Quality Assurance Project Plan

Location:

24 and 32 York Street
Rochester, NY 14611
NYSDEC Spill #1901036

Prepared for:

City of Rochester
Division of Environmental Quality
30 Church Street, Room 300B
Rochester, NY, 14614-1278

LaBella Project No. 2220406

May 2022

In addition to the funding from the USEPA, this project will also be funded by the City of Rochester. Though this project has been funded, wholly or in part, by EPA, the contents of this CAP do not necessarily reflect the views and policies of EPA.

A. Project Management Elements

This Quality Assurance Project Plan (QAPP) will be followed during implementation of environmental remediation and sampling related to the cleanup at 24 and 32 York Street, Rochester, New York 14611. This QAPP has been completed in general accordance with *EPA Requirements for Quality Assurance Project Plans* (EPA QA/R-5) dated March 2001.

1. Approval Sheet

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QAPP Completion Date: 5/19/22

City of Rochester Approval Date: 7/7/22

EPA Approval Date: EPA Approval Not Applicable - QAPP Reviewed and Approved by NYSDEC as Appendix 2 of Corrective Action Plan dated 6/9/22

NYSDEC Approval Date: 7/7/22

2. Table of Contents

3. Distribution List

The following distribution list will receive a copy of any subsequent revisions of this QAPP.

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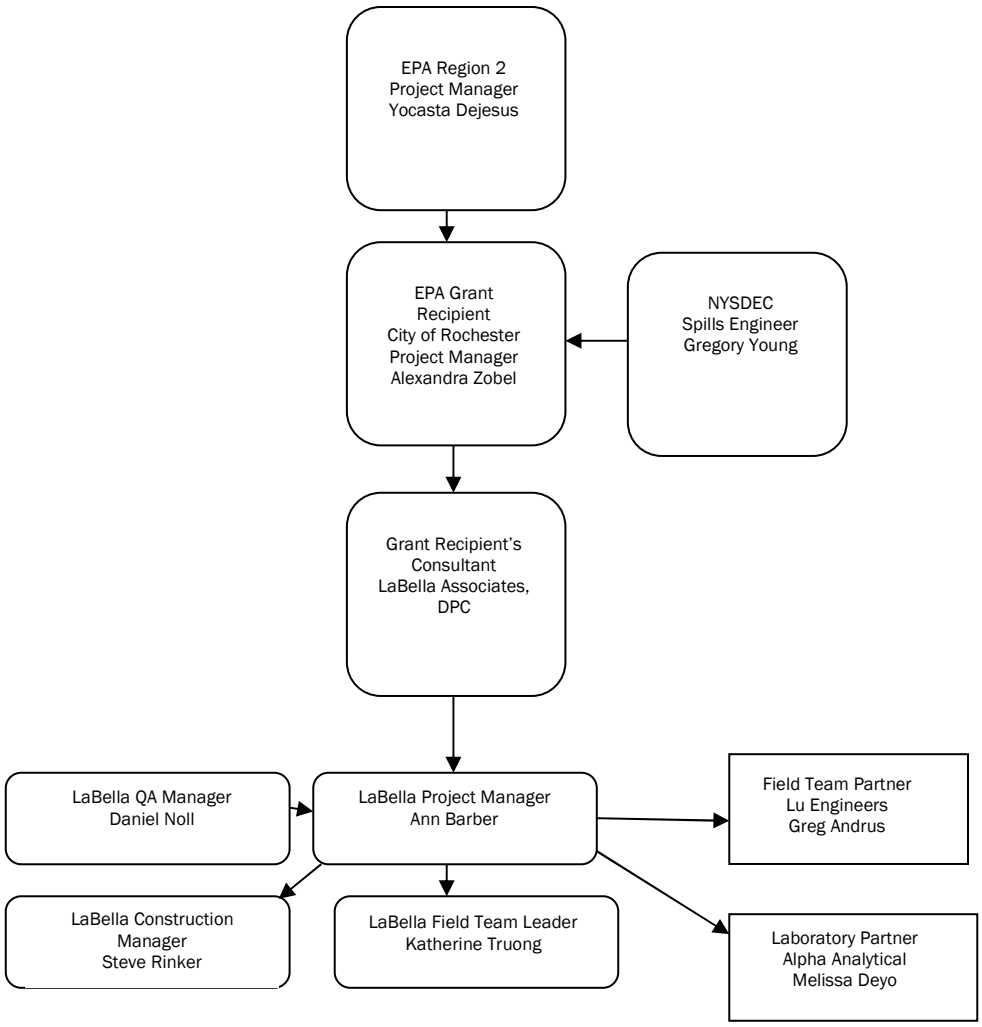
4. Project Organization

The following chart depicts the roles and responsibilities of different organizations involved with this project.



The EPA, NYSDEC and City will oversee the project. LaBella Associates will be providing environmental consulting and remediation services on behalf of the City. The LaBella Project Manager will be the main point of contact for the City and will be responsible for coordinating all project tasks with the project team and making sure the project stays on schedule and budget. The QA Manager will be responsible for reviewing reports for QA/QC purposes. The Field Team Leader will be responsible for all field tasks including sample collection, GPS coordinates, documentation, and community air monitoring. The Construction Manager will be responsible for leading the construction

team including excavation, soil staging, groundwater management, coordination with trucking company and the landfill, well installation, and site security. LaBella has partnered with Lu Engineers. Lu Engineers will be responsible for post-remedial groundwater monitoring. Alpha Analytica is the laboratory that will be used for all sample analyses.

5. Project Definition/ Background

The Site consists of two contiguous parcels located at 24 and 32 York Street in the City of Rochester, Monroe County, New York (Site). As of the date of this report, the Site is owned by the City, and the Monroe County Tax ID numbers for the 24 and 32 York Street parcels are 120.42-2-70 and 120.42-2-71, respectively. The Site parcels are zoned C-2 (Community Center District) which allows a variety of residential and commercial uses, include mixed use. The Site is currently vacant. The former structure including the foundation and footers was demolished by the City in 2020.

Historical uses of the 24 York Street portion of the Site included a blacksmith shop and a wood working shop in at least 1892; a blacksmith shop, wagon shop, and painting and harness shop in at least 1912; an auto repair facility in at least 1924; a gasoline station (with at least eight underground tanks and at least six pump dispensers) from at least 1925 through at least 1954; an auto repair facility and blacksmith shop in at least 1929-30; a blacksmith shop in at least 1935 and 1950; an auto repair facility from at least 1941 to at least 1973; and an auto sales facility in at least 1978, and vacant land and/or a parking lot from about 1981 to the present.

Historical uses of the 32 York Street portion of the Site included residential from at least 1888 to about 1935, a post office from about 1935 to at least 1997, and a church from about 2001 to 2020.

Previous environmental investigations have identified the presence of historic fill material (HFM) and petroleum impacted soil, groundwater and bedrock across approximately 6,500 square feet (sq. ft.) of the Site.

6. Project Task/ Description

This QAPP will be implemented during the remedy which is detailed in the Corrective Action Plan (CAP). Refer to the CAP for specific details regarding the planned cleanup. In summary, the HFM and petroleum impacts will be excavated and disposed of off-Site at a NYS Part 360 permitted landfill. If petroleum impacts are present along the eastern property boundary, excavation will continue onto the eastern adjacent parcel addressed as 42 York Street which is owned by the City. Excavation will continue to bedrock and if evidence of petroleum impacts is present in the top of bedrock, up to 2-feet of bedrock will also be removed and characterized for off-Site disposal. Groundwater that accumulates in the excavation will be pumped into a frac tank, characterized, and disposed of via the public combined sewer system following permit issuance. Following excavation and confirmatory soil sample results that meet 6 NYCRR Part 375 Restricted Residential Use and Protection of Groundwater Soil Cleanup Objectives (SCOs), an oxygen release compound (ORC) will be applied to the excavation to promote further bioremediation. Piping infrastructure will also be installed for potential future ORC application if necessary. The excavation will be backfilled and four (4) bedrock interface wells will be installed for post-remedial groundwater monitoring. Groundwater will be monitored quarterly for one (1) year. In addition, a Soil and Groundwater Management Plan (SGMP) will be prepared for the Site to provide guidance for managing residual petroleum impacts and HFM

if encountered during future subsurface work. Refer to Figure 2 in the CAP for excavation limits and proposed monitoring well locations.

7. Quality Objectives and Criteria

LaBella's Quality Assurance/ Quality Control (QA/QC) Program is an integral part of its approach to environmental investigations and remediation. By maintaining a rigorous QA/QC program, our firm can provide accurate and reliable data. This project-specific QAPP contains procedures which allow for the proper collection and evaluation of data and documents that quality control procedures have been followed during field investigations. This QAPP presents the methodology and measurement procedures used in collecting quality field data. This methodology includes the proper use of equipment, documentation of sample collection, and sample handling procedures. Procedures used in this QAPP are compatible with federal, state, and local regulations, as well as appropriate professional and technical standards. It should be noted that project-specific CAP includes additional project specific details not included in this QAPP. Refer to the CAP and this QAPP when implementing the remedy.

The characteristics of major importance for the assessment of generated data are accuracy, precision, completeness, representativeness, and comparability. Application of these characteristics to specific projects is addressed later in this document. The characteristics are defined below.

- Accuracy is the degree of agreement of a measurement or average of measurements with an accepted reference or "true" value and is a measure of bias in the system.
- Precision is the degree of mutual agreement among individual measurements of a given parameter.
- Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount expected to be obtained under correct normal conditions.
- Representativeness expresses the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Careful choice and use of appropriate methods in the field will ensure that samples are representative. This is relatively easy with water or air samples since these components are homogeneously dispersed. In soil and sediment, contaminants are unlikely to be evenly distributed, and thus it is important for the sampler and analyst to exercise good judgment when removing a sample.
- Comparability expresses the confidence with which one data set can be compared to another. The data sets may be inter- or intra- laboratory.

Accuracy

Accuracy of a particular analysis is measured by assessing its performance with "known" samples. These "knowns" take the form of EPA standard reference materials, or laboratory prepared solutions of target analytes spiked into a pure water or sample matrix. In the case of gas chromatography (GC) or GC/MS (mass spectrometry) analyses, solutions of surrogate compounds are used. These solutions can be spiked into every sample and are designed to mimic the behavior of target analytes without interfering with their determination.

In each case the recovery of the analyte is measured as a percentage, correcting for analytes known to be present in the original sample if necessary, as in the case of a matrix spike analysis. For EPA supplied known solutions, this recovery is compared to the published data that accompany the

solution.

For the firm's prepared solutions, the recovery is compared to EPA-developed data or the firm's historical data as available. For surrogate compounds, recoveries are compared to EPA CLP acceptable recovery tables.

If recoveries do not meet required criteria, then the analytical data for the batch (or, in the case of surrogate compounds, for the individual sample) are considered potentially inaccurate. The analyst or his supervisor must initiate an investigation of the cause of the problem and take corrective action. This can include recalibration of the instrument, reanalysis of the QC sample, reanalysis of the samples in the batch, or flagging the data as suspect if the problems cannot be resolved. For highly contaminated samples, recovery of the matrix spike may depend on sample homogeneity. As a rule, analyses are not corrected for recovery of matrix spike or surrogate compounds.

Precision

Precision of a particular analysis is measured by assessing its performance with duplicate or replicate samples. Duplicate samples are pairs of samples taken in the field and transported to the laboratory as distinct samples. Their identity as duplicates is typically not known to the laboratory. For most purposes, precision is determined by the analysis of replicate pairs (i.e., two samples prepared at the laboratory from one original sample). Often in replicate analysis the sample chosen for replication does not contain target analytes so that quantitation of precision is impossible. For EPA CLP analyses, replicate pairs of spiked samples, known as matrix spike/matrix spike duplicate samples, are used for precision studies. This has the advantage that two real positive values for a target analyte can be compared.

Precision is calculated in terms of Relative Percent Difference (RPD).

- Where X_1 and X_2 represent the individual values found for the target analyte in the two replicate analyses or in the matrix spike/matrix spike duplicate analyses.
- RPDs must be compared to the method RPD for the analysis. The analyst or his supervisor must investigate the cause of RPDs outside stated acceptance limits. This may include a visual inspection of the sample for non-homogeneity, analysis of check samples, etc. Follow-up action may include sample reanalysis or flagging of the data as suspect if problems cannot be resolved.
- During the data review and validation process, field duplicate RPDs are assessed as a measure of the total variability of both field sampling and laboratory analysis.

Completeness

Completeness for each parameter is calculated as follows:

- The firm's target value for completeness for all parameters is 100%. A completeness value of 95% will be considered acceptable. Incomplete results will be reported to the site managers. In planning the field sample collection, the site manager will plan to collect field duplicates from identified critical areas. This procedure should assure 100% completeness for these areas.

Representativeness

The characteristic of representativeness is not quantifiable. Subjective factors to be taken into account are as follows:

- The degree of homogeneity of a site;
- The degree of homogeneity of a sample taken from one point in a site; and
- The available information on which a sampling plan is based.

To maximize representativeness of results, sampling techniques and sample locations will be carefully chosen so that they provide laboratory samples representative of the site and the specific area. Within the laboratory, precautions are taken to extract from the sample bottle an aliquot representative of the whole sample. This includes premixing the sample and discarding pebbles from soil samples.

Comparability

Comparability of laboratory tests is ensured by utilizing only New York State Department of Health (NYSDOH) Environmental Laboratory Accreditation Program (ELAP)- certified laboratories. This certification is the basis for demonstrating proficiency in testing requirements. Using ELAP certified laboratories will result in consistency amongst analytical data within a specific project and across projects.

8. Special Training/ Certification

All samples collected during this project will be submitted to an Environmental Laboratory Accreditation Program (ELAP) certified laboratory for analysis.

Individuals involved with the remedial work on-Site must be 40-hour OSHA HAZWOPER trained with current 8-hour refresher certification.

All LaBella personnel will be familiar with the CAP and its contents including the QAPP prior to working on the Site. The LaBella Project Manager will review the CAP and QAPP with the field team and construction team in advance to ensure everyone is familiar with the procedures outlined herein.

9. Documents and Records

The QAPP is an appendix to the CAP. If the QAPP is revised for any reason, the entire CAP will be updated with the revision number and date on the title page of the CAP. If a revision to this QAPP is made, the title page and footer will be updated with the revision number and date.

Thorough and accurate documentation on-Site is critical to successful completion of the project. Daily logs are necessary to provide sufficient data and observations to enable participants to reconstruct events that occurred during the project and to refresh the memory of the field personnel if called upon to give testimony during legal proceedings. Daily logs may be kept in a project-specific notebook labelled with the project name/ number and contact information.

The daily log is the responsibility of the field personnel and will include:

- Name of person making entry;
- Start and end time of work;

- Names of team members on-site;
- Changes in required levels of personnel protection:
  - Level of protection originally used;
  - Changes in protection, if required; and
  - Reasons for changes.
- Air monitoring locations, start and end times, and equipment identification numbers;
- Summary of tasks completed;
- Detailed site sketch;
- Summary of samples collected including location, matrix, etc.;
- Field observations and remarks;
- Weather conditions, wind direction, etc.;
- Any deviations from the work plan;
- Initials/ signature of person recording the information.

As with any data logbooks, no pages will be removed for any reason. If corrections are necessary, these must be made by drawing a single line through the original entry (so that the original entry can still be read) and writing the corrected entry alongside. The correction must be initialed and dated. Corrected errors may require a footnote explaining the correction.

Sample documents, forms, or field notebooks are not to be destroyed or thrown away, even if they are illegible or contain inaccuracies that require a replacement document. If an error is made on a document assigned to one individual, that individual may make corrections simply by crossing a line through the error and entering the corrected information. The incorrect information should not be obliterated. Any subsequent error discovered on a document should be corrected by the person who made the entry. All corrections must be initialed and dated. All field notes will be scanned and kept on file electronically.

Photographs will be taken to document the work. Documentation of a photograph is crucial to its validity as a representation of an existing situation. Photographs should be documented with date, location, and description of the photograph. Photographs will be kept on file electronically.

A daily email summary will be provided to the City and NYSDEC at the end of each day which will include a summary of work completed that day, any samples collected, site photographs, and planned work for the following day.

B. Data Generation and Acquisition

1. Sampling Process Design

Samples will be collected in accordance with DER Technical Guidance for Site Investigation and Remediation (DER-10). Per DER-10, the confirmatory samples will be collected at a rate of one (1) sidewall confirmatory sample for every 30 linear feet of excavation perimeter, and one (1) bottom confirmatory sample for every 900-sq.ft. of excavation bottom area. Waste characterization samples will be collected in accordance with the requirements of the landfill accepting the material (Waste Management's High Acres or Mill Seat Landfill) which will be 4 samples for approximately 2,000 tons. Soil samples for reuse of material will be collected at a rate of one (1) sample per 100 cubic

yards (CY). The rationale for soil sample frequency is to comply with regulatory requirements and disposal facility requirements.

Four (4) post-remedial groundwater monitoring wells are planned. With a total planned excavation area of approximately 6,500 square feet, one well will be installed for every 1,625 square feet. The planned density of monitoring wells is sufficient to evaluate post-remedial groundwater conditions and trends. Groundwater samples will be collected quarterly for one (1) year. The rationale for groundwater sample frequency is to generate a dataset that has representative data seasonally and four (4) sample events will be sufficient to monitor any trends in the data.

2. Sampling Methods

Prior to drilling or digging, all drill/ dig sites will be cleared with appropriate utility companies to avoid potential accidents relating to underground utilities. Utility drawings will be reviewed, if available.

Geoprobe ® Advanced Borings:
Soil borings and monitoring wells will be advanced with a Geoprobe direct push sampling system. The use of direct push technology allows for rapid sampling, observation, and characterization of relatively shallow overburden soils. The Geoprobe utilizes a four to five-foot macrocore sampler, with disposable polyethylene sleeves. Soil cores will be retrieved in four or five-foot sections and can be easily cut from the polyethylene sleeves for observation and sampling. The macrocore sampler will be decontaminated between boring locations using an alconox and water solution.

Prior to initiating drilling activities, the Macrocores, drive rods, and pertinent equipment, will be steam cleaned or washed with an alconox and water solution. This cleaning procedure will also be used between each boring. Throughout and after the cleaning processes, direct contact between the equipment and the ground surface will be avoided. Plastic sheeting and/or clean support structures (e.g., pallets, sawhorses) will be used.

Test borings will be advanced with 2-inch (or larger) inside diameter (ID) direct push Macrocore through overburden soils. Drilling fluids, other than potable water will not be allowed without special consideration and agreement from NYSDEC. The use of lubricants is also not allowed unless approved by the NYSDEC representative.

During the drilling, a properly calibrated photoionization detector (PID) will be used to screen soil cores retrieved from the Macrocores.

Direct Push Geoprobe advanced groundwater-monitoring wells typically utilize minimum 1.25-inch threaded flush joint PVC pipe with 0.010-in. slotted screen or pre-packed well screens. PVC piping used for risers and screens will conform to the requirements of ASTM-D 1785 Schedule 40 pipe. All materials used to construct the wells will be NSF/ASTM approved. Solvent PVC glue shall not be used at any time in the construction of the wells. The bottom of the screen shall be sealed with a treated cap or plug. No lead shot or lead wool is to be employed in sealing the bottom of the well or for sealant at any point in the well. Stainless steel wells or pre-packed PVC wells may be used if specified in the work plan and approved by the NYSDEC.

Hollow-Stem Auger Advanced Borings:

The drilling and installation of soil borings and monitoring wells will be performed using a rotary drill rig which will have sufficient capacity to perform 4 1/4-inch inside diameter (ID) hollow-stem auger drilling in the overburden, retrieve Macrocore or split-spoon samples, and perform necessary rock coring using NX, NQ, HQ or core barrel size as specified in the project-specific work plan. The borehole may be reamed up to 5 1/2-inch diameter prior to monitoring well installation as cased hole in the bedrock, or may be left as open bedrock hole, with regulatory concurrence. Equipment sizes and diameters may vary based on project-specific criteria. Any investigative derived waste generated during the advancement of soil borings and monitoring well installations will be containerized and characterized for proper disposal.

Prior to initiating drilling activities, the augers, rods, Macrocore, split spoons, and other pertinent equipment will be steam cleaned or washed with an alconox and water solution. This cleaning procedure will also be used between each boring. Steam cleaning activities will be performed in a designated on-site decontamination area. During and after the cleaning processes, direct contact between the equipment and the ground surface will be avoided. Plastic sheeting and/or clean support structures (e.g., pallets, sawhorses) will be used.

Test borings will be advanced with 4 1/4-inch (ID) hollow stem augers through overburden, and cored with a NX, NQ, HQ or core barrel size as specified in the project-specific work plan sized diamond core barrels in competent rock, driven by truck-, track-, or trailer-mounted drilling equipment. Alternative methods of drilling or equipment may be allowed or requested for project-specific criteria, but must be approved by the NYSDEC. Drilling fluids, other than water from a NYSDEC-approved source, will not be allowed without special consideration and agreement from NYSDEC. The use of lubricants is also not allowed unless approved by the NYSDEC representative. During the drilling, visual screening will be utilized to identify any Non-Aqueous Phase Liquid (NAPL) in the soil cores.

Where bedrock wells are required, test borings shall be advanced into rock with NX, NQ, HR (or similar) coring tools. Only water from an approved source shall be used in rock coring. The consultant shall monitor and record the petrology, core recovery, fractures, rate of advance, and water lost or produced in each test boring. The Rock Quality Determination (RQD) value shall be calculated for each 5-foot core. Each core shall be screened with a PID upon extraction. All core samples shall be retained and stored by the consultant in an approved wooden core box for a period of not less than one year.

The method selected may be percussion or rotary drilling. The method and equipment selected must be capable of penetrating the bedrock at each well location to a depth required by the work plan.

Bedrock well installation will involve construction of a rock socket in the weathered bedrock. The socket will be drilled into the top of rock (typically 1-ft. to 5-ft. into the top of rock) at each bedrock well location to allow a permanent steel casing to be grouted securely in place prior to completion of the well. The purpose for this is to provide a seal at the overburden/bedrock interface and into the upper bedrock surface, to prevent the entrance of overburden water into the bedrock. After the grout and casing have set up for a minimum of 12 hours, the remaining bedrock can be NX (or similar) cored through the steel casing to a depth determined by the project-specific work plan.

Bedrock wells will either be open coreholes in the rock or consist of threaded, flush-joint PVC piping. Construction will vary depending on the project and as such, specific construction of the wells will be

detailed in the project-specific work plan. Bedrock wells which do utilized PVC piping for risers and screens will conform to the requirements of ASTM-D 1785 Schedule 40 pipe. All materials used to construct the wells will be NSF/ASTM approved.

Screen and riser sections shall be joined by flush-threaded coupling to form watertight unions that retain 100% of the strength of the casing. Solvent PVC glue shall not be used at any time in the construction of the wells. The bottom of the screen shall be sealed with a treated cap or plug. No lead shot or lead wool is to be employed in sealing the bottom of the well or for sealant at any point in the well.

Sand Pack, Bentonite Seal, Grout, and Surface Protection:
When utilized, granular backfill will be chemically and texturally clean, inert, siliceous, and of appropriate grain size for the screen slot size and the host environment The sand pack will be installed using a tremie pipe, when possible (i.e., a tremie pipe may not fit into smaller, 2-in. diameter boreholes). When utilized, the well screen and casing will be installed, and the sand pack placed around the screen and casing to a depth extending at least 2-ft.. A pre-packed well screen may be used if pre-approved by the NYSDEC. An artificial sand pack will not be utilized in bedrock wells without screens (i.e., open borehole wells).

A minimum 2-ft. thick seal will be placed directly on top of the sand pack, and care will be taken to avoid bridging. In the event that Site geology does not allow for a 2-ft. seal (e.g., only 1-ft. of space remains between the top of the sand pack and ground surface), the remaining space in the annulus will be filled with bentonite.

Upon completion of the bentonite seal, the well may be grouted with a non-shrinking cement grout (e.g., Volclay^{R}) mix to be placed from the top of the bentonite seal to the ground surface. The cement grout shall consist of a mixture of Portland cement (ASTM C 150) and water, in the proportion of not more than 7 gallons of clean water per bag of cement (1 cubic foot or 94 pounds). Additionally, 3% by weight of bentonite powder may be added.

At all times during the progress of the work, precautions shall be used to prevent tampering with or the entrance of foreign material into the well. Upon completion of the well, a suitable cap shall be installed to prevent material from entering the well. Where permanent wells are to be installed, the well riser shall be protected by a flush mounted road box set into a concrete pad or locking well cap for stick-up wells. A concrete pad, sloped away from the well, shall be constructed around the flush mount road box or stick-up casing at ground level.

Any well that is to be temporarily removed from service or left incomplete due to delay in construction shall be capped with a watertight cap.

Geologic Logging and Sampling:
At each investigative location, borings will be advanced through overburden using either a drill rig and hollow-stem auger or direct push technology (split spoons or Macrocore). Soils will be evaluated for visual and olfactory evidence of impairment (i.e., staining, odors, and elevated PID readings) by a qualified individual. Sampling devices will be decontaminated according to procedures outlined in the Decontamination section of this document. When utilized, split-spoon samplers will be driven into the soil using a minimum 140-pound safety hammer and allowed to free-fall 30-inches, in accordance with ASTM-D 1586-84 specifications. The number of blows required to drive the

sampler each 6-inches of penetration will be recorded. When required, samples will be stored in the appropriate bottleware (refer to Section 10) until analysis or deemed unnecessary.

In the event that maximum design depth of investigation is reached and hydrogeologic conditions are not suitable for well installation, the maximum drilling depth may be revised.

Boulders and bedrock encountered during well installation may be cored by standard diamond-core drilling methods using an NX, NQ, HQ size core barrel or other if specified in the project-specific work plan. All rock cores recovered will be logged by a qualified individual, and stored in labeled wooden core boxes. The cores will be stored by the firm until the project is completed or for at least one year. Drilling logs will be prepared by a qualified individual who will be present during drilling operations. One copy of each field boring and well construction log and groundwater data, will typically be submitted as part of the investigation summary report (e.g., Remedial Investigation Report). The RQD value shall be calculated for each 5-foot section. Information provided in the logs shall include, but not be limited to, the following:

- Date(s), test hole identification, and project identification;
- Name of individual developing the log;
- Name of driller and assistant(s);
- Drill, make and model, auger size;
- Identification of alternative drilling methods used and justification thereof (e.g., rotary drilling with a specific bit type to remove material from within the hollow stem augers);
- Standard penetration test (ASTM D-1586) blow counts;
- Field diagram of each monitoring well installed with the depth to bottom of well/ screen, top of screen, length of riser, depth of steel casing, depths of sand pack, bentonite seal, grout, type of well completion etc.;
- Depth of each change of stratum;
- Identification of the material of which each stratum is composed, according to the USCS system or standard rock nomenclature, as appropriate;
- Depth interval from which each sample was taken, sample identification, and sample time;
- Depth at which hole diameters (bit sizes) change;
- Depth at which groundwater is encountered;
- Drilling fluid and quantity of water lost during drilling;
- Depth or location of any loss of tools or equipment;
- Depths of any fractures, joints, faults, cavities, or weathered zones

<u>Groundwater Sampling:</u>
The groundwater in all new monitoring wells will be allowed to stabilize for at least 1week following development prior to sampling. Water levels will be measured to within 0.01 feet prior to purging and sampling. Sampling of each well will typically be accomplished in one of two ways; active or passive.

Active sampling includes bailing or pumping. Purging will be completed prior to active sampling if specified in the project-specific work plan. During purging, the following will be recorded in field books or groundwater sampling logs:

- date
- purge start time
- weather conditions

- presence of NAPL, if any, and approximate thickness
- pump rate
- pH
- dissolved oxygen
- temperature
- conductivity
- redox
- turbidity
- depth of well
- depth to water
- depth to pump intake
- purge end time
- volume of water purged

During low flow sampling, the water quality parameters including pH, conductivity, temperature, dissolved oxygen, redox, water level drawdown, and turbidity will be recorded at five (5) minute intervals. Samples will be collected after the parameters have stabilized for three (3) consecutive 5-minute intervals to within the specified ranges below:

- Water level drawdown (<0.3')
- Turbidity (+/- 10%, < 50-NTU for Metals Samples)
- pH (+/-0.1)
- Temperature (+/- 3%)
- Specific conductivity (+/- 3%)
- Dissolved Oxygen (+/- 10%)
- Oxidation reduction potential (+/- 10 millivolts)

3. Sample Handling and Custody

Sample Identification:
All containers of samples collected from the project will be identified using the following format on a label or tag fixed to the sample container:

AA-BB-CC-DD-EE

AA:	This set of initials indicates an abbreviation for the Site from which the sample was collected.
BB	This set of initials represents the type of sample (e.g., SB for soil boring, MW for monitoring well, ES for excavation side, EB for excavation bottom, and WC for waste characterization)
CC:	These initials identify the unique sample location number (01, 02, 03, etc.)
DD:	These initials identify the sample start depth (if soil sample)
EE	These initials identify the sample end depth (if soil sample)

Each sample will be labeled, chemically preserved (if required) and sealed immediately after collection. To minimize handling of sample containers, labels will be filled out prior to sample collection when possible. The sample label will be filled out using waterproof ink and will be firmly affixed to the sample containers. The sample label will give the following information:

- Date and time of collection

- Sample identification
- Analysis required
- Project name/number
- Preservation

Sample tags attached to or affixed around the sample container must be used to properly identify all samples collected in the field. The sample tags are to be placed on the bottles so as not to obscure any QC lot numbers on the bottles; sample information must be printed in a legible manner using waterproof ink. Field identification must be sufficient to enable cross-reference with the logbook. For chain-of-custody purposes, all QC samples are subject to exactly the same custodial procedures and documentation as "real" samples.

Chain of Custody:

This section describes standard operating procedures for sample identification and chain-of-custody to be utilized for all field activities. The purpose of these procedures is to ensure that the quality of the samples is maintained during their collection, transportation, and storage through analysis. All chain-of-custody requirements comply with standard operating procedures indicated in USEPA sample handling protocol.

Sample identification documents must be carefully prepared so that sample identification and chain-of-custody can be maintained and sample disposition controlled. Sample identification documents include:

- Field notebooks;
- Sample label; and
- Chain-of-custody records.

The primary objective of the chain-of-custody procedures is to provide an accurate written or computerized record that can be used to trace the possession and handling of a sample from collection to completion of all required analyses. A sample is in custody if it is:

- In someone's physical possession;
- In someone's view;
- Locked up; or
- Kept in a secured area that is restricted to authorized personnel.

As few persons as possible should handle samples. Sample bottles will be obtained pre-cleaned from the laboratory. Sample containers should only be opened immediately prior to sample collection. The sample collector is personally responsible for the care and custody of samples collected until they are transferred to another person or dispatched properly under chain-of-custody rules. The sample collector will record sample data in the field notebook and/or field logs.

The chain-of-custody record must be fully completed in duplicate, using black carbon paper where possible, by the field technician who has been designated by the project manager as responsible for sample shipment to the appropriate laboratory for analysis. In addition, if samples are known to require rapid turnaround in the laboratory because of project time constraints or analytical concerns (e.g., extraction time or sample retention period limitations, etc.), the person completing the chain-of-custody record should note these constraints on the chain of custody.

Transfer of Custody and Shipment:
The coolers in which the samples are packed must be accompanied by a chain-of-custody record. When transferring samples, the individuals relinquishing and receiving them must sign, date, and note the time on the chain-of-custody record. This record documents sample custody transfer.
Shipping containers must be sealed with custody seals for shipment to the laboratory. The method of shipment, name of courier, and other pertinent information are entered on the chain-of-custody.
All shipments must be accompanied by the chain-of-custody record identifying their contents. The original record accompanies the shipment. The other copies are distributed appropriately to the site manager.

Custody Seals:
Custody seals are preprinted adhesive-backed seals. Sample shipping containers (coolers, cardboard boxes, etc., as appropriate) are sealed in as many places as necessary to ensure security. Seals must be signed and dated before shipment. On receipt at the laboratory, the custodian must check (and certify, by completing the package receipt log and LABMIS entries) that seals on boxes and bottles are intact. Strapping tape should be placed over the seals to ensure that seals are not accidentally broken during shipment.

Sample Packaging:
Samples must be packaged carefully to avoid breakage or contamination and must be shipped to the laboratory at proper temperatures. The following sample packaging requirements will be followed:

- Sample bottle lids must never be mixed. All sample lids must stay with the original containers.
- The label should not cover any bottle preparation QC lot numbers.
- All sample bottles are placed in a plastic bag and/or individual bubble wrap sleeves to minimize the potential for cross-contamination and breaking.
- Shipping coolers must be partially filled with packing materials and ice when required, to prevent the bottles from moving during shipment.
- The sample bottles must be placed in the cooler in such a way as to ensure that they do not directly come in contact with other samples. Ice will be added to the cooler to ensure that the samples reach the laboratory at temperatures no greater than 4°C.
- Any remaining space in the cooler should be filled with inert packing material. Under no circumstances should material such as sawdust, sand, etc., be used.
- A chain of custody record must be placed in a plastic bag inside the cooler. Custody seals must be affixed to the sample cooler.

Sample Shipment:
Shipping containers are to be custody-sealed for shipment as appropriate. The container custody seal will consist of tape wrapped around the package and custody seals affixed in such a way that access to the container can be gained only by cutting the filament tape and breaking the seal. Chain of custody seals shall be placed on the container, signed, and dated prior to taping the container to ensure the chain of custody seals will not be destroyed during shipment. In addition, the coolers must also be labeled and placarded in accordance with DOT regulations if shipping medium and high

hazard samples.

Field personnel will make arrangements for transportation of samples to the lab. The lab must be notified as early as possible regarding samples intended for Saturday delivery. The transportation and handling of samples must be accomplished in a manner that not only protects the integrity of the sample, but also prevents any detrimental effects due to the possible hazardous nature of samples. Regulations for packaging, marking, labeling, and shipping hazardous materials are promulgated by the United States DOT in the Code of Federal Regulation, 49 CFR 171 through 177. All samples will be delivered to the laboratory and analyzed within the holding times specified by the analytical method for that particular analyte.

All chain-of-custody requirements must comply with standard operating procedures in the USEPA sample handling protocol.

4. Analytical Methods

Analytical methods that will be performed as part of this project are as follows:

Waste Characterization (Soil):

- Toxicity Leachate Characteristics Procedure (TCLP) VOCs using USEPA Method 8260/1311;
- TCLP SVOCs using USEPA Method 8270/1311;
- TCLP Metals using USEPA Method 6010/7470;
- Polychlorinated Biphenyls (PCBs) using USEPA Method 8082;
- Reactivity using USEPA Method 7.3;
- Ignitability using USEPA Method 1030; and,
- pH using USEPA Method 9045

Waste Characterization Water):

- PPL VOCs (EPA 624)
- PCBs (EPA 608)
- PPL Metals & mercury (EPA 200.7/245.1)
- PPL acids/ base/ neutrals, including PAHs (USEPA 625)
- Pesticides (EPA 608)

Confirmatory/ Documentation Soil Samples and Post-Remedial Groundwater Samples:

- NYSDEC Part 375 and CP-51 List VOCs using USEPA Method 8260; and,
- NYSDEC Part 375 and CP-51 List SVOCs using USEPA Method 8270.

Confirmatory soil samples will be analyzed on a 3-day turnaround time. All other samples will be analyzed on a standard turnaround (5-10 business days) unless a need to expedite samples is identified during the project. The laboratory and the data validator will determine if any failures have occurred in the analytical system and the laboratory will correct such errors. Alpha Analytical's

Standard Operating Procedures (SOPs) are included as Appendix 1 to this QAPP. Refer to the SOPs in Appendix 1 for a summary of the analytical procedures.

5. Quality Control

LaBella will be responsible for quality control during sample collection.

Duplicate samples are collected to check the consistency of sampling and analysis procedures. The following types of duplicates will be collected.

- **Blind duplicate** samples consist of a set of two samples collected independently at a sampling location during a single sampling event. Blind duplicates are designed to assess the consistency of the overall sampling and analytical system. Blind duplicate samples should not be distinguishable by the person performing the analysis.
- **Matrix Spike and Matrix Spike Duplicates (MS/MSDs)** consist of a set of three samples collected independently at a sampling location during a single sampling event. These samples are for laboratory quality control checks.

Various types of blanks are used to check the cleanliness of field handling methods. A trip blank will be collected for each groundwater sampling event and analyzed in the laboratory as samples. Their purpose is to assess the sampling and transport procedures as possible sources of sample contamination.

- **Trip Blanks** are similar to routine field blanks with the exception that they are **not** exposed to field conditions. Their analytical results give the overall level of contamination from everything except ambient field conditions. For the RI/FS, one trip blank will be collected with every shipment of water samples for VOC analysis. Each trip blank will be prepared by filling a 40-ml vial with deionized water prior to the sampling trip, transported to the site, handled like a sample, and returned to the laboratory for analysis without being opened in the field. Trip blanks may be provided by the laboratory, shipped with the bottleware, and kept with the sampling containers until analysis.

LaBella will collect a blind duplicate, matrix spike and matrix spike duplicate (MS/MSD) for confirmatory/ documentation soil samples and post-remedial groundwater samples at a rate of 1 per 20 samples. A trip blank will be included for each groundwater sampling event. QC samples will not be collected for waste characterization samples.

Alpha Analytical will be responsible for quality control during the analytical procedures. Each analytical method has varying procedures. Refer to the SOPs for QC criteria.

6. Instrument/ Equipment Testing, Inspection and Maintenance

All instruments and equipment used during sampling and analysis will be operated, calibrated, and maintained according to the manufacturer's guidelines and recommendations as well as criteria set forth in the applicable analytical methodology references. Equipment to be used on site for data collection purposes will include the following:

- PID – MiniRae 3000
- Dust Monitor - TSI 8530 Dust Trak II
- Horiba Multi-Parameter Water Quality Meter
- YSI Pro 20 Water Quality Meter
- Pump - QED MP50 Controller and QED Sample Pro MicroPurge Bladder Pump
- Water Level Meter – Heron Oil/Water Level Meter

Refer to Appendix 2 for product manuals for each piece of equipment. Equipment will be rented from Eco Rental Solutions in Rochester, NY. Eco Rental Solutions will be responsible for testing, inspection and maintenance of their equipment in accordance with the manufacturer's guidelines provided in the product manuals included in Appendix 2. If equipment appears to not be functioning properly on-Site, it will be returned to Eco Rental Solutions for inspection and repair as needed.

Alpha Analytical will be responsible for testing, inspection, and maintenance of laboratory equipment. Refer to Appendix 1 for SOPs regarding laboratory equipment.

7. Instrument/ Equipment Calibration and Frequency

All instruments and equipment used during sampling and analysis will be operated, calibrated, and maintained according to the manufacturer's guidelines and recommendations as well as criteria set forth in the applicable analytical methodology references. Operation, calibration, and maintenance will be performed by personnel properly trained in these procedures. PIDs will be calibrated daily on-Site by LaBella using isobutylene. All other equipment will be calibrated by Eco Rental Solutions prior to use at this Site. Refer to the product manuals in Appendix 2 for calibration procedures.

Alpha Analytical will be responsible for calibration of laboratory equipment. Refer to Appendix 1 for SOPs regarding laboratory equipment.

8. Inspection/ Acceptance of Supplies and Consumables

Consumables to be used on-Site for data collection purposes include the following:

- Gloves
- Deionized water
- Isobutylene (for PID calibration)
- Sample bottleware

Gloves and isobutylene will be obtained from Eco Rental Solutions. Deionized water and sample bottleware will be obtained from Alpha Analytical. All consumables will be unused prior to being brought to the Site and will be obtained specifically for this project. Sample bottleware and deionized water will not be opened until immediately prior to use. If any consumables appear to be opened prior to being brought on-Site, they will not be accepted for use.

9. Non-direct Measurements

No non-direct measurements are expected to be required for implementation of the CAP.

10. Data Management

Field notes will be recorded in accordance with Section B.3 of this QAPP. Handwritten notes will be kept on file at LaBella's office. Any handwritten notes that are typed electronically will be checked by the project manager and QA manager to ensure no errors occur.

Analytical data obtained from the laboratory will be provided to LaBella via email and LaBella will save files electronically. Data tables will be generated from Alpha Analytical's website which will automatically tabulate data in Microsoft Excel and compare to applicable regulatory criteria. This method is time efficient and also prevents errors in transposing lab data from PDFs to tables. All auto-generated tables will be reviewed for accuracy by the project manager and QA manager.

GPS data will be collected using an Arrow Gold GPS which saves data to a cloud-based software as data is collected. This means that even if the GPS unit was damaged or misplaced, the data would be accessible via computer. Data collected on the GPS will be inputted into ArcMap.

All electronic files will be stored on LaBella's secure servers which only LaBella employees have access to and are backed up daily. Files will be shared with the City, NYSDEC and EPA via secure FTP site or via email.

C. Data Validation and Usability

1. Data Review, Verification, and Validation

A Data Usability Summary Report (DUSR) will be generated for confirmatory/ documentation samples and post-remedial groundwater samples in accordance with NYSDEC requirements. The DUSR provides a thorough evaluation of analytical data with the primary objective to determine whether or not the data, as presented, meets the site/project specific criteria for data quality and data use. The development of the DUSR must be carried out by an experienced environmental scientist who is fully capable of conducting a full data validation. The DUSR is developed from an ASP Category B Data Deliverable. The DUSR is developed by reviewing and evaluating the analytical data package.

2. Verification and Validation Methods

In order for the DUSR to be acceptable, during the course of this review the following questions applicable to the analysis being reviewed must be answered in the affirmative.

1. Is the data package complete as defined under the requirements for the most current DEC ASP Category B or USEPA CLP data deliverables?
2. Have all holding times been met?
3. Do all the QC data; blanks, instrument tunings, calibration standards, calibration verifications, surrogate recoveries, spike recoveries, replicate analyses, laboratory controls and sample data fall within the protocol required limits and specifications?
4. Have all of the data been generated using established and agreed upon analytical protocols?
5. Does an evaluation of the raw data confirm the results provided in the data summary sheets and quality control verification forms?
6. Have the correct data qualifiers been used and are they consistent with the most current DEC ASP?
7. Have any quality control (QC) exceedances been specifically noted in the DUSR and have the corresponding QC summary sheets from the data package been attached to the DUSR?

Once the data package has been reviewed and the above questions asked and answered the DUSR proceeds to describe the samples and the analytical parameters, including data deficiencies, analytical protocol deviations and quality control problems are identified and their effect on the data is discussed.

3. Reconciliation with User Requirements

Analytical data will be compared to applicable regulatory criteria consistent with anticipated future Site use as detailed in the CAP. Data collected from the Site will be used for several different purposes and for different data users. LaBella will work with other data users to ensure the data meets the users' requirements or if additional data may be required.

The data user for waste characterization data is the landfill accepting the material. If the waste characterization data does not meet the disposal facility requirements, waste may need to be further segregated and resampled to provided additional data.

The users for confirmatory/ documentation samples and post-remedial groundwater sampling are the City, EPA, and NYSDEC. If confirmatory/ documentation samples do not meet the expected criteria (6 NYCRR Part 375 Restricted Residential Use SCOs), LaBella will notify the data users and expand the excavation to remove additional contamination. If groundwater samples do not meet the expected criteria (6 NYCRR Part 703 Groundwater Quality Standards), LaBella will notify the data users and determine if additional sampling is warranted.

APPENDIX 1

Alpha Analytical Standard Operating Procedures (SOPs)

Metals by Inductively Coupled Plasma EPA 200.7

Reference:

EPA 200.7, Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes. Code of Federal Regulations 40, Part 141 and Part 136, Revision 4.4, May 1994.

EPA 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma – Atomic Emission Spectrometry. Revision 4.4 EMMC Version.

SM 2340B, Hardness by Calculation, Standard Methods for the Examination of Water and Wastewater, APHAAWWA-WPCF, 21st Edition, 1997.

1. Scope and Application

Matrices: Wastewater, Water, Solids

Definitions: Refer to Alpha Analytical Quality Manual.

Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines metals and some nonmetals in solution. This method is a consolidation of existing methods for water, wastewater, and solid wastes. The parameters listed in this method are regulated under the Safe Drinking Water Action (SDWA), Clean Water Act (CWA) and the Resource Conservation and Recovery Act (RCRA).

This is an inductively coupled argon plasma (ICP) method applicable to the determination of the parameters listed above in drinking water, source water and raw water as provided under 40 CFR Part 141.23, municipal and industrial discharges as provided under 40 CFR Part 136.1. Separate SOPs for the digestion of the sample designate the appropriate acids, matrix and quality control samples for the sample matrix and parameters of interest. The parameter list is extended to add metals commonly requested by clients for water samples such as groundwater, surface water and process waters and for solid waste samples such as soil, sludge and other acid digestible materials.

For the determination of total recoverable parameters in aqueous and solid samples a digestion is required prior to analysis when the parameters are not in solution (e.g., soils, sludges, sediments and aqueous samples that may contain particulate and suspended solids). Aqueous samples containing suspended or particulate material ≥1% (w/v) must be digested as a solid type sample.

For drinking water and soluble metals analysis, the method determines certain metal and metalloid contaminants. Samples are analyzed after acid digestion. However, in the determination of some primary drinking water metal contaminants, preconcentration of the sample may be required before analysis in order to meet drinking water acceptance performance criteria.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Metals Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the ICP and in the interpretation of ICP data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability

Parameter	CAS	Parameter	CAS
Aluminum (Al)	7429-90-5	Manganese (Mn)	7439-96-5
Antimony (Sb)	7440-36-0	Molybdenum (Mo)	7439-98-7
Arsenic (As)	7440-38-2	Nickel (Ni)	7440-02-0
Barium (Ba)	7440-39-3	Phosphorus (P)	7723-14-0
Beryllium (Be)	7440-41-7	Potassium (K)	7440-09-7
Boron (B)	7440-42-8	Selenium (Se)	7782-49-2
Cadmium (Cd)	7440-43-9	Silica (SiO)	7631-86-9
Calcium (Ca)	7440-70-2	Silver (Ag)	7440-22-4
Chromium (Cr)	7440-47-3	Sodium (Na)	7440-23-5
Cobalt (Co)	7440-48-4	Strontium (Sr)	7440-24-6
Copper (Cu)	7440-50-8	Thallium (Tl)	7440-28-0
Iron (Fe)	7439-89-6	Tin (Sn)	7440-31-5
Lead (Pb)	7439-92-1	Titanium (Ti)	7440-32-6
Lithium (Li)	7439-93-2	Vanadium (V)	7440-62-2
Magnesium (Mg)	7439-95-4	Zinc (Zn)	7440-66-6

2. Summary of Method

The analysis described in this method involves multiple parameter determinations by ICP-AES using simultaneous instruments. The instruments measure characteristic atomic-line emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Parameter specific emission spectra are produced by radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the line spectra are monitored at specific wavelengths by a photosensitive device. Photocurrents from the photosensitive device are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of the parameters. Background measurement is adjacent to the parameter wavelength during analysis.

Various sample processing techniques must be considered and addressed appropriately for the parameters of interest including: direct analysis (Section 10.3), total parameters using sample digestion techniques found in other standard operating procedures (SOPs) and control of interferences (Section 4.0). Sample processing includes the accurate weighing or measuring of a sample aliquot of a well mixed, homogeneous aqueous or solid sample.

For the determination of dissolved parameters (soluble metals) a filtered aqueous sample aliquot is analyzed by direct analysis for total recoverable parameters. When the sample turbidity is < 1 NTU in drinking water samples, analyze the samples by the direct analysis for total recoverable parameters. Direct analysis of the sample is by using the appropriate addition of nitric acid, diluting if necessary to a predetermined volume, mixing and analyzing.

For total recoverable parameters of a solid or an aqueous sample containing undissolved material, solubilize the parameters first by gentle refluxing with nitric acid. After cooling, the sample is made up to volume, mixed and analyzed. If solids are present after digestion, the sample is either filtered, centrifuged or allowed to settle overnight before analysis. More rigorous digestion techniques such as the technique for total parameters, use sulfuric, perchloric or other acids in combination to breakdown organic or other metal complexes.

This SOP includes the manual calculations for Total Hardness and Calcium Hardness according to SM 2340B

2.1 Method Modifications from Reference

The laboratory reports the % solids at 105°C and not at the 200.7 recommended temperature of 60°C. The percent solids are determined from a separate portion (>20 g) of the sample that is dried to constant weight at 103-105°C. If the data user, program, or laboratory requires that the reported percent solids be determined by drying at 60°C or other temperature, the exception is noted on a nonconformance report and included in the case narrative of the report.

Method 200.7 presents tables listing recoveries for a variety of matrices. These tables are compared to in-house control limits to verify method performance. Routine testing must meet in-house control limits.

Reports Sr wavelengths differ from the reference method. Alpha reports Sr at a wavelength of 421.5

3. Reporting Limits

Detection limits and linear ranges for the parameters will vary with the wavelength selected, the spectrometer, and the matrices. The laboratory follows the procedure found in 40CFR Part 136 to determine the MDL on an annual basis. The method detection limits determined by the laboratory are on file for review.

Table 1 provides estimated instrument detection limits for the listed wavelengths from the reference method and the reported detection limits for the aqueous sample matrix, instrumentation, and selected operating conditions. The reported detection limit for solid samples is calculated from the sample weight and final volume digested.

The reported detection limit is above the laboratory calculated MDL and checked daily by analyzing a standard near the reporting limit concentration. The MDL for a specific solid, wastewater or water matrix may differ from those listed, depending on the nature of the interferences in the sample matrix.

4. Interferences

4.1 Instrumental

4.1.1 Spectral Interferences

Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration parameters, overlap of a spectral line from another parameter, or unresolved overlap of molecular band spectra.

Background emission and stray light are compensated for by subtracting the background emission determined by measurement(s) adjacent to the parameter wavelength peak. Spectral scans of samples or single parameter solutions in the parameter regions may indicate when alternate wavelengths are desirable because of severe spectral interference. The scans determine whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by the measured emission on one side or the other. The determination of the location(s) selected for the measurement of background intensity is by the complexity of the spectrum adjacent to the wavelength peak. The location(s) used for routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak.

Spectral overlaps are avoided by using an alternate wavelength or are compensated for by equations that correct for interelement contributions, which involves measuring the interfering parameters. Some potential on-line spectral interferences observed for the recommended wavelengths are given in Table 2. When operative and uncorrected, these interferences will produce false-positive determinations. The interferences listed are only those that occur between method parameters. Only interferences of a direct overlap nature that were observed with a single instrument having a working resolution of 0.035 nm are listed. More extensive information on interferent effects at various wavelengths and resolutions is available in Boumans' Tables.

Users may apply interelement correction factors determined on their instruments within tested concentration ranges to compensate (off-line or on-line) for the effects of interfering parameters. The analysis of spectral interference check standards (ICS) verifies the accuracy of the interelement corrections. Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating plus the entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line appears must be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Users must not forget that some samples may contain uncommon parameters that could contribute spectral interferences.

See Section 9.7.1 for required spectral interference test criteria. If interelement corrections are not used, document the information and refer to the reference method (Section 4.1.5 Method 200.7) for information. On-going spectral interference check standards must be analyzed to verify the absence of interelement spectral interference or a computer software routine must be employed for comparing the determinative data to limits files for notifying the analyst when an interfering parameter is detected in the sample at a concentration that will produce either an apparent false positive concentration, greater than the parameter IDL, or false negative parameter concentration, less than the 99% lower control limit of the calibration blank. When the interference accounts for 10% or more of the parameter concentration, either an alternate wavelength free of interference or another approved test procedure must be used to complete the analysis.

4.1.2 Physical Interferences

Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by such means as a high-solids nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element.

Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This problem can be controlled by a high-solids nebulizer, wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Also, it has been reported that better control of the argon flow rates, especially for the nebulizer, improves instrument stability and precision; this is accomplished with the use of mass flow controllers.

ICP-AES determines dissolved parameters in aqueous samples after suitable filtration and acid preservation. To reduce potential interferences, dissolved solids must be < 0.2% (w/v).

4.1.3 Chemical Interferences

Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. Normally, these effects are not significant with the ICP-AES technique. If observed, they can be minimized by careful selection of operating conditions (such as incident power and observation height), by buffering of the sample, by matrix matching, and by standard-addition procedures. Chemical interferences are highly dependent on matrix type and the specific parameter.

4.1.4 Memory Interferences

Memory interferences result when parameters in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer, and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the parameter and is minimized by flushing the system with a rinse blank between samples. If memory interference is suspected, the sample must be re-analyzed after a long rinse period, using either a peristaltic pump, or an appropriate internal standard element.

Method interferences are the result of contaminants in acids, reagents, glassware, and other sample processing hardware. Running laboratory reagent blanks as described in Section 10.3 and 9.1 demonstrates the system is free of contamination. The analytical system must be free from contamination under the conditions of the analysis.

4.2 Parameters

4.2.1 When determining **boron** and **silica** in aqueous samples, only plastic, PTFE or quartz labware must be used from time of sample collection to completion of analysis. For accurate determination of boron in solid samples only quartz or PTFE beakers must be used during acid extraction with immediate transfer of an extract aliquot to a plastic centrifuge tube following dilution of the extract to volume. When possible, borosilicate glass must be avoided to prevent contamination of these parameters.

4.2.2 **Silver** is only slightly soluble in the presence of chloride unless there is a sufficient chloride concentration to form the soluble chloride complex. Therefore, low recoveries of silver may occur in samples, spiked sample matrices and spiked blanks or as a dissolved parameter or analyzed by "direct analysis". For this reason, samples are digested using the total recoverable mixed acid digestion before the determination of silver. For the analysis of wastewater samples containing higher concentrations of silver, succeeding smaller volume, well mixed aliquots must be prepared until the analysis solution contains <0.1 mg/L silver. The extraction of solid samples containing concentrations of silver >50 mg/kg must be treated in a similar manner.

4.2.3 The digestion of **tin** from solid samples must be prepared using aliquots of <1 g when expected sample concentrations exceed 1%.

4.2.4 The total recoverable sample digestion procedures solubilize and hold in solution only minimal concentrations of **barium** in the presence of free sulfate. For the analysis of barium in samples having varying and unknown concentrations of sulfate, analysis must be completed as soon as possible after sample preparation.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard.

From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

Specifically, concentrated nitric and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to skin and mucus membranes. Always use these reagents in a fume hood, and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing when working with these reagents.

The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples must be done in a fume hood.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The inductively coupled plasma is only viewed with proper eye protection from the ultraviolet emissions.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Prior to the collection of an aqueous sample, consideration must be taken as to what type of analysis is specified, (i.e. dissolved or total), so that the appropriate pretreatment or preservation steps may be taken.

The laboratory routine practice is to collect a single 1L plastic container for aqueous samples and 250 mL wide mouth jar for soil samples.

6.2 Sample Preservation

6.2.1 Solid samples require no chemical preservation before analysis.

6.2.2 For the determination of <u>total parameters</u>, the sample is preserved with 1:1 HNO_3 to pH < 2, not to exceed 10 ml of 1:1 HNO_3 per liter of sample. Usually 2 ml of 1:1 HNO_3 is sufficient for the preservation of drinking waters Samples must be pH <2 for at least 24 hours prior to digestion if not preserved at the time of collection.

6.2.3 For the determination of <u>dissolved parameters</u>, an aliquot of the unpreserved sample is filtered through a 0.45μm membrane filter within 24 hours of the collection time, and prior to sample digestion and analysis. The sample is filtered through a nitric acid presoaked glass filtration apparatus. Once a sufficient volume of the filtrate is obtained, the filtrate is preserved with 1:1 Nitric acid (HNO_3) to a pH < 2. Soluble samples must be held at pH < 2 for at least 24 hours prior to digestion if not preserved at the time of filtration. A separate SOP lists the sample preparation technique.

6.2.4 For the determination of <u>total recoverable parameters</u> in aqueous samples, samples are not filtered, but acidified with 1:1 Nitric acid to a pH < 2. Usually 3 mL of 1:1 Nitric acid per liter of sample is sufficient for most drinking water samples. Samples must be pH <2 for at least 24 hours prior to digestion if not preserved at the time of collection.

6.3 Sample Shipping

No specific requirement.

6.4 Sample Handling

6.4.1 Samples require ice or refrigeration from the time of collection until analysis. Cool and maintain the sample temperature between 2 and 6 °C from time of sample receipt until analysis.

6.4.2 Samples are digested and analyzed within 180 days of collection. Sample digestates are maintained at room temperature.

6.4.3 Refer to the Project Information Form (PIF) for client specific sample handling, preservation and collection criteria.

7. Equipment and Supplies

7.1 Inductively coupled argon plasma emission spectrometer:

7.1.1 Computer-controlled emission spectrometer with background correction.

7.1.2 Radio-frequency generator compliant with FCC regulations.

7.1.3 Optional mass flow controller for argon nebulizer gas supply.

7.1.4 Peristaltic pump.

7.1.5 Autosampler.

7.1.6 Argon gas supply - high purity.

7.2 Volumetric pipets: 0.5 mL, 1 mL, 5 mL, 10 mL Class A pipets

7.3 Volumetric flasks: 10 mL, 50 mL, 100 mL, Class A with ground glass stoppers

7.4 Autopipetter: Eppendorf, calibrated.

8. Reagents and Standards

8.1 Reagent water: Reagent water in the metals laboratory is water from the RO water system, passed through a column of Milli-Q deionized water system in the laboratory.

8.2 Nitric acid: Concentrated, ACS grade quality and Trace Metals grade.

8.3 10% (v/v) Nitric acid: 100mL concentrated nitric acid diluted to 1 liter with reagent water.

8.4 5% (v/v) Nitric acid: 50 mL concentrated nitric acid diluted to 1 liter with reagent water.

8.5 1:1 Nitric acid: 500 mL concentrated nitric acid diluted to 1 liter with reagent water.

8.6 Hydrochloric acid: Concentrated, ACS grade quality and Trace Metals grade.

8.7 Stock standard solutions: Certified stock standard solutions in nitric acid. The certification includes the concentration, uncertainty and traceability to NIST if available. Stock standards include calibration standards, calibration verification, laboratory controls and spiking solutions.

Select the certified stock standards containing single parameters or multiple parameters of interest. Record the concentration of the certified stock standards, lot number, supplier,

standard name, catalog number, expiration date, date received and receiver's initials in the standards logbook. Record the number of containers prepared and the identifier for the stock standard. Stock standards are initialed and dated upon receipt and certificates are kept in a binder.

Standards are stored in plastic bottles at room temperature.

Standards must be replaced every twelve months, or sooner if comparison with check standards indicates a problem.

8.8 Stock Standard: The stock standards are prepared at concentrations such that the aqueous standards bracket the working range of the analytical system. Prepare the standards using a Class A volumetric pipet, or autopipetter, to transfer the standard (mg/L) into a volumetric flask containing reagent water (mL). Add the appropriate amount of acid to ensure consistent standard and sample acid concentration.

The manufacturer (Inorganic Ventures, Inc.) determines the expiration date of the stock standards. Store stock standards at room temperature.

Other standards may be purchased and prepared. The specific instructions for standard preparation not listed in this SOP are detailed in the standard logbook at the time of preparation.

8.8.1 ICP Calibration Stock #1 (IV-7): Al, Ba, B, Ag, Na at 100 µg/mL; K at 1000 µg/mL; Si at 50 µg/mL

8.8.2 ICP Calibration Stock #2 (IV-19): Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, Se, Tl, Ti, V, Zn at 100 µg/mL

8.8.3 ICP Cal Verification #1 (IQC-007): Al, Ba, B, Ag, Na at 100 µg/mL; K at 1000 µg/mL; Si at 50 µg/mL

8.8.4 ICP Cal Verification #2(IQC-019): Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, Se, Tl, Ti, V, Zn at 100 µg/mL

8.8.5 ICP Ag Standard: Silver standard at 1000 µg/mL

8.8.6 ICP Al Standard: Aluminum standard at 1000 µg/mL

8.8.7 ICP Ba Standard: Barium standard at 1000 µg/mL

8.8.8 ICP B Standard: Boron standard at 1000 µg/mL

8.8.9 ICP Cd Standard: Cadmium standard at 1000 µg/mL

8.8.10 ICP Fe Standard: Iron standard at 1000 µg/mL

8.8.11 ICP K Standard: Potassium standard at 1000 µg/mL

8.8.12 ICP Li Standard: Lithium standard at 1000 µg/mL

8.8.13 ICP Mg Standard: Magnesium standard at 1000 µg/mL

8.8.14 ICP Mo Standard: Molybdenum standard at 1000 µg/mL

8.8.15 ICP Na Standard: Sodium standard at 1000 µg/mL

8.8.16 ICP P Standard: Phosphorus standard at 1000 µg/mL

8.8.17 ICP Pb Standard: Lead standard at 1000 µg/mL

8.8.18 ICP S Standard: Sulfur standard at 1000 µg/mL

8.8.19 ICP Si Standard: Silicon standard at 1000 µg/mL

8.8.20 ICP Sr Standard: Strontium standard at 1000 µg/mL
8.8.21 ICP Sn Standard: Tin standard at 1000 µg/mL
8.8.22 ICP Ti Standard: Titanium standard at 1000 µg/mL
8.8.23 ICP Se Standard: Selenium standard at 1000 µg/mL
8.8.24 ICP Sb Standard: Antimony standard at 1000 µg/mL
8.8.25 ICP As Standard: Arsenic standard at 1000 µg/mL
8.8.26 ICP Be Standard: Beryllium standard at 1000 µg/mL
8.8.27 ICP Co Standard: Cobalt standard at 1000 µg/mL
8.8.28 ICP Cu Standard: Copper standard at 1000 µg/mL
8.8.29 ICP Cr Standard: Chromium standard at 1000 µg/mL
8.8.30 ICP Mn Standard: Manganese standard at 1000 µg/mL
8.8.31 ICP Ni Standard: Nickel standard at 1000 µg/mL
8.8.32 ICP Tl Standard: Thallium standard at 1000 µg/mL
8.8.33 ICP V Standard: Vanadium standard at 1000 µg/mL
8.8.34 ICP Zn Standard: Zinc standard at 1000 µg/mL
8.8.35 ICP Ca Standard: Calcium standard at 1000 µg/mL

8.9 Working Standards: The working standards are prepared from the stock standards and brought to final volume with 5% nitric acid solution. The working standard solutions expire one month from the date of preparation. The CRI stock solution expires six months from the date of preparation or prior to the manufacturer's expiration date. Working standards are stored at room temperature.

8.9.1 ICP CRI Stock Standard: To a 1L volumetric flask, add 500mL DI water and 50mL concentrated HNO_3. Utilizing the 1000µg/mL individual elemental standards (Section 8.8), and an Eppendorf pipette (Section 7.4), add the following volumes of standards to the 1L volumetric flask. Bring to volume with DI water

0.4mL: Be, Cd

0.8mL: As, Ag, Cr, Se, Sn, Tl, Sr, Bi

1.2mL: Mn

1.6mL: Ba, Zn

1.8mL: Pb

2.0mL: Cu

3.2mL: Ni

4.0mL: B, Co, Mo, Sb, V

8.0mL: Fe

6.0mL: Al, Ca, Mg

40.0mL: Si

200mL: Na, K

8.9.2 Low Level Initial Calibration Verification Standard (LLICV) and the Low Level Continuing Calibration Verification Standard (LLCCV)

These standards are actually a series of standards (typically 3) that are at or below the RL for the respective elements included in the calibration sequence. They are prepared

from the same source as the calibration standards but at the laboratory's discretion may be from a second source from the calibration

8.10 Calibration Standards: The instrument calibration standard must include all elements. Other elements may be added or single element standards may be prepared.

Record the stock standard identifier, expiration date for stock standard, acid supplier, log number, preparation date and preparer's initials in the standards logbook. Note any deviations from the routine preparation.

Calibration standards are stored at room temperature and are prepared weekly.

8.10.1 Instrument Cal Std: 10.0 mL IV-7

10.0 mL IV-19

1.0 mL 1000 mg/L Sr, Sn stock standard

Final Volume: 1000 mL

8.11 Spectral Interference Check Solution

These solutions are prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Analysts are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. Single element interference checks - At a minimum, single element SIC checks must be performed for the following elements: Aluminum 500mg/L; Boron 50mg/L, Barium, 50mg/L, Calcium 500mg/L; Copper 50mg/L; Iron 200mg/L; Magnesium 500mg/L; Manganese 50mg/L; Molybdenum 20mg/L; Sodium 1000mg/L; Nickel 20mg/L; Selenium 20mg/L; Silicon 200mg/L; Tin 20mg/L; Vanadium 20mg/L; Zinc 20mg/L The absolute value of the concentration observed for any unspiked analyte in the single element SIC checks must be less than two times the analytes' LLOQ. The concentration of the SIC checks are suggested, but become the highest concentration allowed in a sample analysis, and cannot be higher than the highest established linear range. Samples with concentrations of elements higher than the SIC check must be diluted until the concentration is less than the SIC check solution. Note that reanalysis of a diluted sample is required even if the high concentration element is not required to be reported for the specific sample, since the function of the SIC check is to evaluate spectral interferences on other elements. The single element SIC checks are performed when the instrument is setup and periodically (at least once every 6 months) thereafter.

Mixed element interference check - The mixed element SIC solution is analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than +/- the LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, or alternatively the LLOQ may be raised to twice the concentration observed in the SIC solution. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions These may be present up to the concentration documented plus the LLOQ. Mixed element SIC solution: Aluminum, 500mg/L; Calcium, 500mg/L; Iron, 200mg/L; Magnesium, 500mg/L interference check standard. The routine preparation of the SIC uses the following multielement stock standards. The manufacturer (Absolute Standards, Inc.) sets the expiration date for the stock standards. Store stock standards refrigerated at 4 ± 2°C.

8.11.1 Stock Standards

Interference check standard (ICSA) = Al, Ca, Mg at 5000 µg/mL; Fe at 2000 µg/mL.

8.11.2 Working Standards

The expiration date for the working solutions is six months from the date of preparation or prior to the manufacturer's expiration date. Store at room temperature.

Record the stock standard identifier, expiration date for the standard, date received, opened date and receiver's initials in the standards logbook. Also note any deviations from the routine preparation.

8.11.2.1 ICSA: For Trace ICP, bring 25.0 mL Interference check standard (ICSA) to a final volume of 500mL with 5% nitric acid solution.

Final concentration: Al, Ca, Mg at 250 µg/mL and Fe at 100 µg/mL

8.12 Calibration verification (ICV) standard solution: The CV is also referred to as the initial (ICV) and continuing calibration verification (CCV). This standard is from a different lot number or manufacturer than the calibration standards. Prepare the standard using a Class A volumetric pipet (mL), or Autopipette, to transfer the stock standard (mg/L) into a volumetric flask containing reagent water (mL).

Record the stock standard identifier, expiration date for the CV standard, preparation date and preparer's initials in the standards logbook. Record the exact steps for preparing the standard and the identifier for the CV standard.

The manufacturer (Ultra Scientific, Inc.) sets the expiration date for the stock standards. Store stock standards at room temperature.

8.12.1 Initial Cal Verification (ICV):

5 mL IQC-007

5 mL IQC-019

9.5 mL 1000 mg/L Na stock standard

0.5 mL 1000 mg/L Sr stock standard

0.5 mL 1000 mg/L Sn stock standard

5.0 mL 1000 mg/L Si stock standard

Final Volume: 1000 mL

8.12.2 Reporting Limit (RL) Verification Standard (LLICV/LLCCV): The RL standard consists of a series of standards that are analyzed after the initial calibration verification (LLICV) and at the end of each run (LLCCV). Optionally, the LLCCV may be run every 10 samples with the CCV, CCB pair to eliminate the need for excessive reruns when low level instrument stability is questioned. These standards are at or below the RL included in the multi-point calibration sequence. The standards must have a percent recovery of 70-130%. If an element fails the acceptance criteria to establish a specific RL, the RL standard may be re-analyzed. If the element failure continues, then either re-calibrate the instrument and rerun the affected samples or analyze the affected samples on another instrument with a passing RL verification standard for the element(s) of interest.

The following standards are analyzed:
0.0025 mg/L- Be, Cd;
0.005 mg/L- Ag, As;
0.010 mg/L- B, Ba, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Si, Sn, Sr, Ti, Tl, V;
0.050 mg/L- Al, Sb, Fe, Zn, Ca, Mg, K, Na

8.12.3 RL Standard Solutions:

0.0025 ppm: To a 200ml volumetric flask, add 100mL of DI water and 5mL of concentrated HNO3. Using a calibrated pipette, add 0.5mL of the ICAL standard (Section 8.10.1) and bring to volume with DI water. This standard will have the following concentrations of elements: 0.0025 mg/L- Be, Cd.

0.005 ppm: To a 200ml volumetric flask, add 100mL of DI water and 5mL of concentrated HNO3. Using a calibrated pipette, add 1.0mL of the ICAL standard (Section 8.10.1) and bring to volume with DI water. This standard will have the following concentrations of elements: 0.005 mg/L- Ag, As.

0.010 mg/L: To a 200ml volumetric flask, add 100mL of DI water and 5mL of concentrated HNO3. Using a calibrated pipette, add 2.0mL of the ICAL standard (Section 8.10.1) and bring to volume with DI water. This standard will have the following concentrations of elements: 0.010 mg/L- B, Ba, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Si, Sn, Sr, Ti, Tl, V.

0.050 mg/L: To a 200ml volumetric flask, add 100mL of DI water and 5mL of concentrated HNO3. Using a calibrated pipette, add 10.0mL of the ICAL standard (Section 8.10.1), 2mL of the 200mg/L Na and K intermediate standard (section 8.13.3), and bring to volume with DI water. This standard will have the following concentrations of elements: 0.050 mg/L- Al, Sb, Fe, Zn, Ca, Mg, K, Na.

8.12.4 200 mg/L Na and K intermediate standard: To a 100mL volumetric flask, add 40mL DI water and 5mL concentrated HNO_3. Utilizing the 1000µg/mL individual elemental standards (Section 8.8), and an Eppendorf pipette (Section 7.4), add 20 mL of 1000 ug/mL Na and 20 mL of 1000 ug/mL K standards to the 100mL volumetric flask. Bring to volume with DI water.

8.13 QC check sample (QCS): The reference method requires the analysis of a QCS at least quarterly. The CCV sample is a standard run from a second source and meets the same requirements as the QCS. Whenever a new analyst, new equipment or major method change occurs, the CCV is analyzed in replicate (at least three samples) to verify method performance. Record the stock standard identifier, expiration date for the QCS, preparation date and preparer's initials in the standards logbook. Record the number of containers prepared and the identifier for the standard. Store at room temperature.

8.14 Internal Standards:

The internal standard consists of a single element standard (Y) or, a multi-element solution (Ce, CS, and Lu); each internal standard solution covers a range of the spectrum (low, middle, or high wavelengths) and the elements within that range.

100 mg/L Ce

20 mg/L Cs

2.0 mg/L Lu

1.0 mg/L Y

Note: The standard is used to monitor and compensate for instrument fluctuations including but not limited to nebulization efficiency, plasma variations, environmental temperature changes, peristaltic pump pulsations, etc. Therefore, the solution used to start an analysis calibration cannot be added to or changed out during analysis without requiring subsequent full recalibration.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

The instrument and method blanks must be less than the absolute value of reporting detection limit.

Analyze an acid blank each day to demonstrate that interferences from the instrumental system are under control. The acid blank must contain the same acid concentration as the standards and samples. Do not begin analysis, if parameters are found in the instrument blank at or above the reporting limit.

The possibility of memory interferences must be recognized within an analytical run and suitable rinse times must be used to reduce them. The rinse times necessary for a particular parameter must be estimated prior to analysis. This may be achieved by aspirating a standard containing parameters corresponding to either their LDR or a concentration ten times those usually encountered. The aspiration time must be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce parameter signals to within a factor of two of the method detection limit, must be noted. Until the required rinse time is established, this method requires a rinse period of at least 60 seconds between samples and standards unless proven otherwise through experimentation.

Analyze a method blank from the same preparation batch as the samples. If parameters are found in the method blank, at less than the reporting limit, report the data without qualification. If the parameter in the method blank is above the reporting limit, note the information on a non-conformance form to determine corrective action for all samples in the batch. Data is only reported with qualification when the method blank indicates possible contamination in the system.

9.2 Laboratory Control Sample (LCS)

Compare the measured concentration for the digested LCS with in-house performance data. The percent recovery must be within 85-115% for aqueous samples and 80-120% for soil/solid samples. The LCS is prepared with each digestion batch (every 20 samples).

If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected, prior to sample analysis. A nonconformance form is completed.

9.3 Initial Calibration Verification (ICV)

Demonstrate through the analyses of the initial calibration verification (ICV) standard that the operation of the measurement system is in control. The frequency of the analyses is equivalent to 5% of all samples analyzed. % Recovery of the ICV must be within ± 5% of the true value.

9.4 Continuing Calibration Verification (CCV)

Analyze the CCV to verify the measured concentration for each parameter. The concentration must be recovered within 10% for non-potable wastewater, surface water, domestic water analysis and drinking water analysis.

If the concentration for a parameter does not fall within the range in this second test, instrument maintenance must be performed to determine the cause of the failure. A second CCV standard

with all parameters may be analyzed immediately. The second CCV standard must not fail the acceptance criteria for all parameters.

9.5 Matrix Spike

Spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality.

The concentration of the spike should be at one to five times higher than the sample concentration or at the client requested action level and the same solution used for LCS.

Calculate the matrix spike recovery from the true values. (Table 3). The recovery must be within ±20% or the in-house generated limits for each parameter. If any individual percent recovery falls outside the designated range for recovery (R), that parameter has failed the acceptance criteria. A nonconformance report form is completed to ensure client notification and reporting. A post digestion spike may be performed by adding the same concentration standard to the digested sample. The percent recovery for the post digestion spike is calculated to identify possible sources of the interference; the recovery range of 75-125% is applied. The post digestion spike results are narrated on the final report along with the original failed spike recovery.

9.6 Laboratory Duplicate

Analyze a duplicate sample or reagent water spike at a minimum of 10% of the samples. The percent RPD is determined. The laboratory generated limits for RPD must be met or documented as to the reasons for deviation with a case narrative in the report.

9.7 Method-specific Quality Control Samples

9.7.1 Interference Check Standards

Spectra Interference Check Standard A mixed check solution is analyzed once daily (section 8.11). One solution (SIC) has only elevated concentrations of Fe, Al, Ca, Mg to ensure no interferences occur. The concentrations of the analytes of interest must have an absolute value of the LLOQ. This solution is analyzed at the beginning of the first analytical run of the day. The high level interferences are not evaluated for recovery. If the SIC fails take corrective action which may include re-evaluation of the inter-element correction values (IECs) after running single element SIC. The instrument calibration routine must then be performed and confirmed by the ICV/ICB pair and the SIC reanalyzed before proceeding with analysis. Otherwise, the nonconformance issue is raised to the Department Supervisor and/or the QA Department. .

The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for their automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the user must determine and document both the on-line and off-line spectral interference effect from all method parameters and provide for their automatic correction on all analyses. Tests to determine the spectral interference must be done using parameter concentrations that will adequately describe the interference. Normally, 100 mg/L single parameter solutions are sufficient, however, for parameters such as iron that may be found at high concentration a more appropriate test would be to use a concentration near the upper limit of the linear dynamic range

9.7.2 Internal Standards

Internal Standard The internal standards are added prior to the nebulizer and corrects for intensity differences in the instrument response between the standard's and

sample's matrix. They are monitored for any variation in response during the sample analyses and used to ratio the sample response to the internal standard response of the calibration blank. The ratio is applied to compensate for instrument conditions in the plasma or nebulization caused by the matrix. The internal standard is monitored for 50-150% recovery or laboratory generated control ranges difference from the calibration blank IS response to ensure the proper functioning of the internal standard introduction system and matrix interferences. If an injection falls outside of this acceptance range the sample or QC check is rerun once to check for an introduction error.

If a sample continues to fail it's to be run on successive increasing dilutions until the internal standards associated with the elements of interest are within range. If a QC check fails on the single rerun the analysis is stopped, the root cause investigated, corrected and the instrument re-calibrated/verified. The analysis begins again with all samples that were run after the last acceptable CCV/CCB pair.

9.8 Method Sequence

Instrument Blank (IB)

Instrument Calibration Standard(s)

Calibration Verification Standard (ICV) - second source different concentration (5% of true)

Calibration Blank (ICB)

Interference check standard A (ICS)

Low Level Checks: 2.5, 5.0, 10.0, 50.0 ppb

Continuing calibration verification (CCV) (10% of true)

Continuing calibration blank (CCB)

Samples 1 to 10

(Includes method blank, field blank, sample matrix spike, sample duplicate and spiked blank)

CCV

CCB

Samples 1 to 10

CCV

CCB

10. Procedure

10.1 Equipment Set-up

10.1.1 ICP Set-up and Shut-down

Set-up the instrument with proper operating parameters. (Section 10.3) The instrument must be allowed to become thermally stable before beginning the analysis. Thermal stability requires at least 30 minutes of operation before calibration.

Instrument Start-up steps:

Turn on chiller

Clamp peristaltic pump windings

Start computer (if not currently in operation)

Initialize plasma startup

At the end of each operating day the instrument is shut down. Instrument Shut down steps are:

Flush the instrument for one minute with the rinse solution.

Extinguish plasma by either autoshut down at end of sequence or manually

Turn of chiller once shutdown/cooldown process is complete.

Plasma Optimization: The plasma operating conditions need to be optimized prior to use of the instrument. This routine is not required on a daily basis, but when first setting up a new instrument or following a change in operating conditions. Follow the plasma optimization set-up recommended by the instrument manufacturer.

The purpose of plasma optimization is to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array. The use of a mass flow controller to regulate the nebulizer gas flow or source optimization software greatly facilitates the procedure.

The mass flow controller is set to the recorded optimized flow rate per the manufacturer's suggestions. In order to maintain valid spectral interelement correction routines the nebulizer gas flow rate must be the same (< 2% change) from day to day.

Instrument Profile: The Thermo 6500 series ICP performs an automatic profile optimization each time the software is started which requires no operator interaction.

Interelement Spectral Interference Correction: The laboratory must establish and initially verify an interelement spectral interference correction routine to be used during sample analysis. A general description concerning spectral interference and the analytical requirements for background correction are discussed in the section on interferences. Criteria for determining an interelement spectral interference is an apparent positive or negative concentration for the analyte that falls within ± one reporting limit from zero. The upper control limit is the analyte instrument detection limit. Once established, the entire routine must be verified annually. A portion of the correction routine is verified on a daily basis.

Linear Dynamic Range: The upper limit of the linear dynamic range must be established for each wavelength by determining the signal responses from various different concentration standards across the range. One of the standards is near the upper limit of the range. The data, calculations and rationale for the choice of range is documented and kept on file. The upper range limit is an observed signal no more than 10% below the level extrapolated from lower standards. The linear Dynamic Range is evaluated once per year.

New dynamic ranges are determined whenever there is a significant change in instrument response. For those analytes that periodically approach the upper limit, the range is checked every six months.

NOTE: Many of the alkali and alkaline earth metals have non-linear response curves due to ionization and self absorption effects. These curves may be used if the instrument allows; however the effective range must be checked and the second order curve fit must have a correlation coefficient of 0.995 or better. Third order fits are not acceptable. These non-linear response curves must be revalidated and recalculated every six months.

These curves are much more sensitive to changes in operating conditions than the linear lines and must be checked whenever there have been moderate equipment changes.

10.1.1.1 The essential peak quality control acceptance criteria listed below must be met, otherwise the problem must be found and corrected:

Peak position in terms of wavelength: <1

Peak width at half-height: 10 ±10%

Peak intensity: < ½ the intensity since the instrument was last serviced

10.2 Initial Calibration

Assemble and prepare the instrumental system to the same operation conditions as the sample analysis. (Section 10.3)

Calibrate the instrument according to the instrument manufacturer recommended procedures. Calibration is performed when changes to the operating parameters (Section 10.3) are required for improved instrument performance.

Calibration is checked on a daily basis to verify proper operating conditions are maintained by analyzing the calibration standard, calibration verification, interference check standard and low-level control sample.

Prepare the calibration standards. Record the calibration standard identifier, concentration, and analyst initials in the instrument analysis logbook.

At the beginning of daily analysis (every 24 hours) a single calibration standard is analyzed after the calibration blank. Flush the system with the calibration blank before and after calibration. The average intensity of three (3) exposures is used for calibration to reduce random error. The % RSD for the three replicates must be $\leq$ 5%.

Multiple calibration standards may be analyzed where project specific or client requests require bracketing the working range with daily calibration standards. The calibration points for each element should be representative of the working range from low to high. The high point does not replace the established LDR.

The concentration of the daily calibration standards are near the mid-point of the working range. All parameters of interest are analyzed in the daily calibration standard.

The calibration blank results are to agree within three standard deviations of the mean blank value. If not, repeat the analysis two more times and average the result. If the average of the three results are not within three standard deviation of the background mean, terminate the analysis and correct the problem.

10.3 Equipment Operation and Sample Processing

All glassware must be acid rinsed before use. The glassware is rinsed with 50% hydrochloric acid, then rinsed three times with reagent water, then once with 10% nitric acid solution and then rinsed three times with reagent water.

Changes in acquisition parameters, equipment, and conditions require written authorization from management. Demonstration of method performance based on method modifications must be on file before sample analysis.

Calibration is acceptable if average of three individual runs is %RSD $\leq$ 5% for standards and $\leq$ 25% RSD for samples. Blanks are not controlled on the basis of the %RSD of the three replicates.

The following are the routine instrumental parameters for multielement analysis. Single element analysis or non-routine instrumental parameters are found in the instrument analysis data:

Incident RF power 1100 watts

Reflected RF power <5 watts

Injector tube orifice i.d. 1 mm

Argon supply liquid argon

Argon pressure 100 psi

Sample uptake rate controlled to approximately 1 mL/min.

Rinse time default: 60 seconds

Record the sample number (standard or QC sample identifier), preparation batch identifier, standard solution identifier, dilution, analyst initials, deviations from this procedure and visual observations in the instrument analysis logbook.

Samples digested in a batch must include a method blank (MB), laboratory control sample (LCS), matrix spike and duplicate sample.

Perform a preliminary data review of the sample, standards and blank performance. Note any obvious problems in the instrument analysis logbook. If the absorbance for any parameter exceeds the working range of the system, prepare a dilution of the sample and reanalyze. Record the dilution in the instrument analysis logbook.

10.4 Continuing Calibration

Analyze the calibration verification the same as any sample. (See Section 10.3) The initial calibration verification (ICV) standard is analyzed after the calibration standard and after every ten samples (CCV). Flush the system with the calibration blank before and after every standard and sample. The average intensity of three (3) exposures is used for calibration to reduce random error. The % RSD for the three replicates must be $\leq$ 5%. The initial calibration standard (ICV) must be within $\pm$ 5% of the true value. The continuing calibration standard (CCV) must be recovered within 5% for wastewater analysis and within 10% for drinking water analysis. In order for the analysis to proceed, these acceptance criteria must be met.

Record the calibration verification standard identifier, concentration, analyst initials and any deviations to this procedure in the instrument analysis logbook.

Analyze the calibration blank (CCB) after the calibration verification standard and every ten samples. The absolute value of the calibration blank result must be less than the reporting limit listed in Table 1 for analysis to be acceptable.

10.5 Preventive Maintenance

All preventative maintenance is performed per the manufacturer's instructions for each instrument and is noted in the instrument's Maintenance Logbook.

11. Data Evaluation, Calculations and Reporting

When a parameter is detected, the quantitation of that parameter is based on the absorbance from a known concentration compared to the absorbance from the unknown.

Calculate the sample concentration using the linear regression analysis supplied with the instrument software.

Report results in mg/L without correction for blank and recovery data. Record all QC data and report with the sample results as required by client specifications. Reported detection limits must be corrected for the sample dilution factor.

Sample data is reported in units of mg/L for aqueous samples and mg/kg wet weight for solid samples. Solid samples >5% solids are reported as mg/kg dry weight following solids correction in the LIMS.

For dissolved aqueous parameters or samples analyzed by "direct analysis" report the data generated directly from the instrument with allowance for sample dilution. Do not report parameter concentrations below the Reporting Limits (Table 1).

For total recoverable aqueous parameters, multiply solution parameter concentrations by the dilution factor 0.5, when 100 mL aliquot is used to produce the 50 mL final solution, and report data. If a different aliquot volume other than 100 mL is used for sample preparation, adjust the dilution factor accordingly. Also, account for any additional dilution of the prepared sample solution needed to complete the determination of parameters exceeding 90% or more of the LDR upper limit. Do not report data below the determined parameter MDL concentration or below an adjusted detection limit reflecting smaller sample aliquots used in processing or additional dilutions required to complete the analysis.

For parameters with MDLs < 10 μg/L, round the data values to the ones place and report parameter concentrations up to two significant figures. For parameters with MDLs ≥ 10 μg/L round the data values to the ones place and report parameter concentrations up to three significant figures. Extract concentrations for solids data must be rounded in a similar manner.

For total recoverable parameters in solid samples, round the solution parameter concentrations (mg/L) the same as aqueous samples. Report the data up to three significant figures as mg/kg wet-weight basis unless specified otherwise by the program or data user. Calculate the concentration using the equation below:

$$\textit{Sample Results (mg / Kg)} = \frac{C * V * D}{W}$$

where:

C = Concentration in digestate (μg/L)

V = Volume of digestate (L, 100 mL = 0.1L)

D = Dilution factor (undiluted = 1)

W = Weight of sample aliquot extracted (g x 0.001 = kg)

Do not report parameter data below the estimated solids MDL or an adjusted MDL because of additional dilutions required to complete the analysis.

To report percent solids in solid samples calculate as follows:

$$\%\text{Solids} = \frac{DW}{WW}$$

where:

DW = Sample weight (g) dried at 103 - 105°C.

WW = Sample weight (g) before drying

Calculations for Hardness by SM2340B

The method for determining hardness is to compute it from the results of separate determinations of Calcium and Magnesium on aqueous samples.

Total Hardness:

Total Hardness, mg equivalent $CaCO_3$/L = [2.497 (Ca, mg/L)] + [4.118 (Mg, mg/L)]

Calcium Hardness:

Calcium Hardness, mg equivalent $CaCO_3$/L = [2.497 (Ca, mg/L)]

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

Also refer to Section 9 for Quality Control and acceptance criteria.

If the SIC is outside of the recovery window, then the standard is reanalyzed. If the standard failure continues, the IECs for the element/elements in question are reviewed and recalculated if necessary.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard. If the standard passes the second time then the analysis may be continued. The raw data is noted. If the standard fails again, the problem must be found and corrected and the instrument is recalibrated. The ICV/ICB standard is reanalyzed and all previous data that had failed back to the previous passing CCV/CCB is reanalyzed.

The reanalysis procedure outline above is also conducted for a failing LCS or Method Blank; they may be rerun alone on the new or any subsequent passing bracket. The LCS or Method Blank do not qualify a bracket of samples but the batch run itself.

If the Matrix Spike does not meet acceptance criteria, A post digestion spike may be performed by adding the same concentration standard to the digested sample. The percent recovery for the post digestion spike is calculated to identify possible sources of the interference; the recovery range of 80-120% is applied. The post digestion spike results are narrated on the final report along with the original failed spike recovery.

If sample Duplicates are outside of the acceptance criteria, the analyst examines the sample for homogeneity. If the sample is not homogenous, this is narrated on the final report. Clean, homogenous samples are reanalyzed and if still outside of the acceptance limits, redigested and reanalyzed.

Sample nonconformance regarding a Matrix Spike recovery or a duplicate %RSD is narrated on the final report along with the corrective action(s) taken.

The mixed element SIC solution is analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than +/- the LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, instrument is recalibrated, verified with the ICV/ICB and the SIC is then re-analyzed. Alternatively, the LLOQ may be raised to twice the concentration observed in the SIC solution if approved by the Department Manger or QA Department and the level is below the regulatory action limit or project

specific requirements. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions These may be present up to the concentration documented plus the LLOQ. If failure continues notify the Department Supervisor or Manager.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2124 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1797 Hazardous Waste Management and Disposal SOP

16. Attachments

Table 1: Reporting Limits
Table 2: Interferent Listing
Table 3: LCS and Matrix Spike

Table 1: Reporting Limits

Parameter	Wavelength (nm)	Standard Reporting Limits, aqueous (µg/L)	Standard Reporting Limits, solids (mg/Kg)
Aluminum (Al)	396.1	100	4.0
Antimony (Sb)	206.8	50	2.0
Arsenic (As)	189.0	5	0.2
Barium (Ba)	493.4	10	0.4
Beryllium (Be)	313.0	3	0.12
Boron (B)	249.6	30	1.2
Cadmium (Cd)	226.5	4	0.16
Calcium (Ca)	317.9	100	4.0
Chromium (Cr)	267.7	10	0.4
Cobalt (Co)	228.6	20	0.8
Copper (Cu)	324.7	10	0.4
Iron (Fe)	271.4	50	2.0
Lead (Pb)	220.3	10	0.4
Magnesium (Mg)	279.0	100	4.0
Manganese (Mn)	257.6	10	0.4
Molybdenum (Mo)	202.0	50	2.0
Nickel (Ni)	231.6	25	1.0
Potassium (K)	766.4	2500	100
Selenium (Se)	196.0	10	0.4
Silica (SiO_2)	288.2	500	20.0
Silver (Ag)	328.0	7	0.28
Sodium (Na)	588.9	2000	80.0
Strontium* (Sr)	421.5	10	0.4
Thallium (Tl)	190.8	20	0.8
Tin (Sn)	189.9	50	2.0
Titanium (Ti)	337.2	10	0.4
Vanadium (V)	292.4	10	0.4
Zinc (Zn)	213.8	50	2.0

Notes:
Standard Reporting Limits listed are for undiluted samples, digested or acid matrix matched direct analysis samples.
Calculated Method Detection Limits (MDLs) are on file in the QA Department in a separate file.

**Method modification: Sr wavelength differs from reference method*

Table 2: Interferent Listing

Analyte	Wavelength (nm)	Interferent
Ag	328.068	Ce, Ti, Mn
Al	308.215 / 396.1	V,Mo,Ce,Mn
As	193.759	V,Al,Co,Fe,Ni
B	249.678	None
Ba	493.409	None
Be	313.042	V,Ce
Ca	315.887	Co,Mo,Ce
Cd	226.502	Ni,Ti,Fe,Ce
Ce	413.765	None
Co	228.616	Ti,Ba,Cd,Ni,Cr,Mo,Ce
Cr	205.552	Be,Mo,Ni
Cu	324.754	Mo,Ti
Fe	259.940	None
K	766.491	None
Li	670.784	None
Mg	279.079	Ce
Mn	257.610	Ce
Mo	203.844	Ce
Na	588.995	None
Ni	231.604	Co,Tl
P	214.914	Cu,Mo
Pb	220.353	Co,Al,Ce,Cu,Ni,Ti,Fe
Sb	206.833	Cr,Mo,Sn,Ti,Ce,Fe
Se	196.099	Fe
SiO_2	251.611	None
Sn	189.980	Mo,Ti,Fe,Mn,Si
Sr	421.552	None
Tl	190.864	Ti,Mo,Co,Ce,Al,V,Mn
Ti	334.941	None
V	292.402	Mo,Ti,Cr,Fe,Ce
Zn	213.856	Ni,Cu,Fe

Table 3: LCS and Matrix Spike

Analyte	Liquid Concentration (mg/L)	Soil Concentration * (MS spike only) (mg/Kg)
Antimony	0.5	160
Arsenic	0.12	160
Barium	2.00	160
Beryllium	0.05	80
Cadmium	0.051	80
Chromium	0.20	160
Copper	0.25	160
Lead	0.51	160
Nickel	0.50	160
Selenium	0.12	160
Silver	0.05	40
Thallium	0.12	160
Zinc	0.50	160
Iron	1.00	800
Manganese	0.50	160
Calcium	10.0	800
Magnesium	10.0	800
Potassium	10.0	800
Sodium	10.0	800
Aluminum	2.00	800
Cobalt	0.50	160
Vanadium	0.50	160
Boron	1.0	NA
Molybdenum	1.0	NA
Titanium	1.0	NA

*MS spike of a solid based on 1.25g and a final volume of 50 mL.

Note: Solids LCS is an SRM with certified value provided by the vendor on a lot basis.

Determination of Mercury in Water

(Cold-Vapor Atomic Absorption Spectrometry)

Reference: **EPA 245.1,** Methods for the Chemical Analysis of Water and Wastes. EPA/600/4-79-020. Revision 3, 1994.

1. Scope and Application

Matrices: Method 245.1 is a cold-vapor atomic absorption procedure approved for determining the concentration of total mercury (organic + inorganic) in drinking, surface, ground, sea, brackish waters, industrial and domestic wastewaters. This method may also be used to analyze effluents, and domestic sewages providing potential interferences are not present. This method is approved for NPDES and SDWA. All samples must be subjected to an appropriate dissolution step prior to analysis.

Definitions: See Alpha Analytical Quality Manual Appendix A.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Metals Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Mercury Analyzer and in the interpretation of Mercury data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Prior to analysis, the liquid samples must be prepared according to the procedure discussed in this method.

Method 245.1, is a cold-vapor atomic absorption technique, based on the absorption of radiation at 253.7-nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.1 Method Modifications from Reference

2.1.1 A smaller sample sized is prepared, and therefore proportionately less reagent volumes are used.

2.1.2 Samples are digested in a digestion block.

3. Reporting Limits

The typical reporting limit for Mercury is 0.0002mg/L.

4. Interferences

Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

Copper has also been reported to interfere; however, copper concentrations as high as 10mg/L had no effect on recovery of mercury from spiked samples.

Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine chloride reagent (25mL). Both inorganic and organic mercury spikes have been quantitatively recovered from seawater by using this technique.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Sample preparation is conducted under a laboratory exhaust hood. The analyst must wear chemical resistant gloves when handling concentrated mercury standards.

The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Therefore, the acidification of samples is to be conducted under a laboratory exhaust hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in either glass or plastic containers.

6.2 Sample Preservation

If samples are for soluble metals analysis, filtration must take place prior to preservation with 1:1 HNO3 to a pH < 2. Soluble samples must be held at pH < 2 for at least 24 hours prior to digestion if not preserved at the time of filtration. Samples for total metals analysis are preserved with 1:1 HNO3 to a pH < 2. Samples must be pH <2 for at least 24 hours prior to digestion if not preserved at the time of collection.

6.3 Sample Shipping
No special shipping requirements.

6.4 Sample Handling
Samples are stored at room temperature and analyzed as soon as possible after collection. The samples have a 28-day holding time from the time of collection to analysis.

7. Equipment and Supplies

Instrumentation:

Perkin Elmer FIMS 100 Atomic absorption spectrophotometer: Use instrument settings recommended by the manufacturer. The PE FIMS is designed specifically for the measurement of mercury using the cold-vapor technique with BOC (background offset correction) performed by a survey scan prior to each sample introduction. PE S10 autosampler is coupled to the instrument.

Cetac M-6100 Atomic absorption spectrophotometer: Use instrument settings recommended by the manufacturer. This instrument employs a reference cell off-set correction and full automation through the CETAC software. A Cetac ASX-260 autosampler is coupled to the instrument.

Nippon Instrument model# RA-4300A analyzer with integrated 80 position autosampler:
The instrument adds a stannous chloride (II) solution to the sample post digestion, the divalent mercury ion (Hg2+) is reduced to zero-valent metallic mercury and turns into mercury gas by bubbling. $Hg2{+}{+}SnCl2 \rightarrow Hg0 \uparrow$
After removing the acid mist and water vapor generated by bubbling with an electronic cooling unit, the instrument measures the absorbance of mercury at 253.7 nm absorption wavelength. It measures the known mercury amount, creates a calibration curve, and then calculates the mercury amount from the absorbance.

7.1 Graduated Cylinder: Rinse once with 50% HNO_3 and then rinse with reagent water prior to use.

7.2 Volumetric Flasks, Class A, various volumes: Rinse once with 50% HNO_3 and then rinse with reagent water prior to use.

7.3 Digestion Block: Environmental Express 48 position capacity, capable of 95 °C +/- 3°C.

7.4 Filter Paper: Whatman 41 or Environmental Express filtermates.

7.5 Polypropylene Digestion Vessels: 50mL volume, with plastic screw caps

7.6 Digestion Vessels Rack: 48 position, with rack lock.

7.7 Pump Tubing: Santoprene, or Polypropylene blue/yellow, red/red and orange-green.

7.8 Laboratory Wipes

7.9 Compressed Air

8. Reagents and Standards

8.1 Reagent Water: Reagent water is DI water shown to be interference free. All references to water in this method will refer to reagent water unless otherwise specified.

8.2 Sulfuric Acid (H_2SO_4), concentrated: Reagent grade. Store at room temperature in appropriately designated acid cabinet.

8.3 Hydrochloric Acid, concentrated: Trace metal grade. Store at room temperature in appropriately designated acid cabinet.

8.4 Carrier, Hydrochloric Acid, 3%: This is the ***carrier*** for the PSA Instrument. In a 1L volumetric flask, add 30mL concentrated trace grade HCl (Section 8.3). Bring to volume with reagent water. Store at room temperature; prepare daily as needed.

8.5 Reductant, Stannous Chloride in 3% HCl: This is the ***reductant*** for the PSA Instrument. In a 1L volumetric flask, add 30mL concentrated trace grade HCl and 11g $SnCl_2 \cdot 2H_20$. Mix to dissolve the solid and bring to volume with reagent water. Store at room temperature; prepare daily as needed.

8.6 Nitric Acid (HNO_3), concentrated: Reagent grade of low mercury content. If a high reagent water is obtained, it may be necessary to distill the nitric acid. Store at room temperature in appropriately designated acid cabinet.

8.7 Nitric Acid (HNO_3), 5% Solution: In a 1L volumetric flask, add 50mL reagent grade concentrated HNO_3 (Section 8.6). Bring to volume with reagent water.

8.8 Sodium Chloride-Hydroxylamine Hydrochloride Solution: Dissolve 12g of sodium chloride and 12g of hydroxylamine hydrochloride in reagent water and dilute to 100mL. Store at room temperature; expires one month from date of preparation.

8.9 Potassium Permanganate, mercury-free, 5% solution (w/v): Dissolve 5g of potassium permanganate in 100mL of reagent water. Store at room temperature; expires one month from date of preparation.

8.10 Potassium Persulfate, 5% solution (w/v): Dissolve 5g of potassium persulfate in 100mL of reagent water. Store at room temperature; expires one month from date of preparation.

8.11 Mercury Standard Solutions, 100ppm: Purchased from two different commercial sources with certificates of analysis. Use Absolute Standards as a primary, and High-Purity Standards as a secondary source. Store at room temperature. Expires on manufacturer's specified date.

8.12 Mercury Stock Standard, 0.1ppm: To a 200mL volumetric flask, add 0.2mL of 100ppm Mercury Primary Standard Solution (Section 8.11). Bring to volume with 5% HNO_3. Expires one month from date of preparation. Use this standard for spiking LCS and MS. Store in polyethylene bottle at room temperature.

8.13 Mercury Stock ICV Standard, 0.3ppm: To a 100mL volumetric flask, add 0.3mL of 100ppm Mercury Secondary Standard Solution (Section 8.11). Bring to volume with 5% HNO_3. Store in polyethylene bottle at room temperature. Expires one month from date of preparation.

8.14 Mercury Working Standards: Prepare fresh daily.

8.14.1 0ppm Calibration Standard: Add 25mL of 5% HNO_3 (Section 8.7) to a polypropylene digestion vessel. This aliquot may be used for the CCB.

Another separate aliquot is prepared for use as the ICB and the diluent for any samples with concentration greater than 90 % the highest calibration standard used to define the linear range.

8.14.2 **0.0002ppm Calibration Standard:** To a polypropylene digestion vessel, add 0.05mL of 0.1ppm Mercury Stock Standard (Section 8.12). Bring to 25mL with 5% HNO_3.

8.14.3 **0.001ppm Calibration Standard:** To a polypropylene digestion vessel, add 0.25mL of 0.1ppm Mercury Stock Standard (Section 8.12). Bring to 25mL with 5% HNO_3.

8.14.4 **0.002ppm Calibration Standard:** To a polypropylene digestion vessel, add 0.5mL of 0.1ppm Mercury Stock Standard (Section 8.12). Bring to 25mL with 5% HNO_3.

8.14.5 **0.005ppm Calibration Standard:** To a polypropylene digestion vessel, add 1.25mL of 0.1ppm Mercury Stock Standard (Section 8.12). Bring to 25mL with 5% HNO_3.

8.14.6 **0.010ppm Calibration/CCV Standard:** To a polypropylene digestion vessel, add 2.5mL of 0.1ppm Mercury Stock Standard (Section 8.12). Bring to 25mL with 5% HNO_3.

8.14.7 **0.020ppm Calibration Standard:** To a polypropylene digestion vessel, add 5.0mL of 0.1ppm Mercury Stock Standard (Section 8.12). Bring to 25mL with 5% HNO_3

8.14.8 **0.003ppm ICV Standard:** To a polypropylene digestion vessel, add 0.25mL of 0.3ppm Mercury Stock ICV Standard (Section 8.13). Bring to 25mL with 5% HNO_3.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 ICB, CCB, and Laboratory Reagent Blank (LRB)

ICB, CCB, and LRB consist of the 0ppm standard as prepared in Section 8.14.1. Blank results must be less than the Reporting Limit (RL).

9.1.1 **ICB / CCB / Laboratory Reagent Blank (LRB) Failure Corrective Action:** If an ICB, or CCB contains a mercury concentration that is $\geq$ 10% of the mercury concentration determined for any sample in the batch, or is greater than Reporting Limit (whichever is greater), the ICB or CCB may be rerun once. If the failure continues the instrument must be checked, the issue corrected and then recalibrated. An ICB is analyzed after the initial calibration or re-calibration and the CCB is analyzed at every 10 sample injection interval.

If an LRB has a concentration greater than the RL, the data is rejected, the issue if found and the associated samples sent back for redigestion unless the associated sample concentrations are greater than 10x the blank concentration. In this case the blank is narrated and the results are reported without qualification. The LRB is analyzed once per batch of samples; batch consists of 10 samples.

9.2 Laboratory Control Sample (LCS)

The LCS Standard consists of the 0.001 ppm Mercury Working/LCS Standard (Section 8.14.4). The LCS Standard must be recovered within ± 15% of the true value. The LCS Standard is analyzed once per batch of samples. A batch consists of 10 samples.

9.2.1 **LCS Failure Corrective Action:** If the LCS is not recovered within the specified limits, the LCS is reinjected. If the %Recovery is still outside the acceptance criteria, the entire batch associated with the LCS must be re-digested.

9.3 Initial Calibration Verification (ICV)

The ICV Standard consists of the 0.003ppm Mercury Working ICV Standard (Section 8.14.9). The ICV must be recovered within 5% of the true value.

9.3.1 **ICV Failure Corrective Action:** If the ICV recovery falls outside or acceptance criteria, the ICV is immediately reinjected. If the %Recovery is outside the acceptance criteria, the analysis must stop until the problem is identified and corrected.

9.4 Continuing Calibration Verification (CCV)

The CCV Standard consists of the 0.010ppm Calibration/CCV Standard (Section 8.14.6). The CCV must be recovered within 10% of the true value.

9.4.1 **CCV Failure Corrective Action:** If the CCV falls outside of acceptance criteria, the CCV is immediately reinjected. If the %Recovery is outside the acceptance criteria, then all preceding samples that are not bracketed with a valid CCV must be reanalyzed after the problem has been identified and corrected.

9.5 Matrix Spike

A matrix spike is analyzed once per batch of samples. A batch consists of 10 samples. Prepare the matrix spike at 0.005ppm by adding 1.25mL of 0.1ppm Mercury Stock Standard (Section 8.12) to 25mL of the selected QC sample. The recovery of the matrix spike must be between 70 – 130%. See Section 11.2 for calculation.

If the recovery of the matrix spike is out of range, a post-analytical spike is analyzed. Prepare the post spike by adding 5mL of 0.010ppm Calibration Standard (Section 8.14.6) and 5mL of the sample digestate to a 50mL polypropylene digestion vessel. See Section 11.4 for calculation.

The percent recover of the post-analytical spike must be between 75-125%.

9.5.1 **Matrix Spike / Post Analytical Spike Failure Corrective Action:** If the recovery of the matrix spike is outside the 70 – 130% acceptance range, perform a post spike. If the post spike recovery is outside of a 75 - 125% recovery range, the sample and it's spike are re-digested. If the matrix is visually problematic, the sample extract may be diluted and reanalyzed, and/or a narrative explaining the matrix issue is included with the data to be included on the final report

9.6 Laboratory Duplicate

A sample is analyzed in duplicate once per batch of samples. A batch consists of 10 samples. The relative percent difference must be $\leq$ 20%, as calculated in Section 11.3

9.6.1 **Duplicate Failure Corrective Action:** If the RPD between the sample and its duplicate is > 20%, evaluate the sample matrix. If it is a visually clean matrix, the

sample and duplicate are removed from the batch and re-digested. If the matrix is visually problematic, the sample extract may be diluted and reanalyzed, and/or a narrative explaining the matrix issue is included with the data to be included on the final report.

9.7 Method-specific Quality Control Sample

9.7.1 Calibration Standard Readback (0.0002 PPM)

The 0.0002 ppm calibration standard (see section 8.14.2) is analyzed to confirm the low range of the calibration. The standard should be recovered with 50-150%. If the standard recovery fails outside of this range the problem should be identified, corrected and the instrument calibration sequence repeated. If the standard continues to fall outside of the recovery range a second time the Team Leader or Department Manager is to be contacted for further actions before proceeding.

9.8 Method Sequence

- Calibration Blank
- 0.0002ppm Calibration Standard
- 0.001ppm Calibration Standard
- 0.002ppm Calibration Standard
- 0.005ppm Calibration Standard
- 0.010ppm Calibration Standard
- 0.020ppm Calibration Standard
- ICV
- ICB
- 0.0002ppm Calibration Standard Readback
- Ten analytical samples
- CCV
- CCB
- Ten analytical samples
- CCV
- CCB

10. Procedure

10.1 Sample Preparation:

Transfer 25mL of the well-homogenized sample (or an aliquot of the sample diluted to 25mL with reagent water) to a polypropylene digestion vessel.

10.2 Standard Preparation:

Standard preparation is performed each time samples are digested. See Section 8.14 for details.

10.3 Sample and Standard Digestion:

Add 1.25mL of concentrated H_2SO_4 (Section 8.2), 0.625mL of concentrated HNO_3 (Section 8.6), 3.75mL of Potassium Permanganate Solution (Section 8.9) waiting 15 mins to ensure it is not exhausted (add more if necessary to all samples and QC), and 2mL of Potassium

Persulfate Solution (Section 8.10). Heat for 2 hours in a 95°C (+/- 5°C) digestion block. Cool, and add 1.5mL of Sodium Chloride-Hydroxylamine Hydrochloride Solution (Section 8.8). Filter through Whatman 41 or equivalent filter paper into a polypropylene digestion vessel if needed to remove any sediment or particulate. Analyze samples using the PE FIMS 100 as outlined in Section 10.5. The digested calibration standards (Section 8.14) are used in Section 10.4 to generate a calibration curve on the PE instrument.

10.4 Initial Calibration:

Analyze the digested calibration standards as per Section 10.5. Construct a calibration curve by plotting the absorbances of prepared standards versus micrograms of mercury. Determine the peak height of the unknown from the absorbance maxima on the spectrometer, and read the mercury value from the standard curve. See Section 11.1 for calculation information.

10.4.1 Instrument Calibration

10.4.1.1 Click the "Workspace" button in the toolbar.

10.4.1.2 Select TT.fms and click OK.

10.4.1.3 Click on the Setup tab in the automated analysis window

10.4.1.4 Click the "Browse" button under "Sample Information File" Select the sample information file that you want to open and click OK.

10.4.1.5 Click the "Browse" button under "Sample Information File" Select the sample information file that you want to open and click OK.

10.4.1.6 Click the "X" under the "Use Entire Sample Info File" so that it disappears.

10.4.1.7 Under the "Use Autosampler Locations Listed Below", enter the order of samples to be run. NOTE: Do not include standards and QC checks.

10.4.1.8 Click the "Analyze" tab.

10.4.1.9 Click the "Calibrate' button. The instrument will run the calibration curve.

10.4.2 Initial Calibration Verification

When the calibration is complete (6 – 7 minutes), and the curve has r2 > 0.995, click the "Analyze Samples" button. The instrument will run the ICV and ICB. If the recoveries of these are within the proper ranges (Sections 9.1 and 9.3), the instrument will continue with analysis of samples. If the recoveries of the ICV and/or ICB are not within the proper ranges (Sections 9.1 and 9.3), the problem must be found and corrected, and the instrument recalibrated per Section 10.4.1.

10.5 Equipment Operation and Sample Processing

Sample and standard analysis using the Perkin Elmer FIMS 100:

10.5.1 Instrument Setup

10.5.1.1 Turn the instrument on by flipping the power switch on the face of the instrument. The autosampler will initialize itself.

10.5.1.2 Choose AA Winlab Analyst from the START menu. The autosampler will initialize again. NOTE: The instrument must be turned on before the application is started. Otherwise, an error message will result.

10.5.1.3 Click the button next to "open a custom workspace".

10.5.1.4 Select "TT.fms" from the list and click OPEN. This will open the "FIAS Control" and "Automated Analysis" windows.

10.5.1.5 Click on the "Analyze" tab in the Automated Analysis window, and then click on the "Select Location" button. Click OK and the probe will go to the autosampler rinse.

10.5.1.6 Fill the carrier and reductant bottles. The Carrier is 3% HCl (Section 8.4). The Reductant is 1.1% SnCl2 in 3% HCl (Section 8.5).

10.5.1.7 Allow the instrument to warm up while clearing samples. Samples that are cloudy or with particulate present after clearing must be filtered through Whatman 41 filter paper (Section 7.5) before analysis.

10.5.1.8 Place carrier uptake line (blue/yellow tubing, Section 7.8) and reductant takeup line (red/red tubing, Section 7.8) into graduated cylinders containing reagent water.

10.5.1.9 Load carrier and reductant lines into pump magazines above the roller so that the long ends come out on the right side. The carrier line goes into the inner magazine, and the reductant line goes into the outer magazine.

10.5.1.10 Load the two waste lines into the pump magazines below the roller. The blue/yellow line goes into the inner two-channel magazine so that the long end comes out on the left side. The black line goes into the outer magazine so that the long end comes out on the right side.

10.5.1.11 Lock both the top and bottom magazines into place.

10.5.1.12 Unscrew the fitting from the sample absorption cell leading to the liquid vapor separator and place it into an empty dilution vial.

10.5.1.13 Click the "Pump1" button in the "FIAS Control" window to start the roller.

10.5.1.14 Adjust the tension on the lower pump magazine using the thumbscrews until a steady (but not too fast) stream of bubbles comes out of the liquid vapor separator and through the black tubing.

10.5.1.15 Adjust the tension on the upper pump magazines to obtain the following flow rates: Carrier = 9 – 11mL/minute Reductant = 5 – 7mL/minute When the flow rates are set, click on the "Pump1" button to stop the roller.

10.5.1.16 Place carrier uptake line in the carrier bottle and reductant line in the reductant bottle.

10.5.1.17 Click the "Pump1" button to restart the roller. Allow to run for a couple of minutes to flush the reagent water from the lines. Click on the "Fill/Inject" button several times to flush the sample loop.

10.5.1.18 With the "Fill/Inject" button in the "Fill" position, (button not depressed), click the "Pump1" button to stop the roller.

10.5.1.19 Remove the cap from the liquid/vapor separator and wipe dry with a KimWipe. Blow compressed air through the vapor transfer line to dry it out.

10.5.1.20 Place a PTFE membrane (Section 7.9), rough side up, in the liquid/vapor separator; replace the cap and reattach the vapor transfer line to the sample absorption cell.

10.5.1.21 Click on the "Pump1" button to start the roller.

10.5.1.22 Adjust the gas flow by turning the black knob below the air flow meter to obtain a reading of just over 50. Click on the "Pump1" button to stop the roller

10.5.2 Creating a Sample Information File and Loading the Sample Tray

10.5.2.1 Click the "SampInfo" button on the toolbar.

10.5.2.2 In the description line, type "prep date MM/DD/YY".

10.5.2.3 In the analyst line, type the analyst's initials.

10.5.2.4 Drag the scroll bar so that the autosampler location 13 is showing.

10.5.2.5 Double-click the "Sample Units" cell in line 13.

10.5.2.6 Select "µg/L" from the list and enter the range of locations (13 up to 44) and click OK.

10.5.2.7 Starting with position 13, type in the sample ID

10.5.2.8 When finished, choose "Save As" from the "File" menu, then choose the "Sample Information" file.

10.5.2.9 Save the file as MMDDYYA

10.5.2.10 **Sample Analysis**

Load the samples into the tray as follows:

Position 1 Calibration Blank

Position 2 0.2ppb Standard

Position 3 1.0ppb

Standard Position 4 2.0ppb

Standard Position 5 5.0ppb

Standard Position 6 10.0ppb

Standard Position 7 20.0ppb

Standard Position 8 ICV

Position 9 ICB

Position 10 CCV

Position 11 CCB

Position 12 LRB listed in the sample information file.

Position 13 LCS listed in the sample information file.

Positions 14 – 44 Samples as listed in the sample information file.

10.5.2.11 Click the "Load Tray" button.

10.5.2.12 Replace the empty tray with the tray containing the standards and samples.

10.5.2.13 Click the "Load Tray" button. Click the "Select Location" button and click OK to lower the probe into the autosampler rinse.

Following successful initial calibration:

10.5.2.14 The instrument will now run ten analytical samples, a CCV and CCB, ten analytical samples, CCV, CCB, etc. The CCBs and CCVs must be recovered within the proper ranges (Sections 9.1 and 9.4) for analysis to continue.

10.5.2.15 If the recoveries of the CCB and/or CCV are not within the proper ranges (Sections 9.1 and 9.4), the instrument must be recalibrated per Section 10.4.1. The samples that were analyzed after the last valid CCV/CCB must be re-analyzed.

10.5.2.16 If the sample result is greater than 90% of the concentration of the highest point on the calibration curve used to define the linear range, dilute the extract with a portion of one of the prepared blanks (ICB or CCB) to produce an analytical result that is within the range.

10.5.3 Instrument Shut Down

10.5.3.1 When analysis is complete, click the "Workspace" button in the toolbar.

10.5.3.2 Place reagent uptake lines in a beaker of reagent water.

10.5.3.3 Click on the "Analyze" tab. Click on the "Pump1" button to start the roller Allow to run for several minutes to flush reagents out of the lines. Click on the "Fill/Inject" button several times to rinse the sample loop.

10.5.3.4 Click the "Move Probe Up/Down" button to raise the probe out of the autosampler rinse.

10.5.3.5 Pull the reagent uptake lines out of the reagent water beaker to allow the pump to draw air through the lines.

10.5.3.6 Select "EXIT" from the File menu to exit the WinLab application.

10.6 Continuing Calibration

Continuing Calibration Verification Standards / Continuing Calibration Standard Blanks (Section 9.4) are analyzed after every 10 samples in the sample run, as outlined in Section 10.5.3.

10.7 Preventive Maintenance

Preventative maintenance is conducted per the manufacturer's instructions. All preventative maintenance is recorded in the Instrument Maintenance Logbook

11. Data Evaluation, Calculations and Reporting

11.1 Calculate Mercury concentrations from the daily calibration curve. The curve is generated utilizing a straight-line equation defined as:

$$A = k_1 + k_2C$$

Where: A = Average peak height of the sample/standard integrations
C = Sample/Standard Concentration, ug/L
k_1 = y-intercept
k_2 = slope

The instrument will plot peak height against concentration (ug/L). The result is generated in ug/L. This value is divided by 1000 to convert the units to mg/L. If the sample was diluted (DF), the result is multiplied by the DF to generate the final result.

Mercury, mg/L = (Concentration, ug/L) x (1mg/1000ug/L) x (DF)

11.2 Calculate percent recovery for the Matrix Spike corrected for concentrations measured in the unfortified sample. Percent recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{(Cm - C)}{s} \times 100$$

Where: Cm = measured Mercury in the fortified sample, mg/L
C = measured native Mercury sample concentration, mg/L
S = concentration equivalent of spike added to sample, mg/L

11.3 Calculate the relative percent difference (RPD) for each Duplicate of the initial quantitated concentration (IC) and Duplicate quantitated concentration (Dc) as follows:

$$RPD = \frac{|\ (IC - Dc)\ |}{(\ [IC+Dc] / 2\ )} \times 100$$

11.4 Calculate the post spike concentration as follows:

Post Analytical Spike Sample Concentration (mg/L) =
[Sample Concentration (mg/L) x (0.5)] + 0.005mg/L.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance in a nonconformance action report to the Department Manager.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard.

If the standard passes the second time then the analysis may be continued. If the standard fails again, the problem must be found and corrected and the instrument is recalibrated. The ICV/ICB standard is reanalyzed and all previous data that had failed back to the previous passing CCV/CCB is reanalyzed.

The reanalysis procedure outline above is also conducted for a failing LCS or Method Blank; they may be rerun alone on the new or any subsequent passing bracket. The LCS or Method Blank do not qualify a bracket of samples but the batch run itself.

If the Matrix Spike does not meet acceptance criteria, A post digestion spike may be performed by adding the same concentration standard to the digested sample. The percent recovery for the post digestion spike is calculated to identify possible sources of the interference; the recovery range of 80-120% is applied. The post digestion spike results are narrated on the final report along with the original failed spike recovery.

If sample Duplicates are outside of the acceptance criteria, the analyst examines the sample for homogeneity. If the sample is not homogenous, this is narrated on the final report. Clean, homogenous samples are reanalyzed and if still outside of the acceptance limits, redigested and reanalyzed.

Sample nonconformance regarding a Matrix Spike recovery or a duplicate %RSD is narrated on the final report along with the corrective action(s) taken.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP# 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP #1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15.. Referenced Documents

Chemical Hygiene Plan

SOP #1732 MDL/LOD/LOQ Generation

SOP# 1739 IDC/DOC Generation

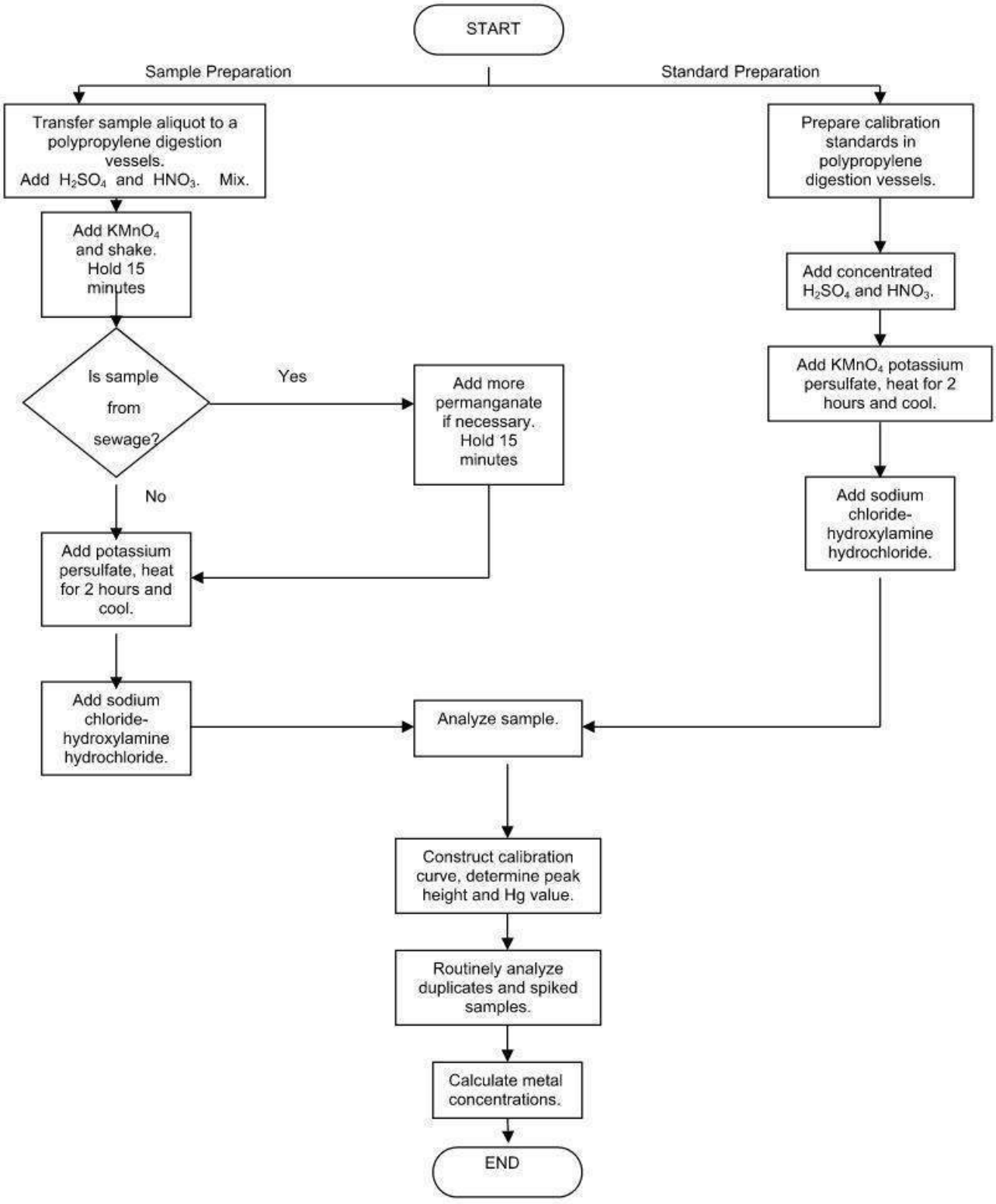
SOP# 1728 Waste Management and Disposal

16. Attachments

Figure 1: Method 245.1 Flow Chart

Figure 1

Method 245.1 Flow Chart



Organochlorine Pesticides
By Capillary Column Gas Chromatography

Reference Method No.: **EPA 608.3**

References: References: Organochlorine Pesticides and PCBs by GC/HSD. Appendix A, Part 136, Code of Federal Regulations. February 14, 2019 edition.

1. Scope and Application

Method 608.3 is used to determine the concentrations of various organochlorine pesticides in extracts from liquid matrices. This SOP details the analysis for these compounds using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD).

Matrices: Extracts liquid matrices.

Definitions: See Alpha Analytical Quality Manual

Regulatory Parameter List: The compounds listed below are determined by this method:

Parameter	CAS#
Aldrin	309-00-2
Alpha-BHC	319-84-6
Beta-BHC	319-85-7
Gamma-BHC (Lindane)	58-89-9
Delta-BHC	319-86-8
Alpha-chlordane	5103-71-9
Gamma-chlordane	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan Sulfate	1031-07-8
Endrin	72-20-8
Endrin Aldehyde	7421-93-4
Endrin Ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
Methoxychlor	72-43-5
Mirex	2385-85-5
Toxaphene	8001-35-2
Chlordane	12789-03-6

This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph (GC) and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial

demonstration of capability (see section 13), analyzing a proficiency test sample and completing the record of training.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a yearly laboratory control samples or acceptable performance from an annual proficiency test sample. A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the QA Officer and/or Laboratory Director on a case-by-case basis.

2. Summary of Method

A measured volume of sample (approximately 1L) is extracted using Separatory Funnel Extraction (Refer to SOP Qualtrax ID 1948).

A variety of cleanup steps may be applied to the extract, depending on the nature of the matrix interferences and the target analytes to be determined. Routine cleanups used include Florisil (Method 3620), Method 3660 for the removal of elemental sulfur from sample extracts.

After cleanup, the extract is analyzed by injecting a 1µL sample into a gas chromatograph equipped with narrow-bore fused silica capillary columns and electron capture (GC/ECD) detectors.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 1 lists our routine reporting limits.

4. Interferences

4.1 Only high purity gases are used in the GC system to eliminate this source of possible contamination. Both the hydrogen (carrier gas – 99.999%) and argon-methane (detector make-up gas) are certified by the gas supplier.

4.2 Preventive instrument maintenance is performed routinely, and whenever highly contaminated extracts are analyzed that could result in chromatographic interferences or result in degradation of system performance. Section 9.5 details the maintenance steps.

4.3 Glassware must be scrupulously cleaned. This procedure is detailed in the extraction SOPs. Store dry glassware in a clean environment.

4.4 All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free. All of these materials are routinely determined to be free of interferences by analysis of extraction blanks with every extraction batch performed.

4.5 Certain compounds (i.e. phthalates) can be extracted from the sample matrix and be detected by the ECD that could possibly result in false positive results or complicate the data interpretation. The use of the cleanup procedures detailed in the extraction SOPs minimize these possible interferences. Analyst experience is also crucial in making compound determinations.

4.6 Interferences co-extracted from the samples will vary considerably from waste to waste. While general cleanup techniques are referenced or provided as part of the method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

4.7 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.

4.7.1 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations.

4.7.2 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled.

4.7.3 Interferences from phthalate esters are minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination.

4.7.4 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides.

4.8 Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Coeluting chlorophenols are eliminated by using Method 3620B (florisil).

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. The following analytes covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, and BHCs. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in 1L amber glass jars with teflon-lined lids. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Upon receipt, samples must be tested for residual chlorine. Refer to the Sample Receipt and Login Qualtrax ID 1559 and the Separatory Funnel Liquid-Liquid Extraction SOP/02-02 for further information.

Also upon receipt, samples must have a pH within the range of 5.0 – 9.0 pH units. If the sample is not within this range, it is adjusted using either NaOH to increase the pH or with H2SO4 to decrease the pH. A record is made on the Sample Delivery Group form to indicate the volume of acid or base that was added to the sample.

The samples are transferred into sample storage refrigerators to be maintained at a temperature of 4 ± 2° C.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Aqueous samples must be extracted within 7 days of sample collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph, Agilent 6890, 7890: An analytical system complete with gas chromatograph configured for split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and data system.

7.2 GC Columns: Alpha utilizes dual-column analyses. The dual-column approach involves either a single injection that is split between two columns that are mounted in a single gas chromatograph, or dual injections of the split extract on a single GC equipped with two columns. Typical column pairs used are listed below. Other columns may be used as long as method performance criteria can be met.

Column pair:

STX-CLPesticides: Cat. #11546 from Restek or equivalent: 30m, 0.32mm, 0.32µm

STX-CLPesticides2: Cat. #11444 from Restek or equivalent: 30m, 0.32mm, 0.25µm

7.3 Guard Column: Cat. #10027 from Restek or equivalent: 5m, 0.32mm

7.4 Class "A" Volumetric Flasks: 10mL and 25mL, for the preparation of standards.

7.5 Microsyringes/Wiretrol syringes: 10 µL – 1000 µL

7.6 Gooseneck splitless injection liner: Cat. #23303 from Restek or equivalent

7.7 Vials: 2 mL clear glass, crimp-top and screw-cap.

7.8 Universal "Y" Press-tight tee split: Cat. #20406 from Restek or equivalent / **Siltek**

MXT Connector: Cat. #21388

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4 ± 2°C in Teflon(R)-sealed containers in the dark. When a lot of standards is prepared, aliquots of that lot are stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC tests indicate a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem.

8.1 n-Hexane: Pesticide quality or equivalent.

8.2 Acetone: Pesticide quality or equivalent.

8.3 Methylene chloride: Pesticide quality or equivalent.

8.4 Organic-free Reagent Water: All references to water in this method refer to organic-free reagent water from Alpha's RO water treatment system.

8.5 Stock Standard Solutions: All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated stock solution is opened, it is transferred to a labeled amber screw-cap vial. The expiration date of the stock solution is either the vendor specified expiration date, or 6 months from the date the ampule was opened, whichever is sooner.

8.6 Calibration Standards: Calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the calibration standard. Calibrations are performed at 10 concentration levels for individual pesticides and 9 concentration levels for Chlordane and Toxaphene; concentrations for all levels are listed below. The list of ampulated calibration standards are obtain from **Accustandard**:

8.6.1 Preparation of individual pesticides initial calibration standards:

PESTICIDES ICAL STOCK: *2000µg/L; FV=25mL, solvent Hexane*

- 50µL of Accustandard M-8081-SC (Pesticides mix; 1000µg/mL)
- 250µL of Accustandard CLP-032-R (TCMX, Deca; 200µg/mL)
- 50µL of Accustandard AS-E0219 (Mirex; 1000µg/mL)

FV=10mL
solvent: Hexane

Level	Concentration µg/L	Amount added of PEST ICAL STOCK µL
1	0.5	2.5
2	1	5.0
3	2	10
4	3	15
5	4	20
6	5	25
7	10	50
8	50	100
9	100	500
10	200	1000

8.6.2 Preparation of Chlordane/Toxaphene initial calibration standards:

Chlordane and Toxaphene ICALs are prepared and ran independently.

CHLORDANE/TOXAPHENE ICAL STOCK #1: *50/100µg/mL*
(Chlordane/Toxaphene);FV=10mL, solvent Hexane

- 100µL of **Ultra Scientific** EPA-1086 (Chlordane Solution; 5000µg/mL)
- 1mL of **Absolute** 20021 (Toxaphene Standard; 1000µg/mL)

CHLORDANE/TOXAPHENE ICAL STOCK #2 (level7): *1000/2000µg/L*
(Chlordane/Toxaphene);FV=10mL, solvent Hexane

- 200µL of STOCK #1
- A 9-point calibration is NOT required for Toxaphene and Chlordane. See note below.

Level	Concentration µg/L	Amount added of ICAL STOCK # µL
1	5/10	50 #2
2	10/20	100 #2
3	50/100	500 #2
4	100/200	1000 #2
5	250/500	50 #1
6	500/1000	100 #1
7	1000/2000	200 #1
8	2500/5000	500 #1
9	5000/10000	1000 #1

FV=10mL
solvent: Hexane

Note: A 9-point calibration is **NOT** required for Toxaphene or Chlordane. The requirement for Toxaphene is at least a 3-point calibration. Levels 2, 7, and 8 are usually used. The requirement for Chlordane is at least a 1-point curve. Level 7 is usually used.

8.7 Second Source Standards (ICV/CCAL): Continuing Calibration/Calibration Verification Standards are prepared volumetrically by diluting the appropriate stock standard(s) with Hexane. They expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the standard.

8.7.1 Preparation of individual pesticides Calibration Verification Standard:

PESTICIDES ICV STOCK: 1000 µg/L; FV=25mL, solvent Hexane

- 25µL of Ultra, Cat. #PPM-808C-1 (1000µg/mL)
- 25µL of Absolute, Cat. #79136 (1000µg/mL)

PESTICIDES ICV: 50µg/L; FV=50mL, solvent Hexane

- 2.5mL of Pest ICV Stock
- 2.5mL of Pest Surrogates Stock

8.7.2 Preparation of Chlordane and Toxaphene Calibration Verification Standards:

CHLORDANE/TOXAPHENE ICV STOCK: *50000/100000µg/L*
(Chlordane/Toxaphene);FV=10mL, solvent Hexane

- 0.5mL of Restek, Cat. #32021 (1000µg/mL)
- 1.0mL of Restek, Cat. #32005 (1000µg/mL)

CHLORDANE/TOXAPHENE ICV: *1000/2000µg/L*
(Chlordane/Toxaphene);FV=25mL, solvent Hexane

- 0.5mL of Chlordane ICV Stock
- 1.0mL of Toxaphene ICV Stock

8.8 Internal Standard Solution: 1-Bromo-2-nitrobenzene is used as the internal standard, and is added to all calibration standards and sample extracts to achieve a concentration of 0.025µg/mL. Solution expires 6 months from the date of preparation, or on the earliest expiration date of the stock solution used to prepare the standard.

8.9 PEM/DEG Solution: is prepared volumetrically by diluting stock standard in Hexane. Solution expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solution used to prepare the standard.

- 5mL of Pesticides Deg. Check Solution, Ultra. #ISM-450-1 into 100mL of Hexane

8.10 Surrogate Standards: Tetrachloro-m-xylene and Decachlorobiphenyl are used as surrogates. They are added to the single-component calibration standards at the concentrations listed in Table 2, and are spiked into all samples and QC samples prior to extraction. Solutions expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the standard.

- **Extraction Surrogate Stock:** is prepared by diluting of 10mL of TCMX&DCB (Accustandard, Cat. #CLP-032-R) to 1000mL of Acetone to achieve concentration of TCMX and DCB at 2.0µg/mL.
- **ICV Surrogate Stock:** is prepared by diluting of 250µL of Pesticides Surrogate Standard (Ultra, Cat. #CS-1947) to 50mL of Hexane to achieve concentration of TCMX and DCB at 1.0µg/mL.

8.11 LCS/MS Spiking Solutions: The LCS/MS spiking solutions are prepared volumetrically by diluting the appropriate stock standards in acetone. Solution expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the standard.

PESTICIDES LCS/MS: 2.0 µg /mL; FV=250mL, solvent Acetone

- 0.5mL of Organochlorine Pesticides Standard, Ultra, Cat. #CR-1559A (1000µg/mL)
- 0.5mL of Mirex, Absolute, Cat. #79136 (1000µg/mL)

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A Method Blank is an aliquot of a clean reference matrix (reagent water) that is carried through the entire analytical procedure. Extraction blanks are performed with each extraction batch of 20 or less samples, according to the extraction SOPs. If any analyte of interest is found in the blank at a concentration greater than the MDL for the analyte or at a concentration greater than 1/10th the concentration in a sample in the batch, whichever is greatest, analysis of samples must be halted and samples in the batch must be re-extracted and the extracts re-analyzed. The surrogate recoveries must also be within the acceptance criteria listed in Table 3. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary. The result for each analyte in the blank is reported at or above the MDL to 2 significant figures unless specified otherwise by the regulatory agency or permit.

9.2 Laboratory Control Sample (LCS)

A Laboratory Control Sample (LCS) is extracted with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same volume. The LCS is spiked with the single component pesticide analytes. The concentration of the spiking solution is listed in Table 2. The recovery acceptance criteria are listed in Table 3. If any recovery criteria are not met, the extract should be reanalyzed. If the criteria are still not met, the entire batch should be re-extracted. If this is not possible, due to insufficient sample or holding time exceedance, the analyst must write up the failure on a narrative sheet for inclusion in the client report.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike (MS)/matrix spike duplicate (MSD) are extracted and analyzed utilizing pesticide spike for each batch of 20 or less samples. The recovery acceptance criteria are listed in Table 3.

9.6 Laboratory Duplicate

NA

9.7 Surrogates

All extracted samples and associated QC are spiked with Extraction Surrogates Stock to achieve concentration of TCMX and DCB at **0.05µg/mL**. The laboratory must evaluate surrogate recovery data from individual samples and QC samples versus the surrogate control limits listed in Table 3. If the surrogate limits are not met, the extract should be reanalyzed to determine if the failure was due to an instrument problem. If the criteria are still not met, the affected samples should be re-extracted to confirm that the failure was due to sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

9.8 Method Sequence

Typical Initial Calibration (each level to identify the standard lot number)

1. Instrument Blank
2. Degradation check standard
3. Std Level 1-10 – Single Pesticides
4. Initial Calibration Verification Standard (ICV)
5. Std Level 1-9 – Chlordane
6. Chlordane Initial Calibration Verification Standard (ICV)
7. Std. Level 7 -Toxaphene (Full Toxaphene calibration must be run in case of any Toxaphene detection in the samples)
8. Toxaphene Initial Calibration Verification Standard (ICV)

   [**NOTE:** If multiple calibration mixtures are analyzed, it is acceptable to analyze appropriate ICVs after all calibration standards have been injected.]

Typical Daily sequence (each standard must to be identified with lot number)

1. Degradation Check Standard
2. Pesticide Continuing Calibration Standard (ICV-Second Source)
3. Chlordane Continuing Calibration Standard (ICV-Second Source)
4. Toxaphene Continuing Calibration Standard (ICV-Second Source)
5. Extraction Blank

6. Laboratory Control Sample
7. Matrix Spike
8. Matrix Spike Duplicate
9. Samples (up to 16 may be analyzed)
10. Pesticide Continuing Calibration Standard (ICV-Second Source)
11. Repeat 5 – 10 (as needed)

10. Procedure

10.1 Equipment Set-up

10.1.1 GC Conditions:

The dual-column / dual-detector approach involves the use of the columns listed in section 7.2. The columns are connected to an injection tee or dual injection GC, and separate electron capture detectors. Typical GC conditions are listed below, but may be altered as long as method performance criteria are met.

Temperature 1:	120 °C
Time 1:	0 minute
Ramp 1:	45 °C/minute
Temperature 2	200 °C
Time 2:	0 minutes
Ramp 2:	15 ° C/minute
Temperature 3:	230 °C
Time 3:	0 minutes
Injector temperature:	250 °C
Ramp 3:	30 °C/minute
Final Temperature:	330 °C
Final Time:	2.0 minutes

Injector mode:	Pulsed Split
	2:1 split, 0.75 min pulse
Injector Flow:	6.0 mL/min split flow
Detector temperature:	375 °C
Carrier gas:	Hydrogen
Carrier flow:	17 mL/min
Carrier mode:	Constant flow
Makeup gas:	Argon/methane (P5)
Total detector flow:	55 mL/min
Injection volume:	1 µL

10.1.2 DDT and Endrin Breakdown (PEM/DEG)

The breakdown of DDT and Endrin must be measured before samples are analyzed and at the beginning of each 12-hour shift. Injector maintenance must be completed if the breakdown in greater than 15% for either compound (See Section 10.5.1). Both analytical columns must pass DDT/Endrin breakdown criteria prior to sample analysis.

10.2 Initial Calibration

10.2.1 Prepare calibration standards using the procedures in Section 8.6.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in section 10.1.1. The same operating conditions are used for calibrations and sample analyses. Create the analytical sequence using the Agilent Chemstation data acquisition software. Record the calibration standard, unique lot number (PP#) and analyst's initials in the analytical sequence list.

10.2.3 A 1µL injection volume of each calibration standard is typically used. Other injection volumes may be employed, provided that the analyst can demonstrate adequate

sensitivity for the compounds of interest. The same injection volume must be used for all standards and samples.

10.2.4 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day or more or after system maintenance. The GC column may be primed (or deactivated) by injecting a pesticide standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

Alternately, the system may be primed by baking at the final analytical temperature for approximately 30 minutes.

Several analytes may be observed in the injection just following system priming. Always run an instrument blank after system priming.

10.2.5 Calibration Factors

Internal standard calibration techniques are employed in this method.

10.2.5.1 Internal Standard Procedure. In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formulae listed in Alpha's Quality Manual.

10.2.6 Initial Calibration Criteria

If the RSD for an analyte is **≤ 15%**, then the response of the instrument for this compound is considered linear over the range and the mean calibration factor can be used to quantitate sample results.

If the RSD for any analyte is > 15%, then linearity through the origin cannot be assumed. The mean response factor cannot be used for quantitation. An alternative calculation may be done by the use of **linear regression** or **quadratic regression** (minimum of six ICAL points are needed and regression must be weighted inversely proportional to concentration) as long as the correlation coefficient is **≥0.990**. If both of these quantitation methods fail criteria for any compound in the initial calibration, then the system must be reevaluated and a new calibration curve must be analyzed.

10.2.7 Retention Time Windows

10.2.7.1 The retention time window used for the identification of target analytes is ± 0.05 minutes. These criteria have been adopted from the EPA CLP Statement of Work (OLM04.2). It has been found that these limits work well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results.

10.2.7.2 The windows listed above are used as guidance; however, the experience of the analyst weighs heavily in the interpretation of the chromatograms. For example, it has been observed that certain oil matrices can cause the retention times to shift more dramatically. Additionally, if any positive results are questionable and at sufficiently high concentration, GC/MS analysis is used for confirmation.

10.2.8 Initial Calibration Verification

An initial calibration verification standard must be run immediately after each initial calibration, near the midpoint of the curve. This standard must be prepared using a second source that is different than the source used for the initial calibration. The %D for each analyte to be quantitated must not exceed limits listed in Table 3 when compared to the initial calibration curve.

10.3 Equipment Operation and Sample Processing

10.3.1 Tentative identification of an analyte occurs when a peak from a sample extract falls within the retention time window for the compound. Each tentative identification is confirmed using a second GC column of dissimilar stationary phase. In particularly difficult matrices, confirmation by GC/MS may be advisable.

10.3.2 The concentration reported for an identified target analyte in an extract is calculated using the Enviroquant data processing software. The Enviroquant methods have been configured to utilize the quantitation formulas found in Alpha's Quality Manual. Proper quantitation requires the appropriate selection of a baseline from which the peak area or height can be determined. See the Manual Integration SOP for integration guidelines.

10.3.2.1 If the responses exceed the calibration range of the system, dilute the extract and reanalyze. The dilution reported is the least dilute level at which the peak area is within the calibration range.

10.3.3 Each sample analysis must begin with an acceptable initial calibration, breakdown standard, calibration verification standard(s) (each 12-hour analytical shift) and be bracketed with passing calibration verification standard. The Internal Standard area of the samples must be within 50%-200% of the opening CCV. When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that met the QC criteria must be re-injected.

10.3.4 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

10.3.5 Use the calibration standards analyzed during the sequence to evaluate retention time stability. The retention time windows are established using the absolute retention time of each analyte in the mid-concentration standard during the initial calibration as the mid-point of the window. The widths of the windows are defined in Section 10.2.7.

10.3.6 Each subsequent injection of a standard during the 12-hour analytical shift (i.e., those standards injected every 20 samples, or more frequently) must be checked against the retention time windows. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

10.3.7 Identification of mixtures (i.e. Chlordane and Toxaphene) is based on the characteristic 'fingerprint' retention time and shape of the indicator peak(s); and quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding calibration peaks(s) of the same retention time and shape generated using internal calibration procedures.

10.3.8 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be

needed. If instrument problems are suspected, rerun the extract on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to the extraction SOPs for the procedures to be followed in sample cleanup.

10.3.9 For secondary column analysis, a second dissimilar column is utilized to confirm positive pesticide results. The laboratory must report the ***LOWER*** of the two results unless obvious interference is present on one of the columns.

10.3.10 GC/MS Confirmation

GC/MS confirmation may be used in conjunction with either single-column or dual-column analysis if the concentration is sufficient for detection by GC/MS.

10.3.11.1 Full-scan GC/MS will normally require a concentration of approximately 10ng/µL in the final extract for each single-component compound.

10.3.11.2 The GC/MS must be calibrated for the specific target pesticides when it is used for quantitative analysis.

10.3.11.3 GC/MS may not be used for confirmation when concentrations are below the sensitivity of the instrument.

10.3.11.4 GC/MS confirmation should be accomplished by analyzing the same extract that is used for GC/ECD analysis.

10.3.11.5 The base/neutral/acid extract and the associated blank may be used for GC/MS confirmation if the surrogates and internal standards do not interfere and if it is demonstrated that the analyte is stable during acid/base partitioning. However, if the compounds are not detected in the base/neutral/acid extract, then GC/MS analysis of the pesticide extract should be performed.

10.3.11.6 A QC reference sample containing the compound should also be analyzed by GC/MS. The concentration of the QC reference sample must demonstrate that those pesticides identified by GC/ECD can be confirmed by GC/MS.

10.4 Continuing Calibration Verification

10.4.1 Verify calibration each 12-hour shift by injecting calibration verification standards (CCV) prior to conducting any sample analyses. A calibration standard must be injected at intervals of not less than once every twenty samples and at the end of the analysis sequence. The calibration verification standards must be obtained from a second manufacturer or manufacturer's batch prepared independently from the batch used for the calibration.

10.4.2 Response factor (for internal standard compounds) for each analyte to be quantitated must not exceed the limits listed in Table 3 when compared to the initial calibration curve. The Enviroquant data processing software automatically calculates the %D for all analytes according to the formulae in Alpha's Quality Manual.

10.4.3 If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis

10.4.4 The Internal Standard area of the CCV must be within 50%-150% of the average area of all initial calibration points for the internal standard. A retention time shift >30 seconds

for the internal standard compare with Initial calibration necessitates reanalysis of all affected samples.

10.5 Preventive Maintenance

Routine preventive maintenance should be performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and replacement of injector seals.

10.5.1 Other Maintenance

Additional maintenance may be required if system performance degrades.

10.5.1.1 GC injector ports are of critical concern, especially in the analysis of DDT and Endrin.

10.5.1.1.1 Injectors that are contaminated or chemically active can cause the degradation ("breakdown") of the analytes. Endrin and DDT breakdown to Endrin aldehyde, Endrin ketone, DDD, or DDE.

Check for degradation problems by injecting a standard containing only 4,4'-DDT and Endrin. Presence of 4,4'-DDE, 4,4'-DDD, Endrin ketone or Endrin indicates breakdown. If degradation of either DDT or Endrin exceeds 15%, take corrective action before proceeding with calibration.

When such breakdown is observed, replacement of the injector liner and seal may solve the problem. If not, clip approximately 3 – 6 inches from the injector end of the GC column. If the degradation does not improve, it may be necessary to replace the column(s).

Calculate percent breakdown as follows:

$$\%\ \text{breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD+DDE)}}{\text{sum of all peak areas (DDT+DDE+DDD)}} \times 100$$

$$\%\ \text{breakdown of Endrin} = \frac{\text{sum of degradation peak areas (aldehyde+ketone)}}{\text{sum of all peak areas (Endrin+aldehyde+ketone)}} \times 100$$

10.5.1.2 ECD detectors may also become contaminated, requiring bake out at elevated temperatures, or repair by the manufacturer.

11. Data Evaluation, Calculations and Reporting

11.1 Quantitation of Single Component Pesticides

The single component pesticide compounds are reported in µg/L units. After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews should be done by two separate individuals.

11.1.1 Quantitation of Multiple-Component Analytes

Quantitation is based on use of 3-5 of the major peaks present for Chlordane and 4-6 of the major peaks present for Toxaphene. Each of these peaks is individually calibrated based on average response factors. The %RSD must meet the criteria of ≤15%. The 3 to 5 or 4 to 6 major peaks are calculated as described in Section 10.3.3. After individual calculation meets criteria, the average of the major 3 to 5 or 4 to 6 peaks is used to determine the final concentration.

11.1.1.1 Toxaphene

Toxaphene is quantitated by the internal standard method, using the 4 – 6 largest peaks found in the standard and averaging the resulting concentrations.

11.1.1.2 Chlordane

Chlordane is a mixture of at least 11 major components and 30 or more minor components. Trans- and cis-Chlordane (alpha and gamma, respectively), are the two major components of Chlordane. However, the exact percentage of each in the chlordane material is not completely defined, and is not consistent from batch to batch.

11.1.1.2.1 The GC pattern of a Chlordane residue may differ considerably from that of the chlordane standard. Depending on the sample substrate and its history, residues of Chlordane can consist of almost any combination of constituents from the Chlordane, plant and/or animal metabolites, and products of degradation caused by exposure to environmental factors such as water and sunlight.

11.1.1.2.2 Whenever possible, when Chlordane residue does not resemble Chlordane, the analyst should quantitate the peaks of alpha-Chlordane, gamma-Chlordane, Heptachlor, and trans-Nonachlor separately against the appropriate reference materials, and report the individual residues.

11.1.1.2.3 When the GC pattern of the residue resembles that of Chlordane, the analyst may quantitate Chlordane residues by comparing the total area of the Chlordane chromatogram using the 3-5 major peaks

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and/or improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Qualtrax ID 1732. These studies performed by the laboratory are maintained on file for review

13.2 Demonstration of Capability Studies

Refer to Qualtrax ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

Qualtrax ID 1732 MDL/LOD/LOQ Generation

Qualtrax ID1739 IDC/DOC Generation

Qualtrax ID 1728 Waste Management and Disposal SOP

16. Attachments

Table 1: REPORTING LIMITS

Table 2: STANDARD SOLUTIONS

Table 3: QC ACCEPTANCE CRITERIA

TABLE 1

REPORTING LIMITS

	RL (Aqueous)
Pesticides	
Alpha-BHC	0.02 µg/L
Gamma-BHC (Lindane)	0.02 µg/L
Heptachlor	0.02 µg/L
Endosulfan I	0.02 µg/L
Dieldrin	0.04 µg/L
Endrin	0.04 µg/L
4, 4'-DDD	0.04 µg/L
4, 4'-DDT	0.04 µg/L
Methoxychlor	0.2 µg/L
Aldrin	0.02 µg/L
Beta-BHC	0.02 µg/L
Delta-BHC	0.02 µg/L
Heptachlor Epoxide	0.02 µg/L
trans-Chlordane	0.02 µg/L
cis-Chlordane	0.02 µg/L
4, 4'- DDE	0.04 µg/L
Endosulfan II	0.04 µg/L
Endrin Aldehyde	0.04 µg/L
Endosulfan Sulfate	0.04 µg/L
Endrin Ketone	0.04 µg/L
Chlordane	0.2 µg/L
Toxaphene	0.2 µg/L
Mirex	0.02 µg/L

TABLE 2

STANDARD SOLUTIONS

	Stock 1 solution (µg/mL)	Stock 2 solution (µg/mL)	Level 10 (µg/L)	Level 9 (µg/L)	Level 8 (µg/L)	Level 7 (µg/L)	Level 6 (µg/L)	Level 5 (µg/L)	Level 4 (µg/L)	Level 3 (µg/L)	Level 2 (µg/L)	Level 1 (µg/L)	Std. Spike Solution (µg/mL)	Std. LCS Solution (µg/mL)
Pesticides	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
TCMX/DCB*	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Chlordane	50000	1000	N/A	5000	2500	1000	500	250	100	50	10	5		
Toxaphene	100000	2000	N/A	10000	5000	2000	1000	500	200	100	20	10		
Internal Standard														
1-Bromo-2-Nitrobenzene	5000	5000	25	25	25	25	25	25	25	25	25	25		

* - surrogates

TABLE 3

SURROGATES ACCEPTANCE CRITERIA

	Aqueous	
Surrogate % Recovery*	**Lower Control Limit**	**Upper Control Limit**
2,4,5,6-Tetrachloro-m-xylene	46%	94%
Decachlorobiphenyl	42%	125%

*One surrogate on one column is allowed to be outside of the control limits and still be within criteria.

QC ACCEPTANCE CRITERIA

Analyte	Calibration Veryfication (%D)	LCS (%)	MS/MSD RPD (%)
Aldrin	75-125	42-140	35
Alpha-BHC	69-125	37-140	36
Beta-BHC	75-125	17-147	44
Gamma-BHC (Lindane)	75-125	32-140	39
Delta-BHC	75-125	19-140	52
Alpha-chlordane	73-125	45-140	35
Gamma-chlordane	75-125	45-140	35
4,4'-DDD	75-125	31-141	39
4,4'-DDE	75-125	30-145	35
4,4'-DDT	75-125	25-160	42
Dieldrin	48-125	36-146	49
Endosulfan I	75-125	45-153	28
Endosulfan II	75-125	D-202	53
Endosulfan Sulfate	75-125	26-144	38
Endrin	5-125	30-144	48
Endrin Aldehyde	75-125	**30-150***	**30***
Endrin Ketone	75-125	**30-150***	**30***
Heptachlor	75-125	34-140	43
Heptachlor Epoxide	75-125	37-142	26
Methoxychlor	75-125	**30-150***	**30***
Mirex	75-125	**30-150***	**30***
Toxaphene	68-134	40-140	41
Chlordane	75-125	**30-150***	**30***

*** - in house limits**

PCBs

By Capillary Column Gas Chromatography

Reference Method No.: **EPA 608.3**

References: References: Organochlorine Pesticides and PCBs by GC/HSD. Appendix A, Part 136, Code of Federal Regulations. February 14, 2019 edition.

1. Scope and Application

Method 608.3 is used to determine the concentrations of Polychlorinated Biphenyls (PCBs) as Aroclors in extracts from liquid matrices. This SOP details the analysis for PCBs using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD).

Matrices: Extracts liquid matrices.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Regulatory Parameter List: The standard compounds listed below are determined by this method.

Parameter	CAS#
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph (GC) and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability (see section 13.2).

2. Summary of Method

A measured volume of sample (approximately 1L or 2L for lower reporting limits) is extracted using the appropriate matrix-specific sample extraction technique.

Liquid samples are extracted at neutral pH with methylene chloride using Separatory Funnel Extraction (Refer to SOP Qualtrax ID 1948), or other appropriate technique.

Sulfuric acid cleanup (Method 3665A), Copper cleanup (Method 3660B) and Silica Gel cleanup (Method 3630) are utilized for PCB extracts. See extraction SOP for details.

After cleanup, the extract is analyzed by injecting 1µL into a gas chromatograph equipped with narrow- or wide-bore fused silica capillary columns and electron capture (GC/ECD) detectors.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

The routine reporting limit for this method is:

- Aqueous samples: 0.25 ug/L / Aroclor (based on a 1L extraction)

4. Interferences

4.1 Instrumental

4.1.1 Only high purity gases are used in the GC system to eliminate this source of possible contamination. Both the helium (carrier gas – 99.999%) and argon-methane (detector make-up gas) are certified by the gas supplier.

4.1.2 Preventive instrument maintenance is performed routinely, and whenever highly contaminated extracts are analyzed that could result in chromatographic interferences or result in degradation of system performance. Section 10.5 details the maintenance steps.

4.1.3 Glassware must be scrupulously cleaned. This procedure is detailed in the Organic Extraction Cleaning and Handling SOP/1953. Store dry glassware in a clean environment.

4.2 Parameters

4.2.1 All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free. All of these materials are routinely determined to be free of interferences by analysis of extraction blanks with every extraction batch performed.

4.2.2 Certain compounds (i.e. phthalates) can be extracted from the sample matrix and be detected by the ECD that could possibly result in false positive results or complicate the data interpretation. The use of the cleanup procedures detailed in the extraction SOP minimizes these possible interferences. Analyst experience is also crucial in making compound determinations.

4.2.3 Interferences co-extracted from the samples will vary considerably from waste to waste. While a general cleanup technique is referenced or provided as part of the method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From

this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. PCBs have been tentatively classified as known or suspected human or mammalian carcinogens. Additional references to laboratory safety are available in the Chemical Hygiene Plan.
All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two 1L amber glass jars with teflon-lined lids. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Aqueous samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of 4 ± 2° C. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of 4 ± 2° C.

6.3 Sample Handling

Aqueous samples must be extracted within 365 days of sample collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph, Agilent 6890, 7890: An analytical system complete with gas chromatograph configured for split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and data system.

7.2 GC Columns: Alpha utilizes dual-column analyses. The dual-column approach involves either a single injection that is split between two columns that are mounted in a single gas chromatograph. Typical column pair used is listed below. Other columns may be used as long as method performance criteria can be met.

Column pair:

RTX-CLP: Cat. #11141 from Restek or equivalent; 30m, 0.32mm, 0.32µm

RTX-CLPII Cat. #11324 from Restek or equivalent; 30m, 0.32mm, 0.25µm

7.3 Guard Column: Cat. #10027 from Restek or equivalent; 5m, 0.32mm

7.4 Class "A" Volumetric Flasks: 10mL and 25mL, for standards preparation

7.5 Microsyringes/Wiretrol syringes: 10 µL – 1000 µL

7.6 Gooseneck splitless injecton liner, Cat #20799-214.5 from Restek or equivalent

7.7 Universal "Y" Press-tight tee split: Cat. #20406 from Restek or equivalent or equivalent / **Siltek MXT Connector:** Cat. #21388

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.
NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4 ± 2° C in Teflon(R)-sealed containers in the dark. When a Lot of standards is prepared, aliquots of that Lot are stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC tests indicate a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem.

8.1 n-Hexane: Pesticide quality or equivalent.

8.2 Acetone: Pesticide quality or equivalent.

8.3 Organic-free Reagent Water: All references to water in this method refer to organic-free reagent water from Alpha's RO water treatment system.

8.4 Stock Standard Solutions: All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated stock solution is opened, it is transferred to a labeled amber screw-cap vial. The expiration date of the stock solution is either the vendor specified expiration date, or 1 year from the date the ampule was opened, whichever is sooner.

8.5 Calibration Standards: Calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the calibration standard. Calibrations are performed at the 6 concentration levels listed in Table 1. The list of ampulated calibration standards are obtain from **Ultra**:

- Aroclor 1016, Cat. #PP-282, at 100ug/ml
- Aroclor 1260, Cat. #PP-361, at 100ug/ml
- Aroclor 1262, Cat. #PP-371, at 100ug/ml

- Aroclor 1268, Cat. #PP-382, at 100ug/ml
- Aroclor 1221, Cat. #PP-292, at 100ug/ml
- Aroclor 1232, Cat. #PP-302, at 100ug/ml
- Aroclor 1242, Cat. #PP-312, at 100ug/ml
- Aroclor 1248, Cat. #PP-342, at 100ug/ml
- Aroclor 1254, Cat. #PP-351, at 100ug/ml

8.6 Second Source Standards: (ICV/CCAL) Continuing Calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Continuing Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the standard. The list of ampulated standards are obtain from **Accustandard**:

- Aroclor 1016, Cat. #C-216S-H-10X, at 1000ug/ml
- Aroclor 1260, Cat. #C-260S-H-10X, at 1000ug/ml
- Aroclor 1262, Cat. #C-262S-H-10X, at 1000ug/ml
- Aroclor 1268, Cat. #C-268S-H-10X, at 1000ug/ml
- Aroclor 1221, Cat. #C-221S-H-10X, at 1000ug/ml
- Aroclor 1232, Cat. #C-232S-H-10X, at 1000ug/ml
- Aroclor 1242, Cat. #C-242S-H-10X, at 1000ug/ml
- Aroclor 1248, Cat. #C-248S-H-10X, at 1000ug/ml
- Aroclor 1254, Cat. #C-254S-H-10X, at 1000ug/ml

8.7 Internal Standard Solution: 1-Bromo-2-nitrobenzene (Ultra, Cat. #PPS-351) is used as the internal standard, and is added to all calibration standards and sample extracts to achieve a concentration of 0.25µg/mL.

8.8 Surrogate Standards: Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl (DCB) are used as surrogates for Aroclor analysis. They are added to the calibration standards at the concentrations listed in Table 1, Continuing Calibration Standards and are spiked into all samples and QC samples prior to extraction.

- **ICAL Surrogates Stock:** is prepared by diluting of 500ul of Pesticides Surrogates Standard Spiking Solution (Ultra, Cat. #ISM-320-1) and 500ul of Decachlorobiphenyl (Accustandard, Cat. #CLP-032-R-01) to 20ml of Hexane to achieve concentration of TCMX at 5ug/ml and DCB at 10ug/ml.
- **CCAL Surrogates Stock:** is prepared by diluting of 1ml of TCMX&DCB (Accustandard, Cat. #CLP-032-R) and 1ml of Decachlorobiphenyl (Accustandard, Cat. #CLP-032-R-01) to 20ml of Hexane to achieve concentration of TCMX at 10ug/ml and DCB at 20ug/ml.
- **Extraction Surrogates Stock:** is prepared by diluting of 10ml of TCMX&DCB (Accustandard, Cat. #CLP-032-R) to 1000ml of Acetone to achieve concentration of TCMX and DCB at 2ug/ml.

8.9 LCS/MS Spiking Solutions: The LCS/MS spiking solution is prepared by diluting of 6.25ml of Arochlor 1016/1260 (Restek, Cat. #32039) to 500ml of Acetone to achieve concentration of Arochlor 1016/1260 at 12.5ug/ml.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A Method Blank is an aliquot of a clean reference matrix (reagent water) that is carried through the entire analytical procedure. Extraction blanks are performed with each extraction batch of 20 or less samples, according to the extraction SOPs. If any analyte of interest is found in the blank at a concentration greater than the MDL for the analyte or at a concentration greater than 1/10th the concentration in a sample in the batch, whichever is greatest, analysis of samples must be halted and samples in the batch must be re-extracted and the extracts re-analyzed. The surrogate recoveries must also be within the acceptance criteria listed in Table 2. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary. The result for each analyte in the blank is reported at or above the MDL to 2 significant figures unless specified otherwise by the regulatory agency or permit.

9.2 Laboratory Control Sample (LCS)

A Laboratory Control Sample (LCS) is extracted with each analytical batch. The LCS consist of an aliquot of a clean (control) matrix and of the same volume. For Aroclor analysis, the LCS is spiked with a mixture of Aroclor 1016 and 1260. The recovery acceptance criteria are listed in Table 2. If any recovery criteria are not met, the extract may be re-analyzed. If the criteria are still not met, the **entire batch is re-extracted**, unless the recoveries are high and the associated samples are non-detect. If this is not possible, due to insufficient sample or holding time exceedances, the analyst must narrate the failure in the LIMS for inclusion in the client report.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.7.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A Matrix Spike and Matrix Spike Duplicate pair is extracted and analyzed with each batch of 20 or less samples. It is a client sample spiked with a mixture of Aroclor 1016 and 1260. The recovery acceptance criteria are listed in Table 2. If the recovery criteria are not met, but are met in the LCS, the failure may be attributed to sample matrix effects.

9.6 Laboratory Duplicate

NA

9.7 Surrogates

All extracted samples and associated QC are spiked with Extraction Surrogates Stock to achieve concentration of TCMX and DCB at **0.5ug/ml.** The laboratory must evaluate surrogate recovery data from individual samples and QC samples versus the surrogate

control limits listed in Table 2. If the surrogate limits are not met, the extract may be reanalyzed to determine if the failure was due to an instrument problem. If the criteria are still not met, the affected samples must be re-extracted to confirm that the failure was due to sample matrix, unless the surrogate recovery is high and the associated sample is non-detect. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

9.8 Method Sequence

Typical Initial calibration (each level identified with the standard lot number)

1. Prime
2. Blank
3. Standard Level 1
4. Standard Level 2
5. Standard Level 3
6. Standard Level 4
7. Standard Level 5
8. Standard Level 6
9. Initial Calibration Verification Standard (ICV)

Repeat steps 3 – 9 as needed for each Aroclor necessary for calibration.

NOTE: If multiple calibration mixtures are analyzed, it is acceptable to analyze appropriate ICVs after all calibration standards have been injected.

Typical Daily Sequence

1.1016/1260 Continuing Calibration Standard - ICV **(identified with the standard lot number)**

2. Extraction Blank

3. Laboratory Control Sample

4. Matrix Spike / Matrix Spike Duplicate

6. Samples up to 16

7. 1016/1260 Continuing Calibration Standard - ICV **(identified with the standard lot number)**

10. Procedure

10.1 Equipment Set-up

10.1.1 GC Conditions:

The dual-column / dual-detector approach involves the use of the columns listed in section 7.2. The columns are connected to an injection tee or dual injection GC, and separate electron capture detectors. Alpha typical GC conditions are listed below, but may be altered as long as method performance criteria are met.

Temperature1: 120 ºC	Injector temerature: 250ºC
Time1: 0 minutes	Injector mode: Pulsed Split
Ramp1: 45ºC/minute	1.4:1 split, 0.20 min pulse
Temperature2: 200ºC	Injector Flow: 5.7 ml/min split flow
Time2: 0 minutes	Detector temperature: 350ºC
Ramp2: 15ºC/minute	Carrier gas: Helium
Temperature3: 230ºC	Carrier flow: 20ml/min
Time3: 0 minutes	Carrier mode: Constant flow
Ramp3: 30ºC/minute	Makeup gas: Argon/methane (P5)
Final temperature 330ºC	Total detector flow: 55ml/min
Final time: 2 minutes	Injection Volume: 1 µL

10.2 Initial Calibration

10.2.1 Prepare calibration standards using the standards listed in Section 8.5 to achieve the concentrations from Table 1.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in section 10.1.1. The same operating conditions are used for calibrations and sample analyses. Create the analytical sequence using the Agilent Chemstation data acquisition software. Record the calibration standard, unique lot number (PP#) and analyst's initials in the analytical sequence list.

10.2.3 A 1µL injection volume of each calibration standard is typically used. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. The same injection volume must be used for all standards and samples.

10.2.4 Column adsorption may be a problem when the GC has not been used for a day or more or after system maintenance. The GC column may be primed (or deactivated) by injecting a PCB standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

Alternately, the system may be primed by baking at the final analytical temperature for approximately 30 minutes.

Several analytes may be observed in the injection just following system priming. Always run an instrument blank after system priming.

10.2.5 **Calibration Factor:** Internal standard calibration techniques are employed in this method.

10.2.5.1 Internal Standard Procedure. In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formula listed in Alpha's Quality Manual.

10.2.6 Initial Calibration Criteria

If the **RSD for an analyte is < 15%**, then the response of the instrument for this compound is considered linear over the range and the mean calibration factor can be used to quantitate sample results.

If the **RSD for any analyte is > 15%**, then linearity through the origin cannot be assumed. The mean response factor cannot be used for quantitation. An alternative calculation may be done by the use of **linear regression** or **quadratic regression** (minimum of six ICAL points are needed and regression must be weighted inversely proportional to concentration) as long as the correlation coefficient is **>0.990**. If both of these quantitation methods fail criteria for any compound in the initial calibration, then the system must be reevaluated and a new calibration curve must be analyzed. .

10.2.7 Initial Calibration Verification

An initial calibration verification standard must be run immediately after each initial calibration, near the midpoint of the curve. The standard must be prepared using a second source that is different than the source used for the initial calibration. (Standards listed in Section 8.6). The **%D** has to be within **25%** (when compared to the mean response factor from the initial calibration).

10.2.8 Retention Time Window

10.2.8.1 The retention time window used for the identification of target analytes is ± 0.05 minutes. These criteria have been adopted from the EPA CLP Statement of Work (OLM04.2). It has been found that these limits work well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results.

10.2.8.2 The windows listed above are used as guidance; however the experience of the analyst weighs heavily in the interpretation of the chromatograms. For example, it has been observed that certain oil matrices can cause the retention times to shift more dramatically.

10.3 Sample Processing

The determination of PCB Aroclors is accomplished by comparing the sample chromatogram to that of the most similar Aroclor standard. The use of PCB overlays is extremely helpful, either by using hardcopies of chromatograms or by utilizing the Enviroquant software. A choice must be made as to which Aroclor is most similar and whether that standard is truly representative of the PCB in the sample. Both retention time and pattern are important when determining PCBs in a sample.

Samples that contained weathered PCB present special analytical challenges. Weathering could alter the Aroclor pattern to the extent that different peaks have to be selected for quantitation. Samples that contained more than one Aroclor present similar problems. For these samples, the analyst may have to consider selecting the earlier eluting peaks for the lower boiling Aroclor and selecting the later eluting peaks for the higher boiling Aroclors to minimize overlapping peaks. A minimum of 3 peaks must be chosen for each Aroclor. In these instances, the analyst may need request the assistance of someone with more expertise in determining the presence of PCB Aroclor.

If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be

needed. If instrument problems are suspected, rerun the extract on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to the extraction SOPs for the procedures to be followed in sample cleanup.

A retention time **shift >30 seconds** for the internal standard necessitates reanalysis of all affected samples. Changes in **Internal Standard** area of more than **50-200%** (when compared to the opening CCAL) must be investigated and all affected samples must to be reanalyzed.

The laboratory must report the ***LOWER*** of the two results unless obvious interference is present on one of the columns.

If any peaks are above the calibration, a dilution must be done. The dilution reported is the least dilute level at which the peak area is within the calibration range.

10.4 Continuing Calibration

10.4.1 Verify calibration each **12-hours** shift by injecting calibration verification standards ***(second source)*** prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than **once every twenty injections**. **A bracketing CCV is required** For Aroclor analysis, the calibration verification standard used is a mixture of Aroclor 1016 and 1260.

10.4.2 The response factor (for internal standard compounds) for each analyte to be quantitated must not exceed a **± 25% difference** when compared to the initial calibration curve. The Enviroquant data processing software automatically calculates the %D for all analytes according to the formulae in Alpha's Quality Manual. A retention time shift >30 seconds for the internal standard necessitates reanalysis of all affected samples. Changes in **Internal Standard** area of more than **50-150%** (when compared to the mean IS area from Initial Calibration) must to be investigated and all affected samples must to be reanalyzed.

10.5 Preventive Maintenance

10.5.1 **Preventive Maintenance:** Routine preventive maintenance is performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and replacement of injector seals.

10.5.2 **Other Maintenance:** ECD detectors may become contaminated, requiring bake out at elevated temperatures, (no greater than 375C) or repair by the manufacturer.

11. Data Evaluation, Calculations and Reporting

11.1 Quantitation of Aroclors

Per Method 608.3, quantitation is based on the use of a minimum of 3 of the major peaks present in the analyte, although the use of 5 of the major peaks is recommended. Each of these peaks is individually calibrated with a **minimum of three calibration points** based on average response factors. The %RSD must meet the criteria of ≤15% for the ICAL. The three to five major peaks are calculated as described below. After individual calculation

meets criteria, the average of the peaks selected for quantitation is used to determine the final concentration.

11.1.1 Aqueous samples

$$\text{Concentration } (\mu g/L) = \frac{C \times DF \times Vf \times 1000}{Vo}$$

where:
C = Extract concentration (µg/L)
DF = Dilution factor
Vf = Final extract volume (mL)
Vo = Sample volume (mL)

11.1.2 Reporting Results

11.1.2.1 After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews must be done by two separate individuals.

11.1.2.2 Summation Rules

"TOTAL" concentrations are calculated for **ALL samples and Quality Control Samples** (i.e. LCS, MS, DUP, BLK).

TOTAL = sum of "reportable" Aroclors

Reportable- all Aroclors reported for associated project.

For dual-column analysis, Total is reported as part of column "A" data, unless all individuals are reported from "B" column. "Total" is calculated based on the associated "Report List". See Work Instruction #14335 for details.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and/or improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written into the LIMS by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method

.

13. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

14. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

15. Attachments

Table 1: STANDARD SOLUTIONS

Table 2: QC ACCEPTANCE CRITERIA

TABLE 1
STANDARD SOLUTIONS

STANDARD SOLUTIONS	Stock solution (ug/mL)	Level 1 (ug/mL)	Level 2 (ug/mL)	Level 3 (ug/mL)	Level 4 (ug/mL)	Level 5 (ug/mL)	Level 6 (ug/mL)	Spike Solution (ug/mL)	LCS Solution (ug/mL)
PCB									
Aroclor 1016/1260	100	0.1	0.5	1	2.5	5	10	12.5	12.5
Aroclors 1221, 1232, 1242, 1254, 1262, 1268	100	0.1	0.5	1	2.5	5	10		
Internal Standard									
1-Bromo-2-Nitrobenzene	5000	0.25	0.25	0.25	0.25	0.25	0.25		
Surrogates:									
Tetrachloro-m-xylene	2.0	0.0064	0.032	0.064	0.16	0.32	0.64	2	2
Decachlorobiphenyl	2.0	0.0126	0.064	0.128	0.32	0.64	1.28	2	2

TABLE 2

QC ACCEPTANCE CRITERIA

Surrogate % Recovery*	Lower Control Limit	Upper Control Limit
2,4,5,6-Tetrachloro-m-xylene	37%	123%
Decachlorobiphenyl	38%	114%

*One surrogate on one column is allowed to be outside of the control limits and still be within criteria.

LCS & MS/MSD	Lower Control Limit	Upper Control Limit	MS/MSD RPD %
Aroclor 1016	50%	140%	36
Aroclor 1260	8%	140%	38

Semivolatile Organics by GC/MS EPA 625.1

Reference: EPA 625.1 Base/Neutrals and Acids by GC/MS. Appendix A, Part 136, Code of Federal Regulations. August 28, 2017 edition.

1. Scope and Application

Matrices: This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from aqueous samples.

Definitions: Refer to Alpha Analytical Quality Manual.

This method is used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone.

The following compounds may require special treatment when being determined by this method:

- Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- n-Nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.
- Pentachlorophenol, 2,4-dinitrophenol, nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The samples are introduced into the GC/MS by injecting 1µL of the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.
Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of standards run on the same GC/MS system. Quantitation is accomplished by comparing the response of the quantitation ion relative to an internal standard using a calibration curve.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 6 lists routine reporting limits.

4. Interferences

4.1 Instrumental

4.1.1 Only high purity helium is used in the GC system to eliminate this source of possible contamination. The helium (carrier gas) is certified by the gas supplier.

4.1.2 Preventive instrument maintenance is performed routinely, and whenever highly contaminated extracts are analyzed that could result in chromatographic interferences or result in degradation of system performance. Section 10.5 details the maintenance steps.

4.2 Parameters

4.2.1 Glassware must be scrupulously cleaned. This procedure is detailed in the Organic Extraction Glassware Cleaning & Handling SOP/1953. Store dry glassware in a clean environment.

4.2.2 Contaminated solvents or reagents are also possible sources or contamination. All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free.

4.2.3 Contamination by carry-over can occur whenever high-concentration and low-concentration samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered (concentrations greater than 2x the highest concentration) and the next sample has reportable hits this sample should to be re-analyzed for confirmation based on analyst discretion.

4.2.4 Matrix interferences may be caused by contaminants co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. Interferences extracted from sample high in total organic carbon (TOC) may result in elevated baselines, or by enhancing or suppressing a signal at or near the retention time of an analyte of interest. Analyses of the matrix spike and matrix spike duplicate may be useful in identifying matrix interferences.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC/MS laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab for future disposal by the Health and Safety Officer. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Flammable solvent bottles must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in two 1L amber glass jars with teflon-lined lids. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Samples are preserved by packing in coolers with ice or ice packs, to maintain a temperature of ≤ 6ºC. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of ≤ 6ºC.

6.3 Sample Shipping

No specific requirement.

6.4 Sample Handling

Samples must be extracted within 7 days of sample collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph/Mass Spectrometer System:

7.1.1 **Gas Chromatograph:** An analytical system complete with a temperature-programmable gas chromatograph configured for split/splitless-injection and all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source.

7.1.2 **Column:** 30m x 0.32mm ID, 0.25 µm film thickness silicone-coated, fused-silica capillary column (RXi-5Sil MS w/5m Integra Guard, Restek), or equivalent.

7.1.3 **Mass Spectrometer:** Scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 1 when 1 µL of the GC/MS tuning standard is injected through the GC (50ng of DFTPP).

7.1.4 **Data System:** A computer system is interfaced to the Mass Spectrometer. The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software allows the analyst to search any GC/MS data file for ions of specific mass and plot such ion abundances versus time. *HP ChemServer* software is used for data acquisition and *Enviroquant MSD Chemstation version E.02.02* is used for data reduction.

7.2 Syringes: 10 µL – 1mL.

7.3 Volumetric Flasks, Class A: Appropriate sizes with ground-glass stoppers.

7.4 Vials: Glass autosampler vials with polytetrafluoroethylene (PTFE)-lined crimp top caps

8. Reagents and Standards

8.1 Stock Standard Solutions

Certified stock standard solutions, traceable to NIST, when available, are purchased from commercial vendors. They can be replaced with different standards as long as they contain all target analytes.

All stock standards, lot number, catalog number, expiration date, preparation date and initials are recorded in a logbook. Standards are stored in the refrigerator or freezer.

Stock standard expire 6 months from the date of preparation or on the earliest expiration date of any of the stock solution used to prepare it.

Please note that the following preparation instructions and stock standards are included for illustration purposes and may be modified as needed (ex. to accommodate standard availability or client requests), however final concentrations for the initial calibration levels shall always follow the example in 8.1.4.

Vendor	Standard	Catalog#	Concentration
Restek			
	8270 Mega Mix	31850	500-1000ug/mL
	Benzoic Acid Mix	31879	2000ug/mL
	Acid Surrogate Mix	31087	10000ug/mL
	B/N Surrogate Mix	31086	5000ug/mL
	Benzaldehyde Standard	33017	2000ug/mL
	Custom AP9 ICAL Standard	571813-FL	2000ug/mL
	Custom ADP Standard	572745-FL	2000ug/mL
	Benzidine Mix	31834	2000ug/mL
	SV Internal Standard Mix	31206	2000ug/mL
	Custom CLP 04.1 BNA Surrogate Mix	571320-FL	1000ug/mL
Ultra			
	Semi-Volatiles GC/MS Tuning Standard	GCM-150-1	1000ug/mL
Accustandard			
	Demeton (Mixed Isomers)	M-622-05	1000ug/mL

8.1.1 ABN Stock Standard, 200ug/mL

Use 5mL of each of the following:
- Benzoic Acid Mix
- Benzidine Mix

and use 10mL of each of the following:
- 8270 Mega Mix
- Custom CLP 04.1 BNA Surrogate Mix

Bring up to 50mL volume with DCM.

8.1.2 AP9 Additional Compounds Stock Standard, 200ug/mL

Use 5mL of each of the following:
Custom AP9 ICAL Standard
Benzaldehyde Standard

Bring up to 50mL volume with DCM.

8.1.3 ADP Stock Standard, 200ug/ml

Use 5ml of:
Custom ADP Standard

Bring up to 50mL volume with DCM.

8.1.4 Calibration Standard

A minimum of 5 calibration standards for each analyte

Level	***Concentration (ug/ml)***
L1	1
L2	2
L3	3
L4	5
L5	10
L6	20
L7	50
L8	100
L9	150
L10	200

8.2 Internal Standard Solution

The internal standards are:
1,4-dichlorobenzene-d_4
naphthalene-d_8
acenaphthene-d_{10}
phenanthrene-d_{10}
chrysene-d_{12}
perylene-d_{12}

Each 500µL of standards, blank and sample extracts are spiked with 10µL of SV Internal Standard Mix, resulting in a concentration of 40ng/ µL.

8.3 GC/MS Tuning Standard

The tuning standard is a methylene chloride solution containing 50ng/µL of decafluorotriphenylphosphine (DFTPP). The standard also contains 50ng/µL each of 4,4'DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance.

Prepare the GC/MS Tuning Standard with 25µL GCM-150 and 475µL Dichloromethane.

8.4 Surrogate Spiking Solution

8.4.1 Extraction Surrogate Preparation

In a 1000mL volumetric flask, add 5ml of 31086 and 31087. Bring up to volume with Acetone. The final concentration is 50µg/mL for the acid surrogates and 25µg/mL for the B/N surrogates.

8.5 Spike Solution (LCS, MS, MSD)

Spike Solution Preparation

ABN SPK1:
In a 500ml volumetric flask, add 20ml of 8270 Mega Mix #31850, 10ml of Benzoic Acid Mix #31879, 10ml Custom AP9 ICAL Standard #571813-FL and 10ml Benzaldehyde Standard #33017. Bring up to volume with Acetone. The final concentration is 40ug/ml.

ABN SPK2:
In a 500ml volumetric flask, add 10ml Benzidine Mix #31834 and 10mL Custom ADP Standard #572945-FL. Bring up to volume with Acetone. The final concentration is 40ug/ml.

8.6 Dichloromethane (DCM): Pesticide quality.

8.7 Acetone: Pesticide quality.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

Extraction blanks are performed with each extraction batch of 20 or less samples, according to the extraction SOPs. If any analytes are detected in the blank at concentrations greater than the MDL for that analyte, but less than the RL, those results shall be reported to 2 significant figures.

The extraction blank must not contain any of the reportable analytes above the reporting limit. If any reportable analytes are detected in the blank but those analytes are not detected in any associated samples, data can be accepted and a narrative shall be written. If detected analytes in blank are also detected in sample, the entire extraction batch is suspect and re-extraction of all associated samples is required. The surrogate recoveries must also be within the

acceptance criteria listed in section 9.7.2. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary.

9.2 Laboratory Control Sample (LCS)

A Laboratory Control Sample (LCS) is extracted with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The spike compounds and levels are listed in Section 8.5. The recovery acceptance criteria are listed in Table 3. If any recovery criteria are not met, the extract must be reanalyzed. If the criteria are still not met, the entire batch must be re-extracted. If this is not possible, due to insufficient sample or holding time exceedence, the analyst must write up the failure on a narrative sheet for inclusion in the client report. If any of analytes are recovered above control limits, this is deemed acceptable as long as the analytes in question are not detected in associated samples.

9.3 Initial Calibration Verification (ICV)

9.4 Continuing Calibration Verification (CCV)

9.5 Matrix Spike

A matrix spike and its duplicate are extracted and analyzed for each batch of 20 or less samples. The spike compounds and levels are listed in Section 8.5. The recovery acceptance criteria are listed in Table 3. If the recovery criteria are not met, but are met in the LCS, this must be noted on a narrative sheet for inclusion in the client report.

9.6 Laboratory Duplicate

Laboratory Duplicate is typically a Matrix Spike Duplicate. See Table 3.

9.7 Method-specific Quality Control Samples

9.7.1 Internal Standard Response

The area responses of the internal standards in each sample and all associated QC must be within 50% to 200% of that in the mid-point standard level of the most recent initial calibration sequence. If this criteria is not met, repeat the CCV and re-analyze the affected samples/QC. If the IS responses are still outside acceptance limits, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

Note that if there are no analytes detected in the sample, the data may be accepted.

9.7.2 Surrogates

All extracted samples and associated QC are spiked with surrogate at the levels listed in Section 8.4. The laboratory must evaluate surrogate recovery data from individual samples and QC samples versus the surrogate control limits listed in Table 2.

Corrective action: Analysis must be repeated once to see if an analytical error has occurred. If the % recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

Re-extraction is not required if surrogate recoveries are high and target analytes are not detected in the sample. Re-extraction is also not required if a surrogate is outside of method acceptance criteria, but the analytes associated with that

surrogate are not requested by the client. Refer to Table 7 to see the IS/Surrogate/Analyte groupings. Both of the above-mentioned scenarios require that a narrative be included in the report.

9.8 Method Sequence

In a 15-hour period, the typical analytical sequence is:

- Degradation Check
- DFTPP
- Continuing or Daily Standards (1 – 2)
  - (1) ABN 50ppm
  - (2) AP9 50ppm
  - (3) ADP 50ppm
- LCS
- Method Blank
- Samples
- QC (as required)

10. Procedure

10.1 Equipment Set-up

10.1.1 GC/MS Operating Conditions:

Typical GC/MS operating conditions are listed below, but may be altered as long as method performance criteria are met.

Mass range:	35 – 500 amu
Scan time:	3.15 scans/second
Initial temperature:	50°C, hold for 1.5 minutes
Temperature program:	28°C/minute to 250°C then 9°C/minute to 320°C
Final temperature:	320°C for 0.58 min
Injector temperature:	300°C
Transfer line temperature:	280°C
Source temperature:	230ºC
Injector:	split ratio 5:1, 11.7mL/min
Injection volume:	1µL
Carrier gas:	helium at 523 cm/second (2.0 mL/min) constant flow

After achieving the key ion abundance criteria for DFTPP, calibrate or verify the calibration of the system daily as described in Sections 10.2 and 10.4. If performance criteria are not achieved, take corrective action as defined in Section 12.

10.1.2 GC/MS Tune:

At the beginning of every each analytical sequence, analyze the 50µg/L DFTPP tuning solution (Section 8.3). This standard is the start of the 15-hour clock.

The resultant mass spectrum for DFTPP must meet the criteria given in Table 1 before sample analysis begins. The mass spectrum of DFTPP should be acquired in the following manner:

(1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.

(2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of DFTPP.

The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Benzidine and pentachlorophenol must be present at their normal responses and no peak tailing must be visible.

The tailing factor for benzidine and pentachlorophenol must be calculated in every DFTPP run. (See Table 4)

10.2 Initial Calibration

10.2.1 Prepare calibration standards for all target analytes at the ten concentration levels specified in Section 8.1.4. Note that a minimum of five concentration levels must be used for a valid linear calibration for each analyte. A minimum of six levels must be used for a non-linear curve (ex. Quadratic regression) to be valid.

10.2.2 Add 10µL of Internal Standard to each calibration standard directly into the autosampler vial containing 500µL of standard. Analyze each calibration standard according to Section 10.1.1.

10.2.3 Record the calibration standard, unique lab identifier code (lot), concentration, and analyst's initials in the analytical sequence list.

10.2.4 In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formulae listed in Alpha's Quality Manual.

Some of the target analytes (2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitrocresol and hexachlorocyclopentadiene) have a tendency to decrease in response as the chromatographic system begins to deteriorate or standard materials begin to deteriorate.

They are usually the first to show poor performance, therefore they must be monitored as indicators of degrading system performance.

10.2.5 Initial Calibration %RSD Criteria:

For all analytes, including the compounds listed above, the RSD must be $\leq$ 35% for the mean response factor to be used for sample quantitation. If the RSD is > 35% an alternate calculation fits may be performed provided that the minimum coefficient of determination (r^2) $\geq$ 0.99 is met.

10.2.6 Evaluation of Retention Times:

The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units.

10.2.7 Initial Calibration Verification (Second Source Verification)

The standards used for the Initial Calibration Verification, or secondary source, (except surrogates) must be of a different production lot than those used to prepare the initial calibration standards.

10.2.7.1 The initial calibration (Section 10.2) for each compound of interest must be verified prior to sample analysis. This is accomplished by analyzing second source calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

10.2.7.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

10.2.7.3 If the % Difference for each analyte is ≤ 30%, then the calibration is assumed to be valid. If this criterion is not met for any analytes, refer to EPA Method 625.1, Table 6, column Q for criteria for those analytes. If the analytes are acceptable by EPA's criteria, the calibration is valid and can be used. If not, then corrective action must be taken prior to the analysis.

10.3 Equipment Operation and Sample Processing

10.3.1 GC/MS Analysis of Samples

10.3.1.1 Allow the sample extracts to warm to room temperature.

10.3.1.2 Add 10µL of the internal standard (Section 8.2) to the 500µL of sample extract.

10.3.1.3 The autosampler is programmed to inject 1µL aliquot of the sample extract into the GC/MS system, using the same instrument conditions that were used for calibration (Section 10.1.1). The injection volume of the sample must be the same as the volume used for the calibration standard.

10.3.1.4 If the response of any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed.

10.3.2 Qualitative Identification

Perform first level data review. Obtain the primary m/z (Table 4) masses for each parameter of interest. The following criteria must be met to make qualitative identification:

- Compare the background subtracted mass spectra for the sample to the reference spectra. The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.
- The retention time must fall within ± 0.1 minutes of the retention time of the compound in the analytical standard. However, analyst experience must be used in making the qualitative identification.
- The relative peak height of the one characteristic mass must fall within 20% of the relative intensity of the mass in a reference mass spectrum. The reference spectrum is obtained from a standard analyzed on the GC/MS system.

Structural isomers that have very similar mass spectra are identified only if the resolution between authentic isomers in a standard mix is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

10.4 Continuing Calibration

10.4.1 Continuing calibration verification is performed at the beginning of each analytical sequence. This is accomplished by analyzing calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The standards used for the CCV must be a second source, different from those used for the calibration standards.

10.4.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

In order to determine if a calibration is valid, the %difference for each analyte must be verified using criteria listed in EPA Method 625.1, Table 6, column Q. If criteria for an analyte is not listed in Table 6, column Q, the %difference criteria is ≤ 40%. If the %

difference for each analyte has met this specified criteria, the calibration is assumed to be valid and can be used. If not, then corrective action must be taken prior to the analysis.

10.4.3 If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.4.4 Allowances may be made for a RF that drifts out high, as long as there are no positive hits for that particular analyte in any of the associated samples.

10.4.5 Internal Standard Retention Time

The retention times of the internal standards in the calibration verification standard is evaluated after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard of the most recent initial calibration, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.4.6 Internal Standard Response

If the area for any of the internal standards in the calibration verification standard changes by a factor of two (50% to 200%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.5 Preventive Maintenance

When poor sensitivity is observed, replacement of the injector liner and seal may solve the problem. If not, clip approximately 3 – 6 inches from the injector end of the GC column. If the sensitivity does not improve it may be necessary to replace the split line or the injector weldment assembly. If the problem persists, it may be necessary to replace the GC column.

Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

11. Data Evaluation, Calculations and Reporting

11.1 When a parameter is identified, the quantitation of that parameter must be based on the integrated abundance of the quantitation characteristic m/z given in Table 5. If the sample produces an interference for the primary m/z, use a secondary characteristic m/z to quantitate.

11.2 Calculate the concentration in the sample using the average response factor (RF) from the initial calibration curve according to the formulae in Alpha's Quality Manual.

$$\text{Concentration } (\mu g/L) = \frac{C \times DF \times Vf \times 1000}{Vo}$$

where:

C = Extract concentration (µg/mL)
DF = Dilution factor
Vf = Final extract volume (mL)
Vo = Sample volume (mL)

11.3 Results for positive hits in samples are reported in µg/L units. After performing technical data review, validating that all QC criteria have been met and confirming all positive hits,

the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews must be done by two separate individuals.

11.4 Results are reported at the lowest dilution factor possible to be within the calibration range, and MS/MSD recovery and RPD are within acceptance criteria (Table 3.) This may require reporting results for some analytes from different runs.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in section 9 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

Analyze four QC check samples spiked with all analytes at 10 – 50 times the MDL.
Calculate the result for each aliquot in µg/L, the relative standard deviation of the four results, and the average percent recovery for each analyte.

The majority of analytes must meet 70-130% acceptance criteria, with the exception of the following analytes, which must meet 70-120% acceptance criteria:

- Acenaphthylene
- Anthracene
- Bis(2-chloroethyl)ether
- 4-Bromophenyl phenyl ether
- 2-Chloronaphthalene
- Di-n-butyl phthalate
- Diethyl phthalate
- Dimethyl phthalate
- 2,4-Dinitrotoluene
- Fluoranthene

- Fluorene
- Hexachlorobutadiene
- Hexachloroethane
- Naphthalene
- Phenanthrene
- Pyrene
- 2-Chlorophenol
- 2,4-Dichlorophenol
- 2,4-Dimethylphenol
- 4-Nitrophenol
- Phenol

Refer to EPA Method 625.1, Table 6, columns "Limit for s" and "Range for X" for analytes that do not pass the above lab-assigned criteria. If those analytes meet EPA criteria, the DOC is acceptable.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

1953 Organic Glassware Cleaning & Handling

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

16. Attachments

Table 1: DFTPP Key Ions and Ion Abundance Criteria

Table 2: Acceptable Surrogate Spike Recovery Limits

Table 3: Acceptable LCS and Matrix Spike Recovery Limits

Table 4: Tailing Factor Calculation

Table 5: Characteristic Ions for Semivolatile Compounds

Table 6: Reported Detection Limits

Table 7: Semivolatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

TABLE 1

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	40-100% of mass 198
443	17-23% of mass 442

TABLE 2

ACCEPTABLE SURROGATE SPIKE RECOVERY LIMITS

Surrogates	Acceptance Limits (%)
2,4,6-Tribromophenol	45-128
2-Fluorobiphenyl	46-121
2-Fluorophenol	25-87
4-Terphenyl-d14	47-138
Nitrobenzene-d5	42-122
Phenol-d6	16-65

TABLE 3

ACCEPTABLE LCS RECOVERY LIMITS

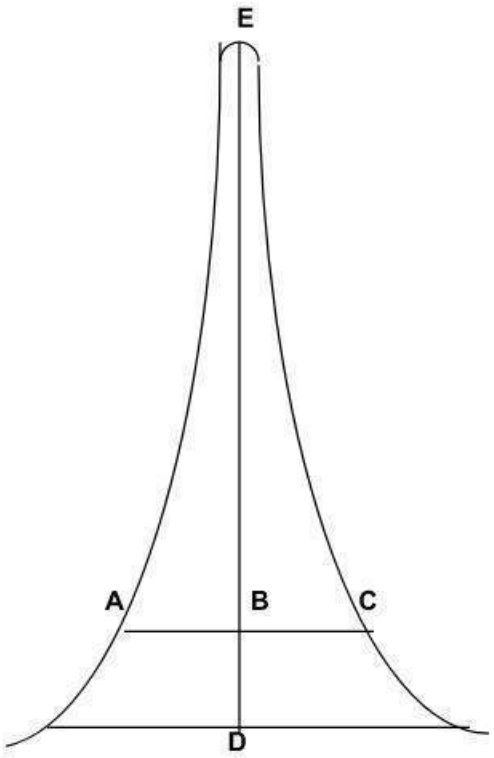
Spike Compound	Acceptance Limits (%)	Spike Compound	Acceptance Limits (%)
1,2,4-Trichlorobenzene	57-130	Benzoic Acid	2-55
1-Methylnaphthalene	40-140	Benzyl Alcohol	31-103
2,4,5-Trichlorophenol	47-126	Bis(2-chloroethoxy)methane	49-165
2,4,6-Trichlorophenol	52-129	Bis(2-chloroethyl)ether	43-126
2,4-Dichlorophenol	53-122	Bis(2-chloroisopropyl)ether	63-139
2,4-Dimethylphenol	42-120	Bis(2-ethylhexyl)phthalate	29-137
2,4-Dinitrophenol	1-173	Butyl benzyl phthalate	1-140
2,4-Dinitrotoluene	48-127	Carbazole	46-114
2,6-Dinitrotoluene	68-137	Chrysene	44-140
2-Chloronaphthalene	65-120	n-Decane	40-140
2-Chlorophenol	36-120	Dibenzo(a,h)anthracene	1-200
2-Methylnaphthalene	40-109	Dibenzofuran	23-126
2-Methylphenol	38-102	Diethyl phthalate	1-120
2-Nitroaniline	43-131	Dimethyl phthalate	1-120
2-Nitrophenol	45-167	Di-n-butylphthalate	8-120
3,3'-Dichlorobenzidine	8-213	Di-n-octylphthalate	19-132
3-Methylphenol/4-Methylphenol	35-103	Fluoranthene	43-121
3-Nitroaniline	27-98	Fluorene	70-120
4,6-Dinitro-o-cresol	56-130	Hexachlorobenzene	8-142
4-Bromophenyl phenyl ether	70-130	Hexachlorobutadiene	38-120
4-Chloroaniline	10-100	Hexachlorocyclopentadiene	7-118
4-Chlorophenyl phenyl ether	38-145	Hexachloroethane	55-120
4-Nitroaniline	41-112	Indeno(1,2,3-cd)pyrene	1-151
4-Nitrophenol	13-139	Isophorone	48-180
Acenaphthene	60-132	Naphthalene	36-120
Acenaphthylene	54-126	NDPA/DPA	45-112
Acetophenone	46-113	Nitrobenzene	54-158
Aniline	1-75	n-Nitrosodimethylamine	15-68
Anthracene	43-120	n-Nitrosodi-n-propylamine	14-198
Azobenzene	44-115	n-Octadecane	40-140
Benzidine	0-70	p-Chloro-m-cresol	41-128
Benzo(a)anthracene	42-133	Pentachlorophenol	38-152
Benzo(a)pyrene	32-148	Phenanthrene	65-120
Benzo(b)fluoranthene	42-140	Phenol	17-120
Benzo(ghi)perylene	1-195	Pyrene	70-120
Benzo(k)fluoranthene	25-146		

TABLE 3 (continued)

ACCEPTABLE MS/MSD RECOVERY LIMITS

Spike Compound	Acceptance Limits (%)	%RPD	Spike Compound	Acceptance Limits (%)	%RPD
1,2,4-Trichlorobenzene	44-142	50	Benzoic Acid	2-55	27
1-Methylnaphthalene	40-140	24	Benzyl Alcohol	31-103	23
2,4,5-Trichlorophenol	47-126	28	Bis(2-chloroethoxy)methane	33-184	54
2,4,6-Trichlorophenol	37-144	58	Bis(2-chloroethyl)ether	12-158	108
2,4-Dichlorophenol	39-135	50	Bis(2-chloroisopropyl)ether	36-166	76
2,4-Dimethylphenol	32-120	58	Bis(2-ethylhexyl)phthalate	8-158	82
2,4-Dinitrophenol	1-191	132	Butyl benzyl phthalate	1-152	60
2,4-Dinitrotoluene	39-139	42	Carbazole	46-114	26
2,6-Dinitrotoluene	50-158	48	Chrysene	17-168	87
2-Chloronaphthalene	60-120	24	n-Decane	40-140	21
2-Chlorophenol	23-134	61	Dibenzo(a,h)anthracene	1-227	126
2-Methylnaphthalene	40-109	18	Dibenzofuran	23-126	22
2-Methylphenol	38-102	23	Diethyl phthalate	1-120	100
2-Nitroaniline	43-131	24	Demeton (O & S)	10-140	
2-Nitrophenol	29-182	55	Dimethyl phthalate	1-120	183
3,3'-Dichlorobenzidine	1-262	108	Di-n-butylphthalate	1-120	47
3-Methylphenol/4-Methylphenol	35-103	26	Di-n-octylphthalate	4-146	69
3-Nitroaniline	27-98	39	Fluoranthene	26-137	66
4,6-Dinitro-o-cresol	1-181	203	Fluorene	59-121	38
4-Bromophenyl phenyl ether	53-127	43	Hexachlorobenzene	1-152	55
4-Chloroaniline	10-100	53	Hexachlorobutadiene	24-120	62
4-Chlorophenyl phenyl ether	25-158	61	Hexachlorocyclopentadiene	7-118	35
4-Nitroaniline	41-112	37	Hexachloroethane	40-120	52
4-Nitrophenol	1-132	131	Indeno(1,2,3-cd)pyrene	1-171	99
Acenaphthene	47-145	48	Isophorone	21-196	93
Acenaphthylene	33-145	74	Naphthalene	21-133	65
Acetophenone	46-113	28	NDPA/DPA	45-112	36
Aniline	1-75	66	Nitrobenzene	35-180	62
Anthracene	27-133	66	n-Nitrosodimethylamine	15-68	17
Atrazine	10-140	23	n-Nitrosodi-n-propylamine	1-230	87
Azobenzene	44-115	23	n-Octadecane	40-140	21
Benzidine	0-70	30	p-Chloro-m-cresol	22-147	73
Benzo(a)anthracene	33-143	53	Pentachlorophenol	14-176	86
Benzo(a)pyrene	17-163	72	Phenanthrene	54-120	39
Benzo(b)fluoranthene	24-159	71	Phenol	5-120	64
Benzo(ghi)perylene	1-219	97	Pyrene	52-120	49
Benzo(k)fluoranthene	11-162	63			

TABLE 4



Tailing Factor = $\frac{BC}{AB}$

Example calculation:

Peak Height = DE = 100mm
10% Peak Height = BD = 10mm
Peak Width at 10% Peak Height = AC = 23mm

AB = 11mm
BC = 12mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

Tailing factor for benzidine < 2.0

Tailing factor for pentachlorophenol < 2.0

TABLE 5

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
Acenaphthene	154	153, 152
Acenaphthylene	152	151, 153
Acetophenone	105	120, 51
Aniline	93	66, 65
Anthracene	178	176, 179
Atrazine	200	202, 215
Azobenzene	77	182, 105
Benzidine	184	92, 185
Benzo(a)anthracene	228	229, 226
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Benzoic acid	105	122, 77
Benzyl alcohol	79	77,108
Bis (2-chloroethoxy) methane	93	95, 123
Bis (2-chloroethyl) ether	93	63, 95
Bis (2-chloroisopropyl) ether	45	77, 121
Bis (2-ethylhexyl) phthalate	149	167, 279
4-Bromophenyl phenyl ether	248	250, 141
Butyl Benzyl phthalate	149	91, 206
Carbazole	166	
4-Chloro-3-methylphenol	107	144, 142
4-Chloroaniline	65	127,129
2-Chloronaphthalene	162	127, 164
2-Chlorophenol	128	64, 130
Chrysene	228	226, 229
Dibenzo(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
3,3'-Dichlorobenzidine	252	254, 126
2,4-Dichlorophenol	162	164, 98
Diethyl phthalate	149	177, 150
2,4-Dimethylphenol	107	121,122
Dimethyl phthalate	163	194, 164
Demeton (O & S)	88	60, 61, 59
4,6-Dinitro-2-methylphenol	198	51, 105

TABLE 5 (continued)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
2,4-Dinitrophenol	184	107,91
2,6-Dinitrophenol	162	164, 126, 98, 63
2,6-Dinitrotoluene	165	63, 89
2,4-Dinitrotoluene	165	63, 89
Di-n-butyl phthalate	149	150, 104
Di-n-octyl phthalate	149	167, 43
Fluoranthene	202	101, 203
Fluorene	166	165, 167
Hexachlorobenzene	284	142, 249
Hexachlorobutadiene	225	223, 227
Hexachlorocyclopentadiene	237	235, 272
Hexachloroethane	117	201, 199
Hexachloropropene	213	211, 215, 117, 106, 141
Indeno(1,2,3-cd)pyrene	276	138, 227
Isophorone	82	95, 138
2-Methylnaphthalene	142	141
2-Methylphenol	108	107,90
3,4-Methylphenol	108	107,90
4-Methylphenol	107	108, 77, 79, 90
Naphthalene	128	129, 127
NDPA/DPA	169	168, 167
Nitrobenzene	77	123, 65
n-Decane	57	43, 41
n-Octadecane	57	43, 71
N-Nitrosodiethylamine	102	42,44
N-Nitrosodimethylamine	74	42,44
N-Nitrosopyrrolidine	100	41,42
N-Nitrosodi-n-propylamine	70	42, 101, 130
2-Nitroaniline	138	92,65
3-Nitroaniline	138	108, 92
4-Nitroaniline	138	65, 108, 92, 80, 39
2-Nitrophenol	139	109, 65
4-Nitrophenol	65	109, 139
Pentachlorobenzene	250	252, 108, 248, 215, 254
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Phenol	94	65, 66
Pyrene	202	200, 203

TABLE 5 (continued)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
1,2,4-Trichlorobenzene	180	182, 145
2,4,5-Trichlorophenol	196	200,198
2,4,6-Trichlorophenol	196	198, 200
2,3,4,6-Tetrachlorophenol	232	214,179,108,143,218
Acenaphthene-d_{10} (IS)	164	162, 160
Chrysene-d_{12} (IS)	240	120, 236
1,4-Dichlorobenzene-d_4 (IS)	152	150, 115
Naphthalene-d_8 (IS)	136	68
Perylene-d_{12} (IS)	264	260, 265
Phenanthrene-d_{10} (IS)	188	94, 80
2-Fluorobiphenyl (Surrogate)	172	171
2-Fluorophenol (Surrogate)	112	64
Nitrobenzene-d_5 (Surrogate)	82	128, 54
Phenol-d_6 (Surrogate)	99	42, 71
Terphenyl-d_{14} (Surrogate)	244	122, 212
2,4,6-Tribromophenol (Surrogate)	330	62,141

TABLE 6
REPORTING LIMITS
FOR SEMIVOLATILE ORGANIC COMPOUNDS

Analyte	RL (µg/L)
Acenaphthene	5.0
Acenaphthylene	5.0
Acetophenone	5.0
Aniline	20.0
Anthracene	5.0
Atrazine	5.0
Azobenzene	5.0
Benzidine	50.0
Benzo(a)anthracene	5.0
Benzo(b)fluoranthene	5.0
Benzo(k)fluoranthene	5.0
Benzo(ghi)perylene	5.0
Benzo(a)pyrene	5.0
Benzoic acid	50.0
Benzyl alcohol	10.0
Bis(2-chloroethyl)ether	5.0
Bis(2-chloroisopropyl)ether	5.0
Bis(2-chloroethoxy)methane	5.0
Bis(2-ethylhexyl)phthalate	2.0
4-Bromophenyl phenyl ether	5.0
Butyl benzyl phthalate	5.0
Carbazole	5.0
4-Chloroaniline	5.0
p-Chloro-m-cresol	5.0
2-Chloronaphthalene	5.0
2-Chlorophenol	5.0
4-Chlorophenyl phenyl ether	5.0
Chrysene	5.0
m/p-Methylphenol	5.0
o-Methylphenol	5.0
Dibenzo(a,h)anthracene	5.0
Dibenzofuran	5.0
Di-n-butylphthalate	5.0
3,3-Dichlorobenzidine	50.0
2,4-Dichlorophenol	10.0
Diethyl phthalate	5.0
2,4-Dimethylphenol	10.0
Dimethyl phthalate	5.0
Demeton (O & S)	10.0

TABLE 6 (continued)
REPORTING LIMITS
FOR SEMIVOLATILE ORGANIC COMPOUNDS

Analyte	RL (µg/L)
4,6-Dinitro-o-cresol	20.0
2,4-Dinitrophenol	30.0
2,4-Dinitrotoluene	5.0
2,6-Dinitrotoluene	5.0
Di-n-octylphthalate	5.0
Fluoranthene	5.0
Fluorene	5.0
Hexachlorobenzene	5.0
Hexachlorobutadiene	10.0
Hexachlorocyclopentadiene	30.0
Hexachloroethane	5.0
Indeno(1,2,3-cd)pyrene	5.0
Isophorone	5.0
1-Methylnaphthalene	5.0
2-Methylnaphthalene	5.0
Naphthalene	5.0
2-Nitroaniline	5.0
3-Nitroaniline	5.0
4-Nitroaniline	5.0
Nitrobenzene	5.0
2-Nitrophenol	10.0
4-Nitrophenol	10.0
n-Decane	50.0
N-Nitrosodiethylamine	5.0
N-Nitrosodimethylamine	50.0
N-Nitrosodiphenylamine	5.0
N-Nitrosodi-n-propylamine	5.0
N-Nitrosopyrrolidine	5.0
n-Octadecane	50.0
Pentachlorophenol	10.0
Phenanthrene	5.0
Phenol	5.0
Pyrene	5.0
1,2,4-Trichlorobenzene	5.0
2,4,5-Trichlorophenol	5.0
2,4,6-Trichlorophenol	5.0
2,3,4,6-Tetrachlorophenol	5.0

Table 7
Semivolatile Internal Standards with Corresponding
Target Compounds and Surrogates Assigned for Quantitation

1,4-dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
1,2,4-Trichlorobenzene	1,2,4,5-Tetrachlorobenzene	2,3,4,6-Tetrachlorophenol	3,3-Dimethylbenzidine	3,3'-Dichlorobenzidine	Benzo(g,h,i)perylene
1,2-Dichlorobenzene	1,2-Dichlorobenzene	2,3,5,6-Tetrachlorophenol	Anthracene	Benzo(a)Anthracene	Dibenzo(a,h)anthracene
1,3-Dichlorobenzene	1,3-Dichlorobenzene	2,4,6-Tribromophenol, surr	Benzidine	Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene
1,4-Dichlorobenezne	1,4-Diclorobenzene	2,4-Dinitrophenol	Benzyl butyl phthalate	Benzo(b)fluoranthene	
2,4-Dichlorophenol	1-chloror-2-nitrobenzene	2,4-Dinitrotoluene	Carbazole	Benzo(k)fluoranthene	
2,4-Dimethylphenol	1-Methylnapthalene	3-Nitroaniline	Di-n-Butylphthalate	Bis(2-ethylhexyl) phthalate	
2-Chloroaniline	2,4,5-Trichlorphenol	4,6-Dinitro-2-methylphenol	Diphenamid	Chrysene	
2-Chlorophenol	2,4,6-Trichlorophenol	4-Bromophenyl-phenyl ether	Fluoranthene	Di-n-octylphthalate	
2-Fluorophenol, surr	2,6-Dichlorophenol	4-Chlorophenyl-phenyl ether	n-Octadecane		
2-Methylphenol	2,6-Dinitrotoluene	4-Nitroaniline	Parathion		
2-Nitrophenol	2-Chloronaphthalene	4-Nitrophenol	Phenanthrene		
3-Methylphenol / 4-Methylphenol	2-Fluorobiphenyl, surr	Acenaphthene	Pyrene		
Acetophenone	2-Methylnaphthalene	Atrazine	Terphenyl-d14, surr		
Aniline	2-Nitroaniline	Azobenzene			
Benzaldehyde	3-Choloroaniline	Dibenzofuran			
Benzyl Alcohol	4-Chloro-3-Methylphenol	Dichloran			
Bis(2-chloroethoxy)methane	4-Chloroaniline	Diethyl phthalate			
Bis(2-chloroethyl)ether	Acenaphthylene	Fluorene			
bis(2-Chloroisopropyl)ether	a-Terpineol	Hexachlorobenzene			
Hexachloroethane	Benzoic Acid	NDPA/DPA			
Isophorone	Biphenyl	Pentachloronitrobenzene			

Table 7 (cont.)
Semivolatile Internal Standards with Corresponding
Target Compounds and Surrogates Assigned for Quantitation

1,4-dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
m-Toluidine	Caprolactam	Pentachlorophenol			
n-Decane	Dimethyl Phthalate	Demeton (O & S)			
Nitrobenzene	Hexachlorobutadiene				
Nitrobenzene-d5, surr	Hexachlorocyclopentadiene				
N-Nitrosodiethylamine					
N-Nitrosodimethylamine	Naphthalene				
N-Nitrosodi-n-propylamine					
n-Nitrosopyrrolidine					
Phenol					
Phenol-d6, surr					
Pyridine					

Ignitability of Solids

Reference Method: **Method 1030**, Rev. 0, December 1996 (Modified)

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1997.

1. Scope and Application

Matrices: Solids including pastes, powders, granular material, soils, clay and solids that can be cut into strips.

Definitions: See Alpha Laboratories Quality Manual Appendix A

This method may be used to meet certain regulatory applications; with respect to the characteristic of ignitability in CFR 261.21, this method may be used, but is not required, to determine whether a solid waste "when ignited, burns so vigorously and persistently that is creates a hazard." If it is impractical to perform the test because the physical form of the sample, generator knowledge should be used to determine the ignitability hazard posed by the material.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by completing the record of training.

2. Summary of Method

In a preliminary test, the test material is formed into an unbroken strip or powder train 250mm in length. An ignition source is applied to one end of the test material to determine whether combustion will propagate along 200mm of the strip within a specified time period. Materials that propagate burning along a 200mm strip within the specified time period are then subjected to a burning rate test. Materials that do not ignite or propagate combustion as described above do not require further testing.

In the burning rate test, the burning time is measured over a distance of 100mm and the rate of burning is determined. Lab doesn't perform Burning rate test and will proceed to Flashpoint analysis by Flash test EPA 1010. SOP 2227

2.1 Method Modifications from Reference

A high temperature gas torch is utilized as the flame source. Lab is using modified method. Lab doesn't proceed to the burning rate test if sample ignites during preliminary test.

3. Reporting Limits

There is no reporting limit for this analysis. Results are reported as either Negative or Positive. Refer to Section 11 for data reporting.

4. Interferences

Particle size of test material can affect not only the burning rate, but also the ignition of the material. Therefore particle size of the test material should be the same for each test run. Report the particle size of the test material in a simple descriptive format (e.g. fine powder, sand, coarse granular).

Temperature of some test material such as sulfur powder affects the burning rate. For reproducible results, all tests must be performed at approximately the same initial temperature (ambient room or laboratory temperature).

All tests must be carried out inside a fume hood with the test apparatus situated perpendicular (90°) to the direction of the airflow. Airflow parallel (0°) to the test apparatus results in non-reproducible burning rates.

The rate of airflow through the fume hood affects the burning rate. Too high an airflow will distort the flame and retard its horizontal propagation. The optimum airflow is within the range of 0.7 – 1 meter per second.

Materials that are moisture sensitive (i.e. readily absorb moisture from the air) should be tested as quickly as possible after removal from the sample container. All materials should be tested as received by the laboratory.

5. Health and Safety

Each sample should be treated as a potential health hazard. From this viewpoint, exposure must be reduced to the lowest possible level by whatever means available. References to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. These practices include, but are not limited to, the use of: laboratory coat, safety glasses, and latex or nitrile gloves, and/or heat resistant gloves. Use caution when operating the high temperature torch. Also use caution in handling the ceramic plate after heating the sample, as it remains hot.

Prior to starting the preliminary test, all sample materials must be tested to determine if that material is explosive or extremely flammable. Use a very small portion of material (1 gram or less). ***If the sample displays explosivity or extreme flammability, do not continue this test.***

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Sample containers should be completely filled and tightly sealed to preserve sample integrity.

6.2 Sample Preservation

No sample preservation is required.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

All samples are tested as received unless requested otherwise. Sample aliquots should be tested as soon as possible after removal from the sample container (i.e. samples must not be allowed to dry or absorb moisture for excessive periods or to lose volatiles). Allow samples to equilibrate to the ambient laboratory temperature in the sample container.

7. Equipment and Supplies

7.1 Ceramic Tile: Low heat conducting, non-combustible and impervious with marks indicating 80mm, 180mm, 200mm and 250mm along the test path. Any material capable to withstand high temperatures that have marks indicating 80mm, 180mm, 200mm and 250mm can be used.

7.2 Powder Train Mold: For molding powdered and granular materials for the burn rate test. The material of construction can be aluminum, brass, stainless steel or plastic. The mold is 250mm in length and has a triangular cross-section, with a width of 20mm, and a depth of 10mm as measured from the bottom of the triangular opening to where the sides meet. On both sides of the mold, in the longitudinal direction, two sheets are mounted as lateral limitations that extend 2mm beyond the upper edge of the triangular cross-section.

7.3 High temperature gas torch: A 6 to 7cm flame, with a minimum diameter of 5mm capable of attaining a temperature of at least 1000 °C. MAPP gas is its fuel source.

7.4 Stop watch.

7.5 Thermocouple: To measure the temperature of the gas flame

7.6 Thermometer: To measure initial temperature of material (i.e. room temperature).

7.7 Anemometer: To measure airflow in the fume hood.

7.8 Fume Hood.

8. Reagents and Standards

No special reagents are required to conduct this test.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Not applicable.

9.2 Laboratory Control Sample (LCS)

Not applicable.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

Not applicable

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Perform preliminary screening test:
  - Prepare test strip or powder train
  - Light flame and measure temperature
    - If it does not burn within specified time, sample is considered a negative, nonflammable solid. Test is complete.
    - If it does burn within specified time, sample is considered Positive for the preliminary screening test. Stop analysis.
- Contact supervisor or team leader; in order to switch method from Ignitability to Flash point.
- Refer to SOP 2227 for Flash point analysis

10. Procedure

10.1 Equipment Set-up

Safety Test: Prior to starting the preliminary test, all sample materials must be tested to determine if that material is explosive or extremely flammable. Use a very small portion of material (1 gram or less). ***If the sample displays explosivity or extreme flammability, do not continue this test.***

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Preliminary Screening Test

10.3.1.1 The preliminary ignitability test is conducted on all waste materials.

10.3.1.2 Place a platform (brick, stone, or other flame-resistant tile) in a fume hood about 20cm (or 8 inches) from the front of the hood in an area of laminar airflow. Position the sample perpendicular to the airflow. The airflow across the perpendicular axis of the sample should be sufficient to prevent fumes from escaping into the laboratory and should not be varied during the test. The air velocity should be approximately 0.7 meters/second. Measure and record the air velocity by using the anemometer (Section 7.7).

10.3.1.3 On the platform, prepare the test material in its “as received” form by forming an unbroken strip or powder train of sample 250mm long by 20mm wide by 10mm high on the platform. Position the sample perpendicular to the airflow. Use the mold (Section 7.3) to form the material as in Section 10.3.2.1 if appropriate.

10.3.1.4 Light the gas torch. Measure and record the temperature of the flame (tip of the flame) by a thermocouple (Section 7.6). The temperature of the flame must be at least 1000 °C.

10.3.1.5 Apply the tip of the flame to one end of the sample strip. Measure the propagation of combustion with the ceramic tile (Section 7.1). The test period will depend on the sample matrix as follows:

10.3.1.5.1 **Non-Metallic Waste, Soils, Clays:** Hold the flame tip on the sample strip until the sample ignites or for a maximum of 2 minutes. If combustion occurs, begin timing with a stopwatch and note whether the combustion propagates up to the 200mm mark within the 2-minute test period.

10.3.1.5.2 **Metal or Alloy Powders:** Hold the flame tip on the sample strip until the sample ignites or for a maximum of 5 minutes. If combustion occurs, begin timing with a stopwatch and note whether the combustion propagates up to the 200mm mark within the 20-minute test period.

10.3.1.6 If waste does not ignite and propagate combustion by either burning with open flame or by smoldering along the 200mm of sample strip within the 2 minute test period (or 20 minute test period for metal powders), the waste is not considered flammable and no further testing is required.

10.3.1.7 If the waste propagates burning of 200mm of the test strip within the 2-minute test period (20 minute test period for metals), test should be stopped and analysis should be switched to Flashpoint test (SOP 2227)

10.4 Continuing Calibration

Not applicable.

10.5 Preventive Maintenance

The ceramic plate and train mold must be cleaned after each sample.

11. Data Evaluation, Calculations and Reporting

Include in the log book the following information for each sample:

Test Material Information:

- Date of test:
- Description of material: e.g. powder or paste, metallic or non-metallic
- Particle size: e.g. fine powder, granular, sand, etc.
- If the preliminary screening test (Section10.3.1) was negative, the result for the sample is Not Ignitable (reported as “NI”) and no further data is required.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the Sample Delivery Group Form and/or Nonconformance Report form. Project manager also have to be notified.

If sample cannot be analyzed using Ignitability method, project manager has to be notified and customer has to be contacted. If matrix of the sample is not appropriate for Ignitability analysis then LIMS product may be changed to Flashpoint product in order to provide customer with accurate data. Project manager/customer have to be notified and project manager has to approve this change.

Supervisor and project manager have to be notified if there is not a sufficient amount of sample provided for analysis.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

This study is not applicable to this method.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

These studies are not applicable to this method.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 MDL/LOD/LOQ Generation

1739 IDC/DOC Generation

1728 Waste Management and Disposal SOP

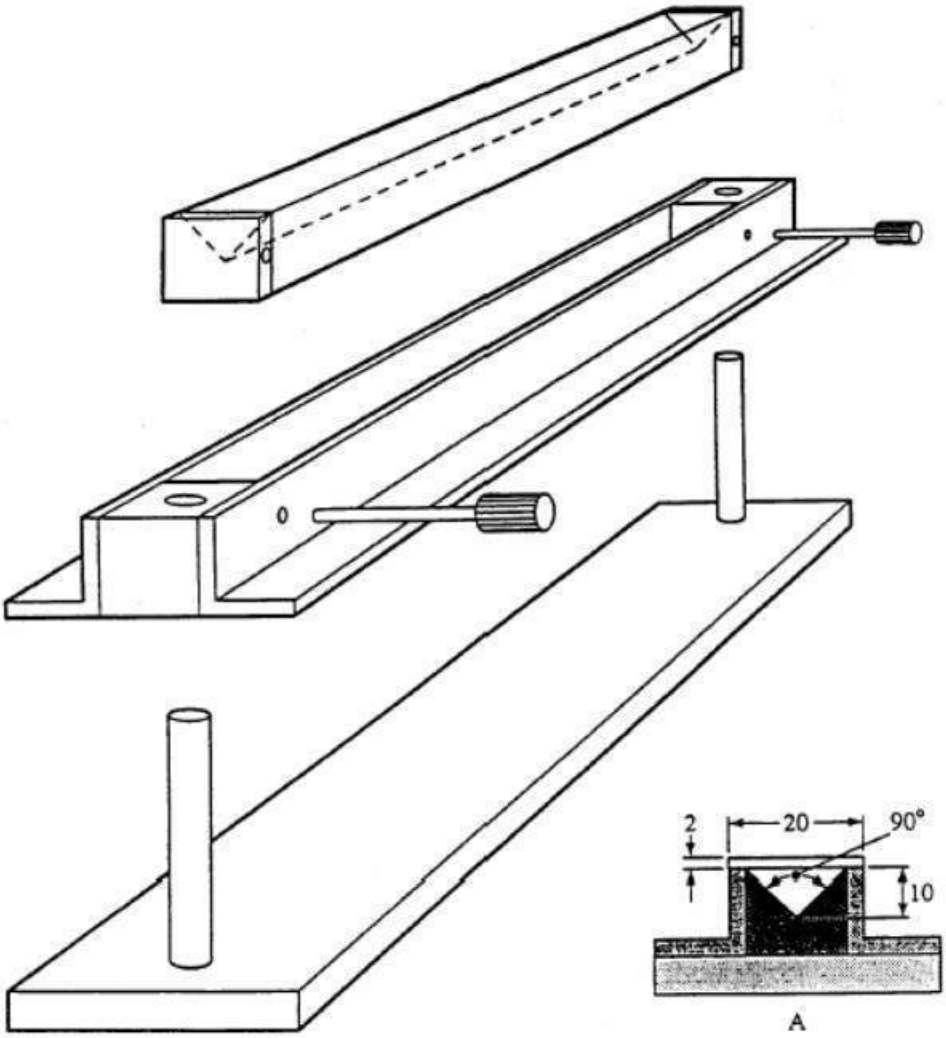
2227 Flashpoint

16. Attachments

- FIGURE 1: Powder Train Mold
- Flowchart

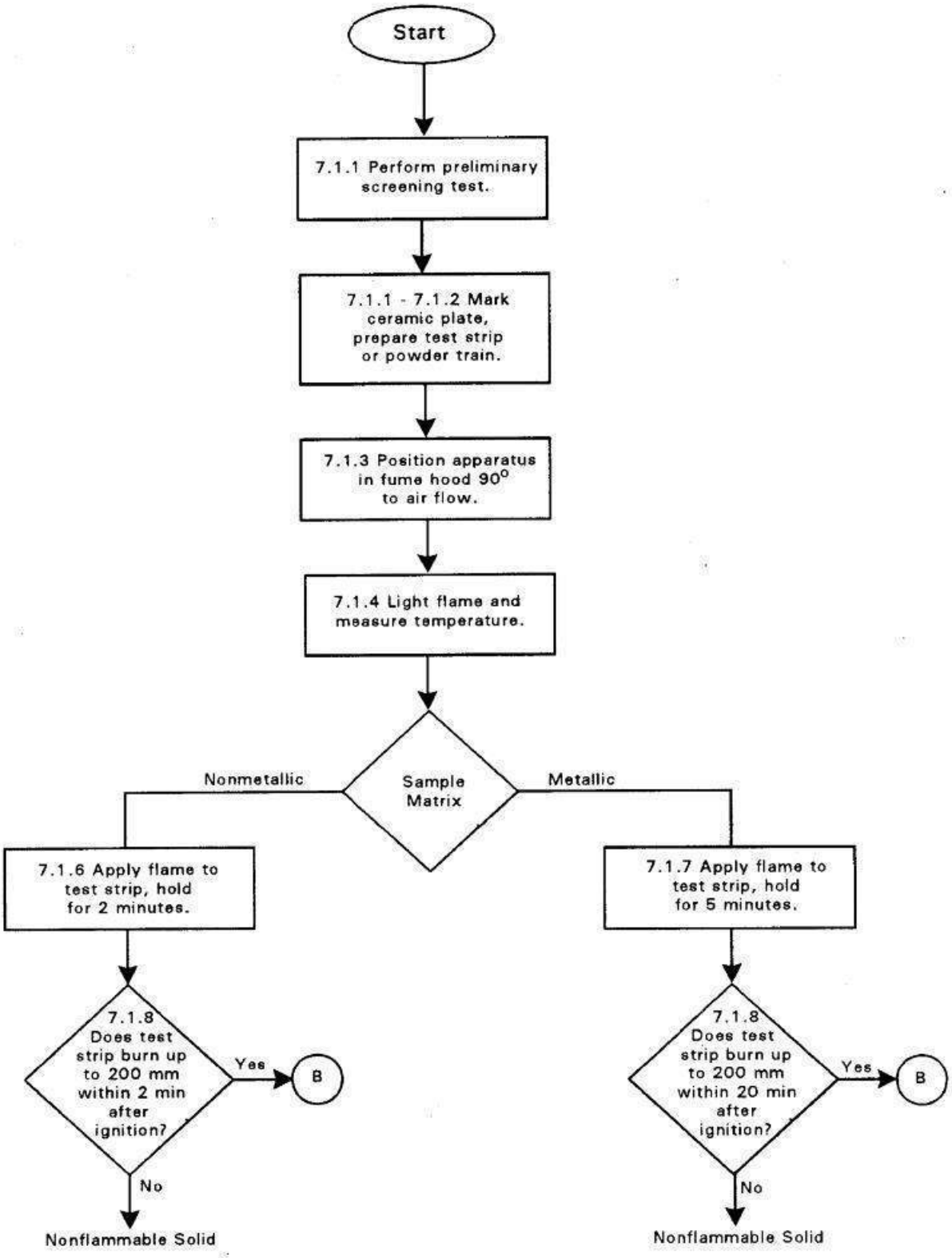
FIGURE 1

Powder Train Mold



(A) Cross-section of 250 mm long mould

Flowchart – Ignitability of Solids Method 1030



TCLP/SPLP Extraction - Volatile Organics EPA 1311/1312

Reference: EPA 1311, EPA 1312 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Update I, July 1992

1. Scope and Application

Matrices: The TCLP/SPLP extraction process is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

Definitions: Refer to Alpha Analytical Quality Manual.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the INSTRUMENT and in the interpretation of INSTRUMENT data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

For liquid wastes, (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP/SPLP extract.

For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. A special extractor vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

The analytical method detection limits determined by the laboratory are on file for review. See the 8260 SOP 2108.

4. Interferences

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. This includes wearing personal protective equipment such as a lab coat, safety glasses, gloves and respirator (as necessary).

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

When the waste is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples are collected in Teflon-lined septum capped vials and stored at 4 ± 2° C). Samples are opened only immediately prior to extraction.

SPLP samples may be collected in 25g En Core sampling device.

6.2 Sample Preservation

Preservatives shall not be added to samples before extraction. For SPLP samples arriving in En Cores they must be extruded and tumbled within 48 hours of collection. Extrude the entire sample or up to 25g if the sample weighs more than 25g since the vessel can only hold 500 mLs of fluid

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) must be extracted.

TCLP/SPLP extracts are prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses.

Samples must undergo TCLP/SPLP extraction within the time periods listed in Table 1.

7. Equipment and Supplies

7.1 Agitation Apparatus: End-over-end 30 ± 2 rpm.

7.2 Zero Headspace Extraction Vessel

7.3 Glass Fiber Filters: 0.6 to 0.8µm, 90mm

7.4 50mL Gastight Syringe

7.5 Graduated Cylinder: 500 mL, 1L

7.6 Laboratory Balance: 0.01g tolerance

7.7 Pressure Gauge: 0-60 psi

7.8 Pressure Filtration Device

7.9 Drying Oven: 100 ± 2 °F

7.10 Fluid Metering Pump

7.11 pH Meter: The meter should be accurate to + 0.05 units at 25°C

8. Reagents and Standards

8.1 Analytical Standards: The standards shall be prepared according to the appropriate volatile organic analysis method 8260 SOP 2108.

8.2 Hydrochloric Acid (HCl), 1N: Dilute 83 mL conc. HCl to 1L with DI water.

8.3 Nitric Acid (HNO_3), 1N: Dilute 64 mL conc. HNO_3 to 1L with DI water.

8.4 Sodium Hydroxide (NaOH), 1N: Dissolve 40 gs NaOH in 1L of water.

8.5 Glacial acetic acid: CH_3COOH.

8.6 Ottawa sand: SiO_2

8.7 Extraction Fluid #1: Add 5.7 mL glacial acetic acid to 500 mL of DI water, add 64.3 mL of 1N NaOH, and dilute to a volume of 1L. Check pH of solution which must be 4.93 $\pm$ 0.05 when correctly prepared. The laboratory reagent number is referenced in the TCLP sample logbook.

NOTE: This extraction fluid is monitored frequently for impurities. The pH is checked prior to use to ensure that this fluid is prepared accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

8.8 Extraction Fluid #2: Organic-free reagent water.

8.9 Methanol: Purge and Trap Grade or equivalent. Store in flammables cabinet.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

A minimum of **one blank** must be analyzed for every batch or 20 extracts, whichever comes first. The blank is prepared by weighing 20g of Ottawa sand (SiO_2) and utilizing the same extraction fluid as used for the samples. The blank is then analyzed following the same procedures as analytical samples.

9.2 Laboratory Control Sample (LCS)

A laboratory control sample is not extracted. Refer to the 8260 procedure for the LCS information.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike

Upon a client's request, a spike solution composed of all regulated compounds can be added to the TCLP/SPLP extract before analysis. See Method 8260 for more information.

9.6 Laboratory Duplicate

Not Applicable.

9.7 Method-specific Quality Control Samples

Not Applicable.

9.8 Method Sequence

The extraction sequence is:

- Disassemble ZHE; clean all parts with warm H_2O and soap; dry.
- Check sample; ensure there is enough sample for analysis.
- Determine %solids of the waste.
- Extract the TCLP/SPLP.
    - Record the temperature of the extraction room.
    - Following extraction, perform a leak test on the ZHE canisters.
- Draw sample with syringe from ZHE after extraction.
- Prepare sample for 8260 analysis

10. Procedure

10.1 Equipment Set-up

10.1.1 Preliminary Evaluation

10.1.1.1 Determine % Solids :

10.1.1.1.1 If waste contains no free liquid, proceed to Section 10.1.1.2 where the waste is the solid portion.

10.1.1.1.2 If the waste contains less than 0.5% solids, proceed to Section 10.3.2.

10.1.1.1.3 Multiphasic waste - separate liquid and solid portion

The following is performed by the Metals Prep Department.

10.1.1.1.3.1 Using a pressure filtration device pre-weigh the filter and the container to receive the initial filtrate. Record the weight in the laboratory notebook.

10.1.1.1.3.2 Assemble the pressure filter according to the manufacturer's directions.

10.1.1.1.3.3 Weigh a subsample of the waste (100 g minimum) and record the weight.

10.1.1.1.3.4 Transfer the waste sample to the filter holder spreading evenly over the filter.

10.1.1.1.3.5 Gradually apply a gentle pressure of 1-10psi until the pressurizing gas moves through the filter, proceed to Section 10.1.1.1.3.8.

10.1.1.1.3.6 If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10psi increments.

10.1.1.1.3.7 Stop the filtration when pressurizing gas passes through the filter, or 50psi is reached and no additional liquid passes through the filter in any 2 minute interval.

10.1.1.1.3.8 The material in the filter holder is defined as the SOLID PHASE of waste and the filtrate is defined as the LIQUID PHASE.

Note: the SOLID PHASE may appear liquid in some samples. Do Not replace original filter. Use only one filter.

10.1.1.1.3.9 Weigh the filtrate filled container and record the weight in the laboratory notebook. Calculate the weight of the LIQUID PHASE as follows:

$$W_{LP} = W_F - W_C$$

Where:

W_{LP} = Weight of the LIQUID PHASE
W_F = Weight of the filtrate filled container
W_C = Weight of container

10.1.1.1.3.10 Calculate the weight of the SOLID PHASE using the following formula:

$$W_{SP} = W_W - W_{LP}$$

Where:

W_{SP} = Weight of the SOLID PHASE
W_W = Weight of the waste sample
W_{LP} = Weight of the LIQUID PHASE

10.1.1.1.3.11 Calculate the percent solids using the following formula:

$$\%Solids = \frac{W_{sp}}{W_w x 100}$$

Where:

W_{SP} = Weight of the SOLID PHASE
W_W = Weight of the waste sample

10.1.1.1.3.12 If the % Solids determined in Section 10.1.1.1.3.11, is less than 0.5%, then proceed to Section 10.3.2, where the filtrate is the TCLP/SPLP extract.

10.1.1.1.3.13 Remove the solid phase and filter from the filtration apparatus, dry at 100 ± 2° F, cool and weigh. Record the weight in the laboratory notebook.

10.1.1.1.3.14 Calculate the percent dry solids using the following formula:

$$\%\ \text{dry solids} = \frac{W_{DW} - W_F}{W_W \times 100}$$

Where:

W_{DW} = Weight of dried waste + filter
W_F = Weight of the filter
W_W = Weight of waste sample

10.1.1.1.3.15 If the % dry solids determine above, 10.1.1.3.14, is less than 0.5%, then proceed to Section 10.3.2, where the filtrate is the TCLP extract.

10.1.1.2 Determine whether waste requires particle size reduction

The following is performed by the Metals Prep Department.

Using fresh portion of the waste, obtain another solid phase of the waste sample by pressure filtration as determined in Sections 10.1.1.1.1. Use enough wet sample to obtain a dry portion for tumbling: at least 100 g. Evaluate the solid phase for particle size. Particle size reduction is required unless the solid has a surface area greater to 3.1 cm^2 per g, or is smaller than 1 cm in its narrowest dimension.

10.1.1.3 Determine Compatibility of Liquids

The following is performed by the Metals Prep Department.

If a LIQUID PHASE was filtered, check its compatibility with the extraction fluid. If the liquids are compatible (miscible), then only one TCLP/SPLP extract needs to

be analyzed. If the liquids are not compatible, then the two liquids will have to be analyzed separately. Record in the laboratory notebook.

10.2 Initial Calibration

Not Applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 After the preliminary examination has been completed, the sample used in the following step comes from supplied VOA sample vials. The extraction for Volatile Organics is accomplished in a zero headspace extractor, ZHE.

10.3.2 Waste containing less than 5% dry solids

10.3.2.1 For waste containing less than 5% dry solids, weigh out up to a 500 g subsample and record its weight. Filter enough of the sample so that two 40 mL VOA vials may be filled with the filtered liquid. The liquid portion of the waste, after filtration, is defined as the TCLP/SPLP extract for volatile organic analysis. Proceed to Section 10.3.4.20.

10.3.3 100% Solid Waste

10.3.3.1 If particle size reduction is required (Section 10.1.1.2.), prepare the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 10.1.1.2. Waste and appropriate reduction equipment should be refrigerated to 4 ± 2° C prior to particle size reduction.

10.3.3.2 Weigh 20 gs of prepared waste and record the weight. If a 25 g Encore is supplied extrude and record the weight.

10.3.3.3 Quantitatively transfer the sample to the ZHE.

10.3.3.4 Assemble the ZHE according to manufacturer's instructions. Add the appropriate type and amount of fluid using the fluid metering pump. The fluid added is 20 times the amount of sample. For example, for a 20 g sample, add 400 mL of extraction fluid for TCLPs or 400 mL of organic free DI water for SPLPs. For a 25 g sample, 500 mLs of the appropriate fluid is added.

10.3.3.5 Position the ZHE in the vertical position with the liquid inlet/outlet valve on top and open.

10.3.3.6 Gradually apply pressure to the ZHE and bleed out any headspace.

10.3.3.7 Proceed to Section 10.3.4.16.

10.3.4 Waste containing greater than 5% dry solids

10.3.4.1 Determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge the ZHE} = \frac{20 \times 100}{\%\ \text{solids}}$$

Weigh out a subsample of the waste of the appropriate size and record the weight

10.3.4.2 If particle size reduction of the solid portion of the waste was required in Section 10.1.1.2, prepare the waste for extraction by crushing, cutting or grinding the solid portion of the waste to a surface area or particle size as described in Section 10.1.1.2. Waste and appropriate reduction equipment should be refrigerated to 4 ± 2° C prior to particle size reduction.

10.3.4.3 Quantitatively transfer the entire sample quickly to the ZHE. Assemble the ZHE in accordance with the manufacturer's instructions.

10.3.4.4 Place the device in the vertical position with the inlet/outlet valve on top and open.

10.3.4.5 Gradually apply pressure to the ZHE and bleed out any headspace quickly.

10.3.4.6 At the first appearance of liquid from the liquid inlet/outlet valve quickly close the valve.

10.3.4.7 Attach a 50mL gastight syringe to the liquid inlet/outlet valve and open the valve.

10.3.4.8 Begin applying gentle pressure to force the liquid phase of the sample into the filtrate collection container. When no additional liquid has passed through the filter in any 2-minute interval, proceed to Section 10.3.4.9.

10.3.4.9 Stop filtration by closing the liquid inlet/outlet valve.

10.3.4.10 Disconnect and weigh the 50mL gastight syringe. Record the weight in the laboratory notebook.

10.3.4.11 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

10.3.4.12 If the liquid phase is compatible with the extract (Section 10.1.1.3), store the filtrate at 4 ± 2°C until analysis. Then combine and analyze the filtrate as one extract.

10.3.4.13 If the liquid phase is not compatible with the solid extract, then analyze the filtrates separately and mathematically combine the results (Section 10.3.4.22).

10.3.4.14 Determine the weight of extraction fluid #1 for TCLP or organic free DI water for SPLP to add to the ZHE as follows:

Weight of extraction fluid = $20(\mathbf{W_S + W_T - W_{TF}})$

Where:

$\mathbf{W_S}$ = Weight Sample
$\mathbf{W_T}$ = Weight of pre-weighed 50mL syringe
$\mathbf{W_{TF}}$ = Weight of filtrate in 50mL syringe

For 100% solid samples, the above formula reduces to:

Weight of extraction fluid = 20 x weight of sample

10.3.4.15 Add extraction fluid by backing piston off the filter and sucking fluid into the ZHE. Do not open the ZHE.

10.3.4.16 Re-pressurize the ZHE to 15 psi (1.03 bar) and place in the rotary agitation apparatus. Rotate at 30 ± 2 rpm for 18 ± 2 hours.

10.3.4.17 Post-extraction measure the final pressure of the ZHE canister. If pressure has decreased below 15 PSI the sample must be reset.

10.3.4.18 Following the agitation period separate the material in the extractor vessel into its liquid and solid phases.

10.3.4.18.1 100% solid waste samples: Filter enough liquid phase to fill two 40 mL vials. The filtrate is defined as the TCLP/SPLP extract. Proceed to Section 10.3.4.20.

10.3.4.18.2 Compatible liquid phases: Filter the entire extract into a 50mL gastight syringe. Transfer the contents of the syringe into 40mL VOA vials. Combine the extracts (Section 10.3.4.22) and analyze together.

10.3.4.18.3 Non-compatible liquid phases. Filter a sufficient quantity of the solid extract to fill two 40 mL VOA vials and analyze. The two liquids are collectively defined as the TCLP/SPLP extract and results are mathematically combined after individual analysis.

10.3.4.19 Following collection of the TCLP/SPLP extract immediately prepare the extract for analysis and store with minimal headspace at 4 ± 2°C until analyzed.

10.3.4.20 Follow the volatile organic analyses method 8260 SOP 2108 to analyze the TCLP/SPLP extract.

10.3.4.21 If the individual liquid phases (i.e. non-compatible liquids) are analyzed separately, determine the volume of the individual phases, conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)\,(C_1) \;+\; (V_2)\,(C_2)}{V_1 \;+\; V_2}$$

Where:

V_1 = Volume of the first phase, (L)
C_1 = Analyte concentration first phase, (mg/L)
V_2 = Volume of the second phase, (L)
C_2 = Analyte concentration second phase, (mg/L)

10.4 Continuing Calibration

Not Applicable.

10.5 Preventive Maintenance

Tumbler rotation speed is verified annually and documented in the TCLP Annual Calibration Log, Form No.: 13855.

Balances are calibrated semi-annually by an instrument service company. Certificates are kept on file.

ZHEs are cleaned after extraction in soapy water. Filter screens and o-rings may be changed if they show degradation or the extraction vessel can't maintain pressure. Extremely contaminated vessels maybe further solvent rinsed with methanol

11. Data Evaluation, Calculations and Reporting

Calculations are included in Section 10.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

Not Applicable.

13.2.2 Continuing (DOC)

Not Applicable.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

2108 8260 SOP

13855 TCLP Annual Calibration Form

16. Attachments

Table1: Maximum Holding Times
Table 2: TCLP Analyte List

Table 1

MAXIMUM HOLDING TIMES

Sample Maximum Holding Times (Days)

	From: Field collection **To:** TCLP/SPLP extraction	**From:** TCLP/SPLP extraction **To:** Preparative extraction	**From:** Preparative extraction **To:** Determinative analysis	**Total Elapsed Time**
Volatiles	14/48 (hrs.)	N/A	14	28
Semi-volatiles	14	7	40	61
Mercury	28	N/A	28	56
Metals, except mercury	180	N/A	180	360

Table 2

TCLP Analyte List

Analyte	CAS No.
1,1 - Dichloroethene	75-35-4
1,2 - Dichloroethane	0107-06-02
1,4 - Dichlorobenzene	106-46-7
2-Butanone	78-93-3
Benzene	71-43-2
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
Tetrachloroethene	127-18-4
Trichloroethene	79-01-6
Vinyl chloride	75-01-4

TCLP Extraction

Metals and Semi-Volatile Organics

Reference Method: **EPA 1311**

Reference: **SW-846**, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Update I, July 1992

State of Connecticut, DEP, TCLP by SW-846 Method 1311, Version 2.0, December 2006

1. Scope and Application

The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

Definitions: See Alpha Laboratories Quality Manual Appendix A.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Extractions Manager, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

For liquid wastes, (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP extract.

For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

For additional detailed instruction, see TCLP and SPLP Work Instruction (ID# 17618).

2.1 Method Modifications from Reference

- 10.1.1.3.3 – Shaker table is used instead of stir bars. Samples not covered when shaking.
- 8.2 – 4N NaOH is used instead of 1N NaOH.

3. Reporting Limits

The Reported Detection Limit is determined by the amount of sample used for preparation. Therefore a review of Client requirements for Reporting Limits is necessary prior to sample preparation. Refer to analytical method SOPs.

4. Interferences

4.1 The most common cause of contamination is from improperly cleaned glassware and lab supplies. All glassware and re-useable extraction equipment must be scrupulously cleaned, following the Organic Extraction Glassware Cleaning and Handling SOP/1953 and Work Instruction 10995, Solvent rinsing guide.

4.2 Impurities in solvents and reagents may also yield artifacts and/or interferences that may compromise the results of sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of extract preparation and analysis by preparing method blanks with each extraction batch. The same solvents and reagents are used for the method blank and the associated samples.

4.3 Phthalate esters contaminate many types of products used in the laboratory. Plastic materials must not contact the samples or extracts, as phthalates could be easily leached from the plastic. The exception is in the use of various pre-packed reagent cartridges (Florisil, Silica gel) used in the extract cleanup steps. Each new lot of cartridges is checked for contamination, and is monitored on an on-going basis through the analysis of method blanks.

4.4 Additional specific interference or contamination concerns are addressed in the various analytical SOPs.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. This includes wearing personal protective equipment such as a labcoat, safety glasses, gloves and respirator (as necessary).

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents and when washing glassware.

5.2 All extract concentration steps must be performed in the extraction hoods. All solvent and extract transfers must also be handled in the hood.

5.3 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods.

5.5 All waste solvents must be transferred to the satellite waste storage containers located in the extraction lab. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

5.6 Inspect all glassware prior to use. Do not use any glassware that is chipped, cracked or etched if it could present a safety hazard. Damaged glassware is put aside for repair, otherwise discard the piece.

5.7 All Field Samples must be opened and handled in a hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass containers. Soils are collected in 8oz amber glass jars, and aqueous samples are collected in 500mL amber glass jars.

6.2 Sample Preservation

Preservatives are not added to samples before extraction.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples may be refrigerated at 4 ± 2°C, unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

Samples must undergo TCLP extraction within the time periods listed in Table 1.

7. Equipment and Supplies

7.1 Agitation Apparatus: End-over-end 30 ± 2 rpm. Calibrated Quarterly.

7.2 Vacuum Filtration Unit

7.3 0.6 to 0.8 µm Glass Fiber Filter Paper: 70 mm, 90 mm, and 120 mm, and acid prewashed 47 mm for all metals analysis.

7.4 2 L Glass/Plastic-coated Bottles

7.5 7 L Plastic Extraction Vessel: For Lamp Extraction

7.6 pH Meter: +/- 0.01 units resolution

7.7 pH test paper: 0-14 pH range

7.8 Laboratory Balance: +/- 0.1 g tolerance

7.9 250 mL Beaker or equivalent

7.10 Watch Glass: Used with Beaker (Section 7.8)

7.11 Stir Bars

7.12 Hot Plate: 1x1 ft (120 V) Barnestead-Thermolyne – Type 2200

7.13 2 L Graduated Cylinder Class A

7.14 250 mL Graduated Cylinder Class A

7.15 Teflon coated Sieve: 9.5 mm (0.375-inch)

7.16 Oven: Capable of maintaining 100 ± 20°C

7.17 Automatic Shaker Table

7.18 Vessel to store fluid #1

7.19 Vessel to store fluid #2

7.20 Calibrated thermometer: Temperature calibration point 50°C. Range: 0-100°C.

7.21 Bottle Top Dispenser: 0-5 mL, 0-100 mL

7.22 1 L Amber Bottle: Thermo Scientific, Item # 341-0950, Jar Tall Amber WM.

7.23 120 mL Plastic Bottle: Greenwood, Product # 07-120WMF24503, Natural HDPE WM Packer assembled w/38-400 F-217 Lined Cap.

7.24 250 mL Plastic Bottle: Greenwood, Product # 07-250OB45F22603, Natural HDPE WM Oblong Bottle Assembled w/45-400 F-217 Lined Cap.

8. Reagents and Standards

8.1 Hydrochloric Acid: 1N: Dilute 83 mL conc. HCl to 1 liter with reagent water. Store at room temperature; expires one year from date of prep.

8.2 Sodium Hydroxide: 4N: Dissolve 160 grams NaOH in 1 liter of reagent water. If larger volumes are required, prepare using similar ratios. Store at room temperature; expires one year from date of prep.

8.3 Glacial acetic acid: CH_3COOH. Store at room temperature; expires according to manufacturer's specifications.

8.4 Reagent Water, (ASTM Type II): All references to water within this SOP refer to reagent water unless otherwise specified. Reagent water is interference-free.

8.5 Extraction Fluid #1: Add 5.7 mL glacial acetic acid to 500 mL of reagent water, add 64.3 mL of 4N NaOH, and dilute to a volume of 4 liters. Check the pH of the solution after preparation and record the value in the Extraction Fluid Logbook. The pH is within 4.93 $\pm$ 0.05 when correctly prepared. In addition, the pH of the fluid is verified prior to each use and the pH recorded in the extraction logbook. Store at room temperature; no expiration date, however the pH must be maintained.

NOTE: Larger volumes or extraction fluid may be prepared, provided the ratios remain the same. For example, a typical preparation of 200 L of Extraction Fluid #1:

1140 mL Glacial Acetic Acid + 3215 mL 4N NaOH diluted to 200 L with reagent water

8.6 Extraction Fluid #2: Dilute 5.7 mL glacial acetic with reagent water to a volume of 1 liter. Check pH of solution which should be 2.88 ± 0.05 when correctly prepared. Record the pH in the Extraction Fluid Logbook.

The pH is checked prior to use to ensure that this fluid is prepared accurately. If the pH is not within the above specifications, the fluid is discarded and fresh extraction fluid prepared. Store at room temperature; no expiration date, however the pH must be maintained.

NOTE: Larger volumes or extraction fluid may be prepared, provided the ratios remain the same. For example, a typical preparation of 20L or 50L Extraction Fluid #2:

19,886 mL of reagent water + 114 mL Glacial Acetic Acid = 20 L of fluid.

49,715 mL of reagent water + 285 mL Glacial Acetic Acid = 50 L of fluid

8.7 Trace Nitric Acid (1:1 HNO3): 500 mL HNO3 diluted to 1 liter with DI water. Store at room temperature in hood. Expires 3 months from prep.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A minimum of **one blank** (using the same extraction fluid lot number as used for the samples) must be prepared and analyzed for every 20 extracts or every 48 hours, whichever comes first. The Blank vessel is a randomly selected container from the stock and is not one designated for blank use only.

9.2 Laboratory Control Sample (LCS)

Not applicable.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Determine analysis to be performed (Metals, ABN, Pest, PCB, Herbicides)
- Check sample; ensure there is enough sample for the extraction
- Determine the %solids of the waste sample

- Determine particle size reduction requirement
- Determine the extraction fluid to be used
- Extract the sample for TCLP
- Filter the extract.
- Aliquot extract to proper preparation departments
- Prepare extracts according to analytical protocols

10. Procedure

10.1 Equipment Set-up

10.1.1 Preliminary Evaluation

Perform preliminary TCLP evaluations on a minimum of 100g aliquot of waste. If there is limited sample volume, the chemist must contact the project manager. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include:

(1) determination of the percent solids (Section 10.1.1.1);

(2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Section 10.1.1.1.2);

(3) determination of whether the solid portion of the waste requires particle size reduction (Section 10.1.1.2); and

(4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste (Section 10.1.1.3)

10.1.1.1 Determine % Solids

10.1.1.1.1 If waste contains no free liquid, proceed to Section 10.1.1.2 where the waste is the solid portion. If the waste contains less than 0.5% solids, proceed to Section 10.1.1.1.2.

10.1.1.1.2 Mulitphasic waste - separate liquid and solid portion

10.1.1.1.2.1 Pre-weigh a filter. Pre-weigh a beaker or graduated cylinder that will hold the initial filtrate. Pre-weigh empty filtrate container. Record weights in the extraction logbook.

10.1.1.1.2.2 Assemble the vacuum filtration unit.

10.1.1.1.2.3 Weigh a subsample of the waste (100 g minimum), or pour 100 mL of the sample into a cylinder, and record the weight or volume in the extraction logbook.

10.1.1.1.2.4 Transfer the waste sample to the filter holder, spreading evenly over the filter.

10.1.1.1.2.5 Allow enough time for the liquid to pass through the filter (at least 15 minutes).

10.1.1.1.2.6 The material in the filter holder is defined as the SOLID PHASE of waste and the filtrate is defined as the LIQUID PHASE.

Note: The SOLID PHASE may appear liquid in some samples. Do Not replace original filter. Use only one filter.

10.1.1.1.2.7 Weigh the filtrate filled container and calculate the weight of the LIQUID PHASE as follows:

$$W_{LP} = W_F - W_C$$

Where:

W_{LP} = Weight of the LIQUID PHASE
W_F = Weight of the filtrate filled container
W_C = Weight of container

10.1.1.1.2.8 Calculate the weight of the SOLID PHASE using the following formula:

$$W_{SP} = W_W - W_{LP}$$

Where:

W_{SP} = Weight of the SOLID PHASE
W_W = Weight of the waste sample (Section 10.1.1.1.2.3)
W_{LP} = Weight of the LIQUID PHASE (Section 10.1.1.1.2.7)

10.1.1.1.2.9 Calculate the percent solids using the following formula:

$$\%\ Solids = \frac{Wsp}{Ww} \times 100$$

Where:

W_{SP} = Weight of the SOLID PHASE (Section 10.1.1.1.2.8)
W_W = Weight of the waste sample (Section 10.1.1.1.2.3)

10.1.1.1.2.10 If the % Solids determined above (section 10.1.1.1.2.9) is less than 0.5%, then the filtrate is the TCLP extract. Filter sample until enough filtrate is obtained for analysis and proceed to Section 10.3.1.3.6.

10.1.1.1.2.11 If the % Solids determined above in section is greater than or equal to 0.5%, all liquids entrained in the filter and waste must be removed to determine the % dry solids. Remove the solid phase and filter from the filtration apparatus. Heat at 100 ± 20° C in an oven (section 7.16) until two successive weights yield the same value within ±1%. Record weights in the extraction logbook.

10.1.1.1.2.12 Calculate the percent dry solids using the following formula:

$$\%\ Dry\ Solids = \frac{W_{DW} - W_F}{W_W} \times 100$$

Where:

W_{DW} = Weight of dried waste + filter (Section 10.1.1.1.2.11)
W_F = Weight of the filter (Section 10.1.1.1.2.1)
W_W = Weight of waste sample (Section 10.1.1.1.2.3)

10.1.1.1.2.13 If the % dry solids determined above (section 10.1.1.1.2.12), is less than 0.5%, then the filtrate is the TCLP extract. Filter sample until enough filtrate is obtained for analysis and proceed to Section 10.3.1.3.6.

10.1.1.1.2.14 If the % dry solids determined above is greater than or equal to 0.5%, the sample is multi-phasic. Determine the weight of the dry waste by subtracting the weight of the filter from the weight of the dry waste and filter as determined follows and record in extraction logbook:

Weight of dry solids= **$W_{DW} . W_F$**

Where:

W_{DW} = Weight of dried waste + filter (Section 10.1.1.1.2.11)
W_F = Weight of the filter (Section 10.1.1.1.2.1)

Proceed to Section 10.3.1.3. (**NOTE:** Do not dispose of dried waste + filter.)

10.1.1.2 Determine whether waste requires particle size reduction

Evaluate the solid phase of the waste sample (Section 10.1.1.1.2.6), to determine if particle size reduction is necessary. Particle size reduction is required unless the solid has a surface area greater than 3.1 cm^2 per gram or is smaller than 1 cm in its narrowest dimension (i.e.: will not pass through a 9.5 mm (0.375-inch) standard sieve).

If the surface area is smaller or the particle size is larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting or grinding the waste to a surface area or particle size as described above.

NOTE: Surface area criteria is meant for filamentous (e.g. paper, cloth, etc.) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, those samples should be reduced to meet the 1 cm dimension criteria.

10.1.1.3 Extraction Fluid Determination

10.1.1.3.1 Weigh out a 5 g subsample of the solid phase waste, if the sample is all solid. If the sample is multiphasic, weigh out a 5 g subsample of the solid waste from Section 10.1.1.1.2.6.

10.1.1.3.2 Transfer the 5 g aliquot to a 250 mL beaker (or equivalent) and add 96.5 mL of reagent water. (NOTE: Less weight and volume may be used, provided the ratio remains the same.)

10.1.1.3.3 Place beaker (or equivalent) in the automatic shaker (Section 7.17) for 5 minutes.

10.1.1.3.4 Measure using pH strip and record the pH in the extraction logbook.

10.1.1.3.5 If pH is less than 5.0, use extraction fluid #1, proceed to Section 10.3.

10.1.1.3.6 If pH is greater than 5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watch glass, heat to 50°C+/- 2°C on a hotplate and hold for 10 minutes.

Note: heating block load tests have shown heating times of up to 25 minutes prior to the sample reaching the specified temperature. The 10 minute hold begins only after the temperature is reached and monitored using a calibrated thermometer placed in a sample or one representative of the batch.

Cool solution to room temperature and record pH. If the pH is less than 5.0, use extraction fluid #1. Otherwise, use extraction fluid #2.

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Extraction of SOLID PHASE

10.3.1.1 A 100 gram minimum aliquot of SOLID PHASE is extracted to produce the TCLP extract, as outlined below. In some cases, a larger sample size may be required in order to produce enough liquid for the required analysis. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analysis, more than one extraction may be performed and the extracts combined for analysis. If there is limited sample volume, the chemist must contact the project manager.

10.3.1.2 100% Solid Waste

10.3.1.2.1 If the waste is 100% solid, homogenize the sample and weigh out an aliquot (100 gram minimum), follow Sections 10.4.1.2 (particle size reduction) and then proceed to 10.3.1.3.2 (extraction fluid amount).

10.3.1.3 Liquid or Multiphasic Waste

10.3.1.3.1 Quantitatively transfer the solid material, along with the filter (from Section 10.1.1.1.2.14), into a glass extractor bottle (plastic may be used if only metals are being analyzed).

Repeat sections 10.1.1.1.2 through .6 if additional final volume is needed for the intended analysis.

10.3.1.3.2 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$V_{EF} = \frac{20 \times \%\,Solid \times W_W}{100}$$

Where:

$\mathbf{V_{EF}}$ = Volume of extraction fluid required.
(Density = 0.998g/L, therefore assume volume is equivalent to weight.)
%Solid = Percent solid as determined in section 10.1.1.1.2.9
(100% if waste contains no free liquid as determined in section 10.1.1.1.1)
$\mathbf{W_W}$ = Weight of waste (Section 10.1.1.1.2.3)

10.3.1.3.3 Add the volume of the appropriate extraction fluid (determined in Section 10.1.1.3) to the extraction vessel. Glass amber 2.5 L for organics and metals, 2.5 L plastic if for metals only analysis. New (never used) 2.5 L HPDE plastic vessels for PFAS (Reference: PFAS by SPE and LC/MS/MS Isotope Dilution (ID#23528).

10.3.1.3.4 Close the extractor bottle tightly. Secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours while maintaining room temperature at 23 ± 2° C. Record the agitation device (Tumbler) ID in the extraction logbook.

10.3.1.3.4.1 The room temperature (°C) and the extraction time are recorded in the extraction logbook at both the beginning and the end of extraction.

10.3.1.3.4.2 To verify that the proper room temperature is maintained during extraction, a Maximum/Minimum thermometer is reset at the beginning of the extraction period. This thermometer is reviewed at the end of the extraction period and the Maximum and Minimum temperatures are noted in the extraction logbook.

10.3.1.3.4.3 If proper room temperature is not maintained during the extraction period, it is considered a variation from the method and must be written into a laboratory narrative and submitted with the reported data. The Department Supervisor is immediately notified to determine the proper corrective action to be taken.

10.3.1.3.5 Following extraction, vacuum filter the extract through glass fiber filters (acid precleaned for metals analysis). For this final filtration, the glass fiber filter may be changed to facilitate quicker filtration but should be kept to a minimum when practical. Use of pre-filters is prohibited for TCLP.

10.3.1.3.6 Prepare the TCLP extract as follows:

10.3.1.3.6.1 If the waste contained no initial liquid phase, the filtered extract obtained is defined as the TCLP extract. Proceed to 10.3.1.3.7.

10.3.1.3.6.2 If the waste contained a liquid phase, check its compatibility with the extraction fluid determined in Section 10.1.1.3. If the liquids are compatible (miscible), then the initial liquid phase (Section

10.1.1.1.2.6) and the filtered extract are combined and analyzed together.

10.3.1.3.6.3 If the initial liquid phase of the waste obtained in Section 10.1.1.1.2.6, is not compatible with the filtered extract, do not combine the liquids. Analyze these liquids, collectively defined as the TCLP extract, separately and mathematically combine the results, as described below. Determine the volume of the individual phases, conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$(\mathrm{mg/L}),\ \mathrm{Final\ Analyte\ Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:

V_1 = Volume of the first phase (L)
C_1 = Analyte concentration first phase (mg/L)
V_2 = Volume of the second phase (L)
C_2 = Analyte concentration second phase(mg/L)

10.3.1.3.7 Record the pH of the TCLP extract in the extraction logbook prior to preservation of samples.

10.3.1.3.8 Metals analysis: transfer filtrate (or combined filtrates) to a 120 mL plastic bottle and add 2.5 mL of 1:1 HNO_3 preservative to all samples.

10.3.1.3.8.1 Check the pH of the preserved samples to ensure that the sample is properly preserved at a pH of less than 2.

10.3.1.3.9 Non-volatile Organics: transfer filtrate (or combined filtrates) to two 1 L glass amber bottles, adhere pre-made sample ID labels and transfer to extraction department refrigerators.

10.3.2 Extraction of Lamps

10.3.2.1 Lamps (for both TCLP extraction and 120x extraction) are broken down into small particles (See Section 10.1.1.2) and extracted using Extraction Fluid #1. The extraction procedure from Section 10.3.1.3.2 through 10.3.1.3.8 is followed with the exception that the entire lamp weight including endcaps is used. 7 L plastic extraction vessels are utilized for larger lamps that exceed 100 g total weight. The vessel size must be recorded in the logbook for lamp samples.

10.3.2.1.1 Check the sample comments for whether Mercuric Oxide (HgO) is required during the extraction. If so, make note in the logbook that the HgO packet was added to the extraction vessel prior to tumbling. Contact the project manager if HgO is requested but no packet is supplied.

10.3.2.2 The excess unfiltered extract is immediately decanted into the TCLP waste drum, making sure to leave behind in the extraction vessel the solid material that was extracted.

10.3.2.3 The remaining solid material is transferred to a plastic bag, labeled as TCLP Lamp Waste and brought to the Hazardous Waste department for proper disposal.

10.4 Continuing Calibration

Not Applicable.

10.5 Preventive Maintenance

None.

11. Data Evaluation, Calculations and Reporting

None.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 All Holding time exceedences, improper preservation and Extraction Anomalies are to be reported to a Supervisor or Manager. Non Conformance Reports may need to be issues through the Qualtrax System.

12.2 Refer to determinative method SOPs for additional Corrective Action information.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732 These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

Not Applicable.

13.2.2 Continuing (DOC)

Not Applicable.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

SOP 2121 Chemical Hygiene Plan

SOP 1732 MDL/LOD/LOQ Generation

SOP 1739 IDC/DOC Generation

SOP 1728 Waste Management and Disposal SOP

WI 17618 TCLP and SPLP Work Instruction

16. Attachments

Table 1: Maximum Holding Times

Table 1

MAXIMUM HOLDING TIMES

Sample Maximum Holding Times (Days)

	From: Field collection **To:** TCLP extraction	**From:** TCLP extraction **To:** Preparative extraction	**From:** Preparative extraction **To:** Determinative analysis	**Total Elapsed Time**
Semi-volatiles	14	7	40	61
Mercury	28	N/A	28	56
Metals, except mercury	180	N/A	180	360

Microwave Extraction

Reference Methods: **EPA 3546**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, February 2007.

1. Scope and Application

Matrices: Soil, solid, clay, sediment, sludge, solid waste

Definitions: Refer to Alpha Analytical Quality Manual.

Method 3546 is a procedure for extracting water insoluble or slightly water soluble organic compounds from soils, clays, sediments, sludges, and solid wastes. The procedure uses microwave energy to produce elevated temperature and pressure conditions (i.e., 75°C and 50 - 175 psi) in a closed vessel containing the sample and organic solvent(s) to achieve analyte recoveries equivalent to those from Soxhlet extraction (Method 3540C), using less solvent and taking significantly less time than the Soxhlet procedure.

This method is applicable to the extraction of a variety of semivolatile organic compounds, some of which are: substituted phenols, PCBs, and PCDDs/PCDFs. The extracts are analyzed by the appropriate chromatographic procedure(s).

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Microwave. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Samples are prepared for extraction following the LEAN one-piece flow methodology and loaded into the extraction vessel. The appropriate solvent (See WI/14825 Microwave Extraction Guide) is added to the vessel and sealed. The extraction vessel containing the sample and solvent is heated to the extraction temperature and extracted for 20- 40 minutes. The extraction mixture is allowed to cool. The vessel is opened and the contents are filtered.

The extract is then concentrated and (as needed) exchanged into a solvent compatible with the cleanup or determinative procedure being employed. Extracts are then vialed and transferred to the Analytical Department.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Reporting Limit information may be found in the various determinative method SOPs.

4. Interferences

4.1 The most common cause of contamination is from improperly cleaned glassware and lab supplies. All glassware and re-useable extraction equipment (i.e. spatulas, extraction vessels) must be scrupulously cleaned, following the Organic Extraction Glassware Cleaning and Handling SOP and Work instruction 10995, Solvent rinsing/filtering guide.

4.2 Impurities in solvents and reagents may also yield artifacts and/or interferences that may compromise the results of sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of extract preparation and analysis by preparing method blanks with each extraction batch. The same solvents and reagents are used for the method blank and the associated samples.

4.3 Phthalate esters contaminate many types of products used in the laboratory. Plastic materials must not contact the samples or extracts, as phthalates could be easily leached from the plastic. The exception is in the use of various pre-packed reagent cartridges (Florisil, Silica gel) used in the extract cleanup steps. Each new lot of cartridges is checked for contamination, and is monitored on an on-going basis through the analysis of method blanks.

4.4 Additional specific interference or contamination concerns are addressed in the various analytical SOPs. If necessary, Florisil, Sulfuric Acid, Silica Gel and/or Sulfur cleanup procedures may be employed.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 The extraction vessels are at elevated temperatures and pressure after the extraction stage. Allow the vessels to cool at room temperature or in the refrigerator before opening.

5.2 During the heating step, some solvent vapors may escape through the vessel liner/seal cover. Follow the manufacturer's directions regarding the vessel assembly and instrument setup to prevent release of solvent vapors to the laboratory atmosphere.

5.3 The instrument contains flammable vapor sensors and should be operated with all covers in place and doors closed to ensure proper operation of the sensors. Follow the manufacturer's directions regarding replacement of extraction vessel seals when frequent vapor leaks are detected.

5.4 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents and when washing glassware.

5.5 All extract concentration steps must be performed in the extraction hoods. All solvent and extract transfers must also be handled in the hood.

5.6 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

5.7 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods.

5.8 All waste solvents must be transferred to the satellite waste storage containers located in the extraction lab. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

5.9 Inspect all glassware prior to use. Do not use any glassware that is chipped, cracked or etched if it could present a safety hazard. Damaged glassware is put aside for repair, otherwise discard the piece.

5.10 All Field Samples must be opened and handled in a hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Sample collection and preservation requirements are described the in the various analytical method SOPs.

6.2 Sample Preservation

None.

6.3 Sample Shipping

See applicable Sample Custody SOP.

6.4 Sample Handling

All soil samples are stored, refrigerated, in the Custody sample refrigerators. Samples are removed by the analyst immediately prior to sample extraction. The chemist must take custody of the samples by signing them out utilizing the LIMS.

When possible, samples must be homogenized prior to taking the sample aliquot, as described in Section 10.1. After the sample aliquot is removed, the samples are returned to the Sample Bank and placed in the appropriate sample refrigerator. Custody of the samples is transferred utilizing the LIMS.

Holding time for soil samples are 14 days, with the following exceptions:

- PCB samples expire after 365 days, except for CT and NJ samples (14 days).

7. Equipment and Supplies

7.1 Spatulas: Stainless steel.

7.2 Beakers: Stainless Steel 250mL.

7.3 Mortar and Pestle: Capable of reducing particle size to <1mm.

7.4 Kuderna-Danish (K-D) apparatus: Assemble by attaching the Concentrator Tube to the Evaporation Flask using the Plastic Kek clip. Add the Macro Snyder column to the Evaporation Flask. The Micro Snyder Column is attached directly to the Concentrator Tube using the Plastic Kek Clip.

7.4.1 Concentrator tube: 25mL, graduated and calibrated. A ground-glass stopper is used to prevent evaporation of extracts.

7.4.2 Evaporation flask: 500mL. Attach to concentrator tube with plastic kek clips.

7.4.3 Snyder column: Three-ball macro.

7.4.4 Plastic Kek Clips.

7.5 S-EVAP Water Bath with Solvent Collection Capability: Heated. Capable of temperature control (0.1C). Baths are located in a hood. Baths are equipped with chilled water condensers (set to 10.0C) for solvent collection.

7.6 Buchi Concentration System: Base Unit, Chiller (set to 3.0C), Pump, Block, Controller and 180mL Glass Vessels.

7.7 Boiling Chips: Solvent-extracted, approximately 10/40 mesh (silicon carbide or equivalent).

7.8 Syringe: 1.0mL, 250µL, 25uL, Various sizes for measuring surrogates and spikes.

7.9 Disposable Borosilicate Transfer Pipets.

7.10 Brady labeling system: Thermal label generator.

7.11 Sodium Sulfate Stainless Steel filtering funnels. Add a plug of glass wool to the base of the 104mm stainless steel funnel. Add approximately 20grams of baked sodium sulfate.

7.12 Glass wool: SUPELCO, silane treated.

7.13 Whatman Filter Paper: used for filtering all Pesticide/8081.(Whatman no.1 or equivalent)

7.14 Graduated Cylinder: 25 and 50mL. Class A.

7.15 N-EVAP: Organomation; Various sizes utilized for micro blowdown.

7.16 Aluminum weighing dishes: Electron Microscopy Sciences Cat#70051-01.

7.17 Solvent pump dispenser: Dispensette Organic 5-100ml, 1-5mL.

7.18 Analytical Balance: Capable of weighing to 0.01g

7.19 Multi-position Stirring Plates

7.20 Magnetic Stirring Bars

7.21 250mL Erlenmeyer Flask

7.22 Microwave Accelerated Reaction System (MARS): CEM Corporation. The temperature performance requirements necessitate that the microwave extraction system be capable of sensing the temperature to within ± 2.0°C and automatically adjusting the microwave field output power within 2 seconds of sensing. Temperature sensor is accurate to ± 2°C and adjustable microwave wattage to 1600W. Temperature feedback control provides the primary performance mechanism for the method.

7.22.1 Microwave extraction vessels: 75ml and 100mL (Plus). With plugs and caps. Capable of accommodating 1g to 30g samples. Vessels are transparent to microwave energy, relatively inert to reagents and sample components, and capable of withstanding the temperature and pressure requirements (minimum conditions of 75°C and 200psi) necessary to perform this procedure. Follow the manufacturer's instructions regarding cleaning, handling, and sealing the vessels.

7.22.2 Kevlar sleeves.

7.22.3 Extraction Vessel Turntable: Used to hold and rotate the extraction vessels during extraction.

7.23 Tongue Depressors: Premiere, Wooden, senior size, No. 95-8801.

8. Reagents and Standards

Reagent grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.1 Reagent Water: All references to water in this method refer to reagent water from Alpha's RO water treatment system.

8.2 Sodium Sulfate (Na_2SO_4): Granular anhydrous; purified by baking at 400ºC for 4 hours in a glass or stainless steel beaker. Store in closed glass containers. All references to sodium sulfate in this method refer to this prepared reagent. Sodium sulfate is also used for filtering.

8.3 Ottwa Sand: VWR catalog #JT3382-5. Purified by baking at 400C for 4 hours in a shallow tray or stainless steel beaker.

8.4 Hexane: Pesticide quality.

8.5 Acetone: Pesticide quality.

8.6 Dichloromethane: Pesticide quality.

8.7 Nitrogen Gas: Reagent grade, used to purge and pressurize the extraction cell and as the concentration gas in the Turbovap II auto-concentrator units and the N-EVAP.

8.8 Spiking Solutions: Commonly used surrogate and LCS/MS spiking solutions used in the extraction steps are listed in WI/14825 Microwave Extraction Guide. Additionally, the WI/14825 Microwave Extraction Guide has a complete listing of all surrogate and LCS/MS spiking solutions. The preparation and expiration dates of these solutions are described in the analytical SOPs.

8.9 Extraction Solvents: This method has been validated using a 1:1 mixture of hexane and acetone, 1:1 mixture of methylene chloride and acetone, or 100% Methylene Chloride for matrices such as soil, glass-fibers, and sand. Other solvent systems may have applicability in microwave extraction, provided that at least one component absorbs microwave energy. The choice of extraction solvent will depend on the analytes of interest and no single solvent is universally applicable to all analyte groups. Whatever solvent system is employed, *including* those specifically listed in this method, the analyst *must* demonstrate adequate performance for the analytes of interest, at the levels of interest. Method 8000 describes procedures that may be used to develop performance criteria for such demonstrations as well as for matrix spike and laboratory control sample results.

Hexane is a water-immiscible solvent and acetone is a water-miscible solvent. The purpose of the water-miscible solvent is to facilitate the extraction of wet solids by allowing the mixed solvent to penetrate the layer of water of the surface of the solid particles. The water immiscible solvent extracts organic compounds with similar polarities. The polarity of acetone may also help extract polar analytes in mixed solvent systems. When 100% Methylene Chloride is used, water is added as a catalyst to absorb microwave energy for method 8270.

8.10 Silica Gel: VWR, Cat# TX4694MAAA. 60 - 200 mesh, chromatography grade. Activated by baking at 140 °C for a minimum of 16 hours in a shallow tray. The silica gel is stored in the oven or desiccator until ready for use. All references to silica gel in this method refer to this prepared reagent.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Each extraction batch contains various QC samples used to ensure the validity of the sample results. The particular QC elements performed for a given extraction batch are determined by the requirements of the determinative method. The purpose and definition of the QC samples performed are listed below. Specific QC requirements of the analytical methods are listed in WI/14825 Microwave Extraction Guide.

9.1 Blank

Blanks, or method blanks, are measured aliquots of clean matrix (typically sodium sulfate or sand for soil extractions) that are treated identically to the associated samples. Surrogates are added, and the blanks are carried through all stages of the sample extraction, concentration, and cleanup procedures. Blanks serve to ensure that no systematic contamination exists. A blank is extracted with each batch or 20 or less samples.

9.2 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

LCS samples are measured aliquots of clean matrix (typically sodium sulfate for soil extractions) that are spiked with a solution containing known amounts of target compounds, in addition to the surrogate solution. The LCS is carried through all stages of the sample extraction, concentration, and cleanup procedures. LCS samples serve as batch specific quantitative checks of the extraction. An LCS is extracted with each batch of 20 or less samples.

An LCSD is performed in addition to an LCS for all Massachusetts Contingency Plan (MCP) methods, as well as in lieu of the MS/MSD or Duplicate when there is insufficient sample volume available. The required solutions are listed in WI/14825 Microwave Extraction Guide.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

MS and MSDs are field samples spiked with a known quantity of the target analyte(s). They are prepared by taking additional sample aliquots, and adding the appropriate amounts of surrogate and spiking solutions. The MS/MSD is carried through all stages of the sample extraction, concentration, and cleanup procedures. MS samples serve as a measure of extraction accuracy, by allowing the comparison of the found amount(s) of target analyte(s) with the spiked amount(s). An MS/MSD set also allows for the calculation of the extraction precision, by comparing the results of the two samples. Requirements for MS and MSD are listed in WI/14825 Microwave Extraction Guide.

For samples with a state of origin of New Jersey, a MS and MSD/DUP must be extracted for every twenty samples within a 24hr period.

9.6 Laboratory Duplicate

Duplicates are laboratory selected replicate samples, prepared by taking an additional sample aliquot of a sample. The duplicate is carried through all stages of the sample extraction, concentration, and cleanup procedures. Duplicates serve as a measure of the extraction precision, by comparing the results of the sample and duplicate. Requirements for Duplicates are listed in WI/14825 Microwave Extraction Guide.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

Surrogates are compounds specified by the analytical method that are added to all samples and QC samples prior to beginning the extraction process. Surrogate recoveries are calculated and serve as a sample specific quantitative check of the extraction. The various spiking solutions are prepared according to the directions found in the analytical SOPs. The required solutions and volumes used are listed in WI/14825 Microwave Extraction Guide.

9.8 Method Sequence

Refer to Section 10.

10. Procedure

All soil microwave soil extracts follow the LEAN "one-piece flow". All extraction information is recorded by the chemist performing the work in the ELN (Electronic Lab Notebook) see WI/2517. In addition to recording the extraction, concentration, clean-up and vialing information, the analyst must note the matrix "type" along with any observations, deviations from the procedure, or difficulties encountered with the samples in the comment section of the logbook.

10.1 Sample Preparation and Extraction

10.1.1 Soil Samples are scanned and removed from Sample Login Custody to Oprep Custody. Immediately after scanning, the samples batched into the ELN to create the Work Group. Labels are printed and placed on the cap of the soil container. See Work Instruction 2421, Labeling and Generating Work Groups and Batches.

10.1.2 All Glassware is cleaned prior to the Extraction following SOP 1953, Organic Extraction Glassware Cleaning and Handling.

10.1.3 During the extraction process, each soil or sediment sample is visually inspected. If a sample contains a significant amount of free water, the chemist must contact their supervisor or manager to determine if the water is to be considered part of the sample. If the water is not to be homogenized with the solid material, decant and discard the water layer. Record this information in the comments section of the ELN.

Any artifacts (rocks, leaves, sticks, or similar materials) are not typically considered part of the soil sample and are not to be included. If necessary, transfer these artifacts to another container prior to homogenizing the sample. Note the presence of sample artifacts in the ELN. Gummy, fibrous, or oily materials not amenable to grinding must be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction.

10.1.4 When possible, homogenize the sample well using a spatula by mixing the contents of the sample container. If this is difficult due to sample matrix, describe the non-homogeneity in the ELN.

10.1.5 The chemist must demonstrate that all equipment used during the extraction process interference-free. This is accomplished through the analysis of a solid matrix (Sodium Sulfate or Ottawa Sand) Method Blank (SB). A Method Blank is extracted with each batch of 20 or less samples.

10.1.6 **MARS Microwave System Operation:** Rinse the reaction vessels, caps, and plugs with 1:1 Acetone/DCM.

10.1.6.1 See Work Instruction WI/2421 for proper labeling procedures and The Microwave Extraction Guide (ID/14825) for one-piece flow operation and appropriate surrogate/spike and solvent to be used. Typically 15-30 grams of the sample and 15grams of sodium sulfate is extracted. Transfer the sample into the reaction vessel from the beaker or weighing tray. Add the appropriate surrogate and spiking solution. Sodium Sulfate is used for the QC substrate for all methods except 8270 and 8270 SIMTECH where Ottawa Sand is used for 8270 QC.

10.1.6.1.1 Note: Samples that are commented to isolate the glassware must be weighed with disposable aluminum weigh dishes (instead of beakers) and wooden tongue depressors (instead of stainless steel spatulas).

10.1.6.2 **For all Methods except 8270:** Add 35mL of 1:1 Hexane:Acetone, 1:1 DCM:Acetone or 100% DCM (or the appropriate extraction solvent) to each

reaction vessel. (Refer to Table 1 or ID#14825). Assure the sample matrix is covered with the extraction solvent prior to microwave extraction.

10.1.6.2.1 Place a plug and a cap on each reaction vessel.

10.1.6.2.2 Place all labeled vessels onto the carousal and place the carousal into the microwave.

10.1.6.2.3 On the front panel of the microwave, select "Use This Method" or UTM from the User Directory. NOTE: UTM-75 is used for the smaller 75mL vessels while UTM-Plus is for the larger 100mL "Plus" vessels.

The following parameters are loaded with "Use this Method" program:

- Stage 1: 1600W @ 100% Power.
- Ramp to 75°C for 10 minutes and hold for 30 minutes.
- This stage is followed by a 5 minute Cool Down step.

10.1.6.2.4 Remove each reaction vessel from the carousel for cooling.

10.1.6.2.5 Once samples have cooled to room temperature, twist the cap off the reaction vessel and filter the sample using The Sample Filtering Guide (ID#25052). The sodium sulfate funnel contains glass wool and approximately 15-30 grams of sodium sulfate. The sample is filtered with the solvent that was used to extract the sample. Due to contamination issues, pesticide samples require filter paper instead of the glass wool. Note- When opening the cap on the Teflon reaction vessel, point away from your body and perform this task in a fume hood.

10.1.6.3 **For 8270 and 8270 SIM:** See Work Instruction WI/2421 for proper labeling procedures and The Microwave Extraction Guide (ID/14825) for one-piece flow operation and appropriate surrogate/spike and solvent to be used. Transfer 30 grams of the sample (a requirement) into the reaction vessel from the beaker or weighing tray. Note: sodium sulfate is **not** mixed with the sample. For QC samples, Ottawa sand is used. Add the appropriate surrogate and spiking solution. After adding the sample to the reaction vessel, using a 1.0mL syringe or 1-5mL pump bottle, add 1.0mL of DI water to the sample.

10.1.6.3.1 Add 40mL of 100% DCM to each reaction vessel. (Refer to Table 1). Additional solvent may be necessary in some cases depending on sample matrix. Assure the sample matrix is covered with the extraction solvent prior to microwave extraction.

10.1.6.3.2 Place a plug and a cap on each reaction vessel.

10.1.6.3.3 Place all labeled vessels onto the carousal.

10.1.6.3.4 On the front panel of the microwave, select "8270" from the User Directory. NOTE: 8270-75 is used for the smaller 75mL vessels while 8270-Plus is for the larger 100mL "Plus" vessels.

The following parameters are loaded with 8270 method:

- Stage 1: 1600W @ 100% Power.
- Ramp to 75°C for 10 minutes and hold for 10 minutes.
- This stage is followed by a 5 minute Cool Down step.

10.1.6.4 Once samples have cooled to room temperature, filter through a sodium sulfate funnel (using DCM only) and into labeled Buchi vessels for DRO, ETPH, and all ABN samples (See Sample Filtering Guide ID#25052). Note- When opening the cap on the Teflon reaction vessel, point away from your body and perform this task in a fume hood.

10.1.6.5 ETPH Analysis: Filter the sample extract through a 20gram sodium sulfate funnel containing glasswool into a 250mL Erlenmeyer flask. Add 5 grams of Deactivated Silica Gel and a stir bar to the extract. Place the sample on a stirring plate and stir for 5 minutes @ 650rpm. Filter the extract through a funnel containing filter paper and collect into a Bucchi vessel for concentration, see Section 10.2.2.

10.1.6.6 Proceed to sample concentration. Note all DRO, ETPH, and ABN products are concentrated using the Buchi Concentration System.

10.2 Sample Concentration Techniques

10.2.1 KD Technique

10.2.1.1 Attach a three-ball Snyder column to the top of the flask. Place the KD apparatus on a hot water bath(SEVAP), (heated to approximately 75°C so that the concentrator tube is partially immersed in the hot water, and so that the entire lower rounded surface of the flask is bathed in hot water vapor. Attach the chilled water condenser to the top of the Synder Column. Adjust the position of the apparatus as required. At the proper rate of distillation, the balls in the column will actively chatter, but the chambers will not flood with solvent.

10.2.1.2 If a Hexane exchange is required (see Table 1), when the sample volume reaches 5-15mL, remove the condenser from the Synder Column and add 25mL of hexane using a graduated cylinder. Add the hexane to the top of the Snyder Column. Allow sample to continue to concentrate to 15-20mL and exchange with another 25mL of hexane. Allow the sample to boil until the intensity decreases (little to no chatter in the Snyder column). Remove the KD concentration setup and move to the 95C bath. Re-attach the condenser and continue with the concentration until the extract volume is reduced to 15mL.

10.2.1.3 Remove the KD apparatus from the water bath and remove the plastic Kek clip. Wipe the joint of the flask and the concentration tube with a dry paper towel to remove any moisture from the outside of the glassware. Allow to cool for 5minutes. Disassemble the KD apparatus. Move the label from the K-D Flask to the concentrator tube (See WI 2421).

10.2.1.4 Place the Concentrator tube on the N-EVAP. Using a disposable pipet, direct the nitrogen over the sample. The N-EVAP is set at 65 °C for samples extracted in Hexane. Adjust the flow of nitrogen so that the surface of the solvent is just visibly disturbed. Samples remain on N-EVAP until they are reduced to the appropriate volume (see S-Evap/N-Evap Concentration Standard Process WI#18528 for listing of appropriate volumes).

10.2.1.5 The extract is now ready for sample cleanup or vialing (See Table 1). Refer to the relevant Clean-up SOP or proceed with extract vialing (See WI 3827 Extract Vialing Procedure, WI 2426 GC Extract Vialing Procedure and WI 2423 GC/MS Extract Vialing Procedure).

10.2.2 Alternative Concentration Technique: Bucchi

The Bucchi is a self-contained sample concentration and solvent recovery system that utilizes vacuum, heat and oscillation to concentrate samples. The Bucchi will recover >95% of solvent emissions. Refer to Alpha SOP/12838 for Buchi concentration set-up and procedure.

10.3 Preventive Maintenance

10.3.1 Microwave System (MARS):

10.3.1.1 The instrument must be kept clean. Wipe the inside of microwave with soap and water and dry with a cloth as needed.

10.3.1.2 All microwave cells, caps and plugs are to be dish washed and rinsed with solvent prior to use. Additionally, the sleeves must be handled with care. If the edge of the sleeve is dented or chipped it will not hold pressure.

10.3.2 Analytical Balance

10.3.2.1 All balances are checked for accuracy daily and calibrated/serviced every six months by an instrument service company. All service records are kept on file.

10.3.2.2 Keep balances clean. Brush of any sample spills immediately.

11. Data Evaluation, Calculations and Reporting

None.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 All Holding time exceedences, improper preservation and Extraction Anomalies are to be reported to a Supervisor or Manager. Non Conformance Reports may need to be issued through the Qualtrax System.

12.2 If the KD Concentrator tube is allowed to run dry, the extract volume is spilled, etc. the sample must be re-extracted.

12.3 Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

12.4 Refer to determinative method SOPs for additional Corrective Action information.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

SOP/2121 Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739 DOC Generation

SOP/1728 Waste Management and Disposal SOP

SOP/1953 Organic Extraction Glassware Cleaning and Handling

WI/2421 Labeling and Generating Work Groups and Batches

WI/2517 LIMS Electronic Laboratory Notebook Procedure

WI/3827 Extract Vialing Procedure

WI/10995 Solvent Rinsing/Filtering

WI/14825 Microwave Extraction Guide

SOP/12838 Buchi Concentration

WI/25052 Sample Filtering Guide

WI/18528 S-Evap/N-Evap Concentration Standard Process

16. Attachments

Table 1 – Specific Extraction Conditions for Various Determinative Methods

Table 1

Specific Extraction Conditions for Various Determinative Methods

LIMS Product Code	Extraction Solvent	Exchange Solvent Required	Typical Final Volume	Appropriate Cleanup Technique
8082	1:1 Hexane/Acetone	hexane	5-10 mL	Sulfuric acid/ Sulfur
8081	1:1 DCM/Acetone	hexane	10 mL	Florisil
8270 SIM	DCM	---	1mL	---
8270/8270SIM	DCM	---	1 mL	---
TPH *	DCM	---	1 mL	---
EPH	DCM	hexane	1 mL	Silica gel Fractionation
EPH-TPH**	DCM	hexane	1 mL	---
ETPH	DCM	---	1 mL	Silica gel

***TPH includes the following LIMS Products: TPH-DRO and TPH-DRO-D**

****EPH-TPH includes the following LIMS Products: NJEPH-TPH-CAT1, NJEPH-TPH-CAT2**

(For a full list of products and final volumes see WI/18528 S-Evap/N-Evap Concentration Standard Process or SOP/12838 Buchi Concentration.)

Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

References: **Method 8260C**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, 2006.

Method 5035A, SW-846, Closed System Purge & Trap and Extraction for Volatile Organics in Soil and Waste Samples. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, Draft Revision I, July 2002.

Method 5030B, Purge & Trap for Aqueous Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, December, 1996.

Method 5030C, Purge & Trap for Aqueous Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, May, 2003.

1. Scope and Application

Matrices: Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Definitions: Refer to Alpha Analytical Quality Manual.

The compounds listed in Table 5 may be determined by this method.

There are various techniques by which these components may be introduced into the GC/MS system. Purge-and-trap, by Methods 5030C (aqueous samples) and 5035A (solid and waste oil samples), is the most commonly used technique for volatile organic analytes. However, other techniques are also appropriate and necessary for some analytes. One technique is direct injection of an aqueous sample (concentration permitting).

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph/mass spectrometers and in the interpretation of mass spectra and their use as a quantitative tool. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by direct injection. The analytes are introduced to a narrow-bore capillary column for analysis. The Gas Chromatograph (GC) is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the GC.

Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with

the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard, comparing sample response to the calibration standards.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 1 lists our typical reporting limits.

4. Interferences

4.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be free from contamination under the conditions of the analysis. Running laboratory reagent blanks as described in Section 9.1 and 10.3 demonstrates the system is free of contamination. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system must be avoided.

4.2 Sample contamination occurs by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A trip blank or a field reagent blank prepared from reagent water and carried through the sampling and handling protocol serves as a check on such contamination.

4.2.1 Storage blanks shall be analyzed if contamination is suspect. If contamination is confirmed by positive detections in the sample storage blanks, all data from samples contained in the relative refrigerator or freezer shall be evaluated for possible contamination. If the samples contain suspected contamination, the Client Services department shall be notified in order to contact the necessary clients regarding the contamination. Samples shall be reanalyzed if so desired by the client. If suspected contamination is not confirmed by storage blanks, no further action shall be pursued concerning said blanks. It is recommended that further action be taken to determine the possible cause of suspected contamination.

4.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. Whenever a highly concentrated sample is being encountered, it should be followed by an analysis of reagent water (instrument blank) to check for potential contamination. If carry-over is suspected, then numerous instrument blanks may be required; additionally all affected samples are rerun for confirmation. In case of severe contamination, preventive maintenance of the entire system may be required.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, standards, or solvents.

5.2 All stock solution standard preparation must be performed in the volatiles hood. Initial calibration, continuing calibration, laboratory control sample and client sample dilutions do not need to be performed in the hood.

5.3 All expired standards must be placed into the waste bucket in the lab for future disposal. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing Methanol must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Storage, Shipping and Handling

6.1 Sample Collection and Preservation

6.1.1 Aqueous Samples

Grab samples are collected in standard 40mL glass screw-cap vials with Teflon lined silicon septa (VOA vial). Two or more VOA vials should be filled per sample location. EPA Method 8260 requires that samples be acidified to eliminate the possibility of biological degradation. Unless otherwise directed for project-specific reasons, all VOA vials are delivered to the client with approximately 2 – 4 drops of 1:1 HCl added to the vial, which is sufficient to adjust the pH of the sample to < 2. Prepared trip blanks are provided to the client to accompany field samples for QC purposes.

Fill the sample vial to the point of overflowing so that no headspace is contained within. Samples must be introduced into the vials gently to reduce agitation, which might drive off volatile compounds or cause loss of the HCl preservative.

Seal the bottle so that no air bubbles are in the VOA vial. If preservative has been added, shake vigorously for one minute. Invert the bottle and tap to check for air bubbles. Recollect the samples if any air bubbles are present.

Maintain the hermetic seal on the VOA vial until time of analysis.

6.1.2 Soil Samples

The recommended sampling method for soil samples is EPA 5035A. Method 5035A provides for two distinct sampling procedures, depending on the required reporting limits and suspected or known concentration levels of target analytes. These methods are referred to as the High Level and Low Level methods. Both are listed below, but depending on the samples only one of the methods may be required. If concentration levels are unknown, it is recommended that samples be collected using both procedures. The Lab will analyze the high level sample first, followed by the low level sample if the

results from the high level analysis show that the sample is clean or contains analytes at low levels. The typical reporting levels of the two methods are listed in Table 1.

6.1.2.1 High Level Soil Samples

Collect sample in a standard 40mL glass screw-cap vial with Teflon lined silicon septa (VOA vial). The vial is provided containing 15mL of Purge and Trap Grade methanol, and is labeled and weighed prior to addition of sample. Record the weight of the vial with methanol on the vial label. Prepared trip blanks are provided to the client to accompany field samples for QC purposes.

Approximately 15g of soil is added to the vial in the field, making sure that the sample is completely covered by the methanol.

Maintain the hermetic seal on the VOA vial until the time of analysis.

An additional sample of the soil must also be obtained (without methanol) to be used for the determination of soil moisture content to allow for the calculation of the dry weight results, and to calculate the methanol dilution effect. (See Sections 11.1.2.2.2 and 11.1.2.2.3)

6.1.2.2 Low Level Soil Samples

Collect sample in a standard 40mL glass screw-cap vials with Teflon lined silicon septa (VOA vial). Two samples should be taken per sample location. Vials are provided containing a magnetic stirring bar and 5 mL of either 200g/L sodium bisulfate solution or water, prepared by a certified vendor. These vials are labeled and weighed prior to addition of sample. Record the weight of the vial with the stirring bar and preservative on the vial label.

Approximately 5g of soil is added to the vial in the field, making sure that the sample is completely covered by the sodium bisulfate solution or water.

Maintain the hermetic seal on the VOA until the time of analysis.

6.1.2.3 Oil Samples

Collect sample in unpreserved 40mL vial or unpreserved jar.

Maintain the hermetic seal on the VOA until the time of analysis.

6.2 Sample Handling and Storage

Document client specific sample handling, preservation and collection criteria in the project file. The laboratory Log-in staff documents sample temperature at the time of receipt.

Record deviations from this SOP or client specific criteria on the chain of custody form.

Record holding time exceedance, improper preservation and observed sample headspace on the nonconformance report form.

6.2.1 Aqueous Samples

Ice or refrigerate all samples from the time of collection until analysis, maintaining the sample temperature between 1 and 4 °C. Sample receiving personnel note on the sample delivery group form when samples received at the laboratory are not within the temperature criteria. If more than one vial is received for a sample the vials are stored in separate refrigerators. Storing the vials apart provides a useful check if laboratory contamination of a sample is suspected. Samples must be analyzed within 14 days of

collection. Unpreserved samples requiring aromatic analysis must be analyzed within 7 days of collection.

6.2.2 High Level Soil and Oil Samples

Ice or refrigerate all samples from the time of collection until analysis, maintaining the sample temperature between 2 and 6 °C. Sample receiving personnel note on the nonconformance report form when samples received at the laboratory are not within the temperature criteria.

6.2.3 Low Level Soil Samples

Ice or refrigerate samples preserved with water or sodium bisulfate from the time of collection until analysis, maintaining the sample temperature between 2 and 6 °C. Samples preserved with water are to be immediately frozen after sampling. Sample receiving personnel note on the nonconformance report form when samples received at the laboratory are not within the temperature criteria.

6.3 Sample Shipping

Samples requiring shipment to the laboratory are shipped in ice-packed coolers via an overnight delivery service in accordance with applicable Department of Transportation regulations.

7. Equipment and Supplies

7.1 Purge and Trap System (For Aqueous samples, High Level Soils and Oils): The purge-and-trap system consists of two separate pieces of equipment: a purging device (autosampler) (Varian Archon/8100, Tekmar Solatek, EST Centurion) coupled to the desorber (concentrator) (Tekmar Velocity or EST Encon).

7.1.1 Purge gas

7.1.1.1 Helium, analytical grade (99.999%).

7.1.1.2 Nitrogen, analytical grade (99.999%).

7.1.2 The purging device is configured with 25 mL sample purge tubes, and the purge gas is introduced at the bottom of the water column as finely divided bubbles

7.1.3 The trap used in the desorber is typically a Supelco “K” trap. Different traps may be used if equivalent performance is demonstrated.

7.1.4 The desorber is capable of rapidly heating the trap to 260°C. The trap is not heated above manufacturer’s specifications

7.2. Purge and Trap System (For Low Level Soil Samples): The purge and trap system consists of two separate pieces of equipment: a purging device (autosampler) coupled to the desorber (concentrator) (Varian Archon/8100, Tekmar Solatek, EST Centurion with EST Encon, Tekmar Velocity, or equivalents).

7.2.1. Purge gas = Helium or Nitrogen, analytical grade (99.999%).

7.2.2. The autosampler purging device is a closed system, designed to accept the 40mL VOA vials. The VOA vial, containing the soil sample, water (or sodium bisulfate), and stirring bar is placed into the autosampler tray. The instrument automatically adds reagent water, internal standards, and surrogates to the unopened VOA vial. The vial

is heated to 40 °C, and the purge gas is introduced into the aqueous portion to purge the volatile components onto the trap.

7.2.3. The trap used in the desorber is typically a Supelco "K" trap. Different traps may be used if equivalent performance is demonstrated.

7.2.4. The desorber is capable of rapidly heating the trap to 260 °C. The trap is not heated above manufacturer specifications.

7.3 Gas Chromatography/Mass Spectrometer/Data System:

7.3.1 Gas Chromatograph, Agilent 6890/7890 or equivalent: An analytical system complete with a temperature-programmable gas chromatograph with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source of the GC/MS system.

7.3.2 Typical Gas Chromatographic Columns:

7.3.2.1 Column 1: Restek 502.2, 40 meter, 0.18mm ID, or equivalent.

7.3.2.2 Column 2: Restek RTX-VMS, 30 meter, 0.25mm ID, or equivalent

7.3.3 Mass Spectrometer, Agilent 5973/5975/5978 or equivalent: Scanning from 35 to 300 amu every 2 seconds or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 3, when 50ng of the GC/MS tuning standard (BFB) are injected through the GC. For all SIM analysis, the mass spectrometer must also be able to acquire data in a dual acquisition mode (SIM and full scan).

7.3.4 Data System: Hewlett-Packard EnviroQuant software is used for data acquisition, and allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

Thruput Target 4.12 software or EnviroQuant E.02.02 (or equivalent) is used for data processing, and allows searching of any GC/MS data file for ions of a specified mass, and plotting such ion abundances versus time or scan-number.

The most recent version of the EPA/NIST Mass Spectral Library is loaded onto the Target / EnviroQuant data system.

7.4 Wiretrol or Micro syringes: 10µL - 1,000µL.

7.5 Syringes: 5mL, 10mL, or 25mL, glass with Luerlock tip.

7.6 Balances: Top-loading, capable of weighing 0.01g.

7.7 Vials: 2mL, 4mL.

7.8 Disposable Pipets.

7.9 Volumetric Flasks: Class A, appropriate sizes, with ground-glass stoppers.

7.10 Eppendorf Pipets.

8. Reagents and Standards

Reagent grade organic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all organic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Great care must be taken to maintain the integrity of all standard solutions. Standards in methanol are stored at –10ºC or less, in amber vials with PTFE-lined screw-caps.

8.1 Organic-free Reagent Water:

All references to water in this method refer to organic-free reagent water, which is tap water passed through activated carbon and air bubbled through.

8.2 Methanol:

Purge and Trap Grade or equivalent. Store in flammables cabinet.

8.3 Stock Solutions:

All stock standard solutions are purchased from commercial vendors as ampule certified solutions. When an ampule stock solution is opened, it is transferred to a labeled amber screw-cap vial with minimal headspace. The expiration date of the stock solution is either the vendor specified expiration date or 6 months from the date the ampule was opened, whichever is sooner. Typical stock standard concentrations are listed in Table 4.

8.4 Intermediate Standards: Intermediate standards are prepared volumetrically by diluting the appropriate stock standard(s) with methanol. Initial Calibration solutions expire 2 months from the date of preparation, or sooner if daily continuing calibration checks do not achieve the method acceptance criteria. If the Intermediate Standards are used as a second source to verify a valid Initial Calibration solution, there is no expiration date.

8.4.1 Internal Standard Solutions:

The internal standards are Fluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. The intermediate IS solution is prepared by diluting the stock solution(s) with methanol to a concentration of 100 µg/mL. The appropriate amount of IS solution is added to the water or soil sample or QC sample to achieve a final concentration of 100 ng/sample or standard. Internal standard is added at the same concentration to all standards, samples, and QC samples.

8.4.2 Surrogate Standard Solutions:

The surrogate standards are Dibromofluoromethane, 1,2-Dichloroethane-d_4, Toluene-d_8, and 4-Bromofluorobenzene. The intermediate surrogate solution is prepared by diluting the stock solution(s) with methanol to a concentration of 100 µg/mL. The appropriate amount of surrogate solution is added to the water or soil sample or QC sample to achieve a final concentration of 100 ng/sample.

8.4.3 Target Compound Solutions:

The target analytes routinely reported by this method are listed in the beginning of this SOP. The intermediate target compound solutions are prepared by diluting the stock solution(s) with methanol. This set of solutions, at concentrations of 200 µg/mL, is used for preparation of the calibration standards.

8.4.4 4-Bromofluorobenzene (BFB) Tune solution:

A solution containing BFB at a concentration of 50 µg/mL is prepared by volumetrically diluting the BFB stock solution. 1 µL of this solution is direct-injected or purged into the GC/MS system to verify system performance prior to any standard or sample analysis.

8.5 Calibration Standards:

There are two types of calibration standards used for this method – initial calibration standards and calibration verification standards.

8.5.1 Initial Calibration Standards:

Initial calibration standards can be prepared at the levels listed in Table 4 (other/different levels are allowed). The Initial Calibration needs to have a minimum of 5 standards, 6 if a quadratic curve fit is used. Prepare these solutions in organic-free reagent water. The standards correspond to the range of concentrations found in typical samples and do not exceed the working range of the GC/MS system. Initial calibration should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

8.5.2 Initial Calibration Verification Standard (ICV):

The initial calibration verification standard is at the same concentration as the level 3 initial calibration standard. This standard is made from a second source than the Initial Calibration Standards.

8.5.3 Continuing Calibration Verification Standard:

The continuing calibration verification standard, or calibration check standard, should be analyzed near the action level of the project. Since most projects are focused on achieving low reporting limits, the continuing calibration verification standard is at the same concentrations as the level 3 initial calibration standard. This standard is run at the beginning of each analytical sequence, following the BFB tune standard, to verify system performance.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Blank samples must be matrix specific, i.e. methanol samples need to have methanol in the blank; sodium bisulfate samples need to have a sodium bisulfate blank analyzed; TCLP samples need a TCLP blank.

Analyze a matrix-specific blank each day prior to sample analysis to demonstrate that interferences from the analytical system are under control. The blank must contain the internal standards and surrogates.

Analyze the reagent water blank from the same source of water used for preparing the standards, QC samples and making sample dilutions. The method blank must not contain any target analytes at or above the compound reporting limits.

9.2 Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)

A LCS/LCSD pair is analyzed at the beginning of each analytical sequence. Since the LCS contains the same compounds at the same concentrations as the continuing calibration check standard, the same analysis is used to satisfy both QC elements. The LCS/LCSD acceptance criteria are based on in-house control limits, unless specified by project/regulation.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.5.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.4.

9.5 Matrix Spike/ Matrix Spike Duplicate

Upon Client Request, a matrix spike/matrix spike duplicate pair may be analyzed with each batch of 20 or less samples. The MS/MSD are sample aliquots spiked with the target compounds at the same concentration as the continuing calibration standard. The MS/MSD acceptance criteria are based on in-house control limits. If the MS/MSD does not meet the criteria, but the LCSD does, the failure may be attributed to sample matrix. Report the MS/MSD, including a narrative sheet for inclusion with the client report.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

9.7.1 Internal Standards

Area counts of the internal standard peaks in all samples and QC samples must be between 50-200% of the areas of the internal standards in the QC check standard.

If any individual percent recovery falls outside the range, that parameter has failed the acceptance criteria. For calibration standards, CCVs, LCS/LCSD or blanks the internal standard must be within the range for data to be reported to the clients. For samples, matrix spikes and duplicates: if the data is not within the range, the sample is rerun to confirm that the failure is due to sample matrix. A nonconformance report form is completed to ensure client notification and reporting if matrix effect is confirmed.

9.7.2 Surrogates

Surrogates are added to each field sample and QC sample. The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. The surrogate acceptance criteria are listed in Table 2. Since the SIM analysis is acquired in dual mode, the surrogates from the full scan are used to evaluate the entire sample (SIM and full scan).

9.8 Method Sequence

In a 12-hour period, the typical analytical sequence is as follows:

- BFB
- QC Check Standard/Laboratory Control Sample/LCSD
- Method Blank
- Samples
- MS/MSD (upon Client request, may be run any time after the Method Blank)

10. Procedure

10.1 Equipment Set-up

Typical instrument operating conditions are listed below. Alternate conditions are allowed, as long as method performance criteria can be met.

10.1.1 GC Conditions:

Temperature 1:	35°C	Carrier gas:	Helium, 99.999%
Hold Time 1:	4 minutes	Carrier mode:	Constant flow
Ramp 1:	6°C/minute	Carrier flow:	1 mL/minute
Temperature 2:	150°C		
Hold Time 2:	0 minutes		
Ramp 2:	8°C/minute		
Temperature 3:	220°C		
Final Time:	1 minute		

10.1.2 MS Conditions:

Mass scan range:	35 – 260 amu
Scan time:	0.5 minutes/scan
Source temperature:	230°C

10.1.3 Velocity Concentrator Purge and Trap Conditions:

Purge time:	11 minutes
Dry purge:	2 minutes
Desorb preheat:	250°C
Desorb temp:	255°C
Desorb time:	2 minutes
Bake temp:	290°C
Bake time:	10 minutes

10.1.4 Encon Concentrator Purge and Trap Conditions:

Purge time:	11 minutes
Dry purge:	1 minute
Desorb preheat:	245°C
Desorb temp:	255°C
Desorb time:	1 minute
Bake temp:	270°C
Bake time:	10 minutes

10.2 Initial Calibration

10.2.1 The initial calibration is performed at a minimum of five (5) concentration levels listed in Table 4, the low level of the either at or below the reporting limit. The calibration is performed using instrument conditions listed in Section 10.1.

BFB must be analyzed prior to analysis of the initial calibration standards, and must pass the criteria listed in Table 3. The mass spectrum of BFB should be acquired in the following manner:

(1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.
(2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of BFB.

This is done automatically with the ThruPut Target / Enviroquant software.

10.2.1.1 Low Level/High Level Soil Curve on Archon or Centurion: To prepare a calibration standard, add the appropriate volume of standard solution(s) to a 50mL volumetric flask using a micro syringe. Remove the needle quickly and mix by inverting the flask 3 times. Pour several mLs of the aqueous standard into the waste vessel, then gently fill a 5mL syringe with standard and transfer to a 40mL VOA vial containing a magnetic stir bar. Load the vial onto autosampler.

10.2.1.2 Aqueous/High Level Soil Curve on Solatek or Centurion: To prepare a calibration standard, add the appropriate volume of standard solution(s) to a 100mL volumetric flask using a micro syringe. Remove the needle quickly and mix by inverting the flask 3 times. Pour several mLs of the aqueous standard into the waste vessel, then gently fill a 40mL VOA vial to the top. Load the vial onto the autosampler.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in Section 10.1. The same operating conditions are used for calibration and sample analyses. Create the analytical sequence using the HP Enviroquant data acquisition software.

Relative Response Factors: The internal standard calibration technique is used. In each calibration standard, calculate the relative response factor for each analyte and the relative standard deviation (RSD) of the response factors using the Target / Enviroquant data processing software. The response factors are calculated using the areas of the characteristic (quantitation) ion for each target analyte and internal standard. The calculations are performed automatically using the Target / Enviroquant software, using the formulae listed in Alpha's Quality Manual.

10.2.3 Initial Calibration Criteria: The following sections outline the method acceptance criteria for an initial calibration curve. All criteria must be met for the calibration to be deemed acceptable, and for sample analysis to proceed.

10.2.3.1 Relative Standard Deviation Criteria: If the RSD for each target analyte is less than or equal to 20%, then the response for this compound is considered linear over the calibration range and the mean calibration factor can be used to quantitate sample results. If the 20% RSD criterion is not met for an analyte linear regression may be used if $\mathbf{r} \geq 0.990$, weighted linear with a weighting factor of 1/SD2 and $r > 0.990$, or quadratic fit if $\mathbf{r^2} \geq 0.995$. A minimum of six points is required and the low point of the calibration must be re-quantitated and recover within 70-130% to be deemed acceptable. The calibration must be repeated for any compounds that fail. If more than 10% of the compounds exceed the 20% RSD limit and do not achieve the minimum correlation coefficient for alternative curve fits, sample analysis cannot proceed.

10.2.3.2 Minimum Response Factors: Table 1 lists the suggested minimum response factors for the most common analytes. Each calibration level must be evaluated against the specified criteria. Analytes that fall below the criteria, but are greater than or equal to 0.05, are narrated for inclusion on the final report. There are certain very poor purgers (1,4-Dioxane, Acrolein, ketones, alcohols and other water soluble compounds) that should meet a 0.001 response factor. If an analyte falls below 0.05 (or 0.001 for 1,4-Dioxane, Acrolein, ketones, alcohols and other water soluble compounds), then corrective action must be taken to resolve the problem before analysis can proceed.

10.2.4 Evaluation of Retention Times: The relative retention times used for identification of target analytes are +/- 0.06 RRT (Relative Retention Time) units, based on the most recent standard run. It has been determined that these limits work well, being wide enough to eliminate false-negative results while being tight enough to eliminate false positive results. Due to the selectivity of the mass spectrometer, compound identification is more definitive than when using a less selective detector.

10.2.5 Initial Calibration Verification: After each calibration and before the analysis of samples, an ICV must be analyzed at or near the midpoint of the curve. The ICV must be prepared using a different source than the Initial Calibration and must contain all target analytes. The percent recoveries must be between 70% and 130% for target analytes except for "difficult" analytes (Table 7), which must exhibit percent recoveries between 40% and 160%. Corrective action is required if greater than 10% of all analytes are outside the prescribed criteria.

10.3 Equipment Operation and Sample Processing

The same GC, MS, and Purge and Trap conditions used for the initial calibration must be employed for sample analysis. After verification of system performance by analysis of BFB, the continuing calibration standard and method blank, samples are analyzed and processed as described below.

10.3.1 Analysis of Samples

10.3.1.1 All samples are initially screened using Tekmar HT3 Static and Dynamic Headspace System.

10.3.1.2 Retrieve sample VOA vials from the sample refrigerator just prior to loading onto the purge and trap system. High level soil and oil samples must be shaken for 1 – 2 minutes to extract the volatile components into the methanol. Let the

sample settle prior to taking methanol aliquot. Low level soil sample should be shaken briefly to ensure that the stir bar is loose, and will spin on the Archon or Centurion unit.

10.3.1.3 Oil Samples:
Take 1g of sample and dilute to 10mL methanol in a volumetric flask. Invert or shake to ensure proper mixing. Transfer to 40mL VOA vial.

10.3.1.4 Low level soil samples: (Archon or Centurion)
Take the low level VOA vial and place directly into the rack of the Archon sampling unit. Surrogate and internal standards are added automatically by the Archon prior to sample purging.

10.3.1.5 Aqueous samples: (Solatek or Centurion)
Load the VOA vial directly on the sampling rack. Dilutions may be prepared volumetrically and poured into VOA vials ensuring there is no headspace left in the vial. The auto-sampler will then sample 10mL from the VOA vial.

10.3.1.6 High level soil and oil samples: (Archon/Solatek/Centurion)

Shake for 2 minutes, ensuring the methanol has completely penetrated the soil in the vial.

10.3.1.6.1 Through liquid path
Load a maximum of 430µL or appropriate dilution of the methanol into a half-full VOA vial. Fill the VOA vial up to the top with water and cap with no headspace. Allow the auto-sampler to sample 10mL out of the VOA vial which would be equivalent to injecting 100µL of the methanol extract. Prepare dilutions accordingly.

10.3.1.6.2 Through soil path
Into a VOA vial with a stir bar added, load 4.9mL of water plus a maximum of 100 µL of methanol or appropriate dilution of methanol extract from a 5mL luerlock syringe or wiretrol. Cap the vial and load onto the auto-sampler.

10.3.2 Qualitative Analysis:

10.3.2.1 The qualitative identification of each compound is based on retention time and on comparison of the sample mass spectrum with the reference mass spectrum. The reference mass spectrum must be generated by the laboratory on the same GC/MS system. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.3.2.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. The Target / Enviroquant data system is configured to make this check.

10.3.2.1.2 The relative retention time (RRT) of the sample component is within ±0.06 RRT units of the RRT of the standard component.

10.3.2.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

10.3.2.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs (i.e., m and p-xylene).

10.3.2.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

10.3.2.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

10.3.2.2 For samples containing non-target analytes, a library search will be performed at client request. Compound identification will be classified as "tentative", and the concentration will be reported as an estimate as no quantitative standards are run for these compounds.

1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
2) The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)
3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks.

10.3.3 Quantitative Analysis:

10.3.3.1 Quantitation of a target compound detected in a sample is performed automatically by the EnviroQuant data processing software, using the formulae

found in Alpha's Quality Manual. Either the average response factor or calibration curve will be used for sample quantitation, depending on how the particular analyte was processed in the initial calibration curve.

10.3.3.2 If non-target compounds are to be reported, the quantitation is performed automatically by the EnviroQuant software using the total area of the compound and the nearest internal standard, and assuming a relative response factor of 1.0.

10.4 Continuing Calibration

Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

10.4.2 Prior to the analysis of samples or calibration standards, inject or purge 1 µL (50 ng) of the 4-Bromofluorobenzene standard (Section 8.4.4) into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 3 before sample analysis begins.

10.4.3 The initial calibration curve for each compound of interest must be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing the continuing calibration check standard (Section 8.5.3).

10.4.4 A method blank must be analyzed prior to any samples, typically immediately following the continuing calibration check standard, to ensure that the analytical system is free of contaminants. The method blank must not contain any target analytes at or above the required compound reporting limits.

10.4.5 The percent difference or drift for each target analyte must be less than or equal to 20% (30% for all SIM compounds). If greater than 20% of target analytes exceed the %D criteria corrective action must be taken prior to the analysis of samples. If less than or equal to 20% of compounds exceed the criteria, corrective action is not required.

10.4.6 The continuing calibration standard must also be evaluated for the suggested minimum response factor criteria, as specified in section 10.2.3.2

10.4.7 Internal Standard Retention Time:

The retention times of the internal standards in the calibration verification standard are evaluated after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.4.8 Internal Standard Response:

If the area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

10.5 Preventive Maintenance

Routine preventive maintenance should be performed on the analytical system. This includes replacement of GC septa and periodic rinsing or replacement of purge and trap tubes and sparge needles. The trap should be replaced every six months, or sooner if performance criteria cannot be met. Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

If system performance deteriorates, additional maintenance may be required. This includes replacement of injector ports and seals, clipping several inches off of the front end of the GC column, or in extreme cases the replacement of the GC column. Flushing or replacement of purge and trap lines may be necessary if they become contaminated or develop active sites.

Perform routine preventative maintenance as described throughout this SOP. Record all maintenance in the instrument logbook.

11. Data Evaluation, Calculations and Reporting

11.1.1 LIMS Data Corrections

Please note that the Laboratory Information Management System (LIMS) automatically adjusts soil sample results to account for the % Total Solids of the sample (as determined per Alpha SOP/07-38) and the methanol preservation dilution effect.

11.1.2 Data Calculations

11.1.2.1 Results of Aqueous Sample Analysis:

$$Concentration\ (ug/L) = \frac{(Conc.)\ (Vp)\ (DF)}{(Vs)}$$

where:

Conc. = On-column concentration obtained from the quantitation report.
Vp = Volume purged, 10 mL is standard
Vs = Volume of sample purged
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".

11.1.2.2 Results of Sediment/Soil, Sludge, and Waste Analysis:

All solids including soils, sediments, and sludges must be reported on a dry-weight basis.

11.1.2.2.1 Low-Level Samples:

$$Concentration\ (ug/Kg) = \frac{(Conc.)\ (Vp)\ (DF)}{(W)\ (\%S)}$$

11.1.2.2.2 High-Level Samples:

$$\text{Concentration (ug/Kg)} = \frac{(\text{Conc.})\ (Vp)\ (5000)\ (DF)}{(W)\ (Ve)\ (\%S)}$$

where:

Conc. = On-column concentration obtained from the quantitation report.
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".
Ve = Extract volume, mL
Vp = Volume purged, 5 mL is standard
W = Aliquot of sample (wet), g
%S = Sample % solid
5000 = Constant representing the final volume of the methanol extraction.

11.1.2.2.3 High-Level Samples Corrected for Total Water/Solvent Mixture (V_t):

Samples that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the water/solvent mixture. The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture calculation.

$$\%\ \text{moisture} = \frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100$$

$$V_t = \frac{[\text{mL of solvent} + (\%\text{moisture} \times \text{g of sample})]}{100} \times 1000\text{mL/mL}$$

The calculated V_t value is now added to the volume of methanol in the sample (typically 5000µL), and the corrected concentration is calculated using the equation below:

$$\text{Corrected concentration (mg/Kg)} = \frac{(\text{Conc.})\ (V_t + \text{methanol vol.})\ (Vp)\ (DF)}{(W)\ (Ve)\ (\%S)}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All batch and sample specific QC criteria outlined in section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/08-05 MDL/LOD/LOQ Generation

SOP/08-12 IDC/DOC Generation

SOP/14-01 Waste Management and Disposal SOP

16. Attachments

TABLE 1: 8260 REPORTING LIMITS

TABLE 2: 8260 QC ACCEPTANCE CRITERIA

TABLE 3: BFB TUNING CRITERIA

TABLE 4: STANDARD SOLUTIONS and ICAL Levels

TABLE 5: 8260C Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

TABLE 6: 8260C Quantitation Ions

TABLE 7: Difficult Analytes

Table 1
Standard Reported Detection Limits
US EPA METHOD 8260C and 5035A/8260C

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL(µg/KG)(1)	RDL (µg/KG) (2)
Acetone (3,4,5)	0.100	5.0	10	250
Acrolein (5)		5.0	25	1250
Acrylonitrile (3,4)		5.0	5	200
Allyl Chloride (7)		N/A	5	250
Benzene (3,4,5)	0.500	0.5	1	50
Bromobenzene (3,4)		2.5	5	250
Bromochloromethane (3,4,5)		2.5	5	250
Bromodichloromethane (3,4,5)	0.200	0.5	1	50
Bromoform (3,4,5)	0.100	2.0	4	200
Bromomethane (3,4,5)	0.100	1.0	2	100
2-Butanone (3,4,5)	0.100	5.0	10	500
Butyl acetate (7)		N/A	5	50
n-Butyl benzene (3,4)		0.5	1	50
sec-Butyl benzene (3,4)		0.5	1	50
tert-Butyl benzene (3,4)		2.5	5	250
Carbon disulfide (3,4,5)	0.100	5.0	10	500
Carbon tetrachloride (3,4,5)	0.100	0.5	1	50
Chlorobenzene (3,4,5)		0.5	1	50
Chlorodifluoromethane (7)		N/A	5	250
Chloroethane (3,4,5)	0.100	1.0	2	100
2-Chloroethylvinyl ether (3)		10.0	20	1000
Chloroform (3,4,5)	0.200	0.75	1.5	75
Chloromethane (3,4,5)	0.100	2.5	5	250
o-Chlorotoluene (3,4)		2.5	5	250
Cyclohexane (5)	0.100	10	20	1000
Cyclohexanone		10	20	1000
p-Chlorotoluene (3,4)		2.5	5	250
cis-Decahydronaphthalene (7)		N/A	5	250
trans-Decahydronaphthalene (7)		N/A	5	250
n-Decane (7)		N/A	5	250
Dibromochloromethane (3,4,5)	0.100	0.5	1	50
1,2-Dibromo-3-chloropropane (3,4,5)	0.050	2.5	5	250
1,2-Dibromoethane (3,4,5)	0.100	2.0	5	250
Dibromomethane (3,4)		5.0	10	500
1,2-Dichlorobenzene (3,4,5)	0.400	2.5	5	250
1,3-Dichlorobenzene (3,4,5)	0.600	2.5	5	250
1,4-Dichlorobenzene (3,4,5)	0.500	2.5	5	250
1,4-Dichlorobutane (3,4)		5.0	10	500
trans-1,4-Dichloro-2-butene (3,4)		2.5	5	250

Dichlorodifluoromethane (3,4,5)		5.0	10	500
1,1-Dichloroethane (3,4,5)	0.200	0.75	1.5	75
1,2-Dichloroethane (3,4,5)	0.100	0.5	1	50
1,1-Dichloroethene (3,4,5)	0.100	0. 5	1	50
cis-1,2-Dichloroethene (3,4,5)	0.100	0.5	1	50
trans-1,2-Dichloroethene (3,4,5)	0.100	0.75	1.5	75

Table 1 (continued)

Standard Reported Detection Limits

US EPA METHOD 8260C and 5035A/8260C

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL(µg/KG)(1)	RDL (µg/KG) (2)
1,2-Dichloropropane (3,4,5)	0.100	1.75	3.5	175
1,3-Dichloropropane (3,4)		2.5	5	250
2,2-Dichloropropane (3,4)		2.5	5	250
1,1-Dichloropropene (3,4)		2.5	2.5	250
cis-1,3-Dichloropropene (3,4,5)	0.200	0.5	1	50
p-Diethylbenzene (4)		2.0	4	200
Diisopropyl Ether (6)		2.0	4	200
1,4-Dioxane (5) (non-SIM)		250	100	5000
trans-1,3-Dichloropropene (3,4,5)	0.200	0.5	1	50
Ethanol (7)		N/A	1000	50000
Ethyl acetate		10.0	20	1000
Ethylbenzene (3,4,5)	0.100	0.5	1	50
Ethyl ether (3,4)		2.5	5	250
4-Ethyltoluene (4)		2.0	4	200
Ethyl methacrylate (3,4)		5.0	10	500
Ethyl-Tert-Butyl-Ether (6)		2.0	4	200
Freon-113 (5)		10.0	20	1000
n-Heptane (7)		N/A	5	250
Hexachlorobutadiene (3,4)		0.5	5	250
Hexachloroethane (7)		N/A	5	250
Hexane		1.0	1.0	50
2-Hexanone (3,4,5)	0.100	5.0	10	500
Iodomethane		5.0	5.0	250
Isopropyl Alcohol (IPA)		25		
Isopropylbenzene (3,4,5)	0.100	0.5	1	50
p-Isopropyltoluene (3,4)		0.5	1	50
Limonene (7)		N/A	5	250
Methyl Acetate (5)	0.100	20	20	1000
Methylene chloride (3,4,5)	0.100	3.0	10	500
Methyl Cyclohexane (5)	0.100	20	4	200
Methyl isothiocyanate (7)		N/A	5	250
Methyl Methacrylate		1.0	5	250

4-Methyl-2-pentanone (3,4,5)	0.100	5.0	10	500
Methyl-tert-butyl-ether (3,4,5)	0.100	1.0	2	100
Naphthalene (3,4)		2.5	5	250
Nitrobenzene (7)		N/A	5	250
2-Nitropropane (7)		N/A	5	250
n-Nonane (7)		N/A	5	250
n-Octane (7)		N/A	5	250
n-Butanol (5)		100	200	10000
n-Propylbenzene (3,4)		0.5	1	50
n-Propyl bromide		5.0		
Pentachloroethane		2.0	N/A	N/A
Styrene (3,4,5)	0.300	1.0	2	100
Tert-Butyl Alcohol (5)		30	100	5000
Tertiary-Amyl Methyl Ether (6)		2.0	4	200
Analyte	**Recommended Minimum Response Factor**	**RDL (µg/L)**	**RDL(µg/K**	**RDL(µg/K**
1,1,1,2-Tetrachloroethane (3,4)		0.5	1	50
1,2,4,5-Tetramethylbenzene (4)		2.0	4	200
1,1,2,2-Tetrachloroethane (3,4,5)	0.300	0.5	1	50
Tetrachloroethene (3,4,5)	0.200	0.5	1	50
Tetrahydrofuran (3)		10.0	20	1000
Toluene (3,4,5)	0.400	0.75	1	75
1,2,3-Trichlorobenzene (3,4,5)		2.5	5	250
1,2,4-Trichlorobenzene (3,4,5)	0.200	2.5	5	250
1,3,5-Trichlorobenzene (6)		2.0	5	250
1,1,1-Trichloroethane (3,4,5)	0.100	0.5	1	50
1,1,2-Trichloroethane (3,4,5)	0.100	0.75	1.5	75
Trichloroethene (3,4,5)	0.200	0.5	1	50
Trichlorofluoromethane (3,4,5)	0.100	2.5	5	250
1,2,3-Trichloropropane (3,4)		5.0	10	500
1,2,4-Trimethylbenzene (3,4)		2.5	5	250
1,3,5-Trimethylbenzene (3,4)		2.5	5	250
n-Undecane (7)		N/A	5	250
Vinyl acetate (3,4)		5.0	10	500
Vinyl chloride (3,4,5)	0.100	1.0	2	100
m/p-Xylenes (3,4,5)	0.100	1.0	2	100
o-Xylene (3,4,5)	0.300	1.0	2	100
1,4-Dioxane (5) SIM		3.0		
1,1,2,2-Tetrachloroethane SIM		0.1		

(1) Detection Limits are for Low-level Aqueous preserved samples.

(2) Detection Limits are for High-level Methanol preserved samples.
(3) Analyte reported by standard 8260 reporting list.
(4) Analyte reported by New York TCL reporting list.
(5) Analyte reported by New Jersey TCL reporting list.
(6) Analyte reported for New Hampshire in addition to standard 8260 reporting list.
(7) Analyte only reported for New York TCL report upon client request.

Note: Reporting Limits are based on standard 8260 reporting list, RL's may vary for New York and New Jersey reporting lists.

Table 2

QUALITY CONTROL ACCEPTANCE CRITERIA

Surrogate Spike Percent Recovery	Aqueous Limits		Soil Limits	
	Lower Control Limit	Upper Control Limit	Lower Control Limit	Upper Control Limit
1,2-Dichloroethane-d_4	70%	130%	70%	130%
4-Bromofluorobenzene	70%	130%	70%	130%
Toluene-d_8	70%	130%	70%	130%
Dibromofluoromethane	70%	130%	70%	130%

Table 3

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

Table 4

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
Fluorobenzene	2500	20	20	20	20	20	20	20	20	20	20
Dichlorodifluoromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorodifluoromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chloromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Vinyl chloride	2000	0.5	1	2	4	20	40	60	100	200	300
Bromomethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Trichlorofluoromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
Ethanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
1,1-Dichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
Carbon disulfide	2000	0.5	1	2	4	20	40	60	100	200	300
Freon-113	2000	0.5	1	2	4	20	40	60	100	200	300
Iodomethane	2000	0.5	1	2	4	20	40	60	100	200	300
Acrolein	2000	0.5	1	2	4	20	40	60	100	200	300
Allyl chloride	2000	0.5	1	2	4	20	40	60	100	200	300
Methylene chloride	2000	0.5	1	2	4	20	40	60	100	200	300
Isopropyl alcohol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
Acetone	2000	0.5	1	2	4	20	40	60	100	200	300
trans-1,2-Dichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl acetate	2000	0.5	1	2	4	20	40	60	100	200	300
Hexane	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl tert-butyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
tert-Butyl alcohol	2000	2.5	5	10	20	100	200	300	500	1000	1500
Diisopropyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
1,1-Dichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Halothane	2000	0.5	1	2	4	20	40	60	100	200	300
Acrylonitrile	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl tert-butyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
Vinyl acetate	2000	0.5	1	2	4	20	40	60	100	200	300
cis-1,2-Dichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
2,2-Dichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
Bromochloromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Cyclohexane	2000	0.5	1	2	4	20	40	60	100	200	300

Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
Chloroform	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl acetate	2000	0.5	1	2	4	20	40	60	100	200	300
Carbon tetrachloride	2000	0.5	1	2	4	20	40	60	100	200	300
Tetrahydrofuran	2000	0.5	1	2	4	20	40	60	100	200	300
Dibromofluoromethane	2500	20	20	20	20	20	20	20	20	20	20
1,1,1-Trichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
2-Butanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
2-Butanone	2000	0.5	1	2	4	20	40	60	100	200	300
1,1-Dichloropropene	2000	0.5	1	2	4	20	40	60	100	200	300
Heptane	2000	0.5	1	2	4	20	40	60	100	200	300
Benzene	2000	0.5	1	2	4	20	40	60	100	200	300
tert-Amyl methyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dichloroethane-d4	2500	20	20	20	20	20	20	20	20	20	20
1,2-Dichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Isobutyl alcohol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
2-Methyl-2-butanol	2000	2.5	5	10	20	100	200	300	500	1000	1500
Methyl cyclohexane	2000	0.5	1	2	4	20	40	60	100	200	300
Trichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
Dibromomethane	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
4-penten-2-ol	2000	2.5	5	10	20	100	200	300	500	1000	1500
2-Chloroethyl vinyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
Bromodichloromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl acrylate	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl methacrylate	2000	0.5	1	2	4	20	40	60	100	200	300
1,4-Dioxane	2000	N/A	40	80	200	1000	2000	3000	5000	10000	15000
cis-1,3-Dichloropropene	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorobenzene-d5	2500	20	20	20	20	20	20	20	20	20	20
Octane	2000	0.5	1	2	4	20	40	60	100	200	300
Toluene-d8	2500	20	20	20	20	20	20	20	20	20	20
Toluene	2000	0.5	1	2	4	20	40	60	100	200	300
4-Methyl-2-pentanone	2000	0.5	1	2	4	20	40	60	100	200	300
Tetrachloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
2-Nitropropane	2000	0.5	1	2	4	20	40	60	100	200	300

Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
Chloropicrin	2000	N/A	50	N/A	100	200	300	400	500	600	700
trans-1,3-Dichloropropene	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl isothiocyanate	2000	0.5	1	2	4	20	40	60	100	200	300
4-Methyl-2-pentanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
Ethyl methacrylate	2000	0.5	1	2	4	20	40	60	100	200	300
1,1,2-Trichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorodibromomethane	2000	0.5	1	2	4	20	40	60	100	200	300
1,3-Dichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dibromoethane	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butyl Acetate	2000	0.5	1	2	4	20	40	60	100	200	300
2-Hexanone	2000	0.5	1	2	4	20	40	60	100	200	300
Nonane	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Ethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,1,1,2-Tetrachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
p/m Xylene	2000	1	2	4	8	40	80	120	200	400	600
o Xylene	2000	1	2	4	8	40	80	120	200	400	600
Styrene	2000	1	2	4	8	40	80	120	200	400	600
1,4-Dichlorobenzene-d4	2500	20	20	20	20	20	20	20	20	20	20
Bromoform	2000	0.5	1	2	4	20	40	60	100	200	300
Butyl acrylate	2000	0.5	1	2	4	20	40	60	100	200	300
Isopropylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
4-Bromofluorobenzene	2500	20	20	20	20	20	20	20	20	20	20
Bromobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Decane	2000	0.5	1	2	4	20	40	60	100	200	300
n-Propylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,4-Dichlorobutane	2000	0.5	1	2	4	20	40	60	100	200	300
1,1,2,2-Tetrachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
4-Ethyltoluene	2000	0.5	1	2	4	20	40	60	100	200	300
2-Chlorotoluene	2000	0.5	1	2	4	20	40	60	100	200	300
1,3,5-Trimethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,3-Trichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
trans-1,4-Dichloro-2-butene	2000	0.5	1	2	4	20	40	60	100	200	300
4-Chlorotoluene	2000	0.5	1	2	4	20	40	60	100	200	300

Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
tert-Butylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Pentachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butyl methacrylate	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,4-Trimethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Limonene	2000	0.5	1	2	4	20	40	60	100	200	300
sec-Butylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
p-Isopropyltoluene	2000	0.5	1	2	4	20	40	60	100	200	300
1,3-Dichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,4-Dichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
trans-Decahydronaphthalene	2000	0.5	1	2	4	20	40	60	100	200	300
Undecane	2000	0.5	1	2	4	20	40	60	100	200	300
p-Diethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Hexachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
cis-Decahydronaphthalene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,4,5-Tetramethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dibromo-3-chloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
1,3,5-Trichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Nitrobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Hexachlorobutadiene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,4-Trichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Naphthalene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,3-Trichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,3-Dioxolane	2000	N/A	25	N/A	50	100	250	500	1000	1500	2000

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
Fluorobenzene	2500	10	10	10	10	10	10	10	10	10	10	10
Dichlorodifluoromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Chloromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Vinyl chloride	2000	0.2	0.5	2	10	30	50	80	100	120	160	200
Bromomethane	2000		0.5	2	10	30	50	80	100	120	160	200
Chloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Trichlorofluoromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
Ethanol	2000		0.5	2	10	30	50	80	100	120	160	200
1,1-Dichloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
Carbon disulfide	2000		0.5	2	10	30	50	80	100	120	160	200
Freon-113	2000		0.5	2	10	30	50	80	100	120	160	200
Iodomethane	2000		0.5	2	10	30	50	80	100	120	160	200
Acrolein	2000		0.5	2	10	30	50	80	100	120	160	200
Methylene chloride	2000		0.5	2	10	30	50	80	100	120	160	200
Isopropyl alcohol	2000		2.5	10	50	150	250	400	500	600	800	1000
Acetone	2000		0.5	2	10	30	50	80	100	120	160	200
trans-1,2-Dichloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
Methyl acetate	2000		0.5	2	10	30	50	80	100	120	160	200
Methyl tert-butyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
tert-Butyl alcohol	2000		2.5	10	50	150	250	400	500	600	800	1000
Diisopropyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
1,1-Dichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Halothane	2000		0.5	2	10	30	50	80	100	120	160	200
Acrylonitrile	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl tert-butyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
Vinyl acetate	2000		0.5	2	10	30	50	80	100	120	160	200
cis-1,2-Dichloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
2,2-Dichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
Bromochloromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Cyclohexane	2000		0.5	2	10	30	50	80	100	120	160	200
Chloroform	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl acetate	2000		0.5	2	10	30	50	80	100	120	160	200

Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
Carbon tetrachloride	2000	0.2	0.5	2	10	30	50	80	100	120	160	200
Tetrahydrofuran	2000		0.5	2	10	30	50	80	100	120	160	200
Dibromofluoromethane	2500	10	10	10	10	10	10	10	10	10	10	10
1,1,1-Trichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
2-Butanol	2000		2.5	10	50	150	250	400	500	600	800	1000
2-Butanone	2000		0.5	2	10	30	50	80	100	120	160	200
1,1-Dichloropropene	2000		0.5	2	10	30	50	80	100	120	160	200
Benzene	2000	0.2	0.5	2	10	30	50	80	100	120	160	200
tert-Amyl methyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dichloroethane-d4	2500	10	10	10	10	10	10	10	10	10	10	10
1,2-Dichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Isobutyl alcohol	2000		2.5	10	50	150	250	400	500	600	800	1000
2-Methyl-2-butanol	2000		2.5	10	50	150	250	400	500	600	800	1000
Methyl cyclohexane	2000		0.5	2	10	30	50	80	100	120	160	200
Trichloroethene	2000	0.2	0.5	2	10	30	50	80	100	120	160	200
n-Butanol	2000		2.5	10	50	150	250	400	500	600	800	1000
Dibromomethane	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
4-penten-2-ol	2000		2.5	10	50	150	250	400	500	600	800	1000
2-Chloroethyl vinyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
Bromodichloromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl acrylate	2000		0.25	1	5	15	25	40	50	60	80	100
Methyl methacrylate	2000		0.25	1	5	15	25	40	50	60	80	100
1,4-Dioxane	2000		100	400	500	600	1000	800	1000	1200	1600	2000
cis-1,3-Dichloropropene	2000		0.5	2	10	30	50	80	100	120	160	200
Chlorobenzene-d5	2500	10	10	10	10	10	10	10	10	10	10	10
Toluene-d8	2500	10	10	10	10	10	10	10	10	10	10	10
Toluene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Methyl-2-pentanone	2000		0.5	2	10	30	50	80	100	120	160	200
Tetrachloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
Chloropicrin	2000		30	50	80	120	200	160	400	200	320	400
trans-1,3-Dichloropropene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Methyl-2-pentanol	2000		2.5	10	50	150	250	400	500	600	800	1000

Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
Ethyl methacrylate	2000		0.5	2	10	30	50	80	100	120	160	200
1,1,2-Trichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Chlorodibromomethane	2000		0.5	2	10	30	50	80	100	120	160	200
1,3-Dichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dibromoethane	2000		0.5	2	10	30	50	80	100	120	160	200
2-Hexanone	2000		0.5	2	10	30	50	80	100	120	160	200
Chlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Ethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,1,1,2-Tetrachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
p/m Xylene	2000		1	4	20	60	100	160	200	240	320	400
o Xylene	2000		1	4	20	60	100	160	200	240	320	400
Styrene	2000		1	4	20	60	100	160	200	240	320	400
1,4-Dichlorobenzene-d4	2500	10	10	10	10	10	10	10	10	10	10	10
Bromoform	2000		0.5	2	10	30	50	80	100	120	160	200
Butyl acrylate	2000		0.25	1	5	15	25	40	50	60	80	100
Isopropylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Bromofluorobenzene	2500	10	10	10	10	10	10	10	10	10	10	10
Bromobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
n-Propylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,4-Dichlorobutane	2000		0.5	2	10	30	50	80	100	120	160	200
1,1,2,2-Tetrachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
4-Ethyltoluene	2000		0.5	2	10	30	50	80	100	120	160	200
2-Chlorotoluene	2000		0.5	2	10	30	50	80	100	120	160	200
1,3,5-Trimethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,3-Trichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
trans-1,4-Dichloro-2-butene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Chlorotoluene	2000		0.5	2	10	30	50	80	100	120	160	200
tert-Butylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Pentachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
n-Butyl methacrylate	2000		0.25	1	5	15	25	40	50	60	80	100
1,2,4-Trimethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
sec-Butylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200

Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
p-Isopropyltoluene	2000		0.5	2	10	30	50	80	100	120	160	200
1,3-Dichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,4-Dichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
p-Diethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
n-Butylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,4,5-Tetramethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dibromo-3-chloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
1,3,5-Trichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Hexachlorobutadiene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,4-Trichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Naphthalene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,3-Trichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,3-Dioxolane	2000		10	40	100	250	N/A	500	N/A	750	N/A	1000
Pentachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
1,4-Dioxane (SIM)	100		0.5	2	10	20	30	50	100	200	N/A	N/A
1,1,2,2-Tetrachloroethane (SIM)	100		0.05	0.1	0.2	0.5	1	2	5	10	N/A	N/A

- For Low Level Soil analysis, the calibration levels are the same in µg/Kg units.
- For High Level Soil and Oil analysis, the calibration levels are at 50x the levels listed due to sample preparation requirements.

TABLE 5

8260C Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Fluorobenzene	Chlorobenzene-d5	1,4-Dichlorobenzene-d4
Dichlorodifluoromethane	Toluene-d8 (surr)	Isopropylbenzene
Chloromethane	Toluene	Bromoform
Vinyl Chloride	Ethyl Methacrylate	1,4-dichloro-2-butane
Bromomethane	Trans-1,3-dichloropropene	1,1,2,2,-tetrachloroethane
Chloroethane	1,1,2-trichloroethane	4-bromofluorobenzene (surr)
Trichlorofluoromethane	2-hexanone	1,2,3-trichloropropane
Ethyl Ether	1,3-dichloropropane	trans-1,4-dichloro-2-butene
Freon 113	Tetrachloroethene	n-propylbenzene
Acrolein	Chlorodibromomethane	Bromobenzene
Acetone	1,2-dibromoethane	4-ethyltoluene
Ethanol	Chlorobenzene	1,3,5-trimethybenzene
1,1,-dichloroethene	1,1,1,2-tetrachloroethane	2-chlorotoluene
Tert-Butyl Alcohol	Ethylbenzene	4-chorotoluene
Methyl Acetate	p/m xylene	tert-butylbenzene
Carbon Disulfide	o xylene	1,2,4-trimethylbenzene
Methylene Chloride	Styrene	sec-butylbenzene
Acrylonitrile	Octane	p-isopropyltoluene
Methyl Tert Butyl Ether	2-Nitropropane	1,3-dichlorobenzene
Halothane	Methyl isothiocyanate	1,4-dichlorobenzene
Trans-1,2-dichloroethene	n-Butyl acetate	n-butylbenzene
Diisopropyl Ether	Nonane	p-diethylbenzene
Vinyl Acetate		1,2-dichlorobenzene
1,1-dichloroethane		1,2,4,5-tetramethylbenzene
Ethyl-Tert-Butyl-Ether		1,2-dibromo-3-chloropropane
2-butanone		1,3,5-trichlorobenzene
2,2-dichloropropane		1,2,4-trichlorobenzene
Cis-1,2-dichloroethene		Hexachlorobutadiene
Chloroform		Naphthalene
Bromochloromethane		1,2,3-trichlorobenzene
Tetrahydrofuran		Cyclohexanone
Dibromofluoromethane		Nitrobenzene

(surr)		
1,1,1-trichloroethane		Pentachloroethane
Cyclohexane		Decane
1,1-dichloropropene		Limonene
Carbon Tetrachloride		Trans-Decahydronaphthalene
Tertiary-Amyl Methyl Ether		Undecane
1,2-dichloroethane-d4 (surr)		Hexachloroethane
1,2-dichloroethane		Cis-Decahydronaphthalene
Benzene		
Trichloroethene		
Methyl Cyclohexane		
1,2-dichloropropane		
Bromodichloromethane		
1,4-Dioxane		
Dibromomethane		
2-Chloroethylvinyl Ether		
4-methyl-2-pentanone		
Cis-1,3-dichloropropene		
Iodomethane		
Methyl methacrylate		
n-Butanol		
Ethyl acetate		
Isopropyl Alcohol (IPA)		
Hexane		
n-Propyl bromide		
Chlorodifluoromethane		
Allyl chloride		
Heptane		

TABLE 6
8260C Quantitation Ions

Analyte	Quantitation Ion	Analyte	Quantitation Ion
Dichlorodifluoromethane	85	Ethyl Methacrylate	69
Chloromethane	50	Trans-1,3-dichloropropene	75
Vinyl Chloride	62	1,1,2-trichloroethane	83
Bromomethane	94	2-hexanone	43
Chloroethane	64	1,3-dichloropropane	76
Trichlorofluoromethane	101	Tetrachloroethene	166
Ethyl Ether	74	Chlorodibromomethane	129
Freon 113	101	1,2-dibromoethane	107
Acrolein	56	Chlorobenzene	112
Acetone	43	1,1,1,2-tetrachloroethane	131
1,1,-dichloroethene	96	Ethylbenzene	91
Tert-Butyl Alcohol	59	p/m xylene	106
Methyl Acetate	43	o xylene	106
Carbon Disulfide	84	Styrene	104
Methylene Chloride	76	Isopropylbenzene	105
Acrylonitrile	53	Bromoform	173
Methyl Tert Butyl Ether	73	1,4-dichloro-2-butane	55
Halothane	117	1,1,2,2,-tetrachloroethane	83
Trans-1,2-dichloroethene	96	1,2,3-trichloropropane	75
Diisopropyl Ether	45	Trans-1,4-dichloro-2-butene	53
Vinyl Acetate	43	n-propylbenzene	91
1,1-dichloroethane	63	Bromobenzene	156
Ethyl-Tert-Butyl-Ether	59	4-ethyltoluene	105
2-butanone	43	1,3,5-trimethybenzene	105
2,2-dichloropropane	77	2-chlorotoluene	91
Cis-1,2-dichloroethene	96	4-chorotoluene	91
Chloroform	83	tert-butylbenzene	119
Bromochloromethane	128	1,2,4-trimethylbenzene	105
Tetrahydrofuran	42	sec-butylbenzene	105
1,1,1-trichloroethane	97	p-isopropyltoluene	119
Cyclohexane	56	1,3-dichlorobenzene	146
1,1-dichloropropene	75	1,4-dichlorobenzene	146
Carbon Tetrachloride	117	n-butylbenzene	91
Tertiary-Amyl Methyl Ether	73	p-diethylbenzene	119
1,2-dichloroethane	62	1,2-dichlorobenzene	146
Benzene	78	1,2,4,5-tetramethylbenzene	119
Trichloroethene	95	1,2-dibromo-3-chloropropane	75
Methyl Cyclohexane	83	1,3,5-trichlorobenzene	180
1,2-dichloropropane	63	1,2,4-trichlorobenzene	180
Bromodichloromethane	83	Hexachlorobutadiene	225
1,4-dioxane	88	Naphthalene	128
Dibromomethane	93	1,2,3-trichlorobenzene	180
2-Chloroethylvinyl Ether	63	Ethanol	45
4-methyl-2-pentanone	58	Cyclohexanone	55
Cis-1,3-dichloropropene	75	Ethyl acetate	43

TABLE 6
8260C Quantitation Ions (continued)

Analyte	Quantitation Ion	Analyte	Quantitation Ion
Toluene	92	Iodomethane	142
Methyl methacrylate	69	n-Butanol	56
Pentachloroethane	167	Isopropyl Alcohol (IPA)	45
Hexane	57	n-Propyl bromide	43
Chlorodifluoromethane	51	Iodomethane	142
Allyl chloride	76	Heptane	71
Octane	85	2-Nitropropane	41
Methyl isothiocyanate	73	n-Butyl Acetate	43
Nonane	57	Decane	57
Limonene	68	Undecane	57
trans-Decahydronaphthalene	138	cis-Decahydronaphthalene	138
Hexachloroethane	117	Nitrobenzene	77

Table 7

List of 8260 Difficult Analytes:

1,1,2,2-Tetrachloroethane
1,2-Dibromo-3-chloropropane (DBCP)
1,4-Dioxane
2-Butanone
2-chloroethylvinyl ether
2-Hexanone
2,2-dichloropropane
4-Methyl-2-pentanone
Acetone
Bromoform
Bromomethane
Carbon disulfide
Chloroethane
Chloromethane
cis-1,3-Dichloropropene
Dichlorodifluoromethane (Freon 12)
Ethanol
Iodomethane
Isobutyl Alcohol
Naphthalene
Nitrobenzene
n-butanol
Styrene
Tert-Butyl Alcohol
Trichlorofluoromethane (Freon 11)
Isopropyl Alcohol (IPA)

pH, Soil and Waste

References: **Method 9045D, Soil and Waste pH,** Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 4, November 2004.

Method 9040C, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 3, November 2004.

NJDEP Site Remediation Program, Data of Known Quality Protocol, Version 1, April 2014

1. Scope and Application

Matrices: Soils and wastes, including solids, sludges, or non-aqueous liquids.

Definitions: See Alpha Laboratories Quality Manual Appendix A

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the pH meter and in the interpretation of pH data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The sample is mixed with reagent water, and the pH of the resulting aqueous solution is measured.

2.1 Method Modifications from Reference

The pH meter that is utilized will compensate for the temperature of the sample. Therefore, the temperature is not reported with the data. However, the sample is analyzed at room temperature. Only one aliquot is used for measurement.

3. Reporting Limits

None.

4. Interferences

4.1 Errors will occur when the electrode becomes coated. If an electrode becomes coated with an oily material that will not rinse free, the electrode can (1) be cleaned with an ultrasonic bath, or (2) be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with water, or (3) be cleaned per the manufacturer's instructions.

4.2 Samples with a very low or very high pH may give incorrect readings on the meter.

For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode.
Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

4.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by rinsing with distilled water. An additional treatment with hydrochloric acid (1:10) may be necessary to remove any remaining film.

4.4 Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference should be controlled with instruments having temperature compensation. The second source of temperature effects is the change of pH due to changes in the sample as the temperature changes. This error is sample-dependent and cannot be controlled. However, prior to analysis the samples are brought to room temperature (20 – 25 °C).

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in 4-ounce glass jars.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are to be analyzed as soon as possible after sampling. Samples are stored at 4 ± 2°C. Samples that are to be run in conjunction with NJ-Hex DAK acceptance criteria must be analyzed within 24 hours of Hex sample preparation.

7. Equipment and Supplies

7.1 **pH Meter: "Black"** Orion Research, expandable ion analyzer EA 940. Laboratory benchtop model. **"White"** Hanna pH temperature bench meter (HI5521-01). **"White 3"** Hanna pH temperature bench meter (HI2002-01) Or equivalent.

7.2 pH electrode: Accuphast electrode with automatic temperature compensation Fisher Catalog #13-620-296. Incorporates measuring and referenced functions; filled with AgCl solution. Hanna (HI 1131B) Glass body, refillable, combination electrode for "white 3".

7.3 Beakers: 50mL glass or plastic

7.4 Magnetic Stirrer

7.5 Teflon-coated stirring bar

7.6 Analytical Balance: Capable of weighing 0.1g

7.7 Kimwipes

8. Reagents and Standards

8.1 Reagent Water: All references to water in this method refer to reagent water.

8.2 pH Buffers: Commercially available pH 4 (or 4.01), pH 7, pH 10 (or 10.01). In addition, an alternate source of pH 7 Buffer is necessary. All Buffers must have been validated by comparison to NIST standards. Certificate of analysis is required. Buffers are stored at room temperature and expire upon manufacturer's specified date.

9. Quality Control

9.1 Blank(s)

Not applicable.

9.2 Laboratory Control Sample (LCS)

One LCS is analyzed with each batch of 20 samples or less. It is a pH 7 buffer of a different source than the calibration buffer. Results must be within ± 0.05.

9.3 Initial Calibration Verification (ICV)

Refer to LCS Section 9.2.

9.4 Continuing Calibration Verification (CCV)

A CCV of pH 7 is analyzed at the end of the analytical run. Results must be within ± 0.2 units.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

One duplicate sample is analyzed per batch of 20 samples or less

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Calibration

- Calibration Verification (LCS)
- Sample analysis
- Duplicate Analysis
- Calibration Verification (CCV)

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation

In a 50mL beaker, weigh 20 grams of soil; record the weight. Add 20mL of reagent water, cover and continuously stir the suspension for 30 minutes, using a teflon stir bar on a magnetic stirrer. Let samples stand for 1 hour before measurement.

Additional dilutions are permissible if working with hydroscopic soils, salts or other problematic matrices.

NOTE: If the sample is hygroscopic and absorbs all the reagent water, begin again using 20 grams of sample and 40mL of reagent water.

NOTE: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous base. The electrode may need to be cleaned (Section 4.1) if it becomes coated with an oily material.

10.2 Initial Calibration

The pH meter is calibrated on a daily basis using three pH buffers (Section 8.1). Follow manufacturer's instructions for a 3-point calibration of the pH meter. The results of the calibration must be recorded in the pH Calibration Log.

10.3 Equipment Operation and Sample Processing

10.3.1 Allow the sample suspension to stand for about 30 minutes to allow suspended material to settle out from the suspension.

10.3.2 Immerse the pH electrode just below the suspension and allow pH meter to stabilize. Note and record the sample pH in the Laboratory notebook. Report sampletemperature at the time of measurement.

10.3.3 Rinse the electrode thoroughly between samples, using reagent water.

10.4 Continuing Calibration

Prior to sample analysis, the calibration is initially verified by using a pH 7 buffer from a source other than the source used for calibration. The results must be within ± 0.2. If this criterion is not met, the meter must be re-calibrated before sample analysis can begin.

10.5 Preventative Maintenance

The pH probe is rinsed with DI and gently dried with a KimWipe between sample readings.

11. Data Evaluation, Calculations and Reporting

pH is read directly from the pH meter. No calculations are necessary.

For pH readings of less than 1, the reported result is: pH <1.

For pH readings greater than 10, report result in 3 significant figures.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the maintenance logbook.

The pH electrode is replaced as necessary.

Review of standards for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the LCS or CCV recovery falls outside the designated acceptance range, the laboratory performance for the parameter is judged to be out of control, and the problem must be immediately identified and corrected. Re-calibration of the meter is necessary prior to sample analysis. All samples analyzed since the last acceptable QC standard must be reanalyzed following re-calibration of the meter.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

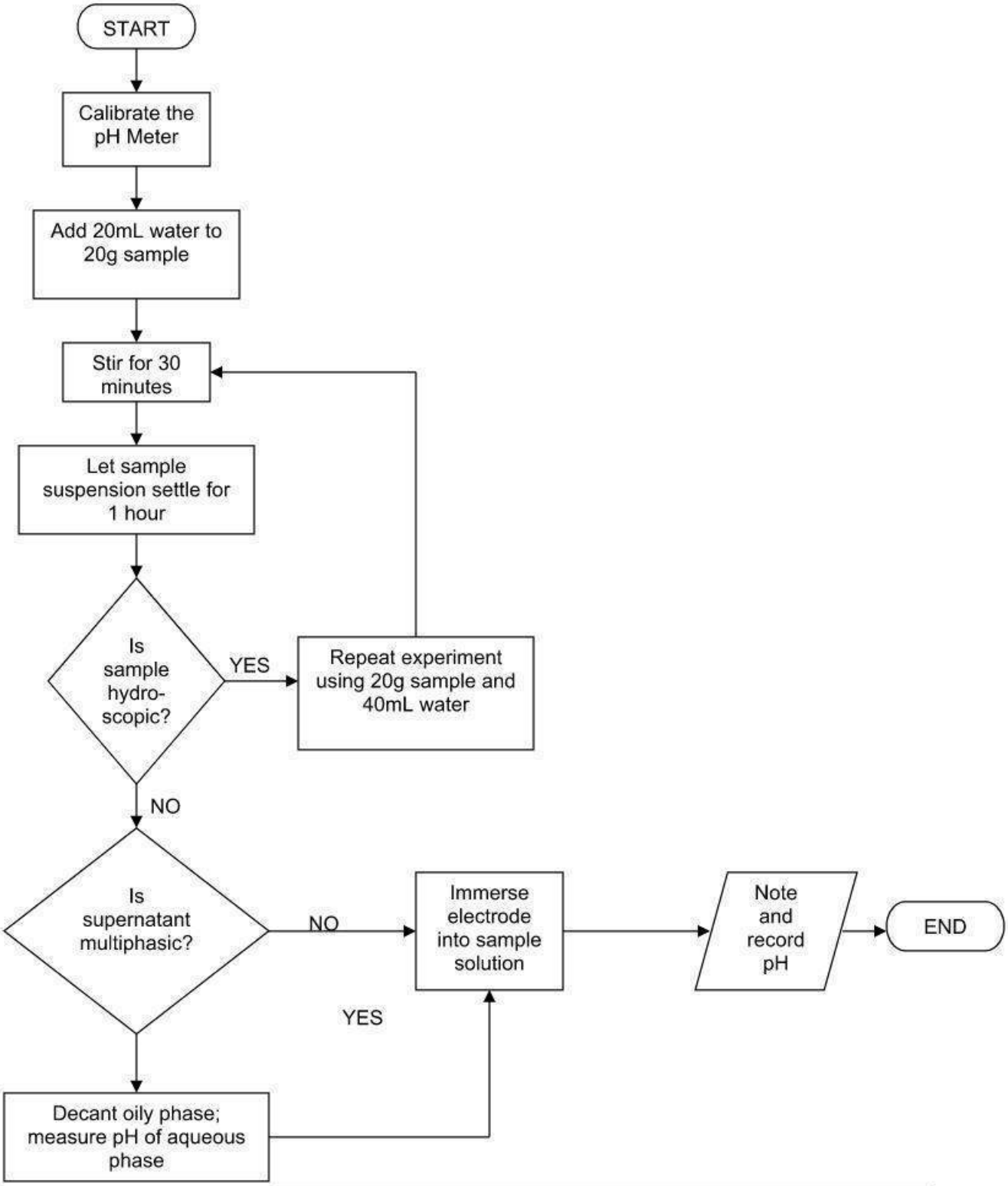
SOP/1728 Waste Management and Disposal SOP

16. Attachments

Flow Chart: Soil and Waste pH

Flow Chart:

Soil and Waste pH



Reactive Sulfide and Reactive Cyanide

Reference Method: This standard operating procedure (SOP) is a performance-based method. This SOP describes the procedure as developed by Alpha Analytical.

1. Scope and Application

Matrices: This method is applicable to water, wastewater, seawater, soil and solid samples with the condition that the samples combined with acids do not form explosive mixtures.

Definitions: See Alpha Analytical Quality Manual

The regulation in 40CFR 261.23 defines reactive wastes to include wastes that have any of the following properties:

(1) readily undergo violent chemical change;

(2) react violently or form potentially explosive mixtures with water;

(3) generate toxic fumes when mixed with water or, in the case of cyanide- or sulfide-bearing wastes, when exposed to mild acidic or basic conditions;

(4) explode when subjected to a strong initiating force;

(5) explode at normal temperatures and pressures; or

(6) fit within the Department of Transportation's forbidden explosives, Class A explosives, or Class B explosives classifications.

This definition is intended to identify wastes that, because of their extreme instability and tendency to react violently or explode, pose a problem at all stages of the waste management process. The definition is to a large extent a paraphrase of the narrative definition employed by the National Fire Protection Association. The Agency chose to rely almost entirely on a descriptive, prose definition of reactivity because most of the available tests for measuring the variegated class of effects embraced by the reactivity definition suffer from a number of deficiencies.

This procedure releases only the hydrogen sulfide evolved at the test conditions. It is not intended to measure forms of sulfide other than those that are evolvable under the test conditions.

This test measures only the hydrocyanic acid evolved at the test conditions. It is not intended to measure forms of cyanide other than those that are evolvable under the test conditions.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

An aliquot of acid is added to a fixed volume or weight of sample in a closed system. The generated gas is swept into a scrubber. The analyte is quantitated.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

The reported reactive sulfide detection limit is 1.0 mg/L (aqueous samples) and 10mg/kg (solid samples).

The reported reactive cyanide detection limit is 1.0 mg/L (aqueous samples) and 10mg/kg (solid samples).

4. Interferences

4.1 Reactive Sulfide Interferences

4.1.1 Strong reducing agents interfere in the methylene blue method by preventing formation of the blue color.

4.1.2 Thiosulfate at concentrations about 10mg/L may retard color formation or completely prevent it.

4.1.3 High Concentration of sulfide itself may inhibit blue color development. To avoid the possibility of reporting false negative results, test a 1000x dilution of the scrubber solution to see if blue color develops. (Refer to Section 10.3.1).

4.1.4 Ferrocyanide is a positive interference since it produces a blue color; distillation process will minimize interference.

4.2 Reactive Cyanide Interferences

Reactive Cyanide interferences are undetermined.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. Therefore, analysts should take every precaution by utilizing latex/nitrile gloves, safety glasses and a lab coat to minimize or eliminate exposure. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected with minimum aeration in either a glass or plastic bottle or jar. Fill the container completely and cover, ensuring no head space is present.

6.2 Sample Preservation

Samples are stored at 4 ± 2° C.

6.3 Sample Shipping
No special shipping requirements.

6.4 Sample Handling
Aqueous samples are analyzed with 7 days of collection. Soil samples are analyzed within 14 days of collection.

7. Equipment and Supplies

7.1 Spectrophotometer: Genesys 10vis (or equivalent); for use at a wavelength of 664nm and 578nm with cells providing light paths of 1cm.

7.2 Centrifuge Tubes: 50mL capacity

7.3 Pipets: Class A, glass, volumetric, various volumes

7.4 Volumetric Flasks: Class A, various volumes

7.5 Non-rubber Flexible Tubing: For connection from pump supply to apparatus.

7.6 Analytical Balance: Capable of weighing to 0.001g.

7.7 Midi Distillation System: Lab-Crest (or equivalent); 10-position distillation unit equipped with a full set of glassware including 10 each of the following:

7.7.1 Reaction Flask: Flat-bottom flask with sufficient volume (over 100mL) to contain violent reactions; has permanent 50mL indicator.

7.7.2 Absorption Flask: Identical to Reaction Flask (Section 7.7.1).

7.7.3 Reflux Impinger: Fits the reaction flask; has air and reagent intake, full-length impinger, and hose barb.

7.7.4 Cold Finger: Fits the Reflux Impinger; has two hose barbs for water circulation, which is not utilized for this test. It can therefore be substituted with a glass or plastic stopper of the same size.

7.7.5 Absorption Impinger: Full-length impinger with 13mm, medium porosity glass filter, and two hose barbs.

8. Reagents and Standards

8.1 Reagent water: Deionized water (DI) produced by Alpha's water treatment system.

8.2 Sulfide Calibration Stock Standard: Weigh 1.5779 gram of $Na_2S \cdot 9H_2O$ in a 200mL volumetric flask and fill to top with DI water. Mix by swirling gently to prevent oxygen entrapment. Store in the refrigerator in the amber glass bottle. Solution expires one month from the date of preparation. Final concentration is 1097 mg/L

8.3 Sulfide 1 Reagent: Available commercially from HACH Company. Store at 4 ± 2°C. Reagent expires on manufacturer's specified expiration date.

8.4 Sulfide 2 Reagent: Available commercially from HACH Company. Store at 4 ± 2°C. Reagent expires on manufacturer's specified expiration date.

8.5 Sulfide Calibration Standards: These standards are used to generate the calibration curve in Section 9.1.1. Prepare a series of sulfide solutions by diluting the Sulfide Calibration Stock Standard (Section 8.2) with 0.1N NaOH (Section 8.6.2).

Into seven 100mL volumetric flasks, pipet 1mL, 2mL, 5mL, 10mL, 15mL, 30mL and 0mL of the sulfide stock standard (Section 8.2) Bring each flask to volume using 0.1N NaOH solution (Section 8.6.2).

The concentration of the calibration standards is determined by the results of the standardization of the stock sulfide standard, as described in Section 8.2.1. The lowest calibration standard must be 0.1mg/L or less. If necessary, add an additional point to the curve by pipetting 0.5mL of Stock Sulfide Standard (Section 8.2) into an eighth 100mL volumetric flask.

Note: This calibration curve is identical to calibration curve, used for Total Sulfide.

8.6 Sodium Hydroxide, NaOH: In pellet form.

8.6.1 Sodium Hydroxide Solution, NaOH, 1N: Dissolve 40g NaOH (Section 8.11) in 1L of DI water. Store at room temperature. Expires three months from the date of preparation.

8.6.2 Sodium Hydroxide Solution, NaOH, 0.1N: In a 100mL volumetric flask, pipet 10mL of 1N NaOH (Section 8.11.1). Bring to volume with DI water. Store at room temperature. Expires three months from the date of preparation.

8.7 Sulfide LCS Stock Standard: Using a different source than was used for the Calibration Stock Standard. (8.2)

8.8 Sulfide LCS Standard: Pipet 0.5mL of LCS Sulfide Stock Standard (Section 8.7) into a 25mL volumetric flask. Bring to volume with DI water. True value: 22 mg/l.

8.8.1 Sulfide ICV / CCV Standard: Pipet 1.0 mL of LCS Sulfide Standard (Section 8.7) into a 50mL volumetric flask, add 5 ml of 1N NAOH. Bring to volume with DI water. Acceptance criteria +/- 25%; if failed outside of acceptance criteria, data is invalid.

8.9 Cyanide LCS/ICV/CCV Stock Standard, 1000ppm: Commercially available with a certificate of analysis. This must be from a different source than the Calibration Standard Stock. Store at 4 ± 2 °C. Expires on manufacturer's specified expiration date.

8.9.1 Cyanide LCS/ICV/CCV Working Standard, 40ppm: To a 25mL volumetric flask, add 1mL of 1000ppm cyanide LCS Stock Standard (Section 8.9). Bring to volume with DI water. Prepare fresh on each day of use.

8.9.1.1 Cyanide ICV/CCV 0.8ppm Standard: Into a 25mL volumetric flask, pipet 0.5mL of the 40ppm cyanide working calibration standard (Section 8.9.1). Bring to volume using 0.1N NaOH (Section 8.6.2) (5 ml of 1N NaOH can be used and volume brought up to 50 ml with DI. This standard is prepared fresh at the time of use.

8.10 Cyanide 1000ppm Stock Calibration Standard Stock Standard: Commercially purchased with a certificate of analysis. The ICV stock must be a different lot than the calibration standards.

8.10.1 Cyanide 10ppm Working Calibration Standard: Into a 100mL volumetric flask, pipet 1mL of 1000ppm cyanide stock calibration standard (Section 8.10) and bring to volume with 0.1N NaOH (Section 8.6.2). This standard expires after one week. Store in a brown bottle at room temperature, out of direct light.

8.10.1.1 0.0ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 0.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.2 0.05ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 0.5mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.3 0.1ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 1.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.4 0.2ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 2.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.5 0.4ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 4.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.6 0.5ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 5.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.11 Chloramine-T Solution: Dissolve 1.0g of Chloramine-T in 100mL DI water. Prepare fresh on each day of use.

8.12 Sodium dihydrogen phosphate monohydrate, $NaH_2PO_4 \cdot H_2O$

8.12.1 Phosphate Buffer: Dissolve 138g sodium dihydrogen phosphate monohydrate (Section 8.12) in DI water and dilute to 1L. Store at room temperature. Expires one month from the date of preparation.

8.13 Barbituric acid: Store at room temperature. No expiration date.

8.14 Pyridine: Store in Flammables cabinet. Expires on manufacturer's specified expiration date.

8.15 Concentrated Hydrochloric Acid, HCl: Store in Acids cabinet. No expiration date.

8.16 6M Hydrochloric Acid (HCl): Dilute 500mL of concentrated HCl (Section 8.20) into 500mL DI water. Store at room temperature. No expiration date.

8.17 Pyridine-Barbituric Acid Reagent: Place 60g barbituric acid (Section 8.13) in a 1000mL volumetric flask and add just enough DI water to wash sides of flask and wet barbituric acid. Add 300mL pyridine (Section 8.14) and mix. Add 60mL conc HCl (Section 8.15), mix and cool to room temperature. Dilute to volume and mix until barbituric acid is dissolved. The solution is stable for one month if stored in an amber bottle at 4 ± 2 °C. Discard if precipitate develops.

8.18 Concentrated Sulfuric Acid, H_2SO_4: Store in Acids cabinet. Expires on manufacturer's specified expiration date.

8.19 Reactivity Stock Solution: Add 2.8mL of conc H_2SO_4 (Section 8.18) to a 1L volumetric flask and bring to volume with DI. Prepare fresh on each day of use.

8.19.1 Reactivity Working Solution, 0.025M: Add 100mL of reactivity stock solution (Section 8.19) to a 1L volumetric flask and bring to volume DI.

8.20 Ottawa sand

8.21 Boiling chips

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Prepare and analyze one Method Blank per batch of 20 samples or less. Blank results must be less than the RL. If this criterion is not met, a batch narrative is included with the Client Report and/or the sample batch is reanalyzed. Refer to Section 12 for corrective actions.

For soils: weigh 1.0 g of Ottawa sand, for water- use 10 ml if DI and analyze with batch.

9.2 Laboratory Control Sample (LCS)

9.2.1 Reactive Sulfide

Run one LCS using the LCS Working Solution per batch of 20 samples or less. .

For waters: to 10 ml of DI add 2.0 ml of LCS Standard (8.8); final concentration is 4.4 mg/l

For soils: to 1.0 g of Ottawa sand add 2.0 ml of LCS Standard (8.8); final concentration 44 mg/kg

The LCS must be recovered within 60 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.2.2 Reactive Cyanide

Run one LCS using the LCS Working Solution per batch of 20 samples or less.

For waters: to 10 ml of DI add 1.0 ml of LCS Standard (8.9.1); final concentration is 4.0 mg/l

For soils: to 1.0 g of Ottawa sand add 1.0 ml of LCS Standard (8.9.1); final concentration 40 mg/kg

The LCS must be recovered within 30 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.3 Initial Calibration Verification (ICV)

9.3.1 Reactive Sulfide

The ICV is analyzed at the beginning of each sample batch. The ICV must be recovered within 75 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.3.2 Reactive Cyanide

An ICV is analyzed at the beginning of each sample batch. The ICV must be recovered within 75 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.4 Continuing Calibration Verification (CCV)

9.4.1 Reactive Sulfide

A CCV is read on the spectrophotometer after every 10 samples and at the end of each sample batch. The CCV must be recovered within 75 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.4.2 Reactive Cyanide

A CCV is read on the spectrophotometer after every 10 samples and at the end of each sample batch. The CCV must be recovered within 75 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

Prepare and analyze one sample in duplicate per batch of 20 samples or less. The %RPD for liquid samples must be $\leq$ 25%. For solid/soil samples, the %RPD must be $\leq$ 40%. If %RPD is not within the specified range, the sample and its duplicate must be narrated and/or reanalyzed. Refer to Section 12 for corrective actions.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Generate calibration curves.
- Prepare samples and QC samples.
- Analyze samples and QC samples.
- Calculate results.

10. Procedure

10.1 Equipment Set-up

10.1.1 Calibration Curve Generation

Calibration curves are generated yearly or when ICV/CCV are not passing acceptance criteria.

10.1.1.1 Reactive Sulfide

Prepare a calibration curve using the standards in Section 8.5. Analyze the standards as described in Section 10.3.1. Plot the concentration vs. absorbance. Determine the slope and the y-intercept. The correlation coefficient must be $\geq$ 0.995.

10.1.1.2 Reactive Cyanide

Prepare a calibration curve using the standards in Section 8.10. Analyze the standards as described in Section 10.3.2. Plot the concentration vs. absorbance. Determine the slope and the y-intercept. The correlation coefficient must be $\geq$ 0.995.

10.1.2 Sample Preparation

10.1.2.1 Add 10mL of 1N NaOH solution to the absorption flask, and bring to a volume of 50mL with DI.

10.1.2.2 Weigh 1g of solid sample or 10mL of liquid sample into a 50mL centrifuge tube and record the weight/volume in the laboratory notebook. Label the tube and secure the cap. For solid Blank and LCS, use PTFE boiling stones or Ottawa sand. Add the contents of the tube to the reaction vessel. Record the tube number and corresponding sample number in the laboratory notebook. Assemble the Midi Distillation Unit.

10.1.2.2.1 Ensure the pump is plugged in and connected to the Midi unit via a Teflon hose. After adding the sample to the reaction vessel (large tube in the back of the unit), place the absorption flask in the front of the unit ; flask should be filled with NaOH. Place the reflux impinger in the reaction flask and place the cold finger (or stopper) into the impinger. Make sure that all of the glass connections are tight. Place the absorption impinger into the absorption flask and make sure that the glass seal is tight. Connect the absorption impinger to the reaction impinger by connecting the front hose of the absorption impinger to the Midi Distillation unit (where the air comes out) and the rear hose to the reaction impinger. Make sure that all glass connections are tight and that all of the hoses are snug.

10.1.2.3 Turn on the pump and adjust the air flow rate of air so that the gas scrubbers produce small bubbles (approx. 60mL/minute).

10.1.2.4 Add the appropriate LCS to their respective reaction vessels. Then add the reactivity solution (Section 8.19.1) to each reaction vessel so that the final volume is about 50mL.

10.1.2.5 Make sure all of the glassware is connected and all of the tubes are connected. All of the reflux impingers should now be producing air bubbles in the reaction (rear) vessels. For any are not, the glassware is not air tight. Run the pump for 30 minutes, and then shut the pump off. Record in the laboratory notebook the time started and the time ended.

10.1.2.6 Disconect the apparatus and bring the volume of the absorption tube to 100mL in DI with a volumetric flask. The normality of this hydroxide solution will be 0.1N.

10.1.2.7 Pour off 25mL into a 50mL centrifuge tube for Sulfide analysis. Pour off 20mL into a 50mL centrifuge tube for Cyanide analysis.

10.2 Initial Calibration

Refer to Sections 10.1.1 and 9.3.

10.3 Equipment Operation and Sample Processing

10.3.1 Reactive Sulfide Analysis

Take a 25mL aliquot of the calibration standards (Section 8.5) or of the volumized scrubber solution for each sample and color as follows:

Add 1.0mL Sulfide 1 reagent (Section 8.3) and swirl to mix. Add 1mL of Sulfide 2 reagent (Section 8.4). Let the reaction continue for 5 minutes. Excessive time between the addition of the "1" and "2" Sulfide coloring reagents causes low results by loss of H_2S as a gas before it has had time to react. The presence of sulfide will be indicated by the appearance of blue color. Wait 5 minutes and read the absorbance on the spectrophotometer (below).

Absorbance Determination: Set the spectrophotometer to a wavelength of 664nm. Zero the instrument with a portion of the blank sample. Read the absorbances of the samples and QC samples and record this information in the laboratory notebook.

If the absorbance of a sample is greater than the highest standard in the calibration curve, the sample must be diluted with 0.1N NaOH and reanalyzed as in Section 10.3.1 above. Record in the laboratory notebook any dilutions that are prepared.

NOTE: Very high sulfide concentrations interfere with blue color formation. For a sample in which high sulfide is suspected, but no blue color develops, make a 1000x dilution of the scrubber solution with 0.1N Color with Reagent 1 (Section 8.3) and Reagent 2 (Section 8.4). Experiment with various dilutions until the absorbance falls within the calibration curve.

10.3.2 Reactive Cyanide Analysis

Take a 20mL aliquot of the calibration standards (Section 8.10.1) or of the volumized scrubber solution and color as follows:

Add 5mL of phosphate buffer solution (Section 8.12.1), 2mL of chloramine-T solution (Section 8.11), 5mL of pyridine (Section 8.14). Bring final volume to 50mL with DI. The presence of a pink color indicates cyanide.

Absorbance Determination: Set the spectrophotometer to a wavelength of 578nm. Zero the instrument with a portion of the blank sample. Read the absorbances of the samples and QC samples and record this information in the laboratory notebook.

If the absorbance is greater than the highest standard in the calibration curve, the sample must be diluted with 0.1N NaOH and reanalyzed as in Section 10.3.2 above. Record in the laboratory notebook any dilutions that are prepared.

10.4 Continuing Calibration

Refer to Section 9.4.

10.5 Preventive Maintenance

The spectrophotometers are calibrated on a semi-annual basis by an instrument service company. Certificates are kept on file.

11. Data Evaluation, Calculations and Reporting

11.1 Reactive Sulfide

Calculate reactive sulfide concentration using the calibration curve generated in Section 10.1.1.1:

$$\text{Reactive Sulfide (mg/L)} = \frac{\text{Absorbance} - \text{y intercept}}{\text{Slope}} \times \text{Dilution factor}$$

$$\text{Reactive Sulfide (mg/kg)} = \frac{\text{Absorbance} - \text{y intercept}}{\text{Slope}} \times \frac{\text{Dilution factor}}{\text{Weight (g)}}$$

11.2 Reactive Cyanide

Calculate reactive cyanide concentration using the calibration curve generated in Section 10.1.1.2:

$$\text{Reactive CN (mg/L)} = \frac{\text{Absorbance} - \text{y intercept}}{\text{Slope}} \times \text{Dilution factor}$$

$$\text{Reactive CN (mg/kg)} = \frac{\text{Absorbance} - \text{y intercept}}{\text{Slope}} \times \frac{\text{Dilution factor}}{\text{Weight (g)}}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence, improper preservation and sample headspace (liquid samples) are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the method blank contains a reportable amount of cyanide of Sulfide, evaluate the method blank as follows. If the samples associated with the method blank are non-detect, or if a sample has an analyte concentration that is greater than 10X the concentration of the contaminated method blank, the data may be reported and the method blank results narrated on the final report. If sample results are positive, but at a concentration less than 10X the concentration of the method blank, then those samples should be re-digested and re-analyzed.

If the LCS fails acceptance criteria, then the entire batch must be re-digested and re-analyzed. If there is insufficient sample volume to re-digest, then the Client Services Department is notified, and the batch results are narrated on the final report as estimated.

If the ICV fails acceptance criteria, a fresh aliquot is poured and re-analyzed. If the standard still fails, prepare a fresh ICV and begin analysis again. If the ICV fails again, and the spectrophotometer is working properly, prepare a new calibration curve.

If the ICB fails acceptance criteria, a fresh aliquot is poured and re-analyzed. If the blank still fails, ensure that the spectrophotometer is “blanked” correctly. If the ICB failure continues, and the spectrophotometer working properly, prepare a new calibration curve.

If the CCV fails acceptance criteria, a fresh aliquot is poured and re-analyzed. If the standard still fails, prepare and analyze a fresh CCV and re-analyze the last 10 samples up to the last passing CCV. If the CCV recovery is greater than 125%, and all associated samples are non-detect, then analysis may be continued. If the CCV recovery is greater than 125%, any associated positive samples are re-prepared and re-analyzed.

If the CCB fails acceptance criteria, a fresh aliquot is poured and re-analyzed. If the CCB still fails, ensure that the spectrophotometer is working properly. Also ensure that the cell is clean and has no smudges, and is indexed correctly. When the problem has been corrected, all samples analyzed since the last passing CCB must be re-analyzed.

If %RPD of duplicates fails acceptance criteria, the non-conformance is narrated and the information is included on the final report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734, 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1734, 1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

Total Solids in Solid and Semisolid Samples

(Percent Solids)

Reference Method: **SM 2540 G,** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

1. Scope and Application

Matrices: Soils, solids and sludges.

Definitions: See Alpha Analytical Quality Manual

This method is applicable to the determination of total solids in such solid and semisolid samples as river and lake sediments, sludges separated from water and wastewater treatment processes, and sludge cakes from vacuum filtration, centrifugation, or other sludge dewatering processes.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results by completing an initial demonstration of capability

2. Summary of Method

A homogenized aliquot of sample is weighed in a tared dish and set in a 103º - 105ºC oven until dry. The sample and dish are cooled and re-weighed, thus the percent of solids in the original sample can be calculated.

2.1 Method Modifications from Reference

Aluminum pans are used instead of porcelain dishes. However, if the sample is corrosive, then the porcelain dishes are used.

5-10 grams of sample are used for soil and solid matricies.

One duplicate is analyzed per batch of 20 samples or less.

3. Reporting Limits

The Reported Detection Limit is 0.1%.

4. Interferences

4.1 Humidity: Humidity in the laboratory may cause samples to pick up moisture. When not being weighed, samples should be kept tightly capped or in a dessicator.

4.2 Large rocks / debris: Large rocks or debris may cause false high results and therefore should not be included in the sample aliquot.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. Personal protective equipment is to be worn at all times within the laboratory areas. At a minimum, a labcoat, gloves and safety glasses are worn.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass or plastic containers with minimal headspace. Containers are covered immediately to minimize the loss of sample moisture.

6.2 Sample Preservation

Samples are refrigerated at 4 ºC.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are kept refrigerated at 4 ºC until the time of analysis.

For samples received, marked and commented as **Foreign Soils** reference SOP 2296 Treatment of Foreign Soils.

For samples received, marked and commented as **Containing or May contain Asbestos** reference WI 2535 Asbestos Handling Procedures.

7. Equipment and Supplies

7.1 Analytical Balance: Capable of weighing to 0.01g

7.2 Aluminum Weighing Dishes or Pans

7.3 Porcelain Evaporation Dishes

7.4 Dessicator: With a color-indicator dessicant.

7.5 Drying Oven: Capable of maintaining 103 – 105 ºC.

7.6 Oven Trays

7.7 Computer: with connection to LIMS and the Analytical Balance (Sect. 7.1)

7.8 Baked Sand: created by and available in the extractions department

8. Reagents and Standards

None.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

One blank is analyzed per batch of 20 samples or less from the state of Maine. Blank recovery must be between 95%-105%. If this criterion is not met, the sample and it's duplicate are reanalyzed. Sand, baked to dryness in the extractions laboratory, is used for the blank sample.

9.2 Laboratory Control Sample (LCS)

Not applicable.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

One duplicate is analyzed per batch of 20 samples or less. Duplicate determinations must agree within 20%. If this criterion is not met, the sample and it's duplicate are reanalyzed.

If sample, used for batch duplicate, is non-homogeneous, then data may be reported with a narrative.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Create a Workgroup in LIMS
- Locate Samples and take possession of them in LIMS
- Create an ELN for the Workgroup
- Homogenize Sample
- Record the Tare weight of the evaporation dish, then the Gross weight for each sample one at a time
- Dry samples in the oven 3+ hours.
- Cool samples in the dessicator
- Record the Net Weight (1)
- Dry samples again for 1+ hour(s), unless samples were originally dried overnight. Due to running of analysis 24 hours a day overnight is interpreted to mean a minimum of 5 hours.

Samples from the State of Maine logged for ME-TS-2540 must always be dried again regardless of the length of time dried.

- Cool samples in the dessicator
- Record the Net Weight (2)
- Save and Send Data to LIMS

10. Procedure

10.1 Equipment Set-up

10.1.1 LIMS Knowledge: Prior to utilizing this SOP, the analyst must first be familiar with the operation of the Laboratory Information Management System (LIMS) and the generation of a sample batch or workgroup.

10.1.2 Porcelain Dish Preparation: Porcelain evaporation dishes are used only if a sample is corrosive to aluminum. To prepare the porcelain dishes, bake them in the 103 – 105 ºC drying oven for a minimum of 1 hour before placing them in the dessicator. Cool in the dessicator for a minimum of one hour.

10.1.3 Matrix and Volume Evaluation: Total Solids analysis should not be performed on plastic, caulking or oil. If logged for total solids analysis see login staff for assistance in altering the login in LIMS.

When taking an aliquot of sample for analysis, it is important that the sample volume is not exhausted to perform total solids before all other analysis has been performed. If the container has analysis printed on the alpha applied label other than total solids, ensure those tests have been performed prior to utilizing the remainder of the sample for total solids analysis.

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Generating a LIMS Batch

Utilizing a computer (Sect. 7.7), generate a LIMS batch of samples and assign a Workgroup (WG) number to the batch. When generating the batch, choose a sample that will be duplicated. Print out a copy of the LIMS batchsheet. For samples from the state of Maine, a blank must be analyzed.

10.3.2 Creating a Gravimetric Laboratory Notebook

From the menu bar in LIMS select: “Sample Data”, “Data”, “Logbooks”, “ELN-Wet Chem” and enter the workgroup number in the space provided. If this is the first time you have attempted to open a lab notebook for this workgroup a window will pop up confirming you wish to create a new gravimetric laboratory notebook.

Once you create the laboratory notebook the samples will populate in the spreadsheet with the Duplicate at the top. Above the list of samples. The reference sample for the Duplicate can be confirmed by double clicking the Sample Number of the Duplicate.

10.3.3 Taking the Tare Weight

10.3.3.1 With the cursor on the tare weight field in the ELN, weigh the corresponding empty dish on a tared balance. When the weight is stable, push the "Print" button on the balance. This will transfer the weight of the empty dish into the ELN.

10.3.4 Taking the Gross Weight

10.3.4.1 Homogenize the sample by mixing with a spatula or spoon.

10.3.4.2 Remove a 5 – 10g aliquot of soil sample or 20 – 25g of a sludge sample and place it in the weighing dish.

10.3.4.3 With the cursor on the cell for the "Gross Weight" corresponding to the sample to be weighed, zero the balance and weigh the dish plus the sample. When the weight is stable, push the "Print" button on the balance. This will transfer the weight into the ELN.

10.3.4.4 Place the dish onto an oven tray.

10.3.5.5 Once the samples have all been weighed and the weights recorded in the ELN, click save.

10.3.6 Drying the Samples : Phase I

10.3.6.1 Place the oven tray in the 103 – 105 ºC drying oven. In the ELN double click the Time In (1) field to populate the current time, edit as needed to reflect the time the samples were placed in the oven. Enter the temperature reading on the oven into the Temperature field.

10.3.6.2 After a minimum of three hours (samples with state of origin ME must be dried at least five hours), if samples appear dry, move the oven tray of dried samples to a dessicator. Allow to cool completely.

10.3.7 Taking the First Net Weight

10.3.7.1 In the ELN with the cursor on the "Net Weight (1)" field corresponding to the sample to be weighed, zero the balance and weigh the dish plus the sample. When the weight is stable, push the "Print" button on the balance. This will transfer the weight into the ELN.

10.3.7.2 Repeat Section 10.3.7.1 until the Net Weight (1) for all samples has been recorded.

10.3.7.3 Click on the "Save" button to save the weights.

10.3.8 Drying the samples : Phase II

10.3.8.1 If the samples were dried in the oven for less than five (but at least three) hours, the tray of samples must be placed back in the 103 – 105 ºC drying oven for a minimum of one hour. If the samples are from the state of Maine the samples must always be placed back in the oven for a minimum of one hour.

10.3.8.2 After drying, move the oven tray of samples to a dessicator. Allow to cool completely.

10.3.9 Taking the Second Net Weight

10.3.9.1 In the ELN with the "Net Weight(2)" field selected corresponding to the sample to be weighed, zero the balance and weigh the dish plus the sample. When the weight is stable, push the "Print" button on the balance. This will transfer the weight into the ELN.

10.3.9.2 Repeat Sections 10.3.9.1 until the Net Weight (2) for all samples has been recorded.

10.3.9.3 Click on the "Save" button to save the weights.

10.3.9.4 If Net Weight (1) and Net Weight (2) are within 4% or 50mg, the ELN will highlight the values light pink and display. If this is the case for all samples in the batch, proceed to Section 10.3.10.

10.3.9.5 If Net Weight (1) and Net Weight (2) are not within 4% or 50mg the ELN will highlight the values red and the words "Not Acceptable" will be displayed, repeat Sections 10.3.8 and 10.3.9 for those samples. This will allow the chemist to record a Net Weight (3), (4) or (5), until the values are within the acceptable range.

If the samples have been dried ≥ 24 hours, and the weights are still not within 4% or 50mg, consult the Department Supervisor as to how to proceed.

10.3.10 Saving the Batch

10.3.10.1 Click on the "Save" button to save the weights in the ELN.

10.3.10.2 Click on the "Send to LIMS" button in the ELN.

10.4 Continuing Calibration

Not applicable.

10.5 Preventative Maintenance

The temperature of the laboratory ovens is recorded constantly on the data logger. The chart recorder and the laboratory ovens are calibrated on an annual basis by an instrument service company. Certificates are kept on file.

Analytical balances are calibrated on a semi-annual basis by an instrument service company. Certificates are kept on file. The calibration of the balances is verified on a daily basis and records are kept in a Logbook.

11. Data Evaluation, Calculations and Reporting

The Excel Spreadsheet is programmed to calculate the Percent Solids results. This is the formula that is used for calculation:

$$\% \text{ Total Solids} = \frac{(A - B)}{(C - B)} \times 100$$

Where: A = Final Net Weight (weight of dried residue + dish, g)
B = Tare weight (weight of dish, g)
C = Initial Gross Weight (weight of wet sample + dish, g)

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Improper preservation is noted on the Sample Delivery Group form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial **(IDC)**

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Unless the containers are labeled as hazardous material (i.e. low flashpoint, ignitable, containing asbestos or high levels of toxic materials), the dried samples are disposed of into the trash.

If sample containers are labeled as hazardous, refer to the Chemical Hygiene Plan for waste handling and disposal instructions.

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

WI 2535 Asbestos Handling Procedures

SOP 2296 Treatment of Foreign Soils

16. Attachments

None.

Microwave Assisted Acid Digestion of TCLP Extracts

Reference Method No.: **EPA 3015A**

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Final Update IV, Revision 1, 2007

1. Scope and Application

Matrices: TCLP extracts only.

Definitions: See Alpha Analytical Quality Manual Appendix A

This digestion procedure is a hot acid leach for determining available metals used for the preparation of mobility-procedure extracts. Subsequently they are analyzed by inductively coupled argon plasma spectroscopy (ICP), for the following:

Aluminum	Antimony	Arsenic	Barium
Beryllium	Cadmium	Calcium	Chromium
Cobalt	Copper	Iron	Lead
Magnesium	Manganese	Molybdenum	Nickel
Potassium	Selenium	Silver	Sodium
Thallium	Vanadium	Zinc	

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Metals Manager, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

TCLP extract: A representative of 5mL of sample extract, and 45mL of DI water is digested in 2.5mL of $cHNO_3$ and 2mL cHCl in a fluorocarbon (PFA or TFM) digestion vessel for 22 minutes using microwave heating. After the digestion process, the sample is cooled and filtered, if necessary, into a clean sample digestion tube prior to analysis.

2.1 Method Modifications from Reference

Open vessels are utilized for digestion, and the system is not pressurized.

3. Reporting Limits

Refer to analytical method SOPs.

4. Interferences

4.1 Many samples that contain organics, such as TCLP extracts, will result in higher vessel pressures which have the potential to cause venting of the vessels. Venting can result in either loss of analytes and/or sample, which must be avoided.

4.2 Other interferences which can cause inconsistent readings are soap, sediment, high pH, and precipitation.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

NOTE: Caution must be taken when using 50% NH_4OH, 50% HCl or 10% HNO_3. They are corrosives and can cause harm to skin and eyes. When using these corrosives, one should wear a lab coat, gloves, and protective eyewear.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in 1L plastic bottles.

6.2 Sample Preservation

Samples for total metals analysis are preserved with conc HNO_3 to a pH <2.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are stored under refrigeration at 4 ± 2 °C.

7. Equipment and Supplies

7.1 Microwave: CEM MARS 6 Xpress

7.1.1 The microwave unit provides programs with a minimum of 574W, which can be programmed to within ±10 watts of the required power. Typical units provide a nominal 600W to 1200W power. The MARS6 provides up to 1600 W. Microwave

temperature and pressure are monitored and controlled. The microwave output for digestions is > 900 W of power.

7.1.2 The microwave unit cavity is corrosion-resistant and well ventilated.

7.1.3 All electronics are protected against corrosion for safe operation.

7.1.4 Fluorocarbon (PFA or TFM) Digestion Vessels: 120mL capacity.

7.1.5 A rotating turntable is employed to insure homogeneous distribution of microwave radiation within the unit. The speed of the turntable should be a minimum of 3rpm.

7.2 Graduated cylinder: 50mL, class A.

7.3 Volumetric Flasks: 50mL, class A.

7.4 Glass Fiber filters: Acid cleaned, 0.7 um nominal pore size

7.5 Whatman 40 or equivalent Filter Paper

7.6 Digestion Tubes: 50mL, calibrated, with caps.

7.7 Microwave Carousel

7.8 Pipets: 5mL.

8. Reagents and Standards

8.1 1:1 Hydrochloric Acid (HCl): Store at room temperature under a hood.

8.2 10% Nitric Acid (HNO_3): Store at room temperature under a hood.

8.3 Concentrated Nitric Acid ($cHNO_3$): Store at room temperature under a hood.

8.4 Concentrated (cHCl): Store at room temperature under a hood.

8.5 DI Water

8.6 1000ppm Single Element Stock Standards: These are commercially prepared standards for various elements. Store at room temperature. Standards expire upon manufacturer's specified date.

8.7 Spiking Solutions

Store at room temperature. Standards expire upon manufacturer's specified date.

8.7.1 ICP Spike Standard #3: Purchased commercially prepared, with a certificate of analysis. Contains the following: 2000ppm Arsenic, 50ppm Cadmium, 500ppm Lead, 2000ppm Selenium, 2000ppm Thallium.

8.7.2 ICP Spike Standard #1: Purchased commercially prepared, with a certificate of analysis. Contains the following: 2000ppm Aluminum, 2000ppm Barium, 50ppm Beryllium, 200ppm Chromium, 500ppm Cobalt, 250ppm Copper, 1000ppm Iron, 500ppm Manganese, 500ppm Nickel, 50ppm Silver, 500ppm Vanadium, 500ppm Zinc

8.7.3 **FPS:** To a 500mL volumetric flask, add 200mL of DI water and 25mL of tHNO3. Add 3mL of the well-shaken, room temperature ICP Spike Standard #3 (Section 8.7.1) and add 25mL of1000ppm Lead standard. Bring to volume with DI water. 0.5mL of this solution per 50mL of sample volume will yield the following concentrations in the spiked sample: 0.12ppm Arsenic, 0.05ppm Cadmium, 0.12ppm Selenium, 0.12ppm Thallium, and 0.51ppm.

8.7.4 **IPS:** To a 500mL volumetric flask, add 100mL DI water and 25mL of tHNO3. Add 50.0mL of the well-shaken, room temperature, ICP Spike Standard #1 (Section 8.7.2), 25.0mL of 1000ppm Antimony standard, and 2.5mL of 1000ppm Cadmium standard. Bring to volume with DI water. 0.5mL of this solution per 50mL of sample volume will yield the following concentrations in the spiked sample: 2ppm Aluminum, 2ppm Barium, 0.05ppm Beryllium, 0.2ppm Chromium, 0.5ppm Cobalt, 0.25ppm Copper, 1.0ppm Iron, 0.5ppm Manganese, 0.5ppm Nickel, 0.05ppm Silver, 0.5ppm Vanadium, 0.5ppm Zinc.

8.7.5 **Mixed Standard:** To a 500mL volumetric flask add 50mL of DI water and 25mL of tHNO3. Add 50mL of each of the following stock standards: 1000ppm Boron, 10,000ppm Calcium, 10,000ppm Magnesium, 1000ppm Molybdenum, 10,000ppm Potassium, 1000ppm Strontium, 10,000ppm Sodium, 1000ppm Titanium, and 1000ppm Tin. Bring to volume with DI water. 0.5mL of this solution per 50mL of sample volume will yield the following concentrations in the spike sample: 1.0ppm Boron, 10ppm Calcium, 10ppm Magnesium, 1.0ppm Molybdenum, 5ppm Potassium, 1.0ppm Strontium, 10ppm Sodium, 1.0 Titanium.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

One Blank is digested per matrix batch of 20 samples or less.

9.2 Laboratory Control Sample (LCS)

One LCS is digested per matrix batch of 20 samples or less.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

One Spiked sample is digested per matrix batch of 20 samples or less.

9.6 Laboratory Duplicate

One Duplicate sample is digested per matrix batch of 20 samples or less.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Acid rinse microwave vessels.
- Shake the sample well and dispense the appropriate volume into a numbered vessel.
- To each sample and QC sample aliquot, add 2.5mL cHNO_3 and 2 mL cHCl.
- Place vessels in a microwave carousel tray.
- Run the appropriate microwave program.
- Allow samples to cool.
- Acid rinse volumetric flasks and Teflon funnels.
- If necessary, filter the samples into 50 mL digestion tubes.
- Bring samples to the appropriate final volume with DI water.
- Transfer the samples in the digestion tubes with caps.

10. Procedure

10.1 Equipment Set-up

10.1.1 Turn on microwave.

10.1.2 Microwave Calibration

10.1.2.1 Microwave Power and Operational Conditions

The power is verified for each Microwave internally and monitored by the instrument software. Additionally, the rotational speed is set to be greater than or equal to 3 RPMs. Corrective action for a failing temperature check (power output) requires a recalibration by the manufacturer (CEM, Inc) before the unit can be returned to use.

Note: A report is generated by the unit on each run, the system automatically shuts down on failure to reach and hold the set temperature requirements or rotational speed. Any samples with failing temperatures are re-digested.

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Sample Digestion

10.3.1.1 Acid Rinse Fluorocarbon Digestion Vessel, as follows:

10.3.1.1.1 Wash with soap and tap water to remove any material if applicable.

10.3.1.1.2 Rinse 1 time with DI water.

10.3.1.1.3 Rinse 1 time with 10% HNO_3.

10.3.1.1.4 Rinse 3 times with DI water.

10.3.1.2 Acid rinse the graduated cylinders following Sections 10.3.1.1.3 through 10.3.1.1.6.

10.3.1.3 TCLP Extraction:

10.3.1.3.1 Shake the sample well, and pipet 5mL into a numbered vessel. Add 45mL of DI water.

10.3.1.3.2 Method Blank: Use 5mL of Method Blank extract and add 45mL of DI water

10.3.1.3.3 LCS: Use 5mL of Method Blank extract and add 45mL of DI water. Add 0.5 mL IPS, 0.5 mL FPS, and 0.5 Mix (Section 8.7). If the desired metal is not present in these standards, add the appropriate amount of desired metal standard stock 1000ppm solution. The appropriate amount will be determined by the Department Manager.

10.3.1.3.4 Matrix Spike (MS): Use 5mL of TCLP sample and add 45mL of DI water. Add 0.5 mL IPS, 0.5 mL FPS, and 0.5 Mix (Section 8.7). If the desired metal is not present in these standards, add the appropriate amount of desired metal standard stock 1000ppm solution. The appropriate amount will be determined by the Department Manager.

10.3.1.3.5 To each sample and QC sample aliquot, add 2.5mL of $cHNO_3$ (Section 8.3) and 2mL cHCl (Section 8.4).

10.3.2 Place vessels in a microwave carousel tray. If the carousel is not full, add extra vessels the volume of water the same as the volumes of the other samples in the carousel.

10.3.3 Place the full carousel in the microwave and run the appropriate program.

10.3.3.1 50mL volume samples, use the Liquid program: 41% of 1600W power, 22 minutes time.

10.3.4 When the microwave program ends, allow the samples to cool.

10.3.5 Transfer the samples into the 50mL digestion tubes.

10.3.6 If any of the samples in the batch contain sediment, those samples along with the method blank and the LCS must be filtered. Place a folded No.40 filter or equivalent into the appropriate digestion tube, and pre-wet with DI water prior to filtering samples. Filter samples into the 50mL digestion tubes.

10.3.7 Rinse each vessel 3 times with DI water and filter each rinseate into the digestion tube.

10.3.8 Bring samples up to the appropriate volume with DI water.

10.3.9 Transfer the sample in the labeled 50mL digestion tube capped.

10.4 Continuing Calibration

Not applicable.

10.5 Preventive Maintenance

Refer to Section 10.1.2 for Microwave Calibration information.

11. Data Evaluation, Calculations and Reporting

Refer to analytical method SOPs.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Refer to analytical method SOPs.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha #1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

SOP #1732 MDL/LOD/LOQ Generation

SOP# 1739 IDC/DOC Generation

SOP# 1728 Waste Management and Disposal

16. Attachments

None.

Separatory Funnel Extraction of Liquid Samples EPA 3510C

Reference Method: **EPA 3510C (EPH 608, EPA 625, EPA 608.3, EPA 625.1, and MA-DEP EPH)** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, December 1996.

1. Scope and Application

Matrices: This method is applicable to aqueous samples.

Definitions: Refer to Alpha Analytical Quality Manual.

This method describes the procedure for extracting water-insoluble and lightly water-soluble organic compounds from aqueous samples. The method also describes concentration techniques suitable for preparing the extract for the various determinative methods listed in Table 1.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, o r Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

A measured volume of sample is serially extracted with methylene chloride using a separatory funnel. Depending on the analytes to be detected, it may be necessary to adjust the pH of the aqueous sample prior to extraction (Table 1).

Any water is removed from the sample extract by filtering through a powder funnel containing approximately 60g of baked anhydrous sodium sulfate. The extract is then concentrated and, as needed, exchanged into a solvent compatible with the cleanup or determinative step being employed. The various cleanup methods used summarized in Table 1.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Reporting Limit information can be found in the analytical method SOPs.

4. Interferences

4.1 The most common cause of contamination is from improperly cleaned glassware and lab supplies. All glassware and re-useable extraction equipment must be scrupulously cleaned, following the Organic Extraction Glassware Cleaning and Handling SOP/1953 and Work instruction 10995, Solvent rinsing/filtering guide.

4.2 Impurities in solvents and reagents may also yield artifacts and/or interferences that may compromise the results of sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of extract preparation and analysis by preparing method blanks with each extraction batch. The same solvents and reagents are used for the method blank and the associated samples.

4.3 Phthalate esters contaminate many types of products used in the laboratory. Plastic materials must not contact the samples or extracts, as phthalates could be easily leached from the plastic. The exception is in the use of various pre-packed reagent cartridges (Florisil, Silica gel) used in the extract cleanup steps. Each new lot of cartridges is checked for contamination, and is monitored on an on-going basis through the analysis of method blanks.

4.4 Additional specific interference or contamination concerns are addressed in the various analytical SOPs.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents and when washing glassware.

5.2 All extract concentration steps must be performed in the extraction hoods. All solvent and extract transfers must also be handled in the hood.

5.3 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods.

5.5 All waste solvents must be transferred to the satellite waste storage containers located in the extraction lab. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

5.6 Inspect all glassware prior to use. Do not use any glassware that is chipped, cracked or etched if it could present a safety hazard. Damaged glassware is put aside for repair, otherwise discard the piece.

5.7 All Field Samples must be opened and handled in a hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Sample collection and preservation requirements are described in the various analytical method SOPs.

6.2 Sample Preservation

None.

6.3 Sample Shipping

See applicable Sample Custody SOP.

6.4 Sample Handling

All aqueous samples are stored, refrigerated, in the Organic Extraction Custody Refrigerators. Samples are removed from the refrigerator by the Chemist immediately prior to sample extraction. The Chemist must take custody of the samples by signing them out utilizing the LIMS, see Work Instruction 2517 ELN Procedure and Work Instruction 2421 Labeling and Generating Work Groups and Batches.

Visually inspect the samples prior to starting the extraction process, as described in Section 10.1. Typically the entire content of the 1L amber jar is used for extraction. After the sample or sample aliquot is measured, the samples or empty sample containers are scanned to "empty" or returned to the Refrigerator.

Holding time for liquid samples are 7 days, with the following exceptions:

- PCB samples expire after 365 days, except for CT and NJ samples (7 days).
- EPH and NJEPH samples expire after 14 days.

7. Equipment and Supplies

7.1 Separatory Funnel: 2 or 3-Liter, glass or Teflon, with polytetrafluoroethylene (PTFE) stopcock and cap. 3L separatory funnels are to be used for extractions requiring 2L of sample.

7.2 Erlenmeyer Flasks: 250 and 500 mL.

7.3 Centrifuge Tubes.

7.4 Syringes: 1mL, 250µL, 100µL

7.5 Sodium Sulfate Stainless Steel filtering funnels. Add a plug of glass wool to the base of the 104mm glass funnel. Add approximately 60grams of baked sodium sulfate.

7.6 Glass wool.

7.7 Kuderna-Danish (KD) Apparatus: Assemble by attaching the Concentrator Tube to the Evaporation Flask using the Plastic clip. Add the Macro column to the Evaporation Flask. The Micro Snyder Column is attached directly to the Concentrator Tube using the Plastic Clip.

7.7.1 Evaporation Flask: 500mL KD flask.

7.7.2 Concentrator Tube: 25mL, graduated.

7.7.3 3-Ball Macro Snyder Column.

7.7.4 Micro Snyder Column.

7.7.5 Plastic Kek clips.

7.8 Boiling Chips: Solvent extracted, approximately 10/40 mesh (silicon carbide, or equivalent).

7.9 Graduated Cylinders: 25, 50, 100, 250, 500, and 1000 mL, class "A".

7.10 S-EVAP Water Bath with Solvent Collection Capability: Heated. Capable of temperature control (0.1C). Baths are located in a hood. Baths are equipped with chilled water condensers for solvent collection.

7.11 Buchi Concentration System: Base Unit, Chiller, Pump, Block, Controller and 180mL Glass Vessels.

7.12 N-EVAP: Organomation; utilized for micro blow down.

7.13 pH Paper: Multibanded, wide range.

7.14 Filter Paper: Whatman #4 185mm

7.15 Screw-top vials: 22mL volume.

7.16 Automatic Separatory Funnel Shaker

7.17 KI Paper Strips: 0.05mg/L residual chlorine sensitivity.

7.18 Multi-Position Stirring Plates.

7.19 Magnetic Stirring Bars.

7.20 Brady Labelling System: Thermal label generator.

8. Reagents and Standards

Pesticide or reagent grade chemicals are used in all tests. All reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.1 Reagent Water: All references to water in this method refer to reagent water from Alpha's DI water treatment system.

8.2 Sodium hydroxide solution (10N), NaOH: Dissolve 400g NaOH in reagent water and dilute to 1000mL. For basification of samples. Reagent expires one year after preparation.

8.3 Sulfuric acid solution (1:1 v/v), H_2SO_4: Prepare by slowly adding 500mL of concentrated H_2SO_4 to 500mL of reagent water, in a 1-Liter beaker placed in an ice water bath. For acidification of all non-EPH samples. Expires 1 year from date of preparation.

8.4 Hydrochloric Acid, 6N, 2:1: Place a 2000mL Beaker or equivalent in an ice water bath. Add 50mL of DI water to the beaker. Slowly add 100mL of Concentrated HCL. Mix with a stirring rod and allow to cool in a hood. Used for the acidification of EPH samples. Expires 1 year from date of preparation.

8.5 Sodium Sulfate (Na_2SO_4): Granular anhydrous; purified by baking at 400ºC for 4 hours in a shallow tray. Store in closed glass containers. All references to sodium sulfate in this method refer to this prepared reagent.

8.6 Methylene Chloride: Pesticide quality or equivalent. No expiration date listed.

8.7 Hexane: Pesticide quality or equivalent. No expiration date listed.

8.8 Acetone: Pesticide quality or equivalent. No expiration date listed.

8.9 1:1 Acetone/Methylene Chloride: Using a Graduated Cylinder measure 2 Liters of Acetone and transfer into a 4-Liter glass bottle. Using a graduated cylinder, add 2 Liters of Methylene Chloride into the same 4-Liter bottle. Mix.

8.10 Spiking Solutions: The various surrogate and LCS/MS spiking solutions used in the extraction steps are listed in the Separatory Funnel Extraction Guide WI# 19781. The preparation and expiration dates of these solutions are described in the analytical SOPs.

8.11 Sodium Thiosulfate Crystals ($Na_2S_2O_3$): J.T. Baker; 5-Hydrate crystal.

8.12 Silica Gel: VWR, Cat# TX4694MAAA. 60 - 200 mesh, chromatography grade. Activated by baking at 140 °C for a minimum of 14 hours in a shallow tray. The silica gel is stored in the oven or desiccator until ready for use. All references to silica gel in this method refer to this prepared reagent.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Each extraction batch contains various QC samples used to ensure the validity of the sample results. The particular QC elements performed for a given extraction batch are determined by the requirements of the determinative method. The purpose and definition of the QC samples performed are listed below. The specific QC requirements of the analytical methods are listed in WI/19781 Separatory Funnel Extraction Guide.

9.1 Blank

Blanks, or method blanks, are measured aliquots of reagent water (for aqueous extractions) that are treated identically to the associated samples. Surrogates are added, and the blanks are carried through all stages of the sample extraction, concentration, and cleanup procedures. Blanks serve to ensure that no systematic contamination exists. A blank is extracted with each batch of 20 or less samples.

For 608 and 625 a blank is extracted with each batch of 10 or less samples.

For 608.3 and 625.1 a blank is extracted with each batch of 20 or less samples.

9.2 Laboratory Control Sample (LCS/LCSD)

LCS samples are measured aliquots of reagent water (for aqueous extractions) that are spiked with a solution containing known amounts of target compounds, in addition to the surrogate solution. The LCS is carried through all stages of the sample extraction, concentration, and cleanup procedures. LCS samples serve as batch specific quantitative checks of the extraction. An LCS is extracted with each batch of 20 or less samples.

For samples to be analyzed by EPA 608 and 625, a LCS is extracted with each batch of 10 or less samples.

For samples to be analyzed by EPA 608.3 and 625.1, a LCS is extracted with each batch of 20 or less samples.

An LCSD is performed in addition to an LCS for most methods, as well as in lieu of the MS/MSD or Duplicate when there is insufficient sample volume available. The required solutions and volumes are listed in the Separatory Funnel Extraction Guide WI# 19781.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike

MS and MSDs are field samples spiked with a known quantity of the target analyte(s). They are prepared by taking additional sample aliquots, and adding the appropriate amounts of surrogate and spiking solutions. The MS/MSD are carried through all stages of the sample extraction, concentration, and cleanup procedures. MS samples serve as a measure of extraction accuracy, by allowing the comparison of the found amount(s) of target analyte(s) with the spiked amount(s). An MS/MSD set also allows for the calculation of the extraction precision, by comparing the results of the two samples.

For samples to be analyzed by EPA 608 and 625, a MS is extracted with batch of 10 samples or less.

For samples to be analyzed by EPA 608.3 and 625.1, a MS and MSD is extracted with batch of 20 samples or less.

For samples with a state of origin of New Jersey, a MS and MSD/DUP must be extracted for every twenty samples within a 24hr period.

9.6 Laboratory Duplicate

Duplicates are laboratory selected replicate samples, prepared by taking an additional sample aliquot of a sample. The duplicate is carried through all stages of the sample extraction, concentration, and cleanup procedures. Duplicates serve as a measure of the extraction precision, by comparing the results of the sample and duplicate.

For samples to be analyzed by EPA 608 and 625, a DUP is extracted with batch of 10 samples or less.

For samples with a state of origin of New Jersey, a MS and MSD/DUP must be extracted for every twenty samples within a 24hr period.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

Surrogates are compounds specified by the analytical method that are added to all samples and QC samples prior to beginning the extraction process. Surrogate recoveries are calculated and serve as a sample specific quantitative check of the extraction. The various spiking solutions are prepared according to the directions found in the analytical SOPs. The required solutions and volumes used are listed in WI/19781 Separatory Funnel Extraction Guide.

9.8 Method Sequence

See Section 10.

10. Procedure

All glassware and Separatory Funnels must be cleaned following the procedure described in the glassware washing SOP/1953. In addition, the glassware must be rinsed with acetone and methylene chloride, or baked until reaching a temperature of 400C just prior to use. The Separatory Funnels are rinsed with a 1:1 mixture of Acetone/Methylene Chloride.

All water extractions follow the LEAN "one-piece flow". All extraction information is recorded by the chemist performing the work in the ELN (Electronic Lab Notebook) see WI/2517. In addition to recording the extraction, concentration, clean-up and vialing information, the analyst must note any anomalies during the extraction procedure in the comments section of the ELN. Generating Work Groups, Batches and Labeling is described in Work Instruction WI/2421.

10.1 Sample Extraction

10.1.1 Carefully examine the sample prior to beginning the extraction process. The sample should be a single phase, with minimal or no sediment or solid material present. If this is not the case, contact the Extraction Lab Supervisor or Manager to determine how to handle the sample. The supervisor may need to contact login or the project manager to determine how the client would like the sample handled.

10.1.2 Common volumes for extraction include 140mL, 275mL, 500mL, 1000mL, 1200mL or 2000mL of sample to meet reporting limits. If 1200mL is required, this will be specified by the client. The containers that samples are received in will determine the extraction volume and procedure (I.e. standard procedure versus low volume initiative).

10.1.3 If the sample volume is less than the top of the sample collection bottle, do not use another container to bring it to 1L. Mark the current volume level on the sample bottle using a permanent marker for later volume determination.

10.1.4 Field and QC samples being analyzed for either EPA 608, EPA 625, EPA 625.1 or EPA 608.3 must be checked for residual chlorine prior to any pH adjustments or extraction. Invert the sample several times to ensure that sample is well mixed, then dip one KI test strip into sample for 10 seconds with a gentle constant back and forth motion. Wait 30 seconds and then refer to the chart on the KI strip container, if chlorine is detected at a

level less than 0.1mg/L proceed with the extraction. Otherwise, record in the ELN that the TRC is positive (+). Then add sodium thiosulfate to the sample, mix and re-check, repeat until the chlorine level is less than 0.1mg/L. Record in the ELN.

10.1.5 For One-piece flow sample processing and labeling, see Labeling and Generating Work Groups and Batches Work Instruction WI/2421 and Separatory Funnel Extraction Guide WI/19781.

10.1.6 Using an appropriate size Class "A" Graduated Cylinder, measure the correct amount of reagent water for blanks and LCSs. For QC requirements, see WI/19781 Separatory Funnel Extraction Guide. For samples, use the entire sample bottle (See10.1.2). Unless high analyte concentrations are anticipated, a smaller sample volume may be taken and diluted to the appropriate volume with reagent water. On occasion, the client may provide limted sample volumes, note this in the ELN. If the sample volume is higher than 3/4 of the container, then mark the water level on the amber and use the entire contents. If the sample volume is less than 3/4 of the container mark the water level on the amber and add reagent water to reach a final volume appropriate for the container (e.g. 1000mL, 500mL, 275mL, 140mL, etc.). Notate in the ELN the sample volume extracted and addition of DI water.

10.1.6.1 See WI# 19781 Separatory Funnel Extraction Guide for specific extraction volumes and spiking requirements. Extraction volume may affect the extract final volume (see WI#18528 S-Evap/N-Evap Concentration Standard Process or WI# 19781 Separatory Funnel Extraction Guide).

10.1.6.2 The for TCLP samples, TCLP fluid is used instead of DI water for the QC. For SPLP samples, QC will require the use of SPLP fluid.

10.1.7 Transfer the sample from the sample container or graduated cylinder into a labeled 2L separatory funnel for all extraction methods requiring less than 2L of sample volume. For extraction methods that require 2L of sample volume use a 3L glass separatory funnel.

10.1.7.1 A colored dot should be placed on the separatory funnel to match the ring stand above the collection glassware for each sample. This is used as a tool to prevent samples being drained into the incorrect collection vessel.

10.1.8 Adjust the pH, if necessary, to the pH indicated in Table 1 or WI# 19781 Separatory Funnel Extraction Guide (including QC samples), using the base and acid solutions listed in Sections 8.2-8.4. (**NOTE:** Do not alter the pH for neutral extractions.) The amount of acid or base required to achieve the desired pH is highly sample dependant, but is typically 1-10 mL.

10.1.9 Using the appropriately sized syringe, add the appropriate volume(s) of the surrogate/spiking solutions to the sample/QC as required; see WI/19781 Separatory Funnel Extraction Guide.

10.1.10 If there is no significant sediment or non-aqueous material present in the sample container, use methylene chloride to rinse the sample cylinder (or bottle) and transfer this rinseate into the separatory funnel.If the sample container does contain sediment or solid material that is not considered part of the sample, add methylene chloride directly into the separatory funnel. A comment must be placed in the ELN stating that the bottle was not rinsed due to the presence of sediment in the container. **NOTE:** Depending on the volume of the container that the sample is received in, it will determine the solvent volume used to rinse the container/extract the sample. See WI# 19781 Separatory Funnel Extraction Guide.

10.1.11 If the sample was transferred directly from the sample bottle, refill the bottle with water to the mark made in Section 10.1.3 and measure the volume using a graduated cylinder. Record the volume of sample that was in the bottle into the ELN.

10.1.12 Tightly cap the separatory funnel and vent the separatory funnel into a hood. Place the funnel into the holder on the Automatic Shaker, cap upward. Secure the locks. Repeat steps 10.1.6-10.1.10 for the remaining samples to be extracted. **NOTE:** If extracting using the 3L glass separatory funnel, the chemist must shake the vessel by hand.

10.1.13 Turn the unit on. Press 'Select' three times to open the timing menu. Press the 'up arrow' to set the number of minutes to be equal to '2'.

NOTE: For EPH extractions set the number of minutes to be equal to '3'.

10.1.14 Press "Start". Press the 'up arrow' to set the rpm to be equal to '150'.

10.1.15 When the Automatic Shaker stops, allow the organic layer to separate from the water phase for a minimum of 10 minutes. Remove the first separatory funnel and align in the hood over the appropriate collection vessel (see WI/2421). If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, addition of sodium chloride, or other physical methods.

10.1.16 **Before decanting the solvent layer**, check the pH of the sample with wide-range pH paper and record the value in the ELN. If the pH of the sample has not been satisfied as required by the determinative method, re-adjust the pH as indicated in Table1 or WI# 19781 Separatory Funnel Extraction Guide, using the base and acid solution. Repeat sections 10.1.10-10.1.13 until the appropriate pH is reached. Do not drain the solvent layer until the appropriate pH is reached.

10.1.17 Filter the extract through a funnel packed with glass wool and one 1/4cup scoop of sodium sulfate, collecting the filtrate in a the appropriate concentration glassware with the appropriate sample label. See WI 10995, Solvent rinsing/filtering guide. Rinse the sulfate funnel with 25mL of DCM. This is done for the first extraction drain only. Add the same amount (60 or 30mL) of solvent to the separatory funnel (unless it is a 625 or 608. See 10.1.17.3) and place the sample back on the shaker table. Repeat steps 10.1.14 through 10.1.16 for each sample on the shaker table.

10.1.17.1 Note: The chemist should drain the solvent until approximately 1mL of solvent remains in the separatory funnel in order to prevent water from reaching the sodium sulfate funnel.

10.1.17.2 Note: Due to contamination issues, pesticide samples should be filtered through a funnel containing a paper filter with one 1/4cup scoop of sodium sulfate, rather than glasswool.

10.1.17.3 For 625 and 608 samples, DCM must be added to the sample container before it is transferred to the separatory funnel before every shake, not just the first shake.

10.1.18 For all analyses repeat the extractions two more times for two minutes each, using fresh portions of solvent (Section 10.1.10-10.1.16). Collect all three solvent extract portions in the same labeled collection vessel (KD apparatus, Erlenmeyer flask. or Buchi vessel).

10.1.18.1 For EPH and EPH-TPH-10 analyses, repeat the extractions two more times for three minutes each, using fresh portions of solvent (Section10.1.9-10.1.15).

10.1.19 If additional pH adjustment during extraction is required (ex. ABN), adjust the pH of the aqueous phase to the desired pH indicated in Table 1 or WI# 19781 Separatory Funnel Extraction Guide. Repeat the extraction process Section 10.1.9-10.1.16.

10.1.19.1 For ETPH and EPH-TPH-10 analyses, the sample extract is filtered through a stainless steel funnel containing glass wool and approximately 60g of sodium sulfate into a 250mL Erlenmeyer flask. Add 3 grams of activated Silica Gel and a stir bar to the extract. Place the sample on a stirring plate and stir for 5 minutes at 650 RPM. Filter the extract through a stainless steel funnel containing filter paper, collecting the filtrate in a Buchi vessel for concentration (See Section 10.2.3).

10.1.19.2 Proceed to sample concentration. Note all DRO, ETPH, and EPH-TPH-10 products are concentrated using the Bucchi Concentration System.

10.2 Sample Concentration Techniques

10.2.1 KD Technique: For samples requiring hexane exchange (See Table 1).

10.2.1.1 S-Evap baths are heated to 75°C and 95°C. Attach a three-ball Snyder column to the top of the flask. Place the KD apparatus on the hot water bath heated to approximately 75°C so that the concentrator tube is partially immersed in the hot water, and so that the entire lower rounded surface of the flask is bathed in hot water vapor. Attach the chilled water condenser to the top of the Synder Column. Adjust the position of the apparatus as required to complete the concentration. At the proper rate of distillation, the balls in the column will actively chatter, but the chambers will not flood with solvent.

10.2.1.2 When the sample volume reaches 10-15mL, remove the condenser from the Synder Column and add 25mL of hexane using a graduated cylinder. Add the hexane to the top of the Snyder Column. Allow the sample to continue to concentrate until the volume reaches 10-15mL or boiling reduces in intensity. Add an additional 25mL of hexane and allow the sample to become fully exchanged with hexane (boiling reduces in intensity and the Snyder columns stop chattering). Remove the KD concentration setup and move to the 95C bath. Re-attach the condenser and continue with the concentration until the extract volume is reduced to 10-15mL.

10.2.1.3 Remove the KD apparatus from the water bath. Wipe the flask and its lower ground glass joint with a dry paper towel to remove any moisture from the outside of the glassware. Allow to cool before disassembling the KD apparatus. Move the sample label from the KD Flask to the concentrator tube (See WI/2421).

10.2.1.4 Place the Concentrator tube on the N-EVAP. The N-EVAP is set at 65 °C for samples extracted in Hexane. Adjust the flow of nitrogen so that the surface of the solvent is just visibly disturbed. Samples remain on N-EVAP until they are reduced to the appropriate final volume (see S-Evap/N-Evap Concentration Standard Process, WI#18528).

10.2.1.5 The extract is now ready for sample cleanup or vialing (See Table 1). Refer to the relevant Clean-up SOP or proceed with extract vialing (See WI/3827, Extract Vialing Procedure, WI/2426, GC Extract Vialing Procedure and WI/2423, GC/MS Extract Vialing Procedure).

10.2.2 Alternative Concentration Technique: Buchi

The Buchi is a self-contained sample concentration and solvent recovery system that utilizes vacuum, heat and oscillation to concentrate samples. The Buchi will recover >95% of solvent emissions.

10.2.2.1 Setup: All DRO, ETPH, EPH-TPH-10, and 8270/SIM products require Buchi concentration. The samples are filtered into the Buchi concentration vessels. The samples are equalized with the extraction solvent. Soil samples will be adjusted to 80mL, while liquid samples will have a 180mL volume. This will allow for even concentration.

10.2.2.2 Empty or drain the solvent collection flask into the appropriate hazardous waste container.

10.2.2.3 Turn "on" the Base Unit, Chiller (set at 5˚C), Pump, Block and Controller. Push "start" button on the chiller to begin circulation and chilling of fluid. Push the "start" button on the block once and turn "off" the oscillation. This will begin cooling the condenser. Pre-heat the block to 65C as noted on the panel readout.

10.2.2.4 On the Controller, press program #1(Gradient and DCM program should be selected). This program will show the following, DCM 50min., 600MBAR for 1 minute, 540MBAR for 17min., and 460MBAR for 32 min. Total run time will be 50 minutes.

10.2.2.5 Add 5mL of water into each block cell position. Place the labeled samples (glass vessel) into the block. Note, all positions on the block must have a vessel in order to form a vacuum. Use empty vessels if necessary to fill the 12-position block.

10.2.2.6 Place the block lid on top of the sample extracts and aligning the set screws to the holes in the lid. Tighten the two knobs on to the block, hand tight. Tighten the knobs evenly or the lid will not form a vacuum.

10.2.2.7 Push "Start" on the controller. The controller should immediately drop and read 600MBAR. If the vacuum does not drop, there is a leak with the vessels. Reposition and retry.

10.2.2.8 Check the sample solvent level after 50min., by shutting off the controller and removing the lid. Remove samples that have a final volume 1.0mL. Place an empty vessel in its place and change the program to "Touchup". If necessary, restart the program and check the solvent volume every 5minutes. Note, by looking at the solvent condensing rate on the condenser, you can gauge the solvent remaining in the vessel.

10.2.2.9 Once all samples have been concentrated to 1.0mL, the extract is ready to be vialed. Refer to the relevant Clean-up SOP or proceed with extract vialing (See WI 3827 Extract Vialing Procedure, WI 2426 GC Extract Vialing Procedure and WI 2423 GC/MS Extract Vialing Procedure).

10.2.2.10 Samples requiring a final volume of less than 1mL should be transferred to a concentrator tube and placed on the N-Evap until the required volume is reached.

10.2.2.11 Samples that will not concentrate to the required final volume should be transferred to a concentrator tube and placed on the N-Evap in order to try to reduce the volume further. Samples that will not continue to concentrate should have the final volume documented in the ELN and proceed to the relevant clean-up or extract vialing procedure.

10.3 Preventive Maintenance

10.3.1 Buchi Concentrators

10.3.1.1 **Refrigeration Re-circulator:** The Refrigeration Re-circulator should be checked once every shift (when in use) to insure it is at the specified temperature, running correctly and that the level of coolant is constant with manufactures recommendation.

10.3.1.2 **Buchi System**: Contact factory representative with inquiries and maintenance issues.

10.3.1.2.1 **Buchi Vacuum Cover**: The Buchi unit vacuum cover requires cleaning in a fume hood using acetone.

10.3.2 Water Bath

10.3.2.1 The water bath should be kept full at all times. Add reagent water as necessary.

10.3.2.2 Keep unit clean. Avoid solvent spills on or around unit. Clean periodically with a damp cloth.

11. Data Evaluation, Calculations and Reporting

Not Applicable.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence, improper preservation and observed sample headspace are noted on the nonconformance report form.

When analysis of samples indicates possible extraction problems, such as poor surrogate recoveries, poor LCS/MS/MSD recoveries, or suspected contamination in blanks or samples, re-extractions are required. Depending on the particular failure, the re-extraction may be of a specific sample or the entire extraction batch.

The analyst that determines the need for re-extraction must fill out a sample re-extract request form. This form notes the reason for the re-extraction request along with any special requirements, and the date and time that the re-extract is needed. Re-extraction request forms are maintained on file to help track the cause for re-extractions, and to be used as a tool in improving systems to minimize the need for re-extractions.

Depending on the results of the re-extraction, the first, second, or both sets of results may be reported to the client, along with a narrative report detailing the problems encountered and the resolution.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

SOP/2121 Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739 DOC Generation

SOP/1728 Waste Management and Disposal SOP

SOP/1953 Organic Extraction Glassware Cleaning and Handling

WI/2421 Labeling and Generating Work Groups and Batches

WI/2517 LIMS Electronic Laboratory Notebook Procedure

WI/3827 Extract Vialing Procedure

WI/10995 Solvent Rinsing/Filtering

WI/19781 Separatory Funnel Extraction Guide

16. Attachments

Table 1 – Specific Extraction Conditions for Various Determinative Methods

Table 1

Specific Extraction Conditions for Various Determinative Methods

Determinative Method	Initial Extraction pH	Secondary Extraction pH	Extraction Solvent	Concentration method	Final Volume	Appropriate Cleanup Technique
Pest/8081	5 – 9	None	DCM	S-Evap/N-Evap	1, 5,10 mL	Florisil *b*c
PCB/8082	5 – 9	None	DCM	S-Evap/N-Evap	1, 5 mL	Sulfuric acid/Copper
608	5 – 9	None	DCM	S-Evap/N-Evap	1, 5, 10mL	Florisil *b
608.3	5 – 9	None	DCM	S-Evap/N-Evap	1, 5, 10mL	Florisil *b
8270	< 2	> 11	DCM	Buchi	1 mL	n/a
625	< 2	> 11	DCM	Buchi	1 mL	n/a
625.1	< 2	=11-13	DCM	Buchi	1 mL	n/a
TPH-DRO	As received	None	DCM	Buchi	1 mL	n/a
NJ TPH	As received	None	DCM	S-Evap/N-Evap	1 mL	n/a
MA-EPH	< 2	None	DCM	S-Evap/N-Evap	1 mL	Silica Gel Fractionation
NJ-EPH	<2	None	DCM	S-Evap/N-Evap	1mL	Silica Gel Fractionation
EPH-TPH-10	<2	None	DCM	Buchi	1mL	Silica Gel
CT-ETPH	As received	None	DCM	Buchi	1 mL	Silica Gel
Herbicide	See relevant SOP					

- 8270, 625, and 625.1 methods are for extraction of both acidic and base/neutral compounds.
- *b - Sample extracts may require additional cleanup by Method 3660A to remove elemental sulfur, as determined by the sample extract appearance once florisil cleanup has been performed.
- *c – Liquid matrix samples do not require florisil cleanup unless requested by the client or required for analysis (exception: Pest-608).
- For the full list of products in each category, see Separatory Extraction Guide WI#19781.

For additional products, QC's, solvent, reagents, surrogate/spike solutions, and extraction volumes see Qualtrax ID 19781 "Separatory Extraction Guide."

Inductively Coupled Plasma - Atomic Emission Spectrometry

Reference Method No.: **Method 6010D** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update V, July, 2014.

SM 2340B, Hardness by Calculation, Standard Methods for the Examination of Water and Wastewater, APHA-AWWA-WPCF, 21st Edition, 1997.

1. Scope and Application

Matrices: Digestates from all matrices.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals, in solution. The method is applicable to all of the elements listed in Table 1. All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludge, sediments, and other solid wastes, require digestion prior to analysis. Groundwater samples that have been prefiltered and acidified will not need acid digestion unless chemical interferences are suspected. Samples which are not digested are matrix matched with the standards. Refer to Metals Preparation SOPs for the appropriate digestion procedures.

Table 1 lists the elements for which this method is applicable. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Table 1 lists the recommended analytical wavelengths for the elements in clean aqueous matrices. Table 3 lists the Reported Detection Limits. The reported detection limit data may be used to estimate instrument and method performance for other sample matrices. Elements other than those listed in Table 1 may be analyzed by this method if performance at the concentration levels of interest (see Section 9) is demonstrated.

Users of the method should state the data quality objectives prior to analysis and must document and have on file the required initial demonstration performance data described in the following sections prior to using the method for analysis.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is made by one of the following laboratory personnel before performing the modification: Area Supervisor, Metals Manager, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences described in this method. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

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2. Summary of Method

Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

This method describes multielemental determinations by ICP-AES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. In one mode of analysis the position used must be as free as possible from spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in Section 4.0 must also be recognized and appropriate corrections made; tests for their presence are described in Section 9.7. Alternatively, users may choose multivariate calibration methods. In this case, point selections for background correction are superfluous since whole spectral regions are processed.

This SOP includes the manual calculations for Total Hardness and Calcium Hardness, according to SM 2340B.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Refer to Table 3 for method Reporting Limits.

4. Interferences

4.1 Spectral

Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

4.1.1 Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for

routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans must be included in the correction algorithm. Off-line spectral interferences are handled by including spectra on interfering species in the algorithm.

4.1.2 To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.

4.1.3 Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

4.1.4 When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that each instrument may exhibit somewhat different levels of interference. The interference effects must be evaluated for each individual instrument since the intensities will vary.

Major known interferences are Fe, Al, Ca, Mg, V, Ni, Cu, and Cr. To minimize any of these interferences, every analyte is analyzed on each instrument at or near its linear range and corrected for these interferences. This is done on an annual basis, and data is kept on file.

4.1.5 Inter-element corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear must be avoided when practical. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users must not forget that some samples may contain uncommon elements that could contribute spectral interferences.

4.1.6 The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.

4.1.7 The primary wavelength for each analyte is based upon the instrument manufacturer's recommendations. An alternate wavelength is chosen if there is an indication of elevated background or overlap of another spectral wavelength. The wavelength for each analyte must be as free from interferences as possible.

4.1.8 If the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution must fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change must be determined and corrected and the correction factor updated. The interference check solutions must be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

4.1.9 When inter-element corrections are applied, their accuracy must be verified, daily, by analyzing the spectral interference check solution. The correction factor or multivariate correction matrices tested on a daily basis. All inter-element spectral correction factors or multivariate correction matrices are verified and updated when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs. The standard solution must be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

4.1.10 When inter-element corrections are <u>not</u> used, verification of absence of interferences is required.

4.1.10.1 One method is to use a computer software routine for comparing the determinative data to limits, files for notifying the analyst when an interfering element is detected in the sample at a concentration that will produce either an apparent false positive concentration, (i.e., greater than) the analyte

instrument detection limit, or false negative analyte concentration, (i.e., less than the lower control limit of the calibration blank defined for a 99% confidence interval).

4.1.10.2 Another method is to analyze an Interference Check Solution(s) which contains similar concentrations of the major components of the samples (>10 mg/L) on a continuing basis to verify the absence of effects at the wavelengths selected. These data must be kept on file with the sample analysis data. If the check solution confirms an operative interference that is >20% of the analyte concentration, the analyte must be determined using (1) analytical and background correction wavelengths (or spectral regions) free of the interference, (2) by an alternative wavelength, or (3) by another documented test procedure.

4.2 Physical

Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, using a peristaltic pump, use of an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also, it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers. The test described in Section 10.3.4.1 will help determine if a physical interference is present.

4.3 Chemical

Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Additionally, if filtered samples are found to have an organic or sulfur like odor they are processed by heating after the addition of the acids to matrix match. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.4 Memory

Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences must be recognized within an analytical run and suitable rinse times must be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample must be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit must be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed after a rinse

period of sufficient length. Alternate rinse times may be established by the analyst based upon their DQOs.

4.5 Other Interferences

4.5.1 Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations must be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in plastic bottles.

6.2 Sample Preservation

If samples are for soluble metals analysis, filtration must take place prior to preservation with 1:1 HNO3 to a pH < 2. Soluble samples must be held at pH < 2 for at least 24 hours prior to digestion if not preserved at the time of filtration. Samples for total metals analysis are preserved with 1:1 HNO3 to a pH < 2. Samples must be pH <2 for at least 24 hours prior to digestion if not preserved at the time of collection.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples to be analyzed for soluble metals, that have not been filtered, must be filtered and preserved within 24 hours of sample collection.

Preserved samples have a hold time of 6 months, and are stored at ambient temperature.

7. Equipment and Supplies

7.1 Inductively coupled argon plasma emission spectrometer:

- **Thermo Scientific ICAP Duo 6500 (Trace7)**

7.1.1 Computer-controlled emission spectrometer with background correction.

7.1.2 Radio-frequency generator compliant with FCC regulations.

7.1.3 Optional mass flow controller for argon nebulizer gas supply.

7.1.4 Optional peristaltic pump.

7.1.5 Optional Autosampler.

7.1.6 Argon gas supply - high purity.

7.2 Volumetric flasks of suitable precision and accuracy.

7.3 Volumetric pipets of suitable precision and accuracy.

8. Standards and Reagents

Reagent semiconductor and/or trace grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration of the contamination is less than the MDL then the reagent is acceptable.

8.1 Hydrochloric acid (conc), HCl. Stored at room temperature in acid resistant cabinet. Expiration date if defined by vendor.

8.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL DI water and dilute to 1 liter in an appropriately sized beaker. Stored at room temperature in polypropylene bottle, expiration date if defined by vendor..

8.3 Nitric acid (conc), HNO_3. Stored at room temperature in acid resistant cabinet. Expiration date if defined by vendor.

8.4 Nitric acid (1:1), HNO_3. Add 500 mL concentrated HNO_3 to 400 mL DI water and dilute to 1 liter in an appropriately sized beaker. Stored at room temperature in polypropylene bottle, expiration date if defined by vendor..

8.5 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be interference free. Refer to Chapter One for a definition of reagent water.

8.6 Standard stock solutions may be purchased or prepared from ultra- high purity grade chemicals or metals (99.99% pure or greater). All stock standards are ordered through ISO and American Association for Lab Accreditation vendors. All standards are in aqueous solutions and are generally at concentrations of 1000ppm and 10,000ppm.

8.7 Mixed calibration standard solutions

Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks. Add the appropriate types and volumes of acids so that the standards are matrix matched with the sample digestates. Care must be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. Transfer the mixed standard solutions to HDPE or polypropylene bottles for storage. Fresh mixed standards must be prepared, as needed, with the realization that concentration can change on aging as evidenced by failures in the ICV.

NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of water and warm the flask until the solution clears. Cool and dilute to 100 mL with water. For this acid combination, the silver concentration must be limited to 2 mg/L. Silver under these conditions is stable in a tap-water matrix for 30 days. Higher concentrations of silver require additional HCl.
Additionally, sulfur standards are stand-alone single element standards and therefore are not to be combined in a mixed calibration standard solution.

8.8 Blanks

Three types of blanks are required for the analysis for samples. The calibration blank is used in establishing the analytical curve, and the method blank is used to identify possible contamination resulting from varying amounts of the acids used in the sample processing. The rinse blank is used to flush the system between all samples and standards.

8.8.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards. Prepare a sufficient quantity to flush the system between standards and samples. The calibration blank will also be used for all initial (ICB) and continuing calibration blank (CCB) determinations (see Sections 10.2 and 10.4). Refer to Section 10.4.1.2 for acceptance criteria and/or corrective actions.

8.8.2 The method blank must contain all of the reagents in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis. Refer to Section 9.1 for acceptance criteria and/or corrective actions.

8.8.3 The rinse blank consists of HNO_3 (1% or 2%) (v/v) in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

8.9 The Initial Calibration Verification Standard (ICV) and the Continuing Calibration Verification Standard (CCV)

These ICV is prepared by the analyst by combining compatible elements from a standard source different than that of the calibration standard at a concentration at or near the mid-point of the calibration curve. The CCV is prepared from the same source as the calibration standards and must be at a concentration near the mid-point of the calibration curve.

8.10 Low Level of Quantification, (LLOQ)

The LLOQ is initially verified by the analysis of at least 7 replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method at or below the lowest calibration point. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be +/- 35% of the true value and RSD should be < 20%. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias.

Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

8.11 Spectral Interference Check Solution

These solutions are prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Analysts are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests.

Single element interference checks - At a minimum, single element SIC checks must be performed for the following elements: Aluminum 500mg/L; Boron 50mg/L, Barium, 50mg/L, Calcium 500mg/L; Copper 50mg/L; Iron 200mg/L; Magnesium 500mg/L; Manganese 50mg/L; Molybdenum 20mg/L; Sodium 1000mg/L; Nickel 20mg/L; Selenium 20mg/L; Silicon 200mg/L; Tin 20mg/L; Vanadium 20mg/L; Zinc 20mg/L The absolute value of the concentration observed for any unspiked analyte in the single element SIC checks must be less than two times the analytes' LLOQ.

The concentration of the SIC checks are suggested, but become the highest concentration allowed in a sample analysis, and cannot be higher than the highest established linear range. Samples with concentrations of elements higher than the SIC check must be diluted until the concentration is less than the SIC check solution. Note that reanalysis of a diluted sample is required even if the high concentration element is not required to be reported for the specific sample, since the function of the SIC check is to evaluate spectral interferences on other elements. The single element SIC checks are performed when the instrument is setup and periodically (at least once every 6 months) thereafter.

Mixed element interference check - The mixed element SIC solution is analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than +/- the LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, or alternatively the LLOQ may be raised to twice the concentration observed in the SIC solution. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions These may be present up to the concentration documented plus the LLOQ.

Mixed element SIC solution: Aluminum, 500mg/L; Calcium, 500mg/L; Iron, 200mg/L; Magnesium, 500mg/L

8.12 Ongoing Low Level of Quantification, (LLOQ)

Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

8.13 Internal Standard

The internal standard consists of a multi-element solution; each internal standard covers a range of the spectrum (low, middle, or high wavelengths) and the elements within that range.

100 mg/L Ce

20 mg/L Cs

2.0 mg/L Lu

Note: The standard is used to monitor instrument fluctuations including but not limited to nebulization efficiency, plasma variations, environmental temperature changes, peristaltic pump pulsations, etc. Therefore, the solution used to start an analysis calibration cannot be added to or changed out during analysis without requiring subsequent full recalibration.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank is a volume of reagent water carried through the same preparation process as a sample.

The method blank results must be less than ½ of the LLOQ for all analytes of concern. If the results of the method blank exceed the RDL for any analyte, perform re-analysis of a new aliquot of the method blank.

If the results continue to exceed the RDL, proceed as follows:

If all of the samples for the analyte are non-detected, and the method blank is at or above the RDL, no action is required.

If one or more associated samples for that analyte have positive results at or above the RDL, those samples must be considered to be out of control, and are re-digested and reanalyzed.

9.2 Laboratory Control Sample (LCS)

Analyze one LCSW/SRM per sample batch. A LCS/SRM sample is a spiked volume of reagent water that is brought through the entire preparation and analytical process. The LCSW must have a % Recovery of ± 20% within the actual value or within vendor control limits (95% confidence limits) for the solid SRM.

If the LCSW or SRM % Recovery is outside the acceptable limits as stated in Table 2, or outside any vendor control limits, the LCS is rerun once. If upon reanalysis the LCS is still out

of control, the failed analytes are re-prepped and re-analyzed. Otherwise, a nonconformance report form is raised to document the exact problem and this form is then authorized by the QA/QC Director and/or the Laboratory Manager(s).

9.3 Initial Calibration Verification (ICV)

For all analytes and determinations, the laboratory must analyze an ICV (Section 8.9), and a calibration blank (ICB, Section 8.8.1), immediately following daily calibration. The results of the ICV are to agree within 10% of the expected value; if not, re-analyze once, if still failing terminate the analysis, correct the problem, and recalibrate the instrument.

9.4 Continuing Calibration Verification (CCV)

A calibration blank (CCB, Section 8.8.1) and a calibration verification standard (CCV, Section 8.9) must be analyzed after every tenth sample and at the end of the sample run. Analysis of the calibration verification (CCV) must verify that the instrument is within 10% of the calibration with the relative standard deviation < 5% from replicate (minimum of two) integrations.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard. If the standard passes the second time then the analysis may be continued. The batch sheet is noted. If the standard fails again, instrument maintenance must be performed and the CCV/CCB standard is reanalyzed. If the standard passes, then all samples run after the last passing CCV/CCB pair must be re-analyzed.

If the standard fails after instrument maintenance, the instrument is recalibrated. A new ICV/ICB is performed, and all previous data after the last passing CCV/CCB is reanalyzed.

9.5 Matrix Spike

Analyze matrix spike samples at a frequency of one per matrix batch. The matrix spike is the same solution as used for the LCS (Table 4). A matrix spike sample is a sample brought through the entire sample preparation and analytical process.

9.5.1 The percent recovery is to be calculated as follows:

$$\% \text{ Recovery} = \frac{MS - S}{C} \times 100$$

where:

MS = Matrix Spike value
S = Sample value.
C = Concentration of the Spiking solution.

9.5.2 If the Matrix Spike falls outside of the limits as stated in Table 2, or outside any historical documentation for analytes of interest a post analytical spike is performed for the failed analytes. The same sample from which the MS/MSD aliquots were prepared should be spiked with a post digestion spike at a minimum level of 10 times and a maximum of 100 times the lower limit of quantitation. The acceptable % Recovery of the post analytical spike is 80-120%. A nonconformance is noted in the LIMS and approved in secondary peer review and/or by the Metals Manager.

9.5.3 If the Post Spike fails the dilution test should be performed**.** If the analyte concentration is sufficiently high (minimally, a factor of 25 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution should agree within ± 20% of the original determination. If not, then a chemical or physical interference effect should be suspected.

9.6 Laboratory Duplicate

A duplicate sample is analyzed once per matrix batch. This sample is brought through the entire sample preparation and analytical process.

9.6.1 The relative percent difference between duplicate determinations is to be calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

RPD = relative percent difference.
D_1 = first sample value.
D_2 = second sample value (replicate).

9.6.2 If the Duplicate falls outside of the limits as stated in Table 2, or outside any historical documentation and the concentrations of the failing analytes are less than 5x the RL or a matrix interference is found a nonconformance is noted in the LIMS and approved in secondary peer review and/or by the Metals Manager.

9.7 Method-specific Quality Control Samples

9.7.1 Spectra Interference Check Standard

A mixed check solution is analyzed once daily (section 8.11). One solution (SIC) has only elevated concentrations of Fe, Al, Ca, Mg to ensure no interferences occur. The concentrations of the analytes of interest must have an absolute value of the LLOQ. This solution is analyzed at the beginning of the first analytical run of the day.

The high level interferences are not evaluated for recovery. If the SIC fails take corrective action which may include re-evaluation of the inter-element correction values (IECs) after running single element SIC. The instrument calibration routine must then be performed and confirmed by the ICV/ICB pair and the SIC re-analyzed before proceeding with analysis. Otherwise, the nonconformance issue is raised to the Department Supervisor and/or the QA Department.

9.7.2 Internal Standard

The internal standards are added prior to the nebulizer and corrects for intensity differences in the instrument response between the standard's and sample's matrix. They are monitored for any variation in response during the sample analyses and used to ratio the sample response to the internal standard response of the calibration blank. The ratio is applied to compensate for instrument conditions in the plasma or nebulization caused by the matrix. The internal standard is monitored for 50-150% recovery or laboratory generated control ranges difference from the calibration blank IS response to ensure the proper functioning of the internal standard introduction system and matrix interferences. If an injection falls outside of this acceptance range the sample or QC check is rerun once to check for an introduction error.

If a sample continues to fail it's to be run on successive increasing dilutions until the internal standards associated with the elements of interest are within range. If a

QC check fails on the single rerun the analysis is stopped, the root cause investigated, corrected and the instrument re-calibrated/verified. The analysis begins again with all samples that were run after the last acceptable CCV/CCB pair.

9.7.3 LDR Check Solution: A multiple element or single element solution run at a point above the highest calibration standard under the same calibration used to quantify the associated sample data. The LDR check must be within +/-10% of the true value of each element of interest to be considered valid.

9.8 Method Sequence

- Calibration of instrument
- Initial Calibration Verification Standard
- Initial Calibration Blank
- Mixed SIC Solution
- Continuing Calibration Verification Standard
- Continuing Calibration Blank
- 10 samples
- Continuing Calibration Verification Standard
- Continuing Calibration Blank
- 10 Samples
- Continuing Calibration Verification Standard
- Continuing Calibration Blank

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation

Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Groundwater samples which have been prefiltered and acidified will not need acid digestion. Samples which are not digested must be matrix matched with the standards.

10.1.2 Instrument Set-Up

Set up the instrument with proper operating parameters established as detailed below. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

Startup Procedures

For iCAP Duo 6500

- Turn on power to the chiller
- Click on ThermoSpec Icon; enter analyst initials in login screen
- Click on Plasma icon to start instrument
- Allow to warm up for 30 minutes
- Enter analytical workgroup number (obtained from LIMS) globally under the Instrument menu by selecting Tools, then Options, then Analyst.

- Click on the Sequence tab and enter the sequence by selecting New Autosampler Table, Add Sequence, Add # of spaces.
- Enter the sample locations and IDs
- Press Run Auto-Session button (►) in menu bar.

10.1.2.1 Specific wavelengths are listed in Table 1. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. The instrument and operating conditions utilized for determination must be capable of providing data of acceptable quality to the program and data user.

Operating conditions for axial plasma will vary from 1100 – 1500 watts forward power, 15-19 Liters/min argon coolant flow, 0.5 – 0.7 L/min argon nebulizer flow, 140 – 200 rpm pump rate and a default 1 minute preflush time and 10 second measurement time is recommended for all simultaneous instruments.

10.1.2.2 The plasma operating conditions need to be optimized prior to use of the instrument. This routine is not required on a daily basis, but only when first setting up a new instrument or following a change in operating conditions. The following procedure is recommended or follow manufacturer's recommendations. The purpose of plasma optimization is to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array. The use of a mass flow controller to regulate the nebulizer gas flow or source optimization software greatly facilitates the procedure.

10.1.2.2.1 The Thermo ICP's typically use a Meinhard Nebulizer. The nebulizer flow for each instrument is 1.0 +/- 0.2 mL/min.

10.1.2.2.2 The 6500 Duo instruments automatically perform a wavelength check at start up without user interaction.

10.1.2.2.3 The instrument operating condition finally selected as being optimum must provide the lowest reliable instrument detection limits and method detection limits.

10.1.2.2.4 If either the instrument operating conditions, such as incident power or nebulizer gas flow rate are changed, or a new torch injector tube with a different orifice internal diameter is installed, the plasma and argon pressures must be reoptimized.

10.1.2.2.5 After completing the initial optimization of operating conditions, but before analyzing samples, the laboratory must establish and initially verify an interelement spectral interference correction routine to be used during sample analysis. A general description concerning spectral interference and the analytical requirements for background correction in particular are discussed in the section on interferences. Criteria for determining an interelement spectral interference is an apparent positive or negative concentration for the analyte that falls within ± ½ LLOQ. The upper control limit is the analyte instrument detection limit. Once established, the entire routine is periodically verified every six months. In between that time, IEC's are done on a need be basis per analyte. Only a portion of the

correction routine must be verified more frequently or on a daily basis. Initial and periodic verification of the routine must be kept on file. Special cases where continual verification is required are described elsewhere.

10.1.2.3 Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

10.1.2.3.1 Method detection limits must be established for all wavelengths utilized for each type of matrix commonly analyzed. The matrix used for the MDL calculation must contain analytes of known concentrations within 3-5 times the anticipated detection limit.

10.1.2.3.2 Determination of limits using reagent water MDLs represent a best case situation and do not represent possible matrix effects of real world samples.

10.1.2.3.3 If additional confirmation is desired, reanalyze the seven replicate aliquots on two more non-consecutive days and again calculate the method detection limit values for each day. An average of the three values for each analyte may provide for a more appropriate estimate.

10.1.2.3.4 The upper limit of the linear dynamic range must be established for each wavelength utilized by determining the signal responses with 10% of the true value of each element from a concentration standard at the upper limit of the range run on the same calibration as required by the sample responses above the calibration range. The range which may be used for the analysis of samples must be no more than 90% of the resulting data. Determined analyte concentrations that are above the upper range limit must be diluted and reanalyzed. The analyst must also be aware that if an inter-element correction from an analyte above the linear range exists, a second analyte where the inter-element correction has been applied may be inaccurately reported.

NOTE: Many of the alkali and alkaline earth metals have non-linear response curves due to ionization and self-absorption effects. These curves may be used if the instrument allows; however the effective range must be checked and the second order curve fit must have a correlation coefficient of 0.995 or better. Third order fits are not acceptable. These curves are much more sensitive to changes in operating conditions than the linear lines and must be checked whenever there have been moderate equipment changes.

10.1.2.4 The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

10.2 Initial Calibration

Calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Section 8.7. Flush the system with the calibration blank (Section 8.8.1) between each standard or as the manufacturer

recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a calibration blank, RL standard and a high level standard. Calibration curve verification is accomplished through the analysis of the ICV, ICB and SIC standards.

10.3 Equipment Operation and Sample Processing

10.3.1 For all analytes and determinations, the laboratory must analyze an ICV (Section 8.9), and a calibration blank (ICB, Section 8.8.1), immediately following daily calibration.

A calibration blank (CCB, Section 8.8.1) and a calibration verification standard (CCV, Section 8.9) must be analyzed after every tenth sample and at the end of the sample run. Analysis of the calibration verification (CCV) must verify that the instrument is within 10% of the calibration with the relative standard deviation < 5% from replicate (minimum of three) integrations.

If the calibration cannot be verified within the specified limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV/ICB, or CCV/CCB must be reanalyzed. The analysis data for the calibration blank, check standard, and ICV or CCV must be kept on file with the sample analysis data.

10.3.2 Rinse the system with the rinse blank solution (Section 8.8.3) before the analysis of each sample. The suggested default rinse time is one minute. Each ICP instrument may establish a reduction in this rinse time through a suitable demonstration.

10.3.3 Dilute and reanalyze samples that exceed the linear range or use a calibrated alternate, less sensitive line for which quality control data is already established.

10.3.4 If less than acceptable accuracy and precision data are generated a series of tests are performed prior to reporting concentration data for analyte elements. At a minimum, these tests should be performed with each batch of samples prepared/analyzed with corresponding unacceptable data quality results. These tests, as outlined in Sections 10.3.5 and 10.3.6, will ensure that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

10.3.5 **Post Digestion Spike Addition:** If the sample concentrations are insufficient to perform a dilution test a post digestion spike added to a portion of a prepared sample, or its dilution for the elements failing the matrix spike recoveries must be run, recovery limits equal to 75% to 125% of the known spike value. If the spike is not recovered within the specified limits If the post-digestion recovery fails to meet the acceptance criteria, the sample results must be reported as estimated values

10.3.6 **Dilution Test:** If the analyte concentration is sufficiently high (minimally, a factor of 25 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution must agree within ± 20% of the original determination. Elements that fail the dilution test are reported as estimated values.

10.3.7 **CAUTION:** If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

10.4 Continuing Calibration

10.4.1 Check calibration with an ICV following the initial calibration (Section 8.9). Verify calibration with the Continuing Calibration Verification (CCV) Standard (Section 8.9) at the end of the initial calibration sequence (ICV, ICB), after every ten samples, and at the

end of an analytical run. Use a calibration blank (Section 8.8.1) immediately following daily calibration, after every 10 samples and at the end of the analytical run.

10.4.1.1 The results of the ICV are to agree within 10% of the expected value, and CCVs are to agree within 10% of the expected value; if not, terminate the analysis, correct the problem, and recalibrate the instrument. Each may be rerun once to confirm or cure the initial failure.

10.4.1.2 The results of the calibration blank should be below ½ of LLOQ or RL (whichever is lower). If not, repeat the analysis and if the failure is repeated terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within ten percent of the action limit, analyses need not be rerun and recalibration need not be performed before continuation of the run.

10.4.2 Verify the inter-element and background correction factors at the beginning of each analytical run. Do this by analyzing the SIC (Section 8.10). Results must be less than +/- LLOQ for all non-spiked elements.

10.4.3 When low-level sensitivity is required, a check standard at the requested limit of quantitation is analyzed to confirm the reported detection limit (RDL). This is performed on a project-by-project basis.

10.5 Preventive Maintenance

Whenever instrument maintenance is performed, it is noted in the instrument's Maintenance Logbook.

10.5.1 Daily

Inspect the nebulizer pump tubing from the Autosampler to the Nebulizer. Replace if necessary.

10.5.2 Monthly or as needed

Remove the torch, "shot glass", nebulizer and spray chamber. Clean each with 10% Nitric Acid and rinse with tap water. Coat the inside of the spray chamber and shot glass with concentrated Sulfuric Acid and soak for one hour, then rinse well with DI water. Soak the torch and nebulizer in aqua regia overnight, then rinse with DI water.

10.5.3 Every 6 months

Preventive Maintenance is performed by the Vendor or in-house personnel as follows:

- check the cooling system
- flush/refill the chiller with distilled water and antibacterial conditioner
- clean the instrument to regain intensity
- clean/replace air filters.

11. Data Evaluation, Calculations and Reporting

11.1 If dilutions were performed, the appropriate factors must be applied to sample values. All results must be reported with up to three significant figures.

11.2 Soil samples

Soil samples are calculated as follows:

$$\mathbf{A} = \frac{\text{Sample weight (grams)}}{\text{Final Volume (mL)}}$$

$$\mathbf{B}\ \text{(concentration in mg/Kg)} = \frac{\text{Concentration of analyte (mg/L)}}{\mathbf{A}}$$

11.2.1 Dry weight correction

The LIMS calculates the dry weight correction, however it is calculated as follows:

$$\text{Final concentration in mg/Kg dry weight} = \frac{\mathbf{B}}{\%\ \text{Solids}}$$

11.3 Liquid samples

Liquid samples are calculated as follows:

$$\text{Dilution Factor} = \frac{\text{Final Volume (mL)}}{\text{Sample Volume (mL)}}$$

$$\text{Final concentration in mg/L} = \text{Concentration of analyte (mg/L)} \times \text{Dilution Factor}$$

11.4 Calculations for Hardness

The method for determining hardness is to compute it from the results of separate determinations of Calcium and Magnesium on aqueous samples.

11.4.1 Total Hardness

Total Hardness, mg equivalent $CaCO_3$/L = [2.497 (Ca, mg/L)] + [4.118 (Mg, mg/L)]

11.4.2 Calcium Hardness

Calcium Hardness, mg equivalent $CaCO_3$/L = [2.497 (Ca, mg/L)]

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Also refer to Section 9 for Quality Control and acceptance criteria.

If the SIC is outside of the recovery window, then the standard is reanalyzed. If the standard failure continues, the IECs for the element/elements in question are reviewed and recalculated if necessary.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard. If the standard passes the second time then the analysis may be continued. The raw data is noted. If the standard fails again, the problem must be found and corrected and the instrument is recalibrated. The ICV/ICB standard is reanalyzed and all previous data that had failed back to the previous passing CCV/CCB is reanalyzed.

The reanalysis procedure outline above is also conducted for a failing LCS or Method Blank; they may be rerun alone on the new or any subsequent passing bracket. The LCS or Method Blank do not qualify a bracket of samples but the batch run itself.

If the Matrix Spike does not meet acceptance criteria, a dilution test is performed. If the levels of the native sample is inadequate (see section 10.3.6) The RPD must be within 20% of the true value of the native sample. If the dilution test fails or the concentrations in the native sample are inadequate, the post spike is analyzed and evaluated (section 10.3.5). If these criteria are met, then the Matrix Spike data is reported, with the post spike narrated on the final report. If the post spike fails the data is reported as estimated.

If sample Duplicates are outside of the acceptance criteria, the analyst examines the sample for homogeneity. If the sample is not homogenous, this is narrated on the final report. Clean, homogenous samples are reanalyzed and if still outside of the acceptance limits, redigested and reanalyzed.

Sample nonconformance regarding a Matrix Spike recovery or a duplicate %RSD is narrated on the final report along with the corrective action(s) taken.

The mixed element SIC solution is analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than +/- the LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, instrument is recalibrated, verified with the ICV/ICB and the SIC is then re-analyzed. Alternatively, the LLOQ may be raised to twice the concentration observed in the SIC solution if approved by the Department Manger or QA Department and the level is below the regulatory action limit or project specific requirements. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions These may be present up to the concentration documented plus the LLOQ. If failure continues notify the Department Supervisor or Manager.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05 unless supersede within this SOP. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP #1732 MDL/LOD/LOQ Generation

SOP# 1739 IDC/DOC Generation

SOP# 1728 Waste Management and Disposal

16. Attachments

TABLE 1: Element Wavelengths

TABLE 2: Precision and Accuracy Acceptance Criteria

TABLE 3: Reporting Limits

TABLE 1
ELEMENT WAVELENGTHS

Element	6500 Duo wavelength (nm)
Pb	220.3
Se	196.0
Sb	206.8
As	189.0
Ba	455.4
Be	313.0
Cd	214.4
Co	228.6
Cu	324.7
Cr	267.7
Fe	259.9
Mn	257.6
Mo	202.0
Ni	231.6
Ag	328.0
Tl	190.8
V	292.4
Zn	206.2
Al	396.1
Ca	315.8
Mg	279.0
B	208.9
Si	212.9
Sn	189.9
Sr	421.5
Ti	334.9
Bi	223.0
Na	589.5
K	766.4
S	180.7

TABLE 2
PRECISION AND ACCURACY ACCEPTANCE CRITERIA

	% Recovery LCS		Aqueous % Recovery MS		Soil % Recovery SRM *		Duplicate	
Element	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**	**Aqueous %RPD**	**Soil %RPD**
Aluminum	80	120	75	125	29	171	20	20
Antimony	80	120	75	125	4	196	20	20
Arsenic	80	120	75	125	81	119	20	20
Barium	80	120	75	125	83	118	20	20
Beryllium	80	120	75	125	83	117	20	20
Boron	80	120	75	125	70	129	20	20
Cadmium	80	120	75	125	82	117	20	20
Calcium	80	120	75	125	83	117	20	20
Chromium	80	120	75	125	80	119	20	20
Cobalt	80	120	75	125	83	117	20	20
Copper	80	120	75	125	83	117	20	20
Iron	80	120	75	125	51	150	20	20
Lead	80	120	75	125	80	120	20	20
Magnesium	80	120	75	125	74	126	20	20
Manganese	80	120	75	125	83	117	20	20
Molybdenum	80	120	75	125	81	119	20	20
Nickel	80	120	75	125	82	117	20	20
Potassium	80	120	75	125	74	126	20	20
Sulfur	80	120	75	125	NA	NA	20	20
Selenium	80	120	75	125	80	120	20	20
Silica (SiO_2)	80	120	75	125	NA	NA	20	20
Silver	80	120	75	125	66	134	20	20
Sodium	80	120	75	125	74	127	20	20
Strontium	80	120	75	125	80	120	20	20
Thallium	80	120	75	125	79	120	20	20
Tin	80	120	75	125	69	131	20	20
Titanium	80	120	75	125	82	118	20	20
Vanadium	80	120	75	125	79	121	20	20
Zinc	80	120	75	125	82	119	20	20

** Ranges of the SRM are presented as an example of a typical SRM; actual limits may vary by lot provided by the vendor.

TABLE 3
REPORTING LIMITS

Element	Aqueous (mg/L)	Soil (mg/Kg)
ALUMINUM	0.10	4.0
ANTIMONY	0.05	2.0
ARSENIC	0.005	0.40
BARIUM	0.01	0.40
BERYLLIUM	0.005	0.20
BORON	0.03	1.2
CADMIUM	0.005	0.40
CALCIUM	0.10	4.0
CHROMIUM	0.01	0.40
COBALT	0.02	0.80
COPPER	0.01	0.40
IRON	0.05	2.0
LEAD	0.01	2.0
MAGNESIUM	0.10	4.0
MANGANESE	0.01	0.40
MOLYBDENUM	0.05	2.0
NICKEL	0.025	1.0
POTASSIUM	2.5	100
SULFUR	0.25	10
SELENIUM	0.01	0.80
SILICA	0.50	20
SILVER	0.007	0.40
SODIUM	2.0	80
STRONTIUM	0.01	2.0
THALLIUM	0.02	0.80
TIN	0.05	4.0
TITANIUM	0.01	0.40
VANADIUM	0.01	0.40
ZINC	0.05	2.0

TABLE 4
LCS and Matrix Spike

Analyte	Liquid Concentration (mg/L)	Soil Concentration * (MS spike only) (mg/Kg)
Antimony	0.5	160
Arsenic	0.12	160
Barium	2.00	160
Beryllium	0.05	80
Cadmium	0.051	80
Chromium	0.20	160
Copper	0.25	160
Lead	0.51	160
Nickel	0.50	160
Selenium	0.12	160
Silver	0.05	40
Thallium	0.12	160
Zinc	0.50	160
Iron	1.00	800
Manganese	0.50	160
Calcium	10.0	800
Magnesium	10.0	800
Potassium	10.0	800
Sodium	10.0	800
Silica	1.0	800
Aluminum	2.00	800
Cobalt	0.50	160
Vanadium	0.50	160
Boron	1.0	NA
Molybdenum	1.0	NA
Titanium	1.0	NA

*MS spike of a solid based on 1.25g and a final volume of 50 mL.

Note: Solids LCS is an SRM with certified value provided by the vendor on a lot basis.

Mercury in Liquid Waste

(Automated Cold-Vapor Technique)

Reference Method No.: **EPA 7470A**

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update II, September 1994.

1. Scope and Application

Matrices: Method 7470 is a cold-vapor atomic absorption procedure approved for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. (Method 7470 can also be used for analyzing certain solid and sludge-type wastes; however, Method 7471 is usually the method of choice for these waste types.) All samples must be subjected to an appropriate dissolution step prior to analysis.

Definitions: See Alpha Laboratories Quality Manual Appendix A.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Mercury Analyzer and in the interpretation of Mercury data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Prior to analysis, the liquid samples must be prepared according to the procedure discussed in this method.

Method 7470, a cold-vapor atomic absorption technique, is based on the absorption of radiation at 253.7-nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.1 Method Modifications from Reference

2.1.1 A smaller sample sized is prepared, and therefore proportionately less reagent volumes are used.

2.1.2 The original method does not address the automated instrument procedure.

3. Reporting Limits

The typical reporting limit for Mercury is 0.0002mg/L. This satisfies Massachusetts, GW1 and GW 2 criteria. Connecticut mobility criteria for SPLP is 0.0004mg/L, Rhode Island is 0.002mg/L, and the Drinking Water reporting limit is 0.0002mg/L.

4. Interferences

Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL). Both inorganic and organic mercury spikes have been quantitatively recovered from seawater by using this technique.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Analysis is conducted under a laboratory exhaust hood. The analyst must wear chemical resistant gloves when handling concentrated mercury standards.

The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Therefore, the acidification of samples is to be conducted under a laboratory exhaust hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in either glass or plastic containers.

6.2 Sample Preservation

Samples are preserved with HNO_3 to a pH of <2.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are stored under refrigeration at 4 ± 2°C and analyzed as soon as possible after collection. The samples have a 28-day holding time from the time of collection.

7. Equipment and Supplies

Instrumentation:

Perkin Elmer FIMS 100 Atomic absorption spectrophotometer: Use instrument settings recommended by the manufacturer. The PE FIMS is designed specifically for the measurement of mercury using the cold-vapor technique with BOC (background offset correction) performed by a survey scan prior to each sample introduction. PE S10 autosampler is coupled to the instrument.

Cetac M-6100 Atomic absorption spectrophotometer: Use instrument settings recommended by the manufacturer. This instrument employs a reference cell off-set correction and full automation through the CETAC software. A Cetac ASX-260 autosampler is coupled to the instrument.

Nippon Instrument model# RA-4300A analyzer with integrated 80 position autosampler:

The instrument adds a stannous chloride (II) solution to the sample post digestion, the divalent mercury ion (Hg2+) is reduced to zero-valent metallic mercury and turns into mercury gas by bubbling. $Hg2{+}{+}SnCl2 \rightarrow Hg0 \uparrow$

After removing the acid mist and water vapor generated by bubbling with an electronic cooling unit, the instrument measures the absorbance of mercury at 253.7 nm absorption wavelength. It measures the known mercury amount, creates a calibration curve, and then calculates the mercury amount from the absorbance.

7.1 Graduated cylinder: Rinse once with 50% HNO_3 and then rinse with reagent water prior to use.

7.2 Volumetric Flasks, Class A, various volumes: Rinse once with 50% HNO_3 and then rinse with reagent water prior to use.

7.3 Heating Block: Environmental Express HotBlock, 48 position capacity, able to maintain 95°C +/-3.

7.4 50 mL Digestion Tubes: Polypropylene, graduated.

7.5 50 mL Digestion Tube Rack: 48 position, racklock

7.6 Pump tubing: Environmental Express, three stop and two in various IDs.

7.7 PTFE membranes: Whatman TE37 disks.

7.8 Dilution vials: 20mL capacity, used to prepare analytical dilutions.

7.9 Low Dust Laboratory Wipes

7.10 Compressed Air

7.11 Whatman 41 filter paper or equivalent

8. Reagents and Standards

8.1 Reagent Water: Reagent water is DI water shown to be interference free. All references to water in this method will refer to reagent water unless otherwise specified.

8.2 Sulfuric acid (H_2SO_4), concentrated: Reagent grade. Store at room temperature in an appropriately designated acid cabinet.

8.3 Hydrochloric acid, concentrated: Trace Metal grade. Store at room temperature in an appropriately designated acid cabinet.

8.4 Carrier, Hydrochloric Acid, 3%: This is the ***carrier*** for the Instrument. In a 1L volumetric flask, add 30mL concentrated trace grade HCl (Section 8.3). Bring to volume with reagent water. Store at room temperature, prepare daily as needed.

8.5 Reductant, Stannous Chloride in 3% HCl: This is the ***reductant*** for the Instrument. In a 1L volumetric flask, add 30mL concentrated trace grade HCl and 11g $SnCl_2 \cdot 2H_20$. Mix to dissolve the solid and bring to volume with reagent water. Store at room temperature, prepare daily as needed.

8.6 Nitric acid (HNO_3), concentrated: Trace metal grade of low mercury content. If a high reagent blank is obtained, it may be necessary to distill the nitric acid. Store at room temperature in an appropriately designated acid cabinet.

8.7 Sodium chloride-hydroxylamine hydrochloride solution: Dissolve 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride in reagent water and dilute to 100mL. Store at room temperature. Expires one month from date of preparation.

8.8 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 5 g of potassium permanganate in 100 mL of reagent water. Store at room temperature. Expires one month from date of preparation.

8.9 Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate in 100 mL of reagent water. Store at room temperature. Expires one month from date of preparation.

8.10 Mercury Stock Standard, 1000ppm: Purchased from a commercial source with a certificate of analysis. Purchase three different sources. Store at room temperature. Expires upon manufacturer's specification.

8.11 Mercury Stock Calibration Standard, 10ppm: To a 100mL volumetric flask, add 5mL of concentrated HNO_3, 2.5mL of concentrated H_2SO_4 and 1000ppm Mercury Stock Standard (Section 8.10, use one source). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.

8.12 Mercury Working Calibration Standard / Matrix Spike Solution, 0.1ppm: To a 100mL volumetric flask, add 5mL of concentrated HNO_3, 2.5mL of concentrated H_2SO_4 and 1mL of 10ppm Mercury Stock Standard (Section 8.11). Bring to volume with reagent water. Store at room temperature. Make fresh daily.

8.13 Mercury Calibration Standards: All calibration standards are prepared daily.

8.13.1 0 ppm Calibration Standard: Add 10 mL of reagent water to a polypropylene digestion vessel. This aliquot may be used for the CCB. Another separate aliquot is prepared in the same manner for use as the ICB and diluent for sample dilutions.

8.13.2 **0.0002ppm Calibration Standard:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 0.05 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.

8.13.3 **0.001ppm Calibration Standard:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 0.25 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.

8.13.4 **0.002ppm Calibration Standard** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 0.5 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.

8.13.5 **0.005ppm Calibration Standard/Continuing Calibration Verification Standard:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 1.25 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.

8.13.6 **0.010ppm Calibration Standard / Post Analytical Spike Solution:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 2.5 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.

8.13.7 **0.020ppm Calibration Standard / Post Analytical Spike Solution:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 5.0 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.

8.14 Mercury Stock LCS Standard, 10ppm: To a 100mL volumetric flask add 25mL of reagent water and 5mL of concentrated HNO_3 (Section 8.6). Add 1mL of 1000ppm Mercury Stock Standard (Section 8.10). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.

8.15 Mercury Working LCS Standard, 0.1ppm: To a 100mL volumetric flask add 25mL of reagent water and 5mL concentrated HNO_3 (Section 8.6). Add 1mL of 10ppm Stock LCS Standard (Section 8.14). Bring to volume with reagent water. Store at room temperature. Expires one week from date of preparation.

8.16 Mercury LCS Standard, 0.001ppm: Prepare daily with each batch of samples. To a 50mL digestion vessel add 10mL of reagent water Add 0.25 mL of 0.1ppm Working LCS Standard (Section 8.15). Bring to a final volume of 25mL and carry through entire digestion process as in Section 10.1.1.

8.17 Mercury Stock ICV Standard, 10ppm: To a 100mL volumetric flask add 25mL of reagent water and 5mL of concentrated HNO_3 (Section 8.6). Add 1mL of 1000ppm Mercury Stock Standard (Section 8.10-use alternate source than that used for both the calibration standards and the LCS Stock Standard). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.

8.18 Mercury Working ICV Standard, 0.3ppm: To a 100mL volumetric flask add 25mL of reagent water and 5mL of concentrated HNO_3 (Section 8.6). Add 3mL of 10ppm Stock ICV Standard (Section 8.5). Bring to volume with reagent water. Store at room temperature. Expires one week from date of preparation.

8.19 Mercury ICV Standard, 0.003ppm: Prepare daily with each batch of samples. To a 25mL digestion vessel add 10mL of reagent water. Add 0.25mL of 0.3ppm Working ICV Standard (Section 8.18). Bring to a 25mL final volume with reagent water and carry through entire digestion process as in Section 10.1.1..

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

The ICB, CCB, and Method Blank: A 25mL aliquot of 0ppm calibration standard brought through the preparation procedure as outlined in Section 10.1.1 . Blank results must be <RL. See Section 12.1 for corrective action. An ICB is analyzed after the initial calibration or re-calibration. The CCB is analyzed at every 10 sample injection interval. Method Blank is analyzed once per batch of samples; batch consists of 20 samples.

9.2 Laboratory Control Samples (LCS)

The LCS Standard consists of a 0.001ppm Mercury LCS Standard (Section 8.16). This standard is brought through the preparation procedure as outlined in Section 10.1.1. The LCS Standard must be recovered within ± 20% of the true value. See Section 12.3 for corrective action. The LCS Standard is analyzed once per batch of samples. A batch consists of 20 samples.

9.3 Initial Calibration verification (ICV)

The ICV Standard consists of a 25mL aliquot of 0.003ppm Mercury ICV Standard (Section 8.19). The ICV must be recovered within 10% of the true value. See Section 12.2 for corrective action.

9.4 Continuing Calibration Verification (CCV)

The CCV Standard consists of a 0.010ppm calibration standard (Section 8.13.3). The CCV must be recovered within 20% of the true value. See Section 12.2 for corrective action.

9.5 Matrix Spike

A matrix spike is analyzed once per batch of samples. A batch consists of 20 samples for monitoring wells, surface waters, influents and effluents. Prepare the matrix spike at 0.005ppm by adding 1.25mL of 0.1ppm Mercury Stock Standard (Section 8.3) to 25mL of the selected QC sample. The final concentration of the matrix spike is 0.005mg/L.

The matrix spike sample is brought through the preparation procedure as outlined in Section 10.1. A matrix spike is analyzed once per batch of samples. A batch consists of 20 samples for monitoring wells and surface waters. The recovery of the matrix spike must be between 75 – 125% (using the calculation in Section 11.2).

If the recovery of the matrix spike is out of range, a post-analytical spike is analyzed. Prepare the post analytical spike by adding 5mL of 0.010ppm Calibration Standard / Post Analytical Spike Solution (Section 8.1.6) and 5mL of the sample digestate to a 50mL centrifuge tube for a final concentration of 0.005mg/L. Analyze the post spike as outlined in Section 10.3.5.

Calculate the post spike concentration as follows:

Post Analytical Spike Sample Concentration (mg/L) =

[Sample Concentration (mg/L) x (0.5)] + 0.005mg/L

The percent recovery of the post-analytical spike must be between 75 – 125%. See Section 12.4 for corrective action.

9.6 Laboratory Duplicate

A sample is analyzed in duplicate once per batch of samples. A batch consists of 20 samples for monitoring wells and surface waters. The RPD between the sample and its duplicate must be 20% or less (using the calculation in Section 11.3), See Section 12.5 for corrective action.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Calibration Blank
- 0.0002 ppm Calibration Standard
- 0.0005 ppm Calibration Standard
- 0.001 ppm Calibration Standard
- 0.002 ppm Calibration Standard
- 0.010 ppm Calibration Standard
- 0.020 ppm Calibration Standard
- ICV
- ICB
- Ten analytical samples
- CCV
- CCB
- Ten analytical samples
- CCV
- CCB
- Etc.

10. Procedure

10.1 Equipment Set-up

10.1.1 Preparation and Digestion

Samples, Standards and All Batch QC

Transfer 25mL of well-homogenized sample (or an aliquot of the sample diluted to 25mL with reagent water) or standards (Sections 8.13.1 through 8.13.7, 8.16 and 8.19) to a 50mL centrifuge tube.

Add 1.25mL of concentrated H_2SO_4 (Section 8.2), 0.625mL of concentrated HNO_3 (Section 8.6), Add 3.75mL of Potassium Permanganate Solution, shake and add additional portions of potassium permanganate solution (if necessary) to all samples and QC, until the purple color persists for at least 15 min. (Section 8.8). Add 2mL of Potassium Persulfate Solution (Section 8.9), and heat samples for 2 hours in a 95°C +/-3

heating block. Cool, and add 1.5mL of Sodium Chloride-Hydroxylamine hydrochloride solution (Section 8.7).
Filter the sample if needed to remove any sediment or particulate.

Analyze samples and the digested calibration standards (Sections 8.13.1 through 8.13.7) are used in Section 9.2 to generate a calibration curve.

10.2 Initial Calibration

Construct a calibration curve by plotting the absorbances of prepared standards (Section 10.1.1) versus micrograms of mercury. Determine the peak height of the unknown from the absorbance maxima on the spectrometer, and read the mercury value from the standard curve. (See Section 11.)

The curve correlation coefficient (cc) must be greater than or equal to 0.995 in order for the curve to be linear. If the correlation coefficient is less than 0.995, find and correct the problem. When the problem has been corrected, re-analyze either the previous standards or new standards. When the curve has generated an acceptable cc then the analysis can continue with the ICV/ICB.

10.3 Equipment Operation and Sample Processing

Sample and standard analysis:

10.3.1 Instrument Setup

10.3.1.1 Turn the instrument on. The autosampler will initialize itself.

10.3.1.2 Choose the instrument software from the desktop menu. The autosampler will initialize again.

FIMS 100 NOTE: The instrument must be turned on before the application is started. Otherwise, an error message will result.

10.3.1.3 Enter the appropriate fields for sample identification, and data storage.

10.3.1.4 Fill the carrier and reductant bottles.

10.3.1.4.1 The Carrier is 3% HCl (Section 8.6).

10.3.1.4.2 The Reductant is 1.1% $SnCl_2$ in 3% HCl (Section 8.5).

10.3.1.5 Allow the instrument to warm up while clearing samples. Samples that are cloudy or with particulate present after clearing must be filtered through Whatman 41 filter paper (Section 7.11) before analysis.

10.3.1.6 Place carrier uptake line and reductant uptake line.

10.3.1.7 Load carrier and reductant lines into pump magazines

10.3.1.8 Load the two waste lines into the pump magazines below the roller.

10.3.1.9 Lock the magazines into place.

FIMS100 only:

10.3.1.10 Remove the cap from the liquid/vapor separator and wipe dry with a Lab Wipe (Section 7.9). Compressed air (Section 7.10) through the vapor transfer line to dry it out.

10.3.1.11 Place a PTFE membrane (Section 7.7), rough side up, in the liquid/vapor separator; replace the cap and reattach the vapor transfer line to the sample absorption cell.

10.3.1.12 Adjust the gas flow by turning the black knob below the air flow meter to obtain a reading of just over 50.

10.3.2 Calibration and Sample Analysis

10.3.2.1 The instrument will now run the calibration standards; verify a CC of 0.995 or better before proceeding with the ICV and ICB. Ten analytical samples, a CCV and CCB, ten analytical samples, CCV, CCB, etc. The CCBs and CCVs must be recovered within the proper ranges (Sections 9.4 and 9.1.3) for analysis to continue.

10.3.2.2 If the sample result is beyond 90% of the concentration of the highest point on the calibration curve or LDR study used to establish the linear range, dilute the sample extract with a portion of one of the prepared blanks (ICB, CCB or PBS) to produce an analytical result that is within the range.

10.3.3 Instrument Shut Down

10.3.3.1 When analysis is complete place reagent uptake lines in a beaker of reagent water.

10.3.3.1.1 Continue to run the pumps for several minutes to flush reagents out of the lines.

10.3.3.1.2 Continue to run the pumps for several minutes to flush reagents out of the lines.

10.3.3.2 Pull the reagent uptake lines out of the reagent water beaker to allow the pump to draw air through the lines.

10.3.3.3 Unlock the top and bottom pump magazines and remove tubing from the magazines.

10.3.3.4 Exit from the software application.

10.3.3.4.1 Dump the samples and instrument waste in the Metals/WetChem waste drum located in the transfer room.

10.4 Continuing Calibration

Continuing calibration verification samples are analyzed after every 10 samples in the sample run, as outlined in Section 10.3.5.

10.5 Preventative Maintenance

Preventative maintenance is conducted per the manufacturer's instructions. All preventative maintenance is recorded in the Instrument Maintenance Logbook.

11. Data Evaluation, Calculations and Reporting

11.1 Calculate Mercury concentration

Calculate Mercury concentration from the daily calibration curve. The curve is generated utilizing a straight-line equation defined as:

$$A = k_1 + k_2C$$

Where:

A = Average peak height of the sample/standard integrations
C = Sample/Standard Concentration, µg/L
k_1 = y-intercept
k2 = slope

The instrument will plot peak height against concentration (µg/L). The result is generated in µg/L . This value is divided by 1000 to convert the units to mg/L. If the sample is diluted (DF), the result is multiplied by the DF to generate the final result.

Result, mg/L = (concentration, µg/L) x (1mg/1000µg) x (DF)

11.2 Matrix Spike Calculation

Calculate percent recovery for the Matrix Spike corrected for concentrations measured in the unfortified sample. Percent recovery is calculated using the following equation:

$$\% \text{ Recovery} = \frac{(Cm - C)}{s} \times 100$$

Where:

Cm = measured Mercury in the fortified sample
C = measured native mercury sample concentration
S = concentration equivalent of spike added to sample

11.3 Relative Percent Difference (RPD) Calculation

Calculate the Relative Percent Difference (RPD) for each Duplicate of the initial quantitated concentration (IC) and duplicate quantitated concentration (Dc) using the following formula:

$$RPD = \frac{|(IC - Dc)|}{\{(IC + Dc) / 2\}} \times 100$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 Method Blank Failure: When a prep blank mercury level constitutes 10% or more of analyte level determined for any sample in the batch, or is greater than 2.2x the MDL value (whichever is greater), the associated samples in the batch must be redigested (Section 10.1).

For method blanks that have concentrations greater than the RL, the data is rejected and the associated samples sent back for redigestion unless the associated sample concentrations are greater than 10x the blank concentration. In this case the blank is narrated and the results are reported without qualification.

12.2 ICV / CCV Failure: If the ICV %Recovery is outside of acceptance criteria, the ICV is reinjected. If the %Recovery is outside the acceptance criteria, the analysis is terminated until the problem is found and corrected. If the CCV %Recovery is outside of acceptance criteria, the CCV is reinjected. If the % Recovery is still outside the acceptance criteria, all samples analyzed since the last acceptable CCV must be reanalyzed following correction of the problem.

12.3 LCS Failure: If the LCS is not recovered within acceptance criteria, the LCS is reinjected. If the % Recovery is still outside the acceptance criteria, either recalibrate and rerun or the associated batch and another LCS must be redigested (Section 10.1).

12.4 Matrix Spike / Post Digestion Spike Failure: If the recovery of the matrix spike is outside of the acceptance criteria, a post digestion spike is performed (Section 9.54). If the post digestion spike is outside of 75 – 125%, a narrative must be included with the data. (Section 10.1).

12.5 Duplicate Failure: If the RPD between the sample and its duplicate is greater than 20%, visually evaluate the sample matrix. If the matrix appears problematic, the sample digestate may be diluted and reanalyzed, or a narrative included with the data to explain the matrix problem.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

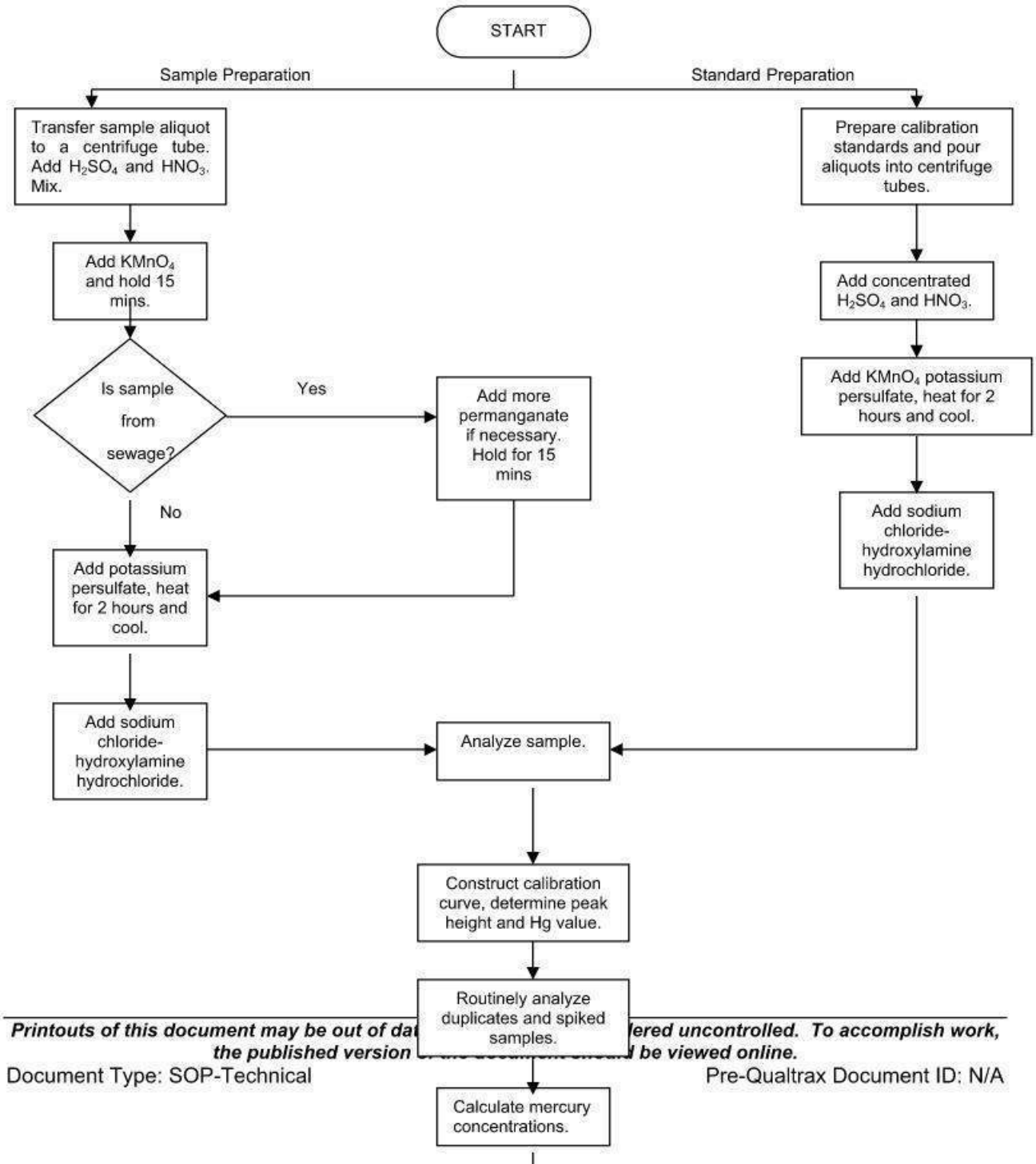
SOP/1797 Waste Management and Disposal SOP

16. Attachments

Figure 1: Method 7470A Flow Chart

Figure 1

Method 7470A Flow Chart



END

PCBs

By Capillary Column Gas Chromatography

Reference Methods: Method 8082A SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, 2007.

Quality Control Requirements and Performance Standards for Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography (GC) in Support of Response Action under the Massachusetts Contingency Plan (MCP), Revision No.1, July 1, 2010.

State of Connecticut, Department of Environmental Protection, RRCP, Version 2.0, July 2006.

1. Scope and Application

Method 8082A is used to determine the concentrations of Polychlorinated Biphenyls (PCBs) as Aroclors in extracts from solid and liquid matrices. This SOP details the analysis for PCBs using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD).
Matrices: Extracts from solid and liquid matrices.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Regulatory Parameter List: The standard compounds listed below are determined by this method.

Parameter	CAS#
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
* Aroclor 1262	37324-23-5
*Aroclor 1268	11100-14-4

*Note – not all states certify for Aroclors 1262 and 1268, including NJ

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.
This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph (GC) and in the interpretation of gas chromatograms. Each analyst must

demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability (see section 13.2).

2. Summary of Method

A measured volume or weight of sample (volumes and weights can vary but approximately 1L or 125 mls (LVI – Low Volume Initiative) for liquids, 15g to 30g for solids) is extracted using the appropriate matrix-specific sample extraction technique.

Liquid samples are extracted at neutral pH with methylene chloride using Method 3510C (separatory funnel), or other appropriate technique. See extraction SOP for details.

Solid samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet), or other appropriate technique. Solid samples may also be extracted with hexane:acetone (1:1) using Method 3546 (microwave). See extraction SOP for details.

Wipe samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet) or other appropriate technique. See extraction SOP for details.

Oil samples are diluted with hexane following the procedure outlined in the extraction SOP.

Sulfuric acid cleanup (Method 3665A), Copper cleanup (Method 3660B) and Silica Gel cleanup (Method 3630) are utilized for PCB extracts. See extraction SOP for details.

After cleanup, the extract is analyzed by injecting 1µL into a gas chromatograph equipped with narrow- or wide-bore fused silica capillary columns and electron capture (GC/ECD) detectors.

2.1 Method Modifications from Reference

Not applicable.

3. Reporting Limits

The reporting limits for this method as outlined is as follows:

- Aqueous samples: 0.25 ug/L / Aroclor (based on a 1L extraction or 125 ml LVI extraction)
- Soil Samples: 33.3 ug/kg / Aroclor (based on a 15g extraction)
- Oil Samples: 5 mg/Kg (based on 1g extraction)
- Solid of Difficult Matrices (i.e Caulking, Concrete, etc. are logged using the Alpha Low Level 8082 products): based on a 15g extraction
    - Aroclors 1016, 1221, 1232, 1242, 1254: 20 ug/kg
    - Aroclors 1248, 1260: 13.3 ug/kg
    - Aroclors 1262, 1268: 6.67 ug/kg

4. Interferences

4.1 Instrumental

4.1.1 Only high purity gases are used in the GC system to eliminate this source of possible contamination. Both the helium (carrier gas – 99.999%) and argon-methane (detector make-up gas) are certified by the gas supplier.

4.1.2 Preventive instrument maintenance is performed routinely, and whenever highly contaminated extracts are analyzed that could result in chromatographic interferences or result in degradation of system performance. Section 10.5 details the maintenance steps.

4.1.3 Glassware must be scrupulously cleaned. This procedure is detailed in the Organic Extraction Cleaning and Handling SOP/1953. Store dry glassware in a clean environment.

4.2 Parameters

4.2.1 All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free. All of these materials are routinely determined to be free of interferences by analysis of extraction blanks with every extraction batch performed.

4.2.2 Certain compounds (i.e. phthalates) can be extracted from the sample matrix and be detected by the ECD that could possibly result in false positive results or complicate the data interpretation. The use of the cleanup procedures detailed in the extraction SOP minimizes these possible interferences. Analyst experience is also crucial in making compound determinations.

4.2.3 Interferences co-extracted from the samples will vary considerably from waste to waste. While a general cleanup technique is referenced or provided as part of the method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. PCBs have been tentatively classified as known or suspected human or mammalian carcinogens. Additional references to laboratory safety are available in the Chemical Hygiene Plan.
All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two 1L or two 125 ml (LVI) amber glass jars with teflon-lined lids. Solid samples are collected in one 250 mL wide-mouth glass jar with a teflon-lined lid. Oil samples are collected in a glass jar with Teflon-lined lids. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of 4 ± 2° C. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of 4 ± 2° C.

6.3 Sample Handling

TCLP/SPLP tumbled extracts, NJ DKQP and RCP CT aqueous samples must be extracted within 7 days of sample collection, solid samples within 14 days of collection (NJ DKQP allows 365 days for solids if frozen). All other samples, both aqueous and solid, have a 365-day hold time. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph, Agilent 6890, 7890: An analytical system complete with gas chromatograph configured for split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and data system.

7.2 GC Columns: Alpha utilizes dual-column analyses. The dual-column approach involves either a single injection that is split between two columns that are mounted in a single gas chromatograph. Typical column pair used is listed below. Other columns may be used as long as method performance criteria can be met.

Column pair:

RTX-CLP: Cat. #11141 from Restek or equivalent; 30m, 0.32mm, 0.32µm

RTX-CLPII Cat. #11324 from Restek or equivalent; 30m, 0.32mm, 0.25µm

7.3 Guard Column: Cat. #10027 from Restek or equivalent; 5m, 0.32mm

7.4 Class "A" Volumetric Flasks: 10mL and 25mL (and other volumes), for standards preparation

7.5 Microsyringes/Wiretrol syringes: 10 µL – 1000 µL

7.6 Gooseneck splitless injecton liner, Cat #20799-214.5 from Restek or equivalent

7.7 Universal "Y" Press-tight tee split: Cat. #20406 from Restek or equivalent / **Siltek MXT Connector:** Cat. #21388 from Restek or equivalent

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4 ± 2° C in Teflon(R)-sealed containers in the dark. When a Lot of standards is prepared, aliquots of that Lot are stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC tests indicate a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem.

8.1 n-Hexane: Pesticide quality or equivalent.

8.2 Acetone: Pesticide quality or equivalent.

8.3 Organic-free Reagent Water: All references to water in this method refer to organic-free reagent water from Alpha's RO water treatment system.

8.4 Stock Standard Solutions: All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated stock solution is opened, it is transferred to a labeled amber screw-cap vial. The expiration date of the stock solution is either the vendor specified expiration date, or 1 year from the date the ampule was opened, whichever is sooner.

8.5 Calibration Standards: Calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the calibration standard. Calibrations are performed at the 6 concentration levels listed in Table 1. The list of ampulated calibration standards are obtain from **Ultra**:

- Aroclor 1016, Cat. #PP-282, at 100ug/ml
- Aroclor 1260, Cat. #PP-361, at 100ug/ml
- Aroclor 1262, Cat. #PP-371, at 100ug/ml
- Aroclor 1268, Cat. #PP-382, at 100ug/ml
- Aroclor 1221, Cat. #PP-292, at 100ug/ml
- Aroclor 1232, Cat. #PP-302, at 100ug/ml
- Aroclor 1242, Cat. #PP-312, at 100ug/ml
- Aroclor 1248, Cat. #PP-342, at 100ug/ml
- Aroclor 1254, Cat. #PP-351, at 100ug/ml

8.6 Second Source Standards: (ICV/CCAL) Continuing Calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Continuing Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the standard. The list of ampulated standards are obtain from **Accustandard**:

- Aroclor 1016, Cat. #C-216S-H-10X, at 1000ug/ml
- Aroclor 1260, Cat. #C-260S-H-10X, at 1000ug/ml
- Aroclor 1262, Cat. #C-262S-H-10X, at 1000ug/ml
- Aroclor 1268, Cat. #C-268S-H-10X, at 1000ug/ml
- Aroclor 1221, Cat. #C-221S-H-10X, at 1000ug/ml
- Aroclor 1232, Cat. #C-232S-H-10X, at 1000ug/ml

- Aroclor 1242, Cat. #C-242S-H-10X, at 1000ug/ml
- Aroclor 1248, Cat. #C-248S-H-10X, at 1000ug/ml
- Aroclor 1254, Cat. #C-254S-H-10X, at 1000ug/ml

8.7 Internal Standard Solution: 1-Bromo-2-nitrobenzene (Ultra, Cat. #PPS-351) is used as the internal standard, and is added to all single-component calibration standards and sample extracts to achieve a concentration of 0.25µg/mL. For LVI, this solution is diluted 10X more, achieving a concentration of 0.025µg/mL.

8.8 Surrogate Standards: Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl (DCB) are used as surrogates for Aroclor analysis. They are added to the calibration standards at the concentrations listed in Table 1, Continuing Calibration Standards and are spiked into all samples and QC samples prior to extraction.

- **ICAL Surrogates Stock:** is prepared by diluting of 500ul of Pesticides Surrogates Standard Spiking Solution (Ultra, Cat. #ISM-320-1) and 500ul of Decachlorobiphenyl (Accustandard, Cat. #CLP-032-R-01) to 20ml of Hexane to achieve concentration of TCMX at 5ug/ml and DCB at 10ug/ml. For LVI, this solution is diluted 10X more, achieving a concentration of 0.5 ug/ml for TCMX and 0.1 ug/ml for DCB.
- **CCAL Surrogates Stock:** is prepared by diluting of 1ml of TCMX&DCB (Accustandard, Cat. #CLP-032-R) and 1ml of Decachlorobiphenyl (Accustandard, Cat. #CLP-032-R-01) to 20ml of Hexane to achieve concentration of TCMX at 10ug/ml and DCB at 20ug/ml. For LVI, this solution is diluted 10X more, achieving a concentration of 1 ug/ml for TCMX and 2 ug/ml for DCB.
- **Extraction Surrogates Stock:** is prepared by diluting of 10ml of TCMX&DCB (Accustandard, Cat. #CLP-032-R) to 1000ml of Acetone to achieve concentration of TCMX and DCB at 2ug/ml. For LVI, this solution is diluted 10X more, achieving a concentration of 0.2 ug/ml for both TCMX and DCB.

8.9 LCS/MS Spiking Solutions: The LCS/MS spiking solution is prepared by diluting of 6.25ml of Arochlor 1016/1260 (Restek, Cat. #32039) to 500ml of Acetone to achieve concentration of Arochlor 1016/1260 at 12.5ug/ml. For LVI, 1.25 ml of the stock solution is diluted to 500 mls of Acetone to achieve a concentration of Aroclor 1016/1260 at 2.5 ug/ml.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A Method Blank is an aliquot of a clean reference matrix (reagent water for water samples, Ottawa sand for soil/sediment samples, or PCB free transformer oil for oil samples) that is carried through the entire analytical procedure. Extraction blanks are performed with each extraction batch of 20 or less samples, according to the extraction SOPs. The extraction blank must not contain any of the reportable analytes above the reporting limit. If any reportable analytes are detected in the blank, the entire extraction batch is suspect and re-extraction of all associated samples is required, unless the associated samples are non-detect or concentration of the analyte in the samples is 10 times greater than the concentration of this analyte in the blank. The surrogate recoveries must also be within the

acceptance criteria listed in Table 2. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary.

9.2 Laboratory Control Sample (LCS)

A Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD) pair is extracted with each analytical batch. The LCS/LCSD consist of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. For Aroclor analysis, the LCS/LCSD are spiked with a mixture of Aroclor 1016 and 1260 (1660). The recovery acceptance criteria are listed in Table 2. If any recovery criteria are not met, the extract may be re-analyzed. If the criteria are still not met, the **entire batch is re-extracted**, unless the recoveries are high and the associated samples are non-detect. If this is not possible, due to insufficient sample or holding time exceedances, the analyst must narrate the failure in the LIMS for inclusion in the client report.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.7.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike

Upon client request, a matrix spike and matrix spike duplicate pair are extracted and analyzed with each batch of 20 or less samples. The MS/MSD pair is extracted and analyzed for standard PCB analysis. The recovery acceptance criteria are listed in Table 2. If the recovery criteria are not met, but are met in the LCS, the failure may be attributed to sample matrix effects and must be narrated for inclusion in the client report.

9.6 Laboratory Duplicate

Upon client request, a Laboratory Duplicate is extracted and analyzed with each batch of 20 or less samples. The relative percent difference (RPD) acceptance criteria are listed in Table 2. If the RPD criteria are not met, the failure may be attributed to matrix effect and must be narrated for inclusion in the client report.

9.7 Surrogates

All extracted samples and associated QC are spiked with Extraction Surrogates Stock to achieve concentration of TCMX and DCB at 0.5ug/ml (0.2 ug/ml for LVI). The laboratory must evaluate surrogate recovery data from individual samples and QC samples versus the surrogate control limits listed in Table 2. If the surrogate limits are not met, the extract may be reanalyzed to determine if the failure was due to an instrument problem. If the criteria are still not met, the affected samples must be re-extracted to confirm that the failure was due to sample matrix, unless the surrogate recovery is high and the associated sample is non-detect. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

9.8 Method Sequence

Typical Initial calibration (each level to identified with the standard lot number)

1. Prime
2. Blank

3. Standard Level 1
4. Standard Level 2
5. Standard Level 3
6. Standard Level 4
7. Standard Level 5
8. Standard Level 6
9. Initial Calibration Verification Standard (ICV)

Repeat steps 3 – 9 as needed for each Aroclor necessary for calibration.

NOTE: If multiple calibration mixtures are analyzed, it is acceptable to analyze appropriate ICVs after all calibration standards have been injected.

Typical Daily Sequence

1. 1660 Continuing Calibration Standard **(identified with the standard lot number)**
2. Extraction Blank
3. Laboratory Control Sample
4. Matrix Spike / Matrix Spike Duplicate (if requested by Client)
5. Duplicate (if included with batch QC)
6. Samples up to 16
7. Repeat 1 – 6 as needed.

10. Procedure

10.1 Equipment Set-up

10.1.1 GC Conditions:

The dual-column / dual-detector approach involves the use of the columns listed in section 7.2. The columns are connected to an injection tee or dual injection GC, and separate electron capture detectors. Alpha typical GC conditions are listed below, but may be altered as long as method performance criteria are met.

Temperature1: 120 ºC	Injector temerature: 250ºC
Time1: 0 minutes	Injector mode: Pulsed Split
Ramp1: 45ºC/minute	1.4:1 split, 0.20 min pulse
Temperature2: 200ºC	Injector Flow: 5.7 ml/min split flow
Time2: 0 minutes	Detector temperature: 350ºC
Ramp2: 15ºC/minute	Carrier gas: Helium
Temperature3: 230ºC	Carrier flow: 20ml/min
Time3: 0 minutes	Carrier mode: Constant flow
Ramp3: 30ºC/minute	Makeup gas: Argon/methane (P5)

Final temperature 330°C
Final time: 2 minutes

Total detector flow: 55ml/min
Injection Volume: 1 µL

10.2 Initial Calibration

10.2.1 Prepare calibration standards using the standards listed in Section 8.5 to achieve the concentrations from Table 1. Alternatively, a standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 (1660) at five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing multi-point initial calibrations for each of the seven Aroclors. In addition, such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. Single standards of each of the other seven Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards have been used to demonstrate the linearity of the detector, these single standards of the remaining seven Aroclors also may be used to determine the calibration factor for each Aroclor when a linear calibration model through the origin is chosen. Prepare a standard for each of the other Aroclors. The concentrations should generally correspond to the mid-point of the linear range of the detector, but lower concentrations may be employed.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in section 10.1.1. The same operating conditions are used for calibrations and sample analyses. Create the analytical sequence using the Agilent Chemstation data acquisition software. Record the calibration standard, unique lot number (PP#) and analyst's initials in the analytical sequence list.

10.2.3 A 1µL injection volume of each calibration standard is typically used. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. The same injection volume must be used for all standards and samples.

10.2.4 Column adsorption may be a problem when the GC has not been used for a day or more or after system maintenance. The GC column may be primed (or deactivated) by injecting a PCB standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

Alternately, the system may be primed by baking at the final analytical temperature for approximately 30 minutes.

Several analytes may be observed in the injection just following system priming. Always run an instrument blank after system priming.

10.2.5 **Calibration Factor:** Internal standard calibration techniques are employed in this method.

10.2.5.1 Internal Standard Procedure. In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The

calculations are performed automatically, using the formula listed in Alpha's Quality Manual.

Alternatively, standards of the other seven Aroclors are necessary for pattern recognition. When employing the traditional model of a linear calibration through the origin, these standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1660 mixture has been used to describe the detector response. The standards for these seven Aroclors should be analyzed before the analysis of any samples with hits above the RL. For example, an Aroclor 1254 standard should be analyzed before a sample with a hit of Aroclor 1254.

10.2.6 Initial Calibration Criteria

- If the **RSD for an analyte is < 20%**, then the response of the instrument for this compound is considered linear over the range and the mean calibration factor can be used to quantitate sample results.
- If the **RSD for any analyte is > 20%**, then linearity through the origin cannot be assumed. The mean response factor cannot be used for quantitation. An alternative calculation may be done by the use of **linear regression** or **quadratic regression** (minimum of six ICAL points are needed and regression must be weighted inversely proportional to concentration) as long as the correlation coefficient is **>0.990**. If both of these quantitation methods fail criteria for any compound in the initial calibration, then the system must be reevaluated and a new calibration curve must be analyzed. If quadratic regression is used for calibration, this must be noted in the laboratory narrative.
- **MCP (Massachusetts Contingency Plan) requirement**: minimum of five unique peaks must be evaluated for Aroclors 1016 and 1260.
- **MCP requirement:** If linear or non-linear regression is used, RL must to be verified by recalculating concentrations in the lowest calibration standard using the final calibration curve. Recoveries must be **70-130%.**
- **MCP requirement:** Minimum of five standards (or six if non-linear regression used) must be used.

Initial Calibration Verification

An initial calibration verification standard must be run immediately after each initial calibration, near the midpoint of the curve. The standard must be prepared using a second source that is different than the source used for the initial calibration. (Standards listed in Section 8.6). The **%D** has to be within **20%** (**15% for CT RCP**) when compared to the mean response factor from the initial calibration.

10.2.7 Retention Time Window

10.2.7.1 The retention time window used for the identification of target analytes is ± 0.07 minutes. These criteria have been adopted from the EPA CLP Statement of Work (OLM04.2). It has been found that these limits work well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the

procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results.

10.2.7.2 The windows listed above are used as guidance; however the experience of the analyst weighs heavily in the interpretation of the chromatograms. For example, it has been observed that certain oil matrices can cause the retention times to shift more dramatically.

10.3 Sample Processing

The determination of PCB Aroclors is accomplished by comparing the sample chromatogram to that of the most similar Aroclor standard. The use of PCB overlays is extremely helpful, either by using hardcopies of chromatograms or by utilizing the Enviroquant software. A choice must be made as to which Aroclor is most similar and whether that standard is truly representative of the PCB in the sample. Both retention time and pattern are important when determining PCBs in a sample.

Samples that contained weathered PCB present special analytical challenges. Weathering could alter the Aroclor pattern to the extent that different peaks have to be selected for quantitation. Samples that contained more than one Aroclor present similar problems. For these samples, the Analyst may have to consider selecting the earlier eluting peaks for the lower boiling Aroclor and selecting the later eluting peaks for the higher boiling Aroclors to minimize overlapping peaks. Minimum of 3 peaks must be chosen for each Aroclor. In these instances, the Analyst may need request the assistance of someone with more expertise in determining the presence of PCB Aroclor.

If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be needed. If instrument problems are suspected, rerun the extract on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to the extraction SOPs for the procedures to be followed in sample cleanup.

The laboratory must report the ***HIGHER*** of the two results unless obvious interference is present on of the columns.

10.4 Continuing Calibration

10.4.1 Verify calibration each **12-hours** shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than **once every twenty injections**. A bracketing CCV is not required with the use of internal standard calibration (Method 8082A 11.6.8) with the exception of samples ran under CT RCP method. For Aroclor analysis, the calibration verification standard should be a mixture of Aroclor 1016 and 1260. The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition (Method 8082A 11.6.2). However, if the one-point calibration is used for the seven other Aroclor, a calibration standard must be analyzed before the sample for any hits.

10.4.2 The response factor (for internal standard compounds) for each analyte to be quantitated must not exceed a **± 20% difference** when compared to the initial calibration curve (**± 15% for CT RCP**). The Target data processing software automatically calculates the %D for all analytes according to the formulae in

Alpha's Quality Manual. A retention time shift >30 seconds for the internal standard necessitates reanalysis of all affected samples.

10.5 Internal Standard

The use of internal standard calibration does not require that all sample results be bracketed with CCV standard. However, when internal standard calibration is used, the retention times of internal standards and the area response of internal standards should be checked for each analysis.

10.5.1 **IS in CCAL** – The measured area of the internal standard must be no more than ± 50% different from the average area calculated during initial calibration **(-50 to 150%).**

10.5.2 **IS in samples** - The measured area of the internal standard must be no more than -50% to +100% different from the area calculated from opening CCV **(-50 to 200%)**

Retention time shifts of more than 30 sec from the retention time of the most recent calibration standard are cause for concern and must be investigated.

10.6 Preventive Maintenance

10.6.1 **Preventive Maintenance:** Routine preventive maintenance is performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and replacement of injector seals.

10.6.2 **Other Maintenance:** ECD detectors may become contaminated, requiring bake out at elevated temperatures, (no greater than 375C) or repair by the manufacturer.

11. Data Evaluation, Calculations and Reporting

11.1 Quantitation of Aroclors

Per Method 8082A, quantitation is based on the use of a minimum of 3 of the major peaks present in the analyte, although the use of 5 of the major peaks is recommended. Each of these peaks is individually calibrated with a **minimum of five calibration points** based on average response factors. The %RSD must meet the criteria of ≤20% for the ICAL. The five major peaks are calculated as described below. After individual calculation meets criteria, the average of the peaks selected for quantitation is used to determine the final concentration.

11.1.1 Aqueous samples

$$\text{Concentration } (\mu g/L) = \frac{C \times DF \times Vf \times 1000}{Vo}$$

where:

C = Extract concentration (µg/mL), **NOTE**: ng on column = ng/injection volume = ng/uL = ug/mL
DF = Dilution factor
Vf = Final extract volume (mL)
Vo = Sample volume (mL)

11.1.2 Soil/sediment samples

$$\text{Concentration } (\mu g/Kg, \text{ dry weight}) = \frac{C \times DF \times Vf \times 1000}{W \text{ (gm)}} \div \%S$$

where:

C = Extract concentration (µg/mL), **NOTE**: ng on column = ng/injection volume = ng/uL = ug/mL
DF = Dilution factor
Vf = Final extract volume (mL)
W = Weight of the sample extracted (10g for high, 30g for low)
%S = Percent solids, as a decimal value

11.1.3 Reporting Results

11.1.3.1 After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews must be done by two separate individuals.

11.1.3.2 Reporting Results for PCBs in Caulk Samples

If in the screen sample Aroclor concentration as calculated above is **> 20000ppm**, the Client is contacted by a Customer Service Representative and these results are sent to the LIMS and reported to the Client.

If the sample concentration as calculated above for any Aroclor is **< 20000ppm**, the sample is sent for re-extraction by Method 3540C (Alpha SOP/1954).

11.1.3.3 Summation Rules

"TOTAL" concentrations are calculated for **ALL samples and Quality Control Samples** (i.e. LCS, MS, DUP, BLK).

TOTAL = sum of "reportable" Aroclors

Reportable- all Aroclors reported for associated project.

For dual-column analysis, Total is reported as part of column "A" data, unless all individuals are reported from "B" column. "Total" is calculated based on the associated "Report List". See Work Instruction #14335 for details.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and/or improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written into the LIMS by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

Table 1: STANDARD SOLUTIONS

Table 2: QC ACCEPTANCE CRITERIA

TABLE 1

STANDARD SOLUTIONS – Suggested Concentrations

STANDARD SOLUTIONS	Stock solution (ug/mL)	Level 1 (ug/mL)	Level 2 (ug/mL)	Level 3 (ug/mL)	Level 4 (ug/mL)	Level 5 (ug/mL)	Level 6 (ug/mL)	Spike Solution (ug/mL)	LCS Solution (ug/mL)
PCB									
Aroclor 1016/1260	100	0.1	0.5	1	2.5	5	10	12.5	12.5
Aroclors 1221, 1232, 1242, 1254, 1262, 1268	100	0.1	0.5	1	2.5	5	10		
LVI		0.01	0.05	0.1	0.25	0.5	1	2.5	2.5
Internal Standard									
1-Bromo-2-Nitrobenzene	5000	0.25	0.25	0.25	0.25	0.25	0.25		
LVI		0.025	0.025	0.025	0.025	0.025	0.025		
Surrogates:									
Tetrachloro-m-xylene	2.0	0.0064	0.032	0.064	0.16	0.32	0.64	2	2
Decachlorobiphenyl	2.0	0.0126	0.064	0.128	0.32	0.64	1.28	2	2
LVI – 10X less								0.2	0.2

LVI is spiked 10X lower

TABLE 2

QC ACCEPTANCE CRITERIA

	Aqueous, Soils	
Surrogate % Recovery	**Lower Control Limit**	**Upper Control Limit**
2,4,5,6-Tetrachloro-m-xylene	30%	150%
Decachlorobiphenyl	30%	150%

	Aqueous, Soils % Recovery		Duplicate and/or MSD	
MS/MSD and LCS	**Lower Control Limit**	**Upper Control Limit**	**Aqueous RPD**	**Soil RPD**
Aroclor 1016, 1260	40%	140%	30%	50%

Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS)

Reference Method No.: **EPA 8270 D**

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update V, February 2007.

1. Scope and Application

Matrices: This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and wastewater samples.

This method is used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone.

Table 9 lists "difficult" compounds that may require special treatment when being determined by this method.

Approval of any method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability (Section 13.2).

2. Summary of Method

The samples are introduced into the GC/MS by injecting 1µL of the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of standards run on the same GC/MS system. Quantitation is accomplished by comparing the response of quantitation ion relative to an internal standard using a calibration curve.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 6 lists our routine reporting limits.

4. Interferences

4.1 Only high purity helium is used in the GC system to eliminate this source of possible contamination. The helium (carrier gas) is certified by the gas supplier.

4.2 Preventive instrument maintenance is performed routinely. Section 10.5 details the maintenance steps.

4.3 Glassware must be scrupulously cleaned. This procedure is detailed in the Organic Extraction Glassware Cleaning & Handling SOP/1953.

4.4 Contaminated solvents or reagents are also possible sources of contamination. All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free.

4.5 Contamination by carry-over can occur whenever high-concentration and low-concentration samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered (concentrations greater than 2x the highest concentration) and the next sample has reportable hits this sample should to be re-analyzed for confirmation based on analyst discretion.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC/MS laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab for future disposal by the Health and Safety Officer. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Flammable solvent bottles must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two 1L amber glass jars with teflon-lined lids. For LVI, aqueous samples are collected in two 275mL amber glass jars with teflon-lined lids. Solid samples are collected in 250mL wide-mouth glass jars with teflon-lined lids. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of 4 ± 2ºC. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of 4 ± 2ºC.

6.3 Sample Handling

Aqueous samples must be extracted within 7 days of sample collection, solid samples within 14 days of collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph/Mass Spectrometer System:

7.1.1 **Gas Chromatograph, Hewlett Packard 6890 (or equivalent):** An analytical system complete with a temperature-programmable gas chromatograph configured for split/splitless-injection and all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source.

7.1.2 **Column:** Rxi-5Sil MS30m x 0.32mm ID, 0.25µm film thickness or column of similar configuration.

7.1.3 **Mass Spectrometer, Hewlett Packard 5973 (or equivalent):** Scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 1 when 1 µL of the GC/MS tuning standard is injected through the GC (50ng of DFTPP).

7.1.4 **Data System:** A computer system is interfaced to the Mass Spectrometer. The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software allows the analyst to search for any GC/MS data file for ions of specific mass and plot such ion abundances versus time or scan number. *HP ChemServer* software is used for data acquisition and *MSD Chemstation/Enviroquant version E.02.02* is used for data reduction.

7.2 **Syringe:** 10 µL.

7.3 **Volumetric Flasks, Class A:** Appropriate sizes with ground-glass stoppers.

7.4 **Vials:** Glass autosampler vials with polytetrafluoroethylene (PTFE)-lined crimp top caps.

8. Reagents and Standards

8.1 Stock Standard Solutions

Certified stock standard solutions, traceable to NIST, when available, are purchased from commercial vendors. They can be replaced with different standards as long as they contain all target analytes.

All stock standards, lot number, catalog number, expiration date, preparation date and initials are recorded in a logbook. Standards are stored in the refrigerator or freezer.

Stock standard expire 6 months from the date of preparation or on the earliest expiration date of any of the stock solution used to prepare it.

Please note that the following preparation instructions and stock standards are included for illustration purposes and may be modified as needed (ex. to accommodate standard availability or client requests), however final concentrations for the initial calibration levels shall always follow the example in 8.1.4.

Vendor	Standard	Catalog#	Concentration
Restek			
	8270 Mega Mix	31850	500-1000ug/mL
	Benzoic Acid Mix	31879	2000ug/mL
	Acid Surrogate Mix	31087	10000ug/mL
	B/N Surrogate Mix	31086	5000ug/mL
	Benzaldehyde Standard	33017	2000ug/mL
	Custom AP9 ICAL Standard	571813-FL	2000ug/mL
	Custom ADP Standard	572745-FL	2000ug/mL
	Benzidine Mix	31834	2000ug/mL
	SV Internal Standard Mix	31206	2000ug/mL
	1,4-Dioxane	30287	2000ug/mL
	Custom CLP 04.1 BNA Surrogate Mix	571320-FL	1000ug/mL
Absolute			
	Aromatic Amines Mix	99410	2000ug/ml
Ultra			
	Semi-Volatiles GC/MS Tuning Standard	GCM-150-1	1000ug/mL

8.1.1 ABN Stock Standard, 200ug/mL

Use 5mL of each of the following:
- Benzoic Acid Mix
- Benzidine Mix

and use 10mL of each of the following:
- 8270 Mega Mix
- Custom CLP 04.1 BNA Surrogate Mix

Bring up to 50mL volume with DCM.

8.1.2 AP9 Additional Compounds Stock Standard, 200ug/mL

Use 5mL of each of the following:
- Custom AP9 ICAL Standard
- Benzaldehyde Standard

Bring up to 50mL volume with DCM.

8.1.3 ADP Stock Standard, 200ug/ml

Use 5ml of:
Custom ADP Standard

Bring up to 50mL volume with DCM.

8.1.4 Calibration Standard

A minimum of 5 calibration standards must be included for each analyte:

Calibration Curve Levels	
Level	Concentration ug/mL
1	1.0
2	2.0
3	3.0
4	5.0
5	10
6	20
7	50
8	100
9	150
10	200

LVI Calibration Curve Levels	
Level	Concentration ug/mL
1	0.2
2	0.4
3	1.0
4	2.0
5	3.0
6	5.0
7	10
8	15
9	35
10	50

*LVI- Low Volume Initiative

8.2 Internal Standard Solution

The internal standards are:
1,4-dichlorobenzene-d_4
naphthalene-d_8
acenaphthene-d_{10}
phenanthrene-d_{10}
chrysene-d_{12}
perylene-d_{12}

Each 500µL of standards, blank and sample extracts are spiked with 10µL of SV Internal Standard Mix, resulting in a concentration of 40ng/ µL.

For the LVI method, a 1:10 dilution is made of the Internal Standard Stock Solution. 500µL of standards, blank and sample extracts are spiked with 10µL of this preparation, resulting in a concentration of 4ng/ µL.

8.3 GC/MS Tuning Standard

The tuning standard is a methylene chloride solution containing 50ng/µL of decafluorotriphenylphosphine (DFTPP). The standard also contains 50ng/µL each of 4,4'DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance.

Prepare the GC/MS Tuning Standard with 25µL GCM-150 and 475µL Dichloromethane.

8.4 Surrogate Spiking Solution

8.4.1 Extraction Surrogate Preparation

In a 1000mL volumetric flask, add 5ml of 31086 and 31087. Bring up to volume with Acetone. The final concentration is 50µg/mL for the acid surrogates and 25µg/mL for the B/N surrogates.

8.4.2 LVI Extraction Surrogate Preparation

The LVI surrogate is a 10 fold dilution of the surrogate solution prepared in 8.4.1. For example, to make 200mL of LVI surrogate, add 20mL of 8.4.1 to a 200mL volumetric flask and fill to volume with Acetone. The resulting surrogate concentration is 5µg/mL for the acid surrogates and 2.5µg/mL for the B/N surrogates.

8.5 Spike Solution (LCS, MS, MSD)

Spike Solution Preparation

ABN SPK1:
In a 500ml volumetric flask, add 20ml of 8270 Mega Mix #31850, 10ml of Benzoic Acid Mix #31879, 10ml Custom AP9 ICAL Standard #571813-FL and 10ml Benzaldehyde Standard #33017. Bring up to volume with Acetone. The final concentration is 40µg/ml.

Note: the LVI ABN SPK1 is prepared by making an 8 fold dilution of the 40µg/ml ABN SPK (in acetone), resulting in a 5µg/ml LVI ABN SPK1.

ABN SPK2:
In a 500ml volumetric flask, add 10ml Benzidine Mix #31834 and 10mL Custom ADP Standard #572945-FL. Bring up to volume with Acetone. The final concentration is 40µg/ml.

Note: the LVI ABN SPK2 is prepared by making an 8 fold dilution of the 40µg/ml ABN SPK (in acetone), resulting in a 5µg/ml LVI ABN SPK2.

8.6 Dichloromethane (DCM): Pesticide quality.

8.7 Acetone: Pesticide quality.

9. Quality Control

9.1 Blank(s)

Extraction blanks are performed with each extraction batch of 20 or less samples. The extraction blank must not contain any of the reportable analytes above the reporting limit. Corrective actions:

- No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample

- If the blank have reportable hits and re-extraction could not be performed due to lack of additional sample volume, the sample results are reported and qualified with “B” flag for any associated samples that concentration is less than 10x the blank concentration

For NJ regulatory work the method blank must have all the target analytes less than RL except for Phthalates which must be less than 5x of the RL. Sample results are qualified with

"B" flag for analytes observed in the blank greater than RL and the Phthalates observed in the blank greater than 5x RL

The surrogate recoveries must also be within the acceptance criteria listed in Table 2. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary.

9.2 Laboratory Control Sample and Laboratory Control Sample Duplicate (LCS / LCSD)

A Laboratory Control Sample/Laboratory Control Sample Duplicate pair (LCS/LCSD) are extracted and analyzed with each analytical batch of 20 or fewer samples.

The LCS/LCSD acceptance criteria are based on in-house control limits. Less than 10% of total compounds may be outside of control limits provided that recoveries are >10%. Note: this does not apply to difficult analytes listed in Table 9 which may be accepted at recoveries <10. If >10% of analytes are recovered above control limits, this is deemed acceptable as long as the analytes in question are not detected in associated samples.

If these criteria are not met, the entire batch is re-extracted. If re-extraction is not possible, due to insufficient sample or holding time exceedance, the analyst must write up the failure on a narrative sheet for inclusion in the client report.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.7.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike and Matrix Spike Duplicate (MS / MSD)

A matrix spike/matrix spike duplicate pair is extracted and analyzed for each batch of 20 or fewer samples per client request. The MS/MSD acceptance criteria are based on in-house control limits. If the recovery criteria are not met, but are met in the LCS/LCSD, this is noted on a narrative sheet for inclusion in the client report.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

All extracted samples and associated QC are spiked with surrogates. The acceptable surrogate recovery limits are listed in Table 2.

Corrective action: Up to one surrogate can be out in each fraction (Acid and Base/Neutral) but not less than 10% recovery, before any corrective action is necessary. Otherwise, analysis must be repeated once to see if an analytical error has occurred. If the % recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

Re-extraction is not required if surrogate recoveries are high and target analytes are not detected in the sample.

9.7.2 Internal Standards

If the area for any of the internal standards in the samples changes by a factor of two (-50% to +100%) from that in the CCV, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

9.8 Method Sequence

In a 12-hour period, the typical analytical sequence is:

- Degradation Check
- DFTPP
- Continuing or Daily Standards (1 – 3)*
  - (1) ABN 50 ppm
  - (2) AP9 50 ppm
  - (3) ADP 50 ppm
- Method Blank
- Samples
- QC (as required)

***Additional Continuing standards may be run at the analyst's discretion or by client request.**

10. Procedure

10.1 Equipment Set-up

10.1.1 GC/MS Operating Conditions:

Typical GC/MS operating conditions are listed below, but may be altered as long as method performance criteria are met.

Mass range:	35 – 500 amu
Scan time:	3.15 second / scan
Initial temperature:	50°C, hold for 1.5 minutes
Temperature program:	28°C/minute to 250°C then 9°C/minute to 320°C
Final temperature:	320°C for 1.50 min
Injector temperature:	300°C
Transfer line temperature:	280°C
Source temperature:	230ºC
Injector:	split ratio 5:1; 11.7mL/min
Injection volume:	1µL
Carrier gas:	helium at 523 cm/second (2.0 mL/min) constant flow

GC/MS Operating Conditions for LVI method:

Mass range:	35 – 550 amu
Scan time:	3.15 second / scan
Initial temperature:	45°C, hold for 4 minutes
Temperature program:	25°C/minute to 250°C then 20°C/minute to 320°C
Final temperature:	320°C for 4.3 min
Injector temperature:	270°C
Transfer line temperature:	280°C
Source temperature:	320ºC
Injector:	split ratio 5:1; 8.57mL/min

Injection volume:	2µL
Carrier gas:	helium at 1.7148mL/min) constant flow

10.1.2 GC/MS Tune:

At the beginning of every 12 hour sequence, analyze DFTPP tuning solution (Section 8.3).

The resultant mass spectrum for DFTPP must meet the criteria given in Table 1 before sample analysis begins. The mass spectrum of DFTPP should be acquired in the following manner:

(1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.
(2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of DFTPP.

The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD must not exceed 20%. Benzidine and pentachlorophenol must be present at their normal responses and no peak tailing must be visible.

The tailing factor for benzidine and pentachlorophenol must be calculated in every DFTPP run. (See Table 4)

If degradation is excessive and/or poor chromatography is noted, the system needs maintenance (see Section 10.5).

10.2 Initial Calibration

10.2.1 Prepare calibration standards for all target analytes at a minimum of five concentration levels as specified in Section 8.1.4.

10.2.2 Add 10µL of Internal Standard to each calibration standard directly into the autosampler vial containing 500µL of standard. Analyze each calibration standard under the conditions specified in Section 10.1.1.

10.2.3 Record the calibration standard, unique lab identifier code (lot), concentration, and analyst's initials in the analytical sequence list.

10.2.4 In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formulae listed in Alpha's Quality Manual.

It is recommended that a minimum response factor for the most common target analytes, as noted in Table 8, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity.

10.2.5 Initial Calibration %RSD Criteria:

For all analytes, the RSD must be ≤ 20% for the mean response factor to be used for sample quantitation.

For RCP, the RSD must be ≤ 15% for the mean response factor to be used for sample quantitation.

An alternate calculation fits may be performed provided that the minimum correlation coefficient ≥ 0.99 is met.

When linear regression model is used a minimum quantitation check of the lowest calibration point is performed. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration.

10.2.6 Evaluation of Retention Times:

The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units.

10.2.7 Initial Calibration Verification (Second Source Verification)

10.2.7.1 The initial calibration (Section 10.2) for each compound of interest must be verified prior to sample analysis. This is accomplished by analyzing second source calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

10.2.7.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

If the % Difference for each analyte is ± 30%, then the calibration is assumed to be valid. If this criterion is not met, then corrective action must be taken prior to the analysis.

For RCP, if the % Difference for each analyte is ± 20%, then the calibration is assumed to be valid. If this criterion is not met, then corrective action must be taken prior to the analysis.

10.2.7.3 In cases where compounds fail (greater than 30% difference), they may still be reported as non-detects.

10.3 Equipment Operation and Sample Processing

GC/MS Analysis of Samples

10.3.1.1 Allow the sample extracts to warm to room temperature.

10.3.1.2 Transfer all of the sample extract to a 1.5mL vial. Remove 500µL of sample extract to another vial, and add 10µL of the internal standard solution (Section 8.2).

10.3.1.3 The autosampler is programmed to inject 1µL aliquot of the sample extract into the GC/MS system, using the same instrument conditions that were used for calibration. The injection volume of the sample must be the same as the volume used for the calibration standard.

10.3.1.4 If the response of any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed.

10.3.2 Qualitative Identification

Perform first level data review. Obtain the primary m/z (Table 5) masses for each parameter of interest. The following criteria must be met to make qualitative identification:

- Compare the background subtracted mass spectra for the sample to the reference spectra. The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.
- The retention time must fall within ± 0.1 minutes of the retention time of the compound in the analytical standard. However, analyst experience must be used in making the qualitative identification.
- The relative peak height of the one characteristic mass must fall within 30% of the relative intensity of the mass in a reference mass spectrum. The reference spectrum is obtained from a standard analyzed on the GC/MS system.

Structural isomers that have very similar mass spectra are identified only if the resolution between authentic isomers in a standard mix is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

10.4 Continuing Calibration

10.4.1 The initial calibration (Section 10.2) for each compound of interest must be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

10.4.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

If the % Difference for each CCV analyte is $\leq$ 20%, then the calibration is assumed to be valid. If the criterion is not met for more than 20% of the compounds then corrective action must be taken.

Due to the large number of analytes present, allowances may be made for a RF that drifts out high, as long as there are no positive hits for that particular analyte in any of the associated samples.

10.4.3 If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.4.4 If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration must be performed.

10.4.5 Internal Standard Retention Time

The retention times of the internal standards in the calibration verification standard is evaluated after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard of the most recent initial calibration, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.4.6 Internal Standard Response

Refer to section 9.7.2

10.5 Preventive Maintenance

When poor sensitivity is observed, replacement of the injector liner and seal may solve the problem. If not, clip approximately 3 – 6 inches from the injector end of the GC column. If the sensitivity does not improve it may be necessary to replace the split line or the injector weldment assembly. If the problem persists, it may be necessary to replace the GC column.

Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

11. Data Evaluation, Calculations and Reporting

When a parameter is identified, the quantitation of that parameter must be based on the integrated abundance of the quantitation characteristic m/z given in Table 5

Calculate the concentration in the sample using the average response factor (RF) from the initial calibration curve according to the formula in Alpha's Quality Manual.

After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews must be done by two separate individuals.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 9 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

Alpha SOP/1732 DL/LOD/LOQ Generation

Alpha SOP/1739 IDC/DOC Generation

Alpha SOP/1729 Waste Management and Disposal SOP

16. Attachments

Table 1: DFTPP Key Ions and Ion Abundance Criteria

Table 2: Acceptable Surrogate Spike Recovery Limits

Table 3A: Acceptable Aqueous QC Limits

Table 3B: Acceptable Soil QC Limits

Table 4: Tailing Factor Calculation

Table 5: Characteristic Ions for Semivolatile Compounds

Table 6: Reported Detection Limits

Table 7: Semivolatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Table 8: Recommended Minimum Response Factor Criteria

Table 9: Difficult analytes

TABLE 1

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	10-80% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	10-80% of mass 198
197	< 2% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-60% of mass 198
365	> 1% of mass 198
441	Present but less than 24% mass 442
442	> 50% of mass 198
443	15-24% of mass 442

TABLE 2

ACCEPTABLE SURROGATE SPIKE RECOVERY LIMITS

Analytical Fraction	Surrogate Compound	Water	Soil/Sediment
BN-8270D	Nitrobenzene-d_5	23-120%	23-120%
BN-8270D	2-Fluorobiphenyl	15-120%	30-120%
BN-8270D	p-Terphenyl-d_{14}	41-149%	18-120%
Acid-8270D	Phenol-d_6	10-120%	10-120%
Acid-8270D	2-Fluorophenol	21-120%	25-120%
Acid-8270D	2,4,6-Tribromophenol	10-120%	10-136%

It is allowable for one surrogate from each fraction be outside acceptance criteria, provided a minimum recovery of 10% has been achieved.

TABLE 3A

ACCEPTABLE AQUEOUS QC LIMITS

	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)		CT TARGET COMPOUND LIST (Aqueous)	
Analyte	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD
1,2,4,5-Tetrachlorobenzene			70-130	20	40-140	20
1,2,4-Trichlorobenzene	39-98	30	70-130	20	40-140	20
1,2-Dichlorobenzene	40-140	30	70-130	20		
1,3-Dichlorobenzene	40-140	30	70-130	20		
1,3-Dinitrobenzene	15-130	30				
1,4-Dichlorobenzene	36-97	30	70-130	20		
1-Methylnaphthalene	41-103	30				
2,3,4,6-Tetrachlorophenol			70-130	20		
2,4,5-Trichlorophenol	30-130	30	70-130	20	30-130	20
2,4,6-Trichlorophenol	30-130	30	70-130	20	30-130	20
2,4-Dichlorophenol	30-130	30	70-130	20	30-130	20
2,4-Dimethylphenol	30-130	30	70-130	20	30-130	20
2,4- Dimethylaniline	40-140	30	70-130	20		
3,4- Dimethylaniline	40-140	30	70-130	20		
2,3- Dimethylaniline	40-140	30	70-130	20		
2,4,5-Dimethylaniline	40-140	30	70-130	20		
4-Chlorotoluidine	40-140	30	70-130	20		
2-Ethylaniline	40-140	30	70-130	20		
O-toluidine	40-140	30	70-130	20		
2-Napthylamine	40-140	30	70-130	20		
2,4-Dinitrophenol	20-130	30	20-130	20	30-130	20
2,4-Dinitrotoluene	24-96	30	70-130	20	40-140	20
2,6-Dinitrotoluene	40-140	30	70-130	20	40-140	20
2-Chloronaphthalene	40-140	30	70-130	20	40-140	20
2-Chlorophenol	27-123	30	70-130	20	30-130	20
2-Methylnaphthalene	40-140	30	70-130	20	40-140	20
2-Methylphenol	30-130	30	70-130	20	30-130	20
2-Nitroaniline	52-143	30	70-130	20	40-140	20
2-Nitrophenol	30-130	30	70-130	20	30-130	20
3,3'-Dichlorobenzidine	40-140	30	70-130	20	40-140	20
3,3'-Dimethylbenzidine			20-160	20		
3-Methylphenol/4-Methylphenol	30-130	30	20-160	20	30-130	20
3-Nitroaniline	25-145	30	70-130	20	40-140	20
4,6-Dinitro-o-cresol	20-164	30	70-130	20	30-130	20
4-Bromophenyl phenyl ether	40-140	30	70-130	20	40-140	20
4-Chloroaniline	40-140	30	20-160	20	40-140	20
4-Chlorophenyl phenyl ether	40-140	30	70-130	20	40-140	20
4-Nitroaniline	51-143	30	70-130	20	40-140	20
4-Nitrophenol	10-80	30	20-160	20	30-130	20
Acenaphthene	37-111	30	70-130	20	40-140	20
Acenaphthylene	45-123	30	70-130	20	40-140	20

	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)		CT TARGET COMPOUND LIST (Aqueous)	
Analyte	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD
Acetophenone	39-129	30	70-130	20		
Aniline	40-140	30	20-160	20	40-140	20
Anthracene	40-140	30	70-130	20	40-140	20
Atrazine			70-130	20		
Azobenzene	40-140	30	70-130	20		
Benzaldehyde			20-160	20		
Benzidine	10-75	30	20-160	20		
Benzo(a)anthracene	40-140	30	70-130	20	40-140	20
Benzo(a)pyrene	40-140	30	70-130	20	40-140	20
Benzo(b)fluoranthene	40-140	30	70-130	20	40-140	20
Benzo(ghi)perylene	40-140	30	70-130	20	40-140	20
Benzo(k)fluoranthene	40-140	30	70-130	20	40-140	20
Benzoic Acid	10-164	30	20-160	20		
Benzyl Alcohol	26-116	30	20-160	20		
Biphenyl	40-140	30	70-130	20		
Bis(2-chloroethoxy)methane	40-140	30	70-130	20	40-140	20
Bis(2-chloroethyl)ether	40-140	30	70-130	20	40-140	20
Bis(2-chloroisopropyl)ether	40-140	30	70-130	20	40-140	20
Bis(2-Ethylhexyl)phthalate	40-140	30	70-130	20	40-140	20
Butyl benzyl phthalate	40-140	30	70-130	20	40-140	20
Caprolactam			20-160	20		
Carbazole	55-144	30	70-130	20	40-140	20
Chrysene	40-140	30	70-130	20	40-140	20
Dibenzo(a,h)anthracene	40-140	30	70-130	20	40-140	20
Dibenzofuran	40-140	30	70-130	20	40-140	20
Diethyl phthalate	40-140	30	70-130	20	40-140	20
Dimethyl phthalate	40-140	30	70-130	20	40-140	20
Di-n-butylphthalate	40-140	30	70-130	20	40-140	20
Di-n-octylphthalate	40-140	30	70-130	20	40-140	20
Fluoranthene	40-140	30	70-130	20	40-140	20
Fluorene	40-140	30	70-130	20	40-140	20
Hexachlorobenzene	40-140	30	70-130	20	40-140	20
Hexachlorobutadiene	40-140	30	70-130	20	40-140	20
Hexachlorocyclopentadiene	40-140	30	20-160	20	40-140	20
Hexachloroethane	40-140	30	20-160	20	40-140	20
Indeno(1,2,3-cd)Pyrene	40-140	30	70-130	20	40-140	20
Isophorone	40-140	30	70-130	20	40-140	20
Naphthalene	40-140	30	70-130	20	40-140	20
Nitrobenzene	40-140	30	70-130	20	40-140	20
NitrosoDiPhenylAmine(NDPA)/ Diphenylamine (DPA)	40-140	30	70-130	20	40-140	20
n-Nitrosodimethylamine	22-74	30	20-160	20		
n-Nitrosodi-n-propylamine	29-132	30	70-130	20	40-140	20
P-Chloro-M-Cresol	23-97	30	70-130	20	30-130	20
Pentachlorophenol	9-103	30	20-160	20	30-130	20
Pentachloronitrobenzene					40-140	20

	STANDARD TARGET COMPOUND LIST (Aqueous)			NEW JERSEY TARGET COMPOUND LIST (Aqueous)		CT TARGET COMPOUND LIST (Aqueous)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**		**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
Phenanthrene	40-140	30		70-130	20	40-140	20
Phenol	12-110	30		20-160	20	30-130	20
Pyrene	26-127	30		70-130	20	40-140	20
Pyridine	10-66	30				40-140	20
2-Fluorophenol	*21-120*			*15-110*		*15-110*	
Phenol-d6	*10-120*			*15-110*		*15-110*	
Nitrobenzene-d5	*23-120*			*30-130*		*30-130*	
2-Fluorobiphenyl	*15-120*			*30-130*		*30-130*	
2,4,6-Tribromophenol	*10-120*			*15-110*		*15-110*	
4-Terphenyl-d14	*41-149*			*30-130*		*30-130*	

TABLE 3B

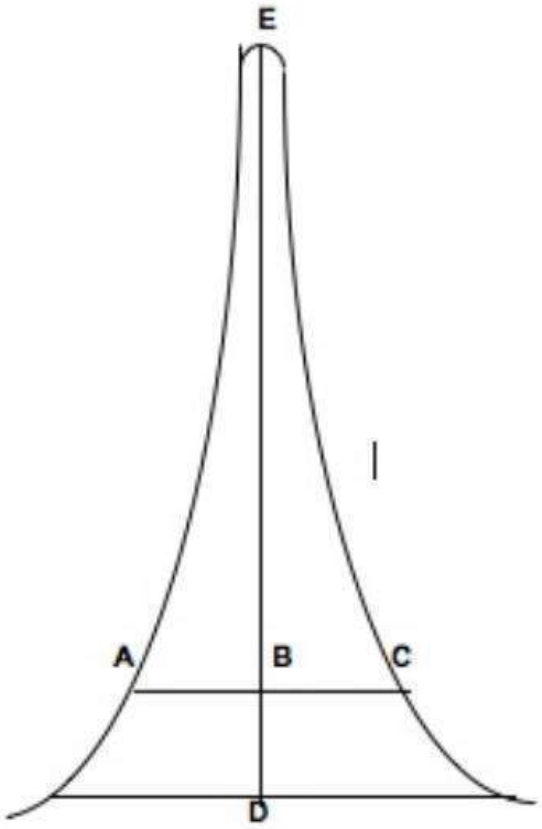
ACCEPTABLE SOIL QC LIMITS

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)		CT TARGET COMPOUND LIST (Soil)	
Analyte	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD
1,2,4,5-Tetrachlorobenzene	40-117	50	70-130	30	40-140	30
1,2,4-Trichlorobenzene	38-107	50	70-130	30	40-140	30
1,2-Dichlorobenzene	40-140	50	70-130	30		
1,3-Dichlorobenzene	40-140	50	70-130	30		
1,3-Dinitrobenzene	40-140	50				
1,4-Dichlorobenzene	28-104	50	70-130	30		
1-Methylnaphthalene	26-130	50				
2,3,4,6-Tetrachlorophenol	40-140	50	70-130	30		
2,4,5-Trichlorophenol	30-130	50	70-130	30	30-130	30
2,4,6-Trichlorophenol	30-130	50	70-130	30	30-130	30
2,4-Dichlorophenol	30-130	50	70-130	30	30-130	30
2,4-Dimethylphenol	30-130	50	70-130	30	30-130	30
2,4-Dinitrophenol	4-130	50	20-160	30	30-130	30
2,4-Dinitrotoluene	28-89	50	70-130	30	40-140	30
2,6-Dinitrotoluene	40-140	50	70-130	30	40-140	30
2-Chloroaniline	30-130	50				
2-Chloronaphthalene	40-140	50	70-130	30	40-140	30
2-Chlorophenol	25-102	50	70-130	30	30-130	30
2-Methylnaphthalene	40-140	50	70-130	30	40-140	30
2-Methylphenol	30-130.	50	70-130	30	30-130	30
2-Nitroaniline	47-134	50	70-130	30	40-140	30
2-Nitrophenol	30-130	50	70-130	30	30-130	30
3,3'-Dichlorobenzidine	40-140	50	70-130	30	40-140	30
3,3'-Dimethylbenzidine	15-115	50				
3-Methylphenol/4-Methylphenol	30-130	50	20-160	30	30-130	30
3-Nitroaniline	26-129	50	70-130	30	40-140	30
4,6-Dinitro-o-cresol	10-130	50	70-130	30	30-130	30
4-Bromophenyl phenyl ether	40-140	50	70-130	30	40-140	30
4-Chloroaniline	40-140	50	20-160	30	40-140	30
4-Chlorophenyl phenyl ether	40-140	50	70-130	30	40-140	30
4-Nitroaniline	41-125	50	70-130	30	40-140	30
4-Nitrophenol	11-114	50	20-160	30	30-130	30
Acenaphthene	31-137	50	70-130	30	40-140	30

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)		CT TARGET COMPOUND LIST (Soil)	
Analyte	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD
Acenaphthylene	40-140	50	70-130	30	40-140	30
Acetophenone	14-144	50	70-130	30	40-140	30
Aniline	40-140	50	20-160	30	40-140	30
Anthracene	40-140	50	70-130	30	40-140	30
Atrazine	40-140	50	70-130	30		
Azobenzene	40-140	50	70-130	30		
Benzaldehyde	40-140	50	20-160	30		
Benzidine	10-66	50	20-160	30		
Benzo(a)anthracene	40-140	50	70-130	30	40-140	30
Benzo(a)pyrene	40-140	50	70-130	30	40-140	30
Benzo(b)fluoranthene	40-140	50	70-130	30	40-140	30
Benzo(e)pyrene	40-140	50				
Benzo(ghi)perylene	40-140	50	70-130	30	40-140	30
Benzo(k)fluoranthene	40-140	50	70-130	30	40-140	30
Benzoic Acid	10-110	50	20-160	30		
Benzyl Alcohol	40-140	50	20-160	30		
Biphenyl	37-127	50	70-130	30		
Bis(2-chloroethoxy)methane	40-117	50	70-130	30	40-140	30
Bis(2-chloroethyl)ether	40-140	50	70-130	30	40-140	30
Bis(2-chloroisopropyl)ether	40-140	50	70-130	30	40-140	30
Bis(2-Ethylhexyl)phthalate	40-140	50	70-130	30	40-140	30
Butyl benzyl phthalate	40-140	50	70-130	30	40-140	30
Caprolactam	15-130	50	20-160	30		
Carbazole	54-128	50	70-130	30	40-140	30
Chrysene	40-140	50	70-130	30	40-140	30
Dibenzo(a,h)anthracene	40-140	50	70-130	30	40-140	30
Dibenzofuran	40-140	50	70-130	30	40-140	30
Diethyl phthalate	40-140	50	70-130	30	40-140	30
Dimethyl phthalate	40-140	50	70-130	30	40-140	30
Di-n-butylphthalate	40-140	50	70-130	30	40-140	30
Di-n-octylphthalate	40-140	50	70-130	30	40-140	30
Diphenamid	40-140	50				
Fluoranthene	40-140	50	70-130	30	40-140	30
Fluorene	40-140	50	70-130	30	40-140	30
Hexachlorobenzene	40-140	50	70-130	30	40-140	30
Hexachlorobutadiene	40-140	50	70-130	30	40-140	30
Hexachlorocyclopentadiene	40-140	50	20-160	30	40-140	30
Hexachloroethane	40-140	50	20-160	30	40-140	30
Indeno(1,2,3-cd)Pyrene	40-140	50	70-130	30	40-140	30
Isophorone	40-140	50	70-130	30	40-140	30

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)		CT TARGET COMPOUND LIST (Soil)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
Naphthalene	40-140	50	70-130	30	40-140	30
Nitrobenzene	40-140	50	70-130	30	40-140	30
NitrosoDiPhenylAmine(NDPA)/ Diphenylamine (DPA)	36-157	50	70-130	30	40-140	30
n-Nitrosodimethylamine	22-100	50	20-160	30		
n-Nitrosodi-n-propylamine	32-121	50	70-130	30	40-140	30
Parathion, ethyl	40-140	50	20-160	30		
P-Chloro-M-Cresol	26-103	50	70-130	30	30-130	30
Pentachloronitrobenzene	42-153	50			40-140	30
Pentachlorophenol	17-109	50	20-160	30	30-130	30
Phenanthrene	40-140	50	70-130	30	40-140	30
Phenol	26-90	50	20-160	30	30-130	30
Pyrene	35-142	50	70-130	30	40-140	30
Pyridine	10-93	50	20-160	30	40-140	30
Thionazin	40-140	50				
2-Fluorophenol	*25-120*		*30-130*		*30-130*	
Phenol-d6	*10-120*		*30-130*		*30-130*	
Nitrobenzene-d5	*23-120*		*30-130*		*30-130*	
2-Fluorobiphenyl	*30-120*		*30-130*		*30-130*	
2,4,6-Tribromophenol	*10-136*		*30-130*		*30-130*	
4-Terphenyl-d14	*18-120*		*30-130*		*30-130*	

TABLE 4 – Tailing Factor Calculation



Tailing Factor = $\frac{BC}{AB}$

Example calculation:

Peak Height = DE = 100mm
10% Peak Height = BD = 10mm
Peak Width at 10% Peak Height = AC = 23mm

AB = 11mm
BC = 12mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

Tailing factor for benzidine < 2.0

Tailing factor for pentachlorophenol <2.0

TABLE 5

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
Acenaphthene	154	153, 152
Acenaphthylene	152	151, 153
Acetophenone	105	71, 51, 120
Aniline	93	66, 65
Anthracene	178	176, 179
Atrazine	200	202, 215
Azobenzene	77	182, 105
Benzaldehyde	105	77
Benzidine	184	92, 185
Benzo(a)anthracene	228	229, 226
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Benzoic acid	105	122, 77
Benzyl alcohol	79	77,108
Biphenyl	154	153,152
Bis (2-chloroethoxy) methane	93	95, 123
Bis (2-chloroethyl) ether	93	63, 95
Bis (2-chloroisopropyl) ether	45	77, 121
Bis (2-ethylhexyl) phthalate	149	167, 279
4-Bromophenyl phenyl ether	248	250, 141
Butyl Benzyl phthalate	149	91, 206
Caprolactam	55	85, 113
Carbazole	167	168, 166
4-Chloro-3-methylphenol	107	144, 142
2-Chloroaniline	127	129, 65
3-Chloroaniline	65	127, 129
4-Chloroaniline	65	127,129
2-Chloronaphthalene	162	127, 164
4-Chlorophenyl phenyl ether	204	206, 141
2-Chlorophenol	128	64,130
Chrysene	228	226, 229
Dibenzo(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
1,2-Dichlorobenzene	146	148, 111

1,3-Dichlorobenzene	**146**	**148, 111**
1,4-Dichlorobenzene	**146**	**148, 111**
3,3'-Dichlorobenzidine	**252**	**254, 126**
2,4-Dichlorophenol	**162**	**164, 98**
Diethyl phthalate	**149**	**177, 150**

TABLE 5 (continued)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
3,3-Dimethylbenzidine	**212**	**211, 213**
Dimethyl phthalate	**163**	**194, 164**
2,4-Dimethylphenol	**107**	**121,122**
Di-n-butyl phthalate	**149**	**150, 104**
Di-n-octyl phthalate	**149**	**167, 43**
4,6-Dinitro-2-methylphenol	**198**	**51, 105**
O-Toluidine	**106**	**107, 77**
2-Ethylaniline	**106**	**121, 77**
2,4-Dimethylaniline	**121**	**120, 106**
2,3-Dimethylaniline	**106**	**121, 120**
3,4- Dimethylaniline	**121**	**120,106**
2,4,5-Trimethylaniline	**120**	**135, 134**
4-Chlorotoluidine	**106**	**141, 140**
2-Napthylamine	**143**	**115, 116**
2,4-Dinitrophenol	**184**	**107,91**
2,4-Dinitrotoluene	**165**	**63, 89**
2,6-Dinitrotoluene	**165**	**63, 89**
Diphenamide	**167**	**72, 165**
1,4-Dioxane	**88**	**58,43**
Ethyl parathion	**109**	**97, 291**
Fluoranthene	**202**	**101, 203**
Fluorene	**166**	**165, 167**
Hexachlorobenzene	**284**	**142, 249**
Hexachlorobutadiene	**225**	**223, 227**
Hexachlorocyclopentadiene	**237**	**235, 272**
Hexachloroethane	**117**	**201, 199**
Indeno(1,2,3-cd)pyrene	**276**	**138, 227**
Isophorone	**82**	**95, 138**
1-Methylnaphthalene	**115**	**141, 142**
2-Methylnaphthalene	**142**	**141**
2-Methylphenol	**108**	**107,90**
3/4-Methylphenol	**108**	**107,90**

Naphthalene	**128**	**129, 127**
2-Nitroaniline	**65**	**92, 138**
3-Nitroaniline	**138**	**92,65**
4-Nitroaniline	**138**	**65, 108, 92, 80, 39**
Nitrobenzene	**77**	**123, 65**

TABLE 5 (continued)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
2-Nitrophenol	**139**	**109, 65**
4-Nitrophenol	**65**	**109, 139**
n-Nitrosodimethylamine	**74**	**42,44**
n-Nitrosodi-n-butylamine	**84**	**57, 41, 116, 158**
n-Nitrosodi-n-propylamine	**70**	**42, 101, 130**
n-Nitrosodiphenylamine/Diphenylamine	**169**	**168, 167**
Pentachlorobenzene	**250**	**252, 108, 248, 215, 254**
Pentachloronitrobenzene	**237**	**142, 214, 249, 295, 265**
Pentachlorophenol	**266**	**264, 268**
Phenanthrene	**178**	**179, 176**
Phenol	**94**	**65, 66**
Pyrene	**202**	**200, 203**
Pyridine	**79**	**52**
1,2,4,5-Tetrachlorobenzene	**216**	**214, 179, 108, 143, 218**
2,3,4,6-Tetrachlorophenol	**232**	**131, 230, 166, 234, 168**
m-Toluidine	**106**	**107, 79**
1,2,4-Trichlorobenzene	**180**	**182, 145**
2,4,5-Trichlorophenol	**196**	**200,198**
2,4,6-Trichlorophenol	**196**	**198, 200**
Acenaphthene-d_{10} (IS)	**164**	**162, 160**
Chrysene-d_{12} (IS)	**240**	**120, 236**
1,4-Dichlorobenzene-d_4 (IS)	**152**	**150, 115**
Naphthalene-d_8 (IS)	**136**	**68**
Perylene-d_{12} (IS)	**264**	**260, 265**
Phenanthrene-d_{10} (IS)	**188**	**94, 80**
2-Fluorobiphenyl (Surrogate)	**172**	**171**
2-Fluoroophenol (Surrogate)	**112**	**64**
Nitrobenzene-d_5 (Surrogate)	**82**	**128, 54**
Phenol-d_6 (Surrogate)	**99**	**42, 71**
Terphenyl-d_{14} (Surrogate)	**244**	**122, 212**
2,4,6-Tribromophenol (Surrogate)	**330**	**62,141**

TABLE 6

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
Acenaphthene	2	133.34
Acenaphthylene	2	133.34
Acetophenone	5	333.34
Aniline	2	133.34
Anthracene	2	133.34
Atrazine	10	666.67
Azobenzene	2	500
Benzaldehyde	5	333.34
Benzidine	20	1333.34
Benzo(a)anthracene	2	133.34
Benzo(b)fluoranthene	2	133.34
Benzo(k)fluoranthene	2	133.34
Benzo(ghi)perylene	2	133.34
Benzo(a)pyrene	2	133.34
Benzoic acid	50.0	3333.34
Benzyl alcohol	2	133.34
Biphenyl	2	366.67
Bis(2-chloroethyl)ether	2	133.34
Bis(2-chloroisopropyl)ether	2	133.34
Bis(2-chloroethoxy)methane	5.0	333.34
Bis(2-ethylhexyl)phthalate	3	200
4-Bromophenyl phenyl ether	2	133.34
Butyl benzyl phthalate	5.0	333.34
Caprolactam	10	666.67
Carbazole	2	166.67
2-Chloroaniline	2	na
3-Chloroaniline	10	na
4-Chloroaniline	5	333.34
p-Chloro-m-cresol (4-chloro-3-cresol)	2	133.34
2-Chloronaphthalene	2	133.34
2-Chlorophenol	2	133.34
4-Chlorophenyl phenyl ether	2	133.34
Chrysene	2	133.34
m/p-Methylphenol (3/4-methylphenol)	5.0	333.34
o-Methylphenol (2-methylphenol)	5.0	333.34

Dibenzo(a,h)anthracene	2	133.34
Dibenzofuran	2	133.34
Di-n-butylphthalate	5.0	333.34
1,2-Dichlorobenzene	2	133.34

TABLE 6 (continued)

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
1,3-Dichlorobenzene	2	133.34
1,3-Dinitrobenzene	2	N/A
1,4-Dichlorobenzene	2	133.34
3,3-Dichlorobenzidine	5	333.34
2,4-Dichlorophenol	5	333.34
O-Toluidine	2	N/A
2-Ethylaniline	2	N/A
2,4-Dimethylaniline	2	N/A
2,3-Dimethylaniline	2	N/A
3,4-Dimetylaniline	2	N/A
2,4,5-Trimethylaniline	2	N/A
4-Chlorotoluidine	2	N/A
2-Napthylamine	2	N/A
2,6-Dichlorophenol	10.0	666.67
Diethyl phthalate	5.0	333.34
3,3-Dimethylbenzidine	4	500
2,4-Dimethylphenol	5	333.34
Dimethyl phthalate	5.0	333.34
4,6-Dinitro-o-cresol	10	666.67
2,4-Dinitrophenol	20	1333.4
2,4-Dinitrotoluene	5.0	333.34
2,6-Dinitrotoluene	5.0	333.34
Di-n-octylphthalate	5.0	333.34
Diphenamide	5	N/A
1,4-Dioxane	5	166.67
Ethyl Parathion	N/A	166.67
Fluoranthene	2	133.34
Fluorene	2	133.34
Hexachlorobenzene	2	133.34
Hexachlorobutadiene	2	133.34
Hexachlorocyclopentadiene	20	1333.34
Hexachloroethane	2	133.34
Indeno(1,2,3-cd)pyrene	2	133.34
Isophorone	5.0	333.34

1-Methylnaphthalene	2	166.67
2-Methylnaphthalene	2	133.34
Naphthalene	2	133.34
2-Nitroaniline	5.0	333.34

TABLE 6 (continued)

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
3-Nitroaniline	5.0	333.34
4-Nitroaniline	5.0	333.34
Nitrobenzene	2	133.34
2-Nitrophenol	10.0	666.67
4-Nitrophenol	10.0	666.67
Nitrosodi-n-butylamine	10.0	666.67
n-Nitrosodimethylamine	2	133.34
n-Nitrosodiphenylamine/Diphenylamine	2	133.34
Nitrosodipiperidine	20.0	2000
n-Nitrosodi-n-propylamine	5.0	333.34
Pentachlorobenzene	20.0	1333.34
Pentachloronitrobenzene	10.0	150
Pentachlorophenol	10.0	666.67
Phenanthrene	2	133.34
Phenol	5.0	333.34
Pyrene	2	133.34
Piridine	5	666.67
1,2,4,5-Tetrachlorobenzene	10	666.67
1,2,4-Trichlorobenzene	5.0	333.34
2,4,5-Trichlorophenol	5.0	333.34
2,4,6-Trichlorophenol	5.0	333.34
2,3,4,6-Tetrachlorophenol	5.0	166.66
m-Toluidine	5	300

* **Note**: Reporting Limits are based on standard 8270 reporting list. RLs may vary for other reporting lists.

Table 7
Semivolatile Internal Standards with Corresponding
Target Compounds and Surrogates Assigned for Quantitation

1,4-dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
O-Toluidine	2-Ethylaniline	2-Naphthylamine	3,3-Dimethylbenzidine	3,3'-Dichlorobenzidine	Benzo(g,h,i)perylene
1,2,4-Trichlorobenzene	2,4-Dimethylaniline	2,3,4,6-Tetrachlorophenol	Anthracene	Benzo(a)Anthracene	Dibenzo(a,h)anthracene
1,2-Dichlorobenzene	3,4-Dimethylaniline	2,3,5,6-Tetrachlorophenol	Benzidine	Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene
1,3-Dichlorobenzene	2,3-Dimethylaniline	2,4,6-Tribromophenol, surr	Benzyl butyl phthalate	Benzo(b)fluoranthene	
1,4-Dichlorobenezne	2,4,5-Trimethylaniline	2,4-Dinitrophenol	Carbazole	Benzo(k)fluoranthene	
2,4-Dichlorophenol	4-Chlorotoludine	2,4-Dinitrotoluene	Di-n-Butylphthalate	Bis(2-ethylhexyl) phthalate	
2,4-Dimethylphenol	1,2,4,5-Tetrachlorobenzene	3-Nitroaniline	Diphenamid	Chrysene	
2-Chloroaniline	1,2-Dichlorobenzene	4,6-Dinitro-2-methylphenol	Fluoranthene	Di-n-octylphthalate	
2-Chlorophenol	1,3-Dichlorobenzene	4-Bromophenyl-phenyl ether	n-Octadecane		
2-Fluorophenol, surr	1,4-Diclorobenzene	4-Chlorophenyl-phenyl ether	Parathion		
2-Methylphenol	1-chloror-2-nitrobenzene	4-Nitroaniline	Phenanthrene		
2-Nitrophenol	1-Methylnapthalene	4-Nitrophenol	Pyrene		
3-Methylphenol / 4-Methylphenol	2,4,5-Trichlorphenol	Acenaphthene	Terphenyl-d14, surr		
Acetophenone	2,4,6-Trichlorophenol	Atrazine			
Aniline	2,6-Dichlorophenol	Azobenzene			
Benzaldehyde	2,6-Dinitrotoluene	Dibenzofuran			
Benzyl Alcohol	2-Chloronaphthalene	Dichloran			
Bis(2-chloroethoxy)methane	2-Fluorobiphenyl, surr	Diethyl phthalate			
Bis(2-chloroethyl)ether	2-Methylnaphthalene	Fluorene			

Table 7 (cont.)
Semivolatile Internal Standards with Corresponding
Target Compounds and Surrogates Assigned for Quantitation

1,4-dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
bis(2-Chloroisopropyl)ether	2-Nitroaniline	Hexachlorobenzene			
Hexachloroethane	3-Choloroaniline	NDPA/DPA			
Isophorone	4-Chloro-3-Methylphenol	Pentachloronitrobenzene			
m-Toluidine	4-Chloroaniline	Pentachlorophenol			
n-Decane	Acenaphthylene				
Nitrobenzene	a-Terpineol				
Nitrobenzene-d5, surr	Benzoic Acid				
N-Nitrosodimethylamine	Biphenyl				
N-Nitrosodi-n-propylamine	Caprolactam				
Phenol	Dimethyl Phthalate				
Phenol-d6, surr	Hexachlorobutadiene				
Pyridine 1,4-Dioxane	Hexachlorocyclopentadiene				
Phenol-d6, surr	Naphthalene				

Table 8

Recommended Minimum Response Factor Criteria from Initial and Continuing Calibration Verification Using the Suggested Ions in Table 5

Analyte	MRF
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010

Table 8 (cont.)

Recommended Minimum Response Factor Criteria from Initial and Continuing Calibration Verification Using the Suggested Ions in Table 5

Analyte	MRF
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Table 9
Difficult analytes

Aniline

Benzaldehyde
Benzidine
Benzoic acid
Benzyl alcohol

Caprolactam
4-Chloroaniline
4-chloro-3-methylphenol (p-chloro-m-cresol)

3,3-Dimethylbenzidine
Dimethylphthalate
2,4 Dinitrophenol
4,6-dinitro-2-methylphenol (4,6-dinitro-o-cresol)

Hexachlorocyclopentadiene
Hexachloroethane

2-Methylphenol
3-Methylphenol/4-Methylphenol

2-nitroaniline
3-nitroaniline
4-nitroaniline
4-Nitrophenol
Nitrosodiphenylamine and diphenylamine (NDPA/DPA)
n-Nitrosodimethylamine

Parathion
Pentachloronitrobenzene
Pentachlorophenol
Phenol
Pyridine

Volatile Organic Compounds by EPA 624.1

Reference: EPA 624.1 Purgeables by GC/MS, Appendix A, Part 136, Code of Federal Regulations. August 28, 2017 edition.

1. Scope and Application

Matrices: Wastewater, Water

Definitions: Refer to Alpha Analytical Quality Manual.

This method covers the determination of a number of purgeable organics regulated under the Clean Water Act. This is a purge and trap gas chromatographic/mass spectrometer (GC/MS) method applicable to the determination of the parameters listed above in municipal and industrial discharges as provided under 40 CFR Part 136.1. The compound list is extended to add analytes commonly requested by clients for water samples such as groundwater, surface water and process waters. The procedure is based on **EPA Method 624.1**.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a quarterly laboratory control sample or acceptable performance from an annual proficiency test sample. Major modification to this procedure requires demonstration of performance. The identification of major method modifications requiring performance demonstration is directed by the QA Officer and Laboratory Director on a case-by-case basis.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Regulatory Analyte List 624.1

Parameter	CAS No.	Parameter	CAS No.
Benzene	71-43-2	1,1 - Dichloroethene	75-35-4
Bromodichloromethane	75-27-4	trans- 1,2 - Dichloroethene	156-60-5
Bromoform	75-25-2	1,2 – Dichloropropane	78-87-5
Bromomethane	74-83-9	cis- 1,3 - Dichloropropene	10061-01-5
Carbon tetrachloride	56-23-5	trans- 1,3 - Dichloropropene	10061-02-6
Chlorobenzene	108-90-7	Ethyl benzene	100-41-4
Chloroethane	75-00-3	Methylene chloride	75-09-2
2 - Chloroethylvinyl ether	110-75-8	1,1,2,2- Tetrachloroethane	79-34-5
Chloroform	67-66-3	Tetrachloroethene	127-18-4
Chloromethane	74-87-3	Toluene	108-88-3
Dibromochloromethane	124-48-1	1,1,1 - Trichloroethane	71-55-6
1,2 - Dichlorobenzene	95-50-1	1,1,2 - Trichloroethane	79-00-5
1,3 - Dichlorobenzene	541-73-1	Trichloroethene	79-01-6
1,4 - Dichlorobenzene	106-46-7	Trichlorofluoromethane	75-69-4
1,1 - Dichloroethane	75-34-3	Vinyl chloride	75-01-4
1,2 - Dichloroethane	107-06-2		

Extended Analyte List:

Parameter	CAS No.	Parameter	CAS No.
Acrolein	107-02-8	4-Methyl-2-pentanone	108-10-1
Acrylonitrile	107-13-1	2-Hexanone	591-78-6
Acetone	67-64-1	m/p- Xylene	1330-20-7
Carbon disulfide	75-15-0	o-Xylene	1330-20-7
Vinyl acetate	108-05-4	Styrene	100-42-5
2-Butanone	78-93-3	Dibromomethane	74-95-3
1,4-Dioxane	123-91-1	Methyl tert-butyl ether	1634-04-4
tert-Butyl alcohol	75-65-0	tert-Amyl methyl ether	994-05-8
cis-1,2-Dichloroethene	156-59-2	1,2-Dibromoethane	106-93-4
1,2-Dibromo-3-chloropropane	96-12-8	1,2,4,Trichlorobenzene	120-82-1
Methylcyclohexane	108-87-2	1,2,3-Trichlorobenzene	87-61-6
1,3-Dichloropropane	142-28-9	Isopropylbenzene	98-82-8
Ethanol	64-17-5		

2. Summary of Method

Purge gas is bubbled through a 5mL water sample contained in a specially designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the purgeables are trapped. After purging is completed, the trap is heated and back flushed with gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables, which are then detected with a mass spectrometer

2.1 Method Modifications from Reference

The following capillary column is substituted for the columns referenced in the method:

RTX 502.2, 40m, 0.18µm df or equivalent.

3. Reporting Limits

The laboratory reporting limits are listed in Table 1. The laboratory reporting limits are adjusted on a sample specific basis to account for dilutions required for target analyte concentrations that exceed the calibration range or sample matrix interference purposes.

4. Interferences

Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be free from contamination under the conditions of the analysis. Running laboratory reagent blanks as described in Section 9.1 and 10.3 demonstrates the system is free of contamination. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system must be avoided.

Sample contamination occurs by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A trip blank or a field reagent blank prepared from reagent water and carried through the sampling and handling protocol serves as a check on such contamination.

Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the sample syringe must be rinsed with reagent water between

sample analyses. Each autosampler position is also monitored for positive hits and subsequent sample analyses are checked for potential carry-over. If carry-over is suspected, the sample is rerun for confirmation. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high purgeable levels, it may be necessary to wash the purging device with a detergent solution, rinse it with reagent water, and then dry it in a 105°C oven between analyses. The trap and other parts of the system are subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

When the sample foams, antifoam is added. One drop of antifoam is use per 10 mLs of sample. The same amount is added to the QC. If the sample is too foamy and one drop per 10 mLs cannot eliminate the foam, then the sample is diluted and then one drop of antifoam per 10 mLs of sample is added. Continue to dilute as necessary keeping the 1 drop per 10 mLs constant. This foam check is done on the screen sample to preserve the integrity of the other vials.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The following parameters covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. Pure standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn if the analyst handles pure (undiluted) materials of these toxic compounds.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Grab samples in standard 40mL glass screw-cap vials with Teflon lined silicon septa (VOA vial). Three VOA vials are filled per sample location. If needed, collect additional sample(s) for the MS/MSD.

Fill the sample bottle just to overflowing. Samples must be introduced into the vials gently to reduce agitation, which might drive off volatile compounds.

Seal the bottle so that no air bubbles are in the sample container. If preservative has been added, shake vigorously for one minute. Invert the bottle and tap to check for air bubbles. Recollect the samples if any air bubbles are present.

Maintain the hermetic seal on the sample bottle until time of analysis. Ice or refrigerate all samples from the time of collection until analysis.

Cool and maintain the sample temperature between 1 and 6 °C. Sample receiving personnel note on the nonconformance form when samples received at the laboratory are not within the temperature criteria

6.2 Sample Preservation

6.2.1 Experimental evidence indicates that some aromatic compounds, notably benzene, toluene, and ethyl benzene are susceptible to rapid biological degradation under certain environmental conditions. Refrigeration alone may not be adequate to preserve these compounds in waters for more than seven days.

6.2.2 Samples suspected of containing residual chlorine are preserved with sodium thiosulfate and the vials are filled completely with sample.

6.2.3 If acrolein is to be determined, analyze the sample within 3 days. Samples that are acidified (pH<2) are not properly preserved for the analyses of acrolein and must be narrated.

6.2.4 Additionally, the analyte 2-Chloroethyl vinyl ether is known to degrade quickly in a low pH environment and must be analyzed using an unacidified sample.

6.2.5 Considering the above preservation issues, Alpha's standard protocol is to preserve all samples with sodium thiosulfate and complete the analysis within 3 days.

6.2.6 The sampling procedure is then completed as per Section 6.1.

6.2.7 Vials that are submitted with improper preservation will be narrated for inclusion in the final report.

6.3 Sample Shipping

Samples requiring shipment to the laboratory are shipped in coolers packed in ice via an overnight delivery service in accordance with applicable Department of Transportation regulations.

6.4 Sample Handling

The laboratory routine practice is to collect three 40mL glass vials and transport the sample, and extra vials for MS/MSD analysis, with ice in coolers. The three sample vials that make up each sample are then split between the three VOC sample storage refrigerators at the laboratory. Storing the vials apart provides a useful check if laboratory contamination of a sample is suspected.

Note: Samples requiring analysis for Acrolein must be analyzed within three (3) days of sample collection.

Document client specific sample handling, preservation and collection criteria in the project file. The laboratory Login staff documents sample temperature at the time of receipt.

Record deviations from this SOP or client specific criterion on the chain of custody form.

Record holding time exceedances, improper preservation and observed sample headspace on the nonconformance report form.

7. Equipment and Supplies

7.1 Vial: glass VOA vials with Teflon-lined septa screw caps. Purchased pre-cleaned to EPA specifications.

7.2 Purge and Trap System: The purge and trap system consists of three separate pieces of equipment: a purging device, coupled to the desorber (EST Centurion/Archon; EST Evolution/Tekmar Velocity, or equivalents) and trap.

7.2.1 Purge gas = Helium, analytical grade (99.999%) or Nitrogen, ultra-high purity.

7.2.2 The purging device accepts 5mL of sample. The 5mL of sample must have a water column at least 3cm deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3mm at the origin. The purge gas must be introduced no more than 5mm from the base of the water column.

7.2.3 The Tekmar Solatek purging device is a closed system, designed to accept the 40mL VOA vials. The instrument automatically adds internal standards and surrogates when transferring the 5 mLs of sample to the purge vessel. The purge gas is introduced into the aqueous portion to purge the volatile components onto the trap.

7.2.4 The trap used in the desorber is typically a Supelco "K" trap. Different traps may be used if equivalent performance is demonstrated.

7.2.5 The desorber is capable of rapidly heating the trap to 260 °C. The trap is not heated above manufacturer specifications.

7.3 Gas chromatograph: An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, and gases. Agilent 6890 or equivalent, Column - RTX 502.2, 40m, 0.18µm df, or equivalent.

7.4 Mass spectrometer: Capable of scanning from 35-270 amu every two seconds or less, utilizing 70V (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the criteria in Table 2 when 50ng of 4-bromofluorobenzene (BFB) is injected through the GC inlet. The GC/MS interface is direct capillary. Agilent 5973 or equivalent.

7.5 Data system: A computer system is interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software allows searching any GC/MS data file for specific m/z (masses) and plotting such m/z abundance versus time or scan number. HP ChemServer software is used for data acquisition and Enviroquant E.02.02 is used for data reduction. Approved data is electronically transferred to the laboratory wide LIMS for final client reporting.

7.6 Syringes: 5mL and 10mL, glass with Luerlock tip.

7.7 Micro syringes: 10, 25, 100, 250, 500, and 1000µL.

7.8 Syringe valve: Two-way, with Luer ends.

7.9 Disposable Pasteur pipets.

7.10 Volumetric flasks: 10mL, 100mL, Class A with ground glass stoppers.

7.11 Vials: 2mL, 4mL with Teflon-lined screw caps.

7.12 Autopipet: 1mL

7.13 Antifoam A: Sigma Catalog #A-6582 or equivalent.

8. Reagents and Standards

8.1 Reagent water: Reagent water in the GC/MS volatiles laboratory is municipal water, passed through a reverse osmosis system. The reagent water after treatment with activated carbon does not contain interferents or the parameters of interest at the reporting limit.

8.2 Sodium Thiosulfate: ACS Reagent Grade or equivalent, Granular.

8.3 Methanol, MeOH: ACS Purge and Trap grade quality or equivalent.

8.4 Trap: Tekmar purge trap K, Vocarb 3000 or equivalent.

8.5 Stock standard solutions:

Certified stock standard solutions in methanol. The certification includes the concentration, uncertainty and traceability to NIST if available. Stock standards include calibration standards, calibration verification, internal, surrogates and spiking solutions. 2 sources are necessary: one utilized for Initial Calibration Standard preparation and the other utilized for ICV Standard (ICVS) preparation.

Select the certified stock standards containing the parameters of interest. Record the concentration of the certified stock standards, lot number, supplier, standard name, catalog number, expiration date, solvent vendor, solvent lot number, preparation date and preparer's initials in the standards logbook. Record the number of containers prepared and the identifier for the stock standard.

Transfer the opened stock standard solution into a Teflon-sealed screw-cap vial. Store, with minimal headspace, at -10 to -20°C and protect from light. Store according to the manufacturer's documented holding time and storage temperature recommendations.

8.5.1 Initial Calibration, primary source:

8.5.1.1 624 Orange:

8.5.1.1.1 Custom VOC Standard – SPEX CertiPrep #VO-ALAMA-10, varied concentrations
8.5.1.1.2 Naphthalene – Restek Catalog #31280; 1000 ug/m

8.5.1.2 624 Yellow

8.5.1.2.1 Dichlorodifluoromethane – AccuStandard #M-502-24-10X, 2000 ug/ml
8.5.1.2.2 MegaMix EPA Method 624 – Restek Catalog #30497, 2000 ug/mL
8.5.1.2.3 624 Calibration Mix #1 – Restek Catalog #30020, 2000 ug/mL
8.5.1.2.4 n-Hexane – Absolute Standards Part #90223, 2000 ug/mL

8.5.1.3 624 Extras ICAL

8.5.1.3.1 Custom VOC Standard 8 compounds - AccuStandard Catalog #S-73335, 200 ug/mL

8.5.1.4 624-SIM ICAL:
8.5.1.4.1 1,4-Dioxane – AccuStandard Catalog # AS-E0480, 10,000 ug/ml

8.5.1.5 Ethanol Ethanol - Absolute Standards # 90572, 20mg/ml

8.5.2 ICVS, QC Check/LCS, MS, MSD, secondary source:
8.5.2.1 624 ICV Orange
8.5.2.1.1 Custom Oxygenates Standard, Restek #559744, Concentration varied
8.5.2.1.2 Naphthalene, Agilent Technologies #PST-4400M1000, 1000ug/ml

8.5.2.2 624 ICV Green
8.5.2.2.1 TCL Ketone Mix, Accustandard #CLP-022K-25X, 5.0mg/ml
8.5.2.2.2 Acrolein & Acrylonitrile Standard, Agilent Technologies #AMN-623-1, 2000ug/ml

8.5.2.3 624 ICV Yellow
- **8.5.2.3.1** Custom VOA Mix, AccuStandard # S-8082A-R1, 2000ug/ml
- **8.5.2.3.2** n-Hexane Standard, AccuStandard #TK-100-01S-10X, 2.0mg/ml
- **8.5.2.3.3** Dichlorodifluoromethane, Restek #30275, 2000ug/ml

8.5.2.4 624 ICV Red:
- **8.5.2.4.1** Dibromomethane Standard, AccuStandard #M502-20-10X, 2.0mg/ml
- **8.5.2.4.2** 2-Chloroethylvinyl ether, AccuStandrd #M-601C-10X, 2.0mg/ml
- **8.5.2.4.3** Methyl t-Butyl Ether standard, Agilent #STS-440-1, 2000ug/ml
- **8.5.2.4.4** Vinyl Acetate Standard, Restek #30216, 2000ug/ml

8.5.2.5 624 ICV 1,4-Dioxane:
- **8.5.2.5.1** Custom 1,4-Dioxane Standard, Restek #566695, 50,000ug/ml

8.5.2.6 624 ICV Extras:
- **8.5.2.6.1** Custom Organic Standard, SPEX CertiPrep #VO-ALAMA-23, 200ug/ml

8.5.2.7 624-SIM ICV 1,4-Dioxane
- **8.5.2.7.1** Custom 1,4-Dioxane Standard, Restek #557629, 10,000ug/ml

8.5.2.8 Ethanol ICV: Ethanol Standard, Restek #30288, 2000ug/ml

8.6 Primary dilution standards: Using the stock standard solutions listed above, prepare the Primary dilution standards in methanol that contain the parameters of interest.

Primary dilution standards are stored with minimal headspace at -10 to -20°C and protected from light. Check for signs of degradation or evaporation, before preparing calibration standards from them. ICVS Standard Green, Yellow, Blue, Red Orange and 1,4-Dioxane Mixes should be replaced when it is suspected that the standard has degraded or by comparison with the check standard or every two months. The 624 Orange and 624 Yellow standards must be prepared every two months or when degradation is suspected.

Record the stock standard identifier, expiration date for primary dilution standard, solvent vendor, solvent lot number, preparation date and preparer's initials in the standards logbook. Record the number of containers prepared and the identifier for the secondary standard.

8.6.1 ICVS, secondary source:

8.6.1.1 ICVS Calibration Green: 400µL of Green-Mix brought to 10mL volume with MeOH.
8.6.1.2 ICVS Calibration Yellow: 1mL of Yellow-Mix brought to 10mL volume with MeOH.
8.6.1.3 ICVS Calibration Blue: 160µL of Blue-Mix brought to 10mL volume with MeOH.
8.6.1.4 ICVS Calibration Red:
- **8.6.1.4.1** 500µL Red-Mix MTBE
- **8.6.1.4.2** 500µL Red-Mix 2-CEVE
- **8.6.1.4.3** 1mL Red-Mix Vinyl Acetate
- **8.6.1.4.4** 500uL of Red-Mix Dibromomethane brought to 10mL volume with MeOH.

8.6.1.5 ICVS Calibration Orange: 1mL of Oxy mix brought to 10mL volume with MeOH.
8.6.1.6 ICVS 1,4-Dioxane: Transfer to a vial.
8.6.1.7 ICVS Dichlorofluoromethane: 1.0 mL of Freon-12 brought to 10 mL with MeOH.
8.6.1.8 624-Extras ICV: Transfer to a vial
8.6.1.9 Hexane ICV: Dilute 1mL to 20mL with methanol
8.6.1.10 Ethanol ICV: Transfer to a vial

8.6.2 Initial Calibration Standard, primary source

8.6.2.1 624 Orange A: 500µL brought to 10 mL with MeOH

8.6.2.2 624 Yellow: 1mL of each stock brought to 10 mL with MeOH.

8.6.2.3 624-Extras ICAL: Transfer to a vial

8.6.2.4 624-Hexane ICAL: Dilute 1mL to 20mL with methanol

8.6.2.5 Ethanol ICAL: Transfer to a vial

8.7 Calibration standards and Matrix Spiking Solutions:

The primary dilution standards are used to prepare the aqueous calibration standards. Prepare the calibration standards using a microliter syringe (µL) to transfer the appropriate volume of primary dilution standard into a 100 mL volumetric flask containing lab reagent water (mL). Five mLs of this aqueous solution is the calibration standard. The aqueous standards can be stored for up to 24 hours at 4 ± 2 °C, if held in sealed vials with zero headspace. Record the primary standard identifier, expiration date for primary dilution standard, preparation date and preparer's initials in the standards logbook. Record the exact preparation steps and the identifier for the calibration standards.

8.7.1 Initial Calibration Standard Preparation / ICVS Standard Preparation:

8.7.1.1 Level 1 Standard (all: 1 ug/L; Acrolein, Acrylonitrile, Vinyl acetate, m,p-Xylene: 2ug/L; Acetone, 2-Butanone, 4-Methyl-2-pentanone, 2-Hexanone: 2.5 ug/L; Total Xylene: 3 ug/L; TBA: 5 ug/L; 1,4-Dioxane: 100 ug/L): Add 25 mL of Level 2 Standard (below) into a 100 mL volumetric flask. Bring to volume with reagent water. Transfer into a 40mL vial.

8.7.1.1.1 Ethanol Level 1 Standard 50 ug/L. Add 25 mL of Level 2 Standard (below) into a 100 mL volumetric flask. Bring to volume with reagent water. Transfer into a 40mL vial.

8.7.1.2 Level 2 Standard (4X the Level 1): Add 4µL of 624 Orange A and 2 µL each of 624 Yellow, 624 Extras and 624 Hexane into a 100mL volumetric flask. Bring to volume with reagent water. Transfer into a 40mL vial.

8.7.1.2.1 Ethanol Level 2 Standard 200 ug/L. Add 1 uL of Ethanol std. (20,000 ppm) into a 100 mL volumetric flask. Bring to volume with reagent water. Transfer into a 40mL vial.

8.7.1.3 Level 3 Standard (20X the Level 1): Add 20µL of 624 Orange A and 10 µL each of 624 Yellow, 624 Extras and 624 Hexane into a 100mL volumetric flask. Bring to volume with reagent water. Transfer to a 40mL vial.

8.7.1.3.1 Ethanol Level 3 Standard 1000 ug/L. Add 5 uL of Ethanol std. (20,000 ppm) into a 100 mL volumetric flask. Bring to volume with reagent water. Transfer into a 40mL vial.

8.7.1.4 Level 4 Standard (40X the Level 1): Add 40µL of 624 Orange A and 20 µL each of 624 Yellow, 624 Extras and 624 Hexane into a 100mL volumetric flask. Bring to volume with reagent water. Transfer to a 40mL vial.

8.7.1.4.1 Ethanol Level 4 Standard 2000 ug/L. Add 10 uL of Ethanol std. (20,000 ppm) into a 100 mL volumetric flask. Bring to volume with reagent water. Transfer into a 40mL vial.

8.7.1.5 Level 5 Standard (60X the Level 1): Add 60µL of 624 Orange A and 30 µL each of 624 Yellow, 624 Extras and 624 Hexane into a 100mL volumetric flask. Bring to volume with reagent water. Transfer to a 40mL vial.

8.7.1.5.1 Ethanol Level 5 Standard 3000 ug/L. Add 15 uL of Ethanol std. (20,000 ppm) into a 100 mL volumetric flask. Bring to volume with reagent water. Transfer into a 40mL vial.

8.7.1.6 Level 6 Standard (100X the Level 1): Add 100µL of 624 Orange A and 50 µL each of 624 Yellow, 624 Extras and 624 Hexane into a 100mL volumetric flask. Bring to volume with reagent water. Transfer to a 40mL vial.

8.7.1.6.1 Ethanol Level 6 Standard 5000 ug/L. Add 25 uL of Ethanol std. (20,000 ppm) into a 100 mL volumetric flask. Bring to volume with reagent water. Transfer into a 40mL vial.

8.7.1.7 Level 7 Standard (200X the Level 1): Add 200µL of 624 Orange A and 100 µL each of 624 Yellow, 624 Extras and 624 Hexane into a 100mL volumetric flask. Bring to volume with reagent water. Transfer to a 40mL vial.

8.7.1.7.1 Ethanol Level 7 Standard 10000 ug/L. Add 50 uL of Ethanol std. (20,000 ppm) into a 100 mL volumetric flask. Bring to volume with reagent water. Transfer into a 40mL vial

8.7.1.8 Level 8 Standard (400X the Level 1): Add 400 µL of 624 Orange A to a 100mL volumetric flask. Bring to volume with reagent water. Transfer to a 40mL.

8.7.1.9 ICVS (same concentration as the Level 3): Add 20µL of Orange, 10ul of 624 Hexane, 25µL of Green, 10µL of Yellow, 25µL of Blue, 20µL Red, 10uL of 624 Extras, 10uL Freon-12 and 100 µL of the 1,4-Dioxane standard into a 100mL volumetric flask. Bring to volume with reagent water. Transfer to a 40mL vial.

8.8 Internal Standard/Surrogate Standard Mixed Solution (IS/SS):

8.8.1 Internal standards and surrogates are automatically added by the autosampler equipment.

8.8.1.1 Restek 624 Internal standard mix, catalog # 30023, or equivalent, 1500µg/mL. Store with minimal headspace at -10 to -20°C and protect from light. Expires 6 months from the date the vial was opened.

8.8.1.2 Restek 624 Surrogate standard mix, catalog # 30243, or equivalent, 2000 µg /mL. Store with minimal headspace at -10 to -20°C and protect from light. Expires 6 months from the date the vial was opened.

8.8.2 Add 1.0 mL Internal Standard mix and 0.75 mL Surrogate Mix to approximately 10mLs MeOH in a 100 mL volumetric flask. Fill to volume to give a final concentration of 15µg/mL.

8.8.3 The autosampler adds 10µL to each sample, standard and blank to give a concentration of 30µg/L in the 5 mL aliquot.

8.8.4 Record the stock standard identifier, expiration date for the internal standard, preparation date and preparer's initials in the standards logbook. Record the exact steps for preparing the standard and the identifier for the internal standard.

8.9 Tune standard: Ultra Scientific Catalog # STS-110N, Bromofluorobenzene (BFB) in methanol, 2000µg/mL or equivalent. Add 250µL stock standard to approximately 5mLs Methanol in a 10mL volumetric flask. Fill to volume to give a final concentration of 50µg/mL. Transfer to screw-cap vials, cap tightly and store amber vials with minimal headspace in freezer at -10 to -20°C. Expires 6 months from the date of preparation.

Record the stock standard identifier, expiration date for the tune solution, preparation date and preparer's initials in the standards logbook. Record the exact steps for preparing the tune solution and the identifier.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

Analyze a reagent water blank each day to demonstrate that interferences from the analytical system are under control. The reagent blank must contain the internal standards.

Analyze the reagent water blank from the same lot of water used for preparing the standards, QC samples and making sample dilutions. If the lot of reagent water is changed during the analysis perform an additional blank to ensure the analytical system is not contaminated.

The Blank results must be less than the MDL for target analytes. If failure occurs, the Blank is reanalyzed. If failure continues, maintenance should be performed and the system recalibrated if necessary.

If blank contamination is less than 1/10th the concentration in the sample, then narrate for inclusion in the final report. Report a result for each analyte in a blank at or above the MDL to 2 significant figures.

9.2 Laboratory Control Sample (LCS)

Demonstrate through the analyses of the QC check standard or LCS that the operation of the measurement system is in control. The LCS must be from a source different from the source used for calibration but may be the same source used for the DOC and ICV. The frequency of the LCS is immediately after calibration and at the beginning of each 12-hour analytical run time.

> **Note:** The 12-hour shift begins after analysis of BFB, the LCS, and the blank, and ends 12 hours later. BFB, the LCS, and blank are outside of the 12-hour shift. The MS and MSD are treated as samples and are analyzed within the 12-hour shift. The total time for the analysis of the BFB, LCS, blank, and 12-hour shift must not exceed 14 hours.

Analyze the QC check standard to determine the concentration measured of each parameter. Calculate each percent recovery. For each parameter listed in Table 5, compare the percent recovery with the corresponding calibration acceptance criteria found in Table 5. If the responses for all parameters of interest fall within the designated ranges, analysis of actual samples can begin. If any individual recovery falls outside the range, proceed according to Section 12. The in-house MS/MSD criteria should be used for the LCS compounds not listed in Table 5.

Note: The large number of analytes in Tables 1 - 2 present a substantial probability that one or more will fail the acceptance criteria when all analytes are tested simultaneously. Because a re-test is allowed in event of failure it may be prudent to analyze two LCSs together and evaluate results of the second analysis against the QC acceptance criteria only if an analyte fails the first test.

9.3 Calibration Verification (ICV)

Because the analytical system is calibrated by purge of the analytes from water, calibration verification is performed using the laboratory control sample (LCS). See Section 9.2 for requirements for calibration verification.

9.4 Matrix Spike

Spike and analyze a minimum of 5% of all samples to monitor and evaluate laboratory data quality. The concentration of the spike should be at one to five times higher than the sample concentration or at the client requested action level. Due to the large number of unknown samples performed, the concentration of the matrix spike is at 20µg/L unless otherwise requested by the client. Refer to Section 8.7 for matrix spike preparation.

If the client doesn't supply enough volume to perform the MS/MSD analysis, the lab will utilize a separate sample source and spike appropriately. Calculate the MS/MSD percent recoveries. Compare the percent recovery for each parameter with the corresponding QC acceptance criteria found in Table 5.

If any individual percent recovery falls outside the designated range for recovery in either aliquot, or the RPD limit is exceeded, the result for the analyte in the unspiked sample is suspect.

The large number of analytes tested in performance tests in this method present a substantial probability that one or more will fail acceptance criteria when many analytes are tested simultaneously, and a re-test is allowed if this situation should occur. If, however, continued re-testing results in further repeated failures, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority. Results associated with a QC failure for an analyte regulated in a discharge cannot be used to demonstrate regulatory compliance. QC failures do not relieve a discharger or permittee of reporting timely results.

Report MS/MSD data to the client whose field sample was spiked.

9.5 Laboratory Duplicate

Duplicates for this method are in the form of the MSD. See section 9.4.

9.6 Method-specific Quality Control Samples

9.6.1 Internal Standards

Area counts of the internal standard peaks in all samples and QC samples must be between 50-200% of the areas of the internal standards in the QC check standard.

If any individual percent recovery falls outside the range, that parameter has failed the acceptance criteria. For calibration standards, LCS or blanks the internal standard must be within the range for data to be reported to the clients. For samples, matrix spikes and matrix spike duplicates: if the data is not within the range, the sample is rerun to confirm that the failure is due to sample matrix. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient

sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

9.6.2 Surrogates

The laboratory must spike all samples with surrogate standards to monitor continuing laboratory performance. Calculate the percent recovery of each surrogate compound. The recovery for the surrogate compounds must be within the 60-140% acceptance criteria, until in house limits are established.

If surrogate recovery fails to meet criteria, sample must be reanalyzed. The only exception to this rule is, if sample shows no detection of target compounds and any surrogate exceeds acceptance range, no further action is required. If the reanalysis also fails, a narrative is submitted for inclusion on the Client report.

9.7 Method Sequence

Tests for BFB, the LCS, and the blank are outside of the 12-hour shift, and the 12-hour shift includes samples and matrix spikes. The total time for analysis of BFB, LCS, blank, and the 12-hour shift must not exceed 14 hours.

The analytical sequence is as follows:

1. BFB Tune Standard
2. QC Check Standard (LCS) – from a second source
3. Method Blank
4. Samples, MS, MSD

10. Procedure

10.1 Equipment Set-up

10.1.1 GC/MS Tune

At the beginning of every 12 hours of instrumental analysis, tune the GC/MS system to demonstrate acceptable performance for BFB. The tune must pass before proceeding with the analysis of any samples, blanks, or standards.

Inject 1µL of BFB tune solution directly on to the column. Analyze the solution using the same mass spectrometer conditions as used for the sample analyses. Obtain a background-corrected mass spectrum of BFB and confirm that all the key m/z criteria in Table 2 are achieved. The mass spectrum of BFB should be acquired in the following manner:

(1) Adjust the scan rate of the MS to produce a minimum of 5 mass spectra across the BFB GC peak, but do not exceed 2 seconds per scan.

(2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of BFB.

If the criteria are not achieved, the analyst must perform needed maintenance, retune the mass spectrometer and repeat the test until all criteria are achieved.

Initial Calibration

Prepare the instrumental system to meet the specifications in Section 10.3. For new systems or systems not in use on a daily basis, condition the trap overnight at 180°C by back flushing with an inert gas flow of at least 20mL/min. The internal standard calibration procedure is used for quantifying all samples. The internal standards are specified in Section 8.8 and are listed in Table 3.

Calibration standards are prepared as specified in Section 8.7. Prepare calibration standards at eight concentration levels for each parameter to achieve the final concentrations for most of the compounds at 1µg/L, 4µg/L, 20µg/L, 40µg/L 60µg/L, 100µg/L and 200µg/L (1000µg/L extra level is used for acetone only). The calibration standards define the working range of the GC/MS system.

Surrogates and Internal Standards are added by the autosampler at a constant concentration in the calibration standard analyses.

Analyze each calibration standard according to Section 10.3.

Record the calibration standards identifier, internal standard identifier, surrogate standard identifier, concentration, analyst initials and any deviations to this procedure in the instrument analysis logbook.

Tabulate the area response of the characteristic m/z against concentration for each compound and internal standard, and calculate response factors (RF) for each compound as follows:

$$RF = \frac{(A_s \times C_{is})}{(A_{is} \times C_s)}$$

Where:

RF	=	Response Factor
A_s	=	Area of the characteristic m/z for the parameter to be measured.
A_{is}	=	Area of the characteristic m/z for the internal standard.
C_{is}	=	Concentration of the internal standard.
C_s	=	Concentration of the parameter to be measured.

If the RF value over the working range is < 35% RSD, the RF can be assumed to be linear and the average RF is used for calculations. Average RF = Sum of RF values from the calibration curve/ Number of RF values. If the % RSD is > 35%, remake the standard and repeat the calibration. If the problem persists, perform maintenance and any other corrective action. Perform a full initial calibration to standardize the system if any other system changes are made.

Alternatively, the results can be used to fit a linear or quadratic regression of response ratios, As/Ais, vs. concentration ratios Cs/Cis. If used, the regression must be weighted inversely proportional to concentration (1/C). The coefficient of determination (R2) of the weighted regression must be greater than 0.920 (this value roughly corresponds to the RSD limit of 35%).

10.2 Equipment Operation and Sample Processing

Changes in acquisition parameters, equipment, conditions and tune criteria require written authorization from management. Demonstration of method performance based on method modifications must be on file before sample analysis.

The following are the routine instrumental parameters:

Electron Energy = 70 V (nominal)
Mass Range = 35-270 amu
Scan Time = At least five scans/peak but not to exceed two seconds/scan
Carrier Gas = Helium
Acquisition mode = Scan

The following are purge parameters:

Purge gas flow rate	= 40 mL/min
Purge time	= 11.0 min
Purge temperature	= 40 °C
Dry purge time	= 4 min
Desorb temperature	= 255 °C
Desorb time	= 4 min
Bake out temperature	= 280°C
Bake out time	= 10 min

After achieving the key m/z abundance criteria for the BFB, calibrate or verify the calibration of the system daily as described in Sections 10.2 and 10.3. If the performance criteria are achieved continue the analysis. If performance criteria are not achieved take corrective action as defined in Section 12.

Analyze a reagent water blank containing 10.0µL IS/SS solution (Section 8.8). If no target parameters are above the reporting limit and the chromatography is acceptable continue the analysis. If poor chromatography or target parameters are above the reporting limit, take appropriate corrective action as defined in Section 12. The purging vessel must be rinsed twice with organic free water between each analysis.

Record the sample number (standard or QC sample identifier), dilution, analyst initials, deviations from this procedure and visual observations in the instrument analysis logbook.

Perform a preliminary data review of the sample, internal standard and surrogate performance at the time of analysis or when the sequence is complete. Note any obvious problems in the instrument analysis logbook. If the concentration for any analyte exceeds the working range of the system, the sample must be reanalyzed at the appropriate dilution. Report analytes from the least diluted analysis that is within calibration range. This may require reporting results for some analytes from different analysis.

Perform first level data review. Obtain the primary m/z (Table 4) and at least two secondary masses for each parameter of interest. The following criteria must be met to make a qualitative identification:

- Compare the background subtracted mass spectra for the sample to the reference spectra. The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.
- The retention time must fall within +/- 0.1 minutes of the retention time of the compound in the analytical standard. However, analyst experience should be used in making the qualitative identification.
- The relative peak heights of the three characteristic masses must fall within 20% of the relative intensities of the masses in a reference mass spectrum. The reference spectrum is obtained from a standard analyzed in the GC/MS system.

Structural isomers that have very similar mass spectra are identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

All 624-MWRA samples are analyzed at a 10x dilution.

10.3 Continuing Calibration

Not applicable.

10.4 Preventive Maintenance

Routine preventive maintenance should be performed on the analytical system. This includes replacement of GC septa and periodic rinsing or replacement of purge and trap tubes and sparge needles. The trap should be replaced every six months, or sooner if performance criteria cannot be met. Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed. If system performance deteriorates, additional maintenance may be required. This includes replacement of injector ports and seals, clipping several inches off of the front end of the GC column, or in extreme cases the replacement of the GC column. Flushing or replacement of purge and trap lines may be necessary if they become contaminated or develop active sites. Perform routine preventative maintenance as described throughout this SOP. Record all maintenance in the instrument logbook.

11. Data Evaluation, Calculations and Reporting

When a parameter is identified, the quantitation of that parameter should be based on the integrated abundance of the quantitation characteristic m/z given in Table 4. If the sample produces an interference for the primary m/z, use a secondary characteristic m/z to quantitate.
Calculate the concentration in the sample using the average response factor (RF) from the initial calibration curve as follows:

$$Concentration(\mu g / L) = D\left(\frac{A_x \times C_{is}}{A_{is} \times \overline{RF}}\right)$$

where:

D	=	Dilution Factor (sample aliquot mL/ 5 mL)
A_x	=	Area of the characteristic m/z for the compound to be measured
A_{is}	=	Area of the characteristic m/z for the internal standard
C_{is}	=	Concentration of the internal standard
$\overline{RF}$	=	Average RF (Section 10.2)

Report results in µg/L without correction for blank and recovery data. Record all QC data and report with the sample results as required by client specifications. Reported detection limits must be corrected for the sample dilution factor.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedances, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of internal standard, surrogates and QC check standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the QC check standard or LCS recovery of any parameter falls outside the designated acceptance range, rerun the LCS for those analytes that failed to meet the acceptance criteria.

Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, repeat the test using a fresh LCS or perform and document system repair.

Manual integration must be minimized. Routine manual integration of the same parameters indicates a system performance problem. Correct this problem or note in the instrument analysis logbook the suspected causes for routine manual integration. Sign and date all quantitation reports, which require manual integration.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

16. Attachments

Table 1: Reporting Limits

Table 2: BFB Key m/z Abundance Criteria

Table 3: Suggested Surrogate and Internal Standards

Table 4: Characteristic Masses for Purgeable Organics

Table 5: EPA 624.1 Calibration and QC Acceptance Criteria

Table 6: 624.1 Quantitation Ions

Table 7: 624.1 Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Table 1: Reporting Limits

Parameter	Reporting Limits (µg/L)
Chloromethane	10.0
Bromomethane	5.0
Vinyl chloride	2.0
Chloroethane	2.0
Methylene chloride	5.0
Trichlorofluoromethane	5.0
1,1- Dichloroethene	1.0
1,1- Dichloroethane	1.5
Trans- 1,2- Dichloroethene	1.5
Chloroform	1.5
1,2- Dichloroethane	1.5
1,1,1- Trichloroethane	2.0
Carbon tetrachloride	1.0
Bromodichloromethane	1.0
1,2- Dichloropropane	3.5
cis- 1,3- Dichloropropene	1.5
Trichloroethene	1.0
Benzene	1.0
Dibromochloromethane	1.0
1,1,2- Trichloroethane	1.5
trans- 1,3- Dichloropropene	1.5
2- Chloroethylvinyl ether	10.0
Bromoform	1.0
Dichlorofluoromethane	1.0
1,2-Dibromo-3-chloropropane	2.0
Methylcyclohexane	1.0
1,3-Dichloropropane	1.0
Isopropylbenzene	1.0
Hexane	1.0
Ethanol	500
1,1,2,2- Tetrachloroethane	1.0
Tetrachloroethene	1.5
Toluene	1.0
Chlorobenzene	3.5
Ethyl benzene	1.0
1,3- Dichlorobenzene	5.0
1,2- Dichlorobenzene	5.0
1,4- Dichlorobenzene	5.0
Xylenes	2.0
Styrene	1.0
Acetone	10.0
Carbon Disulfide	5.0

Parameter	Reporting Limits (µg/L)
2-Butanone	10.0
Vinyl acetate	20.0
4-Methyl-2-pentanone	10.0
2-Hexanone	10.0
Acrolein	8.0
Acrylonitrile	10.0
cis-1,2-dichloroethene	1.0
tert-Butyl alcohol	20.0
MTBE	20.0
tert-Amyl methyl ether	1.0
1,4-Dioxane	2000
Dibromomethane	1.0
1,2-Dibromoethane	1.0
1,2,4,Trichlorobenzene	1.0
1,2,3-Trichlorobenzene	1.0
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon-113)	1.0

Table 2

BFB Key m/z Abundance Criteria

Mass m/z	Abundance criteria
50	15-40% of Mass 95.
75	30-60% of Mass 95.
95	Base Peak, 100% Relative Abundance.
96	5-9% of Mass 95.
173	<2% of Mass 174.
174	>50% of Mass 95.
175	5-9% of Mass 174.
176	>95% but <101% of Mass 174.
177	5-9% of Mass 176.

Table 3

Suggested Surrogate and Internal Standards

Compound	Primary/Secondary Masses (m/z)	Routine Surrogates and Internal Standards
4-Bromofluorobenzene	95 174, 176	S
Fluorobenzene	96 70	S
Pentafluorobenzene	168	S
Bromochloromethane	128 130	I
2-Bromo-1-chloropropane	77 79, 156	I
1,4-Dichlorobutane	55 90, 92	I

Table 4: Characteristic Masses for Purgeable Organics

Parameter	Primary	Secondary
Chloromethane	50	52
Bromomethane	94	96
Vinyl chloride	62	64
Chloroethane	64	66
Methylene chloride	84	49, 86
Trichlorofluoromethane	101	103
1,1-Dichloroethene	96	61, 98
1,1-Dichloroethane	63	65, 85
trans-1,2-Dichloroethene	96	61, 98
Chloroform	83	85, 47
1,2-Dichloroethane	98	62, 64
1,1,1-Trichloroethane	97	99
Carbon tetrachloride	117	119, 121
Bromodichloromethane	127	83, 85
1,2-Dichloropropane	112	65, 114

Parameter	Primary	Secondary
trans-1,3-Dichloropropene	75	77
Trichloroethene	130	95, 132
Benzene	78	52
Dibromochloromethane	127	129
1,1,2-Trichloroethane	97	83, 99
cis-1,3-Dichloropropene	75	77
2-Chloroethylvinyl ether	106	63, 65
Bromoform	173	171, 175
1,1,2,2-Tetrachloroethane	168	83, 131
Tetrachloroethene	164	129, 166
Toluene	92	91
Chlorobenzene	112	114
Ethyl benzene	106	91
1,3-Dichlorobenzene	146	148, 113
1,2-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
EXTENDED ANALYTES		
Xylenes	106	91
Styrene	104	78, 51
Acetone	43	58
Carbon Disulfide	76	78
2-Butanone	43	72
Vinyl acetate	43	86
4-Methyl-2-pentanone	58	43
2-Hexanone	43	42, 58
Acrolein	56	55
Acrylonitrile	53	52
cis-1,2-Dichloroethene	96	61, 98
tert-Butyl alcohol	59	41
MTBE	73	57, 41
tert-Amyl methyl ether	73	55, 87
1,4-Dioxane	88	58
Dibromomethane	95	174
Dichlorodifluoromethane	85	87
1,2-Dibromo-3-chloropropane	75	155
Methylcyclohexane	83	55
1,3-Dichloropropane	76	78
Isopropylbenzene	105	120
1,2-Dibromoethane	107	109
1,2,4,Trichlorobenzene	180	182
1,2,3-Trichlorobenzene	180	182
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon-113)	101	151, 103
Hexane	57	43, 41
Ethanol	45	46, 43

Table 5 : EPA 624.1 Calibration and QC Acceptance Criteria

Parameter	Range for LCS (%)	Range for MS/MSD (%)	Limit for RPD
1,1-Dichloroethene	50 – 150	D - 234	32
1,1-Dichloroethane	70 – 130	59 - 155	40
1,1,1- Trichloroethane	70 – 130	52 - 162	36
1,1,2- Trichloroethane	70 – 130	52 - 150	45
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon-113)	60 – 140	40 - 160	30
1,1,2,2-Tetrachloroethane	60 – 140	46 - 157	61
1,2- Dichlorobenzene	65 – 135	18 - 190	57
1,2- Dichloroethane	70 – 130	49 - 155	49
1,2- Dichloropropane	35 – 165	D - 210	55
1,2-Dibromo-3-chloropropane	60 – 140	40 - 160	30
1,2-Dibromoethane	60 – 140	40 - 160	30
1,2,3-Trichlorobenzene	60 – 140	40 - 160	30
1,2,4-Trichlorobenzene	60 – 140	60 - 140	19
1,3- Dichlorobenzene	70 – 130	59 - 156	43
1,3-Dichloropropane	60 – 140	40 - 160	30
1,4- Dichlorobenzene	65 – 135	18 - 190	57
1,4-Dioxane	60 – 140	60 – 140	30
2- Chloroethylvinyl ether	D – 225	D - 305	71
2-Butanone	60 – 140	40 - 160	41
2-Hexanone	60 – 140	40 - 160	39
4-Methyl-2-pentanone	60 – 140	40 - 160	41
Acetone	40 – 160	40 - 160	39
Acrolein	60 – 140	40 - 160	60
Acrylonitrile	60 – 140	40 - 160	60
Benzene	65 – 135	37 - 151	61
Bromodichloromethane	65 – 135	35 - 155	56
Bromoform	70 – 130	45 - 169	42
Bromomethane	15 – 185	D - 242	61

Parameter	Range for LCS (%)	Range for MS/MSD (%)	Limit for RPD
Carbon disulfide	60 – 140	40 - 160	25
Carbon tetrachloride	70 – 130	70 - 140	41
Chlorobenzene	65 – 135	37 - 160	53
Chloroethane	40 – 160	14 - 230	78
Chloroform	70 – 135	51 - 138	54
Chloromethane	D – 205	D - 273	60
cis-1,2-Dichloroethene	60 – 140	60 - 140	20
cis-1,3-Dichloropropene	25 – 175	D - 227	58
Dibromochloromethane	70 – 135	53 - 149	50
Dibromomethane	70 - 130	70 - 130	13
Dichlorodifluoromethane	70 – 130	70 - 130	30
Ethyl benzene	60 – 140	37 - 162	63
Hexane	60 – 140	40 - 160	30
Ethanol	60 – 140	40 - 160	30
Isopropylbenzene	60 – 140	40 - 160	30
m/p-Xylene	60 – 140	40 - 160	26
Methylcyclohexane	60 – 140	40 - 160	30
Methylene chloride	60 – 140	D - 221	28
MTBE	60 – 140	60 – 140	21
o-Xylene	60 – 140	40 - 160	21
Styrene	60 – 140	40 - 160	22
tert-Butyl alcohol	60 – 140	60 – 140	30
tert-Amyl methyl ether	60 – 140	60 – 140	30
Tetrachloroethene	70 – 130	64 - 148	39
Toluene	70 – 130	47 - 150	41
trans- 1,2- Dichloroethene	70 – 130	54 - 156	45
trans- 1,3- Dichloropropene	50 – 150	17 - 183	86
Trichloroethene	65 – 135	71 - 157	48
Trichlorofluoromethane	50 – 150	17 - 181	84
Vinyl acetate	60 – 140	40 - 160	28

Parameter	Range for LCS (%)	Range for MS/MSD (%)	Limit for RPD
Vinyl chloride	5 - 195	D - 251	66

D = Detected; result must be greater than zero. Criteria were calculated assuming a QC check sample concentration of 20 µg/L.

TABLE 6

624.1 Quantitation Ions Used

Compound	Quantitation Ion	Compound	Quantitation Ion
Benzene	78	Methyl tert-butyl ether	73
Bromodichloromethane	83	tert-Butyl alcohol	59
Bromoform	173	tert-Amyl methyl ether	73
Bromomethane	94	1,1,2,2-Tetrachloroethane	83
Carbon tetrachloride	117	Tetrachloroethene	166
Chlorobenzene	112	Toluene	92
Chloroethane	64	1,1,1-Trichloroethane	97
2-Chloroethylvinyl ether	63	1,1,2-Trichloroethane	97
Chloroform	83	Trichloroethene	130
Chloromethane	50	Trichlorofluoromethane	101
Dibromochloromethane	129	Vinyl chloride	62
1,2-Dichlorobenzene	146	cis-1,2-dichloroethene	96
1,3-Dichlorobenzene	146	Acrolein	56
1,4-Dichlorobenzene	146	Acrylonitrile	53
1,1-Dichloroethane	63	Acetone	43
1,2-Dichloroethane	62	Carbon disulfide	76
1,1-Dichloroethene	96	Vinyl acetate	43
trans-1,2-Dichloroethene	96	2-Butanone	43
1,2–Dichloropropane	63	4-Methyl-2-pentanone	58
cis-1,3-Dichloropropene	75	2-Hexanone	43
trans-1,3-Dichloropropene	75	m/p- Xylene	106
1,4-Dioxane	88	o-Xylene	106
Ethyl benzene	91	Styrene	104
Methylene chloride	84	Dibromomethane	95
Dichlorodifluoromethane	85	1,2-Dibromoethane	107
1,2-Dibromo-3-chloropropane	75	1,2,4,Trichlorobenzene	180
Methylcyclohexane	83	1,2,3-Trichlorobenzene	180
1,3-Dichloropropane	76	Isopropylbenzene	105
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon-113)	101	Hexane	57
Ethanol	45		

TABLE 7

624.1 Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Bromochloromethane	2-Bromo-1-Chloro-Propane	1,4-Dichloro-Butane
Chloromethane	2-Chloroethylvinyl ether	Chlorobenzene
Vinyl chloride	4-Methyl-2-pentanone	Ethylbenzene
Bromomethane	cis-1,3-Dichloropropene	p/m-Xylene
Chloroethane	Toluene	o-Xylene
Trichlorofluoromethane	Trans-1,3-dichloropropene	Styrene
Acrolein	2-Hexanone	Bromoform
Acetone	1,1,2-Trichloroethane	1,1,2,2-Tetrachloroethane
1,1-Dichloroethene	Tetrachloroethene	4-Bromofluorobenzene (surr)
tert-Butyl alcohol	Dibromochloromethane	1,3-Dichlorobenzene
Methylene chloride	1,3-Dichloropropane	1,4-Dichlorobenzene
Carbon disulfide		1,2-Dichlorobenzene
Acrylonitrile		Xylenes (Total)
Methyl-tert-butyl-ether		Isopropylbenzene
trans-1,2-Dichloroethene		1,2,3-Trichlorobenzene
Vinyl acetate		1,2,4-Trichlorobenzene
1,1-Dichloroethane		1,2-Dibromo-3-chloropropane
2-Butanone		
cis-1,2-Dichloroethene		
Chloroform		
Pentafluorobenzene (surr)		
1,1,1-Trichloroethane		
Carbon tetrachloride		
tert-Amyl methyl ether		
1,2-Dichloroethane		
Benzene		
Fluorobenzene (surr)		
Trichloroethene		
1,2-Dichloropropane		
Bromodichloromethane		
1,4-Dioxane		
Dibromomethane		
Dichlorodifluoromethane		
Methylcyclohexane		
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon-113)		
Hexane		
Ethanol		

APPENDIX 2

Field Equipment Product Manuals

DUSTTRAK™ II
AEROSOL MONITOR
MODEL 8530/8531/8532/8530EP

OPERATION AND SERVICE MANUAL

P/N 6001893, REVISION M
DECEMBER 2014

DustTrak II 8530/31 Desktop and 8532 Handheld

DustTrak II 8530EP Monitor

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Printed in U.S.A.

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Fax No.
(651) 490-3824

Limitation of Warranty and Liability (effective April 2014)
(For country-specific terms and conditions outside of the USA, please visit www.tsi.com.)

Seller warrants the goods, excluding software sold hereunder, under normal use and service as described in the operator's manual, shall be free from defects in workmanship and material for twenty-four (24) months, or if less, the length of time specified in the operator's manual, from the date of shipment to the customer. This warranty period is inclusive of any statutory warranty. This limited warranty is subject to the following exclusions and exceptions:

a. Hot-wire or hot-film sensors used with research anemometers, and certain other components when indicated in specifications, are warranted for 90 days from the date of shipment;
b. DustTrak internal pump for Models 8530 and 8533 is warranted for two (2) years or 4000 hours, whichever comes first;
c. DustTrak external pump for Models 8530EP and 8533EP is warranted for two (2) years or 8760 hours, whichever comes first;
d. DustTrak internal pump for Models 8530 and 8533 is warranted for operation within ambient temperatures between 5–45°C. Warranty is void when the internal pump is operating outside of this temperature range;
e. Parts repaired or replaced as a result of repair services are warranted to be free from defects in workmanship and material, under normal use, for 90 days from the date of shipment;
f. Seller does not provide any warranty on finished goods manufactured by others or on any fuses, batteries or other consumable materials. Only the original manufacturer's warranty applies;
g. This warranty does not cover calibration requirements, and seller warrants only that the instrument or product is properly calibrated at the time of its manufacture. Instruments returned for calibration are not covered by this warranty;
h. This warranty is **VOID** if the instrument is opened by anyone other than a factory authorized service center with the one exception where requirements set forth in the manual allow an operator to replace consumables or perform recommended cleaning;
i. This warranty is **VOID** if the product has been misused, neglected, subjected to accidental or intentional damage, or is not properly installed, maintained, or cleaned according to the requirements of the manual. Unless specifically authorized in a separate writing by Seller, Seller makes no warranty with respect to, and shall have no liability in connection with, goods which are incorporated into other products or equipment, or which are modified by any person other than Seller.

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Buyer and all users are deemed to have accepted this LIMITATION OF WARRANTY AND LIABILITY, which contains the complete and exclusive limited warranty of Seller. This LIMITATION OF WARRANTY AND LIABILITY may not be amended, modified or its terms waived, except by writing signed by an Officer of Seller.

Service Policy

Knowing that inoperative or defective instruments are as detrimental to TSI as they are to our customers, our service policy is designed to give prompt attention to any problems. If any malfunction is discovered, please contact your nearest sales office or representative, or call TSI's Customer Service departmentat (800) 874-2811 (USA) or (001 651) 490-2811 (International) or visit www.tsi.com.

CONTENTS

These Application Notes can also be found on TSI's web site:
http://www.tsi.com

EXPMN-001 DustTrak II Theory of Operation.pdf
EXPMN-003 DustTrak II Impactor.pdf

Safety Information

IMPORTANT

There are no user serviceable parts inside the instrument. Refer all repair and maintenance to a qualified factory-authorized technician. All maintenance and repair information in this manual is included for use by a qualified factory-authorized technician.

Laser Safety

- The Model 8530/8531/8532 DustTrak II monitor is a Class I laser-based instrument.
- During normal operation, you will ***not*** be exposed to laser radiation.
- Precaution should be taken to avoid exposure to hazardous radiation in the form of intense, focused, visible light.
- Exposure to this light may cause blindness.

Take these precautions:

- ***DO NOT*** remove any parts from the DustTrak II monitor unless you are specifically told to do so in this manual
- ***DO NOT*** remove the housing or covers. There are no serviceable components inside the housing.

WARNING

The use of controls, adjustments, or procedures other than those specified in this manual may result in exposure to hazardous optical radiation.

WARNING

There are no user-serviceable parts inside this instrument. The instrument should only be opened by TSI or a TSI approved service technician.

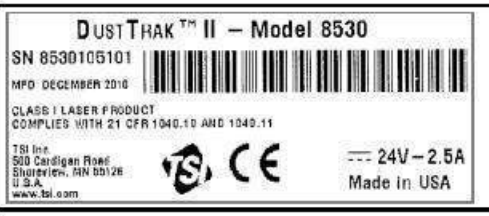
WARNING

If the DustTrak monitor is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.

When operated according to the manufacturer's instruction, this device is a Class I laser product as defined by U.S. Department of Health and Human Services standards under the Radiation Control for Health and Safety Act of 1968. A certification and identification label like the one shown below is affixed to each instrument.

Labels

Advisory labels and identification labels are attached to the instrument.

1. Serial Number Label (bottom)	
2. Laser Radiation Label (internal)	**DANGER!** VISIBLE LASER RADIATION WHEN OPEN. AVOID DIRECT EXPOSURE TO BEAM WARNING: NO USER SERVICABLE PARTS INSIDE. REFER SERVICING TO QUALIFIED PERSONNEL
3. Battery label	**!!WARNING!!** **THIS INSTRUMENT WAS DESIGNED TO USE ONLY TSI SUPPLIED BATTERIES, PN 801680** or **!!WARNING!!** **THIS INSTRUMENT WAS DESIGNED TO USE ONLY TSI SUPPLIED BATTERY, PN 801681**
4. European symbol for non-disposable item. Item must be recycled.	

Description of Caution/Warning Symbols

Appropriate caution/warning statements are used throughout the manual and on the instrument that require you to take cautionary measures when working with the instrument.

Caution

Caution

Failure to follow the procedures prescribed in this manual might result in irreparable equipment damage. Important information about the operation and maintenance of this instrument is included in this manual.

Warning

WARNING

Warning means that unsafe use of the instrument could result in serious injury to you or cause damage to the instrument. Follow the procedures prescribed.

Caution and Warning Symbols

The following symbols may accompany cautions and warnings to indicate the nature and consequences of hazards:

	Warns that the instrument contains a laser and that important information about its safe operation and maintenance is included in the manual.
	Warns that the instrument is susceptible to electro-static discharge (ESD) and ESD protection should be followed to avoid damage.
	Indicates the connector is connected to earth ground and cabinet ground.

Reusing and Recycling

As part of TSI Incorporated's effort to have a minimal negative impact on the communities in which its products are manufactured and used:

- Do ***not*** dispose of used batteries in the trash. Follow local environmental requirements for battery recycling.
- If instrument becomes obsolete, return to TSI for disassembly and recycling.

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Chapter 1

Unpacking and Parts Identification

Carefully unpack the Model 8530/8531/8532 DustTrak™ II Aerosol Monitor from the shipping container. Use the tables and illustrations below to make certain that there are no missing components. Contact TSI immediately if anything is missing or damaged.

Note

If you purchased a DustTrak II Model 8530-NA (no accessories) Aerosol Monitor, it only comes with the following items:

- DustTrak II Model 8530 Aerosol Monitor
- Operations manual
- TrakPro™ Data Analysis Software CD
- One-year calibration certificate
- Service paperwork
- 2-year warranty

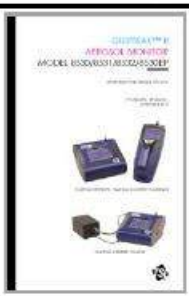
All accessories for the DustTrak II Model 8530 Aerosol Monitor are sold separately. Contact TSI at (800) 874-2811 for information on accessories and how to purchase them through a TSI sales representative.

(continued on next page)

Unpacking the DustTrak II Aerosol Monitor

Compare all the components you received with those listed in the table below. If any parts are missing, contact TSI.

Item	Qty	Part Number	Description
 *or* 	1	8530 8531 8532	Desktop II Desktop II HC Handheld II
	1	801670 801669	Desktop II Carrying Case Handheld II Carrying Case
	1	1090014	Data Analysis Software CD-ROM
	1	800663	Zero Filter

Item	Qty	Part Number	Description
 *or* 	1	801680 801681	6600 mAH Lithium Ion Rechargeable Battery (Desktop) Rechargeable lithium ion battery (Handheld)
	1	1303740	USB cable
	1	801652	Analog/alarm output cable (Desktop models only)
	1	6001893	Operation and Service Manual
	1	N/A	Calibration Certificate

Item	Qty	Part Number	Description
	1	801688	Conductive Tubing
	1	801668	Filter removal tool (Spanner Driver)
	4	801673	Spare Internal Filter Elements Desktop Model Only
	2		37-mm filter includes: Filter body top Filter body bottom Mesh screen
	1		Comes with 37-mm cartridge opening tool
	8	801666	Spare Internal Filters Handheld Model Only

Item	Qty	Part Number	Description
	1	801667	Impactor Kit $PM_{2.5}$ assembled Top Bottom Impaction Plate $PM_{1.0}$ Top $PM_{4.0}$ Top PM_{10} Top Extra Impaction Plate
	1	801691	Dorr-Oliver Cyclone
 	1	801692 801694	Power Supply – Desktop Power Supply – Handheld
	2	N/A	Stylus When shipped, one stylus will be in the accessory bag, the second stylus attached to instrument.
	1	3012094	Screwdriver, dual ended. (For Handheld Models only)

Item	Qty	Part Number	Description
	1	801674	Impactor Oil
	2	801698	Inlet cap When shipped, one inlet will be in the accessory bag, the second inlet attached to instrument.
	1	801675	External Pump Kit *for 8530EP only*
	1	801797	External Pump Power Cable (to DustTrak monitor) *for 8530EP only*
	1	801798	External Pump Flow Tube (to DustTrak monitor) *for 8530EP only*
	1		Exhaust Adapter, DustTrak monitor *for 8530EP only*

Optional Accessories

The following photos and table list optional accessories. If you ordered optional accessories, make certain they have been received and are in working order.

Accessories	Qty	Part Number	Description
	1	801675	External Pump Kit
	2	801795	DustTrak II/DRX External Pump Service Kit for 8530EP only. Contains two filters for External Pump
	1	801685	Battery Charger, 2-Bay, Battery 801680 for Desktop DustTrak monitor
	1	801686	Battery Charger, Battery 801681 for Handheld DustTrak monitor

Parts Identification for the DustTrak II Desktop Aerosol Monitor Models 8530/8531

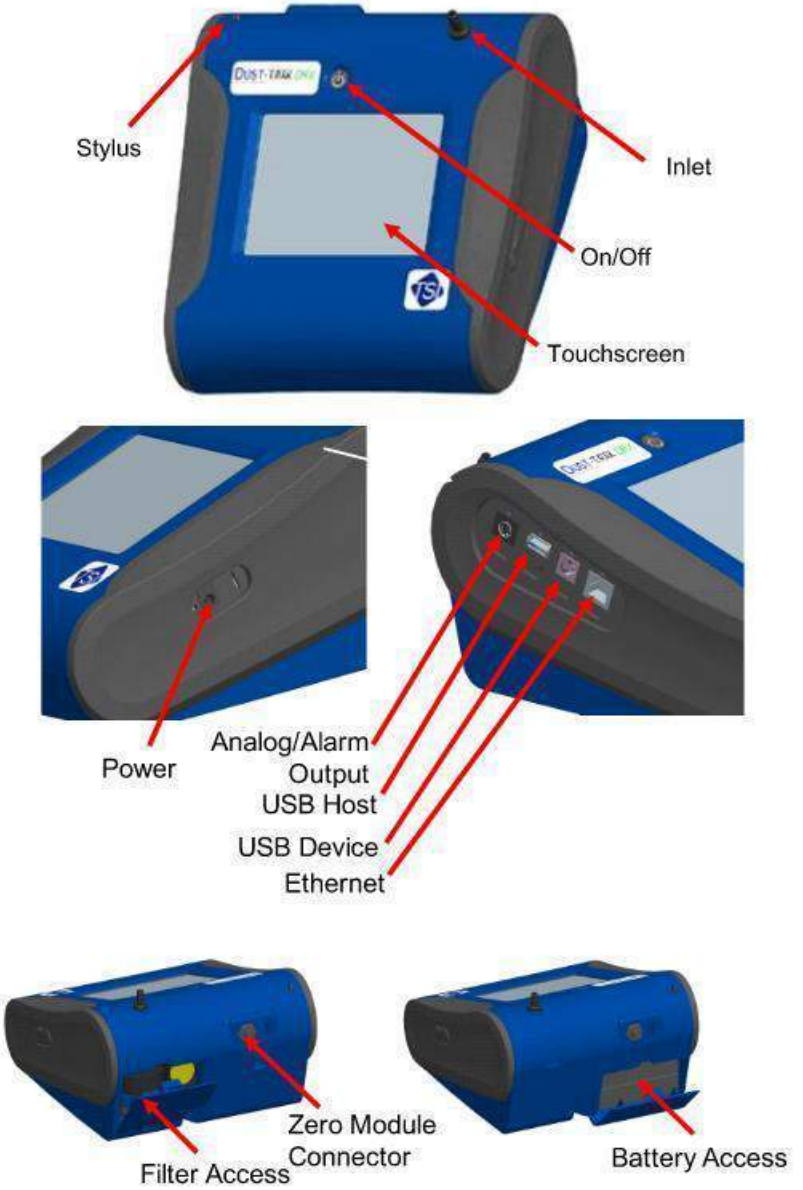
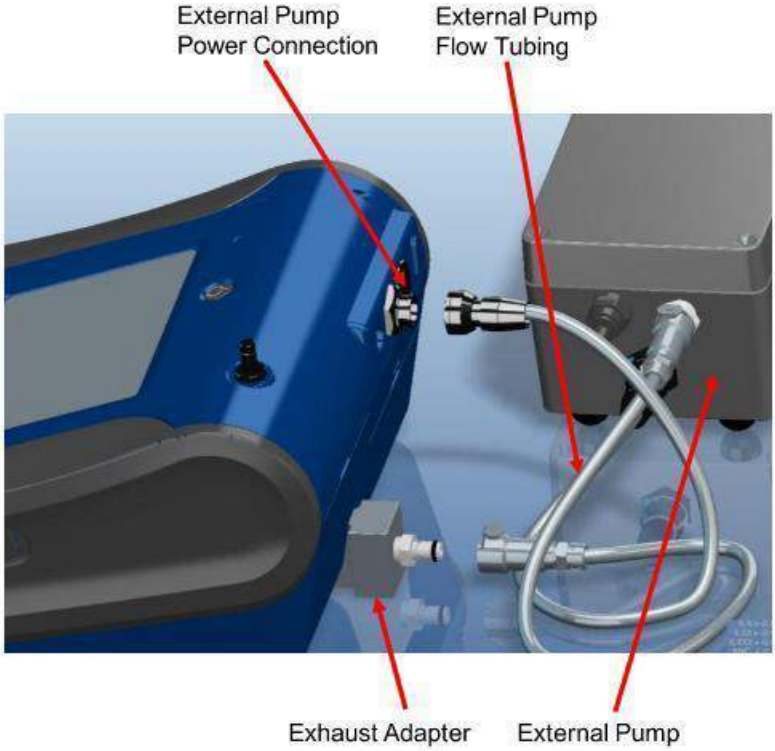


Figure 1-1: Features on Desktop Model 8530/8531

Parts Identification for the DustTrak II Desktop Aerosol Monitor Model 8530EP



External Pump Module (8530EP only)

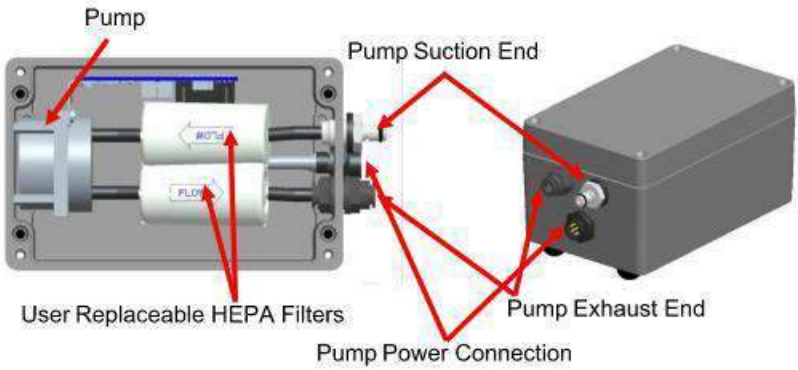


Figure 1-2: Features on Desktop Model 8530EP

Parts Identification for the DustTrak II Handheld Aerosol Monitor Model 8532

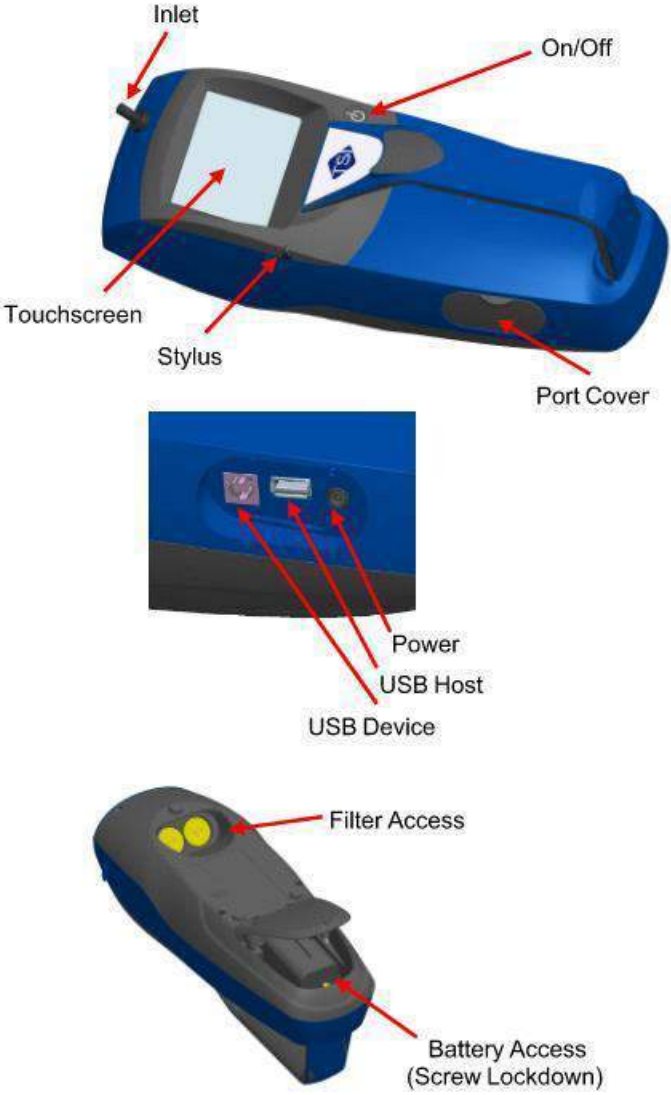


Figure 1-3: Features on Handheld Model

Chapter 2

Setting Up

Supplying Power to the DustTrak II Aerosol Monitor

The DustTrak II Aerosol Monitor must be powered by either batteries or using the external AC adapter.

WARNING

The instrument has been design to be used with batteries supplied by TSI. Do ***not*** use a substitute.

Disposing of old batteries must be recycled in accordance with the local environmental regulations.

WARNING

Do ***not*** use non-rechargeable batteries in this instrument. Fire, explosions, or other hazards may result.

Installing the Batteries in Model 8530/8531/8530EP Desktop

Remove the battery cover and slide one or two batteries into the battery slots. A single battery can be put into either slot. Orient the batteries with the label side facing up (see Figure 2-1).

Figure 2-1: Batteries into Desktop Unit

Installing the Batteries in Model 8532 Handheld

Remove the battery cover by loosening captured screw on the bottom of the unit. Orient battery with brass connectors facing forward. Insert battery into cavity and slide forward to engage into pins. Replace the battery cover and secure by tightening screw (see Figure 2-2).

Figure 2-2: Batteries into Handheld Unit

Connecting the External Pump to DustTrak Model 8530EP

The Model 8530EP is a Desktop DustTrak monitor with an external pump. This DustTrak has no internal pump and will not work with any other external pump other than the one provided by TSI (p/n 801675). The Model 8530EP is intended for applications where the DustTrak is operated continuously over extended periods (several days to months) under wide temperature fluctuations (0 to 50°C). The external pump is designed to be more robust for 24/7 operation of the DustTrak monitor and is warranted to operate continuously for one full year or 8760 hours. The Model 8530EP is ideal for fugitive dust monitoring.

The pump and the DustTrak monitor come separately and require assembly. Follow the steps below to connect the pump with the Model 8530EP DustTrak monitor.

WARNING

Turn the DustTrak monitor OFF before connecting the external pump. Turn the DustTrak monitor ON only after connecting the External Module.

1. Connect the pump end of the quick connect to the pump module (see Figure 2-3).

Figure 2-3: Connect Pump End of Quick Connect to Pump Module

2. Likewise, plug one end of the power connector to the pump module as shown above. Turn the power connector until it clicks and locks in place. This prevents the connector from disconnecting due to vibration or movement.
3. Connect the exhaust adapter to the exhaust of the DustTrak monitor (see Figure 2-4).

Figure 2-4: Connect Exhaust Adapter to Exhaust of DustTrak Monitor

4. Connect the other end of the flow tubing to the exhaust adapter of the DustTrak monitor.
5. Connect the other end of the power connector to the DustTrak monitor (see Figure 2-5).

Figure 2-5: Connect Power Connector to DustTrak Monitor

WARNING

The Pump module design does not allow for installation outdoors without any protection from the elements. Always operate it within an enclosure.

The DustTrak external pump module does not require an A/C adapter. It is always powered off the DustTrak monitor.

Notes
1. The power connector and the flow quick connect "click" when securely connected. The power connector must be rotated clockwise past the locking pin.
2. Do ***not*** hot-plug the External Pump Module when the DustTrak monitor is turned ON. Always connect the External Pump module first and then turn the DustTrak monitor ON.
3. TSI recommends that the DustTrak monitor with the external pump be operated in the Model 8535 Environmental Enclosure.
4. TSI recommends that the pump module be operated when mounted on its feet and avoid operating at other orientations as much as possible.
5. Pump module and the DustTrak monitor should be at the same electrical potential.
6. The additional port on the external pump module is where the pump exhausts the flow. For applications where the DustTrak monitor is sampling from a chamber or a duct at pressures significantly different from the ambient, TSI recommends plumbing the exhaust of the external pump back in to the chamber/duct.

Using the AC Adapter to Run Instrument

The AC adapter allows you to power the DustTrak monitor from an AC wall outlet. When using the AC adapter, the batteries (if installed) are bypassed.

Battery Charging

This instrument will charge the Lithium Ion battery packs. Insert the batteries into the battery compartment, plug the instrument into AC power, and turn the instrument on. Batteries will charge only when the instrument is on and in stand-by mode. Batteries will not charge if the instrument is turned off or is actively taken measurements. Charging will stop when the batteries are fully charged.

WARNING
When Charging Battery the ambient temp must ***not*** exceed 42°C.

Inlet Cap

When using the DustTrak monitor to sample environmental air, the inlet cap should be put over the instrument. This cap will keep large objects from dropping into and plugging the inlet. The cap will also keep direct light from shinning into the chamber and skewing the results.

The inlet cap can simply be pressed onto the instruments inlet.

Figure 2-6: Putting on Inlet Cap

Size-Selective Impactors

Size-selective impactors can be attached to the inlet of the DustTrak II instruments. Size-selective impactors can be used to pre-condition the size range of the particles entering the instrument. PM_1, $PM_{2.5}$, PM_4 (Respirable) and PM_{10} impactors are available. **The instrument must run at the factory default setting of 3.0 L/min for the impactors to achieve the correct cut points.**

The size-selective impactor is composed of three parts; the cap, impaction plate and bottom. Selection of the cap will determine cut size of the impactor. Each cap is labeled with the particle cut size (1 µm, 2.5 µm, 4.0 µm or 10 µm). The same impaction plate and bottom are used on all impactor sizes.

Figure 2-7: Size-Selective Impactor

The impactor assembly is attached to the instrument in place of the inlet cap. The inlet cap does not need to be used if an impactor is being used. See Chapter 4, "Maintenance," for instructions on how to add oil to the impaction plate.

Dorr-Oliver Cyclone

A Dorr-Oliver cyclone is shipped with the instrument. The Dorr-Oliver cyclone removes particles over 4.0 µm in size. The Dorr-Oliver cyclone is attached to the instrument by sliding the cyclone clip over the protruding catch. The tube from the Dorr-Oliver cyclone needs to be routed to the inlet of the instrument.

Figure 2-8: Installing Door-Oliver Cyclone

Do not use Inlet attachments (impactors or inlet cap) when using the Dorr-Oliver Cyclone. **The instrument flow rate must be changed to 1.7 L/min when using the Dorr-Oliver Cyclone in order to achieve a 4 µm (respirable) cut-point.** See the Flow Cal instructions in the Operations chapter for instructions on how to change the instruments flow rate.

Instrument Setup

The DustTrak II monitor can be connected to a computer to download data and upload sampling programs.

Connecting to the Computer

Connect the USB host port of a Microsoft Windows®-based computer to the USB device port on the side of the DustTrak monitor.

Installing TrakPro™ Data Analysis Software

TrakPro software can preprogram the DustTrak monitor, download data, view and create raw data and statistical reports, create graphs, and combine graphs with data from other TSI instruments that use TrakPro software. The following sections describe how to install the software and set up the computer.

Note
To use TrakPro software with the DustTrak Aerosol Monitor, the PC must be running Microsoft Windows® and the computer must have an available Universal Serial Bus (USB) port.

®Windows is a registered trademark of Microsoft Corporation.

1. Insert the TrakPro Data Analysis Software CD into the CD-ROM drive. The install screen starts automatically.

Note
If the software does not start automatically after a few minutes, manually run the program listed on the label of the CD using the **Run** command on the Windows Start Menu.

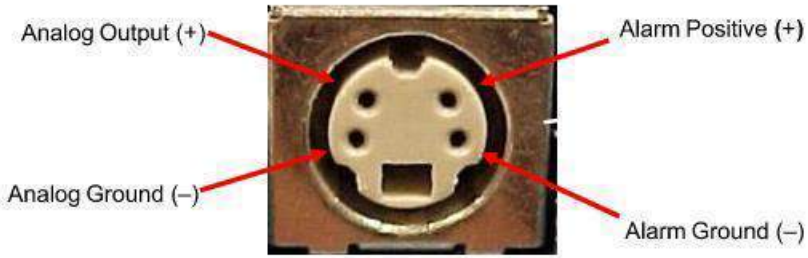
2. Follow the directions to install TrakPro software.

TrakPro software contains a comprehensive installation guide. TSI recommends printing out this guide prior to starting the TrakPro software installation on your computer, so it may be consulted during the installation. The TrakPro Software manual is located in the "Help" file in TrakPro software. There is no separately printed TrakPro Data Analysis software manual.

Connecting Analog/Alarm Output

The Analog/Alarm Output Cable plugs into the alarm connection on the side of the instrument. This feature is on the desktop models (8530/8531) only.

The cable contains a 4-pin, mini-DIN connector. The pin-outs for the connector and the wiring for the cable are shown below.



4-pin miniDIN connector

Cable Wiring Diagram	
Brown Wire	Analog Ground
Orange Wire	Analog Out
Red Wire	Alarm (+)
White Wire	Alarm (-)
Black Wire	Shield

Figure 2-9: Cable Wiring Diagram

®Microsoft and Windows are registered trademarks of Microsoft Corporation.

Wiring the Analog Output

System specifications:

- Output voltage: 0 to 5 VDC. With a maximum output of 15 mA.
- Output Current 4 mA to 20 mA with a maximum load impendence of 250 ohms.
- Correct polarity must be observed (see pin-outs above).

The output cable supplied by TSI (part no. 801652) is labeled with the pin-out wiring diagram. Additional equipment may be needed for making connections to the system that TSI does not supply. It is your responsibility to specify and supply all additional equipment.

Wiring the Alarm

System specifications:

- Maximum voltage: 15 VDC (**DO NOT USE AC POWER**)
- Maximum current: 1 Amp
- Correct polarity must be observed (see pin-outs above)
- The alarm switch, located inside the DustTrak monitor must be located on the ground side of the alarm system.

WARNING

The DustTrak monitor Alarm Output function should ***not*** be used to detect hazardous conditions or to provide an alarm for protecting human life, health or safety.

Caution

The alarm switch must ***not*** be wired to AC power! Failure to install the user alarm properly could damage the DustTrak instrument and/or void the instrument warranty! Please read and follow all instructions before wiring or operating the user alarm.

WARNING

When connected to the analog out and alarm out connector, you **must** use safety certified equipment and/or power sources.

Chapter 3

Operation

Getting Started

The **START UP** screen is displayed initially when the instrument is turned on, following the initial TSI logo splash screen.

Use a stylus or fingertip, touch the "buttons" on the screen to activate different menus.

For Model DustTrak 8530EP only

W A R N I N G

Always setup and operate the DustTrak monitor with External Pump Module with the External Pump Module connected to the DustTrak monitor. Failure to do so will result in communication errors.

Communication errors take place under four different scenarios as follows:

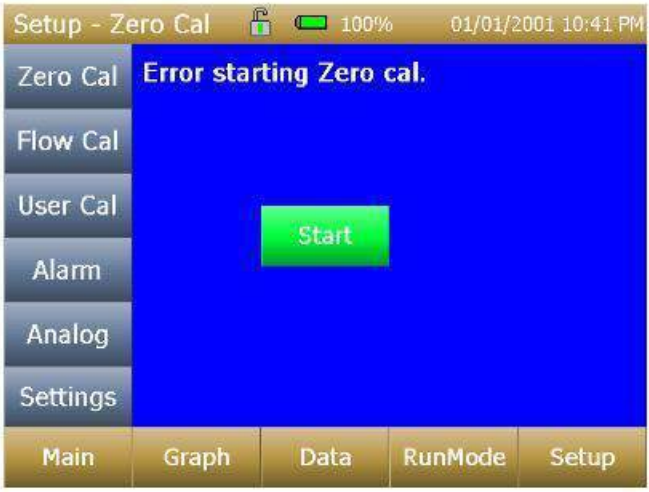
1. When the unit is idle and is ***not*** connected to the External Pump Module, a warning displays on the Main screen.

Note

"No Pump is Connected" is a sticky error. Even after the warning message, if the External Pump Module is connected to the DustTrak, the error will not disappear until the screen is refreshed. Refresh the screen by going into a different menu and returning to the Main menu.

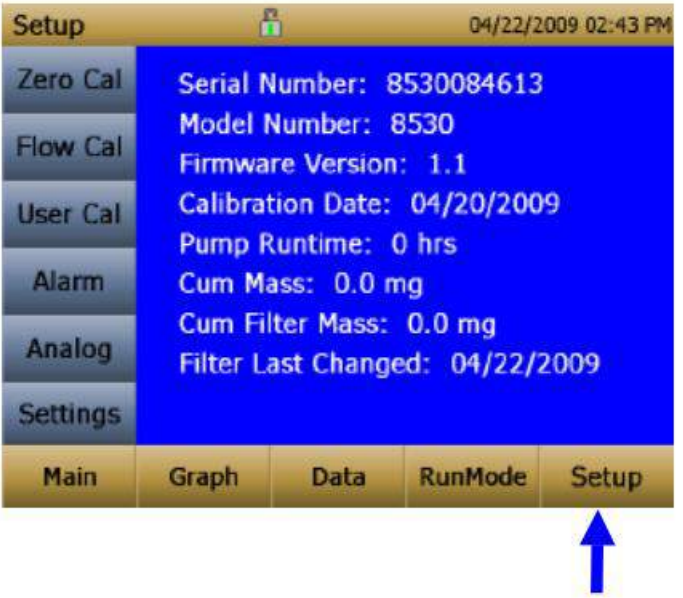
2. When the unit is ***not*** connected to the External Pump Module and an attempt is made to start a run by selecting "Start", an error appears on the Main screen.

3. If the pump is ***not*** connected while attempting to perform a Zero Cal, an error appears on the Setup screen.



4. If the pump is ***not*** connected while attempting to perform a Flow Cal, an error appears on the Setup screen.

Setup Menu

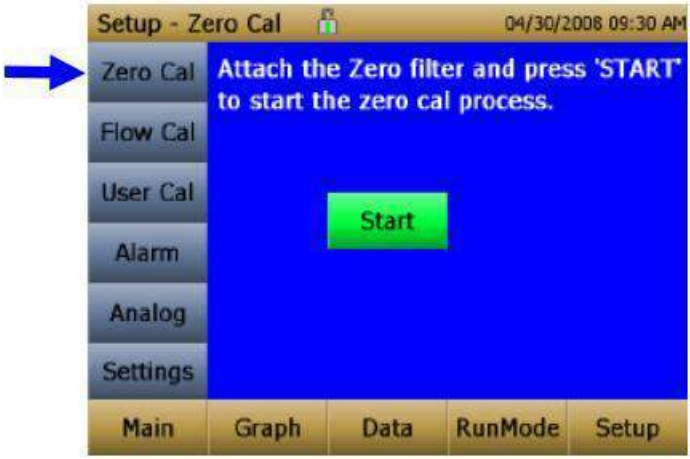


Pressing **Setup** activates the Setup Menu touchscreen buttons along the left edge of the screen. Setup is not accessible when the instrument is sampling.

The main screen of the **Setup** screen displays the following information:

Serial Number	The instruments serial number.
Model Number	The instruments model number.
Firmware Version	Instruments current version of firmware.
Calibration Date	Date of the last factory calibration.
Pump Run Time	Pump running time in hours.
Cum Mass Conc	Amount of mass run through instrument over life.
Cum Filter Conc	Amount of mass run through instrument since last filter change.
Filter Time	Date of last filter change.

Zero Cal



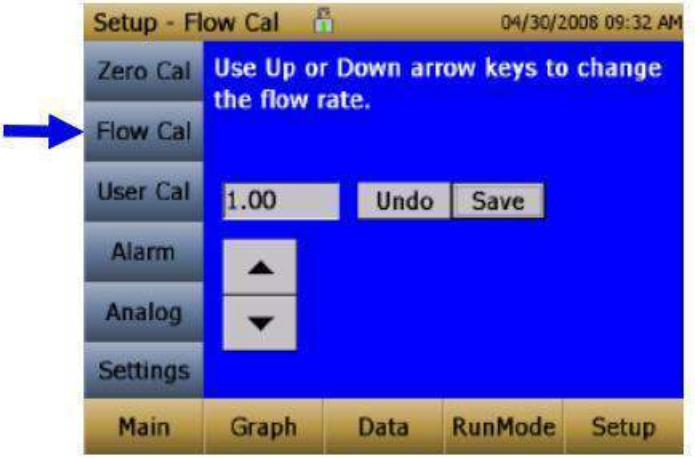
Run **Zero Cal** the first time the instrument is used and repeat prior to every use. Zero Cal requires that the zero filter be attached prior to running. Zero Cal must also be performed if the unit is reading negative concentrations. It is not possible for the DustTrak to read negative concentrations. Negative concentrations are a symptom of zero drift.

Never perform a zero cal without attaching a zero filter.

1. Press **Zero Cal** Button
2. Attach Zero Filter
3. Press the **Start** button to start Zeroing process.
4. A count-down clock will appear indicating the time remaining. The screen with indicate "Zero Cal Complete" when done.

Remove filter after zeroing has been completed. The instrument is now zero calibrated and ready for use.

Flow Cal



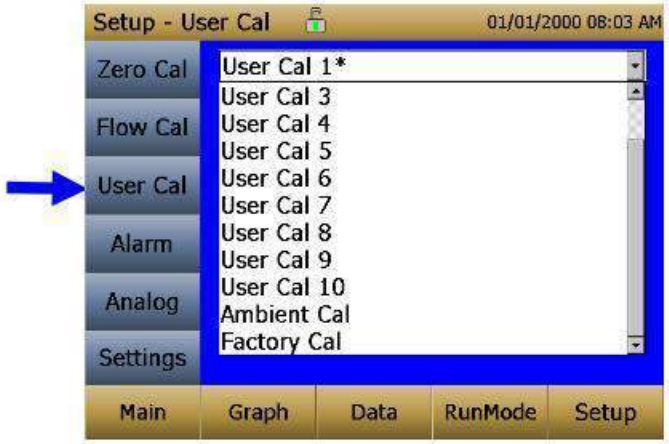
Run **Flow Cal** to change the flow set point. The flow set point is factory set to 3 L/min total flow. 2 L/min of the total flow is measured aerosol flow. 1 L/min of total flow is split off, filtered, and used for sheath flow. There is an internal ΔP flowmeter in the DustTrak II instrument that controls flow rate to ±5% if factory setpoint. TSI recommends checking the flows with an external flow reference meter, especially when collecting data. The pump will automatically start when entering the Flow Cal screen.

1. Attach a flow calibrator (reference flow meter) to inlet port. You may use a bubble buret, mass flow meter, dry piston or rotameter as flow measurement devices.
2. Move the arrows up or down to achieve desired flow on the reference flowmeter. Each up or down arrow will change the flow about 1%. Allow time between button presses to let pump change to the new flow rate.

   Select **Save** once the desired flow rate is achieved. Select **Undo** to return to the factory set point.

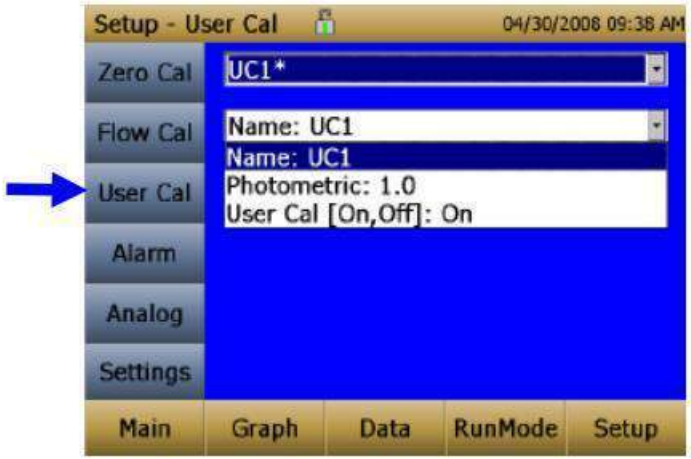
Note
The flow rate can be adjusted from approximately 1.5 to 4.0 L/min. For Model 8533/8534, the FlowCal feature allows you to re-adjust the flow rate to 3.0 L/min. While the flow rate for Model 8533/8534 is fixed at 3.0 L/min, the flow rate for Model 8530/8532 can be changed. This allows for the use of other size selective inlets like cyclones or impactors with Model 8530/8532. No size-selective inlets should be installed on the inlet of Model 8533/8534 during its normal operation.

User Cal



User Cal allows you to store and use 10 different calibration factors. In addition, there are two factory defaults, one is the "Ambient Cal" and the other is the "Factory Cal". The "Ambient Cal" is appropriate for outdoor ambient dust or fugitive dust monitoring. The "Factory Cal" is the calibration to ISO 12103-1, A1 Arizona test dust for which a calibration certificate is provided with the instrument. The "Factory Cal" is appropriate for most workplace aerosol monitoring. The currently active user calibration is highlighted with an asterisk "*".

Four variables can be set for each user calibration.



Name	User can rename calibration to a description name.
Photometric	Changes the factory calibration of particle signal, based on Arizona Road Dust, to actual aerosol being measured. See below for sets to set this calibration.
Size Corr	Changes the factory calibration of the particle distribution, based on Arizona Road Dust, to actual aerosol being measured. See below for sets to set this calibration.
User Cal [on,off]	Selecting **On** will activate current user calibration and deactivate the previously selected user calibration.

Taking a Gravimetric Sample Using the DustTrak Monitor

When sampling with the DustTrak monitor, you can simultaneously take a gravimetric sample either for custom calibration of the DustTrak monitor or for collecting the sample on to the gravimetric filter downstream of the DustTrak monitor without a need for additional gravimetric sampling pump and filter assembly. To accomplish this, follow the instructions given below:

1. Setup the DustTrak monitor to sample how long you want the sample run time to be. The following example shows a sample for 8 hours.
2. Under RunMode menu, put the instrument in Manual Log (Manual Logging is reviewed later in this section), which will enable you to start and stop the pump at any time you choose.
3. Set the logging interval. One minute (i.e., "01:00") is a good choice.
4. Make sure you have a preweighed 37-mm gravimetric filter cassette loaded into the DustTrak monitor. See Chapter 4, "Replacing the Internal Filters" on how to access the filter (see figure 4-8) and replace it.

Note
Use only the conductive plastic filter cassette holder (SKC Part# 225-308).

5. Under the Setup Menu, make sure the DustTrak monitor is set to the desired flow rate. For DustTrak II Models 8530 and 8531, the flows can be varied from 1.7 to 4 L/min for use with various inlet conditioners. For DustTrak DRX Model 8533, ***the flow cannot be changed.*** The flows for DustTrak II monitor can be changed by changing the default flow calibration setpoint from 1.0 to any value between 0.5 to 1.5 in the span adjustment. An external flowmeter is needed to measure the total flow. Flow can be changed by clicking on the UP or DOWN arrow keys shown below:

6. Conduct a preflow calibration on the DustTrak monitor using the same kind of sample media you will sample with. Now, attach the sample media you intend to sample with and start sampling aerosol for the desired time. After the desired run time, stop the sampling. Remove the filter from the DustTrak monitor and follow your laboratory's criteria for filter post weight. Conduct a post-flow calibration with the same sample media done with the pre-flow calibration and determine if these flow calibrations are within ±5% of each other. If they are, use the following to calculate the actual flow rate for the DustTrak monitor. The laboratory will need the following information to calculate mass concentration in mg/m^3:

   - Total sample time in minutes.
   - Flow rate—flow rate of the DustTrak monitor used for gravimetric analysis is only $^2/_3$ the total flow since $^1/_3$ of the flow is used as sheath flow.
   - Total liters of air sampled = total sample time x flow rate.

7. Using this information the laboratory can determine the concentration using the following formula:

$$Concentration, \frac{mg}{m^3} = \frac{\{Filter\ Post\ Weight\ (mg) - Filter\ Pre\ Weight\ (mg)\}}{\frac{2}{3} \times \frac{\{DustTrak™\ Monitor\ Flow\ Rate\ (L/min)\}}{1000}} \times Total\ Sample\ Time\ (min)$$

Note
The flow rate used for gravimetric analysis is only $^2/_3$ the total flow since $^1/_3$ of the flow is used as sheath flow.

8. For instructions on how to calibrate the DustTrak monitor using this data, see section below on "Determining the Calibration Factor for a Specific Aerosol".

Photometric Calibration Factor

In most situations, the DustTrak monitor with its built-in data logging capability can provide very good information on how the concentration of an aerosol changes for different processes over time. Factory calibration to the respirable fraction of standard ISO 12103-1, A1 test dust is fairly representative of a wide variety of workplace aerosols. Because optical mass measurements are dependent upon particle size and material properties, there may be times in which a custom calibration would improve your accuracy for a specific aerosol.

Determining a aerosol specific photometric calibration requires that you determine a true mass concentration (e.g., gravimetric analysis) for the aerosol you want to measure. The true mass concentration is used to calculate the custom calibration factor for that aerosol. Once you have a custom calibration factor, you can reuse it each time you make measurements in the same aerosol environment.

Determining the Calibration Factor for a Specific Aerosol

The DustTrak II monitor is factory calibrated to the respirable fraction of standard ISO 12103-1, A1 test dust. The DustTrak monitor can be easily calibrated to any arbitrary aerosol by adjusting the custom calibration factor. The DustTrak monitor's custom calibration factor is assigned the value of 1.00 for the factory calibration to standard ISO test dust. This procedure describes how to determine the calibration factor for a specific aerosol. Using the value of 1.00 will always revert back to the factory calibration.

To determine a new calibration factor you need some way of accurately measuring the concentration of aerosol, hereafter referred to as the reference instrument. A gravimetric analysis is often the best choice, though it is limited to nonvolatile aerosols. The internal 37 mm filter cartridge, in the desktop units, can be used to collect the reference gravimetric reference sample.

To make an accurate calibration you must simultaneously measure the aerosol concentration with the DustTrak monitor and your reference instrument.

1. Zero the DustTrak II monitor.
2. Put the instrument in Manual Log (Manual Logging is reviewed later in this section).
3. Set the logging interval. One minute (i.e., "01:00") is often a good choice.
4. Co-locate the DustTrak II monitor and the reference sampler together so that they are measuring from the same area. The 37-mm filter cartridge in the desktop unit can be used to collect the particles to be weighed for the gravimetric reference.
5. Start sampling aerosol with both instruments at the same time.

Note
Greater accuracy will be obtained with longer samples. The time you permit for sampling often depends on the reference instrument and characteristics of the measured aerosol. It may take some time to collect sufficient aerosol onto a filter cassette for accurate gravimetric analysis. Refer to instructions of your reference instrument for sampling times.

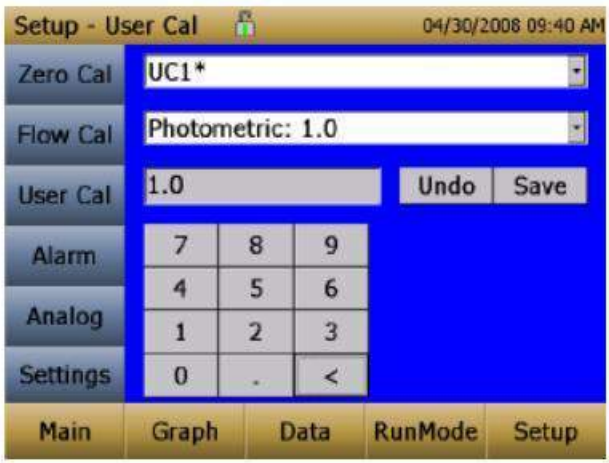
6. Stop sampling with both instruments at the same time.
7. Record the DustTrak monitor average concentration by viewing the sample average in the Data screen. (Data Screen is reviewed later in this chapter.)
8. Determine the mass concentration in mg/m^3 from your reference instrument. For gravimetric sampling this means weighing the gravimetric sample.

Note
If you used the internal gravimetric filter in the DustTrak Model 8530 or 8531, the flow rate used to compute the concentration should be 2 L/min, not 3 L/min since only 2 L/min of aerosol flow reaches the filter.

9. Compute the new calibration constant, NewCal, using the following formula:

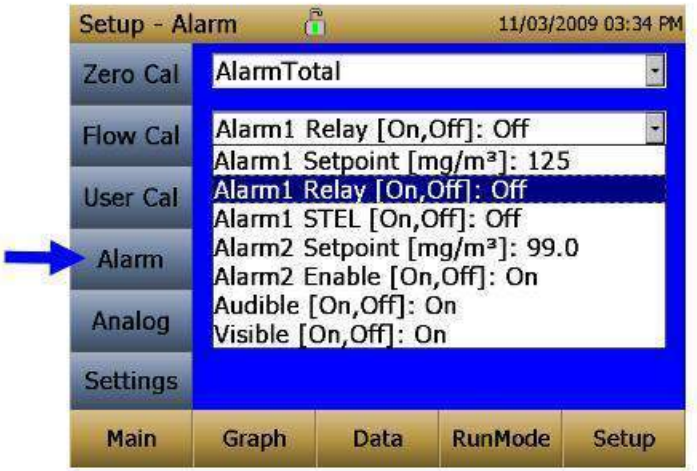
$$NewCal = \left(\frac{\text{Reference Concentraton}}{\text{DustTrak Concentraton}} \right) \cdot CurrentCal$$

10. Select **Photometric** from the User Cal drop down selection and enter the NewCal factor using the onscreen controls.



Alarm

Alarm allows you to set an alarm level that will be triggered if the instrument's reading goes above the setpoint. However, the alarm functioning is determined by the logging interval. The alarm will turn ON only if the average concentration over the logging interval exceeds the set point. If the logging interval is too long and the concentration exceeds the set point and stays at that level, the alarm will not turn ON until after the logging interval has passed. Likewise, the alarm will not stop until after the concentration has dropped below 5% of the threshold and after the logging interval has passed.



Note

The Alarm is dependent on the logging interval. For the DustTrak to alarm as soon as the Alarm Setpoint is exceeded, the logging interval must be set as low as possible (i.e., 1 second or 2 seconds). If a long test duration does not permit setting such a short logging interval, use the STEL alarm instead. The STEL is always based on 1 second concentrations and is independent of the logging interval. For more details on the STEL alarm, see section below on STEL.

In Survey mode, the alarm is dependent on the time constant.

Alarm1 Setpoint [mg/m^3]	The alarm1 setpoint is the mass concentration level upon which the alarm1 is triggered. Alarm will trigger if the mass concentration, taken at the logging interval, rises above the setpoint. ***Note**: Alarm 2 must be lower than Alarm 1 when both alarms are enabled.*

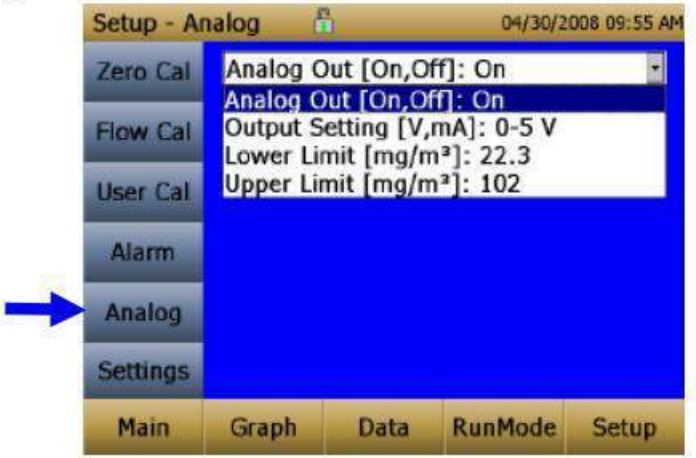
Alarm1 Relay [On, Off]	When the relay alarm is turned on, unit will close relay switch when Alarm1 level is surpassed. Relay selection is available on the 8530 and 8531 desktop models only.
Alarm1 STEL [On, Off]	When the STEL alarm is turned on, STEL data will be collected when Alarm1 level is surpassed. STEL selection is available on the 8530 and 8531 desktop models only. See STEL Note below.
Alarm2 Setpoint [mg/m^3]	The alarm2 setpoint is the mass concentration level upon which the alarm2 triggers. Alarm triggers if the mass concentration, taken at the logging interval, rises above the setpoint. ***Note**: Alarm 2 must be lower than Alarm 1 when both alarms are enabled.*
Alarm2 Enable [On, Off]	Enables Alarm2 to be logged and will activate the Audible or Visible alarms if they are enabled.
Alarm Audible [On, Off]	When the audible alarm is turned on, the instrument will activate internal beeper when Alarm1 or Alarm2 level is surpassed.
Alarm1 Visible [On, Off]	When the visible alarm is turned on, unit will show the alarm icon (Alarm1  , Alarm 2 ) in title bar when Alarm1 or Alarm2 level is surpassed.

STEL Alarm

STEL stands for **S**hort **T**erm **E**xposure **L**imit. When a STEL alarm is selected, the instrument will inspect the data on a second by second basis, independent from the selected logging interval. If the mass exceeds the STEL limit, then a STEL even triggers and the following actions will be taken.

STEL indicator	The STEL indicator STEL will show Red on the main screen.
Data	Data will be taken of the STEL alarm channel at a 1 minute logging interval for **15 minutes**. This data will be stored in a separate file named STEL_XXX, where XXX will be matched to the logged data file. The instrument will also continue to log the mass concentration data at the logging interval selected.
STEL Alarm repeat	If the instrument remains over the STEL limit after the 15 minute interval, or if the instrument exceeds the STEL limit later during the sample period, additional STEL files will be generated.

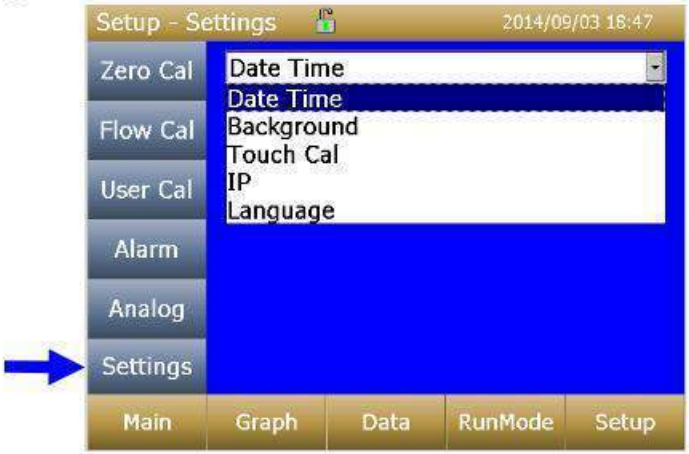
Analog



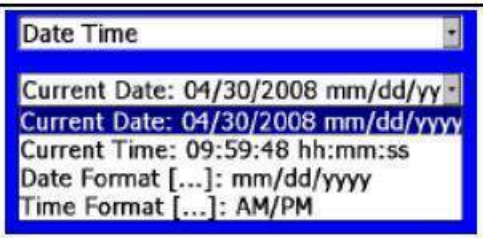
Analog setup screen sets the parameters that will drive the analog out port. Applies to the 8530/8531 Desktop models only.

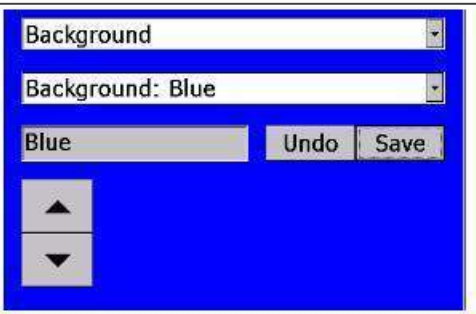
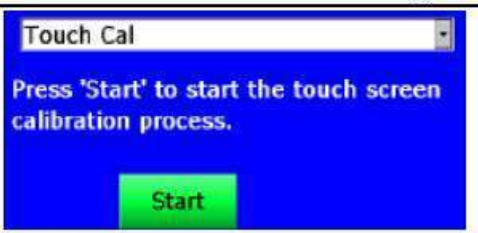
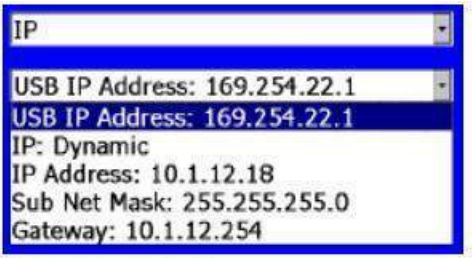
Analog out [On, Off]	Turns analog out port on.
Size Fraction	Selects the size channel that will drive the analog out.
Output Setting [V, mA]	Select between 0 to 5 V and 4 to 20 mA.
Lower Limit [mg/m^3]	Mass concentration reading of the selected channel that will correspond to 0 V or 4 mA.
Upper Limit [mg/m^3]	Mass concentration reading of the selected channel that will correspond to 5 V or 20 mA.

Settings



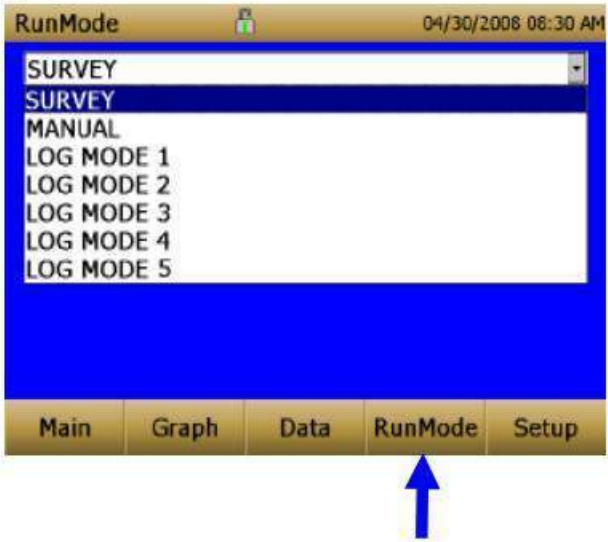
Settings screen sets basic unit parameters.

Date Time	 Sets current date, current time and date/time format. Time can be set in 12 or 24 hour format. Date can be set in yyyy/dd/mm, yyyy/mm/dd or yyyy/dd/mm.

Background	 Switches between blue and white backgrounds.
Touch Cal	 Calibrates the touch cal screen.
IP	 **USB PORT IP Address**: USB IP is the address assigned to the instrument by the NDIS driver. It is shown but cannot be changed. **Ethernet Port IP parameters**: (Model 8530, 8531 Desktop only.) IP method can be set to static or dynamic. For static IP, IP address, default gateway, and subnet mask can be set. For Dynamic, The IP assigned by the network is shown. This cannot be changed. See Note below.
	IP Note After changing the instrument to Dynamic or Static, reboot the instrument. In Dynamic Mode, the unit will show the IP to which is assigned (after being rebooted).

Language	 Switches between display languages. After changing the display language, reboot the instrument.

Run Mode

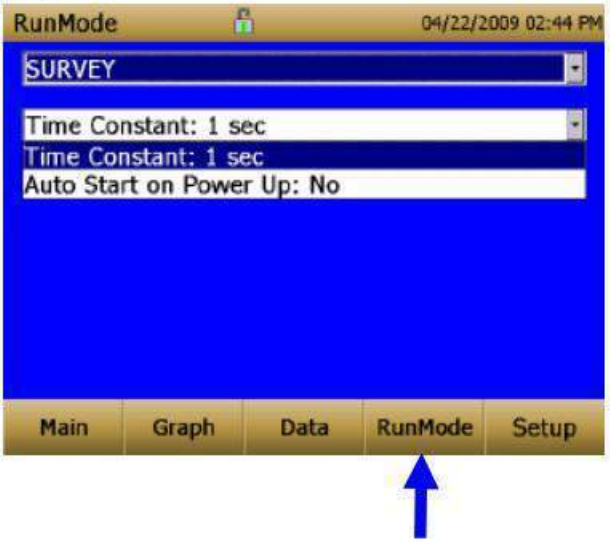


The **RunMode** tab brings up sampling mode options.

Sampling mode options include **Survey Mode**, **Manual Log**, and **Log Mode 1-5**.

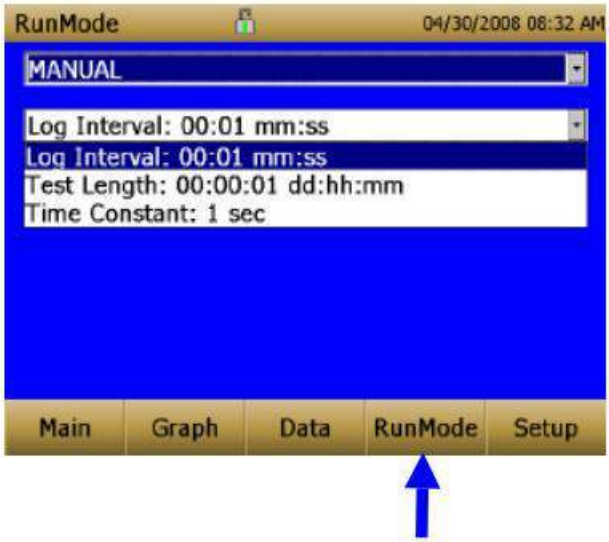
Survey	Survey Mode runs a real time, continuous active sample, but does not log data.
Manual	Manual Log sets the instrument to log data for a specified run time.
Log Modes	Log Mode starts and stops the instrument at specified times, run for a specified test length, and perform multiple tests of the same length with a specified time period between tests.

Survey Mode



Time Constant	Time Constant can be set from 1 to 60 seconds. This will control the update rate of the main screen. It is the rolling average of data displayed on the main screen and is not linked to logged data in either Manual or Program Log modes.
Auto Start on Power Up	When set to "Yes", unit will start a measurement upon being powered on, if the unit was set to "Survey" when it was turned off. When set to "No", the unit will be in idle when it is powered on.

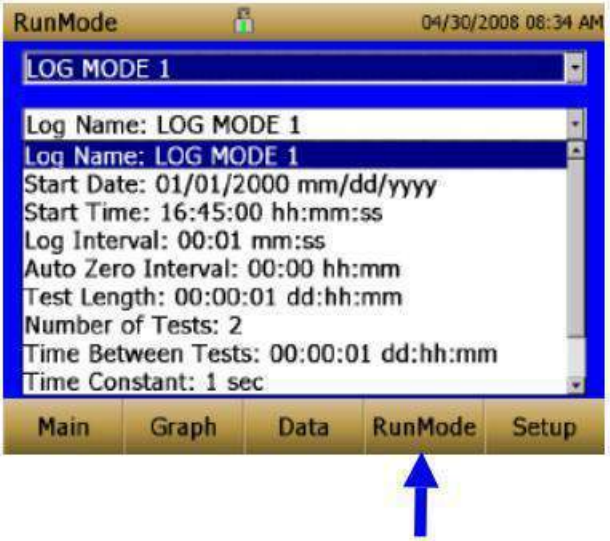
Manual Mode



Log Interval	The log interval can be set from 1 second to 60 minutes. It is the amount of time between logged data points.
Test Length	Test length can be set from 1 minute to the limit of the data storage.
Time Constant	Time Constant can be set from 1 to 60 seconds. This will control the update rate of the main screen. It is the rolling average of data displayed on the main screen and is not linked to logged data in either Manual or Program Log modes.

In Manual mode, data will be stored to a file named "*Manual_XYZ*" where *XYZ* is an incrementing integer.

Log Mode (1–5)



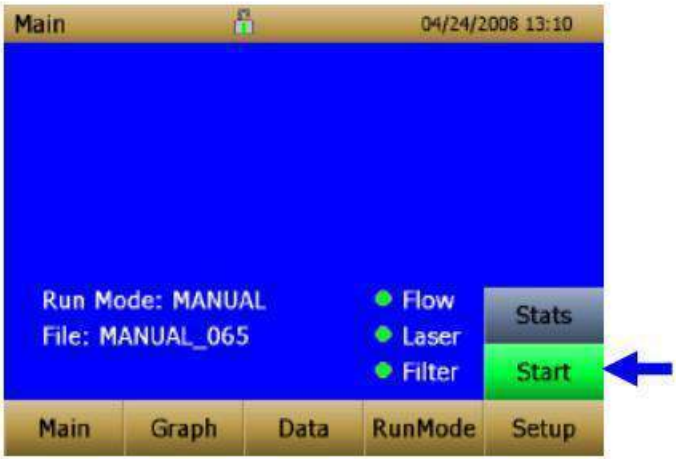
Log Name	Log Name, brings up a virtual keypad to name the Logged Data file.
Start Date	Start Date, select the date the test will start.
Start Time	Start Time, select the time the test will start.
Log Interval	The log interval can be set from 1 second to 60 minutes. It is the amount of time between logged data points.
Auto Zero Interval	Interval between re-zeroing the instrument using the Auto-Zero accessory. Models 8530 and 8531 desktop only.
Test Length	From 1 minute to the limit of the data storage.
Number of Tests	Number of tests, 1 to 999.
Time between Tests	Time between tests, 1 minute to 30 days.
Time Constant	Time Constant can be set from 1 to 60 seconds. This will control the update rate of the main screen. It is the rolling average of data displayed on the main screen and is not linked to logged data in either Manual or Program Log modes.
Use Start Date	Use Start Date, option to use programmed start date or by pass programmed start date.
Use Start Time	Use Start Time, option to use programmed start time or bypass programmed start time.

In Log mode, data will be stored to a file named "*LogName_XYZ*" where *LogName* is the user entered log name and *XYZ* is an incrementing integer.

Taking Mass Concentration Measurements

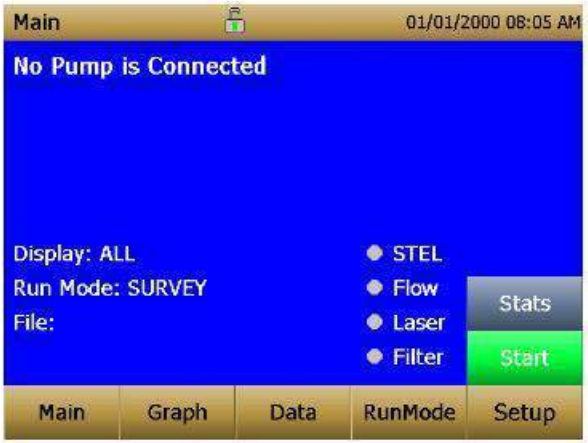
Measurements are started and controlled from the main screen.

Prior to starting a measurement the instrument should be zeroed from the **Setup** screen and the run mode should be configured and selected from the **RunMode** screen.



When the instrument is on, but not taking any mass measurements the start button will be green and instruments pump will not be running. To start taking a measurement, press the green start button.

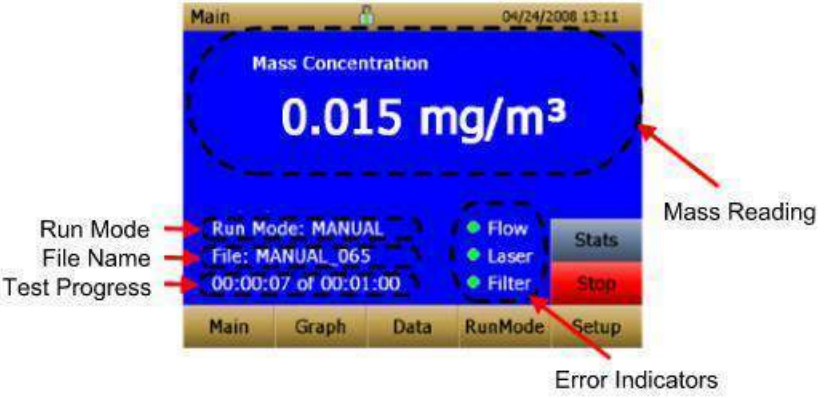
For the Model 8530EP DustTrak monitor with external pump, make sure the external pump is connected to the DustTrak monitor as described in Chapter 2. If the pump is not connected and the green start button is pressed, the DustTrak monitor will identify that the pump is not connected and a warning will be displayed as shown below:



Connect the External Pump Module to the DustTrak monitor and then try again. TSI recommends powering down the DustTrak monitor before connecting the External Pump Module to the DustTrak monitor. Connect the power cable and the flow tubing between the DustTrak monitor and the External pump module, as applicable.

While taking a measurement the screen will display the current measured mass concentration. The various regions of the screen are shown below.

Screen Regions



Mass Reading	Shows the instruments mass measurements.
Run Mode Region	Shows the run mode selected from the RunMode screen.
File Name Region	Displays the file name to which the data is currently being saved.
Test Progress Region	Shows the time-based progress of the test.
Error Indicator Region	Shows the current stats of the instrument STEL: Shows if STEL is in progress (desktop instruments only) Flow: Status of the flow control Laser: Status of the Laser Filter: Status of the Filter See Chapter 5, "Troubleshooting," to resolve any of these error conditions.

Stats

The Stats button shows the statistics of the mass measurement. When the Stats button is pressed, the main mass reading will reduce in font size, and the measurement statistics will show on the right side of the screen.

Graphing

During sampling, pressing the **Graph** button displays current readings in graphical form.

- During Survey Mode, five (5) minutes of running real-time data is displayed graphically.
- During Logging Mode, the entire log test time is displayed on the graph.

Time Display	Pressing the **Time** x-axis label on the graph screen switches between **Time (s)**, **Time (abs)**, and **Time (rel)**. **Time (s):** Elapsed time from first logged point (log interval) to the last logged point (test length). **Time (rel):** Relative time from zero to last logged point (test length – log interval). **Time (abs):** Absolute time from first logged point (test start + log interval) to last logged point (test stop).
Scale Display	Pressing in the Scale Display area will bring up a dialog that will allow changing between auto scaling and user scaling of the Y-axis. 
Data Region	Pressing the data region will bring up a dialog to show TWA or Average lines.  **TWA:** Will show a secondary line on the graph showing the time weighted average of the data. This line will not show if test time is less than 15 minutes. **Average:** Show a secondary line on the graph of the running average of the data.

In Graphing Mode, pressing **Main** returns the instrument to the Main Screen display.

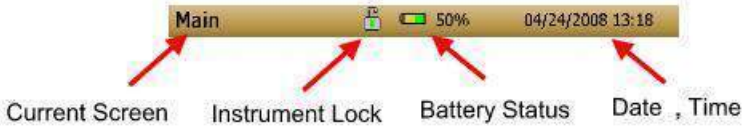
Viewing Data

The **Data** button opens a list of data files for viewing.

Select File	Press the arrows on the right side of the screen to scroll up or down to the data file to be viewed.
Data Statistics	Statistics on the selected file o File Name o Sample Average o Sample TWA o Sample Maximum Reading o Sample Minimum Reading o Number of Data Points in the File
Save All Button	Downloads data to a USB thumb drive. The USB thumb drive must be attached to the USB host port. Data is saved as a .csv file that can be viewed in Microsoft® Excel® spreadsheet software.
Delete Button	Deletes the currently highlighted file.
Delete All Button	Deletes all the files stored on the instrument.
Graph Button	Data can also be viewed in graphical form by pressing the **Graph** button while the data file is highlighted.

Title Bar

The Title Bar shows common instrument information.



Current Screen	Title of the current screen that is being displayed.
Instrument Lock	Icon shows if the instrument touchscreen is in an unlocked or locked condition. Unlocked:  Locked:  To lock the touchscreen controls, touch the "lock" icon, immediately followed by three (3) quick touches on the current screen (**Main)** word along the top tool bar. Repeat the process to unlock the screen.
Battery Status	Show the current % life of the battery and show if the battery is currently being charged: Charging:  (unfilled portion of the icon is filled yellow as well as animated to indicate that the charging is in progress) Not Charging:  (unfilled portion of the icon transparent)
Date and Time	Indicates the instruments current date and time.
Alarm	If the instrument is in an alarm status, an alarm icon  will appear in the title bar.

Chapter 4

Maintenance

The DustTrak II aerosol monitor can be maintained in the field using the instructions below. Additionally, TSI recommends that you return your DustTrak II monitor to the factory for annual calibration. For a reasonable fee, we will quickly clean and calibrate the unit and return it to you in "as new" working condition, along with a Certificate of Calibration. This "annual checkup" helps ensure that the DustTrak II monitor is always in good operating condition.

WARNING

There are no user-serviceable parts inside this instrument. The instrument should only be opened by TSI or a TSI approved service technician.

Maintenance Schedule

The DustTrak II Aerosol Monitor requires maintenance on a regular basis. Table 4–1 lists the factory recommended maintenance schedule.

Some maintenance items are required each time the DustTrak monitor is used or on an annual basis. Other items are scheduled according to how much aerosol is drawn through the instrument. For example, TSI recommends cleaning the inlet sample tube after 350 hours of sampling a 1 mg/m^3 concentration of aerosol. This recommendation should be pro-rated according to how the instrument is used. 350 hours at 1 mg/m^3 is the same amount of aerosol as 700 hours at 0.5 mg/m^3 or 175 hours at 2 mg/m^3, etc.

Table 4–1. Recommended Maintenance Schedule

Item	Frequency
Perform zero check	Before each use.
Clean inlet	350 hr. at 1 mg/m^3*
Clean 2.5 µm calibration impactor	Before every use.
Replace internal filters	350 hr. at 1 mg/m^3* or when indicated by the main screen filter error indicator.
Return to factory for cleaning and calibration (For 8530EP, TSI recommends that both the DustTrak and the External Pump Module be returned to TSI)	Annually
Replace the internal HEPA filters in the External Pump module	Annually

*Pro-rated, see discussion above.

The DustTrak monitor keeps track of the accumulated amount of aerosol drawn through it since its last cleaning. When the internal filter replacement is due, the filter error indicator will turn from green to red.

TSI recommends you perform a zero check prior to each use for the DustTrak monitor and certainly before running any extended tests, and after the instrument experiences a significant environmental change. Examples of significant environmental changes would be ambient temperature changes that exceed 15°F (8°C) or moving from locations with high aerosol concentrations to low concentrations.

Zeroing Instrument

1. Attach the zero filter to the inlet of the instrument.

Figure 4-1: Attach Zero Filter to Inlet

2. Follow zero calibration instructions detailed in the operations section of this manual.

Cleaning the Inlet

The inlet should be cleaned based on the schedule in Table 4–1.

1. Turn the DustTrak monitor off.
2. Unscrew the inlet nozzle from the instrument (Figure 4-2).

Figure 4-2: Unscrew Inlet Nozzle

3. Clean the inlet port. Use a cotton swab to clean the outside of the inlet port. You may dampen the swabs with water or a light solvent (e.g., isopropanol). Clean the inside of the sample tube by using a small brush, along with a light solvent. Dry the tube by blowing it out with compressed air, or let it air-dry thoroughly.

Note
Be *careful* not to blow particles into the DustTrak monitor inlet port.

Figure 4-3: Do NOT Blow into Instrument

4. Screw (hand-tighten) inlet back into instrument.

Cleaning and Oiling Impactors

The calibration impactor should be cleaned prior to every use, using it to perform a Standard Calibration (size correction) on the instrument, as described in the Operations section.

1. Unscrew Impactor. Check O-ring on the impactor base.
2. Clean outside and inside of Impactor and the impactor plate using a clean brush and a light solvent. Dry impactor parts by blowing it out with compressed air, or let it air-dry thoroughly.
3. Apply 2 drops of oil (included) to the impactor plate. Do ***not*** over-fill impaction plate.

Figure 4-4: Apply 2 Drops of Oil to Impactor Plate

4. Screw (hand-tighten) impactor back together.

Replacing the Internal Filters

Replace the internal filters based on the schedule in Table 4–1 or when the filter indicator on the main screen changes to red.

1. Turn the instrument off.
2. Remove old filters from the instrument.

Handheld Model

a. Use the enclosed filter removal tool (PN 801668) tool to unscrew the two filter caps located on the bottom of the instrument.

b. Pull the old filters out of the two filter wells. If filter wells are visibly dirty, blow out with compressed air.

Figure 4-5: Pull Filters Out of Two Filter Wells (Handheld Model)

c. Put two (2) new filters (P/N 801666) into the filter wells and screw filter caps back into place.

Note
Replacement filters were shipped with the new instrument. Order additional filters from TSI under PN 801666.

Desktop Model

a. Open filter access door on the back of the instrument.

b. Use the enclosed filter removal tool (PN 801668) to unscrew filter cap.

c. Pull out single cylindrical filter from filter well. If filter well is visibly dirty, blow out with compressed air.

Figure 4-6: Pull out Single Cylindrical Filter from Filter Well (Desktop Model)

d. Put new filer (P/N 801673) back into filter well and screw filter cap back into place.

e. Open blue retention clip by pinching ends inward and pushing down.

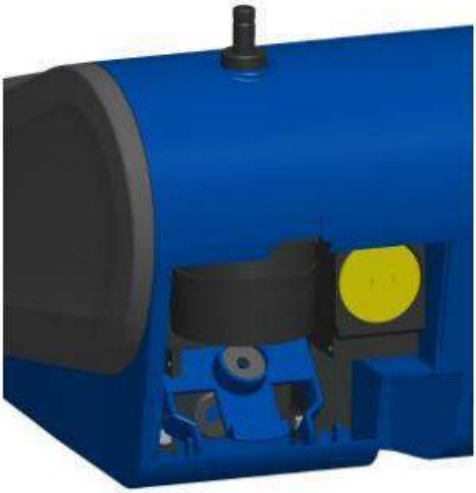


Figure 4-7: Open Blue Retention Clip

f. Remove 37-mm filter cassette by pulling downward and outward.

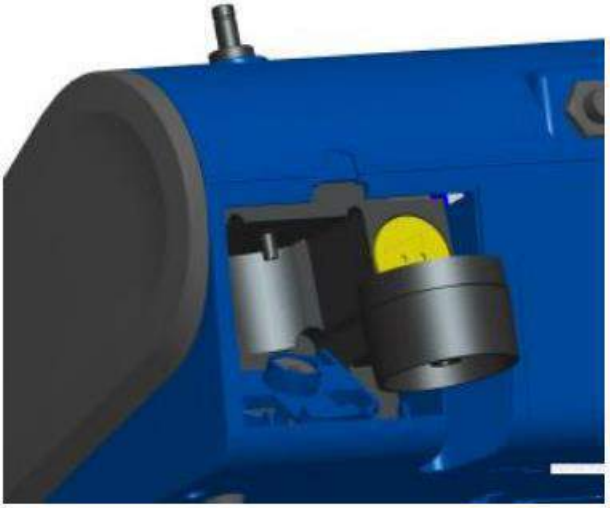


Figure 4-8: Remove 37-mm Filter Cassette

g. Open filter cassette using enclosed tool PN 7001303.

Figure 4-9: Open Filter using Enclosed Tool

h. Remove screen mesh from filter cassette and blow out using compressed air. Blow in reverse direction to remove captured particulate.

i. Replace mesh in filter cassette and press halves together. Make sure filter has been fully closed. The filter tool PN 7001303 can be used to ensure the filter is fully closed.

Figure 4-10: Replace Mesh in Filter Holder

j. Place filter cassette back into position and close blue retaining clip. Make sure retaining clip snaps back into place.

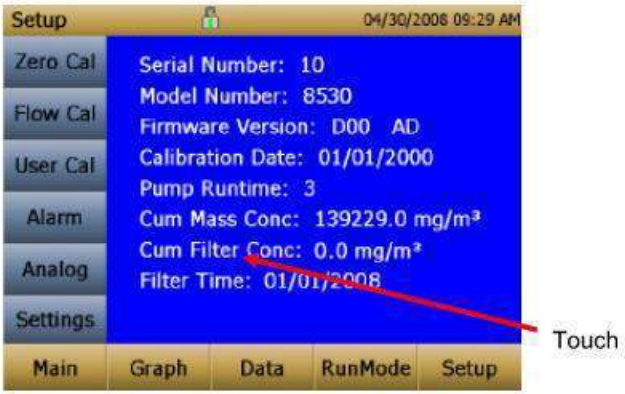
Notes
Replacement filters (HEPA and 3-mm Filter Cassette with mesh filter) were shipped with the new instrument. Order additional filters from TSI under PN 801673. TSI ***does not*** supply any filter media for the filter cassette. Any commercially available 37-mm filter media may be used with the DustTrak II or DRX desktop instruments to collect gravimetric reference samples.

3. **It is important to reset the instruments filter counter after replacing filters. Resetting the counter will clear the filter error condition shown on the main screen.** Reset the counters by the following:

   a. Turn on the instrument.

   b. Press the **Setup** button to go into the setup screen.

   c. Touch the **Cum Filter Conc:** (live key) to reset the aerosol mass.



   d. *Replace user serviceable filters?* Dialog will appear. Press **OK**.

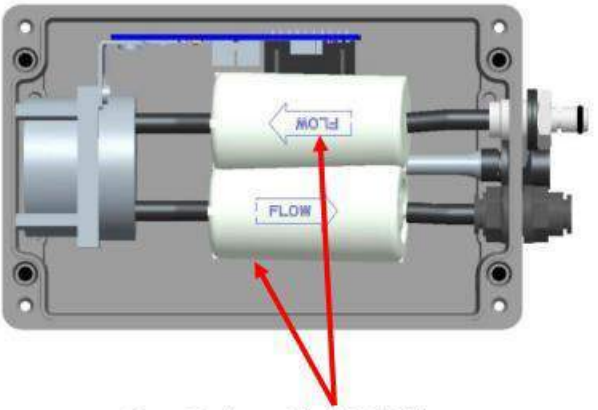
   e. *Reset filter concentration?* Dialog will appear. Press **Yes** to reset the cumulative filter concentration to zero.

   f. The Setup screen will not show zero for the **Cum Filter Concentration and** the current date for the **Filter Time**.

Replacing the Filters in the External Pump Module

The external pump module provided with Model 8530EP is designed to run continuously for about a year (8760 hours). There are two HEPA filters that protect the pump from contamination—one on the suction side of the pump and the other on the discharge side of the pump. The discharge side of the pump collects particles shedding from the vanes of the pump and will turn black over time. The HEPA filters will have to be replaced once a year.

To access the filters open the top cover of the pump module. The two HEPA filters are identified in the figure below. The two filters can be replaced by disconnecting the soft tubing between the filters, pump, and the casing connectors.



User Replaceable HEPA Filters

Caution

When replacing the HEPA filters, make sure they are oriented in the correction direction as shown in the picture above.

Storage Precautions

When storing the DustTrak monitor for more than 30 days, you should charge and remove the batteries. This prevents damage due to battery leakage.

This instrument must be stored in a location where the temperature remains between –20 and 60°C (–4 and 140°F).

Chapter 5

Troubleshooting

The table below lists the symptoms, possible causes, and recommended solutions for common problems encountered with the DustTrak II monitor.

Symptom	Possible Cause	Corrective Action
Erratic zero reading.	Leak	Check connections for leaks Replace zero filter
	Dirty inlet port and/or sample tube	Clean inlet port. Clean or replace tubing
	Internal filter(s) not installed properly (leaking)	Inspect internal filter wells to make certain the filters and o-rings are seated properly. Replace internal filters if necessary
DustTrak reading negative concentrations	Zero Drift	Perform Zero Cal
	Zero Cal was performed without the Zero Filter in-line	Perform Zero Cal again and make sure the Zero Filter is attached to the DustTrak inlet
Error completing Zero Cal	Too much light scatter in the optics chamber due to dust deposits	Clean the inlet nozzle. Attach the zero filter and sample for about 2 minutes. During sampling, pulse the flow going into the DustTrak monitor by intermittently plugging the zero filter. Any dust in the optics chamber will break loose during flow pulsations and will be cleared out by the pump Perform Zero Cal again. If the Zero Cal still cannot be performed, factory service may be required

Symptom	Possible Cause	Corrective Action
Run Mode Error: The start time has passed	The selected Run Mode program has "Use Start Date" selected, but the start date is prior to the current date	Correct or change the run mode program
Run Mode Error: The selected log mode will exceed the allowed number of samples	The selected Run Mode program is programmed to save more samples then is room in memory	Reduce the number of samples by reducing the test length or increasing the logging interval
Instrument runs slow	Large amount of data in memory	Large data files or many small data files will cause instrument to slow, due to need to read and display large amounts of data
No display	Unit not switched on	Switch unit on
	Low or dead batteries	Recharge the batteries or plug in the AC adapter
No touch - screen response	Instrument currently busy	The instrument will take time to open large data files and save configuration information. During this time, the instrument will not respond to additional touchscreen touches
	Instrument Touchscreen is locked	If the lock in the title bar is red, unlock the instrument following the instructions in the Chapter 3, Operation: Title Bar section of this manual
Analog output does not work	Cable/connector not correctly installed	Make sure cable connector is fully seated
	Output wired with reverse polarity	Make sure analog out (+) and analog ground (-) are wired correctly to data-logger

Symptom	Possible Cause	Corrective Action
Analog output is not in proportion to display	Analog output range in DustTrak monitor may be set incorrectly	Check analog output setting in the Setup->Analog screen. Make sure the channel of interest is selected. Make sure that the correct output (0 to 5V, 4 to 20 mA) is selected
	Data logger scaling factor may be set incorrectly	Review the scaling factor set in the Setup-Analog screen
Alarm output does not work	Alarm function not turned on	Turn the alarm function on in the Settings->Alarm screen
Alarm does not turn on correctly	Alarm setting incorrect	Check the alarm settings in the Settings->Alarm screen Make sure the logging interval and time constant are set as short as possible (30 seconds or lower)
	Alarm output wired with reverse polarity	Alarm wires are polarized. Voltage input must be wired to alarm input (+)
Instrument does not store new data	Memory is full	Delete or transfer historic data
	Instrument is in Survey mode	The instrument does not store data in survey mode. Can to manual or program log mode

Symptom	Possible Cause	Corrective Action
Flow Error is indicated on front screen	If sampling from a duct, instrument may have problems overcoming pressure differences	Attach both the input and the exhaust port into the duct
	Flow obstruction	Remove obstruction if still present. Press any key to bypass
	Internal pump failing, indicated by inability to adjust flow rate to full range	Factory service may be required
	Filter Cassette clogged or has mass loading	Replace the filter cassette. See the maintenance section of the manual
	External pump module (for Model 8530EP only) is not connected to the DustTrak monitor	Make sure both the External Pump cable and the flow tubing connector are connected to the DustTrak monitor and the External pump module. Lock the External Pump Cable in place by rotating the connector clockwise until you hear it snap in place Make sure the tubing between the DustTrak monitor and the External pump module is not kinked and is free of any sharp bends Make sure the exhaust adapter is connected to the exhaust of the DustTrak monitor Make sure the External Pump module filters are not clogged. If found dirty, replace the two HEPA filters
Laser Error indicated on front screen	Laser background is too high	Remove and clean inlet nozzle. Pay close attention to the tip of the nozzle that is inserted into the instrument to ensure it is clear of any contamination
	Laser is failing	Factory service may be required

Symptom	Possible Cause	Corrective Action
Filter Error indicated on front screen	Filters need to be replaced	Replaced the filters per instructions in the maintenance section of this manual. Make sure to reset the filter mass and date once the filters have been changed **Note**: This is only a warning. The unit will continue to operate normally until the increase in pressure drop across the filter is so high that the pump can no longer maintain the set flow rate.
System Error has Occurred!	The processor did not receive the input it expected. This can also happen if the optics chamber is saturated with light, or the External Pump Cable is accidentally disconnected during the middle of sampling	Reboot the instrument. If the error does not go away, factory service is required

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Appendix A

Specifications

Specifications are subject to change without notice.

Sensor Type	90° light scattering
Range	8530 Desktop 0.001 to 400 mg/m^3 8531 Desktop HC 0.001 to 400 mg/m^3 8532 Handheld 0.001 to 150 mg/m^3
Resolution	±0.1% of reading of 0.001 mg/m^3, whichever is greater
Zero Stability	±0.002 mg/m^3 24 hours at 10 sec time constant
Particle Size Range	Approximately 0.1 to 10 µm
Flow Rate	3.0 L/min set at factory 1.4 to 3.0 L/min adjustable
Flow Accuracy	±5% factory setpoint Internal flow controlled
Temperature Coefficient	+0.001 mg/m^3 per °C
Operational Temp	0 to 50°C
Storage Temp	-20 to 60°C
Operational Humidity	0-95% RH, non-condensing
Time Constant	Adjustable 1 to 60 seconds
Data Logging	45 days at 1 minute samples
Log Interval	1 second to 1 hour
Physical Size (HWD)	Handheld: 4.9 x 4.75 x 12.45 in. Desktop: 5.3 x 8.5 x 8.8 in. External Pump: 4.0 x 7.5 x 3.5 in.
Weight	Handheld: 2.9 lb, 3.3 lb with battery Desktop: 3.45 lb, 4.45 lb – 1 battery, 5.45 lb – 2 batteries External Pump: 3.0 lb
Communications	8530/31: USB (Host and Device) and Ethernet. Stored data accessible using thumb drive 8532: USB (Host and Device). Stored dada accessible using thumb drive.

Power—DC	Handheld 12 VDC at 2A Desktop 24 VDC at 2.5A
Battery	8530/31: Up to 2 Removable Li-Ion External and Internal charging Life, 1 battery: >6.5 hours (9 hours typical for a new battery) for both internal and external pump Desktop DustTrak monitors Life, 2 battery: >13 hours 8532: 1 Removable Li-Ion External and Internal charging Life: 6 hours typical
Analog out	8530/31: User selectable output 0 to 5 V or 4 to 20 mA User selectable scaling
Alarm Out	8530/31: Relay or sound buzzer Relay No latching MOSFET User selectable set point 5% deadband Connector 4-pin, Mini-DIN connectors 8532: Sound buzzer
Screen	8530/31: 5.7" color touchscreen 8532: 3.5" color touchscreen
Gravimetric Sampling	8530/31: Removable 37-mm Cartridge
EMI/RF Immunity:	Complies with Emissions Directive Standard: EN50081-1:1992 Complies with Immunity Directive Standard: EN50082-1:1992*

*ESD Shock may require instrument reboot

Appendix B

Zero Module

The Zero Module (PN 801690) allows for automatic re-zeroing of the DustTrak Instrument during long sampling runs. The Zero Module works only with the 8530 and 8531 desktop models.

Attach the AutoZero module to the main instrument in two steps.

1. Place the Zero module over the instrument's inlet and press down. The Zero module has an O-ring seal that will engage with the instrument's inlet.

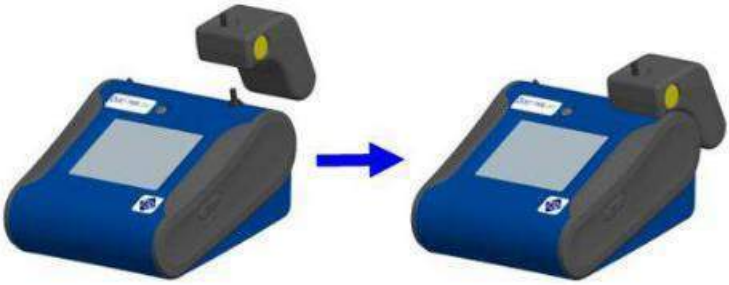

Figure B-1: Place Zero Module Over Inlet and Press Down

2. Attach the cable from the Zero module to the Zero module connector located on the back of the instrument.

Figure B-2: Zero Module Connector

The Zero Module can only be used in a program log mode. The Zero module function is controlled through these two program mode options:

Auto Zero Interval	Interval between re-zeroing the instrument using the Auto-Zero accessory.
Use Auto Zero	Select **Yes** to use the Zero Module. Select **No** to not use the Zero Module.

Important points on Zero Module operation:

- The Zero module will take one (1) minute to take a zero reading. The first 45 seconds of that period is used to clear the chamber of particles. Readings from last 15 second of the period, when the chamber is cleared of particles, will be averaged to determine the Zero offset.
- The log interval, when the Zero module is activated, must be two (2) minutes or greater. Data will not be recorded to the log file when the Zero module is activated.

Index

T

U

V

W–X–Y

Z

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UNDERSTANDING, ACCELERATED

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P/N 6001893 Rev. M

Printed in U.S.A.

IMPORTANT: ENSURE THAT THE PANEL RETAINING THUMB SCREWS (NUTS) ARE TIGHT BEFORE USE*

** not applicable for Sm.OIL*

Figure 1

Item	Part Numbers
H.OIL Electronic Panel	3202
Sm.OIL Electronic Panel	3203
H.OIL Probe	3200
Sm.OIL Probe	3201
Ground Lead	3208
Thumb Screws Set (2)	3250

Figure 2

To remove the probe you will need a 1/2" and 9/16" wrench. Use the wrenches to fully loosen the nut closest to the link and gently separate the link from the probe. Care must be taken to avoid breaking/pinching the wires while removing or replacing the probe. Make sure connections are tight.

Figure 3

Hanger to support the meter at the well head.

Tape Guide to protect the tape from sharp edges.

HERON ALSO MANUFACTURES:

- Water Level Meters
- Data Loggers
- Conductivity Meters
- Temperature Meters
- Well Casing Indicators
- Well Depth Indicators
- Tag Lines
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H.OIL and Sm.OIL Interface & Static Levels

Operating and Maintenance Instructions

www.heroninstruments.com

H.OIL and Sm.OIL Interface Meter Instructions

General Care of the H.OIL/Sm.OIL

The **H.OIL/Sm.OIL** has been designed to provide years of reliable, accurate measurements of floating product (L.N.A.P.L) and sinking layers (D.N.A.P.L.). The **Intrinsically Safe Certification** makes the **H.OIL/Sm.OIL** ideal for use in hazardous environments.

- Avoid sharp edged casing, use the tape guide on the unit to prevent damage to the tape.
- Take care to avoid the tape becoming entangled with other equipment in boreholes or wells, use stilling pipes when possible.
- Neatly rewind and clean the tape after each use.
  Refer to: Cleaning the **H.OIL/Sm.OIL**.

DO NOT use the **H.OIL/Sm.OIL** as a guide to backfilling, bentonite sealing or sand packing in wells. This type of material falls through the water column at a much slower rate than the **H.OIL/Sm.OIL** probe and can result in a trapped tape and probe.

DO NOT allow the tape to "freefall" down the well, it may become caught in other equipment in the well.

Warranty is conditional upon adherence to these guidelines.

Equipment Check

Switch the **H.OIL/Sm.OIL** on by pushing the start button in the center of the electronic panel. The green LED indicates that the unit is ready for use. If the LED does not light, then replace the battery. The green LED will stay on for approximately 8 minutes. Every time the start button is pressed the 8 minute timer will reset. When the 9 volt battery is almost drained, the green LED will start to blink when the unit is signalling. The battery should be replaced as soon as possible (see Figure 1).

NOTE: Accurate battery orientation diagram inside of battery box.

- Inspect the probe lens for any signs of damage or buildup of dirt/residue. The lens should be clean and clear before use.
- To maintain intrinsic safety, ensure the ground lead is securely fastened to the back of the frame and in good condition.
- Ensure the two panel retaining thumb screws (nuts) are tight*. Test unit by lowering probe into water that is shielded from light (intermittent tone will indicate).

NOTE: The probe will not work in ambient light conditions.

Use in the Field

Before using the **H.OIL/Sm.OIL**, ensure the two panel retaining thumb screws (nuts) are tight* and the ground lead is connected to a grounding source. Switch the unit on by pushing the start button in the center of the electronic panel (see Figure 1). The unit will now be active for 8 minutes (the unit switches itself off automatically). The green LED indicates that the unit is ready for use. If the green LED switches off while the meter is being used, push the center start button again to re-start the unit.

- To avoid damaging the tape on the side of the casing, hang the **H.OIL/Sm.OIL** on the casing and run the tape over the guide on the frame leg (see Figure 3). If you cannot hang the unit, hold the **H.OIL/Sm.OIL** away from the side of the casing and guide the tape down the center of the well.
- Swivel the probe holder on the frame to allow the tape free movement down the well (see Figure 3).
- Note the inverted triangle on the probe holder serves as a datum point indicating "top of casing" (see Figure 3).
- When taking measurements, it is suggested to lower the probe down the well until the top of the water/product is reached. **Water is indicated by an intermittent tone and product is indicated by a solid tone**. Note the depth marking on the tape. **DO NOT** try to measure the product/water interface at this stage. Allow the probe to pass through any product into the water below (indicated by the intermittent tone). Now slowly withdraw the probe until the tone changes from intermittent to solid. This point indicates the base of the product layer. Note the depth marking on the tape. This method avoids having the product drawn down into the water giving false interface readings.
- In cold weather, condensation may form on the lens as it contacts the warmer moist air in the well, this causes the unit to falsely sound as product. To overcome this, allow the probe to acclimatize in the well or lower the probe into the water, then take readings.
- When rewinding the tape, remove as much water and debris as possible from the tape and probe.

Removing the Probe

To remove the probe you will need a 1/2" and 9/16" wrench. Use the wrenches to fully loosen the nut closest to the link and gently separate the link from the probe (see Figure 2). Care must be taken to avoid breaking/pinching the wires while removing or replacing the probe.

Make sure connections are tight.

Cleaning the H.OIL/Sm.OIL

Always clean the **H.OIL/Sm.OIL** after use in the field to maintain optimal performance and extend the life of the unit.

If the electronic panel is removed first, the reel and tape can be washed gently with a power washer. Remove the retaining thumb screws (nuts) (see Figure 1) to release the panel*. Take care not to lose the thumb screws as the unit will not work without them*.

We strongly recommend using biodegradable household dishwashing liquid. The reel, tape and probe may be cleaned and de-greased with the following:

- Soap solution
- Joy®
- Formula 409®
- Fantastic®
- Top Job®
- GOO-GONE®
- Windex®
- Mr. Clean®
- Green Clean®

NOTE: DO NOT clean the probe lens with any abrasive cleaners or products that contain alcohol.

Troubleshooting the H.OIL/Sm.OIL

Q. Why doesn't the unit sound when the probe contacts water?

A.
- **Do not** test in ambient light conditions.
- Make sure retaining thumb screws are tight (**H.OIL**) (Figure 1).
- Make sure the connection of the male and female connectors in the probe/link are connected tightly (see Figure 2).

Q. Why does the unit continuously sound when the probe is not in water/product?

A.
- Make sure the electronic panel is oriented in the correct position on the unit (battery box should be under the **Heron** Logo).
- Make sure probe lens is clean and clear.

For more troubleshooting tips please visit our website at **www.heroninstruments.com**

Contact Heron Instruments or your Heron Distributor if you cannot isolate the problem.

Warranty (5 years, probe 1 year)

Heron Instruments Inc. warrants to repair or replace any defective equipment or part upon inspection by a **Heron** service technician. Warranty will be determined to our satisfaction to have a defect in workmanship or original material. The customer is responsible for all shipping fees to return the item to **Heron**.

This warranty shall not apply to damage of equipment caused by improper installation, usage, storage, alteration or inadequate care.

In no event shall **Heron** be held liable for any direct, indirect or consequential damages, abuse, acts of third parties (rental equipment), environmental conditions or expenses which may arise in connection with such defective equipment.

Heron Instruments Warranty coverage does not extend to the following:

- Tape, bag or batteries used with the product.
- Products used as rental equipment.
- Products contaminated by materials which are known to be hazardous and have rendered the unit unserviceable.
- Parts failure due to neglect in cleaning or servicing.
- Failure of parts caused by misuse.

For service information:

- visit **www.heroninstruments.com** under the **CONTACT** heading
- email **service@heroninstruments.com**
- call **1-800-331-2032** or **905-628-4999**

Warranty is conditional upon adherence to these guidelines.

MiniRAE 3000
User's Guide

RAE SYSTEMS

Rev. C
August 2010
P/N 059-4020-000

FCC Information

Contains FCC ID: PI4411B

The enclosed device complies with part 15 of the FCC rules. Operation is subject to the following conditions: (1) This device may not cause harmful interference, and (2) This device must accept any interference received, including interference that may cause undesired operation.

Contents

Read Before Operating

This manual must be carefully read by all individuals who have or will have the responsibility of using, maintaining, or servicing this product. The product will perform as designed only if it is used, maintained, and serviced in accordance with the manufacturer's instructions. The user should understand how to set the correct parameters and interpret the obtained results.

CAUTION!

To reduce the risk of electric shock, turn the power off before removing the instrument cover. Disconnect the battery before removing sensor module for service. Never operate the instrument when the cover is removed. Remove instrument cover and sensor module only in an area known to be non-hazardous.

Special Notes

When the instrument is taken out of the transport case and turned on for the first time, there may be some residual organic or inorganic vapor trapped inside the detector chamber. The initial PID sensor reading may indicate a few ppm. Enter an area known to be free of any organic vapor and turn on the instrument. After running for several minutes, the residual vapor in the detector chamber will be cleared and the reading should return to zero.

The battery of the instrument discharges slowly even if it is turned off. If the instrument has not been charged for 5 to 7 days, the battery voltage will be low. Therefore, it is a good practice to always charge the instrument before using it. It is also recommended to fully charge the instrument for *at least 10 hours* before first use. Refer to this User Guide's section on battery charging for more information on battery charging and replacement.

WARNINGS

STATIC HAZARD: Clean only with damp cloth.

For safety reasons, this equipment must be operated and serviced by qualified personnel only. Read and understand instruction manual completely before operating or servicing.

Use only RAE Systems battery packs, part numbers 059-3051-000, 059-3052-000, and 059-3054-000. This instrument has not been tested in an explosive gas/air atmosphere having an oxygen concentration greater than 21%. Substitution of components may impair intrinsic safety. Recharge batteries only in non-hazardous locations.

Do not mix old and new batteries or batteries from different manufacturers.

The calibration of all newly purchased RAE Systems instruments should be tested by exposing the sensor(s) to known concentration calibration gas before the instrument is put into service.

For maximum safety, the accuracy of the instrument should be checked by exposing it to a known concentration calibration gas before each day's use.

Do not use USB/PC communication in hazardous locations.

AVERTISSEMENT

DANGER RISQUE D'ORIGINE ELECTROSTATIQUE: Nettoyer uniquement avec un chiffon humide.

Pour des raisons de sécurité, cet équipment doit être utilisé, entretenu et réparé uniquement par un personnel qualifié. Étudier le manuel d'instructions en entier avant d'utiliser, d'entretenir ou de réparer l'équipement.

Utiliser seulement l'ensemble de batterie RAE Systems, la reference 059-3051-000 au 059-3052-000 au 059-3054-000. Cet instrument n'a pas été essayé dans une atmosphère de gaz/air explosive ayant une concentration d'oxygène plus élevée que 21%. La substitution de composants peut compromettre la sécurité intrinsique. Ne charger les batteries que dans emplacements désignés non-dangereuse.

Ne pas melanger les anciennes et les nouvelles batteries, ou bien encore les batteries de differents fabriquants.

La calibration de toute instruments de RAE Systems doivent être testé en exposant l'instrument a une concentration de gaz connue par une procédure diétalonnage avant de mettre en service l'instrument pour la première fois.

Pour une securite maximale, la sensibilité du l'instrument doit être verifier en exposant l'instrument a une concentration de gaz connue par une procédure diétalonnage avant chaque utilisation journalière.

Ne pas utiliser de connection USB/PC en zone dangereuse.

Standard Contents

Instrument
Calibration Kit
Charging Cradle
AC/DC Adapter
Alkaline Battery Adapter
Data Cable
CD-ROM With User's Guide, Quick Start Guide, and related materials

General Information

The compact instrument is designed as a broadband VOC gas monitor and datalogger for work in hazardous environments. It monitors Volatile Organic Compounds (VOC) using a photoionization detector (PID) with a 9.8 eV, 10.6 eV, or 11.7 eV gas-discharge lamp. Features are:

Lightweight and Compact

- Compact, lightweight, rugged design
- Built-in sample draw pump

Dependable and Accurate

- Up to 16 hours of continuous monitoring with rechargeable battery pack
- Designed to continuously monitor VOC vapor at parts-per-million (ppm) levels

User-friendly

- Preset alarm thresholds for STEL, TWA, low- and high-level peak values.
- Audio buzzer and flashing LED display are activated when the limits are exceeded.

Datalogging Capabilities

- 260,000-point datalogging storage capacity for data download to PC

The instrument consists of a PID with associated microcomputer and electronic circuit. The unit is housed in a rugged case with a backlit LCD and 3 keys to provide easy user interface. It also has a built-in flashlight for operational ease in dark locations.

Physical Description

The main components of the portable VOC monitoring instrument include:

- Three keys for user to interact with the instrument: 3 operation/programming keys for normal operation or programming
- LCD display with back light for direct readout and calculated measurements
- Built-in flashlight for illuminating testing points in dark environments
- Buzzer and red LEDs for alarm signaling whenever exposures exceed preset limits
- Charge contacts for plugging directly to its charging station
- Gas entry and exit ports
- USB communication port for PC interface
- Protective rubber cover

Specifications

Size:	9.25" L x 3.6" W x 2.9" H
Weight:	28 oz with battery pack
Detector:	Photoionization sensor with 9.8, 10.6, or 11.7 eV UV lamp
Battery:	A 3.7V rechargeable Lithium-Ion battery pack (snap in, field replaceable, at non-hazardous location only)
	Alkaline battery holder (for 4 AA batteries)
Battery Charging:	Less than 8 hours to full charge
Operating Hours:	Up to16 hours continuous operation
Display:	Large dot matrix screen with backlight

Measurement range & resolution

Lamp	Range	Resolution
10.6 eV	0.1 ppm to 15,000 ppm	0.1 ppm
9.8 eV	0.1 ppm to 5,000 ppm	0.1 ppm
11.7 eV	0.1 ppm to 2,000 ppm	0.1 ppm

Response time (T_{90}): 2 seconds

Accuracy (Isobutylene): 10 to 2000 ppm: ±3% at calibration point.

PID Detector: Easy access to lamp and sensor for cleaning and replacement

Correction Factors: Over 200 VOC gases built in (based on RAE Systems Technical Note TN-106)

Calibration: Two-point field calibration of zero and standard reference gases

Calibration Reference: Store up to 8 sets of calibration data, alarm limits and span values

Inlet Probe: Flexible 5" tubing

Radio module: Bluetooth (2.4GHz), RF module (433MHz, 868MHz , 915MHz, or 2.4GHz)

Keypad: 1 operation key and 2 programming keys; 1 flashlight switch

Direct Readout: Instantaneous, average, STEL, TWA and peak value, and battery voltage

Intrinsic Safety: US and Canada: Class I, Division 1, Groups A, B, C, D

Europe: ATEX (0575 Ex II 2G Ex ia IIC/IIB T4 Gb)
KEMA 07 ATEX 0127
Complies with EN60079-0:2009, EN60079-11:2007

IECEx CSA 10.0005 Ex ia IIC/IIB T4 Gb
Complies with IEC 60079-0:2007,
IEC 60079-11:2006
(IIC: 059-3051-000 Li-ion bat pack
or 059-3054-000 NiMH bat pack;
IIB: 059-3052-000 alkaline bat pack)

EM Interference:	Highly resistant to EMI/RFI. Compliant with EMC R&TTE (RF Modules)
Alarm Setting:	Separate alarm limit settings for Low, High, STEL and TWA alarm
Operating Mode:	Hygiene or Search mode
Alarm:	Buzzer 95dB at 30cm and flashing red LEDs to indicate exceeded preset limits, low battery voltage, or sensor failure
Alarm Type:	Latching or automatic reset
Real-time Clock:	Automatic date and time stamps on datalogged information
Datalogging:	260,000 points with time stamp, serial number, user ID, site ID, etc.
Communication:	Upload data to PC and download instrument setup from PC via USB on charging station.
Sampling Pump:	Internally integrated. Flow rate: 450 to 550 cc/min.
Temperature:	-20º C to 50º C (-4º to 122º F)
Humidity:	0% to 95% relative humidity (non-condensing)
Housing (including rubber boot):	Polycarbonate, splashproof and dustproof Battery can be changed without removing rubber boot.

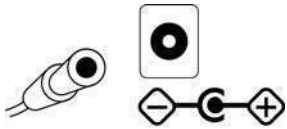
Charging The Battery

Always fully charge the battery before using the instrument. The instrument's Li-ion battery is charged by placing the instrument in its cradle. Contacts on the bottom of the instrument meet the cradle's contacts, transferring power without other connections.

Note: Before setting the instrument into its charging cradle, visually inspect the contacts to make sure they are clean. If they are not, wipe them with a soft cloth. Do not use solvents or cleaners.

Follow this procedure to charge the instrument:

1. Plug the AC/DC adapter's barrel connector into the instrument's cradle.



2. Plug the AC/DC adapter into the wall outlet.
3. Place the instrument into the cradle, press down, and lean it back. It locks in place and the LED in the cradle glow

The instrument begins charging automatically. The "Primary" LED in the cradle blinks green to indicate charging. During charging, the diagonal lines in the battery icon on the instrument's display are animated and you see the message "Charging..."

When the instrument's battery is fully charged, the battery icon is no longer animated and shows a full battery. The message "Fully charged!" is shown. The cradle's LED glows continuously green.

Note: If you see the "Battery Charging Error" icon (a battery outline with an exclamation mark inside), check that the instrument or rechargeable battery has been set into the cradle

properly. If you still receive the message, check the Troubleshooting section of this guide.

Note: If the instrument or battery has been in the cradle for more than 10 hours and you see the "Battery Charging Error" icon and a message that says, "Charging Too Long," this indicates that the battery is not reaching a full charge. Try changing the battery and make sure the contacts between the instrument (or battery) are meeting the cradle. If the message is still shown, consult your distributor or RAE Systems Technical Services.

Charging A Spare Rechargeable Battery

A rechargeable Li-ion battery can be charged when it is not inside the monitor. The charging cradle is designed to accommodate both types of charging. Contacts on the bottom of the battery meet the contacts on the cradle, transferring power without other connections, and a spring-loaded capture holds the battery in place during charging.

1. Plug the AC/DC adapter into the monitor's cradle.
2. Place the battery into the cradle, with the gold-plated contacts on top of the six matching charging pins.
3. Plug the AC/DC adapter into the wall outlet.

The battery begins charging automatically. During charging, the Secondary LED in the cradle blinks green. When charging is complete, it glows steady green.

Release the battery from the cradle by pulling it back toward the rear of the cradle and tilting it out of its slot.

Note: If you need to replace the Li-ion battery pack, replacements are available from RAE Systems. The part number is 059-3051-000.

Note: An Alkaline Battery Adapter (part number 059-3052-000), which uses four AA alkaline batteries (Duracell MN1500), may be substituted for the Li-Ion battery.

WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge and replace batteries only in areas known to be non-hazardous. **Remove and replace batteries only in areas known to be non-hazardous.**

Low Voltage Warning

When the battery's charge falls below a preset voltage, the instrument warns you by beeping once and flashing once every minute, and the "empty battery" icon blinks on and off once per second. You should turn off the instrument within 10 minutes and either recharge the battery by placing the instrument in its cradle, or replace the battery with a fresh one with a full charge.

Clock Battery

An internal clock battery is mounted on one of the instrument's printed circuit boards. This long-life battery keeps settings in memory from being lost whenever the Li-ion battery or alkaline batteries are removed. This backup battery should last approximately five years, and must be replaced by an authorized RAE Systems service technician. It is not user-replaceable.

Data Protection While Power Is Off

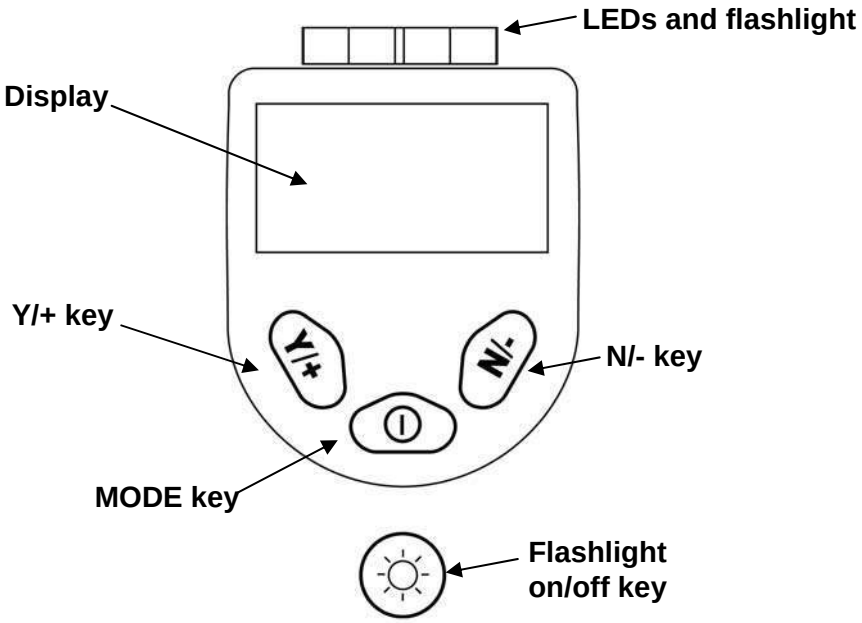
When the instrument is turned off, all the current real-time data including last measured values are erased. However, the datalog data is preserved in non-volatile memory. Even if the battery is disconnected, the datalog data will not be lost.

User Interface

The instrument's user interface consists of the display, LEDs, an alarm transducer, and four keys. The keys are:

Y/+
MODE
N/-
Flashlight on/off

The LCD display provides visual feedback that includes the reading, time, battery condition, and other functions.

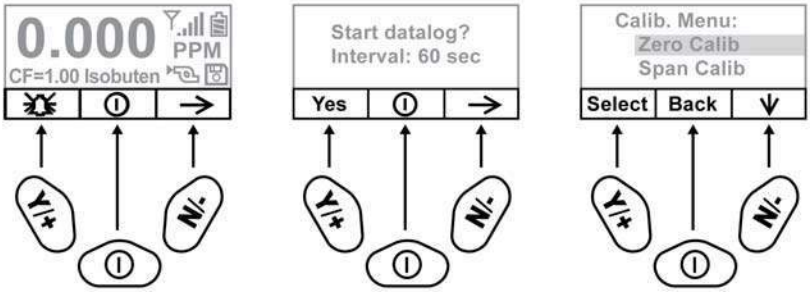


In addition to their labeled functions, the keys labeled Y/+, MODE, and N/- act as "soft keys" that control different parameters and make different selections within the instrument's menus. From menu to

menu, each key controls a different parameter or makes a different selection.

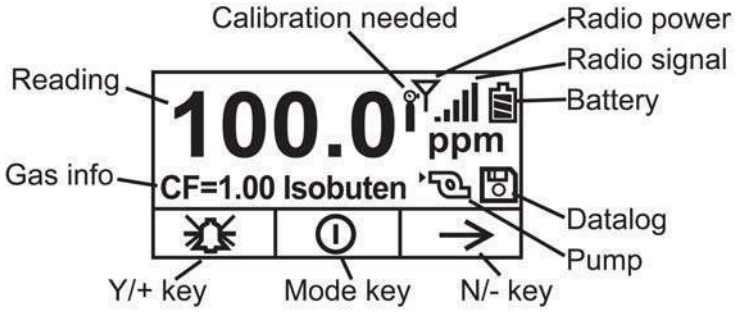
Three panes along the bottom of the display are "mapped" to the keys. These change as menus change, but at all times the left pane corresponds to the [Y/+] key, the center pane corresponds to the [MODE] key, and the right pane corresponds to the [N/-] key. Here are three examples of different menus with the relationships of the keys clearly shown:

RELATIONSHIP OF BUTTONS TO CONTROL FUNCTIONS



Display

The display shows the following information:



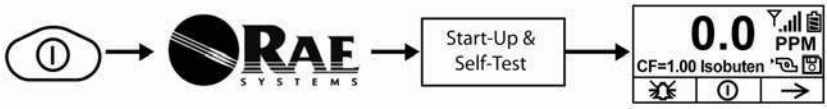
Graph	Graphic representation of concentration plotted over time
Gas info	Tells the Correction Factor and type of calibration gas
Reading	Concentration of gas as measured by the instrument
Calibration needed	Indicates that calibration should be performed
Radio power	Indicates whether radio connection is on or off
Radio signal	Indicates signal strength in 5-bar bargraph
Battery	Indicates battery level in 3 bars
Pump	Indicates that pump is working
Datalog	Indicates whether datalog is on or off
Y/+	Y/+ key's function for this screen
MODE	MODE key's function for this screen
N/-	N/- key's function for this screen

Operating The Instrument

The instrument is designed as a broadband VOC gas monitor and datalogger for work in hazardous environments. It gives real-time measurements and activates alarm signals whenever the exposure exceeds preset limits. Prior to factory shipment, the instrument is preset with default alarm limits and the sensor is pre-calibrated with standard calibration gas. However, you should test the instrument and verify the calibration before the first use. After the instrument is fully charged and calibrated, it is ready for immediate operation.

Turning The Instrument On

1. With the instrument turned off, press and hold [MODE].
2. When the display turns on, release the [MODE] key.



The RAE Systems logo should appear first. (If the logo does not appear, there is likely a problem and you should contact your distributor or RAE Systems Technical Support.) The instrument is now operating and performs self tests. If any tests (including sensor and memory tests fail), refer to the Troubleshooting section of this guide.

Once the startup procedure is complete, the instrument shows a numerical reading screen with icons. This indicates that the instrument is fully functional and ready to use.

Turning The Instrument Off

1. Press and hold the Mode key for 3 seconds. A 5-second countdown to shutoff begins.
2. Once the countdown stops, the instrument is off. Release the Mode key.
3. When you see "Unit off..." release your finger from the [MODE] key. The instrument is now off.

Note: You must hold your finger on the key for the entire shutoff process. If you remove your finger from the key during the countdown, the shutoff operation is canceled and the instrument continues normal operation.

Operating The Built-In Flashlight

The instrument has a built-in flashlight that helps you point the probe in dark places. Press the flashlight key to turn it on. Press it again to turn it off.

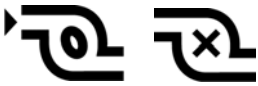
Note: Using the flashlight for extended periods shortens the battery's operating time before it needs recharging.

Pump Status

IMPORTANT!

During operation, make sure the probe inlet and the gas outlet are free of obstructions. Obstructions can cause premature wear on the pump, false readings, or pump stalling. During normal operation, the pump icon alternately shows inflow and outflow as shown here:

During duty cycling (PID lamp cleaning), the display shows these icons in alternation:



If there is a pump failure or obstruction that disrupts the pump, you will see this icon blinking on and off:

If you see this blinking icon, consult the Troubleshooting section of this guide.

Calibration Status

The instrument displays this icon if it requires calibration:

Calibration is required (and indicated by this icon) if:

- The lamp type has been changed (for example, from 10.6 eV to 9.8 eV).
- The sensor has been replaced.
- It has been 30 days or more since the instrument was last calibrated.
- If you have changed the calibration gas type without recalibrating the instrument.

Operating Modes

Your instrument operates in different modes, depending on the model and its factory default settings. In some cases, you can change modes using a password and using the instrument's navigation. In other cases, you must use ProRAE Studio software.

The default setting for your instrument is:

User Mode: Basic
Operation Mode: Hygiene

This is outlined in detail on page 74.

The other options, covered later in this guide, are:

User Mode: Advanced (page 78)
Operation Mode: Hygiene

User Mode: Advanced (page 82)
Operation Mode: Search

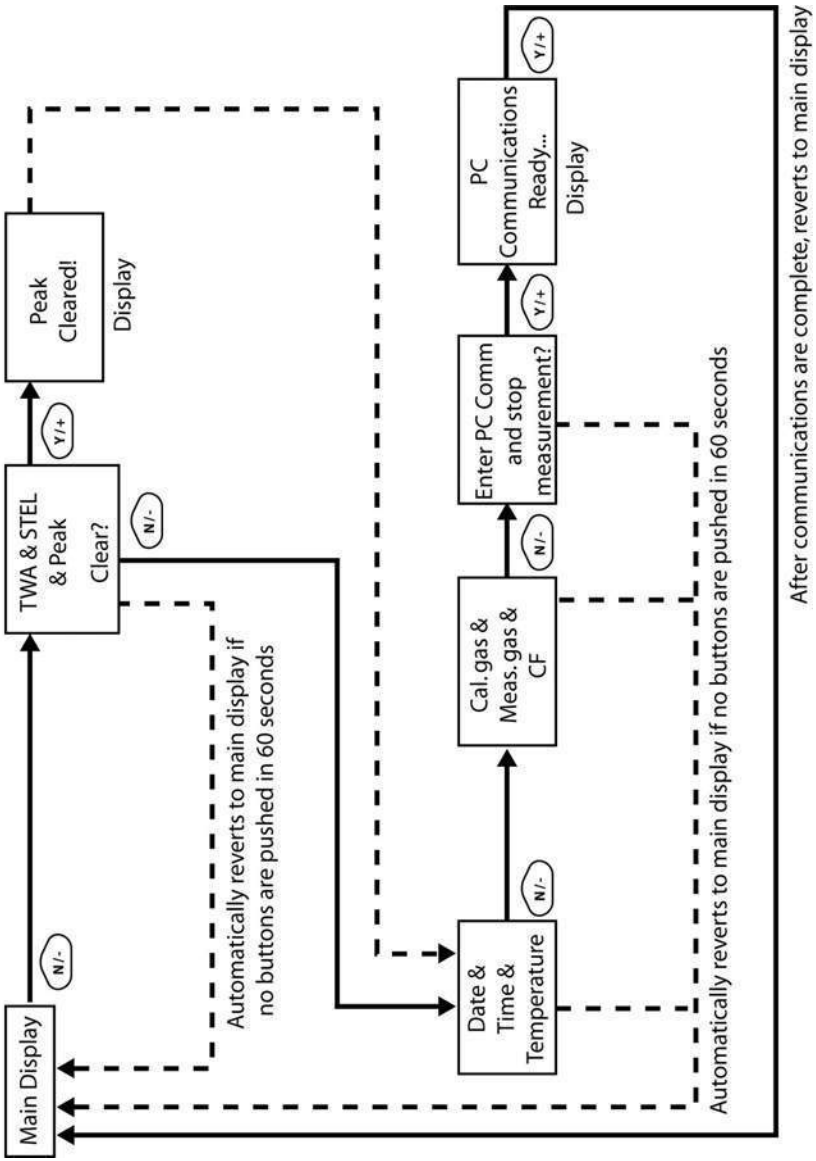
Using ProRAE Studio allows access to other options. In addition, Diagnostic Mode (page 83) is available for service technicians.

Basic User Level/Hygiene Mode (Default Settings)

The instrument is programmed to operate in Basic User Level/Hygiene Mode as its default. This gives you the most commonly needed features while requiring the fewest parameter adjustments.

Pressing [N/-] steps you from one screen to the next, and eventually return to the main display. If you do not press a key within 60 seconds after entering a display, the instrument reverts to its main display.

Note: While viewing any of these screens, you can shut off your instrument by pressing [MODE].



Note: Dashed line indicates automatic progression.

After the instrument is turned on, it runs through the start-up menu. Then the message "**Please apply zero gas…**" is displayed.

At this point, you can perform a zero air (fresh air) calibration. If the ambient air is clean, you can use that. Otherwise, use a cylinder of zero air. Refer to Zero Calibration on page 37 for a more detailed description of zero calibration.

Start zero calibration by pressing Start. You see the message "Zeroing…" followed by a 30-second countdown.

Note: You can press [MODE] to quit, bypassing the zero air calibration.

When zero calibration is complete, you see the message:

Zeroing is done!

Reading = 0.0 ppm

The instrument is now sampling and collecting data.

Note: At the Average & Peak, Date & Time & Temperature, Calibration Gas & Measurement Gas & Correction Factor, and PC Communications screens, the instrument automatically goes to the main display after 60 seconds if you do not push a key to make a selection.

Alarm Signals

During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings). If the concentration exceeds any of the preset limits, the loud buzzer and red flashing LED are activated immediately to warn you of the alarm condition.

In addition, the instrument alarms if one of the following conditions occurs: battery voltage falls below a preset voltage level, failure of the UV lamp, or pump stall.

Alarm Signal Summary

Message	Condition	Alarm Signal
HIGH	Gas exceeds "High Alarm" limit	3 beeps/flashes per second*
OVR	Gas exceeds measurement range	3 beeps/flashes per second*
MAX	Gas exceeds electronics' maximum range	3 beeps/flashes per second*
LOW	Gas exceeds "Low Alarm" limit	2 beeps/flashes per second*
TWA	Gas exceeds "TWA" limit	1 Beep/flash per second*
STEL	Gas exceeds "STEL" limit	1 Beep/flash per second*
Pump icon flashes	Pump failure	3 beeps/flashes per second
Lamp	PID lamp failure	3 beeps/flashes per second plus "Lamp" message on display
Battery icon flashes	Low battery	1 flash, 1 beep per minute plus battery icon flashes on display
CAL	Calibration failed, or needs calibration	1 beep/flash per second
NEG	Gas reading measures less than number stored in calibration	1 beep/flash per second

* Hygiene mode only. In Search mode, the number of beeps per second (1 to 7) depends upon the concentration of the sampled gas. Faster rates indicate higher concentrations.

Preset Alarm Limits & Calibration

The instrument is factory calibrated with standard calibration gas, and is programmed with default alarm limits.

Cal Gas (Isobutylene)	Cal Span	unit	Low	High	TWA	STEL
MiniRAE 3000	100	ppm	50	100	10	25

Testing The Alarm

You can test the alarm whenever the main (Reading) display is shown. Press [Y/+], and the audible and visible alarms are tested.

Integrated Sampling Pump

The instrument includes an integrated sampling pump. This diaphragm-type pump that provides a 450 to 550 cc per minute flow rate. Connecting a Teflon or metal tubing with 1/8" inside diameter to the gas inlet port of the instrument, this pump can pull in air samples from 100' (30 m) away horizontally or vertically.

Note: In Search Mode, the pump turns on when a sample measurement is started, and turns off when the sample is manually stopped.

If liquid or other objects are pulled into the inlet port filter, the instrument detects the obstruction and immediately shuts down the pump. The alarm is activated and a flashing pump icon is displayed.

You should acknowledge the pump shutoff condition by clearing the obstruction and pressing the [Y/+] key while in the main reading display to restart the pump.

Backlight

The LCD display is equipped with an LED backlight to assist in reading the display under poor lighting conditions.

Datalogging

During datalogging, the instrument displays a disk icon to indicate that datalogging is enabled. The instrument stores the measured gas concentration at the end of every sample period (when data logging is enabled). In addition, the following information is stored: user ID, site ID, serial number, last calibration date, and alarm limits. All data are retained (even after the unit is turned off) in non-volatile memory so that it can be down- loaded at a later time to a PC.

Datalogging event

When Datalogging is enabled, measurement readings are being saved. These data are stored in "groups" or "events." A new event is created and stored each time the instrument is turned on and is set to automatic datalogging, or a configuration parameter is changed, or datalogging is interrupted. The maximum time for one event is 24 hours or 28,800 points. If an event exceeds 24 hours, a new event is automatically created. Information, such as start time, user ID, site ID, gas name, serial number, last calibration date, and alarm limits are recorded.

Datalogging sample

After an event is recorded, the unit records a shorter form of the data. When transferred to a PC running ProRAE Studio, this data is arranged with a sample number, time, date, gas concentration, and other related information.

Auto/Manual/Snapshot Datalogging

The instrument has three datalog types:

Auto	Default mode. Collects datalog information when the instrument is sampling.
Manual	Datalogging occurs only when the instrument's datalogging is manually started (see page 63 for details).
Snapshot	Datalogs only during snapshot (single-event capture, initiated by pressing [MODE]) sampling. See page 65 for details.

Note: You can only choose one datalog type to be active at a time.

Accessories

The following accessories are included with the instrument:

- An AC Adapter (Battery Charger)
- Alkaline battery adapter
- External Filter
- Organic Vapor Zeroing kit

Hard-case kits also include these accessories:

- Calibration adapter
- Calibration regulator and Flow controller

Standard Kit & Accessories

AC Adapter (Battery Charger)

WARNING

To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non-hazardous. Remove and replace battery only in area known to be non-hazardous.

Ne charger les batteries que dans emplacements designés non-dangereuses.

A battery charging circuit is built into the instrument cradle. It only needs a regular AC to 12 VDC adapter (wall-mount transformer, part number 500-0114-000) to charge the instrument.

To charge the battery inside the instrument:

1. Power off the instrument.
2. Connect the AC adapter to the DC jack on the instrument's cradle. If the instrument is off, it automatically turns on.
3. While charging, the display message shows "Charging." The Primary LED on the cradle flashes green when charging.
4. When the battery is fully charged, the LED changes to glowing green continuously, and the message "Fully charged" appears on the

display. If there is a charging error, the LED glows red continuously.

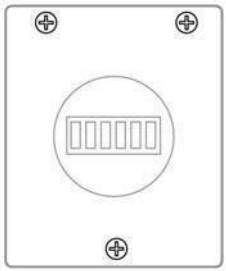
A completely discharged instrument can be charged to full capacity within 8 hours. Batteries drain slowly even if an instrument is off. Therefore, if the instrument has been in storage or has not been charged for several days or longer, check the charge before using it.

The factory-supplied battery is designed to last for 16 hours of normal operation (no alarm), for a new battery under the optimum circumstances. As the battery becomes older or is subject to adverse conditions (such as cold ambient temperature), its capacity will be significantly reduced.

Alkaline Battery Adapter

An alkaline battery adapter is supplied with each instrument. The adapter (part number 059-3052-000) accepts four AA alkaline batteries (use only Duracell MN1500) and provides approximately 12 hours of operation. The adapter is intended to be used in emergency situations when there is no time to charge the Li-ion battery pack.

To insert batteries into the adapter:



1. Remove the three Philips-head screws to open the compartment in the adapter.
2. Insert four fresh AA batteries as indicated by the polarity (+/-) markings.
3. Replace the cover. Replace the three screws.

To install the adapter in the instrument:

1. Remove the Li-ion battery pack from the instrument by sliding the tab and tilting out the battery.
2. Replace it with the alkaline battery adapter
3. Slide the tab back into place to secure the battery adapter.

IMPORTANT!

Alkaline batteries cannot be recharged. The instrument's internal circuit detects alkaline batteries and will not allow recharging. If you place the instrument in its cradle, the alkaline battery will not be recharged. The

internal charging circuit is designed to prevent damage to alkaline batteries and the charging circuit when alkaline batteries are installed inside the instrument. If you try to charge an alkaline batteries installed in the instrument, the instrument's display will say, "Alkaline Battery," indicating that it will not charge the alkaline batteries.

Note: When replacing alkaline batteries, dispose of old ones properly.

WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge the battery only in areas known to be non-hazardous. Remove and replace the battery only in areas known to be non-hazardous.

External Filter

The external filter is made of PTFE (Teflon®) membrane with a 0.45 micron pore size to prevent dust or other particles from being sucked into the sensor manifold, which would cause extensive damage to the instrument. It prolongs the operating life of the sensor. To install the external filter, simply connect it to the instrument's inlet tube.

Optional Accessories

Calibration Adapter

The calibration adapter for the instrument is a simple 6-inch Tygon tubing with a metal adapter on one end. During calibration, simply insert the metal adapter into the regular gas inlet probe of the instrument and the tubing to the gas regulator on the gas bottle.

Calibration Regulator

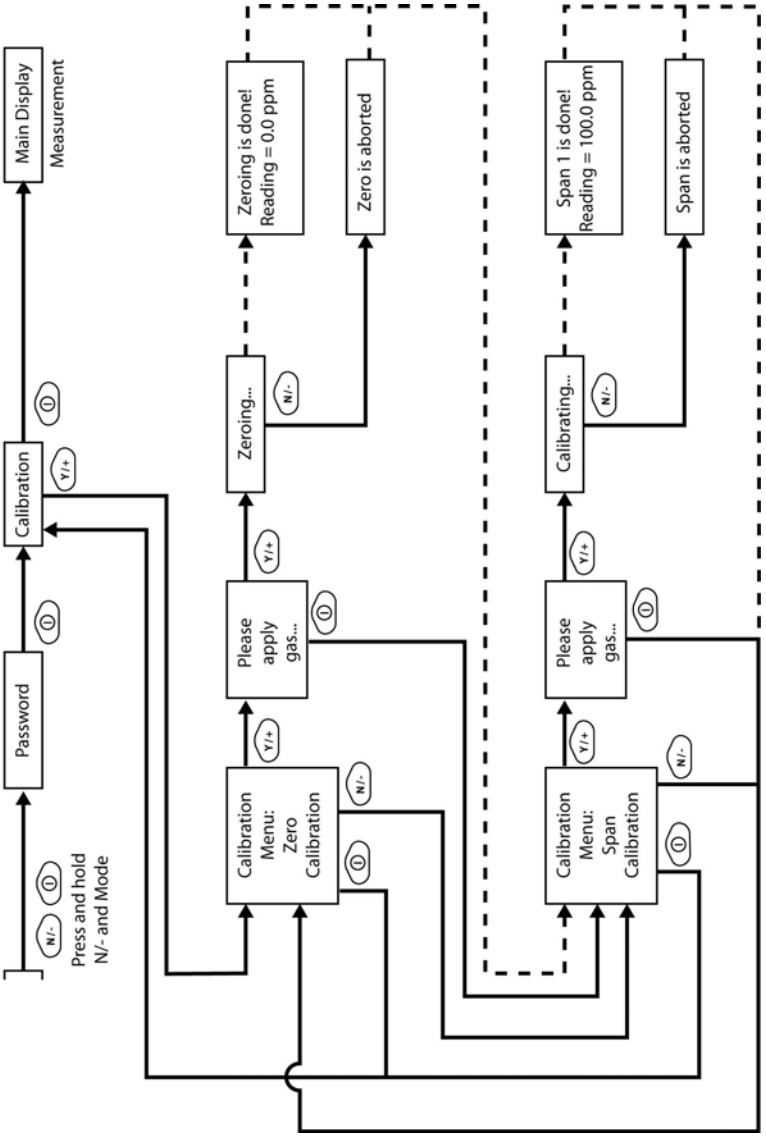
The Calibration Regulator is used in the calibration process. It regulates the gas flow rate from the Span gas cylinder into the gas inlet of the instrument during calibration process. The maximum flow rate allowed by the flow controller is about 0.5L/min (500 cc per min.). Alternatively, a demand-flow regulator or a Tedlar gas bag may be used to match the pump flow precisely.

Organic Vapor Zeroing Kit

The Organic Vapor Zeroing Kit is used for filtering organic air contaminants that may affect the zero calibration reading. To use the Organic Vapor Zeroing Kit, simply connect the filter to the inlet port of the instrument.

Standard Two-Point Calibration (Zero & Span)

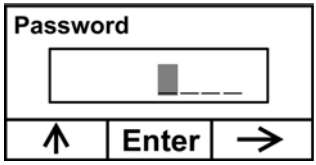
The following diagram shows the instrument's calibrations in Basic/Hygiene mode.



Note: Dashed line indicates automatic progression.

Entering Calibration

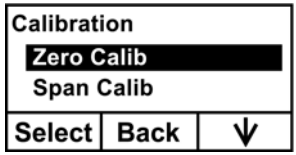
1. Press and hold [MODE] and [N/-] until you see the Password screen.



2. In Basic User Level, you do not need a password to perform calibrations. Instead of inputting a password, enter calibration by pressing [MODE].

   Note: If you inadvertently press [Y/+] and change any of the numbers, simply press [MODE] and you will be directed to the calibration menu.

The Calibration screen is now visible with Zero Calibration highlighted.



These are your options:

- Press [Y/+] to select the highlighted calibration (Zero Calib or Span Calib).
- Press [MODE] to exit calibration and return to the main display and resume measurement.
- Press [N/-] to toggle the highlighted calibration type.

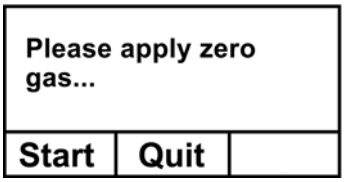
Zero (Fresh Air) Calibration

This procedure determines the zero point of the sensor calibration curve. To perform a fresh air calibration, use the calibration adapter to connect the instrument to a "fresh" air source such as from a cylinder or Tedlar bag (optional accessory). The "fresh" air is clean, dry air without organic impurities and an oxygen value of 20.9%. If such an air cylinder is not available, any clean ambient air without detectable contaminants or a charcoal filter can be used.

At the Zero Calibration menu, you can proceed to perform a Zero calibration or bypass Zero calibration and perform a Span calibration. You may also go back to the initial Calibration menu if you want to exit calibration.

- Press [Y/+] to start calibration.
- Press [MODE] to quit and return to the main calibration display.

If you have pressed [Y/+] to enter Zero calibration, then you will see this message:



1. Turn on your Zero calibration gas.
2. Press [Y/+] to start calibration.

   Note: At this point, you may press [MODE] if you decide that you do not want to initiate calibration. This will take you directly to the Calibration menu, highlighted for Span calibration.

3. Zero calibration starts a 30-second countdown and displays this message:

   Zeroing...

During the zeroing process, the instrument performs the Zero calibration automatically and does not require any action on your part.

Note: To abort the zeroing process at any time and proceed to Span calibration, press [N/-] at any time while zeroing is being performed. You will see a confirmation message that says "Zero aborted!" and then the Span calibration menu appears.

When Zero calibration is complete, you see this message:

Zeroing is done!
Reading = 0.0 ppm

The instrument will then show the Calibration menu on its display, with Span Calib highlighted.

Span Calibration

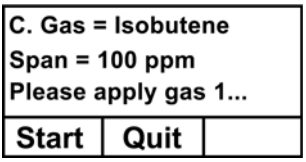
This procedure determines the second point of the sensor calibration curve for the sensor. A cylinder of standard reference gas (span gas) fitted with a 500 cc/min. flow-limiting regulator or a flow-matching regulator is the simplest way to perform this procedure. Choose the 500 cc/min. regulator only if the flow rate matches or slightly exceeds the flow rate of the instrument pump. Alternatively, the span gas can first be filled into a Tedlar bag or delivered through a demand-flow regulator. Connect the calibration adapter to the inlet port of the instrument, and connect the tubing to the regulator or Tedlar bag.

Another alternative is to use a regulator with >500 cc/min flow but allow the excess flow to escape through a T or an open tube. In the latter method, the span gas flows out through an open tube slightly wider than the probe, and the probe is inserted into the calibration tube.

At the Span Calibration menu, you perform a Span calibration. You may also go back to the Zero calibration menu or to the initial Calibration menu if you want to exit calibration.

- Press [Y/+] to enter Span calibration.
- Press [N/-] to skip Span calibration and return to Zero calibration.
- Press [MODE] to exit Span calibration and return to the top calibration menu.

If you have pressed [Y/+] to enter Span calibration, then you will see the name of your Span gas (the default is isobutylene) and the span value in parts per million (ppm). You will also see this message that prompts you:



1. Turn on your span calibration gas.
2. Press [Y/+] to initiate calibration.

Note: You may press [MODE] if you decide that you do not want to initiate calibration. This will abort the span calibration and take you directly to the Calibration menu for Zero calibration.

3. Span calibration starts and displays this message:

   Calibrating...

During the Span calibration process, there is a 30-second countdown and the instrument performs the Span calibration automatically. It requires no actions on your part.

Note: If you want to abort the Span calibration process, press [N/-] at any time during the process. You will see a confirmation message that says "Span is aborted!" and then the Zero calibration menu appears. You can then proceed to perform a Zero calibration, perform a Span calibration, or exit to the topmost Calibration menu.

When Span calibration is complete, you see a message similar to this (the value is an example only):

Span 1 is done!
Reading = 100.0 ppm

The instrument then exits Span calibration and shows the Zero calibration menu on its display.

Note: The reading should be very close to the span gas value.

Exiting Two-Point Calibration In Basic User Level

When you are done performing calibrations, press [MODE], which corresponds with "Back" on the display. You will see the following message:

Updating settings…

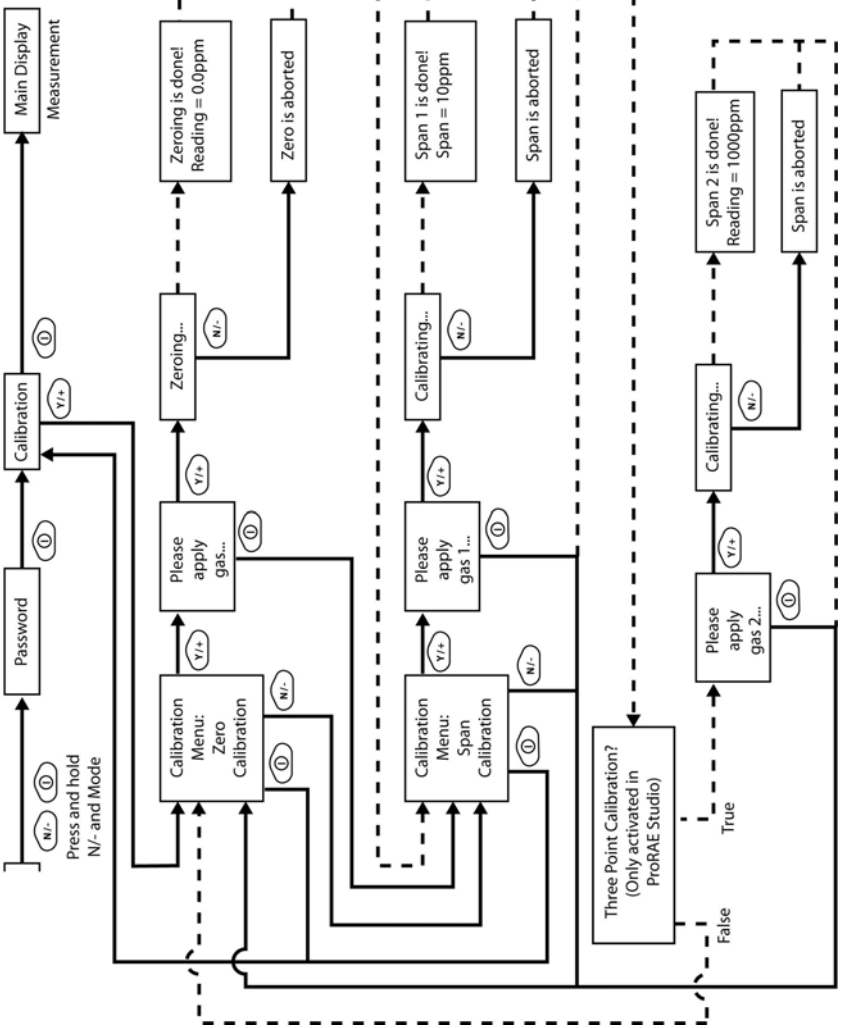
The instrument updates its settings and then returns to the main display. It begins or resumes monitoring.

Three-Point Calibration

For enhanced accuracy, it is possible to perform a second Span calibration in addition to the Zero and Span calibrations outlined in the previous section. Your instrument first must be set to allow this third calibration. This requires using ProRAE Studio software and a PC, as well as a higher concentration of calibration gas.

Note: Once the third calibration is set, you do not need to use ProRAE Studio to allow future 3-point calibrations. Also, you can only disable 3-point calibration capability by using ProRAE Studio again.

Perform the Zero and Span calibrations. After the first Span calibration (Span 1) is completed, the display a second Span calibration (Span 2) can be performed. The process is identical to the first calibration. As in the Span 1 calibration, you may exit and return to the Zero calibration screen if you choose not to perform this calibration or to abort it.



Note: Dashed line indicates automatic progression.

Span 2 Calibration

A cylinder of standard reference gas (span gas) fitted with a 500 cc/min. flow-limiting regulator or a flow-matching regulator is the simplest way to perform this procedure.

Note: This gas should be of a higher concentration than the gas used for Span 1 calibration.

Choose the 500 cc/min. regulator only if the flow rate matches or slightly exceeds the flow rate of the instrument pump. Alternatively, the span gas can first be filled into a Tedlar bag or delivered through a demand-flow regulator. Connect the calibration adapter to the inlet port of the instrument, and connect the tubing to the regulator or Tedlar bag.

Another alternative is to use a regulator with >500 cc/min flow but allow the excess flow to escape through a T or an open tube. In the latter method, the span gas flows out through an open tube slightly wider than the probe, and the probe is inserted into the calibration tube.

At the Span Calibration menu, you perform a Span calibration. You may also go back to the Zero calibration menu or to the initial Calibration menu if you want to exit calibration.

- Press [Y/+] to enter Span 2 calibration.
- Press [N/-] to skip Span calibration and return to Zero calibration.
- Press [MODE] to exit Span calibration and return to the top calibration menu.

If you have pressed [Y/+] to enter Span calibration, then you will see the name of your Span gas (the default is isobutylene) and the span value in parts per million (ppm). You will also see this message that prompts you:

Please apply gas...

4. Turn on your span calibration gas.
5. Press [Y/+] to initiate calibration.

Note: You may press [MODE] if you decide that you do not want to initiate calibration. This will take you directly to the Calibration menu for Zero calibration.

6. Span calibration starts a 30-second countdown and displays this message:

   Calibrating...

During the Span calibration process, the instrument performs the Span calibration automatically and does not require any action on your part.

Note: If you want to abort the Span calibration process, press [N/-] at any time during the process. You will see a confirmation message that says "Span is aborted!" and then the Zero calibration menu will appear. You can then proceed to perform a Zero calibration, perform a Span calibration, or exit to the topmost Calibration menu.

When Span calibration is complete, you will see a message similar to this (the value shown here is for example only):

Span 2 is done!
Reading = 1000 ppm

The instrument then exits Span calibration and shows the Zero calibration menu on its display.

Note: The reading should be very close to the span gas value.

Exiting Three-Point Calibration

When you are done performing calibrations, press [MODE], which corresponds with "Back" on the display. You will see the following message:

Updating settings…

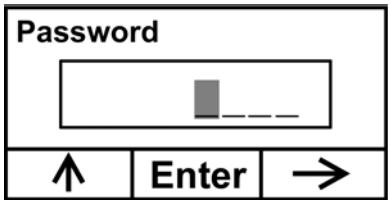
The instrument updates its settings and then returns to the main display. It begins or resumes monitoring.

Programming Mode

Programming Mode can be entered from either Hygiene Mode or Search Mode. If the current user mode is Basic, you must provide a 4-digit password to enter.

Entering Programming Mode

1. Press and hold [MODE] and [N/-] until you see the Password screen.



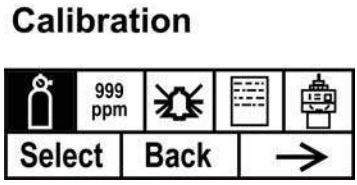
2. Input the 4-digit password:

- Increase the number from 0 through 9 by pressing [Y/+].
- Step from digit to digit using [N/-].
- Press [MODE] when you are done.

If you make a mistake, you can cycle through the digits by pressing [N/-] and then using [Y/+] to change the number in each position.

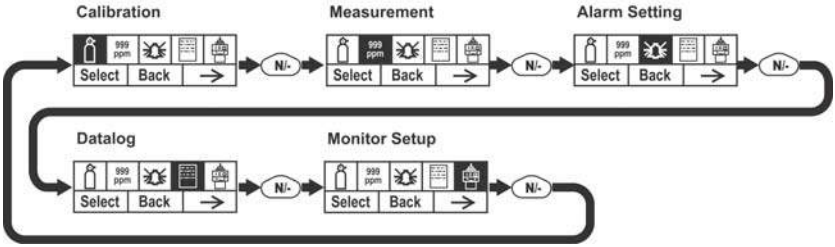
Note: The default password is 0000.

When you have successfully entered Programming Mode, you see this screen:



Note: The password can only be changed by connecting the instrument to a PC running ProRAE Studio software. Follow the instructions in ProRAE Studio to change it.

The Calibration label is shown and its icon is highlighted, but you can press [N/-] to step from one programming menu to the next, with the name of the menu shown at the top of the display and the corresponding icon highlighted. As you repeatedly press [N/-], the selection moves from left to right, and you see these screens:

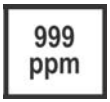
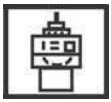


Note: When you reach Monitor Setup and press [N/-], the menu cycles back to Calibration.

Programming Mode Menus

The Programming Mode allows anyone with the password to change the instrument's settings, calibrate the instrument, modify the sensor configuration, enter user information, etc. Programming Mode has five menus. Each menu includes several sub-menus to perform additional programming functions.

This table shows the menus and sub-menus:

				
Calibration	**Measurement**	**Alarm Setting**	**Datalog**	**Monitor Setup**
Zero Calibration	Meas. Gas	High Alarm	Clear Datalog	Op Mode
Span Calibration	Meas. Unit	Low Alarm	Interval	Site ID
		STEL Alarm	Data Selection	User ID
		TWA Alarm	Datalog Type	User Mode
		Alarm Type		Date
		Buzzer & Light		Time
				Pump Duty Cycle
				Pump Speed
				Temperature Unit
				Language
				Radio Power
				Real Time Protocol
				Power On Zero
				Unit ID
				LCD Contrast

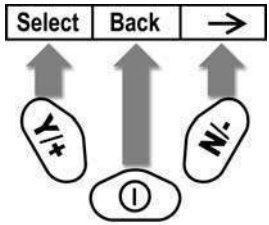
Once you enter Programming Mode, the LCD displays the first menu, Calibration. Each subsequent menu is accessed by pressing [N/-] repeatedly until the desired menu is displayed. To enter a sub-menu of a menu, press [Y/+].

Exiting Programming Mode

To exit Programming Mode and return to normal operation, press [MODE] once at any of the programming menu displays. You will see "Updating Settings…" as changes are registered and the mode changes.

Navigating Programming Mode Menus

Navigating through the Programming Mode menus is easy and consistent, using a single interface format of "Select," "Back" and "Next" at the top level. The three control buttons correspond to these choices as shown:



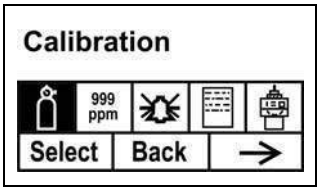
Note: Pressing [MODE] in the Programming Mode's top level causes the instrument to exit Programming Mode and return to monitoring.

The three keys perform the following functions in Programming Mode:

Key	Function in Programming Mode
[MODE]:	Exit menu when pressed momentarily or exit data entry mode
[Y/+]:	Increase alphanumerical value for data entry or confirm (yes) for a question
[N/-]:	Provides a "no" response to a question

Calibration

Two types of calibration are available: Zero (fresh air) and Span.



Select Zero or Span Calibration by pressing [N/+]. Once your choice is highlighted, press [Y/+].

Zero Calibration

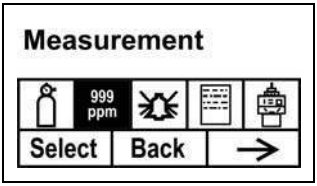
The procedure for performing a zero calibration is covered on page 35.

Span Calibration

The procedure for performing a basic span calibration is covered on page 35.

Measurement

The sub-menus for Measurement are Measurement Gas and Measurement Unit.



Meas. Gas

Measurement gases are organized in four lists:

- My List is a customized list of gases that you create. It contains a maximum of 10 gases and can only be built in ProRAE Studio on a PC and transferred to the instrument. **Note:** The first gas in the list is always isobutylene (it cannot be removed from the list).
- Last Ten is a list of the last ten gases used by your instrument. The list is built automatically and is only updated if the gas selected from Custom Gases or Library is not already in the Last Ten. This ensures that there is no repetition.
- Gas Library is a library that consists of all the gases found in RAE Systems' Technical Note TN-106 (available online at www.raesystems.com).
- Custom Gases are gases with user-modified parameters. Using ProRAE Studio, all parameters defining a gas can be modified, including the name, span value(s), correction factor, and default alarm limits.

1. Scroll through each list by pressing [N/-].
2. Press [Y/+] to select one (My List, Last Ten, Gas Library, or Custom Gases).

3. Once you are in one of the categories, press [N/-] to scroll through its list of options and [Y/+] to select one. (If you press [MODE], you exit to the next submenu.)
4. Press [Y/+] to save your choice or [N/-] to undo your selection.

Leave the sub-menu and return to the Programming Mode menus by pressing [MODE].

Meas. Unit

Standard available measurement units include:

Abbreviation	Unit	MiniRAE 3000
ppm	parts per million	Yes
ppb	parts per billion	
mg/m3	milligrams per cubic meter	Yes
ug/m3	micrograms per cubic meter	

- Scroll through the list by pressing [N/-].
- Select by pressing [Y/+].
- Save your selection by pressing [Y/+] or undo your selection by pressing [N/-].

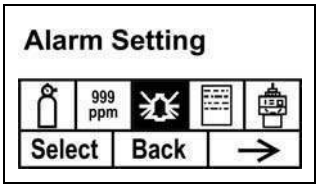
Leave the sub-menu and return to the Programming Mode menus by pressing [MODE].

Alarm Setting

During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings: Low, High, TWA and STEL). If the concentration exceeds any of the preset limits, the loud buzzer and red flashing LED are activated immediately to warn of the alarm condition.

An alarm signal summary is shown on page 27.

In this menu, you can change the High and Low alarm limits, the STEL limit, and the TWA. Press [Y/+] to to enter the Alarm Setting menu. **Note:** All settings are shown in ppb (parts per billion), or µg/m^3 (micrograms per cubic meter), depending on your setting.



1. Scroll through the Alarm Limit sub-menu using the [N/-] key until the display shows the desired limit to be changed (High Alarm, Low Alarm, STEL Alarm, and TWA Alarm)
2. Press [Y/+] to select one of the alarm types. The display shows a flashing cursor on the left-most digit of the previously stored alarm limit.
3. Press [Y/+] to increase each digit's value.
4. Press [N/-] to advance to the next digit.
5. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

Press [MODE] when you are done.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

When all alarm types have been changed or bypassed, press [MODE] to exit to the Programming Menu.

High Alarm

You can change the High Alarm limit value. The value is typically set by the instrument to match the value for the current calibration gas. It is expressed in parts per billion (ppb). **Note:** The default value depends on the measurement gas.

To change the High Alarm value:

1. Press [Y/+] to increase each digit's value.
2. Press [N/-] to advance to the next digit.
3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.
When you have completed your selections, press [MODE]. You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

Press [Y/+] to save the changes.
Press [N/-] to undo the changes and revert to the previous settings.

Low Alarm

You can change the Low Alarm limit value. The value is typically set by the instrument to match the value for the current calibration gas. It is expressed in parts per billion (ppb). **Note:** The default value depends on the measurement gas.

To change the Low Alarm value:

1. Press [Y/+] to increase each digit's value.
2. Press [N/-] to advance to the next digit.
3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

When you have completed your selections, press [MODE]. You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

STEL Alarm

You can change the STEL Alarm limit value. The value is typically set by the instrument to match the value for the calibration gas. It is expressed in parts per billion (ppb). **Note:** The default value depends on the measurement gas.

To change the STEL Alarm value:

1. Press [Y/+] to increase each digit's value.
2. Press [N/-] to advance to the next digit.
3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

When you have completed your selections, press [MODE]. You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

TWA Alarm

You can change the TWA (time-weighted average) Alarm limit value. The value is typically set by the instrument to match the value for the calibration gas. It is expressed in parts per billion (ppb). **Note:** The default value depends on the measurement gas.

To change the TWA Alarm value:

1. Press [Y/+] to increase each digit's value.
2. Press [N/-] to advance to the next digit.
3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

When you have completed your selections, press [MODE]. You will see two choices:

- Save
- Undo

You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

Alarm Type

There are two selectable alarm types:

Latched — When the alarm is triggered, you can manually stop the alarm.
The latched setting only controls alarms for High Alarm, Low Alarm, STEL Alarm, and TWA alarm.

Note: To clear an alarm when the instrument is set to "Latched," press [Y/+] when the main (Reading) display is shown.

Automatic Reset — When the alarm condition is no longer present, the alarm stops and resets itself.

1. Press **[N/-]** to step from one alarm type to the other.
2. Press **[Y/+]** to select an alarm type.

When you have completed your selections, press [MODE].

You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

Buzzer & Light

The buzzer and light alarms can be programmed to be on or off individually or in combination. Your choices are:

- Both on
- Light only
- Buzzer only
- Both off

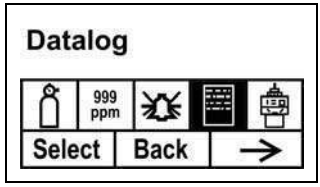
1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates your selection).
3. When you have completed your selections, press [MODE].

   You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

   - Press [Y/+] to save the changes.
   - Press [N/-] to undo the changes and revert to the previous settings.

Datalog

The instrument calculates and stores the concentration and ID of each sample taken. In the datalog sub-menu, a user can perform the tasks and functions shown below.



1. Scroll through the Datalog sub-menu using the [N/-] key until the display shows the desired parameter to be changed:

   Clear Datalog
   Interval
   Data Selection
   Datalog Type

2. Press [Y/+] to make your selection. Exit by pressing [MODE] for Back.

Clear Datalog

This erases all the data stored in the datalog.

Note: Once the datalog is cleared, the data cannot be recovered.

Press [Y/+] to clear the datalog. The display asks, "Are you sure?"

- Press [Y/+] if you want to clear the datalog. When it has been cleared, the display shows "Datalog Cleared!"
- Press [N/-] if you do not want to clear the datalog.

The display changes, and you are taken to the next sub-menu, Interval.

Interval

Intervals are shown in seconds. The default value is 60 seconds. The maximum interval is 3600 seconds.

1. Press [Y/+] to increase each digit's value.
2. Press [N/-] to advance to the next digit.
3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

When you have completed your selections, press [MODE].

You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

Data Selection

Data Selection allows you to select which types of data are stored and made available when you offload your datalog to a computer via ProRAE Studio software.

You can choose any or all of three types of data (you must choose at least one):

- Average
- Maximum
- Minimum

1. Press [N/-] to step from one option to the next. The highlighter indicates your choice.
2. Press [Y/+] to toggle your selection on or off (the check box indicates "on" with an "X").
3. When you have completed your selections, press [MODE].

You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

Datalog Type

The instrument has three datalog types:

Auto	Default mode. Collects datalog information when the instrument is sampling.
Manual	Datalogging occurs only when the instrument's datalogging is manually started (see below for details).
Snapshot	Datalogs only during single-event capture sampling.

Note: You can only choose one datalog type to be active at a time.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
3. When you have completed your selection, press [MODE].

You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.

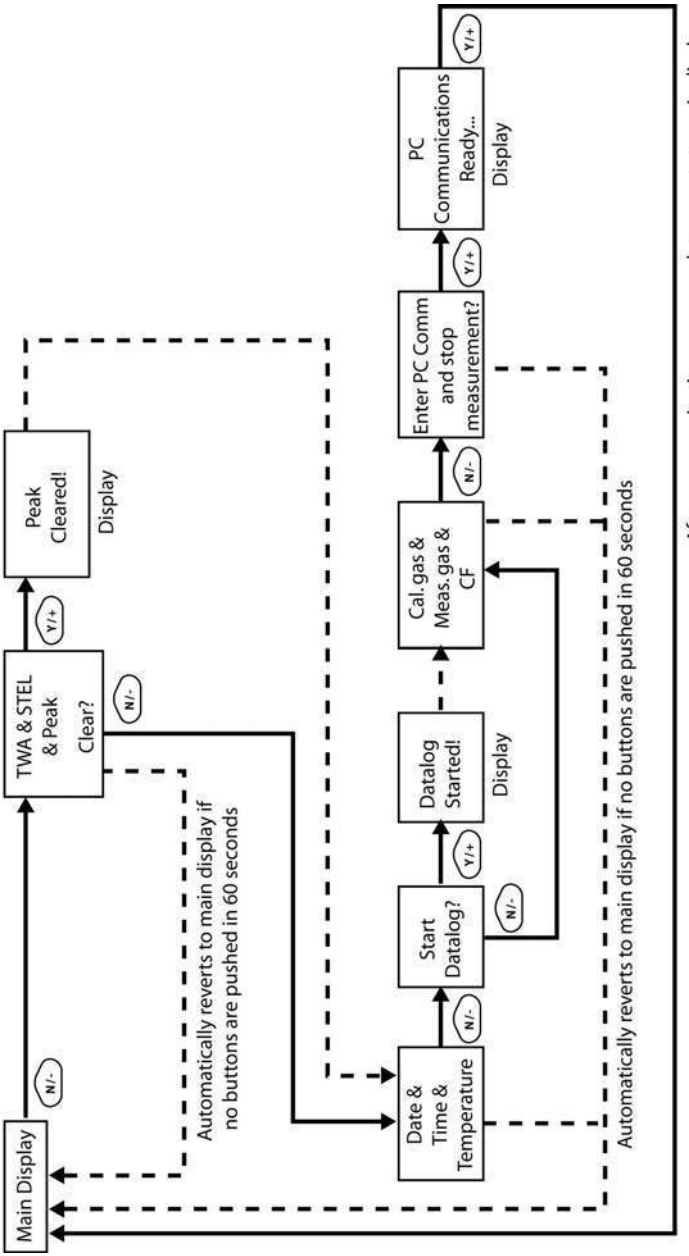
Press [N/-] to undo the changes and revert to the previous settings.

Manual Datalog

When the instrument is set to Manual Datalog, you turn datalogging on and off by stepping through the displays from the Main Display, and then pressing the keys to select datalog on/off functions.

- When you reach the screen that says "Start Datalog?" press [Y/+] to start it. You see "Datalog Started," confirming that datalogging is now on.

When you reach the screen that says "Stop Datalog?" press [Y/+] to stop it. You see "Datalog Stopped," confirming that datalogging is now off.



Note: Dashed line indicates automatic progression.

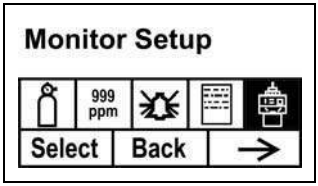
Snapshot Datalog

When the instrument is in Snapshot datalogging mode, it captures a single "snapshot" of the data at the moment of your choosing. Whenever the instrument is on and it is set to Snapshot, all you have to do is press [MODE] each time you want to capture a snapshot of the data at that instant.

When you send the data to a computer using ProRAE Studio, the data snapshots are uniquely identified by time and other parameters.

Monitor Setup

Many settings can be accessed in this menu, including setting the date and time and adjusting the pump's on/off duty cycle.



Op Mode

Under Monitor Setup is "Op Mode."

Press [Y/+] to select.

You see two options (one is highlighted):

Hygiene
Search

The current mode is indicated by a dark circle within the circle in front of either Hygiene or Search.

1. Select Hygiene or Search by pressing [N/-]. The highlighting changes from one to the other each time you press [N/-].
2. Press [Y/+] to select that mode for the instrument.
3. Press [MODE] when you want to register your selection to place the instrument in the selected mode.
4. Press [Y/+] to commit the change and exit to the Monitor Setup screen, or press [N/-] to Undo (exit to the Monitor Setup screen without changing the Mode).

Site ID

Enter an 8-digit alphanumeric/character Site ID in the programming mode. This Site ID is included in the datalog report.

1. Press [Y/+] and the display shows the current site ID. Example: "RAE00001." Note that the left-most digit flashes to indicate it is the selected one.
2. Press [Y/+] to step through all 26 letters (A to Z) and 10 numerals (0 to 9).
   Note: The last four digits must be numerals.
3. Press [N/-] to advance to the next digit. The next digit to the right flashes.

   Repeat this process until all eight digits of the new site ID are entered.

Press [MODE] to exit.

If there is any change to the existing site ID, the display shows "Save?" Press [Y/+] to accept the new site ID. Press [N/-] to discard the change and move to the next sub-menu.

User ID

Enter an 8-digit alphanumeric User ID in the programming mode. This User ID is included in the datalog report.

1. Press [Y/+] and the display shows the current User ID. Example: "RAE00001." Note that the left-most digit flashes to indicate it is the selected one.
2. Press [Y/+] to step through all 26 letters (A to Z) and 10 numerals (0 to 9).
3. Press [N/-] to advance to the next digit. The next digit to the right flashes.

   Repeat this process until all eight digits of the new User ID are entered.

Press [MODE] to exit.

If there is any change to the existing User ID, the display shows "Save" Press [Y/+] to accept the new site ID. Press [N/-] to discard (undo) the change and move to the next sub-menu.

User Mode

The instrument has two user modes:

Basic Basic users can only see and use a basic set of functions.
Advanced Advanced users can see all screens and perform all available functions.

Note: The default value for User Mode is Basic.

To change the User Mode:

1. Press [N/-] to step from one option to the next. The highlighting changes each time you press [N/-].
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
3. When you have completed your selection, press [MODE].
4. Press [Y/+] to accept the new User Mode. Press [N/-] to discard the change and move to the next sub-menu.

Date

The Date is expressed as Month/Day/Year, with two digits for each.

1. Press [Y/+] and the display shows the current date. Note that the left-most digit flashes to indicate it is selected.
2. Press [Y/+] to step through all 10 numerals (0 to 9).
3. Press [N/-] to advance to the next digit. The next digit to the right flashes.

   Repeat this process until all six digits of the new date are entered.

Press [MODE] to exit.

- Press [Y/+] to save the new date.
- Press [N/-] to undo the change and move to the next sub-menu.

Time

The Time is expressed as Hours/Minutes/Seconds, with two digits for each. The time is in 24-hour (military) format.

1. Press [Y/+] and the display shows the current time. Note that the left-most digit flashes to indicate it is selected.
2. Press [Y/+] to step through all 10 numerals (0 to 9).

3. Press [N/-] to advance to the next digit. The next digit to the right flashes.

   Repeat this process until all six digits of the new time are entered.

Press [MODE] to exit.

- Press [Y/+] to save the new date.
- Press [N/-] to undo the change and move to the next sub-menu.

Duty Cycle

The pump's duty cycle is the ratio of its on time to off time. The duty cycle ranges from 50% to 100% (always on), and the period is 10 seconds. Therefore, a duty cycle of 60% means that the pump is on for 6 seconds and off for four seconds. Duty cycling is employed by the instrument to clean the PID. A lower duty cycle has a greater effect on keeping the PID clean than a higher duty cycle.

Important! Pump duty cycling is interrupted when the instrument senses a gas. The pump's duty cycle is disabled when the measurement is greater than the 2ppm threshold and is re-enabled when the reading falls below 90% of the threshold (1.8 ppm).

1. Press [Y/+] to increase the value.
2. When you have completed your selection, press [MODE].
   - Press [Y/+] to save the new duty cycle value.
   - Press [N/-] to undo the change and move to the next sub-menu.

Temperature Unit

The temperature display can be switched between Fahrenheit and Celsius units.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
3. When you have completed your selection, press [MODE].
   - Press [Y/+] to save the new temperature unit.
   - Press [N/-] to undo the change and move to the next sub-menu.

Pump Speed

The pump can operate at two speeds, high and low. Running at low speed is quieter and conserves a small amount of power. There is almost no difference in sampling accuracy.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
3. When you have completed your selection, press [MODE].
   - Press [Y/+] to save the new temperature unit.
   - Press [N/-] to undo the change and move to the next sub-menu.

Language

English is the default language, but other languages can be selected for the instrument.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
3. When you have completed your selection, press [MODE].
   - Press [Y/+] to save your new language choice.
   - Press [N/-] to undo it and return to the previous language selection.

Radio Power

The radio connection can be turned on or off.

1. Press [N/-] to step from one option to the next (on or off).
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates that the option is selected).
3. When you have completed your selection, press [MODE].

   - Press [Y/+] to accept the new radio setting (on or off).
   - Press [N/-] to discard the change and move to the next sub-menu.

Real Time Protocol

Real Time Protocol is the setting for data transmission.

The choices are:

P2M (cable)	Point to multipoint. Data is transferred from the instrument to multiple locations using a wired connection. Default data rate: 19200 bps.
P2P (cable)	Point to point. Data is transferred only between the instrument and one other location, such as a computer. Default data rate: 9600 bps.
P2M (wireless)	Point to multipoint, wireless. Data is transferred wirelessly and can be received by multiple receivers.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
3. When you have completed your selection, press [MODE].
   - Press [Y/+] to save the new real-time communications protocol.
   - Press [N/-] to undo the change and move to the next sub-menu.

Power On Zero

When Power On Zero is on, the instrument performs a zero calibration when it is turned on.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates your selection).
3. When you have completed your selection, press [MODE].
   - Press [Y/+] to save the change.
   - Press [N/-] to discard the change and move to the next sub-menu.

Unit ID

This three-digit number keeps data separated by instrument when more than one instrument is used in a network. If multiple sensing units are attempting to communicate with the same Host, then the units must all have a different Unit ID.

1. Press [Y/+] to step through all 10 numerals (0 to 9). If you pass the numeral you want, keep pressing [Y/+]. After it counts up to 9, it starts counting up from 0 again.
2. Press [N/-] to advance to the next digit. The next digit to the right flashes.

Repeat this process until all three digits of the Unit ID are entered.

3. Press [MODE] when you are done.
   - Press [Y/+] to save the change.
   - Press [N/-] to discard the change and move to the next sub-menu.

LCD Contrast

The display's contrast can be increased or decreased from its default setting. You may not need to ever change the default setting, but sometimes you can optimize the display to suit extreme temperature and ambient brightness/darkness conditions.

- The minimum value is 20.
- The maximum value is 60.

1. Press [Y/+] to increase the value or [N/-] to decrease the value.
2. Press [MODE] to save your selection.
   - Press [Y/+] to save your new contrast value.
   - Press [N/-] to undo it and return to the previous value.

Hygiene Mode

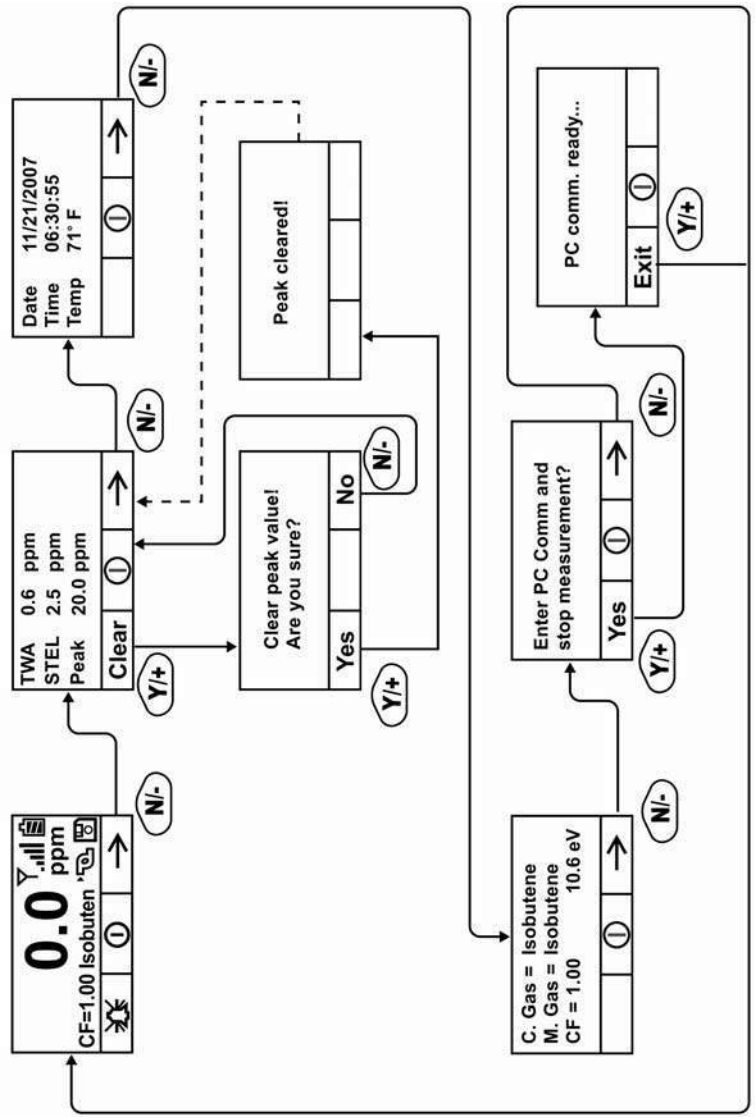
The instrument usually operates in Hygiene Mode, which provides basic functionality. However, it is possible to operate it in a second mode called Search Mode. Here are the primary differences:

Hygiene Mode: Automatic measurements, continuously running and datalogging, and calculates additional exposure values.

Search Mode: Manual start/stop of measurements and display of certain exposure values.

Basic User Level & Hygiene Mode

The default setting is navigated in the following way:



Note: Dashed line indicates automatic progression.

Pressing [N/-] steps you from screen to screen. Options include clearing the Peak value and turning on the instrument's PC Communications for data transfer to a PC.

Entering Search Mode From Hygiene Mode

In order to change the instrument's operational mode from Hygiene Mode to Search Mode, you must enter the password-protected Programming Mode:

1. Hold [MODE] and [N/-] until you see the password screen.
2. Use [Y/+] to increment to the number you want for the first digit. (If you pass by the desired number, press [Y/+] until it cycles through to 0 again. Then press [Y/+] until you reach the desired number.)
3. Press [N/-] to advance to the next digit.
4. Again press [Y/+] to increment the number.
5. Press [N/-] to advance to the next digit.

Continue the process until all four numbers of the password have been input. Then press [MODE] to proceed.

The screen changes to icons with the label "Calibration."

1. Press [N/-] to advance to "Monitor Setup."
2. Press [Y/+] to select Monitor Setup.

Under Monitor Setup, you will see "Op Mode."

Press [Y/+] to select.

You will see:

Hygiene
Search

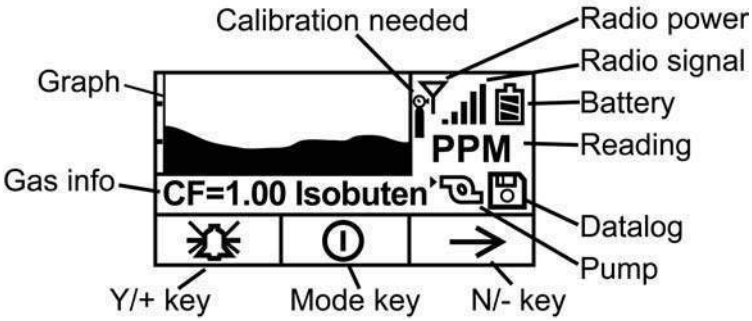
The current mode is indicated by a dark circle within the circle in front of either Hygiene or Search.

1. Select Hygiene or Search by pressing [N/-].
2. Press [Y/+] to place the instrument into the selected mode.

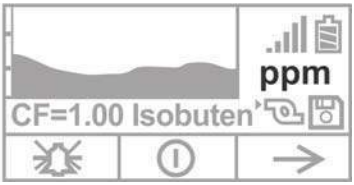
3. Press [MODE] when you want to register your selection to place the instrument in the selected mode.
4. Press [Y/+] to commit the change and exit to the Monitor Setup screen, or press [N/-] to Undo (exit to the Monitor Setup screen without changing the Mode).

Optional Graphic Screen In Search Mode

Using ProRAE Studio, you can set your instrument to show a graphic display instead of a numeric display of ongoing data. Consult your ProRAE Studio disc for information.



During sampling, the display's readings are shown numerically, plus the graph tracks the highest readings over time. The numeric reading alternates between the value and the measurement units, as well:



Advanced User Level (Hygiene Mode Or Search Mode)

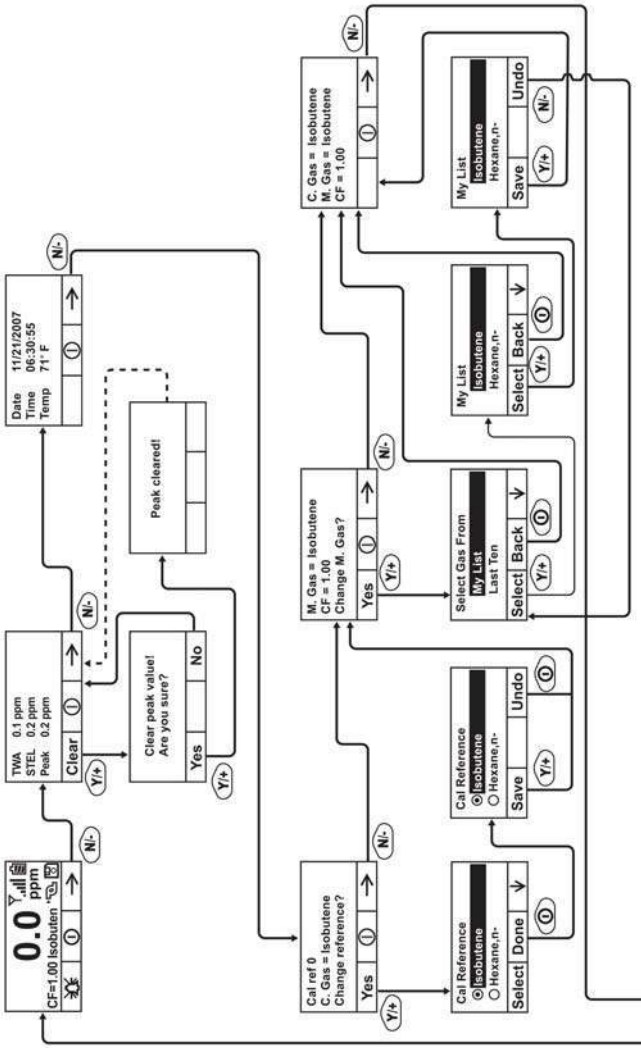
The User Mode called Advanced User Level allows a greater number of parameters to be changed than Basic User Level. It can be used with either of the Operation Modes, Hygiene Mode or Search Mode.

Advanced User Level & Hygiene Mode

With the instrument in Operation Mode: Hygiene Mode, enter User Mode: Advanced User Level (refer to the section called Monitor Mode for instructions).

Once you are in Advanced User Level and Hygiene Mode together, you can change the calibration reference and measurement gas, in addition to performing normal monitoring functions.

Pressing [N/-] progresses through the screens, while pressing [Y/+] selects options. Pressing [MODE] makes menu choices when it is shown for "Done" or "Back." Pressing and holding [Mode] whenever the circle with a vertical line in the middle is shown activates the countdown to shutoff.

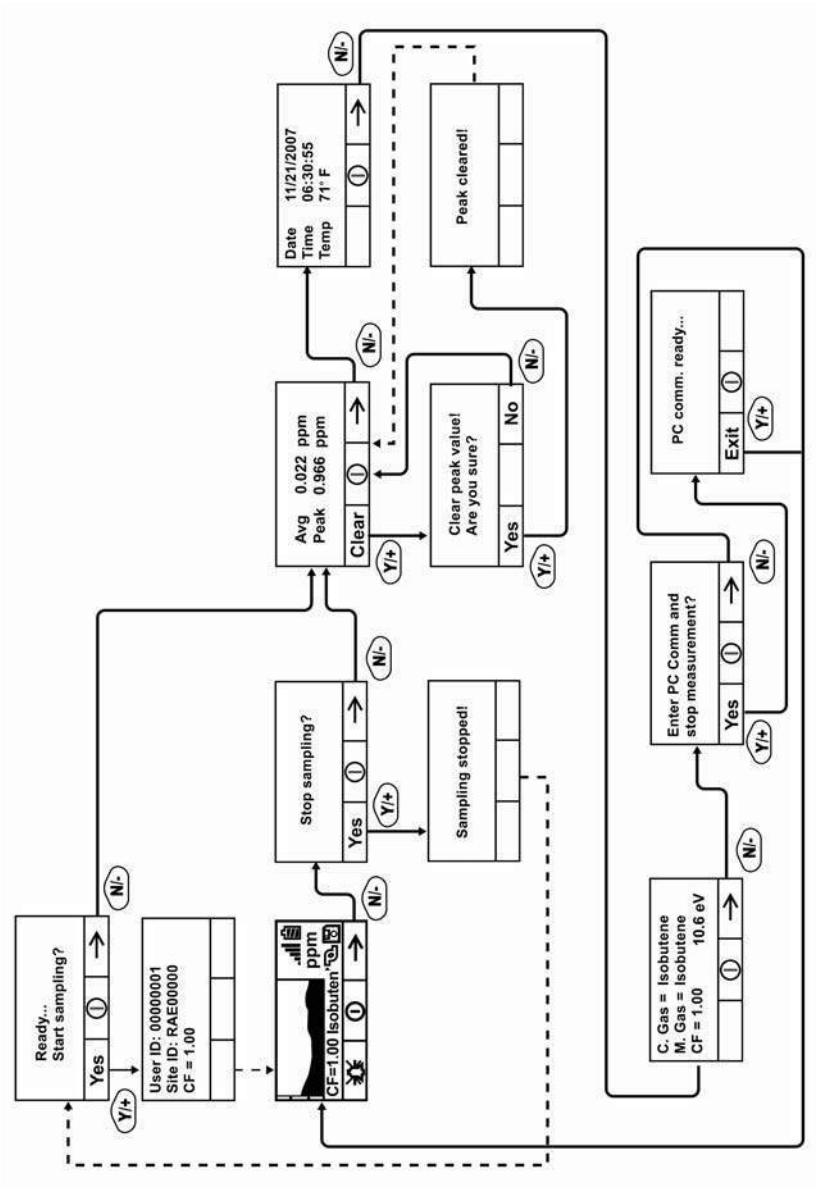


Note: Dashed line indicates automatic progression.

Basic User Level & Search Mode

With the instrument in Operation Mode: Search Mode, enter User Mode and select Basic User Level (refer to the section called User Mode for instructions).

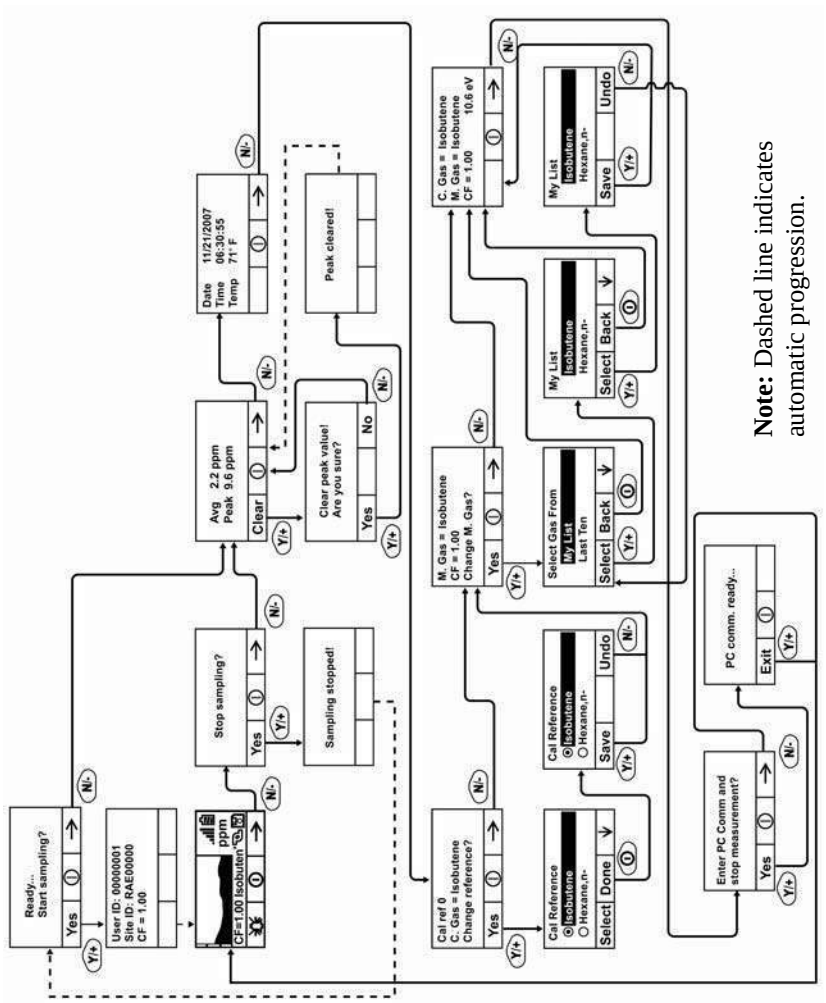
When the instrument is in Search Mode, it only samples when you activate sampling. When you see the display that says, "Ready…Start sampling?" press [Y/+] to start. The pump turns on and the instrument begins collecting data. To stop sampling, press [N/-] while the main display is showing. You will see a new screen that says, "Stop sampling?" Press [Y/+] to stop sampling. Press [N/-] if you want sampling to continue.



Note: Dashed line indicates automatic progression.

Advanced User Level & Search Mode

With the instrument in Operation Mode: Search Mode, enter User Mode and select Advanced User Level (refer to the section called Monitor Mode for instructions). Operation is similar to Basic User Level & Sampling Mode, but now allows you to change calibration and measurement reference gases. Refer to the section on measurement gases on page 52 for more details.



Diagnostic Mode

IMPORTANT! Diagnostic Mode is designed for servicing and manufacturing, and therefore is not intended for everyday use, even by advanced users. It provides raw data from sensors and about settings, but only allows adjustment of pump stall parameters, which should only be changed by qualified personnel.

Note: If the instrument is turned on in Diagnostic Mode and you switch to User Mode, datalog data remains in raw count form. To change to standard readings, you must restart the instrument.

Entering Diagnostic Mode

Note: To enter Diagnostic Mode, you must begin with the instrument turned off.

Press and hold [Y/+] and [MODE] until the instrument starts.

The instrument goes through a brief startup, and then displays raw data for the PID sensor. These numbers are raw sensor readings without calibration. The instrument is now in Diagnostic Mode.

Note: In Diagnostic Mode, the pump and lamp are normally on.

You can enter Programming Mode and calibrate the instrument as usual by pressing both [MODE] and [N/-] for three seconds.

You can enter Monitoring Mode by pressing [MODE] and [Y/+] together for three seconds.

Once the instrument is started up in Diagnostic Mode, you can switch between Diagnostic Mode and Monitoring Mode by pressing and holding [MODE] and [Y/+] simultaneously for two seconds.

In Diagnostic mode, you can step through parameter screens by pressing [MODE].

Adjusting The Pump Stall Threshold

If the gas inlet is blocked but the pump does not shut down, or the pump shuts down too easily with a slight blockage, the pump stall threshold value may be set too high or too low.

Use the following steps to adjust the pump stall threshold:

Pump High

In Diagnostic Mode, press the [MODE] key until "Pump High" is displayed. The display shows the maximum, minimum, and stall values for the pump at its high speed. Write down the "Max" reading.

Block the gas inlet and watch the pump current reading (labeled "I") increase. Write down its blocked reading. **Note:** If the pump current reading does not increase significantly (less than 10 counts), then there may be a leak in the gas inlet or the pump is weak or defective.

Add the two readings you wrote down. This is the average of the maximum block count and the maximum idle count. Divide that number by 2. Use the [Y/+] or [N/-] key to increase or decrease the stall value to equal that number.

Press the [MODE] key to exit this display.

Pump Low

In Diagnostic Mode, press the [MODE] key until "Pump Low" is displayed. The display shows the maximum, minimum, and stall values for the pump at its low speed. Write down the "Max" reading.

Block the gas inlet and watch the pump current reading (labeled "I") increase. Write down its blocked reading. **Note:** If the pump current reading does not increase significantly (less than 10 counts), then there may be a leak in the gas inlet or the pump is weak or defective.

Add the two readings you wrote down. This is the average of the maximum block count and the maximum idle count. Divide that

number by 2. Use the [Y/+] or [N/-] key to increase or decrease the stall value to equal that number.

Press the [MODE] key to exit this display.

Exiting Diagnostic Mode

You can exit Diagnostic Mode and go directly to Programming Mode or Monitor Mode as outlined above, or you can exit Diagnostic Mode completely.

To exit Diagnostic Mode so that it cannot be re-entered without a restart:

Shut down the instrument. When it is off, restart it by holding the [MODE] key. Diagnostic Mode cannot be entered until the instrument is restarted as outlined in "Entering Diagnostic Mode."

Transferring Data To & From A Computer

Once you have connected your instrument cradle to the PC, you can can transfer data, including a download of the datalog to the computer and updates of firmware to the instrument (should this ever be necessary).

Downloading The Datalog To A PC

1. Connect the data cable to the PC and the cradle.
2. Place the instrument into its cradle. The charging LED should be illuminated.
3. Start ProRAE Studio on your PC.
4. From ProRAE Studio, select "Operation" and select Setup Connection.
5. Select the COM port to establish a communication link between the PC and the instrument.
6. To receive the datalog in the PC, select "Downlog Datalog."
7. When you see "Unit Information," click OK.

During the data transfer, the display shows a progress bar.

When the transfer is done, you will see a screen with the datalog information. You can now export this datalog for other use or printing.

Uploading Firmware To The instrument From A PC

Uploading new firmware to your instrument requires connecting the instrument and PC. Follow these steps to make the connection:

1. Connect the data cable to the PC and the cradle.
2. Place the instrument into its cradle. The charging LED should be illuminated.
3. Start RAEProgrammer 7000 on your PC.
4. From RAEProgrammer 7000, select "Operation" and select Setup Connection.
5. Select the COM port to establish a communication link between the PC and the instrument.
6. Select Operation → Download Firmware.

Once communication is established, follow the instructions that accompany RAEProgrammer 7000 and the firmware to upload the new firmware to your instrument.

Note: Check for the latest updates to ProRAEProgrammer 7000 at www.raesystems.com.

Maintenance

The major maintenance items of the instrument are:

- Battery pack
- Sensor module
- PID lamp
- Sampling pump
- Inlet connectors and filters

Note: Maintenance should be performed by qualified personnel only.

NOTE: The printed circuit board of the instrument is connected to the battery pack even if the power is turned off. **Therefore, it is very important to disconnect the battery pack before servicing or replacing any components inside the instrument**. **Severe damage to the printed circuit board or battery may occur if the battery pack is not disconnected before servicing the unit.**

Battery Charging & Replacement

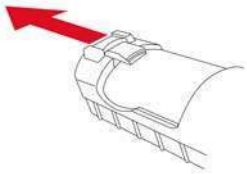
When the display shows a flashing empty battery icon, the battery requires recharging. It is recommended to recharge the instrument upon returning from fieldwork. A fully charged battery runs a instrument for 16 hours continuously. The charging time is less than 8 hours for a fully discharged battery. The battery may be replaced in the field (in areas known to be non-hazardous), if required.

WARNING!

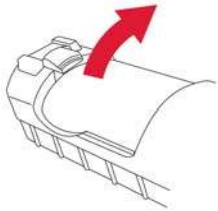
To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non-hazardous. **Remove and replace battery only in areas known to be non-hazardous.**

Replacing The Li-ion Battery

1. Turn off the instrument.
2. Located on the rear of the instrument is a battery tab. Slide it down to unlock the battery.



3. Remove the battery pack from the battery compartment by tilting it out.



4. Replace a fully charged spare battery pack inside the battery compartment. Make sure the battery pack is oriented properly inside the compartment.
5. Slide the capture tab back up to its locked position.

Replacing The Alkaline Battery Adapter

An alkaline battery adapter is supplied with each instrument. The adapter (part number 059-3052-000) accepts four AA alkaline batteries (use only Duracell MN1500) and provides approximately 12 hours of operation. The adapter is intended to be used in emergency situations when there is no time to charge the Li-ion battery pack.

To insert batteries into the adapter:

1. Remove the three Philips-head screws to open the compartment.
2. Insert four fresh AA batteries as indicated by the polarity (+/-) markings.
3. Replace the cover. Replace the three screws.

To install the adapter in the instrument:

1. Remove the Li-ion battery pack from the battery compartment by sliding the tab and tilting out the battery.
2. Replace it with the alkaline battery adapter
3. Slide the tab back into place to secure the battery adapter.

IMPORTANT!

Alkaline batteries cannot be recharged. The instrument's internal circuit detects alkaline batteries and will not allow recharging. If you place the instrument in its cradle, the alkaline battery will not be recharged. The internal charging circuit is designed to prevent damage to alkaline batteries and the charging circuit when alkaline batteries are installed inside the instrument.

Note: When replacing alkaline batteries, dispose of old ones properly.

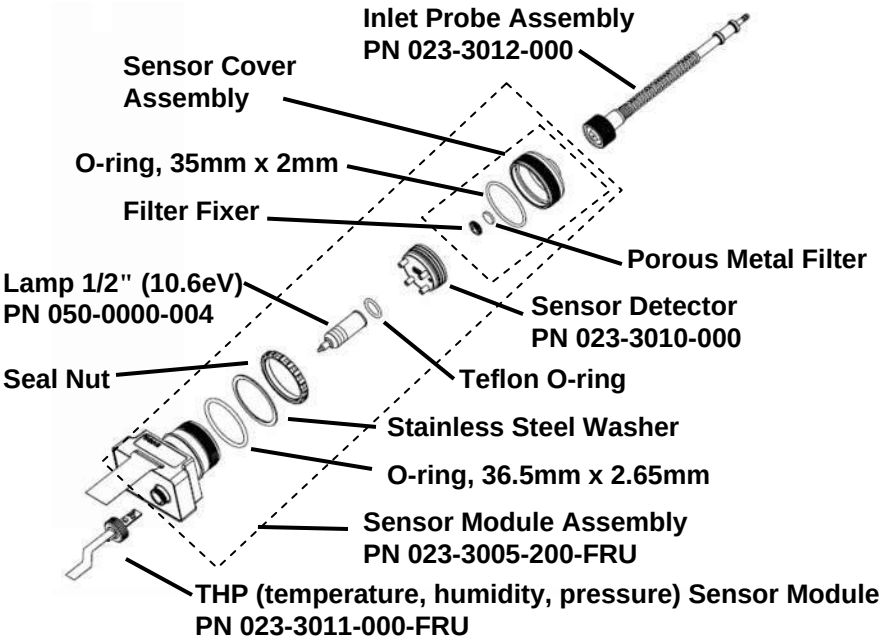
WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge the battery only in areas known to be non-hazardous. Remove and replace the battery only in areas known to be non-hazardous.

Note: The internal charging circuit is designed to prevent charging to alkaline batteries.

PID Sensor & Lamp Cleaning/Replacement

The sensor module is made of several components and is attached to the lamp-housing unit as shown below.



Sensor Components

Note: The cleaning procedure is not normally needed. Clean the PID sensor module, the lamp and the lamp housing only if:

1. The reading is inaccurate even after calibration.
2. The reading is very sensitive to air moisture.
3. A liquid has been sucked into the unit and damaged the unit.

Use of the external filter helps to prevent contamination of the sensor.

To access the sensor components and lamp, gently unscrew the lamp-housing cap, remove the sensor adapter with the gas inlet probe and the metal filter all together. Then hold the PID sensor and pull it straight out. A slight, gentle rocking motion helps release the sensor.

Cleaning The PID Sensor

Place the entire PID sensor module into GC grade methanol. It is highly recommended that an ultrasound bath to be used to clean the sensor for at least 15 minutes. Then dry the sensor thoroughly. Never touch the electrodes of the sensor by hand.
Also use a methanol-soaked cotton swab to wipe off the lamp housing where it contacts the sensor when the sensor is installed.

Turn over the sensor so that the pins point up and the sensor cavity is visible. Examine the sensor electrodes for any corrosion, damage, or bending out of alignment. The metal sensor electrode "fingers" should be flat and straight. If necessary, carefully bend the sensor fingers to ensure that they do not touch the Teflon portions and that they are parallel to each other. Make sure that the nuts on the sensor pins are snug but not overtight. If the sensor is corroded or otherwise damaged, it should be replaced.

Cleaning The Lamp Housing Or Changing The Lamp

If the lamp does not turn on, the instrument will display an error message to indicate replacement of the lamp may be required.

1. If the lamp is operational, clean the lamp window surface and the lamp housing by wiping it with GC grade methanol using a cotton swab using moderate pressure. After cleaning, hold the lamp up to the light at an angle to detect any remaining film. Repeat the process until the lamp window is clean. Never use water solutions to clean the lamp. Dry the lamp and the lamp housing thoroughly after cleaning.

   CAUTION: Never touch the window surface with the fingers or anything else that may leave a film. Never use acetone or aqueous solutions.

2. If the lamp does not turn on, remove the lamp from the lamp housing. Place the lamp O-ring onto the new lamp. Insert the new lamp, avoiding contact with the flat window surface.
3. Reinstall the PID sensor module.
4. Tighten the Lamp Housing Cap.

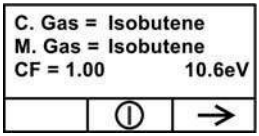
Determining The Lamp Type

The monitor can accommodate three lamp values: 10.6eV (standard), 9.8eV, and 11.7eV. The monitor automatically reads a marking on the side of the lamp to set the proper Correction Factor. There are two ways to determine the lamp type:

Remove the lamp and look for markings (bars) on the side:

- No bars: 10.6eV
- 1 bar: 11.7eV
- 2 bars: 9.8eV

Also, when the monitor is running, the lamp type is shown along with the calibration and measurement gas and Correction Factor:



Note: This screen can be accessed from the reading screen by pressing [N/-] four times.

Sampling Pump

When approaching the end of the specified lifetime of the pump, it will consume higher amount of energy and reduce its sample draw capability significantly. When this occurs, it is necessary to replace or rebuild the pump. When checking the pump flow, make sure that the inlet connector is tight and the inlet tubing is in good condition. Connect a flow meter to the gas inlet probe. The flow rate should be above 450 cc/min when there is no air leakage.

If the pump is not working properly, refer the instrument to qualified service personnel for further testing and, if necessary, pump repair or replacement.

Cleaning The Instrument

Occasional cleaning with a soft cloth is recommended. Do not use detergents or chemicals.

Visually inspect the contacts at the base of the instrument, on the battery, and on the charging cradle to make sure they are clean. If they are not, wipe them with a soft, dry cloth. Never use solvents or cleaners.

Ordering Replacement Parts

If you need replacement parts, contact your local RAE Systems distributor. A list is available online:

http://www.raesystems.com

In the U.S., you can order sensors, replacement batteries, and other accessories online at:

http://istore.raesystems.com/

Special Servicing Note

If the instrument needs to be serviced, contact either:

1. The RAE Systems distributor from whom the instrument was purchased; they will return the instrument on your behalf.

   or

2. The RAE Systems Technical Service Department. Before returning the instrument for service or repair, obtain a Returned Material Authorization (RMA) number for proper tracking of your equipment. This number needs to be on all documentation and posted on the outside of the box in which the instrument is returned for service or upgrade. Packages without RMA Numbers will be refused at the factory.

Troubleshooting

Problem	Possible Reasons & Solutions	
Cannot turn on power after charging the battery	**Reasons:**	Discharged battery. Defective battery.
	Solutions:	Charge or replace battery.
Lost password	**Solutions:**	Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800
Reading abnormally High	**Reasons:**	Dirty filter. Dirty sensor module. Excessive moisture and water condensation. Incorrect calibration.
	Solutions:	Replace filter. Blow-dry the sensor module. Calibrate the unit.
Reading abnormally Low	**Reasons:**	Dirty filter. Dirty sensor module. Weak or dirty lamp. Incorrect calibration.
	Solutions:	Replace filter. Remove Calibration Adapter. Calibrate the unit. Check for air leakage.
Buzzer Inoperative	**Reasons:**	Bad buzzer.
	Solutions:	Check that buzzer is not turned off. Call authorized service center.

Inlet flow too low	**Reasons:**	Pump diaphragm damaged or has debris. Flow path leaks.
	Solutions:	Check flow path for leaks; sensor module O-ring, tube connectors, Teflon tube compression fitting. Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800
"Lamp" message during operation	**Reasons:**	Lamp drive circuit. Weak or defective PID lamp, defective.
	Solutions:	Turn the unit off and back on. Replace UV lamp

Technical Support

To contact RAE Systems Technical Support Team:

Monday through Friday, 7:00AM to 5:00PM Pacific (US) Time
Phone (toll-free): +1 888-723-4800
Phone: +1 408-952-8461
Email: tech@raesystems.com

Life-critical after-hours support is available:

+1 408-952-8200 select option 8

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Controlled Part of Manual

Intrinsic Safety:
US and Canada: Class I, Division 1, Groups A,B,C,D T4

Europe: ATEX (0575 Ex II 2G Ex ia IIC/IIB T4 Gb)
KEMA 07 ATEX 0127
Complies with EN60079-0:2009, EN60079-11:2007
IECEx CSA 10.0005 Ex ia IIC/IIB T4 Gb
Complies with IEC 60079-0:2007, IEC 60079-11:2006

Temperature: -20º C to 50º C (-4º to 122º F)

Humidity: 0% to 95% relative humidity (non-condensing)

Basic Operation

Turning The Instrument On

1. With the instrument turned off, press and hold [MODE].
2. When the display turns on, release the [MODE] key.

The instrument is now operating and performs self tests. Once the self tests are complete, the display shows a graph or numerical gas reading. This indicates that the instrument is fully functional and ready to use.

Turning The Instrument Off

1. Press and hold the Mode key for 3 seconds. A 5-second countdown to shutoff begins.
2. When you see "Unit off..." release your finger from the [MODE] key. The instrument is now off.

Note: You must hold your finger on the key for the entire shutoff process. If you remove your finger from the key during the countdown, the shutoff operation is canceled and the instrument continues normal operation.

Alarm Signals

During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings). If the concentration exceeds any of the preset limits, the loud buzzer and red flashing LED are activated immediately to warn you of the alarm condition.

In addition, the instrument alarms if one of the following conditions occurs: battery voltage falls below a preset voltage level, failure of the UV lamp, pump stall, or when the datalog memory is full.

Alarm Signal Summary

Message	Condition	Alarm Signal
HIGH	Gas exceeds "High Alarm" limit	3 beeps/flashes per second*
OVR	Gas exceeds measurement range	3 beeps/flashes per second*
MAX	Gas exceeds electronics' maximum range	3 beeps/flashes per second*
LOW	Gas exceeds "Low Alarm" limit	2 beeps/flashes per second*
TWA	Gas exceeds "TWA" limit	1 Beep/flash per second*
STEL	Gas exceeds "STEL" limit	1 Beep/flash per second*
Pump icon flashes	Pump failure	3 beeps/flashes per second
Lamp	PID lamp failure	3 beeps/flashes per second plus "Lamp" message on display

Battery icon flashes	Low battery	1 flash, 1 beep per minute plus battery icon flashes on display
CAL	Calibration failed, or needs calibration	1 beep/flash per second
NEG	Gas reading measures less than number stored in calibration	1 beep/flash per second

Preset Alarm Limits & Calibration

The instrument is factory calibrated with standard calibration gas, and is programmed with default alarm limits.

Cal Gas (Isobutylene)	Cal Span	unit	Low	High	TWA	STEL
ppbRAE 3000	10	ppm	10	25	10	25
MiniRAE 3000	100	ppm	50	100	10	25
MiniRAE Lite	100	ppm	50	100	10	25
UltraRAE 3000	100	ppm	50	100	10	25

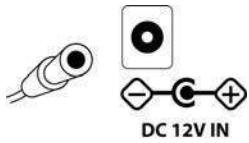
Charging The Battery

Always fully charge the battery before using the instrument. The instrument's Li-ion/NiMH battery is charged by placing the instrument in its cradle. Contacts on the bottom of the instrument meet the cradle's contacts, transferring power without other connections.

Note: Before setting the instrument into its charging cradle, visually inspect the contacts to make sure they are clean. If they are not, wipe them with a soft cloth. Do not use solvents or cleaners.

Follow this procedure to charge the instrument:

1. Plug the AC/DC adapter's barrel connector into the instrument's cradle.



2. Plug the AC/DC adapter into the wall outlet.
3. Place the instrument into the cradle, press down, and lean it back. It locks in place and the LED in the cradle glows.

Note: To release the instrument, press down and tilt the top out of the cradle and lift up.

The instrument begins charging automatically. The LED on the front of the cradle marked "Primary" blinks during charging. During charging, the diagonal lines in the battery icon on the instrument's display are animated and you see the message "Charging..."

When the instrument's battery is fully charged, the battery icon is no longer animated and shows a full battery. The message "Fully charged!" is shown and the Primary LED on the cradle glows continuously green.

Note: A spare Li-ion battery (059-3051-000) or NiMH(059-3054-000) can be charged by placing it directly in the charging port on the back of the cradle. It can be charged at the same time as the instrument. Press the battery in place, sliding it slightly toward the front of the cradle. This locks it in the cradle. To release the battery, slide it forward again and tilt it up.

Note: An Alkaline Battery Adapter (part number 059-3052-000), which uses four AA alkaline batteries (Duracell MN1500), may be substituted for the Li-Ion battery.

WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge and replace batteries only in areas known to be non-hazardous. **Remove and replace batteries only in areas known to be non-hazardous.**

Low Voltage Warning

When the battery's charge falls below a preset voltage, the instrument warns you by beeping once and flashing once every minute, and the battery icon blinks once per second. You should turn off the instrument within 10 minutes and either recharge the battery by placing the instrument in its cradle, or replace the battery with a fresh one with a full charge.

Clock Battery

An internal clock battery is mounted on one of the instrument's printed circuit boards. This long-life battery keeps settings in memory from being lost whenever the Li-ion, NiMH, or alkaline batteries are removed. This backup battery should last approximately five years, and must be replaced by an authorized RAE Systems service technician. It is not user-replaceable.

WARNING

To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non-hazardous. Remove and replace battery only in an area known to be non-hazardous.

Replacing Rechargeable Li-Ion or NiMH Battery

Caution: Turn off the instrument before removing or replacing the battery.

Alkaline Battery Adapter

An alkaline battery adapter is supplied with each instrument. The adapter (part number 059-3052-000) accepts four AA alkaline batteries (use only Duracell MN1500).

Do not mix old and new batteries or different type batteries.

Troubleshooting

Problem	Possible Reasons & Solutions	
Cannot turn on power after charging the battery	**Reasons:**	Discharged battery. Defective battery.
	Solutions:	Charge or replace battery.
Lost password	**Solutions:**	Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800
Reading abnormally High	**Reasons:**	Dirty filter. Dirty sensor module. Excessive moisture and water condensation. Incorrect calibration.
	Solutions:	Replace filter. Blow-dry the sensor module. Calibrate the unit.
Reading abnormally Low	**Reasons:**	Dirty filter. Dirty sensor module. Weak or dirty lamp. Incorrect calibration.
	Solutions:	Replace filter. Remove Calibration Adapter. Calibrate the unit. Check for air leakage.
Buzzer Inoperative	**Reasons:**	Bad buzzer.
	Solutions:	Check that buzzer is not turned off. Call authorized service center.

Inlet flow too low	**Reasons:**	Pump diaphragm damaged or has debris. Flow path leaks.
	Solutions:	Check flow path for leaks; sensor module O-ring, tube connectors, Teflon tube compression fitting. Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800
"Lamp" message during operation	**Reasons:**	Lamp drive circuit. Weak or defective PID lamp, defective.
	Solutions:	Turn the unit off and back on. Replace UV lamp

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Rev. C
August 2010
P/N 059-4020-000

Sample Pro

Portable MicroPurge® Pumps PATENT PENDING QED

MicroPurge® Portable Pump

User's Guide

Part No. 95181 1-14-15

QED

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Introduction

The Portable MicroPurge® Pump, (**Figure 1**) is the first pump designed specifically to meet the needs of portable low-flow sampling: easy to adjust to low-flow purging rates and easy to decontaminate between sampling points. Decon is made easier by the pump having fewer parts, disassembly without tools, and quick-change, disposable bladders. The Portable MicroPurge Pump is compact, can pump from a tall 5-gallon bucket, and is offered with different tube connection sizes and methods. The pump is operated by compressed gas and a bladder pump control unit, and is ideally used with the MicroPurge basics models MP10 and MP15 controllers. The compressed gas is on the outside of the bladder and the pumped liquid is on the inside of the bladder, so there is no contact between the sample and the gas.

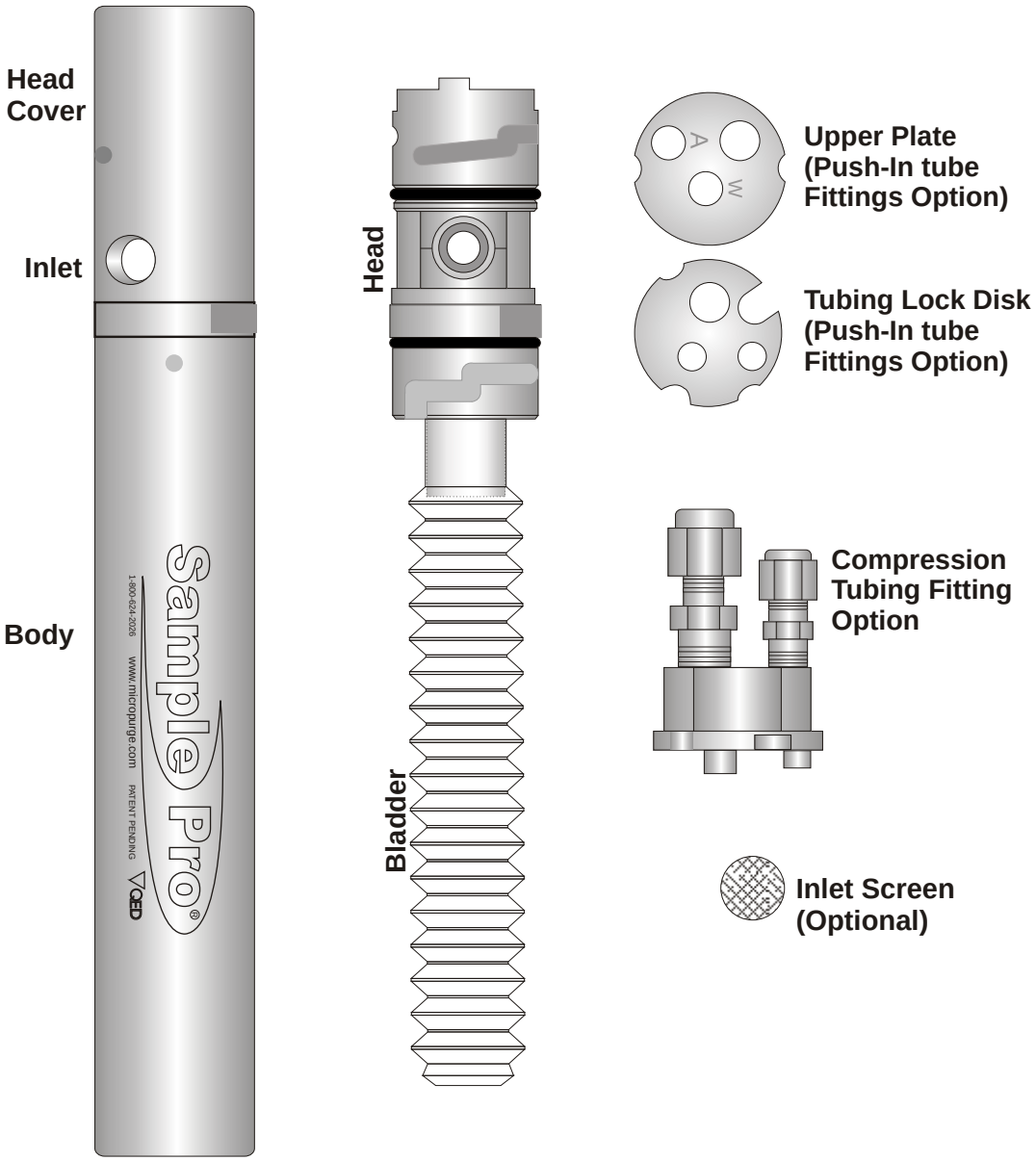


Figure 1

Description

Figure 2 (**Page 3**) shows the main components of the Portable MicroPurge Pump and the assembled pump.The body twists off for quick change of the bladder, which is offered in polyethylene (PE) and optional teflon versions. The PE bladder provides a leak-tight seal for most applications without the use of clamps; a bladder collar is provided for seal assurance for pump submergences over 50 ft.

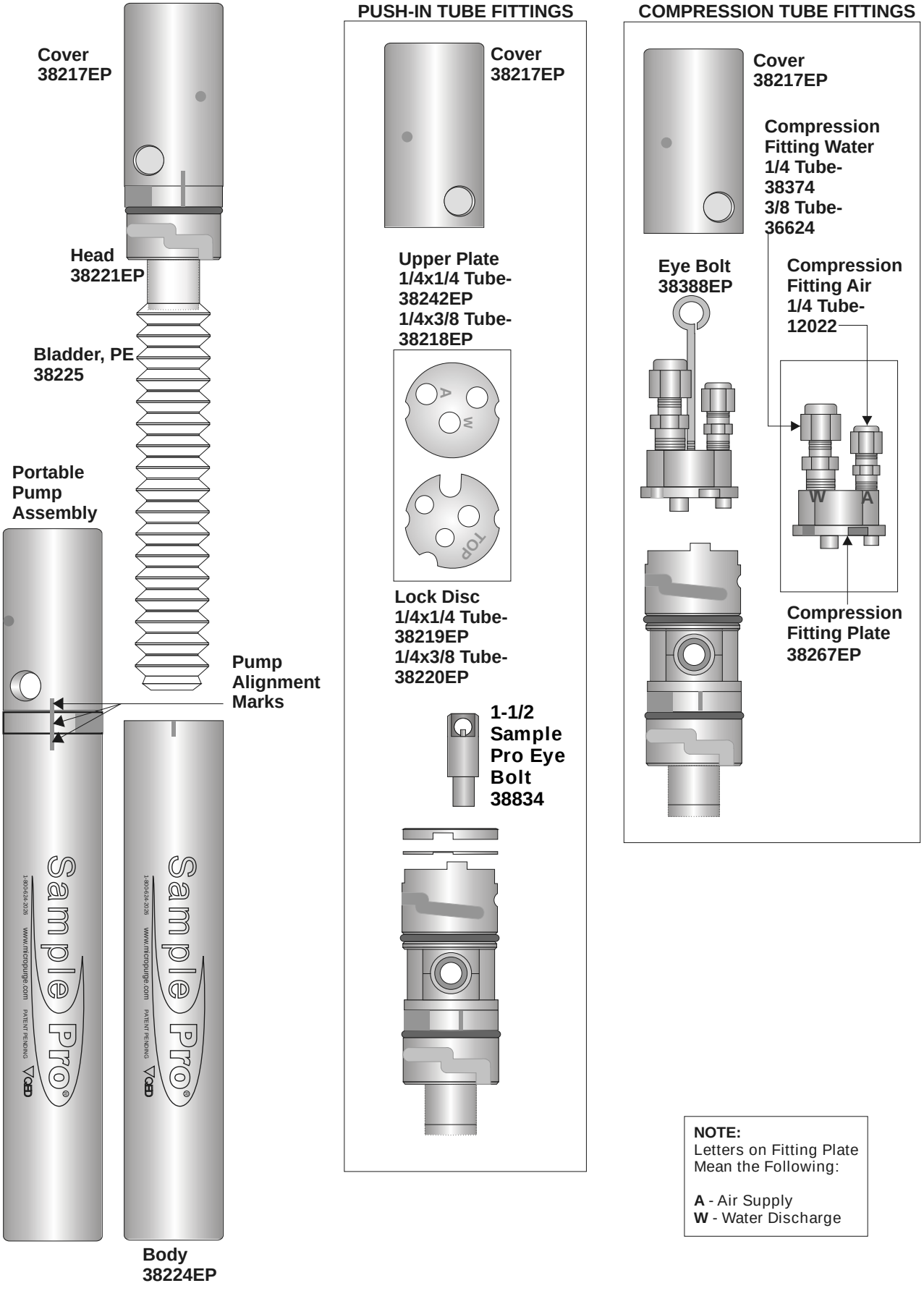
The cap of the pump twists off for full disassembly of the inlet and outlet check valves and the tubing connection components. Tubing connections are offered in two sizes in each of two types:

- Compression-type fittings for ¼" or 3/8" OD water discharge tube, and ¼" OD air supply tube.
  Pump Models **MP-SP-4C** 1/4" O.D. water discharge
  Pump Models **MP-SP-6C** 3/8" O.D. water discharge

- Push-in connections for ¼" or 3/8" OD water discharge tube, and ¼" OD air supply tube.
  Pump Models **MP-SP-4P** 1/4" O.D. water discharge
  Pump Models **MP-SP-6P** 3/8" O.D. water discharge

The push-in connection option is provided for greater ease of use in applications in which the tubing will be frequently changed. The push-in connections provide excellent pull out strength when used with QED tubing, so that use of a support cable is not required. However a connection eye is provided on the top of the push-in assembly for use of a cable when preferred. *The push-in and compression fittings on the SamplePro pump are designed to provide at least 100 lbs. pullout strength when used with QED tubing.* ***QED is not responsible for loss of the pump if non-QED tubing is used****.*

FIGURE 2



NOTE:
Letters on Fitting Plate
Mean the Following:

A - Air Supply
W - Water Discharge

Assembly

Use Figure 2, (**Page 3**) to identify main pump components and Figure 3, (**Page 10**) to identify O-ring locations. Full exploded drawings for each pump model are shown on pages 11 to 14; detailed exploded view drawings for each tubing connection configuration are shown on pages 15 to 18.

1. See figure 3 (**page 10**) to identify locations where O-rings are to be installed and install all O-rings.
2. Connect the bladder to the pump head. The PE bladder pushes onto the pump head barb until the bladder fully covers the barb. A clamping collar (white ring) is provided for pump submergences over 50 ft, to assure a leak tight seal of the bladder. Use the clamp collar by putting it over the pump head barb before pushing the bladder on, then pulling the collar back down firmly over the bladder and barb. The teflon bladder cartridge is installed by inserting the cartridge nipple into the center hole in the bottom of the pump head barb.
3. Attach the pump head to the body by engaging the bayonet dimples into the grooves and twisting them together until the engagement snap is felt and head and body alignment marks line up.
4. With the pump on its side, insert the inlet check ball (same as the discharge check ball) into the side of the pump head, then press in the inlet valve seat by pushing and twisting with your thumb.
5. With the pump vertical, insert the discharge check ball into the top of the pump head, then press in the discharge ball seat by pushing and twisting with your thumb.
6. For the push-in tubing fittings, place the thin metal lock disk in the "TOP" up position on the top of the pump head, with the lock disk edge slots lined up with the posts on the pump head. Then place the thick, upper plate on top of the lock disk, again with the slots and posts lined up. Finally, twist the pump cap onto the pump head until the engagement snap is felt and the hole in the side of the pump cap lines up with the inlet port. Cover and body alignment marks will line up.
7. For the compression nut fittings, place the compression fitting plate onto the top of the pump head, with slots and posts lined up. The fitting nuts may need to be rotated or removed to allow the pump cap to be placed over the compression fitting assembly.Then twist the pump cap onto the pump head until the engagement snap is felt and the hole in the side of the pump cap lines up with the inlet port. Cover and body alignment marks will line up.

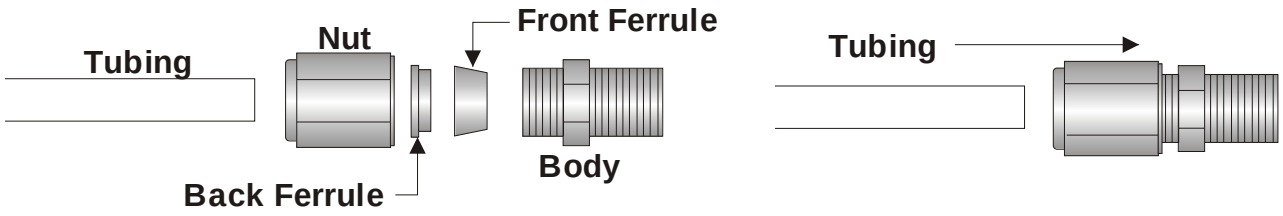
Attaching Tubing

Push-in Fittings

1. Following the previous assembly instructions, use a new lock plate and freshcut end of QED tubing to ensure proper pull-out strength of tubing connection. Re-use of the lock plate or old tube-end and/or use of other brands of tubing may significantly reduce pull-out strength and cause loss of pump in the well.
2. The upper plate is marked "W" for the water discharge tube and "A" for the air supply tube. With QED tubing, the air supply tube is shaded gray to distinguish it from the water discharge tube.
3. Insert each tube separately into the proper opening in the pump head, pushing firmly so that the tube penetrates beyond first resistance at least ½-inch into the pump. As a check on proper assembly, pull back on each tube to see that it is gripped securely.

Compression Fittings

1. Insert tubing insert into I.D. of tubing (if required).
2. Insert tubing into fitting making sure that the tubing rests firmly on the shoulder of the fitting and that the nut is finger tight.
3.Tighten nut to secure tubing in fitting (approximately 1-1/4 turns beyond finger tight).



Disassembly

Use Figure 2, (**Page 3**) to identify main pump components and Figure 3, (Page 10) to identify O-ring locations.

1. Reverse assembly sequence, taking care to position pump to retain check balls during removal of valve seats and stops. A coin or screwdriver can be used if necessary to remove the inlet valve seat and discharge check ball stop. If these parts do not slip out easily, grip the outside edges of the parts with needle nose pliers, rotate the parts back and forth, and remove.
2. Pull the PE bladder off of the pump head barb by pulling firmly, then discard.

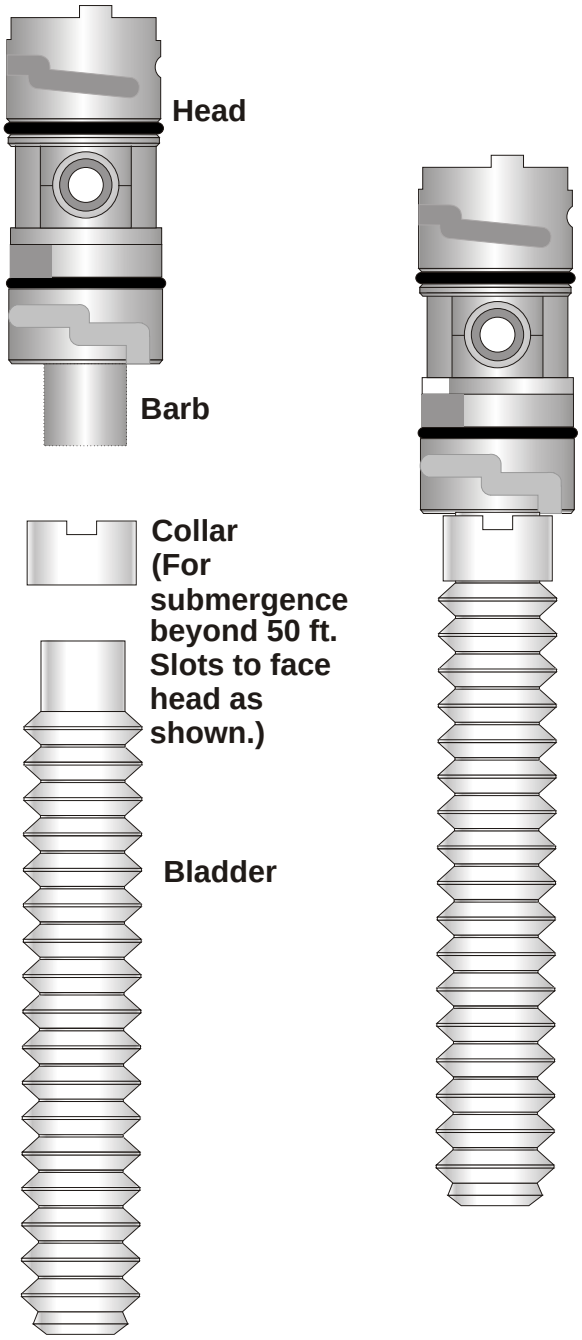
Cleaning / Decontamination Note

If it is desired to operate the pump outside the well, such as in a pail for decontamination purposes, the operating pressure of the pump should be reduced to 35 PSI or less to avoid rupturing the bladder

Standard Bladder Replacement

Connect the bladder to the pump head. The PE bladder pushes onto the pump head barb until the bladder fully covers the barb. A clamping collar (white ring) is provided for pump submergences over 50 ft, to assure a leak tight seal of the bladder. Use the clamp collar by putting it over the narrow, neck section of the bladder, slot side facing toward the pump head; then push the bladder neck fully onto the pump head barb by pushing on the bottom of the bladder, collapsing the bladder. After the bladder neck is in proper position, push the clamp collar up over the bladder and barb. The collar will seal the bladder sufficiently when moderate force (5-10 lbs) is used; excessive force will make later removal difficult.

NOTE: For reasons of contamination and leak integrity, these bladders are designed for one-time use only. ***QED cannot be held responsible for cross contamination or leakage failures if bladders are reused.***

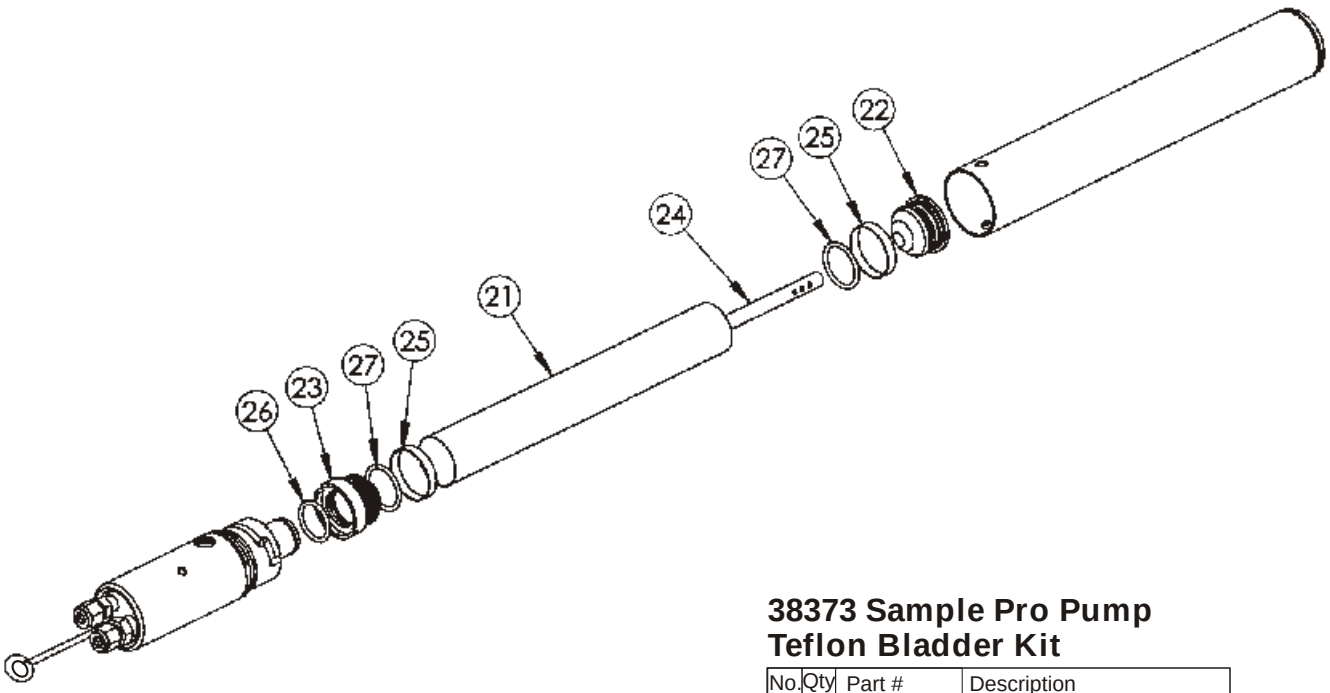


Teflon Bladder Replacement

WARNING: Excessive drive gas pressures can damage the bladder. For normal operation, only 10-15 PSI over the pump depth (hydrostatic head, equivalent to .43 PSI per foot of depth) is sufficient to operate the pump. For pump depths over 200 ft (86 PSI hydrostatic head), it is recommended that initial pumping be performed with approximately 75 PSI drive gas pressure to move approximately 500 ml, equivalent to about 5 full bladder volumes, up into the discharge tubing to provide pumping resistance. Thereafter, full drive gas pressure can be applied. The drive gas pressure gauge on the basics controller is marked in both PSI and feet of hydrostatic head.

NOTE: For reasons of contamination and leak integrity, these bladders are designed for one-time use only. ***QED cannot be held responsible for cross contamination or leakage failures if bladders are reused.***

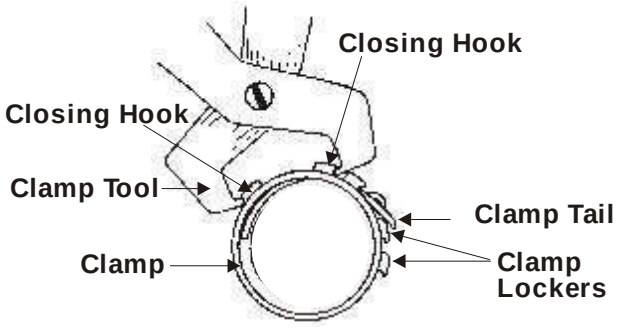
1. Identify the components as shown below.



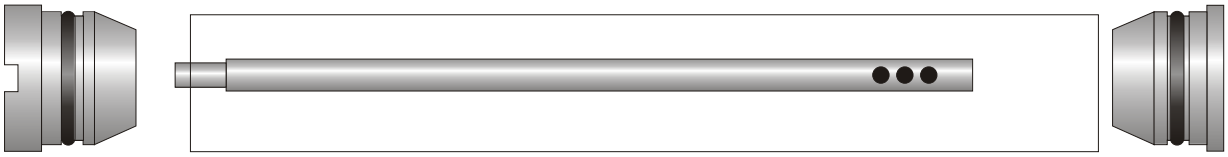
38373 Sample Pro Pump Teflon Bladder Kit

No.	Qty	Part #	Description
21	1	38372	Teflon Bladder
22	1	38371EP	Bottom Spool
23	1	38370EP	Top Spool
24	1	38369EP	Dip Tube
25	2	38016	Clamp 030.2-505R S.S.
26	1	38378	O-Ring 2-118
27	2	38379	O-Ring 2-220

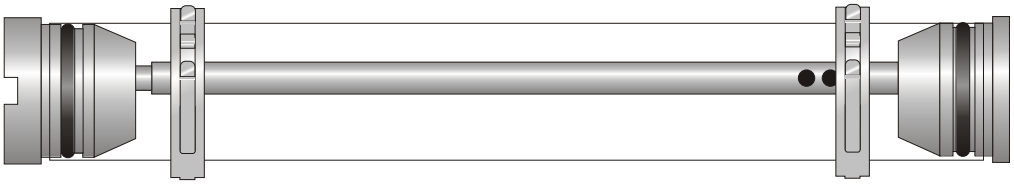
2. Change the teflon bladder by using the clamp tool to remove the bladder clamps.



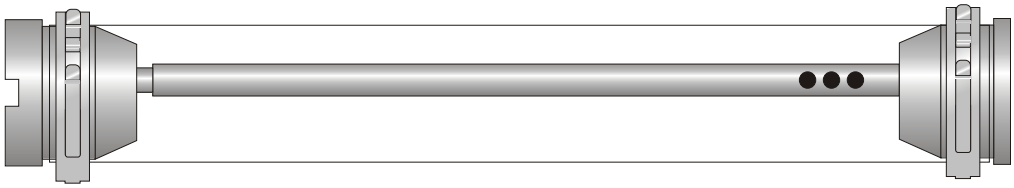
3. Pull apart the cartridge end pieces. Clean cartridge components as desired.



4. Install a new teflon bladder by placing the clamps loosely over the ends of the bladder, pushing the spool ends, with O-rings in place, onto each end of the center rod. Wet both bladder clamps with distilled water prior to clamping them to assure sufficient lubrication.

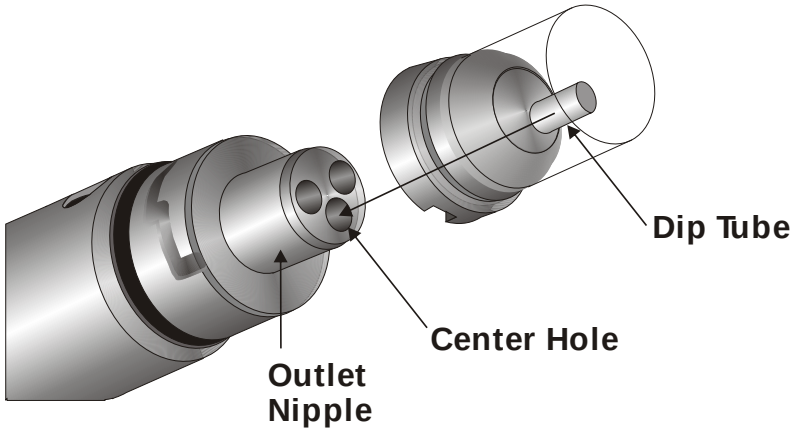


5. Position the clamps directly over the o-rings and then using the clamp tool, clamp the bladder into position onto the cartridge, as shown.



NOTE: *Before clamping clamps down make sure the bladder is visible above the clamp all the way around the clamp.*

6. Insert the outlet nipple from the upper spool piece into the center hole on the bottom of the pump head, as shown.



O-ring Replacement

1. Inspect o-rings with each disassembly and replace as needed. Replacement of Top Spool, inner o-ring replacement shown below.

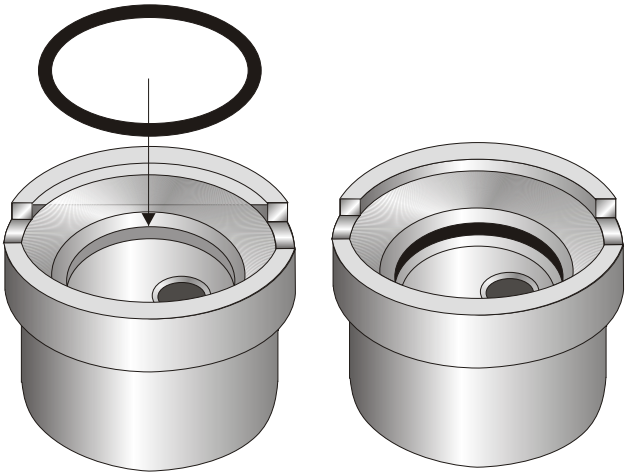
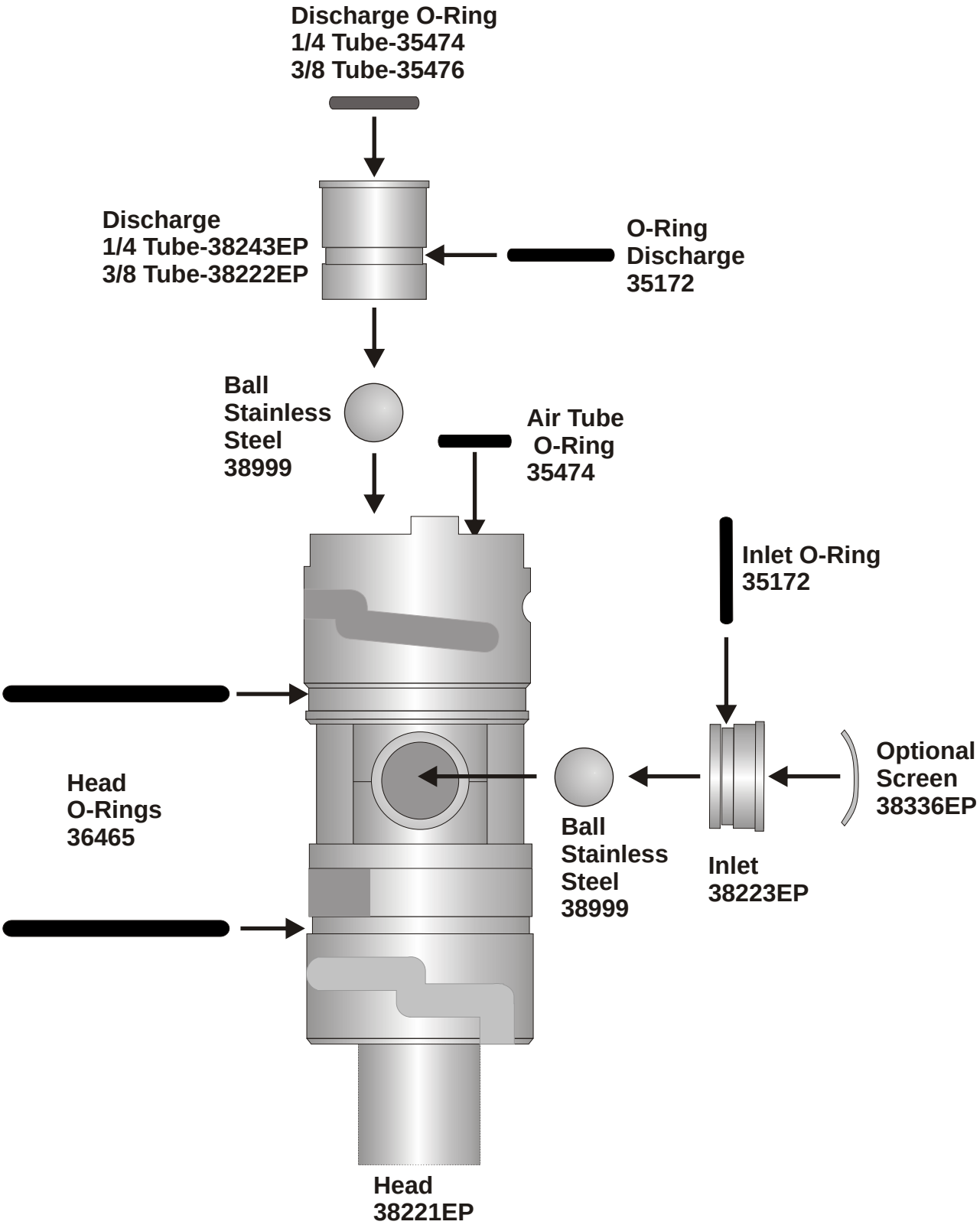


FIGURE 3


Discharge O-Ring
1/4 Tube-35474
3/8 Tube-35476
Discharge
1/4 Tube-38243EP
3/8 Tube-38222EP
O-Ring
Discharge
35172
Ball
Stainless
Steel
38999
Air Tube
O-Ring
35474
Inlet O-Ring
35172
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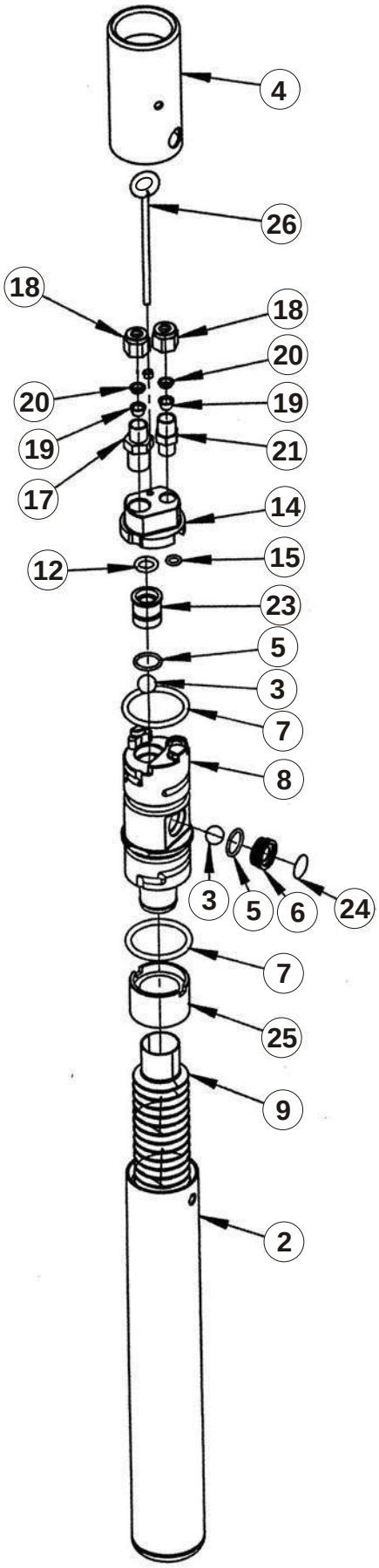
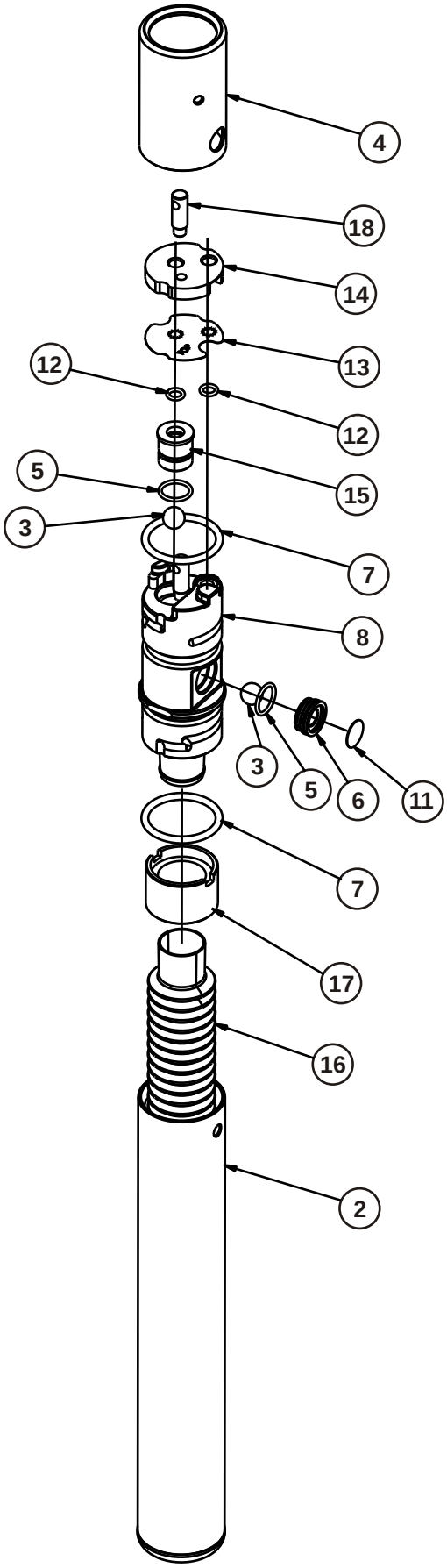
Stainless
Steel
38999
Inlet
38223EP
Head
38221EP



MP-SP-4C SamplePro Pump Assembly

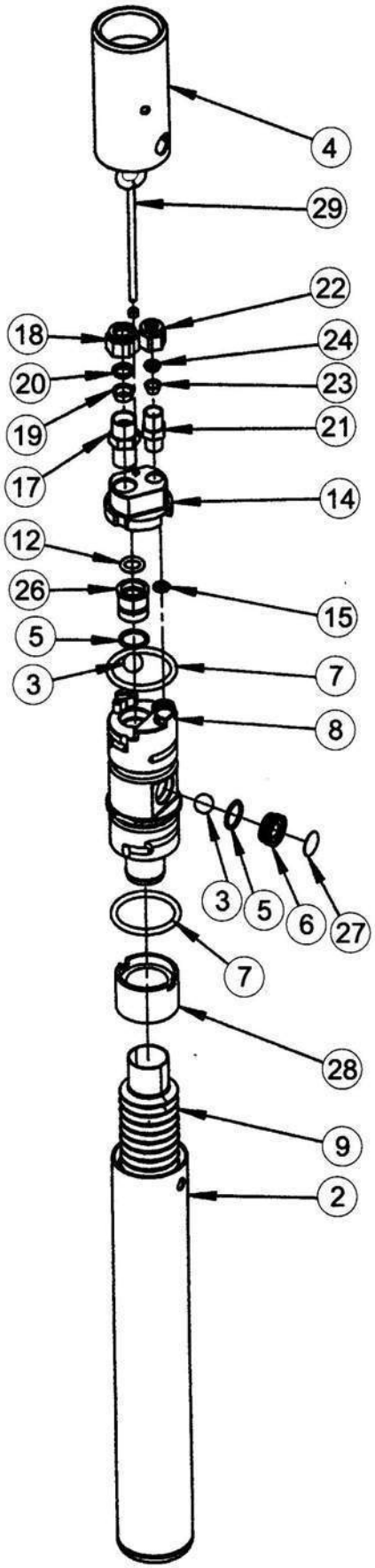
No.	Qty	Part #	Description
2	1	38224EP	Body
3	2	38999	Ball Stainless Steel 7/16 Dia.
4	1	38217EP	Cover
5	2	35172	O-Ring Viton 2-015
6	1	38223EP	Inlet
7	2	36465	O-Ring Viton 2-220
8	1	38221EP	Head
9	1	38225	Bladder PE
12	1	35476	O-Ring Viton 2-110
14	1	38267EP	Compression Fitting Plate
15	1	35474	O-Ring Viton 2-010
17	1	38374	Connector 1/4"T x 1/4" MPT SS
18	2	34954	Nut 1/4" T SS
19	2	34477	Ferrule 1/4" Lower SS
20	2	34476	Ferrule 1/4" Upper SS
21	1	12022	Connector 1/4"T x 1/8" MPT SS
23	1	38222EP	Discharge Tube 3/8"
24	1	38336	Inlet Screen
25	1	38340	Collar
26	1	38388EP	Eye Bolt with Nut

FIGURE 5



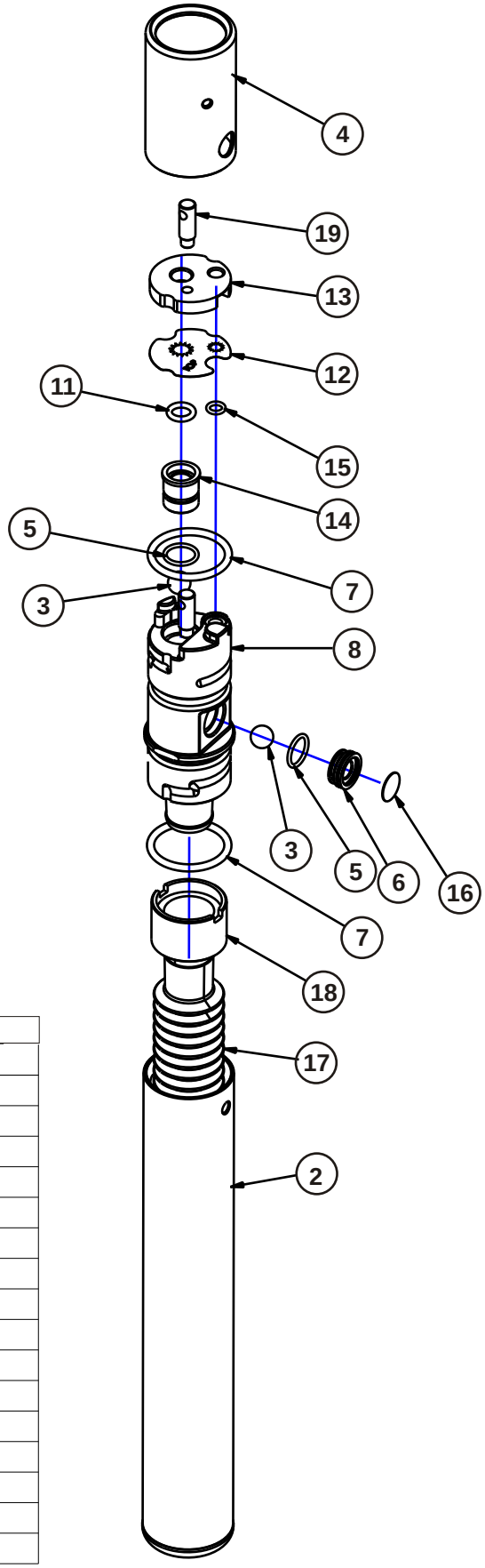
MP-SP-4P Sample Pro Pump Assembly

No.	Qty	Part #	Description
2	1	38224EP	Body
3	2	38999	Ball Stainless Steel 7/16 Dia.
4	1	38217EP	Cover
5	2	35172	O-ring Viton 2-015
6	1	38223EP	Inlet
7	2	36465	O-ring Viton 2-220
8	1	38221EP	Head
11	1	38336	Inlet Screen
12	2	35474	O-ring Viton 2-010
13	1	38219	Grab Plate 1/4" Tube x 1/4" Tube
14	1	38242EP	Plate 1/4" Tube x 1/4" Tube
15	1	38243EP	Discharge 1/4" Tube
16	1	38225	Bladder PE
17	1	38340	Collar
18	2	38834	1 1/2 Sample Pro Eye Bolt



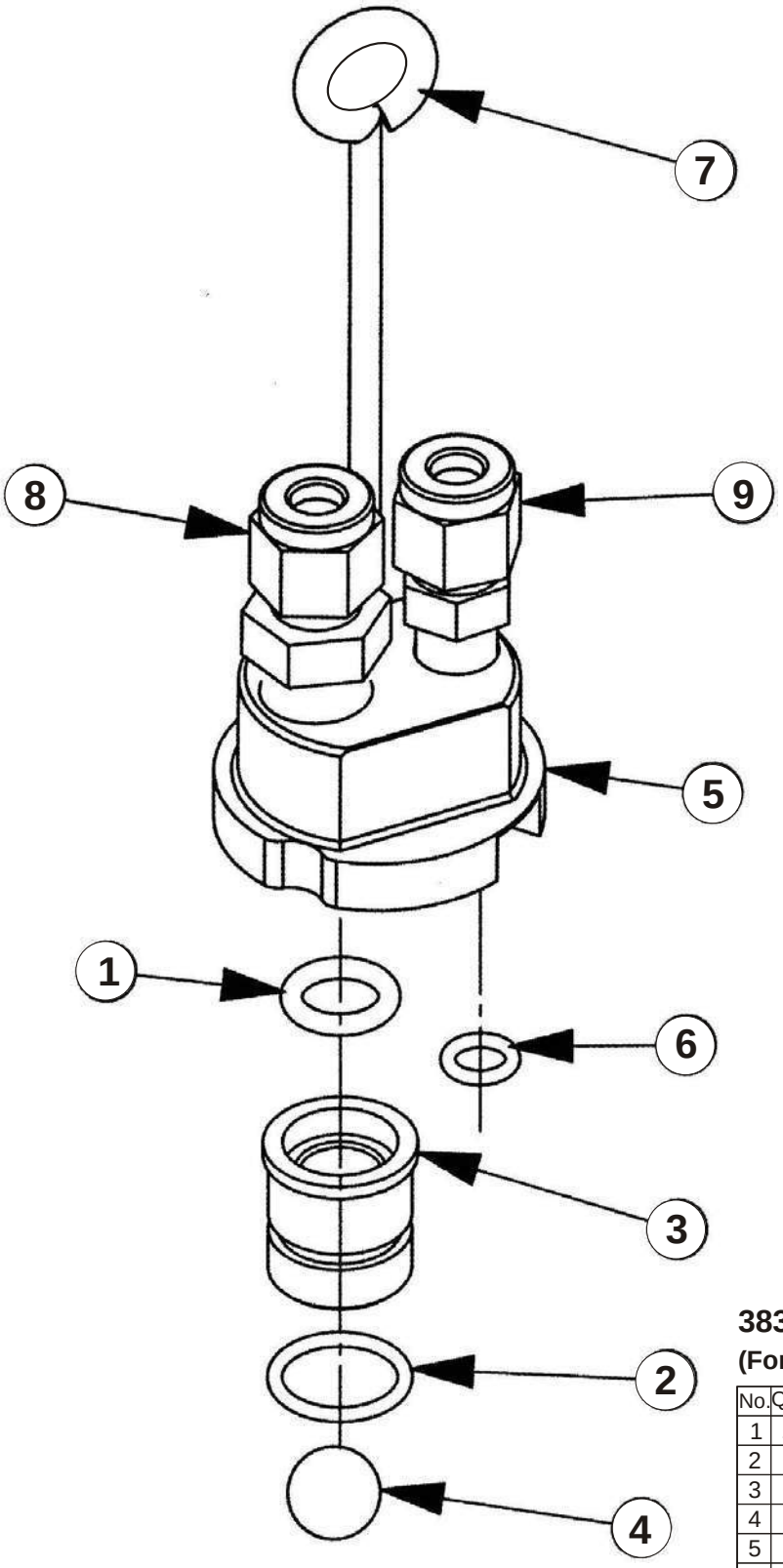
MP-SP-6C SamplePro Pump Assembly

No.	Qty	Part #	Description
2	1	38224EP	Body
3	2	38999	Ball Stainless Steel 7/16 Dia.
4	1	38217EP	Cover
5	2	35172	O-Ring Viton 2-015
6	1	38223EP	Inlet
7	2	36465	O-Ring Viton 2-220
8	1	38221EP	Head
9	1	38225	Bladder PE
12	1	35476	O-Ring Viton 2-110
14	1	38267	Compression Fitting Plate
15	1	35474	O-Ring Viton 2-010
17	1	36624	Connector 3/8"T x 1/4" MPT SS
18	1	37984	Nut 3/8" T SS
19	1	34479	Ferrule 3/8" Lower SS
20	1	34478	Ferrule 3/8" Upper SS
21	1	12022	Connector 1/4"T x 1/8" MPT SS
22	1	34954	Nut 1/4" T SS
23	1	34477	Ferrule 1/4" Lower SS
24	1	34476	Ferrule 1/4" Upper SS
26	1	38222EP	Discharge Tube 3/8"
27	1	38336	Inlet Screen
28	1	38340	Collar
29	1	38388EP	Eye Bolt with Nut



MP-SP-6P SamplePro Pump Assembly

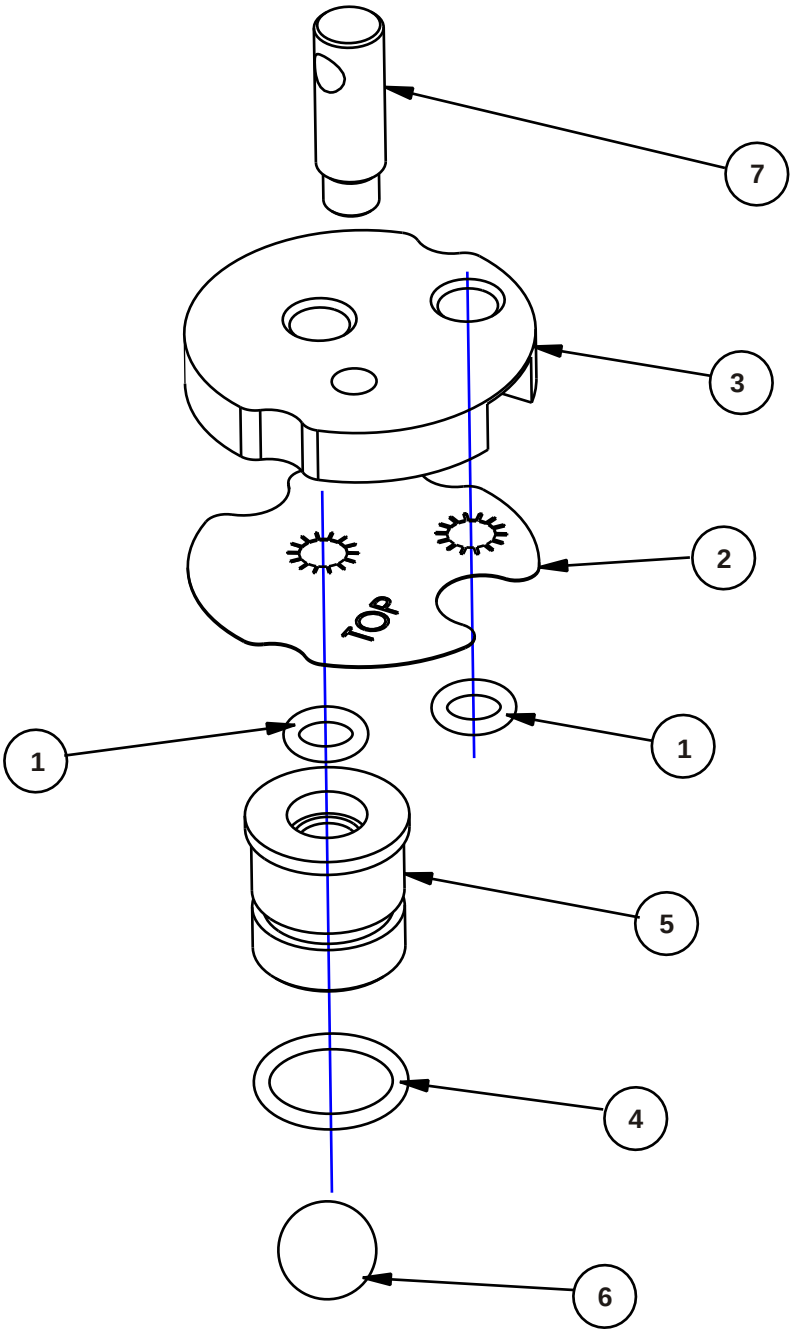
No.	Qty	Part #	Description
2	1	38224EP	Body
3	2	38999	Ball Stainless Steel 7/16 Dia.
4	1	38217EP	Cover
5	2	35172	O-ring Viton 2-015
6	1	38223EP	Inlet
7	2	36465	O-ring Viton 2-220
8	1	38221EP	Head
11	1	35476	O-ring Viton 2-110
12	1	38220	Grab Plate 1/4" Tube x 3/8" Tube
13	1	38218EP	Plate 1/4" Tube x 3/8" Tube
14	1	38222EP	Discharge 3/8" Tube
15	1	35474	O-ring Viton 2-010
16	1	38336	Inlet Screen
17	1	38225	Bladder PE
18	1	38340	Collar
19	2	38834	1 1/2 Sample Pro Eye Bolt



38358 Tube ConnectorAssembly

(For Use on MP-SP-4C)

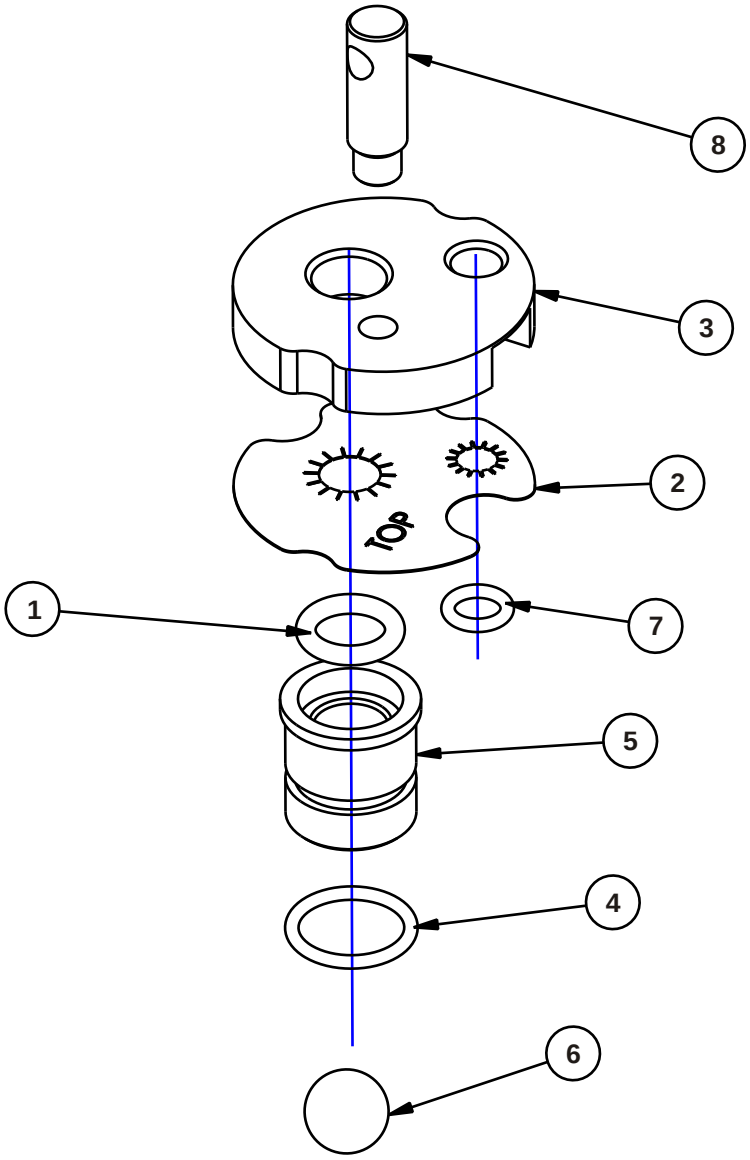
No.	Qty	Part #	Description
1	1	35476	O-Ring Viton 2-110
2	1	35172	O-Ring Viton 2-015
3	1	38222EP	Discharge Tube 3/8"
4	1	38999	Ball Stainless Steel 7/16 Dia.
5	1	38267EP	Compression Fitting Plate
6	1	35474	O-Ring Viton 2-010
7	1	38388EP	Eye Bolt with Nut
8	1	38374	Connector 1/4"T x 1/4" MPT SS
9	1	12022	Connector 1/4"T x 1/8" MPT SS



38356 Tube Connector Use On MP-SP-4P

No.	Qty	Part #	Description
1	2	35474	O-ring Viton 2-010
2	1	38219	Grab Plate 1/4" Tube x 1/4" Tube
3	1	38242EP	Plate 1/4" Tube x 1/4" Tube
4	1	35172	O-ring Viton 2-015
5	1	38243EP	Discharge 1/4" Tube
6	1	38999	Ball Stainless Steel 7/16 Dia.
7	1	38834	1 1/2 Sample Pro Eye Bolt

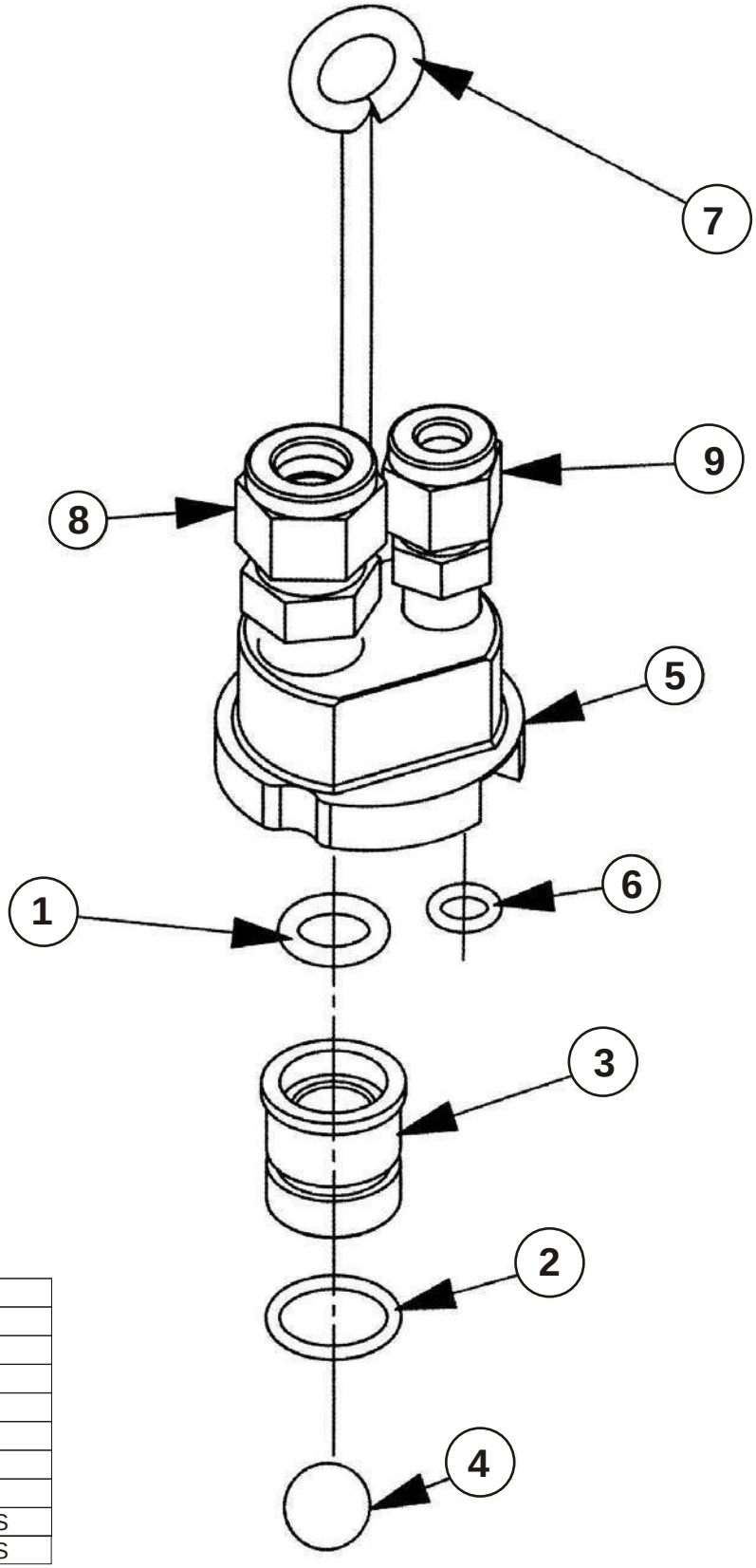
FIGURE 10



38357 Tube Connector Assembly

(For Use on MP-SP-6P)

No.	Qty	Part #	Description
1	1	35476	O-ring Viton 2-110
2	1	38220	Grab Plate 1/4" Tube x 3/8" Tube
3	1	38218EP	Plate 1/4" Tube x 3/8" Tube
4	1	35172	O-ring Viton 2-015
5	1	38222EP	Discharge 3/8" Tube
6	1	38999	Ball Stainless Steel 7/16 Dia.
7	1	35474	O-ring Viton 2-010
8	1	38834	1 1/2 Sample Pro Eye Bolt



38359 Tube Connector Assembly

(For Use on MP-SP-6C)

No.	Qty	Part #	Description
1	1	35476	O-Ring Viton 2-110
2	1	35172	O-Ring Viton 2-015
3	1	38222EP	Discharge Tube 3/8"
4	1	38999	Ball Stainless Steel 7/16 Dia.
5	1	38267EP	Compression Fitting Plate
6	1	35474	O-Ring Viton 2-010
7	1	38388	Eye Bolt with Nut
8	1	36624	Connector 3/8"T x 1/4" MPT SS
9	1	12022	Connector 1/4"T x 1/8" MPT SS

Sample Pro Pump Models

MP-SP-4P - Sample Pro Portable Pump push-in 1/4 x 1/4 tubing connector
MP-SP-6P - Sample Pro Portable Pump push-in 1/4 x 3/8 tubing connector
MP-SP-4C - Sample Pro Portable Pump compression 1/4 x 1/4 tubing connector
MP-SP-6C - Sample Pro Portable Pump compression 1/4 x 3/8 tubing connector

Sample Pro Pump Kits

38355 - Portable pump (Base pump without any tubing connectors.)
38356 -Tubing connector 1/4" x 1/4" tubing push-in38357-Tubing connector 1/4" x 3/8" tubing push-in
38358 -Tubing connector 1/4" x 1/4" tubing compression38359-Tubing connector 1/4" x 3/8" tubing compression
38360 - Bladder PE (10/pieces)
38361 - Screen (10)
38362 - O-ring complete pump (10 sets)
38363 - O-ring head only(10 sets)
38364 - Grab-plate for push-in connector 1/4" x 1/4" (10)
38365 - Grad-plate for push-in connector 1/4" x 3/8" (10)
38366 - Ferrules for compression connectors 1/4" x 1/4" tube (5 sets)
38367 - Ferrules for compression connectors 1/4 x 3/8 (5 sets)
38373 - Bladder Teflon cartridge (parts for optional Teflon bladder)
38380 -Teflon replacement bladders (10) with clamps and O-rings
38407 - Pump controller air fitting, connects to 1/4" air supply tubing
38408 - Ball, Stainless Steel 7/16" dia. (5) for inlet and outlet check valves
38411 - Brushes Sample Pro (3 sizes, 2 of ea.)

SPECIFICATIONS

Materials:
Body - 303 Stainless Steel
Bladder - Polyethylene or Teflon ®
O-rings - Viton
Inlet & Discharge Housing - 303 Stainless Steel

Dimensions:
Diameter - 1.75" (44.5 mm)
Length - 14.75" (37.5 cm) Push-in / 16.5" (41.9 cm) Compression bottom of pump to center line of inlet 12.12" (30.8cm)
Weight - 4.25 Lbs. (1.93 kg)

Fittings:
Stainless Steel Compression or Push-inType
Air - 1/4" (6.4 mm) O.D., 3/16" (4.7 mm) I.D.
Discharge - 1/4" (6.4 mm) O.D., 3/16" (4.7 mm) I.D.
Discharge - 3/8" (9.5 mm) O.D., 1/4" I.D. (6.4 mm)

Maximum Lift:
250 Feet (76.2 m)

Flow Rates (1/4" (6.4 mm) x 1/4" (6.4 mm) O.D.Tubing)
1.2 liters per min @ 25 ft. (7.6 m) (10 ft. (3 m) submergence)
400 ml per min. @ 150 ft. (45.6 m) (10 ft. (3 m) Submergence)

Pump Volume:	Milliliters	Liters	Ounces	Gallons
	100	.100	3.34	.026

For additional assistance contact QED Service at:

Phone: 1-800-624-2026 1-734-995-2547

Fax: 1-734-995-1170

E-mail: service@qedenvcom

24-Hour Service Hot Line: 1-800-272-9559

IMPORTANT WARRANTY NOTE

The push-in and compression fittings on the SamplePro pump are designed to provide at least 100 lbs. pullout strength when used with QED tubing. ***QED is not responsible for loss of the pump if non-QED tubing is used.***

QED ENVIRONMENTAL SYSTEMS, INC. ("Q.E.D.") warrants to the original purchaser of its products that, subject to the limitations and conditions provided below, the products, materials and/or workmanship shall reasonably conform to descriptions of the products and shall be free of defects in materials and workmanship. Any failure of the products to conform to this warranty will be remedied by Q.E.D. in the manner provided herein.

This warranty shall be limited to the duration and the conditions set forth below.All warranty durations are calculated from the original date of purchase.

1. Dedicated-Use Systems Products- 10 year warranty on dedicated bladder pumps equipped with Q.E.D. inlet screens, and purge pumps used in periodic, non continuous groundwater sampling (up to 52 sampling events per year.)All other components, equipment and accessories are warranted for one year.

2. Portable-Use Systems- Pumps, Controllers and water level meters are warranted for one year. Hose reels and Caps are warranted for ninety (90) days. Tubing and Purge Mizers are covered by a ninety (90) day material and workmanship warranty.There will be no warranty for application on tubing and Purge Mizers when used as part of a Portable System.

3. Separately sold parts and Spare Parts Kits- Separately sold parts and spare parts kits are warranted for ninety (90) days. Repairs performed by Q.E.D. are warranted for ninety (90) days from date of repair or for the full term of the original warranty, whichever is longer.

Buyers' exclusive remedy for breach of said warranty shall be as follows:if, and only if, Q.E.D. is notified in writing within applicable warranty period of the existence of any such defect in the said pro-ducts, and Q.E.D. upon examination of any such defects, shall find the same to be within the term of and covered by the warranty running from Q.E.D. to Buyer, Q.E.D. will, at its option, as soon as reasonably possible, eplace or repair any such product, without charge to Buyer. If Q.E.D. for any reason, cannot repair a product covered hereby within four (4) weeks after receipt of the original Purchaser's/Buyer's notification of a warranty claim, then Q.E.D.'s sole responsibility shall be, at its option, either to replace the defective product with a comparable new unit at no charge to the Buyer, or to refund the full pur-chase price. In no event shall such allegedly defective products be returned to Q.E.D. without its con-sent, and Q.E.D.'s obligations of repair, replacement or refund are conditioned upon the Buyer's return of the defective product to Q.E.D.

IN NO EVENT SHALL Q.E.D. ENVIRONMENTAL SYSTEMS, INC. BE LIABLE FOR CON-SEQUENTIAL OR INCIDENTAL DAMAGES FOR BREACH OF SAID WARRANTY.

The foregoing warranty does not apply to major sub-assemblies and other equipment, accessories and parts manufactured by others, and such other parts, accessories, and equipment are subject only to the warranties, if any, supplied by the respective manufacturers. Q.E.D. makes no warranty concerning products or accessories not manufactured by Q.E.D. In the event of failure of any such product acces-sory, Q.E.D. will give reasonable assistance to the Buyer in obtaining from the respective manufacturer whatever adjustment is reasonable in light of the manufacturer's own warranty.

THE FOREGOING WARRANTY IS IN LIEU OFALL OTHER WARRANTIES, EXPRESSED, IMPLIED OR STATUTORY (INCLDING BUT NOT LIMITED TOTHE WARRANTIES OFMERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE), WHICH OTHER WARRANTIES ARE EXPRESSLY EXCLUDED HEREBY, and of any other obligations or liabilities on the part of Q.E.D.,neither assumes nor authorizes any person to assume for it any other obligation or liability in connection with said products, materials and/or workmanship.

It is understood and agreed that Q.E.D. shall in no event be liable for incidental or consequential damages resulting from its breach of any of the terms of this agreement, nor for special damages, nor for improper selection of any product described or referred to for a particular application.

This warranty will be void in the event of unauthorized disassembly of component assemblies. Defects in any equipment that result from abuse, operation in any manner outside the recommended procedures, use and applications other than for intended use, or exposure to chemical or physical environment beyond the designated limits of materials and construction will also void this warranty. Q.E.D. shall be released from all obligations under all warranties if any product covered hereby is repaired or modified by persons other than Q.E.D.'s service personnel unless such repair by others is made with the written consent of Q.E.D.

If any product covered hereby is actually defective within the terms of this warranty, Purchaser must contact Q.E.D. for determination of warranty coverage. If the return of a component is determined to be necessary, Q.E.D. will authorize the return of the component, at owner's expense. If the productproves not to be defective within the terms of this warranty, then all costs and expenses in connection with the processing of the Purchaser's claim and all costs for repair, parts and labor as authorized by owner hereunder shall be borne by the purchaser.

RESPONSIBILITY OF THE PURCHASER

The original Purchaser's sole responsibility in the instance of a warranty claim shall be to notify Q.E.D. of the defect, malfunction, or other manner in which the terms of this warranty are believed to be violated. You may secure performance of obligations hereunder by contacting the Customer ServiceDepartment of Q.E.D. and:

1. Identifying the product invovled (by model or serial number or other sufficent description that will allow Q.E.D. to determine which product is defective).
2. Specifying where, when, and from whom the product was purchased.
3. Describing the nature of the defect or malfunction covered by this warranty.
4. Sending the malfunctioning component, after authorization by Q.E.D. to:

QED Environmental Systems, Inc.
6155 Jackson Rd.
AnnArbor, Michigan 48103

P.O. Box 3726 Ann Arbor, MI 48106-3726 USA
1-800-624-2026 Fax (734) 995-1170
info@qedenv.com www.qedenv.com

Multi Water Quality Checker
U-50 Series

Instruction Manual

CODE:GZ0000144342C

Preface

This manual describes the operation of the Multi Water Quality Checker, U-50 Series.
Be sure to read this manual before using the product to ensure proper and safe operation of the instrument. Also safely store the manual so it is readily available whenever necessary.

Product specifications and appearance, as well as the contents of this manual are subject to change without notice.

■ Warranty and Responsibility

HORIBA warrants that the Product shall be free from defects in material and workmanship and agrees to repair or replace free of charge, at HORIBA's option, any malfunctioned or damaged Product attributable to HORIBA's responsibility for a period of one (1) year from the delivery unless otherwise agreed with a written agreement. In any one of the following cases, none of the warranties set forth herein shall be extended;

- Any malfunction or damage attributable to improper operation
- Any malfunction attributable to repair or modification by any person not authorized by HORIBA
- Any malfunction or damage attributable to the use in an environment not specified in this manual
- Any malfunction or damage attributable to violation of the instructions in this manual or operations in the manner not specified in this manual
- Any malfunction or damage attributable to any cause or causes beyond the reasonable control of HORIBA such as natural disasters
- Any deterioration in appearance attributable to corrosion, rust, and so on
- Replacement of consumables

HORIBA SHALL NOT BE LIABLE FOR ANY DAMAGES RESULTING FROM ANY MALFUNCTIONS OF THE PRODUCT, ANY ERASURE OF DATA, OR ANY OTHER USES OF THE PRODUCT.

■ Trademarks

Generally, company names and brand names are either registered trademarks or trademarks of the respective companies.

Conformable Directive

This equipment conforms to the following directives and standards:

Directives: the EMC Directive 2004/108/EC

Standards: [the EMC Directive]
EN61326-1:2006 Class B, Portable test and measurement equipment

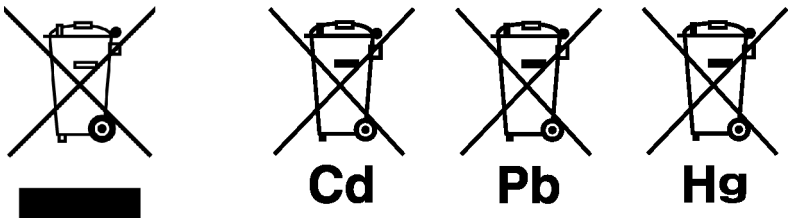
■ Information on Disposal of Electrical and Electronic Equipment and Disposal of Batteries and Accumulators

The crossed out wheeled bin symbol with underbar shown on the product or accompanying documents indicates the product requires appropriate treatment, collection and recycle for waste electrical and electronic equipment (WEEE) under the Directive 2002/96/EC, and/or waste batteries and accumulators under the Directive 2006/66/EC in the European Union.

The symbol might be put with one of the chemical symbols below. In this case, it satisfies the requirements of the Directive 2006/66/EC for the object chemical.

This product should not be disposed of as unsorted household waste.
Your correct disposal of WEEE, waste batteries and accumulators will contribute to reducing wasteful consumption of natural resources, and protecting human health and the environment from potential negative effects caused by hazardous substance in products.

Contact your supplier for information on applicable disposal methods.



FCC Rules

Any changes or modifications not expressly approved by the party responsible for compliance shall void the user's authority to operate the equipment.

■ WARNING

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications.
Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

For your safety

Warning messages are described in the following manner. Read the messages and follow the instructions carefully.

● Meaning of warning messages

⚠DANGER	This indicates an imminently hazardous situation which, if not avoided, will result in death or serious injury. This signal word is to be limited to the most extreme situations.
⚠WARNING	This indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.
⚠CAUTION	This indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. It may also be used to alert against unsafe practices. Without safety alert indication of hazardous situation which, if not avoided, could result in property damage.

● Symbols

Description of what should be done, or what should be followed

Description of what should never be done, or what is prohibited

■ Safety Precautions

This section provides precautions to enable you to use the product safely and correctly and to prevent injury and damage. The terms of DANGER, WARNING, and CAUTION indicate the degree of imminency and hazardous situation. Read the precautions carefully as it contains important safety messages.

⚠ WARNING
Do not disassemble or modify the meter. May cause overheating or fire, resulting in accidents.

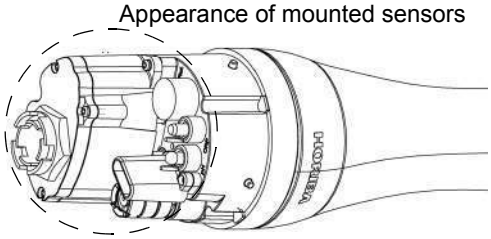
⚠ CAUTION
The pH and ORP sensors are made of glass. Handle them carefully to avoid breakage.
Do not ingest the DO, pH or ORP standard solutions. If it comes into contact with the eyes, rinse thoroughly with water. If swallowed, consult a physician.
Keep away from water when using USB communication. Improper use may result in fire or damage.

Points of concern

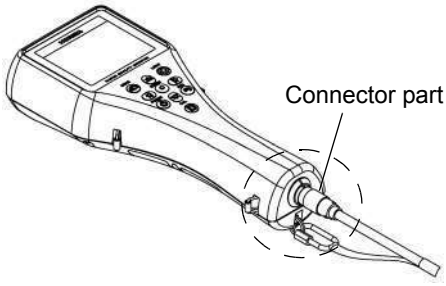
Use of the equipment in a manner not specified by the manufacturer may impair the protection provided by the equipment. It may also reduce equipment performance.

- **Sensor probe**
  - Do not immerse the sensor probe in seawater or other samples with high salinity. Doing so may erode metallic parts. After use, promptly wash the sensor probe thoroughly in water.
  - Do not immerse the sensor probe in alcohol, organic solvent, strong acid, strong alkaline, and other similar solutions.
  - Do not subject to strong shocks.
  - Do not perform measurement in environments of magnetic fields. Measurement errors may result.
  - The sensor probe is no longer waterproof when the sensors are not mounted.

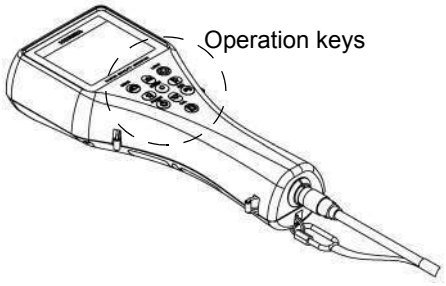
Appearance of mounted sensors



  - Does not support measurement of samples containing fluorine.
  - To disconnect the sensor cable or interface cable, pull them out with holding the connector part. Do not pull the cable part; it may cause breakage.

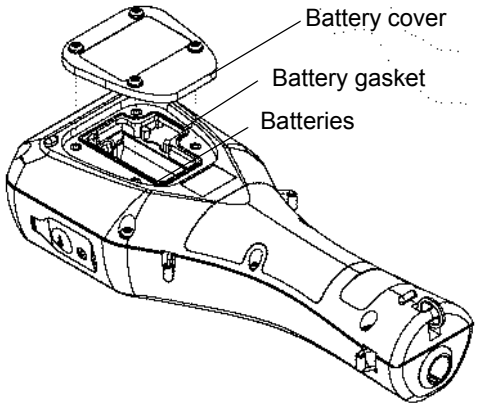


- **Control unit**
  - Do not subject to strong shocks.
  - The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.



  - The control unit is no longer waterproof when the USB cable is connected.
  - When operating the control unit only, protect the connector with the connector cap provided.

- Remove the batteries when not using the control unit for an extended period of time. Battery fluid leakage may cause equipment failure.
- Do not wipe the control unit with organic solvents or powder polish. The surface may deteriorate or its printing may disappear. If the display becomes dirty, wipe the dirt off with a soft cloth soaked in neutral detergent.
- Do not turn the power OFF or disconnect the cable during calibration or setting. Memory data may be erased.
- To perform measurement, connect the sensor probe cable before turning the power ON.
- Do not remove the battery gasket or twist it.
- When opening the battery case, make sure that no foreign matter is attached to the battery gasket.
- Do not use any unspecified batteries; it may cause breakage.



Measurement

- Do not pull the cable when lowering the sensor probe into the sample during measurement. Lower the sensor probe into the sample on a chain or string.
- Before lowering the sensor probe into the sample, do not connect the hook on the unit to a human body.
- The correct values are not displayed if the sensor is not mounted when the measurement display is activated.
- Perform DO measurement with no air bubbles in the internal solution.
- Do not reuse a membrane cap of DO sensor.
- Use the spanner for DO sensor provided to attach or remove the DO sensor.
- Avoid both U-53 and U-53G turbidity measurement in air, since the rubber wiper will quickly become damaged.
- Avoid turbidity measurement in direct sunlight, since the readout may be affected.

Calibration

During atmosphere calibration for the DO electrode with DO salinity compensation set to automatic, values are compensated based on electrical conductivity, but calibration is performed normally.

Location of use and storage

- Storage temperature: −10°C to 60°C
- Relative humidity: Under 80% and free from condensation

Store the meter in locations void of dust, strong vibrations, direct sunlight, corrosive gases, near air conditioners or windy areas.

Disposal of the product

When disposing of the product, follow the related laws and/or regulations of your country for disposal of the product.

Description in this manual

Note

This interprets the necessary points for correct operation and notifies the important points for handling the unit.

Reference

This indicates where to refer for information.

Tip

This indicates reference information.

Contents

1 About this Unit

The U-50 Series Multi Water Quality Checker features an integrated control unit and sensors. It is capable of making a maximum of eleven simultaneous measurements for various parameters, and is perfect for use in the field. The U-50 Series is designed with on-site ease-of-use in mind, provides a wide variety of functions, and can be used for water quality measurements and inspections of river water, groundwater, and waste water.

2 Device Information

2.1 Measurement parameters

Parameters	Model				
	U-51	U-52	U-52G	U-53	U-53G
pH (pH)	✓	✓	✓	✓	✓
pH (mV)	✓	✓	✓	✓	✓
Oxidation reduction potential (ORP)	✓	✓	✓	✓	✓
Dissolved oxygen (DO)	✓	✓	✓	✓	✓
Electrical conductivity (COND)	✓	✓	✓	✓	✓
Salinity (SAL) [expressed as electrical conductivity]	✓	✓	✓	✓	✓
Total dissolved solids (TDS) [expressed as electrical conductivity]	✓	✓	✓	✓	✓
Seawater specific gravity (SG) [expressed as electrical conductivity]	✓	✓	✓	✓	✓
Water temperature (TEMP)	✓	✓	✓	✓	✓
Turbidity (TURB) [LED transmission/front 30° scattering method]	–	✓	✓	–	–
Turbidity (TURB) [tungsten lamp 90° transmission/scattering method] with wiper	–	–	–	✓	✓
Water depth (DEP)	–	–	✓	✓	✓
GPS	–	–	✓	–	✓

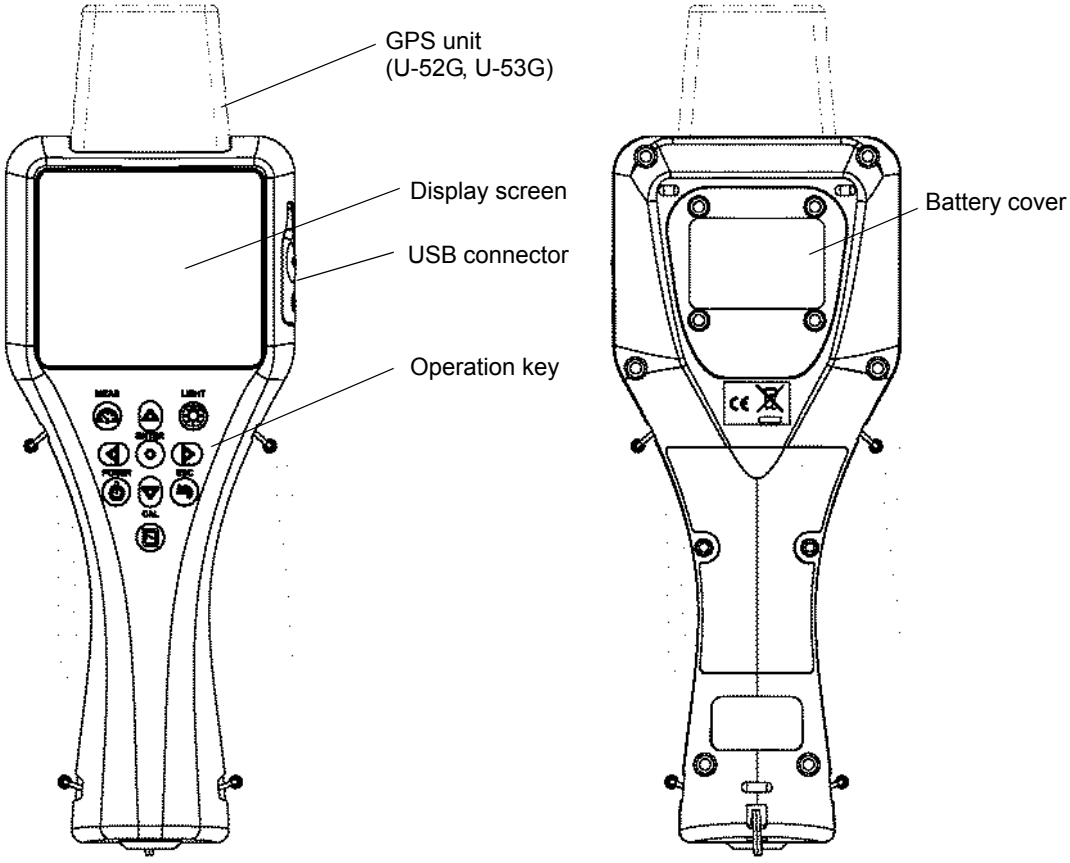
"✓" indicates a measurable parameter.

2.2 Packing list

Parts Name	Quantity	Note
Control unit	1	
Sensor probe	1	
pH sensor (#7112)	1	
ORP sensor (#7313)	1	
Reference electrode (#7210)	1	
DO sensor (#7543)	1	
Turbidity sensor (#7800)	1	With U-52/U-52G only. Attached to the sensor probe.
Turbidity sensor (#7801)	1	With U-53/U-53G only. Attached to the sensor probe.
pH 4 standard solution (#100-4)	1	500 mL
pH reference internal solution (#330)	1	250 mL
DO sensor internal solution set (#306)	1	Internal solution (50 mL), Sandpaper (#8000, #600), Syringe
DO Membrane spare parts set	1	
Spanner for DO sensor	1	
Cleaning brush	1	
calibration cup	1	transparent calibration cup, black calibration cup
Back pack	1	
Strap	1	
Alkaline batteries	4	LR14
Silicon grease	1	
Instruction manual	1	

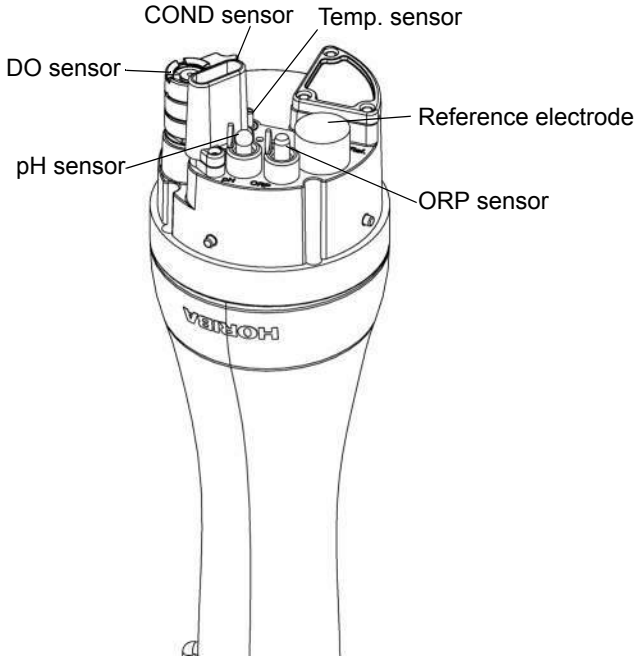
2.3 Parts name and functions

● Display

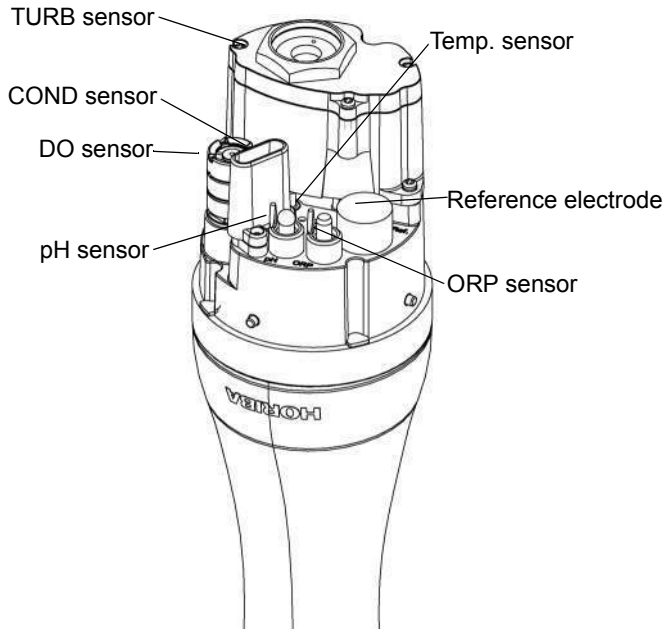


Front view　　Back view

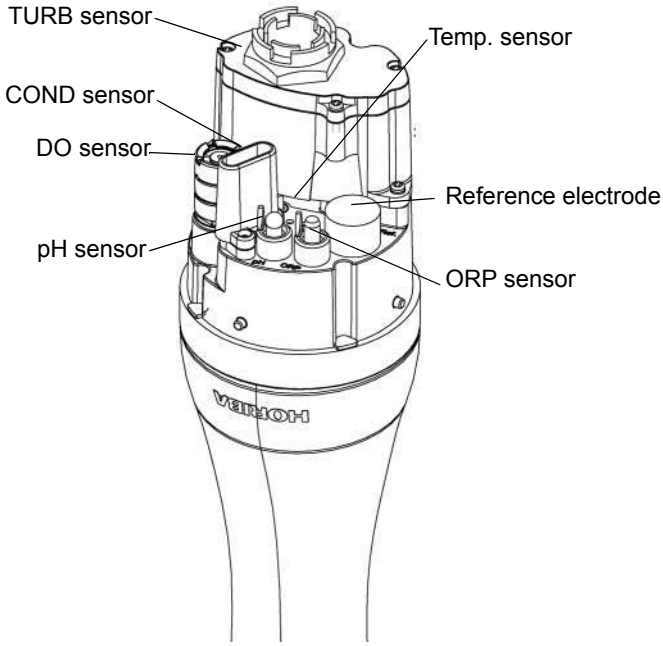
● Sensor probe (U-51)



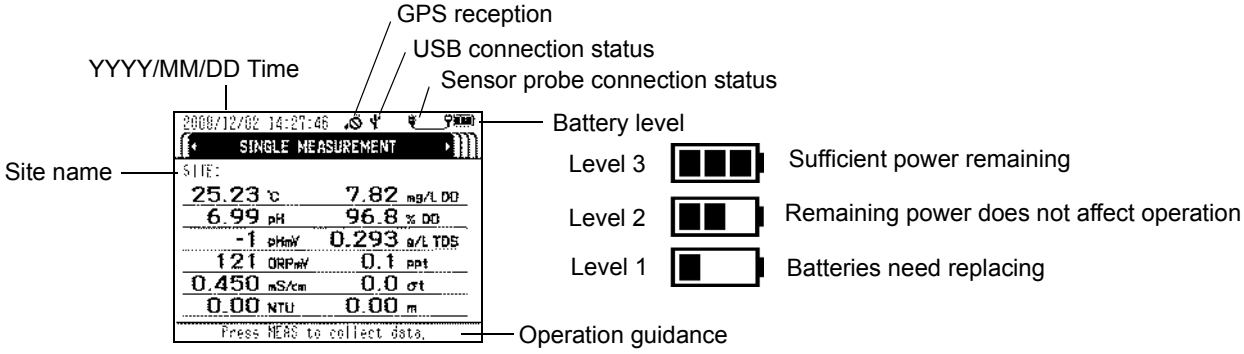
● Sensor probe (U-52)



● Sensor probe (U-53)



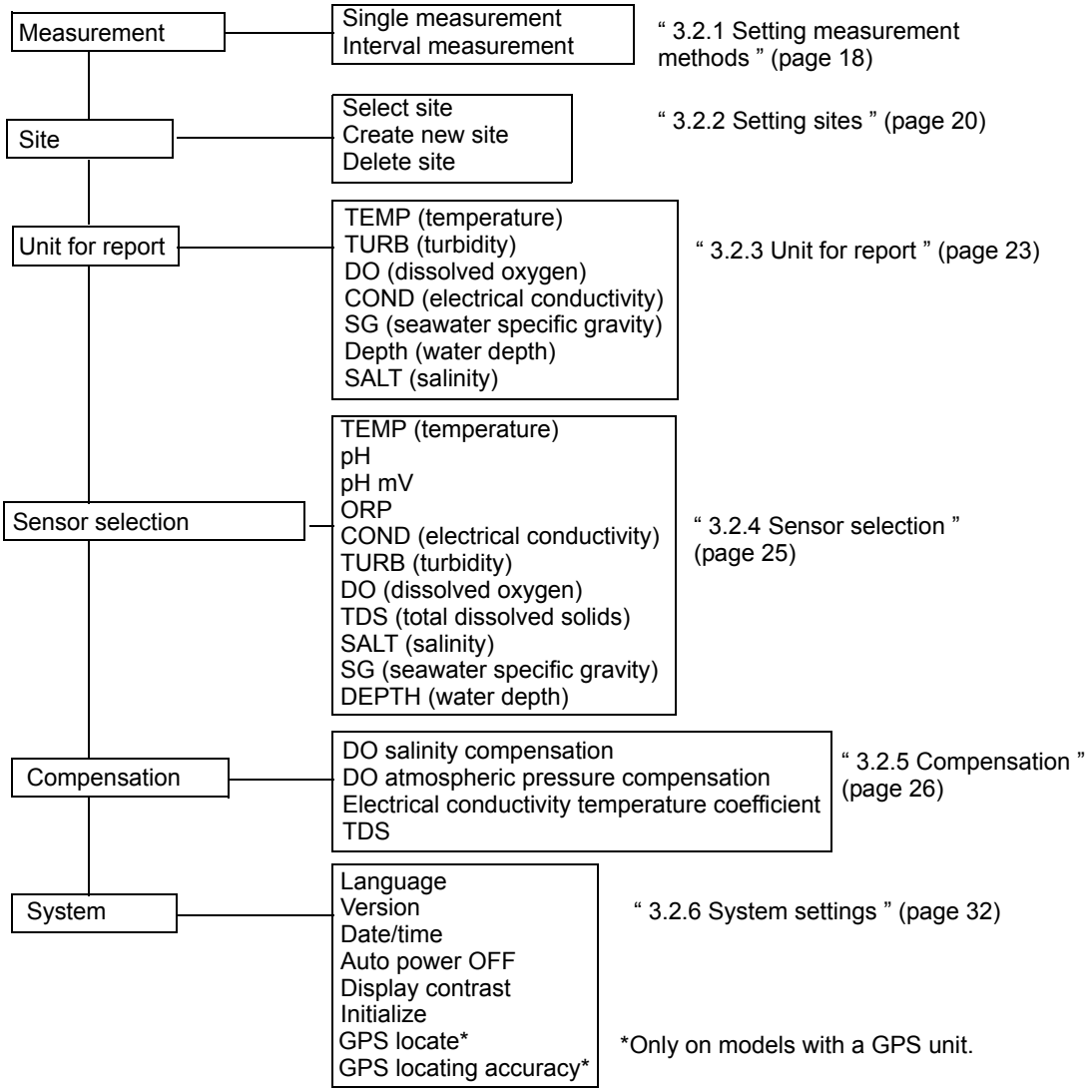
● Display screen



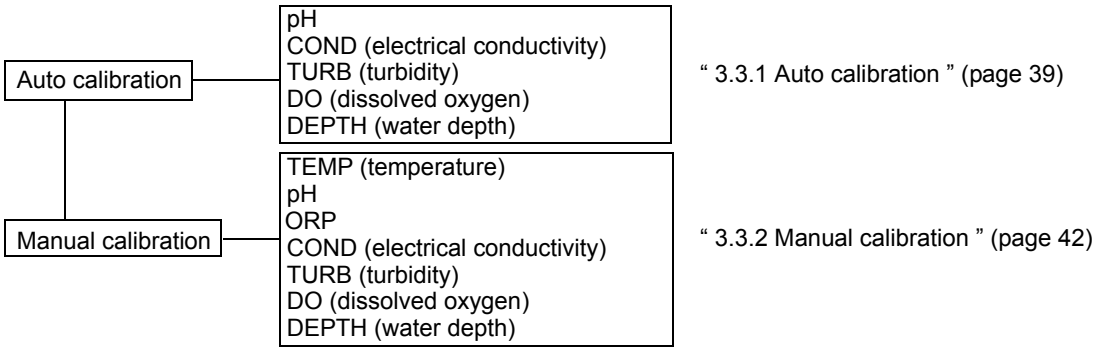
● Operation key

	Key name	description
	POWER key	Turns the system's power ON/OFF. The initial screen appears immediately after turning the power ON. Press and hold down the POWER key for about 3 seconds to turn the power ON and OFF.
	MEAS key	When pressed in the measurement screen, used to set the measurement values of all the measurement parameters. Measurement values flash until the data stabilizes.
		When pressed in the setting, calibration or data operation screen, returns to the measurement screen.
	ENTER key	Used to execute functions, set entered values or store data in memory.
	CAL key	Switches to the calibration screen.
	ESC key	Returns to the immediately preceding operation.
	LIGHT key	Turns the backlight ON/OFF. ● Using the backlight shortens battery life. ● The backlight does not light for about 3 seconds after power ON. ● When the sensor probe is connected while the display's backlight is lit, the backlight goes out for about 3 seconds.
	Left key	Moves the cursor to the left.
	Right key	Moves the cursor to the right.
	Up key	Moves the cursor up.
	Down key	Moves the cursor down.

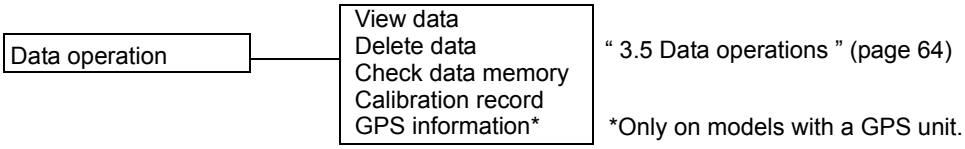
2.4 Setting menu items



2.5 Calibration menu items



2.6 Data operation menu items



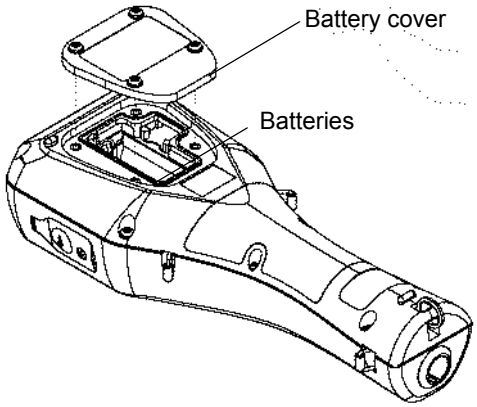
3 Basic Operation

3.1 System setup

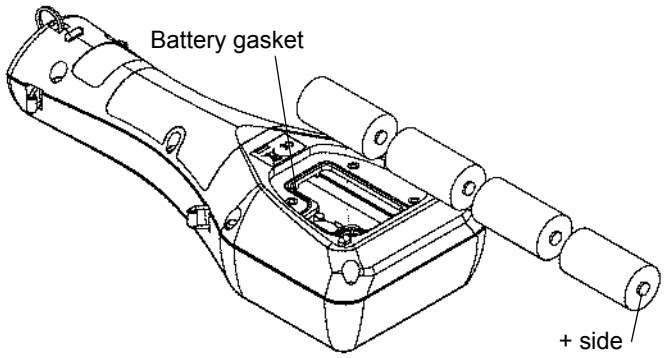
3.1.1 Inserting and replacing the batteries

The control unit is shipped without batteries. Follow the steps below to insert the batteries when using the system for the first time or replacing old batteries.

1. **Loosen the 4 screws on the battery cover by using No. 2 Phillips head screwdriver and remove the cover.**



2. **If replacing the batteries, discard the old batteries.**
3. **Insert new batteries in the control unit.**
   Check that the battery gasket is not dirty or twisted.



4. **Replace the battery cover and fasten it with the 4 screws.**
   Tighten the screws to less than 0.5 N·m.

Note

- **Data and settings will not be lost when the batteries are replaced.**
- **If dirty or twisted, the battery gasket will fail to keep the batteries dry. Check its condition before closing the cover.**
- **To ensure long service life, replacing the battery gasket periodically (once a year) is recommended.**

Precautions when using dry cell batteries

- **Batteries to use: LR14 alkaline dry cell batteries (C-size dry cell batteries) or rechargeable nickel-metal hydride dry cell batteries (C-size)**
  Do not use manganese batteries.
- **Dry cell batteries used incorrectly may leak or burst. Always observe the following**
  - Orient the batteries correctly (positive and negative ends in correct positions).
  - Do not combine new and used batteries, or batteries of different types.
  - Remove the batteries when not using the system for a prolonged period.
  - If batteries leak, have the system inspected at your nearest Horiba service station.

● Battery life

- The battery life for continuous operation when using C-size alkaline dry cell batteries is about 70 hours.
- Using the backlight consumes a proportionate amount of battery power, shortening battery life.
- Searching position information using the GPS unit consumes a proportionate amount of battery power, shortening battery life.
- Nickel-metal hydride secondary batteries can be used, but the battery life is not guaranteed since it will vary according to usage (number of times data is saved, number of charges and amount of each charge). In general, secondary batteries have one-half to one-third the life of C-size alkaline batteries.
- The 70-hour battery life figure applies to a control unit operating temperature of 20°C or more. The battery characteristics shorten the battery life at operating temperatures lower than 20°C, so check the remaining battery level, and replace the batteries before it reaches Level 1.
- The batteries packed with the system at the time of shipment are for checking operation. Their life is not guaranteed.
- The 70-hour battery life figure is the amount of operating time the batteries can provide until the system stops operating. The system may fail during operation if the remaining battery level is low, so it is a good idea to check the remaining battery level and replace the batteries with new ones well before the batteries run out completely.

U-51/52

Battery life: 70 hours (backlight off)

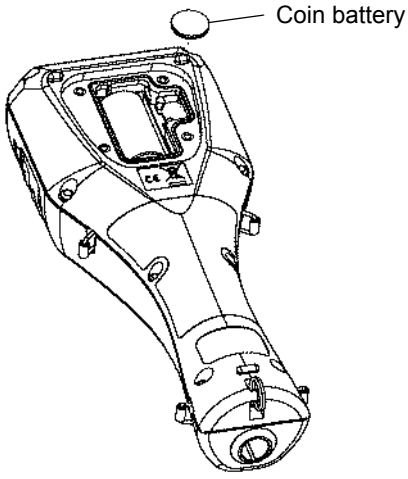
U-53

Battery life: 500 measurements (backlight off)

- Since U-53 is designed for turbidity measurement with wiper, its battery life is estimated in terms of the number of turbidity measurement sequences performed.
- Battery power is also consumed by measurement operations other than turbidity measurement.
- The battery life when turbidity measurement is not performed is about 70 hours.

3.1.2 Replacing the coin battery

- Coin battery to use: CR-2032
- The coin battery is only for the clock. It will provide problem-free operation for three years, but when using the clock continuously, it should be replaced every two years as a precaution.
- When replacing the coin battery for the clock, leave the control unit ON. If the coin battery is replaced when the control unit is turned OFF, the clock will be reset to the default settings.



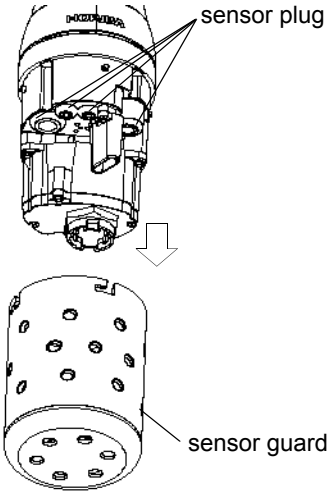
3.1.3 Attaching sensors

Note

- **When attaching or replacing a sensor, wipe any moisture off the sensor probe and sensor.**
- **Be sure to keep water out of sensor connectors. If moisture comes in contact with a sensor connector, blow-dry it with dry air.**
- **The sensor probe is not waterproof when the sensor is not mounted.**
- **Take care not to tighten the sensor too much.**

● Attaching the pH sensor

1. **Remove the sensor guard.**



2. **Remove the sensor plug.**

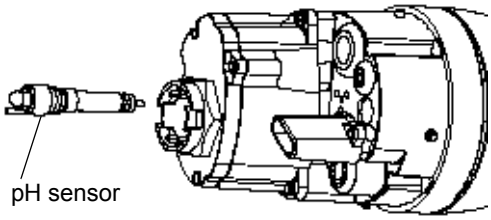
3. **Coat the pH sensor O-ring with a thin layer of silicon grease (part No. 3014017718).**

Note

Be sure no grease from the O-ring gets on the sensor connector. If the sensor connector gets grease on it, wipe it off with a soft cloth soaked in alcohol.

4. **Make sure there is no moisture on the sensor probe's sensor connector (marked "pH").**

5. **Fasten the pH sensor securely by hand.**



6. **Clean the sensor with an alcohol-soaked cloth.**

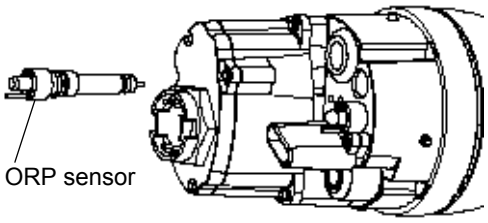
Attaching the ORP sensor

1. **Remove the sensor guard.**
2. **Remove the sensor plug.**
3. **Coat the ORP sensor O-ring with a thin layer of grease (part No. 3014017718).**

Note

Be sure no grease from the O-ring gets on the sensor connector. If the sensor connector gets grease on it, wipe it off with a soft cloth soaked in alcohol.

4. **Make sure there is no moisture on the sensor probe's sensor connector (marked "ORP").**
5. **Fasten the ORP sensor securely by hand.**



6. **Clean the sensor with an alcohol-soaked cloth.**

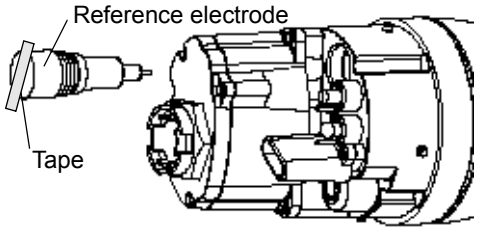
Attaching the reference electrode

1. **Remove the sensor guard.**
2. **Remove the sensor plug.**
3. **Coat the reference electrode O-ring with a thin layer of grease (part No. 3014017718).**

Note

Be sure no grease from the O-ring gets on the sensor connector. If the sensor connector gets grease on it, wipe it off with a soft cloth soaked in alcohol.

4. **Make sure there is no moisture on the sensor probe's sensor connector (marked "REF").**
5. **Fasten the reference electrode securely by hand.**
6. **Remove the tape from the liquid junction part of the reference electrode.**



Attaching the dissolved oxygen (DO) sensor

1. **Remove the membrane cap mounted on the DO sensor beforehand, and replace it with the new membrane cap provided. Replace the internal solution with fresh solution. The main component of the internal solution is potassium chloride (KCl), so the old solution can be disposed of down a sink or other drain.**

Reference

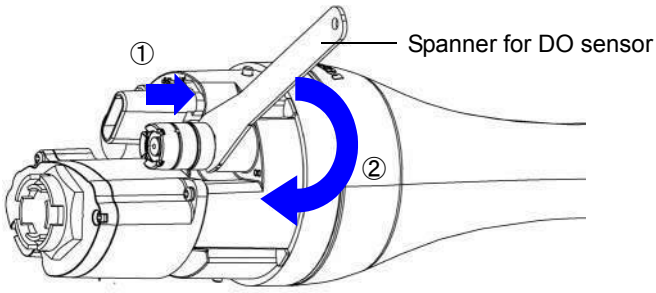
“ 4.5 Replacing the membrane cap ” (page 87)

2. **Screw in the DO sensor to attach it, allowing the internal solution to overflow slightly.**
3. **Use a soft cloth to wipe off the internal solution that overflowed onto the DO sensor.**
4. **Remove the sensor guard.**
5. **Remove the sensor plug.**
6. **Coat the DO sensor O-ring with a thin layer of grease (part No. 3014017718).**

Note

Be sure no grease from the O-ring gets on the sensor connector. If the sensor connector gets grease on it, wipe it off with a soft cloth soaked in alcohol.

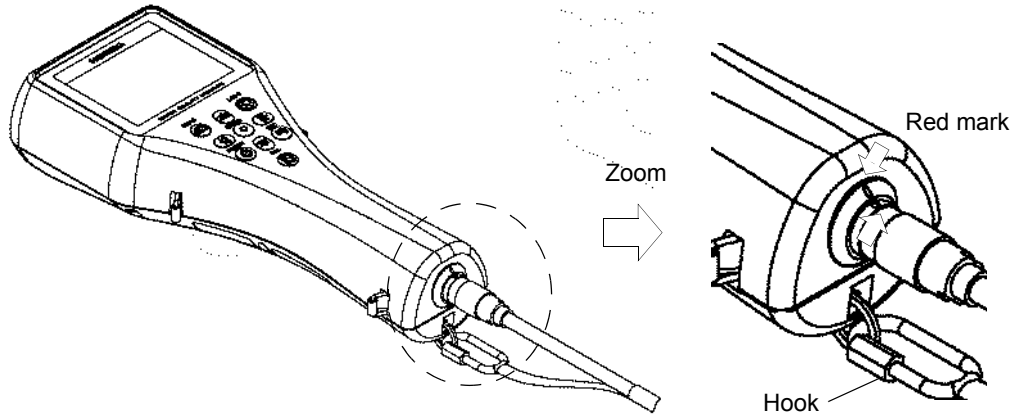
7. **Make sure there is no moisture on the sensor probe’s sensor connector (marked "DO").**
8. **Fasten the DO sensor securely using the spanner for DO sensor.**
   - Hold the DO sensor with the provided spanner for DO sensor and push the sensor down. (Step 1 in figure below)
   - Screw the DO sensor in place. (Step 2 in figure below)



3.1.4 Connecting the control unit and sensor probe

Note

Connect the control unit with its power OFF.

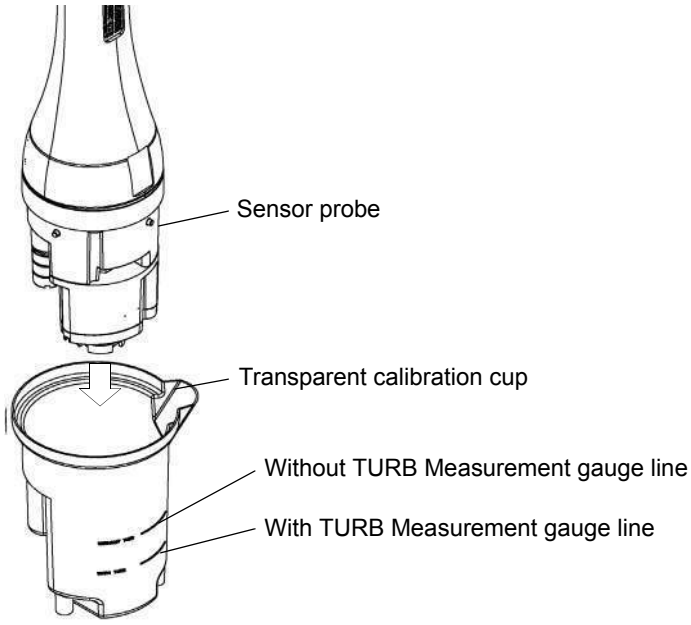


1. **Align the red mark on the connector, and press the connector in until you hear it click.**
2. **Connect the cable's hook to the display.**

3.1.5 Conditioning

Carry out the steps below when using the unit for the first time or when the system has not been used for 3 months or longer.

1. **Fill the transparent calibration cup to the line with pH 4 standard solution.**
   The transparent calibration cup has With TURB Measurement and Without TURB Measurement gauge lines.
2. **Insert the sensor probe in the transparent calibration cup.**



Note

Check that all sensors are attached.

3. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON. Leave the unit for at least 20 minutes to condition the sensors.**

Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

Tip

- **The procedure for immersing the sensor probe in the pH standard solution is the same as that described in " 3.3.1 Auto calibration " (page 39).**
  Auto calibration can be performed using the same pH 4 standard solution that was used in the conditioning procedure.
- **Immersing the sensor in the standard solution is generally required for sensor conditioning, but a voltage supply is required for DO sensor conditioning. Turning ON the power of the control unit is necessary during sensor conditioning.**

3.1.6 GPS (U-52G, U-53G)

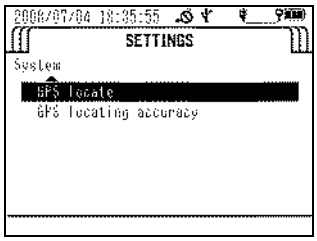
The GPS position measurement precision is proportional to the GPS position measurement time. When the position measurement precision increases, the position measurement time also increases. See " ● GPS locating accuracy" (page 17) for how to set the position measurement precision. See " ● GPS locate" (page 15) below for how to check acquired GPS data.

● GPS locate

1. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

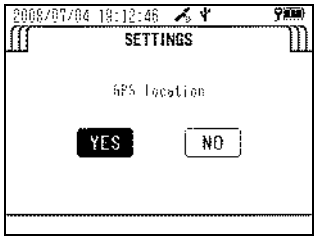
2. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**

3. **Press the down (▽) key to move the cursor to "GPS locate", then press the ENTER key.**



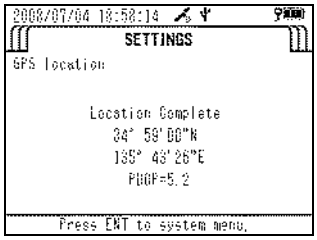
4. **The message "Press ENT key to start position measurement." appears. Press the ENTER key.**

5. **The message "Execute GPS position measurement?" appears. Move the cursor to "YES", then press the ENTER key.**

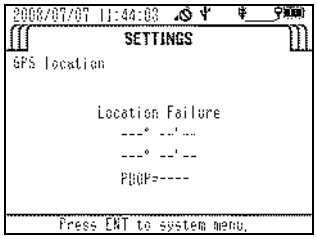


6. **The message "Warming up. Please wait." appears. Wait until the system has finished warming up (about 10 seconds).**

- Position measurement starts automatically when warmup has finished. Position measurement is performed up to 10 times.
- The GPS location complete screen appears after successful position measurement.

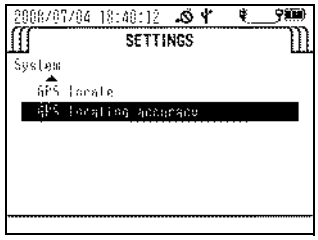


- The GPS location failure screen appears after position measurement has failed. Redo the measurement in a location free from obstacles, or wait for the meteorological conditions to improve before redoing the measurement.

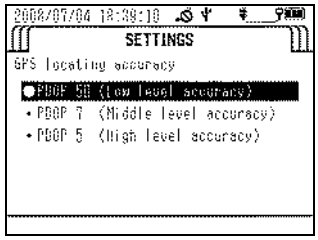


● **GPS locating accuracy**

1. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
2. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**
3. **Press the down (▽) key to move the cursor to "GPS locating accuracy", then press the ENTER key.**



4. **The screen below appears. Move the cursor to the locating accuracy, then press the ENTER key. The black circle (●) indicates the currently set precision.**



3.2 Settings

3.2.1 Setting measurement methods

This section describes how to set the measurement method.

● Measurement methods

● U-51/U-52

Single measurement	Pressing the MEAS key acquires the 5-second average for the selected measurement parameter.
Interval measurement	Pressing the MEAS key acquires and saves the 5-second average for the selected measurement parameter in the set interval. The measurement interval can be set to any value between 10 seconds and 24 hours.

● U-53

The U-53 turbidity sensor uses a tungsten lamp. The lamp lights for about 10 seconds, and the average measurement value acquired during this interval is displayed.

Single measurement	Pressing the MEAS key acquires the 5-second average for the selected measurement parameter after wiper operation. The 10-second average is acquired when measuring turbidity.
Interval measurement	Pressing the MEAS key acquires and saves the 5-second average for the selected measurement parameter in the set interval. The 10-second average is acquired when measuring turbidity. The measurement interval can be set to any value between 10 seconds (final check of this value required; 30 seconds may be better for U-52) and 24 hour.

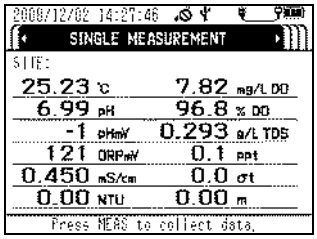
Reference

“ 3.4 Measurement ” (page 61)

● Operation method

1. **Press and hold down the control unit’s POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

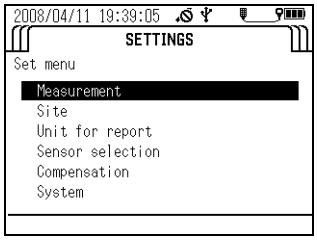


Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

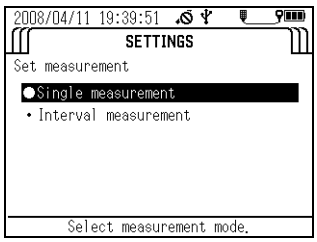
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "Measurement", then press the ENTER key.**



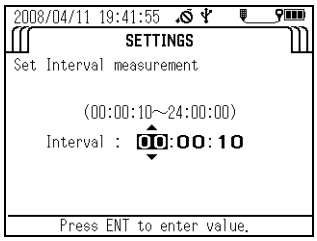
4. **Press the down (▽) key to move the cursor to the desired measurement mode. Press the ENTER key to save the setting.**

The black circle (●) indicates the currently selected measurement mode.



5. **If you selected "Interval measurement", the display switches to the screen used to set the measurement interval. Press the up (△) and down (▽) keys to set the measurement interval.**

The measurement interval can be set to any value between 10 seconds and 24 hours in the case of the U-51 and U-52, or between 30 seconds and 24 hours in the case of the U-53.



3.2.2 Setting sites

The site function allows position data to be connected to corresponding measurement data. Sites have the following specifications and features:

Site names: Text data consisting of up to 20 one-byte alphanumeric characters, spaces, etc.
Site names can be used for control unit searches and as labels for computer processing.
Site names allow measurement data to be saved with a name corresponding to the actual location where it was measured.

You can use site information as a search key when viewing data uploaded by a PC or data saved in the control unit (see " 3.5 Data operations " (page 64)).

- **Selecting sites**

  You can select previously created sites. The black circle (●) indicates the name of the currently selected site. No sites are created at new purchasing or after initialization. Select a site after first creating one from the "Create new site" menu.

- **Creating new sites**

  You can create and save new sites. Up to 20 site names can be registered.

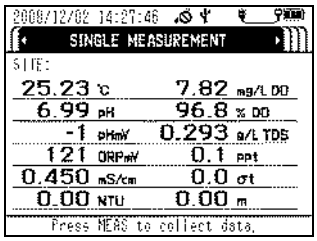
- **Deleting sites**

  You can select a previously created site and delete it.

● **Operation methods**

- **Selecting a site**

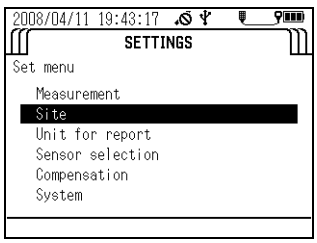
  1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
     The "MEASUREMENT" screen appears after about 10 seconds.



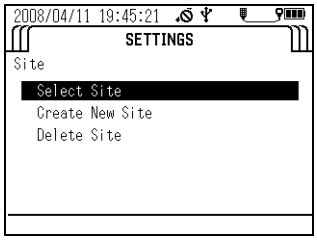
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

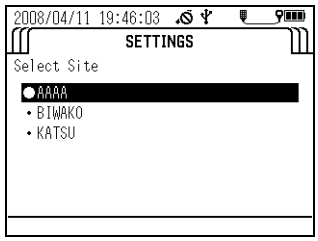
  2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
  3. **Press the down (▽) key to move the cursor to "Site", then press the ENTER key.**



4. **Press the down (▽) key to move the cursor to "Select Site", then press the ENTER key to display the names of the currently saved sites.**

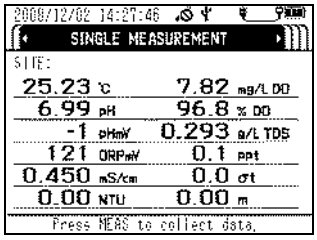


The black circle (●) indicates the currently selected site.



● **Creating a new site**

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
The "MEASUREMENT" screen appears after about 10 seconds.

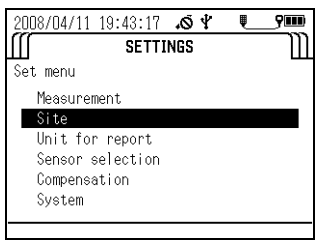


Note

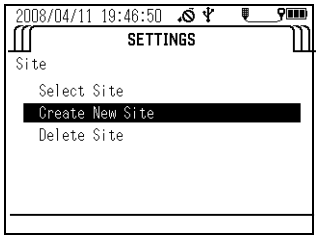
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

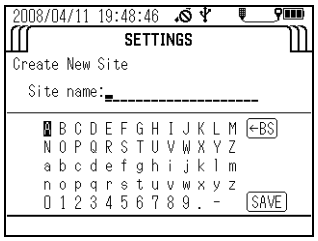
3. **Press the down (▽) key to move the cursor to "Site", then press the ENTER key.**



4. **Press the down (▽) key to move the cursor to "Create New Site", then press the ENTER key.**
Enter the desired site name (up to 20 alphanumeric non-Asian width characters).

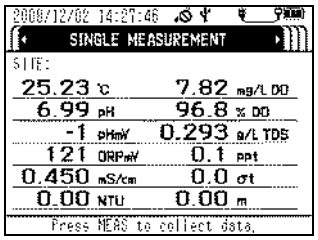


5. **Press the up (△), down (▽), right (▷), and left (◁) keys to move the cursor to each letter or number to use in the name, then press the ENTER key to confirm the entered characters. To delete incorrectly entered characters, move the cursor to "BS" and press the ENTER key to start deleting from the last character. When you have finished entering the name, save it by moving the cursor to "SAVE" and pressing the ENTER key.**



● **Deleting a site**

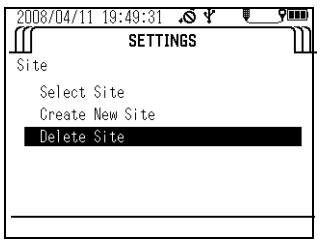
1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
The "MEASUREMENT" screen appears after about 10 seconds.



Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

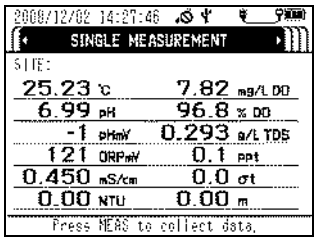
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**



3. **Press the down (▽) key to move the cursor to "Site", then press the ENTER key.**

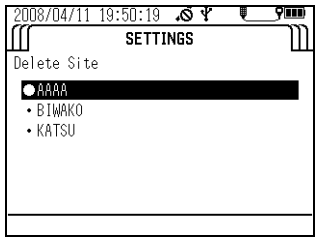
4. **Press the down (▽) key to move the cursor to "Delete Site", then press the ENTER key.**

A list of the currently saved sites appears. The black circle (●) indicates the currently selected site.



5. **Press the down (▽) key to move the cursor to the site to delete, then press the ENTER key to delete it.**

The currently selected site can be deleted after a different site has been selected from the site selection menu or after all unselected sites have been deleted. The same site name cannot be registered more than once.



3.2.3 Unit for report

Note

Units can only be selected when the sensor probe is connected.

Follow the steps below to set the measurement units of measurement parameters. No units are displayed if a measurement parameter has not been selected in the measurement parameter selection screen (see “ 3.2.4 Sensor selection ” (page 25)).

1. **Press and hold down the control unit’s POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

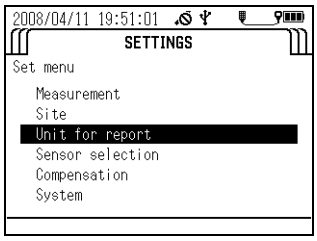
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

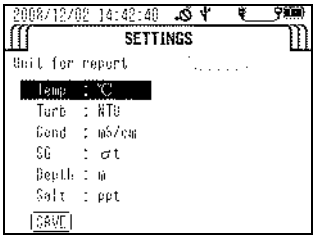
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "Unit for report", then press the ENTER key.**

A list of the currently selected measurement parameters and their units appears. Note that measurement parameters not selected (in the measurement parameter selection screen) are not displayed.

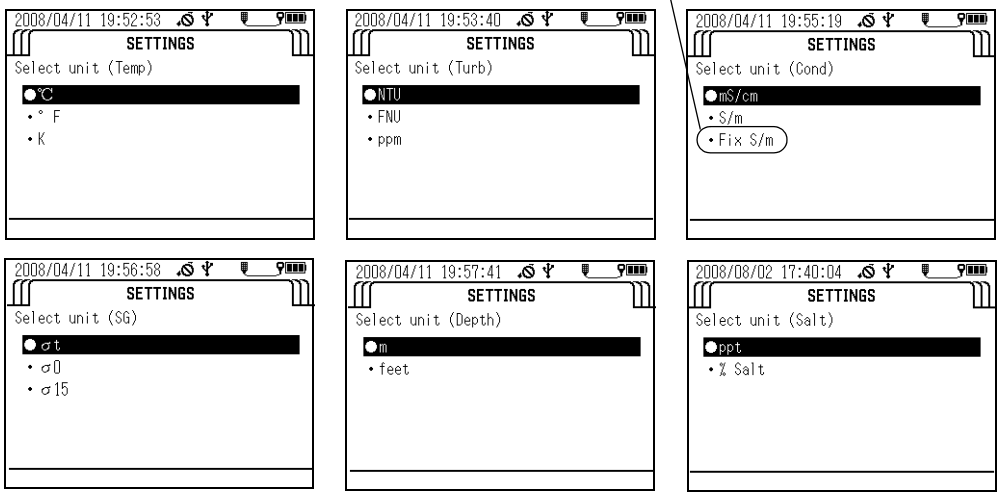


4. **Press the up (△) and down (▽) keys to move the cursor to the item to change, then press the ENTER key.**

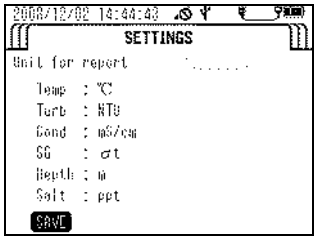


5. **A list of the units that can be selected appears. The black circle (●) indicates the currently selected unit. Press the up (△) and down (▽) keys to move the cursor to the desired unit, then press the ENTER key.**

Fix S/m: Fixes S/m with no auxiliary units for any measurement range.



6. **To save the changes, press the up (△) and down (▽) keys to move the cursor to SAVE, then press the ENTER key. If you do not want to save the changes, press the ESC key.**



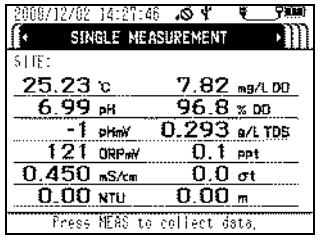
3.2.4 Sensor selection

Note

Measurement parameters can only be selected when the sensor probe is connected.

You can set between 1 and 11 measurement parameters to display in the control unit screen. Follow the steps below to select the desired measurement parameters.

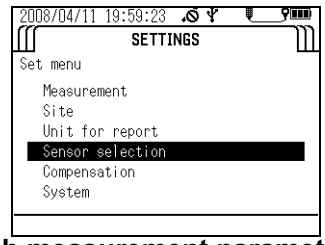
1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
   The "MEASUREMENT" screen appears after about 10 seconds.



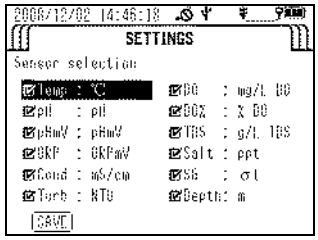
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
3. **Press the down (▽) key to move the cursor to "Sensor selection", then press the ENTER key.**
   A list of the measurement parameters that can be set and the currently set units are displayed.



4. **Move the cursor to each measurement parameter to change, then press the ENTER key.**
   A check in the check box of a measurement parameter indicates it will be displayed.
5. **To save the changes, press the up (△), down (▽), left (◁) and right (▷) keys to move the cursor to SAVE, then press the ENTER key. If you don't want to save the changes, press the ESC key.**



Note

Available measurement parameters differ according to product specifications.

3.2.5 Compensation

Note

Compensation settings can only be made when the sensor probe is connected.

U-50 series have following functions of compensation.

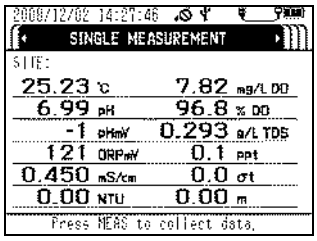
- Salinity compensation and atmospheric pressure compensation for dissolved oxygen (DO)
- Temperature compensation for conductivity (COND)
- Setting total dissolved solid (TDS) coefficient for TDS

● Salinity compensation (DO)

The dissolved oxygen (DO) value is presented higher than actual value if salinity compensation is not added, because the increase of salinity gives higher DO value. To obtain correct value salinity compensation is needed. The following modes are available for calculation of salinity compensation.

AUTO:	Salinity compensation is performed automatically with salinity converted from conductivity.
Value input:	Press the up (△) and down (▽) keys to enter a setting value when the salinity is known.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
The "MEASUREMENT" screen appears after about 10 seconds.

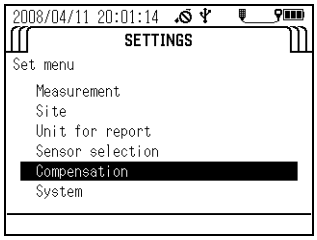


Note

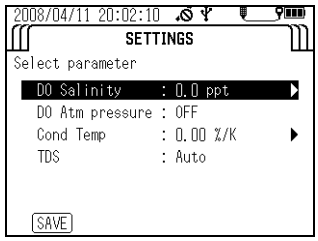
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

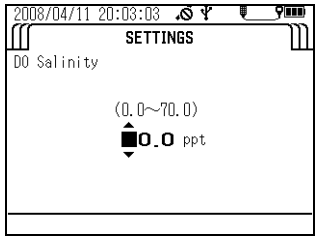
3. **Press the down (▽) key to move the cursor to "Compensation", then press the ENTER key.**



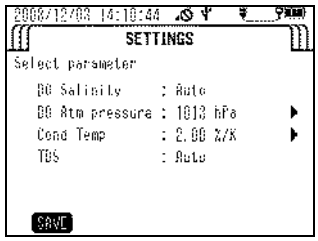
4. **Press the down (▽) key to move the cursor to "DO Salinity", then press the ENTER key to toggle the setting between "Auto" and "Input mode".**
Default: Auto



5. **If you selected "Input mode", press the right (▷) key to display the compensation value input screen. Press the up (△) and down (▽) keys to enter the desired value, then press the ENTER key to set it.**



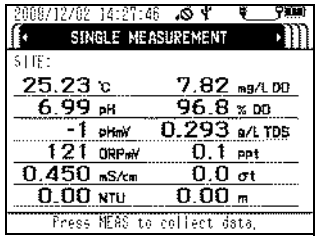
6. **To save the change, press the up (△) and down (▽) keys to move the cursor to SAVE, then press the ENTER key. If you don't want to save the change, press the ESC key.**



● **Atmospheric pressure compensation (DO)**

Differences in the atmospheric pressure of the measurement location influence the Dissolved Oxygen (DO) measurement. By setting (input) the actual atmospheric pressure of the measurement location into the control unit, it is possible to standardize the measured Dissolved Oxygen (DO) value to a value at the standard atmospheric pressure (1013 hPa).

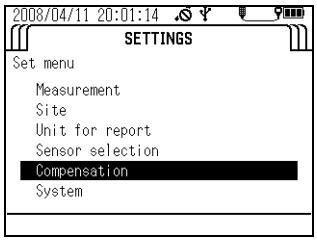
1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
The "MEASUREMENT" screen appears after about 10 seconds.



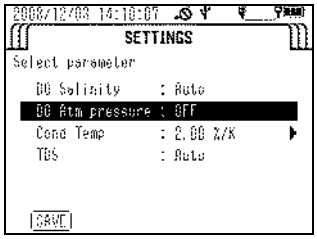
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

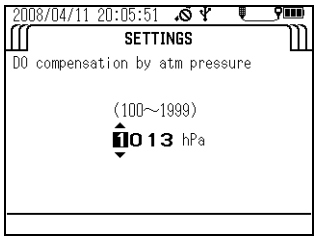
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
3. **Press the down (▽) key to move the cursor to "Compensation", then press the ENTER key.**



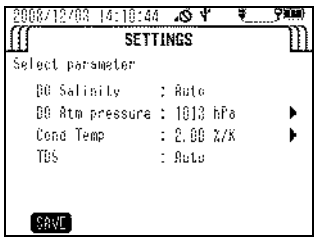
4. **Press the down (▽) key to move the cursor to "Cond Temp", then press the ENTER key to toggle the setting between "OFF" and "Input mode".**
   Default: OFF



5. **If you selected "Input mode", press the right (▷) key to display the compensation value input screen. Press the up (△) and down (▽) keys to enter the desired value, then press the ENTER key to set it.**



6. **To save the change, press the up (△) and down (▽) keys to move the cursor to SAVE, then press the ENTER key. If you don't want to save the change, press the ESC key.**

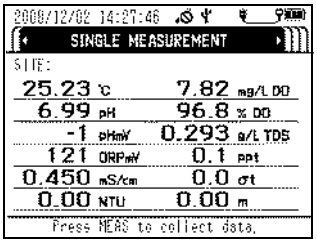


● Temperature compensation for conductivity (COND)

Sample conductivity (COND) varies with temperature, and this control unit uses a temperature compensation coefficient to automatically standardize the conductivity (COND) at 25°C. The initial setting coefficient is 2%/K, which is the generally used.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

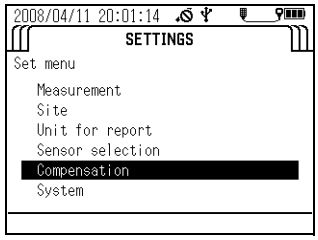


Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

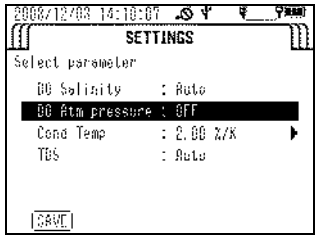
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "Compensation", then press the ENTER key.**

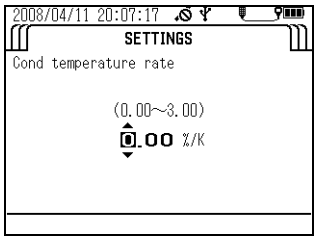


4. **Press the down (▽) key to move the cursor to "Cond Temp", then press the ENTER key to toggle the setting between "OFF" and "Input mode".**

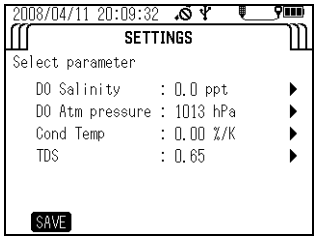
Default: 2.00%/K



5. **If you selected "Input mode", press the right (▷) key to display the compensation value input screen. Press the up (△) and down (▽) keys to enter the desired value, then press the ENTER key to set it.**



6. **To save the change, press the up (△) and down (▽) keys to move the cursor to SAVE, then press the ENTER key.**
If you don't want to save the change, press the ESC key.



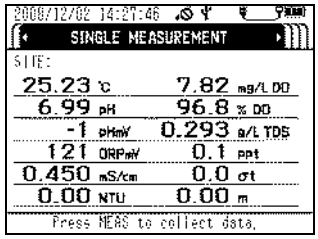
● Setting a total dissolved solid (TDS) coefficient

The total dissolved solid amount (TDS) is a converted value obtained by multiplying the conductivity (COND) by a known coefficient. The coefficient initially set for the control unit is based on a conversion for KCl and $CaCO_3$ solutions and it depends on the conductivity (COND) value as shown below.

Conductivity (COND) (S/m)	Conversion coefficient
< 0.05	0.65
0.05 to 0.5	0.64
0.5 to 1	0.63
1 to 3	0.62
3 to 5	0.61
> 5	0.60

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

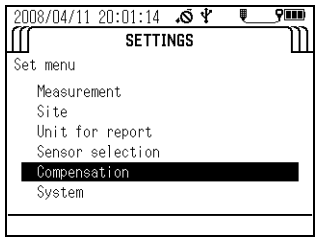


Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

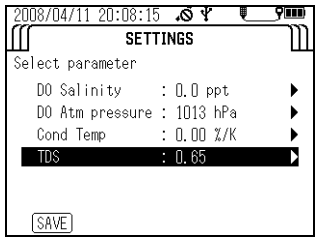
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "Compensation", then press the ENTER key.**

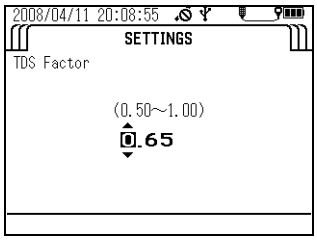


4. **Press the down (▽) key to move the cursor to "TDS", then press the ENTER key to toggle the setting between "AUTO" and "Input mode".**

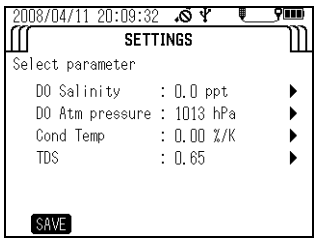
Default: Auto



5. **If you selected "Input mode", press the right (▷) key to display the compensation value input screen. Press the up (△) and down (▽) keys to enter the desired value, then press the ENTER key to set it.**



6. **To save the change, press the up (△) and down (▽) keys to move the cursor to SAVE, then press the ENTER key. If you don't want to save the change, press the ESC key.**



3.2.6 System settings

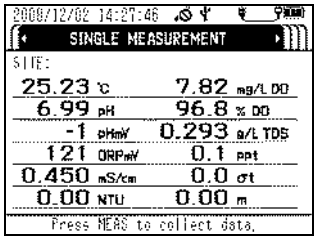
The system settings let you change the display language, check the system software version, set the date/time, set the auto power OFF time, set the display contrast, and initialize the settings.

● **Display language**

Follow the steps below to select either English or Japanese as the display language.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

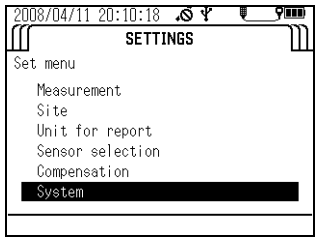


Note

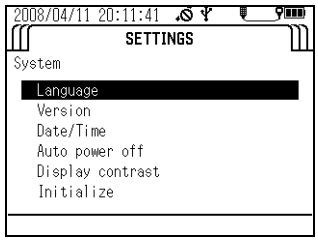
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

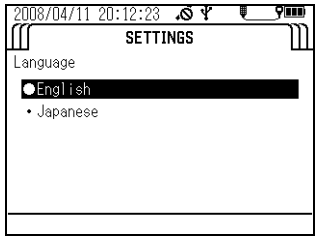
3. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**



4. **Press the down (▽) key to move the cursor to "Language", then press the ENTER key.**



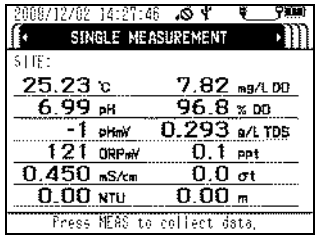
5. **A list of the supported display languages appears. Press the up (△) and down (▽) keys to move the cursor to the desired language, then press the ENTER key.**
The black circle (●) indicates the currently selected display language.



● Version

Follow the steps below to display the program No. and version of the control unit and sensor probe software.
The program No. and version of the sensor probe software will not be displayed if the sensor probe is not connected.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
The "MEASUREMENT" screen appears after about 10 seconds.

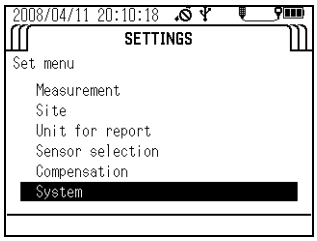


Note

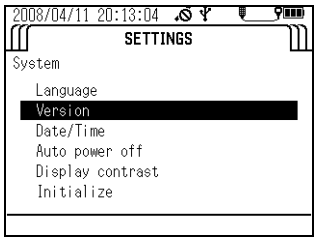
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**



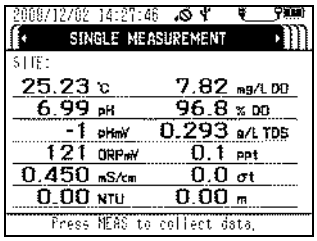
4. **Press the down (▽) key to move the cursor to "Version", then press the ENTER key.**
The program No. of the control unit and sensor probe software appears.



● **Setting the date/time**

Follow the steps below to set the date and time.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
The "MEASUREMENT" screen appears after about 10 seconds.

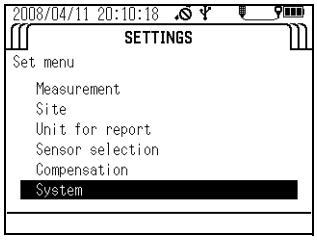


Note

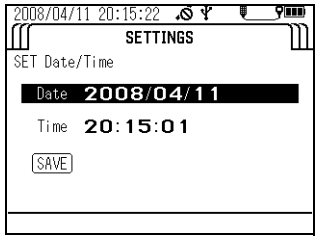
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**

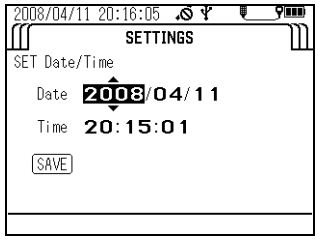


4. **Press the down (▽) key to move the cursor to "Date/time", then press the ENTER key.**

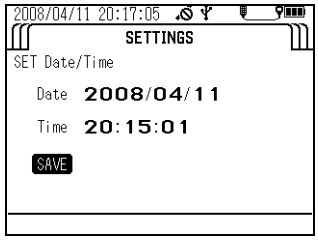


5. **Move the cursor to the date, then press the ENTER key.**

6. **Press the right (▷) key to move the cursor to the year, month, day, hour, minute and second, and press the up (△) and down (▽) keys to enter each value.**



7. **When finished entering settings, press the ENTER key to move the cursor to SAVE, then press the ENTER key again to save the settings.**

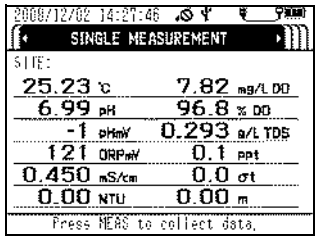


● Setting the auto power OFF time

Follow the steps below to set the time for the auto power OFF function (which turns the power OFF automatically when no operation is performed for the preset amount of time).

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

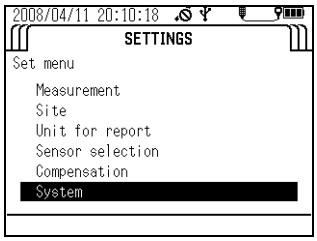
The "MEASUREMENT" screen appears after about 10 seconds.



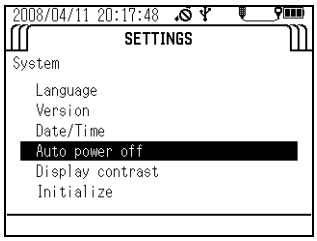
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

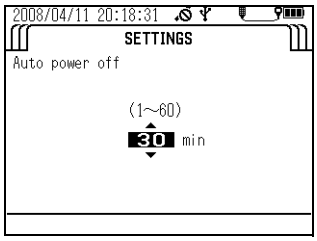
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
3. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**



4. **Press the down (▽) key to move the cursor to "Auto power off", then press the ENTER key.**



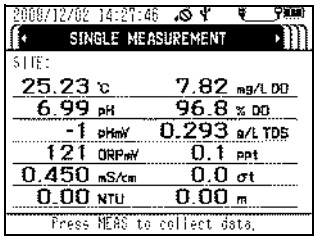
5. **Press the up (△) and down (▽) keys to select the desired time setting, then press the ENTER key.**
   You can select OFF, or settings of 1, 2, 5, 10, 20, 30 or 60 minutes.
   Default: 30 minutes



● Display contrast

Follow the steps below to adjust the display's contrast.

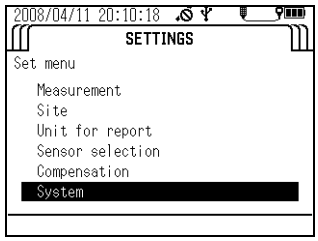
1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
   The "MEASUREMENT" screen appears after about 10 seconds.



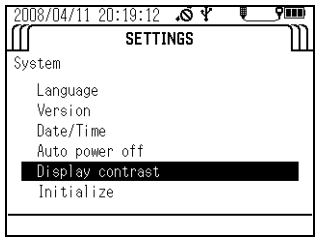
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

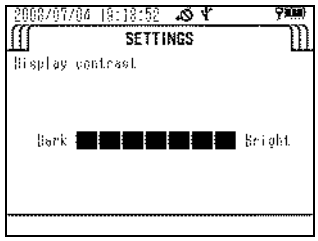
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
3. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**



4. **Press the down (▽) key to move the cursor to "Display contrast", then press the ENTER key.**



5. **Press the left (◁) and right (▷) keys to adjust the contrast.**
   Adjustment can be made in 26 steps.

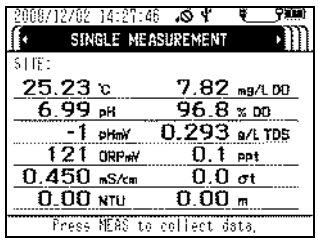


6. **Press the ENTER key.**

● Initialization

Follow the steps below to restore all the settings except date/time to their factory defaults. Factory default calibration data for the electrical conductivity and turbidity sensors will also be deleted at the same time.

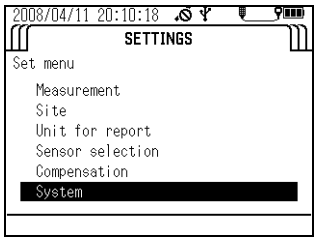
1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
   The "MEASUREMENT" screen appears after about 10 seconds.



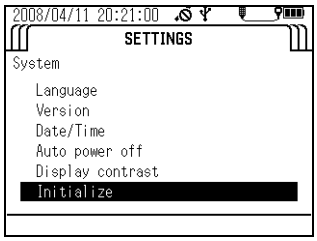
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

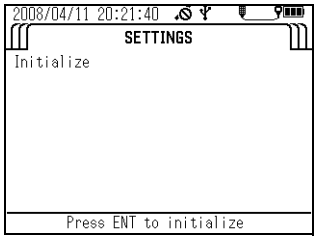
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
3. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**



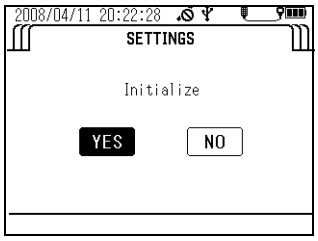
4. **Press the down (▽) key to move the cursor to "Initialize", then press the ENTER key.**



5. **Press the ENTER key again.**



6. **A confirmation message appears asking whether to execute initialization. Press the left (◁) key to move the cursor to YES, then press the ENTER key.**
   The message "Initialize Complete" appears to indicate the process has finished.



3.3 Calibration

To obtain correct measurement values, the sensors need to be calibrated using standard solution before measurement. You can select simultaneous auto calibration of the pH, COND and TURB sensors in pH4 standard solution and DO and DEP sensors simultaneously in air, or manual calibration of individual measurement parameters. You can check the result of the previous calibration using the procedure on " 3.5.4 Checking the calibration record " (page 70).

Note

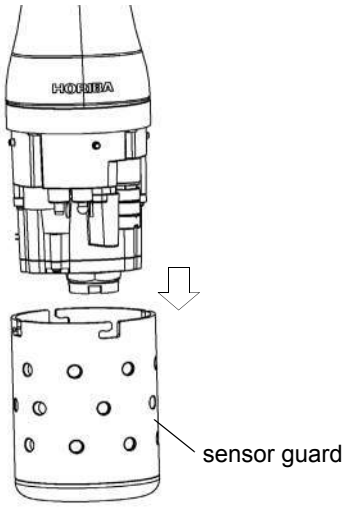
- **Wait at least 20 minutes after turning the system power ON before calibrating the DO sensor.**
- **Make the DO and COND compensation settings before calibration since these settings are applied during calibration.**
- **You can select only the desired parameters for calibration and calibrate just those parameters (see " 3.2.4 Sensor selection " (page 25)).**
- **Use about 200 mL of standard solution in the calibration cup.**
- **Calibration data is stored in the sensor probe.**

3.3.1 Auto calibration

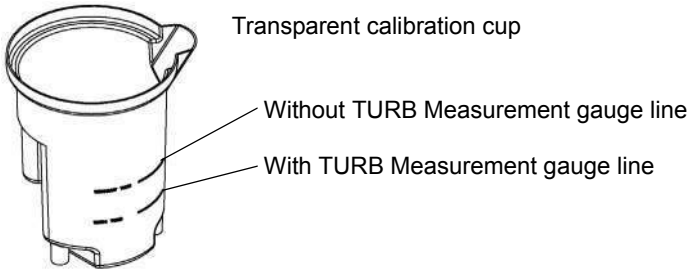
Tip

- **The following parameters are calibrated (at 25°C):**
  pH: Set to 4.01 (zero-point calibration); the span is adjusted to the factory default value.
  COND: 0.449 S/m (4.49 mS/cm, span calibration); the zero point is adjusted to the factory default value.
  TURB: 0 NTU (zero-point calibration); the span is adjusted to the factory default value.
  DO: 8.92 mg/L (span calibration); the zero point is adjusted to the factory default value.
  DEP: 0 m (zero-point calibration); the zero point is adjusted to the factory default value.
- **If the air temperature changes, the readout value may not be stable. Ensure that the ambient air temperature is the same temperature as the calibration solution, because the internal probe temperature sensor and external temperature sensor (in the calibration solution) are used for the auto calibration. Allow the probe and standard solution to equilibrate for 1 hour if a thermometer is not available to verify that these temperatures are the same.**
- **Do not hold the probe while performing the auto calibration. Body temperature may elevate the internal temperature sensor measurement creating DO calibration error.**

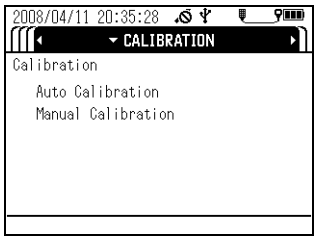
1. **Remove the sensor guard and wash the sensor probe 2 or 3 times with deionized water.**



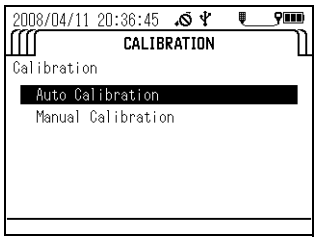
2. **Remove the transparent calibration cup.**
3. **Fill the transparent calibration cup to the line with pH 4 standard solution.**
   The transparent calibration cup has With TURB Measurement and Without TURB Measurement gauge lines.



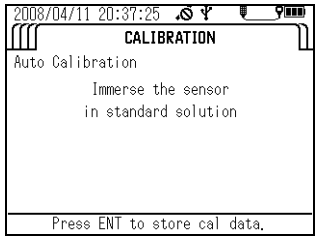
4. **Press the control unit's CAL key to set the calibration mode.**

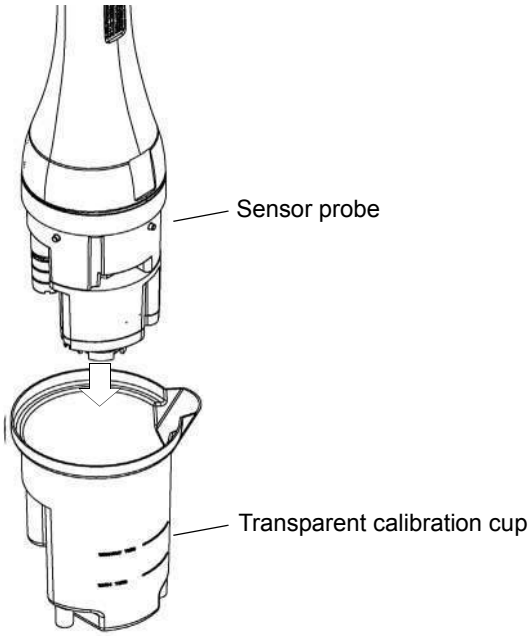


5. **Press the down (▽) key to move the cursor to "Auto Calibration", then press the ENTER key.**

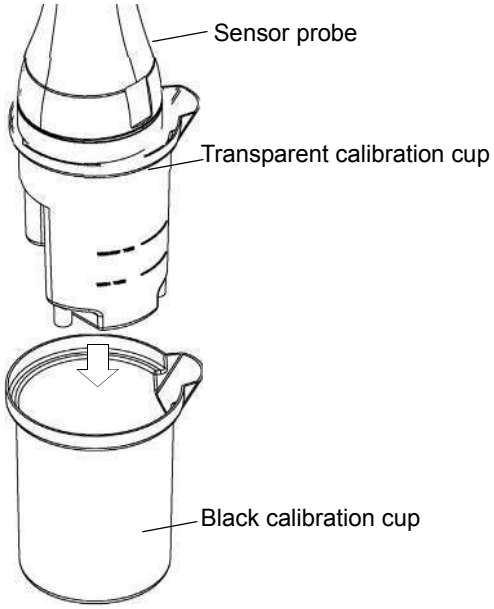


6. **Immerse the sensor probe in the transparent calibration cup.**
Check that the pH sensor, ORP sensor, reference electrode, COND sensor, TURB sensor and temperature sensor are submerged in the pH 4 standard solution and check that there are no air bubbles on the sensor.





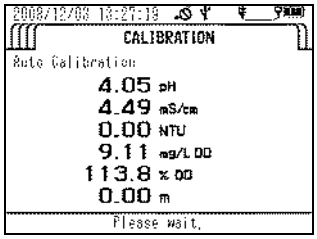
7. **With the sensor probe still in the transparent calibration cup, place the transparent calibration cup into the black calibration cup.**



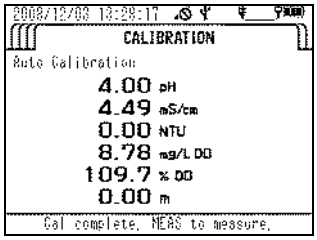
8. **When all the sensor values have stabilized, press the ENTER key to start calibration.**

Note

Do not remove the sensor probe from the calibration solution. U-53 turbidity data will display "----" until the calibration is completed.



Calibration is finished when the message "Cal complete. MEAS to measure." appears. Press the MEAS key to set the measurement screen, then start measurement.



If a calibration error occurs, start calibration after first resolving the issue according to the instructions in " 4.6 Troubleshooting " (page 89).

3.3.2 Manual calibration

The procedures below describe how to calibrate each sensor individually.

Note

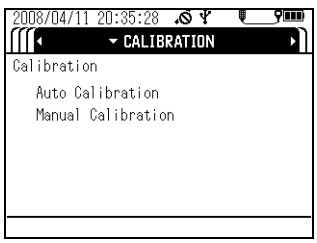
The displayed units are the units set by selecting "Unit for report" in the "SETTINGS" screen.

● **Temperature (TEMP) calibration**

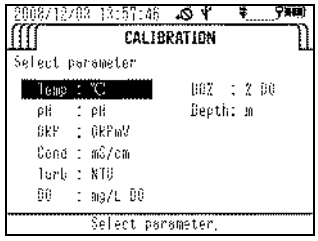
1. **Fill a bucket or similar container with water of a known temperature, and insert the sensor probe in it.**
Wait 5 minutes before starting calibration to allow the sensor probe temperature to stabilize.

2. **Press the control unit's CAL key to set the calibration mode.**

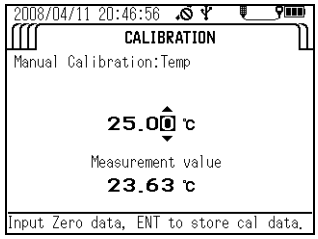
3. **Press the down (▽) key to move the cursor to "Manual Calibration", then press the ENTER key.**



4. **In the parameter selection screen, move the cursor to “Temp”, then press the ENTER key.**



5. **Press the up (△) and down (▽) keys to set the calibration value - the temperature of the water containing the submerged sensor probe.**



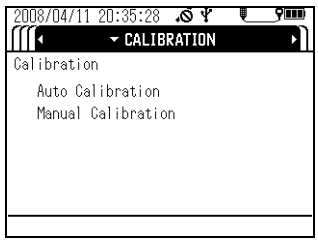
6. **Check that “Measurement value” has stabilized, then press the ENTER key to start calibration.**
   Calibration is finished when the message “Cal complete. CNT to measure.” appears.

● pH calibration

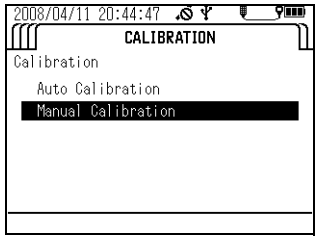
Note

You can select one calibration point (zero-point calibration) or two calibration points (zero-point calibration and span calibration). Carry out two calibration procedures to ensure good measurement precision throughout all measurement ranges.

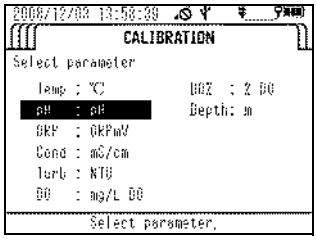
1. **Calibrate the zero point. Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with pH 7 standard solution.**
2. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**
3. **Press the control unit’s CAL key to set the calibration mode.**



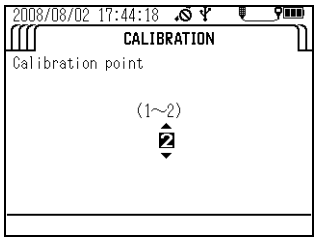
4. **Press the down (▽) key to move the cursor to "Manual Calibration", then press the ENTER key.**



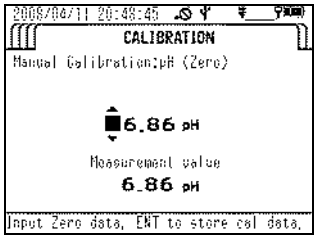
5. **In the parameter selection screen, move the cursor to "pH", then press the ENTER key.**



6. **Set the number of calibration points, then press the ENTER key.**



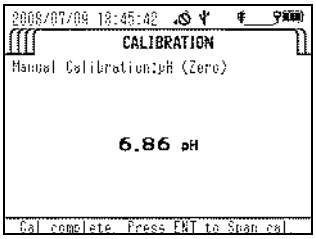
7. **Press the up (△) and down (▽) keys to set the pH value of the pH 7 standard solution containing the submerged sensor probe at the measurement temperature**



Temp. (°C)	pH 4 standard solution Phthalate	pH 7 standard solution Neutral phosphate	pH 9 standard solution Borate
0	4.01	6.98	9.46
5	4.01	6.95	9.39
10	4.00	6.92	9.33
15	4.00	6.90	9.27
20	4.00	6.88	9.22
25	4.01	6.86	9.18
30	4.01	6.85	9.14
35	4.02	6.84	9.10
40	4.03	6.84	9.07
45	4.04	6.84	9.04

8. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

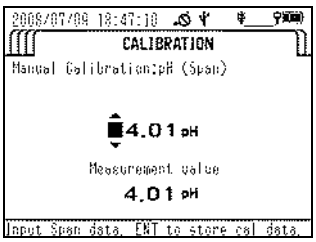
9. **Press the ENTER key to start the span calibration procedure when the message "Cal complete. Press ENT to Span cal." appears.**



10. **Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with pH 4 or pH 9 standard solution.**

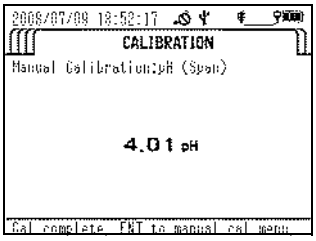
11. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**

12. **Press the up (△) and down (▽) keys to set the pH value of the pH 4 or pH 9 standard solution containing the submerged sensor probe at the measurement temperature.**



13. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

14. **Calibration is finished when the message "Cal complete. ENT to manual cal menu." appears. Press the ENTER key to return to the calibration parameter**

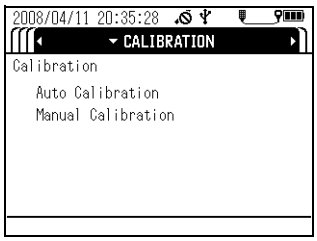


● ORP calibration

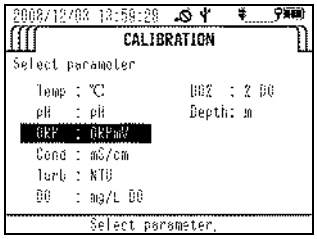
Note

- **If the prepared ORP standard solution is left in open air for one hour or more, the solution may be transformed. For this reason ORP standard solution cannot be stored.**
  Calibrate within one hour of preparing the solution.
- **When measuring sample with low concentrations of oxidants and reductants after conducting an operational check using a standard substance, the measured values may not stabilize or the results of measurement might not be repeatable. If this is the case, start the measurement after immersing the sensors in the sample water sufficiently.**
- **Note that when measuring the ORP of solution with extremely low concentrations of oxidants and reductants, such as tap water, well water, or water treated with purifying equipment, there may be less responsiveness, repeatability, and stability, in general.**
- **When alkaline ion water is left for 5 minutes, its ORP undergoes changes significantly. Always measure alkaline ion water promptly.**

1. **Fill a clean beaker with one bag of ORP standard powder No. 160-22 or No. 160-51. Add 250 mL of deionized water and agitate the solution thoroughly (there will be some excess quinhydrone (a black powder) that floats on the surface when agitating the solution). Fill the transparent calibration cup to the reference line with this standard solution.**
2. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**
3. **Press the control unit's CAL key to set the calibration mode.**
4. **Press the down (▽) key to move the cursor to "Manual Calibration", then press the ENTER key.**



5. **In the parameter selection screen, move the cursor to ORP, then press the ENTER key.**



6. **Press the up (△) and down (▽) keys to set the mV value of the ORP standard solution containing the submerged sensor probe at the measurement temperature.**

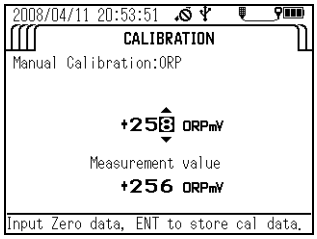


Table 1 Indicated value of ORP standard solution at various temperatures (mV)

Temperature	160-22	16051
5	+274	+112
10	+271	+107
15	+267	+101
20	+263	+95
25	+258	+89
30	+254	+83
35	+249	+76
40	+244	+69

7. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

8. **Calibration is finished when the message "Cal complete. ENT to manual cal menu." appears. Press the ENTER key to return to the calibration parameter selection screen.**

● Conductivity (COND) calibration

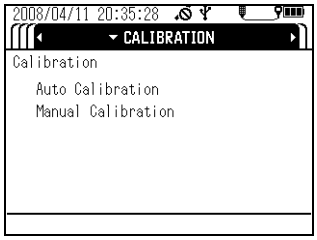
Note

- **To support a wide range of sample concentrations, electrical conductivity is divided into three measurement ranges: 0.0 mS/m to 99.9 mS/m, 0.090 S/m to 0.999 S/m, and 0.9 S/m to 9.99 S/m.**
- **When manually calibrating conductivity, you can select two calibration points (one zero-point calibration point and a span calibration point for one of the three measurement ranges) or four calibration points (one zero-point calibration point and span calibration points for all three measurement ranges). Carry out the four calibration points to ensure good measurement precision throughout all measurement ranges.**
- **Make the compensation setting before calibration since this setting is applied during calibration. (Refer to " 6.5.3 Temperature coefficient " (page 104)).**

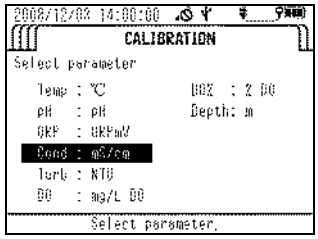
1. **Prepare the standard solution. Dry Potassium chloride (KCl) powder (high-grade commercially available) at 105°C for two hours, and leave it to cool in a desiccator.**
2. **Consult the following table and weigh potassium chloride (KCl), then prepare three standard potassium chloride (KCl) solutions following the procedure below.**

Potassium chloride (KCl) standard solution	Conductivity (COND) value	Potassium chloride (KCl) mass (g) at solution temperature of 25 °C	Calibration range
0.005 mol/L	71.8 mS/m (0.718 mS/cm)	0.373	0.0 mS/m to 99.9 mS/m (0.00 mS/cm to 0.999 mS/cm)
0.050 mol/L	0.667 S/m (6.67 mS/cm)	3.73	0.090 S/m to 0.999 S/m (1.00 mS/cm to 9.99 mS/cm)
0.500 mol/L	5.87 S/m (58.7 mS/cm)	37.2	0.9 S/m to 9.99 S/m (10.0 mS/cm to 99.9 mS/cm)

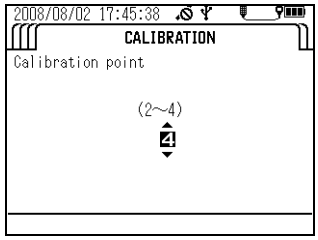
3. **Dissolve the weighed Potassium Chloride (KCl) in deionized water.**
4. **Put the dissolved Potassium Chloride (KCl) into a 1 L measuring flask, and fill to the 1 L mark with deionized water.**
5. **Calibrate the zero point. Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then remove all moisture from the sensor probe (it will be calibrated in air).**
6. **Press the control unit's CAL key to set the calibration mode.**
7. **Press the down (▽) key to move the cursor to "Manual Calibration", then press the ENTER key.**



8. **In the parameter selection screen, move the cursor to “Cond”, then press the ENTER key.**



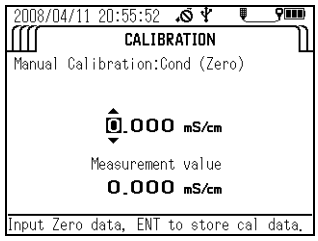
9. **Set the number of calibration points, then press the ENTER key.**



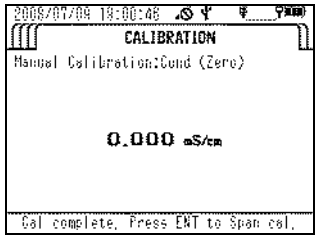
The instructions below assume that four calibration points have been set.

10. **Press the up (△) and down (▽) keys to set the "Cond" value to 0.0 mS/m (0.000 mS/cm).**

11. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**



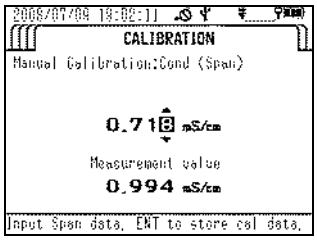
12. **When the message "Cal complete. Press ENT to Span cal." appears, press the ENTER key to start the first span calibration procedure.**



13. **Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with 71.8 mS/m (0.718 mS/cm) standard solution.**

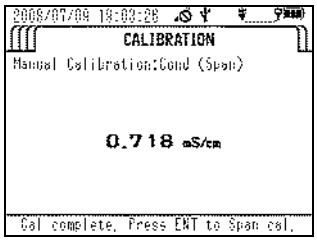
14. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**

15. **Press the up (△) and down (▽) keys to set the "Cond" value to 71.8 mS/m (0.718 mS/cm).**
Calibration range = 0 mS/m to 99.9 mS/m (0 mS/cm to 0.999 mS/cm)



16. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

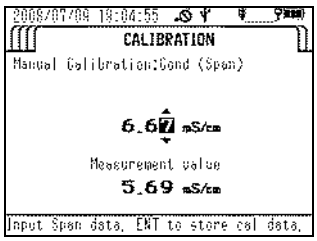
17. **When the message "Cal complete. Press ENT to Span cal." appears, press the ENTER key to start the next span calibration procedure.**



18. **Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with 0.667 S/m (6.67 mS/cm) standard solution.**

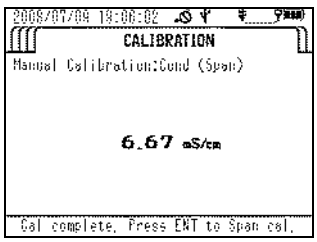
19. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**

20. **Press the up (△) and down (▽) keys to set the "Cond" value to 0.667 S/m (6.67 mS/cm).**
Calibration range = 0.100 S/m to 0.999 S/m (1.00 mS/cm to 9.99 mS/cm)



21. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

22. **When the message "Cal complete. Press ENT to Span cal." appears, press the ENTER key to start the next span calibration procedure.**

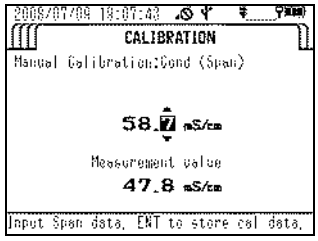


23. **Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with 5.87 S/m (58.7 mS/cm) standard solution.**

24. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**

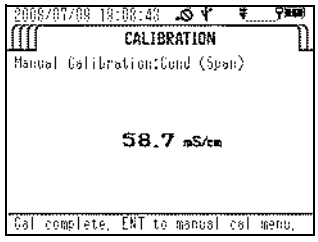
25. **Press the up (△) and down (▽) keys to set the "Cond" value to 5.87 S/m (58.7 mS/cm).**

Calibration range = 1.00 S/m to 10.00 S/m(10.0 mS/cm to 100.0 mS/cm)



26. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

27. **Calibration is finished when the message "Cal complete. ENT to manual cal menu." appears. Press the ENTER key to return to the calibration parameter selection screen.**



● Turbidity (TURB) calibration

Note

- **To support a wide range of sample concentrations, turbidity is divided into three measurement ranges: 0.0 to 9.9 NTU, 10 to 100 NTU, and over 100 NTU.**
- **When manually calibrating turbidity, you can select two calibration procedures (one zero-point calibration procedure and a span calibration procedure for one of the three measurement ranges), three calibration procedures (one zero-point calibration procedure and a span calibration procedure for two of the three measurement ranges) or four calibration procedures (one zero-point calibration procedure and span calibration procedures for all three measurement ranges). Carry out the four calibration procedures to ensure good measurement precision throughout all measurement ranges.**
- **Always use the calibration cup provided. Using other containers can create effects from ambient light that cause incorrect calibration.**

● Preparing the standard solutions

1. **Weigh out 5.0 g of hydrazine sulfate (commercial special grade or above), and dissolve it in 400 mL of deionized water. Dissolve 50 g of hexamethylene tetramine (commercial special grade or above) in 400 mL of deionized water in anther flask.**
2. **Mix the two solutions and add deionized water until the total solution volume is 1000 mL, and mix well. Store this solution at a temperature of 25°C ±3°C for 48 hours.**
   The turbidity value (TURB) of this solution is equivalent to 4000 NTU.
3. **Dilute 4000 NTU-solution 5 times (use a pipette to measure 50 mL of the 4000 NTU solution and pour it into a 250 mL measuring flask, and fill up to 250 mL meniscus)**
   The turbidity value (TURB) of this solution is equivalent to 800 NTU.
4. **Dilute 800 NTU solution 10 times (use a pipette to measure 25 mL of the 800 NTU solution and pour it into a 250 mL measuring flask, and fill up to 250 mL meniscus)**
   The turbidity value (TURB) of this solution is equivalent to 80 NTU.
5. **Dilute 80 NTU solution 10 times (use a pipette to measure 25 mL of the 80 NTU solution and pour it into a 250 mL measuring flask, and fill up to 250 mL meniscus)**
   The turbidity value (TURB) of this solution is equivalent to 8 NTU.

Note

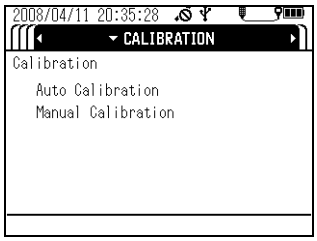
Instead of the standard solutions above, you can use other standard solutions of known concentration measured with other standard instruments.

● U-52, U-53 turbidity calibration

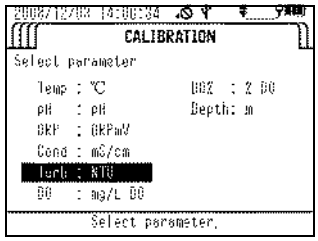
Set the number of calibration points.

You can set between 2 and 4 points.

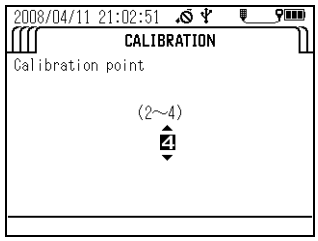
1. **Press the control unit's CAL key to set the calibration mode.**
2. **Press the down (▽) key to move the cursor to "Manual Calibration", then press the ENTER key.**



3. **In the parameter selection screen, move the cursor to "Turb", then press the ENTER key.**



4. **Press the up (△) and down (▽) keys to set the number of calibration points, then press the ENTER key.**

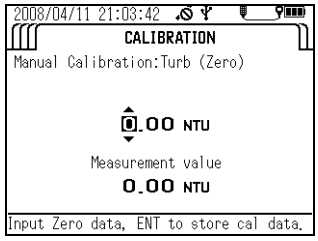


The instructions below assume that four calibration points have been set.

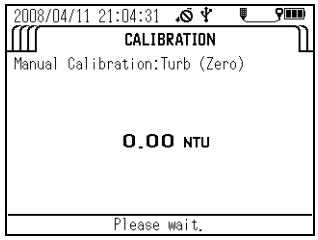
5. **Calibrate the zero point. Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with deionized water.**

6. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**

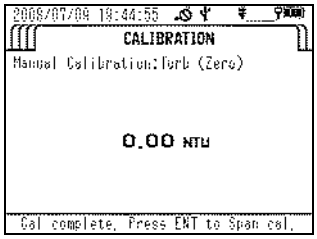
7. **Press the up (△) and down (▽) keys to set the "Turb" value to 0.0 NTU.**



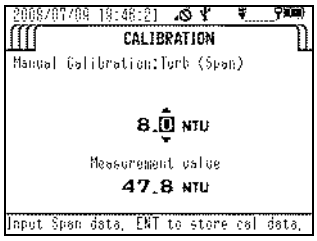
8. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**



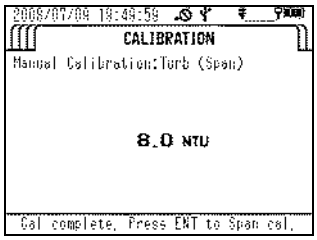
9. **When the message "Cal complete. Press ENT to Span cal." appears, press the ENTER key to start the first span calibration procedure.**



10. **Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with 8 NTU standard solution, or a standard solution of known concentration between 0.1 and 10 NTU.**
11. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**
12. **Press the up (△) and down (▽) keys to set the "TURB" value to 8 NTU, or to the known concentration of the standard solution between 0.1 and 10 NTU. (Input range = 0 NTU to 9.9 NTU (U-51) or 0 NTU to 9.99 NTU (U-52))**

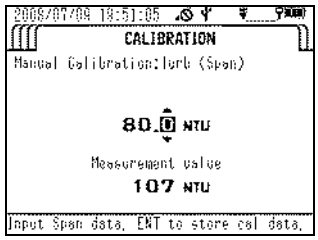


13. **Check that "Current measurement value" has stabilized, then press the ENTER key to start calibration.**
14. **When the message "Cal complete. Press ENT to Span cal." appears, press the ENTER key to start the next span calibration procedure.**



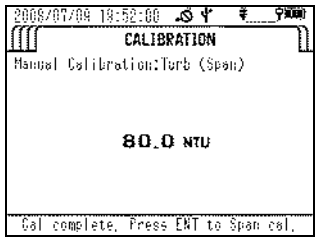
15. **Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with 80 NTU standard solution, or a standard solution of known concentration between 10 and 100 NTU.**
16. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**

17. **Press the up (△) and down (▽) keys to set the "Turb" value to 80 NTU, or to the known concentration of the standard solution between 10 and 100 NTU. (Input range = 10.0 NTU to 99.9 NTU)**



18. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

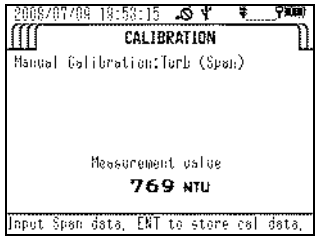
19. **When the message "Cal complete. Press ENT to Span cal." appears, press the ENTER key to start the next span calibration procedure.**



20. **Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with 800 NTU standard solution, or a standard solution of known concentration 100 NTU above.**

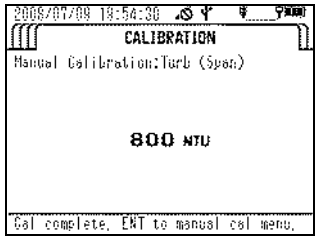
21. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**

22. **Press the up (△) and down (▽) keys to set the "TURB" value to 800 NTU, or to the known concentration of the standard solution 100 NTU above.**
(Input range = 100 NTU to 800 NTU (U-51), 100 NTU to 1000 NTU (U-52))



23. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

24. **Calibration is finished when the message "Cal complete. ENT to manual cal menu." appears. Press the ENTER key to return to the calibration parameter selection screen.**



● Dissolved oxygen (DO) calibration

Note

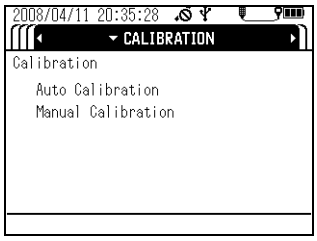
- **You can select one calibration procedure (span calibration) or two calibration procedures (zero-point calibration and span calibration). Carry out the two calibration procedures to ensure good measurement precision throughout all measurement ranges.**
- **It is necessary to prepare new solution before calibration of the Dissolved Oxygen (DO) sensor.**
- **The calibration cup (included) cannot be used to manually calibrate the DO sensor. Use a suitable bottle in which the DO sensor and the temperature sensor can be immersed.**
- **Wait at least 20 minutes after turning the system power ON before calibrating the DO sensor.**
- **Make the compensation setting before calibration since the setting is applied during calibration.**
- **The DO sensor is affected by flow. When performing span calibration with saturated dissolved oxygen water, move the cable slowly up and down (move the sensor probe at a rate of roughly 20 to 30 cm a second) or agitate the saturated dissolved oxygen water.**

1. **Prepare the standard solution.**

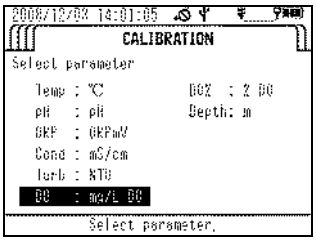
- Add about 50 g of sodium sulfite to 1000 mL of water (either deionized water or tap water) and stir the mixture to dissolve the sodium sulfite in it.
- Pour 1 to 2 liters of water into a suitable flask (either deionized water or tap water). Using a air pump, feed air into the water and aerate the solution until oxygen is saturated.

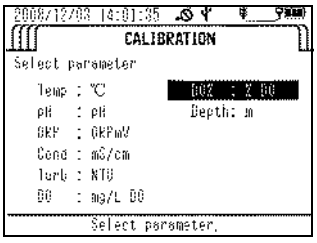
2. **First, calibrate the zero point. Press the control unit's CAL key to set the calibration mode.**

3. **Press the down (▽) key to move the cursor to "Manual Calibration", then press the ENTER key.**

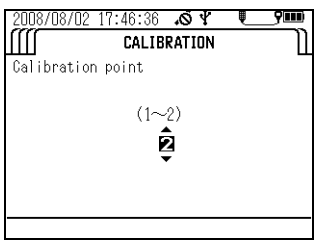


4. **In the parameter selection screen, move the cursor to DO or DO%, then press the ENTER key.**



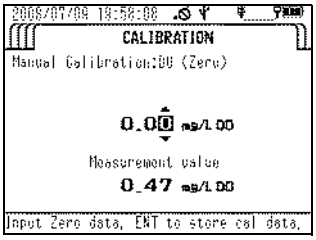


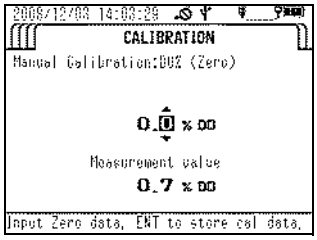
5. **Set the number of calibration procedures, then press the ENTER key.**



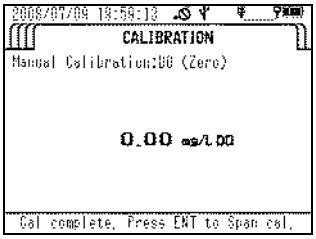
The instructions below assume that two calibration points have been set.

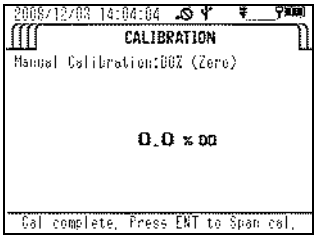
6. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the bottle.**
7. **Press the up (△) and down (▽) keys to set the DO value to 0.00 mg/L or 0.0%.**



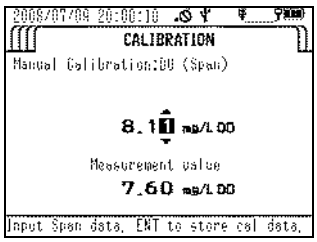


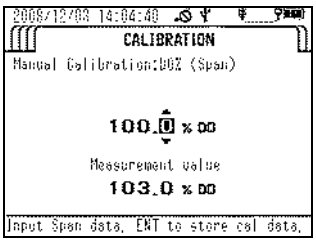
8. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**
9. **When the message "Cal complete. Press ENT to Span cal." appears, press the ENTER key to start the span calibration procedure.**



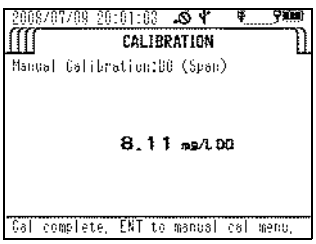


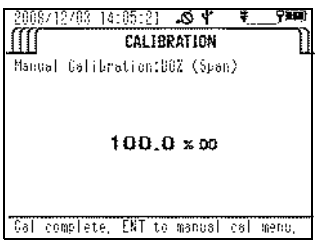
10. **Wash the sensor probe 2 or 3 times with deionized water to remove any dirt, then submerge the sensor probe in the container filled with the span solution.**
11. **Press the up (△) and down (▽) keys to set the DO value to the saturated dissolved oxygen value (mg/L) of the water at that temperature or the dissolved oxygen saturation ratio.**





12. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**
13. **Calibration is finished when the message "Cal complete. ENT to manual cal menu." appears. Press the ENTER key to return to the calibration parameter selection screen.**





Amounts of saturated dissolved oxygen in water at various temperatures (salinity=0.0%)

JIS K0101

Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)
0	14.16						
1	13.77	11	10.67	21	8.68	31	7.42
2	13.40	12	10.43	22	8.53	32	7.32
3	13.04	13	10.20	23	8.39	33	7.22
4	12.70	14	9.97	24	8.25	34	7.13
5	12.37	15	9.76	25	8.11	35	7.04
6	12.06	16	9.56	26	7.99	36	6.94
7	11.75	17	9.37	27	7.87	37	6.86
8	11.47	18	9.18	28	7.75	38	6.76
9	11.19	19	9.01	29	7.64	39	6.68
10	10.92	20	8.84	30	7.53	40	6.59

ISO5814

Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)
0	14.62				
1	14.22	11	11.03	21	8.91
2	13.83	12	10.78	22	8.74
3	13.46	13	10.54	23	8.58
4	13.11	14	10.31	24	8.42
5	12.77	15	10.08	25	8.26
6	12.45	16	9.87	26	8.11
7	12.14	17	9.66	27	7.97
8	11.84	18	9.47	28	7.83
9	11.56	19	9.28	29	7.69
10	11.29	20	9.09	30	7.56

● **Span setting values for calibration in air**

The software should display these values when auto calibration is performed.
Use this table to input values for manual span calibrations in air.

Tip

The DO measurement value of "air-saturated water" and air are different.
Due to the pressure difference against the membrane in air versus the membrane in water, the measurement value in air is about 10% higher than the value of air-saturated water on average.

Amounts of saturated dissolved oxygen in air at various temperatures

Following tables are applicable only to the air calibration of the U-50 DO sensor. Do not use them for other purpose.

Air calibration value in adopting evaluation based on JIS K0101

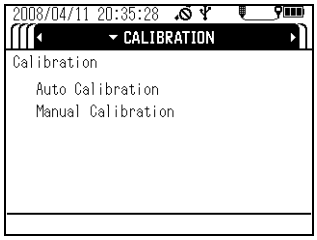
Temp (°C)	DO (mg/L)	Temp (°C)	DO (mg/L)	Temp (°C)	DO (mg/L)	Temp (°C)	DO (mg/L)
0	15.58						
1	15.15	11	11.74	21	9.55	31	8.16
2	14.74	12	11.47	22	9.38	32	8.05
3	14.34	13	11.22	23	9.23	33	7.94
4	13.97	14	10.97	24	9.08	34	7.84
5	13.61	15	10.74	25	8.92	35	7.74
6	13.27	16	10.52	26	8.79	36	7.63
7	12.93	17	10.31	27	8.66	37	7.55
8	12.62	18	10.10	28	8.53	38	7.44
9	12.31	19	9.91	29	8.40	39	7.35
10	12.01	20	9.72	30	8.28	40	7.25

Air calibration value in adopting evaluation based on ISO5814

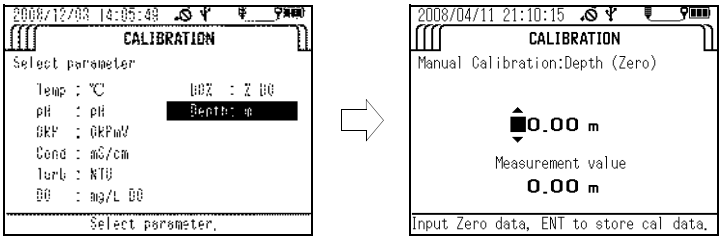
Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)
0	16.08				
1	15.64	11	12.13	21	9.80
2	15.21	12	11.86	22	9.61
3	14.81	13	11.59	23	9.44
4	14.42	14	11.34	24	9.26
5	14.05	15	11.09	25	9.09
6	13.70	16	10.86	26	8.92
7	13.35	17	10.63	27	8.77
8	13.02	18	10.42	28	8.61
9	12.72	19	10.21	29	8.46
10	12.42	20	10.00	30	8.32

● Water depth (DEPTH) calibration

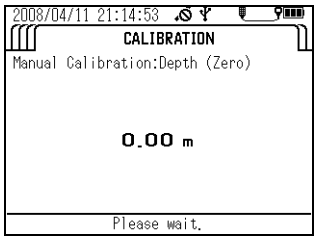
1. **Calibrate the zero point. Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then remove all moisture from the sensor probe (it will be calibrated in air).**
2. **Press the control unit's CAL key to set the calibration mode.**
3. **Press the down (▽) key to move the cursor to "Manual Calibration", then press the ENTER key.**



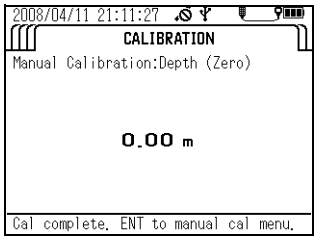
4. **In the parameter selection screen, move the cursor to "Depth", then press the ENTER key.**



5. **Press the up (△) and down (▽) keys to set the "Depth" value to 0.00 m.**
6. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**



7. **Calibration is finished when the message "Cal complete. ENT to manual cal menu." appears. Press the ENTER key to return to the calibration parameter selection screen.**



3.4 Measurement

You can perform measurement by either of the methods below.

- Storing data in memory manually with reference to the measurement value (single measurement)
- Having data stored in memory automatically and continuously
  U-51/U-52: Interval measurement (minimum memory interval of 10 seconds)
  U-53: Interval measurement (minimum memory interval of 30 seconds)

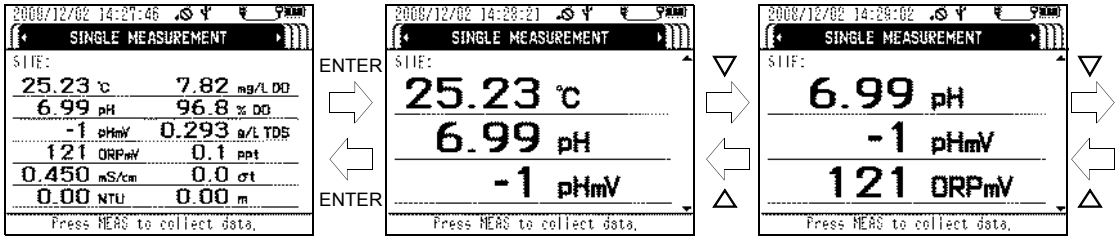
Select the measurement method that meets your requirements.

Note

- **Lower sensor probe slowly when submerging them in samples.**
- **Sensors may break if sensor probe are dropped from a height of 1 meter or more.**
- **Do not submerge sensor probe in water depths of over 30 meters. Sensor probe are only resistant to water pressure of up to 30 meters.**
- **After turning the power ON, check that the DO readout value has stabilized before starting measurement (takes around 20 minutes).**

Tip

- **When on the measurement screen, pressing the ENTER key enlarges the display and shows three measured values at a time.**
- **Pressing the up (△) and down (▽) keys scrolls through the measured values one item at a time.**
- **Pressing the ENTER key again reverts to the normal measurement screen display.**



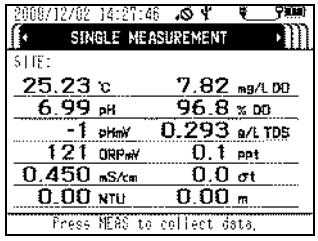
Measurement screen Normal Enlarged

3.4.1 Storing data in memory manually

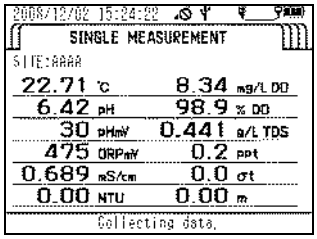
Follow the steps below to manually store data in memory while referring to the measurement value to check the readout value is stable.

● **U-51/U-52**

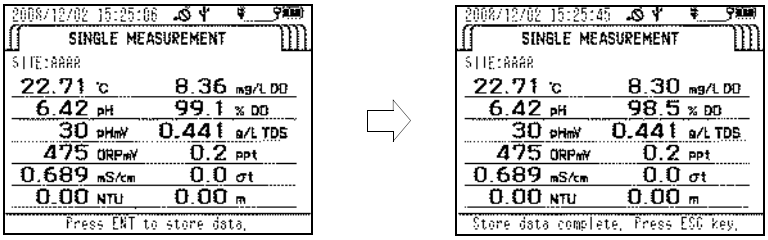
1. **Check that each sensor and sensor guard is mounted.**
2. **Check that "SINGLE MEASUREMENT" has been selected in the measurement screen.**



3. **Submerge the sensor probe in the sample, gently shaking them in the sample to remove any air bubbles from the sensors.**
   If the sample is non-flowing, move the cable slowly up and down (move the sensor probe at a rate of roughly 20 to 30 cm a second) to ensure that fresh sample is continuously supplied to the DO sensor.
4. **When the measurement values are stable, press the MEAS key to acquire the 5-second average.**



5. **Press the ENTER key to save the held measurement values, or press the ESC key to cancel the operation.**

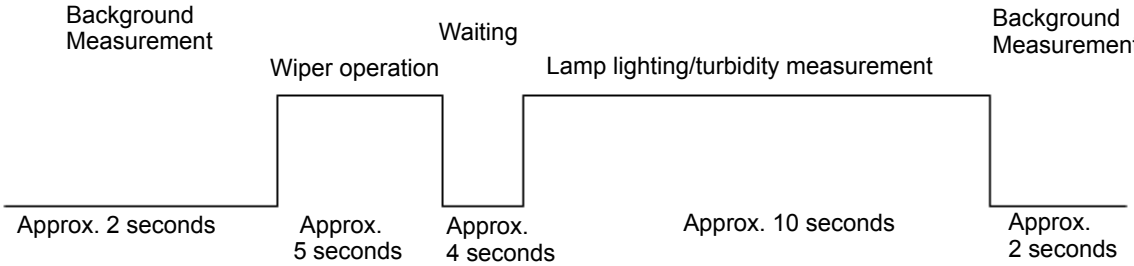


● U-53

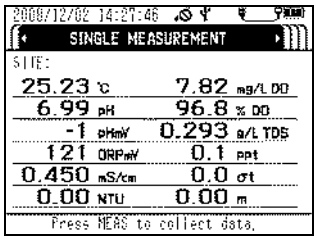
Note

Do not perform turbidity measurement in air as it may damage the wiper.

U-53 turbidity measurement follows the sequence below. The measurement values are held after each sequence.

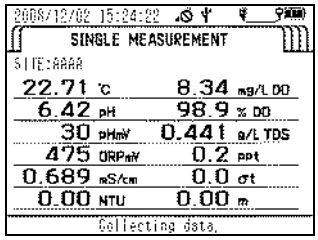


1. **Check that each sensor and sensor guard is mounted.**
2. **Check that "SINGLE MEASUREMENT" has been selected in the measurement screen.**

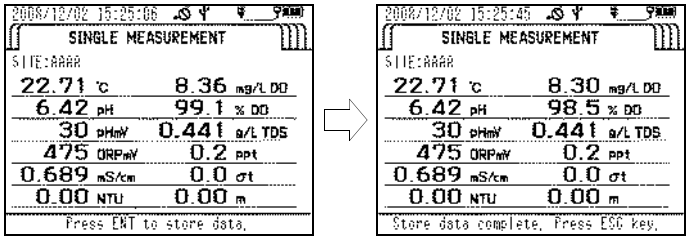


3. **Submerge the sensor probe in the sample, gently shaking them in the sample to remove any air bubbles from the sensors.**
If the sample is non-flowing, move the cable slowly up and down (move the sensor probe at a rate of roughly 20 to 30 cm a second) to ensure that fresh sample is continuously supplied to the DO sensor.

4. **When the non-turbidity meter measurement values are stable, press the MEAS key to start the sequence above.**



5. **When the sequence has finished, hold the measurement values. Press the ENTER key to store the held measurement values, or press the ESC key to cancel the operation.**



3.4.2 Automatic, continuous measurement

● **Interval measurement**

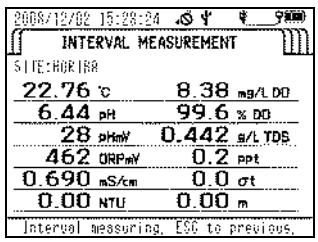
1. **Select the "Interval measurement" measurement setting (see " 3.2.1 Setting measurement methods " (page 18)).**

2. **Press the up (△) and down (▽) keys to set the interval value to the desired value (U-51/U-52: minimum interval: 10 seconds, U-53: minimum interval: 30 seconds), then press the ENTER key.**
The measurement screen appears automatically, and the system becomes ready for measurement.

3. **Check that each sensor and sensor guard is mounted.**

4. **Submerge the sensor probe in the sample, gently shaking them in the sample to remove any air bubbles from the sensors.**
If the sample is non-flowing, move the cable slowly up and down (move the sensor probe at a rate of roughly 20 to 30 cm a second) to ensure that fresh sample is continuously supplied to the DO sensor.

5. **Press the ENTER key to start measurement.**



3.5 Data operations

Use the procedures below to retrieve data stored in memory, delete all the data, check the remaining data memory capacity, and check the calibration record.

3.5.1 Displaying data

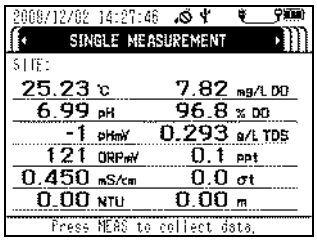
For maximum efficiency, there are 3 methods of displaying data.

- Displaying the data for a specified site
- Displaying the data for a specified date/time
- Displaying all the data

Use the method that best suits your requirements.

● **Displaying the data for a specified site**

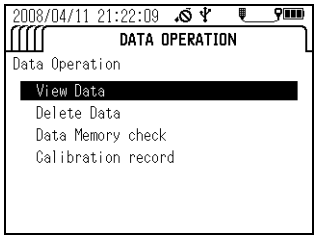
1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
   The "MEASUREMENT" screen appears after about 10 seconds.



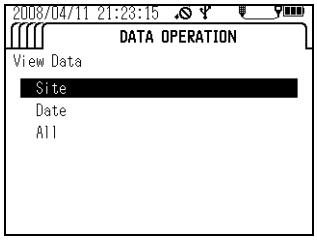
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key 3 times to display the "DATA OPERATION" screen.**
3. **Press the down (▽) key to move the cursor to "View Data", then press the ENTER key.**

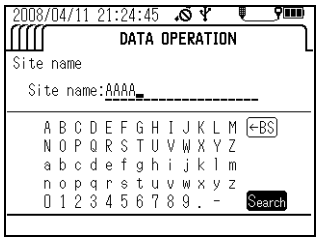


4. **Move the cursor to "Site", then press the ENTER key.**



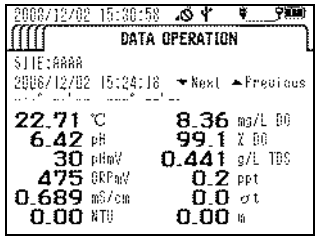
5. **Press the up (△), down (▽), left (◁) and right (▷) keys to enter the site to retrieve.**

6. **Move the cursor to "Search", then press the ENTER key.**



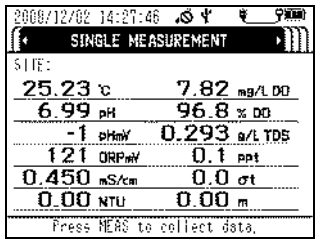
All site names that begin with the entered text are displayed.
The most recently measured data for the entered site is displayed.

7. **Press the up (△) and down (▽) keys to display earlier data.**



● Displaying the data for a specified date/time

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
The "MEASUREMENT" screen appears after about 10 seconds.

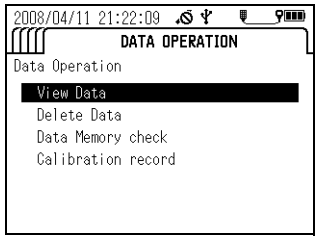


Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

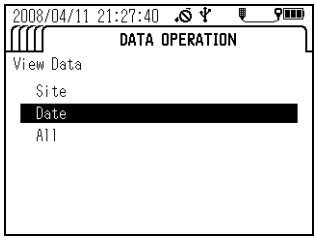
2. **Press the right (▷) key 3 times to display the "DATA OPERATION" screen.**

3. **Press the down (▽) key to move the cursor to "View Data", then press the ENTER key.**



4. **Move the cursor to "Date", then press the ENTER key.**

5. **With the cursor on the Date, press the ENTER key.**



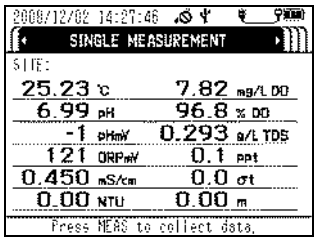
6. **Press the up (△), down (▽), left (◁) and right (▷) keys to enter the desired date/ time, then press the ENTER key to apply the setting.**

7. **The cursor moves to "Search". Press the ENTER key to start the search.**

8. **Press the up (△) and down (▽) keys to display earlier data.**

● **Displaying all the data**

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
The "MEASUREMENT" screen appears after about 10 seconds.

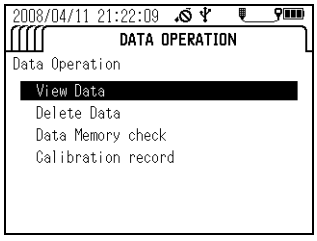


Note

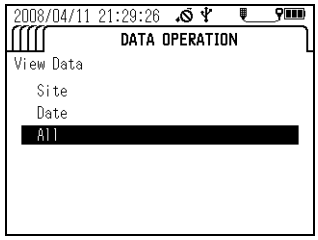
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key 3 times to display the "DATA OPERATION" screen.**

3. **Press the down (▽) key to move the cursor to "View Data", then press the ENTER key.**



4. **Move the cursor to "All", then press the ENTER key.**
The most recently measured data is displayed.

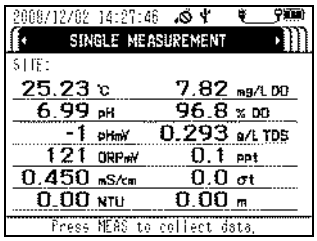


5. **Press the up (△) and down (▽) keys to display earlier data.**

3.5.2 Deleting data

Follow the steps below to delete all the data stored in memory.

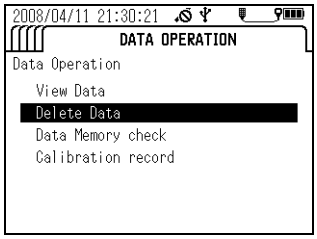
1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
   The "MEASUREMENT" screen appears after about 10 seconds.



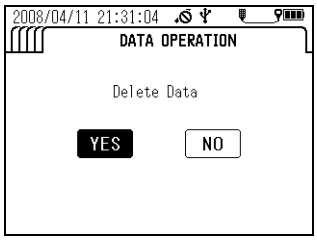
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key 3 times to display the "DATA OPERATION" screen.**
3. **Press the down (▽) key to move the cursor to "Delete Data", then press the ENTER key.**



4. **Press the left (◁) key to move the cursor to YES, then press the ENTER key.**
   All the data has been deleted when the indicator appears along with the message "No data exists".

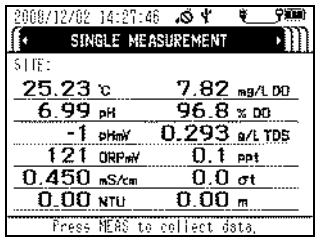


3.5.3 Checking the data memory

You can check the used data capacity and the remaining data capacity.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

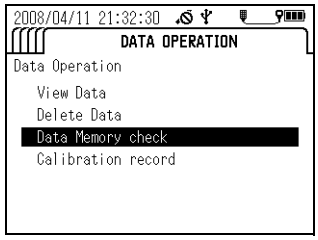


Note

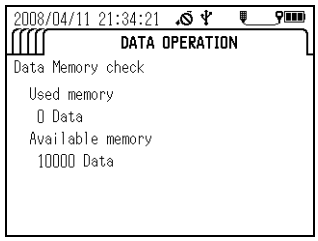
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key 3 times to display the "DATA OPERATION" screen.**

3. **Press the down (▽) key to move the cursor to "Data Memory Check", then press the ENTER key.**



The amount of memory in use and amount of available memory are displayed.

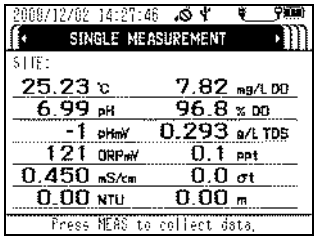


3.5.4 Checking the calibration record

Follow the steps below to check the latest calibration history.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.



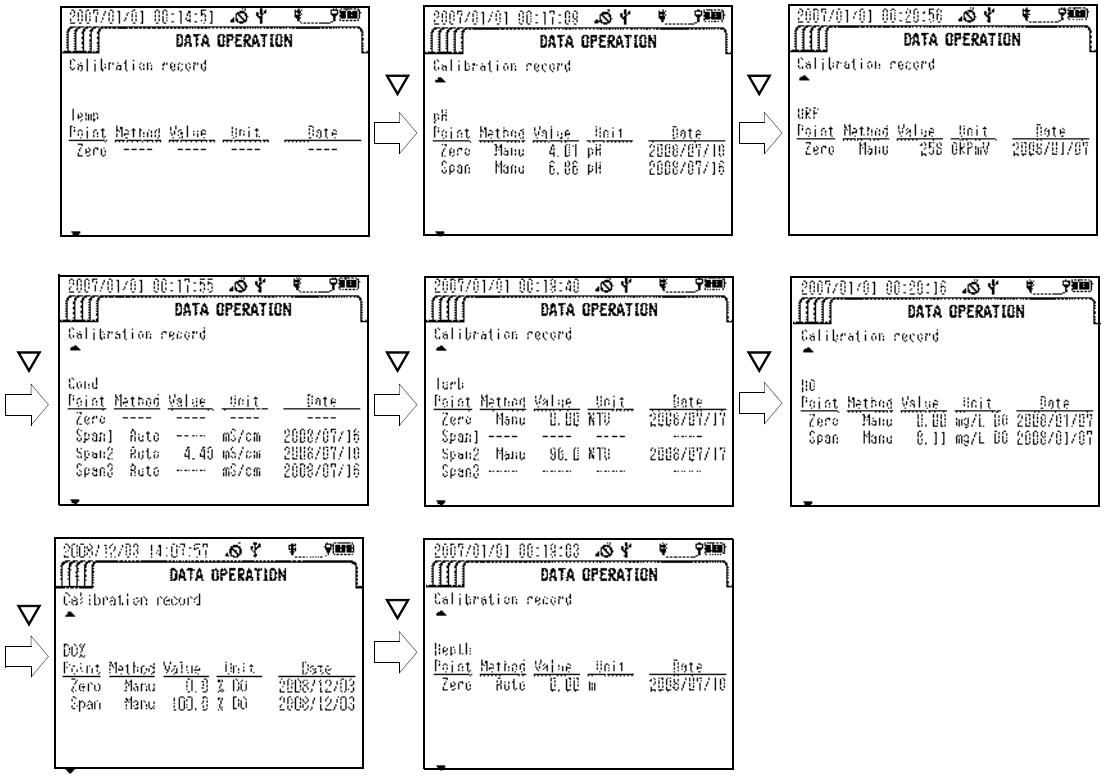
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key 3 times to display the "DATA OPERATION" screen.**

3. **Press the down (▽) key to move the cursor to "Calibration record", then press the ENTER key.**

The latest calibration record is displayed.



3.5.5 GPS data operations

The menu for GPS data operations appears on the display to which the GPS unit is mounted.

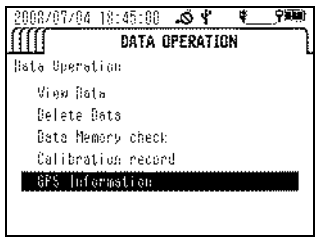
● GPS information

Follow the steps below to display acquired GPS information.

Note

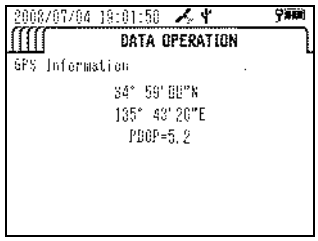
Turning the power OFF erases the GPS information.

1. **Press the right (▷) key to switch the display to the "DATA OPERATION" screen.**
2. **the down (▽) key to move the cursor to "GPS Information", then press the ENTER key.**

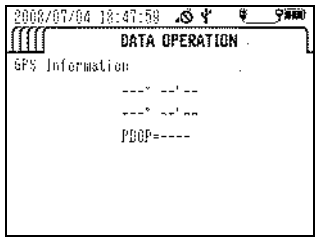


The last GPS information acquired is displayed.

● When received data exists

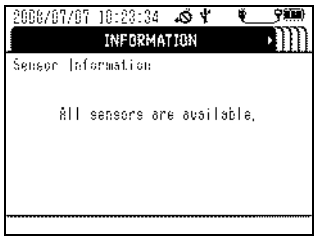


● When no received data exists



3.6 Sensor information

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
   The "MEASUREMENT" screen appears after about 10 seconds.
2. **Press the left (◁) key once to display the "INFORMATION" screen.**
   The "Sensor Information" screen displays the sensor probe's status.
   - When the sensor probe is normal, the display below appears.

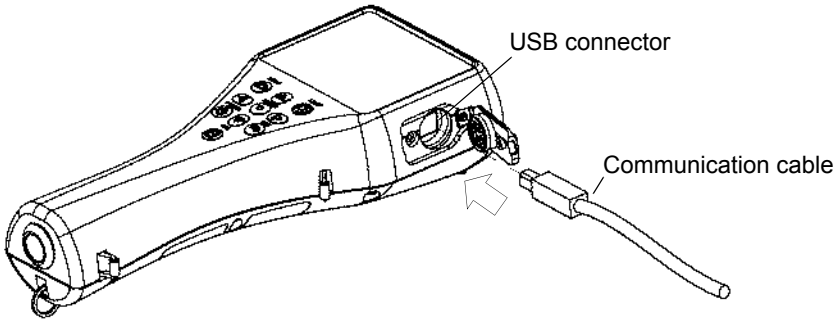


   - When there is a sensor probe problem, individual measurement parameters generate messages such as the one shown below. Follow the troubleshooting information to remove the problem before continuing to operate the system.

3.7 USB communication

The separately-sold, dedicated PC connection cable comes with data collection software. This software allows data to be downloaded from the control unit in CSV format.
This section contains instructions for communication commands used for USB communication.

● Connecting the cable



Dedicated cable
Part name: Communication cable (with data collection software)
Part no.: 3200174823

● Cautions when using USB communication

Take care to observe the following when using USB communication.

- Use the dedicated cable (with data collection software) or a commercially-available USB cable (A-B type) to connect to a PC.
- Be sure to match the transmission format on the control unit and the computer.
  The control unit uses the following transmission format:

Baud rate:	19200 bps
Number of stop bits:	1 bit
Data bit length:	8 bits
Parity:	None
Flow control:	None

Tip

If the transmission formats do not match, a communication error occurs and USB communication will not function normally. After changing the transmission format, restart the control unit and the computer.

- If received data is not sent back or an error occurs after a data request has been sent, adjust the program configuration so that it allows a little waiting time before a data request is sent again. This will enable more stable communication.
- The unit does not use DCD, CTS, or DSR signals. Take care of this when creating programs.

3.7.1 Communication settings

Baud rate:	19200 bps
Number of stop bits:	1 bit
Data bit length:	8 bits
Parity:	None
Flow control:	None

3.7.2 Commands

● Instant data requests

● Request command format

#	RD	@	XX	[CR]	[LF]
1	2	3	4		

1	Header	1 character
2	Command	2 characters
3	Delimiter character	1 character
4	Frame check sequence (FCS)	2 characters

The two ASCII-code characters created by converting the 8 bits of data created by successively combining the value of each character from # through @ in an exclusive OR (XOR) operation with the value of the next character.

Example: #RD@

(1)	0	XOR	35	(ASCII code of # symbol)	⇒	35
(2)	35	XOR	82	(ASCII code of R)	⇒	113
(3)	113	XOR	68	(ASCII code of D)	⇒	53
(4)	53	XOR	64	(ASCII code of @ symbol)	⇒	117 (decimal) ↓ 75 (hex) ↓ Sets "75".

Example: 35 XOR 82 operation

35 in binary	⇒	0	0	1	0	0	0	1	1	
82 in binary	⇒	0	1	0	1	0	0	1	0	
XOR result		0	1	1	1	0	0	0	1	⇒ 113 (decimal)

Note: Set "XX" if you do not want to test for communication frame errors with FCS.

● Response format

#	RD	AAAAAAAAAAAAAAAAAAAA	X	X	XXXX	XX	X	X	XXXXX	X
1	2	3	4	5	6	7	8	9	10	11

XX	X	X	XXXXX	X	XX	X	X	XXXXX	X	XX	X	X	XXXXX	X
12	13	14	15	16	17	18	19	20	21	22	23	24	25	26

XX	X	X	XXXXX	X	XX	X	X	XXXXX	X	XX	X	X	XXXXX	X
27	28	29	30	31	32	33	34	35	36	37	38	39	40	41

XX	X	X	XXXXX	X	XX	X	X	XXXXX	X	XX	X	X	XXXXX	X
42	43	44	45	46	47	48	49	50	51	52	53	54	55	56

```
XX  X  X  XXXXX  X  XX  X  X  XXXXX  X  XX  X  X  XXXXX  X
57  58 59 60      61 62  63 64 65      66 67  68 69 70      71

XX XX XX XX XX XX XX XX XX X  X  XXX XX XX X  X  @  XX [CR] [LF]
72 73 74 75 76 77 78 79 80 81 82 83  84 85 86 87 88 89
```

1	Header		1 character
2	Command		2 characters
3	Site name	Upper- and lowercase letters, numbers, periods (.) hyphens (-) and spaces ( )	20 characters
4	Probe status	(3) Status code	1 character
5	Probe error	(4) Status error code	1 character
6	Unused		4 characters
7	Parameter 1 code	(1) Parameter code	2 characters
8	Parameter 1 status	(5) Parameter status code	1 character
9	Parameter 1 error	(6) Parameter error code	1 character
10	Parameter 1 data	5 characters including decimal point, right-justified with blanks filled	5 characters
11	Parameter 1 unit	(2) Unit code	1 character
12	Parameter 2 code	(1) Parameter code	2 characters
13	Parameter 2 status	(5) Parameter status code	1 character
14	Parameter 2 error	(6) Parameter error code	1 character
15	Parameter 2 data	5 characters including decimal point, right-justified with blanks filled	5 characters
16	Parameter 2 unit	(2) Unit code	1 character
17	Parameter 3 code	(1) Parameter code	2 characters
18	Parameter 3 status	(5) Parameter status code	1 character
19	Parameter 3 error	(6) Parameter error code	1 character
20	Parameter 3 data	5 characters including decimal point, right-justified with blanks filled	5 characters
21	Parameter 3 unit	(2) Unit code	1 character
22	Parameter 4 code	(1) Parameter code	2 characters
23	Parameter 4 status	(5) Parameter status code	1 character
24	Parameter 4 error	(6) Parameter error code	1 character
25	Parameter 4 data	5 characters including decimal point, right-justified with blanks filled	5 characters
26	Parameter 4 unit	(2) Unit code	1 character
27	Parameter 5 code	(1) Parameter code	2 characters
28	Parameter 5 status	(5) Parameter status code	1 character
29	Parameter 5 error	(6) Parameter error code	1 character
30	Parameter 5 data	5 characters including decimal point, right-justified with blanks filled	5 characters
31	Parameter 5 unit	(2) Unit code	1 character
32	Parameter 6 code	(1) Parameter code	2 characters
33	Parameter 6 status	(5) Parameter status code	1 character
34	Parameter 6 error	(6) Parameter error code	1 character

35	Parameter 6 data	5 characters including decimal point, right-justified with blanks filled	5 characters
36	Parameter 6 unit	(2) Unit code	1 character
37	Parameter 7 code	(1) Parameter code	2 characters
38	Parameter 7 status	(5) Parameter status code	1 character
39	Parameter 7 error	(6) Parameter error code	1 character
40	Parameter 7 data	5 characters including decimal point, right-justified with blanks filled	5 characters
41	Parameter 7 unit	(2) Unit code	1 character
42	Parameter 8 code	(1) Parameter code	2 characters
43	Parameter 8 status	(5) Parameter status code	1 character
44	Parameter 8 error	(6) Parameter error code	1 character
45	Parameter 8 data	5 characters including decimal point, right-justified with blanks filled	5 characters
46	Parameter 8 unit	(2) Unit code	1 character
47	Parameter 9 code	(1) Parameter code	2 characters
48	Parameter 9 status	(5) Parameter status code	1 character
49	Parameter 9 error	(6) Parameter error code	1 character
50	Parameter 9 data	5 characters including decimal point, right-justified with blanks filled	5 characters
51	Parameter 9 unit	(2) Unit code	1 character
52	Parameter 10 code	(1) Parameter code	2 characters
53	Parameter 10 status	(5) Parameter status code	1 character
54	Parameter 10 error	(6) Parameter error code	1 character
55	Parameter 10 data	5 characters including decimal point, right-justified with blanks filled	5 characters
56	Parameter 10 unit	(2) Unit code	1 character
57	Parameter 11 code	(1) Parameter code	2 characters
58	Parameter 11 status	(5) Parameter status code	1 character
59	Parameter 11 error	(6) Parameter error code	1 character
60	Parameter 11 data	5 characters including decimal point, right-justified with blanks filled	5 characters
61	Parameter 11 unit	(2) Unit code	1 character
62	Parameter 12 code	(1) Parameter code	2 characters
63	Parameter 12 status	(5) Parameter status code	1 character
64	Parameter 12 error	(6) Parameter error code	1 character
65	Parameter 12 data	5 characters including decimal point, right-justified with blanks filled	5 characters
66	Parameter 12 unit	(2) Unit code (6) Parameter error code	1 character
67	Parameter 13 code	(1) Parameter code	2 characters
68	Parameter 13 status	(5) Parameter status code	1 character
69	Parameter 13 error	(6) Parameter error code	1 character
70	Parameter 13 data	5 characters including decimal point, right-justified with blanks filled	5 characters
71	Parameter 13 unit	(2) Unit code	1 character
72	Year	00 to 99	2 characters

73	Month	01 to 12	2 characters
74	Day	01 to 31	2 characters
75	Hour	00 to 23	2 characters
76	Minute	00 to 59	2 characters
77	Second	00 to 59	2 characters
78	Longitude (degrees)	00 to 90 or "--" (no GPS data)	2 characters
79	Longitude (minutes)	00 to 59 or "--" (no GPS data)	2 characters
80	Longitude (seconds)	00 to 59 or "--" (no GPS data)	2 characters
81	Unused	1 character	1 character
82	North latitude/South latitude	N: North; S: South	1 character
83	Latitude (degrees)	000 to 180 or "---" (no GPS data)	3 characters
84	Latitude (minutes)	00 to 59 or "--" (no GPS data)	2 characters
85	Latitude (seconds)	00 to 59 or "--" (no GPS data)	2 characters
86	Unused		1 character
87	East longitude/West longitude	E: East; W: West	1 character
88	Delimiter character		1 character
89	Frame check sequence (FCS)		2 characters

● Memory data requests

● Request command format

#	RM	X	X	AAAAAAAAAAAAAAAAAAAA	XX	XX	XX	@	XX	[CR]	[LF]
1	2	3	4	5	6	7	8	9	10		

1	Header		1 character
2	Command		2 characters
3	Data specification[*1]	0: Start search; 1: Next data item; 2: Previous data item; 3: Request same data again	1 character
4	Search method specification	0: All data; 1: Site search; 2: Date search	1 character
5	Search site[*2]	Upper- and lowercase letters, numbers, periods (.) hyphens (-) and spaces ( )	20 characters
6	Search year[*3]	00 to 99	2 characters
7	Search month[*3]	01 to 12	2 characters
8	Search day[*3]	01 to 31	2 characters
9	Delimiter character		1 character
10	Frame check sequence (FCS)		2 characters

*1: When sending the RM command, first send 0 [Start search], then 1 [Next data item], 2 [Previous data item] or 3 [Request same data again].

*2: [Search site] is only needed when [Site search] is specified as the search method. If another search method is specified, fill this field with spaces.

*3: [Search year], [Search month] and [Search day] are only needed when [Date search] is specified as the search method. If another search method is specified, fill this field with spaces.

● **Response format**

(when data exists)

```
#    RM  AAAAAAAAAAAAAAAAAAAA   XX  X    X    XXXXX   X
1    2   3                      4   5    6    7       8

XX  X   X   XXXXX  X   XX  X   X   XXXXX  X   XX  X   X   XXXXX  X
9   10  11  12      13  14  15  16  17      18  19  20  21  22      23

XX  X   X   XXXXX  X   XX  X   X   XXXXX  X   XX  X   X   XXXXX  X
24  25  26  27      28  29  30  31  32      33  34  35  36  37      38

XX  X   X   XXXXX  X   XX  X   X   XXXXX  X   XX  X   X   XXXXX  X
39  40  41  42      43  44  45  46  47      48  49  50  51  52      53

XX  X   X   XXXXX  X   XX  X   X   XXXXX  X   XX  X   X   XXXXX  X
54  55  56  57      58  59  60  61  62      63  64  65  66  67      68

XX  XX  XX  XX  XX  XX  XX  XX  XX  X   X   XXX  XX  XX  X   X   @   XX  [CR]  [LF]
69  70  71  72  73  74  75  76  77  78  79  80   81  82  83  84  85  86
```

1	Header		1 character
2	Command		2 characters
3	Site name	Upper- and lowercase letters, numbers, periods (.) hyphens (-) and spaces ( )	20 characters
4	Parameter 1 code	(1) Parameter code	2 characters
5	Parameter 1 selection	0: No selection; 1: Selection made	1 character
6	Parameter 1 error	(6) Parameter error code	1 character
7	Parameter 1 data	5 characters including decimal point, right-justified with blanks filled	5 characters
8	Parameter 1 unit	(2) Unit code	1 character
9	Parameter 2 code	(1) Parameter code	2 characters
10	Parameter 2 selection	0: No selection; 1: Selection made	1 character
11	Parameter 2 error	(6) Parameter error code	1 character
12	Parameter 2 data	5 characters including decimal point, right-justified with blanks filled	5 characters
13	Parameter 2 unit	(2) Unit code	1 character
14	Parameter 3 code	(1) Parameter code	2 characters
15	Parameter 3 selection	0: No selection; 1: Selection made	1 character
16	Parameter 3 error	(6) Parameter error code	1 character
17	Parameter 3 data	5 characters including decimal point, right-justified with blanks filled	5 characters
18	Parameter 3 unit	(2) Unit code	1 character
19	Parameter 4 code	(1) Parameter code	2 characters
20	Parameter 4 selection	0: No selection; 1: Selection made	1 character

21	Parameter 4 error	(6) Parameter error code	1 character
22	Parameter 4 data	5 characters including decimal point, right-justified with blanks filled	5 characters
23	Parameter 4 unit	(2) Unit code	1 character
24	Parameter 5 code	(1) Parameter code	2 characters
25	Parameter 5 selection	0: No selection; 1: Selection made	1 character
26	Parameter 5 error	(6) Parameter error code	1 character
27	Parameter 5 data	5 characters including decimal point, right-justified with blanks filled	5 characters
28	Parameter 5 unit	(2) Unit code	1 character
29	Parameter 6 code	(1) Parameter code	2 characters
30	Parameter 6 selection	0: No selection; 1: Selection made	1 character
31	Parameter 6 error	(6) Parameter error code	1 character
32	Parameter 6 data	5 characters including decimal point, right-justified with blanks filled	5 characters
33	Parameter 6 unit	(2) Unit code	1 character
34	Parameter 7 code	(1) Parameter code	2 characters
35	Parameter 7 selection	0: No selection; 1: Selection made	1 character
36	Parameter 7 error	(6) Parameter error code	1 character
37	Parameter 7 data	5 characters including decimal point, right-justified with blanks filled	5 characters
38	Parameter 7 unit	(2) Unit code	1 character
39	Parameter 8 code	(1) Parameter code	2 characters
40	Parameter 8 selection	0: No selection; 1: Selection made	1 character
41	Parameter 8 error	(6) Parameter error code	1 character
42	Parameter 8 data	5 characters including decimal point, right-justified with blanks filled	5 characters
43	Parameter 8 unit	(2) Unit code	1 character
44	Parameter 9 code	(1) Parameter code	2 characters
45	Parameter 9 selection	0: No selection; 1: Selection made	1 character
46	Parameter 9 error	(6) Parameter error code	1 character
47	Parameter 9 data	5 characters including decimal point, right-justified with blanks filled	5 characters
48	Parameter 9 unit	(2) Unit code	1 character
49	Parameter 10 code	(1) Parameter code	2 characters
50	Parameter 10 selection	0: No selection; 1: Selection made	1 character
51	Parameter 10 error	(6) Parameter error code	1 character
52	Parameter 10 data	5 characters including decimal point, right-justified with blanks filled	5 characters
53	Parameter 10 unit	(2) Unit code	1 character
54	Parameter 11 code	(1) Parameter code	2 characters
55	Parameter 11 selection	0: No selection; 1: Selection made	1 character
56	Parameter 11 error	(6) Parameter error code	1 character
57	Parameter 11 data	5 characters including decimal point, right-justified with blanks filled	5 characters
58	Parameter 11 unit	(2) Unit code	1 character
59	Parameter 12 code	(1) Parameter code	2 characters

60	Parameter 12 selection	0: No selection; 1: Selection made	1 character
61	Parameter 12 error	(6) Parameter error code	1 character
62	Parameter 12 data	5 characters including decimal point, right-justified with blanks filled	5 characters
63	Parameter 12 unit	(2) Unit code	1 character
64	Parameter 13 code	(1) Parameter code	2 characters
65	Parameter 13 selection	0: No selection; 1: Selection made	1 character
66	Parameter 13 error	(6) Parameter error code	1 character
67	Parameter 13 data	5 characters including decimal point, right-justified with blanks filled	5 characters
68	Parameter 13 unit	(2) Unit code	1 character
69	Year	00 to 99	2 characters
70	Month	01 to 12	2 characters
71	Day	01 to 31	2 characters
72	Hour	00 to 23	2 characters
73	Minute	00 to 59	2 characters
74	Second	00 to 5	2 characters
75	Longitude (degrees)	00 to 90 or "--" (no GPS data)	2 characters
76	Longitude (minutes)	00 to 59 or "--" (no GPS data)	2 characters
77	Longitude (seconds)	00 to 59 or "--" (no GPS data)	2 characters
78	Unused		1 character
79	North latitude/South latitude	N: North; S: South	1 character
80	Latitude (degrees)	000 to 180 or "---" (no GPS data)	3 characters
81	Latitude (minutes)	00 to 59 or "--" (no GPS data)	2 characters
82	Latitude (seconds)	00 to 59 or "--" (no GPS data)	2 characters
83	Unused		1 character
84	East longitude/West longitude	E: East; W: West	1 character
85	Delimiter character		1 character
86	Frame check sequence (FCS)		2 characters

When no data exists, or memory is at capacity)

#	RM	@	XX	[CR]	[LF]
1	2	3	4		

1	Header	1 character
2	Command	2 characters
3	Delimiter character\	1 character
4	Frame check sequence (FCS)	2 characters

● Memory data count request

● Request command format

#	RN	@	XX	[CR]	[LF]
1	2	3	4		

1	Header	1 character
2	Command	2 characters
3	Delimiter character\	1 character
4	Frame check sequence (FCS)	2 characters

● Response format

#	RN	XXXXX	@	XX	[CR]	[LF]
1	2	3	4	5		

1	Header		1 character
2	Command		2 characters
3	Total data count	0 to 10000	5 characters
4	Delimiter character\		1 character
5	Frame check sequence (FCS)		2 characters

● Command parse failure response

#	??	X	XX	X	@	XX	[CR]	[LF]
1	2	3	4	5	6	7		

1	Header	1 character
2	Command	2 characters
3	Command parse failure reason*4	1 character
4	Received command*5	2 characters
5	(3) Status code for probe status*5	1 character
6	Delimiter character	1 character
7	Frame check sequence (FCS)	2 characters

*4: List of command parse failure reasons

1: Frame length error
2: FCS mismatch
3: Undefined command
4: Data error
5: Data out of range
6: No "@" delimiter character
7: No "#" header character
8: No [Carriage return] + [Line feed] footer
9: Cannot accept command in this timing.

*5: Only set for command parse failure reason 9, [Cannot accept command in this timing]. Otherwise this field is filled with spaces.

4 Maintenance

Tip

HORIBA recommends regular manufacturer maintenance checks in order to ensure a long product life.

4.1 Routine care

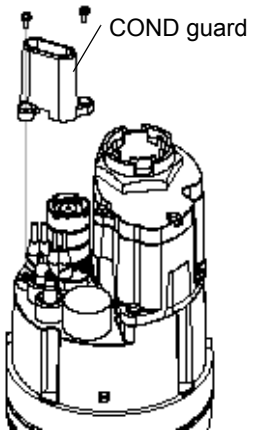
● **After measurement**

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power OFF.**

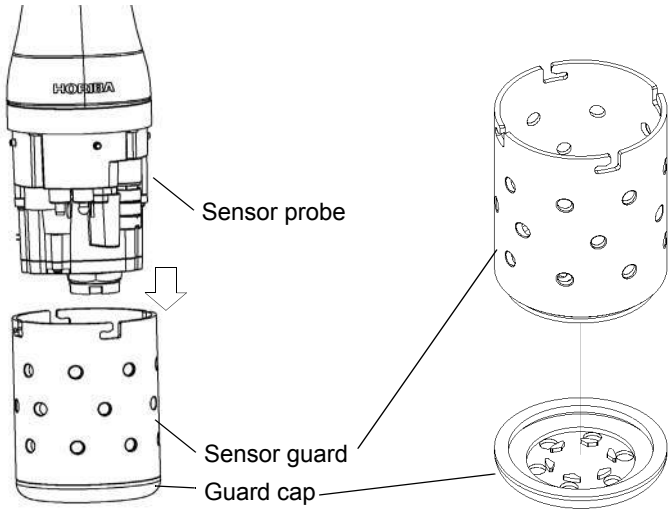
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Remove the sensor guard, and clean the sensor with tap water.**
3. **Clean the turbidity sensor with the cleaning brush provided.**
4. **Remove the two screws securing the COND guard, and the COND guard itself, and use a test tube brush to gently remove any dirt from the electrical conductivity electrode.**



5. **Wipe off any dirt with a soft cloth. If parts are very dirty, clean them with neutral detergent, then rinse them. If parts are contaminated by oil, wipe it off with a soft cloth soaked in alcohol.**
6. **Put the COND guard back in place.**
7. **Remove the sensor guard's guard cap, wash off any dirt with tap water, then put the guard cap back in place.**



4.2 Every 2 months maintenance

● Dissolved oxygen (DO) sensor

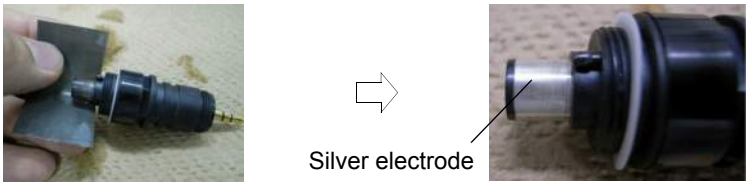
Note

- **The DO sensor's internal solution is potassium chloride (KCl). Although KCl is harmless, protective equipment such as gloves and goggles should be worn when working with it.**
- **Internal solution can be disposed of down a sink.**

- Replace the membrane cap.
- Polish the gold and silver electrodes when replacing the membrane cap.
  The gold electrode does not need to be polished if it is not dirty.

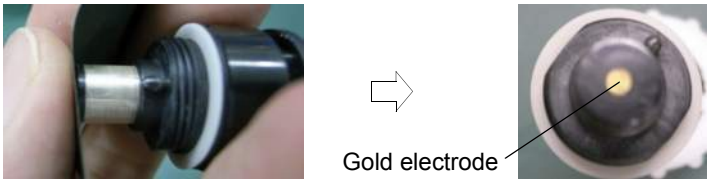
● **Silver electrode**

Polish a silver electrode part with sandpaper (#500) and then wash metal electrode parts with water.



● **Gold electrode**

Polish a gold electrode part with sandpaper (#8000) and then wash metal electrode parts with water.



Replace a membrane cap after clean metal electrodes parts.
Refer to “ 4.5 Replacing the membrane cap ” (page 87).

● **Reference electrode**

Note

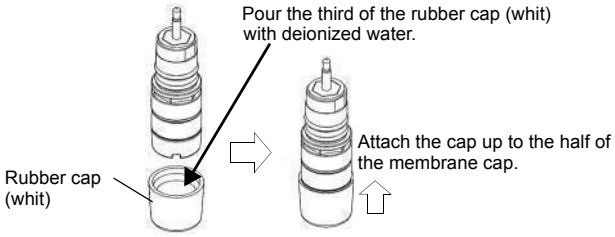
- **The pH reference internal solution is potassium chloride (KCl). Although KCl is harmless, protective equipment such as gloves and goggles should be worn when working with it.**
- **Internal solution can be disposed of down a sink.**

1. **Remove the rubber liquid junction plug from the reference electrode and dispose of the internal solution.**
2. **To prevent air entering, fill the reference electrode to the brim with its internal solution (No. 330).**
3. **Put the rubber liquid junction plug back in place.**
   If the rubber liquid junction plug is dirty, replace the liquid junctions (set of two; No. 9037005100). The reference electrode's internal solution will spill when replacing the liquid junctions. Rinse parts with tap water and dry them with a soft cloth.

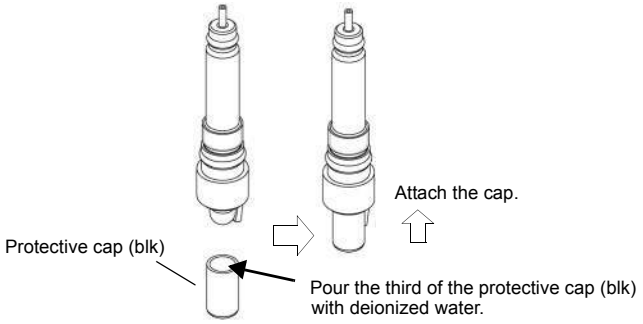
4.3 Storage

● Short-term (under 2 months) storage

- Before storing the DO sensor, pour the third of the rubber cap (whit) provided with deionized water and cover the DO sensor with them.



- Before storing the pH sensor, pour the third of the protective cap (blk) provided with deionized water and cover the pH sensor with them.



Note

Before measuerment, remove the rubber cap (whit) and the protective cap (blk).

● Long-term (2 months or more) storage

- Remove a membrane cap from DO sensor, and wash the gold electrode and silver electrode parts with water. Wipe off the moisture before storing DO sensor in the pack.
- Prevent internal solution seeping out of the reference chip by taping over the point of seepage with electrical tape.
- Before storing the system, remove the control unit's batteries to prevent battery leakage.

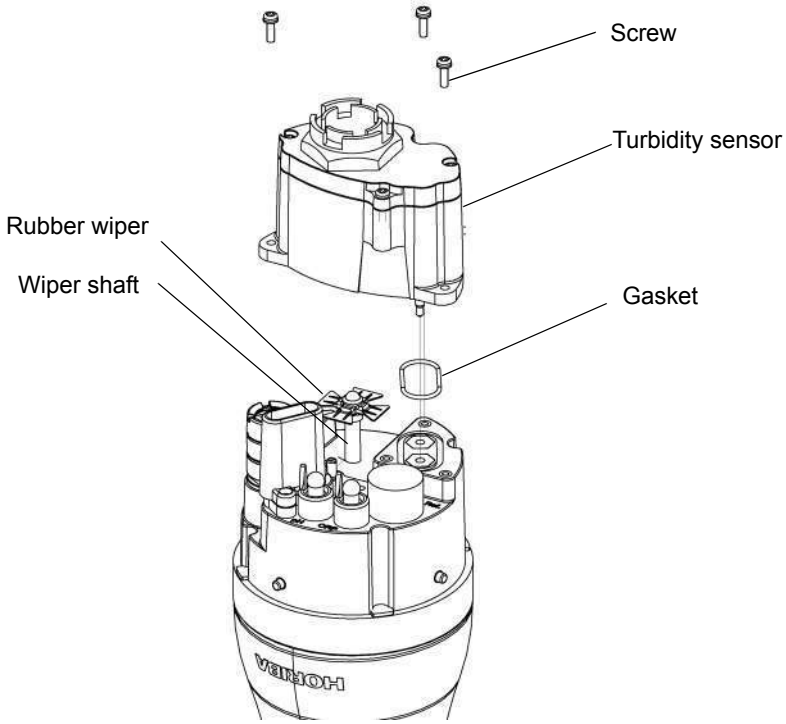
4.4 Replacing the turbidity sensor

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power OFF.**

Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Remove the sensor guard, and clean the sensor probe with tap water.**
3. **Use dry air to blow away and dry off any moisture.**
4. **Remove the three screws holding the turbidity sensor by using No. 2 Phillips head screwdriver.**
5. **Pull out the turbidity sensor horizontally.**
6. **Remove the rubber wiper and gasket, and use a soft cloth to wipe off any dirt from the wiper shaft and turbidity sensor attachment. If parts are very dirty, use a soft cloth soaked in neutral detergent or alcohol.**
7. **Replace the rubber wiper and gasket with new ones. Coat the gasket with a thin layer of grease (No. 3014017718).**
8. **Attach the new turbidity sensor and fasten it in place with the three screws.**
9. **Perform four-point calibration before using the sensor.**



4.5 Replacing the membrane cap

● Replacement procedure

1. **Prepare the DO sensor.**

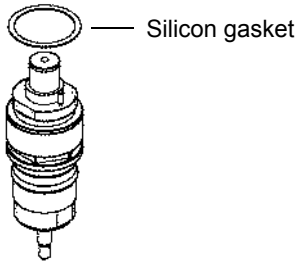
- Take a DO sensor out of pack (newly purchasing).
- Remove a DO sensor from the sensor probe (after use).

Newly purchasing

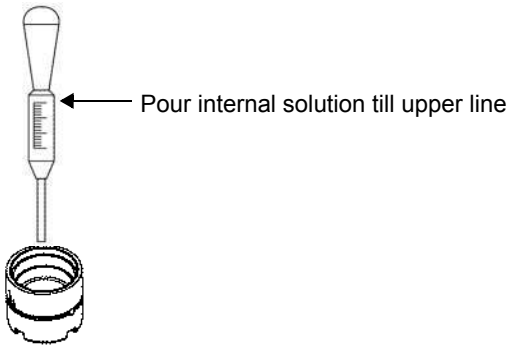
Undo a DO sensor from the sensor probe

- Twist a membrane cap from DO sensor.
- Wash the gold electrode and silver electrode parts with water.

2. **Replace the silicone gasket with a new one.**



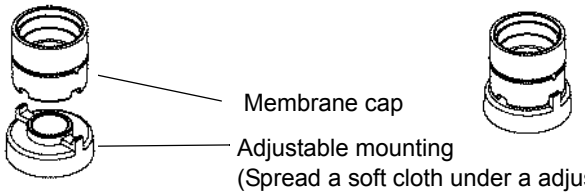
3. **Pour internal solution into a membrane cap with a dropper.**



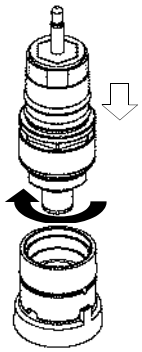
- Check air bubbles in a membrane cap.

Pick a Cap up and drop it down, if there is air bubbles in internal solution of it.

4. **Set up a membrane cap on a adjustable mounting.**



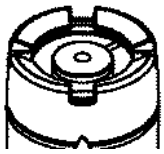
5. **Attach a membrane cap to DO sensor**



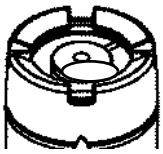
Twist a DO sensor
with holding a membrane cap tight.

6. **Check for membrane surface**

Check air bubbles in a membrane cap.



Good: Limited air bubbles



NG: Air bubbles of more than 5 mm in diameter

- NG → Replace a membrane cap again.
- Check that span calibration can be performed.

If the membrane cap is not attached correctly, sensitivity may be lost or response speed may decrease.

4.6 Troubleshooting

Note

If the sensor probe is removed while the control unit is indicating an error, errors cannot be canceled by using the ESC key. Either reconnect the sensor probe or restart the control unit.

4.6.1 Error displays

Error	Cause	Solution
Probe ADC error	Internal IC failure	Contact your nearest sales outlet to have the sensor probe repaired.
Probe EEPROM error/Factory	Internal IC failure	Turn the power OFF, then restart the system. If the error persists, initialize the system from the "System" menu. If the error still persists, contact your nearest sales outlet to have the sensor probe repaired.
Probe EEPROM error/User	Internal IC failure	Turn the power OFF, then restart the system. If the error persists, initialize the system from the "System" menu. If the error still persists, contact your nearest sales outlet to have the sensor probe repaired.
Turbidity sensor light source error	Turbidity sensor light source failure	Turn the power OFF, wipe off any water droplets on the probe, then remove the turbidity sensor. Check there are no water droplets around the turbidity sensor connector, then mount the sensor again. If the error persists, replace the turbidity sensor.
Turbidity sensor wiper motor error	The turbidity sensor wiper is not operating.	Press the ESC key. Check there are no obstacles near the wiper, then perform the measurement again. If the error persists, the motor will need to be replaced. Contact your nearest sales outlet to have the sensor probe repaired.
Probe capacitor error	Low battery voltage or internal IC failure	Turn the power OFF. Replace the display's batteries. If the error persists, contact your nearest sales outlet to have the sensor probe repaired.
Probe EEPROM error	Internal IC failure	Press the ESC key, then redo the operation. If the error persists, turn the power OFF, then restart the system (the current data will not be saved). If the error still persists, contact your nearest sales outlet to have the display repaired.
Probe board error	Probe board failure	Turn the power OFF. Contact your nearest sales outlet to have the sensor probe repaired.

Error	Cause	Solution
Zero-point calibration error	pH sensor 1. The pH standard solution is contaminated. 2. The pH-responsive membrane is dirty. 3. The concentration of the reference electrode's internal solution has changed. 4. The pH-responsive membrane is torn.	pH sensor 1. Replace the standard solution with new solution. 2. Clean the pH-responsive membrane. 3. Refil the reference electrode's internal solution. 4. Replace the sensor.
	COND sensor 1. There is moisture on the sensor. 2. The sensor is dirty. 3. The COND sensor is broken.	COND sensor 1. Blow-dry the moisture off the sensor. 2. Clean the sensor. 3. Contact your nearest sales outlet.
	TURB sensor 1. There are air bubbles on the cell. 2. The cell window is dirty. 3. The sensor is being affected by ambient light. 4. The solution is dirty. 5. The TURB sensor has failed.	TURB sensor 1. Shake the sensor probe vigorously. 2. Clean the cell window. 3. Calibrate using the calibration cup provided. 4. Replace the solution with new solution. 5. Replace the TURB sensor.
	DO sensor 1. There are air bubbles in the internal solution. 2. The DO sensor has failed.	DO sensor 1. Replace the diaphragm with a new one, and fill the DO sensor with new internal solution. 2. Replace the DO sensor.
	Water depth sensor 1. The water depth sensor is dirty. 2. The water depth sensor has failed.	Water depth sensor 1. Clean the water depth sensor. 2. Contact your nearest sales outlet.

Error	Cause	Solution
Span calibration error	pH sensor 1. The pH standard solution is contaminated. 2. The pH-responsive membrane is dirty. 3. The concentration of the reference electrode's internal solution has changed. 4. The pH-responsive membrane is torn.	pH sensor 1. Replace the standard solution with new solution. 2. Clean the pH-responsive membrane. 3. Refil the reference electrode's internal solution. 4. Replace the sensor.
	ORP sensor 1. The ORP standard solution is contaminated. 2. The ORP electrode is dirty. 3. The concentration of the reference electrode's internal solution has changed. 4. The ORP electrode has failed.	ORP sensor 1. Replace the standard solution with new solution. 2. Clean the ORP electrode. 3. Refil the reference electrode's internal solution. 4. Replace the ORP electrode.
	COND sensor 1. The calibration solution is not correct. 2. The sensor is dirty. 3. The COND sensor has failed.	COND sensor 1. Use the correct calibration solution for calibration. 2. Clean the sensor. 3. Contact your nearest sales outlet.
	TURB sensor 1. There are air bubbles on the cell. 2. The cell window is dirty. 3. The sensor is being affected by ambient light. 4. The solution is dirty. 5. The TURB sensor has failed.	TURB sensor 1. Shake the sensor probe vigorously. 2. Clean the cell window. 3. Calibrate using the calibration cup provided. 4. Replace the solution with new solution. 5. Replace the TURB sensor.
	DO sensor 1. The diaphragm is torn. 2. There are air bubbles in the internal solution. 3. The DO sensor has failed.	DO sensor 1. Replace the diaphragm with a new one, and fill the DO sensor with new internal solution. 2. Replace the diaphragm with a new one, and fill the DO sensor with new internal solution. 3. Replace the DO sensor.
	Temperature sensor The temperature sensor has failed.	Temperature sensor Contact your nearest sales outlet.
Calibration stability error	The calibration value of an individual parameter is not stable. 1. The sensor is dirty. 2. The sensor has not adjusted to the standard solution. 3. The temperature was unstable during calibration.	1. Clean the sensor. 2. Fill the transparent calibration cup with pH 4 standard solution, and wait for at least 20 minutes of conditioning before starting calibration. 3. Start calibration after the temperature has stabilized.
Turbidity calibration error	Error in turbidity measurement sequence	Turbidity calibration failed. Redo calibration after removing the displayed error.
Wet check	The cable connector is submerged.	Turn the power OFF and disconnect the cable connector. Wipe or blow-dry off all the water droplets on the probe. If the error persists, contact your nearest sales outlet to have the display and sensor probe repaired.
Power voltage error	The display's power board has failed.	This error could also be caused by poor cable contact. Turn the power OFF and disconnect the cable connector. Reconnect the connector and turn the power ON. If the error persists, contact your nearest sales outlet to have the display and sensor probe repaired.
Turbidity lamp power voltage error	The remaining battery level is low.	Turn the power OFF and replace the display's batteries with new ones.

Error	Cause	Solution
Display RTC error	The time display is incorrect.	Replace the coin battery.
Display FROM error	Internal IC failure	Contact your nearest sales outlet to have the control unit repaired.
Display EEPROM error	Internal IC failure	Contact your nearest sales outlet to have the control unit repaired.
Display save error	Insufficient memory space	Move data from the display, use the data operations screen to delete data, then redo the measurement.
Measurement sequence error	● When the measurement item is turbidity 1. The battery power is low. 2. The wiper is not operating normally. 3. The light source lamp is not lit. ● If items other than turbidity are also displayed 4. Board failure	1. Replace the batteries with new ones. 2. Check there are no obstacles near the wiper, then redo the measurement. If the error persists, the motor will need to be replaced. Contact your nearest sales outlet to have the sensor probe repaired. 3. Wipe off any water droplets on the probe, then remove the turbidity sensor. Check there are no water droplets around the turbidity sensor connector, then mount the sensor again. If the error persists, replace the turbidity sensor. 4. Contact your nearest sales outlet to have the sensor probe repaired.
Out of measurement range	The attempted measurement is outside the measurement range supported for that item.	The system must be used within its supported measurement ranges.
Last zero-point calibration invalid	pH sensor 1. The pH standard solution is contaminated. 2. The pH-responsive membrane is dirty. 3. The concentration of the reference electrode's internal solution has changed. 4. The pH-responsive membrane is torn.	pH sensor 1. Replace the standard solution with new solution. 2. Clean the pH-responsive membrane. 3. Refil the reference electrode's internal solution. 4. Replace the sensor.
	COND sensor 1. There is moisture on the sensor. 2. The sensor is dirty. 3. The COND sensor has failed.	COND sensor 1. Blow-dry the moisture off the sensor. 2. Clean the sensor. 3. Contact your nearest sales outlet.
	TURB sensor 1. There are air bubbles on the cell. 2. The cell window is dirty. 3. The sensor is being affected by ambient light. 4. The solution is dirty. 5. The TURB sensor has failed.	TURB sensor 1. Shake the sensor probe vigorously. 2. Clean the cell window. 3. Calibrate using the calibration cup provided. 4. Replace the solution with new solution. 5. Replace the TURB sensor.
	DO sensor 1. There are air bubbles in the internal solution. 2. The DO sensor has failed.	DO sensor 1. Replace the diaphragm with a new one, and fill the DO sensor with new internal solution. 2. Replace the DO sensor.
	Water depth sensor 1. The water depth sensor is dirty. 2. The water depth sensor has failed.	Water depth sensor 1. Clean the water depth sensor. 2. Contact your nearest sales outlet.
Out of measurement range	[See above.]	[See above.]
Last zero-point calibration invalid		

Error	Cause	Solution
Last span calibration invalid	pH sensor 1. The pH standard solution is contaminated. 2. The pH-responsive membrane is dirty. 3. The concentration of the reference electrode's internal solution has changed. 4. The pH-responsive membrane is torn.	pH sensor 1. Replace the standard solution with new solution. 2. Clean the pH-responsive membrane. 3. Refil the reference electrode's internal solution. 4. Replace the sensor.
	ORP sensor 1. The ORP standard solution is contaminated. 2. The ORP electrode is dirty. 3. The concentration of the reference electrode's internal solution has changed. 4. The ORP sensor glass is broken.	ORP sensor 1. Replace the standard solution with new solution. 2. Clean the ORP electrode. 3. Refil the reference electrode's internal solution. 4. Replace the sensor.
	COND sensor 1. The calibration solution is not correct. 2. The sensor is dirty. 3. The COND sensor has failed.	COND sensor 1. Use the correct calibration solution for calibration. 2. Clean the sensor. 3. Contact your nearest sales outlet.
	TURB sensor 1. There are air bubbles on the cell. 2. The cell window is dirty. 3. The sensor is being affected by ambient light. 4. The solution is dirty. 5. The TURB sensor has failed.	TURB sensor 1. Shake the sensor probe vigorously. 2. Clean the cell window. 3. Calibrate using the calibration cup provided. 4. Replace the solution with new solution. 5. Replace the TURB sensor.
	DO sensor 1. The diaphragm is torn. 2. There are air bubbles in the internal solution. 3. The DO sensor has failed.	DO sensor 1. Replace the diaphragm with a new one, and fill the DO sensor with new internal solution. 2. Replace the diaphragm with a new one, and fill the DO sensor with new internal solution. 3. Replace the DO sensor.
	Temperature sensor ● The temperature sensor has failed.	Temperature sensor ● Contact your nearest sales outlet.
Out of measurement range Last zero-point calibration invalid	[See above.]	[See above.]
Last span calibration invalid	The calibration value of an individual parameter is not stable. 1. The sensor is dirty. 2. The sensor has not adjusted to the standard solution. 3. The temperature was unstable during calibration.	1. Clean the sensors. 2. Fill the transparent calibration cup with pH 4 standard solution, and wait for at least 20 minutes of conditioning before starting calibration. 3. Start calibration after the temperature has stabilized.
Out of measurement range Last zero-point calibration invalid	[See above.]	[See above.]
Calibration value is factory default value.	Internal IC failure	Turn the power OFF, then restart the system. If the error persists, initialize the system from the "System" menu. If the error still persists, contact your nearest sales outlet to have the sensor probe repaired.

Error	Cause	Solution
Sample is unstable.	1. The concentration of the sample is unstable. 2. External light disturbance has affected the sensor. 3. Water has entered the turbidity sensor's connector.	1. Use a stirrer to agitate the sample during measurement. 2. Perform measurement away from direct sunlight. 3. Turn the power OFF, wipe off any water droplets on the probe, then remove the turbidity sensor. Check there are no water droplets around the turbidity sensor connector, then mount the sensor again. If the error persists, replace the turbidity sensor.

4.6.2 Error displays in sensor information

Error display	Cause	Solution
Measurement sequence error	Measurement sequence error	Turn the power OFF, then restart the system. If the error persists, have the probe repaired.
Out of measurement range	The measurement value is outside the measurement range.	Samples for measurement must be within the measurement range.
Last calibration invalid	The last calibration failed.	Redo calibration.
Calibration invalid	The calibration value is the factory default value.	Redo calibration.
Background unstable	The U-53 turbidity sensor is exposed to direct light.	Mount the guard cap and sensor guard and perform measurement away from direct sunlight.
	The turbidity value changed rapidly during measurement.	Measure a sample that has stable turbidity.

5 Specifications

Specification		Basic value	Model				
			U-51	U-52	U-52G	U-53	U-53G
Sensor probe	Measurement temperature range	−10°C to 55°C	✓	✓	✓	✓	✓
	Maximum sensor outer diameter	Approx. 96 mm					
	Sensor length	Approx. 340 mm					
	Cable length	2 m (standard) 10 m/30 m (options)					
	Mass	Approx. 1800 g					
	Auto calibration function	Uses pH 4 standard solution.					
	Measurement depth	30 m max.	✓	✓	✓	✓	✓
	Wet-part materials*3	PPS, glass, SUS316L, SUS304, FKM, PEEK, Q, titanium, FEP membrane, POM					
	Waterproofing standard	IP-68					
Control unit	Outer dimensions (W × D × H)	115 × 66 × 283 mm	✓	✓	—	✓	—
		115 × 66 × 335 mm	—	—	✓	—	✓
	Mass	Approx. 800 g	✓	✓	✓	✓	✓
	LCD	320 × 240 mm graphic LCD (monochrome) with backlight	✓	✓	✓	✓	✓
	Memory data items	10000	✓	✓	✓	✓	✓
	Communication interface	USB peripheral	✓	✓	✓	✓	✓
	Batteries	C-size dry cells (×4)	✓	✓	✓	✓	✓
	Waterproofing standard	IP-67	✓	✓	✓	✓	✓
	GPS unit	● Reception method (12 channel parallel) ● Measurement precision [With PDOP (high precision): 30 m or less (2 drms)]	—	—	✓	—	✓
	Estimated battery life*1	—	70 hours (no backlight)			500 measurements (no backlight)	
	Storage temperature range	−10°C to 60°C	✓	✓	✓	✓	✓
	Ambient temperature range	−5°C to 45°C					

Specification		Basic value	Model				
			U-51	U-52	U-52G	U-53	U-53G
pH measurement Two calibration	Measurement method	Glass electrode method	✓	✓	✓	✓	✓
	Range	pH 0 to 14					
	Resolution	0.01 pH					
	Precision*2	±0.1 pH					
Dissolved oxygen measurement ● Salinity conversion (0 to 70 PPT, automatic) ● Automatic temperature compensation	Measurement method	Polarographic method	✓	✓	✓	✓	✓
	Film thickness	25 μm					
	Range	0 mg/L to 50.0 mg/L					
	Resolution	0.01 mg/L					
	Precision*2	0 mg/L to 20 mg/L: ±0.2 mg/L 20 mg/L to 50 mg/L: ±0.5 mg/L					
Electrical conductivity measurement ● Auto range ● Automatic temperature conversion (25°C)	Measurement method	Four-AC-electrode method	✓	✓	✓	✓	✓
	Range	0 S/m to 10 S/m (0 mS/cm to 100 mS/cm)					
	Resolution	0.000 mS/cm to 0.999 mS/cm: 0.001 1.00 mS/cm to 9.99 mS/cm: 0.01 10.0 mS/cm to 99.9 mS/cm: 0.1 0.0 mS/m to 99.9 mS/m: 0.1 0.100 S/m to 0.999 S/m: 0.001 1.00 S/m to 9.99 S/m: 0.01					
	Precision*2	1% of full-scale (midpoint of two calibration points)					
Salinity measurement	Measurement method	Electrical conductivity conversion	✓	✓	✓	✓	✓
	Range	0 PPT to 70 PPT (parts per thousand)					
	Resolution	0.1 PPT					
	Precision	±3 PPT					
TDS (total dissolved solid) measurement ● Conversion coefficient setting	Measurement method	Electrical conductivity conversion	✓	✓	✓	✓	✓
	Range	0 g/L to 100 g/L					
	Resolution	0.1% of full-scale					
	Repeatability	±2 g/L					
	Precision	±5 g/L					
Seawater specific gravity measurement ● σt, σ0, σ15 display	Measurement method	Electrical conductivity conversion	✓	✓	✓	✓	✓
	Range	0 σt to 50 σt					
	Resolution	0.1 σt					
	Precision	±5 σt					

Specification		Basic value	Model				
			U-51	U-52	U-52G	U-53	U-53G
Temperature measurement	Measurement method	Platinum temperature sensor	✓	✓	✓	✓	✓
	Range	−10°C to 55°C					
	Resolution	0.01°C					
	Sensor	Platinum temperature sensor, JIS Class B (0.3 + 0.005 |t|)					
Turbidity measurement	Measurement method		—	LED forward 30° transmission/ scattering method		Tungsten lamp 90° transmission scattering method	
	Range			0 NTU to 800 NTU		0 NTU to 1000 NTU	
	Resolution			0.1 NTU		0.01 NTU	
	Precision*2			±5%of readout or ±1 NTU, whichever is larger		● ±0.5NTU (for 0 NTU to 10 NTU measurement range) ● 3% of readout or 1 NTU, whichever is larger (for 10 NTU to 1000 NTU measurement range)	
	Turbidity sensor wiper			—		✓	
Water depth measurement	Measurement method	Pressure method	—	—	✓	✓	✓
	Range	0 m to 30 m					
	Resolution	0.05 m					
	Precision*2	±0.3 m					
ORP (oxidation reduction potential) measurement	Measurement method	Platinum electrode method	✓	✓	✓	✓	✓
	Range	−2000 ～ +2000 mV					
	Resolution	1 mV					
	Precision*2	±15 mV					

*1: Battery life is estimated under following conditions.
- Continuous operation
- Using batteries: C-size alkaline dry cells
- Ambient temperature of the control unit: 20°C or more
- Backlight off

*2: The precision is defined by measuring the standard solution in the following cases.
- Turbidity and conductivity: after four point calibration
- pH and DO: after two point calibration
- Water depth and ORP: after one point calibration

*3: Metallic parts are made of stainless steel. Immersing in seawater may erode metallic parts.

6 Reference

6.1 Consumable parts

● Sensor

Name	Model	No.	Description
pH sensor	#7112	3014057312	Standard type pH sensor
pH sensor ToupH	#7113	3200170923	Tough glass type pH sensor
ORP sensor	#7313	3200170920	
DO sensor	#7543	3200170924	
Reference electrode	#7210	3200043582	
R bush unit	—	3200043587	Reference electrode liquid junction
TURB cell U-52	#7800	3200172803	For U-52/U-52G
TURB cell U-53	#7801	3200172800	For U-53/U-53G
Membrane cap	—	3200170194	For DO sensor

● Standard solution and inner solution

Name	Model	No.	Description
pH 4 (For automatic calibration) 500 mL	#100-4	3200043638	Standard solution for auto calibration. Also used for manual pH span calibration.
pH 4 (For automatic calibration) 4 L	#140-4	3200174430	
pH 7 500 mL	#100-7	3200043637	Standard solution for pH zero-point calibration.
pH 9 500 mL	#100-9	3200043636	Standard solution for pH manual span calibration.
Powder for ORP standard solution 10 packs	#160-51	3200043618	For ORP calibration.
Powder for ORP standard solution 10 packs	#160-22	3200043617	
Inner solution for DO sensor, 50 mL	#306	3200170938	Internal solution for DO sensor.
Internal solution for pH, 250 mL	#330	3200043641	Supplementary internal solution for pH reference electrode.

● Others

Name	Model	No.	Description
Silicone grease	—	3014017718	Silicone grease for coating sensor O-ring.
Sponge brush unit	—	3200169531	Brush for cleaning sensor probe.
O-ring set for reference electrode	—	3200169376	O-rings for reference electrode.
O-ring set for DO sensor	—	3200169426	O-rings for DO sensor.
Rubber cap set for sensor guard	—	3200169428	Rubber caps used between sensor guard and sensor probe.
O-ring set for pH and ORP sensor	—	3200169520	O-rings for pH and ORP sensors.
Wiper unit	—	3200169789	Rubber wiper for U-53/U-53G turbidity sensors.
Protective cap (blk) for pH sensor	—	3200175019	Cap attached to tip of pH sensor for sensor probe storage.
Rubber cap (whit) for DO sensor	—	3200175020	Cap attached to tip of DO sensor for sensor probe storage.

6.2 Options sold separately

Name	Model	No.	Description
Bag	U-5030	3200174772	Storage bag for sensor probes and flow cell. Can be carried in one hand.
Flow cell assy	—	3200156570	Used when collecting measurement samples by pump.
Probe guard	—	3200167002	Used for taking measurements in locations where there is a current or where there is a thick layer of sludge.
Communication cable	—	3200174823	A PC connection cable. Comes with data collection software.

6.3 pH measurement

6.3.1 Principle of pH measurement

U-50 series use the glass electrode method for pH measurements. The glass electrode method measures a potential difference between the glass film for pH and the reference electrode. For more information, refer to “JIS Z 8802 pH measurement method”.

6.3.2 Temperature compensation

The electromotive force generated by the glass electrode changes depending on the temperature of the solution.

Temperature compensation is used to compensate for the change in electromotive force caused by temperature.

This function does not compensate the change in pH caused by the temperature of the solution. When pH is to be measured, the temperature of the solution must be recorded along with that pH value, even if a pH meter has automatic temperature compensation function. If the solution temperature is not recorded, the results of the pH measurement may be meaningless.

6.3.3 Standard solutions

When measuring pH, the pH meter must be calibrated using standard solution. There are five kinds of standard solutions specified in “JIS Z 8802 pH measurement”. For normal measurement, two of standard solutions with pH of 4, 7, and 9 are sufficient to accurately calibrate the meter.

For standard solutions, refer to “JIS Z 8802 pH measurement”.

pH 4 standard solution: (Phthalate)	0.05 mol/L potassium hydrogen phthalate aqueous solution
pH 7 standard solutio: (Neutral phosphate)	0.025 mol/L potassium dihydrogenphosphate, 0.025 mol/L disodium hydrogenphosphate aqueous solution
pH 9 standard solution: (Borate)	0.01 mol/L sodium tetraborate aqueous solution

Table 2 pH values of pH standard solutions at various temperatures settings

Temp. (℃)	pH 4 standard solution Phthalate	pH 7 standard solution Neutral phosphate	pH 9 standard solution Borate
0	4.01	6.98	9.46
5	4.01	6.95	9.39
10	4.00	6.92	9.33
15	4.00	6.90	9.27
20	4.00	6.88	9.22
25	4.01	6.86	9.18
30	4.01	6.85	9.14
35	4.02	6.84	9.10
40	4.03	6.84	9.07
45	4.04	6.84	9.04

6.4 DO measurement

6.4.1 Principle of DO measurement

Dissolved oxygen (DO) refers to the amount of oxygen that is contained in water.
The concentration of dissolved oxygen is generally given as mg/L or as a percentage value (the dissolved oxygen saturation ratio).
Dissolved oxygen is essential for maintaining the self-purifying ability of rivers and seas and also for fish to live. The concentration of dissolved oxygen acts as an indicator of water quality. It is often measured when processing waste water and managing water quality. Fig. 1 provides an overview of the principles behind dissolved oxygen sensor measurement.

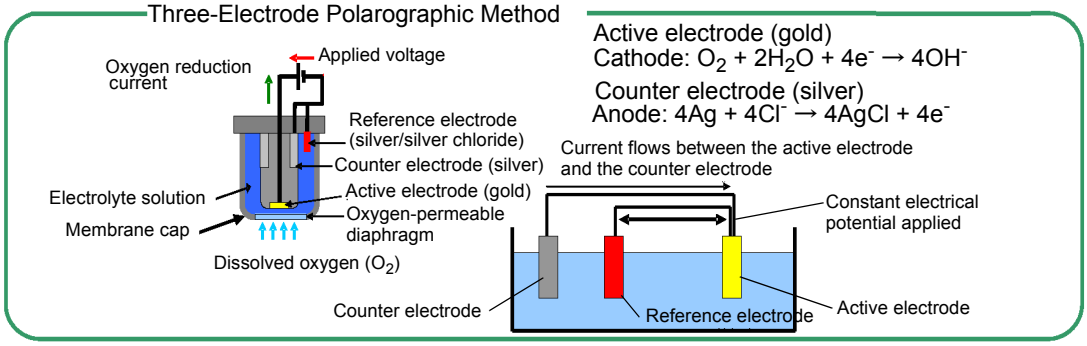


Fig. 1 Overview of principles behind dissolved oxygen sensor

The polarographic oxygen sensor is an enclosed sensor wherein voltage is applied to a cathode made of a precious metal (such as gold or platinum) and an anode also made of a precious metal (such as silver) via an external circuit, and a cap with an oxygen permeable diaphragm (membrane) is filled with electrolyte solution. As indicated in Fig. 1, the concentration of dissolved oxygen can be measured by measuring the current proportional to the amount of reduced oxygen when oxygen that has dispersed through the oxygen permeable diaphragm produces a reductive reaction on the surface of the active electrode (gold). The method of measuring dissolved oxygen based on the above principle is called the Membrane Electrode Method. Compared to the Chemical Analysis Method, which requires complicated pre-processing to alleviate the effect of reduced materials and oxidizing materials, this method allows dissolved oxygen to be measured very easily. It is also easy to remove undesired buildup from the silver electrode by polishing and cleaning if an insulator forms on it due to oxidation, making the method reusable.

6.4.2 Salinity calibration

When the solution and air come into contact and form an equilibrium (i.e. saturation), the relationship between the concentration of dissolved oxygen in the solution, C, [mol/L], and the partial pressure of oxygen in the air, Ps, [MPa/(mg/L)], can be represented by the following formula:

$$C = Ps/H$$

Where H [MPa/(mg/L)] is the Henry constant, a value that changes according to the composition of the solution. As H typically becomes larger as the salinity of the water increases, C becomes smaller.

The DO sensor detects the partial pressure of oxygen (Ps) in the above formula. Accordingly, if the DO sensor is immersed in deionized water saturated with air, or in an aqueous solution containing salt, the output current does not change, resulting in an erroneous measurement. For example, when salt is added to a sample, the amount of oxygen that can be dissolved in the solution decreases, but because the partial pressure of oxygen does not change, the value displayed by the control unit stays the same regardless of salt content. This concept is indicated in graph form below. (Fig. 2)

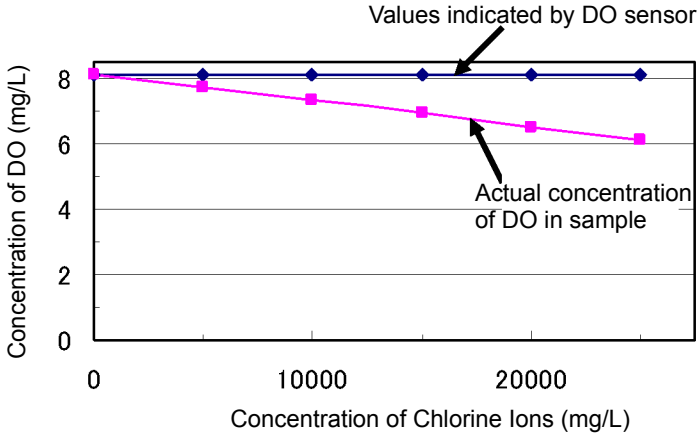


Fig. 2 Relationship between chlorine ion concentration and dissolved oxygen concentration

In samples with a high salt concentration, the solubility of oxygen is lower, but as the partial pressure of oxygen does not change, the value actually indicated on the control unit is higher than the actual value. In order to obtain a measurement of the concentration of dissolved oxygen in an aqueous solution that contains salt, it is therefore necessary to first perform salinity compensation. Conventionally, dissolved oxygen sensors have performed salinity compensation by inputting the salinity of the sample. This is fine as long as the salinity is already known. However, in most cases salinity is unknown, so even if dissolved oxygen sensors contained a salinity compensation function, it was of no practical use.

The U-50 Series can calculate and measure salinity in samples from electrical conductivity values, and can thus be used to automatically compensate for salinity.

6.5 Conductivity (COND) measurement

6.5.1 Four-AC-electrode method

Conductivity is an index of the flow of electrical current in a substance.

Salts dissolved in water are separated into cations and anions. Such solution is called electrolytic solution.

Electrolytic solution has the property of allowing the flow of current according to Ohm's law. This property is referred

to as ionic conductivity, since current flow is caused by ion movement in electrolytic solution.

Metals, on the other hand, allow the flow of current by means of electrons. This property is called electronic conductivity,

which is distinguished from ionic conductivity.

A cube with 1 m on each side, as shown in Fig. 3, is used to demonstrate an electrolytic solution. Two electrode plates are placed on opposite sides, and the cube is filled with solution. If the resistance between these two electrode plates is represented by r(Ω), the conductivity of the solution L($S \cdot m^{-1}$) is represented as L=1/r. S stands for Siemens, a unit of measurement of conductance.

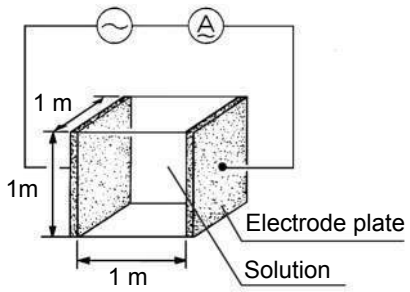


Fig. 3 Definition of conductivity

The most general method for measuring conductivity is based on the above principle, and is called the 2-electrode method.

In the 2-electrode method the influence of polarization cannot be ignored for solutions with high conductivity and conductivity cannot be measure accurately. In addition, contamination on the surface of the electrode increases apparent resistance, resulting in inaccurate measurement of conductivity.

The U-50 series has adopted the 4-electrode method to overcome these disadvantages of the the 2-electrode method.

As shown in Fig. 4, the U-50 series uses two voltage-detecting electrodes and two voltage-applying electrodes, for a total of four electrodes. The voltage-detecting electrodes are for detecting AC voltage, and the voltage-applying electrodes are for applying AC voltage.

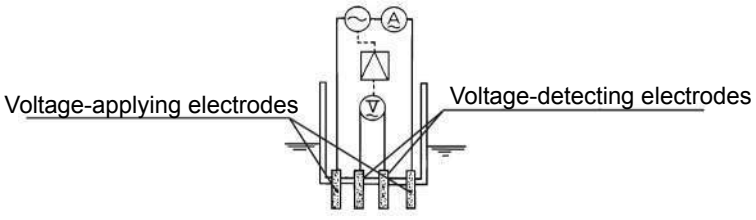


Fig. 4 Principle of the 4-electrode method

Let us assume that the current, I(A), flows in a sample of conductivity L – under automatic control of the voltage-applying electrodes – so that the voltage at the voltage-detecting-electrodes, E(V), remains constant at all times.
Then, the resistance of the sample, R(Ω), across the voltage-detecting electrodes is represented as R=E/I. The resistance, R, of the sample is inversely proportional to its conductivity, L. Accordingly, a measurement of current, Is,
of a standard solution of known conductivity, Ls, enables calculation of conductivity of a sample according to the formula L = Ls (I/Is) from the ratio L : Ls = I : Is.
Even in the 4-electrode method, polarization occurs, since AC current flows in the voltage-applying electrodes. The voltage-detecting electrodes are, however, free from the effects of polarization, since they are separated from the voltage-applying electrodes, and furthermore, current flow is negligible. Therefore, the 4-electrode method is an excellent method to enable measurement of conductivity covering a very high range.

6.5.2 SI units

New measurement units, called SI units, have been in use from 1996. Accordingly, the U-50 series also uses SI units. The following conversion table is provided for people who use the conventional kind of conductivity meter.
Note that along with the change in unit systems, the measurement values and cell counts have also changed.

	Former units	→	SI unit
Measurement value	0.1 mS/cm	→	0.01 S/m
	1 mS/cm	→	0.1 S/m
	100 mS/cm	→	10 S/m

6.5.3 Temperature coefficient

In general, the conductivity of a solution varies largely with its temperature.
The conductivity of a solution depends on the ionic conductivity, described earlier. As the temperature rises, conductivity becomes higher since the movement of the ions becomes more active.
The temperature coefficient shows the change in % of conductivity per °C, with a certain temperature taken as the reference temperature. This is expressed in units of %/°C. The temperature coefficient assumes the premise that the conductivity of a sample changes linearly according to temperature.
Strictly speaking, with actual samples, however, conductivity changes along a curve. Furthermore, the cuve varies with the type of sample. In the ranges of smaller temperature changes, however, samples are said to have the temperature coefficient of 2%/°C (at reference temperature 25°C); this holds for most samples, except in certain special cases.
(The temperature coefficients for various types of solutions are listed on the next page.)
The U-50 series uses an automatic temperature conversion function to calculate conductivity at 25°C at a temperature
coefficient of 2 %/°C based on the measured value of the temperature. Results are displayed on the readout.
The U-50 series's temperature conversion function is based on the following formula.
$L_{25} = L_t / \{ 1 + K (t - 25) \}$
L_{25} : Conductivity of solution converted to 25°C
t : Temperature of solution at time of measurement (°C)
L_t : Conductivity of solution at t (°C)
K : Temperature coefficient (%/°C)

● Conductivity and temperature coefficient for various solutions

Conductivity and related temperature coefficients of representative substances (at 25°C) are shown in the table below.

Substance	Temp. (ºC)	Conc. (wt%)	Cond. (S/m)	Temp.coef. (%/°C)	Substance	Temp. (ºC)	Conc. (wt%)	Cond. (S/m)	Temp.coef. (%/°C)
NaOH	15	5	19.69	2.01	NaCl	18	5	6.72	2.17
		10	31.24	2.17			10	12.11	2.14
		15	34.63	2.49			15	16.42	2.12
		20	32.70	2.99			20	19.57	2.16
		30	20.22	4.50			25	21.35	2.27
		40	11.64	6.48	Na_2SO_4	18	5	4.09	2.36
KOH	15	25.2	54.03	2.09			10	6.87	2.49
		29.4	54.34	2.21			15	8.86	2.56
		33.6	52.21	2.36	Na_2CO_3	18	5	4.56	2.52
		42	42.12	2.83			10	7.05	2.71
NH_3	15	0.1	0.0251	2.46			15	8.36	2.94
		1.6	0.0867	2.38	KCl	18	5	6.90	2.01
		4.01	0.1095	2.50			10	13.59	1.88
		8.03	0.1038	2.62			15	20.20	1.79
		16.15	0.0632	3.01			20	26.77	1.68
HF	18	1.5	1.98	7.20			21	28.10	1.66
		4.8	5.93	6.66	KBr	15	5	4.65	2.06
		24.5	28.32	5.83			10	9.28	1.94
HCl	18	5	39.48	1.58			20	19.07	1.77
		10	63.02	1.56	KCN	15	3.25	5.07	2.07
		20	76.15	1.54			6.5	10.26	1.93
		30	66.20	1.52			—	—	—
H_2S0_4	18	5	20.85	1.21	NH_4Cl	18	5	9.18	1.98
		10	39.15	1.28			10	17.76	1.86
		20	65.27	1.45			15	25.86	1.71
		40	68.00	1.78			20	33.65	1.61
		50	54.05	1.93			25	40.25	1.54
		60	37.26	2.13	NH_4NO_3	15	5	5.90	2.03
		80	11.05	3.49			10	11.17	1.94
		100.14	1.87	0.30			30	28.41	1.68
		—	—	—			50	36.22	1.56
HNO_3	18	6.2	31.23	1.47	$CuSO_4$	18	2.5	10.90	2.13
		12.4	54.18	1.42			5	18.90	2.16
		31	78.19	1.39			10	32.00	2.18
		49.6	63.41	1.57			15	42.10	2.31
		62	49.64	1.57	CH_3COOH	18	10	15.26	1.69
H_3PO_4	15	10	5.66	1.04			15	16.19	1.74
		20	11.29	1.14			20	16.05	1.79
		40	20.70	1.50			30	14.01	1.86
		45	20.87	1.61			40	10.81	1.96
		50	20.73	1.74			60	4.56	2.06

6.6 Salinity (SAL) conversion

The U-50 series is designed to calculate salinity as well as the other parameters.
Note that the “salinity” here is the salinity of sea water. There is a constant relation between conductivity and salinity at certain temperatures.
Therefore, if data on the conductivity and temperature are available, the corresponding salinity can be known. In other words, the salinity measurement of the U-50 series is based on the principle of calculating the salt content, making use of the measured values of conductivity and temperature.
Note therefore, that measured results of all substances whose conductivity is detected are displayed as salinity. For example, the measured result is displayed as NaCl concentration, even if in fact the sample component is, hydrochloric acid (HCl).

6.7 TDS conversion

TDS is short for Total Dissolved Solids and means the total dissolved solid amount.
The conductivity of a solution is affected by the amount of salinity, minerals, and dissolved gases. That is, conductivity is an index that shows the total amount of all substances in the solution. Of these substances, TDS indicates only the amount of dissolved solids.
TDS can be used for a comparison of the state of substances composed of a single component such as NaCl. However, the use of TDS for the comparison of solutions of different types causes serious errors.
Conductivity and TDS are expressed by the following formulas.
Conductivity in SI units (S/m) ..... TDS(g/L) = L (S/m) $\times$ K $\times$ 10
TDS(g/L) = L (mS/m) $\times$ K $\div$ 100
Conductivity in the old units (mS/cm) ..... TDS(g/L) = L (mS/cm)$\times$ K
K = TDS coefficient
Initial settings use the values listed in the table (Page 80) that generally uses TDS coefficients.
For accurate TDS comparisons, find the TDS coefficient from measured conductivity values. Then set the value thus obtained and make measurements.

6.8 σt conversion

- **Specific gravity of seawater**

The density and specific gravity of seawater are equal numerically and generally are not distinguished strictly. Since seawater density ρ is between 1.000 and 1.031, 1 is subtracted from ρ and σ is obtained by multiplying the value by 1000.
The resultant value is used as the specific gravity of seawater.
$\sigma = (\rho - 1) \times 1000$
The density of seawater ρ is expressed by function of temperature, hydraulic pressure, and salinity. The density of seawater under the atmospheric pressure is expressed as σ_t. The density of seawater under the atmospheric pressure is determined by temperature and salinity.
The U-50 Series models make salinity measurement through temperature measurements and conductivity conversion and find σ_t through calculations.
In Japan σ_{15} at 15°C is called a standard specific gravity and widely used while in foreign countries σ_0 at 0°C is employed. σ_{15} and σ_0 are determined by the function of salinity.
In ocean surveys, in particular, these values σ_t, σ_{15}, and σ_0 are more widely used than conductivity and salinity and, in the U-50 Series models, newly added as measurement components.

6.9 Turbidity (TURB) measurement

6.9.1 Principle of turbidity measurement

U-52 and U-53 sensors measure turbidity using the Transmitting and Scattering Method shown in Fig. 5. U-52 sensors use a pulse light LED (infra-red emitting diode) as a light source, and detect scattered light from a 30° angle off center. U-53 sensors use a tungsten lamp as a light source and detect scattered light from a 90° angle. Both models display turbidity as a ratio of scattered light to transmitted light to reduce the affect of the color of the sample. The U-53 method conforms to EPA Method 180.1, and employs wipers to reduce the affect of air bubbles.

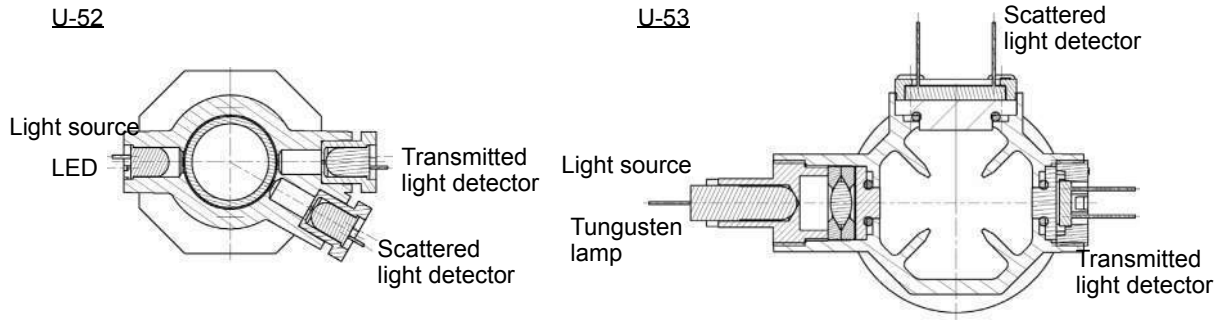


Fig. 5 Turbidity cell

6.9.2 Standard solution

U-50 series can perform calibration using formazin (NTU) or kaolin standard solutions as a turbidity standard solution. However, units for the solution used for calibration should be displayed in measurements. Do not use more than 400 mg/L of kaolin standard solution because it increases precipitation speed, resulting in measurement error.

6.10 Depth (DEPTH) measurement

6.10.1 Principle of depth measurement

For the W-22XD and W-23XD models, depth measurement can be made through use of a pressure gauge. The principle of the depth measurement uses the relation between depth and pressure.

Although the measurement with the depth sensor is affected by atmospheric pressure, the depth sensor, however, makes zero-point adjustments through the automatic calibration before measurements.

6.10.2 Influence of temperature and calibration

The depth sensor depends greatly on temperature. For a wide difference between the temperature at which the sensor has been automatically calibrated and the temperature of the measurement sample, the sensor can make depth measurements with a higher accuracy by the following method:

1. **Immerse the depth sensor of the sensor probe in the sample.**
2. **Keep the sensor immersed in the sample for about 30 minutes until the temperatures of the sensor and the sample are the same.**
3. **Then make the zero calibration of the sensor manually.**

6.11 Oxidation reduction potential (ORP) measurement

6.11.1 Principle of ORP measurement

ORP is an abbreviation for oxidation-reduction potential. ORP is the energy level (potential) determined according to the state of equilibrium between the oxidants (M^{Z+}) and reductants $M^{(Z-N)+}$ that coexist within a solution.

$$M^{Z+} + ne^- \Leftrightarrow M^{(Z-N)+} \quad \cdots (1)$$

If only the solution, forming the ORP measuring system shown in Fig. 6. The difference of potential between two electrodes is generally expressed by the following equation.

$$E = E_0 - \frac{RT}{nF} \ln \frac{a_M{}^{(z-n)+}}{a_M{}^{z+}} \quad \cdots (2)$$

E: Electric potential E_0 : Constant R: Gas constant T : Absolute temperature
n: Electron count F : Faraday constant a : Activity

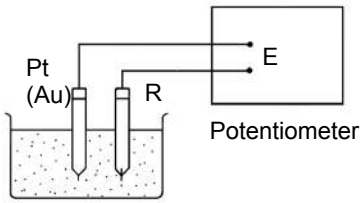


Fig. 6 Measuring mV

For example, for a solution in which trivalent iron ions coexist with bivalent iron ions, equations 1 and 2 would be as follows.

$$Fe^{3+} + e^- \Leftrightarrow Fe^{2+} \quad \cdots (1)$$

$$E = E_0 - \frac{RT}{F} \ln \frac{a_{Fe}{}^{2+}}{a_{Fe}{}^{3+}} \quad \cdots (2)$$

When only one type of state of equilibrium uniquely by equation (Fe^{3+}) and the reductant (Fe^{2+}) (using the equation $a_{Fe}{}^{2+}/ a_{Fe}{}^{3+}$). Actually, however many kinds of states of equilibrium exist simultaneously between various kinds of ions, in most solutions. This means that under actual circumstances, ORP cannot be expressed using the simple equation shown above and that the physical and chemical significance with respect to the solution is not very clear.
In this respect, the value of ORP must be understood to be only one indicator of the property of a solution. The measurement of ORP is widely used, however, as an important index in the analysis of solutions (potentiometric titration) and in the waste water treatment.

6.11.2 Standard electrode (reference electrode) types and ORP

The ORP is obtained comparing with corresponding reference electrode employed.
If different kinds of reference electrodes are used for measurement, the ORP value of the same solution may appear to be different. HORIBA's reference electrode uses Ag/AgCl with 3.33 mol/L KCl as inner solution. According to general technical literature, normal hydrogen electrodes (N.H.E.) are often used as the standard electrode.
The relationship between N.H.E. and the ORP that is measured using an Ag/AgCl with 3.33 mol/L KCl electrode is expressed by the following equation.
$E_{N.H.E.} = E + 206 - 0.7(t - 25)$ mV t = 0 - 60°C
$E_{N.H.E.}$: Measured ORP value using N.H.E. as the reference electrode

E: Measured ORP value using Ag/AgCl with 3.33 mol/L KCl as the reference electrode Potential sign

Standard ORP is expressed in the following way, in literature related to electrochemistry and analytical chemistry.

A $Li^{+} + e^{-} \rightarrow Li$

$E_0 = -3.024$ V VS N.H.E.

However, in some literature, the "+" and "-" signs are reversed.

B $Li \rightarrow Li^{+} + e^{-}$

$E_0 = +3.024$ V VS N.H.E.

In expressions like B, above, the reaction is just reversed and there is no essential difference. But this kind of expression does invite confusion. The majority of the world, today, is consistent in its use of the signs as they are used in A, above.

For this reason, HORIBA, too, uses signs concerning ORP that are consistent with A, above.

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PRO20

USER MANUAL

English

Français

Español

Deutsch

Item # 605597
Rev B
Drawing # A605597
October 2008

CONTENTS

W A R R A N T Y

The YSI Pro20 Instrument is warranted for three (3) years from date of purchase by the end user against defects in materials and workmanship, exclusive of batteries and any damaged caused by defective batteries. Pro20 cables are warranted for two (2) years from date of purchase by the end user against defects in material and workmanship. Pro20 Polarographic sensors are warranted for one (1) year and Galvanic sensors are warranted for six (6) months from date of purchase by the end user against defects in material and workmanship. Pro20 instruments, cables & probes are warranted for 90 days from date of purchase by the end user against defects in material and workmanship when purchased by rental agencies for rental purposes. Within the warranty period, YSI will repair or replace, at its sole discretion, free of charge, any product that YSI determines to be covered by this warranty.

To exercise this warranty, call your local YSI representative, or contact YSI Customer Service in Yellow Springs, Ohio at +1 937 767-7241, 800-897-4151 or visit www.ysi.com (Support tab). Send the product and proof of purchase, transportation prepaid, to the Authorized Service Center selected by YSI. Repair or replacement will be made and the product returned, transportation prepaid. Repaired or replaced products are warranted for the balance of the original warranty period, or at least 90 days from date of repair or replacement.

LIMITATION OF WARRANTY

This Warranty does not apply to any YSI product damage or failure caused by:

1) failure to install, operate or use the product in accordance with YSI's written instructions;
2) abuse or misuse of the product;
3) failure to maintain the product in accordance with YSI's written instructions or standard industry procedure;
4) any improper repairs to the product;
5) use by you of defective or improper components or parts in servicing or repairing the product;
6) modification of the product in any way not expressly authorized by YSI.

THIS WARRANTY IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING ANY WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. YSI's LIABILITY UNDER THIS WARRANTY IS LIMITED TO REPAIR OR REPLACEMENT OF THE PRODUCT, AND THIS SHALL BE YOUR SOLE AND EXCLUSIVE REMEDY FOR ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY. IN NO EVENT SHALL YSI BE LIABLE FOR ANY SPECIAL, INDIRECT, INCIDENTAL OR CONSEQUENTIAL DAMAGES RESULTING FROM ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY.

INTRODUCTION

Thank you for purchasing the YSI Pro20, an instrument from the YSI *Professional Series* product family. The Pro20 features an impact resistant and waterproof (IP-67) case, backlit display, user-selectable sensor options, internal barometer, and a rugged, rubber over-mold case.

The Pro20 provides valuable instructions and prompts near the bottom of the display that will guide you through operation and use. However, reading the entire manual is recommended for a better understanding of the Pro20's features.

The Pro20 can not communicate to a PC via a Pro Plus communications saddle. Connecting the Pro20 to a communication saddle may cause erratic instrument behavior.

GETTING STARTED

INITIAL INSPECTION

Carefully unpack the instrument and accessories and inspect for damage. Compare received parts with materials listed on the packing list. If any parts or materials are missing or damaged, contact YSI Customer Service at 800-897-4151 (+1-937-767-7241) or the Authorized YSI distributor from whom the instrument was purchased.

BATTERY INSTALLATION

This instrument requires 2 alkaline C-cell batteries. Under normal conditions, battery life is approximately 400 hours at room temperature without using the back light. A battery symbol  will blink in the lower, left corner of the display to indicate low batteries when approximately 1 hour of battery life remains.

To install or replace the batteries:

1) Turn the instrument off and flip over to view the battery cover on the back.
2) Unscrew the four captive battery cover screws.
3) Remove the battery cover, and remove the old batteries if necessary.
4) Install the new batteries, ensuring correct polarity alignment (Figure 1).

5) Place the battery cover on the back of the instrument and tighten the four screws. Do NOT over-tighten.

Figure 1, Pro20 with battery cover removed. Note battery symbols indicating polarities.

The waterproof instrument case is sealed at the factory and is not to be opened, except by authorized service technicians. Do not attempt to separate the two halves of the instrument case as this may damage the instrument, break the waterproof seal, and will void the warranty.

KEYPAD

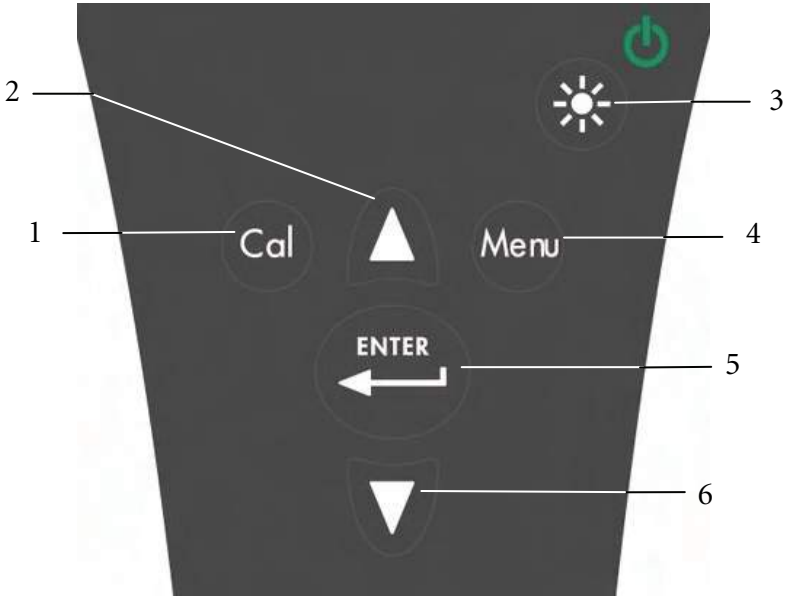


Figure 2, keypad

Number	*Key*	*Description*
1		**Calibrate** Press and hold for 3 seconds to calibrate. Initiates One Touch Calibration. Opens Calibrate menu from the run screen if One Touch Calibration is disabled.
2		**Up Arrow** Use to navigate through menus, to navigate through box options at the bottom of the run screen, and to increase numeric inputs.
3		**Power and Backlight** Press once to turn instrument on. Press a second time to turn backlight on. Press a third time to turn backlight off. Press and hold for 3 seconds to turn instrument off.
4		**Menu** Use to enter the System Setup menu from the run screen.
5		**Enter** Press to confirm entries and selections.
6		**Down Arrow** Use to navigate through menus, to navigate through box options at the bottom of the run screen, and to decrease numeric inputs.

CONNECTING THE SENSOR AND CABLE

CONNECTING THE SENSOR

"Sensor" refers to the removable portion or electrode sensing portion of the cable assembly, i.e. the dissolved oxygen sensor. "Bulkhead" refers to the portion of the cable with the single-pin connector (Figure 3).

The Pro20 has two compatible sensors for use with a field cable:

Polarographic – This sensor has a black sensor body and is engraved with the model number 2003. Polarographic will be abbreviated Polaro in the instrument.

Galvanic – This sensor has a grey sensor body and is engraved with the model number 2002.

For information about the differences on the two sensor types, see Sensor Type in the System Setup menu section and/or the Principles of Operation section of this manual.

If using a ProBOD sensor/cable assembly, there is no need to install a sensor because it has a built in Polarographic dissolved oxygen sensor.

Before installing either sensor or connecting the cable to the instrument, the Sensor Type must be configured for the sensor being installed/connected. Failure to do this may result in damage not covered under warranty. The instrument will step you through this setup the first time it is powered on. See the System Setup menu section of this manual for instructions on configuring the Sensor Type after the first power on.

1) Ensure both the sensor connector and sensor port on the cable are clean and dry.
2) Grasp the sensor with one hand and the cable bulkhead in the other.
3) Push the sensor into the connector on the cable until it is firmly seated and only 1 o-ring is visible. Failure to properly seat the sensor may result in damage.
4) Twist the sensor clockwise to engage threads and finger tighten. Do NOT use a tool. This connection is water-tight.

For more detailed instructions, please refer to the sensor installation sheet that is included with each sensor.

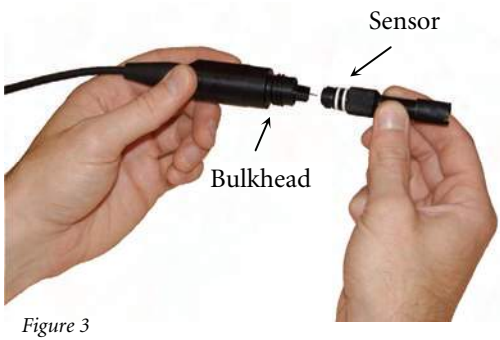


Figure 3

CONNECTING THE CABLE

The Pro20 is designed for field and laboratory use. It is compatible with two different cable options:

1) The field rugged cable is available in standard lengths of 1, 4, 10, 20, 30, and 100 meters with special lengths available between 30 and 100 meters. This cable has a built in temperature sensor and includes a port for the dissolved oxygen sensor.
2) The ProBOD is a 1 meter probe/cable assembly with built in Polarographic dissolved oxygen and temperature sensors. It has an AC powered motor for sample stirring and is designed to fit into a 300 ml BOD bottle.

To connect the cable, align the keys in the cable connector to the slots in the instrument connector. Push together firmly and then twist the outer ring until it locks into place (Figure 4). This connection is water-proof.

Figure 4, Note the keyed connector.

When disconnected, the sensor and cable's sensor connectors are NOT water-proof. Do not submerge the cable without a sensor installed. When disconnected, the cable's instrument connector and the connector on the instrument maintain a waterproof, IP-67 rating.

MEMBRANE INSTALLATION

The dissolved oxygen sensor is shipped with a dry, protective red cap that will need to be removed before using. It is very important to put a new membrane with electrolyte solution on the sensor after removing the red cap.

Prepare the membrane solution according to the instructions on the bottle. After mixing, allow the solution to sit for 1 hour. This will help prevent air bubbles from later developing under the membrane. Ensure you are using the correct electrolyte solution for the correct sensor. Galvanic sensors utilize electrolyte with a light blue label and Polarographic sensors utilize electrolyte with a white label. The Dissolved Oxygen sensor is supplied with cap membranes specific to the sensor type ordered (Polarographic or Galvanic). 5913 and 5914 membrane kits are for Galvanic sensors and the 5908 and 5909 membrane kits are for Polarographic sensors.

Remove and discard or save the red protective cap. Thoroughly rinse the sensor tip with distilled or deionized water. Fill the cap membrane 3⁄4 full of electrolyte solution, then tap the cap with a finger to release any trapped air. Be careful not to touch the membrane portion of the cap. Thread the membrane cap onto the sensor, moderately tight. Do not use a tool. It's typical for some of the electrolyte solution to spill over. It is best to allow the new cap to remain on a new sensor overnight before trying to calibrate. For detailed instructions on changing a membrane cap, see the Care, Maintenance, and Storage section of this manual.

BACKLIGHT

Once the instrument is on, pressing power/backlight key will turn on the display backlight. The backlight will remain on until the key is pressed again or after two minutes of not pressing any key on the keypad.

POWERING OFF

To turn the instrument off, press and hold the power/backlight key for three seconds.

RUN SCREEN

Press the power/backlight key to turn the instrument on. The instrument will run through a self test and briefly display a splash screen with system information before displaying the main run screen (Figure 5). The first time the Pro20 is

turned on, it will step through language, sensor, and membrane selections; see the First Power On section of this manual for more information.

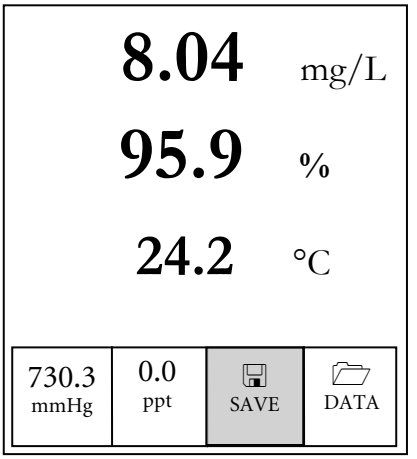


Figure 5, example of main run screen with Save highlighted.

NAVIGATION

The up and down arrow keys allow you to navigate through the functions of the Pro20.

NAVIGATING IN THE RUN SCREEN

When in the run screen, the up and down arrow keys will move the highlighted box along the bottom options. Once a box is highlighted, press enter to access the highlighted option.

Description of run screen box functions from left to right:

Option	*Description*
Barometer reading	Highlight and press enter to calibrate the barometer
Salinity compensation value	Highlight and press enter to adjust salinity compensation value
SAVE	Highlight and press enter to save current data to memory
DATA	Highlight and press enter to view and/or erase saved data

NAVIGATING IN THE SYSTEM SETUP MENU

When in the System Setup menu, the up and down arrow keys will move the highlighted bar up and down the system setup options. See the System Setup menu section of this manual for more information about these options.

FIRST POWER ON

The instrument will step through an initial configuration when powered on for the first time. This will set the language, sensor, and membrane options. Use the up or down arrow keys to highlight the appropriate language, sensor, and membrane, then press enter to confirm (Figures 6, 7, and 8). The Sensor Type must be configured for the sensor installed. Failure to do this may result in damage not covered under warranty. If an incorrect option is selected, it may be changed in the System Setup menu.

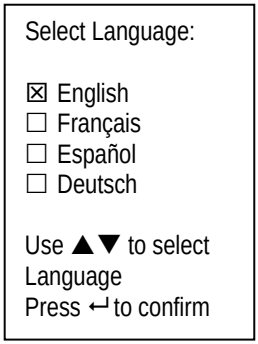


Figure 6, Language selection

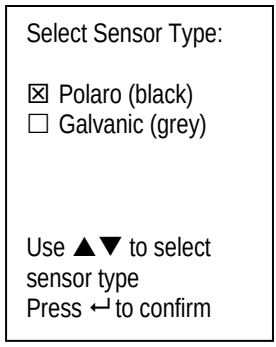


Figure 7, Sensor selection

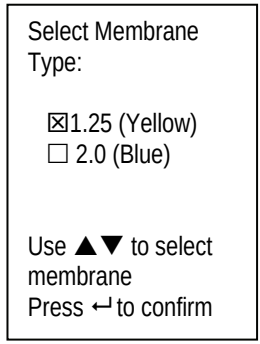


Figure 8, Membrane selection

After selecting a language, sensor, and membrane, the run screen will appear. The next time the instrument is powered up the run screen will appear immediately after the self check. If the sensor type or membrane type is changed, ensure that it updated in the System Setup menu.

SYSTEM SETUP MENU

Press the menu key to access the following System Setup functions.

The System Setup menu contains multiple screens which are notated as 'pages'. The current page is indicated on the display, figure 9.

DO LOCAL%

DO Local% can be enabled or disabled by using the up or down arrow keys to highlight it and then pressing enter. An 'X' in the box next to DO Local% indicates it is enabled (Figure 9).

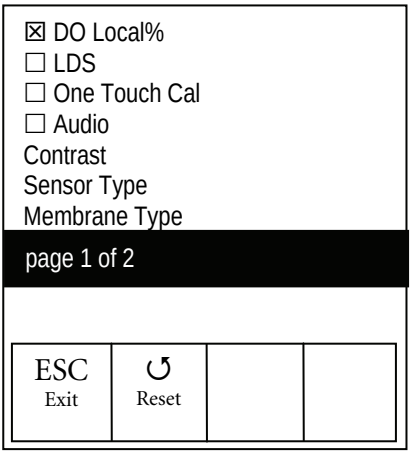


Figure 9, DO %Local is enabled.

When DO Local% is enabled, DO% values will be expressed as %L on the run screen.

DO Local% allows for localized dissolved oxygen measurements. This sets the DO% calibration value to 100% regardless of the altitude or barometric pressure. When DO Local% is enabled, the Pro20 will factor in the barometric pressure on each measurement. For example, if the barometric pressure changes, the DO %L reading would remain constant in air-saturated water or in water-saturated air. Local DO is ideal for EU compliance.

LAST DIGIT SUPPRESSION (LDS)

Last Digit Suppression (**LDS**) can be enabled or disabled by using the up or down arrow keys to highlight it and pressing enter  . An 'X' in the box next to LDS indicates it is enabled.

LDS rounds the DO value to the nearest tenth; i.e. 8.25 mg/L becomes 8.3 mg/L. LDS is automatically disabled during calibrations.

ONE TOUCH CALIBRATION (ONE TOUCH CAL)

One Touch Calibration can be enabled or disabled by using the up or down arrow keys to highlight **One Touch Cal** and pressing enter  . An 'X' in the box next to One Touch indicates it is enabled.

When One Touch Cal is enabled, press and hold the calibration 

key for 3 seconds while in the run screen to calibrate Dissolved Oxygen to the barometer reading and salinity correction value. For more information on One Touch Calibration, see the Calibration section of this manual.

AUDIO

Audio can be enabled or disabled by using the up or down arrow keys to highlight **Audio** and pressing enter  . When enabled, there will be an 'X' in the box next to Audio.

When Audio is enabled, the Pro20 will beep twice to indicate stability when Auto Stable is enabled. The instrument will also beep when a key is pressed. When Audio is disabled, the Pro20 will not beep.

CONTRAST

To adjust the display **Contrast**, use the up or down arrow keys to highlight **Contrast**, then press enter  . Next, use the up or down arrow keys to adjust the contrast. The up arrow will darken the contrast and the down arrow will lighten the contrast. After adjusting the contrast, press enter to save and exit the Contrast adjustment option.

EMERGENCY CONTRAST ADJUSTMENT

If necessary, there is an alternate method of adjusting the contrast. To adjust the contrast, press and hold the menu key, then press the up arrow key to darken the contrast or press the down arrow key to lighten the contrast.

SENSOR TYPE

The instrument's Sensor Type must be configured for the sensor installed. Failure to do this may result in damage not covered under warranty. If you observe readings very close to 0 or extremely high readings, i.e. 600%, your Sensor Type setting may be set incorrectly.

Sensor Type sets the type of oxygen sensor being used; either Polarographic (black) or Galvanic (grey).

Use the up or down arrow keys to highlight **Sensor Type**, then press enter  to open a submenu. Highlight the sensor type corresponding to the sensor installed on the cable and press enter to confirm. The enabled sensor type will have an 'X' in the box next to it. Use the down arrow key to highlight the **ESC – Exit**, then press enter to save changes and to close the sensor submenu.

If using a ProBOD sensor/cable assembly, the sensor type should be set to polarographic.

The Pro20 has two compatible sensors for use with a field cable:

Polarographic – This sensor has a black sensor body and is engraved with the model number 2003. Polarographic will be abbreviated Polaro in the instrument.

Galvanic – This sensor has a grey sensor body and is engraved with the model number 2002.

In terms of physical configuration, membrane material, and general performance, YSI Professional Series Galvanic dissolved oxygen sensors are exactly like the Professional Series Polarographic sensors. The advantage of using Galvanic sensors is convenience. Galvanic sensors provide for an instant-on sensor without the need for warm-up time but this affects the life of the sensor. Polarographic sensors last longer and have a longer warranty but require a 5-15 minute warm-up time before use or calibration.

MEMBRANE TYPE

Membrane Type sets the type of membrane used on the dissolved oxygen sensor; either 1.25 PE (Yellow) or 2.0 PE (blue). Use the up or down arrow keys to highlight **Membrane Type** and press enter  to open the membrane submenu. Highlight the membrane type corresponding to the membrane installed on the sensor and press enter to confirm. The enabled membrane type will have an 'X' in the box next to it. Use the down arrow key to highlight the **ESC – Exit** box and press enter to save changes and to close the membrane submenu.

The dissolved oxygen sensor is supplied with membranes specific to the sensor type ordered and are color coded as described in the following tables.

Galvanic Membrane Kits

Item	*Color*	*Material*	*Description*
5913	Yellow	1.25 mil polyethylene	Faster response time and less flow dependence than traditional Teflon® membranes
5914	Blue	2.0 mil polyethylene	Less flow dependence than 1.25 mil but somewhat slower response

Polarographic Membrane Kits

Item	*Color*	*Material*	*Description*
5908	Yellow	1.25 mil polyethylene	Faster response time and less flow dependence than traditional Teflon® membranes
5909	Blue	2.0 mil polyethylene	Less flow dependence than 1.25 mil but somewhat slower response

Selecting a Dissolved Oxygen Membrane

Membrane Type	*Flow Dependence After 4 Minutes*	*Typical Response Time to 95%*
5913, 5908 - Yellow	25%	8 seconds
5914, 5909 – Blue	18%	17 seconds

AUTO STABLE

Auto Stable utilizes preset values to indicate when a reading is stable. The preset values are adjustable in the System Setup menu. The user can input a % change in dissolved oxygen readings (0.0 to 1.9) over 'x' amount of time in seconds (3-19).

Highlight **Auto Stable** and press enter to expand the submenu. Use the up or down arrow keys to highlight the DO% Change or seconds (secs) input field, then press enter to make the highlighted field adjustable. Use the up and down arrow keys to adjust the selected value, then press enter to confirm changes. Once you have confirmed any changes, highlight the **ESC-Exit** box and press enter to close the Auto Stable submenu.

To disable Auto Stable, set the DO% Change input to 0.0.

When Auto Stable is enabled, a AS will display next to the dissolved oxygen value on the run screen and blink during stabilization. When the dissolved oxygen value has stabilized based on the Auto Stable settings, the AS will display steadily and the instrument will beep twice if Audio is turned on.

DO UNITS

Highlight **DO Units** and press enter to open a submenu that will allow you to select the dissolved oxygen units displayed on the run screen. Highlight the desired unit(s) and press enter to enable or disable. An enabled dissolved oxygen unit will have an 'X' in the box next to it. Highlight the **ESC-Exit** box and press enter to save any changes and to close the DO units submenu.

There are three options for displaying dissolved oxygen:

- **mg/L** will show DO readings in milligrams per liter on a scale from 0 to 50 mg/L.
- **ppm** (parts per million) is equivalent to mg/L and will show the DO reading on a scale from 0 to 50 ppm.
- % will show DO readings in a percent scale from 0 to 500%. This value will be expressed %L when DO Local% is enabled.

Both % or %L and mg/L or ppm can be displayed simultaneously on the screen.

TEMPERATURE UNITS

Highlight **Temperature Units** and press enter to open a submenu that will allow you to change the temperature units displayed in the run screen. Highlight the desired unit (Celsius or Fahrenheit) and press enter to enable. The enabled temperature unit will have an 'X' in the box next to it. Only one unit may be enabled at a time. Highlight the **ESC-Exit** box and press enter to save any changes and to close the Temperature Units submenu.

PRESSURE UNITS

Highlight **Pressure Units** and press enter to open a submenu that will allow you to change the units displayed on the run screen. Highlight the desired unit (mmHg, inHg, mbar, psi, or kPa) and press enter to enable. The enabled pressure unit will have an 'X' in the box next to it. Only one unit may be enabled at a time. Highlight the **ESC-Exit** box and press enter to save any changes and to close the Pressure Units submenu.

LANGUAGE

Highlight **Language** and press Enter to open a submenu that will allow you to change the language. Highlight the desired language (English, Spanish, German, or French) and press enter to enable. The enabled language will have an 'X' in the box next to it. Highlight **ESC-Exit** box and press enter to save any changes and to close the Language submenu.

The text in the boxes along the bottom of the run screen will always be displayed in English regardless of the language enabled in the System Setup menu.

AUTO SHUTOFF

Auto Shutoff allows you to set the instrument to turn off automatically after a period of time. Use the up or down arrow keys to highlight **Auto Shutoff**, then press enter to open the submenu. Press enter while the minute field is highlighted to make it adjustable. Next, use the up and down arrow keys to adjust the shut off time from 0 to 60 minutes. Press enter to confirm and save the new shutoff time. Highlight **ESC-Exit** box, then press enter to close the Auto Shutoff submenu.

To disable Auto Shutoff, set the Time in Minutes to 0 (zero).

RESETTING THE SYSTEM SETUP MENU TO FACTORY DEFAULT

To reset the Pro20 settings to factory default, press the down arrow key until the **Reset** - ↺ box is highlighted, then press enter. The instrument will ask you to confirm the reset. Highlight **Yes** and press enter to continue with the reset or highlight **No** and press enter to cancel the reset. A Factory Reset will not affect data saved in the unit's memory.

The following will be set in the Pro20 after performing a factory reset:

Parameter	*Reset Defaults*
Temperature Units	°C
Dissolved Oxygen Units	mg/L and %
Pressure Units	mmHg
Dissolved Oxygen Sensor Type	Last Setting Confirmed
Membrane Type	Last Setting Confirmed
Salinity Compensation Value	0.0 ppt
DO Local%	Off
One Touch Cal	On
Display Contrast	Set to mid range
Auto Shutoff	30 minutes
Auto Stable	Off (0.0 % Change and 10 secs)
LDS (Last Digit Suppression)	Off
Audio	On
Language	English
Dissolved Oxygen Calibration	Reset to factory default, 100% for enabled membrane and sensor*
Barometer Calibration	Reset to factory default*

*It is recommended to perform a barometer and dissolved oxygen calibration after performing a reset.

EXITING THE SYSTEM SETUP MENU

To exit the System Setup menu, press the down arrow key until the **ESC - Exit** box is highlighted, then press enter  to return to the run screen.

CALIBRATION

TEMPERATURE

All cable assemblies have built-in, temperature sensors. Temperature calibration is not required nor is it available.

BAROMETER

The barometer in the Pro20 is calibrated at the factory. The barometer reading must be accurate to ensure accurate % calibrations and DO readings. If your barometer requires an adjustment, use the up or down arrow keys to highlight the barometer box on the run screen, then press enter. Next, use the up or down arrow keys to adjust the barometer reading to the **local, true barometric pressure.** Continually depress the up or down arrow keys to change the barometer value more rapidly. Press enter to confirm and save the barometer adjustment.

ⓘ *Do not use a barometer value that is corrected to sea level. Laboratory barometer readings are usually "true" (uncorrected) values of air pressure and can be used "as is" for barometer calibration. Weather service readings are usually not "true", i.e., they are corrected to sea level, and therefore cannot be used until they are "uncorrected". An approximate formula for this "uncorrection" is:*

True BP = [Corrected BP] – [2.5 * (Local Altitude in ft above sea level/100)]

ⓘ *Although the barometer range is 400.0 to 999.9 mmHg, you will be unable to adjust the value across the entire range. The barometer is very accurate and the instrument will not allow you to adjust the value drastically beyond what it is measuring during calibration.*

DISSOLVED OXYGEN

The Pro20 can be easily calibrated with the press of one key by enabling One Touch Cal in the System Setup menu and following the One Touch Calibration procedure.

Ensure the barometer is reading accurately before performing a One Touch Calibration, DO %, or DO Local% calibration. These calibration procedures use the barometer reading during calibration. If the barometer reading is erroneous during a calibration, your dissolved oxygen values will be inaccurate.

ⓘ *It is not necessary to calibrate in both % and mg/L or ppm. Calibrating in % will simultaneously calibrate mg/L and ppm and vice versa. YSI recommends calibrating dissolved oxygen in % for both ease and accuracy.*

ONE TOUCH CALIBRATION

Perform this calibration procedure when One Touch Cal is enabled in the System Setup menu.

If using a field cable, install the sensor guard onto the probe. Moisten the sponge in the grey calibration/storage sleeve with a small amount of water and install it over the sensor guard. The sleeve should be moist, but should not have excess water that could cause water droplets to get on the membrane. The storage sleeve ensures venting to the atmosphere.

If using the ProBOD sensor/cable assembly, place the probe in 300 ml BOD bottle with a small amount of water (1/8 inch or 0.3 cm). The dissolved oxygen and temperature sensors should not be immersed in water.

If the calibration/storage sleeve is not available, substitute with a chamber of 100% relative humidity, vented to the atmosphere (not completely sealed).

Power the instrument on and wait approximately 5 to 15 minutes for the storage chamber to become completely saturated and to allow the sensor to stabilize if using a Polarographic sensor. If using a Galvanic sensor, wait approximately 5 to 10 minutes for the chamber to become completely saturated. Auto Shutoff time should be disabled or set to at least 20 minutes, see System Setup menu for more information on adjusting the Auto Shutoff.

Ensure the barometer reading is accurate. If necessary, perform a barometer calibration.

Press and hold the Calibrate Cal key for 3 seconds. The Pro20 will indicate **Calibrating %DO** on the display and automatically calibrate the sensor to the barometer and salinity correction values. This may take up to 2 minutes depending on the age of the sensor and membrane. You may press the Cal key at this time to cancel the calibration.

Calibration Successful will display for a few seconds to indicate a successful calibration and then the instrument will return to the run screen.

If the calibration is unsuccessful, an error message will display on the screen. Press the Cal key to exit the calibration error message and return to the run screen. See the Troubleshooting guide for possible solutions.

CALIBRATING IN PERCENT (DO %)

Perform this calibration procedure when One Touch Cal is disabled in the System Setup menu.

Prepare a 100% humid environment for the sensor as described in the previous calibration section.

Power the instrument on and wait approximately 5 to 15 minutes for the storage chamber to become completely saturated and to allow the sensor to stabilize if using a Polarographic sensor. If using a Galvanic sensor, wait approximately 5 to 10 minutes for the chamber to become completely saturated. Auto Shutoff time should be disabled or set to at least 20 minutes, see System Setup menu for more information on adjusting the Auto Shutoff.

Ensure the barometer reading is accurate. If necessary, perform a barometer calibration.

Press and hold the Calibrate Cal key for 3 seconds. Highlight % and press enter. The Pro20 will display the current DO% and temperature readings along with the % calibration value. The % calibration value is based on the barometer reading.

Wait at least 3 seconds, then, once the DO% and temperature readings are stable, press enter to complete the calibration. Or, press the Cal key to cancel the calibration.

Calibration Successful will display for a few seconds to indicate a successful calibration and then the instrument will return to the run screen.

If the calibration is unsuccessful, an error message will display on the screen. Press the Cal key to exit the calibration error message and return to the run screen. See the Troubleshooting guide for possible solutions.

CALIBRATING IN PERCENT (DO LOCAL% ENABLED)

Perform this calibration procedure when DO Local% is enabled in the System Setup menu.

Prepare a 100% humid environment for the sensor as described in the One Touch Calibration section.

Power the instrument on and wait approximately 5 to 15 minutes for the storage chamber to become completely saturated and to allow the sensor to stabilize if using a Polarographic sensor. If using a Galvanic sensor, wait approximately 5 to 10 minutes for the chamber to become completely saturated. Auto Shutoff time should be disabled or set to at least 20 minutes, see System Setup menu for more information on adjusting the Auto Shutoff.

Ensure the barometer reading is accurate. If necessary, perform a barometer calibration.

Press and hold the Calibrate Cal key for 3 seconds. %Local will be automatically highlight, press enter. The Pro20 will display the current DO% and temperature readings along with the % calibration value. The % calibration value will always be 100% for DO Local%.

Wait at least 3 seconds, then, once the DO% and temperature readings are stable, press enter to complete the calibration. Or, press the Cal key to cancel the calibration.

Calibration Successful will display for a few seconds to indicate a successful calibration and then the instrument will return to the run screen.

If the calibration is unsuccessful, an error message will display on the screen. Press the Cal key to exit the calibration error message and return to the run screen. See the Troubleshooting guide for possible solutions.

CALIBRATING IN MG/L

Power the instrument on and place the sensor into a sample that has been titrated to determine the dissolved oxygen concentration. Continuously stir or move the probe through the sample at a rate of at least ½ foot per second (16 cm per second) during the entire calibration process. A stir plate may be helpful in this calibration.

Allow the dissolved oxygen and temperature readings to stabilize. This may take 5 to 15 minutes, depending on the age of the instrument, type of sensor, and condition of the sensor.

Press the Calibrate Cal key. Highlight **mg/L** and press enter.

Use the up and down arrow keys to adjust the mg/L reading to the value of the titrated sample. Press enter to confirm the value and calibrate or press the Cal key to cancel the calibration.

Calibration Successful will display for a few seconds to indicate a successful calibration and then the instrument will return to the run screen.

If the calibration is unsuccessful, an error message will display on the screen. Press the Cal key to exit the calibration error message and return to the run screen. See the Troubleshooting guide for possible solutions.

SALINITY COMPENSATION CALIBRATION

The Pro20 uses a user inputted salinity value in ppt (parts per thousands) to compensate dissolved oxygen mg/L values. The salinity compensation value entered in the Pro20 should be the salinity value of the water you are testing.

To adjust the salinity compensation value, use the up or down arrow keys to highlight the salinity box on the run screen, and then press enter (Figure 10). Next, use the up or down arrow keys to adjust the salinity compensation value to the salinity of the water you are testing. You may enter a value between 0.0 and 70.0 parts per thousand (ppt). Press enter to confirm and to save the new salinity compensation value.

The salinity compensation value can be adjusted any time without the need to recalibrate dissolved oxygen.

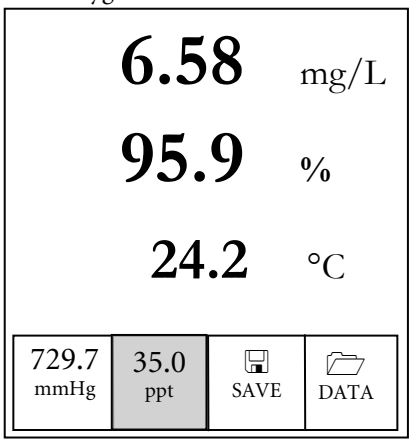


Figure 10, Salinity box highlighted.

TAKING MEASUREMENTS

Before taking measurements, be sure the instrument has been calibrated to ensure the most accurate readings. Turn the instrument on and wait 5-15 minutes if using a polarographic sensor. If using a field cable/sensor, install the sensor guard to protect the sensor and membrane. Place the probe in the sample to be measured and give the probe a quick shake to release any air bubbles. Allow the temperature readings to stabilize. Next, stir the probe in the sample to overcome the stirring dependence of the dissolved oxygen sensor. You must provide at least 6 inches (16 cm) per second of water movement. Once the values plateau and stabilize you may record the measurement and/or store the data set. The dissolved oxygen reading will drop over time if stirring is ceased. If placing the DO sensor into a stream or fast flowing waters it is best to place it perpendicular to the flow and NOT facing into the flow.

If using the DO sensor in an aeration tank/basin it is helpful to make sure bubbles do not burst on the membrane. This may cause unstable readings to occur. You should be able to prevent this by pointing the sensor upwards so it's facing the sky and twist tying, zip tying, or rubber banding the bulkhead to the cable. Essentially making a simple curve to the cable without bending or breaking the cable will allow you to lower the sensor into the aeration tank while the sensor points skyward and the bubbles are no longer bursting on the membrane surface.

SAVING AND VIEWING DATA

The Pro20 can store 50 data sets in non-volatile memory for later viewing. A data set includes the values currently on the display, i.e. temperature in Celsius or Fahrenheit and dissolved oxygen in % and/or mg/L or ppm. Each data point is referenced with a data set number, 01 through 50.

ⓘ *The Pro20 can not communicate to a PC via a Pro Plus communications saddle. Connecting the Pro20 to a communication saddle may cause erratic instrument behavior.*

SAVING DATA

From the run screen, use the up or down arrow keys to highlight the Save box and press enter to save the current readings. The instrument will indicate the data set is saved and display the saved data set's number (Figure 11).

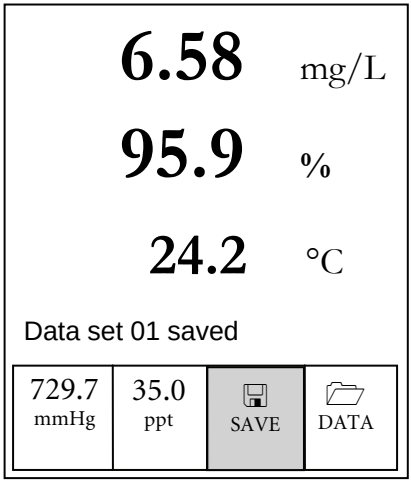


Figure 11, Data set saved

The instrument will display 'Memory Full' if all 50 data sets have been saved and you attempt to save another data set.

VIEWING AND ERASING SAVED DATA – DATA MODE

Data mode allows you to view and erase saved data. From the run screen, use the up or down arrow keys to highlight Data and press enter to access data mode. Note that the function boxes at the bottom of the display are different in data mode (Figure 12).

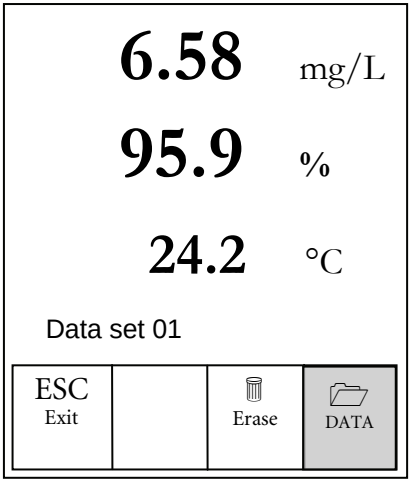


Figure 12, Data mode

VIEWING DATA

Once in data mode, use the up and down arrow keys to view saved data sets in sequential order or press enter to access the bottom functions. After accessing the bottom functions, highlight the Data box and press enter to regain access to viewing data. The data set that is displayed will be indicated by the data set number, 01 through 50.

ERASING DATA

While viewing saved data, press the enter key to access the function boxes at the bottom of the display. Next, use the up or down arrow keys to highlight Erase, then press enter. The instrument will give you the option to erase one or all data sets (Figure 13).

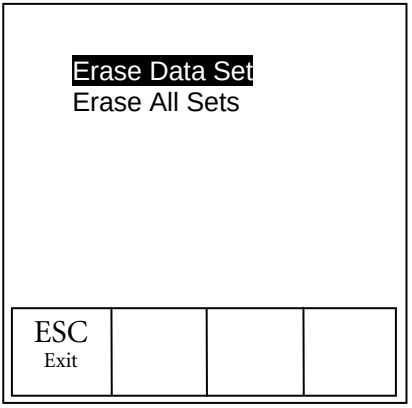


Figure 13, erasing data

Use the up or down arrow key to select Erase Data Set, Erase All Sets, or the ESC-Exit function box, then press enter to confirm.

Select ESC-Exit and press enter to exit erase mode without erasing any data.

Select Erase Data Set and press enter to erase the data set that was displayed before entering erase mode. For example, if data set 12 was displayed before entering erase mode, and Erase Data Set is selected, Data Set 12 will be erased from memory and the sets AFTER that number will move up to keep them sequential. So, if there were 15 records and number 12 is erased then 13 becomes 12, 14 becomes 13, and 15 becomes 14. The instrument will return to data mode after erasing one data set.

Select Erase All Data Sets and press enter to clear the Pro20 memory and return to data mode.

EXITING DATA MODE

While in Data mode, press enter to access the bottom functions. Next, highlight the ESC-Exit box and press enter to return to the run screen.

PRINCIPLES OF OPERATION

The polarographic sensor consists of a silver body as the anode and a circular gold cathode embedded in the end. The galvanic sensor consists of a zinc anode and silver cathode. The polarographic sensor requires an applied voltage for operation while the galvanic sensor electrode potentials are dissimilar enough to reduce oxygen with applied voltage.

Both sensors have a thin semi-permeable membrane, stretched over the sensor, which isolates the electrodes from the environment, while allowing gases to enter. In operation, this end of the sensor is filled with a solution of electrolyte containing a small amount of surfactant to improve wetting action.

When a polarizing voltage is applied to the polarographic sensor electrodes, oxygen that has passed through the membrane reacts at the cathode causing a current to flow. This same reaction takes place with the galvanic sensor without the applied voltage.

For both polarographic and galvanic DO sensors, oxygen diffuses through the membrane at a rate proportional to the oxygen pressure difference across it. Since oxygen is rapidly consumed at the cathode, it can be assumed that the oxygen pressure inside the membrane is zero. Hence, the amount of oxygen diffusing through the membrane is proportional to the absolute pressure of oxygen outside the membrane. If the oxygen pressure increases, more oxygen diffuses through the membrane and more current flows through the sensor. A lower pressure results in less current.

CARE, MAINTENANCE, AND STORAGE

This section describes the proper procedures for care, maintenance and storage of the sensors. The goal is to maximize their lifetime and minimize down-time associated with improper sensor usage.

GENERAL MAINTENANCE

GENERAL MAINTENANCE - O-RINGS

The instrument utilizes o-rings as seals to prevent water from entering the battery compartment and the sensor ports. Following the recommended procedures will help keep your instrument functioning properly.

If the o-rings and sealing surfaces are not maintained properly, it is possible that water can enter the battery compartment and/or sensor ports of the instrument. If water enters these areas, it can severely damage the battery terminals or sensor ports causing loss of battery power, false readings and corrosion to the sensors or battery terminals. Therefore, when the battery compartment lid is removed, the o-ring that provides the seal should be carefully inspected for contamination (e.g. debris, grit, etc.) and cleaned if necessary.

The same inspection should be made of the o-rings associated with the dissolved oxygen sensor connector when it is removed. If no dirt or damage to the o-rings is evident, then they should be lightly greased without removal from their groove. However, if there is any indication of damage, the o-ring should be replaced with an identical o-ring. At the time of o-ring replacement, the entire o-ring assembly should be cleaned.

To remove the o-rings:

Use a small, flat-bladed screwdriver or similar blunt-tipped tool to remove the o-ring from its groove. Check the o-ring and the groove for any excess grease or contamination. If contamination is evident, clean the o-ring and nearby plastic parts with lens cleaning tissue or equivalent lint-free cloth. Alcohol can be used to clean the plastic parts, but use only water and mild detergent on the o-ring itself. Also, inspect the o-rings for nicks and imperfections.

Using alcohol on o-rings may cause a loss of elasticity and may promote cracking.

Do not use a sharp object to remove the o-rings. Damage to the o-ring or the groove may result.

Before re-installing the o-rings, make sure to use a clean workspace, clean hands, and avoid contact with anything that may leave fibers on the o-ring or grooves. Even a very small amount of contamination (hair, grit, etc.) may cause a leak.

To re-install the o-rings:

Place a small amount of o-ring grease between your thumb and index finger. (More grease is NOT BETTER!)

Draw the o-ring through the grease while pressing the fingers together to place a very light covering of grease to the o-ring. Place the o-ring into its groove making sure that it does not twist or roll.

Use the previously grease-coated finger to once again lightly go over the mating surface of the o-ring.

Do not over-grease the o-rings. The excess grease may collect grit particles that can compromise the seal. Excess grease can also cause the waterproofing capabilities of the o-ring to diminish, potentially causing leaks. If excess grease is present, remove it using a lens cloth or lint-free cloth.

GENERAL MAINTENANCE – DO SENSOR PORT

It is important that the entire sensor connector end be dry when installing, removing, or replacing. This will prevent water from entering the port. Once a sensor is removed, examine the connector inside the port. If any moisture is present, use compressed air to completely dry the connector or place directly in front of a steady flow of fresh air. If the connector is corroded, contact YSI Technical Support or the Authorized dealer where you purchased the instrument.

ⓘ *Remove sensors upside down (facing the ground) to help prevent water from entering the port upon removal.*

SENSOR MAINTENANCE

SENSOR MAINTENANCE - TEMPERATURE

You must keep the temperature portion of the sensor free of build up. Other than that, the sensor requires no maintenance. A toothbrush can be used to scrub the temperature sensor if needed.

SENSOR MAINTENANCE – DISSOLVED OXYGEN

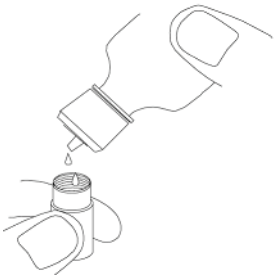
Membrane Cap Installation

The DO sensor (Polarographic and Galvanic) is shipped with a dry, protective red cap that will need to be removed before using. Remove the protective cap or used membrane cap and replace it with a new membrane cap following these instructions:

Remove the sensor guard to access the sensor tip.

Remove the protective red cap or unscrew and remove any old membrane cap by holding the sensor when unscrewing the membrane cap and discard.

Thoroughly rinse the sensor tip with distilled or DI water.



Fill a new membrane cap with O_2 sensor solution that has been prepared according to the directions on the bottle. Be very careful not to touch the membrane surface.

Lightly tap the side of the membrane cap to release bubbles that may be trapped.

Thread the membrane cap onto the sensor. It is normal for a small amount of electrolyte to overflow.

Replace the sensor guard.

Polarographic Sensors – Model # 605203

The KCl (potassium chloride) solution and the membrane cap should be changed at least once every 30 days during regular use. In addition, the KCl solution and membrane should be changed if (a) bubbles are visible under the membrane; (b) significant deposits of dried electrolyte are visible on the membrane; and (c) if the sensor shows unstable readings or other sensor-related symptoms.

During membrane changes, examine the gold cathode at the tip of the sensor and the silver anode along the shaft of the sensor. If either the silver anode is black in color or the gold cathode is dull, the sensor *may* need resurfaced using the fine sanding disks included in the membrane kit. Do not sand the electrode every membrane change as this is not *routine* maintenance. In fact, visually, the anode may appear tarnished and operate just fine. YSI recommends using the 400 grit wet/dry sanding disks after a membrane change if the sensor has difficulty stabilizing or calibrating.

To clean and resurface the sensor, follow the instructions below.

Gold Cathode

For correct sensor operation, the gold cathode must be textured properly. It can become tarnished or plated with silver after extended use. Never use chemicals or abrasives that have not been recommended or supplied by YSI.

First dry the sensor tip completely with lens cleaning tissue. Wet a sanding disc and place it face up in the palm of you hand. Next, with your free hand, hold the sensor in a vertical position, tip down. Place the sensor tip directly down on the sanding disc and twist it in a circular motion to sand the gold cathode. The goal is to sand off any build-up and to lightly scratch the cathode to provide a larger surface area for the O_2 solution under the membrane. Usually, 3 to 4 twists of the sanding disc are sufficient to remove deposits and for the gold to appear to have a matte finish. Rinse thoroughly and wipe the gold cathode with a wet paper towel before putting on a new membrane cap. If the cathode remains tarnished, contact YSI Technical Support or the Authorized dealer where you purchased the instrument.

Silver Anode

After extended use, a thick layer of Silver Chloride (AgCl) builds up on the silver anode reducing the sensitivity of the sensor. The anode must be cleaned to remove this layer and restore proper performance. The cleaning can be chemical and/or mechanical:

Chemical cleaning: Remove the membrane cap and rinse the sensor with deionized or distilled water. Soak the sensing section of the sensor in a 14% ammonium hydroxide solution for 2 to 3 minutes or in a 3% ammonia solution overnight for 8-12 hours (most household ammonia cleaners are typically around 3%). Rinse heavily in cool tap water followed by a thorough rinsing with distilled or deionized water. The anode should then be thoroughly wiped with a wet paper towel to remove the residual layer from the anode. You can smell the tip of the sensor to help ensure all the ammonia has been rinsed off. Trapping residual ammonia under the new membrane cap can quickly tarnish the electrode and/or give false readings.

Chemical cleaning should be performed as infrequently as possible. First attempt a membrane change and recalibrate. If a new membrane does not resolve the problem, then proceed with cleaning.

Mechanical cleaning: In order to sand the silver anode along the shaft of the sensor, simply hold the sensor in a vertical position. Wet the sanding disc and gently wrap it around the sensor and twist it a few times to lightly sand the anode (the goal is to simply sand off any build-up without scratching or removing layers of the anode itself). Usually, 3 to 4 twists of the sanding disc are sufficient to remove deposits. However, in extreme cases, more sanding may be required to regenerate the original silver surface.

After completing the sanding procedure, repeatedly rinse the electrode with clean water and wipe with lens cleaning tissue to remove any grit left by the sanding disc. Thoroughly rinse the entire tip of the sensor with distilled or deionized water and install a new membrane.

***IMPORTANT**: Be sure to: (1) Use only the fine sanding discs provided and (2) Sand as mentioned in the above procedures. Not adhering to either of these instructions can damage the electrodes.*

If this procedure is unsuccessful, as indicated by improper sensor performance, contact YSI Technical Support or the Authorized dealer where you purchased the instrument.

Galvanic Sensors – Model # 605202

We recommend that the Sodium Chloride (NaCl) solution and the membrane cap be changed at least once every 60 days during regular use. In addition, the NaCl solution and membrane should be changed if (a) bubbles are visible under the membrane; (b) significant deposits of dried electrolyte are visible around the membrane; and (c) if the sensor shows unstable readings or other sensor-related symptoms.

The Galvanic dissolved oxygen sensor is continuously reducing oxygen even when the display of the instrument is not active. This factor allows the sensor to be used with no warm-up period as soon as the instrument is powered on (instant on DO). However, because the sensor is "on" all the time, some solid from the oxidation of the zinc anode will form in the electrolyte within 1-2 weeks of activation. Small amounts of the solid will generally cause no performance problems, but excessive amounts may result in jumpy dissolved oxygen readings. The rate of solid formation is dependent on the type of membrane installed. The formation of solids based on membrane type typically form more rapidly with 5913 (1.25 mil PE), and less rapid with 5914 (2 mil PE).

The Galvanic DO sensor solution will appear milky white after use but will NOT affect the accuracy of the sensor unless there is excessive build up. The color change is acceptable and normal as long as DO readings remain stable.

At the time the membrane cap is changed, YSI recommends that you rinse the anode (silver shaft of the sensor) with purified water and wipe with a clean paper towel. If white deposits are evident on the anode after cleaning, YSI recommends that you remove this material by sanding the anode with the sandpaper disk included in the membrane kit. Follow the "Mechanical Cleaning" instructions under the Polarographic Silver Anode section.

IMPORTANT: *Be sure to: (1) Use only the fine sanding discs provided and (2) Sand as mentioned in the above procedures. Not adhering to either of these instructions can damage the electrodes.*

WARNING: *DO NOT PERFORM THE POLAROGRAPHIC CHEMICAL CLEANING ON A GALVANIC SENSOR.*

If this procedure is unsuccessful, as indicated by improper sensor performance, contact YSI Technical Support or the Authorized Dealer where you purchased the instrument.

SENSOR STORAGE

SHORT TERM STORAGE

The instrument is supplied with a grey calibration/storage sleeve that slides over the probe guard. The sleeve is used for short-term storage (less than 30 days). Be sure to keep a small amount of moisture (tap water) on the sponge in the sleeve during storage. This is simply done to maintain a 100% water saturated air environment which is ideal for short-term sensor storage. The sensors should not be submersed in water. The intent is to create a humid air storage environment.

LONG TERM STORAGE

Dissolved oxygen sensors (Polarographic and Galvanic) should always be stored long term in a dry state. When storing for more then 30 days, remove the membrane cap and thoroughly rinse the sensor. Once the sensor has been rinsed either blow it dry with compressed air or allow to air dry completely. Use a clean, dry new membrane cap to screw over the sensor to keep it dry and to protect the anode and cathode.

After storing the sensor for a long period of time it is necessary to "condition" the sensor by putting a new membrane with electrolyte solution on the sensor.

Long Term Storage Temperature: -5 to 70°C (23 to 158°F)

ERROR MESSAGES

Symptom	*Possible Solution*
Instrument will not calibrate; instrument displays "Calibration Over", "Calibration Under", or "Unstable Reading" during calibration.	1) Verify barometer reading 2) Verify correct sensor and membrane type selection in the System Setup menu. 3) Calibration sleeve may not be 100% water saturated, ensure sponge is moisten 4) Ensure adequate sample movement if performing mg/L or ppm calibration 5) Allow sufficient stabilization time for dissolved oxygen and temperature AND wait at least 3 seconds before confirming a DO % or DO %Local calibration 6) Replace membrane and electrolyte 7) Clean sensor electrodes 8) Return system for service
Instrument will not turn on, a battery symbol  appears, or "Critical Shutdown" displays on the screen.	1) Low battery voltage, replace batteries 2) Batteries installed incorrectly, check battery polarity 3) Return system for service
Barometer reads over/under, Dissolved Oxygen and Temperature display Over/Undr, and pressing Cal key results in a Barometric Pressure Over/Undr message.	1) Barometer failure, return system for service

Symptom	*Possible Solution*
Instrument readings are inaccurate.	1) Verify correct sensor/membrane type selection in the System Setup menu. 2) Verify calibration, barometer reading, and salinity settings are correct and recalibrate. 3) Verify accurate temperature readings. 4) Sample temperature is over 45 °C, the temperature compensation range. 5) Probe may not have been in 100% water saturated air during calibration procedure. Moisten sponge in calibration sleeve and recalibrate. 6) Replace membrane and electrolyte, recalibrate. 7) Clean sensor electrodes. 8) Return system for service.
Dissolved Oxygen values display Over or Undr on run screen.	1) Verify correct sensor/membrane type selection in the System Setup menu. 2) If using a polarographic sensor, allow instrument to warm up for 5 – 15 minutes before use. 3) Sample O_2 concentration is more than 50 mg/L or 500%, or less than –0.02 mg/L or -0.3%. 4) Verify barometer and salinity settings are correct and recalibrate. 5) Verify accurate temperature readings. 6) Replace membrane and electrolyte. Recalibrate. 7) Clean sensor electrodes. 8) Return system for service.

Temperature values display Over or Undr on run screen.	1) Sample temperature is less than -5° C or more than +55°C . Increase or decrease the sample temperature to bring within the allowable range. 2) Return system for service.

SPECIFICATIONS

Parameter	Range	Resolution	Accuracy
Temperature	-5 to 55 °C *	0.1 °C	± 0.3 °C
	23 to 113 °F	0.1 °F	± 0.6 °F
Dissolved Oxygen	0 to 200% air saturation	1% or 0.1%, user selectable	± 2% of the reading or 2% air saturation, whichever is greater
	200 to 500% air saturation	1% or 0.1%, user selectable	± 6% of the reading
	0 to 20 mg/L	0.1 or 0.01 mg/L, user selectable	±2% of the reading or 0.2 mg/L, whichever is greater
	20 to 50 mg/L	0.1 or 0.01 mg/L, user selectable	±6% of the reading
Barometer	400.0 to 999.9 mmHg**	0.1 mmHg	± 5 mmHg within 5 °C of calibration temperature***

* Automatic Dissolved Oxygen Temperature Compensation Range is -5 to 45 °C

**Available barometer units include: mmHg, inHg, mbars, psi, or KPa

***For operating temperatures below 10 °C or above 40 °C, the barometer must be recalibrated to maintain accuracy specification.

ACCESSORIES/PART NUMBERS

Part Number	*Description*
6050020	Pro20
60520-1, -4, -10, -20, or -30	1, 4, 10, 20, or 30-meter cable with temperature and a port for Dissolved Oxygen
605202	Galvanic Dissolved Oxygen sensor
605203	Polarographic Dissolved Oxygen sensor
605780	ProBOD, Self-Stirring BOD sensor
603077	Flow cell – For use with any *Professional Series* Instrument
603056	Flow cell mounting spike
603075	Carrying case, soft-sided
603074	Carrying case, hard-sided
603069	Belt clip
063517	Ultra clamp
063507	Tripod
601205	Grease, o-ring
603062	Cable management kit
605978	Weight, sensor/cable, 4.9 oz
063019	Weight, sensor/cable, 24 oz, 3”
063020	Weight, sensor/cable, 51 oz, 6”
603070	Shoulder strap
5908	1.25, Yellow, Polyethylene membrane kit for Polarographic sensors
5909	2.0, Blue, Polyethylene membrane kit for Polarographic sensors
5913	1.25, Yellow, Polyethylene membrane kit for Galvanic sensors
5914	2.0, Blue, Polyethylene membrane kit for Galvanic sensors

DECLARATION OF CONFORMITY

Manufacturer:	YSI Incorporated 1725 Brannum Lane Yellow Springs, Ohio 45387 USA
Product Name:	Pro20
Model Numbers:	
Instrument:	Pro20 (6050020)
Cables:	60520, 605780
Sensors:	605202, 605203
Conforms to the following:	
Directives:	EMC Directive 2004/108/EC
Harmonized Standards:	EN5011 :1998, A1:1999 Class B equipment EN61000-4-2 (ESD) EN61000-4-3 (RF radiated immunity) EN61000-4-4 (EFT) EN61000-4-6 (RF conducted immunity) EN61000-4-8 (50 Hz Radiated Susceptibility) FCC Part 15, Subpart B, Sections 15.107a & 15.109a, Class B
Supplementary Information:	This device complies with the requirements of the EMC Directive 2004/108/EC, and carries the CE mark accordingly. All performance met the continuous unmonitored operation criteria as follows: 1. ESD, IEC 61000-4-2, Performance Criterion B 2. EM, IEC 61000-4-3, Performance Criterion A 3. Burst, IEC 61000-4-4, Performance Criterion B 4. Surge, IEC 61000-4-5, Performance Criterion B 5. Conducted RF, IEC 61000-4-6, Performance Criterion A 6. Voltage Interrupts, IEC 61000-4-11, Performance Criterion B
Authorized EU Representative	YSI Hydrodata Unit 8, Business Centre West, Avenue 1 Letchworth, Hertfordshire, SG6 2HB UK

RECYCLING

YSI takes seriously the commitment to reducing our environmental footprint in our course of doing business. Even though materials reduction is the ultimate goal, we know there must be a concerted effort to responsibly deal with materials after they've served a long, productive life-cycle.

YSI's recycling program ensures that old equipment is processed in an environmentally friendly way, reducing the amount of materials going to landfills.

Printed Circuit Boards are sent to facilities that process and reclaim as much material for recycling as possible.

Plastics enter a material recycling process and are not incinerated or sent to landfills.

Batteries are removed and sent to specialist battery recyclers for dedicated metals.

When the time comes for you to recycle, follow the easy steps as outlined at www.ysi.com/recycle.

CONTACT INFORMATION

ORDERING & TECHNICAL SUPPORT

Telephone: (800) 897-4151
(937) 767-7241
Monday through Friday, 8:00 AM to 5:00 PM ET

Fax: (937) 767-9353 (orders)
(937) 767-1058 (technical support)

Email: environmental@ysi.com

Mail: YSI Incorporated
1725 Brannum Lane
Yellow Springs, OH 45387
USA

Internet: www.ysi.com

When placing an order please have the following information available:

YSI account number (if available)	Name and Phone Number
Model number or brief description	Billing and shipping address
Quantity	Purchase Order or Credit Card

SERVICE INFORMATION

YSI has authorized service centers throughout the United States and Internationally. For the nearest service center information, please visit www.ysi.com and click 'Support' or contact YSI Technical Support directly at 800-897-4151.

When returning a product for service, include the Product Return form with cleaning certification. The form must be completely filled out for an YSI Service Center to accept the instrument for service. The Product Return form may be downloaded at www.ysi.com and clicking on the 'Support" tab.

Item # 605597
Rev B
Drawing # A605597
October 2008