REMEDIAL PLANNING ACTIVITIES
AT SELECTED UNCONTROLLED
HAZARDOUS SUBSTANCE DISPOSAL SITES
IN REGION II
(ARCS II)

U.S. EPA CONTRACT NO.: 68-W9-0024

PROJECT OPERATIONS PLAN
FOR
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
CHEMSOL, INC. SITE
PISCATAWAY, NEW JERSEY

APPENDICES

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APPENDIX A

Standard Operating Procedures

MALCOLM PIRNIE, INC.

Field Standard Operating Procedures

TABLE OF CONTENTS

Title

1.0 OBJECTIVE

This guideline provides instructions for sample packaging and shipping in accordance with United States Department of Transportation (DOT) regulations.

2.0 APPLICABILITY

The guidelines is applicable to all samples taken from uncontrolled hazardous substance sites for analysis at laboratories away from the site.

3.0 DEFINITIONS

Carrier--A person or firm engaged in the transportation of passengers or property.

n.o.s.--Not otherwise specified.

n.o.i.--Not otherwise indicated.

ORM--Other regulated material.

DOT Classifications for Hazardous Materials--The following classifications, set forth by the DOT in the Code of Federal Regulations (49 CFR 173.2):

1. Radioactive material
2. Poison A
3. Flammable liquid
4. Nonflammable gas
5. Flammable liquid
6. Oxidizer
7. Corrosive material (liquid)
8. Poison B

9. Corrosive material (solid)

10. Irritating material

11. Combustible liquid (in containers having capacities exceeding 110 gal)

12. ORM-B

13. ORM-A

14. Combustible liquid (in containers having capacities of 110 gal or less)

15. ORM-E

4.0 GUIDELINES

Samples collected at uncontrolled hazardous substance facilities usually have to be transported elsewhere for analysis. Samples must be transported to protect their integrity, as well as to protect against any detrimental effects from leakage or breakage. Regulations for packaging, marking, labeling, and shipping hazardous materials and wastes are promulgated by the United States Department of Transportation and described in the Code of Federal Regulations (49 CFR 171 through 177, in particular 172.402h, Packages Containing Samples).

4.1 RESPONSIBILITIES

The Project Manager or team leader is responsible for determining that samples are properly packaged and shipped. Sampling personnel and shippers (if used) are responsible for implementing the packaging and shipping requirements. The chain-of-custody procedures and requirements are described SOP 2.

4.2 EQUIPMENT

The following equipment is used in packaging and shipping samples:

1. Samples bottles, provided by designated laboratories

2. Polyethylene bags, 2 mil or thicker

3. Metal paint cans, 1 gal

4. Packing material, vermiculite, bubble pack or similar noncombustible packing material

5. Picnic coolers or ice chests, preferably metal, capable of withstanding impact caused by a 4-ft drop

4.3 PACKAGING, MARKING, AND LABELING METHODS

4.3.1 Environmental Samples

Packing

Environmental samples can be packaged following the procedures for samples classified as flammable liquids or flammable solids. See Standard Operating Procedure 6 for details on the collection of environmental samples. Marking, labeling, and shipping papers do not apply.

Environmental samples can also be packaged without being placed inside metal cans as required for flammable liquids. For example, sample containers properly identified and with a sealed lid can be enclosed in sealed polyethylene bags and packed in metal picnic cooler-type containers. Sufficient noncombustible, absorbent cushioning material such as "bubble pack" must be used to minimize the possibility of sample container breakage. Ice or "blue ice" is added to low-concentration samples. To further reduce the possibility of leakage, the sample container, and the sample bottles and absorbent material can be placed in a larger bag that is also sealed.

Marking and Labeling

A complete sample identification tag or label must be affixed to sample containers. The words "Environmental Sample" should be marked on the outside container.

No DOT marking or labeling is required.

Shipping Papers

No DOT shipping papers are required for environmental samples. However, the appropriate chain-of-custody forms must be included with the shipment.

Transportation

There are no DOT restrictions on the mode of transportation for environmental samples.

4.3.2 Unanalyzed Hazardous Waste Site Samples, Excluding Those from Closed Containers

The procedures to be used to pack, mark, and ship hazardous waste samples are presented below. A checklist summarizing these procedures has been developed and is provided as Table 3-1. This checklist should always be consulted prior to sample shipment to ensure that all sample-handling requirements are satisfied.

Packaging

Packaging procedures are as follows:

1. Collect samples in accordance with the procedures given in SOP 6 of this manual. Allow sufficient ullage (approximately 10 percent by volume) so container is not liquid-full at 130 F. If a solid material is being collected, the container plus contents shall not exceed 1 lb net weight.

2. Attach properly completed sample identification tag or a Malcolm Pirnie, Inc. sample label to sample container.

3. Seal sample container and place in 2-mil-thick (or thicker) polyethylene bag (one sample per bag). Tags should be positioned to enable them to be read through bag.

4. Place sealed bag inside a metal can with incombustible, absorbent cushioning material (e.g., vermiculite or earth) to prevent breakage (one bag per can). Pressure-close the can and sue clips, tape, or other positive means to hold the lid securely, tightly, and effectively.

5. Mark and label this container as indicated below.

6. Place one or more metal cans (or a single 1-gal bottle), surrounded by incombustible packaging material for stability during transport, into a strong outside container, such as a metal picnic cooler or a fiberboard box.

7. Mark and label the outside container and complete shipping papers as described below.

Marking and Labeling

Use abbreviations only where specified. Place the following information (either hand-printed or on preprinted labels) on a metal can (or bottle): laboratory name and address and "Flammable Liquid, n.o.s." (if not liquid, write "Flammable Solid, n.o.s.").* Place the following labels on the outside of the can (or bottle): "Cargo Aircraft Only" and "Flammable Liquid" or, if not liquid, "Flammable Solid." ("Dangerous When Wet" label should be used if the solid has not been exposed to wet environment.)

(NOTE: If the cans are placed in an exterior container, both that container and the inside cans must have the same markings and labels as above. "Laboratory Samples" and "THIS SIDE UP" or "THIS END UP" should also be marked on the top of the outside container, and upward-pointing arrows should be placed on all four sides of the exterior container.)

Shipping Papers

Complete the carrier-provided bill of lading and sign the certification statement. If carrier does not provide these documents, use standard industry form, providing the following information in the order listed (one form may be used for more than one exterior container): "Flammable Liquid, n.o.s." (or "Flammable Solid, n.o.s.," as appropriate); "Cargo Aircraft Only"; "Limited Quantity" or "Ltd. Qty."; "Laboratory Samples"; "Net Weight ____" or "Net Volume____" (of hazardous contents), by item, if more than one metal can is inside an exterior container. The net weight or net volume must be placed just before or just after the "Flammable Liquid, n.o.s." or "Flammable Solid, n.o.s." description.

A chain-of-custody record form (see SOP 2 of this manual) must be properly executed and included in the exterior container.

Unless samples are driven to the laboratory, a team member must accompany shipping container(s) to the transport carrier and, if required, open outside container(s) for freight inspection.

4.3.3 Unanalyzed Hazardous Waste Site Samples Taken From Closed Containers

Slightly different procedures apply to hazardous waste site samples taken from closed containers. The procedures to be followed be site personnel for packaging, marking, and labeling are presented below. They are rarely used and are provided for information only.

This packaging, marking, labeling, and shipping methods provides a worst-case procedure for materials classed as Poison A (49 CFR 173.328). In the absence of reliable data that exclude the possibility of the presence of Poison A chemicals or compounds, these procedures must be followed.

Packaging

The following packaging procedures are to be used:

1. Collect sample in polyethylene or glass container which is of an outer diameter narrower than the valve hole on a DOT spec. 3A1800 or 3AA1800 metal cylinder. Fill sample container allowing sufficient ullage (approximately 10 percent by volume) so it will not be liquid-full at 130 F. Seal sample container.

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*Using "Flammable" does not convey the certain knowledge that a sample is in fact flammable, or how flammable, but is intended to prescribe the class of packaging in order to comply with DOT regulations. just before or just after the "Flammable Liquid, n.o.s." or "Flammable Solid, n.o.s." description.

2. Attach properly completed sample identification tag and EPA sample control label to sample container.

3. With a string or flexible wire attached to the neck of the sample container, lower the container into a metal cylinder that has been partially filled with incombustible, absorbent, loose packaging material (vermiculite or earth). Allow sufficient cushioning material between the bottom and sides of the container and the metal cylinder to prevent breakage. After the cylinder is filled with cushioning material, drop the ends of the string or wire into the cylinder valve hole. Only one sample container may be placed in a metal cylinder.

4. Replace valve, torque to 250 ft-lb (for 1-in. opening) and replace valve protector on metal cylinder using Teflon tape.

5. Mark and label cylinder as described below.

6. One or more cylinders may be placed in a strong outside container.

7. Mark and label outside container and complete shipping papers as described below.

The samples may not be transported by Federal Express Corporation (air cargo) or other common carrier aircraft, or by rented, nongovernment aircraft. (Samples may be shipped by ground transport or government aircraft.)

Marking and Labeling

Use abbreviations only where specified. Place the following information (either handprinted or on preprinted labels) on the side of the cylinder, or on a tag wired to the cylinder valve protector: "Poisonous Liquid or Gas, n.o.s"* and the laboratory name and address. Place the label "Poisonous Gas" on the cylinder ("Poisonous Liquid" label not acceptable here, even if liquid).

(NOTE: If the metal cylinders are placed in an outside container, both the container and cylinders inside must have the same markings and labels as above. In addition, "Laboratory Sample" and "Inside Packages Comply with Prescribed Specifications" should be marked on the top of the outside container. "THIS SIDE UP" marking should be placed on the outside container and upward-pointing arrows on four sides.)

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*Using "Poisonous" does not convey the certain knowledge that a sample is in fact poisonous, or how poisonous, but is intended to prescribe that class of packaging in order to comply with DOT regulations.

Shipping Papers

Complete the shipper-provided bill of lading and sign the certification statement. If carrier does not provide these documents, use standard industry form, providing the following information in the order listed (one form may be used for more than one exterior container; use abbreviation only as specified): "Poisonous Liquid, n.o.s.";

"Limited Quantity" or "Ltd. Qty."; "Laboratory Samples"; "Net Weight ____ or "Net Volume ____" (of hazardous contents), by cylinder if inside an exterior container. The net weight or net volume must be placed just before or just after the "Poisonous Liquid, n.o.s." marking.

A chain-of-custody record form must also be properly executed and included in the container or with the cylinder.

Unless the samples are driven to the laboratory, a team member will accompany shipping containers to the transport carrier and, if required, open outside container(s) for freight inspection.

4.4 RECORDS

A shipping certification form must be completed for all samples to be shipped.

TABLE 3-1

Packaging

1. Check DOT 172.500 table for appropriate type of package for hazardous substance.
2. Check for container integrity, especially the closure.
3. Check for sufficient absorbent material in package.
4. Check for sample tags and log sheets for each sample.

Shipping Papers

1. Check that entries contain only approved DOT abbreviations.
2. Check that entries are in English.
3. Check that hazardous material entries are specially marked to differentiate them from any nonhazardous materials being sent using same shipping paper.
4. Be certain all hazardous classes are shown for multiclass materials.
5. Check total amounts by weight, quantity, or other measure used.
6. Check that any limited-quantity exemptions are so designated on the shipping paper.
7. Offer driver proper placards for transporting vehicle.
8. Check that certification is signed by shipper.
9. Make certain driver signs for shipment.

RCRA Manifest

1. Check that approved state/federal manifests are prepared.

TABLE 3-1
RCRA MANIFEST (Cont'd)

2. Check that transporter has the following: valid EPA identification number, valid driver's license, valid vehicle registration, insurance protection, and proper DOT labels for materials being shipped.

3. Check that destination address is correct.

4. Check that driver knows where shipment is going.

5. Check that driver is aware of emergency procedures for spills and accidents.

6. Make certain driver signs for shipment.

7. Make certain one copy of executed manifest and shipping document is retained by shipper.

Malcolm Pirnie, Inc.
ARCS II QA Program
Standard Operating Procedure

Procedure MP-PMO QA-009 1/92
Date: January 8, 1992
Revisions No. 0
Prepared by: Lisa Szegedi
Fred Lonneker
Approved by: Dennis Stainken

Title: Procedures for Requesting Modification in the Working Documents of a Remedial Investigation/Feasibility Study (RI/FS).

I. Introduction

Variances may become necessary during the course of field investigations as site conditions dictate. Therefore, modifications of the work plans are required to conduct work efficiently without jeopardizing data quality. The purpose of this SOP is to outline the procedures and documentation required for requesting work plan variances.

II. Methods and Procedures

The general steps in the variance process are as follows:

A. When the need for a modification of a project work plan arises, a Variance Request Form must be completed. This form should contain the date of the request, the document to be revised (with page reference), and a detailed description of necessary changes as well as the rationale supporting the modifications. Variance Request Forms are to be completed and signed by the Site Manager.

B. Completed Variance Request Forms must be forwarded to the USEPA Remedial Project Manager (RPM) for approval. A letter summarizing the proposed changes should accompany the request forms.

C. The RPM will review the request forms either approving or amending the proposal. Revisions made by the RPM to the variance request should be incorporated into the original request form, with updated copies being forwarded to the RPM for documentation purposes.

D. The finalized request form should be appended to the affected document and modifications implemented.

1.0 OBJECTIVE

This guideline details the steps required to measure the pH of an aqueous sample while in the field using both a pH meter and pH paper. It is important to obtain a pH measurement soon after taking a sample and thus avoid sample changes such as precipitation, temperature fluctuation, or oxidation which can affect the pH of the sample.

2.0 APPLICABILITY

This guideline is applicable to all aqueous samples such as potable well water, monitoring well water, surface water, leachate, drummed wastewater, and other water samples.

3.0 DEFINITIONS

pH--The negative logarithm (base 10) of the hydrogen ion activity. The hydrogen ion activity is related to the hydrogen ion concentration, and, in relatively weak solution, the two are nearly equal. Thus, for all practical purposes, pH is a measure of the hydrogen ion concentration.

pH paper--Paper that turns different colors depending on the pH of the solution to which it is exposed. Comparison with color standard supplied by the manufacturer will then give an indication of the solution pH.

4.0 GUIDELINES

Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment such as acid-base neutralization, water softening, and corrosion control is pH dependent. Likewise, the pH of leachate can be correlated with other chemical analyses to determine the probable source of contamination. It is therefore important that reasonably accurate pH measurements be taken.

Two methods are given for pH measurement: the pH meter and pH indicator paper. To use the pH meter, the meter and electrode are standardized in pH 7 buffer and then immersed in the unknown sample to obtain a pH reading. No standardization is required when using pH paper. The indicator paper is simply immersed in the sample, and then a color comparison is made.

4.1 RESPONSIBILITIES

The project team leader is responsible for deciding when a pH measurement should be taken.

The field samplers are responsible for measuring the pH and for recording and reporting the results.

4.2 EQUIPMENT

The following equipment is needed for taking field pH measurements:

1. Accumet 150 portable pH meter or equivalent
2. Combination electrode with polymer body to fit the above meter
3. pH indicator paper, such as Hydrion or Alkacid, to cover the pH range 2 through 10

4.3 CALIBRATION

Calibration procedures should be in accordance with those specified in the operations manual of the meter.

4.4 FIELD pH MEASUREMENT

4.4.1 pH Meter

The following procedure is used for measuring pH with a pH meter:

1. Immerse the tip of the electrode in water overnight. If this is not possible due to field conditions, immerse the electrode tip in water for at least an hour before use.
2. Rinse the electrode with demineralized water.
3. Immerse the electrode in pH 7 buffer solution.
4. Adjust the temperature compensator to the proper temperature.

5. Adjust the pH meter to read 7.0. (Note: If the sample is known to have a very acidic or alkaline pH, standardize the meter with pH 4 or pH 10 buffer, respectively.)

6. Remove the electrode from the buffer and rinse with demineralized water.

7. Immerse the electrode in the unknown solution.

8. Read and record the pH of the solution, after adjusting the temperature compensator to the sample temperature.

9. Rinse the electrodes with demineralized water.

10. Keep the electrode immersed in water when not in use.

4.4.2 Indicator Paper

The following procedure is used for measuring pH with pH indicator paper:

1. Immerse a strip of indicator paper into the unknown solution.

2. Remove the paper from the solution and compare the color with the indicator colors given on the pH paper container.

3. Record the pH. (Note: If the indicator paper is suspected of being old or deteriorated, immerse it in pH 7 buffer and check the color that develops against the standards given.)

4.5 RECORDS

All results are to be recorded in the field logbook.

1.0 OBJECTIVE

This guideline details the steps required to measure the specific conductance of an aqueous sample while in the field. It is important to obtain a specific conductance measurement soon after taking a sample since temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect the specific conductance.

2.0 APPLICABILITY

This guideline is applicable to all aqueous samples such as potable well water, monitoring well water, surface water, leachate, drummed wastewater, and other water samples.

3.0 DEFINITIONS

Resistance--The inability of a substance to conduct a current. For metals and solutions the resistance is defined by Ohm's law, $E = IR$, where E is the potential difference, I is the current, and R is the resistance.

Conductance--The reciprocal of the resistance, $1/R$.

Specific conductance--The conductance of a 1-cm cube of electrolyte. Conductivity and specific conductance are used synonymously; this SOP will use the term "specific conductance."

4.0 GUIDELINES

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of the ionized substances dissolved in the water and the temperature at which the measurement is made. Th mobility of each of the various dissolved ions, their valences, and their actual and relative concentrations affect conductivity.

An aqueous system containing ions will conduct an electric current. In a direct-current field, the positive ions migrate toward the negative electrode, while the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases, and salts (such as hydrochloric acid, sodium carbonate, and sodium chloride) are relatively good conductors.

Conversely, molecules of such organic compounds as sucrose and benzene, which do not dissociate in aqueous solution, conduct a current very poorly, if at all.

4.1 RESPONSIBILITIES

The project team leader is responsible for deciding when a specific conductance measurement should be taken. Details will be given in the sampling plan for the site.

The field samplers are responsible for taking the conductance measurement and for recording and reporting the results.

4.2 EQUIPMENT

The following equipment is needed for taking specific conductance measurements:

1. YSI Model 33 portable conductivity meter or equivalent
2. Probe for above meter

4.3 CALIBRATION

Calibration procedures should be in accordance with those specified in the operations manual of the meter.

4.4 SPECIFIC CONDUCTANCE MEASUREMENT

The steps involved in taking specific conductance measurements are listed below.

1. Immerse the electrode in water overnight. If this is not possible due to field conditions, immerse the electrode for at least an hour before use.
2. Rinse the cell with one or more portions of the sample to be tested.
3. Immerse the electrode in the sample and measure the conductivity.
4. Read and record the results. Adjust the temperature setting to the sample temperature.

If the specific conductance measurements become erratic or inspection shows that any of the platinum black has flaked off the electrode, replatinization of the electrode is necessary. See the manufacturer's instruction for details.

Note that specific conductance is occasionally reported at temperatures other than ambient.

4.5 RECORDS

All results are to be recorded in the field logbook.

1.0 OBJECTIVE

The objective of these guidelines is to provide general reference information on sampling.

2.0 LIMITATIONS

These guidelines are for information only and are not to take precedence over the requirements of project-specific plans for sampling.

3.0 DEFINITIONS

Sampling. The physical collection of a representative portion of a population, universe, or environment.

Environmental Samples. Usually offsite samples with mid- or low-contaminant concentrations such as ambient air, streams, ground water, leachates, ditches, soil, and sediments collected at a distance from direct sources of contaminants.

Hazardous Samples. Samples of "raw" wastes, up to 100 percent by concentration, such as those taken from drums, tanks, and other containers; from waste piles, spills, or onsite lagoons or ditches; and from contaminated soil in the immediate vicinity of waste storage or spill areas.

Sampling Plan. A detailed plan that covers the sampling objectives and strategy.

4.0 GUIDELINES

These guidelines identify the sampling equipment, the sequence of operations, and the documents involved in physical sampling at or near uncontrolled hazardous-substance sites. Reference is made to other descriptive or instructional documents as appropriate.

4.1 SAMPLING RESPONSIBILITIES

Project managers are responsible for ensuring that the project specific sampling procedures are followed, maintaining chain-of-custody, and determining that all sampling documents have been completed properly and are accounted for. Samplers are responsible for collecting samples, initiating chain-of-custody forms, and the necessary sample documents as required.

4.2 SAMPLING EQUIPMENT

Typical equipment used for air and radioactivity sampling is summarized in Table 6-1 and sampling equipment for solid or liquid samples is listed in Table 6-2. Table 6-3 presents container and preservation requirements for samples.

4.3 SAMPLING METHODS

4.3.1 Environmental Samples

Air

If initial site atmospheric hazard surveys have been conducted and levels of personnel protection have been established, surveys for organic/inorganic vapors, oxygen content, combustible gases, and radioactivity must be repeated to confirm previous findings. These surveys are to be repeated periodically, as specified in the sampling plan.

Surface Water

Collecting a representative sample from surface water is difficult but not impossible. Samples should be collected near the shore unless boats are feasible and permitted. A small container or dipper attached to a pole is used to obtain the samples. Samples from various locations and depths should be composited; otherwise, separate samples will have to be collected. Approximate sampling points should be identified on a sketch of the water body. The following procedures are used:

1. Record available information for the pond, stream, or other water body, such as its size, location, depth, and probable contents, in the field logbook, on the chain-of-custody form, and on the sample log sheet.

2. Take samples near the shore of the water body and transfer them to appropriate bottles. See Table 6-3.

3. Secure the lid of each sample bottle and attach a label containing sample identification, number, and date. Securely tape the lid to the bottle; then date and initial the tape.

4. Measure the sample radioactivity and record. If readings exceed 10 mR/hr, notify the team leader or the site safety officer immediately.

TABLE 6-1

INSTRUMENTATION FOR AIR MONITORING

Hazard	Direct Reading	Instrument Type/Name	Collection Media
Explosive atmosphere	Combustible gas indicator	ENMET CGS-80 Tritector	Not used
Oxygen-dificient atmosphere	Oxygen meter	ENMET CGS-80 Tritector	Not used
Toxic atmosphere	Photoionization detector, flame ionization detector with gas chromatography option, colorimetric tubes	HNU-PI-101, OVA-Century, OVA 128, Drager, Bendix,	Sampling pumps in conjunction with gas chromatograph, absorption tubes, filters, impingers
Radioactivity	Radiation survey meters (Geiger-Mueller), passive monitors	Eberline, Victoreen	Dosimeters, film badges

TABLE 6-2

SOLID AND LIQUID SAMPLING EQUIPMENT

Sampler	Applications	Limitations
Plastic[a]	Liquids, slurries	Not for wastes containing ketones, nitrobenzene, dimethylformamide, mesityl oxide, tetrahydrofuran, or many common solvents such as acetone.
Glass tubes[a]	--	Not for wastes containing hydrofluoric acid and concentrated alkali solutions.
Pumps, bailers Wells		Pump may be used for precleaning well. Power or gas sources required. Bailers are slower than pumps, require no tubing.
Pnd (dip) sampler[a]	Liquids, sludges	Cannot be used to collect samples beyond 12 ft.
Manual hand pump	Liquids	Requires large amounts of disposable tubing; cannot be used when tubing is not compatible with material.
Weighted bottle sampler	Liquids	Difficult to use with very viscous liquids. Exterior of sample bottle is exposed to hazardous materials.
Buckets	Streams, ponds	Restricted to onshore sampling.
Grain sampler	Granular solids	Limited application for sampling moist and sticky solids with a diameter of 1/4 in.
Sampling trier	Solids	May incur difficulty in retaining core sample of very dry granular materials during sampling.
Trowel/scoop/ spoon[a]	Solids, soil	Not applicable to sampling deeper than 3 in. Difficult to obtain reporducible mass of samples.
Wisk pile sampler	Loose solids	Not applicable to sampling solid wastes with dimensions greater than half the diameter of the sampling tube.
Soil auger (manual)	Soil deeper than 3 to 4 in.	Does not collect undisturbed core sample.

[a]Recommended devices.

TABLE 6-3

CONTAINER AND PRESERVATION REQUIREMENTS

Type of Sample[a]	Concentration[b]	Sample Container	Sample Size	Preservatives
GC ad GC/MS organics Extractables	Low	1.2 gal glass	1 gal	Ice
		40-ml glass ampule	80 ml	Ice
VOAs	Medium and High	8-Oz glass	4 to 6 oz	None
Inorganics	Low	1-liter polyethylene	1-liter[c]	NEIC[c]
	Medium	8-oz glass	4 oz	None
	High	8-oz glass	4 oz	None
General chemistry		1-liter polyehtylene	1 liter	None
COD/TOC		1-liter polyehtylene	0.5 liter	H_2SO_4 to pH $\leq$2
Cyanide		1/2-liter polyethylene	0.5 liter	NaOH to pH $\geq$12
Oil and grease		1-liter glass	1.0 liter	H_2SO_4 to pH $\leq$2
Phenols		1-liter polyethylene	1.0 liter	1 g/liter of copper sulfate H_3PO_4 to pH $\leq$4
Sulfides		1/2-liter glass	0.5 liter	2-ml of 2N zinc acetate solution/liter
		Soils/Solids		
GC and GC/MS organics	Low	8-oz glass	6 oz	None
	Medium	4-oz glass	3 oz	None
	High	8-oz glass	4 oz	None
Inorganics	Low	4-oz glass	3 oz[c]	NEIC[c]
	Medium	4-oz glass	3 oz	None
	High	8-oz glass	4 oz	None

[a] GC = gas chromotagraphy; MS = mass spectroscopy; COD/TOC = Chemical Oxygen Demand/Total Organic Carbon.

[b] Low = sample contains less than 10 ppm of any single contaminant; medium = sample contains between 10 ppm and 15 percent of any one contaminant; high = sample contains greater than 15 percent of any one contaminant.

[c] See NEIC requirements in NEIC Denver's Enforcement Considerations for Evaluations of Uncontrolled Hazardous Waste Disposal Sites by Contractors, April 1980.

5. Carefully pack samples. Custody-seal the shipping package.

Ground Water

Monitoring Wells. Figure 6-1 is a typical well sampling data sheet. Not all the information shown can be obtained at all wells. Critical, required

WELL-MONITORING DATA SHEET

Waste Site Name _____ Analyze for:

Waste Site Location _____

_____

_____

Well I.D. Number _____

Sampler _____

Date and Time _____

Sample I.D. Number _____

Well Depth _____

Water Depth _____

Casing Size _____

Volume Bailed _____

Recharge Wait _____

Depth Sampled _____

Sample Method

- Vacuum
- Bailer
- Pressure
- Other

Sample Temperature _____

Preservation Method _____

Observations _____

CHM 001 0175

Figure 6-1 Example of Well-Monitoring Data Sheet

information includes:

1. Well locations
2. Well radius or diameter
3. Depth to water level
4. Total well depth
5. Amount of water in well

This information should be entered in the logbook.

Wells must be bailed or pumped three to five well volumes before sampling. Samples are taken after the well recharges to initial water depth. Wells that do not recharge within 24 hours will be sampled after the well recharges to a sufficient depth to provide an adequate volume of sample for analysis. Care must be taken not to disturb sediment at the bottom of the well when taking samples. The following procedures are used:

1. Measure the water level in the well using an M-scope or other device and record the elevation at the top of the water surface.

2. Determine the submerged casing volume (standing water volume) in the well from the following equation:

$$V = 3.14\ r^2h$$

where

V = volume
r = radius
h = standing water height as determined from drilling logs and actual measurement.

For example, a 30-ft drilled well with 5 ft of screen has a 2-in. casing with a nominal inside diameter of 1.90 in. (4.83 cm). The standing water level has been determined to be 10 ft or approximately 305 cm. Therefore, the submerged casing volume (in cubic centimeters and liters)

$$= [3.14(2.42)^2]305$$

$$= 5609\ cm^3 \text{ or } 5.6 \text{ liters}$$

3. With a manual bailer, remove three to five casing volumes of water from the well. To avoid disturbing the sediment, do not insert the bailer to the bottom of the screen. (Note: If the casing size allows, the well may be pumped with a submersible electric pump or other device until the appropriate volume has been removed. Do not overpump.)

4. When the well has recharged sufficiently, remove enough water to fill all sample bottles in accordance with Table 6-3. Add preservatives where required. In the event that recovery time of the well is very slow (e.g., 24 hours), attempts to collect samples immediately after bailing or pumping can be delayed until the following day. If the well has been bailed early in the morning, sufficient water may be standing in the well by the day's end to permit sample collection. If the well is incapable of producing a sufficient volume of sample at any time, take the largest quantity available and record in the logbook.

5. Label, tag, and number the sample bottle. Tape the lid on securely and mark the tape with the date and the collector's initials.

6. Replace the well cap. Make sure the well is readily identifiable as the source of the samples.

7. Pack the samples for shipping. Attach a custody seal to the shipping package as described above. Make sure that traffic reports and chain-of-custody forms are properly filled out and enclosed or attached.

Hydrants or Pumped Wells. Sampling from hydrants or pumped wells such as domestic wells requires a modified procedure. The well must be flushed by running the water for 5 minutes through the tap nearest the well. Take the sample from the continuously running tap after the 5-minute period. More detailed procedures can be found in Guideline 1.

Follow the steps above for entering information, packing, preserving, labeling, and marking.

Soils

Environmental soil sampling is generally performed off the site. The sampler to be used is dependent on the parameters to be analyzed, soil type, depth of sample desired, and homogeneity of soil.

For loosely packed earth, appropriately cleaned stainless steel or teflon coated scoops, trowels, and waste pile samplers can be used to collect representative samples. For densely packed soils or deep soil samples, a soil auger or other techniques may be used.

1. Use a soil auger for deep samples (6 to 12 in.) or a scoop or trowel for surface samples. Remove debris, rocks, twigs, and vegetation before collecting 200 to 250 g. Mark the location with a numbered stake if possible and locate sample points on a sketch of the site.

2. Transfer 100 to 200 g of the sample to a 250-ml container. Attach a label, identification number, and tag. Record all required information in the field logbook and on a sample log sheet as described in Section 4.4.3, below.

3. Store the sampler in a plastic bag until decontamination or disposal.

4. Tape the lid on the sample bottle securely and mark the tape with the date and the sample collector's initials.

5. Carefully pack the samples. Attach a custody seal to the shipping package. Make certain that chain-of-custody forms are properly filled out and enclosed or attached.

Sludges and Sediments

Sludge samples and sediments can usually be collected by bucket or longhandled dipper. If the sludges or sediments are relatively dense, waste pile samplers or tiers may be used.

1. Collect at least three small, equal-sized samples for several points along the sludge or sediment deposition area. If possible, mark the location with a numbered stake and locate sample points on a sketch of the site. Deposit sample portions in a clean, 1/2-gal composite.

2. Sediments from large streams, lakes, and the like may be taken with Ekman, Ponar dredges, or equivalent from a boat.

3. Transfer 100 to 200 g of the composite sludges from the 1/2-gal jar to a 250-ml sample bottle. Attach identification label number and tag. Record

all necessary information in the field logbook and on the sample log sheet.

4. Store the sampler and jar in a plastic bag until decontamination or disposal.

5. Tape the lid on the sample bottle securely and mark the tape with the date and the sample collector's initials.

6. Pack the samples for shipping. Attach a custody seal to the shipping package. Make certain that chain-of-custody forms are properly filled out and enclosed or attached.

4.3.2 Hazardous Samples

Air

Air samples are rarely taken to determine a medium or high hazard levels. In fact, site activities are usually suspended if high ambient hazardous substance concentrations are detected by routine monitoring devices or if low oxygen levels are discovered.

Surface Water

When collecting samples from medium- to high-hazard surface waters, such as onsite lagoons or ponds, the steps outlined above for environmental sampling should be followed. Added safety precautions, such as lifelines, are required.

Drums

Probably the most common container at hazardous waste sites is the drum, which is constructed of either metal or paper (fiber). Drum samples should be obtained through a free opening or through the bung hole whenever possible, using the procedure described below. Because drums may fail structurally, losing all or part of their contents, caution must always be exercised when it is necessary to move drums to gain access to them. The wisest course of action is to sample, analyze, and remove the most accessible drums before handling damaged, tipped, or buried drums. Remote-controlled bung wrenches are the best tools for opening drums.

Drums must be opened slowly and carefully. If the drum is bulging because of inside pressure or vacuum, special precautions must be taken in opening it.

It is permissible to place disposable sampling equipment in a drum that was sampled before resealing it. Separately labeled drums may be used as receptacles for contaminated sampling equipment as long as compatibility of the wastes is ensured.

The following procedures are used to obtain samples from drums:

1. Record any markings, special drum conditions, and type of opening in the field logbook, on the sample log sheet, and, later, on the chain-of-custody form. Locate the general area on a sketch of the site.

2. Stencil an identifying number on the drums and record in logbook. Consult the sampling plan for identifications.

3. Make certain that the drum is set on a firm base, preferably in a fully upright position.

4. Using a nonsparking bung wrench or a remote-controlled bung remover, carefully remove the bung and set it aside. Drums with top lids and samp-ring seals may be opened by carefully removing the seal and prying off the lid with a nonsparking tool. Set the lid and samp ring aside.

5. Carefully insert the sampling tube (either metal, glass, or compatible plastic) into the drum contents. Secure the upper end of the tube with the thumb or palm and withdraw the tube. (Note: If the sample is not free flowing and is contained in a drum with a lid, the sample may be removed with a clean scoop or a small shove.).

6. Deliver 100 to 250 ml of the sample (the sampling plan will specify the amount) to a clean, wide-mouth, 500-ml (1 pt) glass sample jar. If the sample is not free flowing and is taken through a bung opening, repeated sampling may be necessary. Replace the bung or cover carefully.

7. Place the used sampling tube, along with paper towels or waste rags used to wipe up any spills, into an empty metal barrel for subsequent disposal. If glass tubing has been used, it may be broken and left inside the drum being sampled.

8. Replace the cap on the sample jar; label, date, and number the jar. Record all information on the chain-of-custody form, sample log sheet, sample tag, and field logbook. The sample jar numbers and dates must match those recorded on all forms.

9. Secure the sample container lid with heavy-duty tape. Date and sign the tape.

10. Measure the sample for radioactivity. If the meter readings exceed 10 mR/hr, notify the site manager immediately.

11. Carefully pack samples. The finished package will be padlocked or custody-sealed for shipment to the laboratory. The preferred procedure includes the use of a custody seal wrapped across filament tape that is

wrapped around the package at least twice. The custody seal (paper, plastic, or metal) is then fold over and stuck to itself so that the only access to the samples is by cutting the filament tape or breaking the seal to unwrap the tape. The seal is signed before the package is shipped.

Tanks

The sampling of tanks is similar to the sampling of drums. Techniques of sampling are the same, except sampling equipment may need to be longer to give a representative sample of deep tanks.

1. Record the tank's condition, markings, opening or valve types, and approximate size in gallons in the field logbook, on the chain-of-custody form, and on the sample log sheet. Note the tank location on the site sketch.

2. Attach an identification number to the tank using a stencil or weatherproof tag. Number succeeding tanks consecutively. Record the numbers in the logbook.

3. Determine whether the tank contents are stratified by inserting a long plastic or glass tube sampler, withdrawing it, and examining the tube contents.

4. Samples of stratified contents can be taken with a bomb or weighted bottle sampler at each level. A segmented tube sampler may also be used if available. Deliver sampler contents, if stratified, to separate 500-ml glass sample jars. Otherwise, a single sample will suffice.

5. Secure the jar lid and label, date, and number the jar as above. Securely tape the lid to the jar; date and initial the tape.

6. Measure the sample radioactivity and record. Notify the site manager if readings exceed 10 mR/hr.

7. Carefully pack samples. Custody-seal the shipping package as described previously.

8. Clean any nondisposable sampling equipment and dispose of cleaning solvents and materials in a metal drum. Wipe up any spills and place rags or paper towels in the metal drum for later disposal.

Solid Waste Piles

Piles of waste usually vary in size and composition. Use scoops or trowels to obtain small discrete samples of homogeneous piles. Layered (nonhomogeneous) piles require the use of tube samplers or triers to obtain cross-sectional samples.

1. Collect small, equal portions of the waste from several points at or near the surface of the pile. Use numbered stakes, if possible, to mark the sampling locations and locate sampling points on the site sketch.

2. Collect a waste sample totaling 100 to 200 g and place it in a 250-ml glass container. Attach a label, identification number, nad tag. Record all the required information in the field logbook and on the sample log sheet.

3. Store the sampling tool in a plastic bag until decontamination or disposal.

4. Tape the lid on the sample bottle securely and mark the tape with the date and the sample collector's initials.

5. Pack samples for shipping. Attach a custody seal to the shipping package. Make sure that the traffic report and the chain-of-custody form are properly filled out and enclosed or attached.

For layered, nonhomogeneous piles, grain samplers, sampling triers, or waste pile samplers must be used to acquire a cross-section of the pile. The basic steps are listed below.

1. Insert a sampler into the pile at a 0- to 45-degree angle from the horizontal to minimize spillage.

2. Rotate the sampler once or twice to cut a core of waste material. Rotate the grain sample inner tube to the open position and then shake the sampler a few times to allow the material to enter the open slits.

3. Move the sampler into position with slots upward (grain sampler closed) and slowly withdraw it from the pile.

4. Transfer 100 to 200 g of sample into a 250-ml container with the aid of a spatula or brush. Attach a label identification number and tag. Record all necessary information in the field logbook and on the sample log sheet.

5. Store the sampler in a plastic bag until decontamination or disposal.

6. Tape the lid on the sample bottle securely and mark the tape with the date and the sample collector's initials.

7. Pack sample for shipping. Attach a custody seal to the shipping package. Make certain that the traffic report and chain-of-custody form are properly filled out and enclosed or attached.

Soils

Guidelines for collecting hazardous soil samples are the same as those for collecting environmental soil samples.

Sludges and Sediments

Guidelines for collecting hazardous sludge and sediment samples are the same as those for collecting environmental sludge and sediment samples.

4.4 SAMPLING DOCUMENTS AND RECORDS

This section identifies the various documents, forms, labels, and tags that sampling personnel will be required to use in the field.

- Field logbook(s)
- Field data records
- Sample log sheet
- Table of contents for sample log sheet notebook
- Other
- Sample identification labels and/or tags
- Chain-of-custody form
- Custody seal

There are additional forms of documentation that may need to be maintained that are not standard in format. These forms are discussed separately.

4.4.1 Field Logbook

A field logbook is a bound notebook with numbered pages in which all pertinent information about a field investigation (data, observations, phone calls, etc.) is entered. It is advised that one field logbook be maintained per site. This logbook is issued by the Project Manager for the life of the project. Logbooks may be issued to other field personnel (including those collecting samples). The Project Manager (or his designee) numbers all logbooks and records their transfer to other individuals as necessary. All project logbooks are to be turned over to the Project Manager and to a central file at the completion of the particular field activity.

4.4.2 Field Data Records

Field data records may include sample log sheets, tables of contents for sample log sheet notebooks, and any other data records that the Project Manager or task leader may designate for use in field data collection. The exact forms used will depend on the scope of the project and the situations presented.

4.4.3 Sample Log Sheet

A sample log sheet is a notebook page 8.5 by 11 in. that is used to record specified types of data while sampling. The data recorded on these sheets is useful in describing the waste source and the sample (if obtained) as well as pointing out any problems encountered during sampling.

4.4.4 Table of Contents for Sample Log Sheet Notebook

The table of contents form is a notebook page 8.5 by 11 inc. on which entries are made as the completed sample log sheets are placed in a three-ring binder. Figure 6-2 is an example of the table of contents form. This form facilitates quick reference to the sample log sheets contained in the notebook and remains in the notebook at all times.

4.4.5 Labeling of Samples

Sample Label

The sample label is a 2- by 4-inc. white label with black lettering and an adhesive backing. Figure 6-3 is an example of Malcolm Pirnie sample identification label.

A sample label must be attached to each bottle that contains a sample. The label must be attached to the bottle just before putting the sample into the bottle. In addition, the label should be covered with clear plastic tape to ensure that it does not peel off or become damaged. The sample number is the number assigned to the waste source under inspection and any samples taken from it.

4.4.6 Chain-of-Custody Form

The chain-of-custody form (8.5 by 11 in.) accompanies a sample or group of samples as it is transferred from person to person. Figure 6-4 is an example of a chain-of-custody form. This form documents custody transfer from person to person. Additional procedures regarding chain-of-custody are given in SOP#2.

4.4.7 Custody Seal

A custody seal is a small paper label with black lettering on an adhesive backing. Figure 6-5 is an example of and EPA custody seal. The custody seal is part of the chain-of-custody process and is used to prevent tampering with samples after they

SAMPLE NUMBER	DATE SAMPLED	SAMPLE NAME	PAGE NO.

FIGURE 6-2

EXAMPLE TABLE OF CONTENTS FOR SAMPLE NOTEBOOK

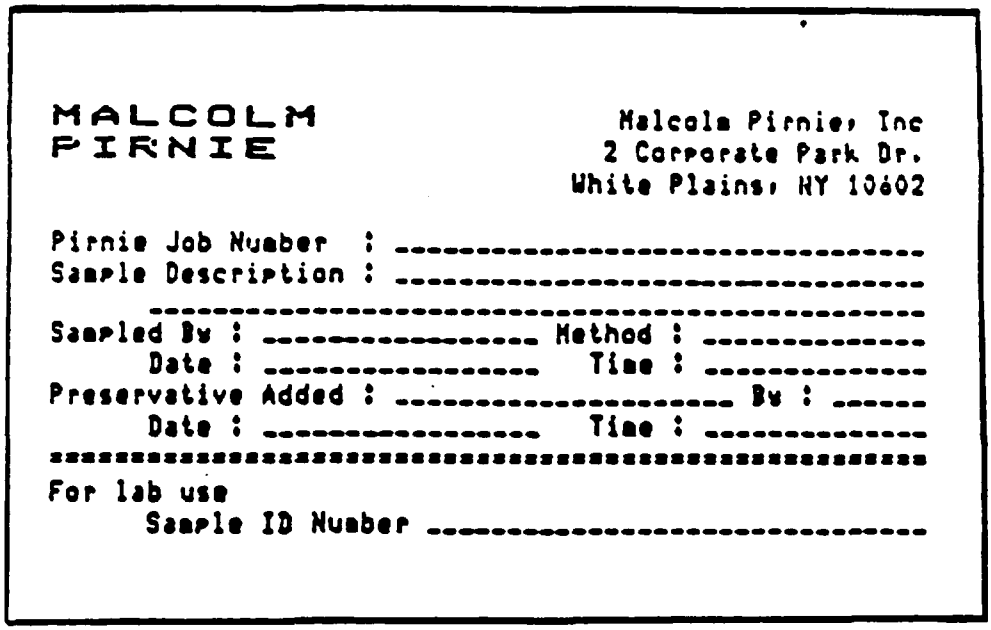

FIGURE 6-3

SAMPLE LABEL

MALCOLM PIRNIE

MALCOLM PIRNIE INC.
SUITE A 1212, NORTHPARK CORPORATE CENTER
1210 WEST COUNTY ROAD E
ST. PAUL, MINNESOTA 55112

CHAIN OF CUSTODY RECORD

OJ. NO. | PROJECT NAME

'LERS: (Signature)

NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS							REMARKS

CHM 001 0189

quished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
quished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
quished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks	

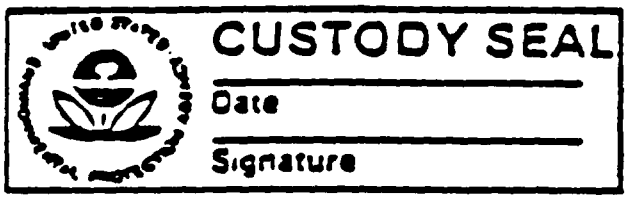

FIGURE 6-6

EPA CHAIN-OF-CUSTODY SEAL

have been collected in the field. Custody seals are provided by the Sampling Management Office and are distributed by the document custodian on an as-needed basis.

4.4.8 Nonstandard Documentation

Photographs

When movies, slides, or photographs are taken of a site or any monitoring location, they are numbered to correspond to logbook entries. The name of the photographer, date, time, site location, site description, and weather conditions are entered in the logbook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook. If possible, such techniques should be avoided, since they can adversely affect the admissibility of photographs as evidence. Chain-of-custody procedures depend upon the subject matter, type of film, and the processing it requires. Film used for aerial photography, confidential information, or criminal investigations require chain-of-custody procedures. Adequate logbook notations and receipts may be used to account for routine firm processing. Once developed, the slides or photographic prints shall be serially numbered and labeled according to the logbook descriptions.

1.0 OBJECTIVE

The objective of these guidelines is to provide general reference information and technical guidance on the measurement of ground-water levels.

2.0 LIMITATIONS

These guidelines give overall technical guidance only and should be modified by specified requirements of project-specific plans for measuring ground water levels in wells.

Cascading water within a borehole can cause false readings with some types of electrical sounding devices. Oil layers may also cause problems in determining the true water level in well.

All water level measurements at a site should ideally be made on the same day.

Ground water contaminated by organic compounds may release toxic vapors into the airspace inside the wellpipe. The release of this air when the well is initially opened is a health/safety hazard which must be considered.

3.0 DEFINITIONS

Hydraulic head-Also piezometric head or ground-water potential pressure is equal to atmospheric pressure.

Water table – A surface in an aquifer where ground-water pressure is equal to atmospheric pressure.

Piezometric (potentiometric) surface – A surface which is defined by the levels to which water will rise in cased wells which penetrate a confined or artesian aquifer.

4.0 GUIDELINES

4.1 General

In measuring ground-water levels, there should be a clearly established reference point of known elevation, which is normally the top of the well casing. The field notes recorded should clearly describe the reference used. To be useful, the

reference point should be tied in with the USGS - Benchmark or a local datum. An arbitrary datum could be used for an isolated group of wells if necessary. (All ground-water level measurements shall be made and recorded to the nearest 0.05 foot.) After the ground-water observation standpipe has been installed or the cased borehole completed and left open, the initial depth to the water should be measured and recorded. The date and time of the reading should also be recorded. Information related to precipitation should be included in the data. The depth of the ground water should be entered.

Appropriate remarks describing the history of the ground-water standpipe or open-cased borehole should be recorded along with the name of the individual who has read th ground-water monitoring well.

Readings should be taken regularly, as required by the site geologist. Ground-water standpipes or open-cased boreholes that are subject to tidal fluctuations should be read in conjunction with a tidal chart; the frequency of such readings should be established by the site geologist.

4.2 SPECIFIC GROUND-WATER LEVEL MEASURING TECHNIQUES

There are several methods for determining water levels in boreholes and monitoring wells. Certain methods have particular advantages and disadvantages depending upon th diameter of the borehole or casing, ground-water quality, and hydraulic conductivity of the formation. A general guideline for obtaining static water level and changes in water level during testing is presented along with a listing of various advantages and disadvantages of each technique. An effective technique should be selected for the particular site conditions by the onsite geohydrologist.

Procedure

1. Check operation of equipment above ground.

2. Record well number, top of casing elevation and surface elevation if available. Water levels should be taken from top of the still protective casing or a reference point at the ground surface for borehole measurements. The distance between the top of the protective casing and inner casing should be recorded.

3. Record water level to the nearest 0.5 foot.

4. Record the time and day of the measurement.

5. Many water level measuring devices have marked metal or plastic bands clamped at intervals along the measuring line used for reference points to obtain depth measurements. The spacing an accuracy of these bands should be checked frequently, as they may loosen and slide up or down the line, resulting in inaccurate reference points.

Chalked Steel Line -- Water level is measured by chalking a steel weighted tape and lowering it a known distance into the well or borehole. Water level is determined by subtracting the wetted chalked mark from th total length lowered into the hole.

The tape should be withdrawn quickly from the well because water has a tendency to rise up the chalk due to capillary action. A paste called "National Water Finder" may be used in place of chalk. The paste is spread on the tape the same way as the chalk, but the part that gets wet turns red. This paste is manufactured by the Metal Hose and Tubing Company, Dover, New Jersey. Disadvantages include the following: Limited to less than 300 ft, ineffective if borehole well is wet or inflow is occurring above the water level, chalking the tape is time consuming, difficult to use during periods of precipitation.

Popper or Bell Sounder -- A A bell or cup shaped weight that is hollow on the bottom is attached to a measuring tape and lowered into the well. A "plopping" or "popping" sound is made when the weight strikes the surface of the water. An accurate reading can be determined by lifting and lowering the weight in short strokes, and reading the tape when the weight barely strikes the water.

Electric Water Level Indicators -- This method consists of a spool of small- diameter steel cable and a probe attached to the end. when the probe comes in contact with the water, the circuit is closed and a meter, light, and/or buzzer attached to th spool will signal the contact. Pen light batteries are normally used for a power source.

There are a number of commercial electric sounders available, none of which is entirely reliable. Especially when there is oil on the water, high specific conductance, water cascading into the well, or a turbulent water surface in the well, measuring with an electric sounder may be difficult. The electric tapes generally are marked at 5-ft intervals with clamped-on metal bands. Before lowering the probe into the well, the circuitry can be checked by dipping the probe in water and observing the indicator. The probe should be lowered slowly into the well. The

electric tape is marked at the measuring point where contact with th water surface was indicated. The distance from the mark to the nearest tape band is measured and added to the band reading to obtain the depth to water. The metal bands have a tendency to move when they are continuously run in and out of a casing. Therefore, these bands should be calibrated periodically and adjustments should be made.

Float Recorder -- A float or an eletromechanically actuated water-seeking probe may be used to detect vertical changes of the water surface in the hole. A recording chart drum is rotated mechanically while a clock drive over a recording pen horizontally across the chart. To ensure continuous records, it should be inspected, maintained, and adjusted periodically.

Air Line -- An air line is especially useful in pumped wells where water turbulence may preclude the use of other devices. A small-diameter tube of known length is installed from the surface to a depth below the lowest water level expected. Compressed air (from a compressor, bottled air, or air pump) is used to purge the water from the tube. The pressure needed to purge the water from the air line multiplies by 2.31 (ft of water for 1 psi) equals the length infeet of submerged air line. The depth to water below the center of of the pressure gauge can be calculated by subtracting the length of air line below the water surface from the total length of the air line.

Capillary Tubing -- In small diameter tubing, water levels are determined by using a capillary tube. Colored or clear water is added to one end of the tube and looped. The other end of the tube is placed in the small diameter tubing until the water in the loop moves. The point on the tube is marked where it intersects the top of the casing. This point is then measured from the bottom of the capillary tube o recorded if the capillary tube is calibrated. This is the best method for very small diameter tubing monitoring systems such as Barcad and other multilevel samples. Unless the capillary tube is calibrated, two people may be required to measure the length of capillary tubing used to reach the ground water.

Pressure Transducer -- Pressure transducers measure the pressure of water on the transducer. The transducer is lowered into a well or borehole below the water. The transducer is wired into a recorder at the surface to record changes in water level with time. The recorder digitizes the information and can transfer the information to a compute for evaluation. The pressure transducer should be initially calibrated with another water level measurement technique to ensure accuracy. This technique is very useful for hydraulic conductivity testing in highly permeable material where

repeated accurate water level measurements are required in a very short period of time.

Borehole Geophysics -- Water levels could be determined during geophysical logging of the borehole. Several logging techniques will indicate water level. One main techniques is the spontaneous potential (SP) log.

Bailer Line Method -- Water levels during a bailing test of a well could be measured by marking and measuring the bailer line from the bottom of the bailer where water is encountered to the point even with the top of the casing. On the last bailing run of the test the bailing line is marked again at the top of the casing where the bailer encounters water. This level would be recorded as the bail down level. this is a useful technique is th bailer hitting the water is heard. It saves time from removing th bailer and placing a measuring line down the hole for th initial measurement of a rising head test.

A ground-water level measurement sheet, shown in Figure 7-1, should be filled out for each round of water level measurements at a site. all pertinent data should be recorded as shown on the sheet. The elevation of reference point is generally the elevation of the top of the well casing. the water level indicator reading is the actual reading on the measuring device. This measurement then must be adjusted by the appropriate amount of each device to determine the adjusted depth and ground-water elevation. It is important to note weather conditions on the form, as changes in barometric pressure will affect the water level within the well.

4.3 SPECIFIC QUALITY CONTROL PROCEDURES

All devices used to measure ground-water levels shall be calibrated against the invar steel surveyor's chain. These devices shall be calibrated to 0.05-ft accuracy periodically. Before each use, these devices shall be prepared according to the manufacturer's instructions (if appropriate) and checked for obvious damage. All calibration and maintenance data shall be recorded in a logbook. All ground-water level measurement devices must be cleaned before and after each use to prevent cross contamination of wells. A water level indicator calibration sheet should be completed after each time the measuring device is checked. A water level indicator calibration form is shown in Figure 7-2. The "actual reading" column on the sheet is the actual length of the interval from the end of the indicator to the appropriate marked depth interval. In many cases, these measurements are different, as the water level measuring device is connected to the end of the measuring tape or line, and may extend beyond "0" ft on the measuring line.

FIGURE 7-1

GROUND -WATER LEVEL MEASUREMENT SHEET

Project Name _____ Local Municipality _____

Project No. _____ County _____

Personnel _____ State _____

Date _____

Weather Conditions

Temperature Range _____ Equipment No. _____

Preceipitation _____ Equipment Name _____

Barometric Pressure _____ Latest Calibration Date _____

Piezometer No.	Date/ Time	Elevation of Reference Point (Ft)	Water Level Indicator Reading (Ft)	Adjusted Depth (Ft)	Ground-Water Elevation (Ft)

FIGURE 7-2

WATER LEVEL INDICATOR CALIBRATION

Project Name _____

Project No. _____ Date

Equipment No. _____ Last Calibration _____

Equipment Name _____ Calibration Period _____

Water Level Indicator Marking (Ft)	Actual Reading* (Ft)
0.0	
5.0	
10.0	
15.0	
20.0	
25.0	
30.0	
35.0	
40.0	
45.0	
50.0	
55.0	
60.0	
65.0	
70.0	
75.0	
80.0	
85.0	
90.0	
95.0	
100.0	

*NOTE: Record readings to the nearest 0.05 ft.

1.0 OBJECTIVE

The objective of these guidelines is to provide general reference information on the sampling of ground-water wells.

This guideline is primarily concerned with the collection of water samples from the saturated zone of the sub-surface. Every effort must be made to assure that the sample is representative of the particular zone of water being sampled.

2.0 LIMITATIONS

These guidelines are for information only and are not to take precedence over the requirements of project-specific plans for ground-water well sampling.

The limitations of analyses of samples collected from wells include changes to the sample from the materials that the water contacts, pressure changes, and temperature changes. The sample will only be representative of a small volume of the aquifer that is being sampled.

3.0 DEFINITIONS

None.

4.0 GUIDELINES

4.1 GENERAL

The primary consideration is to obtain a representative sample of the ground water body by guarding against mixing the sample with stagnant (standing) water in the well casing. In a nonpumping well, there will be little or no vertical mixing of the water, and stratification will occur. The well water in the screened section will mix with the ground water due to normal flow patterns, but the well water above the screened section will remain isolated and become stagnant. Persons sampling should realize that stagnant water may contain foreign material inadvertently or deliberately introduced from the surface, resulting in unrepresentative data and misleading interpretation of the same.

To safeguard against collecting nonrepresentative stagnant water in a sample, the following guidelines and techniques should be adhered to during sample withdrawal:

1. As a general rule, all monitoring wells should be pumped or bailed prior to withdrawing a sample. Evacuation of a minimum of one volume of water in the well casing and preferably three to five volumes is recommended for a representative sample. In a high-yielding ground-water formation and where there is no stagnant water in the well above the screened section, evacuation prior to sample withdrawal is not as critical. However, in all cases where the monitoring data is to be used for enforcement actions, evacuation is recommended.

2. For wells that can be pumped or bailed to dryness with the sampling equipment being used, the well should be evacuated and allowed to recover prior to sample withdrawal. If the recovery rate is fairly rapid and time allows, evacuation of more than one volume of water is preferred.

3. For high-yielding monitoring wells that cannot be evacuated to dryness, bailing without prepumping the well is not recommended; there is no absolute safeguard against contaminating the sample with stagnant water. The following procedures should be used:

   a. The inlet line of the sampling pump should be placed just below the surface of the well water and three to five volumes of water pumped at a rate equal to the well's recovery rate. this provides reasonable assurance that all stagnant water has been evacuated and that the sample will be representative of the ground-water body at that time. The sample can then be collected directly from the pump discharge line.

   b. The inlet line of the sampling pump (or the submersible pump itself) should be placed near the bottom of the screen section, approximately one well volume of water should be pumped at the well's recovery rate and the sample collected directly from the discharge line.

A nonrepresentative sample can also result from excessive prepumping of the monitoring well. Stratification of the leachate concentrations in the ground-water formation may occur or heavier-than-water compounds may sink to the lower portions of the aquifer. Excessive pumping can dilute or

increase the contaminant concentrations from what is representative of the sampling point of interest.

4.2 SAMPLING, MONITORING, AND EVACUATION EQUIPMENT

Appropriate sample containers should be selected for the contaminants present, with most containers being constructed of polyethylene.

The following equipment should be on hand when sampling ground-water wells:

1. Coolers for sample shipping and cooling, chemical preservatives, and appropriate packing cartons and filler.

2. Thermometer; pH paper/meter; dissolved oxygen meter; camera and film; tags; appropriate keys (for locked wells); tape measure; pipe wrenches, torch, hammer, and chisel; water-level indicators; flow meter; specific-conductivity meter; and depth sounder (only where applicable).

3. Pumps

   a. Shallow-well pumps--Centrifugal, pitcher, suction, or peristatic pumps with droplines, air-lift apparatus (compressor and tubing) where applicable.

   b. Deep-well pumps--Submersible pump and electrical power generating unit on air-lift apparatus where applicable.

4. Bailers and monofilament line with tripod-pully assembly (if necessary). Bailers shall be used to obtain samples from shallow and deep ground-water wells.

5. Pails--Plastic, graduated.

6. Decontamination solutions--Distilled water, alconox, methanol.

Sample withdrawal methods require the use of pumps, compressed air, bailers, and samplers. Ideally, sample withdrawal equipment should be completely inert; economical to manufacture; easily cleaned, sterilized, and reused; able to operate at remote sites in the absence of powder resources; and capable of delivering variable rates for well flushing and sample collection. The sample withdrawal equipment

Ground Water Well Sampling

Standard Operating Procedure 8
Figure: 8-1
Date: September 5, 1986

MALCOLM PIRNIE

PROJECT # _____ DATE _____

PROJECT NAME _____ SAMPLERS _____

SITE LOCATION _____

SAMPLE COLLECTION LOGS (WELLS)

WELL I D # _____ DATE: _____

TIME START : _____

TIME FINISH : _____

WELL EVACUATION DEVICE : _____

SAMPLE COLLECTION DEVICE : _____

DEPTH TO WATER (FT. FROM TOC) = _____

WELL DEPTH (FT. FROM TOC) = _____

HEIGHT OF WATER IN WELL = _____

ONE WELL VOLUME (GALLONS) = _____

VOLUME WATER REMOVED(GALLONS) = _____

SAMPLE APPEARANCE : _____

FIELD PARAMETERS	FIRST	SECOND	THIRD	FOURTH	FIFTH

SAMPLES COLLECTED (PARAMETERS AND PRESERVATIONS) : _____

WEATHER CONDITIONS : _____

OTHER NOTES : _____

CASING DIAMETER (IN.)	CASING VOLUME (GALLONS/LINEAR FT.)
1.25	.077
1.50	.1067
2	.1623
2.5	.2431
3	.3662
4	.4929

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(evacuation devices) most commonly used is discussed in Section 4.4.1 below.

4.3 CALCULATIONS OF WELL VOLUME

Calculations are to be done according to the following steps:

1. Obtain all available information on well construction (casing, screens, etc.).
2. Determine well or casing diameter.
3. Determine static water level (feet below ground level).
4. Determine depth of well.
5. Calculate number of linear feet of static water (total depth minus the static water level).
6. Calculate static volume in gallons ($V = Tr^2$ (0.163)), where T is th linear feet of static water and r is th inside radius of the well of casing in inches.
7. Determine the minimum amount to be evacuated before sampling.

4.4 EVACUATION OF STATIC WATER (PURGING)

The amount of using a well receives prior to sample collection depends on the intent of the monitoring program as well as the hydrogeologic conditions. Programs where overall quality determinations of water reserves ar involved may require long pumping periods to obtain a sample that is representative of a large volume of that aquifer. The pumped volume can be determined prior to sampling so that the sample is a composite of a known volume of the aquifer, or the well can be pumped until the stabilization of parameters such as temperature, electrical conductance, and pH has occurred.

However, monitoring for defining a contaminant plume requires a representative sample of a small volume of the aquifer. These circumstances require that the well be pumped enought to remove the stagnant water but not enough to induce flow from other areas. Generally four to six well volumes ar considered effective, or calculations can be made to determine, on the basis o the aquifer parameters and well dimensions, the appropriate volume to remove prior to sampling.

4.4.1 Evacuation Devices

The devices described in this section ar the ones commonly used. Others that have been made on a limited scale have been omitted.

Table 8-1 provides guidance on the proper evacuation device to use for given sampling situations.

Bailers

Bailers are the simplest evacuation devices used and have many advantages. They generally consist of a length of pipe, usually with a ball check-valve at the bottom. A line is used to lower the bailer and retrieve the sample.

Advantages

- only practical limitations on size and materials
- no power source needed
- portable
- inexpensive, so it can be dedicated and hung in a well reducing the chances of cross contamination
- minimal outgassing of volatile organics while sample is in bailer
- readily available

Limitations

- time consuming to flush well of stagnant water
- transfer of sample may cause aeration

Suction Pumps

There are may different types of suction pumps such as centrifugal, peristatic, diaphragm, and pitcher pumps. Diaphragm pumps can be used for well evacuation at a fast pumping rate and sampling at a low pumping rate. The peristaltic pump is a low volume pump that uses rollers to squeeze flexible tubing creating suction. This tubing can be dedicated to a well to prevent cross contamination. Peristaltic pumps, however, require a power source. The pitcher pump is a common farm hand-pump.

Advantages

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TABLE 8-1

PURGING EQUIPMENT SELECTION

	Bailer	Peristaltic Pump	Vacuum Pump	Airlift	Diaphragm "Trash" Pump	Submersible Diaphragm Pump	Submersible Electric Pump	Submersible Electric Pump w/Packer
25-inch								
ater level 125 ft		X	X	X	X			
ater level 125 ft				X				
-inch								
ater level 125 ft	X	X	X	X	X	X		
ater level 125 ft	X			X		X		
-inch								
ater level 125 ft	X	X	X	X	X	X	X	X
ater level 125 ft	X			X		X	X	X
-inch								
ater level 125 ft				X	X		X	X
ater level 125 ft				X			X	X
-inch								
ater level 125 ft				X	X		X	X
ater level 125 ft				X			X	X

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- portable, inexpensive, readily available

Limitations

- restricted to areas with water levels within 20 to 25 ft of the ground surface

- vacuum can cause loss of dissolved gases and volatile organics

Gas-Lift Samplers

This group of samplers includes those that use a gas pressure in the annulus of the well to force the water out a sampling tube and others which use the gas in a venturi to force the sample up a tube.

Advantages

- portable, inexpensive, readily available
- good for well development

Disadvantages

- If air is used, oxidation of the sample may occur
- loss of CO_2 changes the pH and metals concentrations will be lowered
- loss of volatile compounds may occur

Submersible Pumps

There are now many types of pumps which can be considered submersible; that is, they take in water and push the sample to the surface. The power sources for these samplers may be compressed gas or electricity. The operation principles vary. The displacement of the sample can be by an inflatable bladder, sliding piston, gas bubble, or impeller. Industry is now concentrating on the manufacture of devices to obtain samples in 2-in. diameter wells with water levels below 20 to 25 ft.

Advantages

- portable and readily available
- construction materials to match parameters of interest
- available for 2-in. diameter wells

Limitations

- may have low delivery rates
- expensive
- compressed gas or electric power needed
- sediment in water may cause problems with some of these sampler types

4.5 SAMPLING

4.5.1. Sampling Plan

Prior to sampling, a sampling plan and a safety plan should be developed in consultation with the interested parties and submitted for approval. The contents of a sampling plan consist of the following:

1. Background and objective of sampling
2. Brief area and waste characterization
3. Selection of sampling location, with map or sketch
4. Sampling equipment to be used
5. Intended number, volumes, and types of samples
6. Working schedule
7. List of team members
8. List of observers
9. List of contacts
10. Other information, such as th necessity for a warrant, or permission of entry and for split samples

4.5.2 General Sampling Rules

If the samples must be filtered, the appropriate chemical preservatives are to be added as soon as possible.

Recommended sample containers should be used.

Samples of organic analysis should be kept refrigerated or or ice during transport to the laboratory.

Recommended holding times should be adhered to in order to ensure that constituent alteration is minimized.

All samples will require the use of a bailer to maintain the integrity of the sample. Sample handling, labeling, and shipping methods are described in other guidelines of this manual.

4.5.3 Sampling Procedure

The procedure for sampling is made up of the following steps:

1. Open well cap and use detection equipment on the escaping gases at the well head to determine the need for respiratory protection. Using clean equipment, sound well for total depth and water level; then calculate the fluid volume in the casing.

2. Determine depth to main point of screen or well section open to aquifer from casing top. Any dry wells encountered must be noted during the field investigation.

3. Select appropriate purging equipment (see Table 8-1). If an electric submersible pump with packer is chosen, go to step 10.

4. Lower purging equipment or intake into the well to a short distance below the water level and begin water removal. Collect or dispose of purged water in an acceptable manner. Lower purging device, as required, to maintain submergence.

5. If pumping, measure water level with an electric sounder in uniform drawdown increments of 15- to 30-second intervals. If bailing, measure water levels as needed for a good record.*

6. Measure rate of discharge frequently. A bucket and stopwatch are most commonly used; other techniques include using pipe trajectory methods

or constructing weir boxes.

7. Observe peristaltic or vacuum pump intake for degassing "bubbles." If bubbles are abundant and the intake is fully submerged, these devices may not be suitable for collecting samples for volatile organics.

8. Purge a minimum of three to five casing volumes before sampling. In low permeability strata, one volume will suffice.

9. While pumping, lower intake to midscreen or midopen section depth collect sample. If bailing, lower device to sampling level before filling (this requires other than a "bucket-type" bailer). Purgd water should be collected in a designated container or disposed in an acceptable manner.

10. (For pump and packer assembly only.) Lower assembly into well so that packer is positioned just above the screen or open section and inflate. Purge a volume equal to at least twice the screen or open section volume below the packer (whichever is greater) before sampling. Packers should always be tested in a casing section above ground to determine proper inflation pressures for good sealing.

11. Allow the well recharge to the static water level and then sample.

12. After sampling, monitor water level recovery (may not be appropriate with a packer/pump assembly) and decontaminate all equipment. Make sure well is securely capped.

The data required from the sampling program will determine the quantity and preservation needed for each group of constituents. Sample preservation is not completed and may not be possible for some parameters. This may require field analysis for pH, Eh, electrical addition, refrigeration, and freezing. The use of preservatives retards both biological activity and the hydrolysis of some constituents and also reduces volatility. A list of perservatives and their effects is presented in Table 8-2.

Some parameters require special preservation, such as cyanides and phenols. Table 8-3 illustrates the quantities and preservation methods for the 129 priority pollutants identified by the EPA.

4.6 DECONTAMINATION

TABLE 8-2

SAMPLE PRESERVATION METHODS

Preservative	Action	Applicability
$HgCl_2$	Bacterial inhibitor	Nitrogen forms, phosphorous forms
Acid (HNO_3)	Metals solvent, prevents precipitation	Metals
Acid (H_2SO_4)	Bacterial inhibitor	Organic samples (COD, oil and grease, organic carbon)
	Salt formation with organic bases	Ammonia, amines
Alkali (NaOH)	Salt formation with volatile compounds	Cyanides, organic acids
Refrigeration	Bacterial inhibitor	Acidity-alkalinity, organic material, BOD, color, ordor, organic P, organic N, carbon, etc.; biological organisms (coliform, etc.)

TABLE 8-3

SAMPLE REQUIREMENTS FOR THE 129 PRIORITY POLLUTANTS

A. Extractable Organics

1. Fill 1-gal bottle.
2. Cool and maintain sample at 4°C.

B. Metals

1. Fill 1-qt bottle.
2. Adjust to pH of 2 with HNO_3.
3. Cool and maintain sample at 4°C.

C. Volatile Organic Analysis (VOA or purgable organics)

1. Fill two new and cleaned 40ml vials.
2. Seal with cleaned septum and cap; invert; tap; if air bubbles appear, refill.
3. If waste stream contains residual chlorine, collect five vials. Preserve two with either sodium bisulfate or sodium thiosulfate. These vials will be specially marked, and the preservative will then have been added in the lab.
4. Wrap in waterproof packing; cool and maintain at 40°C.

D. All Cyanides

1. Fill a 1-qt cubitainer (polyethylene bottle).
2. Test for chlorine with potassium iodide (KI) starch paper.
3. If paper color is unchanged, go to step 5.
4. If paper turns blue, add ascorbic acid until paper no longer turns blue.
5. Add an additional 0.6 g ascorbic acid (premeasured in a vial).

Decontamination solutions may vary according to type of contamination. To decontaminate pumps used in sampling or evacuation, two separate solutions should be prepared: one containing alconox and water and the other containing water and methanol. The alconox solution should be run through the pump and tubing first, and the methanol solution should be used as a rinse. then the pump should be rinsed with distilled water.

1.0 OBJECTIVE

The objective of these guidelines is to provide general reference information on the use of the HNu Systems PI 101 Photoionization detector.*

2.0 LIMITATIONS

These guidelines are for information only and are not to take precedence over the requirements of project-specific plans for the HNu photoionization detector.

3.0 PREREQUISITES

None.

4.0 GUIDELINES

4.1 PERSONNEL

The HNu Photoionizer Model PI 101 is extremely simple to operate. However, since the interpretation of its readings is often complex, personnel with specialized training and/or knowledge in its operation must be present to evaluate the data obtained from it.

4.2 INSTRUMENT OPERATION

4.2.1 Specifications

1. Range: 0.1 to 2000 ppm (benzene); linear range: 0.1 to 600 ppm
2. Sensitivity: 0 to 2 ppm, maximum for 100 division scale
3. Response time: 3 seconds to 90 percent full scale
4. Operating temperature: Ambient to 40 C
5. Operating humidity: To 95 percent relative humidity

4.2.2 Description

The unit consists of a Readout/Control Assembly and a Sensor/Probe. The Probe connects the Readout/Control Assembly via an electrical cord and 12-pin jack (front panel mounted). The Sensor/Probe can be disassembled and stored in the instrument's cover (see Figure 11-1).

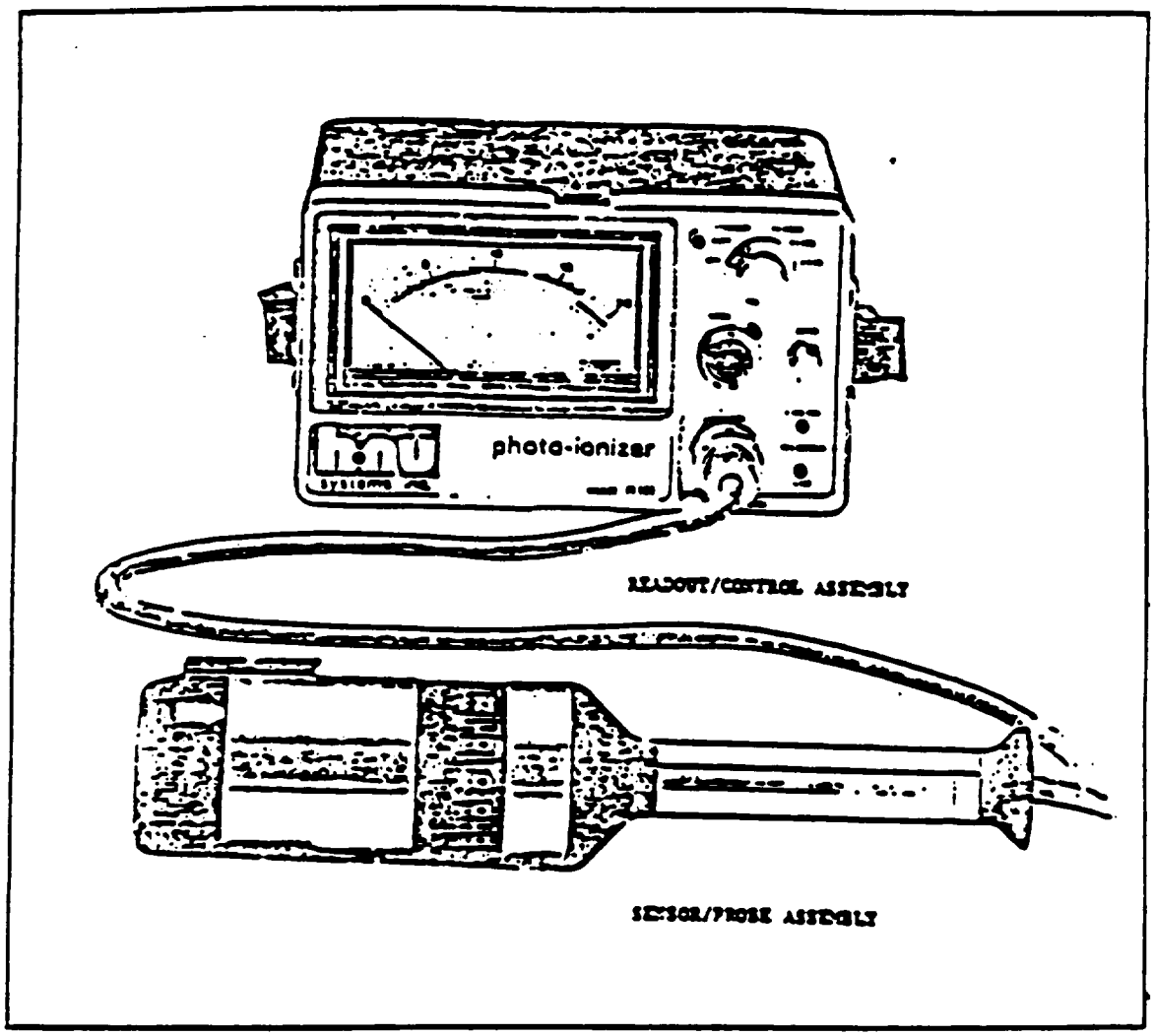

FIGURE 11-1

HNu PORTABLE PHOTOIONIZER

4.2.3 Controls

*These guidelines are adapted from FIT Operation and Field Manual, HNu Systems PI 101 Photoionization Detector and Century Systems Models OVA-128 Organic Vapor Analyzer, Ecology and Environment, Inc., 1982.

Following are the controls on the Readout/Control Assembly (refer to Figure II-2):

1. Six-position function switch: Selects functions according to the following:
   a. OFF: Complete power shutdown.
   b. BATT: Verifies the condition of the battery.
   c. STANDBY: Engergizes entire unit except UV lamp. Used to zero instrument and to conserve power.
   d. Ranges 0 to 20, 0 to 200, 0 to 2000: Direct reading span of the meter face, in ppm.
2. ZERO potentiometer: Electronically zeroes the instrument.
3. SPAN potentiometer: Increases or decreases the sensitivity of the instrument with respect to full-scale deflection. Used to calibrate instrument with specific span gas.
4. RECORDER output jacks: 0 to -5 VDC signal output for recorder.
5. RECORDER power jack: Provides 12 VDC to drive recorder.

4.2.4 Battery Charging

To charge the battery, plug the charger into the jack on th side of the instrument case. The battery is fully charges after 14 hours of charging. Disconnect 120 VAC power before disconnecting the charger plug. A full charge provides about 10 continuous hours of operation. The instrument will always be left on charge when not in use.

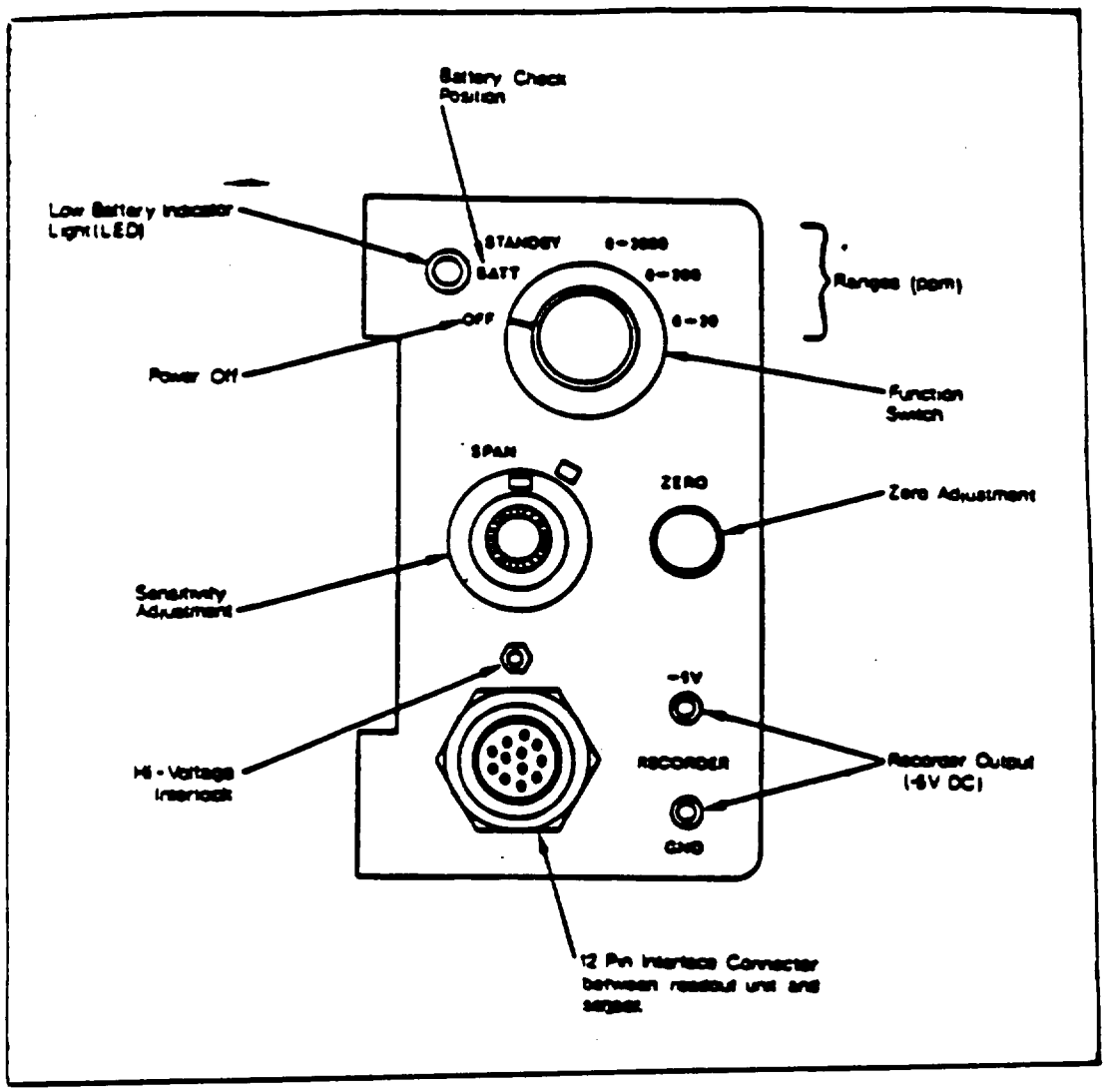

FIGURE 11-2

HNu CONTROL PANEL SCHEMATIC

The instrument is equipped with an automatic cut-off circuit which turns off the power if the battery voltage drops below 11 VDC. This prevents accidental damage to the electronics if it is inadvertently left on. Note that the unit can be operated with the charger on, unless it is in a hazardous (explosive) environment; however, it must be charged in a non-hazardous (non-explosive) area.

4.2.5 Operation

The startup procedure is as follows:

1. Connect sensor probe to readout/control assembly.

2. Turn function switch to BATT and verify condition of the battery.

3. Turn function switch to STANDBY and utilizing ZERO potentiometer, set meter to zero. Hold sensor/probe next to your ear to verify that the fan is working.

4. Set SPAN control to 9.8 or to desired setting (see Section 4.4).

5. Select appropriate range. For most survey operations, the setting used is 0 to 20 ppm. A violet-colored glow from the UV lamp source should be observable at the sample inlet of the Probe/Sensor unit. (Avoid looking directly in since eye damage can result.)

6. Verify instrument operation. A convenient method is to gently blow into the probe. There should be a 1 to 2 ppm deflection.

To shut the unit off, turn function switch to OFF and disconnect the sensor/probe.

4.3 INSTRUMENT SPAN

The SPAN potentiometer increases or decreases the sensitivity of the instrument. (Counter-clockwise rotation increases the sensitivity.) At there recommended span setting of 9.8, it quantitatively response to benzene, if benzene is the sole chemical species present. At this setting it will also respond, but not quantitatively, to all molecules with an IP of less than the energy of the UV lamp. The response to a molecule other than benzene (at the 9.8 setting) may be greater or less than that of benzene (on a volume/volume basis), depending on the molecule's type and structure. If a quantitative response to a specific chemical compound is desired,

then the instrument span must be calibrated with that compound.

4.4 IONIZATION POTENTIALS

Since the HNu can be used as both a safety and a survey device, each unit is supplied with the 10.2 and the 11.7 eV UV sources. This allows the instrument to be used to detect a wide variety of chemical species. A list of the IP Of many chemical compounds appears in Attachment A. Qualitative identification of compounds is not possible.

4.5 CALIBRATION

Primary calibration of the HNu is accomplished at the factory. The calibration standard used is benzene and the SPAN potentiometer reading is 9.8. P rimary calibration is normally stable for a long time. Routine calibration is most easily accomplished by using a manufacturer-supplied cylinder of calibration gas. A sample of the calibration gas is drawn into the instrument and the SPAN potentiometer is adjusted until the instrument is reading the exact concentration of the calibration gas. Small deviations from the span setting over time are normal. Deviations of greater than ± 5% indicate that the lamp window may need cleaning or, if that does not eliminate the deviation, the unit needs servicing. Routine calibration performed prior to each field use serves as an operational check to ensure that the instrument is responding properly. Records of routine calibration should be placed on file.

4.6 DATA

Any quantitative data obtained with the HNu must be reported as the equivalent value of its span gas. That is, with the span set to 9.8, a reading of 20 ppm would be reported as "20 ppm, benzene equivalent, span = 9.8." If a span setting of other than 9.8 is used, the data must be referenced to that particular span and/or calibration gas.

4.7 MAINTENANCE

The following subsections describe the minimum routine maintenance necessary. The instrument contains only one moving part and consumes no gases or reagents.

4.7.1 Cleaning UV Light Source Window

The only routine maintenance procedure specified by the manufacturer is cleaning the light source window every few weeks. The procedure is accomplished as follows:

1. Turn the function switch to the OFF position and disconnect the Sensor/Probe from the Readout/Control Unit.

2. Remove the exhaust screw found near the base of the probe. Grasp the end cap in one hand and the Probe shell in the other and gently pull to separate the end cap and lamp housing from the shell.

3. Loosen the screws on the top of the end cap and separate the end cap and ion chamber from the lamp and lamp housing. Care must be taken so that the ion chamber doesn't fall out of the end cap and the lamp doesn't slide out of the lamp housing. Turn the end cap over in your hand and tap on the top of it; the ion chamber should fall out in your hand.

4. Place one hand over the top of the lamp housing and tilt slightly; the light source will slide out of the housing. The lamp window may now be cleaned with the manufacturer-supplied cleaning compound.

5. Following the completing of cleaning, reassemble the unit by first sliding the lamp back into the lamp housing. Then place the ion chamber on top of the lamp housing, checking to make sure that the contacts are properly aligned.

6. Place the end cap on top of the ion chamber and replace the two screws. The screws should be tightened only enought to seal the "O" ring. DO NOT OVERTIGHTEN. Line up the pins on the base of the lamp housing with pins inside the probe shell. Gently slide the housing assembly into the shell; it only fits one way.

7. Replace the exhaust screw.

Figure 11-3 shows the component parts of the Probe Assembly.

4.7.2 "Fogging" of UV Light Source Window

During cold weather operations, condensation may form on the UV light source

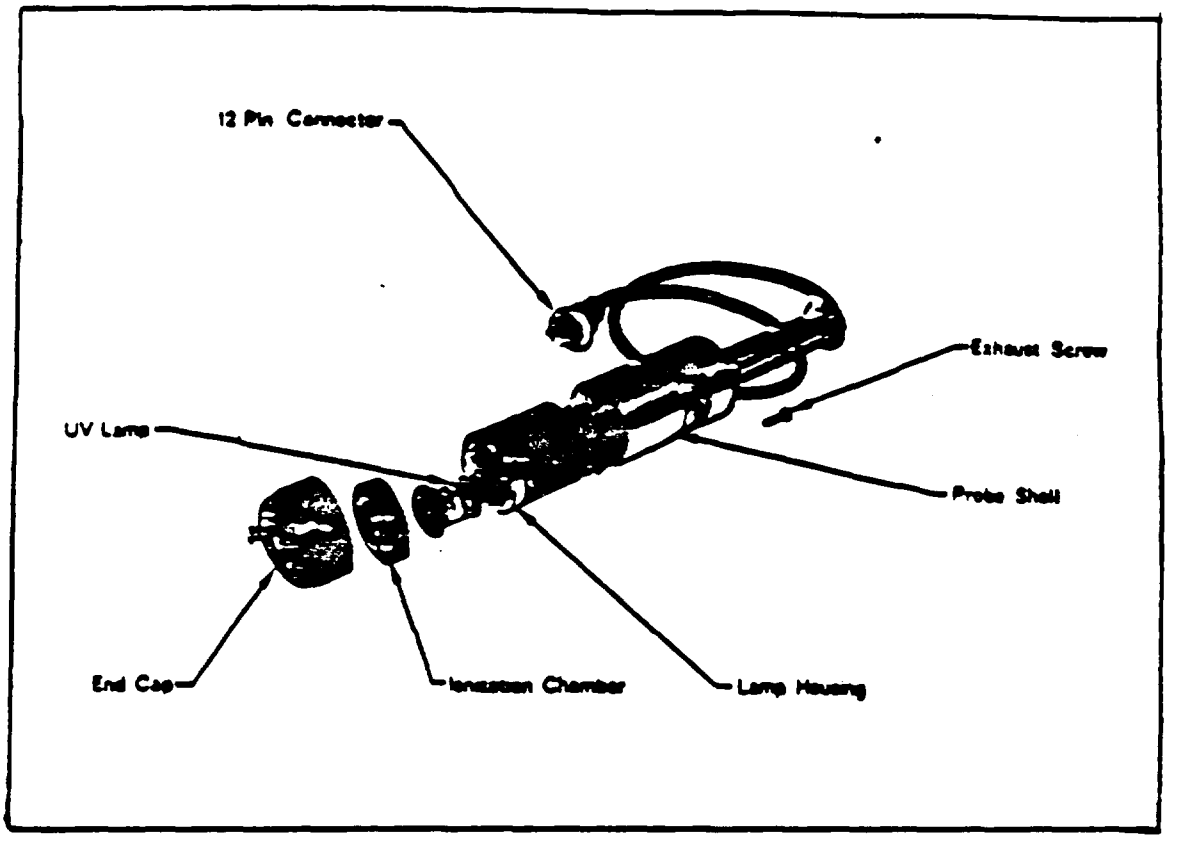

FIGURE 11-3

COMPONENT PARTS OF THE HNu PROBE ASSEMBLY

window, resulting in reduced levels of response. In this case, remove the lamp to clear the UV light source window of condensation. Consideration should also be given to more frequent cleaning when th instrument is used under very dusty conditions, such as on a landfill in dry weather.

4.8 TROUBLESHOOTING

If any malfunctions are noted during the start-up and operational check of the HNu prior to field use, refer to Attachment B for some basic troubleshooting guidance. Any problems which cannot be resolved quickly by the field operator should be referred to the factory for correction. (See the resource list at the end of the manual for the address and telephone number.)

4.9 SAFETY AND SHIPPING

The HNu can be carried only any aircraft as luggage; however, do not check it unless it has been carefully packaged. Commercial airlines will not insure it unless it is shipped in its original container.

The HNu has Factory Mutual (FM) Certification for operation in Class 1, Division 2 of the National Electrical Code. Therefore, the HNu should not be used in environments which are not above 10 percent of the lower explosive limit (LEL), since the circuitry in the probe assembly is relatively open and could be an ignition source.

4.10 GENERAL FIELD APPLICATIONS

This section describes the application of the HNu for general field purposes. The types of application described would typically be associated with a initial involvement with a site. The use of the HNu for more specialized applications such as sample screening and use during hydrogeologic investigations is described in Sections 4.11 and 4.12.

4.10.1 Ambient Air Characterization

A basic application of the HNu is in the characterization of the air quality at a site, which should be a primary objective for an initial site entry operation. The data obtained from a site air characterization study can be integrated with other available data to assist field personnel to evaluate whether the air quality at a site may be endangering the surrounding population and to determine the appropriate level of respiratory protection for subsequent site operations.

The HNu operator can be included as part of an initial site entry team. The other members of the initial site entry team are responsible for operating the characterization equipment which is standard for initial site entry (I.e., oxygen meter, explosimeter, radiation survey equipment, and Draeger tubes). The HNu does not replace any of these pieces of equipment. Once a thorough site characterization has been performed, it may be possible and desirable to eliminate the use of all but the HNu, depending on site activities.

Preparation

The HNu can be prepared for onsite air characterization by first starting it up (Section 4.2.5) and then taking steps to protect it from contamination by bagging the Readout/Control Assembly in plastic, wrapping the power cord in plastic, and bagging th Sensor/Probe Assembly. Care should be taken to avoid covering the air sample inlet. Before anyone crosses the hot line, a background reading should be taken and recorded. Initial operation should take place with th function switch set on he 0 to 20 ppm range.

Operation

Onsite air characterization is carried out is a manner similar to that described in the guidelines for using the OVA. The function switch should remain set on the 0 to 20 ppm range, with changes made to the less sensitive ranges as site conditions dictate.

Use of the 11.7 eV and 10.2 eV Sensor/Probe Assemblies

The initial survey should be made with the 10.2 eV Sensor/Probe Assembly. Although the 11.7 eV assembly detects a greater variety of compounds, its operating life is considerably less than that of the 10.2 eV. If significant readings are noted at

various site locations, the 10.2 eV unit can be exchanged at the hot line for the 11.7 eV unit. (Note: Turn the instrument OFF when charging Sensor/Probe Assemblies.) Second readings can than be made where response to the 10.2 eV unit occurred.

The fact that neither the 10.2 nor 11.7 eV Sensor/Probe Assembly will respond to methane is helpful at a landfill site since the HNu will register response only to those compounds of greater interest. If an OVA is used in tandem, the difference in response between it and the HNu can help to approximate total concentrations of methane and non-methane organics. Additional information can be interpreted from the knowledge that the HNu may also respond to certain inorganic vapors or gases.

Decontamination

Decontamination procedures are the same as outline for the OVA.

Data Evaluation

As is the case with evaluating data obtained from an initial site air characterization using the OVA, the HNu readings must be integrated with other available data. Of possible significance is the fact that the HNu response to inorganic species as well as organic.

Limitations of HNu Use for Ambient Air Characterization

Use of the HNu for site ambient air characterization has the following limitations:

1. The lower limit of detection for the instrument is approximately 1 ppm. Some air contaminants have lower odor thresholds than 1 ppm.

2. Further site work is necessary before decisions can be made about the level of respiratory protection.

3. Since the HNu operates on the principle of photoionization, variations in oxygen concentrations will not affect detector operations.

4. Since the HNu does not respond to methane, an explosimeter and oxygen meter should be used on initial site entry or in areas where potentially explosive concentrations of methane could accumulate.

5. The "capture velocity" of the HNu is less than that of the OVA. This

means that the air sample inlet of the Sensor/Probe Assembly must be closer to a gas/vapor source than the OVA probe. Also, air from a smaller area is drawn in by the HNu. The use of accessory pumps to increase the capture velocity is currently being evaluated.

6. When the air temperature is below 40 F, fogging of the UV lamp source may occur reducing response (see Section 4.9.2).

7. A relative humidity higher than 95 percent will cause inaccurate and unstable responses.

4.10.2 Identification of Potential Sampling Points

The HNu can be of value in helping to locate containers and/or areas of a site which might produce samples having significant concentrations of contaminants. The types of contaminants detected in this application are generally volatile organic compounds, unless the HNu is being used to detect an inorganic vapor or gas. This type of activity should follow the initial site entry/ambient air characterization operations describe in Section 4.11.1. In addition, the level of respiratory and personnel protection used for identifying potential sampling points should be carefully evaluated since the potential for exposure to contaminants is greater.

Containers

The sampling probe of the HNu can be inserted into the headspace of any open containers, such as drums or tanks. This operation should be performed with the HNu function switch set on "0 to 2000." This may provide an indication that the contents are volatile, depending on the length of time that the containers have been exposed to weathering, dilution, etc.

CAUTION: Avoid inadvertent immersion of the probe into the container contents. Should this happen, shut down the instrument immediately and take it offsite for immediate cleaning.

Soil

A "quick-and-dirty" identification of potential subsurface soil sampling points can be made by using a shovel to dig shallow holes and placing the probe of the HNu into the hole to detect the presence of spilled, leaked, or discharged volatile organics. A scaled-up version of this technique could involve the use of a backhole to excavate test pits. This technique is a relatively fast and inexpensive way to track subsurface contaminant migration without actually installing bore holes and wells.

An approach which is in the prototype phase of development is to drive a hollow soil probe into the ground, use a portable vacuum pump to withdraw vapor from the probe and take a reading with the HNu. In this way, it would be possible to track shallow subsurface contamination with minimal disturbance and cost.

CAUTION: Any subsurface exploration should be preceded by a determination that buried hazards do not exist. Care should also be taken to avoid clogging the instruments' sampling probes.

Air

An initial site entry/ambient air characterization should have identified any areas on the site where significant concentrations of contaminants were noted. This information can provide the basis for a more intensive air sampling program. "Hot spots" previously identified may be expedient locations from which real-time or integrated air samples may be taken. (Meteorological and other pertinent data should be evaluated before initiating an air sampling program. Sections 4.11 and 4.12 provide further guidance.)

Water

See Section 4.11 for information on locating potential water sampling points.

4.10.3 Respiratory Protection

One of the objectives of the ambient air characterization/initial site entry procedure is to obtain information to help in evaluating the level of respiratory protection for subsequent site operations. The HNu readings recorded during the initial air characterization are TOTAL vapor/gas concentrations and, unless further work is done, do not provide the qualitative/quantitative data which are required by regulatory agencies such as the Occupational Safety and Health Administration (OSHA) and National Institute for Occupational Safety and Health (NIOSH) for setting levels of respiratory protection. Therefore, it is impossible to safely or legally establish "action levels" for respiratory protection based upon HNu readings.

Even if HNu readings on the site do not exceed the recorded ambient background level, other site information must also point to the absence of a respiratory hazard before a decision can be made. Further, if subsequent site activity such as drum sampling or removal or excavation may alter the air quality, additional monitoring and respiratory protection will be required.

Thus, if it is determined that a respiratory hazard exists at a site, an SCBA must be worn. Air-purifying respirators are permitted only when the contaminant identities and concentrations are known, continued monitoring takes place, and sufficient oxygen levels are present (greater than 19.5 percent).

4.11 SPECIAL APPLICATIONS OF THE HNu

Special applications may include monitoring for vapors or gases and/or actual field analysis.

4.11.1 Monitoring

The use of the HNu for monitoring purposes involves simply focusing the site ambient air characterization process on a specific activity. The objective is to ensure that the personnel involved in the activity are not endangered by the changes in air quality caused by the activity. The following subsections address some specific monitoring applications. Personnel who are trained HNu operators are encouraged to expand these monitoring applications on the basis of personal field experience.

Hydrogeologic Investigations

At sites where volatile organic wastes are buried, the installation of bore holes and monitoring wells can expose personnel to a respiratory hazard. Often, the preliminary air characterization of the site and other data, such as the presence or absence of wastes on the site surface, may indicate that there is no respiratory hazard. However, the drill bit may penetrate contaminant-saturated soils or highly contaminated ground water. Depending upon the temperature and other conditions, dangerous concentrations of volatiles may be present in the area immediately above the bore hole. The personnel in the immediate vicinity of the bore hole are at greatest risk. The OVA in the survey mode or the HNu may be used to monitor the potential for exposure by periodically placing the sampling probe of the instrument directly at the top of the bore hole (drive casing). As is the case with ambient air monitoring, there can be no "action levels" since the readings will be total concentrations. Project personnel must use their best professional judgment, incorporating other conditions, in order to decide whether the use of SCBA is warranted or whether engineering controls such as fans can be used to reduce the possibility of exposure.

The HNu is well suited for this type of monitoring for several reasons. First, it will not respond to naturally occurring methane. Second, it is less cumbersome and readily operated by a wider variety of personnel. Third, if both an HNu and an OVA are available, the HNu can be used to monitor the drilling operation while the OVA is being used in an on-location laboratory
for field analysis. The probe of the HNu can also be used in the drilling area to "sniff" solid core samples as they are brought up in a split-spoon sampler. Care should be taken to identify occasional readings caused by exhaust gases from th drilling rig.

Remedial Response

A variety of remedial response activities can result in altering ambient air quality. Use of the HNu to monitor air quality can provide real-time data which can be used to evaluate respiratory protection needs. The following list is representative of the types of work which, if carried out at sites where volatile organics were the principal compounds, might require HNu monitoring:

1. Overpacking or pump transfer of leaking drums
2. Transfer of individual drum contents to bulk tanks for removal and disposal
3. Staging/containment operations of leaking drums
4. Excavation of buried leaking drums
5. Excavation of contaminated soil for removal and disposal
6. Emergency or planned containment operations (e.g., construction of leachate collection ditches)
7. Emergency treatment operations (e.g., air-stripping of contaminated ground water)
8. Emergency/onsite disposal operations (e.g., onsite incineration, detonation where risk/cost prohibits offsite removal)

4.11.2 Field Analysis

To a limited degree, the HNu can be used in the field to perform preliminary analyses of various air, water, or soil samples. The data generated can be of significant value in making field decisions, responding rapidly to an immediate threat, reducing analytical costs, and selecting future sampling locations. The following is a list of several of these field analytical applications:

1. Preliminary volatile organic analyses of soil, surface water, or ground-water samples obtained during site inspections.

2. Analysis of soil core samples for volatile organics during monitoring well installations

3. Analysis of drilling wash water samples during monitoring well installation to prevent accidental contamination of clean wells

4. Preliminary analysis of air samples to establish best locations for integrated sample stations

5. Rapid preliminary analysis of tap water samples for volatile organics

Once samples have been obtained, the field analytical procedure is essentially common for all types of sample media. Section 4.12 details the procedures for preliminary field analysis, or, sample screening.

4.12 SAMPLE SCREENING WITH THE HNu

Preliminary analysis for volatile organic compounds, or sample screening, can expand the capabilities of a field investigation. A large number of sites investigated have involved the disposal of wastes containing volatile organic compounds. This presents the opportunity to use the HNu to generate data which can facilitate an investigation by providing timely information, helping to focus sampling efforts on appropriate site areas, and reducing field and analytical costs.

Sample screening can be carried out virtually anywhere. All that is required is a small table and th appropriate accessories.

The HNu may be used to determine the presence or absence of volatiles in soil or ground-water samples by using the following technique:

1. Collect the soil or water sample in a glass container (volatile organics analysis septum vial not needed) leaving a 25 percent headspace.

2. Shake water samples or allow soil samples to thermally equilibrate. (Samples may also be warmed a water bath.)

3. Remove sample container cover and insert HNu probe extended into headspace. Record reading.

4.13 HNu TROUBLESHOOTING GUIDE

The following problems may occur with the use of the HNu; solutions ar provided:

1. No meter response in anyswitch position (including BATT CHK)

   a. Broken meter movement

      (1) Tip instrument rapidly from side to side. Meter needle should move freely, and return to zero.

   b. Electrical connection to meter is broken

      (1) Check all wires leading to meter and clean the contacts of quick-disconnects.

   c. Battery is completely dead.

      (1) Disconnect battery and check voltage with a volt-ohm meter.

   d. Check 2 amp fuse.

   e. If none of the above solves the problem, consult the factory.

2. Meter responds in BATT CHK positions, but reads zero or near zero for all others

   a. Input transistor or amplifier has failed

      (1) Rotate zero control; meter should deflect up/down as control is turned.

      (2) Check components on back side of printed circuit board. All connections should be solid, and no wires should touch any other object.

      (3) Check all wires in readout for solid connections.

3. Instrument responds correctly in BATT CHK, and STBY, but not in measuring mode

   a. Check to see that light source is on.

4. Instrument responds correctly in all positions, but signal is lower than expected

   a. Check span setting for correct value.

   b. Clean window of light source.

   c. Double check preparation of standards.

   d. Check for proper fan operation.

   e. Rotate span setting. Response should change if span pot is working properly.

5. Instrument responds in all switch positions, but is noisy (erratic meter movement)

   a. Open circuit in feedback circuit. Consult the factory.

   b. Open circuit in cable shield or probe shield. Consult the factory.

6. Instrument response is slow and/or irreproducible

   a. Indicator comes on if battery charge is low.

   b. Indicator also comes of if ionization voltage is too high.

Attachment A

Ionization Potentials of Selected Molecules

The HNu can be prepared for use in onsite air characterization. The initial survey should be made with th 10.2-eV sensor/probe assembly. In significant readings are noted at various site locations, the 10.2-eV unit can be exchanged at the hot line for the 11.7-eV unit.

When significant differences between the two reading are noted, Tables A-1 through A-13 can be consulted for the possibility of ruling out the presence or absence of certain groups of contaminants on the basis of ionization potential. Quantification identification is not possible

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Table A-1. Ionization Potentials (IP) of Some Atoms and Simple Molecules

Molecule	IP (eV)	Molecule	IP (eV)
H	13.595	I_2	9.28
C	11.264	HF	15.77
N	14.54	HCl	12.74
O	13.614	HBr	11.62
Si	8.149	HI	10.38
S	10.357	SO_2	12.34
P	17.42	CO_2	13.79
Cl	13.01	COS	11.18
Br	11.84	CS_2	10.08
I	10.48	N_2O	12.90
H_2	15.426	NO_2	9.78
N_2	15.580	O_3	12.80
O_2	12.075	H_2O	12.59
CO	14.01	H_2S	10.46
CN	15.13	H_2Se	9.88
NO	9.25	H_2Te	9.14
CH	11.1	HCN	13.91
OH	13.18	C_2N_2	13.8
F_2	15.7	NH_3	10.15
Cl_2	11.48	CH_3	9.840
Br_2	10.55	CH_4	12.98

Table A-2. Ionization Potentials (IP) of Some Paraffins and Cycloparaffins

Molecule	IP (eV)
Methane	12.98
Ethane	11.65
Propane	11.07
n-butane	10.63
i-butane	10.57
n-pentane	10.35
i-pentane	10.32
2,2-dimethylpropane	10.35
n-hexane	10.18
2-methylpentane	10.12
3-methylpentane	10.08
2,2-dimethylbutane	10.06
2,3-dimethylbutane	10.02
n-heptane	10.08
2,2,4-trimethylpentane	9.86
Cyclopropane	10.06
Cyclopentane	10.53
Cyclohexane	9.88
Methylcyclohexane	9.85

Table A-3. Ionization Potentials (IP) of Some Alkyl Halides

Molecule	IP (eV)
HCl	12.74
Cl_2	11.48
CH_4	12.98
Methyl chloride	11.28
Dichloromethane	11.35
Trichloromethane	11.42
Tetrachloromethane	11.47
Ethyl chloride	10.98
1,2-dichloroethane	11.12
1-chloropropane	10.82
2-chloropropane	10.78
1,2-dichloropropane	10.87
1,3-dichloropropane	10.85
1-chlorobutane	10.67
2-chlorobutane	10.65
1-chloro-2-methylpropane	10.66
2-chloro-2-methylpropane	10.61
HBr	11.62
Br_2	10.55
Methyl bromide	10.53
Dibromomethane	10.49
Tribromomethane	10.51
CH_2BrCl	10.77

Table A-3. Ionization Potentials (IP) of Some Alkyl Halides (Continued)

Molecule	IP (eV)
$CHBr_2Cl$	10.59
Ethyl bromide	10.29
1,1-dibromoethane	10.19
1-bromo-2-chloroethane	10.63
CF_2Br_2	11.07
CH_3CF_2Cl (Genetron 101)	11.98
$CFCl_2CF_2Cl$	11.99
CF_3CCl_3 (Freon 113)	11.78
$CFHBrCH_2Br$	10.75
CF_2BrCH_2Br	10.83
CF_3CH_2I	10.00
$n\text{-}C_3F_7I$	10.36
$n\text{-}C_3F_7CH_2Cl$	11.84
$n\text{-}C_3F_7CH_2I$	9.96
1-bromopropane	10.18
2-bromopropane	10.075
1,3-dibromopropane	10.07
1-bromobutane	10.13
2-bromobutane	9.98
1-bromo-2-methylpropane	10.09
2-bromo-2-methylpropane	9.99
1-bromopentane	10.10

Table A-3. Ionization Potentials (IP) of Some Alkyl Halides (Continued)

Molecule	IP (eV)
HI	10.38
I_2	9.28
Methyl iodide	9.54
Diiodomethane	9.34
Ethyl iodide	9.33
1-iodopropane	9.26
2-iodopropane	9.17
1-iodobutane	9.21
2-iodobutane	9.09
1-iodo-2-methylpropane	9.18
2-iodo-2-methylpropane	9.02
1-iodopentane	9.19
F_2	15.7
HF	15.77
$CFCl_3$ (Freon 11)	11.77
CF_2Cl_2 (Freon 12)	12.31
CF_3Cl (Freon 13)	12.91
$CHClF_2$ (Freon 22)	12.45

Table A-4. Ionization Potentials (IP) of Aliphatic Alcohol, Ether, Thiol, and Sulfides

Molecule	IP (eV)
H_2O	12.59
Methyl alcohol	10.85
Ethyl alcohol	10.48
n-propyl alcohol	10.20
i-propyl alcohol	10.16
n-butyl alcohol	10.04
Dimethyl ether	10.00
Diethyl ether	9.53
n-propyl ether	9.27
i-propyl ether	9.20
H_2S	10.46
Methanethiol	9.440
Ethanethiol	9.285
1-propanethiol	9.195
1-butanethiol	9.14
Dimethyl sulfide	8.685
Ethyl methyl sulfide	8.55
Diethyl sulfide	8.430
Di-n-propyl sulfide	8.30

Table A-5. Ionization Potentials (IP) of Some Aliphatic Aldehydes and Ketones

Molecule	IP(eV)
CO_2	13.79
Formaldehyde	10.87
Acetaldehyde	10.21
Propionaldehyde	9.98
n-butyraldehyde	9.86
Isobutyraldehyde	9.74
n-valeraldehyde	9.82
Isovaleraldehyde	9.71
Acrolein	10.10
Crotonaldehyde	9.73
Benzaldehyde	9.53
Acetone	9.69
Methyl ethyl ketone	9.53
Methyl n-propyl ketone	9.39
Methyl i-propyl ketone	9.32
Diethyl ketone	9.32
Methyl n-butyl ketone	9.34
Methyl i-butyl ketone	9.30
3,3-dimethyl butanone	9.17
2-heptanone	9.33

Table A-5. Ionization Potentials (IP) of Some Alphatic Aldehydes and Ketones (Continued)

Molecule	IP(eV)
Cyclopentanone	9.26
Cyclohexanone	9.14
2,3-butanedione	9.23
2,4-pentanedione	8.87

Table A-6. Ionization Potentials (IP) of Some Aliphatic Acids and Esters

Molecule	IP (eV)
CO_2	13.79
Formic acid	11.05
Acetic acid	10.37
Propionic acid	10.24
n-butyric acid	10.16
Isobutyric acid	10.02
n-valeric acid	10.12
Methyl formate	10.815
Ethyl formate	10.61
n-propyl formate	10.54
n-butyl formate	10.50
Isobutyl formate	10.46
Methyl acetate	10.27
Ethyl acetate	10.11
n-propyl acetate	10.04
Isopropyl acetate	9.99
n-butyl acetate	10.01
Isobutyl acetate	9.97
Sec-butyl acetate	9.91
Methyl propionate	10.15
Ethyl propionate	10.00
Methyl n-butyrate	10.07
Methyl isobutyrate	9.98

Table A-7. Ionization Potentials (IP) of Some Aliphatic Amines and Amides

Molecule	IP (eV)
NH_3	10.15
Methyl amine	8.97
Ethyl amine	8.86
n-propyl amine	8.78
i-propyl amine	8.72
n-butyl amine	8.71
i-butyl amine	8.70
s-butyl amine	8.70
t-butyl amine	8.64
Dimethyl amine	8.24
Diethyl amine	8.01
Di-n-propyl amine	7.84
Di-i-propyl amine	7.73
Di-n-butyl amine	7.69
Trimethyl amine	7.82
Triethyl amine	7.50
Tri-n-propyl amine	7.23
Formamide	10.25
Acetamide	9.77
N-methyl acetamide	8.90
N,N-dimethyl formamide	9.12

Table A-8. Ionization Potentials (IP) of Other Aliphatic Molecules with N Atom

Molecule	IP (eV)
Nitromethane	11.08
Nitroethane	10.88
1-nitropropane	10.81
2-nitropropane	10.71
HCN	13.91
Acetonitrile	12.22
Propionitrile	11.84
n-butyronitrile	11.67
Acrylonitrile	10.91
3-butene-nitrile	10.39
Ethyl nitrate	11.22
n-propyl nitrate	
Methyl thiocyanate	10.065
Ethyl thiocyanate	9.89
Methyl isothiocyanate	9.25
Ethyl isothiocyanate	9.14

Table A-9. Ionization Potentials (IP) of Some Olefins, Cyclo-olefins, Polenes, Acetylenes

Molecule	IP (eV)
Ethylene	10.515
Propylene	9.73
1-butene	9.58
2-methylpropene	9.23
Trans-2-butene	9.13
Cis-2-butene	9.13
1-pentene	9.50
2-methyl-1-butene	9.12
3-methyl-1-butene	9.51
3-methyl-2-butene	8.67
1-hexene	9.46
1,3-butadiene	9.07
Isoprene	8.845
Cyclopentene	9.01
Cyclohexene	8.945
4-methylcyclohexene	8.91
4-cinylcyclohexene	8.93
Cyclo-octatetraene	7.99
Acetylene	11.41
Propyne	10.36
1-butyne	10.18

Table A-10. Ionization Potentials (IP) of Some Derivatives of Olefins

Molecule	IP (eV)
Vinyl chloride	9.995
Cis-dichloroethylene	9.65
Trans-dichloroethylene	9.66
Trichloroethylene	9.45
Tetrachloroethylene	9.32
Vinyl bromide	9.80
1,2-dibromoethylene	9.45
Tribromoethylene	9.27
3-chloropropene	10.04
2,3-dichloropropene	9.82
1-bromopropene	9.30
3-bromopropene	9.7
$CF_3CCl=CClCF_3$	10.36
$n\text{-}C_5F_{11}CF=CF_2$	10.48
Acrolein	10.10
Crotonaldehyde	9.73
Mesityl oxide	9.08
Vinyl methyl ether	8.93
Allyl alcohol	9.67
Vinyl acetate	9.19

Table A-11. Ionization Potentials (IP) of Some Heterocyclic Molecules

Molecule	IP (eV)
Furan	8.89
2-methyl furan	8.39
2-furaldehyde	9.21
Tetrahydrofuran	9.54
Dihydropyran	8.34
Tetrahydropyran	9.26
Thiophene	8.860
2-chlorothiophene	8.68
2-bromothiophene	8.63
Pyrrole	8.20
Pyridine	9.32
2-picoline	9.02
3-picoline	9.04
4-picoline	9.04
2,3-lutidine	8.85
2,4-lutidine	8.85
2,6-lutidine	8.85

Table A-12. Ionization Potentials (IP) of Some Aromatic Compounds

Molecule	IP (eV)
Benzene	9.245
Toluene	8.82
Ethyl benzene	8.76
n-propyl benzene	8.72
i-propyl benzene	8.69
n-butyl benzene	8.69
s-butyl benzene	8.68
t-butyl benzene	8.68
o-xylene	8.56
m-xylene	8.56
p-xylene	8.445
Mesitylene	8.40
Durene	8.025
Styrene	8.47
α-methyl styrene	8.35
Ethynylbenzene	8.815
Napthalene	8.12
1-methylnapthalene	7.96
2-methylnapthalene	7.955
Biphenyl	8.27
Phenol	8.50
Anisole	8.22
Phenetole	8.13

Table A-12. Ionization Potentials (IP) of Some Aromatic Compounds (Continued)

Molecule	IP (eV)
Benzaldehyde	9.53
Acetophenone	9.27
Benzenethiol	8.33
Phenyl isocyanate	8.77
Phenyl isothiocyanate	8.520
Benzonitrile	9.705
Nitrobenzene	9.92
Aniline	7.70
Fluoro-benzene	9.195
Chloro-benzene	9.07
Bromo-benzene	8.98
Iodo-benzene	8.73
o-dichlorobenzene	9.07
m-dichlorobenzene	9.12
p-dichlorobenzene	8.94
1-chloro-2-fluorobenzene	9.155
1-chloro-3-fluorobenzene	9.21
1-bromo-4-fluorobenzene	8.99
o-fluorotoluene	8.915
m-fluorotoluene	8.915
p-fluorotoluene	8.785
o-chlorotoluene	8.83
m-chlorotoluene	8.83

Table A-12. Ionization Potentials (IP) of Some Aromatic Compounds (Continued)

Molecule	IP (eV)
p-chlorotoluene	8.70
o-bromotoluene	8.79
m-bromotoluene	8.81
p-bromotoluene	8.67
o-iodotoluene	8.62
m-iodotoluene	8.61
p-iodotoluene	8.50
Benzotrifluoride	9.68
o-fluorophenol	8.66

Table A-13. Ionization Potentials (IP) of Some Miscellaneous Molecules

Molecule	IP (eV)
Ethylene oxide	10.565
Propylene oxide	10.22
p-dioxane	9.13
Dimethoxymethane	10.00
Diethoxymethane	9.70
1,1-dimethoxyethane	9.65
Propiolactone	9.70
Methyl disulfide	8.46
Ethyl disulfide	8.27
Diethyl sulfite	9.68
Thiolacetic acid	10.00
Acetyl chloride	11.02
Acetyl bromide	10.55
Cyclo-$C_6H_{11}CF_3$	10.46
$(n\text{-}C_3F_7)(CH_3)C{=}O$	10.58
Trichlorovinylsilane	10.79
$(C_2F_5)_3N$	11.7
Isoprene	9.08
Phosgene	11.77

Attachment B

Relative Sensitivities for Various Gases

When the 10.2-eV lamp is used for onsite ambient air characterization, Table B-1 should be consulted.

HNu Photoionization Detector Operation
Disk MINN 4

Standard Operating Procedure 11
Attachment B
Date:

Table B-1 Relative Sensitivities For Various Gases (10.2-eV lamp)

Table B-1. Relative Sensitivities for Various Gases (10.2-eV lamp)

Species	Photoionization Sensitivity[a]
p-xylene	11.4
m-xylene	11.2
Benzene	10.0[b]
Toluene	10.0
Diethyl sulfide	10.0
Diethyl amine	9.9
Styrene	9.7
Trichloroethylene	8.9
Carbon disulfide	7.1
Isobutylene	7.0
Acetone	6.3
Tetrahydrofuran	6.0
Methyl ethyl ketone	5.7
Methyl isobutyl ketone	5.7
Cyclohexanone	5.1
Naphtha (86% aromatics)	5.0
Vinyl chloride	5.0
Methyl isocyanate	4.5
Iodine	4.5
Methyl mercaptan	4.3
Dimethyl sulfide	4.3
Allyl alcohol	4.2

Table B-1. Relative Sensitivities for Various Gases (Continued)

Species	Photoionization Sensitivity[a]
Propylene	4.0
Mineral spirits	4.0
2,3-dichloropropene	4.0
Cyclohexene	3.4
Crotonaldehyde	3.1
Acrolein	3.1
Pyridine	3.0
Hydrogen sulfide	2.8
Ethylene dibromide	2.7
n-octane	2.5
Acetaldehyde oxime	2.3
Hexane	2.2
Phosphine	2.0
Heptane	1.7
Allyl chloride (3-chloropropene)	1.5
Ethylene oxide	1.0
Acetic anhydride	1.0
α-pinene	0.7
Dibromochloropropane	0.7
Epichlorohydrin	0.7
Nitric oxide	0.6

Table B-1. Relative Sensitivities for Various Gases (Continued)

Species	Photoionization Sensitivity[a]
β-pinene	0.5
Citral	0.5
Acetic acid	0.1
Nitrogen dioxide	0.02
Methane	0.0
Acetylene	0.0
Ethylene	0.0

[a]Expressed in ppm (V/V).
[b]Reference standard.

Attachment C

Glossary

AC battery charger - A black rectangular box with two cords attached to it. One cord plugs into an AC outlet, while the other cord attaches to the instrument being recharged. The OVA charger has a charge on/off switch.

Backflush valve - On the OVA; used to reverse the hydrogen flow through the column; injected samples then flow directly to the detector.

B-column - One of the three types of columns made for the OVA. This column contains 3 percent Diisodecyl Phthalate on chromosorb WAW 60/80 mesh. This is a low polarity column.

Battery check - Instrument switch on both the OVA and the hNu that indicates the charge in the battery.

Battery pack - Located on the front of the OVA and on the back of the hNu; serves as the power source of the instrument.

Carrier gas - Used in the OVA to carry ambient air through the column and to the detector, or directly to the detector. H_2 is the gas.

Charcoal filter - There are two that can be used on the OVA. One is permanently attached and is used whenever a chromatogram is run. The other may be screwed into the probe/readout assembly.

Chromatogram - A finger print of a sample. Different peaks on a strip chart represent different volatile organic chemicals. A chromatogram is obtained after a syringe injection of headspace gas is made into the column through the T-adapter.

Column - A variable length nickel tube (usually 8", 12", or 24") that contains a certain parking (Type T, B, or G).

Concentration range selector - This is on the OVA and hNu. The desired range can be set when monitoring the ambient air. The OVA and hNu both have three settings: 1 to 10, 1 to 100, and 1 to 1000 on the OVA; while the hNu has 0 to 20, 0 to 200, and 0 to 2000.

DOT exemption - A letter of exemption of the OVA from the aircraft rules which do not allow flammable gas to be shipped. This exemption may allow the OVA to be shipped or carried full on passenger aircraft.

Electronic zero - Found on the hNu; it is used to adjust the zero electronically when the instrument is placed in the standby position with the probe attached.

Fan - In the hNu, it maintains a flow of sample gas through the ion chamber.

GLOSSARY

Flame arrestor - A gold colored metal screen that contains the flame of the flame ionization detector. It is located at the bottom of the OVA.

Flame ionization detector - A detector that uses a flame, fueled by hydrogen, to ionize individual contaminants as they emerge from the column. The ions are then attracted to an oppositely charged electrode, causing a current and finally an electric signal to the Strip Chart Recorder.

Flame-out alarm - Audible alarm that is activated when the flame in the flame ionization detector goes out.

Gas select knob - Found on the OVA, it provides a choice of setting where a certain gas can be used to calibrate the instrument.

G-column - One of the three types of columns made for the OVA. This column contains a 10 percent OV-101 on chromosorb W AW-DMCS treated 60/80 mesh.

Glow plug - Located in the chamber that supports the flame for the flame ionization detector. It provides the ignition source for the flame.

Headspace sample - A VOA vial filled three quarters of the way with water or soil. The remaining quarter of the vial is airspace.

Hydrogen - A diatomic gas that serves as the carrier gas and fuel supply for the OVA.

Hydrogen fill hose - A hose with a pressure gauge, an adaptor to a type 1A hydrogen cylinder, and an adaptor to the refill valve on the OVA. It serves to refill the OVA with hydrogen from the type 1A cylinder.

H_2 refill valve - This valve is only turned on when there is an open path through the hydrogen fill line to the type 1A cylinder.

H_2 supply pressure valve - In the open position this valve allows a measured flow of hydrogen through the column and on to the detector in the OVA.

H_2 tank valve - In the open position this allows the hydrogen from the OVA's cylinder to flow to the H_2 supply pressure valve.

Ignite button - When this is depressed the element in the glow plug glows red hot and causes ignition of the flame of the flame ionization detector.

Injection - This takes place when a syringe containing gas is introduced into the column through the septum contained in the T-adaptor, and depressed so as to enter its contents into the column.

Instrument switch - Found on the OVA, this switch turns on the electronics for the OVA except for the pump and glow plug.

Ionization energy - Energy needed to ionize gaseous molecules. This is usually expressed as ionization potential.

GLOSSARY

Isothermal column - A column that is wrapped around a hollow metal tube and then insulated with styrofoam. The purpose of this structure is to keep the column at a fixed temperature throughout the analysis.

Lamp - Found in the probe of the hNu, it emits ultraviolet light (photons) into the ionization chamber.

Mercury - Used in making standards for the OVA. A small amount is used to cover the septum when the standard is inverted for storage in order to prevent the solvent vapor from escaping.

Mylar bag - Used to hold a gas mixture, which can be introduced into the OVA or hNu through an attached hose or syringe injection.

"Off-scale" - Term used to describe the full scale deflection of the needle for a particular concentration on the OVA or hNu.

Packing - The inner contents of the chromatographic columns used for the OVA. The different packings are designated by letters: T, B, and G.

Parts per billion - 1 part of a chemical in 1 billion parts of air or water (by volume).

Parts per million - 1 part of a chemical in 1 million parts of air or water (by volume).

Photoionization - The absorption of ultraviolet light (a photon) by a molecule that leads to ionization. $RH + h\nu \rightarrow RH^+ + e^-$, where RH is a trace gas and hv is a photon with an energy greater than or equal to ionization potential of RH.

Photon - A quantity of ultraviolet light.

Porous filters - Particle filters that are found in the probe fixtures and at the junction of the umbilical cord and the side pack assembly.

Primary calibration gas - For the OVA this gas is a methane/air mixture, and the gas select knob is set at 3.0; while the hNu uses a benzene/air mixture and the span setting is 9.8.

Probe/Probe readout assembly (OVA) - Attached to the end of the umbilical cord of the OVA, it contains the inlet of the air sampling line and a dial with a linear scale readout.

Probe (hNu) - Attached at the end of the electrical cord that originates from the instrument panel of the hNu. The probe contains a lamp, ionization chamber and a fan to draw in sample gas to the ionization chamber.

Probe extender - both the OVA and hNu have one. It attaches to the end of the probe to shorten the distance one has to get to the source of interest.

GLOSSARY

Pump switch - Found on the OVA, when in the on position it pumps in ambient air at a rate of 2 units.

Retention time - The total time required for a volatile chemical to emerge from the column into the detector from the moment of introduction into the column of the OVA.

Sample screening - Determining total volatile organic chemical content of ambient air or a headspace sample by injection into the T-adaptor of the OVA while the OVA is in the backflush mode.

Sample flow rate gauge - This gauge is used to monitor the intake of ambient air by the pump.

Sample inject valve - When this valve is in the "up" position ambient air is pumped directly to the detector and the instrument is in the survey mode. If depressed during the survey mode, the ambient air is redirected through a charcoal filter before continuing to the detector.

Septum - This is a replaceable, circular, rubber disc with a ten millimeter diameter that fits into the septum adaptor.

Septum adaptor - This screws onto the T-adaptor and provides a guide for the syringe during injections.

Side Pack Assembly - This is the main unit of the OVA. It contains most of the operating controls and indicators, the electronic circuitry, detector chamber, hydrogen fuel supply and electrical power supply.

Standard - This is a known chemical that is in solution with distilled water and contained in a VOA vial in such a way that a headspace is present. A syringe can then withdraw some of the headspace gas after the vial is agitated, and this gas can then be injected into the column for chromatographic analysis. Comparison to unknown samples then follows.

Standby knob - This is found on the HNu and allows the HNu to warm up in a non-emergency demanding position. The instrument can also be electronically zeroed at this position.

Strip Chart Recorder - A ticking type recorder that forms a permanent record of the electronic signals that come from the detector.

Survey mode - The OVA is in this mode when the sample inject valve is in the up position. In this mode ambient air is pumped directly to the detector.

Syringe - A gastight hollow glass tube with a hollow needle on one end and a plunger on the other end that is used to collect headspace gas or ambient air for injections into the OVA.

T-Column - One of the three types of columns made for the OVA. This column contains a 1 percent, 1,2,3 Tris (2 cyanoethoxy) propane (also known as TCEP) on chromosorb W HP, 60/80 mesh.

GLOSSARY

Teflon tape - Used to obtain an airtight seal between connectors in the hydrogen line of the OVA.

Total volatile organic reading - Measurement of total volatile organic chemical content of ambient air.

Ultraviolet light - This light or radiation has a wavelength ($_b$) between 4000-2000 $\mathring{A}$.

Umbilical cord - Two cords intertwined bridging the side pack assembly of the OVA to the probe assembly. This umbilical cord contains an electronic cable and an ambient air sampling line.

UV transmitting window - Window on the UV lamp of the hNu that emits photons of UV light.

VOA Vial - A tubular glass vial with a rubber septum cap that is coated with Teflon on one side (usually 40 to 45 ml).

Volatile organics - Organic chemicals that have low boiling points and high vapor pressures.

SECTION 3

CALIBRATION

Excerpt from "Instruction Manual - Model PI-101, Portable Photoionization Analy HNu Systems, Inc., 1986. (Note: Secti 8.2 of the Appendix of that Manual is attached at end of this excerpt.

3.1 INTRODUCTION

The PI 101 Analyzer is designed for trace gas analysis in ambient air and is calibrated at HNU with certified standards of benzene, vinyl chloride and isobutylene. Other optional calibrations are available (e.g., ammonia, ethylene oxide, H2S, etc.). Calibration data is given in the data sheet. If a special calibration has been done, the data is given in the Application Data Sheet, which notes the sample source, type of calibration (see Section 8, Appendix), and other pertinent information.

Good instrumentation practice calls for calibration on the species to be measured in the concentration range to be used. This procedure assures the operator that the analyzer is operating properly and will generate reliable data.

Some general points to consider when calibrating the PI 101 are that the analyzer is designed for operation at ambient conditions and therefore the gas standards used for calibration should be delivered to the analyzer at ambient temperatures and pressure and at the proper flow rates.

WARNING:

The PI 101 is a non-destructive analyzer; calibrations using toxic or hazardous gases must be done in a hood.

The frequency of calibration should be dictated by the usage of the analyzer and the toxicity of the species measured. If the analyzer has been serviced or repaired, calibration should be done to verify operation and performance. It is recommended that calibration be checked frequently at first (daily or every other day) and then regularly based on the confidence level developed.

The normal meter scaleplate is 0 to 20. If the scaleplate is different, refer to the Application Data Sheet. If there are questions, consult the HNU representative before proceeding with calibration check.

An accurate and reliable method of calibration check is to use an analyzed gas cylinder in a test setup as shown in Figure 3-1 and described below. Additional material on calibration is given in Section 8, Appendix.

3.2 ANALYZED GAS CYLINDER

a. Concentration - The calibration gas cylinder is to contain the species of interest made up in an air matrix at or near the concentration to be analyzed. If the component is unstable in air, another matrix is to be used. The final calibration mixture should be similar to the sample the PI 101 will analyze. If the expected concentration is not known then a concentration should be chosen that will cause a scale displacement of 50 to 80% on the X10 range. Calibration on X10 range will

For use on the 0-2000 range, a two-standard calibration is preferred: one at 70 to 85% of the linear range and the other at 25 to 35% of the linear range. With the linear range of approximately 600 ppm for most compounds these points would lie between 420 to 510 ppm and 150 to 210 ppm, respectively.

b. Stability - The calibration gas must be stable within the cylinder during the period of use. If the calibration is required in the field, then use of a small cylinder is recommended. In addition, the choice of cylinder material in contact with the gas must be considered (steel, aluminum or teflon). If there are any questions, the operator should request stability and usage information from the gas supplier.

WARNING

Extreme care must be taken in the handling of gas cylinders. Contents are under high pressure. In some cases, the contents may be hazardous. Many gas suppliers will provide data sheets for the mixtures upon request.

c. Delivery - The cylinder containing the calibration mixture must be connected to a proper regulator.

WARNING

Never open the valve on a gas cylinder container without a regulator attached.

Leak test all tank/regulator connections as well as the main cylinder valve to prevent toxic or hazardous materials from leaking into the work area. Care must be taken that the materials of construction of the regulator will not interact with the calibration gas.

One method of sampling the calibration gas is illustrated in Figure 3-1. Connect the cylinder to one leg of the tee, a flow meter to the opposite leg, and the probe to the third leg. The flow meter does not require a valve. If there is a valve, it must be left wide open. The flowmeter is only to indicate excess flow. Adjust the flow from the regulator such that only a little excess flow is registered at the flowmeter.

This insures that the PI 101 sees the calibration gas at atmospheric pressure and ambient temperature.

d. Usage - Generally, a gas cylinder should not be used below 200-300 psi as pressure effects could cause concentration variations. The cylinder should not be used past the recommended age of the contents as indicated by the manufacturer. In case of difficulty, verify the contents and concentration of the gas cylinder.

e. Alternate means of calibration are possible. For more information, contact the HNU Service Department.

3.3 PROBE

a. Identify the probe by the lamp label. If a question exists, disassemble the probe and inspect the lamp. The energy of the lamp is etched into the glass envelope.

b. Connect the probe to the readout assembly, making sure the red interlock switch is depressed by the ring on the connector.

c. Set the SPAN pot to the proper value for the probe being calibrated. Refer to the calibration memo accompanying the probe.

d. Check the Ionization Potential (IP) of the calibration gas to be used. The IP of the calibration gas must be at or below the IP of the lamp.

e. Proceed with the calibration as described in Section 3.4. Check the calibration memo for specific data. If any questions develop, call the HNU representative.

f. NOTE: The 11.7eV lamp has a special cleaning compound. Do not use water or any other cleaning compound with the 11.7 eV lamp. Do not interchange ion chambers, amplifier boards or lamps between probes. (See Section 5.2).

3.4 PROCEDURE

a. Battery check - Turn the function switch to BATT. The needle should be in the green region. If not, recharge the battery.

b. Zero set - Turn the function switch to STANDBY. In this position the lamp is OFF and no signal is generated. Set the zero point with the ZERO set control. The zero can also be set with the function switch on the X1 position and using a "Hydrocarbon-free" air. In this case "negative" readings are possible if the analyzer measures a cleaner sample when in service.

c. 0-20 or 0-200 range - For calibrating on the 0-20 or 0-200 range only one gas standard is required. Turn the function switch to the range position and note the meter reading. Adjust the SPAN control setting as required to read the ppm concentration of the standard. Recheck the zero setting (step b.). If readjustment is needed, repeat step c. This gives a two-point calibration; zero and the gas standard point. Additional calibration points can be generated by dilution of the standard with zero air if desired (see Section 8).

d. 0-2000 range - For calibrating on the 0-2000 range, use of two standards is recommended as cited in Section 3.2a. First calibrate with the higher standard using the SPAN control for setting. Then calibrate with the lower standard using the ZERO adjustment. Repeat these several times to ensure that a good calibration is obtained. The analyzer will be appoximately linear to better than 600 ppm, (see Figure 3-2). If the analyzer is subsequently to be used on the 0-20 or 0-200 range, it must be recalibrated as described in steps b. and c. above.

e. Lamp cleaning - If the span setting resulting from calibration is 0.0 or if calibration cannot be achieved, then the lamp must be cleaned (see Section 5.2).

f. Lamp replacement - If the lamp output is too low or if the lamp has failed, it must be replaced (see Section 5.3).

3.5 CALIBRATION CHECKING

Rapid calibration checking in the field can be accomplished by use of a small disposable cylinder containing isobutylene. Immediately after a calibration has been completed, a reading is taken on a special isobutylene standard. This provides a reference concentration measurement for later checking in the field. This can be done at any time with a portable cylinder containing this same special standard, using this reference reading as a check, and making adjustments to the analyzer if necessary. In effect, this is an indirect method of calibration, one maintaining the calibration to give direct readings for the original gas mixture, but using the portable isobutylene cylinder. Details are given in Section 8.2 of the Appendix.

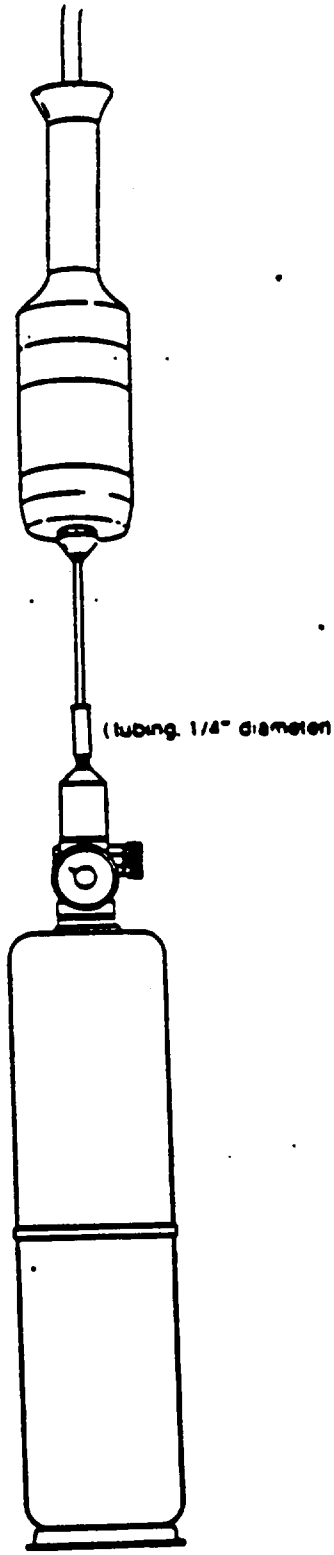
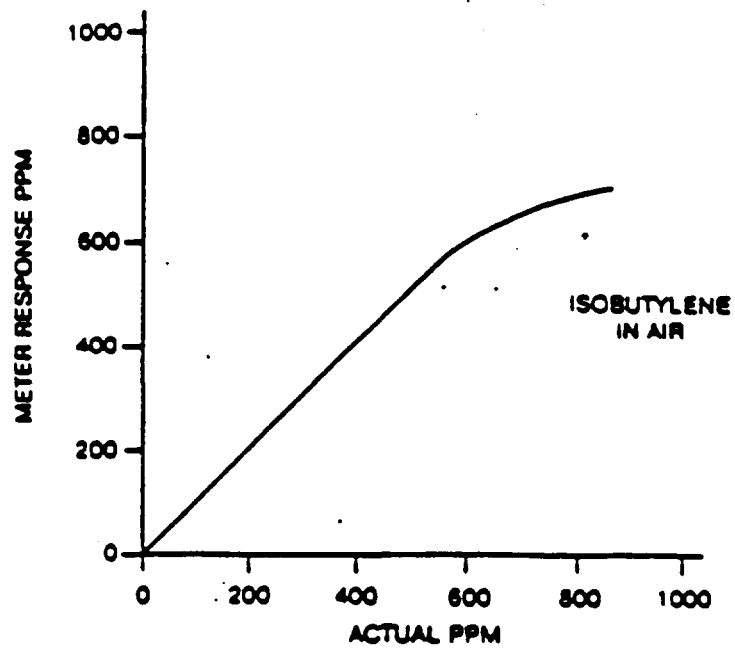


FIGURE 3-1



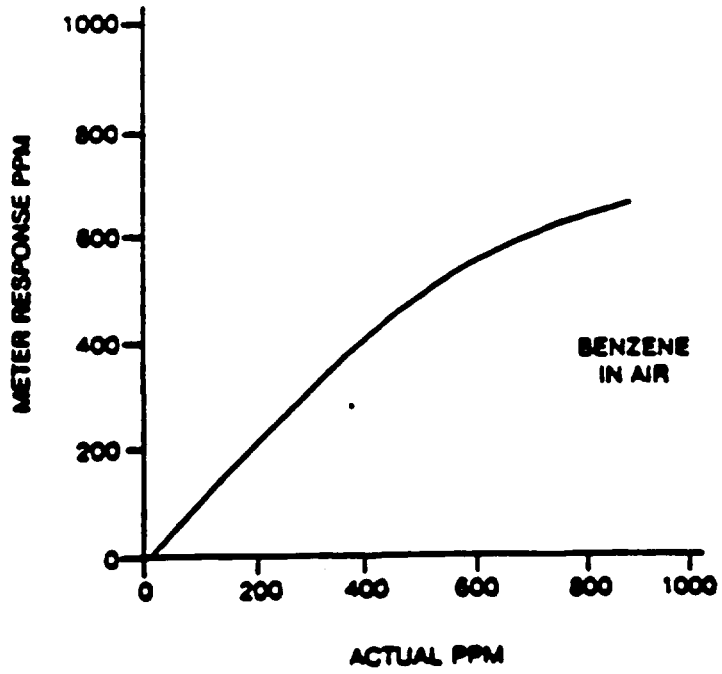


FIGURE 3-2

8.2 CALIBRATION CHECKING WITH ISOBUTYLENE

The calibration of the analyzer can be rapidly checked by the use of an HNU small disposable cylinder containing isobutylene (HNU pn 101-350) with a regulator (HNU pn 101-351).

At the factory, the analyzer is first calibrated on the desired gas standard at the specified concentration. Then a measurement is made with isobutylene.

The ppm reading along with the span setting using isobutylene is recorded in the calibration report.

In service, the analyzer calibration can be checked and readjusted if necessary by using this cylinder and regulator as follows:

a. Connect the analyzer to the regulator and cylinder with a short piece (butt connection) of tubing as shown in Figure 8-1. The calibration gas in the cylinder consists of a mixture of isobutylene and zero air. Isobutylene is nontoxic and safe to use in confined areas. There are no listed exposure levels at any concentration.
   The regulator sets and controls the flow rate of gas at a value preset at the factory. This will be about 250 cc/min.
   It is important that the tubing be clean since contaminated tubing will effect the calibration reading.
   Do not use the cylinder below about 30 psig as readings below that level can deviate up to 10% from the rated value.
   Safely discard the disposable cylinder when empty.
   Do not refill this cylinder.
   It is against the law to transport refilled cylinders.

b. With the SPAN setting and the function switch at the same positions as listed in the Application Data Sheet or Calibration Report, open the valve on the cylinder until a steady reading is obtained.

c. If the reading is the same as the recorded data, the analyzer calibration for the original species of interest is still correct.

d. If the reading has changed, adjust the SPAN setting until the reading is the same.

e. Shut off the cylinder as soon as the reading is established.

f. Record and maintain this new SPAN setting. Then recalibrate the analyzer on the species of interest as soon as possible.

g. Whenever the analyzer is recalibrated, it is to be immediately checked with the small cylinder and the reading recorded. This can then be used for later checking in the field.

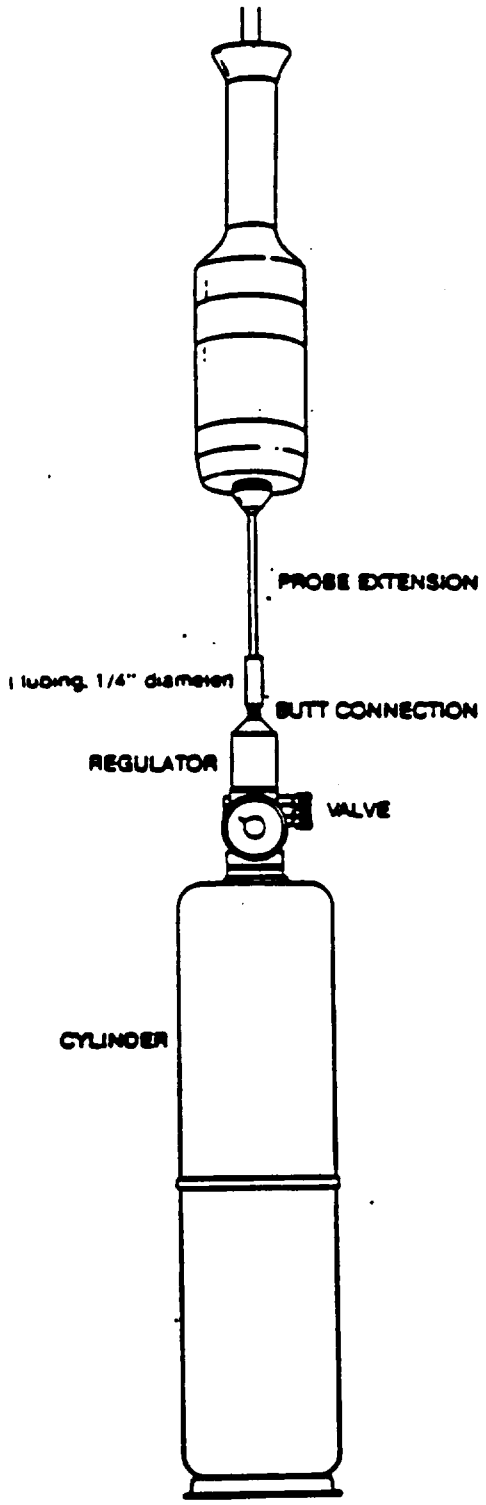


FIGURE 8-1
CALIBRATION CHECKING SET-UP

1.0 OBJECTIVE

The following procedure is a general guideline for the evaluation of existing wells. The guidelines may be modified depending on the project objectives. After completion of the evaluation, a judgment could be made as to the reliability of past data and future usefulness.

2.0 LIMITATIONS

These guidelines give overall technical guidance only and could be modified by specified requirement of project-specific plans for monitoring-well evaluation.

3.0 DEFINITIONS

None.

4.0 GUIDELINES

The following guidelines and procedure should be used when conducting monitoring-well evaluations:

1. Record the identification and general location of the monitoring well, including approximate distance from the site and access to the well.

2. Record the physical condition of the monitoring well including the following:

   a. Existence an condition of the protective steel casing, cap, and lock, including casing diameter.

   b. Existence and condition of cement collar surrounding the protective casing.

   c. Presence or absence of standing water or depressions around the casing.

   d. Presence of any electrical cable and its connections.

3. Before removing well cap, check for and disconnect any wires, cables, or

electrical sources. Remove lock and open cap. Air monitoring equipment should be used to detect the presence of organic vapors in he monitoring well. The following information should be recorded:

a. Cap function.

b. Physical characteristics of the inner casing or riser, including inner diameter and casing composition.

c. Presence of grout between the inner and outer protective casing.

d. Presence of an inner casing or riser cap including how it is attached to the casing and if it is vented.

e. Presence of a submersible pump or dedicated bailer. If possible, remove it and check the diameter, material, and condition of the equipment.

f. Stickup height from top of protective casing with cap open to ground surface and to top of inner casing or riser pipe if it exists.

4. Record initial static water level from top of PVC casing using an M-scope or equivalent.

5. Check dept of the well, if not obstructed, with a calibrated weighted line. Bounce the weight on the hole bottom to check for sediment. The weight will advance slowly if the well contains sediments. Note any sediment or bentonite on the end of the weight when removed. Note any obstructions or discrepancy with construction logs.

6. Check alignment and plumbness of the well. This is not as critical in shallow wells as in deep wells where a vertical line-shaft turbine pump or submersible pump will be installed. A plummert smaller than the inner well diameter should be lowered into the well. Any obstructions should be noted.

7. Check the function of the well to determine the time required for future sampling and estimate the hydraulic conductivity. The extent and method of testing depend on project objectives, well depth and diameter, and estimated hydraulin conductivity. Test methods would include rising head,

falling head, and slug tests. Records recovery using a pressure transducer with recorder or a water level indicator. Record pH, specific conductance, and temperature of groundwater removed if instrumentation is available. In clustered or nested wells record water level in shall wells when deeper wells are tested.

8. After water level equilibrium has been reached, check the integrity of th bentonite seal by pouring several gallons of water around the outside of the casing. Monitor water levels for 10 minutes and note any increase.

9. Close well, secure lock, and properly decontaminate all equipment when necessary.

1.0 OBJECTIVE

The function of a maintenance program is to ensure that any equipment needed for investigative work done by Malcolm Pirnie, Inc. (MPI) personnel is in proper working condition. Specifically, maintenance is needed to prevent the failure or malfunction of equipment and to eliminate erroneous readings resulting from nonmaintained equipment or from equipment in need of repair.

2.0 APPLICABILITY

This guideline is applicable to any monitoring, measuring, or test equipment that will be used for investigations conducted by MPI Personnel.

3.0 DEFINITIONS

General maintenance -- The minimum amount of maintenance needed to keep an instrument in proper working order.

Functional maintenance -- The maintenance needed for the repair or reconditioning of a piece of equipment.

4.0 GUIDELINES

A maintenance program is essential to ensure the continued operation of all instrumentation. The three elements of this program include normal upkeep of equipment, service and repair (when required) and formalized record keeping of all work done on each piece of equipment. This guideline covers the normal-upkeep-of-equipment element of the maintenance program.

For most of the equipment listed in Section 4.2 below, normal maintenance will consist of cleaning (outside surfaces) lubrication (all moving parts) and, if applicable, a battery level check and recharge or replacement as necessary. This program will include the maintenance of all monitoring, measuring, and test equipment returning from use, or any equipment used on a daily basis. The frequency of maintenance checks will be dependent on the individual needs and use of each piece of equipment. As a minimum, most equipment will be maintained according to a monthly schedule.

Maintenance procedures will be only those necessary for keeping an instrument in

service or in preparation for everyday use. It is beyond the scope of this guideline to cover specific maintenance or repair procedures for each piece of equipment. For this purpose, the manufacturer and model number are given for each piece of equipment so that a repair or specific maintenance problem can be referred to the manufacturer or other qualified servicing organization.

4.1 RESPONSIBILITIES

The project team leader's responsibilities include keeping all maintenance records, making sure all equipment used is maintained on a daily basis, and shipping any instrument in need of repair to the correct source.

The equipment custodian's responsibilities include carrying out all maintenance required according to schedule, sending out for service any piece of equipment in need of repair, and informing field team members of any specific maintenance requirements for equipment used at the site. The equipment custodian will also keep on file all records of maintenance under his or her care.

The field personnel responsibilities include the maintenance of every piece of equipment located at the site, on a regular basis, and the maintenance of equipment after each use.

4.2 EQUIPMENT

A list of the equipment that is subject to the maintenance guidelines set forth in Section 4.2- is given below.

1. Altimeter, American Pualin System Model APS-MI
2. Automatic level, Lietz Model B2A
3. Colorimetric tubes, National Draeger Model 67-26065
4. Explosimeter, Mine Safety Appliances Model 2A
5. Flow recorder, Linear Instruments Corporation Models 141 and 142
6. Magnetometer, Omnimag Model PPM-300
7. Oxygen met-r, Mine Safety Appliances Model 245 PA

8. Permissible air-sampling pump, Bendix Models BDx30 and BDX3l

9. pH meters

   a. Coring Scientific Instruments Model 3

   b. Orion Research Models 201 and 301

10. Radiation detectors

   a. Eberline Models E-120 and E-l20E

   b. Solar Electronics Model PA-Mini

   c. Victoreen Model 490, Thyac III

11. Rain gauge, Belfort Instrument Company Model 5-780 series

12. Resistivity meter, Bison Instruments Model 2350

13. Theodolite, Tokyo Optical Company, Ltd., Models TL-6DE, TL-10DE, TL-20DE, and TL-20DEP

14. Vapor detectors

   a. HNU Systems Model Pl-101

   b. Photovac Incorporated Model 10A10

   c. Foxboro Models OVA 128 and 108

15. Water level indicators

   a. SINCO Model 51453

   b. Soiltest Inc. Models DR760A, DR762M, and DR762A

4.3 MONITORING, MEASURING, AND TEST EQUIPMENT MAINTENANCE

All monitoring, measuring, and test equipment should be maintained on a monthly basis (unless otherwise stated) Periodic maintenance should include a general

cleaning with a mild detergent, lubrication of all movable parts, a battery check if applicable, and a generalized check for any equipment that is need of repair. Instrument calibration (Section 12.1 and 12.3) or decontamination (Section 11.11) are not treated in this procedure.

In addition to the monthly scheduled maintenance, equipment that has been used between inspections must undergo a maintenance check after each use, or, if the equipment is used over a prolonged period, a check should be made aster each day of use.

Maintenance requirements are summarized below for the equipment listed in the preceding Section 4.2.

4.3.1 Altimeter

The following is a summary of the maintenance requirements for the Model APS-MI altimeter manufactured by American Paulin System:

1. General maintenance. Protect the altimeter a:id thermometer from the direct rays of the sun.

2. Monthly maintenance. None required.

3. After-use maintenance. Clean the instrument with a mild detergent.

4. Functional maintenance. Refer all specific maintenance and repair requirements to the manufacturer.

4.3.2 Automatic Level

The following is a summary of the maintenance requirements for the Model B2A automatic level manufactured by Lietz:

1. General maintenance. Clean and check the instrument.

2. Semiannual maintenance

   a. Fully check and inspect the instrument.

   b. Periodically check the tripod shoes; they may become loose or the legs may become shaky after being used for a long period.

3.. After-use maintenance

a. Completely wipe off any moisture if the instrument gets wet during survey work. Moisture will adversely affect the instrument.

b. Clean every part of the instrument before putting it back in the case. Breathe on the lenses to moisten them and gently clean them with a clean cloth (worn out cotton) or soft tissue paper.

4. Functional maintenance. If foreign matter appears to have entered any movable parts or screws, or when condensation or fungi appear on the lenses, prisms, etc., of the telescope, promptly consult the manufacturer.

4.3.3 Colorimetric Tubes

The following is a summary of the maintenance requirements for the Model 67-26065 Draeger Multi Gas Detector manufactured by National Draeger:

1. General maintenance. None required.

2. Monthly maintenance

a. Leak test – Insert the unopened tube into the bellows pump. Compress the pump. Following this, the position of the bellows must not change any more for I minute.

b. Suction capacity test – Compress the pump; when the bellows is released, it must open instantly.

3. After-use maintenance. Perform the leak test and suction capacity test.

4. Functional maintenance. Refer any repair or specific maintenance needs to the manufacturer or another qualified service center.

4.3.4 Explosimeter

The following is a summary of the maintenance requirements for the Model 2A Combustible Gas Indicator manufactured by Mine Safety Appliances:

1. General maintenance. None required.

2. Monthly maintenance

   a. Check battery level; replace batteries if necessary.

   b. Check for leaks in the system.

   c. Check flashback arresters.

   d. Check ballast lamp.

   e. Check detector unit.

3. After-use maintenance

   a. Check battery level.

   b. Check filter.

   c. Check detector unit.

4. Functional maintenance

   a. When gaseous concentrations fall above the scale range of the instrument, stop sampling and flush the instrument with fresh air.

   b. Replace battery in accordance with instruction manual (Model 2A).

   c. Change filter in accordance with instruction manual.

   d. Refer all repairs to the manufacturer or other qualified service personnel.

4.3.5 Flow Recorder

The following is a summary of the maintenance requirements for the Models 141 and 142 flow recorders manufactured by Linear Instruments Corporation:

1. General maintenance. Keep on supply chart paper and chart pens

(keep pen tips covered)

2. Monthly maintenance. Check battery level; recharge according to manufacturer's specifications if necessary.

3. After-use maintenance

   a. Check battery and recharge if necessary.

   b. Check the amount of chart paper used; add more if necessary.

4. Functional maintenance

   a. User lubrication is not recommended.

   b. Service or repair should be referred to Linear Instrument Corporation factory technicians or other qualified repair personnel.

4.3.6 Magnetometer

The following is a summary of the maintenance requirements for the Model PPM-300 Omnimag, Total Field Magnetometer, manufactured by EDA Instruments, Inc.

1. General maintenance. Keep the magnetometer clean and free of dust.

2. Monthly maintenance. Check the battery level and, if necessary, change or recharge the batteries.

   a. Rechargeable: nonmagnetic sealed lead-acid batteries

   b. Disposable: C-size cells

3. After-use maintenance

   a. Clean the key pad to prevent buildup of dirt in the key recesses. If necessary, use a small soft brush to remove the dirt.

   b. Clean around the mode selector. Use a small, soft brush to gain access.

c. Clean the display window.

d. Clean the sensor. This is important because foreign matter could contain traces of magnetite which could influence instrument accuracy.

e. Check the battery level; recharge or replace as necessary.

4. Functional maintenance. Refer all specific maintenance or repair needs to the manufacturer or other qualified service personnel.

4.3.7 Oxygen Meter

The following is a summary of the maintenance requirements for the Model 245 RA oxygen meter manufactured by Mine Safety Appliances Company:

1. General maintenance. None required.

2. Monthly maintenance. Check battery level by turning instrument on. If low battery alarm sounds, replace battery (one 9-V alkaline battery)

3. After-use maintenance

a. Check Teflon sensor for damage (easily damaged)

b. Clean the instrument with a cloth and a mild detergent in water.

4. Functional maintenance. For sensor replacement, refer to the manufacturer's manual. All other repairs should be referred to the manufacturer.

4.3.8 Permissible Air-Sampling Pump

Maintenance of the sample pump involves checking for leaks, blockage, and pump capacity. The following is a summary of the maintenance requirements for the Models BDX30 and BDX31 Super Samplers manufactured by Bendix Corporation:

1. Monthly maintenance

a. Check the sampling head for leaks (refer to manufacturer's manual as noted above). If a leak is found, inspect cassette and

hose for cracks and leaks. Repair or replace components as necessary.

b. Inspect the dust collector leaks. If a leak is found, inspect filter holder and hose for cracks and leaks.

2. After-use maintenance

a. Inspect and clean all parts daily. Cleaning should be done with water or alcohol and a pipe cleaner.

b. Check the unassembled parts for cracks or leaks and replace all O-rings at the first sign of wear.

c. Recharge battery after each use.

3. Functional maintenance. Specific maintenance procedures for the Super Sampler pump, gravimetric sampling head assembly, and respirable dust collector are outlined in the manufacturer's manual (*reference*, BDX3O, 3i, 99).

4.3.9 pH Meters

pH Meter Manufactured by Corning Scientific Instruments

The following is a summary of the maintenance requirements for the Model 3 pH meter:

1. General maintenance. None required for meter; store electrodes under conditions described in the instruction manual. The electrode instruction sheet gives maintenance procedures for individual electrodes.

2. Monthly maintenance. Check battery level. Replace if necessary (two 9-V batteries)

3. After-use maintenance. Check battery level after each use.

4. Functional maintenance. Various electrodes can be used with this pH meter, and each electrode will have its own maintenance procedure. Reference to electrode maintenance will be given on the electrode

instruction sheet.

pH Meter Manufactured by Orion Research

The following is a summary of the maintenance requirements for the Models 201 and 301 digital pH meters:

1. General maintenance. Store electrodes according to procedures given on the electrode instruction sheet.

2. Monthly maintenance. Check the battery level, and replace batteries as needed (six AA batteries).

3. After-use maintenance. Check batteries after each use.

4. Functional maintenance. Refer specific maintenance or repair needs to the manufacturer or other qualified service personnel

4.3.10 Radiation Detectors

Radiation Detector Manufactured by Eberline Geiger Counter

The following is a summary of the maintenance requirements for the Models E-120 and E-l20E radiation detectors:

1. General maintenance

   a. Keep instrument clean and dry.

   b. Remove batteries if the instrument is to be inactive for a long period.

2. Monthly maintenance. Check the battery level. Replace batteries when their check reading is below the acceptable level.

3. After-use maintenance. Check battery levels daily.

4. Functional maintenance. Refer all specific maintenance or repair needs to the manufacturer or other qualified service personnel.

Radiation Detector Manufactured by Solar Electronics

The following is a summary of the maintenance requirements for the Radiation Alert Model RA-Mini:

1. General maintenance. None required

   Monthly maintenance. Check the battery level and replace if necessary (9-V alkaline cell)

3. After-use maintenance. Check the battery level and replace if necessary.

4. Functional maintenance. Refer all specific maintenance or repair needs to the manufacturer or other qualified personnel.

Radiation Detector Manufactured by Victoreen Geiger Counter

The following is a summary of the radiation requirements for the Model 490, Thyac III:

1. General maintenance. Keep instrument clean and dust free. Instrument should be turned off when not in use.

2. Monthly maintenance. Check the battery level. Replace batteries as needed (two D cells).

3. After-use maintenance. Check the battery level. Replace batteries as needed. Conduct a performance test in accordance with ANSI N323-1978, paragraphs 4.6 and 5.4-

4. Functional maintenance. Specific maintenance procedures for Geiger tube replacement, high-voltage power supply check, an amplifier, circuit, and monostable multivibrator test are outlined in the manufacturer's manual. Refer all other service to the manufacturer or other qualified service personnel.

4.3.11 Rain Gauge

The following is a summary of the maintenance requirements for the Model 5-780 series (re. book nos. 8777, 12049, 15783) of rain gauges manufactured by Belfort Instrument Company:

1. General maintenance. Inspect the gauge during regular operational visits to ensure correct operation.

2. Monthly maintenance. None required.

3. During-use maintenance. Every 3 months (dusty climates) or once per year (mild climates) clean all Moving parts with a soft brush. Examine the weighing mechanism linkage for evidence of excessive friction. Clean the bucket thoroughly. Inspect the dash pot; add silicone fluid if necessary. Inspect the chart recorder and drive. Clean the pen.

4. Functional maintenance. See additional maintenance procedures provided in the manufacturer 's manual (as referenced above)

4.3.12 Resistivity Meter

The following is a summary of the maintenance requirements for the Model 2350 Earth Resistivity Meter manufactured by Bison Instruments:

1. General maintenance. Keep instrument free of dust, dirt, and moisture.

2. Monthly maintenance. Check battery level (selector switch on test positions) and replace batteries as necessary (three 9-V batteries, Mallory No. TR234R, and three 5.4-v batteries, Everady No. 490).

3. After-use maintenance. Check battery level; replace batteries as necessary.

4. Functional maintenance. For battery replacement refer to instruction manual. Do not attempt any repairs or internal maintenance beyond battery replacement

4.3.13 Theodolite

The following is a summary of the maintenance requirements for theodolites, Models TL-6DE, TL-l0DE, TL-20DE, and TL-20DEP, manufactured by Tokyo OPtical Company, Ltd.:

1. General maintenance. Store instrument in a well-ventilated area. Protect the instrument from shock and vibrations when transporting.

2. Monthly maintenance

   a. Clean the instruMent with a damp cloth.

   b. Check the instrument according to procedure outlined in the manual

3. After-use maintenance. Clean the instrument after each use.

4. Functional maintenance

   a. Do not dismantle the telescope or revolving parts of the instrument.

   b. Refer repair or internal maintenance to the manufacturer or to a qualified service center.

4.3.14 Vapor Detectors

Vapor Detector Manufactured by HNU Systems

The following is a summary of the maintenance requirements for the Model PI-I 01 HNU Photoionization Analyzer:

1. General maintenance. None required

2. Monthly Maintenance

   a. Check battery and recharge if necessary.

   b. Check sample inlet of the probe unit for violet glow (change ultraviolet light source if needed)

3. After-use maintenance. Recharge battery after each use.

4. Functional maintenance. Refer all specific maintenance or repair needs to the manufacturer or other qualified service personnel.

Vapor Detector Manufactured by PhotoVac Incorporated

The following is a summary of the maintenance requirements for the Model 10A10 Portable Photoionizaton Gas Chromatograph:

1. General maintenance. Maintain batteries in a fully charged condition.

2. Monthly maintenance. Check battery level. Recharge batteries when necessary.

3. After-use maintenance. Recondition column by heating overnight at a temperature of 100 C and in an ultra-high-purity helium atmosphere.

4. Functional maintenance

   a. For column replacement, refer to the manufacturer's instruction manual as referenced above.

   b. Because user service is not recommended, forward any repairs to the manufacturer or other qualified service personnel.

Vapor Detector Manufactured by Foxboro

The following is a summary of the maintenance requirements for the Organic Vapor Analyzer, Models OVA 128 and 108:

1. General maintenance. Keep instrument free of dust and moisture.

2. Monthly maintenance

   a. Check the battery charge level. Recharge batteries as needed.

   b. Clean and inspect particle filters, valve rings, and burner chamber.

   c. Check pumping system for leaks.

3. After-use maintenance

   a. Recharge battery after each use.

   b. Recharge hydrogen gas supply as needed.

c. Check chromatograph column; change or regenerate (with a desorber) the column if needed.

4. Functional maintenance. Refer all specific maintenance or repair needs to the manufacturer or to other qualified service personnel.

4.3.15 Water Level Indicators

Water Level Indicator Manufactured by SINCO

The following is a summary of the maintenance requirements for the Model 51453:

1. General maintenance. None required.

2. Monthly maintenance. Check battery level and replace batteries if necessary (three AA batteries)

3. After-use maintenance. Clean the instrument after each use.

4. Functional maintenance. Refer repair or specific Maintenance to the manufacturer or to other qualified service personnel.

Water Level Indicator Manufactured by Soiltest, Inc.

The following is a summary of the maintenance requirements for the Models DR760A, DR762M, and DR762A:

1. General maintenance. None required.

2. Monthly maintenance. Check battery level and replace batteries as needed (one 1-1/2-V penlight cell)

3. After-use maintenance

   a. Clean the instrument after each use.

   b. Check battery level after each use.

4. Functional maintenance. Refer repair or specific maintenance to the manufacturer or to other qualified service personnel.

4.4 RECORDS

A record of all equipment maintenance and repair should be kept in the following places:

1. Field logbook
2. Equipment custodian's logbook
3. Remedial Planning Office (REMPO) files

A record of maintenance form, shown on Figure 4.25-1, will be kept on file at REMPO with the instruction manuals for all instrumentation.

MALCOLM PIRNIE, INC.

RECORD OF MAINTENANCE

Type_____ Date_____

Manufacturer _____

Model No. _____

Serial No. _____

Type of Maintenance _____

Comments _____

_____

_____

_____

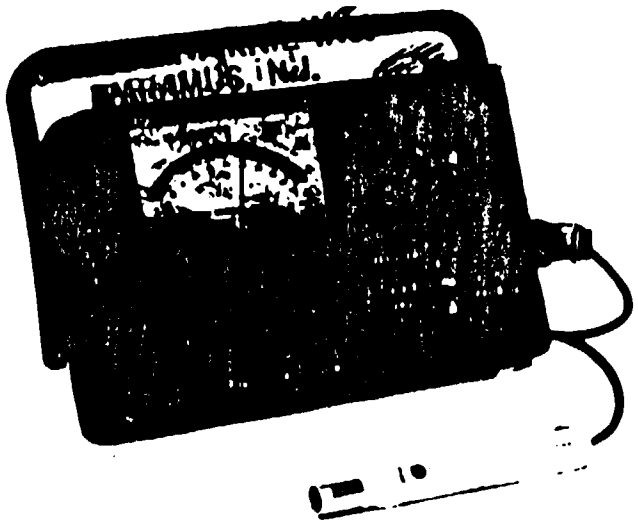
_____

Maintenance Performed by_____

Copy to_____

Quality Assurance Officer_____

INSTRUCTION MANUAL
YSI MODEL 51B
DISSOLVED OXYGEN METER



YSI Scientific

PRICE INCLUDING HANDLING $6.00

SUMMARY OF OPERATING INSTRUCTIONS

1. CALIBRATION

A. Switch instrument to OFF and adjust meter mechanical zero
B. Switch to ZERO and adjust to "0" on mg/l scale
C. Switch to FULL SCALE and adjust to "15" on mg/l scale.
D. Prepare probe for operation, plug into instrument, wait up to 15 minutes for probe to stabilize. Probe can be located in calibration chamber or ambient air.
E. Switch to CALIB. O_2 and adjust CALIB. control until meter indicates local altitude on short scale in upper right corner of meter.
NOTE: It is desirable to calibrate probe in a high humidity environment. See instruction manual for more detail on calibration and other instrument and probe characteristics.

2. MEASUREMENT

A. Place probe in sample and stir.
B. Allow sufficient time for probe to stabilize to sample temperature and dissolved oxygen level.
C. Switch to TEMP AND SET DIAL. Set O_2 SOLUBILITY FACTOR dial to sample temperature. Take care to properly index salinity on sloped white bar.
D. Switch to READ and read mg/l, dissolved oxygen while stirring.
E. We recommend instrument be left on between measurements to avoid the necessity to re-polarize the probe.

3. GENERAL CARE

A. Replace batteries after 1000 hours of operating or if full scale adjustment cannot be made. Use Eveready 935 "C" size or equal.
B. Membranes will last indefinitely depending on use. Average replacement is 2-4 weeks. Probe should be stored in humid environment to prevent drying out.
C. Calibrate daily.

TABLE OF CONTENTS

GENERAL DESCRIPTION

The YSI Model 518 Dissolved Oxygen Meter is intended for dissolved oxygen and temperature measurement in water and wastewater applications, but are also suitable for use in certain other liquids. Dissolved Oxygen is indicated in mg/l (milligrams per liter) on a 0-15 mg/l scale. Temperature is indicated in °C on a -5° to +45°C scale. The dissolved oxygen range is automatically temperature compensated for permeability of the probe membrane, and manual by direct dial for changes in water temperature.

The probes use Clark-type membrane covered polarographic sensors with builtin thermistors for temperature measurement and compensation. A thin, permeable membrane stretched over the sensor isolates the sensor elements from the environment, but allows oxygen and certain other gases to enter. When a polarizing voltage is applied across the sensor, oxygen that has passed through the membrane reacts at the cathode, causing a current to flow.

The membrane passes oxygen at a rate proportional to the 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 force causing the oxygen to diffuse 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.

SPECIFICATIONS

I. Instrument

Oxygen Measurement

Range: 0-15 mg/l

Accuracy: Better than ± 0.2 mg/l when calibrated with ±5°C of actual sample temperature.

Readability: Better than 0.1 mg/l

Temperature Measurement

Range: -5° to +45°C

Accuracy: ±0.7°C, including probe

Readability: 0.25°C

Compensation

Temperature compensation for oxygen probe membrane coefficient is automatic.

Temperature compensation for oxygen solubility is manual by direct dial from 0°C to 45°C for fresh water and -5°C to +37°C for sea water.

Altitude compensation is manual by direct dial from 0 to 11,000 feet.

Salinity compensation is manual by direct dial from fresh water to sea water of 20,000 mg/l chloride concentration.

System Response Time

Typical response for temperature and DO readings is 90% in 10 seconds at constant temperature of 30°C.

DO response at low temperature and low DO is typically 90% in 30 seconds.

If response time under any operating conditions exceeds two minutes, probe service is indicated.

Instrument Ambient Range

Satisfactory operation from -5°C to +45°C.

Power Supply

YSI Model 518 is powered by four disposable "C" size carbon zinc batteries, providing approximately 1000 hour operation. Replace with Eveready 935C or equal.

II. Probe

Cathode: Gold

Anode: Silver

Membrane: .001" FEP Teflon

Electrolyte: Half saturated KCl

Temperature Compensation: (See SPECIFICATIONS, I. Instrument)

Pressure Compensation: Effective 1/2% of reading of pressures of 100 psi (230 ft. water)

Polarizing Voltage: .75 volts nominal

Probe Current: Air at 30°C = 19 microamps nominal
Nitrogen at 30°C = .15 microamps or less

III. Accessories and Replacement Parts

YSI 5720A — Self Stirring B.O.D. Bottle Probe

YSI 5750 — Non Stirring B.O.D. Bottle Probe

YSI 5739 — Oxygen Temperature Probe for field use. Combine with one of the following cables for desired lead length.

Detachable leads for use with YSI 5739.

YSI 5740- 10	10' cable
YSI 5740- 25	25' cable
YSI 5740- 50	50' cable
YSI 5740-100	100' cable
YSI 5740-150	150' cable
YSI 5740-200	200' cable

YSI 5492A — Battery Pack operates YSI 5791A Submersible Stirrer.

YSI 5791A — Submersible Stirrer for field use with YSI 5739 and 5740 probe cable assembly.

YSI 5795A — Submersible Stirrer for field use with YSI 5739 probe (Convenient single cable design does not require YSI 5740 probe cable.)

YSI 5075A — Calibration Chamber for use with field probe.

YSI 5890 — Carrying Case

YSI 5775 — Membrane and KCl Kit Standard — includes 2 each 15 membranes packets (.001" thick standard membranes) and a 30 ml bottle KCl with Kodak Photo Flo.

YSI 5945 — "O" Ring Pack — includes (6) "O" rings for each YSI D.O. Probe

YSI 5486 — Beater Boot Kit — includes (1) A-05486 Boot — (1) A-05484 Tip, (2) A-05485 Springs. Use only on YSI 5720A and discontinued YSI 5420A and 5720.

YSI 5986 — Diaphragm Kit for use only with YSI 5739 D.O. Probe

YSI 5735 — Adaptor makes it possible to use YSI 5739, YSI 5720A and YSI 5750 Probes with discontinued YSI Model 51A.

YSI 5680 — Probe Reconditioning Kit

OXYGEN PROBES AND EQUIPMENT

There are three oxygen probes for use with the YSI Model 51B Dissolved Oxygen Meter. Descriptions of where they are used are contained in the following paragraphs.

I. YSI 5739 D.O. Probe

The YSI 5739 Probe, with built-in lead weight and pressure compensation, is an improved design that replaces the discontinued YSI 5101, 5418, 5419, 5718 and 5719 Probes. (See Figure 1)

For user convenience the probe is equipped with a disconnecting cable to facilitate changing cable lengths and replacing damaged cables or probes. The probe and cable assembly is held together with a threaded retaining nut. The connection is *not* designed for casual disconnection and should only be disconnected when necessary.

To disconnect the cable unscrew the retaining nut and slide it down the cable to expose the connector. Pull gently on the cable and connector until the connector comes away from the probe body.

To reassemble, inspect the connector and "O" ring for cleanliness. If the "O" ring is frayed or damaged remove it by squeezing it in the groove causing it to bulge, then roll it out of the groove and off the connector. A replacement "O" ring is supplied with the cable.

Push the connector into the probe body, rotating it until the two halves mate. A light coating of vaseline or silicone grease on the "O" ring will make reassembly easier. Air trapped between the connector halves which may cause them to spring apart slightly, is normal. Screw on the retaining nut, *hand tight only*. NOTE: If erratic readings are experienced, disconnect the cable and inspect for water. If present, dry out and reconnect, replacing the "O" ring, if necessary.

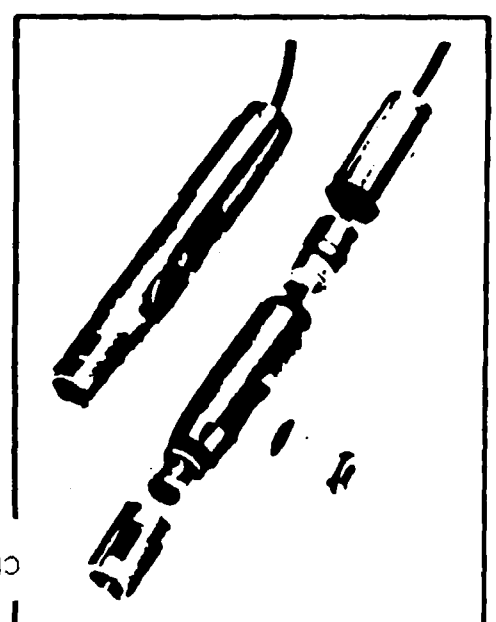


FIGURE 1

Pressure Compensation

The vent on the side of the probe is part of a unique pressure compensating system that helps assure accurate readings at great depths of water. Pressure compensation is effective to 1/2% of reading with pressures to 100 psi (230 ft water). The quantity of air bubbles trapped under the membrane determines how serious the pressure error will be, which is why proper preparation of the probe is essential. (See OPERATING PROCEDURES.) The system is designed to accommodate a small amount of trapped air and still function properly, but the amount should be kept to a minimum.

The compensating system normally does not require servicing and should not be taken apart. However, if electrolyte is leaking through the diaphragm or if there is an obvious puncture, the diaphragm must be replaced. A spare is supplied with the probe. Using a coin unscrew the retaining plug and remove the washer and the diaphragm. Flush any salt crystals from the reservoir, install the new diaphragm (convolution side in), replace the washer, and screw in the retaining plug.

II. YSI 5720A B.O.D. Bottle Probe

The YSI 5720A B.O.D. Bottle Probe replaces the discontinued YSI 5420A B.O.D. Bottle Probe for measuring dissolved oxygen and temperature in standard B.O.D. bottles. It is provided with an agitator for stirring the sample solution, available in models for 115VAC (95-135VAC, 50-60 Hz) or 230VAC (190-250VAC, 50-60 Hz) operation. (See Figure 2)

When using the probe, plug the agitator power supply into line power and the probe plug into the instrument. With the agitator turned off place the tapered probe end into the B.O.D. bottle and switch agitator "ON" with switch on top of probe. The probe should be operated with a minimum of trapped air in the B.O.D. bottle. A slight amount of air in the unstirred region at the top of the bottle may be neglected, but no bubbles should be around the thermistor or oxygen sensor.

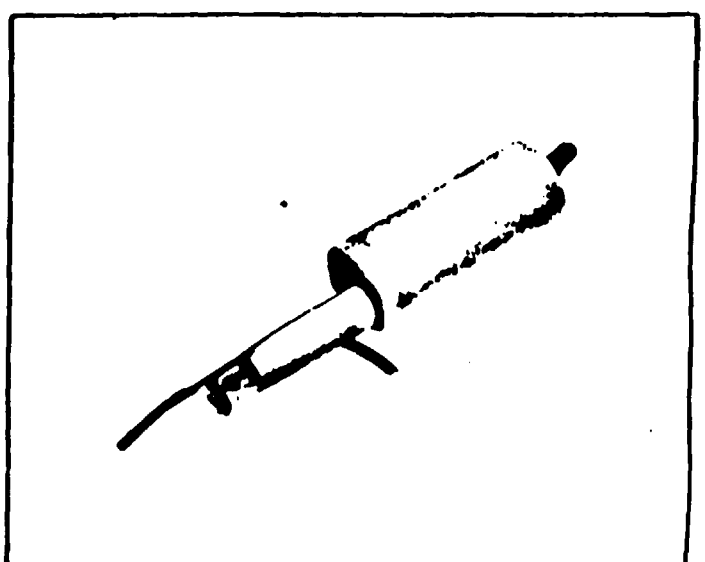


FIGURE 2

Stirrer Boot

The probe uses a flexible stirring boot to transmit motion from the sealed motor housing to the sample. If the boot shows signs of cracking or other damage likely to allow leaking into the motor housing, the boot must be replaced.

In fresh water applications boot life is normally several years, but this may be shortened by exposure to hydrocarbons, moderate to strong acids or bases, ozone, or direct sunlight. For maximum life rinse the boot after use in contaminated samples. (See Figure 3)

Boot replacement is as follows:

1. Pull off old assembly and clean shaft.
2. Slide on new assembly making sure the back spring is on the grooved area of the shaft. A small amount of rubber cement may be used.
3. Check that there is sufficient clearance between the tip and the end of the shaft to permit turning without binding.

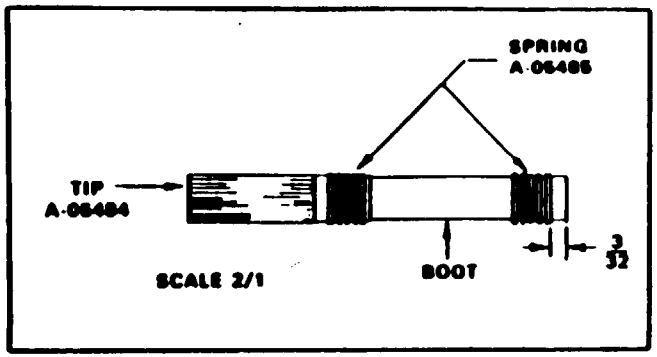


FIGURE 3

III. YSI 5750 B.O.D. Bottle Probe

The YSI 5750 B.O.D. Bottle Probe replaces the discontinued YSI 5450 B.O.D. Bottle Probe. It is similar to the YSI 5720A B.O.D. Bottle Probe, except that it does not have a stirrer. Agitation of the sample must be provided by other means, such as a magnetic stirrer. (See Figure 4)

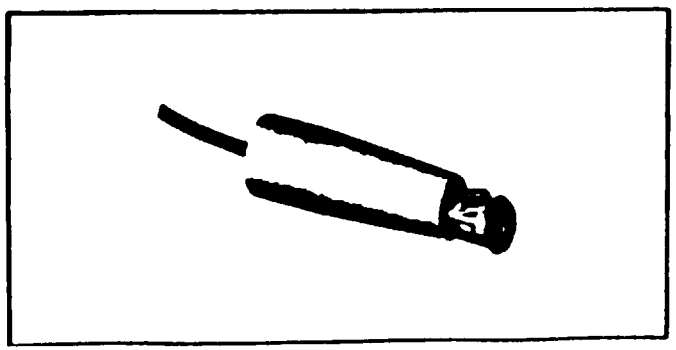


FIGURE 4

IV. Cable Adaptors

All YSI 5700 Series Probes are designed for direct use with the YSI Model 51B Dissolved Oxygen Meter. However, to use YSI 5700 probes with the discontinued YSI Model 51A, cable adaptor YSI 5735 is required.

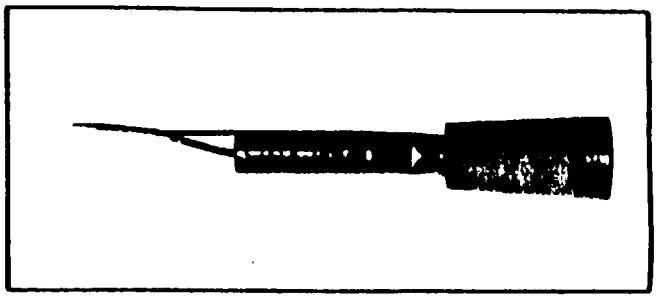


FIGURE 5

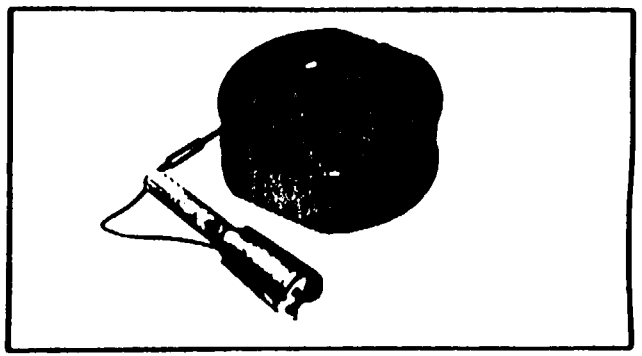


FIGURE 6

V. YSI 5791A and 5795A Submersible Stirrers

The YSI submersible stirrers are accessories that perform the function of stirring the sample being studied when making dissolved oxygen measurements in the field. The YSI 5791A stirrer can be used with the following dissolved oxygen probes: YSI 5101, 5418, 5419, 5718, 5719, and 5739. The YSI 5795A stirrer is only for use with the YSI 5739 Probe. (See Figure 6)

When a stirrer and probe are assembled, the stirrer agitates the sample directly in front of the sensor by means of a rotating eccentric weight which causes the spring-mounted hermetically sealed motor housing to vibrate. An impeller on the end of the motor housing flushes the media across the oxygen sensor. (See sales literature and instruction sheets for further information)

VI. YSI 5492A Battery Pack

The YSI 5492A Battery Back is designed to attach to the case of the YSI Model 51B Dissolved Oxygen Meter to provide power for operating the submersible stirrers. (See sales literature and instruction sheets for further information)

OPERATING PROCEDURES

I. Preparing the Probe

All YSI 5700 Series Probes have similar sensors and should be cared for in the same manner. They are precision devices relying on good treatment if high accuracy measurements are to be made. Prepare the probes as follows (See Figure 7)

ALL PROBES ARE SHIPPED DRY — YOU MUST FOLLOW THESE INSTRUCTIONS

1. Prepare the electrolyte by dissolving the KCl crystals in the dropper bottle with distilled water. Fill the bottle to the top.
2. Unscrew the sensor guard from the probe (YSI 5739 only) and then remove the "O" ring and membrane. Thoroughly rinse the sensor with KCl solution.
3. Fill the probe with electrolyte as follows:
   - A. Grasp the probe in your left hand. When preparing the YSI 5739 probe the pressure compensating vent should be to the right. Successively fill the sensor body with electrolyte while pumping the diaphragm with the eraser end of a pencil or similar soft, blunt tool. Continue filling and pumping until no more air bubbles appear. (With practice you can hold the probe and pump with one hand while filling with the other.) When preparing the YSI 5720A and 5750 probes, simply fill the sensor body until no more air bubbles appear.
   - B. Secure a membrane under your left thumb. Add more electrolyte to the probe until a large meniscus completely covers the gold cathode. NOTE: Handle membrane material with care, keeping it clean and dust free, touching it only at the ends.
   - C. With the thumb and forefinger of your other hand, grasp the free end of the membrane.
   - D. Using a continuous motion *stretch* the membrane *UP, OVER,* and *DOWN* the other side of the sensor. Stretching forms the membrane to the contour of the probe.
   - E. Secure the end of the membrane under the forefinger of the hand holding the probe.
   - F. Roll the "O" ring over the end of the probe. There should be no wrinkles in the membrane or trapped air bubbles. Some wrinkles may be removed by lightly tugging on the edges of the membrane beyond the "O" ring.
   - G. Trim off excess membrane with scissors or sharp knife. Check that the stainless steel temperature sensor is not covered by excess membrane.
4. Shake off excess KCl and reinstall the sensor guard.
5. A bottomless plastic bottle is provided with the YSI 5739 probe for convenient storage. Place a small piece of moist towel or sponge in the bottle and insert the probe into the open end. This keeps the electrolyte from drying out. The YSI 5720A and 5750 probes can be stored in a B.O.D. bottle containing about 1" of water.
6. Membranes will last indefinitely, depending on usage. Average replacement is 2-4 weeks. However, should the electrolyte be allowed to evaporate and an excessive amount of bubbles form under the membrane, or the membrane become damaged, thoroughly flush the reservoir with KCl and install a new membrane.
7. Also replace the membrane if erratic readings are observed or calibration is not stable.
8. "Home brew" electrolyte can be prepared by making a saturated solution of reagent grade KCl and distilled water, and then diluting the solution to half strength with distilled water. Adding two drops of Kodak Photo Flo per 100 ml of solution assures good wetting of the sensor, but is not absolutely essential.
9. The gold cathode should always be bright and untarnished. If it is tarnished (which can result from contact with certain gases) or plated with silver (which can result from extended use with a loose or wrinkled membrane), return it to the factory for service or else clean it with the YSI 5680 Probe Reconditioning Kit. Never use chemicals or any abrasive other than that supplied with this kit.
10. It is also possible that the silver anode may become contaminated, which will prevent successful calibration. Try soaking the probe overnight in a 3% ammonia solution; rinse with deionized water, recharge with electrolyte, and install a new membrane. If still unable to calibrate, return the probe for service.
11. Hydrogen sulfide, sulphur dioxide, halogens, neon, and nitrous and nitric oxide are interfering gases. If you suspect erroneous readings, it may be necessary to determine if these are the cause. These gases have been tested for response:

100% Helium	None	100% Carbon Dioxide	About 1%
100% Ethylene	None	100% Nitrous Oxide	1/3 O_2 response
100% Carbon Monoxide	Less than 1%	100% Nitric Oxide	1/3 O_2 response
100% Hydrogen	Less than 1%	100% Chlorine	2/3 O_2 response

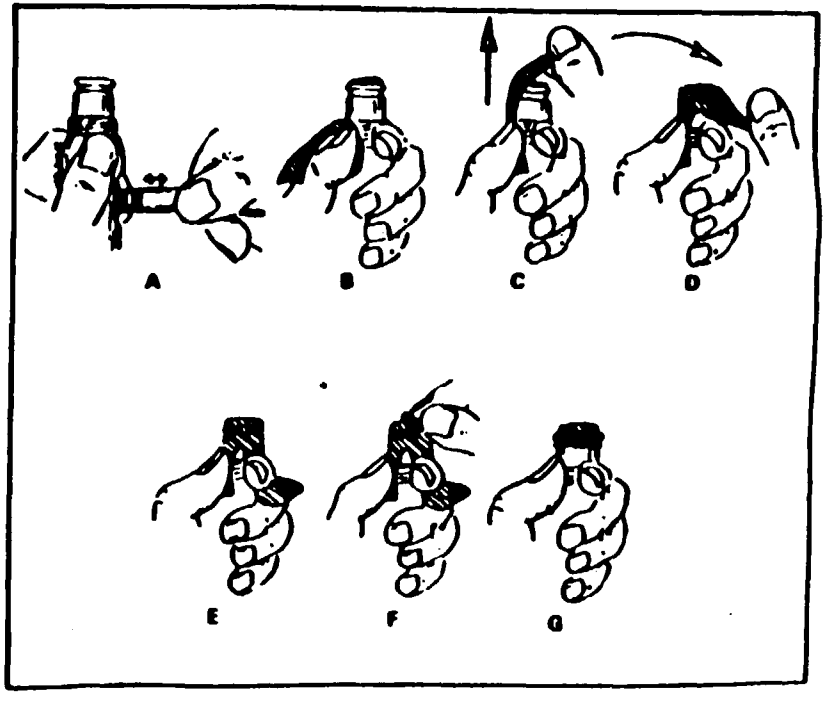


FIGURE 7

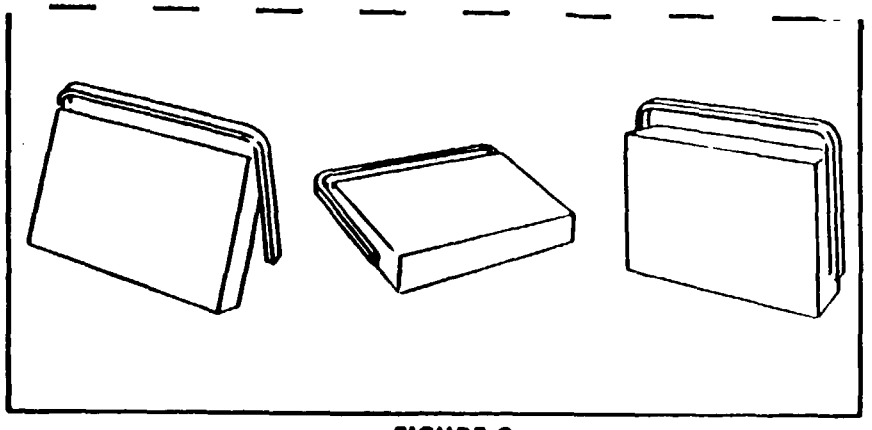


FIGURE 8

II. Preparing the Instrument

It is important that the instrument be placed in the intended operating position vertical, tilted, or on its back — before it is prepared for use and calibrated (See Figure 8). Readjustment may be necessary when the instrument operating position is changed. After preparing the probe proceed as follows

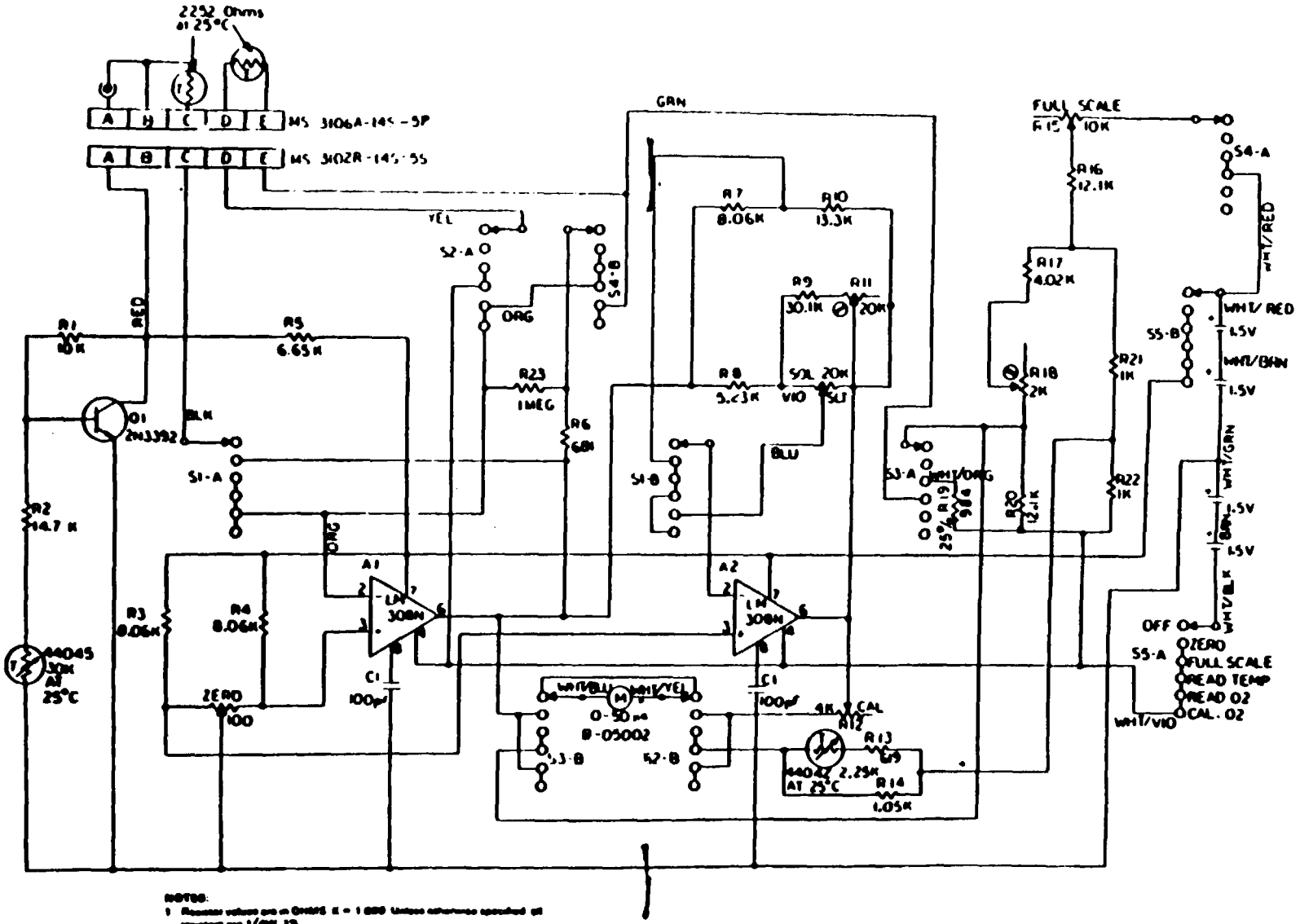
1. With switch in the OFF position, adjust the meter pointer to Zero with the screw in the center of the meter panel. Readjustment may be necessary if the instrument position is changed
2. Switch to ZERO and adjust to zero with zero control knob
3. Switch to FULL SCALE and adjust the FULL SCALE knob until the meter needle aligns with the "15" mark on the mg/l scale.
4. Attach the prepared probe to the PROBE connector of the instrument and adjust the retaining ring finger tight
5. Before calibrating allow 15 minutes for optimum probe stabilization. Repolarize whenever the instrument has been OFF or the probe has been disconnected.

III. Calibration

The operator has a choice of three calibration methods — Winkler Titration, Saturated Water, and Air. Experience has shown that air calibration is quite reliable, yet far simpler than the other two methods. The three methods are described in the following paragraphs.

Winkler Titration

1. Draw a volume of water from a common source and carefully divide into four samples. Determine the oxygen in three samples using the Winkler Titration technique and average the three values. If one of the values differs from the other 2 by more than 0.5 mg/l, discard that value and average the remaining two
2. Place the probe in the fourth sample and stir
3. Read temperature of calibration sample and set solubility dial to sample temperature. Observe correct salinity. Allow the probe to remain in the sample at least two minutes before setting temperature
4. With switch in READ O_2 position, set CALIB O_2 Knob to the average value determined in Step 1. Leave in the sample for an additional two minutes to verify stability



NOTES:

1. Resistor values are in OHMS K = 1,000. Unless otherwise specified all resistors are 1/4W, 1%.
2. 6 batteries are required. Use Eveready 936 cells or equivalent.
3. All switch sections are ganged.
4. The values shown on the schematic may differ from those on the resistors. If so, either value may be used for replacement purposes.

MODEL 910 OXYGEN METER
C 002307

This schematic is representative only, and may be slightly different from the circuit in your instrument.

CHM 001 0297

Saturated Water

1 Air saturate a volume of water (300-500cc) by aerating or stirring for at least 15 minutes at a relatively constant temperature
2 Place the probe in the sample and stir
3 With the switch in the CALIB O_2 position, adjust the CALIB Knob to the mark for the local altitude Leave probe in sample for 2 minutes to verify stability

Air Calibration

1 Switch to CALIB O_2 position
2 Place the probe in moist air B O D probes can be placed in partially filled B O D bottles Other probes can be placed in YSI 5075A Calibration Chamber (Refer to the following section describing CALIBRATION CHAMBER) or the small calibration bottle (the one with the hole in the bottom along with a few drops of water The probe can also be wrapped loosely in a damp cloth taking care the cloth does not touch the membrane Wait approximately 10 minutes for temperature stabilization. This may be done simultaneously while the probe is stabilizing
3 With the CALIB Knob set the meter pointer to the mark for the local altitude Be sure reading is steady. For calibration at altitudes higher than 7000 feet above sea level, see Table II. Recalibration is recommended when altitude is changed A 1000 ft. altitude change can result in a 3% reading error – 0 3 mg/l at 10 0 mg/l

The probe is now calibrated and should hold this calibration value for many measurements Calibration can be disturbed by physical shock touching the membrane, or drying out of the electrolyte Check calibration after each series of measurements and in time you will develop a realistic schedule for recalibration For best results when not in use, follow the storage procedures recommended for the various probes described under OXYGEN PROBES AND EQUIPMENT This will reduce drying out and the need to change membranes

Calibration Chamber

The YSI 5075A Calibration Chamber is an accessory that helps obtain calibrating conditions when air calibrating in the field and is also a useful tool when measuring in shallow water (less than 4 ft) As shown in Figure 9 it consists of a 4 1/2 ft stainless steel tube (1) attached to the calibration chamber (5) and the measuring ring (7) For calibration insert the solid rubber stopper (6) into the bottom of the calibration chamber (5) Push the probe (4) through the hollow rubber stopper (3) as shown in Detail A For maximum accuracy wet the inside of the calibration chamber (5) with fresh water This creates a 100% relative humidity environment for calibration Insert the probe stopper assembly in the top of the calibration chamber

During calibration hold the calibration chamber under water and calibrate as described in the Air Calibration procedure Keep the handle above the water at all times After calibration the chamber can be used as a measuring aid by removing the probe stopper assembly from the calibration chamber (5) and placing it in measuring ring (7) (See Detail C) Slowly stir the water with the wand when measuring

IV Dissolved Oxygen Measurement

With the instrument prepared for use and the probe calibrated place the probe in the sample to be measured and provide stirring

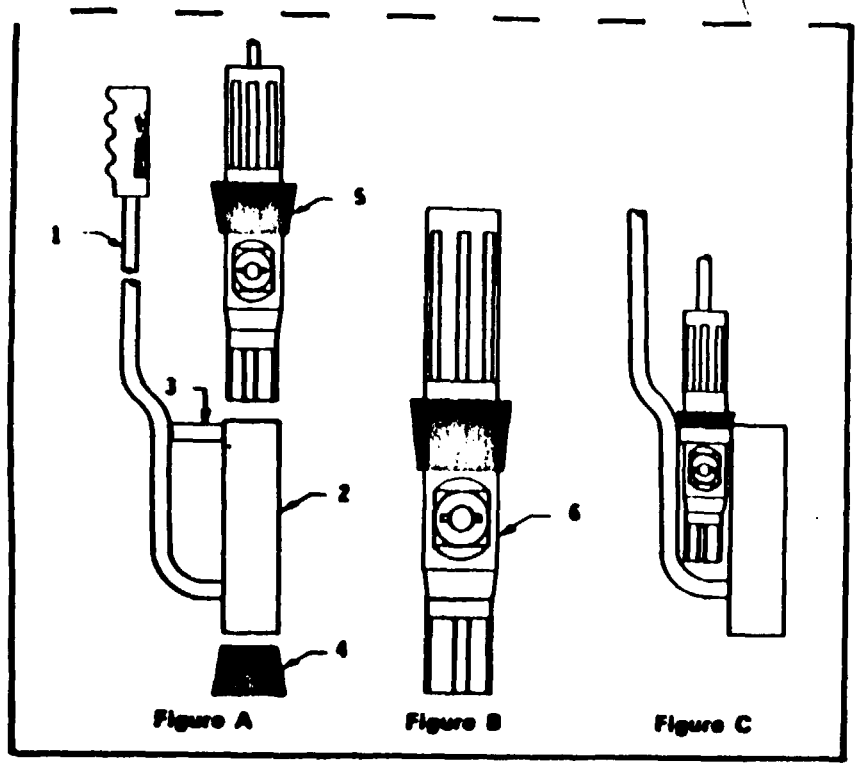


FIGURE 9

1. Stirring for the 5739 Probe can best be accomplished with a YSI Submersible Stirrer. If the Submersible Stirrer is not used, provide manual stirring by raising and lowering the probe about 1 ft. per second. If the 5075A Calibration Chamber is used, the entire chamber may be moved up and down in the water at about 1 ft. per second.
2. The YSI 5720A has a built-in power driven stirrer.
3. With the YSI 5750 sample stirring must be accomplished by other means such as with the use of a magnetic stirring bar.
4. Allow sufficient time for probe to stabilize to sample temperature and dissolved oxygen.
5. Turn the switch to TEMP and read temperature from the lower meter scale Set the O_2 SOLUBILITY FACTOR dial to the observed temperature, taking care to use the appropriate salinity index (See SALINITY CORRECTION)
6. Turn the switch to READ O_2 and read the dissolved oxygen value in mg/l directly from the meter.

Salinity Correction

Less oxygen can be dissolved in salt water than in fresh water. The amount varies directly with the degree of salinity and, at constant temperature, the relationship can be considered linear for the range of fresh water to sea water, which corresponds to the instrument range of 0 to 20,000 mg/l Chloride

It is necessary for the operator to determine salinity by suitable means (such as with the YSI Model 33 S-C-T Meter) and then to choose the correct position along the index scale when dialing in the temperature (See Figure 10)

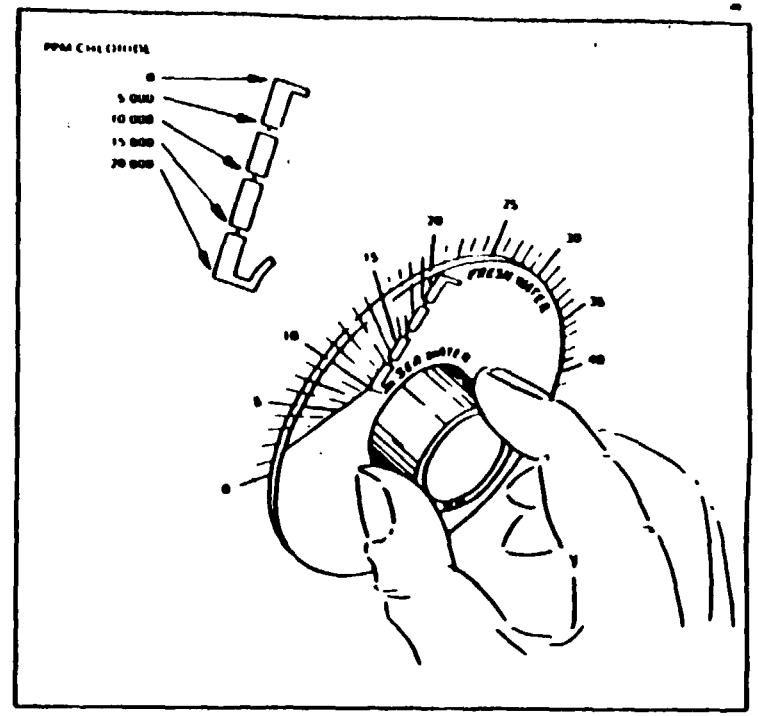


FIGURE 10

Each section of the bar on the O_2 SOLUBILITY FACTOR dial represents 5,000 mg/l Chloride concentration over a range from 0 to 20,000 mg/l The line leading to the correct temperature should intersect the left edge of the bar at the proper salinity concentration The drawing shows the dial set for 0 mg/l Chloride at 20°C, or 10,000 mg/l Chloride at 15°C, or about 18,000 mg/l Chloride at 10°C, etc.

Multiple Measurements

If a series of measurements are made in a short time at about the same temperature (within 5°C of calibration temperature) performance will not be degraded and recalibration is not required, simply:

1 Read temperature of new sample
2 Reset O_2 SOLUBILITY FACTOR DIAL
3 Read oxygen concentration Experience is the best guide for deciding how often recalibration is required Careful probe maintenance and storage aid stability of calibration

V. % Oxygen & % Air Saturation Measurements

Occasionally it is desirable to measure the % oxygen in a sample or the % air saturation of a sample The YSI Model 51B can be used for these measurements with any of the YSI 5700 Series Probes with an altered calibration technique as follows

% Oxygen Readings Calibration Procedure (0-45%)

1. Prepare the probe for operation as previously discussed
2. Connect the probe to the instrument
3. With the instrument OFF adjust the meter to zero on *upper scale* using the adjustment screw
4. Switch to FULL SCALE and adjust the meter to the full scale position
5. Switch to READ O_2 position and leave the instrument on up to 20 minutes to stabilize the probe
6. Set O_2 SOLUBILITY FACTOR dial to 25°C position
7. Switch to ZERO position and adjust meter to zero on the *lower scale* with the ZERO knob
8. Switch to READ O_2 and with the probe in air adjust the meter to 21 on the *lower scale* using the CALIB knob.
9. Repeat Steps 7 and 8 until no further adjustment is required
10. Transfer the probe to the measurement sample and read on the lower scale with the instrument still in the READ O_2 position. All readings will be in $\%O_2$. Accuracy will be ± 1% O_2, worst case

NOTE: Temperature readings may be made with the switch in TEMP position. The SOLUBILITY FACTOR dial must be left in the 25°C position.

% Air Saturation Readings (0-100%)

1 thru 4 Same as in % Oxygen Readings.

5. Turn the selector switch to ZERO and adjust the meter to zero on the *upper scale* using the ZERO adjustment knob.
6. Switch to CALIB O_2 and with the probe in air adjust the meter to 10 on the upper scale using the CALIB knob
7. Transfer the probe to the measurement sample and read on the upper scale with the instrument still in the CALIB O_2 position. Multiply by 10 to obtain % air saturation. For example, if the meter reads 8.5, multiply by 10 for an answer of 85% air saturation.

NOTE: Temperature readings may be made with the switch in the TEMP position. The O_2 SOLUBILITY FACTOR dial is inoperative and unnecessary when making % air saturation readings

NOTE: When $\%O_2$ or % air saturation measurements are to be made in water, greatest accuracy will be achieved if the probe is calibrated *in moist air* by suspending it in a bottle containing a small amount of water, or by wrapping it in a damp cloth during calibration steps.

VI. Recording Data

Although the YSI Model 51B is not designed with recorder output, it is possible for the user to modify the instrument slightly in order to record % O_2 and % air saturation. Connect a 100 mV recorder with minimum 50K ohm input impedance across the meter terminals. Set up the recorder according to the manufacturer's instructions and operate the YSI Model 51B as described.

Dissolved oxygen measurements in mg/l cannot be recorded accurately except under constant temperature conditions. This is because the solubility of oxygen in water is temperature dependent and instrument correction is manual. If recording of dissolved oxygen is required in your application, we recommend either the YSI Model 54 A or Model 57 Dissolved Oxygen Meter, which have automatic temperature compensation and are designed with recorder output terminals.

VII. Calibration Tables

The mg/l tables in accordance with standard practice include the vapor pressure of water in the indicated pressure. The sample of air used for calibration should be saturated with water vapor for greatest calibration accuracy.

The error which may result from use of completely dry air (extreme condition) is small and is maximum at higher temperatures and altitudes. At 25°C and 760 mm pressure the extreme error would be 0.3 mg/l. for 50% RH. 0.15 mg/l. If the error is of concern, wet the inside of the sampler cup before use to provide 100% RH. Then no error will occur using the tables provided.

Table I

SOLUBILITY OF OXYGEN IN WATER (Saturated with Air) IN mg/l AT VARIOUS TEMPERATURES AND PRESSURES

TEMPERATURE IN DEGREE CENTIGRADE	P mm	775	760	750	725	700	675	650	625
	P inches	30.61	29.92	29.53	28.54	27.56	26.57	25.59	24.61
0		14.09	14.00	14.41	13.92	13.44	12.96	12.47	11.99
1		14.47	14.19	14.00	13.53	13.06	12.59	12.12	11.65
2		14.08	13.81	13.63	13.17	12.71	12.26	11.80	11.34
3		13.71	13.44	13.26	12.82	12.37	11.93	11.48	11.03
4		13.35	13.09	12.92	12.48	12.05	11.61	11.18	10.75
5		13.00	12.75	12.58	12.16	11.73	11.31	10.89	10.47
6		12.68	12.43	12.26	11.85	11.44	11.03	10.61	10.20
7		12.36	12.12	11.96	11.56	11.15	10.75	10.35	9.95
8		12.07	11.83	11.67	11.28	10.89	10.49	10.10	9.71
9		11.78	11.55	11.40	11.01	10.63	10.24	9.86	9.47
10		11.50	11.27	11.12	10.74	10.37	9.98	9.62	9.24
11		11.22	11.00	10.85	10.49	10.12	9.75	9.39	9.02
12		10.96	10.76	10.62	10.26	9.90	9.54	9.18	8.82
13		10.73	10.52	10.38	10.03	9.68	9.33	8.97	8.62
14		10.50	10.29	10.15	9.81	9.48	9.12	8.78	8.43
15		10.27	10.07	9.94	9.60	9.28	8.92	8.59	8.25
16		10.05	9.86	9.72	9.39	9.06	8.73	8.40	8.07
17		9.84	9.65	9.52	9.20	8.87	8.55	8.23	7.90
18		9.64	9.45	9.32	9.01	8.69	8.37	8.05	7.74
19		9.45	9.28	9.14	8.82	8.51	8.20	7.89	7.58
20		9.26	9.07	8.95	8.64	8.34	8.03	7.73	7.42
21		9.08	8.90	8.78	8.48	8.18	7.88	7.58	7.28
22		8.90	8.72	8.60	8.31	8.01	7.72	7.42	7.13
23		8.73	8.56	8.44	8.15	7.86	7.58	7.29	7.00
24		8.57	8.40	8.29	8.00	7.72	7.43	7.15	6.88
25		8.41	8.24	8.13	7.85	7.57	7.29	7.01	6.73
26		8.26	8.09	7.98	7.70	7.43	7.15	6.88	6.60
27		8.11	7.95	7.84	7.57	7.30	7.03	6.76	6.49
28		7.97	7.81	7.70	7.44	7.17	6.90	6.64	6.37
29		7.83	7.67	7.56	7.30	7.04	6.78	6.51	6.25
30		7.70	7.54	7.44	7.18	6.92	6.66	6.40	6.14
31		7.58	7.41	7.31	7.05	6.80	6.54	6.29	6.03
32		7.43	7.28	7.18	6.93	6.68	6.43	6.17	5.92
33		7.31	7.16	7.06	6.81	6.57	6.32	6.07	5.82
34		7.20	7.05	6.95	6.71	6.46	6.22	5.97	5.73
35		7.07	6.93	6.83	6.59	6.35	6.11	5.87	5.63
36		6.96	6.82	6.72	6.49	6.25	6.01	5.77	5.53
37		6.85	6.71	6.62	6.38	6.15	5.91	5.67	5.44
38		6.75	6.61	6.52	6.28	6.05	5.82	5.59	5.35
39		6.65	6.51	6.42	6.19	5.96	5.73	5.50	5.27
40		6.55	6.41	6.32	6.09	5.86	5.64	5.41	5.18
41		6.44	6.31	6.22	6.00	5.77	5.55	5.32	5.10
42		6.35	6.22	6.13	5.91	5.69	5.46	5.24	5.02
43		6.26	6.13	6.04	5.82	5.60	5.38	5.16	4.94
44		6.17	6.04	5.95	5.73	5.52	5.30	5.08	4.86
45		6.08	5.95	5.86	5.65	5.43	5.22	5.00	4.78
46		5.99	5.86	5.77	5.56	5.35	5.13	4.92	4.70
47		5.91	5.78	5.70	5.48	5.27	5.06	4.85	4.63
48		5.83	5.70	5.62	5.41	5.19	4.98	4.77	4.56
49		5.75	5.62	5.54	5.33	5.12	4.91	4.70	4.49
50		5.68	5.54	5.46	5.25	5.04	4.83	4.63	4.42

Source Derived from 15 Edition "Standard Materials for the Examination of

It should be noted that the barometric pressure as quoted by the Weather Bureau is not the true atmospheric pressure of the local, but it is corrected to an equivalent sea level reading.

For a reported pressure of 760 mm the true atmospheric pressure at a given altitude is shown in Table II.

Table II
RELATION OF ATMOSPHERIC PRESSURE TO ALTITUDE

Altitude	True Atmospheric Pressure	
Sea Level	760	
1000 Feet	733	
2000 Feet	707	
3000 Feet	681	
4000 Feet	656	
5000 Feet	632	CALIB Setting for Altitudes Above 7000 Feet Set Meter to Read mg/l
6000 Feet	609	
7000 Feet	586	
8000 Feet	564	10 02
9000 Feet	543	9 64
10,000 Feet	523	9 29
11,000 Feet	503	8 93

The temperature-solubility relationship of oxygen in sea water is not the same as that in fresh water.

The solubility of oxygen in sea water is given in Table III.

Table III
SOLUBILITY OF OXYGEN IN SEA WATER
(Chloride concentration 20,000 mg/1)

Temp. °C	Solubility mg/l	Temp. °C	Solubility mg/l
0	11.41	16	7.91
1	11.11	17	7.78
2	10.83	18	7.61
3	10.56	19	7.47
4	10.30	20	7 33
5	10.05	21	7.20
6	9 82	22	7.07
7	9 59	23	6 95
8	9.37	24	6.83
9	9.16	25	6.71
10	8.96	26	6 60
11	8.77	27	6 49
12	8 58	28	6 38
13	8 41	29	6 28
14	8 24	30	6 18
15	8 07		

Source: Derived from 15th Edition "Standard Materials for the Examination of

DISCUSSION OF MEASUREMENT ERRORS

When comparative measures are made on the same sample, under the same conditions, and with the same instrument, the reading should reproduce to ±0 1 mg/l or better. Many factors can contribute to measurement errors. The error figures given below are "worst case absolute error accumulation" where each contributing factor is at its extreme tolerance value and the sum of contributing factors is taken as the worst possible combination. There are three main types of errors.

TYPE 1 Errors due to instrument design, quality control and component limitations. They cannot be reduced without elaborate individual instrument calibration procedures.

1% meter tolerance 0 15 mg/l max

Amp and circuit 0 05 mg/l max

(Worst case Type 1 error ±0 20 mg/l max.)

TYPE 2 Errors arising from temperature and oxygen sensor limitations, non-linear response, and membrane-to-membrane difference. The maximum permissible error is specified, and good maintenance will limit this type of error to those value.

Automatic temperature compensation for membrane temperature coefficient 0 03 mg/l max.

Dial Solubility control error

(1% component 0.10 mg/l max.)

Tracking of panel dial and printing error

-5 to +30°C — 0.05 mg/l max.

(30 to 45°C — 0.10 mg/l max.)

+0.2°C probe error — 0.14 mg/l max.

(Worst case Type 2 error: ±0.32 mg/l max.)

TYPE 3. Errors arising from assumptions made about the environment in which the measurements are made and as such are subject to control by the operator.

Altitude Effect —

1000 ft. change gives about a 3% error or 0 3 mg/l at 10 mg/l level.

Barometric Pressure —

Normal local variation is less than ±0.5" Hg., or 0 15 mg/l max

Humidity —

If less than 100% RH in calibration chamber. Assume only 50% RH

The error varies with temperature.

TEMPERATURE	ERROR/mg/l
0°C	0 02
10°C	0 05
20°C	0 12
30°C	0 27
40°C	0 68

Neglecting the altitude error —

(Worst case Type 3 errors at 20°C. ±0 27 mg/l max.)

Under the worst conditions with all errors additive — The error at 20°C could amount to: ±0.79 mg/l (max.)

If Type 3 errors are omitted — the worst case error would be: ±0.52 mg/l (max.)

If calibration is achieved at or within 1°C of the sample temperature — the error can be further reduced to: ±0.38 mg/l (max.)

Bearing in mind that these accumulated errors represent manufacturing acceptability limits and that probability of all errors adding in the same direction is low, the probable error accumulation is about 1/2 the maximum: 0.26 mg/l if Type 3 errors are eliminated, and 0.19 mg/l if careful temperature matching technique is employed.

CIRCUIT DESCRIPTION, MAINTENANCE & CALIBRATION

The Model 51B contains two separate circuits:

1. A temperature bridge circuit.
2. An amplifier for oxygen measurement.

The amplifier features integrated circuitry for good temperature stability, low voltage power requirements and long battery life. Current from the oxygen probe develops a voltage across a resistor network which includes a thermistor (kept at O_2 probe temperature). This voltage is applied to the input of the circuit. A portion of the amplifier output is applied to the amplifier input in a standard negative feedback configuration.

The amplifier output circuitry is designed to perform specific manipulations on the input signal to achieve dial-in O_2 solubility factor and to provide calibration adjustment means.

The only normal maintenance is battery replacement. Four "C" size flashlight batteries are required. Battery life is at least 1000 hours of operation or 6 months shelf life.

Battery replacement is indicated if the "full scale" adjustment cannot be made or O_2 calibration cannot be achieved. (Warning: a faulty probe will also not permit O_2 calibration.)

Replace batteries every six months to reduce danger of corrosion due to leaky batteries. To replace batteries — remove six screws from bottom plate — battery holders are color coded. Positive (+ button) end of battery must go to red.

It is possible that the O_2 SOLUBILITY FACTOR dial can become loose and slip from its normal position. In an emergency the dial can be repositioned with the following procedure. It must be emphasized that this is an emergency procedure only, and that the instrument should be returned to the factory for proper recalibration at the earliest opportunity.

1. Calibrate in air to the local altitude in the normal fashion.
2. Switch to TEMP and read temperature of the probe.
3. Refer to Table I and determine solubility of oxygen in water at the observed temperature and current barometric pressure. Consult Table II or call the local Weather Bureau for an exact reading. The more accurate the reading the more accurate will be the calibration.
4. Switch to O_2 and set the O_2 SOLUBILITY FACTOR dial to the observed temperature with salinity of fresh water. The mg/l indication should agree with Table I. If it does not, rotate the SOLUBILITY FACTOR dial until the mg/l indication does agree with Table I. Loosen the dial reposition to the pointer indicates the observed temperature. This is a temporary calibration only. As soon as possible the instrument should be returned for factory recalibration. YSI maintains complete facilities for repair and recalibration of all YSI products.

INSTRUMENT BATTERIES

Battery replacement is indicated if full scale adjustment cannot be achieved or O_2 calibration cannot be achieved. (Warning: A faulty probe will also not permit O_2 calibration.)

To replace batteries remove the four screws holding the rear cover of the instrument. The four batteries will be found on the battery terminal board inside. See Figure 11.

Battery holders are color coded. POSITIVE (+ button) end of battery must go to red.

Replace with Eveready No. 935 "C" size or equal.

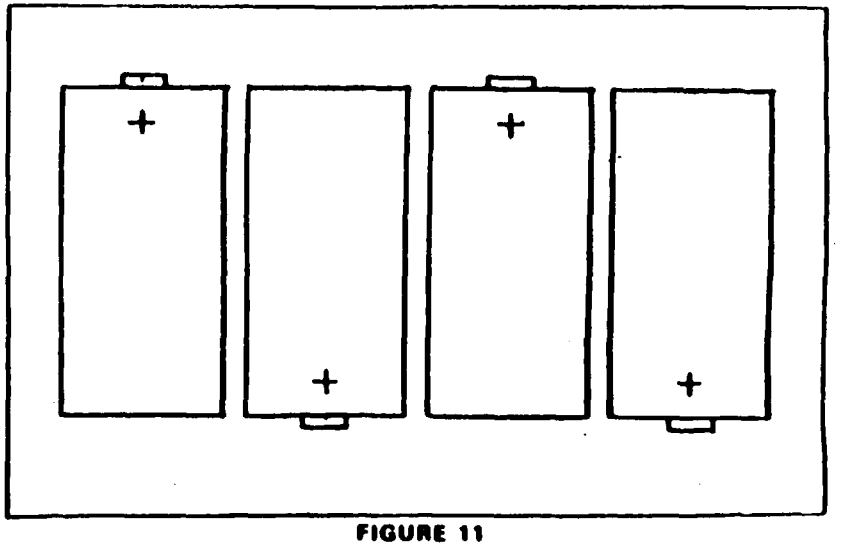


FIGURE 11

WARRANTY AND REPAIR

All YSI products carry a one-year warranty on workmanship and parts, exclusive of batteries. Damage through accident, misuse, or tampering will be repaired at a nominal charge, if possible, when the item is returned to the factory or to an authorized YSI dealer.

If you are experiencing difficulty with any YSI product, it may be returned for repair, even if the warranty has expired. YSI maintains complete facilities for prompt servicing of all YSI products.

Service Department
Yellow Springs Instrument Co., Inc.
P.O. Box 279
Yellow Springs, Ohio 45387, U.S.A.
Phone (513) 767-7241
Telex: 20-5437

MALCOLM PIRNIE

OPERATION PROCEDURES

MINIRAM MODEL PDM-3

MINIRAM PERSONAL MONITOR
MODEL PDM-3
OPERATIONS MANUAL

June 1988

MIE, INC.
213 Burlington Road
Bedford, Massachusetts 01730
(617) 275-5444 Telex 92-3339

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1.0 DESCRIPTION

1.1 Sensing Method

The MINIRAM (for Miniature Real-time Aerosol Monitor) Model PDM-3 is an ultra-compact personal-size airborne particulate monitor whose operating principle is based on the detection of scattered electromagnetic radiation in the near infrared. The MINIRAM uses a pulsed GaAlAs light emitting source, which generates a narrow-band emission (half-power width of 80 nm) centered at 880 nm. This source is operated at an average output power of about 2 mW. The radiation scattered by airborne particles is sensed over an angular range of approximately 45° to 95° from the forward direction by means of a silicon-photovoltaic hydrid detector with internal low-noise preamplifier. An optical interference-type filter is incorporated to screen out any light whose wavelength differs from that of the pulsed source.

The MINIRAM is a light scattering aerosol monitor of the nephelometric type, i.e., the instrument continuously senses the combined scattering from the population of particles present within its sensing volume (approximately 1 cm^3) whose dimensions are large compared with the average separation between the individual airborne particles.

1.2 Open Sensing Chamber Sampling Method

Air surrounding the MINIRAM passes freely through the open aerosol sensing chamber as a result of air transport caused by convection, circulation, ventilation, and personnel motion. The MINIRAM requires no pump for its operation, and the scattering sensing parameters have been designed for preferential response to the particle size range of 0.1 to 10 micrometers, ensuring high correlation with standard gravimetric measurements of both the respirable and thoracic deposition fractions. Optional flow accessories are available for applications requiring specific inertial particle precollection, extractive sampling, concurrent filter collection, etc.

It should be noted that one of the advantages of direct light scattering aerosol sensing is that the rate at which air passes through the sensor does not influence the indicated concentration because the detection is performed directly on every parcel of air traversing the fixed sensing volume.

Therefore, flow velocity through a real-time sensor such as the MINIRAM influences only the response time. So, it should not surprise the first-time user when, upon pressing the MEAS key of the MINIRAM, no pump noise is heard, and this silence will be accompanied by a readout message of "GO" on the liquid-crystal display indicating that the MINIRAM has, indeed, been activated.

1.3 MINIRAM Electronics

The MIE MINIRAM is a very advanced aerosol monitor which incorporates a custom-designed single-chip CMOS microprocessor whose functions are to: process the signal from the light scattering detection circuit, control the measurement sequence program, compute concentration averages, keep record of elapsed time, perform automatic zero correction, control auto-ranging, drive the liquid-crystal-display, store average concentration values as well as timing and identification information, sense battery and overload conditions, sequence playback of stored information, and provide alarm signals.

The MINIRAM derives its power from a set of internal rechargeable Ni-Cd batteries which can provide continuous monitoring operation for over 8 1/2 hours, or retain stored information for up to approximately 6 months. The battery set is packaged as a separable module which allows easy field replacement when recharging is not feasible. The MINIRAM can be run without time limit from an A.C. line using the charger provided with the instrument.

The MINIRAM has two output connectors. One provides a continuous, real-time analog signal output proportional to the aerosol concentration. This signal can be used for continuous recording (e.g., on a strip chart recorder), telemetry, or control purposes, etc. The other connector provides, during the measurement mode, either an ASCII digital output which is updated every 10 seconds, or a switched output for alarm purposes (depending on the user-selected function). Stored information playback can be accomplished either by means of the MINIRAM's own display or through the digital output jack. During the normal monitoring operation, the liquid-crystal-display indicates the aerosol concentration in the units of milligrams per cubic meter, and the displayed reading is updated every 10 seconds. When operating in the measurement or monitoring mode, other functions can be displayed

momentarily, i.e., as long as a corresponding touch switch is pressed. All external controls are performed by pressing one or more of 8 sealed touch switches on the MINIRAM panel.

1.4 Modes of Use and Application

The MINIRAM measures the concentration of any airborne particles, both solid and liquid, and the display indicates this level in the units of milligrams per cubic meter, based on its factory calibration, against a filter-gravimetric reference, using a standard test dust (Arizona road dust). Th MINIRAM can be used to measure the concentration of all forms of aerosol: dusts, fumes, smokes, fogs, etc.

Its small size and weight, and concentration averaging features permit its use as a personal exposure monitor, attached to a belt, shoulder strap, hard hat, etc. Alternatively, it can be used as an area monitor for both indoor and ambient air situations. Test chamber monitoring, visibility measurements, cloud detection (e.g., radio/drop sonde), aerosol dispersion studies, etc. are additional applications of the MINIRAM.

2.0 WHEN YOUR RECEIVE THE MINIRAM ...

Follow these steps when first receiving your MINIRAM:

2.1 Remove the instrument from shipping case.

2.2 Observe display. It should be blank indicating that the MINIRAM is in the minimum power mode.

2.3 Plug charger into A.C. line (standard charger is for 120V, 60 Hz; optional version available for 220V, 50 Hz).

2.4 Connect charger plug into corresponding MINIRAM receptacle.

2.5 Leave charger connected to MINIRAM for a minimum of 8 hours before using instrument without the charger.

2.6 You can operate the MINIRAM immediately after the charger has been connected. Follow operating instructions described in the next section of this manual.

3.0 OPERATING INSTRUCTIONS

Refer to Figure 1 for the location of control switches, display, and connector jacks. Refer to Figure 2 for the display timing sequences.

3.1 Initial Condition

Assuming that the batteries of the MINIRAM have been recharged (see section 2.0), the display may indicate one of the following conditions:

- Blank display: Means the MINIRAM had not been in the measurement mode for 48 hours or more, and is in the minimum power off mode.

- "OFF" display: MINIRAM has been in the off mode for less than 48 hours.

- Concentration display that changes or "blinks" once every 10 seconds: the MINIRAM is in the measurement mode.

3.2 To start Measurement Cycle

- **If the MINIRAM shows a blanked display (see above), press OFF and wait until the display reads "OFF" (approximately 5 seconds after pressing OFF), before pressing MEAS to initiate measurement cycle.**
- **If the MINIRAM shows "OFF" (see above), press MEAS directly to initiate measurement cycle (there is no need to press OFF first, in this case).**

The functions performed by pressing each MINIRAM touch switch are as follows:

3.3 MEAS

To start the monitoring operation of the MINIRAM, Press MEAS (see automatic timing sequence of Figure 2). The first readout displayed is either "GO" (or "CGO" if TIME is also pressed, section 3.4), followed by the last concentration reading or ".00". Approximately 36 seconds after pressing MEAS the first new 10-second-averaged concentration reading is displayed. All subsequent readings are concentration values in milligrams per cubic meter, updated every 10 seconds. Figure 3 shows a typical digital printout of a sequence of 10-second measurements (second data block).

The MINIRAM will now run in the measurement mode for 500 minutes (8 hours and 20 minutes), after which it will stop, displaying the OFF reading, retaining in storage the concentration average and elapsed time information. Once the MEAS mode has been entered this sequence can only be interrupted by pressing OFF; pressing ZERO, TWA, SA, TIME or ID# only affects the display during the time these keys are pressed, without affecting the measurement cycle. Pressing PBK during this cycle has no effect.

The instrument normally operates in the .00 to 9.99 mg/m^3 range. Whenever a 10-second concentration exceeds 9.99 mg/m^3 the MINIRAM display automatically switches to the .0 to 99.9 mg/m^3 range and remains in that range as long a the measured 10-second concentration exceeds 9.99 mg/m^3, otherwise the MINIRAM reverts to its lower range display.

3.4 MEAS and TIME

If both MEAS and TIME are pressed at the same time (press TIME first and while depressing it actuate MEAS) the MINIRAM will display "CGO" (for Continuous "GO"), and will then operate as above (i.e., pressing MEAS only), except that after the first 8.3 hour run it will restart automatically and continue to measure for an indefinite number of 8.3 hour runs, (with the battery charger) until the OFF key is pressed, or until the batteries are exhausted. Concentration averages and timing information for the last seven 8.3 hour runs will remain in storage at any given time.

3.5 OFF

When this key is pressed the MINIRAM will discontinue whatever mode is underway displaying "GCA"* followed by the display segments check ("8.8.8.") and finally "OFF" (see timing diagram of Figure 2). The MINIRAM will then remain in this reduced power condition (displaying "OFF") for a minimum of 10 minutes or a maximum of 48 hours or until the MEAS key is pressed to resume the measurement cycle.

If OFF is pressed during a measurement run the display will read "OFF" for 48 hours (unless another key is pressed during that period), after which the display will be blanked. Thereafter, if OFF is pressed the MINIRAM will display the "OFF" reading for only 10 minutes, after which the display will be blanked again unless another key is pressed during that period.

Every time the OFF key is pressed, during a measurement cycle, the MINIRAM will store the concentration average and elapsed monitoring time up to the time of that OFF command. The duration of the off period (up to 48 hours), i.e., between two consecutive measurement cycles, is also stored for each of up to 7 cycles.

If the MINIRAM is not reactivated (i.e., pressing MEAS) within 48 hours of the OFF Command, it automatically switches to a minimum power level, with blanked display; however, all data remains stored in memory for up to approximately 6 months without battery recharging (indefinitely, with charger).

OFF must be keyed before any other operating mode can be entered: setting ID#, zero referencing, playing back stored data, or changing the program code. Display functions, however, can be activated during the measurement mode.

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*"GCA" is displayed and printed out by the PDM-3 although the instrument is manufactured by MIE, Inc.

3.6 TIME

During the measurement mode, if TIME is pressed the display will show the elapsed time, in minutes, to three significant figures, from the start of the last measurement run. The MINIRAM will automatically return to concentration display after the TIME key is released.

3.7 TWA

This key stands for Time-Weighted-Average. During the measurement mode, if TWA is pressed the display will indicate the average concentration in milligrams/m^3 up to that instant, from the start of the last run. This average is computed by the MINIRAM applying the equation:

$$TWA = \frac{1}{t} \int_0^t C dt$$

where t is the elapsed run time and C is the instantaneous concentration at time t. The value of TWA is updated every 10 seconds. After releasing the TWA key the MINIRAM display returns to the 10-second concentration display.

3.8 SA

This key stands for Shift-Average. During the measurement mode, pressing SA will provide a display of the aerosol concentration, up to that moment, averaged over an 8-hour shift period. This average is computed by the MINIRAM applying the equation:

$$TWA = \frac{1}{480 \text{ Min.}} \int_0^t C dt$$

The shift-average value corresponds to the exposure from the start of the measurement cycle. Thus, for example, if the MINIRAM has been measuring for 3 hours, and the time-weighted average over that period has been 6 mg/m^3

(TWA reading), the shift average value at that time, (SA reading) would be 2 mg/m^3, which is equivalent to an 8-hour exposure at an average concentration of 2 mg/m^3.

The value of SA is updated every 10 seconds. When releasing the SA key the MINIRAM display returns to the 10-second concentration display.

3.9 PBK

With the MINIRAM in the off mode (i.e., not in the measurement mode), the stored information can be played back by pressing PBK. If the PBK key is initially pressed the display will indicate "P" for one second. If PBK continues to be pressed for more than 1 second, then the stored data is automatically played back through the MINIRAM display: First, the identification number is displayed with the ID indicator bar on; next the shift or run number (7 through 1, i.e., starting with the last run) is shown (with the OVR indicator bar on as identification); followed by the sampling (i.e., measurement) time in minutes, for that run; followed by the off-time between the last and next run (in tens of minutes); finally, the average in mg/m^3.* This sequence is repeated seven times. An average reading of 9.99 indicates that a significant overload condition occurred during that run. The total time required for the complete automatic playback on the MINIRAM display is approximately 70 seconds.

If PBK is pressed for less than one second "PA" will be displayed, and the stored data will be fed out through the digital output jack of the MINIRAM for printout, magnetic storage, telemetry, etc. A printout consists of 8 lines of data. Figure 3 shows a typical stored data printout (see data block labeled "Playback of Stored Data"). The first 7 lines show the data for the last 7 measurement periods, and the last line shows the identification number (I), the programmable selection code (F), and the zero value for that data block (Z). In addition a check sum is printed out on a 9th line for

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*Either the TWA or the SA values, depending on selected user-programmable code (see Section 4.2).

modem/computer data transfer purposes. The first 7 data lines are subdivided into 4 columns. The first column identifies the measurement period (starting with the last or 7th); the next column lists the corresponding duration of each measurement period, in minutes; the third column lists the off time between consecutive measurement periods, in minutes divided by 10; and the last column lists the average concentration values for each period in mg/m^3.*

Either time-weighted, or shift average values can be printed, depending on the selected programmable code (see section 4.2). The example shown on Figure 3 (F=0012) indicates that the TWA values are listed. Although the printout heading will indicate "PDM-2 LISTING" (as shown in Figure 3), this format applies equally to the MINIRAM model PDM-3.

The speed of the digital transfer to a printer or other digital device can be user selected through the programmable selection code (see section 4.2). For a 300 baud rate the transfer time for the stored data block is approximately 45 seconds. See sections 10.0 and 11.0 for instructions on how to connect the MINIRAM to a printer or other digital recording/processing device.

3.10 ZERO

The interior walls of the MINIRAM sampling chamber reflect a small amount of the light from the infrared source into the detector. This background level is referred to as the "zero value", and is automatically subtracted from all aerosol concentration readings during the measurement mode. The result is that the displayed readings depend only on the actual dust concentration present within the sensing chamber.

The zero value varies from instrument to instrument as well as with different sensing chambers. It will increase somewhat as the chamber inner walls and windows become contaminated with dust. A zero update should be performed after cleaning the sensing chamber (see section 12.0).

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*Either the TWA or the SA values, depending on selected user-programmable code (see Section 4.2).

Pressing ZERO during a measurement period provides momentary display of the stored zero concentration value used by the MINIRAM to correct all digital concentration readings (the analog output signal is not zero-corrected). To update the ZERO value the MINIRAM must be in its off condition (press OFF in case of doubt). Then, press ZERO and wait until the display again indicates "OFF".

The average of 4 consecutive 10-second zero level measurements will then be stored by the MINIRAM as the new ZERO reference value. (See timing diagram in Figure 2 and digital printout obtained during a typical zero check on Figure 3). When operating the MINIRAM in high particle concentration environments (5 mg/m^3) the zero value update should be performed approximately every 8 hours. At aerosol concentrations below approximately 1 mg/m^3 this update may only be required once a week, or even less frequently. The zero update should be performed either within a clean-air environment (ideally, a clean room or clean-bench) for dust measurements in the concentration range below 0.5 mg/m^3, approximately, or by flowing clean air through the sensing chamber of the MINIRAM (e.g., by means of an optional clean-air adaptor, MIE model PDM-1FZ Zero Check Module) (see section 17.2) for use at dust concentrations above 0.5 mg/m^3, approximately. Air conditioned offices (without smokers) usually have concentrations below approximately 0.05 mg/m^3 and can thus be used for zeroing purposes. When measurements are performed under essentially clean air conditions, e.g., in the same environment where the zero check was performed, the MINIRAM readings will indicate 0.00 mg/m^3 with small random fluctuations around that value. Positive values (e.g., 0.02) will thus be indicated on the LCD display. Negative values (e.g., -0.02) are suppressed and are also indicated as 0.00. The digital output, however, does include such negative values and these will be printed out by a digital printer (see sections 10.0 and 11.0)

3.11 ID#

Pressing ID# during a measurement period provides momentary display of the identification number stored within the MINIRAM memory.

The ID# key, in combination with other keys, is used for several additional programming functions described in the next section (4.0).

4.0 PROGRAMMABLE FUNCTIONS

4.1 ID# Selection

In order to change the instrument identification number the MINIRAM must first be in the off mode (i.e., press OFF). Then press the ID# key, and the presently stored number (between 1 and 999) will be displayed, as well as the ID indicator bar. To increment the identification number press the ▲ key (same key as TWA), and to decrement the number press the ▼ key (same key as SA). Any number between 1 and 999 can thus be selected and will remain in storage until the batteries are disconnected, or if the MINIRAM is not recharged over a 6-month period.

Pressing the OFF key after the above identification number selection will remove the MINIRAM from the ID# selection routine and lock-in that number until a new number is selected. A complete ID# lock-out (i.e., a routine to preclude panel-control change of that number) can be accomplished by a separate programmable code selection (see section 4.2).

4.2 Programmable Selection Code

The programmable code allows the user to panel-select several alternate functions and operating modes.

The program codes to select specific alternate operating modes are:

1 selects the alarm instead of ASCII digital output

2 selects the ID# lock-out

4 selects the TWA instead of the SA to be stored for playback

8 selects a 1-second pause after each printer carriage return (for slow printers)

32 selects 110 baud digital output rate instead of 300 baud

64 selects 600 baud digital output rate instead of 300 baud

These numbers are entered as a sum, e.g., to implement ID# lock out, TWA storage, and 1-second carriage return delay, the code number would be 14 (2+4+8).

To enter the desired code (e.g., 14) follow these steps:

- Press OFF key and wait until "OFF" is displayed.
- Press ID# key and set program code to desired number (e.g., 14) by means of the ▲ and ▼ keys.
- Press TIME key (this will show previously entered code).
- Press ID# key again to lock in the new program code which will then be displayed.
- The preceding steps will cause the ID# to become equal to the programmable selection code. To restore the desired ID# (without affecting the selected code number which is now locked in), use the ▲ and ▼ keys again to select the ID# for the instrument as described in section 4.1.
- Press OFF to exit the ID# selection routine.
- To look at the programmed code number, at any time, start from the off condition; press ID#, then press TIME ("F" will then be displayed momentarily), after which the code number will be displayed. Press OFF to exit the code number routine.

If no specific alternate code is entered the MINIRAM will operate in its standard mode (equivalent to code 12) consisting of the following:

- ASCII digital output
- Panel-selectable ID number (preset to 999)
- Time-Weighted Average (TWA) values in memory storage
- 7-bit ASCII resolution
- 300 baud digital output
- Printer carriage return followed by a 1 second delay

4.3 ID# Lock-out

If the ID# lock-out code has been selected (i.e., a 2 as part of the sum, as described in section 4.2) then both the ID# and the programmable code can only be displayed (and printed out), but neither of the two can then be

changed by means of the panel keys. In this case, in order to change the ID# if the lock-out code has been selected, or too alter the programmable code, the battery must be unplugged momentarily. Disconnecting the battery, however, causes the MINIRAM to lose all stored data, and cancels all alternate program codes which may then be restored following the procedure described in section 4.2.

4.4 Alarm Level Adjustment

If the selected program code includes a 1, the MINIRAM will not provide an ASCII digital output but instead a switched output (at the digital output connector) which will close every time the measured 10-second concentration value exceeds a presettable threshold concentration level. If a 1 has been included in the code, then the ID# divided by 10 becomes the alarm level in milligrams/m^3. This level can be adjusted following the ID# selection procedure of section 4.1, that is using the ▲ and ▼ keys to increment or decrement the number. For example, if an alarm level of 12.5 mg/m^3 is desired (and starting from the off mode), press ID#, adjust displayed number to 125 with the ▲ and ▼ keys, and press OFF. This number (e.g., 125) then becomes the ID# as well. It is not possible to enter a separate alarm level and ID# number.

5.0 OVERLOAD AND ERROR CODE INDICATORS

5.1 Bar Displays

There are three bar indicators on the MINIRAM display, identified as OVR, ID, and BAT. If the OVR bar is displayed at any time during operation in the measurement mode the MINIRAM detection circuit has been overloaded. A momentary overload can be caused by the insertion of an object into the sensing chamber, sudden exposure to sunlight, etc. If the cause of overload is eliminated, the OVR bar will disappear during the next 10-second display period, unless the overload persists for more than a total of 1 1/2 minutes over an 8 1/3 hour measurement cycle.

The ID bar display is activated only for display identification purposes and not for error conditions.

The BAT bar is displayed when the battery voltage becomes insufficient, indicating that the charger should be plugged into the MINIRAM.

5.2 Error Codes

The MINIRAM will display and output (at the digital output jack) error code numbers along with the corresponding overload indicator bars on the LCD readout. These codes will appear only if the problem persists for more than about 30 seconds.

The error code numbers are as follows:

.0.1: low battery condition

.0.2: RAM (digital processing)

.0.3: A/D (signal) overload

If an overload condition persists for more than approximately 1 1/2 minutes the selected concentration average value (SA or TWA) automatically registers 9.99 and that number will be indicated (or digitally transmitted) upon data playback, signifying an invalid measurement cycle. The OVR bar will then remain on for the rest of that run.

6.0 SENSING CHAMBER REMOVAL AND INSERTION

During normal operation of the MINIRAM the removable sensing chamber (see Figure 1) must be properly inserted, i.e., pushed all the way into the MINIRAM towards the display/control panel end of the instrument. When this chamber is properly positioned the surface on the opposite end from the display/control panel will be approximately flush with the body of the MINIRAM.

To remove the sensing chamber, gently push it away from the display/control panel end, using both thumbs, sliding it out of its channel. This will expose the shouldered metal button with its small spring-loaded plunger, and the two lenses (illumination and detection lenses). Touching of

these lenses should be avoided to prevent their soiling. Lens tissue should be used if cleaning of these lenses becomes necessary. Also, the inside surface of the removable sensing chamber is coated with a special anti-reflectant paint and these surfaces should not be touched, if at all possible. (Newer models have a special finish which is not touch-sensitive).

The removable sensing chamber has two small glass windows which should be kept clean (see section 12.0 on routine maintenance).

The sensing chamber is partially closed at one of its open ends. This end is inserted first when sliding the chamber back into the MINIRAM channel. A small shouldered slot is provided on the underside of the removable sensing chamber for the metal button that serves to retain the chamber.

To reinsert the sensing chamber simply slide it back into position making sure that the chamber is moved parallel to the MINIRAM body. Ensure complete insertion, as mentioned above.

7.0 BATTERY PACK REPLACEMENT

The battery pack of the MINIRAM (MIE Part No. PDM-3B) constitutes an intrinsically safe sealed module that can be removed and replaced. To do so, remove the four central screws from the back of the MINIRAM case (not the two corner screws), and gently lift the battery pack up and out, and gently pull apart the battery connector freeing the battery pack. Reverse order of steps when installing another pack.

CAUTION: All stored data will be lost when disconnecting battery.

After reconnecting battery pack, the ID resets to 999 and an automatic zero reference check is performed by the MINIRAM.

Separate battery packs can be used whenever a.c. line power is unavailable to recharge the pack within the MINIRAM. These spare parts can be recharged independently from the MINIRAM by plugging the charger into the charge receptacle which is an integral part of the battery pack (see figure 1).

8.0 CALIBRATION ADJUSTMENT

Although every MINIRAM has been factory-calibrated using a representative dust (see section 1.4), the user may wish to change the calibration constant of the instrument for a specific type of aerosol. Such a calibration should be performed by obtaining a concurrent filter collection (e.g., by means of a personal filter sampler), sampling from the same environment within which the MINIRAM is placed. The average concentration obtained by the MINIRAM (i.e. TWA reading) at the end of the test should be compared with the filter-gravimetric-determined concentration. The ratio of the two concentration values can then be used to correct the MINIRAM calibration. The comparison run should be replicated several times (to minimize errors) to obtain an average ratio.

To change the MINIRAM calibration proceed as follows:

8.1 Place MINIRAM in a clean environment (e.g. air conditioned office).

8.2 Remove battery pack (follow procedure of section 7.0).

8.3 Disconnect battery connector (remember that all stored data will thus be lost/erased from MINIRAM memory).

8.4 While leaving battery pack lying next to MINIRAM, re-connect the two units (i.e. plug in connector).

8.5 Immediately observe MINIRAM display. It will be performing a slow segment-by-segment display checkout. As soon as it displays ".00", press OFF, thus interrupting the initial automatic zero check (see section 7.0). Wait until the display indicates "OFF" and then press MEAS and wait approximately 36 seconds.

8.6 Observe 10-second readings (typically in the range of 1 to 3 mg/m^3) and record manually a few consecutive readings. Calculate the average of these values.

8.7 Identify small potentiometer screw (visible through an opening in the foil shield of the open MINIRAM) opposite the digital output jack. Adjust this potentiometer, using a fine screw driver, until the average MINIRAM reading is increased or decreased (with respect to the average obtained in 8.6) by the desired ratio (e.g. as determined by previous gravimetric comparison runs).

8.8 Shut off MINIRAM, reposition and secure battery pack, and re-zero instrument as usual. All subsequent concentration readings are now corrected by the desired ratio.

If an optional Reference Scatterer is available, insert in the MINIRAM instead of the normal sensing chamber and follow the same procedure (i.e., follow steps 8.1 through 8.8).

9.0 ANALOG RECORDER CONNECTION

The analog output of the MINIRAM is a negative voltage of 0 to 1.5 V. A high input impedance recorder (100K), or other signal processing device can be connected to that output. The 0 to -1.5V range corresponds approximately to 0 to 100 mg/m^3 as displayed by the MINIRAM.

This analog output (as opposed to the digital output and readings) is not zero-corrected, and thus a zero concentration results in a bias level of the order of several millivolts.

It is advisable to connect a capacitor in parallel with (i.e. across) the analog output (e.g. 100 microfarads or larger) in order to obtain a steady output signal. The internal time constant of the analog output of the MINIRAM is only 0.2 seconds which, in the absence of an external capacitor, results in excessive signal fluctuations.

Two miniature plugs are provided with the instrument to connect to the analog and/or digital output jacks (both can be used concurrently).

10.0 USE OF OPTIONAL MIE DIGITAL PRINTER

The MINIRAM can be connected to the MIE model DP-2-80C digital printer, an optional accessory designed for direct coupling to the MINIRAM. This printer can be used both to print out the continuous concentration data (updated every 10 seconds) in the normal measurement mode, and to print out the data stored in the MINIRAM memory as described in section 3.9.

An example of the printout formats when using the printer in combination with the MINIRAM is presented in figure 3. The DP-2-80C, a very compact impact dot matrix printer, is provided with a special interconnection cable to the MINIRAM digital output jack. When using the MINIRAM with this printer, the output data rate should be left at 300 baud (the normal MINIRAM default value), as described in section 4.2.

The following are specific operation procedures for use of the printer in combination with the MINIRAM. Other operating and maintenance information is contained in the instruction manual that accompanies the printer.

10.1 Printer Connection

A 20 mA current must flow through the DP-2-80C printer for it to operate. Plug its cable into the MINIRAM digital output receptacle and press OFF. Turn on the printer power switch (on its right side) and the two green lights on the front will be on if printing paper is in the unit. The ribbon cartridge should have been previously loaded. Refer to the printer User's Manual for details.

10.2 Printer Test

In order to test whether the DP-2-80C printer is operating correctly, hold down the LINE FEED button while turning the printer on. Once the printer is on, release the LINE FEED button and the printer will then print out all of its characters. To stop this operation press SELECT.

10.3 Printout of Stored Data

Plug printer connector into the MINIRAM digital output receptacle. Turn off printer power switch. Press OFF on MINIRAM and wait until it reads "OFF". Turn on printer power switch. Press PBK on MINIRAM for less than one second (see section 3.9) and the LCD display should then indicate "PA". The printer will then print out the stored data block.

10.4 Printout of Zero or of Measurement Data

Interconnect MINIRAM and printer as indicated before and switch on printer power. Press OFF on the MINIRAM. Press either ZERO or MEAS on MINIRAM (depending on which information should be printed out). Printer will print out zero data approximately 72 seconds after pressing ZERO on MINIRAM (see figure 2). The first line of measurement data will be printed out approximately 126 seconds after pressing MEAS, and thereafter every 100 seconds (each line contains ten 10-second measurements). The printer power can be turned off any time during the measurement cycle, and turned on again during a cycle to resume printing. The data line numbers (see figure 3) will then be the current ones as sequenced by the MINIRAM whose output is independent of the operations of the printer.

11.0 DIGITAL OUTPUT CONNECTIONS

A digital printer (other than MIE model DP-2-80C), data logger (MIE PDL-1), or modem may be coupled to the MINIRAM. The data output is in the form of 20 mA current loop, 300 baud (110 or 600 baud by alternate programming) asynchronous ASCII characters. The output load should be less than 50 ohms.

Figure 4 is a diagram showing the connections and components required for a 20 mA loop interconnection to a printer. A similar diagram is shown for standard RS232 interfacing with a printer (see Figure 5). These connections do not apply when using the DP-2-80C printer.

The MINIRAM does not send parity information, but does provide an ASCII check sum which is the sum of all ASCII characters, to insure data integrity.

To use the check sum the host computer must add the ASCII value of all digits, spaces, carriage returns, and line feeds except for the first two carriage returns and line feeds which are sent immediately after pressing the PBK switch. The last eight bits of this sum should then be expressed as a decimal number (0-255) and should agree with the decimal value of the MINIRAM check sum.

12.0 ROUTINE MAINTENANCE

When the MINIRAM is not being operated it should be placed in its shipping case which should then be closed. This will minimize the amount of particle contamination of the inner surfaces of the sensing chamber.

After prolonged operation within, and exposure to particlate-laden air, the interior walls and the two glass windows of the sensing chamber may have become contaminated with particles. Although repeated updating of the zero reference following the procedure of section 3.10 will correct errors resulting from such particle accumulations, eventually this contamination could affect the accuracy of the measurements as a result of excessive spurious scattering, and significant attenuation to the radiation passing through the glass windows of the sensing chamber.

An indication of excessive chamber contamination is provided by the zero level reading (section 3.10), which should not exceed 3 mg/m^3, approximately.

In order to clean a soiled sensing chamber remove that chamber as described in section 7.0 and wash it with soap and water, rinsing thoroughly to remove any residues from the glass windows and interior of the chamber. Do not use solvents of any type. Do not rub interior surfaces of the chamber (coated version). Allow the sensing chamber to dry completely and re-insert into the MINIRAM as indicated in section 7.0

13.0 PRECAUTIONS AND OPERATING POSITIONS

The interior of the MINIRAM sensing chamber should not be exposed to fluctuations of intense light; flashes of sunlight or bright daylight especially, are to be avoided. Such excessive variable illumination of the

scattering detector can result in significant measurement errors that may persist over several 10-second display cycles. In order to operate the MINIRAM under those conditions it is advisable to use the Sunshield accessory (MIE model PDM-SNS, see section 17.5).

Another potential source of error is the presence of reflecting surfaces in close proximity to the sensing chamber openings. Such objects should be kept at least 2 cm (3/4 inch) from the chamber openings.

The removable sensing chamber should not be used as a carrying handle, especially not while operating the MINIRAM; holding this chamber may affect the measurements.

When using the MINIRAM for personal monitoring it should be positioned vertically, i.e., with the display/control panel facing upwards, by either clipping the MINIRAM to the belt, shoulder strap, etc.

In general, an approximate vertical position is to be preferred for any long-term monitoring purposes, in that this position minimizes potential particle deposition within the removable sensing chamber.

Other monitoring positions are:

a) horizontal, resting on belt clip

b) hand held (while ensuring that hand and fingers are away from edges of sensing chamber)

c) Using the optional MINIRAM table stand

d) Wall mounted using belt clip, or the four battery pack attachment screws on the back of the MINIRAM.

14.0 INTRINSIC SAFETY

The MINIRAM has been designed to satisfy the requirements for intrinsically safe operation in methane-air mixtures. The sealed battery pack incorporates a current-limiting resistor that limits the battery short circuit current to less than 14A. MSHA 2G-3532-0 approval has been granted to the PDM-3.

15.0 SPECIFICATIONS

- Measurement ranges: 0.01 to 10 mg/m^3 and 0.1 to 100 mg/m^3
- Precision and stability (for 10 sec. readings)*: $\pm$ 0.03 mg/m^3 (2-sigma)
- Precision and stability of time-averaged measurements*:
  $\pm$ 0.02 mg/m^3 (for 1 minute averaging)
  $\pm$ 0.006 mg/m^3 (for 10 minute averaging)
  $\pm$ 0.003 mg/m^3 (for 1 hour averaging)
  $\pm$ 0.001 mg/m^3 (for 8 hour averaging)
- Temperature coefficient: 0.005 mg/m^3 per °C (typical)
- Readout resolution: 0.02 mg/m^3 or 0.1 mg/m^3 depending on automatically selected range (3 digit LCD)
- Digital readout updating time: 10 seconds
- Analog output time constant: 0.2 seconds
- Total measurement period: 8 1/3 hours, or indefinite 8 1/3 hour cycles
- Particle size range of maximum response: 0.1 to 10 μm in diameter
- Measurement display: normally 10 second real time measurement; or momentarily: time-weighted average, or 8-hour equivalent shift average, or elapsed sample time (in minutes), or zero value, or identification number, or programmable code
- Data storage: seven concentration averages, sampling periods in minutes (3 significant figure resolution), off time (10 minute resolution), identification number, zero value, programmable code, and check sum
- Real time outputs: analog (0 to 1.5V full scale), and digital ASCII
- Memory playback: either by own LCD display, or by 110, 300 or 600 baud, ASCII digital output (20 mA current loop, or RS232 terminals may be connected with appropriate interface)
- Nominal battery voltage: 7.5V
- Average battery current drain: 40 mA

---

*At constant temperature (typ. 25°C)

- Continuous operating time with full battery charge: 10 hours, approximately
- Operating temperature: 0° to 50°C (32 to 120°F) Storage: -20 to 60°C
- Outside dimensions: main body: 10 x 10 x 4 cm (4 x 4 x 2 inches); sensing chamber cover: 7.7 x 3.8 x 1.5 cm (3 x 1.5 x 0.6 inches)
- Weight: 0.45 kg. (16 oz.)

16.0 STANDARD ACCESSORIES

Accessories provided with each MINIRAM are detailed in the following subsections.

16.1 Battery Charger

The battery charger (MIE model PDM-1-157-1) serves the following functions: recharge or maintain the charge of the nickel-cadmium batteries within the MINIRAM, permit continuous a.c. power line operation, and provide power for the operation of pump of the optional MIE model PDM-1FZ Zero Check Module (see section 17.2). The charger cannot be used to power the MINIRAM without its batteries, however, it can be used to charge a separate or spare battery pack (MIE model PDM-3B, see section 7.0).

The standard battery charger is designed for a 120V/60 Hz input, however, it can be obtained for 220V/50 Hz if so specified.

16.2 Other Standard Accessories

Other accessories supplied with the MINIRAM are:

- Output connectors (can be used for the analog, and/or the digital output jacks);
- Shipping Case;
- Instruction Manual.

17.0 OPTIONAL ACCESSORIES

Several optional accessories are available from MIE for the MINIRAM, these are described in the following subsections.

17.1 Flow Adapter (MIE model PDM-1F)

The Flow Adapter when used in conjunction with the MINIRAM and any pump or external flow system, allows a sample to be drawn through the instrument sensing chamber. A personal monitoring pump at flow rates of 2 l/minute or less may be used.

To attach the Flow Adapter to the MINIRAM loosen the two thumbscrews and pull the front sealing plate forward. Slide the Adapter over the MINIRAM sensing chamber as illustrated above; secure the Adapter to the MINIRAM by tightening the two allen-head screws through the hold down tabs. Tighten the thumbscrews to seal the two end plates to the MINIRAM sensing chamber.

Typically, this accessory would be used when extracting samples from aerosol chambers, detecting leaks from pressurized ducting, or for isokinetic sampling using probes.

Notes:

When using any of the optional accessories that are attached on and around the sensing chamber (models PDM-1F, -1FZ, -1FS, -1FR, and -SNS) to perform measurements at concentrations below 0.5 mg/m^3, it is advisable to zero check the MINIRAM with the accessory in place, making sure that its mounting and sealing screws are properly tightened.

Use an external pump or pressurized air source (well filtered) to drive clean air through the sensing chamber to zero the MINIRAM with any of those accessories (except in the case of the Zero Check Module). To zero check the MINIRAM when using the Sunshield place instrument with the attached sunshield in a clean room environment (see Section 3.10).

17.2 Zero Check Module (MIE model PDM-1FZ)

The Zero Check Module can be used to zero the MINIRAM when a clean air environment is not available. The Zero Check Module should be used when concentrations in the range above 0.5 mg/m^3 are to be measured (see section 3.10).

In addition, this accessory can be used to draw a sample stream through the MINIRAM sensing chamber (in lieu of a separate pump) by disconnecting the small tube at the sensing chamber inlet fitting.

This accessory consists of a pump, filter and the necessary tubing to circulate clean filtered air through the MINIRAM. The pump may be powered by the MINIRAM battery charger (or a 5-10 VDC power supply). The battery within the MINIRAM cannot be used to operate the Zero Check Module.

To zero the MINIRAM, first attach the Zero Check Module following the same procedure described for attaching the basic Flow Adapter (see section 17.1). Connect the battery charger to the Zero Check Module and to an A.C. source. Allow at least one minute of operation to purge the sample chamber with clean air. Press the ZERO button on the MINIRAM and continue operating the Zero Check Module until the final average zero reading is displayed (see section 3.10).

17.3 Personal Sampler Adapter (MIE models PDM-1FS and PDM-2FS)

This accessory, when used with the MINIRAM and a personal monitoring pump, permits active sampling of respirable (cyclone preselected) particles through the instrument sensing chamber and collection on a filter. The aerosol sample is drawn through a 10 mm nylon cyclone (with a 50% cut point at 3.5 m when operated at 2 ℓ/minute), through the sensing chamber of the MINIRAM, and then collected on a filter located in the cassette/filter holder for subsequent gravimetric or other analysis.

The model PDM-1FS is for use with an MSA 37mm filter cassette no. 457193. The model PDM-2FS is compatible with a Millipore 37mm disk filter holder no. M000 037 A0.

To attach the Personal Sampler Adapter to the MINIRAM, follow the same procedure as described for attaching the basic Flow Adapter (see Section 17.1). Connect a length of tubing from the exhaust fitting on the filter holder to a personal sampling pump (not provided with the Adapter).

The use of the Personal Sampler Adapter permits concurrent MINIRAM readings and filter collection to facilitate calibration of the MINIRAM for a specific aerosol, or to determine both concentration and chemical composition of the aerosol.

17.4 Respirator Adapter (MIE model PDM-1FR)

The Respirator Adapter, when used in conjunction with the MINIRAM, provides a means of measuring aerosol concentrations inside a respirator. The external concentration can also be measured with the MINIRAM and thus the values obtained with the MINIRAM after connecting it to the respirator can be used to determine protection factors; consequently, quantitative fit checks are possible under field conditions.

WARNING: DO NOT USE THIS ACCESSORY IN A HAZARDOUS (TOXIC DUSTS, FUMES, GASES, ETC.) ENVIRONMENT, SINCE RESPIRATOR INTEGRITY CANNOT BE GUARANTEED BECAUSE OF THE POSSIBILITY OF LEAKS.

A tube should be attached from a tap on the respirator to the inlet (which is located on the smaller sealing plate) of the Respirator Adapter. When the respirator wearer exhales, a slight positive pressure develops inside the mask resulting in an air flow to the MINIRAM sample chamber, where the concentration is measured. This air then passes through a check valve as it exits the chamber. When the wearer inhales, the check valve closes to prevent exposure to ambient conditions. A back-up filter is also used after the check valve as an additional safety precaution in the event of check valve failure.

To attach the Respirator Adapter to the MINIRAM, follow the same procedure for attaching the basic Flow Adapter (see Section 17.1).

17.5 Sunshield (MIE model PDM-SNS), included with units ordered after 1 April 1987).

The sunshield accessory serves to protect the MINIRAM sensing elements from excessive ambient light fluctuations (see section 13.0). It should be used whenever the MINIRAM is to be operated outdoors or under fluctuating bright light illumination. It is also advisable to use the sunshield to prevent loose clothing or other objects from touching or entering the open sensing chamber. The use of the sunshield causes only a slight retardation of the air exchange rate between the outside and inside of the sensing chamber, an effect that is negligible except when using the analog output in order to follow rapid fluctuations of particle concentration. The sunshield attaches by its two support tabs to the body of the MINIRAM.

17.6 Shoulder Strap (MIE model PDM-SS)

The PDM-SS is a leather strap, worn over the shoulder and across the chest, which attaches to the wearer's belt in front and back. The MINIRAM mounting loop in the upper chest area allows exposure measurements close to the breathing zone while still permitting comfort and freedom of movement.

17.7 Table Stand (MIE model PDM-TS)

The table stand accessory provides a convenient mounting support for the MINIRAM when it is used for area monitoring. The MINIRAM is simply clipped onto the table stand which holds it in a position where reflections from the table surface do not interfere with its operation.

17.8 Dot Matrix Digital Printer (MIE model DP-2-80C)

This printer is supplied with a special interconnecting cable which is plugged into the digital output jack of the MINIRAM. The DP-2-80C is normally provided for 120V/60 Hz operation. Operation with 220V/50 Hz line can be provided upon customer request. The DP-2-80C weighs 4 kg (9 lbs), and its dimensions are 36 W x 27 D x 8H cm (14 x 11 x 3 inches).

The use of the DP-2-80C in combination with the MINIRAM is described in Section 10.0. A separate instruction manual for the printer is supplied with that unit.

17.9 Reference Scatterer (MIE model PDM-RS)

The PDM-RS is a specially modified sensing chamber that includes a diffusing optical filter mounted within the sensing region of the MINIRAM. It is designed to scatter a controlled amount of light from the infrared source to the detector, providing a stable and repeatable reading on the MINIRAM display. The reference scatterer is inserted into the MINIRAM instead of the normal sensing chamber, and the readings are obtained operating in the MEAS mode. If the PDM-RS is ordered from MIE concurrently with a MINIRAM the reference scatterer will be factory marked with the calibration reading to be obtained when inserted into that particular MINIRAM whose serial number will also be shown on the PDM-RS tag. The readings displayed by the MINIRAM when inserting the PDM-RS should be within +5% of the value marked on that reference scatterer.* The readings obtained with the reference scatterer may show a small warm-up drift (i.e. gradual change) during the initial 5 to 10 minutes after pressing MEAS.

If the reference scatterer is ordered separately from the MINIRAM, the user will then determine the calibration reading obtained on the MINIRAM and mark it (together with the MINIRAM serial number) on the PDM-RS tag.

Because of small differences in the optical configuration of each reference scatterer, the readings obtained with a given reference scatterer are unique to a given MINIRAM. The response to a given population of airborne particles, however, is the same for all factory calibrated MINIRAMs, within approximately ± 5%.

17.10 Carrying Case (MIE model PDM-HC)

The PDM-HC is a convenient and compact hard shell carrying case designed to house a MINIRAM and a battery charger. The inside is foam padded for full

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*Prior to the use of the reference scatterer the MINIRAM should be zeroed with a clean standard sensing chamber as described in Section 3.10.

protection. The outside dimensions of the PDM-HC-1 are: length - 9 1/2 inches, depth - 7 inches, and height - 3 1/2 inches.

17.11 Cable (MIE model PDM-CB)

The PDM-CB cable is used to connect the digital output of the PDM-3 with the input of the 80 column digital printer MIE model DP-2-80C.

17.12 Portable Data Logger (model PDL-1)

This data logger can be used to record, average, peak detect, etc. concentration levels measured by the PDM-3. A separate instructional manual is provided for the PDL-1.

List of Figures

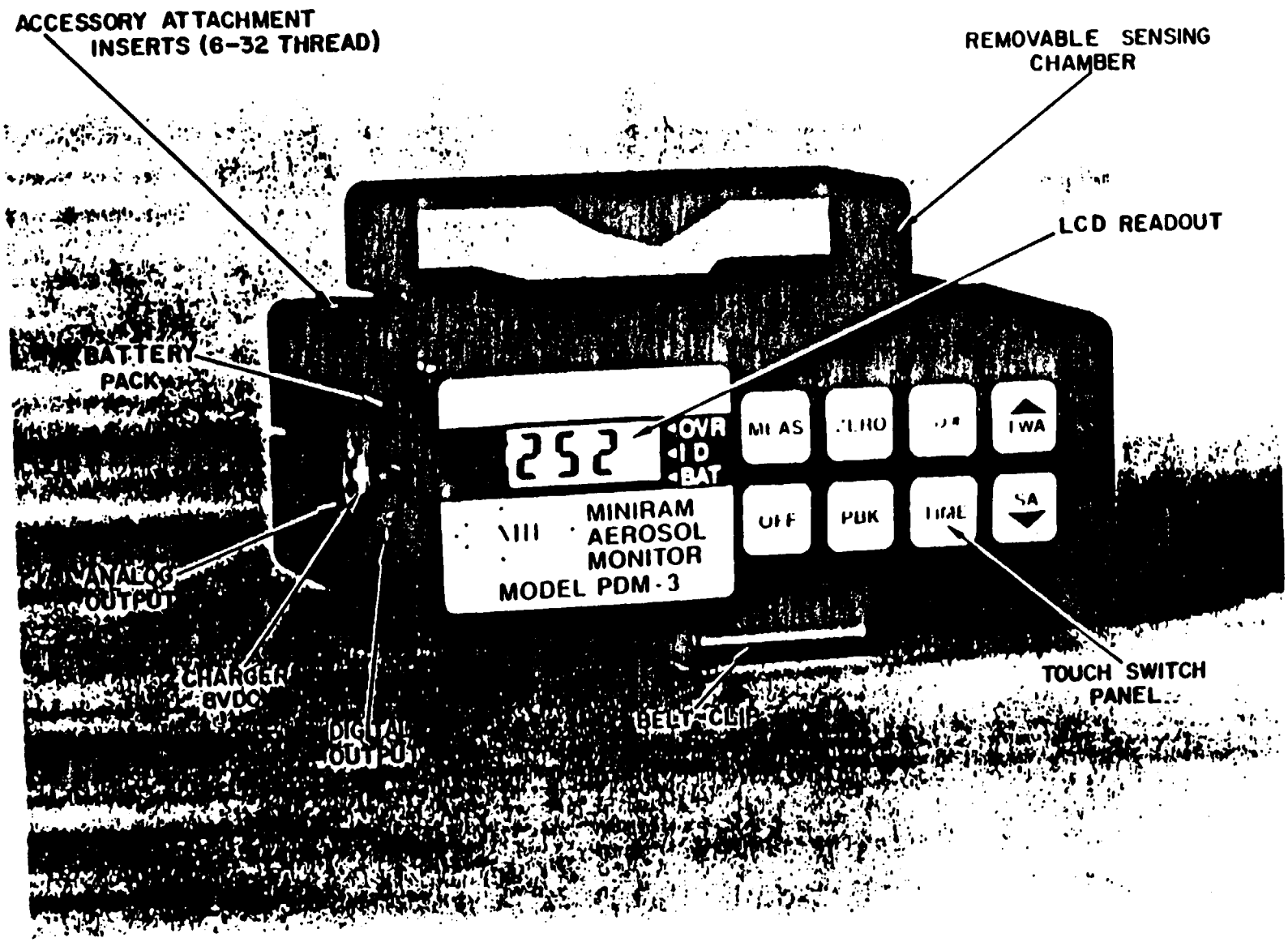


Figure 1. Main view of MINIRAM.

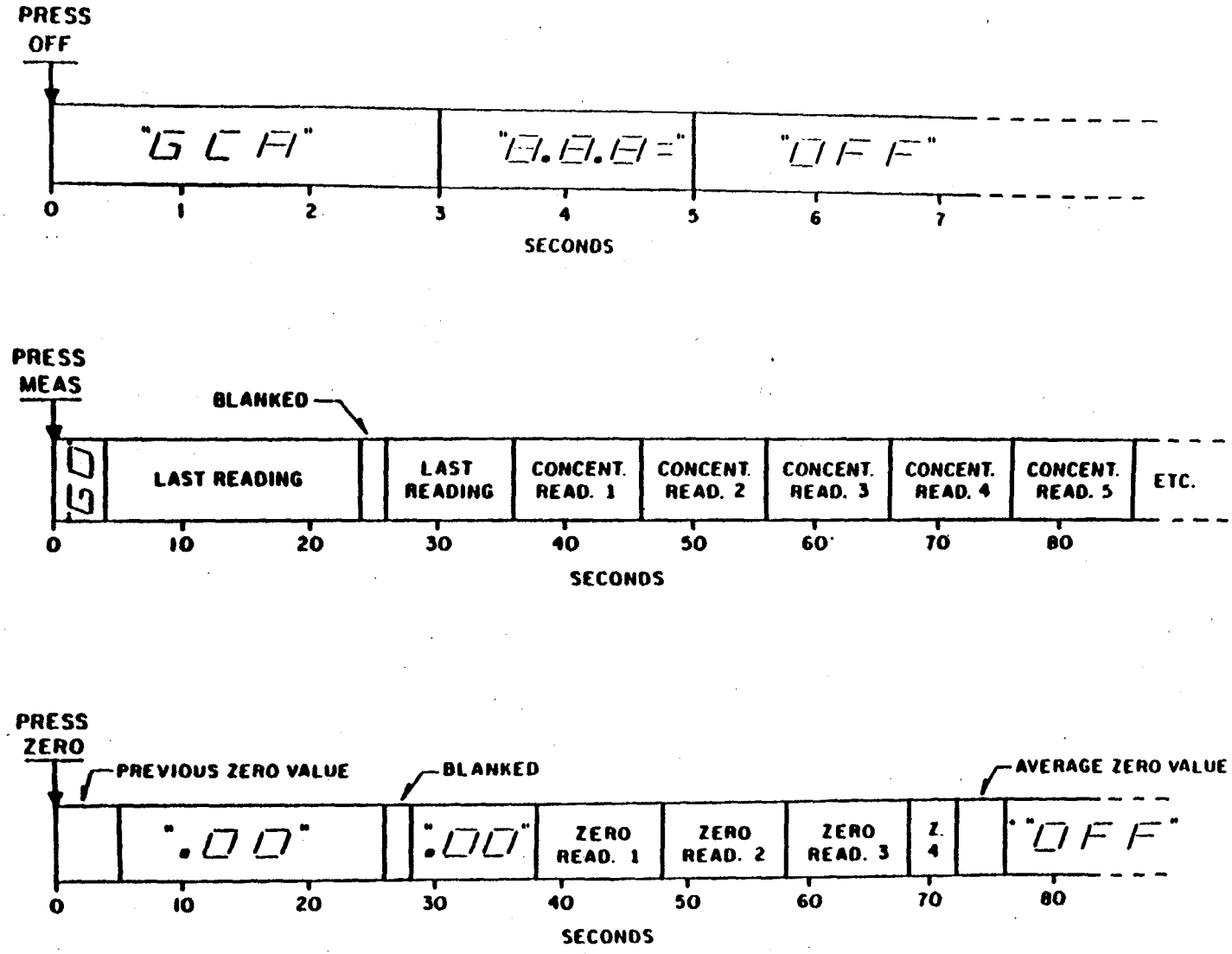


Figure 2. Timing diagram of MINIRAM model PDM-3 when pressing OFF, MEAS, or ZERO (typical times).

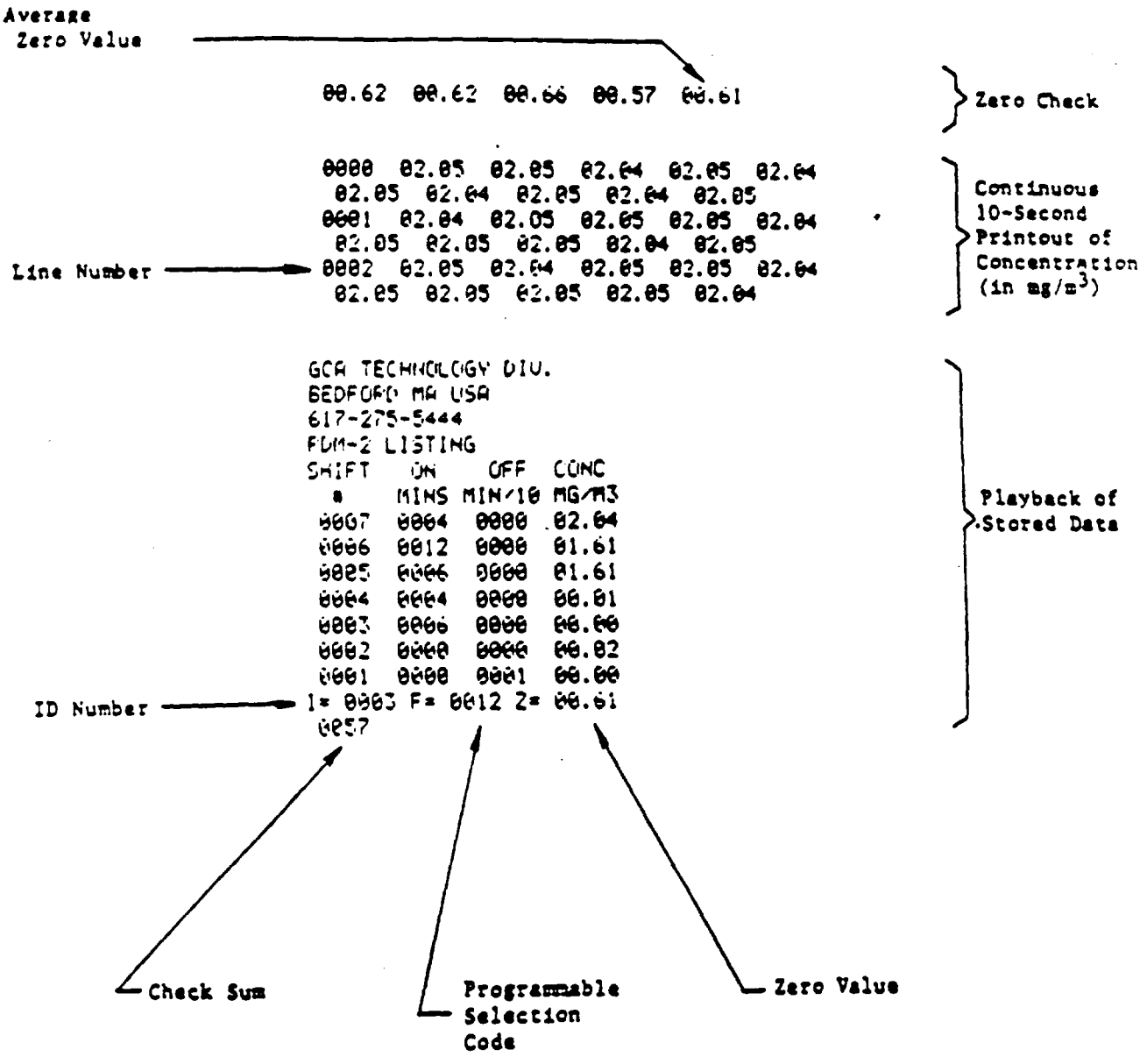


Figure 3. Typical MINIRAM Model PDM-3 Digital Printout Format.

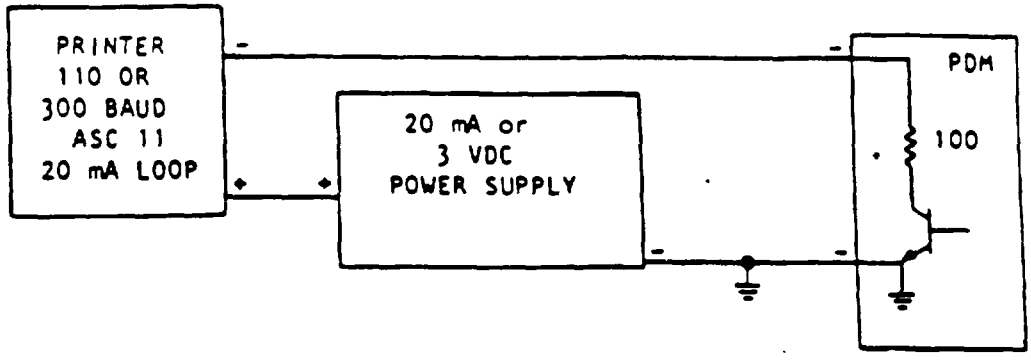


Figure 4. 20 mA loop connection.

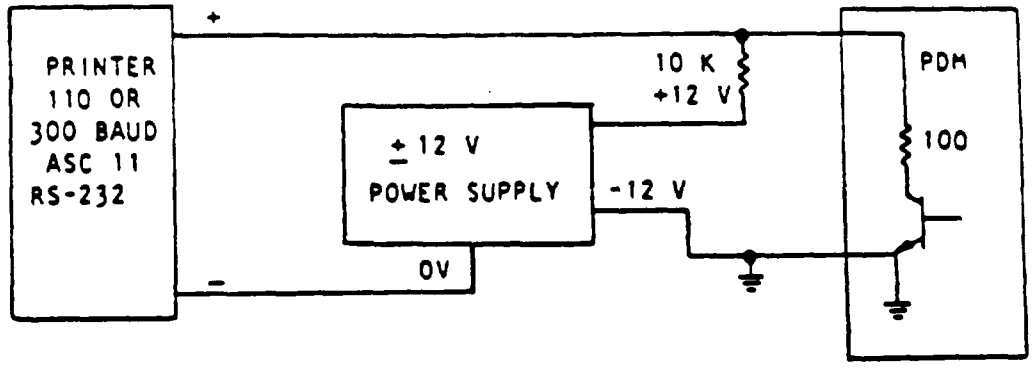


Figure 5. RS-232 connection.

Interface Meter

Model 121 Data Sheet

Model 121

Interface Meter

For accurate measurements of product level and thickness, the Solinst Interface Meter allows quick and easy determination of any air/product or water/product interface. The meter measures both floating and sinking layers.

Unique patented design* uses a sturdy probe with infra-red refraction to detect liquids and conductivity to distinguish water. The probe is 1-1/2" diameter and is ideal for use in monitoring wells.

The infra-red and conductivity sensors are both located at the zero measuring point. This avoids errors inherent in flotation devices. The narrow infra-red beam allows accurate measurement of product layers 50 thousandths of an inch or greater. Thinner layers are also detectable.

High Quality Design

Accurate

Datum allows easy measurement to 1/100 ft
Detects layers thinner than 0.050 inches.
Highly responsive sensors.
Both sensors located at zero measurement point.
Lengths from 25 ft. - 1500 ft.

Sturdy

Designed for rugged field use.
Cable uses stranded stainless steel conductors:
- non-stretch.
- resists kinking and breakage.
- inexpensive to repair/replace.

Rugged free-standing reel with carrying handle.

Simple

Simple to operate.
Easily cleaned.
Light weight.
Permanently attached operating instructions.
Easily replaced, long-life 9V batteries.

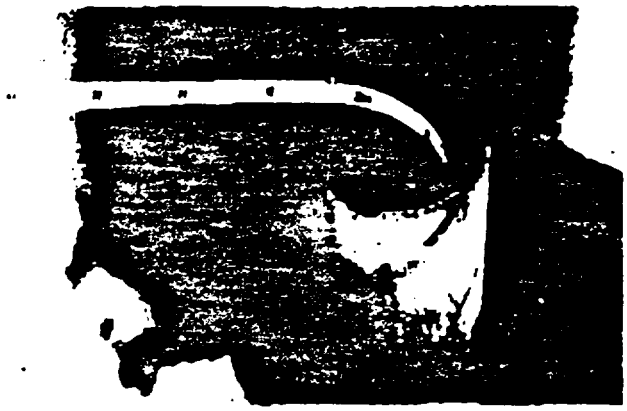
*Patents pending

Instrumentation to measure the properties of

Operating Principles

To detect liquids, the Solinst Interface Meter uses an infra-red beam and receptor. The beam is powered by a standard 9V battery housed within the probe. When the probe enters a liquid the beam is refracted, activating a buzzer and two lights. As the infra-red beam is only 0.050 inches diameter, it allows measurement of any layer of non-conductive liquid greater than 0.050".

To detect water, a conductivity circuit is used. This is powered by a second 9V battery housed in the hub of the reel. The conductivity of water completes the circuit when the zero point enters water. This overrides the optical circuit, deactivating one of the lights and the buzzer.



Measurement

After grounding the instrument, the probe is lowered into a well or tank. Upon entering a liquid, a single light indicates water. A buzzer and two lights indicates product.

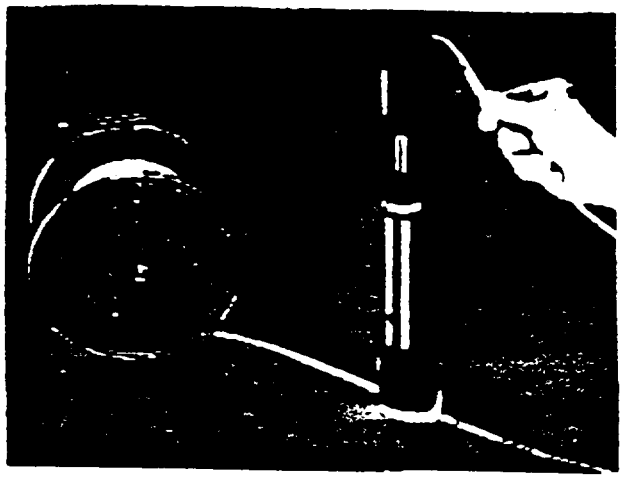
The depth to the air/product interface is determined from the permanently marked cable. Use of the tape guide/datum allows measurements to 1/100 ft., or better. The probe is lowered further and a reading is taken at the product/water interface. The thickness of the product layer is then determined by subtracting the first reading from the second.

The presence/absence of dense sinking layers is determined by continuing to lower the probe to the bottom of the well.

Tape Guide

A tape guide/datum is provided to ensure easy, repeatable measurements to 1/100 ft.

The tape guide also protects the cable from damage on rough edges of well casing. It guides the cable such that the probe is held away from the casing as it is lowered and raised in the well.



Tape

The easy-to-read, flat-tape cable is made with stranded stainless steel conductors embedded within polyethylene. The conductors provide strength and prevent cable stretch. The smooth surface of the tape is easy to clean and decontaminate.

Markings are permanently heat-embossed onto the cable, with the following options available:

M2	Each 1/50 ft. in black; feet in red.
M3	Each cm in black; metres in red.
M4	Both sides: any scale combination.

Probe

The probe is 1-1/2" diameter by 9" long. The measurement point for both optical and electronic sensors is located 1-1/4" from the lower end of the probe.

When the probe is switched "ON" and is in air, a warning light on the faceplate of the reel reminds the user to turn the probe off after use.

The sturdy probe is modular to simplify maintenance. The pressure proof design allows submersion for the detection of sinking hydrocarbons (DNAPLs).

Ordering Information

Interface Meter

Specify: Model 121*
Measurement Option
Length
Spare Tape Guides
Spare Probe Cleaners

*Supplied with a carrying bag, tape guide and probe cleaner.

Printed in Canada

MALCOLM PIRNIE

DETERMINATION OF STREAM FLOW

The flow rate of a stream can be calculated using the following formula:

$$Q = wdu$$

Where Q = discharge in cubic feet per second
w = width of the stream, in feet
d = average depth of the stream, in feet
u = velocity in feet per second

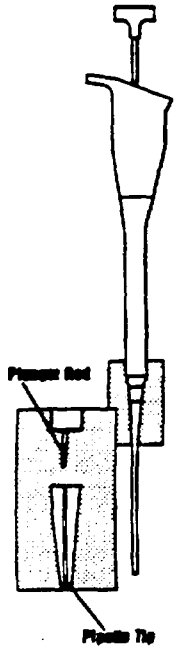
The stream velocity is measured by placing a floating object in the stream above the location where the stream cross-section will be measured. The time required for the object to float unobstructed from upstream to downstream of the stream cross-section measurement location is noted. This figure is then divided into the distance between the start and end points to obtain a stream volocity in units of length per unit of time.

To determine the cross-sectional area of the strea, sufficient measurements must be collected at regular intervals across the stream bed to permit accurate assessment of the stream depth. The depth will be measured by lowering a weighted line into the water. The collected depth measurements are then averaged for inclusion in the stream flow equation.

STANDARD OPERATING PROCEDURES FOR THE

ENSYS PCB RISc SOIL TEST KIT

HOW TO OPERATE THE MECHANICAL PIPETTE



To Set Or Adjust Volume

Turn lower part of push-button to adjust volume up or down. Meter should read "030".

To Assemble Pipette Tip

Slide larger mounting end of pipette tip onto end of pipette. Holding tip in place, press push-button until plunger rod enters pipette tip (see illustration).

To Withdraw Sample

With tip mounted in position on pipette, press push-button to first stop and hold it.

Place tip at bottom of liquid sample and slowly release push-button to withdraw measured sample.

To Dispense Sample

Place tip into dispensing vessel (immersing end of the tip if vessel contains liquid) and slowly press push-button to first stop. (Do not push to second stop or tip will eject).

Remove tip from vessel and release push-button.

To Eject Tip

Press push-button to second stop. Tip is ejected.

For additional information regarding operation and use of pipette, please refer to your pipette manual.

[redacted]

PCB RISC™ SOIL TEST SYSTEM

5 ppm

RAPID IMMUNOASSAY SCREEN

User's Guide

This method correctly identifies 98% of samples that are PCB-free and those containing 5 ppm of PCBs. A sample that develops less color than the standard is interpreted as positive. It contains PCBs. A sample that develops more color than the standard is interpreted as negative. It contains less than 5 ppm PCBs.

IMPORTANT NOTICE

The Test System performs accurately only when used as directed. This User's Guide is brief. Read it carefully prior to using the Test System. It will increase understanding of test objectives and help ensure a successful test.

System Description

Each PCB RISc Soil Test System contains enough material to perform four complete tests, each at 5 ppm.

The PCB RISc Soil Test is divided into four phases. The instructions and notes should be reviewed before proceeding with each phase.

Hotline Assistance

If you need assistance or are missing necessary Test System materials, call toll free: 1-800-262-RISC (7472).

Validation and Warranty Information

Product claims are based on validation studies carried out under controlled conditions. Data has been collected in accordance with valid statistical methods and the product has undergone quality control tests of each manufactured lot.

PCB-free soil and soil containing 5 ppm of PCBs were tested with the EnSys PCB RISc analytical method. The method correctly identified 95% of these samples. A sample that has developed less color than the standard is interpreted as positive. It contains PCBs. A sample that has developed more color than the standard is interpreted as negative. It contains less than 5 ppm PCBs.

The company does not guarantee that the results with the PCB RISc Soil Test System will always agree with instrument-based analytical laboratory methods. All analytical methods, both field and laboratory, need to be subject to the appropriate quality control procedures.

EnSys, Inc. warrants that this product conforms to the descriptions contained herein. No other warranties, whether expressed or implied, including warranties of merchantability and of fitness for a particular purpose shall apply to this product.

EnSys, Inc. neither assumes nor authorizes any representative or other person to assume for it any obligation or liability other than such as is expressly set forth herein.

Under no circumstances shall EnSys, Inc. be liable for incidental or consequential damages resulting from the use or handling of this product.

How It Works

Standards, Samples, and color-change reagents are added to test tubes, coated with a chemical specific to PCBs. The concentration of PCBs in an unknown Sample is determined by comparing its color intensity with that of a Standard.

Note: PCB concentration is inversely proportional to color intensity: the lighter the color development of the sample, the higher the concentration of PCBs.

Quality Control

Standard precautions for maintaining quality control:

- Do not use reagents or test tubes from one Test System with reagents or test tubes from another Test System.
- Do not use the Test System after any portion has passed its expiration date.
- Do not attempt the test using more than 3 antibody coated tubes (two of which are Standards) at the same time.
- Do not exceed incubation periods prescribed by the specific steps.
- Always dispense correct number of drops and wash the number of times indicated in this guide.
- Use EPA Method 8080 or Code of Federal Regulations Title 40, Part 136, Appendix A, Method 680 to confirm results.

Storage and Handling Precautions

- Wear protective gloves and eyewear.
- Store kit at room temperature and out of direct sunlight (less than 80°F).
- Keep aluminized pouch (containing unused antibody coated tubes) sealed when not in use.
- If Stop Solution or liquid from the extraction jar comes into contact with eyes, wash thoroughly with cold water and seek immediate medical attention.
- If Stop Solution or liquid from the extraction jar comes into contact with skin or clothing, wash thoroughly with cold water.
- Standard Solution contains PCBs. Test samples may contain PCBs. Handle with care.

PHASE ONE

EXTRACTION & PREPARATION OF THE SAMPLE

PHASE TWO

DILUTION & BUFFERING OF SAMPLE & STANDARDS

→ Following completion of Phase two steps, proceed directly with Phase Three

NOTES BEFORE PROCEEDING WITH PHASE ONE

- Items that you will need that are not provided in the test kit include: a permanent marking pen, laboratory tissue, a timer or stopwatch, liquid waste container, and disposable gloves.

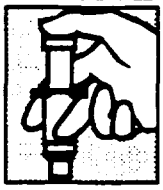
WEIGH SAMPLE

1 Place weigh boat on pan balance.
2 Press ON/MEMORY button on pan balance. Balance will beep and display 0.0.
3 Weigh out 10 +/- 0.1 grams of soil.
4 If balance turns off prior to completing weighing, use empty weigh boat to retare, then continue.

EXTRACT PCBs

5 Remove lid from extraction jar and transfer 10 grams of soil from weigh boat into extraction jar.
6 Recap extraction jar tightly and shake vigorously for one minute.
7 Allow to settle for one minute.

FILTER SAMPLE

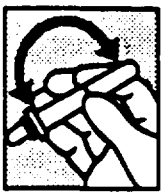


8 Remove lid from extraction jar.
9 Disassemble filtration plunger from filtration barrel.
10 Insert bulb pipette into top (liquid) layer in the extraction jar and draw up sample. Transfer at least ½ bulb capacity into filtration barrel. Do not use more than one full bulb.
11 Press plunger firmly into barrel until at least ½ mL of filtered sample is available (place on table and press if necessary).

Sample is now ready to be tested with the immunoassay.

PREPARE ENZYME DROPPER

12 Crush glass ampule contained within enzyme dropper by pressing tube against hard edge.



13 Mix enzyme by turning dropper end-over-end 5 times. Do not shake.
14 Remove seal from enzyme dropper.

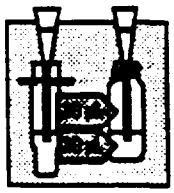
NOTES BEFORE PROCEEDING WITH PHASE TWO

- Using a permanent marking pen (not included), write **Standard 1** near the top of one blue buffer tube and one antibody coated tube. Then, write **Standard 2** near the top of one blue buffer tube and one antibody coated tube. Place the **Standard** tubes in the workstation.

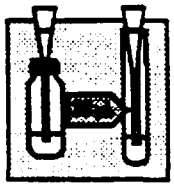
- **For each sample to be tested:**
  Place one 8 ppm dilution vial in the workstation.
  Write **8 ppm** near the top of one blue buffer tube and one antibody coated tube.

- Following instructions on reverse of insert, assemble new tip onto mechanical pipette.

DILUTE AND BUFFER SAMPLE

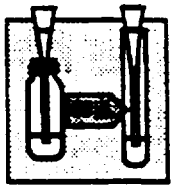


15 Remove cap from 8 ppm dilution vial.
16 Withdraw 30 µL of filtered sample using mechanical pipette and dispense below the liquid level in 8 ppm dilution vial. Then, withdraw another 30 µL of filtered sample and dispense below the liquid level into the same 8 ppm dilution vial for a total of 60 µL; replace cap and gently shake vial for 5 seconds.



17 Remove cap from 8 ppm blue buffer tube.
18 Withdraw 30 µL of diluted sample from 8 ppm dilution vial and dispense below the liquid level in 8 ppm blue buffer tube. Do not recap blue buffer tube.
19 Gently shake 8 ppm blue buffer tube for 5 seconds.
20 Discard mechanical pipette tip.

BUFFER STANDARDS



21 Assemble new tip onto mechanical pipette.
22 Remove tops from PCB Standard vial and two blue buffer tubes marked **Standard 1** and **Standard 2**.
23 Withdraw 30 µL of PCB Standard and dispense below the liquid level in **Standard 1** blue buffer tube.
24 Wipe pipette tip with laboratory tissue.
25 Withdraw 30 µL of PCB standard and dispense below the liquid level in **Standard 2** blue buffer tube.
26 Immediately replace cap on PCB Standard vial.
27 Discard mechanical pipette tip.
28 Gently shake **Standard 1** and **Standard 2** blue buffer tubes for 5 seconds.

PHASE THREE

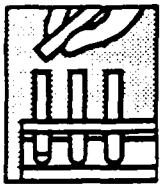
THE IMMUNOASSAY

Overview: The timing used in performing Phase Three steps is critical to obtaining accurate test results.

NOTES BEFORE PROCEEDING WITH PHASE THREE

- This phase of the procedure requires critical timing and care in handling the antibody coated tubes.
- Instructions to gently shake any of the vials mean to gently but thoroughly mix the contents with special care not to spill or splash.
- All washing must be done thoroughly and with force to remove all unbound material. The wash solution is a harmless, dilute solution of detergent. Do not hesitate to wash vigorously even if the solution contacts gloved hands.

INCUBATION I



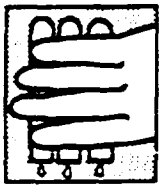
29 Start timing and immediately pour solution from each **Standard** blue buffer tube (1 and 2) into appropriate **Standard** antibody coated tube.

30 Pour solution from 8 ppm blue buffer tube into 8 ppm antibody coated tube.

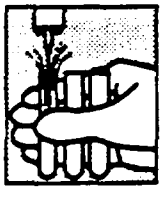
31 When pouring is complete, gently shake all 3 tubes for 5 seconds.

32 Let tubes stand exactly 10 minutes.

WASHING I



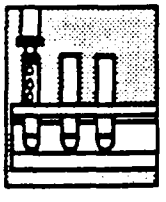
33 After the 10 minute incubation, discard solution from each antibody coated tube into liquid waste container.



34 Keeping nozzle of wash solution bottle just above top of antibody coated tube, forcefully squeeze wash solution into tube with a strong, vigorous stream to fill each tube. Empty all 3 washed tubes into liquid waste container. Repeat wash 3 times (total of 4 washes).

35 After final wash, tap antibody coated tubes upside down on a laboratory tissue.

INCUBATION II

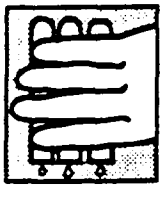


36 Remove cap and dispense first drop from enzyme dropper into liquid waste container.

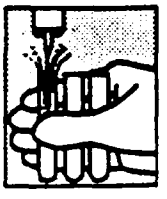
37 Start timing and immediately dispense 4 drops into each antibody coated tube (**Standards** and **Sample**) by squeezing the dropper. When complete, gently shake antibody coated tubes for 5 seconds.

38 Let tubes stand exactly 5 minutes.

WASHING II



39 After the 5 minute incubation, discard solution from each antibody coated tube into liquid waste container.



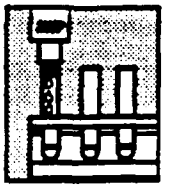
40 Keeping nozzle of wash solution bottle just above top of antibody coated tube, forcefully squeeze wash solution into each tube with a strong, vigorous stream to fill each tube. Empty all 3 washed tubes into liquid waste container. Repeat wash 3 times (total of 4 washes).

41 After final wash, tap antibody coated tubes upside down on a laboratory tissue.

PHASE THREE (CONT.)

COLOR DEVELOPMENT

COLOR DEVELOPMENT



42 Remove top from Substrate A (yellow cap).

Note: Keep Substrate dropper bottles vertical and direct each drop at bottom of antibody coated tubes. Addition of more or less than indicated number of drops (of Substrate A or B) may give inaccurate results.

43 Add 5 drops of Substrate A to each antibody coated tube.

44 Remove top from Substrate B (green cap).

45 Start timing and immediately add 5 drops of Substrate B to each antibody coated tube.

46 Shake all 3 tubes for 3-5 seconds, and let stand for exactly 2 ½ minutes. Solution will turn blue in some or all antibody coated tubes.

47 Stop reaction at end of 2 ½ minutes by adding 5 drops of Stop Solution (red cap).

Note: Blue solution will turn yellow when Stop Solution is added.

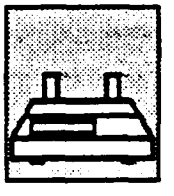
PHASE FOUR

INTERPRETING TEST RESULTS

NOTES BEFORE PROCEEDING WITH PHASE FOUR

- In this step, the standards are evaluated first in order to identify which is darker. To be conservative, the sample will be measured against the darker of these two standards.

SELECT STANDARD



48 Wipe outside of **Standard 1** and **Standard 2** antibody coated tubes with laboratory tissue.

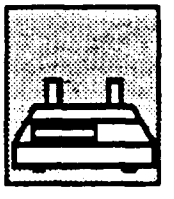
49 Place both **Standard** tubes in photometer.

50 If photometer readout is negative or zero, the tube in the left well is the darker standard. Remove tube from right well and discard it.

However

If photometer reading is positive, the tube in the right well is the darker standard. Remove tube from left well, discard it, and move tube from right well to left well.

MEASURE SAMPLE



51 Wipe outside of 8 ppm antibody coated tube with laboratory tissue.

52 Place 8 ppm tube in right well of photometer and record reading shown on display.

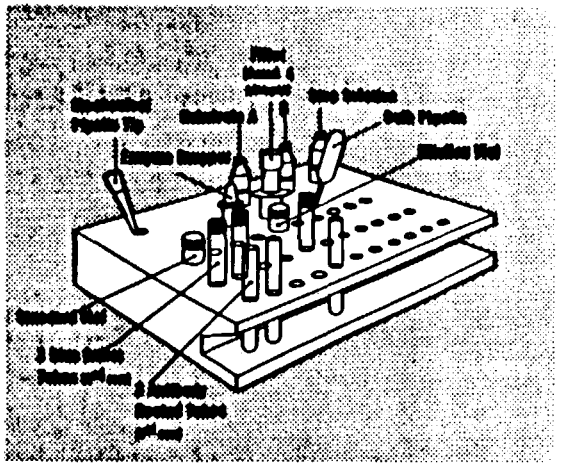
If photometer reading is negative or zero, PCBs are present.

If photometer reading is positive, concentration of PCBs is less than 8 ppm.

WORKSTATION SET-UP

Assemble the following components in the workstation:

- ☐ 3 antibody-coated tubes
- ☐ PNS standard vial
- ☐ Enzyme dropper
- ☐ Bulb pipette
- ☐ Substrate A
- ☐ Stop solution
- ☐ 3 blue buffer tubes
- ☐ 3 green dilution vial
- ☐ Filtration barrel & plunger
- ☐ 2 recalcified plastic tips
- ☐ Substrate B

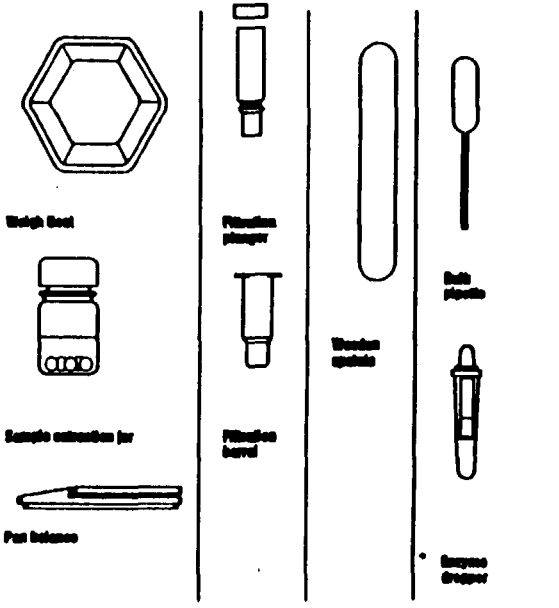


PHASE ONE

COMPONENTS FOR EXTRACTION & SAMPLE PREPARATION

Assemble the following components:

- ☐ Weigh boat
- ☐ Pan balance
- ☐ Wooden spatula
- ☐ Sample extraction jar
- ☐ Filtration barrel
- ☐ Filtration plunger
- ☐ Bulb pipette
- ☐ Enzyme dropper



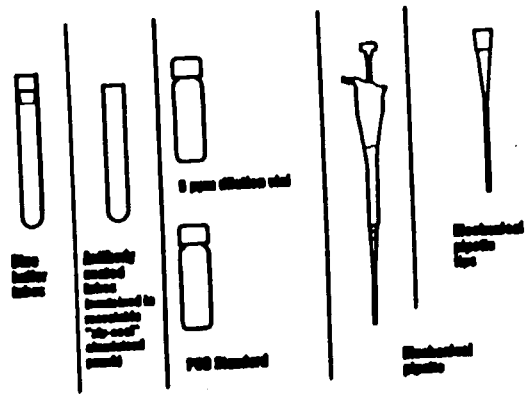
PHASE TWO
COMPONENTS FOR DILUTION OF SAMPLE & STANDARDS

Use the following component from Phase One:

- Filtered sample

Use the following additional components:

- Permanent marking pen (not included in kit)
- 3 Blue buffer tubes
- 3 Antibody coated tubes
- Dilution vial marked "S"
- P48 standard vial marked "run control"
- Disclosed pipette
- 2 unclosed pipette tips



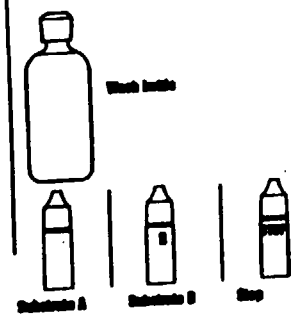
PHASE THREE
COMPONENTS FOR IMMUNOASSAY & COLOR DEVELOPMENT

Use the following components from earlier phases:

- Sample
- Blue buffer tube marked "S"
- Blue buffer tube marked "Standard 1"
- Blue buffer tube marked "Standard 2"
- Antibody coated tube marked "S"
- Antibody coated tube marked "Standard 1"
- Antibody coated tube marked "Standard 2"
- Pasteur dropper
- Laboratory timer (not included)

Use the following additional components:

- Wash bottle
- Liquid waste container (not included)
- Stop watch or timer
- Substrate A (yellow cap)
- Substrate B (green cap)
- Stop Solution (red cap)



PHASE FOUR
COMPONENTS FOR INTERPRETING TEST RESULTS

Use the following components from earlier phases:

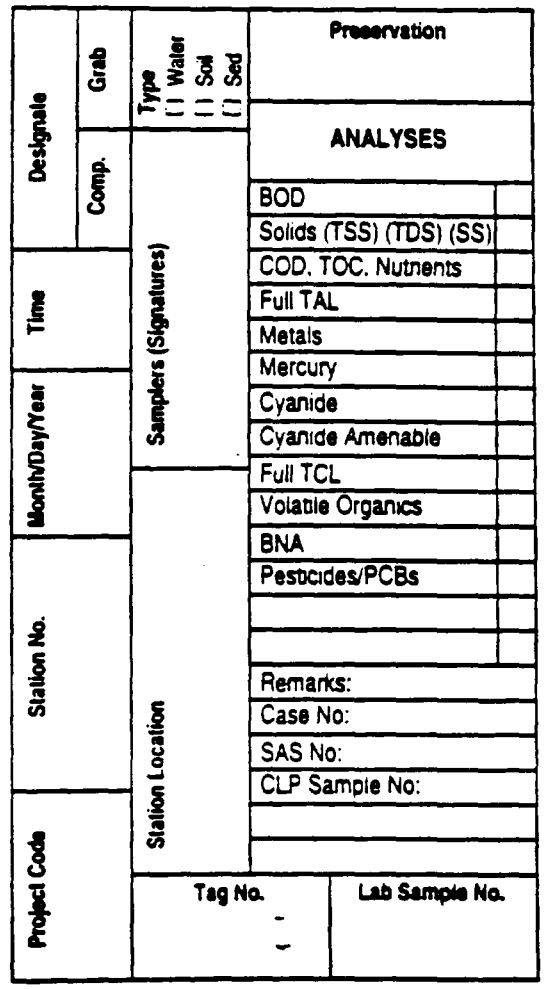
- Sample
- Antibody coated tube marked "S"
- Antibody coated tube marked "Standard 1"
- Antibody coated tube marked "Standard 2"
- Laboratory timer (not included)

Use the following additional component:

- Photometer

APPENDIX B

Sample Custody and Documentation



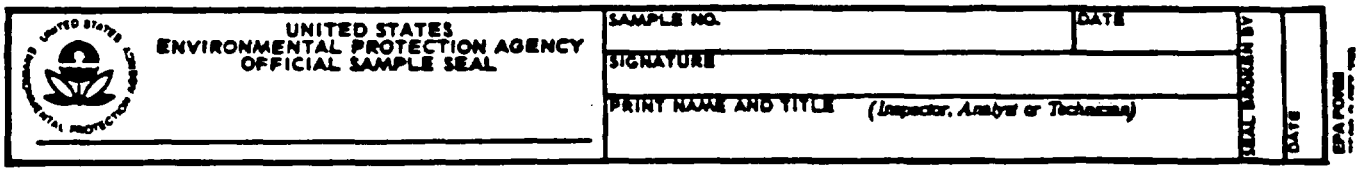
CHM 001 0352

MALCOLM PIRNIE

SAMPLE ANALYSIS TAG

MALCOLM P

FIGURE B-1



CHM 001 0353

MALCOLM PIRNIE

SAMPLE CUSTODY SEAL

MALCOLM P INC

FIGURE B-2

159361

EPA

United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Organic Traffic Report

(For Organic CLP Analysis)

SAS No. (if applicable)

Case No.

1. Project Code | Account Code

2. Region No. | Sampling Co.

4. Date Shipped | Carrier

6. Preservative (Enter in Column D)
1. HCl
2. HNO_3
3. Na_2SO_4
4. H_2SO_4
5. Other (SAS) (Specify)
6. Ice only
N. Not preserved

7. Sample Description (Enter in Column A)
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) (Specify)

Triple volume required for matrix spike/matrix spike duplicate analysis sample.
Ship medium and high concentration samples in paint cans.
See reverse for additional standard instructions.
Please indicate sample to spike/duplicate.

Regional Information

Sampler (Name)

Airbill Number

Non-Superfund Program

Sampler Signature

5. Ship To

Site Name

3. Type of Activity
ENF ☐ PA ☐ O&M ☐
ER ☐ RA ☐ RIFS ☐
LSI ☐ RD ☐ SSI ☐
NPLD ☐ STSI ☐ ST ☐
Other ☐ STPA ☐

City, State | Site Spill ID

CLP Sample Numbers (from labels)	A Enter # from Box 7	B Conc. Low Med High	C Sample Type Comp / Grab	D Preservative from Box 6	E RAS Analysis VOA	BNA	Pest/ PCB	High ARO/ TOX	F Regional Specific Tracking Number or Tag Numbers	G Station Location Number	H Mo/Day/ Year/Time Sample Collection	I Sampler Initials	J Corresp. CLP Inorg. Samp. No.

Shipment for Case complete? (Y/N)

TR

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Received by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks Is custody seal intact? Y/N/none	

Split Samples ☐ Accepted (Signature)
☐ Declined

EPA Form 9110-2 (Rev. 10-90) Replaces EPA Form (2075-7), previous edition which may be used

DISTRIBUTION:
Blue - Region Copy Pink - SMO Copy White - Lab Copy for Return to SMO Yellow - Lab Copy

00667

CHM 001 0354

MALCOLM PIRNIE

ORGANIC ANALYSIS TRAFFIC REPORT

MALCOLM INC

FIGURE B-3

159381

EPA United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Inorganic Traffic Report
(For Inorganic CLP Analysis)

SAS No. (if applicable) | Case No.

1. Project Code	Account Code	2. Region No.	Sampling Co.	4. Date Shipped	Carrier
Regional Information		Sampler (Name)		Airbill Number	
Non-Superfund Program		Sampler Signature		5. Ship To	
Site Name		3. Type of Activity: ENF ☐ ER ☐ LSI ☐ NPLD ☐ PA ☐ RA ☐ RD ☐ STSI ☐ O&M ☐ RIFS ☐ SSI ☐ ST ☐ STPA ☐ Other ☐			
City, State	Site Spill ID				

6. Preservative (Enter in Column D)
1. HNO_3
2. NaOH
3. HCl
4. H_2SO_4
5. Ice only
6. Other (SAS) (Specify)
N. Not preserved

7. Sample Description (Enter in Column A)
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) (Specify)

Double volume required for spike/duplicate analysis sample.

Ship medium and high concentration samples in paint cans.

See reverse for additional standard instructions.

For total or dissolved metals, check only one RAS analysis per each sample.

CLP Sample Numbers (from labels)	A Enter # from Box 7	B Conc. Low Med High	C Sample Type Comp / Grab	D Preservative from Box 6	E - RAS Analysis: Metals Total	Metals Dissolved	Cyanide	Low Conc. Nitrate/Nitrite	Low Conc. Fluoride	High pH	High Conductivity	F Regional Specific Tracking Number or Tag Numbers	G Station Location Number	H Mo/Day/Year/Time Sample Collection	I Sampler Initials	J Corresp. CLP Org. Samp. No.

Shipment for Case complete? (Y/N)

TR

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Received by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks Is custody seal intact? Y/N/none	

EPA Form 9110-1 (Rev. 11-90) Replaces EPA Form (2075-6), previous edition which may be used

DISTRIBUTION:
Green - Region Copy Pink - SMO Copy White - Lab Copy for Return to SMO Yellow - Lab Copy

Split Samples ☐ Accepted (Signature)
☐ Declined

011543

CHM 001 0355

MALCOLM PIRNIE

INORGANIC ANALYSIS TRAFFIC REPORT

MALCOLM PIRNIE, INC

FIGURE B-4

1893B1

EPA United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria VA 22313
703 557-2490 FTS 557-2490

Special Analytical Service

Packing List/Chain of Custody

SAS No.

1. Project Code	Account Code	2. Region No.	Sampling Co.	4. Date Shipped	Carrier

Regional Information	Sampler (Name)	Airbill Number
Non-Superfund Program	Sampler Signature	5. Ship To
Site Name	3. Type of Activity (Removal / Remedial)	
	Lead / Pre-Remedial: SF, PRP, ST, FED; PA, SSI, LSI; RIFS, RD, RA, O&M, NPLD; Removal: CLEM, REMA, REM, OIL, UST	
City, State / Site Spill ID		

6. Sample Description (Enter in Column A)
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil
7. Waste
8. Other (Specify)

7. Preservative (Enter in Column C)
1. HCl
2. HNO_3
3. $NAHSO_4$
4. H_2SO_4
5. NaOH
6. Other (SAS) (Specify)
7. Ice only
N. Not preserved

Sample Numbers	A Matrix Enter from Box 6	B Conc Low Med High	C Preserv-ative Used from Box 7	D Analysis	E Sample used for spike and/or duplicate	F Regional Specific Tracking Number or Tag Number	G Station Location Identifier	H Mo/Day/ Year/Time Sample Collection	I Sampler Initials	J Designated Field QC
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										

Shipment for SAS complete? (Y/N)

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by (Signature)
Received by (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks Is custody seal intact? Y/N/none	

Split Samples ☐ Accepted (Signature)
☐ Declined

EPA Form

DISTRIBUTION
White - Region Copy Yellow - SMO Copy Pink - Lab Copy for Return to SMO Gold - Lab Copy

S 004108

CHM 001 0356

MALCOLM PIRNIE

SPECIAL ANALYTICAL SERVICES
TRAFFIC REPORT

MALCOLM PIRNIE, INC.

FIGURE B-5

159361

APPENDIX C

SEE CDM FEDERAL ARCS II

QUALITY ASSURANCE MANAGEMENT PLAN

REVISION 3, DATED JULY 1991

FOR FIELD AUDIT FORMS

APPENDIX C

Field Audit Forms

QUALITY CONTROL FIELD AUDIT REPORT

SUMMARY INFORMATION

1. Project Name: _____

2. Project Address: _____

_____

3. Project Status:

CERCLA- ☐

ACO/Enforcement ☐ ECRA ☐ RCRA ☐

Other: _____

4. Project Phase:

Preliminary Assessment ☐ RI/FS ☐ RD ☐ Construction ☐ O&M ☐

Other: _____

5. Dates of QC Field Audit: _____

6. Auditor's Name: _____ Phone: _____

7. Facility Contact: _____ Phone: _____

8. Contractor Contact: _____ Phone: _____

9. Personnel On-site:

Name	Representing	Phone

10. Work Plan Submitted & Reviewed: Yes ☐ No ☐ Status: _____

Outstanding Problems with Work Plan: _____

11. Weather Conditions:

Sunny ☐ Partly Sunny ☐ Partly Cloudy ☐ Cloudy ☐ Rain ☐ Drizzle ☐ Snow ☐ Sleet ☐

Temperature: _____ Wind Speed: _____ Wind Direction: _____

12. Aqueous Matrices Sampled:

Potable well ☐ Groundwater ☐ Surface Water ☐ Leachate Runoff ☐ Storm Sewer ☐ Sanitary Sewer ☐

Other: _____

13. Non-Aqueous Matrices Sampled:

Soil ☐ Sediment ☐ Sludge ☐ Chemical Solids ☐ Waste Pile ☐

Other: _____

14. Level of Personnel Protection Required in Work Plan: Level of Personnel Protection Actually Donned:

A ☐ B ☐ C ☐ D ☐ A ☐ B ☐ C ☐ D ☐

15. Field Survey Equipment:

Instrument	Model	Calibration Check	Calibration Standard	Span Setting
Conductivity Meter				
Dissolved Oxygen Meter				
pH Meter				
Combustible Gas Indicator				
Flame Ionization Detector				
Photoionization Detector				
Toxic Gas Indicator (CO, H_2S)				
Other:				

Observations: _____

16. Did the sampling team take periodic surveys of the ambient air conditions: Yes ☐ No ☐ N/A ☐

17. Did the sampling team provide a decon zone designating a clean and contaminated area: Yes ☐ No ☐ N/A ☐

18. Were photographs taken: Yes ☐ No ☐ Total number taken: _____

CHM 001 0360

AQUEOUS SAMPLE INFORMATION

EVACUATION PROCEDURES

1. Well Casing Construction: Stainless Steel ☐ Teflon ☐ PVC ☐ Other: _____

2. Diameter of Well Casing: 2" ☐ 4" ☐ 6" ☐ Other: _____

3. Locking Caps on the Wells: Yes ☐ No ☐ N/A ☐ Protective Casing: Yes ☐ No ☐ N/A ☐

4. Method utilized to determine the static water level: Water Level Indicator ☐ Other: _____

5. Reference point that the static water level was measured from:

   Survey Point ☐ Top of Inner Casing ☐ Top of Protective Casing ☐

6. Was the water level indicator decontaminated according to standard NJDEP/DHSM procedures between each well:

   Yes ☐ No ☐ N/A ☐

   If No, method utilized: _____

7. Evacuation Method:

   Bailer ☐ Centrifugal Pump ☐ Peristaltic Pump ☐ Bladder Pump ☐ Submersible Pump ☐

   Gas Displacement Pump ☐ Gas Lift Pump ☐ Other: _____

8. Type of Hose Utilized:

   Polyethylene (ASTM Drinking Water Grade 2239) ☐ Teflon ☐ Silastic ☐ N/A ☐

   Other: _____

9. Was the hose equipped with a foot check valve: Yes ☐ No ☐ N/A ☐

10. Was the hose dedicated to each well location: Yes ☐ No ☐ N/A ☐

    If No, method of decontamination: _____

11. Was the pump dedicated to each well location: Yes ☐ No ☐ N/A ☐

12. Was the pump: Laboratory Decontaminated ☐ Field Decontaminated ☐ N/A ☐

13. Was the pump decontaminated according to standard NJDEP/DHSM procedures: Yes ☐ No ☐

    If No, method of decontamination: _____

14. Was the pump head or end of hose within 6 feet of the dynamic water level during evacuation: Yes ☐ No ☐ N/A ☐

15. Was the decontamination area located away from the source of contamination: Yes ☐ No ☐ N/A ☐

16. Power Source for the Pump:

    Gasoline Powered Generator ☐ Gasoline Powered Compressor ☐ Diesel Air Compressor ☐

    Other: _____

17. Was the gasoline powered generator/compressor set up downwind of the sampling site: Yes ☐ No ☐ N/A ☐

18. Was the gasoline transported in the same vehicle as the sample bottles and sampling equipment: Yes ☐ No ☐ N/A ☐

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PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: _____

LAB: _____

SITE: _____

	YES	NO	N/A
1.0 Data Completeness and Deliverables			
1.1 Have any missing deliverables been received and added to the data package.	[___]	___	___
ACTION: Call lab for explanation / resubmittal of any missing deliverables. If lab cannot provide them, note the effect on review of the package under the "Contract Problems/Non-compliance" section of reviewer narrative.			
1.2 Was SMO CCS checklist included with package?	[___]	___	___
2.0 Cover Letter/Case Narrative			
2.1 Is the Narrative or Cover Letter present?	[___]	___	___
2.2 Are Case Number and/or SAS number contained in the Narrative or Cover Letter?	[___]	___	___
3.0 Data Validation Checklist			
The following checklist is divided into three parts. Part A is filled out if the data package contains any VOA analyses, Part B for any BNA analyses and Part C for Pesticide/PCBs.			
Does this package contain:			
VOA data?		___	___
BNA data?		___	___
Pesticide/PCB data?		___	___
ACTION: Complete corresponding parts of checklist.			

PART A: VOA ANALYSES

	YES	NO	N/A
1.0 Traffic Reports and Laboratory Narrative			
1.1 Are the Traffic Report Forms present for all samples?	[___]	___	___
ACTION: If no, contact lab for replacement of missing or illegible copies.			
1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?	___	[___]	___
ACTION: Use professional judgement to evaluate the effect on the quality of the data.			
ACTION: If any sample analyzed as a soil contains more than 50% water, all data should be rejected.			
ACTION: If both VOA vials for a sample have air bubbles, flag all positive results "J" and all non-detects "R".			
2.0 Holding Times			
2.1 Have any VOA holding times, determined from date of collection to date of analysis, been exceeded?	___	[___]	___

If unpreserved, aqueous aromatic volatiles must be analyzed within 7 days of collection and non-aromatic volatiles must be analyzed within 14 days. If preserved with hydrochloric acid and stored at 4°C, then both aromatic and non-aromatic volatiles must be analyzed within 14 days. If uncertain about preservation, contact the sampler to determine whether the samples were preserved.

A ten-day holding time for soil samples is recommended.

Table of Holding Time Violations

			(See Traffic Report) Date Sampled	Date Lab Received	Date Analyzed
Sample	Sample Matrix	Preserved ?			
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

ACTION: If holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

	YES	NO	N/A

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon reanalysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. The reviewer may determine that non-detect data are unusable ("R").

3.0 Surrogate Recovery (Form II)

3.1 Are the VOA Surrogate Recovery Summaries (Form II) present for each of the following matrices:

a. Low Water	[___]	___	___
b. Med Water	[___]	___	___
c. Low Soil	[___]	___	___
d. Med Soil	[___]	___	___

3.2 Are all the VOA samples listed on the appropriate Surrogate Recovery Summaries for each of the following matrices:

a. Low Water	[___]	___	___
b. Med Water	[___]	___	___
c. Low Soil	[___]	___	___
d. Med Soil	[___]	___	___

ACTION: Call lab for explanation / resubmittals. If missing deliverables are unavailable, document effect on data under "Conclusions" section of reviewer narrative.

3.3 Were outliers marked correctly with an asterisk?	[___]	___	___

ACTION: Circle all outliers in red.

3.4 Was one or more VOA surrogate recovery outside of contract specifications for any sample or method blank?	___	[___]	___
If yes, were samples reanalyzed?	[___]	___	___
Were method blanks reanalyzed?	[___]	___	

ACTION: If surrogate recoveries are > 10% but all do not meet SOW specifications:

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ").

	YES	NO	N/A
If any surrogate has a recovery of <10% : 1. Flag all positive results as estimated ("J"). 2. Flag all non-detects as unusable ("R"). Professional judgement should be used to qualify data that have method blank surrogate recoveries . out of specification in both original and re-analyses. Check the internal standard areas.			
3.5 Are there any transcription/calculation errors between raw data and Form II?	___	[__]	__
ACTION: If large errors exist, call lab for explanation / resubmittal, make any necessary corrections and note errors under "Conclusions".			
4.0 Matrix Spikes (Form III)			
4.1 Is the Matrix Spike Duplicate/Recovery Form (Form III) present?	[__]	__	__
4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:			
a. Low Water	[__]	__	__
b. Med Water	[__]	__	__
c. Low Soil	[__]	__	
d. Med Soil	[__]	__	__
ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.			

4.3 How many VOA spike recoveries are outside QC limits?

Water	Soils
_____ out of 10	_____ out of 10

4.4 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water	Soils
_____ out of 5	_____ out of 5

ACTION: If MS and MSD both have less than 10% recovery for an analyte, negative results for that analyte should be rejected, and positive results should be flagged "J". The above applies only to the sample used for the MS/MSD analysis. Use professional judgement in applying this criterion to other

	YES	NO	N/A
5.0 Blanks (Form IV)			
5.1 Is the Method Blank Summary (Form IV) present?	[___]	___	___
5.2 Frequency of Analysis: for the analysis of VOA TCL compounds, has a reagent/method blank been analyzed for each set of samples or every 20 samples of similar matrix (low water, med water, low soil, medium soil), whichever is more frequent?	[___]	___	___
5.3 Has a VOA instrument blank been analyzed at least once every twelve hours for each GC/MS system used?	[___]	___	___
ACTION: If any method blank data are missing, call lab for explanation / resubmittal. If not available, reject all associated positive data ("R").			
5.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.			
Is the chromatographic performance (baseline stability) for each instrument acceptable for VOAs?	[___]	___	___
ACTION: Use professional judgement to determine the effect on the data.			
6.0 Contamination			
NOTE: "Water blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.			
6.1 Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for VOAs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample Dilution Factor.	___	[___]	___
6.2 Do any field/trip/rinse blanks have positive VOA results (TCL and/or TIC)?	___	[___]	___
ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)			
NOTE: Only field/rinse blanks taken the same day as the samples are used to qualify data. Trip blanks are used to qualify only those samples with which they were shipped. Blanks may not be qualified because of contamination in another blank. Blanks may be qualified for surrogate, spectral, tuning or calibration QC problems.			

YES NO N/A

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

	Sample conc > CRQL but < 10x blank	Sample conc < CRQL & is < 10x blank value	Sample conc > CRQL value & >10x blank value
Methylene chloride Acetone Toluene 2-butanone	Flag sample result with a 'U'; cross out 'B' flag	Reject sample result and report CRQL; cross out 'B' flag	No qualification is needed

	Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL value & > 5 blank value
Other Contaminants	Flag sample result with a 'U'; cross out 'B' flag	Reject sample result and report CRQL; cross out 'B' flag	No qualification is needed

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R" (unusable).

6.3 Are there field/rinse/equipment blanks associated with every sample? [___] ___ ___

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 GC/MS Tuning and Mass Calibration (Form V)

7.1 Are the GC/MS Tuning and Mass Calibration Forms (Form V) present for Bromofluorobenzene (BFB)? [___] ___ ___

7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift? [___] ___ ___

7.3 Has a tuning performance compound been analyzed for every twelve hours of sample analysis per instrument? [___] ___ ___

ACTION: If any tuning data are missing, take action specified in 3.2 above.

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS

YES NO N/A

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

7.4 Have the ion abundance criteria been met for each instrument used? [___] ___ ___

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If tuning calibration is in error, flag all associated sample data as unusable ("R"). However, if expanded ion criteria are met (See 1988 Functional Guidelines), the data reviewer may accept data with appropriate qualifiers.

7.5 Are there any transcription / calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) ___ [___] ___

7.6 Have the appropriate number of significant figures (two) been reported? (Check at least two values, but if errors are found check more values.) ___ [___] ___

ACTION: If large errors exist, call lab for explanation / resubmittal, make necessary corrections and note errors under "Conclusions".

7.7 Are the spectra of the mass calibration compound acceptable? [___] ___ ___

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

8.0 Target Compound List (TCL) Analytes

8.1 Are the Organic Analysis Data Sheets (Form I VOA) present with required header information on each page, for each of the following:

a. Samples and/or fractions as appropriate [___] ___ ___

b. Matrix spikes and matrix spike duplicates [___] ___ ___

c. Blanks [___] ___ ___

	YES	NO	N/A
8.2 Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			
a. Samples and/or fractions as appropriate	[___]	___	___
b. Matrix spikes and matrix spike duplicates (Mass spectra not required)	[___]	___	___
c. Blanks	[___]	___	___
ACTION: If any data are missing, take action specified in 3.2 above.			
8.3 Are the response factors shown in the Quant Report?	[___]	___	___
8.4 Is chromatographic performance acceptable with respect to:			
Baseline stability	[___]	___	___
Resolution	[___]	___	___
Peak shape	[___]	___	___
Full-scale graph (attenuation)	[___]	___	___
Other:_____	[___]	___	___
ACTION: Use professional judgement to determine the acceptability of the data.			
8.5 Are the lab-generated standard mass spectra of the identified VOA compounds present for each sample?	[___]	___	___
ACTION: If any mass spectra are missing, take action specified in 3.2 above. If Lab does not generate their own standard spectra, make note in "Contract Problems/Non-compliance".			
8.6 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?	[___]	___	___
8.7 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	[___]	___	___
8.8 Do sample and standard relative ion intensities agree within 20%?	[___]	___	___
ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected, flagged "N" (presumptive evidence of the presence of the compound) or changed to not detected (at the calculated detection limit).			

	YES	NO	N/A
9.0 Tentatively Identified Compounds (TIC)			
9.1 Are all Tentatively Identified Compound Forms (Form I, Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "J" qualifier?	[___]	___	___
9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:			
a. Samples and/or fractions as appropriate	[___]	___	___
b. Blanks	[___]	___	___
ACTION: If any TIC data are missing, take action specified in 3.2 above.			
ACTION: Add "J" qualifier if missing and "N" qualifier to all identified TIC compounds on Form I, Part B.			
9.3 Are any TCL compounds (from any fraction) listed as TIC compounds (example: 1,2-dimethylbenzene is xylene—a VOA TCL—and should not be reported as a TIC)?	___	[___]	___
ACTION: Flag with "R" any TCL compound listed as a TIC.			
9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	[___]	___	___
9.5 Do TIC and "best match" standard relative ion intensities agree within 20%?	[___]	___	___
ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change identification to "unknown" or to some less specific identification (example: "Cl substituted benzene") as appropriate.			
10.0 Compound Quantitation and Reported Detection Limits			
10.1 Are there any transcription / calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found?	___	[___]	___
10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture?	___	[___]	___

	YES	NO	N/A

ACTION: If errors are large, call lab for explanation / resubmittal, make any necessary corrections and note errors under "Conclusions".

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

11.0 Standards Data (GC/MS)

11.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant. Reports) present for initial and continuing calibration? [___] ___ ___

ACTION: If any calibration standard data are missing, take action specified in 3.2 above.

12.0 GC/MS Initial Calibration (Form VI)

12.1 Are the Initial Calibration Forms (Form VI) present and complete for the volatile fraction? [___] ___ ___

ACTION: If any calibration standard forms are missing, take action specified in 3.2 above.

12.2 Are response factors stable for volatiles over the concentration range of the calibration (RSD <30%)? [___] ___ ___

ACTION: Circle all outliers in red.

ACTION: When RSD >30%, non-detects may be qualified using professional judgement. Flag all positive results "J". When RSD >90%, flag all non-detects as unusable ("R"). (Region II policy.)

12.3 Do any compounds have a RRF < 0.05? ___ [___] ___

ACTION: Circle all outliers in red.

ACTION: If any volatile compound has an average RRF < 0.05, flag positive results for that compound as estimated ("J"), and flag non-detects for that compound as unusable ("R").

	YES	NO	N/A
12.4 Are there any transcription / calculation errors in the reporting of average response factors (RRF) or %RSD? (Check at least two values but if errors are found, check more.)	___	[___]	___

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation / resubmittal, make any necessary corrections and note errors under "Conclusions".

13.0 GC/MS Continuing Calibration (Form VII)

13.1 Are the Continuing Calibration Forms (Form VII) present and complete for the volatile fraction?	[___]	___	___
13.2 Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	[___]	___	___

ACTION: List below all sample analyses that were not within twelve hours of the previous continuing calibration analysis.

_____

_____

_____

ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, call lab for explanation / resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").

13.3 Do any continuing calibration standard compounds have a RRF < 0.05?	___	[___]	___

ACTION: Circle all outliers in red.

ACTION: If any volatile compound has a RRF < 0.05, flag positive results for that compound as estimated ("J"), and flag non-detects for that compound as unusable ("R").

13.4 Do any compounds have a % difference between initial and continuing calibration RRF > 25%?	___	[___]	-

ACTION: Circle all outliers in red and qualify associated sample data as outlined in the table below:

YES NO N/A

% DIFFERENCE

25-50	50-90	>90
'J' positive results, no action for non detects	'J' positive results, 'UJ' non detects	'J' positive results, "R" non detects

13.5 Are there any transcription / calculation errors in the reporting of average response factors (RRF) or difference (%D) between initial and continuing RRFs? (Check at least two values but if errors are found, check more.) ___ [___] ___

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation / resubmittal, make any necessary corrections and note errors under "Conclusions".

14.0 Internal Standards (Form VIII)

14.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits for each continuing calibration? [___] ___ ___

ACTION: List all the outliers below.

Sample #	Internal Std	Area	Lower Limit	Upper Limit
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

(Attach additional sheets if necessary.)

ACTION: If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard. If extremely low area counts are reported, or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable ("R").

14.2 Are the retention times of the internal standards within 30 seconds of the associated calibration standard? [___] ___

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

	YES	NO	N/A
15.0 Field Duplicates			
15.1 Were any field duplicates submitted for VOA analysis?	[___]	___	___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

PART B: BNA ANALYSES

	YES	NO	N/A
1.0 Traffic Reports and Laboratory Narrative			
1.1 Are the Traffic Report Forms present for all samples?	[___]	___	___
ACTION: If no, contact lab for replacement of missing or illegible copies.			
1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?	___	[___]	___
ACTION: Use professional judgement to evaluate the effect on the quality of the data.			
ACTION: If any sample analyzed as a soil contains more than 50% water, all data should be rejected.			
2.0 Holding Times			
2.1 Have any BNA holding times, determined from date of collection to date of extraction, been exceeded?	___	[___]	___

Samples for BNA analysis, both soils and waters, must be extracted within seven days of the date of collection. Extracts must be analyzed within 40 days of the date of extraction.

Table of Holding Time Violations

Sample	Sample Matrix	Date Sampled	(See Traffic Report) Date Lab Received	Date Extracted	Date Analyzed
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

ACTION: If holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

	YES	NO	N/A

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon reanalysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. The reviewer may determine that non-detect data are unusable ("R").

3.0 Surrogate Recovery (Form II)

3.1 Are the BNA Surrogate Recovery Summaries (Form II) present for each of the following matrices:

a. Low Water	[___]	___	___
b. Med Water	[___]	___	___
c. Low Soil	[___]	___	___
d. Med Soil	[___]	___	___

3.2 Are all the BNA samples listed on the appropriate Surrogate Recovery Summaries for each of the following matrices:

a. Low Water	[___]	___	___
b. Med Water	[___]	___	___
c. Low Soil	[___]	___	___
d. Med Soil	[___]	___	___

ACTION: Call lab for explanation / resubmittals. If missing deliverables are unavailable, document effect on data under "Conclusions" section of reviewer narrative.

3.3 Were outliers marked correctly with an asterisk?	[___]	___	___

ACTION: Circle all outliers in red.

3.4 Were two or more base-neutral OR acid surrogate recoveries out of specification for any sample or method blank?	___	[___]	___
If yes, were samples reanalyzed?	[___]	___	___
Were method blanks reanalyzed?	[___]	___	___

ACTION: If all BNA surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet SOW specifications, for the affected fraction only (i.e. base-neutral OR acid compounds):

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ").

	YES	NO	N/A
If any base-neutral or acid surrogate has a recovery of <10% : 1. Flag all positive results for that fraction (i.e. all acid or base-neutral compounds) "J". 2. Flag all non-detects for that fraction "R". Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and re-analyses. Check the internal standard areas.			
3.5 Are there any transcription/calculation errors between raw data and Form II?	___	[___]	___
ACTION: If large errors exist, call lab for explanation / resubmittal, make any necessary corrections and note errors under "Conclusions".			
4.0 Matrix Spikes (Form III)			
4.1 Is the Matrix Spike Duplicate/Recovery Form (Form III) present?	[___]	___	___
4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:			
a. Low Water	[___]	___	___
b. Med Water	[___]	___	___
c. Low Soil	[___]	___	___
d. Med Soil	[___]	___	___
ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.			

4.3 How many BNA spike recoveries are outside QC limits?

Water	Soils
_____ out of 22	_____ out of 22

4.4 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water	Soils
_____ out of 11	_____ out of 11

ACTION: If MS and MSD both have less than 10% recovery for an analyte, negative results for that analyte should be rejected, and positive results should be flagged "J". The above applies only to the sample used for MS/MSD analysis. Use professional judgement in

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	YES	NO	N/A
5.0 Blanks (Form IV)			
5.1 Is the Method Blank Summary (Form IV) present?	[___]	___	___
5.2 Frequency of Analysis: for the analysis of BNA TCL compounds, has a reagent/method blank been analyzed for each set of samples or every 20 samples of similar matrix (low water, med water, low soil, medium soil), whichever is more frequent?	[___]	___	___
5.3 Has a BNA instrument blank been analyzed at least once every twelve hours for each GC/MS system used?	[___]	___	___
ACTION: If any method blank data are missing, call lab for explanation / resubmittal. If not available, reject all associated positive data ("R").	[___]	___	___
5.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.			
Is the chromatographic performance (baseline stability) for each instrument acceptable for BNAs?	[___]	___	___
ACTION: Use professional judgement to determine the effect on the data.			
6.0 Contamination			
NOTE: "Water blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.			
6.1 Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for BNAs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample Dilution Factor.	___	[___]	___
6.2 Do any field/rinse blanks have positive BNA results (TCL and/or TIC)?	___	[___]	___
ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)			
NOTE: Only field/rinse blanks taken the same day as the samples are used to qualify data. Blanks may not be qualified because of contamination in another blank. Blanks may be qualified for surrogate, spectral, tuning or calibration QC problems.			

YES NO N/A

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

	Sample conc > CRQL but < 10x blank	Sample conc < CRQL & is < 10x blank value	Sample conc > CRQL value & >10x blank value
Common Phthalate Esters	Flag sample result with a 'U'; cross out 'B' flag	Reject sample result and report CRQL; cross out 'B' flag	No qualification is needed

	Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL value & > 5 blank value
Other Contaminants	Flag sample result with a 'U'; cross out 'B' flag	Reject sample result and report CRQL; cross out 'B' flag	No qualification is needed

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R" (unusable).

6.3 Are there field/rinse/equipment blanks associated with every sample? [___] ___ ___

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 GC/MS Tuning and Mass Calibration (Form V)

7.1 Are the GC/MS Tuning and Mass Calibration Forms (Form V) present for Decafluorotriphenylphosphine (DFTPP)? [___] ___ ___

7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve hour shift? [___] ___ ___

7.3 Has a tuning performance compound been analyzed for every twelve hours of sample analysis per instrument? [___] ___ ___

ACTION: If any tuning data are missing, take action specified in 3.2 above.

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.

YES NO N/A

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

7.4 Have the ion abundance criteria been met for each instrument used? [___]

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If tuning calibration is in error, flag all associated sample data as unusable ("R"). However, if expanded ion criteria are met (See 1988 Functional Guidelines), the data reviewer may accept data with appropriate qualifiers.

7.5 Are there any transcription / calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) ___ [___] ___

7.6 Have the appropriate number of significant figures (two) been reported? (Check at least two values, but if errors are found check more values.) [ ]

ACTION: If large errors exist, call lab for explanation / resubmittal, make necessary corrections and note errors under "Conclusions".

7.7 Are the spectra of the mass calibration compound acceptable? [ ]

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

8.0 Target Compound List (TCL) Analytes

8.1 Are the Organic Analysis Data Sheets (Form I BNA) present with required header information on each page, for each of the following:

a. Samples and/or fractions as appropriate [ ]

b. Matrix spikes and matrix spike duplicates [ ]

c. Blanks [ ]

	YES	NO	N/A
8.2 Are the BNA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			
a. Samples and/or fractions as appropriate	[___]	___	___
b. Matrix spikes and matrix spike duplicates (Mass spectra not required)	[___]	___	___
c. Blanks	[___]	___	___
ACTION: If any data are missing, take action specified in 3.2 above.			
8.3 Are the response factors shown in the Quant Report?	[___]	___	___
8.4 Is chromatographic performance acceptable with respect to:			
Baseline stability	[___]	___	___
Resolution	[___]	___	___
Peak shape	[___]	___	___
Full-scale graph (attenuation)	[___]	___	___
Other:_____	[___]	___	___
ACTION: Use professional judgement to determine the acceptability of the data.			
8.5 Are the lab-generated standard mass spectra of the identified BNA compounds present for each sample?	[___]	___	___
ACTION: If any mass spectra are missing, take action specified in 3.2 above. If Lab does not generate their own standard spectra, make note in "Contract Problems/Non-compliance".			
8.6 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?	[___]	___	___
8.7 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	[___]	___	___
8.8 Do sample and standard relative ion intensities agree within 20%?	[___]	___	___
ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected, flagged "N" (presumptive evidence of the presence of the compound) or changed to not detected (at the calculated detection limit).			

	YES	NO	N/A
9.0 Tentatively Identified Compounds (TIC)			
9.1 Are all Tentatively Identified Compound Forms (Form I, Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "J" qualifier?	[___]	___	___
9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:			
a. Samples and/or fractions as appropriate	[___]	___	___
b. Blanks	[___]	___	___
ACTION: If any TIC data are missing, take action specified in 3.2 above.			
ACTION: Add "J" qualifier if missing and "N" qualifier to all identified TIC compounds on Form I, Part B.			
9.3 Are any TCL compounds (from any fraction) listed as TIC compounds (example: 1,2-dimethylbenzene is xylene—a VOA TCL—and should not be reported as a TIC)?	___	[___]	___
ACTION: Flag with "R" any TCL compound listed as a TIC.			
9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	[___]	___	___
9.5 Do TIC and "best match" standard relative ion intensities agree within 20%?	[___]	___	___
ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate.			
10.0 Compound Quantitation and Reported Detection Limits			
10.1 Are there any transcription / calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found?	___	[___]	___
10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture?	___	[___]	___

	YES	NO	N/A

ACTION: If errors are large, call lab for explanation / resubmittal, make any necessary corrections and note errors under "Conclusions".

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher, CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

11.0 Standards Data (GC/MS)

11.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant. Reports) present for initial and continuing calibration? [___] ___ ___

ACTION: If any calibration standard data are missing, take action specified in 3.2 above.

12.0 GC/MS Initial Calibration (Form VI)

12.1 Are the Initial Calibration Forms (Form VI) present and complete for the BNA fraction? [___] ___ ___

ACTION: If any calibration standard forms are missing, take action specified in 3.2 above.

12.2 Are response factors stable for BNAs over the concentration range of the calibration (RSD <30%)? [___] ___ ___

ACTION: Circle all outliers in red.

ACTION: When RSD >30%, non-detects may be qualified using professional judgement. Flag all positive results "J". When RSD >90%, flag all non-detects as unusable ("R"). (Region II policy.)

12.3 Do any compounds have a RRF < 0.05? ___ [___] ___

ACTION: Circle all outliers in red.

ACTION: If any BNA compound has an average RRF < 0.05, flag positive results for that compound as estimated ("J"), and flag non-detects for that compound as unusable ("R").

	YES	NO	N/A
12.4 Are there any transcription / calculation errors in the reporting of average response factors (RRF) or %RSD? (Check at least two values but if errors are found, check more.)	___	[___]	___

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation / resubmittal, make any necessary corrections and note errors under "Conclusions".

13.0 GC/MS Continuing Calibration (Form VII)

13.1 Are the Continuing Calibration Forms (Form VII) present and complete for the BNA fraction?	[___]	___	___
13.2 Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	[___]	___	___

ACTION: List below all sample analyses that were not within twelve hours of the previous continuing calibration analysis.

_____

_____

_____

ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, call lab for explanation / resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").

13.3 Do any continuing calibration standard compounds have a RRF < 0.05?	___	[___]	___

ACTION: Circle all outliers in red.

ACTION: If any BNA compound has a RRF < 0.05, flag positive results for that compound as estimated ("J"), and flag non-detects for that compound as unusable ("R").

13.4 Do any compounds have a % difference between initial and continuing calibration RRF > 25%?	___	[___]	.

ACTION: Circle all outliers in red and qualify associated sample data as outlined in the table below:

YES NO N/A

% DIFFERENCE

25-50	50-90	>90
'J' positive results, no action for non detects	'J' positive results, 'UJ' non detects	'J' positive results, "R" non detects

13.5 Are there any transcription / calculation errors in the reporting of average response factors (RRF) or difference (%D) between initial and continuing RRFs? (Check at least two values but if errors are found, check more.) ___ [___] ___

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation / resubmittal, make any necessary corrections and note errors under "Conclusions".

14.0 Internal Standards (Form VIII)

14.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits for each continuing calibration? [___] ___ ___

ACTION: List all the outliers below.

Sample #	Internal Std	Area	Lower Limit	Upper Limit
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

(Attach additional sheets if necessary.)

ACTION: If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard. If extremely low area counts are reported, or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable ("R").

14.2 Are the retention times of the internal standards within 30 seconds of the associated calibration standard? [___] ___ ___

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

	YES	NO	N/A
15.0 Field Duplicates			
15.1 Were any field duplicates submitted for BNA analysis?	[___]	___	___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

	YES	NO	N/A
PART C: PESTICIDE/PCB ANALYSES			
1.0 Traffic Reports and Laboratory Narrative			
1.1 Are the Traffic Report Forms present for all samples?	[ ]	—	—
ACTION: If no, contact lab for replacement of missing or illegible copies.			
1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?	—	[ ]	—
ACTION: Use professional judgement to evaluate the effect on the quality of the data.			
ACTION: If any sample analyzed as a soil contains more than 50% water, all data should be rejected.			
2.0 Holding Times			
2.1 Have any PEST/PCB holding times, determined from date of collection to date of extraction, been exceeded?	—	[ ]	—
Samples for PEST/PCB analysis, both soils and waters, must be extracted within seven days of the date of collection. Extracts must be analyzed within 40 days of the date of extraction.			
3.0 Surrogate Recovery (Form II)			
3.1 Are the PEST/PCB Surrogate Recovery Summaries (Form II) present for each of the following matrices:			
a. Low Water	[ ]	—	—
b. Med Water	[ ]	—	—
c. Low Soil	[ ]	—	—
d. Med Soil	[ ]	—	—
3.2 Are all the PEST/PCB samples listed on the appropriate Surrogate Recovery Summaries for each of the following matrices:			
a. Low Water	[ ]	—	—
b. Med Water	[ ]	—	—
c. Low Soil	[ ]	—	—
d. Med Soil	[ ]	—	—

	YES	NO	N/A

ACTION: Call lab for explanation / resubmittals. If missing deliverables are unavailable, document effect on data under "Conclusions" section of reviewer narrative.

3.3 Were outliers marked correctly with an asterisk? [__] __ __

ACTION: Circle all outliers in red.

3.4 Was surrogate (DBC) recovery outside of the contract specification for any sample or blank? __ [__] __

ACTION: No qualification is done if surrogates are diluted beyond detection. If recovery is below contract limit (but above zero), flag all results for that sample "J". If recovery is zero, flag positive results "J" and non-detects "R". If recovery for the blank is zero, flag non-detects for all associated samples "R". If recovery is above contract limit, flag all positive results for that sample "J", unless in the reviewers professional judgement the high recovery is due to co-eluting interference (check the associated blank - if recovery is high there also, flag the sample data).

3.5 Are there any transcription/calculation errors between raw data and Form II? __ [__] __

ACTION: If large errors exist, call lab for explanation / resubmittal, make any necessary corrections and note errors under "Conclusions".

4.0 Matrix Spikes (Form III)

4.1 Is the Matrix Spike Duplicate/Recovery Form (Form III) present? [__] __ __

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Low Water [__] __ __

b. Med Water [__] __ __

c. Low Soil [__] __

d. Med Soil [__]

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

4.3 How many PEST/PCB spike recoveries are outside QC limits?

Water	Soils

YES NO N/A

4.4 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water	Soils
_____ out of 6	_____ out of 6

ACTION: If MS and MSD both have less than zero recovery for an analyte, negative results for that analyte should be rejected, and positive results should be flagged "J". The above applies only to the sample used for MS/MSD analysis. Use professional judgement in applying this criterion to other samples.

5.0 Blanks (Form IV)

5.1 Is the Method Blank Summary (Form IV) present? [___] ___

5.2 Frequency of Analysis: for the analysis of Pesticide TCL compounds, has a reagent/method blank been analyzed for each set of samples or every 20 samples of similar matrix (low water, med water, low soil, medium soil), whichever is more frequent? [___] ___ ___

5.3 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for PEST/PCBs? [___] ___ ___

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent blanks have positive results for PEST/PCBs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample Dilution Factor. [ ]

6.2 Do any field/rinse blanks have positive PEST/PCB results? ___ [ ] ___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

YES NO N/A

NOTE: Only field/rinse blanks taken the same day as the samples are used to qualify data. Blanks may not be qualified because of contamination in another blank. Blanks may be qualified for surrogate, spectral, tuning or calibration QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL & > 5x blank value
Flag sample result with a "U"; cross out "B" flag	Reject sample result and report CRQL; cross out "B" flag	No qualification is needed

6.3 Are there field/rinse/equipment blanks associated with every sample? [___] ___ ___

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 Calibration and QC Performance

7.1 Are the following Gas Chromatograms and Data System Printouts for both Primary and Confirmation (confirmation standards not required if there are no positive results above CRQL) column present:

a. Evaluation Standard Mix A [___] ___ ___

b. Evaluation Standard Mix B [___] ___ ___

c. Evaluation Standard Mix C [___] ___ ___

d. Individual Standard Mix A [___] ___

e. Individual Standard Mix B [___] ___ ___

f. Multi-component Pesticides Toxaphene & Chlordane [___]

g. Aroclors 1016/1260 [___]

h. Aroclors 1221, 1232, 1242, 1248, and 1254 [___] ___ ___

CHM 001 0390

ACTION: If no, take action specified in 3.2 above

	YES	NO	N/A
7.2 Is Form VIII Pest-1 present and complete for each GC column (primary and confirmation) and each 72 hour sequence of analyses?	[___]	___	___
ACTION: If no, take action specified in 3.2 above.			
7.3 Are there any transcription/calculation errors between raw data and Form VIII?	___	[___]	___
ACTION: If large errors exist, call lab for explanation/ resubmittal, make any necessary corrections and note errors under "Conclusions".			
7.4 Has the total breakdown on quantitation or confirmation column exceeded 20% for DDT?	___	[___]	___
- for Endrin?	___	[___]	___
or if Endrin aldehyde and 4,4'-DDD co-elute and there is a peak at their retention time, has the combined DDT and Endrin breakdown exceeded 20%?	___	[___]	___

ACTION:

a. If DDT breakdown is greater than 20% on quantitation column beginning with the samples following the last in control standard:

   1. Flag all positive DDT results "J".
   2. If DDT was not detected but DDD and/or DDE are positive, flag the DDT non-detect "R".
   3. Flag positive DDD and DDE results "JN".
   4. If DDT breakdown is > 20% on confirmation column and DDT is identified on quantitation column but not on confirmation column, use professional judgement to determine whether DDT should be reported on Form I (if reported, flag result "N").

b. If Endrin breakdown is > 20% on quantitation column, beginning with the samples following the last in control standard:

   1. Flag all positive Endrin results "J".
   2. If Endrin was not detected, but Endrin Aldehyde and/or Endrin Ketone are positive, flag the Endrin non-detect "R".
   3. Flag Endrin Ketone positive results "JN".
   4. If Endrin breakdown is > 20% on confirmation column and Endrin is identified on quantitation column but not on confirmation column, use professional judgement to determine whether Endrin should be reported on Form I (if reported, flag result "N").

c. If the combined breakdown is used (it can only be used if the conditions in 7.4 above are met) and is > 20% on quantitation column beginning with the last in control standard, take the actions specified in 7.4 a and b above. If the combined breakdown is >20% on confirmation column and Endrin or DDT is identified on quantitation column but not on confirmation column, use professional judgement to determine whether Endrin or DDT should be reported on Form I (if reported, flag result "N").

	YES	NO	N/A
7.5 Is the linearity check RSD of all four calibration factors <10% for the quantitation column?	[___]	___	___
ACTION: If no, flag positive hits for all pesticide and PCB analytes "J" for all associated samples. Do not flag toxaphene or DDT if they are quantified from a 3-point calibration curve.			
7.6 Is the % difference between the EVAL A and each analysis, (quantitation and confirmation) DBC retention time within QC limits (2% for packed column, 0.3% for capillary [I.D. < 0.32 mm], 1% for megabore [0.32 < I.D. < 2 mm]) ?	[___]	___	___
ACTION: DBC retention time cannot be evaluated if DBC is not detected. If it is present and has a retention time out of QC limits, then use professional judgement to determine the reliability of the analysis and flag results "R", if appropriate.			
7.7 Was the proper analytical sequence followed for each 72 hour period of analyses (page PEST D-36 in 8/87 SOW).	[___]	___	___
ACTION: If no, use professional judgement to determine the severity of the effect on the data and accept or reject it accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.			
8.0 Pesticide/PCB Standards Summary			
8.1 Is Form IX present and complete for each GC column and 72 hr sequence of analyses?	[___]	___	___
ACTION: If no, take action specified in 3.2 above.			
8.2 Are there any transcription/calculation errors between raw data and Form IX?	___	[___]	___
ACTION: If large errors exist, call lab for explanation / resubmittal, make any necessary corrections and note errors under "Conclusions".			
8.3 Is DDT retention time for packed columns > 12 min (except OV-1 and OV-101 columns)?	[___]	___	___
ACTION: If no, check that there is adequate resolution between individual components. If not, flag results for compounds that interfere with each other (co-elute) "R".			
8.4 Do all standard retention times fall within the windows established for the first IND A and IND B analyses?	[___]	___	___

	YES	NO	N/A
ACTION: Beginning with the samples following the last <u>in control</u> standard, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and, DBC is visible non-detects are valid. If peaks are present and cannot be identified through "pattern recognition" or a consistent shift in standard retention times, flag all affected compound results "R".			
8.5 Are the continuing calibration standard calibration factors within 15% (for quantitation column) or 20% (for confirmation column) of the initial (at beginning of 72 hr sequence) calibration factors?	[___]	___	___
ACTION: If no, flag all associated positive results "J". Use professional judgement to determine whether or not to flag non-detects.			
9.0 <u>Pesticide/PCB Identification</u>			
9.1 Is Form X complete for every sample in which a pesticide or PCB was detected?	[___]	___	___
ACTION: If no, take action specified in 3.2 above.			
9.2 Are there any transcription errors between raw data and Form X?	___	[___]	___
ACTION: If large errors exist, call lab for explanation / resubmittal, make any necessary corrections and note errors under "Conclusions".			
9.3 Are retention times of sample compounds within the calculated retention time windows for both quantitation and confirmation analyses?	[___]	___	___
Was GC/MS confirmation provided when required (when compound concentration is > 10 ug/ml in final extract)?	[___]	___	___
ACTION: Reject ("R") all positive results (meeting quantitation column criteria, but missing confirmation by a second column or GC/MS (if appropriate). Also, reject ("R") all positive results not meeting retention time window criteria unless associated standard compounds are similarly biased (i.e. base on RRT to DBC).			
9.4 Check chromatograms for false negatives, especially for the multiple peak components toxaphene and PCB's. Were there any false negatives?	___	[___]	___
ACTION: If appropriate PCB standards were not analyzed, or if the lab performed no confirmation analysis, flag the appropriate data with an "R".			

	YES	NO	N/A

10.0 Compound Quantitation and Reported Detection Limits

10.1 Are there any transcription / calculation errors in Form I results? Check at least two positive values. Were any errors found? ___ [___] ___

NOTE: Simple peak pesticide results can be checked for rough agreement between quantitative results obtained on the two GC columns. The reviewer should use professional judgement to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interfering compound is indicated, the lower of the two values should be reported and qualified as presumptively present at an estimated quantity ("JN"). This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that the presence of interferences has obscured the attempt at a second column confirmation.

10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture? [___] ___ ___

ACTION: If errors are large, call lab for explanation / resubmittal, make any necessary corrections and note errors under "Conclusions".

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

11.0 Chromatogram Quality

11.1 Were baselines stable? [___] ___ ___

11.2 Were any electropositive displacement (negative peaks) or unusual peaks seen? ___ [___] ___

11.3 Were early eluting peaks (for early eluting analytes) resolved to baseline? [___] ___ ___

ACTION: For 11.1 and 11.2, comment only. For 11.3, reject ("R") those analytes that are not sufficiently resolved.

	YES	NO	N/A
.2.0 Field Duplicates			
12.1 Were any field duplicates submitted for PEST/PCB analysis?	[___]	___	___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

DRAFT

LABORATORY DATA VALIDATION

FUNCTIONAL GUIDELINES FOR EVALUATING INORGANICS ANALYSIS

Prepared for the

HAZARDOUS SITE EVALUATION DIVISION
U. S. ENVIRONMENTAL PROTECTION AGENCY

October 1989 Revision

DRAFT

TABLE OF CONTENTS

INTRODUCTION

This document is designed to offer guidance in analytical data evaluation and validation for Inorganic data produced by laboratories participating in the EPA Contract Laboratory Program (CLP) or operating under CLP analytical protocols. These Guidelines have been updated to include requirements in the Inorganic Statement of Work (SOW) 7/88, including the June 1989 revisions.

In some aspects, the document is equivalent to a Standard Operating Procedure (SOP). In other, more subjective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples. Those areas where specific SOPs are possible are primarily areas in which definitive performance requirements are established. These requirements are concerned with specifications that are not sample dependent, but rather are associated with performance criteria on matters that should be fully under a laboratory's control. These specific areas include blanks, calibration standards, calibration verification standards, laboratory control standards, and interference check standards.

The document is intended to assist technical review of the data. While some areas of overlap between technical review and Contract Compliance Screening (CCS) may exist, determining contract compliance is not intended to be an objective of these guidelines.

At times, there may be an urgent need to use data which do not meet all contract requirements and technical criteria. Use of these data does not constitute either a new requirement standard or full acceptance of the data. Any decision to use data for which performance criteria have not been met is strictly to facilitate the progress of projects requiring the availability of the data. A contract laboratory submitting data which are out of specification may be required to reanalyze samples or resubmit data even if the previously submitted data have been utilized due to urgent program needs. Data which do not meet specified requirements are never fully acceptable. The only exception to this is in the area of specifications for individual sample analyses. If the nature of the sample itself limits the attainment of specifications, appropriate allowances must be made. The overriding concern of the Agency is to obtain data which are technically valid and legally defensible.

All data reviews must have, as a cover sheet, the Inorganic Regional Data Assessment (IRDA) form. A copy of this form is attached at the end of this document. If mandatory actions are required, they should be specifically noted on this form. In addition, this form is to be used to summarize deficiencies requiring attention, as well as general laboratory performance and any discernible trends in the quality of the data. This form is not a replacement for the data review. Sufficient supplementary documentation must accompany the form to clearly identify the problems associated with a Case or Sample Delivery Group. The form and any supplemental review documentation must accompany the laboratory data forwarded to the intended data recipient (client) or user. A copy of the form and review documentation must be submitted to the Contract Laboratory Program Quality Assurance Officer (CLP/QAO), the Regional Deputy Project Officer (DPO) having assigned oversight responsibility for the laboratory

producing the data, and the Environmental Monitoring Systems Laboratory in Las Vegas, Nevada (EMSL-LV).

It is the responsibility of the data reviewer to notify the appropriate Regional DPO concerning problems and deficiencies with regard to laboratory data. If there is an urgent requirement, the DPO may be contacted by telephone to expedite corrective action. It is recommended that all items for DPO action be presented at one time. In any case the Inorganic Regional Data Assessment form must be completed and submitted.

PRELIMINARY REVIEW

In order to use this document effectively, the reviewer should have a general overview of the Case or Sample Delivery Group at hand. The exact number of samples, their assigned number, their matrix, and the number of laboratories involved in their analysis are essential information. Background information on the site is helpful, but often this information is very difficult to obtain. The site Project Manager is the best source for information of this type or for further direction.

The CCS report, when available, is a source of a large quantity of summarized information. It can be used to alert the reviewer of systematic problems in the Case or to problems that may be sample specific. This information may be used in data validation. If CCS is unavailable, those criteria affecting data validity must be addressed by the data reviewer.

Cases routinely have unique samples which require special attention by the reviewer. Field blanks, field duplicates, and performance audit samples need to be identified. The sampling records should provide:

1. Name of Site Project Manager.
2. Complete list of samples with notations on
   a. sample matrix
   b. field blanks*
   c. field duplicates*
   d. field spikes*
   e. QC audit sample*
   f. shipping dates
   g. labs involved.

* If applicable.

The chain-of-custody record includes sample descriptions and date of sampling. Although sampling date is not addressed by CLP contract requirements, the reviewer must take into account lag time between sampling and shipping when assessing sample holding times.

INORGANICS DATA REVIEW PROCEDURE

The requirements to be checked in data validation are listed below. (The notation "CCS" indicates that the contractual requirements for these items will also be checked by CCS. CCS requirements are not always the same as the data review criteria.)

I. Holding Times (CCS-Lab holding times only)

II. Calibration

- Initial (CCS)
- Initial and Continuing Calibration Verification (CCS)

III. Blanks (CCS)

IV. ICP Interference Check Samples (CCS)

V. Laboratory Control Sample (CCS)

VI. Duplicate Sample (CCS)

VII. Matrix Spike Sample (CCS)

VIII. Furnace Atomic Absorption QC (CCS)

IX. ICP Serial Dilution (CCS)

X. Sample Result Verification (CCS)

XI. Field Duplicates

XII. Overall Data Assessment

I. HOLDING TIMES

A. Objective:

The objective is to ascertain the validity of analytical results based on the holding time of the sample from the date of collection to the date of analysis.

Note: For data review and assessment purposes, the holding time is based on the elapsed time between the date of sample collection and date of analysis, rather than the elapsed time between the verified time of sample receipt (VTSR) and the date of analysis as allowed by SOW 7/88. It is thus a technical evaluation, not a contract requirement.

B. Criteria:

Technical requirements (as opposed to contractual) for sample holding times have only been established for water matrices. The following holding time and preservation requirements were promulgated under 40 CFR 136 (Clean Water Act) and are also found in volume 49, Number 209, page 43260, of the Federal Register issued on October 26, 1984.

METALS:	6 months; preserved to pH < 2
MERCURY:	28 days; preserved to pH < 2
CYANIDE:	14 days; preserved to pH > 12

C: Evaluation Procedure:

Actual holding times are established by comparing the sampling date on the EPA Sample Traffic Report with the dates of analysis on the analysis run logs (Form 14 (XIV-IN) of SOW 7/88). Examine the digestion and/or distillation logs included with the raw data to determine if samples had been preserved at the proper pH.

Analyte Holding Time (days) = Anal. Date - Coll. Date

D. Action:

1. If 40 CFR criteria for holding times or preservation are not met for a specific analyte in a water matrix, qualify all water matrix results for that analyte which are greater than the Instrument Detection Limit (> IDL) as estimated (J) and results less than the Instrument Detection Limit (< IDL) as estimated (UJ). For Cyanide and Mercury the appropriate detection limit for this judgement is the Contract Required Detection Limit (CRDL) rather than the Instrument Detection Limit (IDL).

2. If technical holding times for a specific analyte are exceeded, the reviewer may use professional judgement to evaluate the reliability of the data and the probable effects of additional storage on the analytical results. The expected bias would be low and the reviewer may determine that results < IDL (< CRDL for CN and Hg) are unusable (R).

3. Due to limited information concerning holding times for soil samples, it is left to the discretion of the data reviewer whether to apply 40 CFR water holding time criteria to soil samples. If the data are qualified when water holding time criteria are applied to soil samples, it must be clearly documented in the review.

Note: Contractual holding time criteria based on VTSR and analysis date are equally applicable to water and soil samples and will be subject to CCS review. Preservation requirements for water samples are not applicable to soil samples under any conditions.

II. CALIBRATION

A. Objective:

Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration verification demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration remains valid throughout the course of the analytical run.

B. Criteria:

1. Initial Calibration

Instruments must be calibrated daily and each time the instrument is set up.

a. ICP Analysis

A calibration blank and at least one standard must be used in establishing the calibration curve.

b. Atomic Absorption Analysis (AA) (other than Mercury)

(1). A blank and at least three standards must be used in establishing the calibration curve. One of the standards must be at the Contract Required Detection Limit (CRDL).

(2). The correlation coefficient should be ≥ 0.995.

Note: The correlation coefficient ≥ 0.995 is a technical, not a contractual, criterion. The reviewer should exercise discretion in applying this criterion to data qualification. See D.2 under "Action".

c. Mercury Analysis

(1). A blank and at least four standards must be used in establishing the calibration curve.

(2). The correlation coefficient should be ≥ 0.995.

Note: The correlation coefficient ≥ 0.995 is a technical, not a contractual, criterion. The reviewer should exercise discretion in applying this criterion to data qualification. See D.2 under "Action".

d. Cyanide Analysis

(1). A blank and at least three standards must be used in establishing the calibration curve.

(2). A mid-range standard must be distilled. (See B.2.c. below).

(3). The correlation coefficient for the photometric calibration curve should be ≥ 0.995.

Note: The correlation coefficient ≥ 0.995 is a technical, not a contractual, criterion. The reviewer should exercise discretion in applying this criterion to data qualification. See D.2 under "Action".

2. Initial and Continuing Calibration Verification (ICV and CCV).

a. Analysis results must fall within the control limits of 90 - 110 Percent Recovery (%R) of the true value for all ICV and CCV analytes except mercury and cyanide.

b. Analysis results for mercury ICV and CCV must fall within the control limits of 80 - 120 %R.

c. Analysis results for cyanide ICV and CCV must fall within 85 - 115 %R. SOW 7/88, page E-5 requires that the Cyanide ICV must be distilled with the batch of samples analyzed in association with it. If this has been done and meets specifications, this may suffice for the mid-range standard specified in B.1.d.(2).

C. Evaluation Procedure:

1. Verify that the instrument was calibrated immediately prior to the subject analytical run or within the twenty-four hour period preceding and including the subject analytical run.

2. Verify that the correlation coefficient is $\geq$ 0.995.

3. Check the distillation log and verify that the Cyanide ICV was distilled. Also determine if a mid-range standard was distilled and used during the course of procedural calibration.

4. Recalculate one or more of the ICV and CCV %R per type of analysis (ICP,GFAA, etc.) using the following equation and verify that the recalculated value agrees with the laboratory values on Form 2A (Form II (Part 1)-IN). Due to possible rounding discrepancies, allow results to fall within 1% of the contract windows (e.g., 89 - 111%).

$$\%R = \frac{Found}{True} \times 100$$

Where: *Found =* *Concentration (in ug/l) of each analyte measured in the analysis of the ICV or CCV solution.*

True = *True value concentration (in ug/l) of each analyte in the ICV or CCV source solution.*

D. Action:

1. If the minimum number of standards as specified in section B were not used for initial calibration, or if the instrument was not calibrated immediately prior to the analytical run or within the twenty-four hour period preceding and including the analytical run, qualify all associated analytical data as unusable (R).

2. If the calibration curve correlation coefficient is < 0.995, qualify results > IDL (> CRDL for CN and Hg) as estimated (J) and results < IDL (< CRDL for CN and Hg) as estimated (UJ).

Note: Reviewer should use discretion in applying this non-contractual criterion. Further evaluation of the calibration curve, ICV and CCV specifications, and sample concentration levels may be necessary to determine if data qualification is necessary.

3. If the ICV for Cyanide was not distilled with the associated sample batch, qualify all associated results as estimated (J) or (UJ), as appropriate. If the ICV for Cyanide was not distilled with the associated sample batch as required by SOW 7/88, but it is documented that a mid-range standard was distilled and met procedural

specifications, the reviewer may use professional judgement in determining whether data qualification is necessary.

4. If the ICV or CCV %R falls outside the acceptance windows for one or more analytes, use professional judgement to qualify all associated data for the affected analytes. If possible, indicate the expected bias in the review. The following guidelines are recommended:

Note: For the guidelines in a - e below, the appropriate limit on which to base judgments for CN and Hg is the CRDL for those analytes.

a. If the ICV or CCV %R falls outside the acceptance windows, but within the ranges of 75 - 89% or 111 - 125% (CN, 70 - 84% or 116 - 130%; Hg, 65 - 79% or 121 - 135%), qualify associated results > IDL for the affected analytes as estimated (J).

b. If the ICV or CCV %R is within the range of 111 - 125% (CN, 116 - 130%; Hg 121 - 135%), associated results < IDL are acceptable.

c. If the ICV or CCV %R is 75 - 89% (CN, 70 - 84%; Hg, 65 - 79%), qualify associated results < IDL as estimated (UJ).

d. If the ICV or CCV %R is <75%, (CN, <70%; Hg, <65%), qualify all associated results as unusable (R).

e. If the ICV or CCV %R is >125%, (CN, >130%; Hg, >135%), qualify associated results > IDL as unusable (R); associated results < IDL are acceptable.

III. BLANKS

A. Objective:

The assessment of blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to any blank associated with the samples. If problems with <u>any</u> blank exist, all data associated with the SDG must be carefully evaluated to determine whether or not there is an inherent variability in the data for the SDG, or if the problem is an isolated occurrence not affecting other data.

B. Criteria:

The criteria listed below are a mixture of the criteria of the July, 1988, Functional Guidelines document (Criterion 1) and criteria based on requirements and allowances of SOW 7/88 (Criteria 2 & 3).

1. No contaminants should be in the blank(s).

2. No analyte should be present in a calibration blank associated with an analytical run in excess of the CRDL for that analyte.

3. No analyte should be present in the preparation blank associated with an SDG or digestion batch in excess of the CRDL unless samples associated with that preparation blank contain a concentration level ≥ 10 X CRDL for that analyte.

C. Evaluation Procedure:

1. Review the results reported on the Blank Summary Form 3 (Form III-IN) as well as associated raw data (ICP printouts, strip charts, printer tapes, bench sheets, etc.) for all blanks and verify that results were accurately reported.

Note: Only calibration blanks and preparation blanks are reported on Form 3. Blanks originating with the sampling operation (field blanks, trip blanks, QA audit blanks, etc.) and submitted to the laboratory will be processed by the laboratory as field samples and reported on a Form 1 (Form I-IN). Therefore, if an assessment is to be made of these blanks, the appropriate Forms 1 and associated raw data must be evaluated.

2. From Form 3, determine that no analyte is present at a concentration > CRDL for that analyte in the Calibration Blanks (ICB or CCB).

3. From Form 3, determine that no analyte is present at a concentration > CRDL for that analyte in the preparation blank(s). In the event any analyte exceeds CRDL, evaluate associated samples to determine that the concentration level for that analyte is ≥ 10 X blank concentration.

4. From the raw data determine that no analyte in the calibration blanks or preparation blanks has a negative result whose absolute value exceeds the CRDL for that analyte.

D. Action:

1. Sample results must <u>not</u> be corrected by subtracting any blank value.

2. Qualification of sample results based on the analyses of blanks submitted from the field is left to the discretion and professional judgement of the reviewer; however, no corrective action is required of the laboratory when such blanks show evidence of contamination.

3. For analytes with positive levels of contamination in calibration or preparation blanks, values < IDL (< CRDL for CN or Hg) in associated samples are acceptable and may be reported as (U).

4. For analytes whose concentration level in a calibration blank exceeds the CRDL, all associated sample result data > IDL (> CRDL for CN or Hg) should be qualified as unusable (R).

5. For analytes with negative results in a calibration blank whose absolute value exceeds the CRDL, all associated sample result data should be qualified as unusable (R).

6. For analytes with positive levels exceeding the CRDL in the preparation blank, all associated sample results > IDL and less than 10 X blank concentration level should be qualified as unusable (R). Results $\geq$ 10 X blank concentration level may be accepted without qualification, although reviewer should use discretion in doing so.

7. For analytes with negative results in the preparation blank whose absolute values exceed the CRDL, all associated sample results < CRDL should be qualified as unusable. Associated sample results $\geq$ CRDL may be accepted.

IV. INTERFERENCE CHECK SAMPLE (ICS)

A. Objective:

The ICP interference check samples verifies the validity of the laboratory's interelement and background correction factors.

B. Criteria:

1. The ICP Interference Check Sample must be run at the beginning and end of the analytical run or a minimum of twice per eight hour working shift, whichever is more frequent.

2. Results for the ICP analysis of Solution AB during the analytical run must fall within the limits of $\pm$ 20% of the true value for the analytes included in the Interference Check Samples.

C. Evaluation Procedure:

1. Recalculate from the raw data (ICP printout) one or more of the recoveries using the following equation for %R, and verify that the recalculated value agrees with the laboratory reported values on Form 4 (Form IV-IN).

$$\%R = \frac{\text{Found Analyte Value, Solution AB}}{\text{True Analyte Value, Solution AB}} \times 100$$

2. Check ICS raw data for results with an absolute value > IDL for those analytes which are not intended to be present in the ICS solution.

D. Action:

1. For samples with concentrations of Al, Fe, Ca, or Mg which are comparable to or greater than their respective levels in the Interference Check Sample:

   a. If the ICS recovery for an analyte is > 120% and the analyte results are < IDL in the associated samples, the data are acceptable for use.

   b. If the ICS recovery for an analyte is > 120%, or falls between 50 - 79 %, and the analyte results are > IDL in the associated samples, qualify the affected data as estimated (J).

   c. If the ICS recovery for an analyte falls between 50 - 79% and the analyte results are < IDL in the associated sample, the possibility of false negatives exists. Qualify the affected analyte data for these samples as estimated (UJ).

   d. If ICS recovery for an analyte is < 50%, qualify the affected analyte data for the associated samples as unusable (R).

   Note: If possible, indicate the bias for the estimated sample results in the review.

2. If results > IDL are observed for elements which are not intended to be present in the EPA provided ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with comparable or higher levels of interferents and with suspect analyte concentrations that approximate those levels found in the ICS (false positives), qualify sample results > IDL as estimated (J).

3. If negative results are observed for analytes that are not intended to be present in the EPA ICS solutions, and their absolute value is > IDL, the possibility of false negatives in the samples exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with comparable or higher levels of interferents, qualify results for the affected analytes < IDL as estimated (UJ).

4. In general, the sample data can be accepted if the concentrations of Al, Fe, Ca, and Mg in the sample are found to be less than or equal to their respective concentrations in the ICS. If these elements are present at concentrations greater than the level in the ICS, or other elements are present in the sample at > 10 mg/l, the reviewer should investigate the possibility of other interference effects by using Table 2 on page D-22 of SOW 7/88. These analyte concentration equivalents presented in the Table should be considered

only as estimated values, since the exact value of any analytical system is instrument specific. Therefore, estimate the concentration produced by an interfering element. If the estimate is > 2 X CRDL and also greater than 10% of the reported concentration of the affected element, qualify the affected results as estimated (J).

V. LABORATORY CONTROL SAMPLE (LCS)

A. Objective:

The laboratory control sample serves as a monitor of the overall performance of all steps in the analysis, including the sample preparation.

B. Criteria:

1. All aqueous LCS results must fall within the control limits of 80 - 120 %R, except for Sb and Ag which have no control limits.

2. All solid LCS results must fall within the control limits established for each element by EPA. This information is available from EMSL-LV.

C. Evaluation Procedure:

1. Review Form 7 (Form VII-IN) and verify that results fall within the control limits.

2. Check the raw data (ICP printout, strip charts, bench sheets, etc.) to verify the reported recoveries on Form 7. Recalculate one or more of the recoveries (%R) using the following equation:

$$\%R = \frac{\text{LCS Found}}{\text{LCS True}} \times 100$$

Where,

LCS Found = *concentration (in ug/l for aqueous; ug/kg for solid) of each analyte measured in the analysis of the LCS solution.*

LCS True = *stated true value concentration (in ug/l for aqueous; ug/kg for solid) of each analyte in the LCS source.*

D. Action:

1. Aqueous LCS:

a. If the LCS recovery for an analyte falls within the range of 50 - 79% or > 120%, qualify associated sample results with affected analyte values > IDL as estimated (J).

b. If the LCS recovery for an analyte is > 120%, associated sample results with affected analyte values < IDL are acceptable.

c. If the LCS recovery for an analyte falls within the range of 50 - 79%, qualify associated sample results with affected analyte values < IDL as estimated (UJ).

d. If the LCS recovery for an analyte is < 50%, qualify associated sample results for the affected analyte as unusable (R).

2. Solid LCS:

a. If the LCS recovery for an analyte falls outside its established EPA control limit, qualify associated sample results with affected analyte values > IDL as estimated (J).

b. If the LCS recovery for an analyte is higher than its established EPA control limit, associated sample results with affected analyte values < IDL are acceptable.

c. If the LCS recovery for an analyte is lower than its established EPA control limit, qualify associated sample results with affected analyte values < IDL as estimated (UJ).

VI. DUPLICATE SAMPLE ANALYSIS

A. Objective:

Duplicate analyses are indicators of a laboratory's analytical precision based on each sample matrix.

B. Criteria:

1. Samples identified as Field Blanks cannot be used for duplicate analyses.

2. A control limit of ± 20% (± 35% for soil) for the Relative Percent Difference (RPD) shall be used for sample values > 5X CRDL.

3. A control limit of ± CRDL (± 2X CRDL) shall be used for sample values < 5X CRDL, including the case when only <u>one</u> of the duplicate sample values is < 5X CRDL.

C. Evaluation Procedure:

1. Review Form 6 (Form VI-IN) and verify that results fall within the control limits.

2. Check the raw data and recalculate one or more RPD using the following equation to verify that results have been correctly reported on Form 6.

$$RPD = \frac{|S - D|}{(S+D)/2} \times 100$$

Where,

S = First Sample Value (original)

D = Second Sample Value (duplicate)

3. Verify that the Field Blank was not used for duplicate analysis.

D. Action:

1. If duplicate analysis results for a particular analyte fall outside the appropriate control limits, qualify the results for that analyte in all associated samples of the same matrix as estimated (J).

2. If the Field Blank was used for duplicate analysis, all other QC data must be carefully checked and professional judgement exercised when evaluating the data.

Note: This information must be included on the IRDA form.

VII MATRIX SPIKE SAMPLE ANALYSIS

A. Objective:

The matrix spike sample analysis provides information about the effect of each sample matrix on the digestion and measurement methodology.

B. Criteria:

1. Samples identified as Field Blanks must not be used for matrix spike sample analysis.

2. Spike recovery (%R) must be within the limits of 75 - 125%. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more. If the latter condition exists for an analyte, matrix spike recovery cannot be used to qualify the associated sample data for that analyte.

C. Evaluation Procedure:

1. Review Form 5 (Form V-IN) and verify that results fall within the specified control limits.

2. Check raw data and recalculate one or more %R using the following equation to verify that results were correctly reported on Form 5.

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

Where,

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

3. Verify that the Field Blank was not used for matrix spike sample analysis.

D. Action:

1. If the spike recovery for an analyte is > 125% and reported associated sample results for that analyte are < IDL (< CRDL for CN or Hg), the affected data are acceptable for use.

2. If the spike recovery for an analyte is > 125% or < 75% and reported sample results for that analyte are > IDL, qualify the affected data for that analyte in the associated samples as estimated (J).

3. If the spike recovery for an analyte falls within the range of 30 - 74% and reported sample results for that analyte are < IDL, qualify the affected data in the associated samples as estimated (UJ).

4. If spike recovery results for an analyte are < 30% and the reported sample results for that analyte are < IDL, qualify the affected data in the associated samples as unusable (R).

5. If the Field Blank was used for matrix spike analysis, all other QC data must be carefully checked and professional judgement exercised when evaluating the data. This information must be included on the IRDA form.

Note: For all analytes except Ag, Hg, and those elements analyzed by GFAA, if the matrix spike recovery does not meet criteria, a post digestion spike is required; The results of post digestion spike recovery are not used to qualify the data; however, the information must be included in the IRDA report.

VIII. FURNACE ATOMIC ABSORPTION QC

A. Objective:

Duplicate injections and furnace post digestion spikes establish the precision and accuracy of the individual analytical determinations. In some instances, Method of Standard Additions may be required.

B. Criteria:

1. For sample concentrations > CRDL, duplicate injections must agree within ± 20% Relative Standard Deviation (RSD) or Coefficient of Variation (CV). Otherwise, the sample must be rerun once (at least two additional injections).

2. Analytical (post digestion) spike recovery must be > 85% and < 115%.

3. The Furnace Atomic Absorption Scheme must be followed as described in SOW 7/88, p. E 16-18.

C. Evaluation Procedure:

1. Check raw data to verify that duplicate injections agree within ±20% RSD (or CV) for sample concentrations > CRDL.

2. Review Furnace AA raw data to verify that the Furnace Atomic Absorption Scheme has been followed.

D. Action:

1. If duplicate injections are outside the ± 20% RSD (or CV) limits and the sample has not been rerun once as required, qualify the data as estimated (J). If the rerun sample results are outside the ± 20% limits as well, qualify the sample data as estimated (J).

2. If the analytical (post digestion) spike recovery is < 40%, qualify results > IDL as estimated (J).

3. If the analytical (post digestion) spike recovery is ≥ 10%, but <40%, qualify results < IDL as estimated (UJ).

4. If the analytical (post digestion) spike recovery is < 10%, qualify results < IDL as unusable (R).

5. If sample absorbance is < 50% of the analytical (post digestion) spike absorbance, then: If the furnace analytical (post digestion) spike is not within 85 - 115 %, qualify the sample results > IDL as estimated; qualify the results < IDL as estimated (UJ).

6. If Method of Standard Additions (MSA) is indicated as being required but has not been performed, qualify the data as estimated (J).

7. If any of the samples analyzed by MSA were not spiked at the appropriate levels, qualify the data as estimated (J).

8. If the MSA correlation coefficient is < 0.995, qualify the data as estimated (J).

IX. ICP SERIAL DILUTION

A. Objective:

The serial dilution analysis performed in association with the ICP procedure indicates whether significant physical or chemical interferences exist due to sample matrix effects.

B. Criteria:

If the analyte concentration is sufficiently high (concentration in the original sample is minimally a factor of 50 above the IDL), an analysis of a five fold dilution must agree within 10% Difference (%D) of the original results after correction for dilution.

C. Evaluation Procedure:

1. Check the raw data and recalculate the %D for one or more analytes using the following equation to verify that the dilution analysis results agree with results reported on Form 9 (Form IX-IN).

$$\%D = \frac{I - S}{I} \times 100$$

Where,

I = Initial Sample Result
S = Serial Dilution Result (Instrument Reading X 5)

2. Check the raw data for evidence of negative interference; i.e., results of the diluted sample significantly higher than results of the original sample.

D. Action:

1. When criteria are not met for an analyte, qualify the associated sample data for that analyte as estimated (J).

2. If evidence of negative interference is found, use professional judgement to qualify the data.

X. SAMPLE RESULT VERIFICATION

A. Objective:

The objective is to ensure that the reported quantitative results are appropriately and correctly calculated.

B. Criteria:

Analyte quantitation must be calculated according to appropriate SOW instructions or requirements.

C. Evaluation Procedure:

The raw data should be examined to verify the correct calculation of sample results reported by the laboratory. Digestion and distillation logs, instrument printouts, strip charts, etc. should be compared to the reported sample results.

1. Examine the raw data for any anomalies (e.g., baseline shifts, negative absorbances, omissions, illegibility, etc.).

2. Verify that there are no transcription or data reduction errors (e.g., dilutions, percent solids, sample weights) on one or more samples.

3. Verify that results fall within the linear range od the ICP (Form XII-IN) and within the calibration range for the non-ICP parameters.

4. Verify that sample results are > 5 X ICP-IDL if ICP analysis results are used for As, Tl, Se, or Pb.

Note: When the laboratory provides both ICP and GFAA results for an analyte in a sample and the concentration is > ICP-IDL, the results can assist in identifying quantitation problems.

D. Action:

If there are any discrepancies found, the laboratory may be contacted by the designated contact personnel to obtain resubmissions or additional information in an effort to resolve the discrepancy. If a discrepancy remains unresolved, the reviewer may determine that appropriate qualification of the data is warranted.

XI. FIELD DUPLICATES

A. Objective:

Field duplicate samples may be taken and submitted for analysis as an indication of overall precision. These analyses measure combined field sampling and laboratory analytical precision; therefore, the results may have greater variability than lab duplicates alone which measure only intra-laboratory and analytical method precision. It is also expected that soil field duplicate results will have greater variability than water than water matrices due to the difficult associated with collecting identical field samples and the natural non-homogeneity of soils.

B. Criteria:

There are no review criteria for field duplicate analyses comparability.

C. Evaluation Procedures:

Sample which are field duplicates should be identified using EPA Sample Traffic Reports other appropriate documents. The reviewer should compare the results reported for each sample and calculate the Relative Percent Difference, if appropriate.

D. Action:

Any evaluation of the field duplicates should be included with the review documentation attached to the IRDA form.

XII. OVERALL ASSESSMENT OF DATA FOR A CASE (OR SDG)

It is appropriate for the data reviewer to make professional judgments and express concerns and provide comments on the validity of the overall data for a Case or Sample Delivery Group. This is particularly true when there are several QC criteria out of specification. The additive nature of QC factors out of specification is difficult to assess in an objective manner, but the reviewer has a responsibility to inform the user concerning data quality and data limitations in order to assist that user in avoiding inappropriate use of the data, while not precluding any considering of the data at all. If qualifiers other than those used in this document are employed to describe or qualify the data, it is necessary to thoroughly document/explain/define the additional qualifiers used. The data reviewer would be greatly assisted in this data review effort if the data quality objectives for the project were provided.

In transmitting the reviewed data to the data user or other appropriate client, the IRDA cover form and supplementary documentation must be included.

APPENDIX A

REVISED DATA QUALIFIER DEFINITIONS

FOR INORGANIC DATA REVIEW

U - The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Instrument Detection Limit (IDL) for all analytes except Cyanide (CN) and Mercury (Hg). For CN and Hg, the associated value is the Contract Required Detection Limit (CRDL).

If a decision requires quantitation of the analyte below the associated numerical level, reanalysis or alternative methods should be considered.

J - The analyte was analyzed for and was positively identified, but the associated numerical value may not be consistent with the amount actually present in the environmental sample.

One or more of the following quality control criteria were not met:

- *Blank contamination: indicates possible high bias and/or false positives.*
- *Calibration range exceeded: indicates possible low bias.*
- *Holding times not met: indicates possible low bias and/or false negatives.*
- *Other QC outside control limits: bias not readily determined.*

R - The analyte was analyzed for, but the presence or absence of the analyte has not been verified. Resampling and reanalysis are necessary to confirm or deny the presence of the analyte.

The data are unusable for any purpose.

UJ - A combination of the "U" and the "J" qualifier. The analyte was analyzed for but was not detected above the level of the associated value. The associated value may not accurately or precisely represent the sample detection limit.

If a decision requires quantitation of the analyte close to the associated numerical level, reanalysis or alternative methods should be considered.

APPENDIX B

CONTRACT REQUIRED DETECTION LIMITS

FOR INORGANIC TARGET ANALYTES

(Inorganic Statement of Work 7/88)

ANALYTE	CRDL (ug/l)
Aluminum, Al	200
Antimony, Sb	60
Arsenic, As	10
Barium, Ba	200
Beryllium, Be	5
Cadmium, Cd	5
Calcium, Ca	5000
Chromium, Cr	10
Cobalt, Co	50
Copper, Cu	25
Iron, Fe	100
Lead, Pb	3
Magnesium, Mg	5000
Manganese, Mn	15
Mercury, Hg	0.2
Nickel, Ni	40
Potassium, K	5000
Selenium, Se	5
Silver, Ag	10
Sodium, Na	5000
Thallium, Tl	10
Vanadium, V	50
Zinc, Zn	20
Cyanide, CN	10

APPENDIX C

GLOSSARY OF TERMS AND ACRONYMS

Associated Samples	All samples processed and/or analyzed in association with a particular Quality Control Sample. For example: All samples in an analytical run initiated with an Initial Calibration Verification Sample are associated samples to that ICV; All samples of a given matrix digested and analyzed with a Laboratory Control Sample are associated samples to that LCS.
AA	Atomic Absorption
Calibration Curve	A plot of absorbance (or other measurement unit) versus concentration of prepared standards.
Case	A finite, usually predetermined, number of samples collected in a defined time period for a particular site. A Case consists of one or more Sample Delivery Groups.
CCB	Continuing Calibration Blank. A deionized water sample run immediately following the CCV, designed to detect carryover contamination.
CCS	Contract Compliance Screening. The process of inspection of analytical data submitted through the Contract Laboratory Program to assure adherence to contractual specifications for sample processing and analysis contained in the Statement of Work.
CCV	Continuing Calibration Verification. A standard solution analyzed at specified frequency during an analytical run to assure continued validity of the calibration curve under which the analyses are performed.
CLP	Contract Laboratory Program.
CRDL	Contract Required Detection Limit.
CV	Coefficient of Variation.
DPO	Deputy Project Officer.
EMSL/LV	Environmental Monitoring Systems Laboratory/Las Vegas. (P.O. Box 15027, Las Vegas, NV 89114)

Field Blank	Field blanks are intended to detect contaminants that may have been introduced in the field. Examples of field blanks are trip blanks, travel blanks, rinsate blanks, and decontamination blanks.
Field Duplicate	A duplicate sample set collected in the field intended to provide a measure of the overall precision of the sampling and analytical process.
GFAA	Graphite Furnace Atomic Absorption.
Holding Time	The elapsed time in days between the date of sample collection and the date of analysis. (Contractual holding time measures from the verified time of sample receipt to the date of analysis.)
ICB	A deionized water sample run immediately following the Initial Calibration Verification Sample at the beginning of an analytical run.
ICP	Inductively Coupled Plasma.
ICS	Interference Check Sample.
ICV	Initial Calibration Verification. A standard run immediately following instrument or method calibration as the first sample in an analytical run to confirm the validity of the calibration curve.
IDL	Instrument Detection Limit.
Initial Calibration	The establishment of a calibration curve with the appropriate number of standards and concentration range prior to the beginning of an analytical run.
IRDA	Inorganic Regional Data Assessment.
LCS	Laboratory Control Sample. An aqueous or solid sample of known composition, generally supplied by EPA, to be processed and analyzed in association with a defined set of field samples of unknown composition.
Matrix Spike (MS)	Introduction of a known concentration of one or more analytes into a submitted sample to provide information on the effect of the sample matrix on the sample preparation and measurement methodology.
MSA	Method of Standard Additions.

Post Digestion Spike	The addition of a known amount of analyte into a prepared sample after digestion. (Also identified as analytical spike or spike for GFAA analyses.)
QAC (QAO)	Quality Assurance Coordinator. (Quality Assurance Officer).
RPD	Relative Percent Difference. A measure of duplicate analysis precision.
RSCC	Regional Sample Control Center.
RSD	Relative Standard Deviation.
Serial Dilution	A sample analyzed at a specific dilution for comparison with the undiluted sample analysis to determine if significant chemical or physical interference exists due to sample matrix effects. (Run with ICP only.)
SDG	Sample Delivery Group. Defined by one of the following, whichever occurs first: o Case (if less than 20 samples of a single matrix type), o Each twenty samples within a Case. or o Each 14 day calendar period during which samples are received, beginning with the first sample in the Case or SDG. *At the option of the laboratory, samples may be assigned to SDGs by matrix; i.e., all waters in one SDG, all soils in another.*
SMO	Sample Management Office.
SOP	Standard Operating Procedure.
SOW	Statement of Work.
VTSR	Verified Time of Sample Receipt.

CHM 001 0421

Region_____

INORGANIC REGIONAL DATA ASSESSMENT

CASE NO._____ SITE_____

LABORATORY_____ NO. OF SAMPLES/ MATRIX_____

SDG NO._____ REVIEWER (IF NOT ESD)_____

SOW NO._____ REVIEWER'S NAME_____

DPO ACTION_____FYI_____ COMPLETION DATE_____

DATA ASSESSMENT SUMMARY

		ICP	AA	Hg	CYANIDE
1.	HOLDING TIMES				
2.	CALIBRATION				
3.	BLANKS				
4.	ICS				
5.	LCS				
6.	DUPLICATE ANALYSIS				
7.	MATRIX SPIKE				
8.	MSA				
9.	SERIAL DILUTION				
10.	SAMPLE VERIFICATION				
11.	OTHER QC				
12.	OVERALL ASSESSMENT				

O : Data had no problems/or qualified due to minor problems.
M : Data qualified due to major problems.
I : Data unacceptable.
X : Problems, but do not affect the data.

ACTION ITEMS_____

AREAS OF CONCERN_____

NOTABLE PERFORMANCE_____

APPENDIX E

DETERMINATION OF TOTAL ORGANIC CARBON IN SEDIMENT

July 27, 1988

PREPARED BY: Lloyd Kahn, Quality Assurance Specialist

AFFILIATION: U.S. Environmental Protection Agency, Region II
Environmental Services Division
Monitoring Management Branch
Edison, New Jersey 08837

DETERMINATION OF TOTAL ORGANIC CARBON IN SEDIMENT

1. Scope and Application

1.1 This method describes protocols for the determination of organic carbon in ocean sediments.

1.2 Although the detection limit may vary with procedure or instrument, a minimum reporting value of 100 mg/kg will be required for the ocean dumping/dredging program.

1.3 Several types of determinations, which are considered equivalent are presented.

1.4 Data are reported in mg/kg on a dry weight basis.

1.5 Wet combustion methods are not considered to be equivalent to the pyrolytic methods herein described.

2. Summary of Method

2.1 Inorganic carbon from carbonates and bicarbonates is removed by acid treatment.

2.2 The organic compounds are decomposed by pyrolysis in the presence of oxygen or air.

2.3 The carbon dioxide that is formed is determined by direct non-dispersive infrared detection, flame ionization gas chromatography after catalytic conversion of the carbon dioxide to methane; thermal conductivity gas chromatography, differential thermal conductivity detection by sequential removal of water and carbon dioxide; or thermal conductivity detection following removal of water with magnesium perchlorate.

2.4 Water content is determined on a separate portion of sediment.

3. Sample Handling and Preservation

3.1 Collect sediments in glass jars with Teflon or aluminum foil. Cool and maintain at 4°C. Analyse within 14 days.

4. Interferences

4.1 Volatile organics in the sediments may be lost in the decarbonation step resulting in a low bias.

4.2 Bacterial decomposition and volatilisation of the organic compounds are minimized by maintaining the sample at 4°C, analysing within the specified holding time, and analysing the wet sample.

5. Apparatus

5.1 Drying oven maintained at 103° to 105°C.

5.2 Analytical instrument options:

5.2.1 Perkin Elmer Model 240C Elemental Analyzer or equivalent.

5.2.1 1 In this instrument, the sample from Section 7.2 is pyrolysed under pure oxygen, water is removed by magnesium perchlorate and the carbon dioxide is removed by ascarite. The decrease in signal obtained by differential thermal conductivity detectors placed between the combustion gas stream before and after the ascarite tube is a measure of the organic carbon content.

5.2.2 Carlo Erba Model 1106 CHN Analyser, or equivalent.

5.2.2.1 In this apparatus, the sample is pyrolysed in a induction type furnace, and the resultant carbon dioxide is chromatographically separated and analysed by a differential thermal conductivity detector.

5.2.3 LECO Models WR12, WR112, or CR-12 carbon determinators, or Models 600 or 800 CHN analysers.

5.2.3.1 In the LECO WR-12, the sample is burned in high frequency induction furnace, the carbon dioxide is selectively adsorbed at room temperature in a molecular sieve. It is subsequently released by heating and is measured by a thermal conductivity detector. The WR-112 is an upgraded WR-12 employing microprocessor electronics and a printer to replace the electronic digital voltmeter.

5.2.3.2 In the LECO CR-12 carbon determinator, the sample is combusted in oxygen, moisture and dust are removed by appropriate traps and the carbon dioxide is measured by a selective, solid state, infrared detector. The signal from the detector is then processed by a microprocessor and the carbon content is displayed on a digital readout and recorded on an integral printer.

5.2.3.3 In the LECO CHN-600 and CHN-800 elemental analysers, the sample is burned under oxygen in a resistance furnace and the carbon dioxide is measured by a selective infrared detector.

5.2.4 Dohrman Model DC85 Digital High Temperature TOC Analyser.

5.2.4.1 In this instrument, the sample is burned in resistance furnace under oxygen, the interfering gases are removed by a sparger/scrubber system and the carbon dioxide is measured by a non-dispersive infrared detector and shown on a digital display in concentration units.

5.3 No specific analyzer is recommended as superior. The above listing is for information only and is not intended to restrict the use of other unlisted instruments capable of analysing TOC. The instruments to be used must have the following specifications:

5.3.1 A combustion boat which is heated in a stream of oxygen or air in a resistance or induction-type furnace to completely convert organic substances to CO_2 and water.

5.3.2 A means to physically or by measurement technique to seperate water and other interferants from CO_2.

5.3.3 A means to quantitatively determine CO_2 with adequate sensitivity (100 mg/kg), and precision (25% at the 95% confidence level as demonstrated by repetitive measurements of a well mixed ocean sediment sample).

5.4 A strip chart or other permanent recording device to document the analysis.

6. Reagents

6.1 Distilled water used in preparation of standards and for dilution of samples should be ultra pure to reduce the carbon concentration of the blank.

6.2 Potassium hydrogen phthalate, stock solution, 1000 mg carbon/liter: Dissolve 0.2128 g of potassium hydrogen phthalate (Primary Standard Grade) in distilled water and dilute to 100.0 ml.

NOTE 2: Sodium oxalate and acetic acid are not recommended as stock Solutions.

6.3 Potassium hydrogen phthalate, standard solutions: Prepare standard solutions from the stock solution by dilution with distilled water.

6.4 Phosphoric acid solution, 1:1 by volume.

7. Procedure

7.1 Weigh the well mixed sample (up to 500 mg) into the combustion boat or cup. Add 1:1 phosphoric acid drop wise until effervescence stops. Heat to 75°C.

NOTE: This procedure will convert inorganic carbonates and bicarbonates to carbon dioxide and eliminate it from the sample.

7.2 Analyse the residue according to the instrument manufacturer's instructions.

7.3 Determine percent residue on a separate sample aliquot as follows:

7.3.1 Heat a clean 25 ml beaker at 103° to 105°C for one hour. Cool in desiccator, weigh to the nearest mg and store in desiccator until use.

7.3.2 Add 1 g, weighed to the nearest mg, of an aliquot of the well-mixed sample .

7.3.3 Dry and heat in the 103° to 105°C oven for one hour. Cool in desiccator. Weigh to the nearest mg.

8. Calibration

8.1 Follow instrument manufacturer's instructions.

8.2 Prepare calibration curve plotting mg carbon vs. instrument response. using four standards and a blank covering the analytical range of interest.

9. Precision and Accuracy

9.1 The precision and accuracy will differ with the various instruments and matrices and must be determined by the laboratories reporting data. To initiate a control chart, a representative sample of well mixed sediment should be analyzed 15 times to determine the analytical precision. Set up a control chart showing 3 times the standard deviation limits for precision.

9.2 Subsequently during analysis of environmental samples, take one sample per batch of 20 or less and run in quadruplicate. Calculate standard deviation and report with initial control chart data.

9.3 If the sample being run in quadruplicate exceeds the 3 standard deviation limit, identify error and rerun environmental samples in that batch along with the quadruplicate sample.

ATTACHMENT 1
SOP NO. HW-6

TOTAL REVIEW

CLP DATA ASSESSMENT

Functional Guidelines for Evaluating Organics Analysis

Case No. _____SDG No. _____LABORATORY_____. SITE

DATA ASSESSMENT:

The current functional guidelines (1988) for evaluating organic data have been applied.

All data are valid and acceptable except those analytes which have been qualified with a "J" (estimated), "U" (non-detects), "R" (unusable),or "JN" (presumptive evidence for the presence of the material at an estimated value). All action is detailed on the attached sheets.

Two facts should be noted by all data users. First, the "R" flag means that the associated value is unusable. In other words, due to significant QC problems the analysis is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on data tables because they cannot be relied upon, even as a last resort. The second fact to keep in mind is that no compound concentration, even if it has passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data but any value potentially contains error.

Reviewer's Signature:_____ **Date:** __/__/19__

Verified By:_____ **Date:** _/_/19

DATA ASSESSMENT:

1. HOLDING TIME:

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detects (sample quantitation limits) will be flagged as estimated, "J", or unusable, "R", if the holding times are grossly exceeded.

The following action was taken in the samples and analytes shown due to excessive holding time.

ATTACHMENT 1
SOP NO. HW-6

DATA ASSESSMENT:

2. BLANK CONTAMINATION:

Quality assurance (QA) blanks, i.e., method, trip field, rinse and water blanks are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Trip blanks measure cross-contamination of samples during shipment. Field blanks measure cross- contamination of samples during field operations. If the concentration of the analyte is less than 5 times the blank contaminant level (10 times for the common contaminants), the analytes are qualified as non- detects, "U". The following analytes in the samples shown were qualified with "U" for these reasons:

A) Method blank contamination

B) Field or rinse blank contamination ("water blanks" or "distilled water blanks" are validated like any other sample)

C) Trip blank contamination

ATTACHMENT 1
SOP NO. HW-6

DATA ASSESSMENT:

3. MASS SPECTROMETER TUNING:

Tuning and performance criteria are established to ensure adequate mass resolution, proper identification of compounds, and to some degree, sufficient instrument sensitivity. These criteria are not sample specific. Instrument performance is determined using standard materials. Therefore, these criteria should be met in all circumstances. The tuning standard for volatile organics is bromofluorobenzene (BFB) and for semi-volatiles is decafluorotriphenyl- phosphine (DFTPP).

If the mass calibration is in error, all associated data will be classified as unusable, "R".

DATA ASSESSMENT:

4. CALIBRATION:

Satisfactory instrument calibration is established to ensure that the instrument is capable of producing acceptable quantitative data. An initial calibration demonstrates that the instrument is capable of giving acceptable performance at the beginning of an experimental sequence. The continuing calibration checks document that the instrument is giving satisfactory daily performance.

A) RESPONSE FACTOR:

The response factor measures the instrument's response to specific chemical compounds. The response factor for the Target Compound List (TCL) must be $\geq$ 0.05 in both the initial and continuing calibrations. A value < 0.05 indicates a serious detection and quantitation problem (poor sensitivity). Analytes detected in the sample will be qualified as estimated, "J". All non-detects for that compound will be rejected ("R").

DATA ASSESSMENT:

5. CALIBRATION:

A) PERCENT RELATIVE STANDARD DEVIATION (%RSD) AND PERCENT DIFFERENCE (%D):

Percent RSD is calculated from the initial calibration and is used to indicate the stability of the specific compound response factor over increasing concentration. Percent D compares the response factor of the continuing calibration check to the mean response factor (RRF) from the initial calibration. Percent D is a measure of the instrument's daily performance. Percent RSD must be <30% and %D must be <25%. A value outside of these limits indicates potential detection and quantitation errors. For these reasons, all positive results are flagged as estimated, "J" and non-detects are flagged "UJ" (if %D or RSD >50%). If there is a gross deviation of %RSD and %D, the non-detects may be rejected ("R").

For the PCB/PESTICIDE fraction, %RSD for aldrin, endrin, DDT, and dibutylchlorendate must not exceed 10%. Percent D must be within 15% on the quantitation column and 20% on the confirmation column.

DATA ASSESSMENT:

6. SURROGATES:

All samples are spiked with surrogate compounds prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate concentrations were outside contract specifications, qualifications were applied to the samples and analytes as shown below.

DATA ASSESSMENT:

7. INTERNAL STANDARDS PERFORMANCE:

Internal standard (IS) performance criteria ensure that the GC/MS sensitivity and response are stable during every experimental run. The internal standard area count must not vary by more than a factor of 2 (-50% to +100%) from the associated continuing calibration standard. The retention time of the internal standard must not vary more than ±30 seconds from the associated continuing calibration standard. If the area count is outside the (-50% to +100%) range of the associated standard, all of the positive results for compounds quantitated using that IS are qualified as estimated, "J", and all non-detects as "UJ", or "R" if there is a severe loss of sensitivity.

If an internal standard retention time varies by more than 30 seconds, the reviewer will use professional judgment to determine either partial or total rejection of the data for that sample fraction.

DATA ASSESSMENT:

8. COMPOUND IDENTIFICATION:

A) VOLATILE AND SEMI-VOLATILE FRACTIONS:

TCL compounds are identified on the GC/MS by using the analyte's relative retention time (RRT) and by comparison to the ion spectra obtained from known standards. For the results to be a positive hit, the sample peak must be within ± 0.06 RRT units of the standard compound and have an ion spectra which has a ratio of the primary and secondary m/e intensities within 20% of that in the standard compound. For the tentatively identified compounds (TIC) the ion spectra must match accurately. In the cases where there is not an adequate ion spectrum match, the laboratory may have provided false positive identifications.

B) PESTICIDE FRACTION:

The retention times of reported compounds must fall within the calculated retention time windows for the two chromatographic columns and a GC/MS confirmation is required if the concentration exceeds 10 ng/ml in the final sample extract.

DATA ASSESSMENT:

9. MATRIX SPIKE/SPIKE DUPLICATE, MS/MSD:

The MS/MSD data are generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MSD may be used in conjunction with other QC criteria for some additional qualification of the data.

DATA ASSESSMENT:

10. OTHER QC DATA OUT OF SPECIFICATION:

11. SYSTEM PERFORMANCE AND OVERALL ASSESSMENT (continued on next page if necessary):

12. CONTRACT PROBLEMS____NON-COMPLIANCE:

13. This package contains re-extraction, re-analysis or dilution. Upon reviewing the QA results, the following form I(s) are identified to be used.

The chain-of-custody record includes sample descriptions and date of sampling. Although sampling date is not addressed by CLP contract requirements, the reviewer must take into account lag time between sampling and shipping when assessing sample holding times.

INORGANICS DATA REVIEW PROCEDURE

The requirements to be checked in data validation are listed below. (The notation "CCS" indicates that the contractual requirements for these items will also be checked by CCS. CCS requirements are not always the same as the data review criteria.)

I. Holding Times (CCS-Lab holding times only)

II. Calibration

- Initial (CCS)
- Initial and Continuing Calibration Verification (CCS)

III. Blanks (CCS)

IV. ICP Interference Check Samples (CCS)

V. Laboratory Control Sample (CCS)

VI. Duplicate Sample (CCS)

VII. Matrix Spike Sample (CCS)

VIII. Furnace Atomic Absorption QC (CCS)

IX. ICP Serial Dilution (CCS)

X. Sample Result Verification (CCS)

XI. Field Duplicates

XII. Overall Data Assessment

I. HOLDING TIMES

A. Objective:

The objective is to ascertain the validity of analytical results based on the holding time of the sample from the date of collection to the date of analysis.

Note: For data review and assessment purposes, the holding time is based on the elapsed time between the date of sample collection and date of analysis, rather than the elapsed time between the verified time of sample receipt (VTSR) and the date of analysis as allowed by SOW 7/88. It is thus a technical evaluation, not a contract requirement.

B. Criteria:

Technical requirements (as opposed to contractual) for sample holding times have only been established for water matrices. The following holding time and preservation requirements were promulgated under 40 CFR 136 (Clean Water Act) and are also found in volume 49, Number 209, page 43260, of the Federal Register issued on October 26, 1984.

METALS:	6 months; preserved to pH < 2
MERCURY:	28 days; preserved to pH < 2
CYANIDE:	14 days; preserved to pH > 12

C: Evaluation Procedure:

Actual holding times are established by comparing the sampling date on the EPA Sample Traffic Report with the dates of analysis on the analysis run logs (Form 14 (XIV-IN) of SOW 7/88). Examine the digestion and/or distillation logs included with the raw data to determine if samples had been preserved at the proper pH.

Analyte Holding Time (days) = Anal. Date - Coll. Date

D. Action:

1. If 40 CFR criteria for holding times or preservation are not met for a specific analyte in a water matrix, qualify all water matrix results for that analyte which are greater than the Instrument Detection Limit (> IDL) as estimated (J) and results less than the Instrument Detection Limit (< IDL) as estimated (UJ). For Cyanide and Mercury the appropriate detection limit for this judgement is the Contract Required Detection Limit (CRDL) rather than the Instrument Detection Limit (IDL).

2. If technical holding times for a specific analyte are exceeded, the reviewer may use professional judgement to evaluate the reliability of the data and the probable effects of additional storage on the analytical results. The expected bias would be low and the reviewer may determine that results < IDL (< CRDL for CN and Hg) are unusable (R).

3. Due to limited information concerning holding times for soil samples, it is left to the discretion of the data reviewer whether to apply 40 CFR water holding time criteria to soil samples. If the data are qualified when water holding time criteria are applied to soil samples, it must be clearly documented in the review.

Note: Contractual holding time criteria based on VTSR and analysis date are equally applicable to water and soil samples and will be subject to CCS review. Preservation requirements for water samples are not applicable to soil samples under any conditions.

II. CALIBRATION

A. Objective:

Requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration verification demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration remains valid throughout the course of the analytical run.

B. Criteria:

1. Initial Calibration

Instruments must be calibrated daily and each time the instrument is set up.

a. ICP Analysis

A calibration blank and at least one standard must be used in establishing the calibration curve.

b. Atomic Absorption Analysis (AA) (other than Mercury)

(1). A blank and at least three standards must be used in establishing the calibration curve. One of the standards must be at the Contract Required Detection Limit (CRDL).

(2). The correlation coefficient should be $\geq$ 0.995.

2. Check ICS raw data for results with an absolute value > IDL for those analytes which are not intended to be present in the ICS solution.

D. Action:

1. For samples with concentrations of Al, Fe, Ca, or Mg which are comparable to or greater than their respective levels in the Interference Check Sample:

   a. If the ICS recovery for an analyte is > 120% and the analyte results are < IDL in the associated samples, the data are acceptable for use.

   b. If the ICS recovery for an analyte is > 120%, or falls between 50 - 79 %, and the analyte results are > IDL in the associated samples, qualify the affected data as estimated (J).

   c. If the ICS recovery for an analyte falls between 50 - 79% and the analyte results are < IDL in the associated sample, the possibility of false negatives exists. Qualify the affected analyte data for these samples as estimated (UJ).

   d. If ICS recovery for an analyte is < 50%, qualify the affected analyte data for the associated samples as unusable (R).

   Note: *If possible, indicate the bias for the estimated sample results in the review.*

2. If results > IDL are observed for elements which are not intended to be present in the EPA provided ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with comparable or higher levels of interferents and with suspect analyte concentrations that approximate those levels found in the ICS (false positives), qualify sample results > IDL as estimated (J).

3. If negative results are observed for analytes that are not intended to be present in the EPA ICS solutions, and their absolute value is > IDL, the possibility of false negatives in the samples exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with comparable or higher levels of interferents, qualify results for the affected analytes < IDL as estimated (UJ).

4. In general, the sample data can be accepted if the concentrations of Al, Fe, Ca, and Mg in the sample are found to be less than or equal to their respective concentrations in the ICS. If these elements are present at concentrations greater than the level in the ICS, or other elements are present in the sample at > 10 mg/l, the reviewer should investigate the possibility of other interference effects by using Table 2 on page D-22 of SOW 7/88. These analyte concentration equivalents presented in the Table should be considered

only as estimated values, since the exact value of any analytical system is instrument specific. Therefore, estimate the concentration produced by an interfering element. If the estimate is > 2 X CRDL and also greater than 10% of the reported concentration of the affected element, qualify the affected results as estimated (J).

V. LABORATORY CONTROL SAMPLE (LCS)

A. Objective:

The laboratory control sample serves as a monitor of the overall performance of all steps in the analysis, including the sample preparation.

B. Criteria:

1. All aqueous LCS results must fall within the control limits of 80 - 120 %R, except for Sb and Ag which have no control limits.

2. All solid LCS results must fall within the control limits established for each element by EPA. This information is available from EMSL-LV.

C. Evaluation Procedure:

1. Review Form 7 (Form VII-IN) and verify that results fall within the control limits.

2. Check the raw data (ICP printout, strip charts, bench sheets, etc.) to verify the reported recoveries on Form 7. Recalculate one or more of the recoveries (%R) using the following equation:

$$\%R = \frac{\text{LCS Found}}{\text{LCS True}} \times 100$$

Where,

LCS Found = *concentration (in ug/l for aqueous; ug/kg for solid) of each analyte measured in the analysis of the LCS solution.*

LCS True = *stated true value concentration (in ug/l for aqueous; ug/kg for solid) of each analyte in the LCS source.*

D. Action:

1. Aqueous LCS:

a. If the LCS recovery for an analyte falls within the range of 50 - 79% or > 120%, qualify associated sample results with affected analyte values > IDL as estimated (J).

b. If the LCS recovery for an analyte is > 120%, associated sample results with affected analyte values < IDL are acceptable.

c. If the LCS recovery for an analyte falls within the range of 50 - 79%, qualify associated sample results with affected analyte values < IDL as estimated (UJ).

d. If the LCS recovery for an analyte is < 50%, qualify associated sample results for the affected analyte as unusable (R).

2. Solid LCS:

a. If the LCS recovery for an analyte falls outside its established EPA control limit, qualify associated sample results with affected analyte values > IDL as estimated (J).

b. If the LCS recovery for an analyte is higher than its established EPA control limit, associated sample results with affected analyte values < IDL are acceptable.

c. If the LCS recovery for an analyte is lower than its established EPA control limit, qualify associated sample results with affected analyte values < IDL as estimated (UJ).

VI. DUPLICATE SAMPLE ANALYSIS

A. Objective:

Duplicate analyses are indicators of a laboratory's analytical precision based on each sample matrix.

B. Criteria:

1. Samples identified as Field Blanks cannot be used for duplicate analyses.

2. A control limit of ± 20% (± 35% for soil) for the Relative Percent Difference (RPD) shall be used for sample values > 5X CRDL.

3. A control limit of ± CRDL (± 2X CRDL) shall be used for sample values < 5X CRDL, including the case when only <u>one</u> of the duplicate sample values is < 5X CRDL.

C. Evaluation Procedure:

1. Review Form 6 (Form VI-IN) and verify that results fall within the control limits.

2. Check the raw data and recalculate one or more RPD using the following equation to verify that results have been correctly reported on Form 6.

$$RPD = \frac{|S - D|}{(S+D)/2} \times 100$$

Where,

S = First Sample Value (original)

D = Second Sample Value (duplicate)

3. Verify that the Field Blank was not used for duplicate analysis.

D. Action:

1. If duplicate analysis results for a particular analyte fall outside the appropriate control limits, qualify the results for that analyte in all associated samples of the same matrix as estimated (J).

2. If the Field Blank was used for duplicate analysis, all other QC data must be carefully checked and professional judgement exercised when evaluating the data.

Note: This information must be included on the IRDA form.

VII MATRIX SPIKE SAMPLE ANALYSIS

A. Objective:

The matrix spike sample analysis provides information about the effect of each sample matrix on the digestion and measurement methodology.

B. Criteria:

1. Samples identified as Field Blanks must not be used for matrix spike sample analysis.

2. Spike recovery (%R) must be within the limits of 75 - 125%. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more. If the latter condition exists for an analyte, matrix spike recovery cannot be used to qualify the associated sample data for that analyte.

C. Evaluation Procedure:

1. Review Form 5 (Form V-IN) and verify that results fall within the specified control limits.

2. Check raw data and recalculate one or more %R using the following equation to verify that results were correctly reported on Form 5.

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

Where,

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

3. Verify that the Field Blank was not used for matrix spike sample analysis.

D. Action:

1. If the spike recovery for an analyte is > 125% and reported associated sample results for that analyte are < IDL (< CRDL for CN or Hg), the affected data are acceptable for use.

2. If the spike recovery for an analyte is > 125% or < 75% and reported sample results for that analyte are > IDL, qualify the affected data for that analyte in the associated samples as estimated (J).

3. If the spike recovery for an analyte falls within the range of 30 - 74% and reported sample results for that analyte are < IDL, qualify the affected data in the associated samples as estimated (UJ).

4. If spike recovery results for an analyte are < 30% and the reported sample results for that analyte are < IDL, qualify the affected data in the associated samples as unusable (R).

5. If the Field Blank was used for matrix spike analysis, all other QC data must be carefully checked and professional judgement exercised when evaluating the data. This information must be included on the IRDA form.

Note: For all analytes except Ag, Hg, and those elements analyzed by GFAA, if the matrix spike recovery does not meet criteria, a post digestion spike is required; The results of post digestion spike recovery are not used to qualify the data; however, the information must be included in the IRDA report.

VIII. FURNACE ATOMIC ABSORPTION QC

A. Objective:

Duplicate injections and furnace post digestion spikes establish the precision and accuracy of the individual analytical determinations. In some instances, Method of Standard Additions may be required.

B. Criteria:

1. For sample concentrations > CRDL, duplicate injections must agree within ± 20% Relative Standard Deviation (RSD) or Coefficient of Variation (CV). Otherwise, the sample must be rerun once (at least two additional injections).

2. Analytical (post digestion) spike recovery must be > 85% and < 115%.

3. The Furnace Atomic Absorption Scheme must be followed as described in SOW 7/88, p. E 16-18.

C. Evaluation Procedure:

1. Check raw data to verify that duplicate injections agree within ±20% RSD (or CV) for sample concentrations > CRDL.

2. Review Furnace AA raw data to verify that the Furnace Atomic Absorption Scheme has been followed.

D. Action:

1. If duplicate injections are outside the ± 20% RSD (or CV) limits and the sample has not been rerun once as required, qualify the data as estimated (J). If the rerun sample results are outside the ± 20% limits as well, qualify the sample data as estimated (J).

2. If the analytical (post digestion) spike recovery is < 40%, qualify results > IDL as estimated (J).

3. If the analytical (post digestion) spike recovery is ≥ 10%, but <40%, qualify results < IDL as estimated (UJ).

4. If the analytical (post digestion) spike recovery is < 10%, qualify results < IDL as unusable (R).

5. If sample absorbance is < 50% of the analytical (post digestion) spike absorbance, then: If the furnace analytical (post digestion) spike is not within 85 - 115 %, qualify the sample results > IDL as estimated; qualify the results < IDL as estimated (UJ).

6. If Method of Standard Additions (MSA) is indicated as being required but has not been performed, qualify the data as estimated (J).

7. If any of the samples analyzed by MSA were not spiked at the appropriate levels, qualify the data as estimated (J).

8. If the MSA correlation coefficient is < 0.995, qualify the data as estimated (J).

IX. ICP SERIAL DILUTION

A. Objective:

The serial dilution analysis performed in association with the ICP procedure indicates whether significant physical or chemical interferences exist due to sample matrix effects.

B. Criteria:

If the analyte concentration is sufficiently high (concentration in the original sample is minimally a factor of 50 above the IDL), an analysis of a five fold dilution must agree within 10% Difference (%D) of the original results after correction for dilution.

C. Evaluation Procedure:

1. Check the raw data and recalculate the %D for one or more analytes using the following equation to verify that the dilution analysis results agree with results reported on Form 9 (Form IX-IN).

$$\%D = \frac{I - S}{I} \times 100$$

Where,

I = Initial Sample Result
S = Serial Dilution Result (Instrument Reading X 5)

2. Check the raw data for evidence of negative interference; i.e., results of the diluted sample significantly higher than results of the original sample.

D. Action:

1. When criteria are not met for an analyte, qualify the associated sample data for that analyte as estimated (J).

2. If evidence of negative interference is found, use professional judgement to qualify the data.

X. SAMPLE RESULT VERIFICATION

A. Objective:

The objective is to ensure that the reported quantitative results are appropriately and correctly calculated.

B. Criteria:

Analyte quantitation must be calculated according to appropriate SOW instructions or requirements.

C. Evaluation Procedure:

The raw data should be examined to verify the correct calculation of sample results reported by the laboratory. Digestion and distillation logs, instrument printouts, strip charts, etc. should be compared to the reported sample results.

1. Examine the raw data for any anomalies (e.g., baseline shifts, negative absorbances, omissions, illegibility, etc.).

2. Verify that there are no transcription or data reduction errors (e.g., dilutions, percent solids, sample weights) on one or more samples.

3. Verify that results fall within the linear range od the ICP (Form XII-IN) and within the calibration range for the non-ICP parameters.

4. Verify that sample results are > 5 X ICP-IDL if ICP analysis results are used for As, Tl, Se, or Pb.

Note: When the laboratory provides both ICP and GFAA results for an analyte in a sample and the concentration is > ICP-IDL, the results can assist in identifying quantitation problems.

D. Action:

If there are any discrepancies found, the laboratory may be contacted by the designated contact personnel to obtain resubmissions or additional information in an effort to resolve the discrepancy. If a discrepancy remains unresolved, the reviewer may determine that appropriate qualification of the data is warranted.

XI. FIELD DUPLICATES

A. Objective:

Field duplicate samples may be taken and submitted for analysis as an indication of overall precision. These analyses measure combined field sampling and laboratory analytical precision; therefore, the results may have greater variability than lab duplicates alone which measure only intra-laboratory and analytical method precision. It is also expected that soil field duplicate results will have greater variability than water than water matrices due to the difficult associated with collecting identical field samples and the natural non-homogeneity of soils.

B. Criteria:

There are no review criteria for field duplicate analyses comparability.

C. Evaluation Procedures:

Sample which are field duplicates should be identified using EPA Sample Traffic Reports other appropriate documents. The reviewer should compare the results reported for each sample and calculate the Relative Percent Difference, if appropriate.

D. Action:

Any evaluation of the field duplicates should be included with the review documentation attached to the IRDA form.

XII. OVERALL ASSESSMENT OF DATA FOR A CASE (OR SDG)

It is appropriate for the data reviewer to make professional judgments and express concerns and provide comments on the validity of the overall data for a Case or Sample Delivery Group. This is particularly true when there are several QC criteria out of specification. The additive nature of QC factors out of specification is difficult to assess in an objective manner, but the reviewer has a responsibility to inform the user concerning data quality and data limitations in order to assist that user in avoiding inappropriate use of the data, while not precluding any considering of the data at all. If qualifiers other than those used in this document are employed to describe or qualify the data, it is necessary to thoroughly document/explain/define the additional qualifiers used. The data reviewer would be greatly assisted in this data review effort if the data quality objectives for the project were provided.

In transmitting the reviewed data to the data user or other appropriate client, the IRDA cover form and supplementary documentation must be included.

APPENDIX A

REVISED DATA QUALIFIER DEFINITIONS

FOR INORGANIC DATA REVIEW

U - The analyte was analyzed for but was not detected above the level of the associated value. The associated value is the Instrument Detection Limit (IDL) for all analytes except Cyanide (CN) and Mercury (Hg). For CN and Hg, the associated value is the Contract Required Detection Limit (CRDL).

If a decision requires quantitation of the analyte below the associated numerical level, reanalysis or alternative methods should be considered.

J - The analyte was analyzed for and was positively identified, but the associated numerical value may not be consistent with the amount actually present in the environmental sample.

One or more of the following quality control criteria were not met:

- *Blank contamination: indicates possible high bias and/or false positives.*
- *Calibration range exceeded: indicates possible low bias.*
- *Holding times not met: indicates possible low bias and/or false negatives.*
- *Other QC outside control limits: bias not readily determined.*

R - The analyte was analyzed for, but the presence or absence of the analyte has not been verified. Resampling and reanalysis are necessary to confirm or deny the presence of the analyte.

The data are unusable for any purpose.

UJ - A combination of the "U" and the "J" qualifier. The analyte was analyzed for but was not detected above the level of the associated value. The associated value may not accurately or precisely represent the sample detection limit.

If a decision requires quantitation of the analyte close to the associated numerical level, reanalysis or alternative methods should be considered.

APPENDIX B

CONTRACT REQUIRED DETECTION LIMITS

FOR INORGANIC TARGET ANALYTES

(Inorganic Statement of Work 7/88)

ANALYTE	CRDL (ug/l)
Aluminum, Al	200
Antimony, Sb	60
Arsenic, As	10
Barium, Ba	200
Beryllium, Be	5
Cadmium, Cd	5
Calcium, Ca	5000
Chromium, Cr	10
Cobalt, Co	50
Copper, Cu	25
Iron, Fe	100
Lead, Pb	3
Magnesium, Mg	5000
Manganese, Mn	15
Mercury, Hg	0.2
Nickel, Ni	40
Potassium, K	5000
Selenium, Se	5
Silver, Ag	10
Sodium, Na	5000
Thallium, Tl	10
Vanadium, V	50
Zinc, Zn	20
Cyanide, CN	10

APPENDIX C

GLOSSARY OF TERMS AND ACRONYMS

Associated Samples	All samples processed and/or analyzed in association with a particular Quality Control Sample. For example: All samples in an analytical run initiated with an Initial Calibration Verification Sample are associated samples to that ICV; All samples of a given matrix digested and analyzed with a Laboratory Control Sample are associated samples to that LCS.
AA	Atomic Absorption
Calibration Curve	A plot of absorbance (or other measurement unit) versus concentration of prepared standards.
Case	A finite, usually predetermined, number of samples collected in a defined time period for a particular site. A Case consists of one or more Sample Delivery Groups.
CCB	Continuing Calibration Blank. A deionized water sample run immediately following the CCV, designed to detect carryover contamination.
CCS	Contract Compliance Screening. The process of inspection of analytical data submitted through the Contract Laboratory Program to assure adherence to contractual specifications for sample processing and analysis contained in the Statement of Work.
CCV	Continuing Calibration Verification. A standard solution analyzed at specified frequency during an analytical run to assure continued validity of the calibration curve under which the analyses are performed.
CLP	Contract Laboratory Program.
CRDL	Contract Required Detection Limit.
CV	Coefficient of Variation.
DPO	Deputy Project Officer.
EMSL/LV	Environmental Monitoring Systems Laboratory/Las Vegas. (P.O. Box 15027, Las Vegas, NV 89114)

AQUEOUS SAMPLING PROCEDURES

1. Aqueous Matrix Sampled:

Potable Well ☐ Groundwater ☐ Surface Water ☐ Leachate Runoff ☐ Storm Sewer ☐ Sanitary Sewer ☐

Other: _____

2. Type of Sample: Grab ☐ Composite ☐ If Composite, # samples/composite: _____

3. Was the VOA sample collected first: Yes ☐ No ☐ N/A ☐

4. Type of Sampling Equipment:

	Material of Construction: Stainless Steel	Teflon	Glass	Other
Bailer	☐	☐	☐	_____
Bladder Pump	☐	☐	☐	_____
Pond Sampler	☐	☐	☐	_____
Coliwasa	☐	☐	☐	_____
Kemmerer Depth Sampler	☐	☐	☐	_____
Wheaton Dip Sampler	☐	☐	☐	_____
Thief Sampler	☐	☐	☐	_____
Other: _____	☐	☐	☐	_____

5. Type of leader line that comes in contact with the well water:

Teflon ☐ Teflon Coated Stainless Steel ☐ N/A ☐ Other: _____

6. Length of the leader line: _____

7. Was the Sampling Equipment Dedicated: Yes ☐ _____ No ☐ _____

8. Was the Sampling Equipment: Lab Decontaminated ☐ _____ Field Decontaminated ☐ _____

9. Was the sampling equipment decontaminated according to standard NJDEP/NESH procedures: Yes ☐ No ☐ N/A ☐

If No, method of decontamination: _____

10. Was the decontamination area located away from the source of contamination: Yes ☐ No ☐ N/A ☐

11. Are disposable gloves worn and changed between each sample location: Yes ☐ No ☐

12. Auditor's Comments: _____

_____

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_____

_____

_____

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Field Blank	Field blanks are intended to detect contaminants that may have been introduced in the field. Examples of field blanks are trip blanks, travel blanks, rinsate blanks, and decontamination blanks.
Field Duplicate	A duplicate sample set collected in the field intended to provide a measure of the overall precision of the sampling and analytical process.
GFAA	Graphite Furnace Atomic Absorption.
Holding Time	The elapsed time in days between the date of sample collection and the date of analysis. (Contractual holding time measures from the verified time of sample receipt to the date of analysis.)
ICB	A deionized water sample run immediately following the Initial Calibration Verification Sample at the beginning of an analytical run.
ICP	Inductively Coupled Plasma.
ICS	Interference Check Sample.
ICV	Initial Calibration Verification. A standard run immediately following instrument or method calibration as the first sample in an analytical run to confirm the validity of the calibration curve.
IDL	Instrument Detection Limit.
Initial Calibration	The establishment of a calibration curve with the appropriate number of standards and concentration range prior to the beginning of an analytical run.
IRDA	Inorganic Regional Data Assessment.
LCS	Laboratory Control Sample. An aqueous or solid sample of known composition, generally supplied by EPA, to be processed and analyzed in association with a defined set of field samples of unknown composition.
Matrix Spike (MS)	Introduction of a known concentration of one or more analytes into a submitted sample to provide information on the effect of the sample matrix on the sample preparation and measurement methodology.
MSA	Method of Standard Additions.

Post Digestion Spike	The addition of a known amount of analyte into a prepared sample after digestion. (Also identified as analytical spike or spike for GFAA analyses.)
QAC (QAO)	Quality Assurance Coordinator. (Quality Assurance Officer).
RPD	Relative Percent Difference. A measure of duplicate analysis precision.
RSCC	Regional Sample Control Center.
RSD	Relative Standard Deviation.
Serial Dilution	A sample analyzed at a specific dilution for comparison with the undiluted sample analysis to determine if significant chemical or physical interference exists due to sample matrix effects. (Run with ICP only.)
SDG	Sample Delivery Group. Defined by one of the following, whichever occurs first: o Case (if less than 20 samples of a single matrix type), o Each twenty samples within a Case. or o Each 14 day calendar period during which samples are received, beginning with the first sample in the Case or SDG. *At the option of the laboratory, samples may be assigned to SDGs by matrix; i.e., all waters in one SDG, all soils in another.*
SMO	Sample Management Office.
SOP	Standard Operating Procedure.
SOW	Statement of Work.
VTSR	Verified Time of Sample Receipt.

Region_____

INORGANIC REGIONAL DATA ASSESSMENT

CASE NO._____ SITE_____

LABORATORY_____ NO. OF SAMPLES/ MATRIX_____

SDG NO._____ REVIEWER (IF NOT ESD)_____

SDN NO._____ REVIEWER'S NAME_____

DPO ACTION_____FYI_____ COMPLETION DATE_____

DATA ASSESSMENT SUMMARY

		ICP	AA	Hg	CYANIDE
1.	HOLDING TIMES				
2.	CALIBRATION				
3.	BLANKS				
4.	ICS				
5.	LCS				
6.	DUPLICATE ANALYSIS				
7.	MATRIX SPIKE				
8.	MSA				
9.	SERIAL DILUTION				
10.	SAMPLE VERIFICATION				
11.	OTHER QC				
12.	OVERALL ASSESSMENT				

O : Data had no problems/or qualified due to minor problems.
M : Data qualified due to major problems.
I : Data unacceptable.
X : Problems, but do not affect the data.

ACTION ITEMS_____

_____

_____

AREAS OF CONCERN_____

_____

_____

NOTABLE PERFORMANCE_____

_____

_____

APPENDIX E

DETERMINATION OF TOTAL ORGANIC CARBON IN SEDIMENT

July 27, 1988

PREPARED BY: Lloyd Kahn, Quality Assurance Specialist

AFFILIATION: U.S. Environmental Protection Agency, Region II
Environmental Services Division
Monitoring Management Branch
Edison, New Jersey 08837

DETERMINATION OF TOTAL ORGANIC CARBON IN SEDIMENT

1. Scope and Application

1.1 This method describes protocols for the determination of organic carbon in ocean sediments.

1.2 Although the detection limit may vary with procedure or instrument, a minimum reporting value of 100 mg/kg will be required for the ocean dumping/dredging program.

1.3 Several types of determinations, which are considered equivalent are presented.

1.4 Data are reported in mg/kg on a dry weight basis.

1.5 Wet combustion methods are not considered to be equivalent to the pyrolytic methods herein described.

2. Summary of Method

2.1 Inorganic carbon from carbonates and bicarbonates is removed by acid treatment.

2.2 The organic compounds are decomposed by pyrolysis in the presence of oxygen or air.

2.3 The carbon dioxide that is formed is determined by direct non-dispersive infrared detection, flame ionization gas chromatography after catalytic conversion of the carbon dioxide to methane; thermal conductivity gas chromatography, differential thermal conductivity detection by sequential removal of water and carbon dioxide; or thermal conductivity detection following removal of water with magnesium perchlorate.

2.4 Water content is determined on a separate portion of sediment.

3. Sample Handling and Preservation

3.1 Collect sediments in glass jars with Teflon or aluminum foil. Cool and maintain at 4°C. Analyse within 14 days.

4. Interferences

4.1 Volatile organics in the sediments may be lost in the decarbonation step resulting in a low bias.

4.2 Bacterial decomposition and volatilization of the organic compounds are minimized by maintaining the sample at 4°C, analysing within the specified holding time, and analysing the wet sample.

5. Apparatus

5.1 Drying oven maintained at 103° to 105°C.

5.2 Analytical instrument options:

5.2.1 Perkin Elmer Model 240C Elemental Analyzer or equivalent.

5.2.1.1 In this instrument, the sample from Section 7.2 is pyrolyzed under pure oxygen, water is removed by magnesium perchlorate and the carbon dioxide is removed by ascarite. The decrease in signal obtained by differential thermal conductivity detectors placed between the combustion gas stream before and after the ascarite tube is a measure of the organic carbon content.

5.2.2 Carlo Erba Model 1106 CHN Analyzer, or equivalent.

5.2.2.1 In this apparatus, the sample is pyrolyzed in a induction type furnace, and the resultant carbon dioxide is chromatographically separated and analyzed by a differential thermal conductivity detector.

5.2.3 LECO Models WR12, WR112, or CR-12 carbon determinators, or Models 600 or 800 CHN analyzers.

5.2.3.1 In the LECO WR-12, the sample is burned in high frequency induction furnace, the carbon dioxide is selectively adsorbed at room temperature in a molecular sieve. It is subsequently released by heating and is measured by a thermal conductivity detector. The WR-112 is an upgraded WR-12 employing microprocessor electronics and a printer to replace the electronic digital voltmeter.

5.2.3.2 In the LECO CR-12 carbon determinator, the sample is combusted in oxygen, moisture and dust are removed by appropriate traps and the carbon dioxide is measured by a selective, solid state, infrared detector. The signal from the detector is then processed by a microprocessor and the carbon content is displayed on a digital readout and recorded on an integral printer.

5.2.3.3 In the LECO CHN-600 and CHN-800 elemental analyzers, the sample is burned under oxygen in a resistance furnace and the carbon dioxide is measured by a selective infrared detector.

5.2.4 Dohrman Model DC85 Digital High Temperature TOC Analyzer.

5.2.4.1 In this instrument, the sample is burned in resistance furnace under oxygen, the interfering gases are removed by a sparger/scrubber system and the carbon dioxide is measured by a non-dispersive infrared detector and shown on a digital display in concentration units.

5.3 No specific analyzer is recommended as superior. The above listing is for information only and is not intended to restrict the use of other unlisted instruments capable of analysing TOC. The instruments to be used must have the following specifications:

5.3.1 A combustion boat which is heated in a stream of oxygen or air in a resistance or induction-type furnace to completely convert organic substances to CO_2 and water.

5.3.2 A means to physically or by measurement technique to separate water and other interferants from CO_2.

5.3.3 A means to quantitatively determine CO_2 with adequate sensitivity (100 mg/kg), and precision (25% at the 95% confidence level as demonstrated by repetitive measurements of a well mixed ocean sediment sample).

5.4 A strip chart or other permanent recording device to document the analysis.

6. Reagents

6.1 Distilled water used in preparation of standards and for dilution of samples should be ultra pure to reduce the carbon concentration of the blank.

6.2 Potassium hydrogen phthalate, stock solution, 1000 mg carbon/liter: Dissolve 0.2128 g of potassium hydrogen phthalate (Primary Standard Grade) in distilled water and dilute to 100.0 ml.

NOTE 2: Sodium oxalate and acetic acid are not recommended as stock Solutions.

6.3 Potassium hydrogen phthalate, standard solutions: Prepare standard solutions from the stock solution by dilution with distilled water.

6.4 Phosphoric acid solution, 1:1 by volume.

7. Procedure

7.1 Weigh the well mixed sample (up to 500 mg) into the combustion boat or cup. Add 1:1 phosphoric acid drop wise until effervescence stops. Heat to 75°C.

NOTE: This procedure will convert inorganic carbonates and bicarbonates to carbon dioxide and eliminate it from the sample.

7.2 Analyse the residue according to the instrument manufacturer's instructions.

7.3 Determine percent residue on a separate sample aliquot as follows:

7.3.1 Heat a clean 25 ml beaker at 103° to 105°C for one hour. Cool in desiccator, weigh to the nearest mg and store in desiccator until use.

7.3.2 Add 1 g, weighed to the nearest mg, of an aliquot of the well-mixed sample .

7.3.3 Dry and heat in the 103° to 105°C oven for one hour. Cool in desiccator. Weigh to the nearest mg.

8. Calibration

8.1 Follow instrument manufacturer's instructions.

8.2 Prepare calibration curve plotting mg carbon vs. instrument response. using four standards and a blank covering the analytical range of interest.

9. Precision and Accuracy

9.1 The precision and accuracy will differ with the various instruments and matrices and must be determined by the laboratories reporting data. To initiate a control chart, a representative sample of well mixed sediment should be analyzed 15 times to determine the analytical precision. Set up a control chart showing 3 times the standard deviation limits for precision.

9.2 Subsequently during analysis of environmental samples, take one sample per batch of 20 or less and run in quadruplicate. Calculate standard deviation and report with initial control chart data.

9.3 If the sample being run in quadruplicate exceeds the 3 standard deviation limit, identify error and rerun environmental samples in that batch along with the quadruplicate sample.

NON-AQUEOUS SAMPLE INFORMATION

1. Non-Aqueous Matrix Sampled:

   Soil ☐ Sediment ☐ Sludge ☐ Chemical Solids ☐ Waste Pile ☐

   Other: _____

2. Type of Sample: Grab ☐ Composite ☐ If Composite Sample, # samples/composite: _____

3. Was the VOA sample collected first from a discrete location prior to homogenization: Yes ☐ No ☐ N/A ☐

4. Was the sample homogenized prior to acquistion into the sample containers: Yes ☐ No ☐

5. Type of Sampling Equipment:

	Material of Construction: Stainless Steel	Carbon Steel	Teflon	Other
Spoon/Spatula	☐	☐	☐	_____
Trowel/Scoop	☐	☐	☐	_____
Bucket Auger	☐	☐	☐	_____
Split Spoon	☐	☐	☐	_____
Shelby Tube	☐	☐	☐	_____
Trier	☐	☐	☐	_____
Vichmeyer Sampler	☐	☐	☐	_____
Penar Dredge	☐	☐	☐	_____
Other: _____	☐	☐	☐	_____

6. Was the drill rig, auger flights, rods, etc. decontaminated according to standard NJDEP/DHSM procedures between each sa

   location: Yes ☐ No ☐ N/A ☐

   If No, method of decontamination: _____

7. If mud rotary drilling was utilized what was the souce of the water: _____

8. Was the Sampling Equipment Dedicated: Yes ☐ _____ No ☐ _____

9. Was the Sampling Equipment: Lab Decontaminated ☐ _____ Field Decontaminated ☐ _____

10. Was the sampling equipment decontaminated according to standard NJDEP/DHSM procedures: Yes ☐ No ☐

    If No, method of decontamination: _____

11. Was the decontamination area located away from the source of contamination: Yes ☐ No ☐ N/A ☐

12. Are disposable gloves worn and changed between each sample location: Yes ☐ No ☐

13. Auditor's Comments: _____

_____

_____

_____

QA/QC Information

1. Laboratory:

Name: _____ Phone: _____

Contact Person: _____

CLP ☐ CLP Capable ☐ NJDEP Certified ☐ NJDEP Contract ☐ Other: _____

2. Sample Information:

Matrix	Parameter	Preservative	Container Description

3. What order, by analytical parameter, are samples collected: _____

4. Field Blanks: Yes ☐ No ☐ N/A ☐ Frequency: _____

Method: _____

Was identical bottle to bottle transfer of water utilized: Yes ☐ No ☐

5. Trip Blanks: Yes ☐ No ☐ N/A ☐ Frequency: _____

Parameters: _____ Container: _____

6. What was the source of the blank water: Laboratory provided analyte free ☐ Other: _____

7. Sample Packaging and Handling:

Sample Containers Labeled: Yes ☐ No ☐ N/A ☐

COC Forms Completed: Yes ☐ No ☐ N/A ☐

Custody Seals Used: Yes ☐ No ☐ N/A ☐

Samples Preserved to 4°C: Yes ☐ No ☐ N/A ☐

Method: Blue Ice ☐ Cube Ice ☐ Other: _____

NJDEP/DWSR "Handling Time" Exceeded: Yes ☐ No ☐

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TABLE

NON-AQUEOUS and AQUEOUS SAMPLES

...PLE ...TION	SAMPLE NUMBER	SAMPLE DEPTH	SAMPLE TIME	PARAMETERS	COMMENTS

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TABLE

GROUNDWATER SAMPLES

AMPLE CATION	SAMPLE NUMBER	EVACUATION TIME	VOLUME PURGED	SAMPLE TIME	PARAMETERS	COMMENTS

TABLE

POTABLE WELL SAMPLES

LOCATION (SEQUENCE)	SAMPLE NUMBER	PURGE TIME	SAMPLE TIME	PARAMETERS	COMMENTS

APPENDIX D

Data Validation SOPs

SOP NO. HW-6
Revision #6

CLP ORGANICS DATA REVIEW AND PRELIMINARY REVIEW

CONCURRED BY: _____ Date: 4/6/89
Louis Bevilacqua
Monitoring Management Branch

APPROVED BY: _____ Date: 4/14/89
Gerard F. McKenna, Chief
Monitoring Management Branch

INTRODUCTION TO DATA VALIDATION

1.0 Scope

1.1 This procedure is applicable to organic data obtained from contractor laboratories working for the Contract Laboratory Program (CLP).

1.2 The data validation is based upon analytical and quality assurance requirements specified in the Statement of Work (SOW).

2.0 Responsibilities

Data reviewers will complete the following tasks as assigned by the Data Review Coordinator:

2.1 Data Assessment - The reviewer must answer every question on the checklist. All response shall be in ink.

2.2 Data Assessment Narrative (Attachment 1) - Data reviewer is required to use these forms and must match the action in the narrative with the action taken on the Form I(s).

2.3 Rejection Summary Form (Attachment 2) - Fill in the total number of analytes measured by different analyses and the number of analytes rejected or flagged as estimated due to corresponding quality control criteria. Place an "X" in the boxes where analyses were not performed or criteria do not apply.

2.4 Organic Regional Data Assessment - Data reviewer is also required to fill out Organic Regional Data Assessment Form (Attachment 3).

2.5 Telephone Record Log - The data reviewer should enter the bare facts of inquiry before initiating any authorized telephone conversation with a CLP laboratory. After the case review has been completed, mail the white copy of the Telephone Record Log to the laboratory and the pink copy to SMO. File the yellow copy in the Telephone Record Log folder and attach a photocopy of the Telephone Record Log to the completed Data Assessment Narrative.

2.6 Forwarded Paperwork - Upon completion of the review, the following are to be forwarded to the Regional Sample Control Center (RSCC) located in the Surveillance and Monitoring Branch:

a. data package
b. completed assessment checklist
c. SMO Contract Compliance Screening (CCS)

Forward four (4) copies of the completed Data Assessment Narrative along with four (4) copies of the Organic Data Assessment Form: one each for the appropriate Regional DPO, the Sample Management Office (SMO), and to the last two addresses of the Data Reviewers Mailing List.

2.7 Filed Paperwork - Upon completion of the review, the following are to be filed within the Monitoring and Management Branch (MMB) files:

a. Telephone record Log (copy)
b. Record of Communication (original)
c. Rejection Summary Form

3.0 Rejection of Data - All values determined to be unacceptable on the Organic Analysis Data Sheet (Form I) must be flagged with an "R". As soon as review criteria causes data to be rejected, that data can be eliminated from any further review or consideration.

4.0 Acceptance Criteria - In order that the reviews be consistent among reviewers, this Standard Operating Procedure (SOP) should be used. Additional guidance can be found in the Functional Guidelines.

5.0 SMO Contract Compliance Screening (CCS) - This is intended to aid the reviewer in locating any problems, both corrected and uncorrected. However, the validation should be carried out even if CCS is not present. Resubmittals received from the laboratory in response to CCS must be used by the reviewer.