

**U.S. Environmental Protection Agency
Region 4
SESD - EAB**



Ecological Assessment Standard Operating Procedures
and
Quality Assurance Manual

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SECTION 1

PREFACE

1.1 Introduction

The purpose of this manual is to document the standard operating procedures of the USEPA, Region 4, Science and Ecosystem Support Division, Ecological Assessment Branch (EAB). The specific procedures/guidance defined in this manual are based on the experience of Branch personnel and/or are specifically referenced. This manual is not intended to be “prescriptive” as to stifle professional judgement, but is intended to assure that acceptable field methods and quality assurance/quality control procedures are used when performing environmental investigations.

This manual is a dynamic document which will be periodically reviewed and updated. It will serve as a training text for new employees as well as a reference text for current employees. When changes are made in the manual, a memorandum from the branch chief will be sent to each EAB employee with appropriate documentation of the changes. Each employee will be responsible for keeping his or her manual current.

The standard operating procedures and quality assurance/quality control procedures in this manual will be incorporated by reference into the study plans issued by EAB personnel. Any deviation of these protocols, procedures or methods must be documented in the plan of study and approved by the employee’s section chief. If deviation from this manual occurs as a result of unforeseen field events, then justification for the deviations must be documented in the field note book(s).

SECTION 2
SAMPLE CONTROL, FIELD RECORDS, AND DOCUMENT CONTROL

SECTION OBJECTIVES:

- Present standard procedures for sample and evidence identification.
- Present standard procedures for sample control.
- Present standard procedures for chain-of-custody.
- Present standard procedures for maintenance of field records and document control.

2.1 Introduction

Sample identification, chain-of-custody records, receipt for sample forms, and field records (with the exception of surveying notes) shall be recorded with waterproof, non-erasable ink. If errors are made in any of these documents, corrections should be made by crossing a single line through the error and entering the correct information. All corrections shall be initialed and dated. If possible, all corrections should be made by the individual making the error.

If information is entered onto sample tags, logbooks, or sample containers using stick-on labels, the labels should not be capable of being removed without leaving obvious indications of the attempt. Labels should never be placed over previously recorded information. Corrections to information recorded on stick-on labels should be made as stated above.

Following are definitions of terms used in this section:

Project Leader: The individual with overall responsibility for conducting a specific field investigation in accordance with this SOP.

Field Sample Custodian: Individual responsible for maintaining custody of the samples and completing the sample tags, and Chain-of-Custody Records.

Sample Team Leader: An individual designated by the project leader to be present during and responsible for all activities related to the collection of samples by a specific sampling team.

Sampler: The individual responsible for the actual collection of a sample.

Transferee: Any individual who receives custody of samples subsequent to release by the field sample custodian.

Laboratory Sample Custodian: Individual responsible for accepting custody of samples from the field sample custodian or a transferee.

One individual may fulfill more than one of the roles described above while in the field.

2.2 Sample and Evidence Identification

PERFORMANCE OBJECTIVES:

- To accurately identify samples and evidence collected.
- To adequately insure that chain-of-custody is maintained.

2.2.1 Sample Identification

The method of sample identification utilized depends on the type of sample collected. In-situ field samples are those collected for specific field analysis or measurement where the data are recorded directly in bound field logbooks, logged directly into field instrument memory/computers or on the Chain-of-Custody Record, with identifying information, while in the custody of the sampling team. Examples of such in-situ field measurements and analyses include pH, temperature, dissolved oxygen and conductivity. Samples other than those collected for in-situ analysis are identified by using a standard sample tag (Figure 2-1) which is attached to the sample container. In some cases, particularly with biological samples, the sample tag may have to be included with or wrapped around the sample. However, after the samples have been analyzed, the tags will be disposed with the sample containers. The following information shall be included on the sample tag using waterproof, non-erasable ink:

- project number;
- field identification or sample station number;
- date and time of sample collection;
- designation of the sample as a grab or composite;
- brief description of the sampling location. If foreign soil sample, label as "FOREIGN SOIL";
- the signature of either the sampler(s) or the designated sampling team leader and the field sample custodian (if appropriate);
- whether the sample is preserved or unpreserved;
- the general types of analyses to be performed (checked on front of tag); and
- any relevant comments (such as readily detectable or identifiable odor, color, or known toxic properties).

Samples or other physical evidence collected during criminal investigations are to be identified by using the "criminal sample tag." This tag is similar to the standard sample tag shown in Figure 2-1, except that it has a red border around the front and a red background on the back of the tag. If a criminal sample tag is not available, the white sample tag may be used and should be

marked "Criminal" in bold letters on the tag.

If a sample is split with a facility, state regulatory agency, or other party representative, the recipient should be provided (if enough sample is available) with an equal weight or volume of sample. The split sample should be clearly marked and identified.

Tags for blank or duplicate samples will be marked "blank" or "duplicate," respectively. This requirement does not apply to blind-spiked or blank samples which are to be submitted for laboratory quality control purposes. Blind-spiked or blank samples shall not be identified as such. This identifying information shall also be on the Chain-Of-Custody Record and in the bound field log books as outlined in Sections 2.3 and 2.5.

2.2.2 Photograph, Digital Still Image and Video Identification

Photographs and Digital Still Images

When photographs or digital images are taken, to document field activities, a record of each exposure or image shall be kept in a bound field logbook. The following information shall be recorded in the logbook:

- an accurate description of what the photograph or image shows, including the name of the facility or site and the specific project name and project number;
- the date and time that the photograph or image was taken;
- the name of the individual who took the photograph or digital image.

When photographs are used in technical reports or placed in the official files, the film shall be developed with the negatives supplied uncut. The identifying information that was recorded in the field logbook shall be entered on the back of the prints. For criminal investigations, the negatives must be maintained with the bound field logbook in the project file and stored in a secured file cabinet.

When digital images are used in technical reports or placed in the official files, the disk or memory card/stick with the original, unaltered file of the images or a printed copy of the unaltered images shall be placed in the official files as well. If printed copies of the images are used, each image shall be identified using the information that was recorded in the field logbook. For enforcement cases, it is imperative that the individual who took the image be identified in the field logbook in the event their testimony is required.

Video

When a video tape is made for use as evidence in an enforcement case, the following information should be recorded in a bound field logbook:

- the date and time that the video was recorded;
- a brief description of the subject of the video tape;
- the person recording the video

Video records shall include a visual notation (placard) at the beginning of the of the video with the information (i.e., location, date, time), or an audio record may also be included in the video tape with the above logistical information as well as a narrated description of the video record.

A label shall be placed on the video tape with the appropriate identifying information (i.e., project name, project number, date, location etc.). In the event testimony regarding a video tape recording is required for an enforcement case, one individual should be responsible for recording the video for each case. The original, unaltered tape shall be placed in the official files.

2.2.3 Identification of Physical Evidence

Physical evidence, other than samples, shall be identified by utilizing a sample tag or recording the necessary information on the evidence. When samples are collected from vessels or containers which can be moved (drums for example), mark the vessel or container with the field identification or sample station number for future identification, when necessary. The vessel or container may be labeled with an indelible marker (e.g., paint stick or spray paint). The vessel or container need not be marked if it already has a unique marking or serial number; however, these numbers shall be recorded in the bound field logbooks. In addition, it is suggested that photographs of any physical evidence (markings, etc.) be taken and the necessary information recorded in the field logbook.

Occasionally, it is necessary to obtain recorder and/or instrument charts from facility owned analytical equipment, flow recorders, etc., during field investigations and inspections. Mark the charts and write the following information on these charts while they are still in the instrument or recorder :

- Starting and ending time(s) and date(s) for the chart.
- Take an instantaneous measurement of the media being measured by the recorder. The instantaneous measurement shall be entered at the appropriate location on the chart along with the date and time of the measurement.
- A description of the location being monitored and any other information required to interpret the data such as type of flow device, chart units, factors, etc.

All of the above information should be initialed by the field investigator. After the chart has been removed, the field investigator shall indicate on the chart who the chart (or copy of the chart) was received from and enter the date and time, as well as the investigator's initials.

Documents such as technical reports, laboratory reports, etc., should be marked with the field investigator's signature, the date, the number of pages, and from whom they were received. Confidential documents should not be accepted, except in special circumstances when approval has been granted from EPA legal counsel.

2.3 Chain-of-Custody Procedures

PERFORMANCE OBJECTIVE:

- To maintain and document the possession of samples or other evidence from the time of collection until they or the data derived from the samples are introduced as evidence.

2.3.1 Introduction

Chain-of-custody procedures are comprised of the following elements; 1) maintaining sample custody and 2) documentation of samples for evidence. To document chain-of-custody, an accurate record must be maintained to trace the possession of each sample from the moment of collection to its introduction into evidence.

2.3.2 Sample Custody

A sample or other physical evidence is in custody if:

- it is in the actual possession of an investigator;
- it is in the view of an investigator, after being in their physical possession;
- it was in the physical possession of an investigator and then it was secured to prevent loss or tampering; and/or
- it is placed in a designated secure area.

2.3.3 Documentation of Chain-of-Custody

Sample Tag

A sample tag (Figure 2-1) should be completed for each sample using waterproof, non-erasable ink as specified in Section 2.2.

Sample Seals

Samples should be sealed as soon as possible following collection utilizing the EPA custody seal shown in Figure 2-2. The sample custodian should write the date and their initials on the seal. The use of custody seals may be waived if field investigators keep the samples in their custody as defined in Section 2.3.2 from the time of collection until the samples are delivered to the laboratory analyzing the samples. Custody seals should be used for criminal investigations.

Chain-of-Custody Record

The field Chain-Of-Custody Record (Figures 2-3 & 2-4) is used to record the custody of all samples or other physical evidence collected and maintained by investigators. All physical evidence or sample sets shall be accompanied by a Chain-Of-Custody Record. This Chain-Of-Custody Record documents transfer of custody of samples from the sample custodian to another person, to the laboratory, or other organizational elements. To simplify the Chain-of-Custody Record and eliminate potential litigation problems, as few people as possible should have custody of the samples or physical evidence during the investigation. This form shall not be used to document the collection of split samples where there is a legal requirement to provide a receipt for samples (see Section 2.4). The Chain-Of-Custody Record also serves as a sample logging mechanism for the laboratory sample custodian. A separate Chain-of-Custody Record should be used for each final destination or laboratory utilized during the investigation.

The following information must be supplied in the indicated spaces (Figure 2-3) to complete the field Chain-Of-Custody Record.

1. The project number.
2. The project name.
3. The project leader.
4. If the individual serving as the field sample custodian is different from the individual serving as the project leader, the field sample custodian's name and the title of the sample custodian (e.g., Jane Doe, Sample Custodian) should be recorded in the "Remarks/AirBill" section of the Chain-of-Custody Record. This section may also be used to record airbill numbers, registered or certified mail serial numbers, or other pertinent information.
5. All samplers or sampling team leaders (if applicable) must sign in the designated signature block.
6. The sampling station ID (if positional data is recorded for the sample), Station ID, Media Code, date, and time of sample collection, grab or composite sample designation, and a brief description of the type of sample and/or the sampling location must be included on each line. One sample should be entered on each line and a sample should not be split among multiple lines.
7. If multiple sampling teams are collecting samples, the sampling team leader's name should be indicated in the "Remarks" column.
8. The total number of sample containers must be listed in the "Total Containers" column for each sample. The number of individual containers for each analysis must also be listed. There should not be more than one sample type per sample. Required analyses should be circled or entered in the appropriate location as indicated on the Chain-of-Custody Record.
9. The tag numbers for each sample and any needed remarks are to be supplied in the "Tag Number" column.

10. The sample custodian and subsequent transferee(s) should document the transfer of the samples listed on the Chain-of-Custody Record. The person who originally relinquishes custody should be the sample custodian. Both the person relinquishing the samples and the person receiving them must sign the form. The date and time that this occurred should be documented in the proper space on the Chain-of-Custody Record.
11. Usually, the last person receiving the samples or evidence should be the laboratory sample custodian or their designee(s).

The Chain-of-Custody Record is a serialized document. Once the Record is completed, it becomes an accountable document and must be maintained in the project file. The suitability of any other form for chain-of-custody should be evaluated based upon its inclusion of all of the above information in a legible format.

If chain-of-custody is required for documents received during investigations, the documents should be placed in large envelopes, and the contents should be noted on the envelope. The envelope shall be sealed and an EPA custody seal placed on the envelope such that it cannot be opened without breaking the seal. A Chain-Of-Custody Record shall be maintained for the envelope. Any time the EPA seal is broken, that fact shall be noted on the Chain-Of-Custody Record and a new seal affixed. The information on the seal should include the sample custodian's signature or initials, as well as the date.

Physical evidence such as video tapes or other small items shall be placed in Zip-Loc® type bags or envelopes and an EPA custody seal should be affixed so that they cannot be opened without breaking the seal. A Chain-Of-Custody Record shall be maintained for these items. Any time the EPA seal is broken, that fact shall be noted on the Chain-of-Custody Record and a new seal affixed. The information on the seal should include the sample field custodian's signature or initials, as well as the date.

EPA custody seals can be used to maintain custody of other items when necessary by using similar procedures as those previously outlined in this section.

Samples should not be accepted from other sources unless the sample collection procedures used are known to be acceptable, can be documented, and the sample chain-of-custody can be established. If such samples are accepted, a standard sample tag containing all relevant information and the Chain-Of-Custody Record shall be completed for each set of samples.

2.3.4 Transfer of Custody with Shipment

- Samples shall be properly packaged for shipment in accordance with the procedures outlined in Appendix D.
- All samples shall be accompanied by the Chain-Of-Custody Record. The original and one copy of the Record will be placed in a plastic bag inside the secured shipping container if samples are shipped. When shipping samples via common carrier, the "Relinquished By" box should be filled in; however, the "Received By" box should be left blank. The laboratory sample custodian is responsible for receiving custody of the samples and will fill in the "Received By" section of the Chain-of-Custody Record. One copy of the Record will be retained by the project leader. The original Chain-of-Custody

Record will be transmitted to the project leader after the samples are accepted by the laboratory. This copy will become a part of the project file.

- If sent by mail, the package shall be registered with return receipt requested. If sent by common carrier, an airbill should be used. Receipts from post offices and airbills shall be retained as part of the documentation of the chain-of-custody. The airbill number or registered mail serial number shall be recorded in the "Remarks/airbill" section of the Chain-Of-Custody Record or in another designated area if using a form other than that shown in Figure 2-3.

2.4 Receipt for Samples Form (CERCLA/RCRA/TSCA)

2.4.1 Introduction

Section 3007 of the Resource Conservation and Recovery Act (RCRA) of 1976 and Section 104 of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund) of 1980 require that a "receipt" for all facility samples collected during inspections and investigations be completed and offered to the owner/operator of each facility before the field investigator departs the premises. The Toxic Substances Control Act (TSCA) contains similar provisions. The laws do not require that homeowners or other off-site property owners be given this form.

2.4.2 Receipt for Samples Form

The Receipt for Samples form (Figure 2-5) is to be used to satisfy the receipt for samples provisions of RCRA, CERCLA, and TSCA. The form also documents that split samples were offered and either "Received" or "Declined" by the owner/operator of the facility or site being investigated. The following information must be supplied and entered on the Receipt for Samples form.

- The project number, project name, name of facility or site, and location of the facility or site must be entered at the top of the form in the indicated locations.
- The sampler(s) must sign the form in the indicated location. If multiple sample teams are collecting samples, the sample team leader's name should be indicated in the "EPA Sample Tag No.'s/Remarks" column.
- Each sample collected from the facility or site must be documented in the sample record portion of the form. The sample station number, date and time of sample collection, composite or grab sample designation, whether or not split samples were collected (yes or no should be entered under the split sample column), the tag numbers of samples collected which will be removed from the site, a brief description of each sampling location, and the total number of sample containers for each sample must be entered.
- The bottom of the form is used to document the site operator's acceptance or rejection of split samples. The project leader must sign and complete the information in the "Split Samples Transferred By" section (date and time must be entered). If split samples were not collected, the project leader should initial and place a single line through "Split Samples Transferred By" in this section. The operator of the site must indicate whether

split samples were received or declined and sign the form. The operator must give their title, telephone number, and the date and time they signed the form. If the operator refuses to sign the form, the sampler(s) should note this fact in the operator's signature block and initial this entry.

The Receipt for Samples form is serialized and becomes an accountable document after it is completed. A copy of the form is to be given to the facility or site owner/operator. The original copy of the form must be maintained in the project files.

2.5 Field Records

PERFORMANCE OBJECTIVE:

- To accurately and completely document all field activities.

Each project should have a dedicated logbook(s). The project leader's name, the sample team leader's name (if appropriate), the project name and location, and the project number should be entered on the inside of the front cover of the logbook. It is recommended that each page in the logbook be numbered and dated. The entries should be legible and contain accurate and inclusive documentation of an individual's project activities. At the end of all entries for each day, or at the end of a particular event if appropriate, the investigator should draw a diagonal line and initial indicating the conclusion of the entry. Since field records are the basis for later written reports, language should be objective, factual, and free of personal feelings or other terminology which might prove inappropriate. Once completed, these field logbooks become accountable documents and must be maintained as part of the official project files. All aspects of sample collection and handling, as well as visual observations, shall be documented in the field logbooks. The following is a list of information that should be included in the logbook:

- sample collection equipment (where appropriate);
- field analytical equipment, and equipment utilized to make physical measurements shall be identified;
- calculations, results, and calibration data for field sampling, field analytical, and field physical measurement equipment;
- property numbers of any sampling equipment used, if available;
- sampling station identification;
- time of sample collection;
- description of the sample location;
- description of the sample;

- who collected the sample;
- how the sample was collected;
- diagrams of processes;
- maps/sketches of sampling locations; and
- weather conditions that may affect the sample (e.g., rain, extreme heat or cold, wind, etc.)

2.6 Document Control

Document control refers to the maintenance of inspection and investigation project files. All documents as outlined below shall be kept in project files. Investigators may keep copies of reports in their personal files, however, all official and original documents relating to inspections and investigations shall be placed in the official project files. The following documents shall be placed in the project file, if applicable:

- request memo from the program office, if appropriate;
- copy of the study plan;
- original Chain-Of-Custody Records and bound field logbooks;
- copy of the Receipt for Sample forms;
- records obtained during the investigation;
- complete copy of the analytical data and memorandums transmitting analytical data;
- official correspondence received by or issued by the Branch relating to the investigation including records of telephone calls;
- photographs, negatives, video, or digital image memory cards, etc., associated with the project;
- one copy of the final report and transmittal memorandum(s); and
- relevant documents related to the original investigation/inspection or follow-up activities related to the investigation/inspection.

The project leader shall review the file at the conclusion of the project to insure that it is complete.

2.7 Disposal of Samples or Other Physical Evidence

Disposal of samples or other physical evidence obtained during investigations is conducted on a case-by-case basis. After samples have been analyzed, the laboratory sample custodian shall contact the project leader via e-mail, indicating that the samples and tags will be disposed of by a

certain date unless the project leader indicates otherwise. If the sample custodian does not receive a reply from the project leader within the time specified in the e-mail, the samples and tags will be disposed. Personnel may want to check with the EPA Program Office requesting the inspection or investigation before granting permission to dispose of samples or other physical evidence. The following general guidance is offered for the disposal of samples or other physical evidence:

- No samples, physical evidence, or any other document associated with a criminal investigation shall be disposed without written permission from EPA's Criminal Investigations Division.
- Samples and sample tags associated with routine projects may be disposed following approval from the project leader.

2.8 Field Operations Records Management System (FORMS)

FORMS is a computer program designed to streamline the documentation required by SESD and/or the Contract Laboratory Program (CLP) for sample identification and chain-of-custody. Once the appropriate information is entered into the computer, FORMS will generate stick-on labels for the sample tags, sample containers (CLP), and field logbooks, and will generate the sample receipt and chain-of-custody reports for the appropriate laboratory. The advantages to this system include faster processing of samples and increased accuracy. Accuracy is increased because the information is entered only once, and consequently, consistent from the log book to the tags, bottle labels, and chain-of-custody forms. Operating instructions are available for use with the FORMS program.

Figure 2-1
Sample Tag

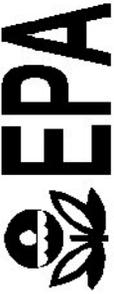
<p>UNITED STATES ENVIRONMENTAL PROTECTION AGENCY REGION 4 960 COLLEGE STATION RD. ATHENS, GA 30605-2720</p> 					
Project No.	Station I.D.	Month/Day/Year	Time	Designate Comp.	Grab
Station Location		Samplers (Signatures)			
Tag No. 4A-		Lab Sample No.			
<p>Preservative No <input type="checkbox"/> Yes <input type="checkbox"/>: _____</p>					
<p>ANALYSES</p>					
COD, TOC, Nutrients					
BOD, Solids					
Metals					
Extractable Organics					
Pesticides/PCB's					
Volatile Organics					
Cyanide					
<p>Remarks:</p>					

Figure 2-2
Custody Seal

<p>United States Environmental Protection Agency Athens, Georgia REGION 4</p>		<p>Date _____ Initial _____</p>	<p>OFFICIAL SAMPLE SEAL</p>
--	---	---	--

Figure 2-4
Chain-of-Custody Form – Back

① **Station ID** - Station ID is required *if* positional data is recorded for the sample. Any combination of letters, numbers, or other characters. Maximum of 20 characters. Use this column to identify a sampling station where one or more samples or field measurements are taken. A few examples are well numbers, NPDES permit numbers, Air permit numbers, AOC numbers, Grid numbers, Site designations, etc., or combinations of these as appropriate.

② **Sample ID** - Required. Any combination of letters or numbers. Maximum of 8 characters. **NOTE: For QA/QC samples, the Sample ID must begin with the letters "QA for the sample to be properly identified.**

③ **Media Code** - Required. Choose the code that most closely describes the sample:

Environmental Samples

SF	-Surface Soil (0"-12")	WP	-Wipe Sample
SB	-Subsurface Soil (>12")	FI	-Fish Sample
PW	-Potable Water	VG	-Vegetation
MS	-Municipal Water Supply	MI	-Macroinvertebrates
IW	-Industrial Well	WW	-Wastewater
WA	-Waste	SL	-Sludge (non-RCRA)
SW	-Surface Water	UI	-UIC Injection Wells
SD	-Sediment	US	-Underground Storage Tanks
GW	-Groundwater	PT	-Petroleum Tanks
PC	-Precipitation	AA	-Ambient Air
OT	-Other (Unknown)	IA	-Indoor Air
OB	-Other Biota	ME	-Municipal Eff. Wastewater
MP	-Municipal Proc. Wastewater	IE	-Industrial Eff. Wastewater
IP	-Industrial Proc. Wastewater	PE	-Periphyton
		TI	-Tissue

Field QA/QC Samples

TS	-Trip Blank-Soil	FB	-Filter Sand Blank
TW	-Trip Blank-Water	OW	-Organic Free Water Blank
TP	-Trip Blank-Wipe	PB	-Preservative Blank
EB	-Equipment Rinse Blank	GB	-Glove Blank
GR	-Grout Blank	BO	-Bottle Blank
MB	-Drilling Mud Blank	FL	-Field Blank
DB	-Potable Decon Blank	IB	-Dry Ice Blank
BB	-Bentonite Blank	BK	-Other Blank
FA	-Field Blank Air		

SECTION 3
ECOLOGICAL ASSESSMENT BRANCH SAFETY PROTOCOLS

SECTION OBJECTIVE:

- Present safety protocols to ensure that all operations are conducted in a manner which protects worker safety and meets compliance with all OSHA regulations and EPA safety policies.

3.1 Introduction

The following parts of this section define safety protocols that are to be used by Branch personnel while conducting field operations. This section also covers the necessary training, equipment, and experience that is needed to conduct safe environmental investigations.

The Division's safety program is jointly coordinated by the Regional Occupational Health and Safety Designee (OHSD); a Division Safety, Health and Environmental Manager (SHEM) coordinator; and a Branch Safety Officer. The Division Director appoints the SHEM to perform the following duties: 1) classify employees into safety categories based upon the type of work they are engaged in; 2) make requests for hazardous duty status; 3) provide and track safety related training; 4) notify management of safety deficiencies; and 5) reviews project specific safety plans. The employees immediate supervisor is responsible for ensuring that employees meet training and medical monitoring requirements. Field studies require a safety plan that must be approved by the Branch Safety Officer. It is the responsibility of the project leader to make a copy of the safety plan available to all project team members. It should be noted that the responsibility for obtaining and reading a copy of the safety plan and for the safe conduct of field operations is ultimately the responsibility of each individual worker. On large or complex studies a Field Safety Officer (FSO) may be selected to insure all safety requirements are performed in accordance with the safety plan. The FSO duties are listed in 3.3.1.

Field personnel will not be required to participate in any operation which violates OSHA and EPA policies. The safety protocols in this section are written in accordance with those defined by the following regulations, policies, and manuals;

29 CFR Part 1910.120, Hazardous Waste Operations and Emergency Response: These OSHA regulations govern workers at hazardous waste sites and include requirements for training, equipment, and practices involved in handling of hazardous materials.

29 CFR Part 1910.1200, Hazard Communication: These OSHA regulations govern workers handling hazardous materials and include requirements for training, labeling, and documentation involved in handling hazardous materials.

Occupational Safety and Health Guidance Manual for Hazard Waste Activities: This

NIOSH, OSHA, USCG, and EPA guidance manual is for those who are responsible for occupational safety and health programs at hazardous waste sites. It assumes a basic knowledge of science and experience in occupational safety and health. It is the product of four Agencies (NIOSH, OSHA, USCG, and EPA) mandated by CERCLA section 301 (f) to study the problem of protecting the safety and health of workers at hazardous waste sites.

Safety, Health and Environmental Management Program Procedures and Policy Manual: Science and Ecosystem Support Division, Environmental Protection Agency (USEPA), Region IV, Athens, Georgia, August 2001: This manual covers safety practices and rules governing activities at EPA facilities in Athens, Georgia. Included in its contents are accident reporting procedures, procedures for natural and man-made emergencies, safety guidelines for offices and laboratories, and special rules for storage of equipment and wastes.

Field Health and Safety Manual: USEPA, Region IV, 1990: This manual covers safety involved in all field activities performed in Region 4. It includes regional policy regarding training requirements, medical monitoring, and personal protection.

The remaining parts of this section cover hazard communication, safety protocols, training, and equipment that are to be used when conducting environmental investigations.

3.2 Hazard Communication Procedure

3.2.1 Introduction

The purpose of this hazard communication procedure is to ensure that the hazards of all chemicals used by the Branch are evaluated, and that information concerning their hazards are transmitted to Branch personnel. The transmittal of information is to be accomplished by means of a comprehensive hazard communication program which includes container labeling and other forms of warning, material safety data sheets (MSDS), and employee training.

3.2.2 Scope

This hazard communication procedure covers activities involving the use and storage of hazardous chemicals.

3.2.3 Labels and Other Forms of Warnings

Personnel who receive and store hazardous chemicals from manufacturers and suppliers will ensure that the containers are marked with the following information:

- Identity of the hazardous chemical(s);
- Appropriate hazard warnings; and
- Name and address of the chemical manufacturer, importer, or other responsible party.

Containers of hazardous chemicals generated during field investigations will be labeled with the following information:

- Identity of the hazardous chemical(s) contained therein; and
- Appropriate hazard warnings.

Exempt from labeling requirements are any containers into which hazardous chemicals are transferred from labeled containers, and which are intended only for use by the person who performs the transfer during the same work day in which the transfer is made. Labels on containers or hazardous chemicals will not be removed or in any way defaced. Labels for containers of hazardous chemicals will be provided by the SHEM or a designee. Information on the labels will be in English. Information in other languages may be added as long as the information presented in English is in no way obscured.

3.2.4 Material Safety Data Sheets (MSDSs)

Personnel responsible for receiving hazardous chemicals from manufacturers or suppliers will ensure that MSDSs are obtained for each shipment received. Receipt of hazardous chemicals will be contingent upon both the provision of MSDSs and compliance of the MSDS with requirements set forth in OSHA's Hazardous Communication Final Rules, part (g).

3.2.5 The Hazardous Chemical Inventory

To insure all chemicals are included in the inventory all chemical purchase orders must be approved and ordered by the Divisional Chemical Hygiene Officer (CHO)

The CHO will compile a list of hazardous chemicals used or stored within the Branch. The list will include the following:

- Name used in-house for the chemical or mixture of chemicals;
- Correct chemical name for the chemical or each component of a mixture of chemicals;
- Location(s) of the chemical; and
- Location(s) of the posting of MSDSs related to the chemical or mixture of chemicals.

Employee Information and Training

The Branch Safety Officer or a designee will insure that personnel are provided with information and training on hazardous chemicals in their work area at the time of their initial assignment, and whenever a new hazard is introduced into their work area.

Information provided to personnel will consist of the following:

- Requirements of this Hazard Communication Procedure;
- Operations in their work area where hazardous chemicals are present; and
- Location and availability of this Hazard Communication Procedure in this SOP, the Hazardous Chemical Inventory List in the Branch Safety Officer's office, and the locations of MSDSs as stated in Section 3.2.4.

Training provided to personnel will consist of:

- Methods and observations that may be used to detect the presence or release of a hazardous chemical in the work area (e.g., monitoring conducted by the Branch Safety Officer or a designee, continuous monitoring devices, visual appearance or odor of hazardous chemicals when being released, etc.);
- The physical and health hazards of the chemicals in the work area;
- Measures such as appropriate work practices, emergency procedures, and personal protective equipment to be used by personnel to protect themselves from these hazards, and specific procedures to be implemented to protect them from exposure to hazardous chemicals; and
- The details of this Hazard Communication Procedure, including an explanation of the labeling system and the MSDSs, and how personnel can obtain and use the appropriate hazard information.

3.3 Safety Protocols

3.3.1 Field Safety Officer Duties

The following is a list of duties that are required if an individual is designated to be a Field Safety Officer (FSO). Branch safety protocols are to be administrated by the Division Safety, Health, and Environmental Manager (SHEM) and Branch Safety Officer. Safety protocols are to be followed by the FSO as well as each individual that is a part of the field team. Safety during field investigations begins with the individual. However, it is the responsibility of the FSO to plan and coordinate the following during an investigation:

1. Ensure that each member of the field team is up to date on their field safety training (i.e. Annual Safety Refresher, CPR and First Aid) or has received an over-ride by the Branch Chief.
2. Meet with the project leader to gain knowledge of field operations and sampling strategies.
3. Prepare and enforce the field safety plan.
4. Make sure that necessary project specific safety equipment is available and operational.

5. The FSO is also responsible for oversight of safety during the study and has the authority to stop all field activities if a severe safety problem is identified.

3.3.2 Safety Equipment

Field personnel will be provided with the following safety equipment as appropriate:

- rain suit
- snow suit and ski mask (if necessary)
- work gloves
- safety glasses (prescription if necessary)
- goggles
- hearing protection
- hard hat
- steel toe/shank safety boots (leather and rubber)
- first aid supplies.

Field personnel will be responsible for properly operating and maintaining the safety equipment in the field. Should the safety equipment malfunction or be broken, field investigators are responsible for reporting the condition to branch management. The report will include as accurate a description or account of the problem as possible.

Field personnel will not operate any equipment for which they have not received training or have insufficient familiarity to conduct safe operations.

Activities which will require a familiarization exercise for personnel prior to the actual execution of the work include:

- Boating operations (Sec. 3.3.3)
- Trailing of equipment (Sec. 3.3.4)
- Diving operations (Sec 3.3.5)
- Electrofishing (Sec 3.3.6)
- Back-pack shocking operations (Sec 3.3.6)
- Crane truck operations;
- Field monitoring equipment operations
- Helicopter operations (Sec 3.3.7)
- Brush cutting with power equipment;

3.3.3 Boat Operations

The skipper must be familiar with the capabilities of the vessel (care and maintenance of engines, batteries, emergency procedures, and rules of navigation as prepared by the United States Coast Guard). The skipper is responsible for everything that happens on the vessel. The following requirements must be adhered to while using SESD boats.

- Personnel involved with the operation of boats over 20 feet in length must have completed the Boating Skills and Seamanship Course provided by the U.S. Coast Guard

Auxiliary and proved proficiency in boating operations to the Chief of the Ecological Assessment Branch.

- Personnel involved with the operation of boats under 20 feet in length should have completed the Boating Skills and Seamanship Course provided by the U.S. Coast Guard Auxiliary and shown proficiency in operations with an experienced skipper.
- A float plan must be completed and submitted to the safety officer before departure. When participating in field work, the field team should know where each boating team will be working and the expected hour of return.
- When deploying equipment at a station make sure that it is secured and not a danger to fellow workers. When the vessel is underway all equipment should be placed in the boat and secured.
- Boats should be checked out through the Boat Log.
- Upon return to the SESD facility, all temporary/portable gasoline cans must be removed from the boats and placed in the white, flammable storage building.
- Upon return to the SESD facility, all batteries must be removed from the boats and returned to the battery storage location near the Hazardous Materials Storage Building.
- Consumption of alcohol will not be permitted on any boat at any time.
- Smoking will not be permitted on any boat at any time.
- The law prohibits the throwing, discharging, or depositing of any refuse matter of any kind (including trash, garbage, oil or hazardous substances) into the waters of the United States to a distance of three miles from the coastline.

Required Equipment:

Outboard motorboats less than 26 feet in length which are so constructed that entrapment of flammable vapors cannot occur are not required to carry fire extinguishers but they are recommended and should be capable of extinguishing fires involving flammable liquids and grease (class “B” fires).

All recreational boats less than sixteen (16) feet in length, including sailboats and rowboats, and all kayaks and canoes, must carry at least one Coast Guard Approved Type I, II, III or IV Personal Flotation Device (PFD) for each person on board, and carry at least one Type I, II, or III (wearable) PFD for each person on board and one Type IV (throwable) PFD in each boat. Weather or working conditions may dictate the wearing of PFD’s.

All recreational boats 16 feet or more are required to be equipped with visual distress signaling devices (VDS) at all times when operating on coastal waters. Also, boats less than 16 feet long are required to carry visual distress signals when operating on coastal waters at night. Coastal

waters are defined as (1) the ocean (territorial sea); (2) the Great Lakes; (3) bays or sounds that empty into those waters; and (4) rivers over two miles across at the mouth, upstream to a point where they narrow to two miles. The simplest VDS is a bright orange flag bearing a black square and a black circle (other forms can be found in the U.S. Coast Guard Auxillary Boating Skills and Seamanship).

Figure 3-1

FLOAT PLAN

Complete this plan, before going boating and leave it with a reliable person who can be depended upon to notify the Coast Guard, or other rescue organization, should you not return as scheduled. Do not file this plan with the Coast Guard.

PROJECT DATES _____ (if overnight, date returning) _____

1. NAME OF PERSON REPORTING _____
 TELEPHONE NUMBER _____

BOAT MAKE	COLOR	LENGTH	ENGINES	OCCUPANTS
Wahoo	White	26	IB	
Privateer (New)	White	25	2 OB	
Privateer #241	White	24	2 OB	
Parker	White	23	2 OB	
Parker	White	21	OB	
Mako	White	20	OB	
Privateer	White	18	OB	
Shocker (Schaffer)	Tan	18	OB	
Shocker (Fisher)	Green	18	OB	
Boston Whaler	White	17.5	OB	
River Hawk	Camouflage	15	OB	
Jon	Gray	14	OB	
Jon	Gray	14	OB	
Jon	Gray	14	OB	
Jon	Gray	12	OB	
Jon	Gray	12	OB	

4. TRIP EXPECTATIONS: LEAVE AT _____ (TIME)
 FROM _____
 GOING TO _____
 EXPECTED TO RETURN BY _____ (TIME)
 AND IN NO EVENT LATER THAN _____ (TIME)

5. IF NOT RETURNED BY _____ (TIME), CALL THE COAST GUARD, OR
 _____ (LOCAL AUTHORITY)
 TELEPHONE NUMBERS _____

6. SURVIVAL EQUIPMENT: (CHECK AS APPROPRIATE)
 ___ PFDs ___ FLARES ___ MIRROR ___ SMOKE SIGNALS ___ EPIRB

CLOTHING FLASHLIGHT FOOD PADDLES
 WATER OTHERS ANCHOR RAFT OR DINGHY

7. RADIO: YES NO
TYPE _____ FREQS. _____

8. ANY OTHER PERTINENT INFO: _____

9. FOR SINGLE BOAT OPERATION: AUTOMOBILE LICENSE _____ TYPE _____
TRAILER LICENSE _____ COLOR/MAKE OF AUTO _____
WHERE PARKED _____

10. OTHER: _____

Trailing of boats

- Make sure to use the proper class of hitch for the weight of the trailer being towed;
- Check to have the proper size coupler for the ball being used;
- Trailer must have safety chains;
- A tire-pressure gauge should be used to check tire pressure frequently;
- Running lights, brake lights and turn signals must be functioning;
- Every unattached piece of gear in the trailered boat should be firmly secured, and the boat itself should be firmly lashed in place;

A more detailed description of boating regulations and safety can be found in the United States Coast Guard Auxiliary Manual Boating Skills and Seamanship. ISBN #0-930028-03-1

3.3.4 EPA Diving Safety Rules

The following Diving Safety Rules are taken from the U.S. Environmental Protection Agencies Diving Safety Manual (Revision 2.1) Office of Administration and Resources Management Safety, Health and Environmental Management Division, Washington, D.C., May, 1997. For more detailed information on specific types of dives refer to this manual.

Certification

Each diver must have a valid EPA certification or EPA-approved equivalent.

Solo Diving

No one may dive unattended.

Depth Limits

Normally, dives shall not exceed 130 feet. Proposals for planned dives to depths greater than 130 feet will require written approval by the EPA DSB (Diving Safety Board) Chairman or designee. Dives to be conducted to depths greater than 130 ft. require that a working recompression chamber attended by trained personnel be ready at the dive site.

Dives conducted in excess of 100 ft. Require that divers have commensurate training and or experience working at the proposed depth. If no prior experience exists, the diver is to complete a checkout dive to the planned water depth or greater within six weeks of the scheduled working dive. Depending upon conditions and at the recommendation of the divemaster or UDO (Unit Diving Officer), an alternate or redundant air source may be required.

Decompression Tables

All dives using compressed air (i.e., other than NOAA Nitrox I or II) will be conducted using the U.S. Navy Standard Air Decompression Tables. Decompression tables should be copied for use by some photographic method that reproduces an exact copy.

Diving Logs

All EPA divers are required to maintain an EPA personal dive log. The information logged must include the dive location, purpose or function, maximum water depth, and bottom time. In addition, the dive tender shall also record (on the Dive Tender's Log) any other information that is needed by the Divemaster or the UDO. The dive tender must also record the diver's surface interval time for repetitive dives. The diver must log his bottom (subsurface) time and surface interval time in the case of repetitive dives. A dive is completed when a diver leaves the water after completing an activity or, after surfacing for more than ten minutes, before resubmerging to perform a different activity.

- a. Bottom Time is the total elapsed time in next whole minutes from when a diver leaves the surface to begin his descent until the time he begins his direct ascent to the surface. A "dive" is that time and activity spent beneath the surface of the water by a person equipped with diving gear. At the Divemasters discretion, however, when it is practical to do so, bottom time shall be defined as the time from when the diver leaves the surface until the diver returns to the surface (i.e., including the time spent surfacing). This is the more conservative approach, and it has the advantage that both submerging and surfacing are events easily recorded by the tender on the surface.
- b. Surface Interval is the time which the divers have spent on the surface following a dive, beginning as soon as the divers surface and ending as soon as they begin their next descent. For surface intervals less than ten (10) minutes, add the total bottom time of the previous dive to that of the repetitive dive and choose the decompression schedule for the bottom time and the deepest water depth achieved for the sequence.

Decompression Dives

Routine working dives shall not exceed the U.S. Navy no-decompression limits. Diving activities that exceed the limits of no decompression will be permitted only under the following conditions.

- a. Proposal. A detailed dive plan has been reviewed and approved by the UDO or the DSB Chairperson
- b. Competence. The project leader must have demonstrated to the Unit Diving Officer, or his designee, that the Divemaster and all members of the diving team have a thorough knowledge of decompression and repetitive dive principles.
- c. Dive Team. The team must be composed of no fewer than three people: two divers and a tender. All diving activities must have a dive master on-scene.
- d. Equipment. Each participating diver must wear a watch or bottom timer, depth gauge, and have on hand a decompression schedule for the maximum proposed depth of dive. Recompression chamber must be on site and attended by trained operator.

High Altitude Diving

Decompression tables, depth of stops, rate of ascent, and repetitive dive planning must be altered for safe diving at altitudes above 1,000 feet. The current edition of the NOAA Diving Manual should be used as a guide for diving at high altitudes.

Flying After Diving

Wait a minimum surface interval of 12 hours prior to flying after diving. When making daily, multiple dives for several days or making a dive requiring an emergency decompression stop, extend the surface interval beyond twelve hours. Whenever possible wait 24 hours before flying.

Over-Bottom Dives

Dives in waters where a diver could sense a loss of orientation or descent below safe diving depths are to be considered over-bottom dives. No over-bottom dives shall be made unless some direct contact with the surface is maintained, such as net web, a marked line suspended from a surface float, or depth gauges for all participants, which permits the diver to determine when ascent or descent occurs. Additional procedures can be found in the current edition of the NOAA Diving Manual.

Boat Tending

During dives beyond swimming distance from shore or those in areas of strong currents, a small boat with a qualified operator will tend the divers (see item 16.j, regarding use of “diver down” flag).

Ship Activities

When appropriate during ship-related diving activities, the Safety Manual 4 “Dive Safe Ship Operations Checklist” (e.g., NOAA Form 64-3) will be completed and used.

Recompression Chamber

The location, accessibility, and telephone number of all accessible and operable recompression chambers shall be listed in the dive plan and be available to all participating divers during each diving operation.

Emergency Procedures

The Unit Diving Officers, or their designee, with the approval of the EPA Diving Safety Board will prescribe emergency procedures to be used in handling diving-related accidents in the operational area, and all divers shall be familiar with these procedures.

Diving Accident Management Training

All divers shall have diving accident management training, and certification for cardiopulmonary resuscitation (CPR), and first-aid training, and shall complete appropriate refresher training to

maintain skills. First Aid kit will be required.

Emergency Oxygen Resuscitators

An oxygen resuscitator of at least 650 liters (e.g., single Jumbo D or E cylinders or multiple smaller cylinders) capable of ventilating a non-breathing person, shall be immediately available at each dive site, local laws regarding resuscitation equipment permitting. Divers and diver support personnel shall be trained in the use of this equipment. Two such units are highly recommended since both divers of a buddy pair will likely have the same exposure and thus may exhibit the same symptoms.

Equipment.

- a. Life Support. Open-circuit SCUBA using compressed air or oxygen enriched air shall be standard. (There will be a 12 month, after the first use, service interval on regulators.) Other types of equipment (i.e., surface-supplied diving equipment, closed-circuit rebreathers, semiclosed units or other types of diving apparatus using gas mixtures) may be approved for use by the EPA Diving Safety Board DSB Chairperson, Technical Director, or Training Director. Individuals requesting use of these other types of equipment must have been trained and qualified in their use.
- b. Alternate Air Source. To allow for the possible termination of a team member's air supply, each free swimming scuba diver will have available on his system an alternate air source. The alternate air source may be a spare second stage regulator (i.e., an "octopus") or a redundant air system (e.g., "pony" bottle or dual manifold system).
- c. Harness and Weight Belt. All harnesses and weight belts must have a quick release.
- d. Flotation Device. Each free swimming SCUBA diver shall wear an adequate flotation device, such as a buoyancy compensator, that has a means of inflation other than oral (also should have an oral inflation device). (See note regarding variable volume dry suits)
- e. Variable Volume Dry Suits. Variable volume dry suits (VVDS) will be used only after satisfactory completion of a minimum of three (3) hours of training in the use of these suits [two (2) hours of which must have been in open water] from qualified persons designated by the EPA DSB Training Director, or equivalent prior experience verified by a qualified EPA Unit Diving Officer or designee.

Caution: Compensating devices that might obstruct inflation or exhaust valves should not be worn over VVDS. It is recommended that divers wear B.C. Otherwise, they are on their own.

As variable volume suits do not qualify for flotation in use of scuba, another appropriate buoyancy compensating device (BCD) should be used which will not obstruct the valve systems. A BCD is not required or recommended for use in any surface supplied system. If used, in contaminated water environments, the BCD should be capable of being decontaminated or be considered expendable.

[**Note:** VVDS (particularly shell type) manufacturers do not warranty their suits for floatation. Therefore, the user assumes full risk if no additional buoyancy control device (BCD) is used. As such the user should use a BCD which meets the following requirements: 1.) It does not obstruct the operation of the valves on the VVDS; 2.) If the BCD is to be used in chemically, biologically or radiologically contaminated environments, then the BCD must be capable of being decontaminated (interior as well as exterior) by a method appropriate to the contamination present without degradation of the device or operation of the device. If no BCD is available to meet the criteria, the VVDS must be thoroughly inspected for abnormal wear or seam stress which may indicate a potential failure before/after each use. By the same token: dry suit should be “qualified” for contaminated environment use by manufacturer warranty or suit materials (especially seams, seals and closures/zipper) should be compatibility tested.]

- f. Compass. An underwater compass shall be carried by each free swimming diver on all dives.
- g. Depth Gauge. A depth gauge shall be carried by each diver on all dives.
- h. Decompression Meters. Use of decompression meters will be authorized only by the EPA DSB Chairman, the DSB Technical Director of the UDO.
- i. Diving Timer. A diving watch or other suitable timing device shall be worn by each member of a SCUBA diving team. In all cases, an accurate time record of any dive must be kept.
- j. Diving Flag. An appropriate diving flag shall be shown at all times while actively diving.
- k. Air Compressor. No person shall operate a SCUBA air compressor without having first read the instructions and assisted an operator experienced in its operation. An operational log shall be maintained for all EPA SCUBA compressors. Compressed air, from all active compressors, shall be tested every 100 hours or six (6) months, whichever comes first, by an approved method. (Reference: NOAA Diving Manual)
- l. Submersible Pressure Gauge. Each diver shall have a submersible gauge capable of directly reading the breathing gas pressure in his gas supply as an integral part of his scuba regulator system.
- m. Line Cutter/Dive Knife. Each diver shall carry at least one line cutter (e.g., dive knife, scissors, or other cutting tool) for use in release of line entrapment.
- n. Emergency Signaling Device. Each diver shall carry or have as integral part of his dive equipment an emergency signaling device (e.g., whistle, compressed air horn/whistle, mirror, light, or inflatable signal tube).

Equipment Maintenance

All diving gear and accessory equipment shall be maintained in a safe operating condition. Manufacturers' recommended servicing policy shall be followed. Equipment in questionable condition shall be tested, repaired, overhauled, or discarded. Such equipment shall be kept separate from operational equipment and clearly identified. All regulators, regulatory valves, depth gauges,

submersible pressure gauges, and decompression meters must be critically examined, checked for accuracy, and calibrated by a competent mechanic or appropriate specialist every twelve (12) months after the first date of use.. A record of the inspection and repair will be filed with the Unit Diving Officer.

SCUBA Cylinder Inspection and Testing

All SCUBA cylinders must be visually inspected annually by a qualified SCUBA tank inspector, who will attach a dated visual inspection sticker to the cylinder. Cylinders will be hydrostatically tested at least every five (5) years. The dates of the last hydrostatic test must be stamped on the cylinder.

Air

Scuba cylinders shall be charged only with air or an oxygen enriched air mixture certified as meeting established air standards.

Minimum Air Supply

Divers must surface with a minimum of 500 psig in the tank as a safety factor for reaching the shore or boat and to prevent inclusion of water in the cylinder.

3.3.5 Safety Guidelines for Electrofishing:

Electrofishing is a necessary and essential technique for collecting fish samples in lakes, rivers, and streams, but the mixture of electricity and water make for a potential hazard. With safety a primary concern in electrofishing, the following procedures are recommended:

- a. All electrofishing equipment should be inspected before each trip into the field. The crew leader must make all initial settings on the equipment but all crew members should be familiar with the electrical system and basic operation of the boat electrofishing unit and/or backpack shocker. The crew leader will brief the crew on their tasks and responsibilities prior to the initiation of the electrofishing activities. Electrofishing should proceed slowly and deliberately. All crew members must be on guard for potential hazards (logs, overhanging branches, etc.) during the electrofishing operation. The crew leader will also be responsible for evaluating the fitness of the sampling crew and scheduling adequate rest periods.
- b. Electrical power on boat units is always supplied via a control box/generator. Power for electrofishing must never be supplied from the boat's main power source. A circuit breaker must be used with a generator to protect the generator from an electrical overload. Before refueling the generator, allow the engine on the generator to cool down in case fuel is accidentally spilled. In addition, the crew working in close proximity to the generator must wear hearing protection while the generator is in use.
- c. All samplers must wear rubber gloves (class 0, 1, 2, or 3 lineman's gloves) during electrofishing operations. The crew leader will also decide whether the sampling crew will

wear hip boots or chest waders. Hats, polarized sunglasses, and rain suits may also be worn when sampling. The crew leader will indicate to the members of the sampling crew when it is safe to remove any protective equipment. The crew leader is responsible for the safety of the crew. If some activity or procedure appears unsafe, electrofishing must be terminated and the crew must discuss the “safety concern” and what corrective action needs to be taken.

d. Other safety requirements for electrofishing operations are:

- All electrofishing operations must be discontinued at the first sign of lightening, heavy rains, or high winds;
- Avoid electrofishing during high water periods and in public recreation areas;
- Remove all rings and metallic jewelry prior to electrofishing. Be certain that all ancillary equipment is made of non-conducting material (e.g., buckets, nets);
- **Never** electrofish alone;
- Fishing electrodes must never be energized unless immersed in water.

The following will apply mainly to the backpack shocking unit but also should be considered in all electrofishing operations:

- e. Except for leads and electrodes, the entire backpack shocker unit should be housed in a weatherproof metallic container that is securely fastened to a comfortable pack frame. The power source is either a 12-volt sealed battery (gel cells preferred) or 115- volt AC generator. The DC method is preferred unless sampling in waters with low conductivity (<10 micromhos). If a battery is used, the unit should allow selection of output voltage, frequency, and duty cycle. Lightweight generators can be carried on a pack frame and connected to electrodes with the appropriate connections. Positively activated switches (“tip switches”) are an essential safety feature. The power source should have both automatic and manual circuit breakers.
- All samplers, including the person operating the shocking unit, must wear rubber gloves (class 0, 1, 2, or 3 lineman’s gloves) during backpack electrofishing operations.
 - Never touch the water or electrodes while the current is on
 - All members of the sampling crew should be aware of the person operating the shocking unit’s position and intentions at all times.

3.3.6 Helicopter Safety:

Regulations:

1. Aircraft and pilots engaged for sampling routines must be approved and licensed by the Office of Aircraft Safety (OAS)

2. Participants in helicopter work should have completed courses in Basic Aviation Safety and Aircraft Water Ditching And Survival sponsored by the Office of Aircraft Safety.

Safety Around Helicopters:

1. Approach and depart helicopter from the side or front in a crouching position, in view of the pilot and with the pilot's permission.
2. Approach and depart on the down slope side (to avoid main rotor).
3. Approach and depart in pilot's field of vision (never towards the tail rotor).
4. Use chin strap or secure hard hat when working around main rotor.
5. Carry tools horizontally, below waist level (never upright or over the shoulder).
6. Fasten seat belt upon entering helicopter and leave buckled until pilot signals to exit. Fasten seat belt behind you before leaving.
7. Use the door latches as instructed; caution should be exercised around moving parts or plexiglass.
8. Keep landing areas clear of loose articles that may "fly" in the rotor downwash.
9. Do not throw items from the helicopter.
10. Eye and hearing protection should be worn when working in close proximity to helicopters.
11. Secure items internally and externally on the helicopter. Provide the pilot with accurate weights and types of baggage or cargo to be secured.
12. Passengers should exit the aircraft when it is being refueled unless specifically told not to by the pilot.

Personal Protective Equipment:

1. Utilization of the SPH-4 or SPH-5 Gentex Corporation helmet is mandatory anytime the rotor is operational.
2. A loose fitting Nomex flight suit should be fastened over the boots. Sleeves are to be worn down, zipper zipped to the top, and collar turned up. Nomex gloves shall be worn under the sleeve cuffs to prevent accidental snagging. To obtain maximum protection, it is important to wear cotton, wool, cotton-wool blend, or Nomex undergarments. Synthetic or petroleum-based materials (nylon, polyester, polypropylene, or plastic) impose a hazard of high flammability and melting to the skin when worn under fire resistant clothing.
3. Leather boots which extend above the ankle. An exception would be working in an

environment (water, snow) not conducive to wearing leather boots.

3.3.7 OSHA Confined Space Entry

According to 29 CFR Part 1910.146 an individual must have a permit to enter a space that meets the following definition for a confined space. Confined space means a space that is: 1) large enough and so configured that an investigator can bodily enter and perform assigned work; 2) has limited or restricted means for entry or exit (e.g., tanks, vessels, silos, storage bins, hoppers, vaults, or pits are spaces that may have limited means of entry); and 3) is not designed for continuous occupancy. Field investigators shall not enter a space if it meets this definition.

3.3.8 Training Status Tracking System

A computer system is used for tracking the status of required safety training for all personnel involved in field operations within the Division. The system tracks the following safety training:

- Medical monitoring physical (annual renewal);
- 40-hour hazardous waste training (no required renewal);
- 8-hour refresher training (annual renewal);
- Cardio-pulmonary resuscitation (CPR) certification (annual renewal);
- First aid certification (tri-annual renewal);
- Fit testing (annual renewal);
- Fire extinguisher operation (annual renewal);
- Hazard Communication (no required renewal).

It is the responsibility of the Branch safety officer or their designee to notify field personnel or their supervisor when renewals of required training are due. Notification will be at least 60 days prior to the actual renewal date. Scheduling training will be the responsibility of each individual unless otherwise stipulated in the notification. Upon successful completion of training, a copy of the certificate received will be sent by the individual to the Branch safety officer who will then forward the information to the SHEM for inclusion in the safety training file.

In the event that a field investigator's OSHA required training has lapsed by more than 90 days, the individual will not be allowed to participate in field work. When lapses in training required by EPA policy occur, the individual will be allowed to perform field work at the discretion of the Branch Chief. The individual and their supervisor will be notified of the change in status. Upon successful completion of the required training, the individual and their supervisor will be notified of their return to prior status.

3.3.9 Field Operations

Prior to and during the initial field operations, the work location will be surveyed by field personnel to:

- Determine the hazards that may exist which could affect field personnel.
- Verify existing information or obtain new information about the location.

Field attire for Ecological Assessment Branch personnel conducting routine ecological assessments will be at the discretion of the individual. Ecological assessments are considered routine when the working area contains no known or anticipated hazards and work conditions preclude splashes, immersion or the potential for unexpected inhalation of or contact with hazardous levels of any chemical. Shirts, pants (shorts) and shoes are typical, except for operations where personnel are working in and under water. Swim wear is appropriate in those circumstances. Shoes must be worn during boating operations. Optional attire may include:

- gloves
- rubber boots with steel toe and shank
- disposable boot covers
- safety glasses, goggles, or face shield (not for chemical splash protection)
- hard hat, sun hat
- sunscreen
- thermal weather protection (coat, overalls, sweater, hat, rain gear, cool vests, and Heat stress monitors.

Stress

Field personnel on sites are exposed to both psychological and physiological stress. Psychological stress is countered with adequate training and job proficiency. Physiological stress is primarily due to exposure of the worker to extremes of heat and cold.

Heat Stress

Heat stress can be the result of working during hot weather or wearing protective clothing that inhibits natural ventilation. It can occur even under moderate temperature conditions. Whenever possible, work should be scheduled during cooler parts of the day or night. The following protocols are to be used to counter heat stress:

- Allow workers to replace lost body fluids.
- Adequate shade will be provided to shelter workers from direct exposure to the sun during rest periods.
- Work teams will be rotated so that an individuals time on stressful jobs is minimized.
- Field personnel are encouraged to maintain their physical fitness.
- Intake of diuretics (coffee or alcohol) should be minimized prior to field work.

Cold Stress

Exposure to extreme cold can result in hypothermia. Field work during periods of low temperatures and high winds should be conducted to minimize the possibility of hypothermia. The following protocols are to be followed:

- Workers will dress as warmly as possible using the principle of layering their clothing to maximize protection.
- Gloves should be worn when handling metal equipment.
- When possible, work tours will be limited to minimize exposure to the cold.
- Warm shelter will be made available for workers during breaks. Use of vehicles for warm shelter is discouraged due to the possibility of carbon monoxide exposure.

The FSO will carefully observe workers for signs of hypothermia/frostbite.

3.3.10 Reconnaissance Safety Plans and Field Safety Plans

When a field reconnaissance is to be done to consider sites to be used in the regular field study, an abbreviated safety plan must be completed and contain the following item:

- a map of the probable sites to be visited
- the location of the nearest hospital
- the expected duration of the trip

If the reconnaissance is to be a day trip, the safety information for the reconnaissance is to be given to the Branch Secretary who will attach it to the day trip form and after the trip is completed will give the information to the Branch Safety Officer for filing.

Field safety plans will be developed for every field project conducted. The plan will use the form included in this section. MSDSs will be attached for contaminants anticipated at the location.. The plan will be submitted to the Branch Safety Officer and the Section Chief for approval.

Prior to commencing site activities, investigators will be briefed on the contents of the safety plan. The plan's emergency instructions and directions to the closest hospital will be posted in a conspicuous location at a designated location and in each field vehicle/boat. When there is more than one organization involved at the location, the development of the safety plan should be coordinated among the various groups.

FIELD SAFETY PLAN

SAFETY PLAN	
Site Name:	Contact:
Address:	
Phone Number:	
Purpose of Visit:	
Proposed Date of Work:	
Directions to Site:	

SITE INVESTIGATION TEAM:

PERSONNEL *	SAFETY CATEGORY	RESPONSIBILITIES
* All employees have been trained/medically monitored in accordance with OSHA 29 CFR 1910.12 requirements and US-EPA Region IV Field Health and Safety Manual, 1990 edition.		

PLAN PREPARATION:

Site Safety Officer		Date
Branch Safety Officer		Date
Section Chief:		Date

SITE STATUS:

Active	Inactive	Unknown
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EMERGENCY INFORMATION:

Local Resources:

Ambulance (Name):	Phone:
Hospital (Name):	Phone:
Police (Local or State):	Phone:
Fire Department:	Phone:

Office Resources:

OFFICE/POINT of CONTACT	WORK PHONE	HOME PHONE
EAB Office - Marilyn Vidmar	(706) 355-8701	
EPA - Emergency Response - Atlanta	(800) 564-7577	
Ecological Evaluation Section - Philip Murphy	(706) 355-8711	(706) 546-7895
Toxics Section - Anne Keller	(706) 355-8767	(706) 227-2336
Safety - Phyllis Meyer	(706) 355-8709	(706) 549-8533
OHSD - Jim Gray	(706) 355-8613	(706) 742-8467
Branch Chief - Bill Bokey	(706) 355-8604	(706) 549-2611

EMERGENCY CONTACTS:

Poison Control Center	Phone: (800) 282-5846
National Response Ctr (ENVIRONMENTAL EMERGENCY ONLY)	Phone: (800) 424-8802

Directions to Hospital (Attach Map if Available):

SAFETY AND HEALTH RISK ANALYSIS

Waste Types/Chemicals (Attach MSDS for each):

HAZARD EVALUATION:

Known or Suspected Hazardous/Toxic Materials

OVERALL HAZARD:

SERIOUS	MODERATE	LOW	UNKNOWN
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SITE PERIMETER ESTABLISHMENT:

Map/Sketch attached?	
Perimeter identified?	
Zone(s) of contamination identified?	

Modifications of typical field attire:

Field Dress:	
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Above Ground Utilities:

All above ground utilities must be located prior to commencing augering activities. Locations of power lines, telephone lines, video cables, guy wires, and other objects which could pose a hazard to personnel operating a hand auger with multiple extensions. The FSO will insure that all operations are kept well clear of such hazards.

3.4 Transport of Hazardous Materials

Vehicles operated by SESD are exempt from DOT regulations governing the transport of hazardous materials. However, to protect the safety and health of SESD personnel, and to minimize the potential for accidental releases to the environment, the following procedures will be adhered to when hazardous materials are carried aboard SESD vehicles.

1. MSDS's for the materials will be added to the vehicle's MSDS notebook
2. Materials will be packaged in DOT approved containers. Batteries will be in approved battery boxes with lids secured.
3. Appropriate spill containment and clean-up materials will be available when quantities of hazardous materials being transported exceed 5 gallons.
4. DOT compatibility requirements will be observed.
5. DOT labeling requirements for containers of hazardous materials as well as the labeling requirements given in the Hazard Communication Plan will be followed.
6. Gasoline in tanks for powered equipment or boats shall not be carried in the passenger carrying compartment of vehicles.
7. Compressed gas cylinders, with safety caps in place, must be secured at all times.

3.4.1. Shipping note

When samples are to be shipped by common carrier or sent through the United States mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of 40 CFR, Part 136, Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric Acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium Hydroxide (NaOH) in water solutions at concentrations of 0.08% by weight or less (pH about 12.30 or less). This footnote is wholly reproduced from 40 CFR 136.3, which is definitive.

SECTION 4 SAMPLING DESIGN AND QUALITY ASSURANCE PROCEDURES

SECTION OBJECTIVES:

- Define planning and quality assurance elements that must be incorporated in all sampling operations.
- Define sampling quality assurance procedures.

4.1 Introduction

This section discusses the standard practices and procedures used by Branch personnel during field operations to ensure the collection of representative samples. Sampling activities conducted by field investigators are conducted with the expectation that information obtained may be used for enforcement purposes, unless specifically stated to the contrary in advance of the field investigation. Therefore, correct use of proper sampling procedures is essential. Collection of representative samples depends upon:

- Ensuring that the sample is representative of the material being sampled.
- The use of proper sampling, sample handling, preservation, and quality control techniques.

4.2 Definitions

Sample -- part of a larger lot, usually an area, a volume, or a period of time.

Representative Sample -- a sample that reflects one or more characteristics of a population.

Sample Representativeness -- the degree to which a set of samples defines the characteristics of a population, where each sample has an equal probability of yielding the same result.

Variability -- the range or “distribution” of results around the mean value obtained from samples within a population. There are three types of variability which must be measured or otherwise accounted for in field sampling.

1. Temporal Variability

Temporal variability is the range of results due to changes in contaminant concentrations over time. An example would be the range of concentrations obtained for a given

parameter in wastewater samples collected at different times from an outfall where contaminant concentrations vary over time.

2. Spacial Variability

Spacial variability is the range of results due to changes in contaminant concentrations as a function of their location. An example would be the range of concentrations obtained for a given parameter in surface soil from a site where discrete "hot spots" are present due to localized releases of contaminants on otherwise uncontaminated soil.

3. Sample Handling Variability

Sample handling variability is the range of results due to the sample collection and handling by the sampler. This variability manifests itself as a positive bias due to errors such as unclean sampling equipment, cross contamination, etc., or a negative bias due to improper containers or sample preservation.

Accuracy -- a measure of agreement between the true value and the measured value of a parameter.

Precision -- measure of the agreement among individual measurements of identical samples.

Bias -- consistent under or over-estimation of the true value due to sampling errors, sample handling errors, or analytical errors.

Grab Sample -- an individual sample collected from a single location at a specific time or period of time. Grab samples are generally authoritative in nature.

Composite Samples -- a sample collected over a temporal or spacial range that typically consists of a series of discrete, equal samples (or "aliquots") which are combined or "composited". Four types of composite samples are listed below:

1. Time Composite (TC) - a sample comprised of a varying number of discrete samples (aliquots) collected at equal time intervals during the compositing period. The TC sample is typically used to sample wastewater or streams.
2. Flow Proportioned Composite (FPC) - a sample collected proportional to the flow during the compositing period by either a time-varying/constant volume (TVCV) or time-constant/varying volume (TCVV) method. The TVCV method is typically used with automatic samplers that are paced by a flow meter. The TCVV method is a manual method that individually proportions a series of discretely collected aliquots. The FPC is typically used when sampling wastewater.
3. Areal Composite - sample composited from individual, equal aliquots collected on an areal or horizontal cross-sectional basis. Each aliquot is collected in an identical manner.

Examples include sediment composites from quarter-point sampling of streams and soil samples from within grids.

4. Vertical Composite - a sample composited from individual, equal aliquots collected from a vertical cross section. Each aliquot is collected in an identical manner. Examples include vertical profiles of soil/sediment columns, lakes, and estuaries.

Quality Control Samples

Quality control samples are collected during field studies for various purposes which include the isolation of site effects (control samples), define background conditions (background sample), evaluate field/laboratory variability (spikes and blanks, trip blanks, duplicate, split samples).

The definitions for specific quality control samples are listed below:

Control Sample -- typically a discrete grab sample collected to isolate a source of contamination. Isolation of a source could require the collection of both an upstream sample at a location where the medium being studied is unaffected by the site being studied, as well as a downstream control which could be affected by contaminants contributed from the site under study.

Background Sample -- a sample (usually a grab sample) collected from an area, water body, or site similar to the one being studied, but located in an area known or thought to be free from pollutants of concern.

Split Sample -- a sample which has been portioned into two or more containers from a single sample container or sample mixing container. The primary purpose of a split sample is to measure sample handling variability.

Duplicate Sample -- two or more samples collected from a common source. The purpose of a duplicate sample is to estimate the variability of a given characteristic or contaminant associated with a population.

Trip Blanks -- a sample which is prepared prior to the sampling event in the actual container and is stored with the investigative samples throughout the sampling event. They are then packaged for shipment with the other samples and submitted for analysis. At no time after their preparation are trip blanks to be opened before they reach the laboratory. Trip blanks are used to determine if samples were contaminated during storage and/or transportation back to the laboratory (a measure of sample handling variability resulting in positive bias in contaminant concentration). If samples are to be shipped, trip blanks are to be provided with each shipment but not for each cooler.

Temperature Blanks-- a container of water shipped with each cooler of samples requiring preservation by cooling to 4°C (ice). The temperature of the blanks are measured at the time of sample receipt by the laboratory. No temperature measurement is necessary for samples

designated as “waste”.

Spikes -- a sample with known concentrations of contaminants. Spike samples are often packaged for shipment with other samples and sent for analysis. At no time after their preparation are the sample containers to be opened before they reach the laboratory. Spiked samples are normally sent with each shipment to contract laboratories only. Spiked samples are used to measure negative bias due to sample handling or analytical procedures, or to assess the performance of a laboratory.

Equipment Field Blanks -- a sample collected using organic-free water which has been run over/through sample collection equipment. These samples are used to determine if contaminants have been introduced by contact of the sample medium with sampling equipment. Equipment field blanks are often associated with collecting rinse blanks of equipment that has been field cleaned

Pre- and Post-Preservative Blanks -- a sample that is prepared **in the field** and used to determine if the preservative used during field operations was contaminated, thereby causing a positive bias in the contaminant concentration. One preservative blank should be collected for each bottle of preservative used to preserve samples. On small studies, usually only a post-preservative blank is prepared at the end of all sampling activities. On studies extending beyond one week, a pre-preservative blank should also be prepared prior to beginning sampling activities. At the discretion of the project leader, additional preservative blanks can be prepared at intervals throughout the field investigation. These blanks are prepared by putting organic/analyte-free water in the container and then preserving the sample with the appropriate preservative.

Field Blanks -- a sample that is prepared in the field to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected (for example air-borne dust or organic vapors which could contaminate a soil sample). Organic-free water is taken to the field in sealed containers or generated on-site. The water is poured into the appropriate sample containers at pre-designated locations at the site. Field blanks should be collected in dusty environments and/or from areas where volatile organic contamination is present in the atmosphere and originating from a source other than the source being sampled.

4.3 Sampling Design

4.3.1 Introduction

Development of a sampling design may follow the seven steps outlined in the EPA publication, "Guidance for the Data Quality Objectives Process" (1). The Data Quality Objectives (DQOs) process is a logical step-by-step method of identifying the study objective, defining the appropriate type of data to collect, clarifying the decisions that will be based on the data collected, and considering the potential limitations with alternate sampling designs.

Sampling designs are typically either non-probabilistic (directed sampling designs) or probabilistic (random sampling designs) in nature. The sampling design ultimately must meet specific study objectives. The location and frequency of sampling (number of samples) should be clearly outlined in the sampling design, as well as provisions for access to all areas of the site, the use of special sampling equipment, etc. Development of the sampling design in the context of DQOs and sampling optimization are discussed in the ASTM documents "Standard Practice for Generation of Environmental Data Related to Waste Management Activities: Development of Data Quality Objectives" (2), and "Standard Guide for the Generation of Environmental Data Related to Waste Management Activities" (3).

4.3.2 Representative Sampling

A "representative sample" is often defined as a sample that reflects one or more characteristics of the population being sampled. For example, the characteristic which is desired to be reflected by the sample may be the average, minimum, or maximum concentration of a constituent of concern. Ultimately a representative sample is defined by the study objectives. For instance, the objective of the study may be to determine the maximum concentration of lead in the sludge from a surface impoundment. One sample collected near the inlet to the impoundment may provide that information. The collection of a representative sample may be influenced by factors such as equipment design, sampling techniques, and sample handling.

4.3.3 Stratification and Heterogeneous Sample Matrices

Environmental media may be stratified, i.e., different portions of the population, which may be separated temporally or spatially, may have similar characteristics or properties which are different from adjacent portions of the population. An example would be a landfill that contains a trench which received an industrial waste contaminated with chromium was the contaminant of concern. Stratified sampling designs are discussed later which incorporate independent sampling of each strata, thereby reducing the number of samples required.

Some environmental matrices may be, for purposes of the field investigation, homogeneous (for instance the surface water in a limited segment of a small stream). If the composition of the matrix and the distribution of contaminants are known, or can be estimated, less sampling may be necessary to define the properties of interest. An estimate of the variability in contaminant distribution may be based on knowledge, or determined by preliminary sampling. The more heterogeneous the matrix, the greater the planning and sampling requirements.

A population could also have very localized strata or areas of contamination that are referred to as "hot spots". Specific procedures for hot spot identification and characterization are available in Statistical Methods for Environmental Pollution Monitoring (4).

4.3.4 Specific Sampling Designs

Sampling strategies used by the Branch typically fall into two general groups: **directed** or **probabilistic**. Directed or "authoritative" approaches typically rely on the judgement and experience

of the investigators, as well as available information on the matrix of concern. Probabilistic, or "statistical" approaches may be appropriate when estimates on uncertainty and specific confidence levels in the results are required. The probabilistic approaches include: simple random sampling, stratified random sampling, and systematic grid sampling. The main feature of a probabilistic approach is that each location at the site has an equal probability of being sampled, therefore statistical bias is minimized.

4.3.5 Determining the Number of Samples to Collect

The number of samples to collect as part of a sampling design will typically be based on several factors, e.g., the study objectives, properties of the matrix, degree of confidence required, access to sampling points, and resource constraints. Additionally, water quality and ecological studies conducted by EAB usually involve *in situ* measurements that employ specific methods which inherently provide guidance or sampling protocols. Additional programmatic guidance is available for various types of investigations (ie. RBP, ocean disposal, etc.)

4.3.6 Authoritative Sampling

Authoritative sampling is based on the judgement of the investigator, and does not necessarily result in a sample that reflects the average characteristics of the entire population or aquatic community. There are two types of authoritative designs: judgmental sampling and biased sampling. Judgmental sampling uses the knowledge and experience of the investigator to attempt to derive "average" conditions at a site. In contrast, biased sampling attempts to determine the maximum or minimum value for the parameter or process of concern.

The primary advantages of authoritative sampling are the designs tend to be quick and simple to implement, and the designs have relatively low costs. Because the experience of the investigator is often the basis for sample collection, personal bias (depending on the study objectives) may be introduced and should be recognized.

4.3.7 Simple Random Sampling

Simple random sampling insures that each element in the population has an equal chance of being included in the sample. This is often the method of choice when, for purposes of characterization studies when the population is randomly heterogeneous. If the population contains trends or patterns of contamination, a stratified random sampling or systematic grid sampling strategy would be more appropriate.

4.3.8 Stratified Random Sampling

Stratified random sampling may be useful when distinct strata or "homogeneous sub-groups" are identified within the population. The strata could be located in different areas of the population, or the strata may be comprised of different layers. This approach is useful when the individual strata may be considered internally homogeneous, or at least have less internal variation, in what would otherwise be considered a heterogeneous population. Information on the site is usually required to

establish the location of individual strata. A grid may be utilized for sampling several horizontal layers if the strata are horizontally oriented. A simple random sampling approach is typically utilized for sample collection within each strata. The use of a stratified random sampling strategy may result in the collection of fewer samples.

4.3.9 Systematic Grid Sampling

Systematic grid sampling involves the collection of samples at fixed intervals when the parameter or targeted community component (ie sediment, organism, etc.) is assumed to be randomly distributed. This method is commonly used with populations when estimating trends or patterns. This approach may also be useful for identifying the presence of strata within the population. The grid and starting points should be randomly laid out over the site, yet the method allows for rather easy location of exact sample locations within each grid. Also, the grid size would typically be adjusted according to the number of samples that are required.

4.3.10 Adaptive Clustering Sampling

Adaptive sampling designs are ones in which additional decisions units or sample locations are selected depending on the interpretation of measurements or observations made during an initial survey (8). Additional sample locations are selected when a parameter of concern requires more intense sampling to meet DQO's or the best professional judgement of the scientist. Simple random or systematic grid sampling can be used in conjunction with adaptive cluster sampling designs.

4.4 Surface Water and Sediment Sampling Designs

4.4.1 Introduction

Surface water quality and sediment characterization studies (both physical and chemical) are consistently major components of EAB activities. Data are collected for purposes such as wasteload allocations, water quality model calibration/verification, oxygen dynamics, biological community characterizations and contamination, TMDL's as well as many other technical support functions. Accordingly, the study design is dependent upon the type of investigation and is ultimately governed by the data quality objectives and the professional judgement of the project leader and/or scientific team. Subsequent sections of this SOP contain guidance related to activities specific to those sections. However, following below are general considerations associated with designing water and sediment sampling activities. These considerations are not to be viewed as prescriptive or absolute, nor substituted for best professional judgement.

4.4.2 Sampling Site Selection

The following factors may be considered in the selection of surface water and sediment sampling locations:

- Study objectives;
- Water use;

- Point source discharges;
- Nonpoint source discharges;
- Tributary locations;
- Changes in stream characteristics;
- Type of stream bed;
- Depth of stream;
- Turbulence;
- Presence of structures (weirs, dams, etc.);
- Accessibility; and
- Tidal effect (estuarine).

If the study objective is to investigate a specific water use such as a source of water supply, recreation, or other discrete use, then considerations such as accessibility, flow, velocity, physical characteristics, etc., are not critical from a water quality investigation standpoint.

If the objective of a water quality study is to determine patterns of pollution, provide data for mathematical modeling purposes, conduct assimilative capacity studies, etc., where more than a small area or short stream reach is to be investigated, then several factors become interrelated and need to be considered in sampling location selection. An excellent guide to conducting surface water stream studies is F. W. Kittrells, "A Practical Guide to Water Quality Studies" (7).

Before any sampling is conducted, an initial reconnaissance may aid in the selection of sampling locations. Bridges and piers are normally good choices as sites since they provide ready access and permit water sampling at any point across the width of the water body. However, these structures may alter the nature of water flow and thus influence sediment deposition or scouring. Additionally, bridges and piers are not always located in desirable locations with reference to waste sources, tributaries, etc. Wading for water samples in lakes, ponds, and slow-moving rivers and streams must be done with caution since bottom deposits are easily disturbed, thereby resulting in increased sediments in the overlying water column. On the other hand, wadeable areas may be best for sediment sampling. In slow-moving or deep water, a boat is usually required for sampling. Sampling station locations can be chosen without regard to other means of access if the stream is navigable by boat, especially in estuarine systems where boats frequently provide the only access to critical sampling locations.

Fresh water environments are commonly separated into two types:

- Lotic - Flowing water, including rivers, creeks, and small to intermittent streams; and
- Lentic - Water that is contained, with restricted flow including lakes, ponds, and manmade impoundments

Since these waterways differ considerably in general characteristics, site selection must be adapted to each. Estuarine environments are a special case and are discussed separately.

4.4.3 Rivers, Streams, and Creeks

In the selection of a surface water sampling site in rivers, streams, or creeks, areas that exhibit the greatest degree of cross-sectional homogeneity should be located unless study objectives dictate otherwise. When available, previously collected data may indicate if potential sampling locations are well mixed or vertically or horizontally stratified. Since mixing is principally governed by turbulence and water velocity, the selection of a site immediately downstream of a riffle area will insure good vertical mixing. These locations are also likely areas for deposition of sediments since the greatest deposition occurs where stream velocities decrease provided that the distance is far enough downstream from the riffle area for the water to become quiescent. Horizontal (cross-channel) mixing occurs in constrictions in the channel, but because of velocity increases, the stream bottom may be scoured, and therefore, a constriction is a poor location to collect sediment.

Typical sediment depositional areas are located:

- Inside of river bends;
- Downstream of islands;
- Downstream of obstructions; and
- Areas of flow reversals.

Sites that are located immediately upstream or downstream from the confluence of two streams or rivers should generally be avoided since flows from two tributaries may not immediately mix, and at times due to possible backflow can upset the depositional flow patterns.

When several locations along a stream reach are to be sampled, they should be strategically located:

- At intervals based on time-of-water-travel, not distance, e.g., sampling stations may be located about one-half day time-of-water-travel for the first three days downstream of a waste source (the first six stations) and then approximately one day through the remaining distance.
- At the same locations if possible, when the data collected is to be compared to a previous study.
- Station locations should be such so as to integrate the physical change that occurs in the stream channel.
- To isolate major discharges as well as major tributaries. Dams and weirs cause changes in the physical characteristics of a stream. They usually create quiet, deep pools in river reaches that previously were swift and shallow. The affect of such impoundments should be considered in regards to sampling locations and study objectives.

When major changes occur in a stream reach, an upstream station, a downstream station, and an intermediate station may be appropriate. Major changes may consist of:

- A wastewater discharge;
- A tributary inflow;

- Non-point source discharge (farms or industrial sites); and
- A significant difference in channel characteristics.

To determine the effects of certain discharges or tributary streams on ambient water quality, stations should be located both upstream and downstream from the discharges. In addition to the upstream and downstream stations bracketing a tributary, a station should be established on the tributary at a location upstream and out of the influence of the receiving stream if contributions to the tributary flow are anticipated to substantially influence the parameters of concern.

Actual sampling locations will vary with the size of the water body and the mixing characteristics of the stream or river. Generally, for small streams a sampling site should be selected where the water is well mixed. In such cases, a single grab sample taken at mid-depth at the center of the channel is adequate to represent the entire cross-section. Large streams may require vertical or horizontal independent or composite sampling based on the study object uses.

Composit sampling must take into account various factors, with stream geometry and flow being major considerations. The number of composites required and the number of depths sampled for each are usually determined in the field by the investigators. This determination is based on a reasonable balance between the following two considerations:

- The larger the number of subsamples, the more closely the composite sample will represent the water body; and
- Subsample collection is time-consuming and expensive, and increases the chance of cross-contamination.

In most circumstances, a number of sediment samples should be collected along a cross-section of a river or stream in order to adequately characterize the bed material. A common procedure is to sample at quarter points along the cross-section. When the sampling technique or equipment requires that the samples be extruded or transferred on site, they may be combined into a single composite sample. However, samples of dissimilar composition should not be combined but should be stored for separate analysis in the laboratory. Diver deployed coring tubes is one method used often by EAB to enhance the representativeness of sediment samples.

4.4.4 Lakes, Ponds, and Impoundments

Lakes, ponds, and impoundments have a much greater tendency to stratify than rivers and streams. The relative lack of mixing generally requires that more samples be obtained. Occasionally, an extreme turbidity difference may occur where a highly turbid river enters a lake. For these situations, each layer of the vertically stratified water column needs to be considered and may require independent sampling based on study objectives. Since the stratification is caused by water temperature differences, the cooler, more dense river water is beneath the warmer lake water. A temperature profile of the water column as well as visual observation of lake samples can often detect the different layers which can be sampled separately. Light extinction profiles are also useful to distinguish water strata.

The number of water sampling stations on a lake, pond, or impoundment will vary with the objective of the investigation as well as the size and shape of the basin. In ponds and small impoundments, a single vertical composite at the deepest point may be sufficient. Dissolved oxygen, pH, and temperature are generally profiled or measured for each vertical composite aliquot. In naturally-formed ponds, the deepest point is usually near the center; in impoundments, the deepest point is usually near the dam.

In lakes and larger impoundments, several vertical subsamples may be composited to form a single sample. These vertical sampling locations are often collected along a transect or grid. The number of vertical subsamples and the depths at which subsamples are taken are usually at the discretion of the field investigators. In some cases, it may be of interest to collect separate composites of epilimnetic and hypolimnetic zones (above and below the thermocline or depth of greatest temperature change).

The shape, inflow pattern, bathymetry, and circulation must be considered when selecting sediment sampling sites in lakes or reservoirs. Generally, the coarser grained sediments are deposited near the headwaters of the reservoir, and the bed sediments near the center of the water mass will be composed of fine-grained materials.

4.4.5 Estuarine Waters

Estuarine areas are zones where inland freshwaters (both surface and ground) mix with oceanic saline waters. Estuaries are generally categorized into three types, dependent upon freshwater inflow and mixing properties:

- Mixed estuary -- Characterized by an absence of vertical halocline (gradual or no marked increase in salinity in the water column) and a gradual increase in salinity seaward. Typically this type of estuary is found in major freshwater sheetflow areas, featuring shallow depths.
- Salt wedge estuary -- Characterized by a sharp vertical increase in salinity and channelized freshwater inflow into a deep estuary. In these estuaries, the vertical mixing forces cannot override the density differential between fresh and saline waters. In effect, a salt wedge tapering inland moves horizontally, back and forth, with the tidal phase.
- Oceanic estuary -- Characterized by salinities approaching full strength oceanic waters. Seasonally, freshwater inflow is small with the preponderance of the fresh and saline water mixing occurring near, or at, the shore line.

Southeastern estuaries often exhibit all these characteristics both spatially and seasonally. A reconnaissance investigation should be conducted for each estuarine study unless prior knowledge of the estuarine type is available. The reconnaissance should focus upon the freshwater and oceanic water dynamics with respect to the study objective. National Oceanic Atmospheric Administration (NOAA) tide tables and United States Geological Survey (USGS) freshwater surface water flow records provide valuable insights into the estuary hydrodynamics. The basic in-situ measurement

tools for reconnaissance are:

- Boat;
- Recording fathometer;
- Salinometer;
- Multiparameter sonde for DO, temp, sal; and
- Global Positioning System (GPS) equipment and charts.

These instruments coupled with the study objective or pollution source location, whether it is a point or nonpoint source problem, provide the focus for selecting sampling locations.

Water sampling in estuarine areas is normally based upon the tidal phases, with samples collected on successive slack tides unless study objectives dictate otherwise. All estuarine sampling should include vertical salinity measurements at one to five-foot increments coupled with vertical dissolved oxygen and temperature profiles. A variety of water sampling devices are used, but in general, the Van Dorn (or similar type) horizontal sampler or pumps are suitable.

Generally, estuarine investigations are two phased, with study investigations conducted during wet and dry periods. Depending upon the freshwater inflow sources, estuarine water quality dynamics cannot normally be determined by a single season study.

4.5 Data Quality Objectives

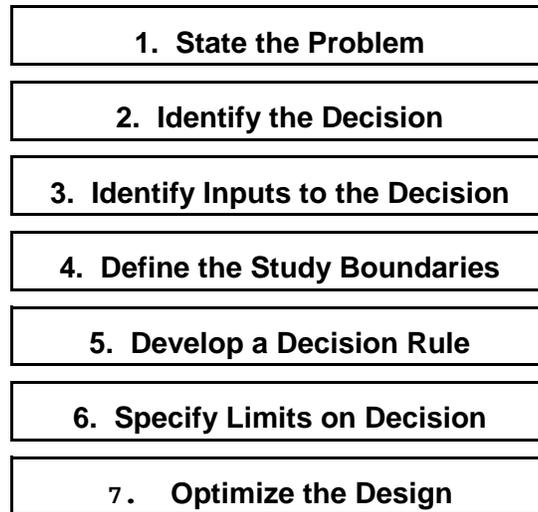
PERFORMANCE OBJECTIVE:

- To provide guidance on systematic planning and the use of DQO Process.
- To describe the steps, the purpose and activities of DQO Process

The US-EPA's Quality Assurance Division has developed guidance as part of its Quality System. One component of this Quality System is the requirement that field investigators use a systematic planning process as mandated in EPA Order 5360.1: Policy and Program Requirements for Mandatory Agency-wide Quality System (10). The US-EPA has developed a nonmandatory systematic planning process, the Data Quality Objectives (DQO) Process. The DQO process is an important tool for project managers, planners, and field investigators to define the type, quality, and quantity of data needed to make defensible decisions. The DQO process offers a way to plan field investigations so that the quality of data collected can be evaluated with respect to the data's intended use. For a detailed discussion of the complete DQO process, refer to the referenced guidance documents: Guidance for the Data Quality Objectives Process (1), Data Quality Objectives Process for Hazardous Waste Sites (2), and Standard Practice for Generation of Environmental Data Related to Waste Management Activities: Development of Data Quality Objectives (3).

The DQO process contains seven steps that will assist in preparing plans for environmental data collection activities (Figure 4-1). The steps are iterative and should be revisited as new information about a problem is learned. It provides a systematic approach for defining the requirements that a field investigation will attempt to fulfill. Such requirements may include when, where and how to collect samples, the number of samples, and the limits on tolerable error rates.

Figure 4-1. Steps of the DQO Process



Below are the steps, the purpose and recommended activities of DQO Process.

Step 1. State the Problem

Purpose: Summarize the environmental problem that will require the collection of data, and identify resources available.

Activities:

- ▶ Identify members of the planning team.
- ▶ Develop or refine the conceptual site model.
- ▶ Specify the available resources and constraints.
- ▶ Write a brief summary of the contamination problem.

Step 2. Identify the Decision

Purpose: To identify the decision that requires new environmental data to address the problem.

Activities:

- ▶ Identify the principal study question(s).
- ▶ Define alternative actions that could result from the resolution of the principal study question(s).
- ▶ Combine the principal study question and the alternative actions into a decision statement.
- ▶ Organize multiple decisions.

Step 3. Identify Inputs to the Decision

Purpose: To identify information that will be required to support the decision and specify which inputs require new environmental measurements.

Activities:

- ▶ Identify the information that will be required to resolve the decision statement.
- ▶ Determine the sources for each item of information identified.
- ▶ Identify the information needed to establish the action level(s).
- ▶ Confirm the appropriate analytical methods exist to provide the necessary data.

Step 4. Define the Study Boundaries

Purpose: To define the spatial and temporal boundaries that data must represent to support the decision.

Activities:

- ▶ Specify the characteristics that define the population of interest.
- ▶ Define the geographical area which the decision statement applies.
- ▶ When appropriate, divide the population into strata that have relatively homogenous characteristics.
- ▶ Determine the time frame to which the decision applies.
- ▶ Determine when to collect the data.
- ▶ Define the scale of the decision making.
- ▶ Identify any practical constraints on data collection.

Step 5. Develop a Decision Rule

Purpose: Develop a logical “If ..., then ...” statement that defines the conditions that would cause the decision maker to choose among alternate actions.

Activities:

- ▶ Specify the parameter that characterizes the population of interest.

- ▶ Specify the action level for the decision.
- ▶ Confirm the measurement detection limits will allow reliable comparisons with action level.
- ▶ Combine the outputs from the previous DQO steps and develop a decision rule.

Step 6. Specify Limits of Decision Errors

Purpose: To specify the decision maker's tolerable limits on decision errors, which are used to establish performance goals for limiting uncertainty in the data.

Activities:

- ▶ Determine the possible range of the parameter of interest.
- ▶ Define both types of decision errors and their potential consequences and select the baseline condition.
- ▶ Specify a range of possible parameter values where the consequences of a false negative decision error are relatively minor (the gray region).
- ▶ Assign probability values to points above and below the action level that reflect the tolerable probability for the occurrence of decision errors.

Step 7. Optimize the Design for Obtaining Data

Purpose: To identify resource-effective sampling and analysis design for generating data that are expected to satisfy the DQO's.

Activities:

- ▶ Review the DQO outputs and existing environmental data.
- ▶ Develop general data collection design alternatives.
- ▶ Formulate the mathematical expression necessary for each design alternative.
- ▶ Select the sample size that satisfies the DQOs for each design alternative.
- ▶ Select the most resource-effective design that satisfies all DQOs.
- ▶ Document the operational details and theoretical assumptions of the selected design in the study plan or quality assurance project plan (QAPP).

There are several benefits of using the DQO process including; providing a reliable methodology for clarifying how decisions about the site will be supported by environmental data and for establishing site-specific performance criteria for these decisions. Other benefits are: it helps conserve resources by determining which data collection and analytical methods are most appropriate, and it provides field personnel with an end-point to make defensible decisions.

The DQO process has both qualitative and quantitative components. The qualitative steps encourages logical and practical planning for environmental data collection activities while the quantitative steps may use statistical methods to design a data collection operation that will efficiently control the probability of making an incorrect decision. Although the quantitative steps

of the DQO process are important, investigators and decision makers may choose not to apply statistics to every environmental field investigation. In some cases, the planning team may only utilize the qualitative steps of the DQO process during the investigation planning phases to generate authoritative data.

4.6 Specific Sample Collection Quality Control Procedures

4.6.1 Introduction

This subsection provides guidelines for establishing quality control procedures for sampling activities. Strict adherence to all of the standard operating procedures outlined in this subsection form the basis for an acceptable sampling quality assurance program.

4.6.2 Experience Requirements

There is no substitute for field experience. Field experience shall be gained by on-the-job training using the "buddy" system. Each new investigator should accompany an experienced employee on as many different types of field studies as possible. During this training period, the new employee will be permitted to perform all facets of field investigations, including sampling, under the direction and supervision of senior investigators.

4.6.3 Traceability Requirements

All sample collection activities shall be traceable through field records to the person collecting the sample and to the specific piece of sampling equipment (where appropriate) used to collect that sample. All maintenance and calibration records for sampling equipment (where appropriate) shall be kept so that they are similarly traceable. See Sections 2.1 through 2.6 for specific procedures to be utilized that insure traceability.

4.6.4 Chain-of-Custody

Specific chain-of-custody procedures are included in Sections 2.1 through 2.6 of this SOP. These procedures will insure that evidence collected during an investigation will withstand scrutiny during litigation. To assure that procedures are being followed, it is recommended that field investigators or their designees audit chain-of-custody entries, tags, field notes, and any other recorded information for accuracy.

4.6.5 Sampling Equipment Construction Material

Sampling equipment construction materials can affect sample analytical results. Materials used must not contaminate the sample being collected and must be easily decontaminated so that samples are not cross-contaminated.

4.6.6 Sample Preservation

Samples for some analyses must be preserved in order to maintain their integrity. Preservatives required for routine analyses of samples collected are given in Appendix A of this SOP. All chemical preservatives used will be supplied by the Analytical Support Branch. All samples requiring preservation should be preserved immediately upon collection in the field. Samples that **should not** be preserved in the field are:

- Those collected within a hazardous waste site that are known or thought to be highly contaminated with toxic materials which may be highly reactive. Barrel, drum, closed container, spillage, or other source samples from hazardous waste sites are not to be preserved with any chemical. These samples may be preserved by placing the sample container on ice, if necessary.
- Those that have extremely low or high pH or samples that may generate potentially dangerous gases if they were preserved using the procedures given in Appendix A.

All samples preserved with chemicals shall be clearly identified by indication on the sample tag that the sample is preserved. If samples normally requiring preservation were not preserved, field records should clearly specify the reason.

4.6.7 Special Precautions for Trace Contaminant Sampling

Some contaminants can be detected in the parts per billion and/or parts per trillion range. Extreme care must be taken to prevent cross-contamination of these samples. The following precautions shall be taken when trace contaminants are of concern:

- A clean pair of non-powdered, disposable latex gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come into contact with the media being sampled.
- If possible, one member of the field sampling team should take all the notes, fill out tags, etc., while the other members collect the samples.
- When sampling surface waters, the water sample should always be collected before the sediment sample is collected.
- Sample collection activities should proceed progressively from the least suspected contaminated area to the most suspected contaminated area.
- Investigators should use equipment constructed of Teflon[®], stainless steel, or glass that has been properly precleaned (Appendix B) for collection of samples for trace metals or organic compounds analyses. Teflon[®] or glass is preferred for collecting samples where trace metals are of concern. Equipment constructed of plastic or PVC shall not be used to collect samples for trace organic compounds analyses.

4.6.8 Sample Handling and Mixing

After collection, all sample handling should be minimized. Investigators should use extreme care to ensure that samples are not contaminated. If samples are placed in an ice chest, investigators should ensure that melted ice cannot cause the sample containers to become submerged, as this may result in sample cross-contamination. Plastic bags, such as Zip-Lock[®] bags or similar plastic bags sealed with tape, should be used when small sample containers (e.g., VOC vials or bacterial samples) are placed in ice chests to prevent cross-contamination.

Once a sample has been collected, it may have to be transferred into separate containers for different analyses. The best way to transfer liquid samples is to continually stir the sample contents with a clean pipette or precleaned Teflon[®] rod and allow the contents to be alternately siphoned into respective sample containers using Teflon[®] or PVC (Tygon[®] type) tubing (and a siphon bulb to start the flow). Teflon[®] must be used when analyses for organic compounds or trace metals are to be conducted. Any device used for stirring, or tubing used for siphoning, must be cleaned in the same manner as other equipment (Appendix B). However, samples collected for volatile organic compound, oil and grease, bacteria, sulfides, and phenols analyses may not be transferred using this procedure.

It is extremely important that soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

1. The material in the sample pan should be divided into quarters and each quarter should be mixed individually.
2. Two quarters should then be mixed to form halves.
3. The two halves should be mixed to form a homogenous matrix .

This procedure should be repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction, and occasionally turning the material over.

4.6.9 Special Handling of Samples for Volatile Organic Compounds (VOCs) Analysis

Water samples to be analyzed for volatile organic compounds should be stored in 40-ml septum vials with screw cap and Teflon[®]-silicone disk in the cap to prevent contamination of the sample by the cap. The disks should be placed in the caps (Teflon[®] side to be in contact with the sample) in the laboratory prior to the beginning of the sampling program.

The vials should be completely filled to prevent volatilization, and extreme caution should be exercised when filling a vial to avoid any turbulence which could also produce volatilization. The sample should be carefully poured down the side of the vial to minimize turbulence. As a rule, it

is best to gently pour the last few drops into the vial so that surface tension holds the water in a convex meniscus. The cap is then applied and some overflow is lost, but the air space in the bottle is eliminated. After capping, turn the bottle over and tap it to check for bubbles. If any bubbles are present, repeat the procedure with another clean 40-ml vial. Since the VOC vials are pre-preserved, caution should be exercised when the vials are used as the collection device for surface water samples in order to prevent the loss of the preservative. When collecting water samples for VOCs, three 40-ml vials containing preservative should be filled the with sample.

Specific procedures for the collection and handling of sediment and soil samples for VOC analysis are presented in Sections 7 and 10, respectively.

4.6.10 Estimating Variability

Spacial Variability

The following spacial duplicate sampling procedures should be used during the collection of samples as a measure of variability within the area represented by the sample. Spacial duplicate grab and/or composite samples should be collected during all major investigations and studies conducted by the Branch. A "major study" would include all investigations where more than twenty (4) samples were collected, or those studies where the study objectives dictate that additional quality control samples be collected. No more than ten percent of all samples should be collected as spacial duplicates. These samples should be collected at the same time, using the same procedures, the same type of equipment, and in the same types of containers as the original samples, but collected from a different location within the area represented by the original. They should also be preserved in the same manner and submitted for the same analyses as the required samples. The collection of spacial duplicate composite samples requires that the sample aliquots be arrayed in a manner different from the original sample and spaced within the same area of representativeness. Data from spacial duplicates will be examined by the lead investigator to determine if the samples represent the areas intended in the project work plan.

Temporal Variability

When required, temporal variability at a given sampling location will be measured by collecting temporal duplicate samples. These samples will be collected from the same sampling location, using the same techniques and the same type of equipment, but at a time different from the original sample. The time selected for the temporal duplicate sample will be within the same span of time for which the original sample is designed to be representative in the project work plan. Data from temporal duplicates will be examined by the project leader to determine if samples represent the time span intended in the project work plan.

Sample Handling Variability

The effectiveness of sample handling techniques will be measured by collecting split and blank samples.

Split Samples

Split samples will be collected by initially collecting twice as much material as normally collected for a sample. After mixing, the material will be apportioned into two sets of containers. Both sets of containers will be submitted for analyses with one set designated as an "original sample", the other designated as a "split sample". Data from split samples will be examined by the project leader to determine sample handling variability. On large studies (more than 20 samples), no more than 10 percent of all samples will be collected as split samples.

Blank Samples

The following blank samples will be prepared by the laboratory and obtained by the project leader prior to traveling to a sample site.

1. Water Sample VOC Trip Blank -- A water sample VOC trip blank is required for every study where water samples are collected for VOC analysis. Two sealed preserved (or unpreserved if appropriate) 40-ml VOC vials will be transported to the field. For routine studies these samples will be prepared by lab personnel. Investigators shall request that these samples be provided at least one week in advance of scheduled field investigations and inspections and never (except in emergency situations) less than two days in advance of scheduled field investigations and inspections. These samples should not be picked up earlier than the morning of departure for the scheduled inspection/investigation. These field blanks will be handled and treated in the same manner as the water samples collected for volatile organic compounds analysis on that particular study. These samples will be clearly identified on sample tags and Chain-of-Custody Records as trip blanks.
2. Soil and Sediment Sample VOC Trip Blank -- Soil and sediment sample VOC trip blanks are required for every study where soil and sediment samples are collected for VOC analysis. The type of blank will be dictated by the method utilized to collect the soil or sediment samples. This field blank will be handled and treated by Branch personnel in the same manner as the soil samples collected for volatile organic compounds analysis on that particular study. These samples will be clearly identified on sample tags and Chain-Of-Custody Records as trip blanks.

The following blanks are prepared in the field:

1. Inorganic Sample Preservative Blanks -- Metals and general inorganic sample containers filled with analyte-free water will be transported to the field and preserved and submitted for the same analyses as the other inorganic samples collected. These samples will be clearly identified as preservatives blanks on sample tags and the Chain-Of-Custody Record(s). At least one preservative blank for each type of preserved sample should be collected at the end of routine field investigations. A minimum of one preservative blank should be prepared in the field at the beginning and end of all major field investigations

that last more than one week. One preservative blank should be collected for each bottle of preservative used to preserve samples.

2. Equipment Field Blanks -- When field cleaning of equipment is required during a study for sampling trace contamination, a piece of the field-cleaned equipment will be selected for collection of a rinse blank. At least one rinse blank will be collected during each week of sampling operations. After the piece of equipment has been field cleaned and prior to its being used for sample operations, it will be rinsed with organic/analyte free water. The rinse water will be collected and submitted for analyses of all constituents for which normal samples collected with that piece of equipment are being analyzed.
3. Organic/Analyte Free Water System Blanks -- When using a portable organic/analyte-free water generating system in the field, a sample of the water generated will be collected at least once during each week of operations. The collected water sample will be submitted for analyses of all constituents for which normal samples are being analyzed.
5. Automatic Sampler Blanks -- In general, cleaning procedures outlined in Appendix B of this SOP should be adequate to insure sample integrity. However, it is the standard practice of the Branch to submit automatic sampler blanks for analyses when automatic samplers are used to collect samples for true organic compounds and metals analyses.

The Branch Field Quality Assurance Officer will inform the project leaders and management when blank samples are found to be unacceptably contaminated. The Branch Field Quality Assurance Officer will immediately initiate an investigation to determine the cause of the problem. The results of this investigation will be promptly reported to appropriate personnel so that corrective action and/or qualifications to the data can be initiated.

4.6.11 Special Quality Control Procedures for Water Samples for Extractable Organic Compounds, Pesticides, or Herbicides Analyses (Matrix Duplicate)

Duplicate water samples shall be submitted to the laboratory for extractable organic compounds, pesticides, and/or herbicides analyses from at least one sampling location per project and laboratory used. These samples should be collected from a location expected to be relatively free from contamination, since the samples will be used for laboratory quality control purposes. The duplicate samples should be clearly identified as "Duplicate Sample for Matrix Spike" on the sample tag, Chain-Of-Custody Record, in the field logbook, and on the Contract Laboratory Program (CLP) Traffic Report Form (if appropriate). This procedure shall be followed for all projects where water samples are collected for the indicated analyses.

4.6.12 Special Quality Control Procedures for EPA Contract Laboratories

On a case-by-case basis, field investigators may be required to collect split samples (or duplicate samples if appropriate) for analyses by both the Region 4 laboratory and contract laboratories. The split samples are to be submitted to the Region 4 laboratory using established

procedures. The contract laboratory involved shall not be notified that samples were split, i.e., there should be no indication on Chain-Of-Custody Records or CLP Traffic Report Forms submitted to the contract laboratories that these samples were split with the Region 4 laboratory.

4.6.13 Special Quality Control Procedures for Dioxins and Furans

All samples collected for dioxins and furans analyses are analyzed by other EPA laboratories or through contract laboratories. The Region 4 laboratory does not conduct in-house analyses for dioxins and furans. The Region 4 laboratory should be consulted for the current quality control procedures for dioxin and furan samples prior to the sampling event.

4.7 Internal Quality Control Procedures

4.7.1 Introduction

Internal quality control refers to monitoring operations at the Field Equipment Center involving the preparation of sampling and support equipment used to collect samples for trace (Ppb/PPT) contaminants. Quality control checks of these operations insure that equipment used for collecting samples is properly maintained and decontaminated. The Enforcement and Investigations Branch (EIB) Quality Assurance Officer is responsible for overseeing these activities, therefore specific requirements for these task are found in the Environmental Investigation Standard Operating Procedures and Quality Assurance Manual, 2001.

The Ecological Assurance Branch Quality Assurance Officer is responsible for compiling quarterly reports summarizing the following information for each project conducted during the quarter:

- Field split samples (not to include inter-lab splits);
- Water VOC trip blank samples;
- Soil VOC trip blank samples;
- Inorganic sample preservative blanks;
- Equipment field rinse blanks;
- Field organic/analyte free water system blanks; and
- Material blanks.

The QA Officer will evaluate all data received and immediately attempt to resolve any problems identified.

4.8 Investigation Derived Waste (IDW)

4.8.1 Types of IDW

Materials which may become IDW are:

- Personnel protective equipment (PPE) -- This includes disposable coveralls, gloves,

booties, etc.

- Disposable equipment -- This includes plastic, aluminum foil, Teflon® tubing, broken or unused sample containers, sample container boxes, tape, etc.
- Analytical reagents and
- Packing and shipping materials.

Table 4.8.1 lists the types of IDW commonly generated during field studies, and current disposal practices.

4.8.2 Management of Non-Hazardous IDW

Most of the field studies conducted by EAB will generate non-hazardous IDW, such as, used plastic containers, aluminum foil, tubing, broken sample containers, cardboard boxes, tape, etc. The disposal of this material must comply with local and state solid waste disposal requirements and may be disposed at the study location. For instance, EAB often establishes a command post at a local wastewater treatment plant (WWTP) located in the area a major water quality investigation is being conducted. Non-hazardous IDW may be disposed in the WWTPs' dumpster, if permission is granted by the plant management. However, if permission is not granted, then the waste must be placed in garbage bags and taken to a nearby permitted landfill or returned to SESD for disposal.

4.8.3 Management of Hazardous IDW

Typically EAB does not generate site specific hazardous IDW. However, if EAB personnel are involved in a study that may generate site specific hazardous IDW, i.e. a Superfund Investigation, then the waste must be properly identified and disposed. The appropriate methods for dealing with site generated hazardous IDW are provided in the Enforcement and Investigations Branch, Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, 2001.

However, EAB personnel may use chemical reagents for field analysis, preservation and instrument calibration that may be considered hazardous waste if discarded. These spent chemicals must be properly contained, labeled and returned to SESD for disposal. The disposal of these chemicals must be coordinated with the SESD Hazardous Waste Disposal Officer.

**TABLE 4.8.1
DISPOSAL of IDW**

TYPE	HAZARDOUS	NON-HAZARDOUS
PPE-Disposable	Containerize in plastic 5-gallon bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise return to SESD for proper disposal.	Bag waste and place in dumpster with permission of site operator, otherwise return to SESD for disposal.
Spent Chemical/Reagents	Containerize in original containers or a similar container. Clearly identify contents. Return to SESD for proper disposal.	N/A
Trash	N/A	Bag waste and place in dumpster with permission of site operator, otherwise return to SESD for disposal in dumpster.

4.9 References

1. US-EPA, Guidance for the Data Quality Objectives Process (EPA QA/G-4, 1994)
2. ASTM, Standard Practice for Generation of Environmental Data Related to Waste Management Activities: Development of Data Quality Objectives (D-34.02.10-Draft).
3. ASTM, Standard Guide for the Generation of Environmental Data Related to Waste Management Activities (D-34.01.11-Draft).
4. Gilbert, Richard O., Statistical Methods for Environmental Pollution Monitoring, Van Nostrand Reinhold Co., New York, NY, 1987.
5. ASTM, Standard Guide for General Planning of Waste Sampling.
6. US-EPA, Characterization of Hazardous Waste Sites - A Methods Manual, Volume 1 - Site Investigations (EPA 600/4-84/075).
7. Kittrell, F.W., A Practical Guide to Water Quality Studies.

SECTION 5 WASTEWATER SAMPLING

SECTION OBJECTIVE:

- To provide guidance for the proper collection of wastewater samples.

5.1 Introduction

The variety of conditions that occur at sampling locations require that considerable judgment be exercised regarding the methodologies and procedures for the collection of representative samples of wastewater. Each sampling location warrants attention commensurate with its complexity. There are, however, basic rules and precautions generally applicable to sample collection. Acceptable procedures are generally those outlined in the NPDES Compliance Inspection Manual (1) and the Handbook for Sampling and Sample Preservation of Water and Wastewater (2). Some important considerations for obtaining a representative wastewater sample include:

- The sample should be collected where the wastewater is well mixed. Therefore, the sample should be collected near the center of the flow channel, at approximately 40 to 60 percent of the water depth, where the turbulence is at a maximum and the possibility of solids settling is minimized. Skimming the water surface or dragging the channel bottom should be avoided. However, allowances should be made for fluctuations in water depth due to flow variations.
- In sampling from wide conduits, cross-sectional sampling should be considered. Tracers such as Rhodamine WT dye may be used as an aid in determining the most representative sampling locations.
- If manual compositing is employed, the individual sample portions must be thoroughly mixed before pouring the individual aliquots into the composite container. For manual composite sampling, the individual sample aliquots should be preserved at the time of sample collection (2).
- When collecting samples or installing sampling equipment, field investigators should use precaution to prevent contamination of the sample and reduce exposure to hazardous substances.

5.2 Site Selection

Where applicable, wastewater samples should be collected at the location specified in the NPDES permit (if the source has such a permit). In some instances the sampling location specified in the permit, or the location chosen by the permittee, may not be acceptable for the collection of a representative wastewater sample. In such instances, the investigator is not limited by permit specifications and may collect a sample at a more representative location. When a conflict exists between the permittee and the regulatory agency regarding the most representative sampling location,

both sites should be sampled, and the reason for the conflict should be noted in the inspection or study report and field notes. Recommendations and reasons for a change in sampling locations should be given to the appropriate permitting authority.

5.2.1 Influent

Influent wastewaters are preferably sampled at locations of highly turbulent flow in order to ensure good mixing; however, in many instances the most desirable location is not accessible. Preferable influent wastewater sampling locations include: 1) the upflow siphon following a comminutor (in absence of grit chamber); 2) the upflow distribution box following pumping from main plant wet well; 3) aerated grit chamber; 4) flume throat; 5) pump wet well when the pump is operating; or 6) downstream of preliminary screening. When possible, influent samples should be collected upstream from sidestream returns.

5.2.2 Effluent

Effluent samples should be collected at the site specified in the permit, or if no site is specified in the permit, at the most representative site downstream from all entering wastewater streams prior to discharge into the receiving waters. If a conflict exists between the permittee and inspector regarding the source being sampled or the location of the most representative site, follow the procedures previously described under "Site Selection".

5.2.3 Pond and Lagoon Sampling

Generally, composite effluent wastewater samples should be collected from ponds and lagoons. Even if the ponds or lagoons have long retention times, composite sampling is preferable because of the tendency of ponds and lagoons to have flow paths that short circuit which changes the design detention time.

5.3 Sample Types

For NPDES sampling, two types of sampling techniques are used: grab and composite. For these procedures, the NPDES permit specifies the appropriate sample type. A complete description of all NPDES sampling procedures and techniques is presented in the NPDES Compliance Inspection Manual (1).

5.3.1 Grab Samples

Grab samples consist of either a single discreet sample or individual samples collected over a period of time not to exceed 15 minutes. The grab sample should be representative of the wastewater conditions at the time of sample collection. The sample volume depends on the type and number of analyses to be performed.

5.3.2 Composite Samples

Composite samples are collected over time, either by continuous sampling or by mixing discrete samples. A composite sample represents the average wastewater characteristics during the compositing period. Various methods for compositing are available and are based on either time or

flow proportioning. The choice of a flow proportional or time composite sampling scheme depends on the permit requirements, variability of the wastewater flow or concentration of pollutants, equipment availability, and sampling location. If an investigator suspects that there is significant variability in the wastewater flow, a flow proportional sample is preferable. Otherwise, a time composite sample would be acceptable.

A time composite sample consists of equal volume discrete sample aliquots collected at constant time intervals into one container. A time composite sample can be collected either manually or with an automatic sampler.

A flow proportional composite sample can be collected using one of two methods. One method consists of collecting a constant sample volume at varying time intervals proportional to the wastewater flow. For the other method, the sample is collected by varying the volume of each individual aliquot proportional to the flow, while maintaining a constant time interval between the aliquots.

Flow proportional samples can be collected with an automatic sampler and a compatible flow measuring device, semi-automatically with a flow chart and an automatic sampler capable of collecting discrete samples, or manually.

5.4 Use of Automatic Samplers

Automatic samplers may be used to collect composite or grab samples when several aliquots are to be collected at frequent intervals or when a continuous sample is required. For composite sampling applications, the automatic samplers may be used to collect time composite or flow proportional samples. In the flow proportional mode, the samplers are activated by a compatible flow meter. Flow proportional samples can also be collected using an automatic sampler equipped with multiple containers and manually compositing the individual sample portions proportional to the flow. Detailed procedures are outlined in the NPDES Compliance Inspection Manual (1), Handbook for Sampling and Sample Preservation of Water and Wastewater (2) and in the SESD EISOPQAM (4).

5.5 Manual Sampling

Manual sampling is normally used for collecting grab samples and/or for immediate *in situ* field analyses. However, it can also be used in lieu of automatic equipment over extended periods of time for composite sampling, especially when it is necessary to evaluate unusual waste stream conditions.

The best method to manually collect a sample is to use the actual sample container which will be used to transport the sample to the laboratory. In some cases it may be best to use a pump, either power or hand operated, to withdraw a sample from the water or wastewater stream. If a pump is used, it is imperative that all components of the pump that come in contact with the sample are properly cleaned to insure the integrity of the sample.

In general, samples are manually collected by first selecting a location in the wastestream that is well mixed then dipping the container in the water or wastewater stream so the mouth of the container faces upstream. The container should not be overfilled if preservatives are present in the

container.

5.6 Special Sample Collection Procedures

5.6.1 Organic Compounds and Metals

Trace organic compounds and metals detection limits are usually in the parts per billion or parts per trillion range, so extreme care must be exercised to insure sample integrity.

All containers, composite bottles, tubing, etc., used for sample collection for trace organic compounds and metals analyses should be prepared as described in Appendix B.

When possible, the sample should be collected directly into the appropriate sample container. If the material to be sampled cannot be physically reached, an intermediate collection device may be used. For organic compounds this should be a Teflon®, glass, or stainless steel vessel attached to a stainless or Teflon® pole. In the case of metals this should be a Teflon® or glass vessel attached to a stainless or Teflon® pole. Another option is using Teflon® tubing via a peristaltic type pump and a Teflon® vacuum container attachment which converts a sample container into a vacuum container. The device which is used should be cleaned as described in Appendix B.

5.6.2 Microbiological Sampling

Microbiological samples are collected from surface water to determine the degree of contamination from wastes. Extreme caution should be utilized to insure the samples are not contaminated by sampling personnel. Region 4 has developed the following sampling protocols based on Method 9060 from Standard Methods for the Examination of Water and Wastewater (1998).

Sampling Protocols: Samples should be collected directly into sterilized (autoclaved) glass or plastic containers. If the presence of residual chlorine is possible, sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) should be added to the containers prior to sample collection. The sample container should remain capped until it is filled. When the cap is removed, care should be taken not to contaminate the cap or inside of the bottle. Samples should always be collected from mid-stream and if possible, mid-depth. When collecting the sample by dipping the container, the bottle should be held near its base and plunged, neck downward, below the surface. Turn the bottle until the neck points slightly upward and the mouth is directed toward the current. If there is no current, push the bottle forward horizontally to fill. If possible, adequate head space should be left in the sample container to facilitate mixing by shaking before analysis. If samples cannot be collected by dipping the container directly into the water, subsurface sampling devices are available to assist with collection. Method 9060 contains information regarding subsurface sampling devices.

5.6.3 Immiscible Liquids/Oil and Grease

Oil and grease may be present in wastewater as a surface film, an emulsion, a solution, or as a combination of these forms. Since it is very difficult to collect a representative sample for oil and grease analysis, the inspector must carefully evaluate the location of the sampling location. The most desirable sampling location is the area of greatest mixing. Quiescent areas should be avoided. The sample container should be plunged into the wastewater using a swooping motion with the mouth

facing upstream. Care should be taken to insure that the bottle does not over fill during sample collection.

Because losses of oil and grease will occur on sampling equipment, an automatic sampler should not be used to collect samples for oil and grease analysis. Individual portions collected at prescribed time intervals must be analyzed separately to obtain the average concentrations over an extended period.

5.6.4 Volatile Organic Compounds

Samples to be analyzed for volatile organic compounds (VOCs) should be collected in 40-ml septum vials with screw caps with a Teflon® lined silicone disk in the cap to prevent contamination of the sample by the cap. If the water contains chlorine, ascorbic acid is used as a preservative (see Appendix A).

The 40-ml vials should be completely filled to prevent volatilization, and extreme caution should be exercised when filling each vial to prevent any turbulence which could also produce volatilization. As a rule, it is best to gently pour the last few drops into the vial so that surface tension holds the water in a convex meniscus. The cap is then applied and some overflow is lost, but air space in the bottle is eliminated. After capping, turn the bottle over and tap it to check for bubbles; if any are present, repeat the procedure using a new 40-ml vial.

Sampling containers with preservatives should be pre-labeled prior to any field activities. This will reduce the chances of confusion during sampling activities by the investigation team. Sample preservation, containers, holding times, and sample volumes are listed in Appendix A.

5.7 Supplementary Data Collection

While conducting wastewater sampling, the following information will also be obtained (if applicable):

- Field measurements -- pH, dissolved oxygen, conductivity, and temperature.
- Flows associated with the samples collected -- continuous flows with composite samples and instantaneous flows with grab samples.
- Diagrams and/or written descriptions of the wastewater treatment systems (if available).
- Photographs of pertinent wastewater associated equipment, such as flow measuring devices, treatment units, etc.
- Process control information on the wastewater treatment process (if applicable).
- Completion of applicable forms required during specific investigations.

All observations, measurements, diagrams, etc., will be entered in bound field logbooks or attached thereto.

5.8 References

1. NPDES Compliance Inspection Manual, United States Environmental Protection Agency, Office of Environment and Compliance Assurance, September 1994.
2. Handbook for Sampling and Sample Preservation of Water and Wastewater, United States Environmental Protection Agency, Office of Research and Development, September 1982.
3. Code of Federal Regulations, 40 CFR, Part 136.3, Table II, 2001.
4. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, 2001.

SECTION 6 SURFACE WATER SAMPLING

PERFORMANCE OBJECTIVES:

- To collect a representative sample of the surface water of interest.

6.1 Introduction

As used in this section, the term surface water refers to any body of water on the earth's surface as opposed to ground water. Surface water sampling techniques and equipment are designed to ensure the chemical and physical integrity of the sample. General considerations for all sampling efforts are provided in Section 4 of this SOP.

If surface water samples are required, direct dipping of the sample container into the water is desirable. This may be possible from a small boat, a pier, etc., or by wading in the waterbody. However, wading may cause the resuspension of bottom deposits and bias the sample. Wading is acceptable if the waterbody has noticeable current and the samples are collected while facing upstream. If the waterbody is too deep to wade or the sample must be collected from more than one depth, supplemental sampling equipment may be required (see Section 6.5).

Representative sampling sites are dependent on the type of investigation undertaken and are generally determined based on the professional judgement of the project leader and the individual conducting the sampling. A sufficient number of samples should be collected to adequately represent the area of interest in both time (e.g., hourly grab samples, slack tide sampling) and space (e.g., stream quarter-point sampling, mid-depth or stratified samples). Where water column stratification is expected, the quality assurance project plan shall define what conditions dictate stratified sampling.

6.2 Definitions

6.2.1 Grab Sample

Grab samples consist of either a single discreet sample or individual samples collected over a period of time not to exceed 15 minutes.

6.2.2 Composite Sample

Composite samples can be time or depth integrated. Time integrated composite samples are collected over time, either by continuous sampling or mixing discrete samples. Depth integrated composite samples are collected throughout the water column.

6.4 Equipment

6.4.1 Dipping Using Sample Container

A sample may be collected directly into the sample container when the surface water source is accessible by wading or other means. The sampler shall face upstream and collect the sample without disturbing the sediment. If disturbance of sediments is unavoidable, the surface water sample should be taken upstream of the disturbed sediment or the investigator should wait for the disturbed material to be moved downstream before collecting the sample. Surface water samples must be collected prior to a sediment sample at the same location. The sampler should be careful not to displace the preservative from a pre-preserved sample container.

6.4.2 Peristaltic Pumps

Another device that can be effectively used to sample a water column is the peristaltic pump. The use of a weighted line to which the tubing is attached allows for the collection of a vertical sample (to about 25' deep) which is representative of the water column.

6.4.3 Submersible Pumps

Submersible pumps may also be used to collect surface water samples throughout the water column directly into a sample container. Prior to filling the sample container at each discrete sample depth within the water column, water must be pumped through the system for a short time based on the sample depth to purge sample water from the previous depth. A submersible pump also allows for collection of a water column composite sample by lowering and raising the pump throughout the water column during sample collection.

6.4.4 Discrete Depth Samplers

Another option for collecting discrete samples from a specific depth is the Van Dorn sampler. Once the sampler is lowered to depth on a line, a weighted messenger is sent down the line causing stoppers to close on the ends of the sampler. If the stoppers are not seated properly, the sample may leak out during retrieval and redeployment is required. A rubber tube may be attached to the Van Dorn outlet valve to allow filling of sample bottles (e.g., DO bottles) without additional aeration of the sample.

6.4.5 Depth Integrated Sampler

Depth integrated samplers are designed for use in streams less than 15 feet deep. The samplers are streamlined and accommodate round 1-pint glass sample containers. The sampler has tail vanes which orient the sampler into the direction of flow. While sampling, the nose of the sampler is oriented upstream and the sampler is lowered from the water surface to the stream bed and then raised. A continuous sample of water is collected during raising and lowering of the sampler. See Section 11 of this SOP for detailed instructions regarding use of depth integrated samplers.

6.4.6 Buckets

A plastic bucket can be used to collect samples for measurement of water quality

parameters in the field (e.g., pH, temperature, conductivity) and specific water quality samples. The bucket must be rinsed twice with the sample water prior to collection of the sample.

6.4.7 Automatic Samplers

Where unattended sampling is required (e.g., TMDL storm-event sampling, time-of-travel studies), an automatic sampler may be used. The sampler may be used to collect grab samples based on time, instream flow, or water level or used to collect composite samples as dictated by the study data needs. A test volume should be checked prior to deployment to prevent overflow of sample bottles. The manufacturer's instruction manual should be consulted for automatic sampler operation.

6.4.8 Vacuum Chamber (Trace Level Mercury)

A specially design vacuum chamber sampler is available for sampling associated with low level water column mercury studies. This device is utilized in conjunction with specially cleaned sample bottles. Section 6.6.2 below addresses low level mercury sampling.

6.6 Low Level Mercury Sampling

6.6.1 Introduction

Sampling for certain constituents requires special procedures. This section addresses sample collection for low level mercury analyses. One of the greatest difficulties in measuring trace levels of mercury (< 1 part per trillion) is preventing contamination during collection, transport and analyses. Region 4 has developed the procedures in this section based on EPA Method 1669.

6.6.2 Equipment

Vacuum Chamber

Region 4 utilizes a vacuum chamber assembly to collect water samples for mercury analyses. The vacuum chamber assembly consists of the following: 1) an acrylic chamber to hold the sample container, 2) Teflon[®] sample tubing that connects to the top of the chamber, passes through a rigid Teflon[®] pole for stability and has a magnetic screen holder at the intake, and 3) a hand pump to deliver the sample to the container. The chamber is designed to hold a 2 liter sample container, however, smaller sample containers may be utilized with a spacer inserted into the chamber. A two inch square of 105 μm Nitex screen is used in the magnetic screen holder at the intake to prevent large pieces of debris from entering the sample. The screen does not prevent the passage of particulate organic matter which is often prevalent in surface water. A hand pump, which is connected to the top of the chamber with Teflon[®] tubing, is then used to create a vacuum within the chamber, thus drawing the sample into the container. (Figure 1).

Sample Containers

All sample containers used for collection of low level mercury samples must be precleaned in a laboratory as described in EPA Method 1631. The containers should also be etched with a unique number for identification. Water samples collected for total, inorganic, methyl and ethyl mercury analyses are placed into either 500 ml or 2L Teflon[®] bottles and preserved. Preservation can be performed in the field but the laboratory is preferred. Region 4 utilizes laboratory preservation of low level mercury samples in order to minimize the potential for contamination, and if split samples are required, they must be split in a clean lab.

Quality Assurance/Quality Control

The following quality assurance/quality control (QA/QC) samples are collected in conjunction with low level mercury samples:

- bottle blanks
- equipment blanks
- air depositional blanks
- trip blanks.
- duplicates
- splits

A bottle blank is run with reagent water in the laboratory which prepares the mercury sample containers to insure the cleanliness of the bottles prior to use in the field. After decontamination of the chamber assembly by pumping and discarding several sample container volumes of reagent water through the Teflon[®] sample tubing, an equipment blank sample is collected into a clean container .

Air depositional blanks are collected to determine if airborne mercury was present at the time of sample collection. The air depositional blanks consist of a precleaned mercury sample container, which was filled with reagent water by the laboratory that prepared the containers, and shipped with the containers to the field. The air depositional blank is uncapped using “clean hands”/”dirty hands” procedures and set near the sampling location throughout the duration of the mercury sample collection. Once the mercury sample is collected, the air depositional blank is recapped and handled and processed with the other mercury samples. Typically, one air depositional blank is collected each day unless atmospheric conditions warrant otherwise.

Trip blanks are utilized to determine if any contaminants of interest to the study are potentially introduced to the samples during storage and transport to the laboratory. Trip blanks are prepared by the laboratory which supplies the mercury sample containers. The trip blanks consist of cleaned bottles which are filled with reagent water by the laboratory and shipped with the other clean sample containers. One trip blank is placed in each cooler of mercury samples and returned to the laboratory with the samples. The trip blanks are never opened in the field.

Duplicate samples are discrete samples collected at the same site and time to obtain within site variance. Sample splits are in number portions of a sample (minimum 500 ml) that

are developed from a single sample in a clean laboratory.

6.6.3 Sampling Protocols

In order to determine trace levels of mercury in ambient surface waters, clean sampling protocols must be employed throughout the sampling effort. For each sampling event, one sampling team member is designated as “clean hands” and one as “dirty hands”. All operations involving contact with the sample bottle and transfer of the sample from the sample collection device to the sample bottle are handled by the individual designated as “clean hands”. “Dirty hands” is responsible for preparation of the sampling device (except the sample container itself) and for all other activities that do not involve direct contact with the sample.

Prior to sample collection with the vacuum chamber assembly, the sampling line should be cleaned at each station by rinsing three times with ambient water. A 2 liter bottle should be placed into the chamber, filled and rinsed with ambient water. The same 2 liter bottle can be used at each station. Additional cleaning measures are not recommended as long as the chamber assembly is only used to collect ambient surface water samples. Detergents washes and acid rinses are not conducted due to potential mercury contamination from these solutions. If applicable, samples for other analyses can be collected with the vacuum chamber assembly and should be collected first as an additional means of flushing the sampling line prior to collection of the mercury samples. It is not necessary to implement the “clean hands”/“dirty hands” method for collection of non-mercury samples.

Following are procedures for cleaning the vacuum chamber assembly and collection of ancillary water quality samples, if applicable:

1. Approach the sampling station from downstream and downwind if possible.
2. Wearing latex gloves, place an uncapped 2 liter bottle into the chamber and secure the chamber lid by attaching the spring-loaded clamps.
3. Place a new square of 105 μm Nitex[®] screen in the magnetic screen holder. Place the intake beneath the surface of the water (mid-depth or six inches, which ever is less) and hold firmly in place. Care should be taken not to stir up sediment particles in very shallow waters (< 4 inches deep).
4. Squeeze the hand pump until liquid starts to fill the bottle in the chamber. When the bottle is approximately half full, release the vacuum on the chamber, remove the bottle, swirl the contents and discard the water downstream. Repeat this rinse two times. If ancillary water quality samples are to be collected, return the 2 liter bottle to the chamber and pump the required volume of water to fill the appropriate sample containers. Remove the 2 liter bottle from the chamber and cap. Fill the ancillary sample bottles upon completion of the mercury sample collection.

Water samples for trace level mercury analyses should be collected according to the following procedures:

5. “Clean hands” should put on a pair of vinyl gloves, then a pair of shoulder length polyethylene gloves.
6. “Dirty hands” should retrieve a sample bottle and open the outer bag. “Clean hands” should open the inner bag and remove the bottle.
7. “Dirty hands” should open the lid on the chamber. “Clean hands” should place the sample container in the chamber, remove the container lid, and place it on top of the bottle.
8. “Dirty hands” should close and secure the chamber lid and using the hand pump, fill the container. The sample container should be filled to overflowing. “Dirty hands” should then release the vacuum and open the lid on the chamber.
9. “Clean hands” should place the lid on the sample container, remove it from the chamber and place it in and seal the inner bag. “Dirty hands” should seal the outer bag and place the sample in a cooler. Only coolers dedicated to storage and transport of low level mercury samples should be used.

6.7 Microbiological Sampling

6.7.1 Introduction

Microbiological samples are collected from surface water to determine the degree of contamination from wastes. Extreme caution should be utilized to insure the samples are not contaminated by sampling personnel. Region 4 has developed the following sampling protocols based on Method 9060 from Standard Methods for the Examination of Water and Wastewater (1998).

6.7.2 Sampling Protocols

Samples should be collected directly into sterilized (autoclaved) glass or plastic containers. If the presence of residual chlorine is possible, sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) should be added to the containers prior to sample collection. The sample container should remain capped until it is filled. When the cap is removed, care should be taken not to contaminate the cap or inside of the bottle. Samples should always be collected from mid-stream and if possible, mid-depth. When collecting the sample by dipping the container, the bottle should be held near its base and plunged, neck downward, below the surface. Turn the bottle until the neck points slightly upward and the mouth is directed toward the current. If there is no current, push the bottle forward horizontally to fill. If possible, adequate head space should be left in the sample container to facilitate mixing by shaking before analysis. If samples cannot be collected by dipping the container directly into the water, subsurface sampling devices are available to assist with collection. Method 9060 contains information regarding subsurface sampling devices.

6.8 General Quality Assurance Procedures

Recommended sample containers, preservatives, and maximum sample holding times vary by analyte and are presented in Appendix A. Latex gloves should be worn whenever direct contact poses a sample contamination risk or a health risk to the samplers (e.g., in vicinity of wastewater outfall). For samples requiring preservation following collection (e.g., nutrients), space should be left in the bottle to allow addition of preservative without causing overflow.

For each analyte, duplicate samples should be collected to allow for laboratory quality assurance tests. The number of duplicate samples should be approximately ten percent (10 %) of the total number of samples collected for each analyte. Sample handling, collection, preservation, and shipping of the duplicate samples should be performed in the same manner as for the regular samples; however, the sample custody sheet and sample tag should reflect that the sample is a duplicate for QA purposes.

6.9 Data/Records Management

All details of sample collection (date, time, depth, etc.) should be documented in field records. Section 2 of this SOP describes appropriate sample control, chain-of-custody, and shipping requirements applicable to surface water samples.

SECTION 7 SEDIMENT SAMPLING

PERFORMANCE OBJECTIVES:

To collect a sediment sample that is representative of conditions as they exist at the sampling site

- By selecting the appropriate sampling device(s).
- By taking measures to avoid introducing contamination as a result of poor sampling and/or handling technique.
- By reducing the potential of cross contamination between samples.

7.1 Introduction

This section discusses sampling equipment and collection methods which have typically been used by EPA Region 4 that have proven technically appropriate for collecting representative sediment samples. These techniques and equipment are designed to minimize effects on the chemical and physical integrity of the sample.

The physical location of the investigator when collecting a sample may dictate the equipment to be used. If the surface water body is shallow or if sediments are exposed (i.e., marsh, tidal flats, etc.), wading may be the preferred method for reaching the sampling location, particularly if a water body has a noticeable current (is not impounded). If wading is utilized, one should always approach the sampling location from downstream to minimize disruption of the sampling site. If the water body is too deep to wade, the sediment sample can be collected by divers utilizing hand corers or from a boat or bridge, utilizing another type of coring device or dredge. Regardless of the method used, care should be taken when collecting sediment samples not to lose the smaller particle size material or “fines”. Fines are typically composed of clays and silts, which can bind many chemical constituents, (especially metals) and which generally have the highest concentrations of contaminants in a given sample.

7.2 Equipment

Equipment used to collect sediment samples for contaminant analyses should be constructed of inert materials such as stainless steel. Sampler parts that come in contact with the sediment sample must not contain substances that may contaminate the sample. Ancillary equipment such as auger flights, winch cables/ropes, etc. may be constructed of other materials since this equipment does not come in contact with the sample.

Selection of equipment is usually based on accessibility (depth of overlying surface water), the nature of the bottom material, and the depth of sediment sample to be collected. The following

equipment is typically used by EPA Region personnel for collecting surface and shallow subsurface sediment samples:

- coring devices (coring tubes, stainless steel auger buckets)
- dredges (Peterson, Eckman, Ponar, Young, etc.)
- scoops and spoons

7.3 Sampling Methodology

This discussion of sediment sampling methodology reflects both the equipment used to collect the sample, as well as how the sample is handled and processed after retrieval of the material. This guide emphasizes general principles, not step-by-step instructions. Because sediment sampling is a field-based operation, methods and equipment must often be modified to suit local conditions. This modification process requires operator skill and judgement, which cannot be replaced by written instructions.

Sediment samples may be collected using manual (hand operated) devices, or powered devices (drilling rig, vibracore). Simple, manual techniques and equipment, such as coring tubes, hand augers, dredges, and scoops are usually selected when sampling depth is less than two feet. As the sediment depth of the sample increases, some type of powered sampling equipment may be needed to overcome torque induced by resistance and friction as well as to prevent compaction of the sample being collected. If powered equipment is required to assist in collecting subsurface sediment samples, refer to the Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, November 2001 for guidance.

Sediment samples are removed from the ground, placed in pans, and mixed as instructed in Section 4.6.8. It should be noted that samples collected for volatile organic analyses are never mixed. Specific procedures for collecting samples for volatile organic compound analysis are in Section 7.5.

7.3.1 Coring

Core samplers are utilized to sample vertical columns of sediment. They are particularly useful when it is desirable to minimize the loss of material at the sediment-water interface, when a discrete sediment depth is required, and also when a historical picture of sediment deposition is desired since core samplers preserve the sequential layering of the deposit. Many types of coring devices have been developed depending on the depth of water from which the sample is to be obtained, the nature of the bottom material, and the length of core to be collected. Coring devices vary from hand push tubes to vibration, weight, or gravity driven devices. Coring tubes are available in stainless steel, teflon, and HDPE.

Sampling tubes should be of sufficient length to collect the desired core depth and to extend far enough above the substrate or sediment-water interface to prevent disturbance of the core sample and to provide for retrieval of the coring device. Soft or semi-consolidated sediments such as mud and clays have a greater adherence to the inside of the tube and thus can be sampled with larger diameter tubes. A tube of approximately two inches in diameter is usually the best size for a hand

operated corer. For deep cores or core tubes of larger diameter, it is recommended that the corer have a checkvalve built into the driving head which allows water and air to escape from the cutting core. This not only diminishes the pressure wave in front of the corer, minimizing disturbance to the surficial sediments, but also creates a partial vacuum which helps to hold the sediment core in the tube. Most larger tubes also have a core catcher, i.e. a device in the end of the corer that prevents sediment from falling out. Because coarse or unconsolidated sediments such as sands and gravel tend to fall out of the tube, a small diameter is required for them. However, the relatively small surface area and sample size obtained with core samplers, it is sometimes necessary to collect replicate samples in order to obtain the required amount of material for analysis. In spite of the above, due to the minimal disturbance to the surficial sediments, core samplers are recommended in sampling sediments for trace organic compounds or metals analyses.

Before extracting the sediment from the coring tubes, the clear supernatant above the sediment-water interface in the core should be decanted from the tube. This is accomplished by simply turning the core tube to its side, and gently pouring the liquid out until fine sediment particles appear. The core sample is then extruded (pushed from the core) into a clean container.

7.3.2 Grabs or Dredges

There are many different types of grabs samplers (dredges), each with varying applications depending upon substrate, water depth and accessibility. Grab samplers are extremely useful when a larger sample size is necessary or where use of a core tube is impractical. Depending upon the grab sampler chosen and the analyses to be performed, the sample can be deposited directly into a mixing pan or a subsample may be taken from the grab with a hand core tube.

Listed below are some of the more common types of grab samplers utilized by EPA Region 4:

The Peterson Grab has a 12"x12" or .09 m² sampling area. The Peterson dredge may be used when the bottom is rocky, in very deep water, or when the stream velocity is high. The dredge should be lowered very slowly as it approaches bottom, since it can displace and miss fine particle size sediment if allowed to drop freely.

The Eckman Grab is available as a 6"x6" (.023 m²) or a 9"x9" (.05 m²) sampling area. The Eckman dredge performs well where the bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high velocities. It should not be used from a bridge that is more than a few feet above the water, because the spring mechanism which activates the sampler can be damaged by the messenger if dropped from too great a height.

The Ponar Grab has a 9"x9" (.05 m²) sampling area. The Ponar dredge is a modification of the Peterson dredge and is slightly smaller in size and weight. Modifications include the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the dredge as it descends thus reducing turbulence around the dredge. The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates.

The "mini" or "petite" Ponar Grab is a smaller, much lighter version of the Ponar dredge. It has a sampling area of 6"x 6" (.023 m²). It is used to collect smaller sample volumes when working in industrial tanks, lagoons, ponds, and shallow water bodies. It is a good device to use when collecting sludge and sediment containing hazardous constituents because the size of the dredge makes it more amenable to field cleaning.

The Van Veen Grab has a surface area of 14"x11" (0.1 m²). The Van Veen Grab is useful in soft sediments when a large surface area or volume of sediment is needed for analysis or if multiple subsamples are collected from one grab. The Van Veen normally requires a davit or "A" frame with a winch for deployment and retrieval, due to the heavy combined weight of sampler and sediment.

The Young Grab is available with a surface area of either 8"x8" (0.04 m²) or 12.5"x12.5" (0.1 m²). The Young Grab is a modified VanVeen Grab. It is used by federal and state agencies for the collection of benthic macroinvertebrate samples in estuarine and marine environments. The Young grab collects a uniform, relatively undisturbed sample. It is attached in a frame that not only allows the addition of weights for different substrate types, but also adds stability for deeper water sampling. The Young Grab requires a boat or ship with a winch and "A" frame or davit for deployment and retrieval.

7.3.3 Scoops and Spoons

If the sampling area is wadeable and sediment is exposed (i.e., a tidal or freshwater marsh with little or no standing water or a shallow stream with slight current), a sediment sample may be collected using a stainless steel scoop or spoon. A sufficient quantity of sample is deposited directly into the mixing pan. This method should not be utilized if there is a lens of water over the sediment as this would likely result in a loss of fine material. In this case some other method, such as a core tube, should be utilized to prevent loss of fines.

7.4 Sample Handling

Sediment samples collected for chemical or toxicity analysis, (with the exception of those collected for volatile organics analysis), should be placed in glass, stainless or teflon lined pans, (depending upon analysis to be performed), quartered and homogenized according to the method described in Section 4.6.8 of this SOP. It is extremely important that sediment samples be mixed as thoroughly as possible to ensure that the sample is representative of the interval sampled. Sediment samples collected for volatile organics analysis (VOAs) should not be mixed due to the loss of volatile organics from mixing. Procedures for collecting and preserving samples for volatile organic constituent (VOC) analysis vary depending upon whether the samples are freshwater or marine sediments as well as with collection method (i.e. diver collected versus dredge or core tube). Section 7.5 describes sample handling for VOCs.

7.5 Sediment Sampling for Volatile Organic Constituents

7.5.1 Introduction

The following sampling protocol is recommended for assessing the extent of (VOCs) in sediments. These procedures will provide representative VOC concentrations in sediment samples.

Because of the large number of options available, careful coordination between field and laboratory personnel is needed. The specific sampling containers and sampling tools required will depend upon the detection levels and intended data use. Once this information has been established, selection of the appropriate sampling procedure and preservation method can be made.

7.5.2 Equipment

The sampling devices and procedures described previously in this section can be used to collect the sample media from either the surface or shallow subsurface. If surface soil samples are collected, the sample should be placed directly into the VOC sampling device if possible (i.e., from ground to EnCore™ or syringe). If it is not possible to collect the sample directly with one of the devices in Section 10.4.2, then a standard 2 ounce glass VOC container can be used. The EnCore™, syringe, or spatula can be used to subsample from the 2 ounce container. Subsampling from the 2 ounce container should take place as soon as possible but no longer than 30 minutes from the time the container is filled. The subsample should be collected within one minute of opening the 2 ounce container. If subsurface samples are collected, the procedures in Section 10.3 should be used to bring the sample media to the surface and then the procedures described for collecting shallow VOC samples should be followed.

Following is a list of equipment available for collecting soil samples for VOC analysis:

- the EnCore™ VOC sampler;
- syringes; and
- stainless steel spatulas.

The specific sample containers and the sampling tools required will depend upon the data quality objectives established for the sampling investigation.

7.5.3 Sampling Methodology

The sampling methodology utilized is dependent upon the anticipated concentration of VOCs in the samples. The concentration range of each sampling methodology is dependent upon the determinative method, matrix, and compound. **The sampling methodology should be determined based on the data quality objectives (DQOs) and the detection level requirements for the study.** Environmental samples usually contain low concentrations of VOCs and should be collected using low-concentration sampling methodology. If high-concentration sampling methodology is required based upon the study DQOs and detection limit requirements, consult SW846 Method 5035 for sample collection guidance.

Sample Container Preparation

For low-concentration VOC sediment samples, the Analytical Support Branch (ASB) supplies pre-weighed 40 ml VOC vials. Depending on field conditions, the vials may be pre-preserved with sodium bisulfate. If un-preserved samples are collected, they must either be frozen while in the field or transported or shipped to the laboratory for preservation within 48 hours. If the

samples are frozen, a certified thermometer should be obtained from ASB to keep with the samples so that the temperature in the freezer can be monitored. The temperature in the freezer should be $-12^{\circ}\text{C} \pm 3^{\circ}\text{C}$. Dry ice is not acceptable for freezing the samples. The sample vials should be stored horizontally to reduce stress on the sides of the glass vials. VOC trip blanks and spikes should be utilized to insure the integrity of the septum seal on the vial is not compromised due to freezing.

EnCore™ VOC Sampler

The EnCore™ VOC sampler is a commercially available device which has been approved by EPA for collection, storage, and shipping of unpreserved soil VOC samples (Figure 1). EnCore™ samplers are available in 5 gram and 25 gram volumes. The volume of sample required is based upon the VOC analysis that will be conducted. Following are the instructions for use:

Before Taking Sample:

- 1) hold coring body and push plunger rod down until small o-ring rests against tabs. This will assure that plunger moves freely.
- 2) Depress locking lever on EnCore™ T-Handle. Place coring body, plunger end first, into open end of T-Handle, aligning the (2) slots on the coring body with the (2) locking pins in the T-Handle. Twist coring body clockwise to lock pins in slots. Check to ensure sampler is locked in place. Sampler is ready for use.

Taking Sample:

- 3) Turn T-Handle with T-up and coring body down. This positions plunger bottom flush with bottom of coring body (ensure that plunger bottom is in position). Using T-Handle, push sampler into soil until coring body is completely full. When full, small o-ring will be centered in T-Handle viewing hole. Remove sampler from soil. Wipe excess soil from coring body exterior.
- 4) Cap coring body while it is still on T-Handle. Push cap over flat area of ridge and twist to lock cap in place. **CAP MUST BE SEATED TO SEAL SAMPLER.**

Preparing Sampler for Shipment:

- 5) Remove the capped sampler by depressing locking lever of T-Handle while twisting and pulling sampler from T-Handle.
- 6) Lock plunger by rotating extended plunger rod fully counter-clockwise until wings rest firmly against tabs.
- 7) Attach label or tag to cap on coring body.
- 8) Return full EnCore™ sampler to plastic bag. Place a custody seal on the bag and place on ice.

Samples collected with an EnCore™ sampler must be transported or shipped to the

laboratory for preservation. The samples must be preserved within 48 hours of collection. Therefore, daily transport or shipment to the laboratory is required.

Syringes

Disposable syringes can be obtained from the ASB for collection of VOC samples. Approximately 5 grams of sample should be collected with the syringe. The sample should be extruded into a 40 mL pre-prepared vial. Three 40 mL vials should be filled for each sample location. The containers along with the identifying tag should be placed in a plastic bag. Do not place the tag on the container. A custody seal should be placed on the bag, not the containers. When using the syringes, it is important that air is not allowed to become trapped behind the sample prior to extrusion into the 40 mL vial.

Stainless Steel Spatula

Stainless steel spatulas can be obtained from the Analytical Support Branch for collecting 5 grams of sample media to place into 40 mL pre-prepared containers. Three containers should be filled for each sample location. The containers along with the identifying tag should be placed in a plastic bag. Do not place the tag on the container. A custody seal should be placed on the bag rather than the sample container.

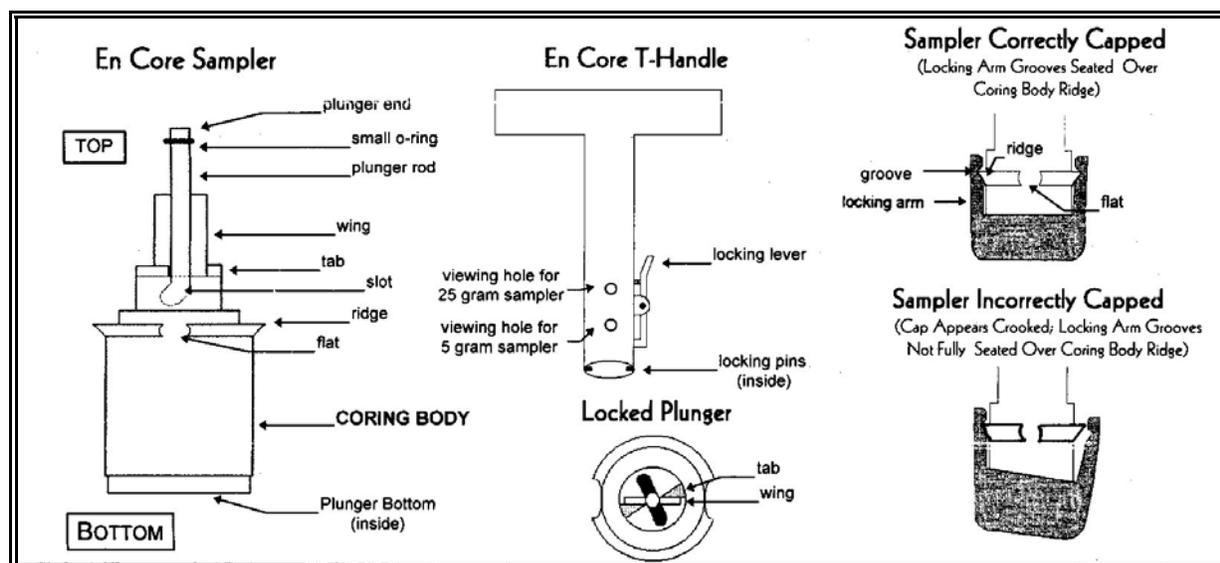


Figure 1: EnCore Soil VOC Sampler

7.5.4 Special Techniques and Considerations

Effervescence

Effervescence, the rapid formation of bubbles, occurs when there is a reaction between the sample media and the preservative. If there is uncertainty regarding low concentration samples effervescing from contact with the preservative, then either a test for effervescence must be

performed during a reconnaissance, the field investigator should be equipped to collect samples both preserved or un-preserved, or all samples should be collected un-preserved.

To check for effervescence, collect a test sample and add to a pre-preserved vial. If preservation of the sample results in effervescence, the preservation by acidification is not acceptable, and the sample must be collected un-preserved.

In the event effervescence occurs unexpectedly and only pre-preserved vials are available, the preservative solution may be placed into an appropriate hazardous waste container and the vials triple rinsed with organic free water. An appropriate amount of organic free water, equal to the amount of preservation solution, should be placed into the vial. The sample may then be collected as an un-preserved sample. Note that the amount of organic free water placed into the vials will have to be accurately measured and the weight recorded.

Marine Sediments

Due to the calcium carbonates found in almost all marine sediments in the southeast, effervescence will occur if VOC vial are preserved with sodium bisulfate. Therefore, VOC collection in marine sediments should be conducted utilizing the SW846 Method 5035 to limit the loss of volatile organics and reduce the possibility of contamination from site conditions, (i.e. diesel fumes from ship operations). Divers collect the VOC sample utilizing the standard 2 ounce sediment container with a septum sealed lid. The container will be filled with organic free water at the laboratory prior to the survey. This is to allow the diver to remove the lid once the container is under pressure during the dive. Once the VOC sample is back on board the diving vessel, approximately 5 grams of sediment from the 2 oz container will be added to a pre-weighed, non-preserved, 40 ml vial containing 10 mls of milli-Q water that was added at the laboratory prior to the survey. Two to three replicates will be taken from the 2 oz container. These samples will then be placed in a whirl-pak or other plastic bag along with the sample tag and placed in the freezer on their sides in a protective container. The freezer temperature should be kept at $-12^{\circ}\text{C} \pm 3^{\circ}\text{C}$ in order to prevent failure of the septum seal on the container. Samples should be transported to the laboratory for analysis as soon as possible.

Sample Size

While this method of sampling is an improvement over earlier methods, field investigators must be aware of an inherent limitation. Due to the extremely small sample size, sample representativeness of VOCs may be reduced when compared to samples with larger volumes collected for other constituents. The sampling design and objectives of the investigation should take this into consideration.

Percent Moisture

Sufficient sample volume must be sent to the laboratory to determine percent moisture in the VOC soil sample to correct the results to dry weight. If other analyses requiring percent moisture determination are being performed upon the sample, these results may be used. If not, a separate sample (minimum of 2 ounces) for percent moisture determination is required

Sample Holding Times

Sample holding times are specified in Appendix A. Field investigators should note that the holding time for an unpreserved VOC soil/sediment sample is 48 hours. Arrangements should be made to ship the soil/sediment VOC samples to the laboratory by overnight delivery the day they are collected so the laboratory may prepare and preserve the sample within 48 hours of collection.

7.5.5 Summary

The following options are suggested for compliance with SW846 Method 5035. The advantages and disadvantages for each option are noted.

OPTION	PROCEDURE	ADVANTAGES	DISADVANTAGES
1	Collect 2 - 40 mL vials with 5 grams of sample and 1 - 2-oz., glass w/septum lid for screening and % moisture	Screening conducted by lab.	Presently, a 48 hour holding time for unpreserved samples.
2	Collect 3 Encore™; and 1 - 2-oz., glass w/septum lid for screening and % moisture	Lab conducts all preservation/preparation procedures.	Presently, a 48 hour holding time for preparation of samples.
3	Collect 2 - 40 ml vials with 5 grams of sample and preserve with sodium bisulfate and 1 - 2-oz., glass w/septum lid for screening and % moisture	Longer holding time.	Hazardous materials used in field.

7.5.6 Shipping

Sodium bisulfate is considered a hazardous material. Therefore, shipping of the sample containers with the preservative is regulated by the U.S. Department of Transportation and the International Air Transport Association (IATA). The rules of shipment set in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179) and the current edition of the IATA Dangerous Goods Regulation must be followed when shipping sodium bisulfate between the laboratory and the field. Consult the above documents or the shipping company for additional information. The shipment of the quantity of sodium bisulfate used for the sample preservation falls under the exemption for small quantities. A summary of the requirements for shipping samples follows. Refer to the code for a complete review of the requirements.

- (1) The maximum volume of sodium bisulfate in a sample container is limited to thirty (30) mls.
- (2) The sample container must be stored upright and have the lid held securely in place. The mechanism used to hold the cap in place must be able to be completely removed so weight is not added to the sample container.
- (3) Sample containers must be packed in a sorbent material capable of absorbing spills from leaks or breakage of the sample containers.

- (4) The maximum sample shuttle weight must not exceed 64 pounds.
- (5) The maximum volume of sodium bisulfate per shipping container is 500 mls.
- (6) The shipper must mark the sample shuttle in accordance with shipping dangerous goods in acceptable quantities.
- (7) The package must not be opened or altered until no longer in commerce.

7.6 References

- 1. En Novative Technologies, Inc. 1998. Disposable EnCore® Sampler: Sampling Procedures. 2pp. (Photocopy)
- 2. U.S. EPA. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846) Third Edition, Method 5035.

SECTION 8 FLUVIAL SEDIMENT SAMPLING

PERFORMANCE OBJECTIVE:

- The objective of this section is to provide guidance on collecting fluvial sediment (bedload and suspended).

8.1 Introduction

8.1.1 Bedload Sediment

Bedload is defined as sediment that is transported along the streambed by sliding, rolling, or bouncing (Hubbell, 1964, Leopold et al., 1964, Emmett, 1980a). Bedload samplers are designed to estimate mobilized bedload which is conveyed usually within 3 inches of the streambed.

Interferences/sample collection error. Obtaining bedload measurements representative of the actual bedload is problematic. Sampling personnel should recognize and take steps to minimize the following potential error associated with bedload sampling and sampling equipment:

- If the streambed is irregular, the sampler could be posed unevenly on the streambed and a representative sample would not be collected;
- When deployed on the streambed, the sampler may disturb the existing stream flow and bed material; and
- There is a time and space (in cross-section and planform) variation in bedload transport rate and stream velocity, consequently, the sample collected at a given point may not be representative of the actual mean sediment transport rate.

8.1.2 Suspended Sediment

The premise behind integrated sampling is to composite the vertical water column resulting in a sample which is representative of the relative quality and quantity of suspended sediment throughout the water column at a point in space and time.

Interferences/sample collection error. Sampling personnel should recognize and take steps to minimize the following potential error associated with suspended sediment sampling:

- The stream may not be completely mixed in cross section;
- When deploying the suspended sediment sampler, the sampler may cause resuspension of bed material; and
- There is a time and space (in cross-section and planform) variation in suspended sediment transport rate and stream velocity, consequently, the sample collected at a

given point may not be representative of the actual mean sediment transport rate.

8.2 Equipment

8.2.1 Bedload Sediment

Helley-Smith bedload samplers or an equivalent should be used to sample sediment being transported along the streambed. Both hand held and suspended models are acceptable with either 3-inch or 6-inch intake. The preferred intake size will depend upon the anticipated channel particle size. For instance, bed material found within the Piedmont and Coastal Plain physiographies are fine-grained, thus the 3-inch intake size may be adequate. However, a comparative study between different sized samplers should be conducted before proceeding with the 3-inch sampler *in lieu* of the 6-inch sampler. A sampler bag constructed of Nitex™ nylon or equivalent is attached to the bedload sampler. For most applications, the sampler bag should have a 0.25 mm mesh size.

8.2.2. Suspended Sediment

An integrated sampler (e.g., DH-59 or equivalent) should be capable of compositing the vertical water column and collecting a sample which is representative of the fluvial sediment being conveyed in the water column of the stream. The integrated sediment sampler should be capable of isokinetic sampling (i.e., the nozzle orientation and orifice are designed to simulate the flow and sediment transport rate of the stream).

8.3 Sampling Methodology

8.3.1 Site Selection

In order to collect representative bedload samples, reduction in the error associated with sample collection identified above is important. The step-wise field procedure for bedload deployment is meant for general guidance and not a replacement for judgement in identifying station locations that reduce error associated with bedload collection. Selection of sampling locations requires evaluation of local conditions. The following criteria should be utilized to identify representative stations:

- The stream should be relatively free of obstructions including large boulders, center bars, detrital debris, and standing vegetation;
- The stream should have relatively even velocity in cross-section;
- Upstream of the station, the stream should be free of recent activities (e.g., beaver dams, man-made structures) that impede the “natural” conveyance of sediment; and
- In general, the station should not be located immediately downstream of a sediment source that does not have an adequate mixing zone resulting in an uneven distribution of sediment at the sample collection cross-section.

8.3.2 Bedload Deployment

Once an appropriate cross section is established, the cross section should be cleared of large boulders and debris and a relatively flat surface maintained throughout sampling. The width of the

wetted surface of the stream should be determined by locating zero tape on the left-edge-of-water (LEW) and measuring across to the right-edge-of-water (REW). LEW and REW are determined looking down stream. It may be required to repeat this step during storm event sampling, given the temporal variation in stage and wetted surface width. However, once cross sectional spacing for bedload and suspended load sampling is established during baseflow conditions, it is at the discretion of the project leader if horizontal spacing is to be re-established. In general, the more intervals sampled per traverse, the greater the probability that the composited sample is representative of the true bedload discharge. However, the number of points sampled per traverse must be weighed against the allowable time at each station during a storm event. In general for stream widths less than 100 feet, the bedload sampler should be deployed at five (5) pre-determined, equally-spaced points per traverse for 2 minutes. Two traverses are recommended for a total of 20 minutes of bedload sampling per station.

Depending on stream conditions (i.e., stream depth and velocity), the bedload sampler is outfitted with a 6 to 8-foot pipe handle for wading or is lowered off the culvert or bridge from a cable and winch system.

8.3.3 Integrated Sampler Deployment

Sampling stations should be established taking into consideration the criteria identified above in Bedload Sampling. Sampling personnel should be familiar with manufacturers instructions on use of specific integrated sampler to be used. In general, the container (glass milk bottle) should not be completely filled during vertical integration of the water column. If the container is completely filled, it is not representative (both quality and quantity) of the suspended sediment throughout the water column and should be discarded. The optimal volume of sample collected should be between 375 and 440 ml. Sampling personnel should develop the skills required to collect a fully integrated sample given variation in local conditions (e.g., water depth, velocity).

Once an appropriate cross section is established, deploy the integrated sampler at evenly-spaced points across the stream cross-section. It is at the discretion of the project leader to determine the number of sample points required. In general, in streams less than 50 feet wide that are well mixed, two evenly-spaced sample points are adequate. Larger stream widths may require 3 or 4 sample points. The sample bottle should be filled according to manufacturer's specifications. Depending upon the required sample volume, the sample can be composited across sample points within the bottle or samples from each sample point can be decanted into a small mixing pail and additional samples collected until an adequate volume of sample is produced. While gently swirling the pail, fill a glass cubette, and measure turbidity using a LaMotte™ Model 2020 or equivalent.

8.3.4 Bedload and Integrated Suspended Load: Step-wise Procedure

1. Install safety equipment and traffic control as required (crew chief to determine level of protection);
2. Record tape down (TD) for stage/discharge determination - *RECORD TD, Date, Time, weather conditions, and field personnel*;
3. Establish location for bedload and suspended load cross-section:
 1. locate zero tape on left bank or bridge;
 2. identify left-edge-of-water (LEW) and right-edge-of-water (REW) -

RECORD;

Note: steps 4 and 5 can be conducted simultaneously being cautious of preventing cross-contamination:

4. Deploy bedload sampler at five (5) pre-determined, equal intervals across the stream cross-section for 2 minutes each and repeat cross-section for a total of 2 traverses totaling 20 minutes;
5. Carefully, concentrate sediment into one corner of the nitex bag and decant into 1-liter, wide-mouth Nalgene™ jar;
6. Deploy integrated sampler at 2 evenly distributed intervals across the stream cross-section (larger x-sections may require 3 or 4 intervals);
 1. allow milk bottle to fill according to manufacturer's specifications;
 2. estimate volume of sample collected in milk bottle - *RECORD*;
 3. decant milk bottle contents into small mixing pail and repeat (a) and (b) until an adequate volume of sample is collected;
 4. while gently swirling the pail, fill a glass cubette, and measure turbidity-*RECORD*;
 5. discard cubette contents;
 6. decant remainder of sample from pail to the appropriate sample containers;
 7. preserve sample according to the guidelines in Appendix A of this SOP;
 8. Clean DH-59 nozzle and glass bottle with three (3) rinses of deionized water;
7. Record end tape down and time.

8.4 Sample Handling

8.4.1 Bedload Sediment

The composited sample is concentrated into one corner of the bedload mesh bag and decanted into 1-liter, wide-mouth Nalgene™ jar or equivalent. The samples are preserved at 4°C and transported to the laboratory for analysis.

8.4.2 Suspended Sediment

Following field determination of turbidity, decant the remainder of sample into a 1 liter plastic container for TSS/TDS and/or suspended sediment concentration (SSC) analysis or ½-gallon milk jug for nutrients (if required). Preserve the samples at 4°C and nutrients with 10% H₂SO₄ to pH < 2 and 4°C.

8.5 Safety Equipment

Health and safety of personnel while collecting sediment samples from bridges, culverts, and roadways is a priority. Traffic control will be maintained during sampling events conducted off bridges and culverts, whenever lane closure is required. Procedures on lane closure recommended in the Manual on Uniform Traffic Control Devices-MUTCD (USDOT/FHWA 1988) will be adopted. MUTCD offers several applications depending on visibility and traffic volume.

Consequently, it will be at the discretion of the crew chief to determine the required level of protection. In scenarios where visibility is obscured (in both directions) and traffic volume is high, the maximum level of protection will be employed and, in general, will include the following (Figure 8.1):

Deployed on each end of bridge or culvert (when required):

1. Two (2) barricades equipped with amber strobe lights will be positioned from maximum visibility and effect for on-coming traffic;
2. One (1) each warning signs: LANE CLOSED AHEAD, BE PREPARED TO STOP, and FLAGGER; and
3. One (1) traffic-control personnel with flame orange vest, orange flag, and handheld, two-way radio.

Deployed on bridge or in closed lane:

1. One crane truck equipped with amber strobe light and two-way radio; and
2. One safety vehicle equipped with amber strobe light posed toward on-coming traffic.

Orange safety cones should be distributed throughout the lane closure zone. The number and spacing will depend upon the site conditions (e.g., driver visibility). Flagmen will be trained and practiced on communications and traffic control methods. All sample and traffic control personnel will be equipped with 2-way radios. The crew chief will be in constant communications with the flagmen as well as samplers.

8.6 References

1. Emmett, W.W. 1980a. *A field calibration of the sediment-trapping characteristics of the Helley-Smith bedload sampler*. U.S. Geological Survey Professional Paper 1139. 44 p.
2. Hubbell, D.W. 1964. *Apparatus and techniques for measuring bedload*. U.S. Geological Survey. Water-Supply Paper 1948. 74 p.
3. Leopold, L.B. M.G. Wolman, and J.P. Miller. 1964. *Fluvial processes in geomorphology*. W.H. Freeman and Company, San Francisco, California. 522 p.
4. USDOT/FHWA. 1988. *Manual on Uniform Traffic Control Devices* (MUTCD, 1988 Edition).

SECTION 9 PORE-WATER SAMPLING

PERFORMANCE OBJECTIVE:

- To collect a representative sample of pore-water from sediment.

9.1 Introduction

Sampling techniques and equipment are designed to minimize effects on the chemical and physical integrity of the sample. If the guidance in this section is followed, a representative sample of pore water should be obtained.

9.2 Sampling Equipment

9.2.1 Collection Device

Pore-water may be collected using a vacuum-operated pore-water extractor, also known as a sipper (Figure 9-1). A sipper consists of a filter attached with flexible tubing to a 60-cc syringe. Pore-water is extracted by inserting the filter into the sediment and creating a vacuum by retracting and bracing the syringe plunger. To provide structure and protection, the filter and tubing may be encased in a stainless steel pipe (insertion tool) fitted with a flange. Water tight extensions may be used for sampling in deeper water.

9.3 Sampling Methodology

Method limitations resulting from small sample volumes may be overcome by deployment of multiple sippers placed at least 30 centimeters (cm) apart. To prevent recruitment of surface water, no more than 30 milliliters (ml) should be extracted per sipper site.

Specific methods for collecting pore-water samples for nutrient/anion and sulfide analyses are described below.

9.3.1 Nutrient/anion samples

1. Load each insertion tool with a sipper and attached tubing.
2. Locate sites at least 30 cm apart which are relatively free of standing vegetation.
3. Press the insertion tool into the soil/sediment firmly assuring the flange is tight against the soil/sediment surface with the flange ring imbedded into the soil/sediment.
4. Use the insertion rod to push the sipper into the undisturbed soil/sediment.

5. Connect a syringe (60 ml) to the female luer fitting on each sipper.
6. Apply suction and pull ten ml into the syringe. [The void volume of an empty sipper, with 65cm length tubing, is approximately 1.5 ml. Pulling slightly greater volumes than this assures flushing.] Disconnect the syringe from the luer fitting and attach a syringe filter-holder with enclosed filter (for filtering nutrient/anion samples). Filter this water into the sample bottle in three (3) ml increments to rinse the filter and the bottle discarding each in succession. Reapply suction to collect approximately 20 ml of interstitial soil/sediment water. No more than 30 mls should be collected from one sipper site. Reapply filter and filter-holder and place remaining 20 mls in the rinsed 30 ml polyethylene bottle.
7. Samples should be placed on ice immediately.
8. Pore-water samples (nutrients and anions) should be stored either in a refrigerator and analyzed immediately (<24 h) or stored frozen until analyzed (<30 d).
9. If surface water depth exceeds the height of the insertion tube, add an extension at step 1 and proceed as directed. The amount of water for flushing will be increased to 15 ml when the extension is used.

9.3.2 Sulfide samples

The syringe used for the sulfide samples will be pre-preserved in the laboratory with zinc acetate and sodium hydroxide, a Luer-Lok stopcock (four-way polycarbonate) will be attached to the syringe, valve closed and capped.

Follow Steps 1 through 4 in Section 9.3.1.

- 5 Remove stopcock cap and attach syringe to the sipper tube. Take a second non-preserved syringe and attach it to one of the other ports of the stopcock. Carefully draw all the air out of the sipper tube with the non-preserved syringe and while keeping a vacuum on the non-preserved syringe, turn the valve toward the preserved syringe and draw the 30 ml interstitial soil water sample into the preserved syringe. Once 30 ml has been drawn into the preserved syringe, turn the valve back to the non-preserved syringe, disconnect the non-preserved syringe from the stopcock and the preserved syringe and stopcock from the sipper tube, replace the cap on the stopcock, label the preserved syringe and place in a container for transport **No more than 30 mls should be collected from one site.**
- 6 Preserved sulfide samples will be analyzed immediately upon return from the field.
- 7 If surface water depth exceeds the height of the insertion tube add extension at step 1 and proceed as directed. The amount of water for flushing will be increased to 15 ml when the extension is used.

9.4 Quality Control

1. One nutrient/anion blank should be provided for every 20 samples collected or one for every batch shipped. The blank consists of a syringe with appropriate labeling attached. The syringe should be filled with organic/analyte free water in the laboratory.
2. One sulfide interstitial soil/sediment water blank should be provided for every 20 samples collected or one for every batch shipped. The blank consists of a pre-preserved syringe with appropriate labeling attached. The pre-preserved syringe should be filled with Milli-Q water in the laboratory.
3. A duplicate interstitial soil water sample should be collected at 10% of the field locations.

Specifications (measurements, parts, etc.) of sipper and insertion tool used by EPA Region 4 may be found in:

Stober, Q.J., K. Thornton, R. Jones, J. Richards, C. Ivey, R. Welch, M. Madden, J. Trexler, E. Gaiser, D. Scheidt, and S. Rathbun. 2001. South Florida Ecosystem Assessment: Phase I/II (Technical Report) - Everglades Stressor Interactions: Hydropatterns, Eutrophication, Habitat Alteration, and Mercury Contamination. EPA 904-R-01-003. U.S. Environmental Protection Agency, Region 4, Science & Ecosystem Support Division and Office of Research and Development.

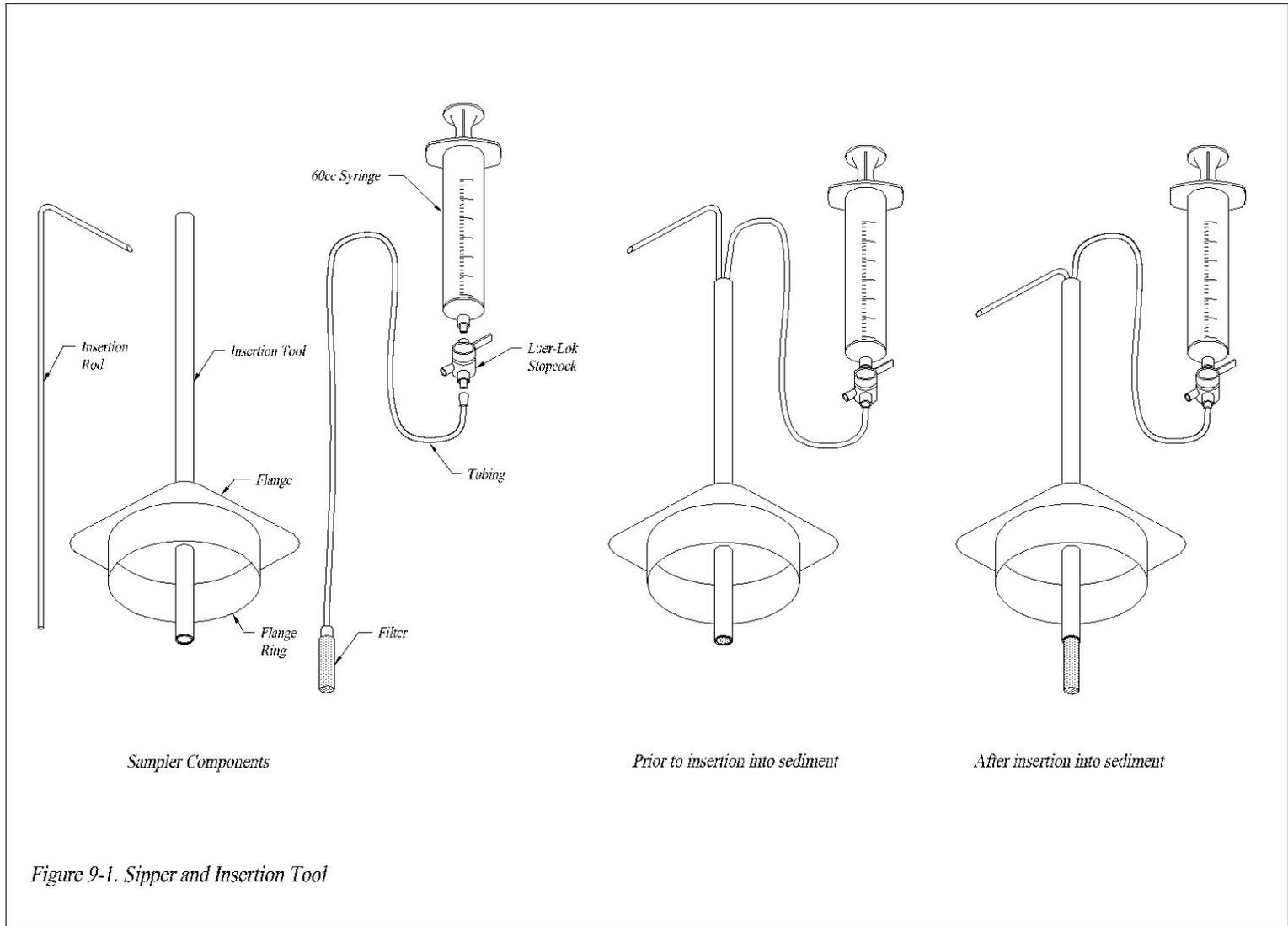


Figure 9-1. Sipper and Insertion Tool

SECTION 10 SOIL SAMPLING

PERFORMANCE OBJECTIVES:

To collect a soil sample that is representative of conditions as they exist at the site

- By selecting the appropriate sampling device(s).
- By taking measures to avoid introducing contamination as a result of poor sampling and/or handling technique.
- By reducing the potential of cross contamination between samples.

10.1 Introduction

This section discusses the sampling equipment available and collection methods which have been shown to be technically appropriate for collecting representative soil samples.

10.2 Equipment

Equipment used to collect soil samples for trace contaminant analyses should be constructed of inert materials such as stainless steel. Ancillary equipment such as auger flights, post hole diggers, etc. may be constructed of other materials since this equipment does not come in contact with the samples. However, plastic, chromium, and galvanized equipment should not be used routinely in soil sampling operations. Painted or rusted equipment must be sandblasted before use.

Selection of equipment is usually based on the depth of the samples to be collected, but it is also controlled by the characteristics of the soil. The following equipment is available at the EPA Field Equipment Center for collecting surface and shallow subsurface soil samples:

- stainless steel spoons;
- stainless steel hand augers;
- push tubes; and
- post hole diggers.

10.3 Sampling Methodology

This discussion of soil sampling methodology reflects both the equipment used to collect the sample, as well as how the sample is handled and processed after retrieval of the material. There are two sampling methodologies for collecting soil samples: 1) collection with manual or hand operated devices, and 2) collection with powered devices. Simple, manual techniques and equipment, such as hand augers, are usually selected for surface or shallow, subsurface soil

sampling. As the depth of the sampling interval increases, some type of powered sampling equipment may be needed to overcome torque induced by soil resistance and depth. If powered equipment is required to assist in collecting subsurface soil samples, refer to the Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, November 2001 for guidance.

Surface Soil Sampling

Surface soils are generally classified as soils between the ground surface and 6 to 12 inches below ground surface. The shallow subsurface interval may be considered to extend from approximately 6 to 12 inches below ground surface to a site-specific depth at which sample collection using manual methods becomes impractical.

Surface samples are removed from the ground, placed in pans, and mixed as instructed in Section 4.6.8. It should be noted that samples collected for volatile organic analyses are never mixed. If a thick, matted root zone is encountered at or near the surface, it should be removed before the sample is collected. Specific procedures for collecting samples for volatile organic compound analysis are in Section 10.4.

Shallow Sub-surface Soil Sampling

Hand-augering is the most common manual method used to collect subsurface samples. Typically, 4-inch auger-buckets with cutting heads are twisted into the ground and removed as the buckets are filled. The auger holes are advanced one bucket at a time. The practical depth of investigation using a hand-auger is related to the material being sampled. At depths approaching 20 feet, torquing of hand-auger extensions becomes so severe that in resistant materials, powered methods must be used if deeper samples are required. Most powered methods are not acceptable for actual sample collection, but are used to gain access to the required sample depth, where hand-augers or push tubes are used to collect the sample.

When a vertical sampling interval is required, one auger-bucket is used to advance the auger hole to the first desired sampling depth. If the sample is a vertical composite, the same bucket may be used to advance the hole, as well as to collect subsequent aliquots in the same hole. However, if discrete grab samples are to be collected, a clean bucket must be placed on the end of the auger extension immediately prior to collecting the next sample. The top several inches of soil should be removed from the bucket to minimize the chances of cross-contamination of the sample from fall-in of material from the upper portions of the hole.

Another hand-operated piece of soil sampling equipment commonly used to collect shallow subsurface soil samples is the Shelby® tube or "push tube". This is a thin-walled tube, constructed of stainless steel and has a beveled leading edge. The tube is twisted and pushed directly into the soil. This type of sampling device is useful if an undisturbed sample is required. The sampling device is removed from the push-head, then the sample is extruded from the tube into the pan with a spoon or special extruder. Even though the push-head is equipped with a check valve to help retain samples, the Shelby® tube will not retain loose or watery soils, particularly at greater depths.

Sample Mixing

It is extremely important that soil samples be mixed as thoroughly as possible to ensure that

the sample is representative of the interval sampled. Soil samples with the exception of samples collected for VOC analysis should be mixed as specified in Section 4.6.8.

10.4 Soil Sampling for Volatile Organic Constituents

10.4.1 Introduction

The following sampling protocol is recommended for assessing the extent of volatile organic compounds (VOCs) in soils. These procedures will provide representative VOC concentrations in soil samples. Because of the large number of options available, careful coordination between field and laboratory personnel is needed. The specific sampling containers and sampling tools required will depend upon the detection levels and intended data use. Once this information has been established, selection of the appropriate sampling procedure and preservation method can be made.

10.4.2 Equipment

The sampling devices and procedures described previously in this section can be used to collect the sample media from either the surface or shallow subsurface. If surface soil samples are collected, the sample should be placed directly into the VOC sampling device if possible (i.e., from ground to EnCore™ or syringe). If it is not possible to collect the sample directly with one of the devices in Section 10.4.2, then a standard 2 ounce glass VOC container can be used. The EnCore™, syringe, or spatula can be used to subsample from the 2 ounce container. Subsampling from the 2 ounce container should take place as soon as possible but no longer than 30 minutes from the time the container is filled. The subsample should be collected within one minute of opening the 2 ounce container. If subsurface samples are collected, the procedures in Section 10.3 should be used to bring the sample media to the surface and then the procedures described for collecting shallow VOC samples should be followed.

Following is a list of equipment available for collecting soil samples for VOC analysis:

- the EnCore™ VOC sampler;
- syringes; and
- stainless steel spatulas.

The specific sample containers and the sampling tools required will depend upon the data quality objectives established for the sampling investigation.

10.4.3 Sampling Methodology

The sampling methodology utilized is dependent upon the anticipated concentration of VOCs in the samples. The concentration range of each sampling methodology is dependent upon the determinative method, matrix, and compound. **The sampling methodology should be determined based on the data quality objectives (DQOs) and the detection level requirements for the study.** Environmental samples usually contain low concentrations of VOCs and should be collected using low-concentration sampling methodology. If high-concentration sampling methodology is required based upon the study DQOs and detection limit requirements, consult SW846 Method 5035 for

sample collection guidance.

Sample Container Preparation

For low-concentration VOC samples, the Analytical Support Branch (ASB) supplies pre-weighed 40 ml VOC vials. Depending on field conditions, the vials may be pre-preserved with sodium bisulfate. If un-preserved samples are collected, they must either be frozen while in the field or transported or shipped to the laboratory for preservation within 48 hours. If the samples are frozen, a certified thermometer should be obtained from ASB to keep with the samples so that the temperature in the freezer can be monitored. The temperature in the freezer should be -10°C . Dry ice is not acceptable for freezing the samples. The sample vials should be stored horizontally to reduce stress on the sides of the glass vials. VOC trip blanks and spikes should be utilized to insure the integrity of the septum seal on the vial is not compromised due to freezing.

EnCore™ VOC Sampler

The EnCore™ VOC sampler is a commercially available device which has been approved by EPA for collection, storage, and shipping of unpreserved soil VOC samples (Figure 1). EnCore™ samplers are available in 5 gram and 25 gram volumes. The volume of sample required is based upon the VOC analysis that will be conducted. Following are the instructions for use:

Before Taking Sample:

- 1) hold coring body and push plunger rod down until small o-ring rests against tabs. This will assure that plunger moves freely.
- 2) Depress locking lever on EnCore™ T-Handle. Place coring body, plunger end first, into open end of T-Handle, aligning the (2) slots on the coring body with the (2) locking pins in the T-Handle. Twist coring body clockwise to lock pins in slots. Check to ensure sampler is locked in place. Sampler is ready for use.

Taking Sample:

- 3) Turn T-Handle with T-up and coring body down. This positions plunger bottom flush with bottom of coring body (ensure that plunger bottom is in position). Using T-Handle, push sampler into soil until coring body is completely full. When full, small o-ring will be centered in T-Handle viewing hole. Remove sampler from soil. Wipe excess soil from coring body exterior.
- 4) Cap coring body while it is still on T-Handle. Push cap over flat area of ridge and twist to lock cap in place. **CAP MUST BE SEATED TO SEAL SAMPLER.**

Preparing Sampler for Shipment:

- 5) Remove the capped sampler by depressing locking lever of T-Handle while twisting and pulling sampler from T-Handle.
- 6) Lock plunger by rotating extended plunger rod fully counter-clockwise until wings rest firmly against tabs.

- 7) Attach label or tag to cap on coring body.
- 8) Return full EnCore™ sampler to plastic bag. Place a custody seal on the bag and place on ice.

Samples collected with an EnCore™ sampler must be transported or shipped to the laboratory for preservation. The samples must be preserved within 48 hours of collection. Therefore, daily transport or shipment to the laboratory is required.

Syringes

Disposable syringes can be obtained from the ASB for collection of VOC samples. Approximately 5 grams of sample should be collected with the syringe. The sample should be extruded into a 40 mL pre-prepared vial. Three 40 mL vials should be filled for each sample location. The containers along with the identifying tag should be placed in a plastic bag. Do not place the tag on the container. A custody seal should be placed on the bag, not the containers. When using the syringes, it is important that air is not allowed to become trapped behind the sample prior to extrusion into the 40 mL vial.

Stainless Steel Spatula

Stainless steel spatulas can be obtained from the Analytical Support Branch for collecting 5 grams of sample media to place into 40 mL pre-prepared containers. Three containers should be filled for each sample location. The containers along with the identifying tag should be placed in a plastic bag. Do not place the tag on the container. A custody seal should be placed on the bag rather than the sample container.

10.4.4 Special Techniques and Considerations

Effervescence

Effervescence, the rapid formation of bubbles, occurs when there is a reaction between the sample media and the preservative. If there is uncertainty regarding low concentration samples effervescing from contact with the preservative, then either a test for effervescence must be performed during a reconnaissance, the field investigator should be equipped to collect samples both preserved or un-preserved, or all samples should be collected in an un-preserved container.

To check for effervescence, collect a test sample and add to a pre-preserved vial. If preservation of the sample results in effervescence, the preservation by acidification is not acceptable, and the sample must be collected un-preserved.

In the event effervescence occurs unexpectedly and only pre-preserved vials are available, the preservative solution may be placed into an appropriate hazardous waste container and the vials triple rinsed with organic free water. An appropriate amount of organic free water, equal to the amount of preservation solution, should be placed into the vial. The sample may then be collected as an un-preserved sample. Note that the amount of organic free water placed into the vials will have to be accurately measured and the weight recorded.

Sample Size

While this method of sampling is an improvement over earlier methods, field investigators must be aware of an inherent limitation. Due to the extremely small sample size, sample representativeness of VOCs may be reduced when compared to samples with larger volumes collected for other constituents. The sampling design and objectives of the investigation should take this into consideration.

Percent Moisture

Sufficient sample volume must be sent to the laboratory to determine percent moisture in the VOC soil sample to correct the results to dry weight. If other analyses requiring percent moisture determination are being performed upon the sample, these results may be used. If not, a separate sample (minimum of 2 ounces) for percent moisture determination is required

Sample Holding Times

Sample holding times are specified in Appendix A. Field investigators should note that the holding time for an unpreserved VOC soil/sediment sample is 48 hours. Arrangements should be made to ship the soil/sediment VOC samples to the laboratory by overnight delivery the day they are collected so the laboratory may prepare and preserve the sample within 48 hours of collection.

10.4.5 Summary

The following options are suggested for compliance with SW846 Method 5035. The advantages and disadvantages for each option are noted.

OPTION	PROCEDURE	ADVANTAGES	DISADVANTAGES
1	Collect 2 - 40 mL vials with 5 grams of sample and 1 - 2-oz., glass w/septum lid for screening and % moisture	Screening conducted by lab.	Presently, a 48 hour holding time for unpreserved samples.
2	Collect 3 Encore™; and 1 - 2-oz., glass w/septum lid for screening and % moisture	Lab conducts all preservation/preparation procedures.	Presently, a 48 hour holding time for preparation of samples.
3	Collect 2 - 40 ml vials with 5 grams of sample and preserve with sodium bisulfate and 1 - 2-oz., glass w/septum lid for screening and % moisture	Longer holding time.	Hazardous materials used in field.

10.4.6 Shipping

Sodium bisulfate is considered a hazardous material. Therefore, shipping of the sample containers with the preservative is regulated by the U.S. Department of Transportation and the International Air Transport Association (IATA). The rules of shipment set in Title 49 of the Code

of Federal Regulations (49 CFR parts 171 to 179) and the current edition of the IATA Dangerous Goods Regulation must be followed when shipping sodium bisulfate between the laboratory and the field. Consult the above documents or the shipping company for additional information. The shipment of the quantity of sodium bisulfate used for the sample preservation falls under the exemption for small quantities. A summary of the requirements for shipping samples follows. Refer to the code for a complete review of the requirements.

- (1) The maximum volume of sodium bisulfate in a sample container is limited to thirty (30) mls.
- (2) The sample container must be stored upright and have the lid held securely in place. The mechanism used to hold the cap in place must be able to be completely removed so weight is not added to the sample container.
- (3) Sample containers must be packed in a sorbent material capable of absorbing spills from leaks or breakage of the sample containers.
- (4) The maximum sample shuttle weight must not exceed 64 pounds.
- (5) The maximum volume of sodium bisulfate per shipping container is 500 mls.
- (6) The shipper must mark the sample shuttle in accordance with shipping dangerous goods in acceptable quantities.
- (7) The package must not be opened or altered until no longer in commerce.

10.5 References

1. En Novative Technologies, Inc. 1998. Disposable EnCore® Sampler: Sampling Procedures. 2pp. (Photocopy)
2. U.S. EPA. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846) Third Edition, Method 5035.

SECTION 11 OXIDATION - REDUCTION POTENTIAL

SECTION OBJECTIVE:

- The objective of this section is to provide guidance on calibration, application, and interpretation of oxidation - reduction potential.

11.1 Introduction

Oxidation is the process of liberating electrons or gaining oxygen. Examples of oxidation include conversion of elemental iron to rust, elemental sulfur to sulfate, and elemental hydrogen to water (Pankow 1991). Reduction is the process of gaining electrons or hydrogen ions and/or liberating oxygen resulting in the charge on some atomic unit in the species to be reduced. Oxidation-reduction potential (ORP) or redox potential (hereafter, referred to as redox) is a measure of the intensity or activity of an aqueous environment or soil to mediate reactions of important elements in biological systems (e.g., O, N, Mn, Fe, S, and C) and other metallic elements.

11.2 Redox Chemistry

Pankow (1991) described the negative logarithm of the electron activity (pe) as the master variable for describing the equilibrium position for all redox couples in a given system:

$$pe \equiv -\log \{e\} \quad (1)$$

According to Faulkner et al. (1989) redox is a quantitative measure of electron availability and is indicative of the intensity of oxidation or reduction in both chemical and biological systems. When based on a hydrogen scale, redox (E_H) is derived from the Nernst Equation (Stumm and Morgan 1981):

$$E_H = E_H^\circ + 2.3 \times (R \times T)/nF \times \log \left(\prod_i \{ox\}^{m_i} / \prod_j \{red\}^{n_j} \right) \quad (2)$$

where

E_H° = potential of reference, mV

R = gas constant = 81.987 cal deg⁻¹ mole⁻¹

T = temperature, °K

n = number of moles of electrons transferred

F = Faraday constant = 23.061 cal/mole-mv

{ox} and {red} = activity of the oxidants and reductants, respectively

At standard conditions, pe can be estimated from E_H by:

$$pe = E_H / 0.05916 \quad (3)$$

Sulfates are transformed to sulfides at an approximate E_H range of -0.075 to -0.150 V (Stumm and Morgan 1981, Mitsch and Gosselink 1993). Similar to sulfide emissions, variability

in E_H can be attributed to spatial heterogeneity in texture, structure, organic matter, aeration, and moisture (Megonigal et al. 1993).

11.3 Applications

When calibrated, measured, and interpreted properly, redox combined with other conventional water quality parameters is useful in developing a more complete understanding of water chemistry. Several applications of redox have been identified below:

1. "Red flag": conditions are too reduced to use Clarke-type dissolved oxygen (DO) probes without poisoning the silver anode. In general, anaerobic conditions occur at a redox range of +150 mV to +300 mV (pH-dependent and adjusted to hydrogen reference electrode). When redox drops below this level, DO as determined with a Clarke-type probe are highly suspect since the semi-permeable membrane does not discriminate between partial O_2 and sulfides. Consequently, the meter may be reading sulfides.
2. Redox could be viewed as an extension of the oxygen scale. Consequently, the DO probe spans the aerobic scale and the redox probe extends that scale to measure anaerobic conditions. Inferences to geochemistry and chemical speciation can be made from the oxidative state of the system. Application to metal sequestration, metal-iron, -sulfide, -methane complexation, and the subsequent bioaccumulation potential is possible;
3. Redox can be used to identify anaerobiosis at or near the water column and sediment interface in streams, lakes, and estuaries. In addition, based on the partial pressure of oxygen, a relationship can be developed between redox and the consumption of oxygen by anaerobic and even highly reducing sediments (anaerobic metabolism) in lakes, rivers, and estuaries;
4. Based on a redox, a pe (or E_H) vs. pH stability diagram can be developed to aid in nutrient exchange studies including the timing, release, and partitioning of important water and sediment quality pollutants such as nitrogen and phosphorus species. Most importantly, redox can be used to address error associated with chamber-effect during closed chamber measurements of the water-sediment interface. Redox probes placed inside the contact chamber and inserted approximately ten centimeters into the underlying sediment can be used to monitor changes in sediment redox caused by the chamber, and steps can be taken to reduce chamber-effect;
5. Redox is one of the two means of determining reducing conditions in hydric soils applicable to the Clean Water Act, Section 404, wetland delineation (Soil Survey Staff 1998);
6. Redox is critical in establishing water and sediment quality standards applicable to wetlands; and
7. Redox can be used to develop technical standards applicable to hydric soils across wetland hydrogeomorphic gradients (Pruitt 2001).

11.4 Calibration

Redox probes should be calibrated to quinhydrone ($C_6H_{10}OH_2$) or an equivalent redox standard (e.g., Light's solution, ZoBell's solution). Quinhydrone is used in this example. It must be prepared "fresh" prior to calibration. However, field preparation is as simple as adding pH buffered standards to a pre-weighed aliquot of quinhydrone and mixing thoroughly as follows:

1. Standards are made by mixing equal parts quinhydrone with predetermined pH buffered

standards based on anticipated *in-situ* redox conditions as follows*:

- a. pH 4 = redox standard +218.4 mV;
- b. pH 7 = redox standard +41.0 mV; and
- c. pH 10 = redox standard -36.5 mV.

*A three-point calibration curve can be developed by calibrating the redox probe to all three standards.

2. The standard is thoroughly mixed to reach saturation (a magnetic stirring table is recommended);
3. The resulting saturated solution is decanted into a suitable container and the redox probe and the reference probe are placed in the solution;
4. Once the reading is stable, the water, sediment, or soil quality instrument is calibrated; or
5. If a three-point calibration curve is desired, each of the three readings are recorded and a calibration curve is developed.

11.5 Interferences

Measurement of redox in water, sediment, and soil media is relatively simple, and in general, stable redox values can be observed in most natural settings. However, field personnel should exercise caution when interpreting redox data. Even though inferences can be made to the activity of redox couples, relative concentrations of these elements should be determined analytically. Several factors common in natural settings have been identified that may cause interferences with redox measurements including irreversible reactions, electrode poisoning, the presence of multiple redox couples, very small exchange currents, and inert redox couples (Standard Methods 1992).

11.6 Safety

Hydroquinone or quinhydrone (CAS# 106-34-3) is considered an irritant and is poisonous if ingested. Safety precautions when handling quinhydrone should include rubber gloves to prevent dermal contact and a mask to avoid inhaling dust particles. Unused hydroquinone/ calibration standards must be returned to SESD for proper disposal.

11.7 Calculations

Typically, redox is reported in millivolts (mV) as referenced to a hydrogen probe (E_H). However, the use of a hydrogen reference probe for field applications is problematic. Consequently, either a saturated calomel electrode (E_{SCE}) or a silver chloride (E_{AgCl}) reference probe is recommended for field applications. A saturated calomel electrode is used in the following example. Silver chloride reference probes would have a different correction factor. *In situ* redox (E_{SCE}) needs to be corrected in the following steps:

- 1) correct observed redox to the calibration curve specific to the electrode (if calibration curve is used):

$$\text{Corrected } E_{sce} = b + a (\text{Observed } E_{sce}) \quad (4)$$

where:

b = y-intercept; and

a = slope.

2) normalize to a predetermined pH (e.g., pH 5) for comparison between sample populations:

$$E_{\text{sce, pH5}} = \text{Corrected } E_{\text{sce}} + 59.16 (\text{Field pH} - 5) \quad (5)$$

and

3) correct to a hydrogen probe using the Nernst Equation and accounting for a calomel reference electrode and temperature (Pankow 1991):

$$E_{\text{H}} = E_{\text{sce, pH5}} + 0.268 - (2.303 \times RT)/2F \log \left(\frac{\{\text{Hg}_{(l)}\}^2 \{\text{Cl}^-\}^2}{\{\text{Hg}_2\text{Cl}_{2(s)}\}} \right) \quad (6)$$

At standard conditions, the correction from a E_{SCE} and E_{AgCl} is +244 mV and +222 mV, respectively.

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SECTION 12 TISSUE SAMPLING

PERFORMANCE OBJECTIVE:

- To collect a representative tissue sample from the surface water or area of interest.

12.1 Introduction

Tissue sampling techniques and equipment are designed to minimize effects on the chemical and physical integrity of the sample. The collection techniques described in this section should provide tissue samples that are suitable for use in ecological assessments, Total Maximum Daily Loads (TMDL) and other studies for which tissues are needed for chemical analysis.

The physical location of the investigator when collecting a sample may dictate the equipment to be used. If tissue samples are required from a wadeable waterbody, backpack electrofishing or seining is possible. Physical barriers could hinder the use of a seine net, making backpack electrofishing a more desirable technique. If the stream is too deep to wade, boat electrofishing, nets or hook and line techniques may be used to collect the sample. Current, velocity and conductivity will determine which technique is best. Because boat electrofishing is more time efficient and less destructive of the fish community, it is the preferred technique. However, if water at the sampling location has extremely high ($>1300 \mu\text{S}$) or low ($< 20 \mu\text{S}$) conductivity making electrofishing ineffective, then nets or hook and line techniques may be used to collect the sample.

12.2 Collection Techniques for Fish and Shellfish

12.2.1 Backpack Electrofishing

A sample may be collected using backpack electrofishing equipment when the surface water source is accessible by wading. All samplers must be familiar with safe electrofishing techniques including wearing chest waders and insulated gloves. A typical 24 volt backpack electrofisher must have an operating range for conductivity that allows collecting fish in the water source to be sampled. The electrofishing unit must also be capable of producing pulsed DC current. The electrofishing unit must be set on the lowest voltage setting that allows fish to be stunned without injury. The electrofishing unit must have a tip-over safety switch as well as other engineered safety features. A commercially built model of electrofisher is preferred due to the inherent safety features designed into the unit by the manufacturer. All members of the sampling crew be certified in CPR as well as First Aid. Consult the operating manual for specific instructions on electrofishing units. Scientific collection permits are required to collect fish.

The samplers should collect fish by moving in an upstream direction collecting targeted fish species from any available habitat. Fish stunned by the electrofisher are collected with dip nets and held in a bucket of water until euthanized on ice, or released if not needed for the study.

12.2.2 Seine Nets

A sample may also be collected using a seine net when the surface water source is accessible by wading. The samplers should seine in an upstream direction collecting targeted fish species from any available habitat. The selection of seine mesh size and length depends on the species being targeted and stream size. The mesh size of the seine must be small enough to capture the smallest targeted species. The length of the seine should be long enough to span the width of the water body being sampled without having extra material. In some cases net that exceeds the stream or cove width may be rolled around the seine pole/handle to keep it from getting in the way. The seine must be tall (deep) enough to keep fish from swimming over the top or under the bottom of the net. Typical seine dimensions are 10-20 feet long by 3 feet tall (deep) with a mesh opening of 3/8 of an inch.

Fish collected with a seine net are removed by the samplers wearing latex gloves and may be held in a bucket of water until euthanized on ice or released if not needed.

12.2.3 Boat Electrofishing

A sample may be collected using a boat electrofisher when the surface water source is not accessible by wading. A typical boat electrofisher must have an operating range for conductivity that will carry adequate current to stun fish in the water body to be sampled. The electrofishing unit must also be capable of producing pulsed DC current. The electrofishing unit should be set on the lowest voltage setting that allows fish to be stunned without injury. The electrofishing unit must have a positive activated foot power control switch as well as other engineered safety features. A commercially built electrofisher model is preferred to a home-made device due to the inherent safety features designed into the unit by the manufacturer. All samplers must be familiar with safe electrofishing techniques including using net poles made of an insulating material such as fiberglass, wearing personal flotation devices, and wearing insulated lineman gloves. All members of the sampling crew should be certified in CPR as well as First Aid. See operating manual for the specific make and model of electrofishing unit being used for specific information on how to operate it. Scientific collection permits are required when collecting fish.

Fish that are stunned by the electrofisher are collected with dip nets, placed in a clean plastic bag and euthanized on wet ice.

12.2.4 Gill Nets

When fish samples are desired from a water source where conductivity is too high or too low to electrofish or where multiple targeted fish species are needed the use of gill nets is possible. Unlike electrofishing gill nets to a certain extent are non selective in the capture of fish. A mesh size must be selected that allows for the capture of the targeted fish species while minimizing the catch of unwanted species. Placement of gill nets in the waterbody should optimize the chances of collecting the targeted species of fish therefore knowledge of fish habitats and behavior is critical to collecting the desired sample. Gill nets must be checked once per hour when they are deployed to minimize the capture of unwanted species. Gill nets should not be placed in a manner which would cause entanglement in boat propellers or hinder boat traffic. Samplers should always be within sight of gill nets deployed. Fish are removed from the gill net by samplers wearing latex gloves.

When fish are removed from the net, they are euthanized (if not already dead) in a clean plastic bag on wet ice.

12.2.5 Hook and Line

When fish samples are desired from a water source where electrofishing and gill nets are unsuitable, hook and line angling can be used to collect a sample. Most standard angling gear is suitable for collecting fish in this manner. Knowledge of fish habitats and behavior is critical to collecting the desired sample. If live bait is used to catch fish an aliquot of the bait being used must be analyzed for the same contaminants as the fish tissue samples. Fish caught with hook and line techniques must be handled by samplers wearing latex gloves.

12.2.6 Shellfish (crabs, shrimp, etc.) Collection Using Cast Nets

A standard cast net is suitable for collection of shrimp or crab from a water body. Knowledge of shellfish habitats and behavior is critical to collecting the desired sample. Net mesh size must be small enough to collect targeted species. A typical cast net should be about as tall as the sampler throwing it and have a mesh opening of 3/8 of an inch. Scientific collection permits are required to collect shellfish. A valid state fishing license may also be required.

12.2.7 Crab Collection Using Traps

Traps are usually the best method to collect crabs. Knowledge of crab behavior and habitat preferences improves the likelihood of obtaining the necessary sample. A typical trap can be baited with dead fish, deployed and left undisturbed for a number of hours to provide sufficient time for targeted species to enter the trap. Traps should be checked approximately every 12 hours to minimize overcrowding of the crabs and resultant cannibalization, or trap loss from tidal changes. Traps should have an attached buoy identifying it as U.S. EPA equipment. Scientific collection permits are required to collect crabs for field studies. A valid state fishing license may also be required.

12.3 Tissue Sampling of Small Mammals Posing a Human Health Risk Due to Hantavirus

12.3.1 Introduction

Because of the high morbidity and mortality associated with hantavirus pulmonary syndrome (HPS) and the possibility of aerosol transmission, special precautions will be taken to reduce the risk of infection. Transmission of the virus from rodent to human occurs by inhalation of virus present in aerosols of urine, feces, or saliva of infected rodents. Transmission also may occur when broken skin, mucous membranes, or the conjunctiva of the eye come into direct contact with materials containing the virus (Kunz et al. 1996). The onset of HPS symptoms occurs within 45 days after exposure. Symptoms include fever, headache, muscle ache, fatigue, vomiting, shortness of breath, and a dry cough. Later symptoms include rapid heartbeat and difficult breathing, which can lead to respiratory failure (Kunz et al. 1996).

Sin Nombre virus (SNV) is the most common hantavirus in North America; the deer mouse (*Peromyscus maniculatus*) is the primary reservoir for SNV throughout much of the continent (Mills et al. 1995). Other species of Cricetid rodents host other hantaviruses. There is an apparent one-to-one correspondence between virus types and rodent species. Thus, Sin Nombre is restricted to *P.*

maniculatus, New York to *P. leucopus*, Prospect Hill to *Microtus pennsylvanicus*, Bayou to *Oryzomys palustris*, Black Creek Canal to *Sigmodon hispidus*, and so on. Rodent species will be competent hosts insofar as their own virus type is concerned, but not for other viruses. Species in other families of rodents, as well as non-rodent animals, are dead-end hosts. Hispid cotton rats (*Sigmodon hispidus*) are responsible for at least one case of HPS in Florida (Rollin et al. 1995), while rice rats (*Oryzomys palustris*) have been linked to HPS cases in Louisiana and eastern Texas (Mills et al. 1995). The Prospect Hill hantavirus has not been associated with human disease (Mills et al. 1995). Norway rats (*Rattus norvegicus*) are responsible for the transmission of Seoul virus, which causes mild hemorrhagic fever and renal syndrome, primarily in Asia (Glass et al. 1993, Childs et al. 1995).

12.3.2 Setting Traps

Small mammals will be captured using transects of pitfall traps, with interception fencing, and grids of snap-traps or live traps. Size and location of transects and grids will be determined in the field according to standard practices in vertebrate ecology (Markham 1978, Smith 1980, Wilson 1996).

12.3.3 Checking and Collecting Set Traps

Persons checking or retrieving traps will wear protective clothing including long sleeves, long pants, lace-up shoes, rubber gloves, and a surgical mask. When a closed live trap is encountered, workers should lift it without shaking it and hold the trap at arms length with the wind to the left or right side. Occupancy may be assessed by “hand weighing” the trap. Traps that have caught rodents will be placed in zipper closure plastic bags on wet ice and returned expeditiously to the processing site. After handling occupied traps, gloves will be disposed of as prescribed below. Workers will wash their hands thoroughly with soap and water as soon as practicable after removing gloves.

12.3.4 Handling Rodents and Traps that Have Caught Rodents

Rodents will be processed in an area away from other humans or domestic animals. Work surfaces will be covered with a non-porous material that will be rolled up and disposed of into a biohazardous waste bag during clean-up. Outdoor processing is preferred because of increased ventilation and the presence of UV light. Workers will sit with the wind coming from behind them at a 45-degree angle, with occupied traps downwind and any other workers, vehicles, or equipment upwind. If it is necessary to process animals indoors, there should be adequate means of ventilating the area to the outdoors (such as a hood). Workers will wear the protective clothing described above, with addition of a disposable surgical gown. Anyone handling live animals will wear thick gloves to protect against rodent bites. Traps that have caught rodents will be placed in zipper closure plastic bags until they are decontaminated. Any bedding, bait or feces will be treated as biohazardous waste and incinerated prior to disposal.

Standard measurements (DeBlase and Martin 1981) will be obtained from captured animals. Specimens will then be frozen in doubled zipper closure plastic bags until further processing in the laboratory.

12.3.5 Clean-up

After animals have been processed, all used equipment will be cleaned thoroughly with disinfectant. Traps should be placed into a plastic bucket containing disinfectant for approximately 10 minutes after the animal is removed. Snap traps floating on the surface of the disinfectant will be turned upside down. The disinfectant will be one labeled by the manufacturer as capable of killing viruses. Bleach is not recommended because it may corrode metal traps. Dirt, blood, and fecal material will be removed with a long-handled brush while the trap is submerged in disinfectant. Disinfected traps will be rinsed in two successive container rinses of clean tap water. Heavy arm-length rubber gloves should be worn over latex gloves while handling traps. Any disposable gloves or other contaminated trash, such as surgical wear, will be placed into biohazard bags and disposed of in accordance with standard procedures (USEPA 2001). If animals are processed indoors, the area will be ventilated for at least 30 minutes before personnel can enter without a surgical mask. Upon completion of clean-up, hands will be thoroughly washed with soap and water after any protective clothing is removed.

All other traps placed on CERCLA/RCRA sites will be decontaminated according to standard procedures (USEPA 1996).

12.4 Tissue Sampling of Herpetiles that Pose a Human Health Risk Due to Poisonous Bites

12.4.1 Introduction

Reptiles and amphibians (herpetiles) should be collected in the same general areas where soil or sediment is collected as part of a comprehensive investigation. No individual will be collected whose apparent home range, as indicated by its current position, does not overlap the area where soil and sediment are collected.

12.4.2 Capture

Herpetiles will be collected by any legal, practicable means that do not pierce the body. A variety of techniques and devices will be available and will be employed according to the judgement of experienced members of the field team. Species identifications will be done in the field. Animals will be handled by standard methods employed by professional herpetologists. Venomous snakes will be collected in accordance with customary professional practice (Conant 1975). Only trained personnel will handle them, using specialized equipment. Venomous snakes will be bagged and tied separately, with orange flagging tied on to mark the bag. Bags will be handled with leather gloves.

12.4.3 Processing

Venomous snakes will be chilled on wet ice and then sacrificed by smothering in a cooler with dry ice. All other herpetiles will only be chilled on wet ice. All specimens will then be weighed and measured, double-bagged in self-sealing plastic bags, and frozen in a portable freezer.

12.5 Sampling identification, preservation and transfer of custody

Fish or crabs must be handled and/or removed from the traps or nets by samplers wearing latex gloves. Samples collected from a site must be placed in a plastic bag set on wet ice in a clean cooler until relinquished to the sample custodian. The plastic bag must be tied closed and identified with a tag indicating the date of collection, station number, and species collected. An entry will be made on a chain-of-custody sheet indicating how many fish were collected at each station, and the time and date collected. Samples of fish and shellfish collected at the same site must be kept in separate, labeled bags. Samples collected at different sites must also be bagged and tagged separately. Tissue samples (fish filets or muscle from crabs) must be removed from fish or crabs within 48 hours of their collection, and frozen prior to further processing.

12.6 References

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SECTION 13 BENTHIC MACROINVERTEBRATES

PERFORMANCE OBJECTIVE:

- To collect a representative sample of the benthic macroinvertebrates.

13.1 Introduction

Benthic macroinvertebrates are an excellent tool for detecting stress in aquatic ecosystems. Due to their limited mobility and relatively long life span, benthic macroinvertebrates integrate and reflect water quality effects over time. State and federal water quality agencies for some time have employed trained and experienced benthic ecologists. This has led to considerable background data on benthic macroinvertebrates. In addition, sampling methodology has been refined and published in national guidance documents (EPA/440/4-89-001 and EPA 841-B-99-002). All these factors, in addition to the fact that U.S. EPA, Region 4 maintains a close rapport with its state partners, has led to consistency in benthic macroinvertebrate sampling methodology.

13.2 Benthic Macroinvertebrate Sampling

13.2.1 Equipment and Supplies

- Quantitative samplers such as Ponar, Peterson, Young, Ekman dredges; coring devices; Surber or square foot bottom samplers; artificial substrates (multiplate or rock-filled baskets)
- Qualitative samplers such as rakes, dip nets, sieves and drift nets
- Tub, sieve buckets, waste buckets
- U.S. Standard No. 30 Sieve (U.S. Standard No. 35 for marine waters)
- Soft brush
- Tweezers
- Wash bottle
- Wide mouth jars in crates
- White enamel pans, gridded
- Tablespoon
- Vials, vial holder
- Taxonomic keys
- Labels and tags
- Global Positioning System (GPS)
- Field record book
- Waterproof pen/pencils
- Benthic macroinvertebrate field data sheets
- Habitat assessment field data sheets
- Physical characterization/water quality field data sheets
- Chest waders/hip boots
- Rubber gloves

- First aid kit
- 90% ethanol

Further reference on equipment and supplies is provided in *Rapid Bioassessment Protocols for Use in Wadeable Streams and Rivers.*, 2nd Edition 1999.

13.2.2 Field Sampling Procedures: Freshwater

Selection of sampling stations will vary depending on the scope of the project and study objectives. Sample site location guidance is provided in Standard Methods, Part 10500-B (1998) and EPA 841-B-99-002, Chapter 7.

Detailed guidance of quantitative and qualitative sampling approaches is provided in *Macroinvertebrate Field and Laboratory Methods for Evaluating the Biological Integrity of Surface Water*, 1990. The specific method for benthic macroinvertebrate collection is dictated by the water body and study objectives.

13.2.3 Wadeable streams and rivers: Freshwater

The method of choice utilized by U.S. EPA, Region 4 for freshwater wadeable streams and rivers involves multi-habitat sampling. This approach is modified from *Rapid Bioassessment Protocols for Use in Wadeable Streams and Rivers.*, 1989, and is consistent with methodology adopted by states within Region 4. Generally, this qualitative sampling effort shall encompass a minimum of one work-hour per sampling site and shall include the following habitats:

- riffles - 3 kicknet samples from faster current, 3 kicknet samples from slower current,
- snags/woody debris - collect 5-6 pieces (snags, limbs, submerged logs) and wash in the sieve bucket to dislodge organisms,
- Coarse Particulate Organic Material (CPOM) - collect leaf packs equivalent to one half dip net (approximately 2-3 handfuls),
- undercut banks - 6 “jabs” with D-frame dip net (a “jab” equals a one meter thrust or sweep with the net), and
- bottom substrate - 3 sweeps or kicks (disturb sediment to 3 cm. depth).

Collected material shall be “coarse sorted” in the field, utilizing a white enamel pan, to remove larger sticks, leaves, and rocks in order to keep the sample size manageable and also assure adequate sample preservation.

13.2.4 Non-wadeable streams and rivers: Freshwater

In non-wadeable freshwater streams and rivers, quantitative grab samplers (Ponar, Peterson, or Ekman dredges) are appropriate sampling devices and may be used where the bottom substrate is predominantly sand. Diver-deployed coring devices are also suitable for this type of bottom substrate. A minimum of three replicates, as dictated by Standard Methods, Part 10500 B (1998), shall be collected from bottom substrates. In addition to the grab sampling devices and/or diver-deployed cores, artificial substrate samplers offer another alternative for sampling non-wadeable streams and rivers. Further guidance on these sampling approaches for non-wadeable streams and rivers is provided in *Macroinvertebrate Field and Laboratory Methods for Evaluating the Biological Integrity of Surface Water*, 1990.

13.2.5 Preservation of Benthic Macroinvertebrate Samples

Samples collected for benthic macroinvertebrates should be adequately sieved through a U.S. Standard No. 30 sieve or equivalent mesh to remove unnecessary silt and clays. To preserve benthic macroinvertebrates, 90% ethanol shall be used. Wide-mouth plastic containers (one quart size) shall be used to store benthic macroinvertebrate samples; containers should not be filled more than half full with collected material. If collected material exceeds one half container, sample should be split in two separate containers. All sample collections shall be labelled (inside and outside) with appropriate station information (station number, project name, date, type sample).

13.2.6 Marine Macroinvertebrate Collection

Benthic macroinvertebrate collection in the marine or estuarine environment is usually collected by divers using screen coring devices to ensure sample integrity. Sample replication is based on the species saturation curve; generally, fifteen replicates per station are collected. For QA purposes, thirty replicates will be taken from one or two stations in order to verify sample replication size via the species saturation curve. Each replicate is collected with a stainless steel corer measuring 10 cm in diameter and screened at the top with 0.5mm wire mesh. Core penetration is limited to 15 cm or the point of refusal if less than 15 cm. Each core is capped in place, secured into cloth bags, and returned to the dive vessel. On board processing involves washing the bagged core sample contents through a #35 screened (0.5 mm) sieve bucket with raw sea water. The sample retained on the screen after washing is then placed in a sample bag, properly labeled, and placed in a five gallon bucket containing a 10% seawater formalin with Rose Bengal staining solution. Sample bags and buckets are labeled both internally and externally and stored for transfer to contract lab facilities for taxonomic identification.

In some instances where the number of stations are too numerous or sea state conditions preclude the collection by divers, a .04 meter square Young Grab is utilized to sample sediments from aboard a ship or small boat equipped with a davit or "A" frame for raising and lowering the grab. At least three replicates need to be taken at a site in order to cover the approximate surface area of fifteen 10 cm cores. Once the sample is aboard the vessel handling procedures are identical to diver collected samples.

13.3 Quality Control/Quality Assurance

Guidelines for field collection of benthic macroinvertebrates shall adhere to that presented in Section 2, Quality Assurance and Quality Control, of EPA/600/4-90-030.

13.4 References

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SECTION 14 HYDROLOGICAL STUDIES

PERFORMANCE OBJECTIVES:

- To outline standard practices for obtaining hydrological data during water quality surveys;
- To present acceptable flow measurement techniques;
- To present general and specific quality assurance procedures for hydrological/flow measurement equipment and techniques.

14.1 Scope and Applicability

Hydrological studies are an important component of virtually all Branch field studies and include activities such as time-of-travel studies, current/circulation studies, dye dilution surveys, flow measurement and stage/discharge relationship development. Time-of travel surveys are frequently required as part of water quality model calibration surveys or as a component of other activities such as reaeration measurements. Dye dilution studies are extremely useful for evaluating the mixing of effluents with receiving waters. Activities such as water quality enforcement studies, NPDES permit compliance monitoring, water quality survey monitoring, reconnaissance surveys, and research rely on accurate flow measurement. For example, NPDES permit limits often limit the mass loading of a particular pollutant that may be discharged. Stage measurement and the determination of stage-discharge relationships are also important hydrological data collected by the Branch. For example, stage-discharge studies are extremely useful for determining flow in conjunction with TMDL storm event sampling efforts. As much attention and care should be given to hydrological measurements in the design of a sampling program as to the collection of samples and subsequent laboratory analysis.

14.2 Summary of Method

14.2.1 Surface Water Stage/Tape Down

Water level recorders provide a time series record of water levels. When necessary, these instruments should be referenced to National Geodetic Vertical Datum (NGVD). All notes on water level tracings should include beginning and ending date and time; site location; stage scale; time scale; and the name of the field investigators responsible for the data. Standard USGS staff gages should be employed at each water level recorder site to provide a reference and check on the recorder trace. Water stage should be recorded to the nearest 0.01 foot where possible.

Tape downs provide instantaneous water stage as referenced to a known elevation. An engineering tape is fashioned with a plumb bob to measure from a bridge deck or other reference point to the water surface. The plumb bob provides weight for the tape as well as providing a discernible contact with the water surface. All measurements should be to the nearest 0.01 foot accompanied by a date, time, and station location. The exact reference or point from which a tape down is measured should be permanently marked on the reference (wing wall or bridge rail by

etching a reference with a chisel, etc.) and a complete description of the reference should be made in the field records. Photographs are also helpful for referencing a site.

Both of these procedures (water stage and tape downs) are predicated upon accurate references to established measuring points. As mentioned above, the NGVD is an established datum that provides correlation of water surface recordings to engineering structures (bridge, wing walls, sea wall caps, clarifier cat walks, etc.). When recording water level dynamics in relation to a particular flow device, the datum is established in relation to the flow device reference point. The flow through rectangular and V-notch weirs, for instance, are proportional to the water level referenced to the weir crest or, in the case of partially filled pipes, the flow rate is proportional to the depth of flow. Therefore, when employing a water level recorder or tape down on primary flow devices, the reference or datum is the weir crest or in the case of pipes, the invert.

14.2.2 Time-of-Travel

Three principal methods are used to determine time-of-water-travel in streams: surface floats, measurements of cross sectional velocity, and tracers such as dye.

A very rough method for preliminary estimates of time-of-water-travel consists of dropping sticks or other buoyant objects in the stream reach under observation, and noting the time required for them to float an estimated 10 feet or some other convenient distance. The velocity estimates are too inaccurate for use in interpretation of data or final reporting, but can be useful in preliminary planning of studies and in subsequent more precise measurements of time-of-water-travel.

Stream velocities at gaging stations, measured by the U. S. Geological Survey in developing rating curves, may be applied to the entire reach under observation to estimate time-of-water travel. This is somewhat more refined than the floating objects estimates, but can still be far from accurate. There rarely are more than one or two gaging stations in most stream reaches being studied. Stream channels generally are restricted at gaging stations and velocities there are generally higher than average velocities throughout the reach. Cross sectional velocities can also be determined at locations designated for a particular study.

Tracer dyes provide a direct and highly accurate method of determining time-of-travel. This is the preferred method if resources are available.

Floats

Surface floats may be followed downstream and timed for known distances to determine time-of-water-travel. This requires the use of considerable judgment as floats tend to travel into quiet or eddy areas, or become stuck on tree limbs, the stream bank, or other obstacles. The floats must frequently be retrieved and returned to the stream current. The principal judgment factors are how long the floats should be left in quiet areas before retrieval and where they should be placed in the current.

Surface water velocity is greater than the average for the entire stream, and a correction factor must be applied to the surface velocity. An average velocity of about 85 percent of that of the surface velocity is a reasonable rule-of-thumb value.

Cross Section Measurements

The measurement of cross sectional velocities at frequent longitudinal intervals and the calculation of average velocity in the stream constitutes a time consuming method of obtaining time-of-water-travel.

The longitudinal intervals at which cross sections should be measured vary with the characteristics of the stream channel. One cross section per mile may be adequate for streams with reasonably uniform channels. Cross sections at every tenth of a mile may be desirable for streams with irregular channels.

Tracers

The most accurate method of measuring time-of-travel involves following and measuring a tracer. Some conservative constituents such as salt, or radioisotopes may serve as tracers; however, Rhodamine WT dye is the most common tracer used. Rhodamine WT dye is water soluble and can be detected at concentrations as low as 0.01 ppb by a fluorometer.

Prior to injection into the stream, the concentrated dye is often diluted with stream water. This insures immediate maximum dispersion. Addition of concentrated dye without dilution may result in incomplete dispersion, particularly in shallow streams.

The dye should be distributed across the stream at the upstream point, as nearly instantaneously as possible. The ideal distribution produces a narrow band of tracer in a uniform concentration across the stream. The band of tracer mixes with water ahead of and behind it by diffusion, or longitudinal mixing, as it moves downstream to produce an increasingly wider band. The peak concentration remains near, but somewhat downstream of, the center line of the band and decreases as longitudinal mixing proceeds. The times-of-water-travel to downstream points are the differences between the time the dye was added to the stream and the times the centroid of the dye mass arrives at downstream points. The length of the dye cloud and the peak concentrations produces a measure of in stream dispersion.

If Rhodamine WT dye is used as the tracer, peak concentrations from 1.0 to 50 ppb allow satisfactory definition of the dye concentration curve.

Most methods of calculating the dosage of dye needed at the upstream point involve estimates of one or more stream characteristics, such as flow, velocity, length of reach, volume in the reach, cross-sectional area, average depth, or the roughness coefficient "n" of Manning's formula. The USGS has produced excellent publications regarding time-of-travel techniques such as "Measurement of Time of Travel in Streams by Dye Tracing" (See Section 14.7, Reference #7) and "Fluorometric Procedures for Dye Tracing" (Sect. 14.7, Ref. #5).

The stream should be sampled frequently as the dye arrives at the downstream point to define the tracer concentration versus time curve with special emphasis on the peak. The frequency may be varied from continuously to every 60 minutes or more depending on the duration of the dye cloud at the sampling point. The dye may be missed altogether by overestimating the time required for it to travel downstream. Much time may be wasted, on the other hand, waiting for it to arrive if the time-of-travel is underestimated. All information that will contribute to the best possible preliminary estimate of the time required should be used.

There are two primary methods by which the stream water can be sampled and analyzed for dye. A submersible pump can be used to pump the dye continuously through a fluorometer or the

stream samples can be grabbed (either by hand or by automatic sampler) at specified frequencies and then placed into the fluorometer individually. Readings directly from the fluorometer scale or conversion to dye concentration corrected for temperature can be manually plotted against time.

A version of the grab sampling technique is to use an automatic water sequential sampler which discharges into separate bottles. The samples collected at preset intervals are analyzed and the concentrations plotted against time.

Ideally, dye samples should continue to be analyzed until the stream background concentration following the peak is measured. With a time versus concentration plot from background level to peak to background level, the centroid, and thus actual travel time, can be determined. Where it is infeasible to continue monitoring to the stream background concentration, the trailing edge of the dye cloud should at least be monitored until the in stream tracer concentration is no more than 2 to 5 % of the peak concentration.

Prior to conducting tracer studies in freshwater systems, water supplies should be inventoried to insure that the dye tracer concentrations will not impart color to downstream public or private water supplies. Rhodamine WT concentrations in the dye cloud should be maintained below 10 ppb at water supply intakes. Commercially available Rhodamine WT is a 20% solution.

14.2.3 Dilution

A great deal of the previous section (time-of-travel studies) applies to this section and USGS publications provide references to appropriate techniques, in particular "Measurement of Discharge by Using Tracers" (Sect.14.7, Ref. #6).

Dilution studies using tracer dyes evolved from "mass conservation" principles, i.e., a known mass of tracer is introduced at an upstream point, and after mixing with the water to be traced, this mass should be accountable at downstream locations. Rhodamine WT provides an adequate tracer for most investigations. This dye is slightly photoreactive. Decay rates (e^{kt} where $k=0.034/\text{day}$ for exposure to full sunlight) are reported in the literature. Due to limited light penetration, actual rates are normally insignificant or can be established through on-site bottle tests. Other tracers either introduced into an upstream point or in some instances occurring at the upstream point are often used. The high degree of accuracy and detection ability of fluorometers plus the solubility properties of tracer dyes make them the technique of choice.

In dilution studies, the tracer dye is precisely metered into the waters to be traced and then monitored after mixing via a fluorometer at downstream stations. This series of events requires highly controlled metering rates and very accurate fluorometric analyses. State-of-the-art fluorometers make the dilution study methods valuable assessment tools.

The principal of superposition as developed by Kilpatrick et al. (Sect. 14.7, Ref. # 8) of the USGS is a reliable method to determine dilution levels of wastewaters in receiving estuaries. A tracer dye is metered into the wastewater stream during a tidal cycle. Successive slack tide measurements of dye concentrations in the estuary at selective distances from the outfall produce a series of concentration curves. By superposition, the accumulative concentration at each station provides a determination of the ultimate concentrations or steady-state concentration of a continuous discharge. By simple proportioning, with due regard to any tracer photo decay, the dilution levels of the discharge can be produced for selective points in the estuary.

Calculation Procedure:

$$C_w = \frac{(C_d)(e^{-kt})(V_w)}{V_d}$$

Where:

- C_w = Ultimate concentration of wastewater at point of interest
- C_d = Ultimate concentration of dye tracer (by superposition) at point of interest
- e^{-kt} = Photo decay of tracer
- V_d = Wastewater discharge per tidal day
- V_t = Volume dye tracer released in tidal day

Investigations of industrial and municipal facilities for NPDES permit compliance require measurements of discharge rates. Often encountered during these investigations are flow measuring devices such as orifices and magnetic meters which are inaccessible for measurements of flow by standard equations relating to hydraulic head and structure size. The following provides a direct technique for measurement of flow through these devices using dye tracers.

Calculation:

The discharge rate through any structure can be defined by the following mass balance equation:

MASS BALANCE EQUATION

$$(C_1) (q_1) = (C_2) (Q_2 + q_1)$$

then, $Q_2 = \{(C_1) (q_1) - (C_2) (q_1)\} / C_2$

Where:

Q_2 = flow rate in pipe

C_2 = tracer concentration after mixing

q_1 = tracer injection rate

C_1 = tracer injection concentration

Assuming a constant discharge rate and complete mixing of the tracer in the waste stream, the task is (1) to inject into the waste stream a tracer at a constant rate and constant concentration and (2) to measure the concentration of the tracer after mixing with the waste stream.

It is suggested that at least three injection rates and resulting mixed tracer concentration measurements be used to calculate the discharge rate.

EPA's Technical Support Document also provides guidance for conducting a "quick saltwater dilution assessment" using a dye tracer (Sect. 14.7, Ref. #10).

14.2.4 Surface Water Flow

Surface waters are considered to be open channels for flow measurement purposes. Where installation of a primary flow device is practical, open channel flow measurement shall adhere to EISOPQAM, October 2001. Where installation of a primary flow device is not practical, flow measurements shall be made using stream gaging or acoustic doppler techniques.

Stream Gaging

Where practical, flow data and/or rating curves shall be obtained from existing permanent stream gaging stations maintained by the USGS, Army Corps of Engineers, or other federal or state agency. Where permanent stations do not exist, flow may be measured using stream gaging techniques. In making stream gaging measurements, Branch personnel shall utilize the procedures outlined in the USGS publication *Discharge Measurements at Gaging Stations* (Sect. 14.7, Ref. #4) to (1) select the flow measurement site, (2) perform stream gaging, and (3) calculate flow. If a station is to be used more than one time during a water quality survey, a rating curve may be developed for that station. A rating curve is constructed by making a series of independent flow measurements and simultaneous tape down or staff gage measurements for the same section of a particular station at different water levels and plotting the resulting data pairs on a semi-log graph. At least two (preferably three) flow measurement-tape downs shall be made to develop a rating curve.

Available current meters for conducting stream gaging include vertical-axis mounted Price AA and Price pygmy meters. For wadeable streams, these meters may be deployed using a top-setting wading rod. For non-wadeable streams, a Price AA meter may be deployed on a weighted line using a bridge rig system. Depth may be determined using Raytheon or Lowrance fathometers, taken from a standard top setting wading rod, or by taking the difference of tape down measurements of the river bottom and surface. Incremental width measurements as part of stream gaging may be made using a Lee-Au galvanized steel tag line segmented into equal lengths, steel tapes, or cloth tapes. Measurement of total width may be made using these systems or a laser range finder.

ADCP

Measuring flow by Acoustic Doppler Current Profiler (ADCP) is a fairly new method of flow measurement relative to stream gaging. ADCP meters used in flow measurements may be deployed via a boat-mounted configuration for larger river/estuarine systems or mounted on a tethered float for smaller stream measurement applications. ADCP meters may also be used to determine water velocity, depth, and width in conjunction with a laser range finder or other means for determining edge distances. Branch personnel measuring flow by ADCP will follow instrument manufacturer's specifications. In addition, the USGS publication *Discharge-Measurement System Using an Acoustic Doppler Current Profiler with Applications to Large River and Estuaries* (Sect. 14.7, Ref. #9) provides guidance in the use of ADCP for flow measurement.

14.2.5 Wastewater Flow Measurement

Basic guidance for making wastewater flow measurements and a basic description of all acceptable wastewater flow measurement systems are given in the *NPDES Compliance Inspection Manual* (Sect. 14.7, Ref. #2). This manual shall be used as Branch guidance for such measurements. The USDA Water Measurement Manual (Sect. 14.7, Ref. #1) is an acceptable reference for procedures for checking the installation of primary open channel flow devices. Field procedures for measuring wastewater flow will follow the Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, 2001, Section 18.2.

14.2.6 Current Measurement

Current measurements may be made by either an axial-flow, ducted impeller recording current meter (Endeco 174) or an Acoustic Doppler Current Profiler (ADCP). Both meter types may be programmed via appropriate connection to a PC prior to deployment, deployed in an unattended mode, and interrogated for data download by PC following data collection. Likewise, the meters may be used for real-time data collection in profiling applications. For unattended applications, deployment in and out times/dates should be recorded in the field record. The field record should also include the location and depth(s) of the deployment and serial number or other appropriate identifier of the meter(s) used in the deployment.

For unattended applications, the impeller-type meters are deployed on a weighted tether line with a subsurface float (to keep the tether line taut) and a surface float (for locating the meter). Multiple meters can be deployed at any depth(s) on the tether. For a non-stratified system, one meter will generally be deployed at mid-depth. For a stratified system, a meter will generally be deployed at the mid-depth of each strata. Anticipated deployment depths for a given application should be provided in the Quality Assurance Project Plan.

Care should be taken during deployment of the impeller-type meters to prevent tangling of the tether line around the meter and/or floats. The meter should also be checked for level deployment in the water. Lead weights attached at the nose and tail ends of the meter should be added or removed as needed to ensure the meter is axially deployed (horizontal) in the water column. Meters should be deployed in a way that minimizes potential equipment damage or interference from ship traffic or other obstructions.

ADCP meters may be mounted in a variety of ways; however, for unattended current measurements, these meters will typically be deployed in upward facing configuration with the meter mounted to a weighted platform specifically designed to minimize potential impacts from drag lines or nets. The meter may also be boat mounted in a downward facing configuration for real-time data collection and profiling.

14.3 Definitions

ADCP - Acoustic Doppler Current Profiler
NPDES - National Pollutant Discharge Elimination System
USGS - United States Geological Survey

14.4 Equipment

The following equipment is available for surface water stage/tape down measurements:

- Model F Stevens Stage Recorder(s) (mechanical, horizontal drum system);
- Model A-71 Stevens Stage Recorder(s) (mechanical drum system);
- Stevens Model GS-93 Endcoders and Loggers;
- Stevens AxSys System (pressure transducer);
- Model 1870, 2870, 3210, 3230, and 4220 ISCO flow meter(s) and Recorder(s) (pressure transducer, bubbler, and ultrasonic reflection systems)
- USGS staff gage(s); and
- Weighted steel tape-down systems

The following equipment is available for time-of-travel and dilution studies:

- Turner 10-AU and Turner 10-005 fluorometers,
- Rhodamine WT dye tracer and standards,
- ISCO automatic samplers,
- Peristaltic, submersible, and metering pumps,
- recorders,
- flow meters, and
- floats

The following equipment is available for flow/current measurement:

- cup-type current meters (Pygmy/Price),
- impeller-type current meters (Endeco),
- sounding (depth) equipment, and
- ADCP

14.5 General Quality Assurance Procedures

No field investigator shall make flow measurements until they have had at least six months of actual field experience and have performed these measurements under the supervision of a senior field investigator.

Wastewater flow shall be expressed in million gallons per day (MGD) or the metric equivalent (m^3/day). Stream flow shall be expressed in cubic feet per second (cfs) or the metric equivalent (m^3/sec). Current velocities shall be expressed in feet per second (fps) or the metric equivalent (m/sec). Time records associated with hydrological studies shall be (1) kept in local time, (2) recorded in 24 hour military format, and (3) recorded to at least the nearest five minutes.

All field equipment shall be operated, calibrated, and maintained according to manufacturer's specifications. All equipment shall be visually inspected prior to deployment to ensure proper operation.

14.6 Data/Records Management

All hydrological measurements shall be thoroughly documented in field records. All measurements shall be traceable to the personnel making the measurements and the equipment utilized.

14.7 References

1. *Water Measurement Manual*, Second Edition, Revised, United States Department of Interior,

- Bureau of Reclamation, 1981.
2. *NPDES Compliance Inspection Manual*, United States Environmental Protection Agency, September 1984.
 3. *Stevens Water Resources Data Book*, Third Edition, Leopold Stevens, Inc. Beaverton, Oregon, 1978.
 4. “Discharge Measurement at Gaging Stations”, *Applications of Hydraulics*, Book 3, Chapter A8, United States Department of Interior, Geological Survey, 1969.
 5. “Fluorometric Procedures for Dye Tracing”, *Applications of Hydraulics*, Book 3, Chapter A12, United States Department of Interior, Geologic Survey, Revised, 1986.
 6. “Measurement of Discharge Using Tracers”, *Applications of Hydraulics*, Book 3, Chapter A16, United States Department of Interior, Geologic Survey, 1985.
 7. “Measurement of Time of Travel in Streams by Dye Tracing”, *Applications of Hydraulics*, Book 3, Chapter A9, United States Department of Interior, Geologic Survey, 1989.
 8. *Simulation of Soluble Waters Transport and Buildup in Surface Waters Using Tracers*, United States Geological Survey, Open File Report 92-457, 1992.
 9. *Discharge-Measurement System Using an Acoustic Doppler Current Profiler with Applications to Large Rivers and Estuaries*, Water Supply Paper 2395, United States Geological Survey, 1993.
 10. *Technical Support Document for Water Quality-based Toxics Control*, US EPA, Office of Water, EPA/505/2-90-001, March 1991.
 11. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, 2001.

SECTION 15 OXYGEN DYNAMIC STUDIES

15.1 Introduction

Dissolved oxygen is one of the most, if not the most important, parameter of focus for water quality investigations and water quality standards. Adequate dissolved oxygen concentrations are essential to sustain viable and diverse aquatic biological communities. The importance of this parameter to the chemical and biological processes, community diversity and structure, and water quality model sensitivity dictates that the rates that dissolved oxygen is produced and consumed in the aquatic environment be accurately measured to the extent that present state of the art methods will permit. Oxygen budget assessments have been at the point of EAB activities for over twenty five years and present and former EAB staff are recognized nationally and internationally for the development and/or refinement of methods for partitioning the rates and coefficients that oxygen is consumed, produced, and accrued in the aquatic environment. When the physical, biological, and chemical processes that produce and consume oxygen are perturbed by insitu as well as allochthonous activities, the oxygen budget becomes out of balance and water quality and associated resources are adversely affected and water quality standards may be violated. This section presents methods utilized by EAB scientists in the assessment of oxygen dynamics. Included is the assessment of total community oxygen metabolism, which represents the total available oxygen “bank account”, followed by methods which are aimed at partitioning the various oxygen debits and credits along with other parameters which strongly influence oxygen production and respiration.

15.2 Total Community Oxygen Metabolism (Diel Curve Method)

SECTION OBJECTIVE:

- To provide guidance for the determination of total community oxygen Gross Primary Production (GPP) and Respiration (R) rates in water bodies.

15.2.1 Introduction

Total Community Oxygen Metabolism involves the assessment of Gross Primary Production (GPP) and Respiration (R) associated with the aquatic community at selected locations. Although examined by various researchers, the technique employed by EAB is a modification of that described in: **Odum, H.T. and C. M. Hoskins. 1958. Comparative Studies on the Metabolism of Marine Waters. *Publ. Inst. Of Marine Science, Univ. of Texas.* 4:115.** Generally, the method involves the monitoring of dissolved oxygen concentrations over a diel period (diurnally) followed by the graphical analysis of dissolved oxygen concentrations, oxygen saturation, and the rate of change of dissolved oxygen at prescribed intervals as corrected for diffusion of oxygen at the air/water interface based on saturation deficits. The rate of change in dissolved oxygen is described by the following equation: $q = p - r + d + a$ where q is the rate of change in DO; p is production (rate of photosynthesis); r is respiration; d is diffusion (at air/water interface); and a is accrual. Hence, because of the potential affect of accrual, it is critical to this method that the history of the water at the station being examined remains constant through the diel period, thus not being influenced by the accrual of dissolved oxygen from unaccounted sources. If the history of the waters being

examined cannot be accounted, and/or if adjustments for accrual cannot be accomplished with confidence, then the data should not be subjected to the diel curve method analysis. Review of the diel dissolved oxygen concentration curve, in combination with hydrologic data for the waterbody, should provide the scientist with sufficient information to govern water history determinations. Of additional importance is an assessment of the average depth of the waters being investigated to effect a more accurate computation of GPP and R on a gmO_2/m^2 basis.

EAB's modification to the Odum & Hoskins diel curve methods involves the application of diffusion rates. Odum & Hoskins used an equation requiring a subjective decision to compute a diffusion constant (k) which involved the rate of change in DO along with percent saturation deficits at before dawn (sun-up) and after sunset. Subjectiveness entered into this assessment as one selected the appropriate point on the diel curve for sun-up and sun-down values. Often, particularly in marine systems, the initial and end points of the usual bell-shape DO curve during daylight hours do not directly coincide with dawn and sundown as indicated on a pyranograph. Since publication of the diel curve method by Odum & Hoskins, advances in other assessment techniques for directly measuring diffusion and/or reaeration coefficients have been made which allows insitu measurement of these coefficients for application to the diel curve analysis. These advances, many of which occurred specifically within EAB, enhance the diel curve analysis by providing diffusion constants measured in the field under ambient conditions, thus removing the subjectiveness.

The GPP & R values resulting from total community oxygen metabolism measurements are typically used as the overall baseline ("bank account") for oxygen budget assessments. As with any budget, various debits and credits are involved which affect the overall status of the account. In the case of the oxygen budget, the production and respiration of various components of the aquatic ecosystem governs the oxygen balance. If the goal is to partition the P & R process to account for the credits and liabilities to the system, then measurement of specific oxygen production and respiration rates must be conducted in concert with the diel curve assessment. Such processes include water column P & R, reaeration, and sediment oxygen demand (SOD). Since light plays a major role in production rates, records of solar energy as well as light profiles through the water column enhance data interpretation. If measurements of P & R span multiple days, solar energy values are essential for comparison and normalization of rates. Methods for determination of rates and coefficients associated with these processes, as well as solar energy and light transmission, are contained elsewhere in this document.

15.2.2 Total Community Sampling Equipment

The equipment list below represents only that applicable for accurate measurement and monitoring of dissolved oxygen and temperature throughout a diel period. As previously stated, corresponding assessment of diffusion or reaeration is critical to adjustment of the DO rate of change curve. Techniques and equipment associated with measurement of diffusion constants or reaerations coefficients are described elsewhere in this manual.

- DO meters with stirrers or multiparameter Sondes with data logging capability
- Salinometer (for marine systems - not required if Sondes are used)
- Calibration accessories
 - DO calibration chamber
 - Calibration standards for sondes
 - NBS certified thermometer
 - Lap-top computer for sonde interrogation/calibration

- Pyrheliometer
 - 7 day and 24 hr charts
 - Pens for inking chart
- Supplies for deployment of DO monitors
 - Rope or line
 - Conduit or rebar
 - Plastic ties
 - Electrical tape
 - Anchors (ie. concrete blocks, lead weights, PVC cast concrete, etc)
 - Floats/buoys

15.2.3 Total Community Oxygen Metabolism/Continuous DO Monitoring Procedures

EAB's standard operating procedures for assessment of total community oxygen metabolism employ multiparameter data logging sondes for acquisition of DO, temperature, conductivity, salinity, and pH. However, other hardware and/or techniques can be substituted for data acquisition. Procedures detailed below will focus on the use of sondes and associated equipment.

Predeployment

- Service sondes (ie. batteries, membranes, etc. - see mfg's specs)
- Check of sonde sensor performance is recommended
- Conduct calibration of all selected sensors using fresh, traceable standards (see manufacturer's operation manuals for YSI or Hydrolab equipment)

Deployment

- Set up data logging profiles in sondes, double checking time, date, and data logging intervals.
- Label sondes, using lab tape, with appropriate station number information.
- Assess station characteristics and select location and depth based on guidance specified in project specific study plan and/or data quality objectives
- Secure probes or sondes at appropriate depth by fastening to rods driven into stream bottom or by hanging from floating buoys. Weighting of probes or sondes in moderate to fast currents may be required.
- Where appropriate, secure additional safety lines to probes/sondes. Keep in mind possibilities for changes in climatic/flow conditions that may be encountered during the course of the survey or at the time that instruments may be retrieved.
- Retrieve sondes/probes at end of deployment period and perform end calibration checks.
- Download data to diskettes (or hard drives) but do not erase files on sondes until review of data has been completed.

15.2.4 Total Community Oxygen GPP and R Calculations

Since sondes are the method of choice for conducting Total Community P & R assessments, this section will address processing of dissolved oxygen and temperature data retrieved from sondes. As stated earlier in this section, Odum & Hoskins (1958) provides the primary guidance for community metabolic calculations. Additional guidance, incorporating use of computer spreadsheets

is as follows:

- Import sonde data into spreadsheets and arrange spreadsheet equations to;
- Plot diurnal dissolved oxygen curves;
- Compute oxygen saturation and plot diurnal dissolved oxygen saturation curves;
- Compute and plot dissolved oxygen rate of change curves;
- Compute and plot dissolved oxygen corrected rate of change curves as by adjusting for saturations deficits and diffusion rates;
- Calculate or select “r” (hourly respiration) and plot as baseline respiration line for 24 hour period;
- Compute GPP by intergrating area under curve to the corrected rate of change curve during photoperiod (this area is bracketed by the baseline respiration line and the corrected rate of change curve) and multiplying by average depth of station or by depth of euphotic zone if less than total station depth;
- Compute R (daily respiration): $R = r(24\text{hrs})(\text{average depth})$.

15.2.5 Quality Control Checks

- End calibration for DO should be within 0.5 mg/L of end calibration DO Winkler titrations;
- If sondes are to be deployed greater than 72 hours, calibration checks and/or recalibration for DO should be performed generally around the third day of deployment by temporarily placing the sonde in a DO calibration chamber (containing water of a known DO concentration) and adjusting DO as appropriate;

15.3 Reaeration

SECTION OBJECTIVE:

- To provide guidance for the measurement/and determination of the rate of reaeration of water bodies.

15.3.1 Introduction

The determination of the stream reaeration rate coefficient is a measure of the rate at which atmospheric oxygen can move across the air water interface. This is a measure of the rate of potential oxygen transfer. The actual quantity of oxygen transferred to the water column is a function of the water column dissolved oxygen deficit and the reaeration rate coefficient.

The most notable existing methods for measuring stream reaeration rate coefficients are gaseous tracer procedures. The US Geological survey "Determination of Stream Reaeration Coefficients by Use of Tracers" (1989) has developed a measurement technique using propane gas. However, due to the bio-sorption problems associated with propane this technique has some inherent limitations. For these reasons EPA Region 4, has pursued the application of a modified non-radioactive krypton technique for measuring stream reaeration rate coefficients. The chief consideration in developing this new technique was to build upon the strengths of the past Kr 85 and propane work.

In general, the technique involves the simultaneous and continuous injection of rhodamine WT dye and noble krypton gas. The purpose is to create a sustained steady state plateau of gas and dye in the stream. The plateau is then sampled at successive downstream locations for the purpose of measuring desorption of krypton in the waterbody. This provides the information necessary to compute associated reaeration rate coefficients.

15.3.2 Reaeration Sampling Equipment

Gas Injection

- cylinder of noble gas krypton
- two stage gas regulator
- gas flow meters
- quick disconnects
- nipples
- gas hose
- ceramic diffuser plates

Dye Injection

- 50 liter graduated polyethylene container
- Rhodamine WT tracer dye
- tubing
- dye injection pump
- 12 volt deep cycle battery

Gas Sampling

- specialized gas sampling bucket
- specialized gas sample bottles
- sample rack
- ice chest
- custody sheets
- field labels/tags
- marking pens

Dye Monitoring

- 12 volt batteries
- submersible pump
- fluorometer
- automatic sampler
- sample intake tubing

15.3.3 Reaeration Procedure

Injection Site Selection

A reaeration injection requires the introduction of a tracer gas and liquid dye into the water

column. The EPA methodology uses a steady state injection of both the noble gas krypton and Rhodamine WT dye. The desorption gas (krypton) is dispersed into the water through the use of micro bubbler aerator plates. Simultaneous dye tracer injection at a center point relative to the gas plates is controlled via a constant rate injection pump. The following criteria should be utilized to identify a representative station:

- The stream at this site should be reasonably deep (>2 feet) with an even velocity in cross-section;
- Downstream of the station, the stream should have some degree of sinuosity allowing for the greatest mixing potential;
- In general, if this is a bank side injection the location should preferably be accessible with minimal climbing.

Gas and Dye Injection

Calculations must be made to insure that the formation of the dye and gas tracer clouds created during the injection meet the steady state requirements of the technique. Obtain preliminary information about the geometry of the study reach under investigation. General information should include the width, depth, and mean reach velocity as they are necessary for estimation of dye and gas requirements. The gas injection equipment should be set first at the injection site. The configuration of the system should follow in order: gas cylinder, regulator, air hose, gas flowmeter, diffuser plates. Placement of plates should be in the main hydraulic conveyance channel. Next the constant rate dye injection equipment should be set up. The pump should be calibrated on site to deliver the requisite dye volume over the predetermined period of injection. The dye discharge tube should be positioned in the main hydraulic conveyance section of the stream. The dye and gas should be started simultaneously and the injection rates of both should be constant throughout the duration of the injection period. The following criteria should be met:

- The rhodamine WT dye should be injected at a constant rate to maintain a plateau concentration in the subject stream less than 100 ppb;
- At no time should the dye concentration exceed 50 ppb at the point of withdrawal for any water supply within the study reach;
- The noble gas krypton should be injected at a constant rate to achieve a plateau of sufficient concentration and duration to allow for the measurable loss of gas along the entire study reach;
- The dye and associated gas should have at least three hours of plateau at the farthest downstream station;
- Specifically; station locations should be sited to allow for a target 60 percent reduction in gas due to loss to the atmosphere from upstream to downstream.

Dye Monitoring and Gas Sampling

At each successive downstream station the protocol for sampling is repeated. First, a

submersible pump connected to a flow thru fluorometer is used to qualify the extent of dye mixing in the stream. Dye must be mixed throughout the width and depth of the water column at that station. If the mixing is complete in the cross section, monitoring continues until an equilibrium plateau dye concentration is achieved and sustained. It is mandatory that a steady state dye concentration plateau be achieved before krypton and argon gas tracer sampling is initiated. Gas sampling is conducted with specially designed weighted buckets that allow for the sampling of four replicate krypton and argon samples without inducing aeration. This procedure is repeated through four equally spaced intervals in time (normally 30 minutes).

After the gas sampling is completed an automatic sequential sampler is then installed at the site to collect aliquots from the stream. These samples are later analyzed for dye concentration. This provides the data necessary for computing time of travel.

Samples are held on ice until analysis is started. A dedicated gas chromatographic mass spectrometer (GCMS) system equipped with a unique gas extraction cleanup line is used to determine the Krypton: Argon ratio of gas in traced water samples. Krypton/Argon ratio analysis is presently conducted at the UGA-CAIS Laboratory in Athens, Georgia.

15.3.4 Reaeration Rate Coefficient Calculation

Once the field data have been processed, desorption rate coefficients are calculated for each reach. Desorption measurements require stream travel time, flowrate, and krypton/argon ratios at consecutive downstream stations. Travel times are based on calculated centroids of successive downstream dye clouds. Flows are computed using a mass balance dye dilution calculation on dye plateau concentration results and/or by gaging measurements. Computed desorption rate coefficients are converted to oxygen absorption rates (reaeration rate coefficients) using the relative transfer rates. The earlier work on this project conducted by Whittemore and Krause had established the relative transfer rate of krypton desorption to oxygen absorption at 0.83 +/- 0.04. Lastly reaeration rate coefficients are temperature corrected to a base temperature of 20°C.

The following equations are used to compute the reaeration rate coefficient for each reach.

$$(1) K_r = 1/tt * (\ln((Q_{up} * (Kr/Ar)_{up}) / (Q_{down} * (Kr/Ar)_{down}))) = \text{day}^{-1}$$

$$(2) K_a@t = K_r / 0.83 = \text{day}^{-1}$$

$$(3) K_a@20^\circ\text{C} = K_a@t * (1.0241^{(20.0 - ((T_{up} + T_{down}) / 2)))}$$

tt = travel time (days)

Q_{up} = flow at upstream station (cfs)

Q_{down} = flow at downstream station (cfs)

$(Kr/Ar)_{up}$ = Krypton:Argon ratio upstream (unitless)

$(Kr/Ar)_{down}$ = Krypton:Argon ratio downstream (unitless)

T_{up} = upstream temperature (°C)

T_{down} = downstream temperature ($^{\circ}\text{C}$)

$K_a@t$ = reaeration rate coefficient at ambient temperature (1/day)

$K_a@20^{\circ}\text{C}$ = reaeration rate coefficient corrected to 20°C

15.4 Water Column Oxygen Metabolism

SECTION OBJECTIVE:

- To provide guidance for the determination of water column oxygen Gross Primary Production (GPP), Net Primary Production (NPP) and Respiration (R) rates in water bodies.

15.4.1 Introduction - (Light and Dark Bottle Oxygen Method)

Water Column Oxygen Metabolism involves the assessment of Gross Primary Production (GPP), Net Primary Production (NPP), and Respiration (R) associated with phytoplankton communities in the aquatic community at selected locations. This method is described in detail in **Standard Methods for the Examination of Water and Wastewater (various editions)**. EAB's Light and Dark Bottle Method is generally consistent with Standard Methods with limited procedural modifications which will be described below. Generally, the method involves the deployment of clear (Light) and Dark bottles at selected locations. Bottles are deployed at intervals through the water column based on percent light transmission profiles which provides for full integration of the water column. Since light plays a major role in production rates, records of solar energy as well as light profiles through the water column are required for bottle deployment and data interpretation. If measurements of P & R span multiple days, solar energy values are essential for comparison and normalization of rates. Procedures and equipment required for determination of solar energy and light transmission, are contained elsewhere in this document. As with community P and R assessments, an assessment of the average depth of the waters being investigated is essential to effect a more accurate computation of GPP and R on a $\text{gm}_{\text{O}_2}/\text{m}^2$ basis.

Discussions elsewhere in this SOP indicated the use of Total Community GPP & R values resulting from total community oxygen metabolism measurements to serve as the overall baseline ("bank account") for oxygen budget assessments. Water Column P and R rates are used as a means of partitioning the credits and deficits to the oxygen budget associated with water column metabolic processes. Rates developed through this method can also serve as essential input parameters in water quality models.

15.4.2 Water Column Oxygen Metabolism Sampling Equipment

- Pyranograph (pyrheliometer) for recording daily solar energy
- Marine photometer for determination of percent light transmission profiles.
- Winkler titration kits (fully stocked with fresh reagents, sodium thiosulfate (0.0375N)), and fresh starch. (It is recommended that sufficient reagents, sodium thiosulfate, and starch be available for assessing one and one half the actual number

of stations expected, allowing for extreme oxygen concentrations due to supersaturation in the light bottles. Preproject calculations should be conducted to estimate sodium thiosulfate requirements.)

- Light and Dark bottles (300ml bottles)
- 4-liter horizontal water sampler or submersible 12-volt pump for filling bottles.
- Plastic caps for light and dark bottles. Black plastic caps for dark bottles are required. Aluminum foil can be substituted for black plastic caps.
- Floats with chains (graduated at 1 foot intervals) for bottle deployment.
- Anchors with rope for securing floats with bottles on stations.
- Rubber bands for securing paired bottles together to prevent breakage during rough sea conditions states that may be encountered during incubations periods.
- Ice chest to be used (if necessary for darkness or icing) when bottles are retrieved.

15.4.3 Water Column Oxygen Metabolism - Procedures

As previously stated, EAB's standard operating procedures for assessment of water column P and R is generally consistent with that described in Standards Methods with a few procedural modifications. The procedures described below include those modifications.

Deployment

- Deploy pyrhelimeters with 24 hr and 7 day charts.
- Obtain percent light transmission profiles on station.
- Select bottle deployment depths based upon light transmission percentages that will permit integration of the water column/euphotic zone. As an example, in very clear waters, bottles would be deployed at percent transmission values such as 90, 50, 10, and 1 percent. Such excellent percent transmission values are rarely encountered so onsite selection of appropriate depths based on percentages that fully integrate the water column must be made.
- At each selected depth, fill two bottles for initial DO determination, two light bottles and two dark bottles for deployment. If the total depth at the station is greater than the depth of the photic zone (defined as the zone with 1% or greater ambient light), then two additional dark bottles should be filled within one foot of the bottom and deployed.
- Light and dark bottle measurements should occur generally during the middle of the day when solar intensity usually greatest. Duration of incubation periods should be adjusted based on productivity of the waters being assessed but under no circumstances should incubation periods include early and late portions of the day when insufficient light could affect production inordinately. Ideal incubation times are usually between 0900-1500 hours.
- While bottles are being filled from selected depths, care should be taken that bottles are not allowed to sit in intense sunlight above water before being deployed.
- Immediately "fix" initial bottles with manganous sulfate, and iodide azide solutions (Winkler reagents "1" and "2") and set aside for later processing via the Winkler method. Preserved in this manner, the bottles can sit for several hours before addition of sulfuric acid and titration. If reagent "3" (sulfuric acid) is added immediately after settling, then the bottles should be placed in the dark to prevent photodegradation of the iodine which result in erroneous DO measurement.
- Deploy paired light and paired dark bottles at the depths from which they were filled

being sure to record time at which deployment was initiated. This should take into account time at which bottles were filled. Filling of all bottles should usually take no more than 15 to 20 minutes.

- Allow deployed bottles to incubate at depth for approximately 4 hours. As previously stated, incubation times should be adjusted for productivity of waters.
- Retrieve bottles being sure to record retrieval time.
- “Fix” bottles with Winkler reagents immediately upon retrieval. The exception to this is if supersaturation in the light bottles is suspected and oxygen bubbles are observed in the light bottles, then they should be submersed in ice in an ice chest and cooled to approximately 4 degrees Centigrade to increase the solubility of oxygen to its maximum before preservation with Winkler reagents. This additional time should not be added to the incubation time.
- As with the initial bottles, only Winkler reagents “1” and “2”, (manganous sulfate and alkaline iodide azide) should be added if the titrations are not to be performed immediately.
- Conduct Winkler titrations for determination of DO in the light and dark bottles.

Water Column P and R Calculations

Using the oxygen light and dark bottle method, light bottles represent Net Primary Production (NPP) and dark bottles represent Respiration (R) at each respective depth. Calculations and data analyses are as follows:

- Average DO concentrations for the paired initial bottles, light bottles, and dark bottles at each respective depth.
- Light bottle DO - Initial bottle DO = net primary production
- Initial bottle DO - Dark bottle DO = respiration
- $npp + r = gpp$
- Plot npp values (Y axis) from each respective depth against bottle depth (X axis) and integrate area under the curves to obtain NPP as $gmO_2/m^2/incubation\ period$. Divide by incubation time (hrs) to obtain NPP in $gmO_2/m^2/hr$ and multiply by 24 to obtain $gmO_2/m^2/day$.
- Repeat for Respiration.
- Repeat for Gross Primary Production.

Quality Control Considerations

- In as much as respiration can be minimal even over a 4 hours or more incubation period, extreme care should be taken with the mechanics of titrations to achieve as much accuracy as possible under field conditions.
- Pay close attention to recording the beginning and end times of incubation.
- Handling and care of bottles, avoiding exposure of light bottles to intense sunlight before deployment is essential.
- Observation of light bottles for bubbles after retrieval and icing as necessary is critical as previously discussed.

15.5 Radiant Energy

SECTION OBJECTIVE:

- To provide guidance for the acquisition of radiant energy data while conducting Primary Production (P) and Respiration (R) assessments.

15.5.1 Introduction

Radiant energy is a primary factor governing aquatic community primary production rates. When assessments of total community oxygen metabolism, water column oxygen metabolism, and benthic gross and net primary production are being conducted, it is essential that daily records of total available solar energy be acquired. Often, such measurements will span several days, or be conducted during different segments of a daily photoperiod. Accordingly, records of radiant energy must be available and used to normalize primary production rates to a standard energy level for data comparability.

The instrument of choice for the EAB is the pyranograph, a recording pyrliometer, which can be equipped with gears for recording solar energy over a 24 hour or 8 day period. For EAB P & R assessments, it is suggested that two instruments be used, one equipped for 24 hours records, which yields more discrete hourly analysis, and the other equipped for 8 day recording. With appropriate gear installation, both instruments are deployed and operated as follows:

15.5.2 Calibration and Maintenance

The pyranographs used in the recording of radiant energy require no calibration unless the instrument is damaged. Such repair and calibration must be conducted by the manufacturer. The instrument does, however, require periodic adjustment for zero. Zero adjustment should be checked prior to use in each field activity by bringing the instrument indoors and allowing it to stand for one hour and then reading the pen position. Should zeroing of the instrument be required, specific instructions contained in the manufacturer's manual which accompanies the pyranograph should be followed by the analyst. The dark bimetallic strip should be observed for blackness prior to putting each strip chart in place, and observing the time at 2-hour intervals over the course of a working day. Deviations in accuracy beyond one hour require clock replacement. The dome must be kept clean and unshaded. Daily morning and afternoon checks of the instrument should be performed during field operation. Time of the check versus pyranograph time should be recorded along with climatic conditions. (Note: If zero adjustment is not done prior to deployment, corrections on the pyranograph chart can be accomplished during post processing of data by inserting a "zero" baseline along the nighttime portion of the graph.

Reporting Units - All data are recorded in langleys/hour or gram calories/hour.

15.6 Light Transmission

SECTION OBJECTIVE:

- To provide guidance for the acquisition of percent light transmission profiles and light extinction coefficients while conducting Primary Production (P) and Respiration (R) assessments and water quality investigations.

15.6.1 Water Column Light Transmission Profiles

Available light is a governing factor effecting production of oxygen by phytoplankton and attached algal and rooted aquatic plant communities. Accordingly, for station to station comparison of production rates in waterbody P and R assessment, determination of light transmission through the water column is essential. Assessment methods such as the Light and Dark Bottle Oxygen method require accurate determination of light transmission in order to conduct the assessment in a scientifically valid manner. Additionally, in other types of assessments, such as monitoring of ocean disposal sites, where resuspension and movement of sediments can affect sensitive biological resources, determination of light extinction coefficients at specific depth intervals can be a useful assessment parameter.

Percent light transmission and light extinction coefficients are obtained using an irradiator ("marine photometer"). The instrument consists of a monitor with either two analog scales or two digital scales which are coupled to two photo cells, one for ambient light ("deck cell") and the other for underwater light ("sea cell").

15.6.2 Calibration and Maintenance

Presurvey checkout and calibration of the underwater irradiator should be conducted in accordance with the manufacturer's manual. Checkout procedures involve calibration (zeroing) and battery checks for power supply before and after field use each day or more frequently if meter performance is suspected of going out of control during a monitoring period.

- General guidance for checking instrument performance is as follows: perform a battery check using the appropriate switch on the monitor; after verifying sufficient battery power, zero each analog scale by assuring that the black rubber cap and red cover are providing complete darkness for the deck and sea cells. (Use any other available material to assure complete darkness of the cells if necessary.) Use the zero adjustments as required (Note: Direction of turn for the zero knob is counter to the expected direction.) Once zero is acquired for each cell, be sure that each scale is set on 10K; uncover each cell and place them side by side in a completely level position and read each meter. If meter readings vary more than 0.5, perform an adjustment to synchronize the two meters by opening the monitor and using a small screw driver to adjust the electronic "pots" inside the unit that are appropriately marked for the deck cell and sea cell. (NOTE: a tolerance of 0.5 difference between the two analog meters is permissible due to the difficulty of assuring precise leveling when working on boats. Any deviation greater than 0.5 should be corrected using the above guidance.)

- Maintenance of the instrument is also specifically outlined in the manual and should be performed after each field activity upon returning to the laboratory. Calibration, other than zeroing or analog meter (deck vs. sea cell) synchronization, must be performed by the manufacturer based on (1973) National Bureau of Standards Scale of Spectral Irradiance.

Presurvey checkout information should be recorded in the field data book for each individual survey. Recalibration information accompanies the instrument when returned from the factory and is placed in the operation manual.

Instrument readout is based on $\mu\text{W}/\text{cm}^2/\text{nm}$ and multiples thereof including 10, 30, 100, 300, 1K, 3K and 10K. Field readings are recorded directly from meter observations to the nearest tenth accompanied by the scale setting (10-10K).

15.6.3 Light Transmission Field Procedures

Light transmission profiles are obtained by lowering the sea cell a prescribed depth increment through the water column. Guidance is as follows:

- Perform battery checks, zero adjustment, and analog meter synchronization as previously specified.
- Obtain a deck and sea cell reading with the cells side by side and as level as possible and unshaded (except for possible cloud cover). Record reading in the data book as a "deck reading", recording both the scale setting and the observed light value.
- With the deck cell always remaining in an unshaded location, lower the sea cell into the water until only the thinnest film of water covers the white photo cell. This can be obtained by simply allowing the "well" surrounding the white photo cell to fill with water without further submergence. Record both deck and sea cell scales and light values as the "surface" reading.
- Continue profiling at 0.5 foot increments, recording scales and light values until below the 1% light transmission depth or the sea cell reaches the bottom. Adjust scale on sea cell to greater sensitivity as required during the profile, being sure to log scale change in data book. In water of high clarity, wider spaced increments may be used to establish the profile, using best professional judgement. Suggested log book setup:

Date		Station		Time	
Deck Cell			Sea Cell		
Depth	Scale	Reading	Scale	Reading	% Light

- Always assure that the sea cell does not drift into the shade of the boat during deployment since this would create an error in the light profile.
- If cells will not synchronize with acceptable tolerance during "deck" reading, or if

deck cell fails, light profiles can still be acquired using only the sea cell if conditions of full sun or steady cloud cover prevail through the profile. Acquire deck reading in full sun or steady light with the sea cell, then conduct profile with the sea cell. Upon returning the sea cell to the deck, measure and record the final light value for comparison with the beginning deck reading.

15.6.4 Quality Control Checks and Considerations for Light Transmission Measurements

- Conduct zero and synchronization adjustments as prescribed.
- Assure both deck and sea cells remain unshaded while obtaining the light profile, excepting cloud cover.
- Allow no greater than 0.5 deviation between the deck and sea cell during "deck" comparison. Strive for the minimum deviation possible.

15.7 Sediment Oxygen Demand (SOD)

15.7.1 Chamber Design and Other Considerations

The determination of benthic metabolic rates is accomplished through the placement of clear or opaque chambers over bottom substrate and then recording the oxygen decay or production rate at intervals during the course of an investigation. In the case of SOD, only opaque chambers are used.

The shape of the chamber maximizes water volume entrapped to sediment surface area isolated by the chamber. By maximizing water volume to surface area, sufficient dissolved oxygen resource is usually enclosed within the chamber to reduce rate fluctuations or changes attributable to resuspension during deployment, yet, sustaining the investigation for a length of time necessary to produce highly correlated data. In maximizing volume to surface area, factors such as circulation, deployment, and stability in currents must be considered in relation to the shape of the chamber. Regardless of the chamber size or design, circulation of the enclosed waters should be sufficient to prohibit variation in DO concentration or "dead spots" within the chamber.

An SOD chamber should be equipped with a flange and cutting edge along its perimeter of contact with the sediment. The horizontal flange and vertical cutting edge effectively stabilize and secure the chamber to the sediment surface. Additionally, the flange limits intrusion into the sediment, and by maintaining vertical position, provides a degree of certainty respective to chamber volume. In harder substrates and rubble where penetration of the cutting edge is limited, the flange can be fitted with a soft flexible collar to aid in sealing the chamber to the substrate. In soft, mushy substrate, the chamber can be fitted with a larger flange such as plywood to limit its penetration but still create an effective chamber to substrate seal.

Isolation of the chamber environment from surrounding ambient waters is essential to prohibit replenishment of the oxygen resource within the dome by leakage. Diver deployment and placement of the chamber provides an effective means of achieving a seal with the bottom substrate. Deployment by divers eliminates many uncertainties in chamber placement, particularly regarding the effectiveness of the seal as well as such things as substrate type, topography and presence or absence of benthic macrophytes.

The chamber is designed with a port or moveable lid to pass water and emit trapped air as it is lowered through the water column. Passage of water through the chamber as it is lowered assures that ambient bottom water is ultimately enclosed within the chamber for the SOD experiment and, further, reduces shock wave disturbance and resuspension of the bottom sediment upon impact. The lid or port should be closed once the chamber is seated by a diver.

Circulation of chamber enclosed waters is necessary to achieve uniform DO concentrations throughout the chamber. Commercially available self-stirring oxygen probes are not adequate. Accordingly, either a propeller or pump with diffuser mechanism is necessary. Mixing rate should be uniform within the chamber and comparable between all chambers used and each replicate conducted. Variation between chambers and replicates affects data comparability. To assist in data comparability, mixing rates, dispersion coefficient, or turnover rates should be reported for each experiment. Regardless of which mixing device is employed, the operator of the chamber must be concerned with debris fouling the devices. Orientation of the circulation device should be in a manner to prevent sediment resuspension during operation. Resuspension tends to increase apparent SOD. Graphical analysis of DO concentration showing inconsistencies with subsequent data points during the course of the experiment should be used in detecting resuspension.

Because a portion of the water column is enclosed within the chamber, monitoring of dissolved oxygen changes in the chamber will also reflect metabolic processes occurring both in the water column as well as the sediment. A relative measure of the water column metabolic impact to the chamber's dissolved oxygen resource can be achieved by dark bottle experiments conducted simultaneously with the SOD experiment using bottles filled with ambient bottom water suspended alongside the chamber. A more effective way, and the method used by Region 4, is to deploy chambers identical to the SOD chambers, but with a sealed bottom. These "blank" chambers are filled with bottom waters using the circulation pump to purge the chamber and the reduction in DO concentrations is monitored simultaneously with the SOD chambers. Since the "blank" chambers bottom eliminates any contact with the sediment, only a water column respiration rate is observed and used during rate calculations to partition the water column from sediment respiration in the SOD chambers. Adjustments to the sediment oxygen demand rate should then be made by application of the water column production and respiration rate.

The SOD chamber presented in Figure 15-1 is the primary type used by personnel of the Ecological Assessment Branch. Annular in configuration, the chamber encloses 65 liters of water over 0.27 m² of sediment. Circulation is achieved with one 160 gph 12V DC pump. The chamber lid is on stanchions and is secured in the raised position during deployment. Divers seal the chamber to the bottom and lower the lid to the closed position which results in isolating a known volume of water over a defined area of sediments. The dissolved oxygen concentration of the enclosed water is monitored for approximately 2 ½-4 hours. The average rate of decrease in DO concentration is determined and used in calculating SOD as defined in section 15.7.4.

15.7.2 Equipment and Supplies

- Chambers as described in Figure 15-1
- Dissolved oxygen meters, probes or multiparameter sondes
- Salinometer or conductivity meter
- Opaque BOD bottles with dark caps
- Data book
- 12v batteries

- Battery charger
- Extra pumps
- Extra diffusers
- Current meter
- KCL & Membranes to DO probes
- Scotch-kote for coating electrical connections
- Vulcanizing tape
- Electrical tape
- Fiber tape
- 3/8" Tygon tubing
- 3/8", or larger, yellow poly. line
- Silicone and caulking gun
- Extra flange gasket for chambers
- Rubber stoppers
- C-clamps
- Winkler kit with reagents, thiosulfate, starch
- C-size batteries for DO meters or AA's for multiparameter sondes
- van-Dorn (horiz) sampler
- Bucket

15.7.3 Procedures

The following procedures are recommended for conducting field measurements of sediment oxygen demand using the chamber configuration shown in Figure 15-1.

1. Obtain preliminary information about the study area to determine general sediment types and near bottom current velocities. Perform functional checks of all equipment before survey.
2. Calibrate dissolved oxygen meters and/or other monitoring equipment such as multiparameter sondes, salinometer or conductivity meters according to manufacturer's instructions. Maintain record of calibration. A DO probe having a stirring device or pulsed probes is required in order to maintain appropriate water velocities across the membrane of the probe.
3. Measure vertical profiles of dissolved oxygen, temperature, salinity or conductivity. Near bottom concentrations of dissolved oxygen less than 1.0 mg/L are generally inadequate for the measurement of SOD using chambers. Attempts at measuring SOD rates under such conditions must be done with great caution or the dissolved oxygen will be depleted within too short a period of time for adequate data acquisition.
4. Check delivery of power and operation of circulation pump.
5. Deploy chambers. Lower the chamber with rope by a person in a boat or wade in from the bank taking care to avoid placement of chambers in an area disturbed by wading. When on bottom, divers place, position and seal chamber. Deploy blank chamber first and position upstream from other chambers. Purging of blank chambers with bottom water occurs while other chambers are being deployed with

care taken to avoid suspension of sediments which could be sucked into the blank chamber. In a period of 20 minutes, the contents will turnover approximately three times.

6. Allow approximately 10-20 minutes for settlement of material that might have been resuspended during deployment of chamber. Install monitoring probes and engage circulation pump. Secure probe into hole in lid with number 12 rubber stopper fitted to probe housing, assuring that the split stopper is completely sealed.
7. Lower ambient probe to chamber level; approximately one foot above bottom.
8. Record initial monitoring data.
9. Deploy a minimum of two dark bottles (300 ml each) alongside the chambers for incubation during the course of the SOD experiments. Use water column respiration values obtained from the dark bottles as back-up to blank chamber experiments in case of chamber failure. General procedures for conducting the light-dark bottle tests are provided in Standard Methods, 17th or later editions.
10. Record monitoring data either continuously or at prescribed intervals (usually 15 minutes).
11. Continue experiment for approximately 2 ½-4 hours or long enough to determine a rate sufficient to mask any precision error in post calibration. Depending upon the meter used, oxygen readings at 5-15 minute intervals for two or more hours provide sufficient data points (6-9) for determining the rate of change of the DO concentration of the chamber contents.
12. With conclusion of the monitoring period, remove probes from chambers and recheck their calibration. Record calibration check. Check operation of circulation pump just prior to termination of experiment.
13. Relocate chambers to nearby position and begin replicate measurement. Use of additional chambers as replicates will eliminate need for relocation of chambers. Three to six replicates are suggested. Allow dark bottle experiment to continue.

15.7.4 SOD Rate Calculations and Data Assessment

During in-situ SOD rate determinations, the decrease in dissolved oxygen concentration is recorded as the instantaneous value at specific time intervals, usually 10 or 15 minutes. The specified interval should be adhered to throughout each individual experiment.

The SOD rate is then calculated using the following equation:

$$\text{SOD} = .06 \frac{V}{A} (b_1 - b_2)$$

where SOD is the sediment oxygen demand rate, in g/m² hr; b₁ is the rate of change in the SOD chamber oxygen concentration, in mg/L/min; b₂ is the rate of change in the "blank" chamber oxygen

concentration, in mg/L/min (b_2 is in effect the water column respiration in the chamber); V is the volume of chamber, in liters; A is the area of chamber, in square meters; and .06 is the constant converting mg/l/min to g/m²/hr. Values for b_1 and b_2 may be determined graphically or from a linear regression analysis, where b_1 and b_2 are the slopes of the curves obtained by plotting SOD chamber and blank chamber DO concentrations versus time.

If dark bottle respiration values are substituted for "blank" chamber respiration, the b_2 value is obtained by subtracting the final dark bottle DO concentration from the initial bottle DO concentration and dividing by the incubation time in total minutes.

15.7.5 Precision (see section 20.2.3)

15.7.6 Quality Control Checks

- All DO meters should be calibrated via the Winkler method (Standard Methods; 17th ed. or later) prior to determination of dissolved oxygen profile and initiation of SOD experiment. Salinometer and conductivity meters should be calibrated electronically.
- Sealing of chamber to substrate should be conducted and confirmed by divers in both salt and fresh water.
- Pump operation should be determined prior to chamber deployment and confirmed by divers prior to securing the lid to the chamber.
- Resuspension should be visually checked by divers in waters with sufficient clarity, but in all cases a 10-minute time delay is observed prior to beginning pump operation to allow time for settlement. Plotting of observed data points during and after experiments also should be accomplished to determine resuspension and its effect on SOD rates.
- Between station, and post experiment, calibration checks should be accomplished by removing DO probes from chambers during relocation or retrieval checking them in a bucket where the DO concentration has been determined via the modified Winkler titration method. Failure of the meter to post calibrate to within ± 0.2 mg/l voids the replicate.
- All light and dark bottle DO concentrations should be determined via the Winkler Method.

15.7.7 Sediment Nutrient Flux

An additional capability afforded with the Sediment Oxygen Demand (SOD) chambers used by Region 4, SESD, is the acquisition of the rate of Nutrient Flux (sometimes referred to as Nutrient Exchange (NUTS-X)) at the sediment/water interface. Through isolation of a known volume of water over a known area of sediment, nutrient flux can be measured by periodic extraction of water samples from each chamber and analytically determining the change of concentration selected nutrients over an extended incubation period. Sampling of water from the chamber may occur only at the beginning and end of the deployment period. Nutrient Flux assessments are usually conducted in conjunction with SOD measurement. Accordingly, the deployment and dissolved oxygen

monitoring techniques are essentially the same (see Section 15.7.3 SOD Procedures), with minor modification of the DO monitoring due to the extended deployment times associated with nutrient flux assessments.

15.7.8 Equipment and Supplies

Basic equipment and associated supplies are the same as those listed in Section 15.7.2 (SOD Equipment and Supplies). Additional supplies required for Nutrient Flux assessment are those associated with the collection, holding, and filtering of water samples to be extracted from each chamber.

- Narrow mouth glass, pint or quart, bottles. Number of bottles required is based upon the number of stations, number of chambers, and the sampling regime (ie. initial plus final plus any intermediate samples). Glass bottles, as opposed to plastic or nalgene, are essential since the underwater sampling procedure depends upon a siphoning effect for the water sample to replace the air within the sampling container. Plastic bottle walls will compensate (collapse) for the water pressure and, thus, will not fill properly.
- Sample collection siphon for extracting water sample from chamber into glass bottle. Siphoning device consists of a stopper sized properly to fit into the mouth of the glass sample bottle. The stopper has two holes through which glass, or rigid plastic tubes are fitted. Plastic pipetts (10 mls) cut to appropriate lengths work well for this. One tube serves as the inlet for injecting sample into the bottle. The inlet tube should extend into the bottle three inches. The second tube serves as an outlet to allow the air to escape from the bottle during sampling. This outlet tube is cut flush with the bottom of the stopper to ensure that all air is purged from the sample bottle. Each tube should extend approximately one inch above the top of the stopper. Tygon tubing, equipped with clamps, attached to the outboard end of each tube is required to seal and open the sampling device during deployment and sampling, respectively. The inlet tubing should be approximately 15 inches in length. At the distal end of the tubing is another stopper, sized appropriately to the sampling port in the chamber lid, through which a single tube has been fitted. The portion of the tube that is inserted into the chamber should be approximately 6 inches in length while the section extending from the top of the stopper outside the chamber need only to be approximately one inch in length.
- Depending upon the targeted analyses, appropriate preservatives are required for sample preservation upon return to the surface. Sample preservation is dictated in the Appendix A of this SOP.

15.7.9 Procedures

Deployment procedures for chambers used for nutrient flux (NUTS-X) measurements are the same as those outlined for SOD measurements. As stated previously, NUTS-X measurements can be initiated concurrently with SOD. However, incubation times (chamber deployment) must be considerably longer for NUTS-X than for SOD. Measurement of nutrient flux rates can be conducted under aerobic or anaerobic conditions. However, the strategy for such measurements must be developed prior to initiation of field activities and include an assessment of available

oxygen resources immediately prior to deployment of chambers. This approach is required since accomplishment of NUTS-X measurements must be initiated and completed without transitioning from aerobic to anaerobic conditions, or vice versa. Such a change in the status of oxygen resources of the water column entrapped within the chamber will yield inconsistent results not reflective of ambient conditions. Accordingly, chamber incubation times (the time period from collection of the initial sample to the final sample) must be estimated by using the observed SOD rate in consideration of the chamber dissolved oxygen concentrations. Following is the sampling approach for collection of samples for determination of sediment nutrient flux rates.

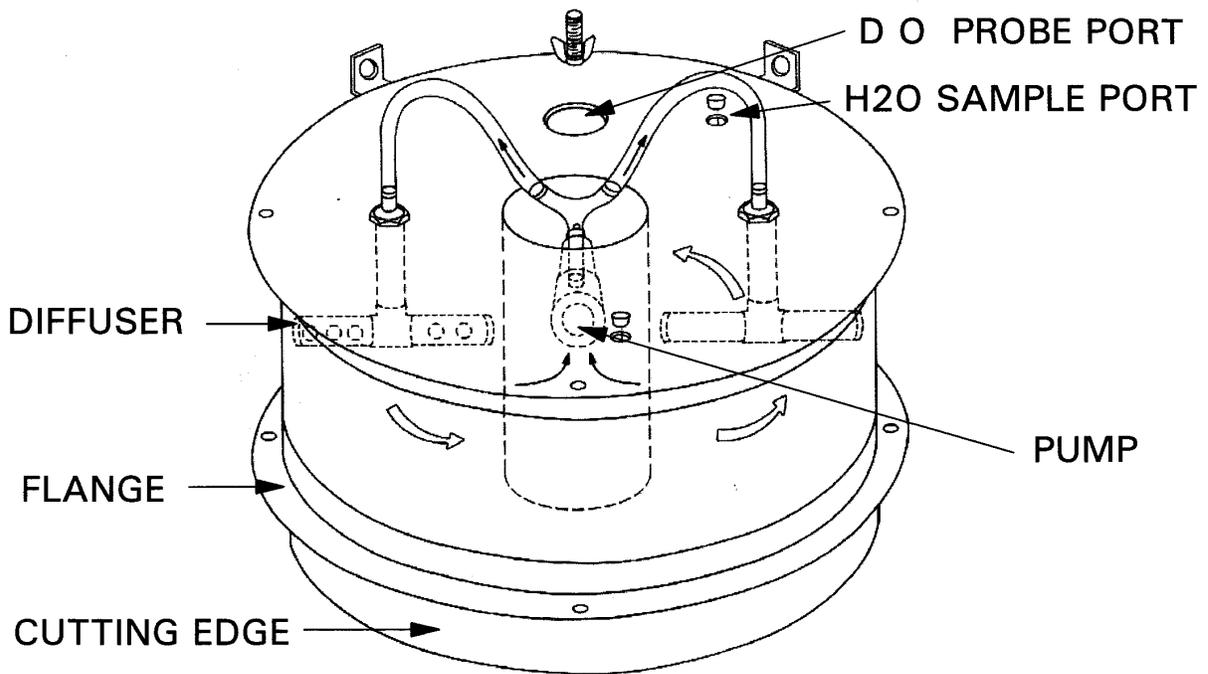
1. Deploy chambers as described in Section 15.7.3 regarding SOD measurements.
2. After the chambers have been seated into the substrate, the chamber lids secured, and the circulation pumps started, extract the initial water sample for nutrient analysis. Procedurally, water samples from each chamber are extracted as follows:
 - Attach siphoning device to the bottle and confirm that tubing clamps are tight and stopper is secure in the bottle mouth.
 - Diver, or wader, submerges the bottle with siphon to chamber depth and attaches siphon to chamber by removing solid (size 1) rubber stopper in chamber lid and inserting inlet tube stopper into chamber lid. All tubing clamps remain closed.
 - With the bottle held above the chamber and inverted, the clamp on the inlet tube is released.
 - Move the bottle to the upright position, release the clamp on the outlet tube, and position the bottle beside the chamber taking care not to accidentally loosen or remove the inlet tube's stopper from the chamber lid.
 - A second (size 1) stopper is positioned on the chamber lid across from the location where the inlet tube has been plugged into the chamber. This second stopper should be immediately removed when the siphoning begins to allow recruitment into the chamber of the sample volume removed during sampling. This is necessary because it is advisable to recruit from the water column rather than from sediment interstitial water.
 - Observe or listen for air bubbles being purged from the sample bottle as it fills. When the bubbling ceases, the bottle is full. Secure clamps on both the exhaust and inlet port, tubes, remove siphon stopper and from chamber lid and replace #1 rubber stoppers into the sampling and recruitment ports.
 - Return sample to surface and process according to procedures appropriate for sample handling and preservation based upon the target analyses.
 - A final sample at the end of the incubation period is required for NUTS-X rate determinations. The final sample is acquired in the same manner as the initial sample. Again, it is important to state that the dissolved oxygen concentration within the chamber must be monitored, and recorded, to make

sure that if the experiment was initiated under aerobic conditions, then it must be completed under aerobic conditions within the chamber. The same requirement applies to experiments conducted under anaerobic (anoxic) conditions.

- After the final sample is collected from each chamber, and pump circulation has been checked the activity is complete and the chambers can be retrieved.

Figure 15-1 SOD Chamber

insitu SOD CHAMBER



64.5 Liters 0.27 m² over bottem velocity 0.8-1.2 fps

15.8 Light Transmission

SECTION OBJECTIVE:

- To provide guidance for the acquisition of light transmission profiles and light extinction coefficients while conducting Primary Production (P) and Respiration (R) assessments and water quality investigations.

15.8.1 Water Column Light Transmission Profiles

Available light is a governing factor effecting production of oxygen by phytoplankton and attached algal and rooted aquatic plant communities. Accordingly, for station to station comparison of production rates in waterbody P and R assessment, determination of light transmission through the water column is essential. Assessment methods such as the Light and Dark Bottle Oxygen method require accurate determination of light transmission in order to conduct the assessment in a scientifically valid manner. Additionally, in other types of assessments, such as monitoring of ocean disposal sites, where resuspension and movement of sediments can affect sensitive biological resources, determination of light extinction coefficients at specific depth intervals can be a useful assessment parameter.

Percent light transmission and light extinction coefficients are obtained using an irradiator ("marine photometer"). The instrument consists of a monitor with either two analog scales or two digital scales which are coupled to two photo cells, one for ambient light ("deck cell") and the other for underwater light ("sea cell").

15.8.2 Calibration and Maintenance

Presurvey checkout and calibration of the underwater irradiator should be conducted in accordance with the manufacturer's manual. Checkout procedures involve calibration (zeroing) and battery checks for power supply before and after field use each day or more frequently if meter performance is suspected of going out of control during a monitoring period.

- General guidance for checking instrument performance is as follows: perform a battery check using the appropriate switch on the monitor; after verifying sufficient battery power, zero each analog scale by assuring that the black rubber cap and red cover are providing complete darkness for the deck and sea cells. (Use any other available material to assure complete darkness of the cells if necessary.) Use the zero adjustments as required (Note: Direction of turn for the zero knob is counter to the expected direction.) Once zero is acquired for each cell, be sure that each scale is set on 10K; uncover each cell and place them side by side in a completely level position and read each meter. If meter readings vary more than 0.5, perform an adjustment to synchronize the two meters by opening the monitor and using a small screw driver to adjust the electronic "pots" inside the unit that are appropriately marked for the deck cell and sea cell. (NOTE: a tolerance of 0.5 difference between the two analog meters is permissible due to the difficulty of assuring precise leveling when working on boats. Any deviation greater than 0.5 should be corrected using the above guidance.)

- Maintenance of the instrument is also specifically outlined in the manual and should be performed after each field activity upon returning to the laboratory. Calibration, other than zeroing or analog meter (deck vs. sea cell) synchronization, must be performed by the manufacturer based on (1973) National Bureau of Standards Scale of Spectral Irradiance.

Presurvey checkout information should be recorded in the field data book for each individual survey. Recalibration information accompanies the instrument when returned from the factory and is placed in the operation manual.

Instrument readout is based on $\mu\text{W}/\text{cm}^2/\text{nm}$ and multiples thereof including 10, 30, 100, 300, 1K, 3K and 10K. Field readings are recorded directly from meter observations to the nearest tenth accompanied by the scale setting (10-10K).

15.8.3 Light Transmission Field Procedures

Light transmission profiles are obtained by lowering the sea cell a prescribed depth increment through the water column. Guidance is as follows

- Perform battery checks, zero adjustment, and analog meter synchronization as previously specified.
- Obtain a deck and sea cell reading with the cells side by side and as level as possible and unshaded (except for possible cloud cover). Record reading in the data book as a "deck reading", recording both the scale setting and the observed light value.
- With the deck cell always remaining in an unshaded location, lower the sea cell into the water until only the thinnest film of water covers the white photo cell. This can be obtained by simply allowing the "well" surrounding the white photo cell to fill with water without further submergence. Record both deck and sea cell scales and light values as the "surface" reading.
- Continue profiling at 0.5 foot increments, recording scales and light values until below the 1% light transmission depth or the sea cell reaches the bottom. Adjust scale on sea cell to greater sensitivity as required during the profile, being sure to log scale change in data book. In water of high clarity, wider spaced increments may be used to establish the profile, using best professional judgement. Suggested logged book setup:

Date		Station		Time	
Deck Cell			Sea Cell		
Depth	Scale	Reading	Scale	Reading	% Light

- Always assure that the sea cell does not drift into the shade of the boat during deployment since this would create an error in the light profile.

- If cells will not synchronize with acceptable tolerance during "deck" reading, or if deck cell fails, light profiles can still be acquired using only the sea cell if conditions of full sun or steady cloudy cover prevail through the profile. Acquire deck reading in full sun or steady light with the sea cell, then conduct profile with the sea cell. Upon returning the sea cell to the deck, measure and record the final light value for comparison with the beginning deck reading.

15.8.4 Quality Control Checks and Considerations for Light Transmission Measurements

- Conduct zero and synchronization adjustments as prescribed.
- Assure both deck and sea cells remain unshaded while obtaining the light profile, excepting cloud cover.
- Allow no greater than 0.5 deviation between the deck and sea cell during "deck" comparison. Strive for the minimum deviation possible.

SECTION 16 WETLANDS CHARACTERIZATION

PERFORMANCE OBJECTIVE:

- To delineate or demarcate a wetland, or to assess its ecological functionality, with a minimum of subjectivity.

16.1 Introduction

Procedures have been established at the national level for meeting the objectives of this Section. These procedures are incorporated by reference herein. Where indicated, their use is mandatory because it is required by executive order in Federal programs.

As used in this Section, delineation means the marking of a wetland's boundary in the field. Demarcation is reserved for the drawing of a boundary on an aerial photograph.

16.2 Wetland Delineation

16.2.1 General Field Procedures

The procedures, equipment, and data forms described in the U.S. Army Corps of Engineers' (COE) wetland delineation manual (Environmental Laboratory, 1987) shall be used on the ground to establish the boundary between wetland and upland. To enable accurate mapping of the boundary, a global positioning system should be used to locate each marked point along the boundary.

Prior to delineating, individuals shall be trained in use of the 1987 manual by passing the Federal 5-day training course (COE, 1994). This course is offered by COE and by the USDA Natural Resources Conservation Service (NRCS).

The 1987 manual is available on-line at:

<http://www.wes.army.mil/el/wetlands/pdfs/wlman87.pdf>.

The on-line version includes data forms and many helpful user notes. Some of these notes direct the reader to revised lists of wetland plants (FWS 1988, 1991), hydric soil series (SCS 1991), and hydric soil field indicators (NRCS 2001). Other notes cite Regulatory Guidance Letters that provide clarification on certain issues raised in the original version of the manual. The on-line version also includes ancillary COE documents that provide additional clarification on various topics.

16.2.2 Soil RedOx Measurement

Low redox potential is indicative of saturated soil, an essential feature of wetlands. Details on the measurement of oxidation-reduction potential in soil are included in Section 11.

16.3 Wetland Demarcation

Procedures, equipment, and decision documentation forms described in the National Food Security Act manual (NRCS, 14) may be used to demarcate wetlands. On lands without a history of agricultural production, demarcation is not a legal substitute for delineation. Demarcation is an exercise in photointerpretation. Where a zoom transfer scope is available to copy the final line onto the photograph, acetate or mylar overlays should be used to draft the boundary. Where a heads-up digitizer is available, a digital map may be made from the interpreted photograph.

16.4 Wetland Functional Assessment

16.4.1 General Field Procedures

The hydrogeomorphic (HGM) method of classifying (Brinson 1993) and assessing (Smith et al. 1995) wetlands should be used wherever an HGM guidebook has been developed. A guidebook exists for low-gradient riverine wetlands in the Western Kentucky Coal Field physiographic province (Ainslie et al. 1999). Guidebooks for other HGM subclasses in other eco-physiographic regions are under development, including one for mineral pine flats in the Lower Atlantic and Gulf Coastal Plains (Rheinhardt et al. 1999). National guidebooks for riverine (Brinson et al. 1995) and tidal fringe (Wetlands Ecology Branch, 1998) wetlands are available for future application to specific regions. Guidance has been developed for future application of HGM in the Piedmont of the Carolinas and Georgia (Brinson et al. 1996). The procedures, equipment, and data forms described in an applicable guidebook (i.e., one written for a particular HGM subclass in a particular region) should be followed where one is available.

Because guidebooks have yet to be written for most subclasses in most regions, site-specific methods will be required. These methods may be developed on a case-by-case basis, as in the Advance Identification (ADID) program (EPA 1984). Methods for disturbed freshwater marshes in the southern peninsula of Florida (Pruitt unpub.) and for fringing/depressional systems in the Florida Keys (McNeese et al. 2000) were written for ADID studies. Where no established method exists that can be reasonably applied or adapted to a specific wetland, best professional judgement should be used. In such cases, documentation of field observations shall be combined with references to the literature to infer a general degree of functionality. Parameters to be recorded shall follow the general HGM methodology, i.e, hydrological, edaphic, and vegetational characteristics shall be documented.

16.4.2 Remote Assessment

Where limitations on logistical resources preclude on-the-ground assessment, and where reliable information on topography, soils, and land use/land cover (LU/LC) is available, wetlands may be assessed using desk-top methods. Remotely sensed information on LU/LC, typically an aerial photograph or high-resolution satellite image, shall be considered reliable only if less than a year old in counties on the perimeter of metropolitan areas or less than five years old in rural counties. The hydrological modifiers on National Wetland Inventory maps (Cowardin et al. 1979), where available, should be used as one indicator of probable changes in the functionality of wetlands

that have been disturbed by human activity (e.g. ditching and partial draining) or by natural events (e.g. damming by beavers). Modern county soil surveys, those published after 1970, contain information useful for inferring bio-geochemical function in wetland soils (Soil Survey Division Staff 1993). The hydric phase of such soils should be validated by landscape position and comparison to the county hydric soils list. State lists may be used where no county list has been prepared. Degraded wetlands may exist on areas with probable wet-phase hydric soils that have been converted from native land cover to agricultural or urban land uses. Published synoptic assessment methods such as those in Kentula (1997) and Kalla (in prep.) should be used where applicable.

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SECTION 17
SURVEYING AND GEOGRAPHICAL POSITIONING

PERFORMANCE OBJECTIVE:

- Present standard practices used for making field physical measurements by land surveying and global positioning system (GPS) techniques;

17.1 Introduction

Field measurements of topographic features, water levels, geophysical parameters, physical dimensions, etc., are frequently required during field investigations conducted by EPA Region 4. The purpose of the investigation will determine the scope and accuracy required for these measurements.

All sampling locations used during field investigations should be depicted on an accurate drawing, topographic or other standard map, or be referenced in such a manner that the location(s) can be firmly established.

Each field measurement made should be traceable to the person(s) making the measurement and to the field equipment used to make that measurement. Equipment maintenance and calibration shall be performed in accordance with manufacturer specifications. Time records shall be kept in local time using the military time format, with the time recorded to the nearest five minutes or less.

New employees should perform each of the physical field measurements described in this section under the supervision of a senior technical staff member at least once before being permitted to make these measurements on their own.

17.2 Horizontal Location Surveys

17.2.1 Introduction

Several field methods, from traditional or classical methods to Global Positioning System (GPS) techniques, may be used to horizontally locate sampling locations or various site features during field investigations. EPA guidance documents (U.S. EPA 1991) recommends that location be described with a 25 meter level of accuracy for purposes of integrating data resources. For the vast majority of projects conducted by the EPA, Region 4, standard non-differentially corrected GPS, which is accurate to within 10 meters is sufficient for horizontal location information. The level of precision and accuracy required to support a particular project should be defined in the project plan of study.

Traditional traverse methods used by the branch utilize horizontal angle or direction (azimuth/bearing) measurements and calculated horizontal distances from a starting point to a

second point.

GPS methods utilize radio frequency measurements with multichannel receivers of the signals from the global network of satellites that the Department of Defense has established. Measurements of the horizontal sampling locations or site features by GPS technology is based on the same principles used in traditional surveying methods. However, with GPS, hand held receivers measure distances to three or more satellites of known positions and triangulate the position of the sampling location, site feature, or point on earth. More fundamental information on GPS technology may be found in U.S. EPA (1992).

Regardless of the method(s) used, horizontal location surveys should be based on established control points. A network of horizontally (and vertically) located control points has been established and is continually maintained by the National Oceanic and Atmospheric Administration (NOAA) through its National Ocean Survey (formerly U.S. Coast and Geodetic Survey). The old horizontal datum, called the North American Datum of 1927 (NAD27), has been replaced with the newer datum of 1983 (NAD83). The system of horizontal control points has established latitude and longitude positions and provides the basis for the coordinate grid systems used by many States. Currently the database of US Geological Survey (USGS) topographic quadrangles maintained by the GIS group in Athens is based on the older NAD27 datum, therefore if data points collected are to be overlain on one of the NAD27 USGS quad sheets, then that data needs to also be collected in the NAD27 format. If the data is not going to be overlain on a NAD27 map, then data should be collected in the NAD83 format.

Existing information on horizontal control stations or coordinate grid data and their exact locations may be obtained from local, state, or federal departments or agencies. Typically, engineering or public works departments of counties, cities, or towns may have data on file that is near the particular site being investigated. State or federal agencies which are good sources of useful data include:

- State highway or transportation departments
- State geodetic or land surveying offices
- State natural or water resources bureaus
- State geological surveys
- NOAA/National Ocean Survey
- United States Geological Survey
- Corps of Engineers, Department of the Army
- Soil Conservation Service
- Tennessee Valley Authority
- Bureau of Land Management

When exact locations of sampling points or other physical features at a site are needed, surveying methods must be based on existing control data. If unavailable at the time of the investigation, and if necessary, the site property boundary survey, legal description, and any physical property corners or monuments must be established by a professional Registered Land Surveyor (RLS).

If no existing control data exists in the site vicinity, an arbitrary point or points may be established at a permanent location, e.g., set a nail or spike beneath the ground or set a nail and cap

in asphalt or foundation. Coordinates for those points (and, therefore all other points) should be determined at a later date. Location of all control data used and all field measurements should be recorded in the field logbook.

17.2.2 Equipment Available

The following equipment is available for field use in conducting horizontal surveys:

- Topcon GTS-2, total station theodolite/electronic distance meter (EDM)
- 2 - Trimble Pathfinder Pro XRS 12 channel GPS receiver
- 2 - Trimble GeoExplorer II 8 channel GPS receivers
- 2 - Trimble Ensign 6 channel GPS receivers
- 2 - Garmin Etrex Legend 12 channel GPS receivers
- 6 - Apelco fixed mount GPS receivers in boats
- steel tape
- cloth tape
- right angle prism
- compass

17.2.3 Equipment Quality Control Procedures

Field surveying methods using appropriate and available equipment should be made only by those personnel who have been trained to use them. Field investigators must be trained and checked out in surveying procedures by qualified staff before using this equipment.

Maintenance and calibration procedures used for all surveying equipment should follow equipment manufacturers specifications. More detailed specific equipment quality control procedures may be found in the *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*, 2001.

17.2.4 Procedures for Traversing

When traverse methods are used, at least two stations or control points of known horizontal location (expressed in terms of a local or State Plane coordinate system) must be in the site vicinity. These control points can usually be set for the specific site by a governmental agency or registered land surveyor.

The total station theodolite, which measures horizontal angles, vertical and/or zenith angles, and slope distances, is set up over an existing control point. The rodman has either a range pole equipped with a reflector prism (single or triple) or a tripod with the reflector prism. The difference in location between the point where the theodolite is set up and the point where the prism is held is determined trigonometrically. A compass and measuring tape could also be used to reference field measurements to a map or vice versa. A much greater level of detail and examples of applications can be found in the *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*, 2001.

17.2.5 Procedures for Differential GPS

Differential GPS involves the use of two or more multichannel receivers. One or more

receivers are used as the rover receiver(s) and one is used as the base station. The base is set up at a control point of known horizontal location (usually expressed in terms of latitude, longitude, and elevation). Triangulated coordinate positions from the satellites are recorded at the base and then used for the development of a correction factor to be applied to the roving GPS units.

Realtime differentially corrected positions are available for branch riverine, estuarine and marine surveys by means of boat mounted DGPS units. These units receive signals from USCG land based transmitters for realtime differential corrections. Coverage for the USCG transmitters actually extends inland for several hundred miles and includes stations located on the Mississippi River, therefore most of US-EPA Region 4 is covered by realtime differential coverage.

All professional staff and field technicians must be trained in the use of the GPS equipment by qualified staff before using this equipment. Specific procedures on the operation and setup of the GPS equipment are described in detail in the operations manuals for each of the instruments.

17.2.6 GPS Accuracy

Measurement accuracy is dependent upon a number of factors including: 1) the instrument employed, 2) signal strength, 3) selective availability, and 4) differential corrections. The following is an approximation of the accuracy for measurements made with Branch equipment (assuming selective availability remains disabled by DOD).

Handheld non-differentially corrected GPS -----	10 Meters
Boat Mounted Realtime DGPS -----	1 Meter
Rover with base station differential correction –	<1 Meter

17.3 Vertical Location (Elevation) Surveys

17.3.1 Introduction

The field of surveying that pertains to measuring the relative differences in elevation of two or more points is called "running levels" or "Leveling". The two most commonly used methods are Differential Leveling and Trigonometric Leveling. Differential leveling is the most precise and easiest method because it utilizes "level" measurements with simple addition and subtraction. Trigonometric leveling is slightly less precise and more difficult as it uses vertical angle and distance measurements combined with the principles of trigonometry. Global Positioning System (GPS) equipment can obtain elevation measurements, however this new technology is less accurate than horizontal measurements and is not recommended for vertical locations.

Regardless of the method(s) used, elevation surveys should be based on established control points. A network of vertically (and horizontally) located control data points has been established and is continually maintained by the National Oceanic and Atmospheric Administration (NOAA) through its National Ocean Survey (formerly U. S. Coast and Geodetic Survey). The system of vertical control points, or Benchmarks (B.Ms.), are referenced to a surface of fixed and precisely known elevation above mean sea level and is referred to as the datum or datum plane. The datum for vertical control (elevation) is called the National Geodetic Vertical Datum of 1929 (NGVD29), formerly known as the 1929 sea level datum, or the soon to be established North American Vertical Datum of 1988 (NAVD88).

Sources of existing information on benchmark data and their locations may be obtained from

local, state, or federal departments or agencies. Typically, engineering or public works departments of counties, cities, or towns may have data on file that is near the particular site being investigated. State or federal agencies that are good sources of useful data include:

- State highway or transportation departments
- State geodetic or land surveying offices
- State natural or water resources bureaus
- State geological surveys
- NOAA/National Ocean Survey
- United States Geological Survey
- Corps of Engineers, Department of the Army
- Soil Conservation Service
- Tennessee Valley Authority
- Bureau of Land Management

When the exact elevations of sampling locations or other physical features are needed, benchmarks of precisely known elevation should be used when leveling. If necessary, a registered land surveyor could be requested to set at least two vertical control points or benchmarks. The vertical control points should have established elevations referenced to NGVD29 or NAVD88.

If no benchmark is located in the site vicinity, an arbitrary temporary benchmark should be established on a permanent location, e.g., bridge wingwall, foundation, or a nail or spike in a tree or a metal rod driven into the ground. The elevation of the temporary benchmark (and, therefore all other points) could be determined at a later date. As with all field work, the location of benchmarks used should be shown on the site sketch map and all field measurements should be recorded in the field logbook.

17.3.2 Equipment Available

The following equipment is available for field use in conducting elevation surveys in support of site investigations:

Differential Leveling

- Sokkia B20, K&E or Lietz B2C, precision automatic level

Trigonometric Leveling

- Topcon GTS-2, total station theodolite/electronic distance meter (EDM)
- cloth or steel tape
- compass

17.3.3 Equipment Quality Control Procedures

Field surveying methods using this equipment should be made only by those personnel who have been trained to use them. All field investigators must be trained and checked out in surveying procedures by qualified staff before using this equipment.

The specific maintenance and calibration procedures should follow manufacturers specifications.

17.3.4 Procedures for Differential Leveling

The level, or instrument, is set up by the instrument operator at a location not more than 300 feet from the benchmark and at a height above the benchmark and the next point(s). The rodman holds the rod as plumb (vertical) as possible and rocks the rod through vertical on the benchmark so that the instrument operator can read where the horizontal cross-hair in the telescope of the level intersects the graduations on the rod. The differences between the first reading obtained from the benchmark and subsequent readings at point of interest is the difference in elevation between the point and the benchmark. Note that the distance between each sighted reading should not ordinarily exceed 300 feet with turning point back sight and foresight distances deviating no more than 50 feet from one another.

When practical, leveling should be conducted to form a closed circuit. That is, the level circuit or loop should close back in close agreement to a benchmark. If the level circuit does not close within the accuracy required for the survey as defined in the plan of study, then the level circuit must be repeated until this accuracy is attained. Fourth order accuracy, with an accepted tolerance defined by the formula: $0.10 \text{ foot} \times (\sqrt{\text{number of miles run}})$ is the recommended requirement. First, second third and fourth order accuracy tolerance is defined by, 0.02, 0.04, 0.05 and 0.10 foot respectively $\times (\sqrt{\text{number of miles run}})$.

17.3.5 Procedures for Trigonometric Leveling

The total station theodolite, or instrument, is usually set up above a benchmark and the elevation of the instrument (H.I.) must be obtained. The rodman has either a level rod or a range pole equipped with a reflector prism. The difference in elevation between the theodolite and the point of interest is determined trigonometrically. A compass with a clinometer and a measuring tape could also be used for field measurements or as a map reference.

17.4 Bathymetry

17.4.1 Procedures

Recording fathometers are used to provide bathymetric traces of water depths. Because water depths are time dependent (especially in tidal areas), the date and time of all traces should be noted. Operation manuals provide operation and calibration procedures to be followed. In particular, tide and draft adjustments provide datum calibration in regard to the respective tidal amplitude and sensor probe depth. All traces should be noted with transect description, direction of travel, pertinent reference points, and GPS (if available) and then indexed to a site map. When working in tidal areas, a water stage recorder should be positioned or referenced to provide a histogram of water levels to correlate with the bathymetric trace.

17.4.2 Equipment Available

The following equipment is available for bathymetric surveys:

- recording fathometers;
- water level recorder and/or referenced gaging station(s);
- calibrated sounding line(s); and
- GPS instrumentation.

17.4.3 Equipment Quality Control Procedures

All equipment used for bathymetric studies should be maintained and calibrated in accordance with manufacturers specifications.

17.5 References

1. United States Environmental Protection Agency. 1991. Information Resource Management (IRM) manual, Chapter 13: Location Data
2. United States Environmental Protection Agency. 1992. GIS Technical Memorandum 3: Global Positioning Systems Technology And Its Application In Environmental Programs. US EPA Document # EPA/600/R-92/036.

SECTION 18
FIELD INSTRUMENT, CALIBRATION AND MAINTENANCE

PERFORMANCE OBJECTIVE:

To measure physical and/or chemical characteristics of a sample that are representative of field conditions as they exist at the time of sample collection:

- By selecting the appropriate meter/instrument
- By properly calibrating each meter/instrument.

18.1 Introduction

The information contained within this section pertains to the equipment available for *in situ* data collection including but not limited to pH, temperature, dissolved oxygen, salinity, turbidity, and specific conductance. Several different meters may be available for measuring the same parameter. The field investigator is responsible for choosing the appropriate equipment for measuring physicochemical parameters. Proper maintenance and calibration are required for insuring the quality of the data collected with *in situ* instrumentation. General information and calibration and maintenance procedures are presented in this section. The manufacturer's recommendations should be followed for detailed instructions on calibration and maintenance procedures.

18.2 YSI® 6920 Multi-Parameter Water Quality Monitor

18.2.1 Introduction

The YSI® 6920 Environmental Monitoring System is a multiparameter, water quality measurement and data collection system. Measurement parameters include:

- Dissolved Oxygen
- Specific Conductance
- Salinity
- Total Dissolved Solids
- Resistivity
- Temperature
- pH/ORP
- Depth/Level
- Turbidity.

The YSI® 6920 sonde can be used for profiling or continuous monitoring of water conditions. The sonde operates using eight internal AA-size alkaline batteries. The YSI® 6920s are programmed using PC-based software which comes with each unit. It should be noted that sondes equipped with level sensors rather than depth sensors use vented cables. The vented level eliminates error due to changes in barometric pressure. This is accomplished by using a sensor that is vented to the outside atmosphere by way of a tube that runs up through the sonde and cable. Never expose the sonde or the cable to the atmosphere for more than a few minutes without an active desiccant system in place.

18.2.2 Calibration

The YSI® 6920 should be calibrated for each parameter that will be measured according to the manufacturer's recommendations in the YSI® 6920 Multi-Parameter Water Quality Monitor Instruction Manual and Service Manual. To insure the best possible calibration, all sensors should be immersed in the calibration solutions. During many calibrations, readings from other sensors (e.g., temperature probe) are factored into the calibration algorithm. The sensors should be thoroughly rinsed between calibration solutions. Clean, absorbent paper towels or cotton cloths should be used to dry the sensor guard between rinses and calibration solutions. Drying the probes and guards reduces carry-over contamination of calibration solutions and increases the accuracy of the calibration. It is imperative that port plugs are installed in all ports where probes are not installed. It is extremely important to keep the electrical connectors dry.

18.2.3 Maintenance

Maintenance and storage procedures for each probe associated with the YSI® 6920 sonde vary and are found in Section 7 and Appendix G, respectively, of the YSI® 6920 Multi-Parameter Water Quality Monitor Instruction Manual and Service Manual.

18.3 YSI® Model 57/58 Dissolved Oxygen Meter

18.3.1 Introduction

The YSI® Model 58 Dissolved Oxygen Meter is used by EAB for collecting *in situ* dissolved oxygen measurements. Dissolved oxygen is indicated in mg/l (1 mg/l \approx 1 part per million) or in % air saturation. The meters are powered by eight C-cell batteries.

18.3.2 Calibration

Prior to calibration, the meters should be turned on for 15 minutes for the probe to stabilize. A 15 minute warm-up is necessary whenever the meter has been off or the probe has been disconnected. The meters can be calibrated using either the Winkler Titration method or air calibration. See the YSI® Model 57/58 Dissolved Oxygen Meter Instruction Manuals for detailed calibration instructions.

18.3.3 Maintenance

The batteries should be checked and/or changed before each use. The probe membranes and the electrolyte solution should be changed before each study. Refer to the YSI® Model 57/58 Dissolved Oxygen Meter Instruction Manuals for instructions regarding probe maintenance.

18.4 Hydrolab Datasonde® 3 and Datasonde® 4a

18.4.1 Introduction

The Hydrolab Datasonde® 3 (DS3) and Datasonde® 4a (DS4a) have multiparameter probes capable of measuring the following parameters:

- dissolve oxygen

- pH/ORP
- temperature
- conductivity
- turbidity
- salinity
- depth
- total dissolved solids

Both the DS3 and DS4a can be used for profiling or deployed for continuous monitoring of water conditions. The DS3 is powered by 10 internal AA cell batteries. The DS4a is powered by 8 internal C cell batteries.

18.4.2 Calibration

The DS3 and DS4a should be calibrated for each parameter to be measured by exposing the sensors to known standards and adjusting the unit to agree with the standard. Calibration should be conducted according to the manufacturer's recommendations in the Datasonde® 3 and Datasonde® 4a User's Manuals.

18.4.3 Maintenance

Maintenance procedures for the two units are very similar. However, the individual operating manuals should be consulted for detailed maintenance procedures. At a minimum, the membranes and electrolyte solution associated with the dissolved oxygen probes should be checked and changed as needed. Battery voltages should be monitored and batteries replaced as needed. The pH reference solution should be checked and changed as needed.

18.5 Hydrolab Quanta® and H20®

18.5.1 Introduction

The Hydrolab Quanta® and H20® water quality monitoring units have multiparameter probes capable of measuring the following:

- dissolved oxygen
- temperature
- pH/ORP
- conductivity
- total dissolved solids
- depth
- salinity.

The Quanta® is powered by 3 internal C cell batteries. The H20® is powered by an external Hydrolab rechargeable 12 V gel cell battery. Both the Quanta® and H20® are capable of *in situ* readings but neither are able to store the data electronically.

18.5.2 Calibration

Both units are calibrated by exposing the sensors to known standards and adjusting the

readout from the unit to agree with the standard. Consult the Hydrolab Quanta® Water Quality Monitoring System Operating manual (February 2000) and the Hydrolab Multiparameter Water Quality Monitoring Instruments H20® Operating manual for calibration instructions.

18.5.3 Maintenance

Maintenance procedures for the two units are very similar. However, the individual operating manuals should be consulted for detailed maintenance procedures. At a minimum, the membranes and electrolyte solution associated with the dissolved oxygen probes should be checked and changed as needed. Battery voltage in the Quanta® should be monitored and batteries replaced as needed. The H20® rechargeable battery should be charged daily or as needed. The pH reference solution should be checked and changed as needed.

18.6 Turner Design Model 10 and 10-AU Field Fluorometers

18.6.1 Introduction

Fluorometers are used to detect fluorescent tracers during time-of-travel, dye dispersion, dye dilution and reaeration studies. Fluorometric analysis is based on quantitation of the ability of fluorescent materials to absorb light at one wavelength and convert it into light at a longer wavelength. The fluorescent tracer most commonly used by EAB is Rhodamine WT dye.

The two generations of fluorometers; namely, the Model 10 and Model 10AU field units can be operated in either a cuvette or flow through mode. The earlier Model 10 fluorometer is an analog unit whereas the Model 10AU is a digitally equipped temperature compensating unit. Both fluorometers can be operated with either 110 volt alternating current or 12 volt direct current.

Operation of the fluorometer consists of two basic steps:

1. Calibration; and
2. Operation.

18.6.2 Calibration

Calibration consists of setting the basic sensitivity of the instrument to a level appropriate to the samples and the blank. When direct concentration readout is desired, a sample of known concentration must be used as a standard. The following should be considered during fluorometer calibration:

- Temperature fluctuation of the sample can produce significant errors in fluorometer readings. Therefore, when using the fluorometer units, standards, blanks and samples should preferably be held at the same temperature or the fluorescence readings should be corrected to what they would be at some common temperature.
- At dye concentrations below 500 ppb dye tracer, a single-point calibration (one standard and a blank) may be used for calibration..
- The basic sensitivity of the fluorometers was set by the manufacturer for optimum

performance. If the cuvette size, filters or lamp are changed, the sensitivity will need to be adjusted.

Refer to the Model 10-AU-005 Field Fluorometer User's Manual for detailed instructions regarding calibration.

18.6.3 Maintenance

Maintenance should be performed according to the procedures found in Section 4.4 of the Model 10 unit and Appendix 7 of the Model 10-AU User's Manual.

18.7 ADCP Current Meters

18.7.1 Introduction

The SESD/EAB collection of Acoustic Doppler Current Profiling (ADCP) meters currently includes two 600 kHz Workhorse Sentinels (internal battery) and one 600kHz Workhorse Rio Grande (external battery). These ADCP meters measure current speed and direction. Both types of meters can be utilized in profiling applications while the Sentinel units can also be used in unmanned deployments. In addition, bottom tracking capabilities have been installed on all three meters to allow for flow measurement. More discussion on ADCP use in current and flow measurement is provided in Chapter 15 (Hydrological Studies) of this SOP.

18.7.2 Calibration

The internal compass in each unit was calibrated according to manufacturer's specifications prior to initial use. No additional routine calibration is required; however, the compass should be recalibrated in the Sentinel units following battery replacement. The compass should be calibrated according to the manufacturer's procedures (RDI Workhorse ADCP Technical Manual, p. 4-7).

18.7.3 Maintenance

All equipment shall be visually inspected prior to deployment. Chapter 4 of the RDI Workhorse ADCP Technical Manual provides details on the maintenance of the instrument including proper procedures for inspection and maintenance of the O-rings and transducer faces as well as for end cap and transducer assembly removal and replacement, if needed.

18.8 Stevens Axsys™ System Stage Recorder

18.8.1 Introduction

The Stevens Axsys™ System consists of data acquisition products based on the Axsys MPU (monitoring/processing unit). A submersible pressure transducer is connected to the Axsys MPU SDI-12 data bus to measure depth/level. The Axsys MPU is capable of storing date, time and depth/level measurements. Recording time intervals range from once per second to once per day. The data is processed, formatted and stored in non-volatile Flash memory. Approximately 60,000 readings can be stored in the basic MPU. Data can be extracted through the serial port from the MPU. Utility programs such as Stevens MODTERM® and LOGTERM® can be used to

format the data for standard word processing and spread sheet programs.

18.8.2 Calibration

No calibration is necessary.

18.8.3 Maintenance

The following maintenance should be performed on each unit after use:

- The seal on the lid of the MPU should be checked periodically to maintain the condition of the gasket. Replace the gasket when damaged.
- Care should be taken to prevent moisture from entering the MPU when the lid is open.
- Clean the case of the MPU with a clean, damp cloth and mild liquid soap when necessary. The pressure transducer can be cleaned in the same manner, taking care not to damage the tip where the sensor is located.

18.9 Tipping Bucket Rain Gauge

18.9.1 Introduction

The Met One Instruments Model 370 Tipping Bucket Rain Gauge measures rainfall on a continuous basis. Water is drained each time an internal bucket fills with 0.01 inches of rainfall. Each time the internal bucket is filled a pulse is sent to a datalogger for counting. The tipping bucket rain gauge can be connected to the Stevens Axsys System for logging of the data.

When selecting a location for the rain gauge, choose a site where the height of any nearby trees or other objects above the sensor is no more than about twice their distance from the sensor. A uniform surrounding of objects is beneficial as a wind break. Nonuniform surroundings create turbulence which affects accuracy. Once a location has been chosen and the rain gauge installed, it should be leveled.

18.9.2 Calibration

The sensor is factory calibrated; recalibration is not necessary unless damage has occurred or the adjustment screws have loosened.

18.9.3 Maintenance

Approximately every six months, the funnel and buckets should be cleaned. Do not use any lubricants on the pivots. Lubricant may attract dust and dirt and cause wear of the bearings. Verify that the buckets move freely and that the data logger registers 0.01" for each bucket tip.

18.10 Recording Pyrheliometer

18.10.1 Introduction

Radiant energy is a primary factor governing aquatic community primary production rates. When assessments of total community oxygen metabolism, water column oxygen metabolism, and benthic gross and net primary production are being conducted, it is essential that daily records of total available solar energy be acquired. Often, such measurements will span several days, or be conducted during different segments of a daily photoperiod. Accordingly, records of radiant energy must be available and used to normalize primary production rates to a standard energy level for data comparability.

A recording pyrhelimeter or pyranograph should be used. The pyranograph can be equipped with gears for recording solar energy over a 24 hour or 8 day period. All data are recorded in langleys/hour or gram calories/hour.

18.10.2 Calibration

The pyranographs used in the recording of radiant energy require no calibration unless the instrument is damaged. Such repair and calibration must be conducted by the manufacturer. The instrument does require periodic adjustment for zero. Zero adjustment should be checked prior to used in each field activity by bringing the instrument indoors and allowing it to stand for one hour and then reading the pen position. Should zeroing of the instrument be required, specific instructions contained in the manufacturer's manual which accompanies the pyranograph should be followed.

18.10.3 Maintenance

The dark bimetallic strip should be observed for blackness prior to putting each strip chart in place, and observing the time at 2-hour intervals over the course of a working day. Deviations in accuracy beyond one hour require clock replacement. The dome must be kept clean and unshaded. Daily morning and afternoon checks of the instrument should be performed during field operation. Time of the check versus pyranograph time should be recorded along with climatic conditions.

18.11 Irradiameter (Marine Photometer)

18.11.1 Introduction

Percent light transmission and light extinction coefficients are obtained using an irradiameter (marine photometer). The instrument consists of a monitor with either two analog scales or two digital scales which are coupled to two photo cells, one for ambient light (deck cell) and the other for underwater light (sea cell).

Instrument readout is based on $\mu\text{W}/\text{cm}^2/\text{nm}$ and multiples thereof including 10, 30, 100, 300, 1K, 3K and 10K. Field readings are recorded directly from meter observations to the nearest tenth accompanied by the scale setting (10-10K).

18.11.2 Calibration

Presurvey checkout and calibration of the underwater irradiameter should be conducted in accordance with the manufacturer's manual. Checkout procedures involve calibration (zeroing)

and battery checks for power supply before and after field use each day or more frequently if meter performance is suspected of going out of control during a monitoring period.

General guidance for checking instrument performance is as follows: perform a battery check using the appropriate switch on the monitor; after verifying sufficient battery power, zero each analog scale by assuring that the black rubber cap and red cover are providing complete darkness for the deck and sea cells. (Use any other available material to assure complete darkness of the cells if necessary.) Use the zero adjustments as required (Note: Direction of turn for the zero knob is counter to the expected direction.) Once zero is acquired for each cell, be sure that each scale is set on 10K; uncover each cell and place them side by side in a completely level position and read each meter. If meter readings vary more than 0.5, perform an adjustment to synchronize the two meters by opening the monitor and using a small screw driver to adjust the electronic "pots" inside the unit that are appropriately marked for the deck cell and sea cell. (NOTE: a tolerance of 0.5 difference between the two analog meters is permissible due to the difficulty of assuring precise leveling when working on boats. Any deviation greater than 0.5 should be corrected using the above guidance.)

18.11.3 Maintenance

Maintenance of the instrument is also specifically outlined in the manual and should be performed after each field activity upon returning to the laboratory. Calibration, other than zeroing or analog meter (deck vs. sea cell) synchronization, must be performed by the manufacturer based on (1973) National Bureau of Standards Scale of Spectral Irradiance.

Presurvey checkout information should be recorded in the field data book for each individual survey. Recalibration information accompanies the instrument when returned from the factory and is placed in the operation manual.

18.12 ISCO 6700 Automatic Sampler

18.12.1 Introduction

The ISCO 6700 Automatic Sampler can be used to collect water and wastewater samples. The samples are collected by an internal vacuum pump. Therefore, there is a suction head limitation of 28 at 30 inHg. The sampler is powered by an external 12V DC battery and can be programmed to collect samples of various volumes at varying times. The samplers are available with several different sampler base configurations. Currently, EAB's sampler bases have 24-1000 ml polyethylene bottles. Consult the ISCO 6700 Portable Sampler Instruction Manual for detailed instruction regarding programming and operation of the sampler.

18.12.2 Calibration

The sampler is calibrated based on the length of the suction tubing line and the amount of suction head. The 6700 delivers the sample by counting pump revolutions and automatically compensating for the suction head. Consult the ISCO 6700 Portable Sampler Instruction Manual for detailed instruction regarding calibration procedures.

18.12.3 Maintenance

New suction tubing line and sample discharge tubing should be installed in the sampler at each new sampling location. The sample discharge tubing must be the exact length recommended in the 6700 Instruction Manual based upon the sampler base configuration. If not, sample volumes will be adversely affected. The pump tubing can be reused. However, appropriate decontamination procedures as outlined in Appendix C of this SOP should be followed. The pump tubing should be inspected regularly and replaced when it cracks or appears worn. Detailed maintenance procedures are contained in the ISCO 6700 Portable Sampler Instruction Manual.

18.13 ISCO 4210 and 4220 Flowmeters

18.13.1 Introduction

Both the ISCO 4210 and 4220 Flowmeters are capable of measuring level and converting the level measurements to flow data if the appropriate measuring device is used in conjunction with the flowmeter. The ISCO 4210 and 4220 Flowmeters operate under the same principle but with different level sensing devices. The 4210 Flowmeter uses an ultra-sonic probe which is mounted several feet above the surface of the water. The 4220 Flowmeter uses a submerged probe with a pressure transducer. Each of EAB's flowmeters is equipped with a 4200T Modem. The modem enables the user to transmit stored data over standard dial-up telephone lines. The flowmeters can be programmed to work in conjunction with the ISCO 6700 Samplers for sampler initialization. The flowmeters are powered by an external 12V DC battery.

18.13.2 Calibration

No calibration is necessary. The flowmeters can be programmed to measure stage rather than level by entering the stage of the stream or waterbody based upon tape down measurements as described in Section 14.2.1 of this SOP.

18.13.3 Maintenance

Very little maintenance is required for the flowmeters. The desiccants inside the flowmeter case should be recharged when the color changes from blue to pink. The submerged probes should be cleaned with a soft cloth and mild soap to remove build-up from submersion. Care should be taken not to damage the pressure transducer. Consult Chapter 5 of the ISCO 4220 Flowmeter Instruction Manual for detailed instructions regarding the cleaning of the probe.

18.14 LaMotte 2020 Turbidimeter and Hach Model 2100P Portable Turbidimeter

18.14.1 Introduction

The LaMotte 2020 Turbidimeter and the Hach Model 2100P Portable Turbidimeter are portable, microprocessor controlled nephelometers. The LaMotte 2020 measures turbidity over a range of 0-1100 Nephelometric Turbidity Units (NTU). The 2020 Turbidimeter is powered by either a 9 V alkaline battery or an AC power adapter. The Hach 2100P measures turbidity over a range of 0-1000 NTU and requires 4 AA cell batteries. The turbidity is determined by filling a vial (which comes with the units) with sample, wiping the outside of the vial dry with a clean, lint-free tissue and placing the vial in the turbidimeter. The lid of the meter should be closed and the "read" button pushed.

18.14.2 Calibration

A one point calibration is performed on the LaMotte 2020 and the Hach 2100P. A standard in the range of the samples to be tested should be used. Consult the LaMotte 2020 Turbidimeter Instruction Manual or the Hach Model 2100P Portable Turbidimeter Instruction Manual for detailed instructions regarding calibration of the instrument.

18.14.3 Maintenance

It is important that the sample vials and the calibration vials are clean, dry and unscratched. Scratches on the vials can cause interferences with the lamps and result in erroneous readings.

The batteries should be replaced as necessary. If readings in the LaMotte 2020 become unstable during calibration, the tungston lamp within the meter may need to be replaced. The tungston lamp has a life of approximately 800 hours. The turbidimeter must be returned to the manufacturer for lamp replacement.

The lamp in the Hach 2100P can be replaced by the user. Instructions for lamp replacement can be found in the Hach 2100P Instruction Manual.

APPENDIX A
RECOMMENDED CONTAINERS, HOLDING TIMES, & PRESERVATION

ANALYTICAL GROUP	Soil/Sediment			Water/Wastewater ¹		
	Container	Preservative	Holding Time	Container	Preservative	Holding Time
BIOLOGICAL						
Bacteriological*	--	--	--	B	I	6 hours
Toxicity, Acute	--	--	--	CU	I	36 hours
Toxicity, Chronic	--	--	--	CU	I	36 hours
INORGANICS						
pH*	8G	NA	--	--	--	--
Ash free dry weight	4P	NA	--	--	--	--
Particle Size	WP	Freeze	--	--	--	--
Bed Load Samples	BJ	I	--	--	--	--
Freshwater TSS	--	--	--	SM	I	7 days
Freshwater TDS	--	--	--	SM	I	7 days
TOC	--	--	--	SM	I/S	28 days
AGPT	--	--	--	2LP	I	24 hours to filter then freeze
Chlorophyll	--	--	--	Filter kit	I/F	3.5 weeks
Krypton Gas	--	--	--	Vacuum bottle	I	NS
Ammonia (preserved)	--	--	--	8G	S/I	28 days

ANALYTICAL GROUP	Soil/Sediment			Water/Wastewater ¹		
	Container	Preservative	Holding Time	Container	Preservative	Holding Time
Ammonia (un-preserved)	--	--	--	8G	I	48 hours
Residual Chlorine*	--	--	--	SM	NA	Immediately
Turbidity	--	--	--	SM	I	2 days
Conductivity	--	--	--	SM	I	28 days ¹¹
Temperature	--	--	--	SM	NA	Immediately
BOD5	--	--	--	HP ²	I	2 days
Solids Series	--	--	--	HP	I	7 days
Settleable Solids	--	--	--	HP	I	2 days
Nutrients (N,P) preserved	8G	I	NS	HP	S/I	28 days
Nutrients (N,P) un-preserved	--	--	--	HP	NA	48 hours
Chloride	--	--	--	LP	NA	28 days
Ortho-P	8G	I	NS	LP	I ⁴	2 days
Dissolved P	--	--	--	LP	S ⁴ /I	28 days
COD	8G	I	NS	LP	S/I	28 days
Alkalinity	--	--	--	LP	I	14 days
Color	--	--	--	GP/LP	I	2 days
Oil & Grease*	--	--	--	LG	S/I	28 days
Metals	8G	I	180 days	LP	N	180 days
Mercury	8G	I	180 days	LP	N	28 days

ANALYTICAL GROUP	Soil/Sediment			Water/Wastewater ¹		
	Container	Preservative	Holding Time	Container	Preservative	Holding Time
Mercury - Ultra Trace Level	4P	I	NS	PTFE	HCl	28 days
Chromium VI	--	--	--	LP	I	1 day
Cyanide	--	--	--	LP	A ⁵ /C ⁶ /I	14 days
Sulfate	--	--	--	LP	I	28 days
Sulfide	--	--	--	LP	Z/C ⁷ /I	7 days
Porewater Sulfide	Pre-preserved syringe	Z/C ⁷ /I	7 days	--	--	--
Nitrite	--	--	--	LP	I	2 days
Nitrate	--	--	--	HP	I	2 days
Hardness	--	--	--	LP	N	180 days
Fluoride	--	--	--	LP	NA	28 days

ANALYTICAL GROUP	Soil/Sediment			Water/Wastewater ¹		
	Container	Preservative	Holding Time	Container	Preservative	Holding Time
ORGANICS						
VOCs*	2G	I	14 days	V	H or B ⁸ /I	14/7days ¹³
Extractables ¹⁶	8G	I	54 days ¹⁵	GG	I ⁹	47days ¹⁴
Dioxins ¹⁷	8G	I	75 days ¹²	LA ³	I ¹⁰	75 days ¹²
Phenols	--	--	--	LA	F/S/I	28 days
Org Halide (TOX)	8G	I	28 days	LA	S/I	28 days
TISSUES/PLANT MATERIAL						
ANALYTICAL GROUP	Container		Preservative	Holding Time		
Organic Analysis Tissue Samples	Wrap in aluminum foil and place in plastic bag/or 8G		Freeze	Not Specified		
Mercury Tissue Samples	Wrap in aluminum foil and place in plastic bag/ or 8G		Freeze	28 days		
Metals and other Inorganic Compounds Tissue Samples	Wrap in aluminum foil and place in plastic bag/ or 8G		Freeze	Not Specified		
Periphyton	4P		Freeze	Not Specified		
Floc	4P		Freeze	Not Specified		
Sawgrass/Cattails	4P		Freeze	Not Specified		

APPENDIX B STANDARD FIELD CLEANING PROCEDURES

PERFORMANCE OBJECTIVE:

- To remove contaminants of concern from sampling, and other field equipment to concentrations that do not impact study objectives using a standard cleaning procedure.

B.1 Introduction

Cleaning procedures in this appendix are intended for use by field personnel for cleaning sampling and other equipment in the field. Emergency field sample container cleaning procedures are also included; however, they should not be used unless absolutely necessary. Cleaning procedures for use at the Field Equipment Center (FEC) are in Appendix C.

Sampling and field equipment cleaned in accordance with these procedures must meet the minimum requirements for Data Quality Objectives (DQO) definitive data collection. Alternative field decontamination procedures may be substituted as outlined in Section 4.5 when samples are to be analyzed for data uses at a lower DQO level. Deviations from these procedures should be documented in the approved study plan, field records, and investigative reports.

These are the materials, methods, and procedures to be used when cleaning sampling and other equipment in the field.

B.1.1 Specifications for Cleaning Materials

Specifications for standard cleaning materials referred to in this appendix are as follows:

- Soap shall be a standard brand of phosphate-free laboratory detergent such as Liquinox[®]. Use of other detergent must be justified and documented in the field logbooks and inspection or investigative reports.
- Solvent shall be pesticide-grade isopropanol. Use of a solvent other than pesticide-grade isopropanol for equipment cleaning purposes must be justified in the study plan. Otherwise its use must be documented in field logbooks and inspection or investigation reports.
- Tap water may be used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.
- Analyte free water (deionized water) is tap water that has been treated by passing through a standard deionizing resin column. At a minimum, the finished water should contain no detectable heavy metals or other inorganic compounds (i.e., at or above analytical detection limits) as defined by a standard Inductively Coupled Argon Plasma Spectrophotometer (ICP) (or equivalent) scan. Analyte free water obtained by other methods is acceptable, as long as it meets the above analytical criteria.

- Organic/analyte free water is defined as tap water that has been treated with activated carbon and deionizing units. A portable system to produce organic/analyte free water under field conditions is available. At a minimum, the finished water must meet the analytical criteria of analyte free water and should contain no detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels as determined by the Region 4 laboratory for a given set of analyses. Organic/analyte free water obtained by other methods is acceptable, as long as it meets the above analytical criteria.
- Other solvents may be substituted for a particular purpose if required. For example, removal of concentrated waste materials may require the use of either pesticide-grade hexane or petroleum ether. After the waste material is removed, the equipment must be subjected to the standard cleaning procedure. Because these solvents are not miscible with water, the equipment must be completely dry prior to use.

Solvents, laboratory detergent, and rinse waters used to clean equipment shall not be reused during field decontamination.

B.1.2 Handling and Containers for Cleaning Solutions

Improperly handled cleaning solutions may easily become contaminated. Storage and application containers must be constructed of the proper materials to ensure their integrity. Following are acceptable materials used for containing the specified cleaning solutions:

- Soap must be kept in clean plastic, metal, or glass containers until used. It should be poured directly from the container during use.
- Solvent must be stored in the unopened original containers until used. They may be applied using the low pressure nitrogen system fitted with a Teflon[®] nozzle, or using Teflon[®] squeeze bottles.
- Tap water may be kept in clean tanks, hand pressure sprayers, squeeze bottles, or applied directly from a hose.
- Analyte free water must be stored in clean glass, stainless steel, or plastic containers that can be closed prior to use. It can be applied from plastic squeeze bottles.
- Organic/analyte free water must be stored in clean glass, Teflon[®], or stainless steel containers prior to use. It may be applied using Teflon[®] squeeze bottles, or with the portable system.

Note: Hand pump sprayers generally are not acceptable storage or application containers for the above materials (with the exception of tap water). This also applies to stainless steel sprayers. All hand sprayers have internal oil coated gaskets and black rubber seals that may contaminate the solutions.

B.1.3 Disposal of Solvent Cleaning Solutions

Procedures for the safe handling and disposition of investigation derived waste (IDW),

including used wash water, rinse water, and spent solvents are in Section 4.8.

B.1.4 Equipment Contaminated with Concentrated Wastes

Equipment used to collect samples of hazardous materials or toxic wastes or materials from hazardous waste sites, RCRA facilities, or in-process waste streams should be field cleaned before returning from the study. At a minimum, this should consist of washing with soap and rinsing with tap water. More stringent procedures may be required at the discretion of the field investigators.

B.1.5 Safety Procedures for Field Cleaning Operations

Some of the materials used to implement the cleaning procedures outlined in this appendix can be harmful if used improperly. Caution should be exercised by all field investigators and all applicable safety procedures should be followed. At a minimum, the following precautions should be taken in the field during these cleaning operations:

- Safety glasses with splash shields or goggles, and latex gloves should be worn during all cleaning operations where high pressure equipment or solvents are used.
- Solvent rinsing operations will be conducted in the open (never in a closed room).
- No eating, smoking, drinking, chewing, or any hand to mouth contact should be permitted during cleaning operations.

B.1.6 Handling of Cleaned Equipment

After field cleaning, equipment should be handled only by personnel wearing clean gloves to prevent re-contamination. In addition, the equipment should be moved away (preferably upwind) from the cleaning area to prevent recontamination. If the equipment is not to be immediately re-used it should be covered with plastic sheeting or wrapped in aluminum foil to prevent re-contamination. The area where the equipment is kept prior to re-use must be free of contaminants.

B.2 Field Equipment Cleaning Procedures

Sufficient clean equipment should be transported to the field so that an entire study can be conducted without the need for field cleaning. However, this is not possible for some specialized items. During large scale studies, it is not practical or possible to transport all of the precleaned field equipment required into the field. In these instances, sufficient pre-cleaned equipment should be transported to the field to perform at least one day of work. The following procedures are to be utilized when equipment must be cleaned in the field.

B.2.1 "Classic Parameter" Sampling Equipment

"Classic Parameters" are analyses such as oxygen demand, nutrients, certain inorganics, sulfide, flow measurements, etc. For routine operations involving classic parameter analyses, water quality sampling equipment such as Kemmerers, buckets, dissolved oxygen dunkers, dredges, etc., may be cleaned with the sample or analyte-free water between sampling locations. A brush may be used to remove deposits of material or sediment, if necessary. If analyte-free water is used, samplers should be flushed at the next sampling location with the substance (water) to be sampled, but before

the sample is collected.

Flow measuring equipment such as weirs, staff gages, velocity meters, and other stream gaging equipment may be cleaned with tap water between measuring locations, if necessary.

The previously described procedures are not to be used for cleaning field equipment to be used for the collection of samples undergoing trace organic or inorganic constituent analyses.

B.2.2 Sampling Equipment used for the Collection of Trace Organic and Inorganic Compounds

The following procedures are to be used for all sampling equipment used to collect routine samples undergoing trace organic or inorganic constituent analyses:

1. Clean with tap water and soap using a brush if necessary to remove particulate matter and surface films. Equipment may be steam cleaned (soap and high pressure hot water) as an alternative to brushing. Sampling equipment that is steam cleaned should be placed on racks or saw horses at least two feet above the floor of the decontamination pad. **(PVC or plastic items should not be steam cleaned.)**
2. Rinse thoroughly with tap water.
3. Rinse thoroughly with analyte free water.
4. Rinse thoroughly with solvent. **(Do not solvent rinse PVC or plastic items.)**
5. Rinse thoroughly with organic/analyte free water. If organic/analyte free water is not available, equipment should be allowed to completely dry. Do not apply a final rinse with analyte water. Organic/analyte free water can be generated on-site utilizing the portable system.
6. Remove the equipment from the decontamination area and cover with plastic. Equipment stored overnight should be wrapped in aluminum foil and covered with clean, unused plastic.

B.4 Emergency Disposable Sample Container Cleaning

New one-pint or one-quart mason jars may be used to collect samples for analyses of organic compounds and metals in waste and soil samples during an emergency. These containers would also be acceptable on an emergency basis for the collection of water samples for extractable organic compounds, pesticides, and metals analyses. These jars cannot be used for the collection of water samples for volatile organic compound analyses.

The rubber sealing ring should not be in contact with the jar and aluminum foil should be used, if possible, between the jar and the sealing ring. If possible, the jar and aluminum foil should be rinsed with pesticide-grade isopropanol and allowed to air dry before use. Several empty bottles and lids should be submitted to the laboratory as blanks for quality control purposes.

APPENDIX C FIELD EQUIPMENT CENTER STANDARD CLEANING PROCEDURES

PERFORMANCE OBJECTIVE:

- To remove contaminants of concern from sampling, and other field equipment to concentrations that do not impact study objectives using a standard cleaning procedure.

C.1 Introduction

Cleaning procedures outlined in this appendix are intended for use at the Field Equipment Center (FEC) for cleaning sampling and other field equipment prior to field use. These procedures are not intended to be used in the field. Cleaning procedures for use in the field are in Appendix B.

Sampling and other field equipment cleaned in accordance with these procedures will meet the minimum requirements for Data Quality Objective (DQO) Definitive Data Collection. Alternative cleaning procedures may be substituted as outlined in Section 4.5 when samples are to be analyzed for data to be used at a lower DQO level. Deviations from these procedures should be documented in the approved study plan, field records, and/or investigative reports.

C.1.1 Specifications For Cleaning Materials

The specifications for standard cleaning materials referred to in this appendix are as follows:

- Soap shall be a standard brand of phosphate-free laboratory detergent such as Liquinox[®].
- Disinfectant soap shall be a standard brand of disinfectant cleaner.
- Solvent shall be pesticide-grade isopropanol.
- Tap water may be obtained from any spigot at the FEC.
- Nitric acid solution (10%) shall be made from reagent-grade nitric acid and deionized water.
- Analyte free water (deionized water) is tap water that has been treated by passing it through a standard deionizing resin column. At a minimum, it should contain no detectable heavy metals or other inorganic compounds (i.e., at or above analytical detection limits) as defined by a standard Inductively Coupled Argon Plasma Spectrophotometer (ICP) (or equivalent) scan.
- Organic/analyte free water is defined as tap water that has been treated with activated carbon and deionizing units. At a minimum, it must meet the analytical criteria of analyte free water and should contain no detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels

determined by the Region 4 laboratory for a given set of analyses. Organic/analyte free water obtained by other methods is acceptable, as long as it meets the above analytical criteria.

- Other solvents may be substituted for a particular investigation if needed. Pesticide-grade acetone or methanol are acceptable. However, it should be noted that if pesticide-grade acetone is used, the detection of acetone in samples collected with acetone rinsed equipment is considered suspect. Pesticide-grade methanol is much more hazardous to use than either pesticide-grade acetone or isopropanol, therefore its use is discouraged.

Solvents, nitric acid solution, laboratory detergent, and rinse waters used to clean equipment cannot be reused.

C.1.2 Handling and Containers for Cleaning Solutions

Improperly handled cleaning solutions may easily become contaminated. Containers should be constructed of the proper materials to ensure their integrity. Following are the materials to be used for storing the specified cleaning materials:

- Soap should be kept in clean containers until use. It should be poured directly from the container.
- Disinfectant soap should be kept in clean containers until use. It should be poured directly from the container.
- Solvents should be stored in the unopened original containers until used. Solvents may be applied using the low pressure nitrogen system fitted with a Teflon[®] nozzle, or by using Teflon[®] squeeze bottles.
- Tap water may be kept in clean tanks, hand pressure sprayers, squeeze bottles, or applied directly from a hose.
- Analyte free water should be stored in cleaned containers that can be closed when not being used. It may be applied from squeeze bottles.
- Organic/analyte free water should be stored in cleaned glass, Teflon[®], or stainless steel containers prior to use. It may be applied using Teflon[®] squeeze bottles, or directly from the system.
- Nitric acid should be kept in the glass container it is received in, and placed in squeeze bottles prior to application.

C.1.3 Disposal of Spent Cleaning Solutions

Procedures for safe handling and disposition of spent cleaning solutions, including washwater, rinse water, spent acid solutions, and spent solvents are as follows:

Washwater

Since equipment is decontaminated before its return to the FEC, the washwater may be disposed in the sanitary drain in the washroom. When large equipment (vehicles, augers, etc.) is washed outside, it may wash onto the ground without recovery of the washwater.

Rinsewater

Since equipment is decontaminated before its return to the FEC, the rinsewater may be disposed in the sanitary drain in the washroom. When large equipment (vehicles, augers, etc.) is rinsed outside, it may go onto the ground without recovery.

Nitric Acid

Nitric acid cleaning solutions are to be diluted to a pH greater than 2.0, and flushed down the sanitary drain in the washroom. If used outdoors, this material should be captured and diluted to a pH greater than 2.0, and flushed down the sanitary drain in the washroom.

Solvent

All solvents used should be captured, properly labeled, and stored on the premises of the FEC until arrangements for proper disposal are made. Used solvents can be classified as either "solvent for recovery" or "solvent for disposal". Solvent for recovery is that which was used in the standard field cleaning or FEC cleaning of equipment. Solvent used for cleaning badly contaminated equipment (e.g., tar removal, etc.) should be designated for disposal. The two groups should be labeled "For Recovery" or "For Disposal" and stored separately at the FEC.

C.1.4 Safety Procedures for Cleaning Operations

Some materials used to implement the cleaning procedures outlined in this Appendix are harmful if used improperly. Caution should be exercised and all applicable safety procedures shall be followed. At a minimum, the following precautions shall be taken in the washroom during these cleaning operations:

- Safety glasses with splash shields or goggles, a neoprene apron, and neoprene gloves will be worn during all cleaning operations. When cleaning heavy items such as hollow-stem augers or other drill rig equipment, safety boots will be worn.
- All solvent rinsing operations will be conducted under a fume hood or in the open (never in a closed room).
- No eating, smoking, drinking, chewing, or any hand to mouth contact shall be permitted during cleaning operations.

C.1.5 Handling and Labeling of Cleaned Equipment

After cleaning, equipment should be handled only by personnel wearing clean latex gloves to prevent re-contamination.

After the cleaned equipment is wrapped in aluminum foil and sealed in plastic, the date that the equipment was cleaned should be written on the plastic. If the equipment was not cleaned according to the procedures outlined in this appendix, this should also be noted on the plastic.

C.1.6 Initial Processing of Returned Equipment

Field or sampling equipment that needs to be repaired will be identified with a "repair" tag. Any problems encountered with the equipment and specific required repairs shall be noted on this tag, as well as the date and the initials of the investigator. Field equipment or reusable sample containers needing cleaning or repairs will not be stored with clean equipment, sample tubing, or sample containers.

All coolers, plastic wrapped equipment, containers, and tubing not used in the field may be placed back into stock after the following precautions are taken:

- Soap and hot water rinse plastic containers. Allow to air dry.
- If plastic wrapping leaks after soap/water rinse, remove the equipment and place it into the standard cleaning process.

C.2 Trace Organic and Inorganic Constituent Sampling Equipment

Sampling equipment used to collect samples undergoing trace organic and/or inorganic constituent analyses should be thoroughly cleaned. The following procedures are to be used.

C.2.1 Teflon® and Glass

1. Wash equipment thoroughly with soap and hot tap water using a brush or scrub pad to remove any particulate matter or surface film.
2. Rinse equipment thoroughly with hot tap water.
3. Rinse equipment with 10 percent nitric acid solution. Small and awkward equipment such as vacuum bottle inserts and well bailer ends may be soaked in the nitric acid solution instead of being rinsed with it. Fresh nitric acid solution should be prepared for each cleaning session.
4. Rinse equipment thoroughly with analyte free water.
5. Rinse equipment thoroughly with solvent and allow to air dry for at least 24 hours.
6. Wrap equipment in one layer of aluminum foil. Roll edges of foil into a "tab" to allow for easy removal. Seal the foil wrapped equipment in plastic and label.

When this sampling equipment is used to collect samples that contain oil, grease, or other hard to remove materials, it may be necessary to rinse the equipment several times with pesticide-grade acetone, hexane, or petroleum ether to remove the materials before proceeding with the first step. In extreme cases, it may be necessary to steam clean the field equipment before proceeding with Step 1. If the equipment cannot be cleaned utilizing these procedures, it should be discarded.

C.2.2 Stainless Steel or Steel

1. Wash equipment thoroughly with soap and hot tap water using a brush or scrub pad to remove any particulate matter or surface film.
2. Rinse equipment thoroughly with hot tap water.
3. Rinse equipment thoroughly with analyte free water.
4. Rinse equipment thoroughly with solvent and allow to air dry for at least 24 hours.
5. Wrap equipment in one layer of aluminum foil. Roll edges of foil into a "tab" to allow for easy removal. Seal the foil wrapped equipment in plastic and label.

When this sampling equipment is used to collect samples that contain oil, grease, or other hard to remove materials, it may be necessary to rinse the equipment several times with pesticide-grade acetone, hexane, or petroleum ether to remove the materials before proceeding with the first step. In extreme cases, it may be necessary to steam clean the field equipment before proceeding with Step 1. If the equipment cannot be cleaned utilizing these procedures, it should be discarded.

C.3 Automatic Wastewater Sampling Equipment

C.3.1 ISCO[®] and Other Automatic Samplers

- The exterior and accessible interior (excluding the waterproof timing mechanism) portions of the automatic samplers will be washed with soap and tap water then rinsed with tap water.
- Desiccant in the flow meters should be checked and replaced, if necessary, each time the equipment is cleaned.
- The face of the timing case mechanism will be cleaned with a clean damp cloth.
- Tubing (sample intake and pump tubing) will be discarded after each use.
- New precleaned, Silastic pump tubing (see Appendix C.4.1) will be installed.

C.3.2 ISCO[®] 3700 and 6700 Rotary Funnel, Distributor, and Metal Tube

1. Clean with hot tap water, soap, and a brush.
2. Rinse thoroughly with analyte free water.
3. Replace in sampler.

C.3.3 All Automatic Sampler Headers

1. Disassemble header and using a bottle brush, wash with hot tap water and soap.

2. Rinse thoroughly with analyte free water.
3. Dry thoroughly, then reassemble header and wrap with aluminum foil.
4. Seal in Plastic

C.3.4 Reusable Glass Composite Sample Containers

1. Wash containers thoroughly with hot tap water and laboratory detergent, using a bottle brush to remove particulate matter and surface film.
2. Rinse containers thoroughly with hot tap water.
3. Rinse containers with at least 10 percent nitric acid.
4. Rinse containers thoroughly with tap water.
5. Rinse containers thoroughly with analyte free water.
6. Rinse twice with solvent and allow to air dry for at least 24 hours.
7. Cap with aluminum foil or Teflon[®] film.

When these containers are used to collect samples that contain oil, grease, or other hard to remove materials, it may be necessary to rinse the containers several times with pesticide-grade acetone, hexane, or petroleum ether to remove the materials before proceeding with Step 1. Any bottles that have a visible film, scale, or discoloration remaining after this cleaning procedure shall also be discarded.

C.3.5 Plastic Reusable Composite Sample Containers (3700 - 4 gal.)

1. Wash containers thoroughly with hot tap water and laboratory detergent, using a bottle brush to remove particulate matter and surface film.
2. Rinse containers thoroughly with hot tap water.
3. Rinse containers with at least 10 percent nitric acid.
4. Rinse containers thoroughly with tap water.
5. Rinse containers thoroughly with analyte free water.
6. Cap with aluminum foil or Teflon[®] film.

Any plastic composite sample containers that have a visible film, scale, or other discoloration remaining after this cleaning procedure will be discarded.

C.3.6 ISCO[®] 3700 and 6700 Plastic Sequential Bottles

1. Wash bottles thoroughly with soap and hot tap water.

2. Rinse thoroughly with hot tap water.
3. Rinse with analyte free water.
4. Allow to air dry.
5. Place in ISCO® sampler base and cover with aluminum foil for storage.

C.3.7 Bottle Siphons for Composite Containers

Tubing should be rinsed with solvent and dried in the drying oven overnight before use. The ends of the siphon should be capped with aluminum foil and/or Teflon® film for storage. The tubing will be sealed in plastic and labeled. The siphon should be flushed with sample thoroughly before use.

C.3.8 Reusable Teflon® Composite Mixer Rods

1. Wash equipment thoroughly with soap and hot tap water using a brush or scrub pad to remove any particulate matter or surface film.
2. Rinse equipment thoroughly with hot tap water.
3. Rinse equipment with at least a 10 percent nitric acid solution.
4. Rinse equipment thoroughly with tap water.
5. Rinse equipment thoroughly with analyte free water.
6. Rinse equipment thoroughly with solvent and allow to air dry for at least 24 hours.
7. Wrap equipment in one layer of aluminum foil. Roll edges of foil into a "tab" to allow for easy removal. Seal the foil wrapped equipment in plastic and label.

When this sampling equipment is used to collect samples that contain oil, grease, or other hard to remove materials, it may be necessary to rinse the equipment several times with pesticide-grade acetone, hexane, or petroleum ether to remove the materials before proceeding with Step 1. In extreme cases, it may be necessary to steam clean the field equipment before proceeding with Step 1. If the equipment cannot be cleaned utilizing these procedures, it should be discarded.

C.4 Cleaning Procedures for Tubing

C.4.1 Silastic® Pump Tubing

The Silastic® pump tubing in the automatic samplers and peristaltic pumps should be replaced after each study. After installation, the exposed ends should be capped with clean, unused aluminum foil.

C.4.2 Teflon® Sample Tubing

Use only new Teflon® tubing which has been precleaned as follows for the collection of samples for trace organic compound or ICP analyses:

1. Teflon® tubing shall be precut in 10, 15 or 25-foot lengths before cleaning.
2. Rinse outside of tubing with solvent.
3. Flush interior of tubing with solvent.
4. After flushing with solvent, pressurize the tubing to a safe level (one end only) to flush out solvent.
5. Dry overnight in the drying oven.
6. Coil. Cap ends with aluminum foil. Wrap tubing in one layer of aluminum foil. Roll edges of foil into a "tab" to allow for easy removal. Seal the foil wrapped tubing in plastic and label.

C.4.3 Stainless Steel Tubing

1. Wash with soap and hot tap water using a long, narrow, bottle brush.
2. Rinse equipment thoroughly with hot tap water.
3. Rinse equipment thoroughly with analyte free water.
4. Rinse equipment thoroughly with solvent and allow to air dry for at least 24 hours.
5. Cap ends with aluminum foil. Wrap tubing in one layer of aluminum foil. Roll edges of foil into a "tab" to allow for easy removal. Seal the foil wrapped tubing in plastic and date.

When this sampling equipment is used to collect samples that contain oil, grease, or other hard to remove materials, it may be necessary to rinse the equipment several times with pesticide-grade acetone, hexane, or petroleum ether to remove the materials before proceeding with Step 1. If the equipment cannot be cleaned utilizing these procedures, it should be discarded.

C.4.4 Glass Tubing

New glass tubing should be cleaned as follows:

1. Rinse thoroughly with solvent.
2. Air dry for at least 24 hours.
3. Wrap tubing completely with aluminum foil and seal in plastic (one tube/pack) to prevent contamination during storage.

C.5 Cleaning Procedures for Miscellaneous Equipment

C.5.1 Miscellaneous Sampling and Flow Measuring Equipment

Flow measuring equipment such as weirs, staff gages, velocity meters, and other stream

gaging equipment, and other miscellaneous sampling equipment shall be washed with soap and hot tap water, rinsed with hot tap water, rinsed thoroughly with analyte free water, and completely air dried before being stored. This procedure is not to be used for equipment utilized for the collection of samples for trace organic or inorganic constituent analyses.

C.5.2 Field Analytical Equipment

Field instruments for in-situ water analysis should be wiped with a clean, damp cloth. The probes on these instruments (pH, conductivity, DO, etc.), should be rinsed with analyte-free water and air dried.

Any desiccant in these instruments should be checked and replaced, if necessary, each time the equipment is cleaned.

C.5.3 Ice Chests and Shipping Containers

Ice chests and reusable containers shall be washed with soap (interior and exterior) and rinsed with tap water and air dried before storage. If in the opinion of the field investigators the container is severely contaminated with concentrated waste or other toxic material, it shall be cleaned as thoroughly as possible, rendered unusable, and properly disposed.

C.5.4 Pressure Field Filtration Apparatus

1. Wash equipment thoroughly with soap and hot tap water using a brush to remove any particulate matter or surface film.
2. Rinse equipment thoroughly with hot tap water.
3. Rinse equipment with 10 percent nitric acid solution.
4. Rinse equipment thoroughly with analyte free water.
5. Rinse equipment thoroughly with solvent and allow to air dry for at least 24 hours.
6. Assemble the apparatus and cap both the pressure inlet and sample discharge lines with aluminum foil to prevent contamination during storage.
7. Wrap equipment in one layer of aluminum foil. Roll edges of foil into a "tab" to allow for easy removal. Seal the foil wrapped equipment in plastic and date.

During steps 1 through 5 as outlined above and immediately after assembling, pressure should be applied to the apparatus after each rinse step (water and acid) to drive the rinse material through the porous glass filter holder in the bottom of the apparatus.

When this sampling equipment is used to collect samples that contain oil, grease, or other hard to remove materials, it may be necessary to rinse the equipment several times with pesticide-grade acetone, hexane, or petroleum ether to remove the materials before proceeding with the first step. In extreme cases, it may be necessary to steam clean the field equipment before proceeding with Step 1. If the equipment cannot be cleaned utilizing these procedures, it should be discarded.

C.5.5 Organic/Analyte Free Water Storage Containers

NOTE: These containers will be used only for transporting organic/analyte free water.

1. Wash containers thoroughly (interior and exterior) with hot tap water and laboratory detergent, using a bottle brush to remove particulate matter and surface film.
2. Rinse containers thoroughly with hot tap water.
3. Rinse containers with at least 10 percent nitric acid.
4. Rinse containers thoroughly with tap water.
5. Rinse containers thoroughly with analyte free water.
6. Rinse containers thoroughly with solvent and allow to air dry for at least 24 hours.
7. Cap with aluminum foil or Teflon® film.
8. Store in plastic bags.

When transporting organic/analyte free water to the field, use only containers cleaned as specified above. Thoroughly rinse the interior of the container with organic/analyte free water prior to filling. Cap with one layer of Teflon® film, one layer of aluminum foil, and label the container as "organic/analyte free water" and include the date it was prepared. Do not store the organic/analyte free water at the FEC for more than three days.

C.5.6 Portable Solvent Rinse System

1. Replace Teflon® tubing if necessary. Wash nozzle and tubing fittings with hot, soapy water.
2. Rinse with analyte-free water.
3. Wrap nozzle and tubing ends with aluminum foil.

C.5.7 Garden Hose

1. Brush exterior with soap and tap water
2. Rinse with tap water.
3. Flush interior with tap water until clear (minimum of one gallon).
4. Let completely air dry.
5. Coil and place in clean plastic bag.

C.5.8 Portable Tanks for Tap Water

1. Scrub interior and exterior with soap and tap water.

2. Rinse with tap water.
3. Let completely air dry.
4. Close.

C.5.9 Vehicles

Vehicles utilized by field investigators should be washed (if possible) at the conclusion of each field trip. This should minimize contamination of equipment or samples due to contamination of vehicles.

When vehicles are used in conjunction with hazardous waste site inspections, or on studies where pesticides, herbicides, organic compounds, or other toxic materials are known or suspected to be present, a thorough interior and exterior cleaning (using soapy tap water) is mandatory at the conclusion of such investigations. It shall be the responsibility of the field investigators to see that this procedure is followed. Personnel involved will use appropriate safety measures.

Vehicles shall be equipped with trash bags and/or trash containers to facilitate vehicle cleaning. Field investigators are responsible for keeping field vehicles clean by removing trash and other debris. Contaminated trash and equipment should be kept separate from ordinary trash and should be properly disposed on-site or upon return (Section 20.8).

C.6 Preparation of Disposable Sample Containers

C.6.1 Introduction

No disposable sample container (with the exception of the glass and plastic compositing containers) may be reused. All disposable sample containers will be stored in their original packing containers. When packages of uncapped sample containers are opened, they will be placed in new plastic garbage bags and sealed to prevent contamination during storage.

Specific precleaning instructions for disposable sample containers are given in the following sections.

C.6.2 Plastic Containers used for "Classical" Parameters

Plastic containers used for oxygen demand, nutrients, classical inorganics, and sulfides have no precleaning requirement. However, only new containers may be used.

C.6.3 Light/Dark BOD Bottles

Light/dark BOD bottles used for dissolved oxygen measurements should be cleaned in the laboratory according to the following procedures:

1. Wash inside and outside of bottles with soap and water.
2. Rinse thoroughly with analyte-free water and allow to dry.
3. Cover openings of bottles with aluminum foil.

APPENDIX D

SAMPLE SHIPPING PROCEDURES

D.1 Introduction

Samples collected during field investigations or in response to a hazardous materials incident must be classified prior to shipment, as either environmental or hazardous materials samples. In general, environmental samples include drinking water, most groundwater and ambient surface water, soil, sediment, treated municipal and industrial wastewater effluent, biological specimens, or any samples not expected to be contaminated with high levels of hazardous materials.

Samples collected from process wastewater streams, drums, bulk storage tanks, soil, sediment, or water samples from areas suspected of being highly contaminated may require shipment as dangerous goods. Regulations for packing, marking, labeling, and shipping of dangerous goods by air transport are promulgated by the International Air Transport Authority (IATA), which is equivalent to United Nations International Civil Aviation Organization (UN/ICAO) (1). Transportation of hazardous materials (dangerous goods) by EPA personnel is covered by EPA Order 1000.18 (2)

D.2 Shipment of Dangerous Goods

The project leader is responsible for determining if samples collected during a specific field investigation meet the definitions for dangerous goods. If a sample is collected of a material that is listed in the Dangerous Goods List, Section 4.2, IATA, then that sample must be identified, packaged, marked, labeled, and shipped according to the instructions given for that material. If the composition of the collected sample(s) is unknown, and the project leader knows or suspects that it is a regulated material (dangerous goods), the sample may not be offered for air transport. If the composition and properties of the waste sample or highly contaminated soil, sediment, or water sample are unknown, or only partially known, the sample may not be offered for air transport.

In addition, the shipment of pre-preserved sample containers or bottles of preservatives (e.g., NaOH pellets, HCL, etc.) which are designated as dangerous goods by IATA is regulated. Shipment of nitric acid is forbidden on all aircraft. Dangerous goods must not be offered for air transport without contacting the SESD dangerous goods shipment designee.

D.3 Shipment of Environmental Laboratory Samples

Guidance for the shipment of environmental laboratory samples by personnel is provided in a memorandum dated March 6, 1981, subject "Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Laboratory Samples" (3). By this memorandum, the shipment of the following unpreserved samples is not regulated:

- Drinking water
- Treated effluent
- Biological specimens
- Sediment
- Water treatment plant sludge
- POTW sludge

In addition, the shipment of the following preserved samples is not regulated, provided the amount of preservative used does not exceed the amounts found in 40 CFR 136.3 (4) (see Appendix A). It is the shippers' (individual signing the airway bill) responsibility to ensure that proper amounts of preservative are used:

- Drinking water
- Ambient water
- Treated effluent
- Biological specimens
- Sediment
- Wastewater treatment plant sludge
- Water treatment plant sludge

Samples determined by the project leader to be in these categories are to be shipped using the following protocol, developed jointly between US-EPA, OSHA, and DOT. This procedure is documented in the "Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Environmental Laboratory Samples" (3).

Untreated wastewater and sludge from POTW's are considered to be "diagnostic specimens" (not environmental laboratory samples). However, because they are not considered to be etiologic agents (infectious) they are not restricted and may be shipped using the procedures outlined below.

Environmental samples should be packed prior to shipment by air using the following procedures:

1. Allow sufficient headspace (ullage) in all bottles (except VOC containers with a septum seal) to compensate for any pressure and temperature changes (approximately 10 percent of the volume of the container).
2. Be sure the lids on all bottles are tight (will not leak).
3. Place bottles in separate and appropriately sized polyethylene bags and seal the bags with tape (preferably plastic electrical tape). Up to three VOC bottles may be packed in one Whirl-Pak container.
4. Optionally, place three to six VOC vials in a quart metal can and then fill the can with vermiculite.
5. Select a sturdy cooler in good repair. Secure and tape the drain plug with fiber or duct tape. Line the cooler with a large heavy duty plastic bag.
6. Place two to four inches of vermiculite in the bottom of the cooler and then place the bottles and cans in the cooler with sufficient space to allow for the addition of vermiculite between the bottles and cans.
7. Put "blue ice" (or ice that has been "double bagged" in heavy duty polyethylene bags and properly sealed) on top of and/or between the samples. Fill all remaining space between the bottles or cans with vermiculite.

8. Securely fasten the top of the large garbage bag with tape (preferably plastic electrical tape).
9. Place the Chain-of-Custody Record and the CLP Traffic Report Form (if applicable) into a plastic bag, and tape the bag to the inner side of the cooler lid.
10. Close the cooler and securely tape (preferably with fiber tape) the top of the cooler shut. Chain-of-custody seals should be affixed to the top and sides of the cooler within the securing tape so that the cooler cannot be opened without breaking the seal.
11. Shipping containers must be marked "THIS END UP", and arrow labels which indicate the proper upward position of the container should be affixed to the container. A label containing the name and address of the shipper should be placed on the outside of the container. Labels used in the shipment of hazardous materials (e.g., Cargo Only Air Craft, Flammable Solids, etc.) are not permitted to be on the outside of containers used to transport environmental samples.

D.4 References

1. Dangerous Goods Regulations, International Air Transport Authority (IATA). Current Edition. which changes annually.
2. EPA Order 1000.18, February 16, 1979.
3. "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), US-EPA, April 13, 1981.
4. 40 CFR 136.3. July 1, 2001. See Table 11, Footnote 3.

APPENDIX E
BATTERY USE AND STORAGE OPERATIONS

PERFORMANCE OBJECTIVES:

- To insure that field batteries are handled in a safe and efficient manner.
- To insure that field batteries are properly stored.

CAUTION
DANGER OF EXPLODING BATTERIES

Batteries generate explosive gasses. Keep sparks, flames, burning cigarettes, cigars, or other ignition sources away at all times. Always shield eyes when working near batteries. Charge batteries only in well ventilated areas.

DANGER OF ACID BURNS

In case of skin contact with acid, immediately wash affected area for 15 minutes, using safety shower, eye wash, or sink as required. Seek medical attention as soon as possible. Notify the designated Safety Officer or management in the event of injury.

E.1. Procuring Batteries from the Battery Storage Area

All Marine and 12 volt batteries procured from the Battery Storage Area (located near the Hazardous Materials Storage Building) must be in DOT approved containers with lids. Batteries that have been charged will be placed in the “charged battery” section and be in approved DOT containers. Batteries must be kept in approved containers with tops secured at all times.

E.2. Returning Batteries from the Field

Upon return from the field, Marine and 12 volt batteries must be placed in the Battery Storage Area in the section for “discharged batteries”.

E.3. Charging Batteries

Battery charging equipment is to be operated only by trained personnel who are familiar with the procedures.

E.4. Small Batteries

Small batteries such as 9V, AA, etc. are to be stored in an upright position at all times, preferably in the boxes in which they were received, so that the terminals will not be exposed and contact other terminals.