WATER QUALITY

QUALITY ASSURANCE MANUAL

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Georgia Department of Natural Resources Environmental Protection Division Water Protection Branch 4220 International Parkway, Suite 101 Atlanta, GA 30354

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INTRODUCTION EPD WATER PROTECTION BRANCH QUALITY ASSURANCE MANUAL

The sections that follow detail the standard field Quality Assurance procedures used by the Water Protection Branch of the Georgia Environmental Protection Division. This guidance was developed to ensure and document the validity of measurements and analyses and the representativeness of samples collected. This is necessary in the event of a dispute with other parties regarding data collection techniques and the resulting quality of field information. Enforcement activities by the Branch require full documentation on particulars of data collection and the equipment used to collect it. All Branch associates who collect samples or field data must be familiar with the measures outlined in this document.

Requirements pertaining to specifics of sample collection for certain parameters are specified in federal regulations under the authority of the Clean Water Act (CWA) and the National Pollutant Discharge Elimination System (NPDES) permitting program. The most widely applicable guidance at this level is *Title 40 of the Code of Federal Regulations (40 CFR)*. The procedures and techniques given in *40 CFR* are updated periodically by the United States Environmental Protection Agency and field workers are advised to consult the latest revision for proper procedures and developments. Other references are cited at the conclusion of each section.

In the process of outlining "quality assurance" measures it is difficult not to stray into the areas of procedures, methods and techniques. The distinction between "QA measures" and sampling techniques is also blurred by the fact that the collection protocols in *40 CFR* are in many instances based on the concern for quality assurance. Certain of the discussions that follow may fulfill the role of Standard Operating Procedures (SOPs) for this reason. The emphasis, however, is on quality and validation rather than methodology.

This document is dynamic and will be continually revised as new developments warrant. As the Branch assumes more responsibilities for study and sampling in new investigational areas, it is anticipated that additional QA measures will be implemented.

SECTION 1 PLANNING, DOCUMENTARY PROTOCOLS AND DOCUMENT CONTROL FOR WATER QUALITY ASSESSMENTS

Introduction

The water quality inspections, investigations and studies conducted by the Georgia EPD Water Protection Branch (WPB) can be broadly categorized as either enforcement or non-enforcement related activities.

The enforcement related fieldwork includes water enforcement case investigations, National Pollutant Discharge Elimination System (NPDES) compliance sampling inspections (CSIs), some diagnostic evaluations of municipal and industrial wastewater treatment plants, and monitoring of sewage spills.

Field work conducted that does not have a specific enforcement objective includes technical assistance studies at municipal wastewater treatment plants, trend monitoring, surveys to verify issued permit limits, waste load allocation and model calibration studies, and other intensive surveys for documenting water quality. Because studies and data derived from non-enforcement type investigations could be used for enforcement purposes, the quality assurance measures for both follow the same basic guidelines. Field investigations and studies in the WPB primarily involve sampling and analysis of wastewater, surface water, aquatic sediments and assessment of fish tissue residue data, but may also include other environmental media (ground water, soils or other waste).

In all aspects of water quality planning and field assessment activities, safety is to be addressed and treated as a critical element of any WPB activity. The Georgia DNR *Safety Manual* is to be consulted and its policies, protocols and procedures are to be incorporated and implemented in WPB field activities.

Water Quality Survey Procedures

Routine NPDES inspections and special response investigations do not require written study plans. Compliance sampling inspections (CSIs) are scheduled well in advance through monthly calendars covering staffing and laboratory analytical support needs.

Detailed investigations such as for lake and coastal monitoring, model calibration studies and other intensive surveys or large-scale technical evaluations require more planning and a formalized plan of study before initiation. The WPB has "Water Quality Survey Procedures" for these types of studies that follows a step-wise process from the initial request through planning, conducting and reporting of the results.

General Documentary Guidelines

All sample identification, chain-of-custody records, and field records should 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 should be initialed and dated. If possible, all corrections should be made by the individual making the error. Some data forms may not be amenable to using indelible waterproof ink and the use of pencil is acceptable as long as the reviewing professional certifies the accuracy of the data.

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.

Field Notes (Logbook and Other Original Written Field Records)

Preferably each study or project should have a logbook dedicated to it, shared with other related projects or studies, or with other work conducted in the same river basin, or watershed unit (HUC). Reasons for this include document control and consistency in filing original completed field records, ability to segregate records that may become legal evidence in enforcement proceedings from current study record keeping, and security of past data should a logbook become damaged or lost in subsequent work. The WPB has logbooks with different numbers of pages and the ability to produce specialized logbooks using resin coated paper stock that can be preprinted on laser jet printers and bound.

The study leader's name, the sample team leader's name (if appropriate), names of any additional staff collecting data, and the study name, location and study dates 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 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 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 field notes logbook:

- sample collection equipment (where appropriate);
- field analytical equipment, and equipment used for physical measurements shall be identified;
- calculations, results, and calibration data for field sampling, field analytical, and field

physical measurement equipment;

- property and serial numbers of any sampling equipment used, if available;
- sampling station identification;
- time of sample(s) collection;
- description of the sample(s) location;
- description of the sample(s);
- sample collector(s);
- how the sample(s) 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.)

Original field data that is recorded by an instrument and saved directly into a logging component (internal or accessory), are to be downloaded and secured as an electronic file. A hard copy of the data file should be printed and labeled by the investigator and included and filed with the original field notes.

Sample (and Evidence) Identification

Sample Identification

The method of sample identification used depends on the type of sample collected. Samples collected for specific field analyses or measurement data are recorded directly in bound field logbooks and/or recorded directly on the chain-of-custody record, with identifying information, while in the custody of the samplers. Samples collected for laboratory analyses are identified by using sample tags which are attached to the sample containers, writing on the bottle label when provided and appropriate, or both depending on the type of sample. In some cases, particularly with biological samples, the sample tags may have to be included with or wrapped around the samples. Sample tags can be accountable documents after they are completed and attached to a sample or other physical evidence. Examples of a combination sample tag and field information form containing both sample identification and field data follow. The following information may be included on the sample tag using waterproof, non-erasable ink (the first three must always be provided; additional critical information is recorded on accompanying laboratory source document):

- study or project;
- field identification or sample station number;
- date and time of sample collection;
- designation of the sample as a grab or composite;
- type of sample (water, wastewater, leachate, soil, sediment, etc.) and a very brief description of the sampling location;
- the name (or signature) of either the sampler(s) or the designated sampling team leader and the field sample custodian (if appropriate);
- the general types of analyses to be performed (if necessary); and
- relevant comments (such as detectable or identifiable odor, color, or known toxic properties).

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 or identified.

Tags for blank or duplicate (or replicate) samples will be marked "blank" or "duplicate," respectively. This identifying information shall also be recorded in the bound field logbooks and on the chain-of-custody record.

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| Е | Example Tag (Hybrid also providing field information and chain of custody record) |
|---|--|
| | Purpose: Annual Bacteriological Monitoring of Ga. State Parks Operated Beaches |
| | Sample I.D.#: Park: |

| Date Collected: Time Collected: | Collector | • | |
|---|--------------------------|------------|---------|
| Swimming Area Open?: If Yes | s, Level of Use (low, hi | gh, etc.): | |
| Weather Today: Wea | ather Previous 3 Days | : | |
| Water Clarity (Secchi disk): | Water Color | : | |
| Waterfowl (Ducks, Geese, etc.) Recently Pro | esent at or near Beach | Area? | |
| SAMPLE RELINQUISHED BY: | on | at | A / P M |
| SAMPLE RECEIVED BY: | on | at | A / P M |
| SAMPLE RELINQUISHED BY: | on | at | A / P M |
| SAMPLE RECEIVED BY: | on | at | A / P M |

Photographs, Video and Digital Media Documentation

Photographs used in investigative reports or placed in the official files shall be identified on the back of the print with the following information:

- A brief, but accurate description of what the photograph shows, including the name of the facility or site and the location.
- The date and time that the photograph was taken.
- The name of the photographer.

When photographs are taken, a record of each frame exposed shall be kept in the field notes along with the information required for each photograph. The film shall be developed with the negatives supplied. The field investigator shall then enter the required information on the prints, using the photographic record from the field notes, to identify each photograph. For criminal investigations, the negatives are to be maintained and stored in a secured file or other location. Video recorded for documentary purposes is treated in a similar fashion. When in use, the videographer (or a designated at hand narrator) should state at the beginning of the tape the investigation purpose, what is being videotaped, who is present, the date and time, and the location/area that the following recording represents. During the taping, the videographer and/or narrator are to describe what is being video taped at the time for the audio part of the recording. It may be helpful to the videographer (and assistants), to discuss how the video taping will proceed and prescript an approach prior to beginning the taping.

Upon return to the office or as soon as possible, the field tape should be copied on to a blank tape that is labeled (on the tape directly and the tape cover), with all of the summary information that accurately reflects the tape contents. If the tape is to be used for an enforcement action, at least one duplicate tape should be produced and stored in a secured file or location with the original copy.

Images obtained using digital still or video cameras are secured as documentary evidence similar to that described above for film and video media. Image records made with digital media are electronic graphic files that can be modified or altered using software. If a digital record is to be used for evidence in an enforcement proceeding, the original graphic file is to be saved and secured with chain of custody certification by the originator. Guidance should be obtained from the Georgia Attorney General's Office if digital image documentation for enforcement is anticipated.

Identification of Other Physical Evidence

Physical evidence, other than samples or images, shall be identified by utilizing a sample tag or recording the necessary information on the evidence. In addition, it is suggested that photographs of any physical evidence be taken and the necessary information recorded in the field logbook.

Occasionally, it is necessary to obtain recorder and/or instrument charts from WPB or facility owned analytical equipment (flow recorders, etc.), during field investigations and inspections. 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.
- Results of an instantaneous measurement 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.

Chain-of-Custody Procedures

Introduction

Chain-of-custody procedures maintain and document the sample custody record and provide 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.

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 they secured it to prevent tampering; and/or,
- it is placed in a designated secure area.

Chain-of-Custody Record

The field chain-of-custody record 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. The chain-of-custody record also serves as a sample logging mechanism for the laboratory sample custodian. In the WPB, the EPD Laboratory Source Document may serve as the chain of custody record for most samples and is completed for all samples collected and submitted to the EPD 14th Street Laboratory for analysis. The EPD Laboratory Source Document may be customized to a format specific to special types of samples or monitoring projects. A separate chain-of-custody record may be used for special samples. Field notes should also note the sample holding and disposition concerning custody, transfer and laboratory delivery as appropriate. An example of a Laboratory Source Document and a chain-of custody form are provided on the following pages.

The following information is recorded in the indicated spaces to complete the Laboratory Source Document or field Chain-of-Custody Record.

- The study or project name.
- Name of study leader and/or sample collector
- The sampling station number or I.D., date and time of sample collection, grab or composite sample designation, and a brief description of the sampling location must be included.
- Required analyses should be checked off in the appropriate location.
- 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.

• Usually, the last person receiving the samples or evidence should be the laboratory sample custodian or their designee(s).

Physical evidence for criminal investigations such as video tapes or other small items shall be placed in Zip-Loc® type bags or envelopes and a 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 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.

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.

LABORATORY SOURCE DOCUMENT

COLLECTED BY: PROJECT COORDINATOR: DATE TIME STATION NUMBER GEORGIA DEPARTMENT OF NATURAL RESOURCES COLLECTED COLLECTED ENVIRONMENTAL PROTECTION DIVISION WATERSHED PLANNING AND MONITORING UNIT DEDULECTE

PROJECT:

| SAMPLE SITE | |
|--------------|--|
| DESCRIPTION: | |

FIELD DATA:

| PARAME | STORET CODE | Value | |
|--------------------|----------------|-------|--|
| H2O Temperature | deg. C | 00010 | |
| Air Temperature | deg. C | 00020 | |
| Field Conductivity | umho/cm | 00094 | |
| Dissolved Oxygen | mg/L | 00300 | |
| pН | Std. Units | 00400 | |
| Depth | ft. | 00003 | |
| Gage Height | ft. | 00065 | |
| Photic Zone | m. | 00204 | |
| Secchi Disk | m. | 00078 | |
| Tapedown | ft. | 82580 | |
| Stream Flow | cfs | 00061 | |
| Salinity | mg/mL | 00096 | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

LABORATORY DATA:

| PARAM | PARAMETER | | | | |
|---------------------|---------------|-------|--|--|--|
| Turbidity | NTU | 00076 | | | |
| Color | PCU | 00080 | | | |
| Specific Cond. | umho/cm | 00095 | | | |
| BOD – 5 day | mg/L | 00310 | | | |
| рН | Std. Units | 00403 | | | |
| Tot. Alkalinity | mg/L CaCO3 | 00410 | | | |
| Hardness | mg/L CaCO3 | 00900 | | | |
| Suspended Solids | mg/L | 00530 | | | |
| Tot. Dissolved Soli | ids mg/L | 70300 | | | |
| Ammonia | mg/L (N) | 00610 | | | |
| Nitrate + Nitrite | mg/L (N) | 00630 | | | |
| Tot. Kjeldahl Nitre | ogen mg/L (N) | 00625 | | | |
| Tot. Phosphorous | mg/L (P) | 00665 | | | |
| Chloride | mg/L | 00940 | | | |
| COD | mg/L | | | | |
| Tot. Organic Carb | on mg/L | 00680 | | | |
| Fecal Coliform | MPN/100mL | 31615 | | | |

| - |
|----------------------|
| PARAMETER |
| TMDL Metals (ICP/MS) |
| Mercury |
| Semi Volatiles |
| Volatiles |
| Pesticides |
| Ortho Phosphorous |
| |
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| |
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| Type & Number of Samples: | Delivered by & Date: | Received by & Date: | Lab Label |
|---------------------------|----------------------|---------------------|-----------|
| | | | |
| Comments: | | | |

Chain of Custody Record

Georgia Department of Natural Resources Environmental Protection Division Laboratories 455 14th Street NW, Atlanta, GA 30318

| Matrix T | Гуре Defir | nition: S | 5 = Soil | or Semi | Solid, | W = Wa | ter (Aqueous), A | a = Air, | $\mathbf{N}\mathbf{A} = \mathbf{N}$ | lon Ac | ueou | ıs Liq | uid (| Oil, So | olvent | , Etc | .) | | | | | |
|---|------------|---------------|---------------------|--|-----------|------------------------------|-----------------------|-------------------|-------------------------------------|---------|--------|--------|-------|-----------|--------|-------|------|-------|-----------------|---|--|--|
| Facility: | | | | | | Location: Analysis Requested | | | | | | | | | | 1 | | | | | | |
| Sampler Name: | | | | | | Phone: | Phone: | | | | | | | | | | | | | | | |
| Address: | | | | | | FAX: | | | | | | | | | | | | | | | | |
| | | | | | | | 1 / 1/1. | | | | | | | | | | | | | | | |
| San | nple | Sam | nla Idant | fication | (In aluda | unique communie i | dantifian analy as as | mm la la / | | | | Matri | x Typ | <u>be</u> | N | mhor | of C | antai | iners Submitted | | | |
| Date | Time | Sam | pie ident | meation | (Include | unique sample i | dentifier such as sa | imple log | g number | 8.) | S | W | Α | NA | INU | mber | | ontan | | | | |
| | | | | | | | | | | | | | | | | | | | | | | |
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| | | | | | | | | | | | | | | | | | | | | | | |
| Relinquished By (Signature) Date Time Relinqui | | | iished By (Signatur | e) | Date | Tim | e | F | Reling | uished | By (Si | gnatu | ire) | | Date | Tin | ne | | | | | |
| Received By (Signature) Date Time | | | Recei | Received By (Signature) Date Time Received | | | | | eived B | y (Sigi | nature | e) | | Date | Tin | ne | | | | | | |
| 1 | | | | | · · | • | Laboratory | | i | • | | | | | | | | | | • | | |
| Received For Laboratory By Date Time Custody Intact (Signature) Image: Construction of the second sec | | Ustody Intact | Custody Seal No | Labo | ratory Re | emarks: | | | | | | | | | | | | | | | | |

Transfer of Custody with Shipment

- Samples shall be properly packaged for shipment.
- All samples shall be accompanied by the Laboratory Source Document, sealed in a watertight Zip-Loc® type bag if samples are shipped. The laboratory sample custodian is responsible for receiving custody of the samples and will fill in the "Received By" section.

Document Control

The term document control refers to the maintenance of records and reports produced during and as a result of field inspection, investigation, or survey activities. All files shall be maintained in accordance with WPB guidelines. All documents as outlined below shall be kept in the WPB files when completed, and approved if applicable. Investigators may keep copies of reports in their personal files, however, all official and original documents relating to inspections, investigations and surveys shall be placed in the official WPB files. The following documents shall be placed in the WPB file, if applicable:

- request memo from the program office;
- copy of the study plan;
- original bound field logbooks;
- 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;
- photographs and negatives 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.

Under no circumstances are any inappropriate personal observations or irrelevant information to be filed in the official WPB files. The project or study leader shall review the file at the conclusion of the project to insure that it is complete.

References

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SECTION 2 SAMPLING DESIGN AND QUALITY ASSURANCE PROCEDURES

Introduction

This section discusses the standard practices and procedures used by WPB personnel during field operations to ensure the collection of representative samples. Sampling activities are conducted by field investigators 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 requires:

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

Definitions

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

<u>Representative Sample:</u> a sample that reflects accurately one or more characteristics of a population.

<u>Sample Representativeness</u>: the degree of accuracy that a set of sample results defines the characteristics of a population, where each sample has an equal probability of yielding the same result.

<u>Variability</u>: 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 concentrations or values over time. An example would be the variable dissolved oxygen concentrations measured in one location in a reservoir over a period of twenty-four hours.

2. Spatial Variability

Spatial variability is the range of results due to changes in concentration or value as a function of their location. An example would be the variation in a metal concentration in sediment due to variations in sediment composition, depositional rates and other factors.

3. Sample Handling Variability

Sample handling variability is the range of results due to improper sample collection and handling. This variability is not a result of improper time or place of sampling. 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.

<u>Accuracy</u>: 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.

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

<u>Grab Sample:</u> an individual sample collected from a single location at a specific time or period of time. Grab samples are generally authoritative in nature.

<u>Composite Samples:</u> 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. An example is a sediment sample composited from quarter-points of a stream.
- 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 in 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), or evaluate field/laboratory variability (spikes and blanks, trip blanks, duplicate, split samples). The definitions for specific quality control samples are listed below:

<u>Control Sample:</u> a discrete grab sample collected to isolate a source of contamination. Isolation of a source may require the collection of both an upstream sample at a location where the medium being studied is unaffected by the site being studied, and a downstream control which could be affected by contaminants contributed from the site under study.

<u>Background Sample:</u> 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.

<u>Duplicate Sample:</u> 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.

<u>Trip Blanks</u>: 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.

<u>Equipment Field Blanks</u>: a sample collected using analyte-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. An example is a purified water chlorophyll <u>a</u> blank which is subjected to the chlorophyll filtering process.

<u>Field Blanks</u>: 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 vapors which could contaminate an organics and/or metals sample). Purified 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 predesignated 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.

Sampling Design

Introduction

The sampling design must meet specific study objectives. The location and frequency of sampling (number of samples) should be clearly outlined in the sampling design. Information on sampling design may be found in *Standard Methods for the Examination of Water and Wastewater*.

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.

Stratification and Heterogeneous Wastes

Environmental media, as well as waste matrices, 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. The trench would be considered a strata within the landfill if chromium was the contaminant of concern. A special case, "stratification by component", is often observed with waste matrices when the constituent of interest is associated with one component of the matrix. An example would be slag contaminated with lead that is mixed with otherwise uncontaminated fire brick. Thus the lead is stratified by component, that being the slag. Stratified sampling designs are discussed later which incorporate independent sampling of each strata, thereby reducing the number of samples required. A population with very localized strata or areas of contamination that are referred to as "hot spots".

Some environmental and waste 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.

Surface Water and Sediment Sampling Designs

Sampling Site Selection

The following factors should 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.

Before any sampling is conducted, an initial reconnaissance should be made to locate suitable 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. 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:

- Flowing water, including rivers, creeks, and small to intermittent streams; and
- 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.

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. 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;
- Areas of flow reversals; and
- Low or reduced velocity areas.

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. Considerations and criteria for station selection include:

- 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 historical locations when the data collected will be compared to a previous study.
- Whenever a marked physical change occurs in the stream channel. Example: A stream reach between two adjacent stations should not include both a long rapids section of swift shallow water with a rocky bottom, and a long section of deep, slow-moving water with a muddy bottom. Stations at each end of the reach would yield data on certain rates of change, such as reaeration, that would be an unrealistic average of two widely different rates. The actual characteristics of the stream would be better defined by inserting a third sampling station within the reach, between the rapids and the quiet water sections.
- 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. Such impoundments may be bracketed with sampling stations. When time-of-water-travel through the pools is long, stations should be established within the impoundments.

Some structures, such as dams, permit overflow and cause swirls in streams that accomplishes significant reaeration of oxygen deficient water. In such cases, stations should be located short distances upstream and downstream from the structures to measure the rapid, artificial increase in dissolved oxygen, which is not representative of natural reaeration.

When major changes occur in a stream reach, an upstream station, a downstream station, and an intermediate station should be selected. 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.

The use of three stations is especially important when rates of change of unstable constituents are being determined. If results from one of only two stations in a sub-reach are in error for some unforeseen reason, it may not be possible to judge which of the two sets of results indicate the actual rate of change. Results from at least two of three stations, on the other hand, may support each other and indicate the true pattern of water quality in the sub-reach.

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. Unless a stream is extremely turbulent, it is nearly impossible to measure the effect of a waste discharge or tributary immediately downstream from the source. Inflow frequently "hugs" the stream bank due to differences in density, temperature, and specific gravity, and consequently lateral (cross-channel) mixing does not occur for some distance. Tributaries should be sampled as near the mouth as feasible. Frequently, the mouths of tributaries are accessible by boat. Care should be exercised to avoid collecting water samples from stratified locations which are due to differences in density resulting from temperature, dissolved solids, or turbidity.

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 less than 20 feet wide, 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. A sediment sample could also be collected in the same vicinity if available.

For slightly larger streams, a vertical composite may be collected from mid-stream. Samples may be collected just below the surface, at mid-depth, and just above the bottom. For larger streams and rivers, at least quarter point (1/4, 1/2, and 3/4 width) composite samples may be collected.

For large rivers, several locations across the channel width may be sampled. Vertical composites across the channel width should be located in a manner that is roughly proportional to flow, i.e., they should be closer together toward mid-channel, where most of the flow is, than toward the banks, where the proportion of total flow is less. The number of vertical 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 sub-samples, the more closely the composite sample will represent the water body; and
- Sub-sample collection is time consuming and expensive, and increases the chance of crosscontamination.

In most circumstances, a number of sediment samples should be collected along a crosssection 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.

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. Since the stratification is caused by water temperature differences, the cooler, more dense river water is beneath the warmer lake water. A depth profile of the water column as well as visual observation of lake samples can often detect the different layers which can be sampled separately. 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 photic zone composite at the deepest point may be sufficient. The photic or euphotic zone is defined as that depth from the surface where 1% of the surface level irradiation remains (depth having 99% light extinction). The photic zone is measured using an underwater photometer with a surface light sensor, or is estimated from the measured Secchi depth. A composite sample is collected over the photic zone either by a continuously pumped sampling over this depth, or by compositing subsamples collected over this depth. 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).

A depth profile of dissolved oxygen, conductivity, pH, and temperature are generally measured from the surface to the bottom, at 1 meter intervals, for each vertical composite sample. In naturally-formed ponds, the deepest point is usually near the center; and in impoundments the deepest point is usually near the dam, over the original river channel.

In lakes with irregular shapes and with several bays and coves that are protected from the wind, additional separate composite samples may be needed to adequately determine water quality. Similarly, additional samples should be collected where discharges, tributaries, land use characteristics, etc., are suspected of influencing water quality.

When collecting sediment samples in lakes, ponds, and reservoirs, the sampling site should be approximately at the center of the water mass. This is particularly true for reservoirs that are formed by the impoundment of rivers or streams. 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. The shape, inflow pattern, bathymetry, and circulation must be considered when selecting sediment sampling sites in lakes or reservoirs.

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 sheet flow 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 shoreline.

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. Because of the dynamics of estuaries, the initial sampling results often reveal that the study objective could be better served by relocating, adding, or deleting sampling locations.

Water sampling in estuarine areas is normally based upon the tidal phases, with samples collected on successive slack tides. Estuarine sampling may 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 peristaltic pump are suitable.

A single vertical photic zone composite at the deepest point may be sufficient. The photic or euphotic zone is defined as that depth from the surface where 1% of the surface level irradiation remains (depth having 99% light extinction). The photic zone is measured using an underwater photometer with a surface light sensor, or is estimated from the measured Secchi depth. A composite sample is collected over the photic zone either by a continuously pumped sampling over

this depth, or by compositing sub-samples collected over this depth. Alternatively, samples may be collected at mid-depth in areas where the depths are less than 10 feet, unless the salinity profile indicates the presence of a halocline (salinity stratification). In that case, samples are collected from each stratum. Depending upon the study objective(s), when depths are greater than 10 feet, samples may be collected at the one-foot depth from the surface, mid-depth, and one-foot from the bottom.

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.

Control Stations

In order to have a basis of comparison of water quality, the collection of samples from control stations is always necessary. A control station upstream from the waste source is as important as are stations down gradient, and should be chosen with equal care to ensure representative results. In some situations it is desirable to have background stations located in similar, nearby estuaries which are not impacted by the phenomena or pollutants being investigated. At times it may be desirable to locate two or three stations downstream from the waste inflow to establish the rate at which the unstable material is changing. The time-of-water-travel between the stations should be sufficient to permit accurate measurement of the change in the constituent under consideration.

References

- American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF), 1992, *Standard Methods for the Examination of Water and Wastewater*, 18th Edition, Washington, D.C.
- APHA-AWWA-WEF, 1995, *Standard Methods for the Examination of Water and Wastewater*, 19th Edition, Washington, D.C.
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SECTION 3 WASTEWATER SAMPLING

Introduction & General Considerations

The objective of waste stream sampling is to obtain a representative quantity to analyze for parameters of concern. In some cases it is desired to accurately represent contents of the waste stream at a given moment; this is accomplished by a <u>grab sample</u>. It may also be advantageous for certain purposes to represent the waste stream characteristics over a period of time; this may be done by <u>composite sampling</u>. Considerations unique to each of these approaches will be discussed below.

Samples collected at wastewater treatment facilities are "split" with the permittee during certain types of Water Quality inspections. Sample splitting involves providing the facility representative with a set of samples that duplicate those taken by Branch personnel to the Water Quality Laboratory for analysis. Comparison of analytical results on such sample splits is an important component of self-monitoring program validation.

The QA/QC guidance set forth herein and the procedures referenced were originally developed for the purpose of compliance sampling at various types of wastewater-generating facilities regulated by the Branch. With the passage of time, the Branch has assumed additional responsibilities for environmental protection that go beyond traditional compliance sampling of influent and effluent. Storm water, Combined Sewer Overflows (CSOs), land-use associated runoff and other discharges to the State's surface waters are now or will soon be the object of sampling excursions by Branch associates. The guidance and requirements set forth below apply and may be adapted to these additional sampling tasks.

A general statement regarding protective gear for the sampling personnel is in order at this point. It is imperative that the health and safety of Branch personnel not be at risk during waste-water sampling operations. The threats from wastewaters encountered by Branch associates are manifold. Municipal/sanitary wastes pose obvious pathogenic hazards. Process wastewaters from industry may contain harmful concentrations of metals, solvents or chemicals such as sulfides, cyanide or chlorine. Some waste streams may be excessively corrosive or caustic. For these reasons, any Branch personnel engaged in or assisting with wastewater sampling should wear protective gloves and, in some cases, eye shields. (Gloves should be consistent with the level of threat as well as the nature of the parameters to be analyzed. For example, during metals sampling, latex gloves dusted with talc should <u>not</u> be worn). Industrial settings may require hearing protection and/or steel-toed boots. At a minimum, Branch associates should be familiar with *Part III* of the *DNR Safety Manual* prior to going into the field.

Protective equipment serves another purpose; that of ensuring the integrity of the sample collected. For the collection of samples to be analyzed for volatile organics, for example, it is required that a clean pair of Latex gloves be worn. Various products such as hand creams, cologne or hair preparations can contain chemicals that could be detected in a worst-case contamination

scenario. Branch sampling personnel should use good judgment when approaching each unique field assignment with these considerations in mind.

For any type of waste stream sampling the analytical parameters largely determine the major sampling protocols. That is, sampling methods, equipment, QA/QC procedures and sample containers must be consistent with the analytes of concern. For <u>any</u> sampling of wastewater, the sampling equipment must be clean and free of contamination that may have occurred during previous sampling events, equipment storage or transportation to the site. "Clean" in the sense of suitability for conventional parameter sampling (oxygen demand, solids, nutrients, et cetera) means either new equipment from a closed container or equipment that has been washed with a laboratory grade phosphorous-free detergent and rinsed with potable water.

Sample bottles/containers must also be appropriate for the analyses requested. The definitive reference and guidance for determination of sample containers, sample types (grab versus composite), required preservation techniques, maximum holding times and parameter-specific sampling considerations is *Title 40* of the *Code of Federal Regulations, Part 136*. Samples collected for NPDES reporting by the regulated community must adhere to the requirements of 40 CFR Part 136; Branch personnel must also follow these procedures for sampling results to be valid for enforcement purposes. A complete reiteration of the Federal guidelines is beyond the scope of this document. The *Code* is continually updated and revised by publications of notices in the *Federal Register*. In the event of a conflict regarding a method or technique, the most recent guidance appending the *Code* will rule.

The sample bottles used by Branch personnel are provided by EPD's Water Quality Laboratory. The correct types and amounts of preservatives are added to the containers by Laboratory personnel. All bottles and preservatives are consistent with the requirements of 40 CFR Part 136. Samples for certain parameters are required to be collected as grab samples only. Certain other samples must be collected directly into the bottle containing the preservative (examples include the Oil & Grease, Volatile Organics and Bacteriological samples).

EPD's Water Quality Laboratory has an extensive QA/QC program that includes such measures as analytical duplicates, spikes, blanks and analysis of known standards. Certain similar measures are carried out by field sampling personnel in the form of equipment blank samples (for metals sampling) and inclusion of "travel blanks" (for Volatile Organics sampling).

Sample bottles are labeled in the field either by writing with an indelible, waterproof marker directly on the bottle itself or by securing a special waterproof label to the bottle which has been inscribed with the necessary information. Bottle labeling information shall include, at a minimum, the following items:

- Name of facility where sample was collected
- Nature of the sampled substance (influent, effluent, etc).
- The date of sample collection
- The time or time interval represented by the sample
- The name of the sampling team leader

• Any special sample characteristics that could affect analysis for the parameters requested (facility dechlorinates with sulfur-dioxide, sample suspected of high concentrations of certain pollutants, etc.)

Records of samples collected are made at the time of collection in bound field books. This is to ensure that sample analysis results are "traceable" from the moment of sample collection to laboratory result report preparation. The records required include the name of the facility being sampled, sampling location, the substance sampled, the date and time of sampling and the identity of the sample collector and other team members present. If an automatic sampler is used, its serial number or DNR Property Control number must be recorded. Additional observations regarding unusual conditions, poor effluent quality or potential for adverse environmental impact should be noted. Primary responsibility for field book entry falls on the sampling team leader but may be delegated as the leader deems appropriate to expedite operations. Documentation of photographs taken, diagrams of sampling locations or treatment vessel layout and field data information must also be recorded in the field book. Calibration data for field instruments may be entered in the field book or separate QA/QC forms designed and used by individual work units.

Various specialized evaluation forms have been designed and implemented by Branch work units. Forms corresponding to the different types of inspections, investigations, audits, overviews and reconnaissance should be filled out on site with the necessary field observations as appropriate.

Metals Sampling

Sampling for metals (when other than "trace metal" detection limits are required) necessitates another level of equipment cleaning and preparation. Any component of the sampling equipment train that the sample contacts <u>must</u> meet these guidelines when metals are to be analyzed. The material of the sampling equipment must be plastic (polyethylene, etc.), Teflon, Tygon, Silastic, PVC or similar substances. (The use of glass sample bottles is now discouraged as detection limits become ever more sensitive). Materials that are <u>not</u> acceptable for metals sampling include brass, cast iron, aluminum, copper or galvanized containers. Although acceptable for conventional parameters sampling, leaching of metals into the sample makes these items unsuitable for samples collected for metals analyses.

The metals sampling equipment items must be either new or acid rinsed. Acid rinsing involves three separate rinse cycles with dilute (10%) laboratory-grade nitric acid, followed by three rinsing cycles with de-ionized water. Following acid-rinsing and air-drying, the equipment is covered or sealed to avoid contamination prior to use.

Trace-metal detection sampling involves yet another level of equipment preparation, QA/QC documentation and anti-contamination measures. Although the specialized methodologies are beyond the scope of this document, the Branch has developed guidance for trace-metal sampling which is used on those occasions when Branch personnel must sample for trace level metals. (*INTERIM PROTOCOL:Clean Laboratory and Sampling Techniques for Determination of Trace Metals*, DNR, EPD, WPB, March 1995).

Organics Sampling

Sampling for those analytes generally termed "organics" (volatiles, PCBs, pesticides, base neutrals, et cetera) also requires specialized containers and sampling equipment. Acceptable materials include glass, stainless steel and Teflon. Cleaning between sampling events must include a solvent rinse (laboratory- grade hexane or methanol) in addition to conventional cleaning, followed by a de-ionized water rinse. The equipment should then be covered with aluminum foil and/or clean plastic sheeting.

Sampling for Volatile Organic Compounds (VOCs) requires special attention. One set of VOCs consists of three (3) 40 milliliter septum vials with screw caps and a Teflon coated cap insert. An amount of concentrated hydrochloric acid is placed in each vial in the laboratory before delivery to the sampling personnel. The associate filling VOC vials must don a clean pair of Latex gloves. The vials must be filled with a minimum amount of turbulence to prevent volatilization; pouring the sample down the side of the vial is the generally accepted procedure. Care must be taken not to overfill the vials and lose the preservative. As the vials are filled, an amount of sample sufficient to leave a convex (or "positive") meniscus above each vial mouth should be poured. The Teflon liners and the securing threaded caps are then placed on the vials and sealed. The vials must be inverted to ensure that no air bubbles remain. If air bubbles are found, the procedure must be repeated.

If the wastewater to be sampled for VOCs contains chlorine, the sample aliquot must be dechlorinated with sodium thiosulfate or ascorbic acid before placement in the septum vials. The vessel used for the dechlorination procedure must meet the requirements for organics sampling equipment. Eight (8) ounces of sample may be dechlorinated with two (2) drops of a 25% ascorbic acid solution. Five-hundred milliliters (500 ml) of sample may be dechlorinated with eight (8) drops of a 10% sodium-thiosulfate solution. The sample being treated should be gently swirled to avoid volatilization as the dechlorinating agent is mixed. The sample may then be transferred to the 40 ml vials as noted above.

Bacteriological Sampling

Bacteriological samples must be collected directly into sterile containers provided specifically for this purpose by EPD's Water Quality Laboratory. If residual chlorine is present, the bottles must have an appropriate amount of sodium-thiosulfate added prior to sterilization. The cap must be removed from the container for the minimum amount of time necessary to collect the sample. Care must be taken not to contaminate either the cap or the inside of the sample bottle. During sampling the bottle should be inserted into the waste stream with the neck partially below the surface and tilted slightly upward with the mouth directed against the current. The bottle should be filled to within approximately one inch of the top in order to provide adequate mixing space.

If the sample container must be lowered into the waste stream for reasons of safety or accessibility, extra care must be taken to avoid extraneous contamination and to prevent loss of the sodium thiosulfate through overfilling the vessel.

Oil & Grease / Immiscible Liquids Sampling

Oil and grease may be present as a surface film, a solution, an emulsion or a combination of these forms. The area of greatest mixing is the preferred sampling location. The sample must be collected directly into the bottle provided by the Water Quality Laboratory. With the mouth of the bottle facing upstream, it should be filled as completely as possible without losing the preservative.

Process Control Sampling

Branch personnel perform Technical Evaluations at treatment works of particular concern. Process control tests are run on activated sludge system component streams such as aeration basin mixed liquor, return activated sludge, settled sludge and primary and secondary effluents. Due to the very high solids concentrations of some of these fluids, obtaining a representative sample is especially critical. Recommended sampling sites include return sumps or pits, junction boxes, aeration basin overflow weirs and near mechanical aerators or compressed air diffusers because flow at these locations is typically very turbulent, providing a well mixed sample. In the case of very large aeration basins which are not uniformly mixed or aerated, an areal composite may be necessary. Several samples from widely dispersed locations in the basin are mixed to form a composite basin sample.

Most of these types of samples will be manual grab samples, although in some cases an automatically collected time-composite may be required. For certain substances of extremely high solids content (digester contents, dewatered solids and return sludge) the Water Quality Laboratory may provide specialized containers for certain tests.

Although procedures on process control testing and trouble-shooting are beyond the scope of this document, the reader is referred to the EPA's *Activated Sludge Process Control Testing* handbook and their *Process Control Manual: Aerobic Biological Treatment Facilities*. The California State University at Sacramento's *Operation of Wastewater Treatment Plants* (Volumes I, II, and III) also has valuable practical information.

Sampling Site Selection

Additional consideration must be given to selection of the sampling location. Branch personnel encounter a wide variety of conditions and wastewater conduit configurations during their field work. It would be impossible to design guidance that would address every possible sampling situation. On site judgment must be exercised on each occasion to assure that the samples collected satisfy the purpose and intent of the sampling effort. General guidelines may be detailed, as follows:

- Samples should be taken from a location where the waste-stream is well mixed, near the center of the channel.
- Care should be taken to select a location that will not unduly bias any parameter positively or negatively.
- Note that the sampling location defined in the facility's permit (if a permit has been issued) is not necessarily the optimum location for sampling.

- In the case of influent sampling at a wastewater treatment works, it is desirable to sample <u>upstream</u> of any "return" flows, such as filtrates, centrates, washdown water, return activated sludge, supernatants, etc., to the extent possible.
- When sampling wastewater treatment effluents, the <u>final</u> effluent is often specified as being the point of application of discharge limitations; however, there are many exceptions to this relevant chemical addition (chlorine and dechlorinating agents, defoamer, supplemental oxygen, etc. In the event of conflicting protocols, more than one sample may be collected.

Additional guidance may be found in EPA's *NPDES Compliance Sampling Inspection Manual*, which has been adopted by the Branch as its main reference for wastewater sampling standard procedures.

Grab Sampling & Manual Sampling

Grab samples consist of either a single sample or individual samples collected over a period of fifteen minutes or less. The quantity collected is determined by the type and number of analytical parameters. Grab sampling is useful when the waste stream is not continuous, such as the cases of batch discharges and intermittent flows. Grab sampling may also be appropriate when the characteristics of the waste stream are known to be constant through time. Samples for certain parameters are required to be collected as grab samples. By collecting a series of grab samples on a continuous discharge, valuable data on maximum and minimum concentrations may be obtained that would be missed by the compositing process.

As discussed above, sampling equipment and preparation must be consistent with any special requirements of the parameters. In the case of sampling for a broad suite of analytes, multiple protocols may need to be followed, necessitating a variety of sampling devices of different materials.

Grab samples are most often collected manually. In-situ field determinations and on-site analyses are also performed on manually collected grab samples. When grab sampling for laboratory parameters, it is desirable to collect the sample directly in the bottle(s) provided by the laboratory. In the case of bottles containing preservatives, care must be taken not to overfill the bottle and lose the preservative. When an intermediate sample container must be used for reasons of safety or accessibility, it must meet the material and cleaning requirements for the parameters being sampled. Samples collected for analysis of Oil & Grease, Bacteria, Phenols, Sulfides and Volatile Organics must always be collected directly into the laboratory sample bottle.

Sometimes it is advantageous to collect grab samples by use of a pump. If a pump is used, the same considerations apply as for an intermediate sample container. All internal pump components that contact the sample must meet the appropriate guidelines for the parameters being analyzed.

Certain grab samples need to be taken from a tap, valve or other outlet as opposed to an open channel. When this is necessary, allow sufficient fluid to run out of the source in order to purge the piping of stagnant material prior to sampling.

Whatever method is chosen, the preferred location for grab sampling is one that is well mixed so that a representative sample may be obtained. The accepted procedure is to dip the sampling vessel in the waste stream so the mouth of the container faces upstream.

Composite Sampling

A composite sample consists of a number of individual sample aliquots that are collected over a period of time. The purpose of composite sampling is to accurately reflect the average characteristics of the waste stream over this time period. Composite sampling is most appropriate when the waste stream flow is continuous (or nearly so). Concentration data from composite samples allow pollutant mass loadings to be calculated. Composite samples may be collected manually, but the most common method utilized by Branch personnel involves the use of automated sampling equipment.

As with all sampling methods, the equipment used and its preparation must be consistent with the corresponding requirements of the parameters to be analyzed. In the case of automatic sampling devices, this includes sample intake lines, strainers, internal pump components, sample aliquot bottles and any other compositing vessels which contact the sample. Teflon sample tubing is available for compositing organics (except for volatiles which must always be collected as grab samples). Sample strainers of various materials are in use, including Teflon and stainless steel models. Internal sample aliquot bottles made of glass and plastic are on hand. All internal sampler components must also be cleaned according to the corresponding parameter protocol. For example, when sampling for metals, dilute nitric acid must be circulated through the pump mechanism; individual aliquot bottles must be acid-rinsed. The Branch no longer uses older model sampling equipment which had numerous internal components for routing sampled fluid to individual bottles. The current designs distribute sample directly to the aliquot bottles by way of a flexible tube which is part of the pump assembly itself. This greatly reduces the amount of material which must be properly prepared.

Most parameters of interest to Branch personnel require that samples collected for their analysis be kept "cool" (approximately four degrees Centigrade) during the compositing period. All automatic samplers in use by the Branch have internal chambers for the placement of ice to meet this requirement. In the case of high ambient temperatures during summer months or locations inside factories, replenishment of the ice during the compositing interval is necessary.

Additional concerns pertaining to the use of automatic sampling equipment include:

- The pumping mechanism must be able to lift fluid a vertical distance of at least twenty (20) feet at a velocity of at least two (2) feet per second.
- Aliquot volume must be adjustable; aliquot volume should be verified at the start of the compositing period with a graduated cylinder (100 milliliters minimum).
- The sample intake line must be purged prior to collection of each aliquot.
- Clean or new sample intake tubing must be used for each installation.
- The minimum inside diameter of the sample intake line must be 1/4 inch.

- Care must be taken to ensure suitable mixing when decanting sample from one container to another. This applies to the compositing of individual aliquot bottles as well as the filling of laboratory sample bottles.
- When using an automatic sampler to composite samples for analysis of metals, an equipment blank must be collected. The complete sample collection train (including strainer, sample intake line, pump tubing and a number of aliquot bottles) must be rinsed with organic/analyte free water and a portion directed into a metals sample bottle for analysis by the Water Quality Laboratory.
- If the sample intake tubing must be attached to metal pipe for proper positioning in the wastestream, the tubing should be mounted upstream of and away from the pipe or conduit. If possible, the submerged portion of the pipe should be wrapped with protective material.
- When using an automatic sampler for Organic, PCB or Pesticide sample collection, internal glass bottles must be used that have been solvent-rinsed. The sample intake line and strainer must be Teflon. An equipment blank must be collected and analyzed for the same parameters as the sample itself. New pump tubing must be installed. The power source must be capable of running the equipment for the entire compositing period, plus a reserve capacity for grab sample pumping (if necessary). Facility electrical outlets may be used. For locations where power is not available, nickel-cadmium or lead-acid batteries will be used. For extended sampling investigations, deep-cycle marine batteries may be used.

In some situations it may not be possible to install a Branch automatic sampler. The desired sample point may be within a force-main or may be inaccessible for safety or other reasons. In such cases the facility often has installed specialized sampling devices to accommodate the unusual condition. It is permissible to use a facility's sampler if it meets the protocols required for the parameters to be analyzed.

The Branch's automatic samplers are assumed to be in the custody of the facility while they are on their property. This assumption should be presented to the facility representatives when equipment is left unattended. Samplers may be locked in such a way that the composite sample bottles and the sampler's controls cannot be accessed, however, this does not prevent tampering with the sample intake line and strainer. Seals may be placed on the intake line to reduce the possibility of tampering.

Specific operating instructions for each type of automatic sampler are beyond the scope of this document. The reader is referred to the user manuals which accompany the equipment. These manuals also provide valuable data on pump performance, battery life and maintenance requirements.

The automatic samplers must be kept clean and maintained to ensure proper performance and avoid sample contamination. Each unit should have a log maintained of its maintenance and repair history. The log should identify the samplers by their manufacturer's serial number, model number and DNR Property Control number. Entries should be made in the log at least quarterly denoting the status of the sampler (operable, inoperable, sent off for repair, et cetera). The following procedures

should be performed at the frequency indicated:

- Prior To Field Trip Sample pump operation; forward, reverse and automatic modes tested. Desiccant, fuses and batteries checked; batteries charged.
- After Each Field Trip Outside of sampler cleaned; inside of sample bottle tub rinsed. Used sample bottles replaced with clean ones. Distributor retention nut tightened. Pump tubing cleaned. Sampler body checked for cracks and leaks and repairs made.
- Quarterly Verify forward, reverse and automatic pump operation modes. Tighten control box mounting bolts. Examine internal pump hose for splits and cracks, replace if worn. Lubricate pump hose with silicone. Lubricate pump impeller drive spindle with light machine oil.

The following identification protocol should be followed to denote the status of sampler bottle tubs and mechanical condition:

- The wide latch on the bottle tub should be tagged "Acid Rinsed" when bottles prepared for Metals sampling are installed. The tag should be removed when the sampler is placed in use in the field. If the sample bottles have been prepared for Organics sampling, the tub should be tagged "Solvent Rinsed."
- After use in the field, the wide latch of the bottle tub should be flagged with brightly colored surveyor's tape to indicate that the bottles require cleaning prior to use again.
- A sampler which suffers a mechanical problem should have one of the carrying handles on the sampler body tagged with bright tape <u>and</u> a sample tag inscribed with a brief description of the malfunction.

Composite samples may be "flow-proportioned" or "composited according to flow" in order to more accurately reflect the nature of the waste stream over time. The basic concept is that periods of greater discharge should be represented in the composite by a correspondingly larger volume of sample. This procedure is a requirement of most Water Quality permits issued by the Branch. It is vital that all Branch personnel engaged in composite sampling be familiar with flow proportioning, not only to enhance the representativeness of their own samples but to be able to demonstrate the procedure to members of the regulated community on request.

There are two methods of flow proportioning a composite sample:

- 1. Constant-interval/variable volume
- 2. Variable-interval/constant volume

The first of these, constant-interval/variable volume consists of individual aliquots collected at uniform time intervals. At the end of the compositing period, varying amounts of each aliquot are composited into a separate vessel depending on the available flow information for the respective

aliquot collection time. For example, aliquots of 800 milliliters (ml) could be collected at one-hour intervals. If the maximum flow rate during the compositing period was 4.0 MGD, then the amount of sample to be used from each aliquot could be calculated by multiplying the flow (in MGD) by a "proportioning factor" of 200. Thus, for periods of maximum flow, all of the aliquot would be used, intervals of flow rate 2.0 MGD would be represented by 400 milliliters per aliquot, etc. This approach is subject to certain assumptions and requirements:

- 1. The sampler must be equipped with a sufficient number of individual aliquot bottles.
- 2. Reliable flow information for the aliquot collection times must be available.
- 3. The amount of sample obtained after flow proportioning must be adequate for all parameters to be analyzed, plus provide for sample "splitting" with the facility.
- 4. Volume of the smallest aliquot portion composited must be ≥ 100 milliliters.
- 5. The vessel used for aliquot measurement (graduated cylinder) must be cleaned in accordance with the protocols for the sampling parameters.

The other flow-proportioning method, variable-interval/ constant volume entails interfacing the automatic sampling device with a flow measurement system. The composite is proportioned by collecting a uniform volume aliquot per set discharge amount. For example, 200 milliliters of sample could be composited for every 50,000 gallons of discharge. It may be seen that during intervals of greater discharge rates, the aliquots will be collected more frequently, representing such periods with proportionally greater sample volume. The advantages of this approach include:

- 1. Compositing is done automatically, reducing time required for this task.
- 2. Any internal sampler bottle configuration may be used (including a single large vessel, again reducing time requirements).
- 3. Sample aliquot handling is reduced, avoiding a possible contamination vector.
- 4. The minimum composite aliquot volume requirement of 100 milliliters is never compromised.

Possible drawbacks of the variable-interval/constant volume method may include:

- 1. Appropriate flow measurement system may not be available, or may not be accurate.
- 2. The flow pattern during the compositing period may not be as anticipated resulting in an insufficient volume of sample being collected (or too much sample may be collected causing shutdown of the sampler before the compositing interval is complete).

If neither flow-proportioning method is possible, sample aliquots will have to be composited on a constant-volume/uniform interval basis. In some cases, samples collected in this manner may not be acceptable for enforcement purposes. The flow proportioning requirement does not apply to those instances where it can be shown that the instantaneous flow rate did not vary by more than 15% during the compositing interval.

Groundwater Sampling

Certain types of wastewater treatment facilities (most notably Land Application Systems and Overland Flow facilities) pose a threat to subsurface waters. Monitoring wells are mandated by the Water Quality permits for such facilities. Sampling of subsurface waters via these constructed wells is required of the permittees at varying frequencies for different parameters.

Occasions arise when Branch associates are called upon to sample groundwater monitoring wells. EPD has a guidance document, *Manual For Groundwater Monitoring* (1991), which contains the necessary QA/QC measures and sampling methodologies to assure integrity and representativeness of samples collected from the subsurface. When sampling fluids from monitoring wells, the guidelines of the *Manual* must be combined with guidance from this document regarding appropriateness of sample containers, preservatives and other considerations for the parameters to be analyzed.

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SECTION 4 FLOW MEASUREMENT

Wastewater Flow Measurement

Introduction & General Considerations

Flow measurement is necessary on either an instantaneous or cumulative basis for a variety of uses. Peak hydraulic load and piping requirement calculations depend on accurate flow information. Pollutant loading (mass) calculations require representative flow data. Analyses of transient events at treatment works are greatly aided by high-resolution flow records.

The standard references for wastewater flow measurement include the EPA's *NPDES Compliance Sampling Inspection Manual*, the *ISCO Open Channel Flow Measurement Handbook* and the *Stevens Water Resources Data Book*. The *Water Measurement Manual* from the U.S. Department of the Interior is also frequently cited.

Locations For Measuring Flow

Flow is commonly measured at various places in wastewater treatment works. Influent, effluent, returned and recirculated flows are the most often measured fluids. The preferred location for flow measurement will depend on the wastestream in question. Untreated wastewater prior to any return flows should be monitored in the case of influent. For effluent flow measurement, a site that is downstream of all diverted amounts (utility or washdown water, for example) should be measured so that the true discharge amount may be determined. Process flows require unique considerations depending on the purpose of measurement. If measurement excluding diverted amounts is not possible, the quantities attributable to such flows must be accounted for (or estimated).

The various methods of flow measurement have in common the need for a restriction in the flow channel so that the measurement can be made. In the case of open channel measurement (the most common) unique properties of the flowing-head/discharge relationship make accurate flow determinations possible. Other systems rely on pressure differences, induced electrical current, acoustic and other properties in specially prepared pipe or channel runs to determine flow rates and volumes. Flow measurement location will depend on availability of a suitable place in the flow conduit where such a prepared restriction may be positioned. Certain devices are susceptible to errors induced by unfavorable piping configurations upstream or downstream of the metering run.

It may be seen that the most suitable location for flow measurement from a "plumbing" standpoint is not always favorably sited in relation to return or recycled flows. Each situation is unique and the team leader must use judgment in evaluation of the existing system or placement of his own temporary flow monitoring equipment.

Open Channel Flow Measurement

Wastewater measurement by the use of weirs, flumes and similar primary devices in open channels is most often encountered in the field. Their accessibility for manual measurements is a major advantage; calibration of any associated mechanical or electronic devices may be easily verified. In the absence of such secondary devices instantaneous flow measurements may be made manually and may be adequate for some applications. Manual instantaneous flow readings require only the primary device in the wastestream and a means to measure the flowing head (staff gauge, carpenter's square, or other rule). Several of the references cited above contain discharge tables for the various types and sizes of common weirs and flumes. In the case of a primary device unique to a given facility, the discharge performance table or curve in the Operation & Maintenance Manual for that facility should be consulted. (These are most often some type of "open flow nozzle").

Some type of flow indicator or recording device is more commonly found in conjunction with the weir or flume. Instantaneous flow indicators may be analog or digital and they may be calibrated in various units (GPM, CFS, MGD, etc). Recording graphs that represent the "ups and downs" of the flow regime may be made on strip paper or circular charts or even computer generated at various time compression/expansion scales. Some type of flow accumulator or "totalizer" is almost always included in such a system. This allows cumulative flows to be determined for any desired interval (usually daily).

The interface between the weir or flume and the recording instruments consists of a sensor to measure the depth of flowing head and the necessary connection (mechanical or electronic) to the flow meter. Mechanisms or circuits in the flow meter convert data on flowing head to indications of flow rate. Head sensors include floats, bubble systems, electrical conductance units, ultrasonic transducers, submerged pressure probes and other less common types. The circuitry or mechanism in the flow meter must be appropriate for the primary device in use; that is, it must be properly configured or programmed.

Each component of the flow measurement system must be evaluated by the field inspector to determine if the values obtained are suitably accurate. In wastewater measurement, an error of +/-10% is generally considered acceptable. If a facility's flow monitoring system is found to be within a ten percent calibration accuracy compared to the actual flow (determined by manual head measurement), it may be used for purposes of the investigation or inspection. If the existing flow meter is outside of this calibration range and cannot be readily adjusted, the field investigators will have to install an EPD flow meter at the primary device. Flow meters in use by the Water Protection Branch include both bubblers and submerged pressure transducer types. Both have proven to be highly accurate; both are especially suited for certain applications. Installation of a portable meter will also be necessary at facilities where the permit requires instantaneous flow measurement but continuous flow data is needed for the purposes of the investigation.

The primary device itself must also be evaluated for conformity to standard dimensions, proper installation and free flow conditions. Turbulence through the metering run should not be excessive, especially at the head measurement point. Weirs should be of the "sharp" or "narrow-crested" variety (approximately 1/8" thickness, preferably with a 45 degree bevel on the downstream

side) unless documentation is available detailing the head/discharge relationship for the particular installation. The references should be consulted for guidance on requirements unique to each type of device. Prefabricated flume structures may be considered to conform to standard dimensions if they are properly oriented and installed in the wastestream.

In primary device evaluation, such instruments as spirit levels, carpenter's squares, protractors and tape measures or rules are helpful. Weirs should be checked for squareness across the channel and level orientation as viewed from directly downstream. V-notch weirs should be checked for the proper angle. Ventilation of the nappe should be verified along with the minimum distance to the channel floor downstream of the weir.

The most common problem noted with flumes is submerged or restricted flow in or immediately downstream of the flume throat. If the conditions of submergence are severe the head/discharge relationship is affected and the flume may not be sufficiently accurate. Measuring the flowing head at an incorrect location is also common. Excessive turbulence in flumes is often caused by piping irregularities upstream of the converging or approach section. Prefabricated flumes have been found installed backwards in the channel; flow measurements based on such an improperly installed flume are invalid.

When setting up a flow meter in the field, certain considerations are common to both types of meter and all types of open channel primary devices. The head sensor must be installed at the proper location dictated by the type and size of the primary device. For weirs, this is generally four or more times the maximum anticipated head measurement <u>upstream</u> of the weir crest. This is to offset the "drawdown" effect as the wastewater discharges over the weir plate. Flumes of the various standard designs have definite locations which are meant for head measurement; a reference should be consulted prior to each field installation. In the case of very small flumes (throat width of three inches or less) care must be taken to ensure that the placement of the sensor does not restrict or cause turbulence in the flow.

Technical details regarding operating procedures of the various flow meters in use in the Branch are beyond the scope of this document. The operating manuals that accompanied the equipment should be consulted for particulars. The same considerations apply to Branch field meter installations as for the permittees, that is, the meter must be properly programmed for the type and size of primary device in use and the chart recorder must be set to encompass the full variation in flow rates while also giving the degree of resolution required.

Sufficient information on the installation must be recorded in the field book (or dedicated form) to document the accuracy and validity of the flow measurements obtained. The data to be recorded must include at a minimum:

- Model and serial number (or DNR number) of the meter used;
- The type and size of the primary device;
- Scaling constants and maximum allowable head values programmed into the meter, if applicable;
- Units flow is to be measured in (gallons, cubic meters);

- Chart recorder "full scale" value;
- The date and time of the beginning and end of the flow monitoring interval, as well as any calibration checks;
- "Zero" adjustment of the meter (if applicable);
- The results of a calibration check of the Branch meter at the start of the measurement interval including an actual head measurement and the head readout on the meter at the same time; and
- The results of a calibration check at the conclusion of the measurement interval.

Some of the latest model flow meters in use by the Branch will print a "report" on the chart itself which contains most or all of the required information. This is considered adequate documentation.

Closed-Channel Flow Measurement

Methods of accurately measuring flow in pipes and other closed conduits have been developed and are in use. They include venturi meters, orifice meters, magnetic meters, turbine meters, acoustic meters, pump meters and several others. Each type of meter has its own installation and application idiosyncracies.

At this writing the Branch has no meters that operate on closed-channel principles. Although these types of meters are very accurate in permanent installations, the portable equipment available for these situations is not acceptably accurate for regulatory purposes. In the case of the need to determine if such a meter is in proper calibration, a different flow measurement method will have to be employed.

Alternate Flow Measurement Techniques

Other methods are available for determining instantaneous and cumulative flow values. Each field situation is unique and the need to improvise and adapt these methods is evident. Some of the more common approaches are detailed below:

- Vessel Volume This is particularly well-suited to batch discharges. When the liquid capacity of a vessel is known it is a simple matter to note the time required to wholly or partially fill it. Information on multiple fill cycles is necessary when the vessel is small relative to the discharge volume. If the vessel is a nonstandard size or the capacity is not known it must be measured and the volume calculated.
- Sump and Wet Well Measurements These are variations of the volumetric technique above. In many cases the vessel will continue to receive flow while it is being decanted or pumped down; the incoming flow volume during these cycles can be significant and must be accounted for. The other considerations given for vessel volume also apply.

- Bucket & Stopwatch This method is best suited for very small flows; it is another adaptation of the vessel volume approach. A bucket, drum or tank of known capacity is inserted into the discharge stream and the fill time is recorded. A minimum of ten seconds is recommended as the time needed to fill the container. Three measurements should be made and the results averaged.
- Dilution Methods Dilution methods for measuring flow are based on the quantifiable property of a tracing substance injected into the wastestream. They require specialized equipment and an experienced investigator. They are also time consuming. They have the advantage of being adaptable to situations where flow measurement is not possible by any other means. They are useful for verifying the calibration of closed-conduit devices. The references contain specific methodology on dilution methods of flow measurement. The Rhodamine WT dye method is the preferred method; the dye is available to Branch personnel when this approach must be used.
- Manning Equation (also known as the Slope/Area Method) If the slope of the water surface in a conduit or pipe is known along with the cross-sectional area of the waste-stream the flow rate may be calculated. Other data on the degree of smoothness of the piping material must also be available. Errors in slope values and smoothness data greatly affect the accuracy of the calculation. The latest model flow meters in use by the Branch may be programmed to measure flow by this method.
- Velocity-Area Method Where the cross-sectional area of the wastestream can be measured and the average velocity determined the flow may be readily calculated. The average velocity may be quantified by the use of a current meter such as is used in stream gauging. This technique may be used in an approach channel upstream of a weir or flume as a calibration check for these devices.

Surface Water Flow Measurements

Introduction

Surface waters are considered to be open channels for flow measurement purposes. All of the techniques utilized by field investigators to measure open channel flows have been discussed in the wastewater flow measurement portion of this section. Except for very small surface streams, the installation of primary flow devices is not practical. Most surface water flow measurements are made utilizing classic stream gaging techniques. These techniques involve the use of the velocity-area open channel technique which was discussed in the wastewater portion of this section. Water Protection Branch personnel shall use the techniques outlined in the referenced United States Geological Survey (USGS, Department of the Interior) publications:

- select the flow measuring site
- perform stream gaging
- calculate flow

Techniques

Whenever possible stream studies shall be conducted utilizing existing permanent stream gaging stations operated by the USGS, the U. S. Army Corps of Engineers (COE), or other federal or state agencies. These permanent gaging stations have established water stage-discharge relationships that permit the flow to be obtained from water stage measurements. Staff gage or recorder readings of water stage at these stations may be converted to flow by using the rating curve for that gaging station. The rating curve is generally available from the operator of the permanent gaging station. An additional benefit of utilizing these permanent gaging stations is that long-term flow records are generally available. These long-term flow records are invaluable in planning water quality studies and assessing data trends.

Where permanent gaging stations do not exist, surface water flow will have to be measured utilizing classic stream gaging techniques. If a station is to be used on a long term basis, rather than a few times during a water quality survey, a rating curve may be developed for that station. This may be done by making a series of independent flow measurements and simultaneous tape down or staff gage measurements for that station at different water levels. The rating curve is developed utilizing the same measurement section each time. The flow measurements used to develop the rating curve must bracket the lowest and highest flows encountered and have sufficient points that the rating curve is well defined. A stage rating curve is constructed by plotting the stage measurements versus flows on log or semi-log graph paper, or by regression analysis using computer software.

Equipment

The equipment available for the measurement of surface water or wastewater flows is categorized as follows: water level/stage hardware and recorders, velocity measuring equipment and assemblies, and direct flow measurement equipment and instrumentation.

The hardware available to determine the rise and fall of a water surface with time (the water stage) includes Stevens stage drum recorders. Non-recording equipment available includes vertical staff gages and tape-down systems.

Instruments and equipment available to make velocity cross-sectional area measurements include current meters and sounding (depth) equipment. The current meters available are the vertical-axis mounted Price AA and Price pygmy meters (including cat-whisker and magnetic head with data logging). Sounding (determination of depth) is accomplished using a Raytheon recording fathometer or with a standard top setting wading rod. Width measurements are made using metal tapes, steel tag lines which are segmented into equal divisions of length by metal beads or marked Kevlar tag lines.

Specific Equipment Quality Control Procedures

A log book shall be kept of all equipment utilized for measuring water flows, water stage/tape downs, velocity measurements, flow recordings.

Price AA and Price Pygmy Current Meters

All meters shall be examined before and after each discharge measurement. The examination shall include the meter cups or vanes, pivot and bearing, and shaft for damage, wear, or faulty alignment. Meter balance and alignment shall be checked prior to each use in the field. Meters shall be cleaned and oiled daily when in use. Surfaces that shall be cleaned and oiled on a yearly basis are the pivot bearing, pentagear teeth and shaft, cylindrical shaft bearing, and thrust bearing at the cap.

Top Setting Wading Rod

This equipment shall be cleaned and examined before and after each discharge measurement. The examination shall include a check on the sliding rod and lock set mechanism.

Tag Lines and Tapes

This equipment shall be inspected for damage and cleaned before and after each discharge measurement. Tag line(s) and tapes shall be accurate to 0.1 foot per 100 feet. Tag lines or tapes that do not conform to this accuracy specification shall be repaired, recalibrated, or discarded. Yearly maintenance shall include an inspection for potential breaks, a thorough washing, and a finish oiling.

General Quality Assurance Procedures

Techniques and procedures for making wastewater and surface water flow measurements are outlined in this section. The United States Department of the Interior (USDI) *Water Measurement Manual*, the USGS *Discharge Measurements at Gaging Stations*, the U.S. EPA *NPDES Compliance Inspection Manual*, and a set of weir and flume tables are basic flow references for field investigations. The measurement of waste and stream flow requires considerable experience. Therefore, no field investigator shall make flow measurements until they have been trained 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 (second feet) or the metric equivalent (m^3/sec) . Time records associated with flow measurements shall be kept in local time, be made in the 2400 hour military time format, and shall be recorded to the nearest five minutes. All flow measurements conducted shall be thoroughly documented in field records. All measurements shall be traceable both to the individual making the measurements and the equipment utilized.

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SECTION 5 SURFACE WATER SAMPLING

Introduction

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

Sampling station characteristics often dictate the equipment to be used. If surface-level water samples are required, direct dipping of the sample container into the stream is desirable. This is possible using a boat, pier, or bridge for access, or by wading when stream depth permits. Wading, however, may cause the re-suspension of bottom deposits and bias the sample. Wading is acceptable if the stream has a noticeable current, and the samples are collected while facing upstream. If the stream is too deep to wade, more than one water depth must be sampled, or the sample must be collected from a bridge or other access point, supplemental sampling equipment may be needed.

Surface Water Sampling Equipment

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 should face upstream and collect the sample without disturbing the sediment. The surface water sample should always 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 such as a nutrient sample bottle.

<u>Scoops</u>

Stainless steel scoops are useful for reaching out into a body of water to collect a surface water sample. The scoop may be used directly to collect and transfer a surface water sample to the sample container, or it may be attached to an extension in order to access the selected sampling location.

Peristaltic Pumps

Another device that can be effectively used to sample a water column is the peristaltic pump/vacuum jug system. The use of a metal conduit to which the tubing is attached, allows for the collection of a vertical sample (to about a 25 foot depth) which is representative of the water column. Commercially available pumps vary in size and capability, with some being designed specifically for the simultaneous collection of multiple water samples.

Discrete Depth Samplers

When discrete samples are desired from a specific depth, and the parameters to be measured do not require a Teflon® coated sampler, a standard Kemmerer or Van Dorn sampler may be used. The Kemmerer sampler is a brass cylinder with rubber stoppers that leave the ends of the sampler open while being lowered in a vertical position, thus allowing free passage of water through the cylinder. The Van Dorn sampler is plastic and is lowered in a horizontal position. In each case, a messenger is sent down a rope when the sampler is at the designated depth, to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill respective sample containers. With a rubber tube attached to the valve, sample bottles can be filled. With multiple depth samples, care should be taken not to stir up the bottom sediment and thus bias the sample.

A second type of discrete depth sampler has sample intake and air discharge ports that have plastic plugs in place until pulled out via spring loaded tethers at the desired sampling depth. The air being evacutated creates a siphoning effect pulling water into the chamber. The housing is polypropylene and is designed to accept a 300 mL Winkler BOD bottle if used for collection of dissolved oxygen samples. The WPB has two sizes of these samplers that will collect either 1400 or 2000 mL of sample.

Bailers

Teflon® bailers may also be used for surface water sampling, if the study objectives do not necessitate a sample from a discrete interval of the water column. A closed top bailer with a bottom check-valve is sufficient for many studies. As the bailer is lowered through the water column, water is continually displaced through the bailer until the desired depth is reached, at which point the bailer is retrieved. This technique may not be successful where strong currents are found.

Buckets

A plastic bucket can be used to collect samples for *in-situ* analyses, (e.g., pH, temperature and conductivity). However, the bucket should be prerinsed with the sample water prior to collection of the sample.

Sample Handling

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 should be used when small sample containers (e.g., VOC vials or bacterial samples) are placed in ice chests to prevent cross-contamination. Trace metals sampling is to be conducted according to the WPB document, *Interim Protocol: Clean Laboratory and Sampling Techniques for Determination of Trace Metals*, and U.S. EPA trace metal sampling protocols.

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SECTION 6 SEDIMENT SAMPLING

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 the sediment should be obtained.

Station characteristics may dictate the equipment to be used. Wading is the preferred method for reaching the sampling location, particularly if the stream has a noticeable current. However, wading may disrupt bottom sediments causing biased results. Deeper areas where stream velocities are reduced are sediment deposition areas. If the stream is too deep to wade, the sediment sample may be collected from a boat or from a bridge. Regardless of the method used, precautions should be taken to insure that the sample collected is representative. These methods are discussed in the following pages. To collect a sediment sample a variety of equipment can be used such as:

- Dredges (Peterson, Eckman, Ponar),
- Coring (tubes, augers)
- Scoop

Sediment Sampling Equipment

Scoop

If the sampling station is wadeable, the easiest way to collect a sediment sample is by using a stainless steel scoop. This sampling method is accomplished by wading into the stream while facing upstream (into the current), scooping the sample along the bottom in the upstream direction. Excess water may be removed from the scoop or spoon. However, this may result in the loss of some material. In water bodies that are too deep to wade, but less than eight feet deep, a stainless steel scoop attached to a piece of conduit can be used either from the banks, if the surface water body is narrow, or from a boat. If trace metal analysis is to be conducted on the sample, scoops made of polypropylene or Teflon® may be required.

<u>Dredges</u>

For routine analyses, the <u>Peterson</u> dredge can 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 sizes if allowed to drop freely.

The <u>Eckman</u> dredge has only limited usefulness. It 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 <u>Ponar</u> dredge is a modification of the Peterson dredge and is similar in size and weight. It has been modified by 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 sampler 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 <u>"mini" or Petite Ponar</u> dredge is a smaller, much lighter version of the Ponar dredge. 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.

Coring

Core samplers are used to sample vertical columns of sediment. They are particularly useful when a historical picture of sediment deposition is desired since they preserve the sequential layering of the deposit, and when it is desirable to minimize the loss of material at the sediment-water interface. 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. They vary from hand push tubes to weight or gravity driven devices.

Coring devices are particularly useful in pollutant monitoring because turbulence created by descent through the water is minimal, thus the fines of the sediment-water interface are only minimally disturbed; the sample is withdrawn intact permitting the removal of only those layers of interest; core liners manufactured of glass or Teflon® can be purchased, thus reducing possible sample contamination; and the samples are easily delivered to the lab for analysis in the tube in which they were collected.

The disadvantage of coring devices is that a relatively small surface area and sample size is obtained often necessitating repetitive sampling in order to obtain the required amount of material for analysis. Because it is believed that this disadvantage is offset by the advantages, coring devices are recommended in sampling sediments for trace organic compounds or metals analyses.

In shallow, wadeable waters, the direct use of a core liner or tube manufactured of Teflon®, plastic, or glass is recommended for the collection of sediment samples. (Plastic tubes are principally used for collection of samples for physical parameters such as particle size analysis). Their use can also be extended to deep waters when SCUBA diving equipment is utilized. Teflon® or plastic are preferred to glass since they are unbreakable which reduces the possibility of sample loss. Stainless steel push tubes are also acceptable and provide a better cutting edge and higher strength than Teflon®. The use of glass or Teflon® tubes eliminates any possible metals contamination from core barrels, cutting heads, and retainers. The tube should be approximately 12 inches in length if only recently deposited sediments (8 inches or less) are to be sampled. Longer tubes should be used when the depth of the substrate exceeds 8 inches. 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. Because coarse or unconsolidated sediments such as sands and

gravel tend to fall out of the tube, a small diameter is required for them. A tube about two inches in diameter is usually the best size. The wall thickness of the tube should be about 1/3 inch for Teflon®, plastic, or glass. The inside wall may be filed down at the bottom of the tube to provide a cutting edge and facilitate entry of the liner into the substrate.

Caution should be exercised not to disturb the bottom sediments when the sample is obtained by wading in shallow water. The core tube is pushed into the substrate until four inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is being pushed will facilitate greater penetration and decrease core compaction. The top of the tube is then capped to provide suction and reduce the chance of losing the sample. A Teflon® plug or a sheet of Teflon® held in place by a rubber stopper or cork may be used. After capping, the tube is slowly extracted with the suction and adherence of the sediment keeping the sample in the tube. Before pulling the bottom part of the core above the water surface, it too should be capped.

When extensive core sampling is required, such as a cross-sectional examination of a streambed (with an objective of profiling both the physical and chemical contents of the sediment), a whole core must be collected. A strong coring tube such as one made from aluminum, steel or stainless steel is needed to penetrate the sediment and underlying clay or sands. A coring device can be used to collect an intact sediment core from streambeds that have soft bottoms which allows several inches of penetration. It is recommended that the corer have a check valve built into the driving head which allows water and air to escape from the cutting core, thus creating a partial vacuum which helps to hold the sediment core in the tube. The corer is attached to a standard auger extension and handle, allowing it to be corkscrewed into the sediment from a boat or while wading. The coring tube is easily detached and the intact sediment core is removed with an extraction device.

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 in the waste liquid. The loss of some of the fine sediments usually occurs with this technique.

Sample Handling and Mixing

It is extremely important that 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.

SECTION 7 BIOLOGICAL SAMPLING

Introduction

Five types of biological sampling are currently conducted by the Water Protection Branch: 1) aquatic macroinvertebrate sampling; 2) plankton sampling; 3) the light-bottle/dark-bottle test; 4) chlorophyll *a*; and, 5) fish tissue assessment project planning and coordination.

Macroinvertebrate Sampling and Identification

Methodologies and QA/QC for macroinvertebrates, including sampling, habitat assessment, and laboratory identification of samples may be found in the EPD Water Protection Branch document, *Standard Operating Procedures: Freshwater Macroinvertebrate Biological Assessment*.

Plankton and Periphyton

Plankton/Periphyton sampling and analysis for enumeration ("counts") are done in accord with that described in *Standard Methods for the Examination of Water and Wastewater*.

Light-Bottle/Dark-Bottle Test for Primary Productivity in Phytoplankton

The light-bottle/dark-bottle test for phytoplankton productivity is conducted according to the methods and QA/QC protocols in *Standard Methods for the Examination of Water and Wastewater*.

Chlorophyll Sampling and Analysis

The WPB Watershed Planning and Monitoring Program (WPMP) collects samples for the analysis of chlorophyll a (pheophytin a corrected), as a part of the parametric coverage in lake and coastal monitoring. Chlorophyll a is monitored monthly, from April through October each year at six lakes where site specific standards have been set. Samples are submitted to the EPD 14th Street Laboratory for analysis. Other major lakes are monitored according to the river basin schedule.

In routine and standards monitoring surveys, the photic (euphotic) zone is measured and a composite is collected for filtration of chlorophyll samples. In some cases, such as special model calibration studies, discrete depth samples are collected for chlorophyll analysis. Samples are filtered in the field, in duplicate (at a minimum), using 0.7 micron glass fiber filters and the filters are immediately frozen using dry ice. Field blank filtrations are conducted at the beginning and end of the sample filtrations at a minimum, and additionally for every 10 of the filtrations conducted during a monitoring event. At a minimum, 5% occurrence rate (calculated from multiple monitoring events), two additional replicated sample filtrations are provided to the EPD Laboratory for matrix

spike analysis. Field replicate analysis is conducted by the EPD Laboratory at a minimum 5% rate. Additional samples are collected and submitted to the EPD Laboratory when requested for additional quality control determinations such as analyst performance assessments. Field sampling and field filtrations are performed in accordance with the USEPA (Method 446.0) and *Standard Methods for the Examination of Water and Wastewater*.

The spectrophotometric method (USEPA Method 446.0 and Standard Methods 10200 H. 2.) is used for the analysis of chlorophyll *a*, and the EPD Laboratory has the capability to analyze fluorometrically (USEPA Method 445.0 and Standard Methods 10200 H. 3).

Fish Tissue Assessment

The Georgia Environmental Protection Division (EPD), the Wildlife Resources Division (WRD), and the Coastal Resources Division (CRD) work cooperatively to conduct fish tissue monitoring. The primary goals of this effort are to assess fish tissue for contamination, evaluate human health risks, and provide information for the public in an easily understandable format. This program began in 1988, with fish consumption advisories based on USFDA action or threshold limits as the triggering mechanism.

In 1991, the Georgia Department of Natural Resources (DNR) formed a committee to evaluate and develop guidelines for monitoring fish tissue contamination and issuing fish consumption recommendations. The Fish Tissue Advisory Committee (FTAC) was represented by DNR, USEPA, U.S. Fish and Wildlife Service and University of Georgia staff. The DNR used a communications consultant (Ringo Research Associates, Atlanta, Georgia) to assist in acquiring public input for the proposed model and developing a communication strategy. In the fall of 1993, six meetings were held around the state to provide the public an opportunity to learn about the proposed method and provide input. Among those invited to these stakeholder/ customer meetings were representatives of environmental groups, lake associations, lake side business owners, fishing guides and sportsmen. A seventh meeting was held for the Georgia Power Company and the U. S. Army Corps of Engineers. The program currently in use to develop consumption guidance is based on USEPA methods and was conservatively developed using available scientific information regarding likely intake rates of fish and toxicity values for contaminants detected. The Georgia Risk-Based Consumption Guidance was first published in early 1995.

The fish tissue assessment project is coordinated by the Watershed Planning and Monitoring Program of the Water Protection Branch of EPD. In the spring of each year the fish tissue advisory group begin planning the sampling plan for that year. The advisory group consists of Fisheries Regional Managers and Fisheries Department Supervisors of WRD, Coastal Resources Division (CRD) fisheries staff, representatives from the Watershed Planning and Monitoring Program of EPD and the EPD Toxicologist. Sites are chosen based on their importance and resources available each year for testing. Two criteria are evaluated for listing sites for testing: potential for contamination (point and nonpoint) or changed on level of contamination, and fishing pressure. Target species are selected to represent bottom feeders and top predators. Popularity of certain species (what is actually caught and consumed) is also considered. After sites and species have been ranked and selected, the Watershed Planning and Monitoring Program prepares a Field Monitoring Plan. It is provided to all Regional Fisheries Programs and sampling begins in early fall. The Fisheries Biologists are instructed to collect only fish that would be considered common creel for anglers in that area.

Samples are collected by WRD and CRD Fisheries Biologists and the Watershed Planning and Monitoring Program coordinates collection and transport of filet samples to both the University of Georgia Laboratory and the EPD State Laboratory for work-up and analysis. The samples are routinely analyzed for 43 contaminants, including metals, organic compounds, and pesticides. In some instances other contaminants have been analyzed (i.e., PAHs) where known sources existed.

Results are reported to the Watershed Planning and Monitoring Program where they are reviewed and provided to the EPD Toxicologist. The data are evaluated by the EPD Toxicologist, and integrated into the historical data set to produce a risk-based assessment. This information is incorporated into the *Guidelines for Eating Fish from Georgia Waters* published annually by the EPD Watershed Planning and Monitoring Program, and incorporated in an abbreviated format into the Georgia Freshwater and Saltwater Fishing Regulations by the WRD.

EPD's "risk-based" fish consumption guidelines are developed using USEPA's potency factors for carcinogenicity and reference doses for noncancer toxicity, whichever is most protective. Therefore, the recommendations are protective for both endpoints. EPD uses a risk level of 1×10^{-4} for cancer with the potency factor, a 30-year exposure for both carcinogens and toxic contaminants, 70 kg as body weight for an adult, and 70 years as a lifetime duration. A range of meal sizes (¼ to ½ pound) and frequencies are evaluated and one of four recommendations made: No restriction, Limit consumption to 1 meal per week, Limit consumption to 1 meal per month, or Do not eat. Each water body has its own advisory showing how often each species can be eaten. In lakes, species are also broken down further into three categories based on size of fish, < 12 inches, 12 to 16 inches, and > 16 inches. Advisories are only provided on legal size limits.

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SECTION 8 FIELD PHYSICAL MEASUREMENTS

Introduction

Field measurements of topographic features, water levels, time-of-travel, geophysical parameters, physical dimensions, etc., are frequently required during field investigations. The purpose of the investigation will determine the scope of 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 clearly established. The Water Protection Branch has a variety of maps including Georgia Department of Transportation county maps, and a complete collection of 7.5 minute USGS (1:24,000 scale) and 1:100,000 topographic maps. In addition, digital GIS 1:24,000 scale hydrologic unit (HUC) maps were developed through contract with the USGS as part of the Georgia River Basin Management Planning (RBMP) process. Commercial mapping software is utilized for planning, reference and for production of report maps.

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 records shall be kept in logbooks and field records so that the procedures are traceable.

Time records shall be kept in local time using the hour format, with the time recorded to the nearest five minutes or less. Georgia is contained entirely within the Eastern Time Zone. Time is recorded as Eastern Standard Time or Eastern Daylight Savings Time. Time adjustments must be noted and made in records when contractors such as the USGS submit data because all records by the USGS are in Eastern Standard Time.

New employees should perform each of the physical field measurements under the supervision and review of a senior technical staff member at least once before being permitted to make these measurements on their own.

Global Positioning System (GPS)

Introduction

GPS methods utilize radio frequency measurements with multi channel receivers of the signals from the global network of satellites that the Department of Defense has established. Measurements of the sampling locations or site features by GPS technology are 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.

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.

Existing information on 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:

- Georgia Department of Transportation
- State geodetic or land surveying offices
- Georgia Clearinghouse
- Georgia DNR-EPD Geologic Survey Branch
- NOAA/National Ocean Survey
- United States Geological Survey
- Corps of Engineers, Department of the Army
- Georgia Power Company
- Natural Resources Conservation Service
- Tennessee Valley Authority
- Bureau of Land Management

Location of all control data used and all field measurements should be recorded in the field logbook..

Specific Equipment Quality Control Procedures

Field investigators must be trained and checked out in surveying procedures by qualified staff before using this equipment. Each piece of field equipment (as appropriate) should be provided an inventory control number and a log book should be kept containing maintenance and calibration records for the equipment.

GPS Receivers

This equipment should:

- be serviced and calibrated by the manufacturer if damaged or suspected to be in error;
- be checked out using procedures outlined in the appropriate users manuals before use; and,
- be cleaned and maintained using procedures outlined in the appropriate users manuals during field use and before being returned to storage.

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 usually only one is used as the base station. The base station and the rover(s) must be within 200 to 300 miles of each other (accuracy increases as separation between base and rover decreases) and have an unobstructed view of the sky. 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, which will be compared to the actual control point coordinates for the development of a correction factor to be applied to other roving GPS units. Since the base station receiver and the rover receiver(s) synchronize with the satellites clocks, data must be recorded or logged by both at the exact same time in order for the correction factor to be applicable. Often times, base station data will be obtained via modem or disk after the field data collection by the rovers. It is therefore extremely important to coordinate the logistics and planning for using GPS techniques before leaving for the field.

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. All instruments will be used consistent with the instructions contained within these manuals. A copy of each of the manuals will be maintained by a designated person.

Vertical Location (Elevation) and Horizontal Location Surveys

Introduction

The field work conducted by the WPB does not require the ability to perform Horizontal Location Surveying with any measurable frequency. It is not discussed in this document. Vertical location surveying is employed for establishing new elevations from existing control or established reference points.

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; the WPB uses this method. Trigonometric leveling is slightly less precise and more difficult as it uses vertical angle and distance measurements combined with the principles of trigonometry; the WPB does not use this. 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. This subsection discusses the standard procedures and techniques used to obtain differences in elevation and are described in more detail in basic surveying and field geology textbooks.

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 (BMs.), 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 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 were listed in the previous discussion on Global Positioning Systems (GPS).

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 third-order accuracy 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 wing wall, foundation, or a nail or spike in a tree or telephone pole. 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.

Specific 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. Each piece of field equipment (as appropriate) should be numbered, and a log book should be kept containing all maintenance and calibrations made on the equipment. The manufacturer's specific maintenance and calibration procedures should be used for all equipment listed above.

Procedures for Differential Leveling

The level, or instrument, is set up by the instrument man at a location not more than 250 feet from the benchmark and at a height above the benchmark and the next point(s). The level is attached to the plate of the tripod by a fastening screw and the bubble in the bulls eye level is centered, or brought level by adjusting the three-screw leveling heads accordingly. Once the bulls eye bubble is centered, the level is rotated 90 degrees at a time and the horizontal level bubble is checked and brought level using the three-screw leveling heads. The level is ready for use when, after repeated rotations, the bubble in the horizontal level remains exactly in the center or middle of its housing.

The rod man holds the rod as plumb (vertical) as possible on the benchmark so that the instrument man can read where the horizontal cross-hair in the telescope of the level intersects the graduations on the rod. The rod man "rocks" the rod in two planes, when instructed by the instrument man, to obtain a level reading. The rod is white with large red numbers which indicate the foot-marks and smaller black numbers which indicate the tenths of feet and has black graduations the entire length which indicate hundredths of feet. The instrument man sights through the telescope and takes the first rod reading which is called a back sight (denoted BS or + in the field log book). The back sight (+) reading added to the elevation of the benchmark gives the height of the level, or instrument, (denoted H.I. in the field log book). Next the rod man holds the rod on a point (called a turning point and denoted TP) of fixed but unknown elevation such as a nail in the ground, spike in a tree or telephone pole, or the top of a fire hydrant. The instrument man then takes his second rod reading which is called a foresight (denoted FS or - in the field log book). If the foresight (-) reading is subtracted from the H.I., the result is the elevation of the point. That is, the difference between the first reading obtained from the benchmark and the second reading obtained from the point is the difference in elevation between the point and the benchmark. Note that the distance between each sighted reading should not ordinarily exceed 250 feet with turning point back sight and foresight distances deviating no more than 50 feet from one another.

The instrument man then goes ahead of the rod man, sets the level up as stated before and takes a rod reading (back sight) from the previous turning point. The rod man then moves ahead of the instrument man for a new turning point rod reading (foresight) and so forth until the desired final point is located vertically. Once the final point is located, the instrument man breaks the set up of the level (i.e., changes the H.I.) and re-levels the level. The instrument man and rod man then run levels from the last or final point to the first point or benchmark. This is called making a closed circuit or closed level loop.

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 by within 0.02 foot of the original reading or third order accuracy whichever is greater. If the level circuit does not close within these limits of accuracy, then the level circuit must be repeated until this accuracy is attained. Third order accuracy is defined by the formula: 0.05 foot H (/number of miles run), which means for a one-mile level circuit, the closure should be within five hundredths of a foot.

Bathymetry

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. During the initial setup of each survey, the fathometer calibration should be checked against a field measurement of water depth made using a graduated sounding line. All traces should be noted with transect description, chart speed, direction of travel, pertinent reference

points, and GPS and then indexed to a site map. When working in tidal areas, a water stage recorder should be positioned to provide a histogram of water levels to correlate with the bathymetric trace.

Specific Equipment Quality Control Procedures

All equipment used for bathymetric studies should be numbered and a record should be kept of all maintenance and calibration procedures. The following procedures should be used to maintain and calibrate bathymetric measurement equipment.

Recording Fathometers

These fathometers should be:

- Calibrated and maintained according to the manufacturer's instructions before use. The chart speed should be checked against a reference time source before instrument use.
- Checked in the field against a field measurement of water depth utilizing a calibrated sounding line or accurate depth recorder.
- Cleaned daily after use and prior to being stored.

Sounding Lines

All sounding lines will be calibrated against a steel surveyors tape with an accuracy of $\pm 3\%$.

Surface Water Stage/Tape Downs

Procedures

Water level recorders provide a time series record of water levels. These instruments should be referenced to National Geodetic Vertical Datum (NGVD). All water level tracings should be noted with beginning and ending date and time, site location, stage scale, and time scale and initialed by the field investigators. Standard USGS staff gages can 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 0.05 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.

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.

Specific Equipment Quality Control Procedures

A logbook will be kept of all equipment used for making water stage/tape down measurements. The following maintenance and calibration procedures should be used and recorded in the logbook for all equipment used for water stage and tape down measurements.

Stevens Stage Recorders

These stage recorders should be:

- Maintained according to the manufacturer's instructions. The time and vertical scale should be adjusted to read within two percent of full scale.
- Checked in the field by comparison with a staff gage. During field measurements, the vertical accuracy should check within 0.05 foot.
- Cleaned and maintained before storage.

USGS Staff Gage(s)

USGS staff gages should:

- Be checked for damage, warpage, legibility, etc., before use. Any damaged or illegible staff gages should be discarded.
- Be cleaned after use before being stored.

Weighted Steel Measuring Tapes

Weighted steel measuring tapes should:

- Be calibrated; the calibration should be within 0.01 foot per 10 feet of length.
- Be checked for damage before use; damaged tapes should be recalibrated or discarded.
- Be cleaned after use and prior to being stored.

Time-of-Travel

Introduction

Three principal methods are used to determine time-of-travel in streams. These are: surface floats or drogues; measurements of cross sectional velocity; and dye tracers.

A very rough method for preliminary estimates of time-of-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.

Procedures Using Floats and Drogues

Surface floats or drogues may be used in limited circumstances. Surface floats may be followed downstream and timed for known distances to determine time-of-travel. This requires the use of considerable judgment, for floats are susceptible to wind currents, tend to travel into quiet or eddy areas, or to become stuck on tree limbs, the stream bank, or other obstacles. The floats must frequently be retrieved and returned to the stream current. Drogues are weighted sea anchor floats and are sometimes used in estuarine studies. 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.

Procedures Using Cross Section Measurements

The measurement of cross sectional velocities at frequent longitudinal intervals and the calculation of average velocity in the stream is a time consuming method of obtaining time-of-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 1/10th mile may be desirable for streams with irregular channels.

Procedures Using Tracers

The most accurate method of measuring time-of-travel involves following and measuring a tracer. The WPB uses Rhodamine WT dye as a tracer. Rhodamine WT can be detected at concentrations as low as 0.01 ppb using a fluorometer.

Prior to injection into the stream, the concentrated dye is diluted with stream water. This insures immediate maximum dispersion. Addition of concentrated dye without dilution may result in incomplete dispersion, particularly in shallow streams. Calibration curves should be developed for each study with particular emphasis on accounting for natural background fluorescence.

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 or peak 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 published methods regarding time-of-travel techniques. These are *Measurement of Time-of-Travel and Dispersion by Dye Tracing* and *Fluorometric Procedures for Dye Tracing*.

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 or from once each minute to once every 30 to 60 minutes, depending on how wide the band of dye has become at the sampling point. The dye may be missed altogether by over estimating 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 read individually using the fluorometer. With the "flow-through" version, a strip chart recorder connected to the fluorometer can be used to plot the tracer concentration versus time. Some fluorometers have internal data loggers to provide this function. Readings directly from the fluorometer scale or conversion to dye concentration can be manually plotted against time when the grab sampling technique is used.

A version of the grab sampling technique would be to use an automatic water sampler which discharges into separate bottles. The sampler is pre-set to collect samples at certain intervals; at the end of the sample collection time, the discrete samples should be analyzed and the concentration determined for each. The concentrations are then plotted against time.

For proper determination of travel times, 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. The trailing edge of the dye cloud should generally 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.

Dilution Studies

Introduction

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 Dye-Dilution Methods*. The WPB rarely conducts dye dispersion/dilution studies. These data are typically estimated in table-top modeling using the Georgia Estuary Model.

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 photo reactive. Decay rates (e^{kt} where k=0.034/day for exposure to full sunlight) are reported in the literature. Due to limited light penetration, actual rates are much lower than this value and can be established through on-site bottle tests. 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 requires highly controlled metering rates and very accurate fluorometric analyses.

Procedures

The principal of superposition as developed by Kilpatrick et al. of the USGS is a reliable method to determine dilution levels of wastewater 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 tracer photo decay (and loss due to sorption on fine suspended solids), the dilution levels of the discharge can be produced for selective points in the estuary.

Calculation Procedure:

$$C_w = (\underline{C_t})(\underline{e^{kt}})(Vw)$$

Vt

Where:

| $C_w =$ | Ultimate concentration of wastewater at point of interest |
|-------------------|--|
| C _t = | Ultimate concentration of tracer (by superposition) at point of interest |
| t = | Tidal days to ultimate concentration |
| e ^{kt} = | Photo decay of tracer |
| $V_{\rm w}$ = | Wastewater discharge per tidal day |
| V_t = | Volume dye released in tidal day |

Example Calculation:

| Assume: | | After 5 tidal days the tracer clears to the area of interest and; | | |
|---------|-----------------|---|---|--|
| | C_t | = | 100 ppb | |
| Then: | k | = | 0.005 per tidal day (based on on-site bottle tests) | |
| | e ^{kt} | = | 1.03 (ultimate concentrations obtained in 5 tidal days) | |
| | $V_{\rm w}$ | = | 100,000 gal/tidal day | |
| | V_t | = | 10 gal/for one tidal day | |
| | $C_{\rm w}$ | = | 1,030,000 ppb | |
| | | | or | |
| | | = | 0.103% wastewater | |
| | | | | |

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.

Example Calculation:

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

MASS BALANCE EQUATION

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

Where:

 $Q_2 = pipe flow rate$

 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 to inject into the waste stream a tracer at a constant rate and constant concentration, and 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.

Specific Equipment Quality Control Procedures

See previous section. The metering pump should be calibrated before and during use. Field calibration data should be recorded in the field records.

References

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SECTION 9 FIELD MEASURABLE PHYSICAL/CHEMICAL CHARACTERISTICS

Introduction

Temperature, specific conductance (conductivity), hydrogen ion concentration (pH), turbidity, dissolved oxygen (DO), residual chlorine, salinity, and Sediment Oxygen Demand (SOD) tests will be the parameters discussed in this section. The sections will be discussed in the most applicable order. References can be found at the end.

Numerous meters/instruments are commercially available. Some meters are capable of numerous measurements which may include: pH, temperature, conductivity, DO, salinity, and turbidity. Individual meters discussed here are not necessarily the only ones available. However, the setup and use of all instruments should follow a basic format to imply a consistency of use. Regardless of the brand of meter used, all should be properly maintained and operated in accordance with the manufacturer's instructions and the calibration should be verified before and after use.

Field data may be measured and recorded instantaneously (in field books or in instrument electronic log files), by field personnel, or *in-situ* remotely using programmed data logging instrumentation. The WPB utilizes single and multiparametric instrumentation, with and without programmed data logging capability.

Temperature

Temperature is a measure of hotness or coldness on a defined scale. Three types of thermometers are available:

- Digital (thermocouple) thermistor
- Glass bulb, mercury or alcohol filled
- Bi-metal strip/dial indicator

Calibration:

Whichever type of thermometer is used, it should be calibrated against a National Instrumentation Standards and Technology (NIST) certified thermometer. Temperature may also be checked using a thawing ice water bath using de-ionized or purified water for both ice and liquid.

Note: Thermistors should be checked against a mercury bulb thermometer prior to use and should agree within ± 0.5 °C.

Inspection:

All thermometers should be inspected for leaks, cracks, and/or function prior to use. A broken glass bulb-mercury filled thermometer can contaminate samples by the release of

mercury. Information on the proper recycling of mercury is available from the Georgia DNR Pollution Prevention Assistance Division.

<u>Procedures:</u> (Make measurements *in-situ* when possible)

- 1. Clean the probe end with de-ionized water and immerse into sample.
- 2. Swirl the thermometer in the sample. Immerse according to the recommended immersion depth printed on the thermometer, or listed for the probe.
- 3. Allow the thermometer to equilibrate with the sample.
- 4. Suspend the thermometer away from the sides and bottom to observe the reading.
- 5. Record the reading in the logbook.

Note: Always clean the thermometer prior to storage and/or use. When measuring air temperature the thermometer or probe must be dry to prevent a bias due to evaporative cooling.

Units:

Conversion Formulas for Degrees Celsius (°C) or Degrees Fahrenheit (°F):

$$^{\circ}F = (9/5 \ ^{\circ}C) + 32 \text{ or} \qquad ^{\circ}C = 5/9 \ (^{\circ}F - 32)$$

Specific Conductance (Electrolytic Conductivity)

Conductivity is defined as the reciprocal of the resistance of a 1 meter cube of liquid at a specified temperature (25°C). Conductivity reflects the capacity of a liquid to conduct or transmit an electrical current, and provides an indirect measure of the ion content of a solution. A variety of meters are used in the WPB that use conductivity cells based on the Wheatstone bridge. Most of these meters will temperature correct readings to standard conductance at 25°C.

Standard(s) available:

Prepared primary standards using potassium chloride (KCl) are commercially available covering a range of conductance values. The WPB uses commercially prepared standards in the 1412 micromhos per centimeter (μ mhos/cm or μ Siemens/cm) and other ranges. Georgia waters are typically soft, low ionic strength with values less than 100 μ mhos/cm at 25°C.

Calibration:

The meter should be calibrated in accordance with the manufacturer's instructions. Conductivity is affected by temperature (conductivity increases in a nonlinear relationship with increases in ion concentration). Therefore, temperature should be read first so that appropriate adjustments can be made in accordance to the manufacturer's instructions if the meter does not provide temperature correction to 25°C.

1. Check and record the temperature of the standard solutions.

- 2. Rinse the probe with analyte-free water before immersing it in the standards solution.
- 3. Immerse the probe in the first standard solution and record the results. If the meter allows correction, adjust to the standard value and record.
- 4. If a second standard is used, rinse the probe and immerse it into the second standard solution and record results.
- Note: If the meter is not accurate to within $\pm 10\%$ of the standards, correct the problem before proceeding.

Procedures:

- 1. Collect the sample and check and record its temperature.
- 2. Correct the instruments temperature adjustment to the temperature of the sample (if instrument does not automatically temperature compensate).
- 3. Pre rinse conductivity cell with sample and discard the standard used in the rinse (moisture on cell or in container will dilute standard, introducing error. Always rinse with de-ionized water followed by a rinse with the next standard or sample before taking a reading. Discard standard or sample used for rinses).
- 4. Immerse the probe in the sample keeping it away from the sides and bottom of the container. It is important that the bridge portion of the probe electrodes be wetted by the sample.
- 5. Record the results in a logbook.
- 6. Rinse probe.

Units:

Conductivity units are measured in micromhos per centimeter (μ mhos/cm), or the International System of Units siemens (S) at 25°C. A siemens is a more recent unit designation and is becoming more prevalent as the reported unit utilized in instrumentation measuring conductance. A siemens is the reciprocal of the ohm (a mho is also the reciprocal of the ohm, which resistance is measured in); 1 μ S/cm is equivalent to 1 μ mho/cm.

Salinity

Salinity is the measure of salts of the alkali metals or of magnesium found in water. This measurement is based on the direct proportionality between the magnitude of an induced electric current and the electrical conductivity of the water in which it is induced.

Calibration/Maintenance:

- Follow the manufactures instructions.
- Follow calibration procedures as listed in previous section for conductivity.

Units:

Units are reported as salinity in the nearest tenth of a parts per thousand (0.1 ppt).

Hydrogen Ion Concentration (pH)

The pH is defined as the negative logarithm of the effective hydrogen ion concentration or hydrogen ion activity in gram equivalents per liter. It is used in expressing both acidity and alkalinity on a scale which ranges from 0 to 14 with 7 representing neutrality.

Temperature affects pH measurements in two ways. Mechanical effects are caused by changes in the properties of the electrodes and chemical effects are caused by equilibrium changes. Standard pH buffers have certain values at specific temperatures. Automatic temperature compensation (ATC) or manually setting the sample temperature will correct for the temperature effects on the electrode(s). Always measure and record the solution temperature of the sample.

Calibration:

Follow manufacturer's instructions with the following as a minimum. Two-point standardization is preferred when possible. Some meters allow for three-point standardization. Standard buffers selected should be near the expected values anticipated in the field monitoring, and when a two-point calibration is done, the chosen standards should bracket this expected range. Standardize the meter with buffers that are near or at the temperature that will be present when the meter is being used for monitoring.

- 1. Remove the meter from storage and allow it to equilibrate to ambient temperature. If the probe is dry or new, follow manufacturer's directions for preconditioning probe(s) prior to use.
- 2. Use a thermometer and determine the temperature of the buffering solutions and record. Meters provided with ATC probes allow standardization with temperature compensation.
- 3. Select either pH 4 and pH 7 or pH 7 and pH 10 solutions as described above.
- 4. Rinse the probe with analyte-free water and immerse it into the first buffer (pH 7) and record.
- 5. Rinse the probe and immerse it into the second buffer and record.
- 6. Rinse and store the electrode(s) in a container(s) filled with analyte-free water, or as manufacturer recommends (some probes are best temporarily stored in pH 4 buffer such as pH probes; reference or combination electrodes in 4 molar KCl, or dry for long term storage).

Procedures:

Meters with automatic read and lock features for picking the stable pH can sometimes show an error when field readings are being taken in flowing stream. This is because the pH is changing as water flows across the sensor and the meter programming interprets this as an unstable probe error. Turn the automatic read and lock feature off and record what appears to be the representative pH or take a grab sample of the stream water and read the pH of that.

1. Collect a sample or immerse the probe in the water. Measure and record the

temperature prior to measuring the pH.

- 2. Immerse the probe in the sample keeping it away form the sides and bottom of the sample container. Allow ample time for the probe to equilibrate with the sample.
- 3. While suspending the probe away from the sides and bottom of the sample container, record the pH.
- 4. Rinse the probe with analyte-free water and store it in an analyte-free water filled container until the next sample is ready.

Operational check:

- 1. While in use, periodically check the pH by rinsing the probe with analyte-free water and immersing it into the pH 7 buffer solution.
- 2. Perform a post calibration at the end of the day and record all findings.

Units:

Units of pH are Standard Units (SU) and should be read in one-hundredths (0.01) and recorded in tenths (0.1).

Turbidity

The majority of turbidity measurements are performed by the EPD Water Quality Laboratory (WQL), on samples collected and submitted by WPB personnel. A nephelometer/turbidimeter is used in comparing the turbidity of liquids by viewing light through them and determining how much light is scattered from the light path. Some WPB multiparametric sonde units are furnished with a turbidity probe. Calibration checks against the WQL turbidimeter are recommended in conjunction with the use of latex sphere standards.

Operational check:

- 1. Periodically check the turbidity meter by using the standards provided.
- 2. Perform a post calibration at the end of the day and record all findings.

Units:

Turbidity measurements are reported in nephelometric turbidity units (NTUs).

Dissolved Oxygen (DO)

Introduction

The solubility of oxygen in water is affected by pressure (atmospheric and hydrostatic), temperature and ionic strength or salinity. When all other factors remain the same:

- DO solubility decreases with increases in temperature.
- DO solubility decreases with increases in ionic strength/salinity.

• DO solubility increases with increases in pressure, both atmospheric (barometric pressure), and hydrostatic pressure (hydrostatic pressure increases with depth because of the weight of overlying water). Atmospheric pressure decreases with increases in elevation, and is usually referred to as above the mean sea level datum. Total pressure at depth is the sum of atmospheric and hydrostatic pressure.

Accurate measurement of dissolved oxygen with instrumentation requires that calibrations and readings allow for or automatically compensate for the effects of temperature, pressure and ionic strength, so that correction of the reading for these variables is made.

Method Selection

The WPB has a variety of instrumentation for measuring DO by the electrometric method. Measurement of DO by the Winkler titrametric method is occasionally done on samples collected from shallow depths. Dissolved oxygen is measured using meters dedicated to that parameter and multiparametric units that include DO probes in the sonde units. Temperature and pressure compensation is provided by all of the instrumentation used for DO measurement. Some of the WPB instrumentation also provides salinity compensation, and depth that the reading is taken from as provided by a depth sensor in the sonde unit.

Choice of instrumentation or method used for DO measurement is indicated by station characteristics, depth that readings are required at, water quality characteristics (freshwater, estuarine/marine, interferences), or time period over which reading are required (single or multiple over a set period). Discrete depth water samplers may be used to collect samples in shallow to moderate depths for subsequent determination of DO by ME or iodometric methods if oxygen introduction (as prone with low DO or anoxic samples), or oxygen loss (as with saturated or supersaturated samples such as from an active algal photosynthetic zone or deep gas saturated zone) is not a factor of concern.

Meter Inspection

- Prior to field use, the membrane of the DO meter should be inspected for air bubbles and/or holes. If air bubbles or holes exist, replace the membrane. Flush chamber well with electrolyte solution provided. Some membranes should be allowed an overnight stabilization period prior to calibration. Certain probes take cap membranes of different designs and require the use of special electrolyte filling solutions. Consult manufacturer's recommendations.
- The membrane should be checked for dryness. If the membrane is dry, replace and soak it in analyte-free or analyte-free water prior to calibration of the meter.
- Multiparametric units deployed in-situ for periods of a week or more are prone to biological fouling by diatoms, algae, other microorganisms or invertebrate organisms that may affect the sensitivity of the reading (diffusion rate across membrane and respiratory effects), or perforate the membrane. End calibrations should be done on sonde units with any material present upon retrieval from deployment still on the DO membrane so that any drift in sensitivity may be documented. Upon completing end calibration, the probe is cleaned, and an electrolyte flush and new membrane installation should be considered

before subsequent field use.

• ME DO probes typically use a gold cathode and a silver anode. Gray to black deposits on the silver anode indicate that hydrogen sulfide has diffused across the membrane and formed silver sulfate deposits (common in anoxic environments), and sensitivity of the DO probe may be affected. A dull or whitish color on the surface of the gold cathode may indicate plating of chloride or other anions and may also affect probe sensitivity. The manufacturer's recommendations should be consulted and utilized in maintenance and cleaning procedures to be employed when these conditions occur.

Meter Calibration:

- Air calibrate according to the manufacturer's instructions in a stable water saturated air environment. Use of the readings from an accurate, calibration checked barometer at the calibration site will increase the accuracy of the air calibration.
- The ME meter can be checked and/or calibrated against the Winkler method if desired.

Meter Procedures:

- When making measurements, be sure that the DO stirring apparatus, when provided is working. Some probes utilize low flow membranes or other design and do not require stirring for accurate readings.
- Adjust the temperature and salinity compensators (if equipped, and if compensation is not automatic).
- Read the display to the nearest 0.1 mg/l and record the measurement. Multiparametric sonde interfaces typically have record storage programming for electronic data logging.

When Use of a Sampler is Required:

- When possible, measure the DO *in-situ* with a field probe attached to a sufficient length of cable for the height and depth present.
- Generally, in depths of less than 1 meter collect the sample(s) at mid-depth in a shallow water sampler and fill a 300 mL Winkler BOD bottle allowing 3 volume changes and introduction of no air bubbles, and measure the DO by ME or the Winkler method.
- For moderate depths of 2 to 4 meters, if insufficient cable length is available for direct measurement, a discrete depth water sampler may be used to collect the sample(s) for analysis, by ME or iodometrically, but the possibility of DO loss or addition to the sample increases.
- When using discrete depth water samplers for DO analysis by the Winkler method, collect samples in duplicate or triplicate at the same depth at the same time (or as close a possible). Use samplers that collect sample directly in the 300 mL Winkler DO bottle placed inside the sampler and such that the sample flows through and overflows out of the bottle as the sampler fills. Allow at least 3 exchanges of sample to flush bottle.
- Special care should be exercised to avoid entrainment of atmospheric oxygen or loss of DO. The WPB has shallow water samplers constructed out of bottles and tubing (used to fill BOD bottles), and Kemmerer type and Polyethylene plug-pull (LabLine, 1400 and

2000mL) samplers that allow collection in 300 mL Winkler BOD.

• Units should be reported in mg/L.

Limitations:

- Dissolved inorganic salts are a factor with the performance of DO probes.
- Reactive gases which pass through the ME probes may interfere with the DO analysis. For example, chlorine will depolarize the cathode and cause a high probe output. Longterm exposures to chlorine will coat the anode with the chloride of the anode metal and eventually desensitize the probe. Hydrogen sulfide will interfere with the ME probes if the applied potential is greater than the half-wave potential of the sulfide ion.
- Dissolved oxygen ME probes are temperature sensitive, and temperature compensation is normally provided by the manufacturer (see manufacturer's instructions).

DO Analysis by the Iodometric Method (Azide Modification of the Winkler Method)

The azide modification of the Winkler method is performed as described in the U. S. EPA Chemical Methods Manual (EPA Method 360.2) and Standard Methods (Method 4500-O A/BC). Samples analyzed by this method should be collected in duplicate at a minimum.

Stock reagents are ordered by and stored in the WPB laboratory/field support area as follows:

- Manganous Sulfate, prepared reagent #1; stored in routine chemical storage cabinet.
- Alkaline Iodide Azide, prepared reagent #2; stored in alkali corrosive chemical cabinet.
- Concentrated Sulfuric Acid, prepared reagent #3; stored in acid corrosive chem. cabinet.
- Primary standard 0.0375N Sodium Thiosulfate titrant, prepared reagent with preservative and expiration date; stored in routine chemical storage cabinet.
- Starch Indicator solution, either purchased as prepared indicator solution or prepared from potato starch heated to saturation, cooled, decanted and refrigerated. Opened starch solutions are kept in the laboratory refrigerator.

All reagent bottles are dated with receipt or preparation date, and the open date is recorded on the container bottle label. Field Winkler kits are to be refreshed with new titrant and indicator prior to use at a minimum if they have not been used recently. All reagent bottles need to have chemical safety labels identifying reagents and safety codes. Material safety data sheets (MSDS) are maintained in a notebook in the calibration room for the above chemicals (and others used).

Total and Free Residual Chlorine

The HACH reagents and colorimeter or spectrophotometer are accepted by the U.S. EPA for NPDES monitoring if used in accordance with approved procedures. The preprinted calibration scales provided by the manufacturer are based on factors developed under ideal conditions and are only acceptable if verified. The calibration scale must be initially verified using multiple standards and a blank. A calibration curve using a blank and multiple standards should be conducted with a meter prior to taking it to the field for use. The calibration scale or curve should be verified in the

field prior to use in the field using a blank and a standard representative concentration range expected in samples. These standard checks must agree within $\pm 10\%$ of the original scale or a new curve must be prepared. Verification data should be recorded and maintained on file.

Inspection:

- Each meter should be visually inspected before and after each use.
- Check the battery strength.
- Insure that the reagents are fresh before field trips.

Calibration:

- The calibration scales should be checked for calibration prior to field use with a a blank and 5 known standards that bracket the expected sample concentrations (recommended 0.02, 0.05, 0.1, 1.0 and 2.0 mg/L, prepared using Potassium Permanganate).
- Calibration in the field should be verified with a blank and standard in the range expected in the sample (standard is prepared in the field from a lab prepared stock solution or a commercially prepared standard).
- When DPD is used, adhere to the requirements set forth in Standard Methods.

Reagents/Standards:

- DPD total chlorine powder packets. The packets deteriorate in the presence of moisture. The packets should be discarded if they have caked or have turned brown. The DPD oxalate is very toxic. <u>DO NOT</u> handle with unprotected hands or ingest. If accidentally spilled on skin or ingested, seek medical attention immediately.
- Chlorine demand-free water (See Standard Methods, Method 4500 Cl for directions in preparing the ASTM Standard D1193 "Consumption of Potassium Permanganate").
 Note: KMnO₄ standards will fade rapidly (within 15 minutes) if chlorine demand-free water is not used.
- Potassium permanganate stock -- Prepare a stock solution containing 891 mg/1000 mL.
- Potassium permanganate working stock 10 ppm -- Prepare working stock solution containing 10 mg/L KMnO₄ by diluting 10 mLs of stock solution to 1 liter.
- The stock should be stable for approximately 5 days if kept cool and away from light.
- Potassium permanganate calibration standards -- Prepare calibration standards from the working stock solution and/or KMnO₄ calibration standard solutions for each day of use.

Interferences:

Positive errors using the DPD reagent may occur if the sample contains strong oxidants such as nitrite, iodide, and divalent manganese. Common errors in Georgia surface waters will result from reaction with manganese in the range of 0.02 to 0.1 mg/L, due to the prevalence of the manganese ion in Georgia waters. Following runoff producing rains, false positive readings of total residual chlorine in surface waters as high as 0.8 mg/L may result from the reaction of DPD with manganese or other non residual chlorine constituents.

Sediment Oxygen Demand (SOD) Studies

Introduction

Sediment oxygen demand is a term used to describe the combined effects of all oxygen reducing factors associated with benthic material. Some of these factors are macroinvertebrates, aerobic bacteria, chemical reactions and floral respiration. Sediment oxygen demand is used to describe a component of the oxygen budget and is necessary for mathematical modeling of aquatic systems. Oxygen demand by benthic sediments and organisms can represent an appreciable component of oxygen consumption in surface waters. Sediment oxygen demand can be measured in the laboratory or *in-situ*. Models can also be used to estimate SOD.

In-Situ Procedure

The WPB has the capability to perform SOD studies *in-situ*. Determination of SOD rates is accomplished through placement of special chambers on bottom substrates and recording the dissolved oxygen content of the contained overlying volume of water at intervals for a period of time. Each chamber is equipped with a flange and cutting edge on the bottom to seat it to the substrate and a pump for circulation of water with in the chamber. A multiparametric sonde unit either programed to collect readings over the interval and time period required, or connected through cabling to a display unit for real time readings, is inserted through a gasketed opening such that the sonde probes are in contact with the inner chamber waters.

Three replicate and one control chamber are typically used at each test site. The control chamber resembles the other chambers but has a bottom to isolate the water from the sediment surface (to factor oxygen uptake background demand of water from that due to benthic demand). The number of stations to be sampled is determined on the basis of data needs and by the bottom types existing in the study area. The chambers are deployed by certified divers using SCUBA (self contained underwater breathing apparatus). Because of the potential adverse exposures that may occur to divers, use of WPB dry suits and full face masks with diver communication equipment is recommended. Quality control measures include:

- Initial and final calibration of multiparametric units.
- Flushing of chamber contents upon initial seating to substrate.
- Chamber seal checks .
- Samples pulled from inside water for laboratory analysis.
- Samples of sediments at study site and surrounding area (for comparison to assessed sites), for laboratory analysis.
- Velocity measurements within chamber to verify acceptable water circulation.

Chamber Design

The SOD chambers were specially manufactured using aluminum plate following a custom design from the U.S. EPA. The WPB chambers are 29 inches (71.1 cm) in diameter by 11.25 inches (28.6 cm) deep. Two flanges are located on the outer rim of the chambers in which the upper and lower flanges function as the cap plate and cutting edge. The lower flange also increases the surface

area of the chamber. This reduces the possibility of the chamber sinking when deployed on soft bottoms. In very unstable sediments having a pudding-like consistency, the chambers can be "floated" on a sheet of cut out plywood that distributes the weight of the chamber over a larger surface area. Centered on the cap plate is a core which houses a direct current (DC) water pump. The battery is attached to the top of the cap plate. An opening for the insertion of the multiparametric sonde unit is cut in the top of the cap plate and a sealing plug is employed when the sonde is installed. Volumes are calculated to the nearest liter. The chamber volume and the surface area of the bottom sediment covered are 70 liters and 0.274 square meters, respectively.

SOD Data Analysis

The dissolved oxygen data (salinity corrected in estuarine/marine studies), is graphed, and a linear regression analysis is performed to obtain the slope (rate of oxygen depletion). The SOD rate is calculated using the following formula:

SOD Rate = (S) x [(V)(c)]) A

where:

S = Slope derived from linear regression (mg/liter/minute)
V = Volume of chamber (liters)
A = Surface Area of Sediment Covered (square meters)
c = Constant (1.44) used to convert mg oxygen/min. To grams oxygen/day

Correction of the SOD Rate to a temperature of 20°C can be made using the following formula:

$$\mathbf{R}_{20} = \mathbf{R}_{\rm CT}^{(1.065)_{20-\rm CT}}$$

where:

 R_{CT} = Rate at Observed Temperature (°C)

CT = Observed Temperature

1.065 = Temperature Coefficient used in USGS-Steady water quality model for determining SOD rate coefficients. Note that the temperature coefficient is raised to the (20-CT) power in the above formula.

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