

NPDES Compliance Monitoring Inspector Training: Sampling



NPDES COMPLIANCE MONITORING INSPECTOR TRAINING MODULE

SAMPLING

U.S. ENVIRONMENTAL PROTECTION AGENCY

ENFORCEMENT DIVISION OFFICE OF WATER ENFORCEMENT AND PERMITS ENFORCEMENT SUPPORT BRANCH

1990

DISCLAIMER

This module has been reviewed by the Office of Water Enforcement and Permits, U.S. Environmental Protection Agency, and approved for publication. This module represents EPA's introductory training on selected topics related to conducting NPDES compliance inspections. Failure on the part of any duly authorized official, inspector, or agent to comply with its contents shall not be a defense in any enforcement action, nor shall failure to comply with this guidance alone constitute grounds for rendering evidence obtained thereby inadmissible in a court of law. The mention of trade names or commercial products constitutes neither endorsement nor recommendation for use.

ACKNOWLEDGMENTS

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TABLE OF CONTENTS

| | | Page |
|----|--|-------------|
| FC | DREWORD | vii |
| 1. | INTRODUCTION | 1-1 |
| | 1.1 OVERVIEW OF THE NPDES PROGRAM | 1-1 |
| | PROGRAM | 1-2 |
| | 1.3 OBJECTIVES OF NPDES SAMPLING | 1-3 1-3 |
| | The state of the s | |
| 2. | SAMPLE COLLECTION | 2-1 |
| | 2.1 IMPORTANCE OF SAMPLE COLLECTION | 2-1 |
| | 2.2 SAMPLING PLAN | 2-2 |
| | 2.3 PREPARATION FOR SAMPLING | 2-4 |
| | 2.4 SAMPLING SAFETY | 2-4 |
| | 2.5 SAMPLING LOCATION | 2-5 |
| | 2.6 SELECTION AND PREPARATION OF SAMPLE CONTAINERS | 2-6 |
| | 2.7 SAMPLE TYPES | 2-8 |
| | 2.8 SAMPLE COLLECTION TECHNIQUES | 2-10 |
| | 2.9 SAMPLE VOLUME | 2-14 |
| | 2.10 SAMPLE PRESERVATION AND HOLDING TIMES | 2-14 |
| | 2.11 SAMPLE DOCUMENTATION | 2-16 |
| | 2.12 SAMPLE IDENTIFICATION AND LABELING | 2-17 |
| | 2.13 SAMPLE PACKAGING AND SHIPPING | 2-18 |
| | 2.14 CHAIN-OF-CUSTODY PROCEDURES | 2-18 |
| | 2.15 SPECIAL SAMPLING REQUIREMENTS | 2-20 |
| 3. | ANALYTICAL METHODS FOR ONSITE ANALYSIS | 3-1 |
| 4. | AUTOMATIC SAMPLERS | 4-1 |
| 5. | FLOW MEASUREMENT | 5- 1 |
| | 5.1 IMPORTANCE OF FLOW MEASUREMENT | 5-1 |
| | 5.2 OPEN CHANNEL FLOW | 5- |
| | 5.3 CLOSED CHANNEL FLOW | 5- |

TABLE OF CONTENTS (Continued)

| | | <u>Page</u> |
|----|--|-------------|
| 6. | QUALITY CONTROL PROCEDURES FOR SAMPLING | 6-1 |
| | 6.1 QUALITY CONTROL PROCEDURES FOR SAMPLING | 6-1 |
| | 6.2 QUALITY ASSURANCE PROCEDURES FOR SAMPLING | 6-2 |
| | 6.3 LABORATORY QUALITY ASSURANCE/QUALITY CONTROL | 6-3 |
| 7. | SUMMARY | 7-1 |

LIST OF TABLES AND FIGURES

| <u>Tabl</u> | <u>le</u> | Page |
|-------------|---|------|
| 2-1 | COMPOSITING METHODS | 2-11 |
| <u>Figu</u> | <u>ire</u> | |
| 5-1 | PROFILE AND NOMENCLATURE OF SHARP-CRESTED WEIRS | 5-3 |
| 5-2 | FOUR COMMON TYPES OF SHARP-CRESTED WEIRS | 5-4 |
| 5-3 | DIMENSIONS AND CAPACITIES OF THE PARSHALL MEASURING FLUMES FOR VARIOUS THROAT WIDTHS | 5-5 |
| 5-4 | CONFIGURATION AND NOMENCLATURE OF VENTURI METER | 5-8 |
| 5-5 | ELECTROMAGNETIC FLOW METER | 5-10 |
| AP | PENDIX A - GLOSSARY | |
| AP | PENDIX A - GLOSSARY | |
| AP | PENDIX B - REFERENCES | |
| AP | PENDIX C - REVIEW QUESTIONS AND ANSWERS ON NPDES SAMPLING PROCEDURES | |
| AP. | PENDIX D - VOLUME OF SAMPLE REQUIRED FOR DETERMINATION OF THE VARIOUS CONSTITUENTS OF INDUSTRIAL WASTEWATER | |
| AP. | PENDIX E - REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, HOLDING TIMES, AND TEST METHODS (EXCERPTED FROM 40 CFR PART 136) | |
| ΑP | PENDIX F - EPA ORDER 1440.2 - HEALTH AND SAFETY REQUIREMENTS FOR EMPLOYEES ENGAGED IN FIELD ACTIVITIES | |
| ΑP | PENDIX G - LIST OF FIELD SAMPLING EQUIPMENT | |
| ΑP | PENDIX H - SAMPLE IDENTIFICATION LABELS | |

LIST OF APPENDICES (Continued)

- APPENDIX I EXAMPLE RECORD OF FIELD SAMPLE DATA AND CHAIN-OF-CUSTODY RECORD
- APPENDIX J CRITERIA FOR SELECTION OF AUTOMATIC SAMPLING EQUIPMENT
- APPENDIX K QUALITY CONTROL PROCEDURES FOR FIELD ANALYSIS AND EQUIPMENT

FOREWORD

This document is one of five training modules developed by the Office of Water Enforcement and Permits (OWEP), U.S. Environmental Protection Agency (EPA) to introduce the National Pollutant Discharge Elimination System (NPDES) program to new inspectors. Information in each module provides training to an inspector unfamiliar with the NPDES program. The modules address the following topics:

- The Overview Module presents an overview of the entire NPDES program and briefly summarizes different types of inspections conducted under this program
- The Legal Issues Module discusses the legal issues which must be addressed during an inspection and provides legal information to assist inspectors in performing their duties
- The Biomonitoring Module outlines the principles of biomonitoring and the role of biological testing in the NPDES program
- The Sampling Procedures Module details procedures to be used when conducting sampling and flow monitoring
- The Laboratory Analysis Module outlines procedures and information necessary to perform an effective evaluation of a permittee's laboratory.

The modules are best used in a classroom setting where there is discussion between instructors and students and where questions can be asked. Yet, they can also stand alone as reference sources. Additional discussion of the topics covered in these modules appears in the 1988 NPDES Compliance Inspection Manual.

These training modules were developed primarily for in-house training of Regional and State NPDES inspectors. However, they are available as well to other interested parties such as attorneys, other program offices, facility owners and operators, and members of the general public.

Regional and State personnel are encouraged to provide EPA Headquarters with suggested changes or information which they believe would improve these modules. The content of the modules will be updated and revised periodically. Comments, information, and suggestions to improve the modules should be addressed to the:

Enforcement Support Branch (EN-338)
Office of Water Enforcement and Permits
U.S. Environmental Protection Agency
401 M Street, S.W.
Washington, DC 20460

1. INTRODUCTION

1.1 OVERVIEW OF THE NPDES PROGRAM

The Federal Water Pollution Control Act of 1972, as amended by the Clean Water Act (CWA) of 1977 and by the Water Quality Act of 1987, specifies the objectives of restoring and maintaining the chemical, physical, and biological integrity of the Nation's waters. The CWA provides broad authority to the U.S. Environmental Protection Agency (EPA) to:

- Establish the National Pollutant Discharge Elimination System (NPDES) program and the National Pretreatment Program
- Define acceptable pollution control technologies and establish effluent limitations based thereon
- Obtain information through reports and compliance inspections
- Take enforcement actions, both civil and criminal, when violations of the CWA occur.

The NPDES program, mandated by Section 402 of the CWA, regulates the discharge of pollutants from point sources, such as municipal treatment plants, industries, animal feedlots, aquatic animal production facilities, and mining operations. In order to discharge, each point source is required to obtain a NPDES permit which specifies effluent limits, monitoring and reporting requirements, and any other terms and conditions necessary to protect water quality.

To determine whether these NPDES permit conditions are being met, Section 308 of the CWA authorizes inspections and monitoring of permittee facilities. Under the authority of Section 308, two types of monitoring have been established: self-monitoring by the permittee and compliance monitoring by the permit-issuing agency. According to the CWA, an inspection may be conducted where there is an existing NPDES permit, or where a discharge exists or is likely to exist and no permit has been issued.

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Compliance with NPDES permit conditions is often monitored by States. Sections 308 and 402 of the CWA provide for the transfer of Federal program authority to conduct NDPES permit compliance monitoring to State agencies. Currently, over 75 percent of the States and territories are approved by EPA to implement State NPDES programs.

1.2 PURPOSE OF THE NPDES COMPLIANCE MONITORING PROGRAM

As mentioned above, each NPDES permit contains specific, legally enforceable effluent limitations and monitoring and reporting requirements. The purposes of the NPDES compliance monitoring program (and the various inspections conducted under the program) are to collect information that supports enforcement of the Water Quality Act by:

- Evaluating the compliance or dischargers with permit limitations
- Assessing compliance with orders or consent decrees
- Furnishing information which supports permitting.

This compliance evaluation involves two aspects: (1) collection of effluent samples by a NPDES inspector during a Compliance Sampling Inspection (CSI), a Toxic Sampling Inspection (XSI), or a Compliance Biomonitoring Inspection (CBI); and (2) evaluation of a permittee's self-monitoring procedures during a Performance Audit Inspection (PAI) or a Compliance Evaluation Inspection (CEI). Under certain circumstances, the inspection may also evaluates the industrial monitoring and enforcement efforts conducted as part of a municipality's pretreatment program. This type of inspection is called a Pretreatment Compliance Inspection (PCI).

To familiarize new NPDES inspectors with proper sampling procedures and to establish consistent procedures throughout the NPDES compliance inspection program, this module outlines procedures to collect, preserve, and transport wastewater samples. This module also discusses sampling equipment, methods for onsite analytical procedures, flow measurement, and quality assurance and quality control procedures.

Appendix A of this module contains a glossary of terms with which inspectors should be familiar. In addition

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to the discussion in this module, inspectors may also wish to consult the references listed in Appendix B. Finally, after reviewing the module, each inspector should complete the questions in Appendix C to test his/her understanding of its contents. Answers to these questions are also provided in Appendix C.

1.3 OBJECTIVES OF NPDES SAMPLING

Data obtained from sampling play a vital role in the NPDES program. Sampling is conducted to accomplish one or more of the following objectives:

- Determine discharge quality at the time of the inspection
- Determine compliance with effluent limitations and permit conditions
- Collect information for use in permit development
- Assess the quality of self-monitoring data
- Provide a basis for enforcement proceedings in the event such proceedings become necessary.

Whether sampling is used as a part of an enforcement proceeding or to verify or compile data, sampling activities should always be performed with great care.

1.4 SAMPLING TASKS

To achieve these objectives, one or more of the following sampling tasks must be performed:

- Sample at the location and for the parameters specified in the NPDES permit. Additionally, sample at locations and for parameters requested by enforcement personnel but not specified in the permit
- Verify accuracy of permittee flow measuring device, either by verifying accuracy of in-plant equipment or by actual independent flow measurement
- Verify that the sampling location(s) specified in the permit include all of the process and nonprocess discharges and is adequate to collect a representative sample of the effluent

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| • | Verify that the permittee collects self-monitoring samples at the location specified in the permit |
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| • | Verify that the permittee's sampling and preservation techniques are adequate to ensure the collection of a representative sample. |
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2. SAMPLE COLLECTION

2.1 IMPORTANCE OF SAMPLE COLLECTION

Actual sample collection is an extremely important part of any sampling program. Without proper sample collection techniques, even the most precise and accurate analytical procedures will produce results which do not reflect the actual pollutant levels in the facility where sampling is performed prior to the inspection. Such information includes:

- EPA guidance materials (manuals such as the NPDES Compliance Inspection Manual, Pretreatment Compliance Monitoring and Enforcement Guidance, Samplers and Sampling Procedures For Hazardous Waste Streams, and the Handbook for Sampling and Sample Preservation of Water and Wastewater) may be helpful in developing a sampling plan.
- Thorough knowledge of the Department of Transportation (DOT) shipping regulations applied to the constituents and preservatives contained in the samples in case any materials must be transported in a specific manner.
- 40 Code of Federal Regulations (CFR) Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants."
- NPDES permit and other pertinent information contained in the compliance files.
- Descriptions and photographs of the waste treatment process used, obtained through such materials as the EPA development documents and prior inspection reports.
- Familiarity with production processes and sources of wastewaters or, in a municipal plant, a knowledge of the raw waste and treatment systems. (For industrial processes, the EPA development documents are a good source of information.)
- Knowledge of travel or shipping schedules in the area of the facility to be sampled in case samples must be shipped to an offsite laboratory.

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2.2 SAMPLING PLAN

Before sampling a discharger for the first time, the inspector should clearly define the data needs and the data quality objectives. The inspector should, if possible, refer to available file information; consult with the appropriate compliance, legal, permitting, and laboratory personnel; and walk through the facility to become familiar with its operation and layout.

Once the inspector understands the needs and objectives of the visit, a complete and comprehensive quality assurance and sampling plan can be developed. This plan should contain the following items:

- Sampling Locations Sampling locations should include all outfalls that appear in the NPDES permit. Due to accessibility, needs, and objectives of the survey, and/or safety hazards, the sampling location specified in the permit may not be adequate. Therefore, locations other than those specified in the NPDES permit may need to be sampled. The number of samples to be taken at each location should be indicated as well.
- Type of Sample Type of sample depends on the parameters to be measured and/or the discharge characteristics (i.e., batch discharge). This information is specified in 40 CFR Part 136 and the NPDES permit.
- Type of Flow Measurement Type of flow measurement is dependent on the flow rate, condition of the wastewater, and variability of the discharge. Flow measurements are necessary to determine the mass loading of a discharge. Flow should be measured or the permittee flow measurement device should be verified.
- Parameters for Analysis The NPDES permit specifies pollutant parameters monitoring by the permit holder; these parameters are given as mass- or concentration-based discharge limitations. These same parameters will be selected for compliance sampling but other parameters may be chosen as well, if new processes or products have been incorporated in the plant or new or added sources of wastewater are in evidence. If new processes or products have been incorporated in the plant, additional sampling will help provide the basis for necessary permit modifications.

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- Sample Volume The volume of sample collected depends on the type and number of analyses necessary, based on parameters to be measured. The volume of the sample obtained should be sufficient to perform all the required analyses [including laboratory Quality Assurance/Quality Control (QA/QC) and repeat analyses] plus additional amounts to provide for any split samples that may be required. A summary of required sample volumes for determination of various constituents is provided in Appendix D.
- Type of Sample Containers Selection and preparation of sample containers are based on the parameters to be measured and wastewater characteristics. Required containers are specified in 40 CFR Part 136, which is summarized in Appendix E.
- <u>Sample Preservation Techniques</u> To preserve samples correctly, the appropriate chemicals must be used and temperature control must be ensured. Preservation techniques and recommended holding times are specified in 40 CFR Part 136 (see Appendix E).
- Sample Identification Procedures Each container should have an acceptable identification label so the sample can be tracked accurately and an uninterrupted chain-of-custody can be maintained.
- <u>Sample Packaging and Delivery Concerns</u> Once a sample is collected, it must be delivered to the laboratory for analysis to be conducted within the prescribed holding time. Holding times are specified in 40 CFR Part 136.
- <u>Safety Concerns</u> Sampling personnel should have complete information on any relevant plant safety regulations and safety procedures to be followed during onsite sampling activities. Personnel should be familiar with EPA Order 1440.2 (see Appendix F).
- <u>Hazardous Waste Concerns</u> Samples of potentially hazardous effluent or process waste; samples with extremely high or low pH; and samples that may contain extremely toxic, volatile, or explosive substances will require special handling. DOT regulations for shipping these types of samples must be followed.
- <u>Chain-of-Custody Procedures</u> Procedures for chain-of-custody must be followed for all samples. Chain-of-custody forms should be used for this purpose.
- <u>QA/QC Procedures</u> To ensure that data collected is valid, systematic checks must show that test results are accurate.

| Several of these considerations must be coordinated with the laboratory. The inspector should contact the |
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| laboratory in advance of any sampling to discuss the sampling plan and QA/QC procedures, allocate laboratory |
| time, and obtain sample identification numbers. |

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2.3 PREPARATION FOR SAMPLING

The success of each sampling task hinges on adequate preparation. Because personnel may not be familiar with the facility to be inspected, the sampling plan should be reviewed prior to going out into the field. Personnel should be briefed, as well, on all field procedures, particularly safety requirements. The inspector should make sure that the appropriate sampling equipment is available and in good working order. When sample analyses are to be performed in the field (such as pH), the necessary instruments should also be included. Equipment must be checked prior to going into the field to ensure accurate operation and calibration. In addition, a review of necessary safety equipment should be made and the inspector should be aware of any hazards. The inspector and plant staff should discuss any unusual circumstances and formulate a plan for dealing with them during the inspection.

A checklist of field sampling items (see Appendix G) can be used to ensure proper preparation. When the type of waste to be sampled is known ahead of time, the list can be narrowed to the actual pieces necessary.

2.4 SAMPLING SAFETY

In developing the sampling plan, the inspector should not sample at locations which pose a threat to health and safety. Under hazardous conditions, a two-person inspection team is necessary. All required safety equipment and protective clothing should be used as well. EPA Order 1440.2 (see Appendix F) specifies the equipment and clothing required for EPA personnel at various levels of exposure.

Extensive and continuous education is essential to a successful safety program. The inspector should be familiar with hazards associated with sampling in addition to the safety measures to be followed. For example, if the inspector is required to enter a manhole or other confined space to obtain a sample, training in confined space entry and rescue procedures is required. Potential hazards in a confined space include toxic gases, such as hydrogen sulfide, chlorine, and carbon monoxide; or explosive gases, such as gasoline vapors or methane. In addition, an atmosphere may be hazardous because there is not enough oxygen to support life due to the

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presence of other gases, such as hydrogen sulfide and/or carbon dioxide. A confined space, such as a manhole, should not be entered until the atmosphere has been tested for sufficient oxygen and the lack of toxic or explosive gases. Such a confined space should never be entered alone or without a lifeline. The ability to recognize hazards and to follow proper procedures will eliminate unnecessary accidents.

2.5 SAMPLING LOCATION

The inspector should always collect samples from a representative sampling that reflects total effluent flow. Convenience and accessibility are important, but are secondary to the representativeness of a sample. A representative location is where specific conditions or parameters are measured that adequately reflect the actual conditions of those waters or wastewaters. The most representative samples will be drawn from a depth where the flow is turbulent and well mixed and the chance of solids settling is minimal. Depending on the sampling location, the depth may range from a few inches below the wastestream's surface to 40 to 60 percent of the wastestream's total flow. Stagnant areas must be avoided as well, particularly if the wastewater contains immiscible liquids or suspended solids. The inspector should take care to collect samples from the center of the flow with the container facing upstream to avoid contamination. Wide channels or paths of flow may require dye testing to determine the most representative sampling site. Dye testing involves placing a colored dye in a wastestream and following the color to the outfall. If dye testing is inconclusive, cross-sectional sampling may be required.

If the sample location specified in the NPDES permit is not adequate to collect a representative sample, the inspector should determine an alternative location. This determination should be based on the inspector's knowledge of the plant itself, the production processes, and the outfalls. If there is a conflict between the sample location described in the permit and the location the inspector feels is most representative, samples should be collected at both sites. The reason for the conflict should be thoroughly documented for later resolution by the permitting authority.

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In addition to sampling effluent, the NPDES permit will often specify that the influent or internal wastestreams be sampled, particularly when there is a percent removal requirement. Limits on the influent or internal wastestreams of an industrial facility may be imposed only when exceptional circumstances make such limitations necessary, such as when:

- The final discharge point is inaccessible (for example, under 10 meters of water)
- The wastes at the point of discharge are so dilute that monitoring is impractical
- Interferences among pollutants at the point of discharge make detection or analysis impossible.

When the permit requires that influent to the wastewater treatment facility be sampled (i.e., where treatment efficiencies need to be determined), the preferred sample collection locations are those that provide the best mixing, such as an influent line upflow distribution box from the plant wet well, or a flume throat. These samples should be collected upstream of any sludge or supernatant recirculation. If samples are taken from a closed conduit via a valve or sample tap or from a well equipped with a hand or mechanical pump, the inspector should allow sufficient flushing time to ensure a representative sample, taking into account the diameter, the length of pipe to be flashed, and the velocity of the flow.

2.6 SELECTION AND PREPARATION OF SAMPLE CONTAINERS

Sample containers must be made of chemically resistent material that does not affect the concentration of pollutants to be measured. The containers used should be either glass or plastic. For most analyses, the option of using either glass or plastic sample containers is open, and the selection of the sample container is based on the organization's operating procedures. It is important that the inspector become familiar with these procedures. If either type of sample container is acceptable and available, the inspector should use plastic ones because they are less likely to break.

Plastic sample bottles are usually made of polyethylene; however, containers with teflon bottoms and lid liners are available. The teflon provides added chemical resistance to strong mineral acids or organic solvents, although this added chemical resistance is not normally needed. Glass sample bottles are required when

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collecting samples for priority pollutants, oil and grease, and phenols, while plastic sample bottles are most often used for Biochemical Oxygen Demand (BOD), Total Suspended Solids (TSS), metals, and nutrients. Containers with wide mouths are recommended to facilitate the transfer of samples from sampler to sample containers. In addition, the container must be large enough to contain the required volume for laboratory analysis. The inspector should use dark containers for samples that contain constituents which will oxidize from exposure to sunlight, such as iron cyanide (which is oxidized to hydrogen cyanide).

Container lids and closure linings must also be intact so they do not interfere with the pollutant parameters to be measured. Most containers have tight, screw-type lids. Plastic containers are usually provided with screw caps made of the same material as the container, so cap liners are usually not required. Glass containers usually come with rigid plastic screw caps. Liner materials may be polyethylene, polypropylene, neoprene, or teflon.

The inspector should make sure that all sample containers are clean and uncontaminated. The general cleaning procedure for a sample container is to:

- Wash with hot water and detergent
- Rinse thoroughly with tap water followed by three or more rinses with organic-free water
- Rinse glass containers with an interference-free, redistilled solvent, such as acetone
- Dry in a contaminant-free room.

Precleaned and sterilized disposable containers are available for sampling use. The most commonly used container of this type is the molded polyethylene cubitainer shipped (nested) to the buyer.

All tubing and other sampling system parts must be scrubbed with hot water and detergent, rinsed several times with tap water, and then rinsed with distilled or deionized water. Further rinsing with acetone is advised only when the type of tubing (e.g., teflon) is not susceptible to dissolution by the solvent.

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In most cases, the container should be rinsed three times with the wastewater to be sampled before the sample is taken. However, some sample containers, such as those used for bacteriological sampling, require special cleaning procedures. Bacteriological sample containers must be sterilized prior to sample collection. The inspector should refer to Standard Methods for the Examination of Water and Wastewater and 40 CFR Part 136 for proper procedures on sample container preparation.

2.7 SAMPLE TYPES

There are two primary types of samples: grab samples and composite samples. Each type has distinct advantages and disadvantages. To obtain a complete characterization of a specific facility's effluent, the two sample types can be used in combination. However, the inspector must use the appropriate sample type for compliance monitoring based on the requirements specified in the NPDES permit.

A grab sample is an individual sample collected over a period of time not exceeding 15 minutes. Grab samples represent the conditions that exist at the moment the sample is taken and do not necessarily represent conditions at any other time. Grab sampling is the preferred method of sampling under the following conditions:

- When the effluent is not discharged on a continuous basis. The true characteristics of a wastestream may be obtained only when the batch discharge occurs.
- When specific pollutant parameters are immediately affected by biological, chemical, or physical interactions, such as pH, temperature, chlorine, soluble sulfide, volatile organics, cyanide, and dissolved oxygen. Individual grab samples should always be taken for oil and grease and when bacteriological analysis will be performed.
- When the waste conditions are relatively constant over the period of discharge. In lieu of complex sampling activities, a grab sample provides a simple and accurate method of establishing waste characteristics.
- When it is necessary to check for extreme conditions. For example, composite samples would tend to conceal peaks in the pH of a discharge. Extreme acidic and alkaline conditions may cancel each other out, causing a composite sample to appear neutral.

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In addition, grab samples may be used to determine consistency in an industry's self-monitoring data and to corroborate results of composite samples.

A composite sample is a sample collected over time, formed either by continuous sampling or by mixing discrete samples. Composite samples reflect the average characteristics of the wastestream during the compositing period. Composite samples are used when stipulated in a permit and when:

- Determining average pollutant concentration during the compositing period
- Calculating mass/unit time loadings.

Various methods for compositing samples are available. Composite samples may be collected individually at equal time intervals if the flow rate of the sample stream does not vary more than plus or minus 10 percent of the average flow rate, or they may be collected proportional to the flow rate. The permit may specify which composite sample to use, either time composites or flow-proportional composites. The compositing methods, all of which depend on either continuous or periodic sampling, are described below:

- <u>Time Composite Sample</u> Composed of discrete sample aliquots collected in one container at constant time intervals. This method provides representative samples when the flow of the sampled stream is constant. This type of sample is similar to a sequential composite sample (described below).
- Flow-Proportional Composite Sample There are two methods used to collect this type of sample. One method collects a constant sample volume per stream flow [e.g., 200 milliliters (ml) sample collected for every 5,000 gallons of stream flow] at time intervals proportional to stream flow. This method provides representative samples of all wastestreams when the flow is measured accurately. For this reason, it is used most frequently. In the other method, the sample is collected by increasing the volume of each aliquot as the flow increases, while maintaining a constant time interval between the aliquots.
- Sequential Composite Sample Composed of discrete samples composited in individual containers at
 constant time intervals or constant discharge increments. For example, samples collected every 15
 minutes are composited each hour. The 24-hour composite is made up from the individual 1-hour
 composites. Each of the 24 individual samples is manually flow-proportioned according to the flow
 recorded for the hour the sample represents. Each flow-proportioned sample is then added to the
 composite samples. The actual compositing of the samples may be done in the field or the

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laboratory. In most cases, compositing in the field is preferable since only one sample container must be cooled, transported to, and handled in the laboratory. This method of compositing is frequently used since an automatic sampler can easily collect the individual samples. A variation of this method is to collect a constant volume of sample taken at constant discharge increments, which are measured with a totalizer. For example, one aliquot is collected for every 10,000 gallons of flow.

• <u>Continuous Composite Sample</u> - Collected continuously from the wastestream. The sample may be a constant volume which is similar to the time composite, or the volume may vary in proportion to the flow rate of the wastestream, in which case the sample is similar to the flow-proportional composite.

Table 2-1 lists the advantages and disadvantages of each sampling method. Either manual or automatic sampling techniques can be used. If a sample is composited manually, sample manipulation should be minimized to reduce the possibility of contamination.

The inspector must always use the method required by the permit and also weigh advantages and disadvantages when choosing between the use of grab or composite sampling methods. While grab sampling allows observation of unusual conditions that may exist during discharge, such as sudden bursts of color or turbidity, this method is labor-intensive and impractical when sampling is performed at many locations over extended periods of time. When sampling a large number of locations, the use of automatic samplers is more practical. Automatic samplers also help reduce human error, specifically in complex sampling activities, such as flow-proportional sampling, and reduce exposure to potentially hazardous environments. The primary disadvantage to automatic sampling is the cost of the equipment and maintenance requirements. Many automatic samplers in use today are electronically controlled and must be sent back to the manufacturer when a malfunction occurs. There is also a greater possibility of tampering when using an automatic sampler.

2.8 SAMPLE COLLECTION TECHNIQUES

To obtain a representative sample, sampling must be conducted where wastewater flow is adequately mixed. Ideally, a sample should be taken in the center of the flow where velocity is highest and there is little possibility of solids settling. The inspector should avoid skimming the surface of the wastestream or dragging the channel bottom. Mixing of the flow is particularly important for ensuring uniformity. Sampling personnel

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TABLE 2-1. COMPOSTING METHODS

| Method | Advantages | Disadvantages | Comments |
|--|--|--|--|
| Time Composite Constant sample volume, constant time interval between samples | Minimal instrumen- tation and manual effort; requires no flow measurement | May lack represen- tativeness especi- ally for highly variable flows | Widely used in both automatic samplers and manual handling |
| Flow-Proportional Composite Constant sample volume, time interval between samples proportional to stream flow | Minimal manual | Requires accurate flow measurement reading equipment; manual compositing from flow chart | Widely used in automatic as well as manual sampling. |
| Constant time interval between samples, sample volume proportional to total stream flow at time of sampling | Minimal instrumentation | Manual compositing from flow chart in absence of prior information on the ratio of minimum to maximum flow; chance of collecting too small or too large individual discrete samples for a given composite volume | Used in automatic samplers and widely used as manual method |

TABLE 2-1. COMPOSITING METHODS (Continued)

| Method | Advantages | Disadvantages | Comments |
|--|---|---|--|
| Constant time interval between samples, sample volume proportional to total stream flow since last sample | Minimal instrumentation | Manual compositing from flow chart in absence of prior information on the ratio of minimum to maximum flow; chance of collecting either too small or too large individual discrete samples for a given composite volume | Not widely used in automatic samplers but may be done manually |
| Sequential Composite Series of short period composites, constant time intervals between samples | Useful if fluctua- tions occur and time history is desired | Requires manual compositing of aliquots | Commonly used; however, manual compositing is labor intensive |
| Series of short period composites, aliquots taken at constant dis- charge increments | Useful if fluctua- tions occur and the time history is desired | Requires flow totalizer; re- quires manual compositing of aliquots | Manual compositing is labor intensive |

TABLE 2-1. COMPOSITING METHODS (Continued)

| Method | Advantages | Disadvantages | Comments |
|--|--|---|-------------------------------|
| Continuous Composite | | | |
| Constant sample volume | Minimal manual effort, requires no flow measurement highly variable flows | Requires large sample capacity; may lack represen- tativeness for highly representative flows | Practical but not widely used |
| Sample volume proportional to stream flow flows; minimal manual effort | Most representa- tive especially for highly variable sample volume, variable pumping capacity, and power | Requires accurate flow measurement equipment, large sample volume, variable pumping capacity and power | Not widely used |

should be cautious when collecting samples near a weir because solids tend to collect upstream and floating oil and grease accumulate downstream. If the sample is not to be tested for volatile organics or will not be affected by stripping dissolved gases, mechanical stirring may be used or a stream of air may be introduced into the wastestream.

When taking a grab sample, the entire mouth of the container should be submerged below the wastestream's surface. A wide mouth bottle with an opening of at least two inches is recommended for this type of sampling. When using a composite sampler, the sample line should be kept as short as possible and sharp bends, kinks, and twists in the line (where solids can settle) should be avoided. The sample line should be placed so that changes in flow will not affect sample collection.

2.9 SAMPLE VOLUME

The volume of samples collected depends on the type and number of analyses needed, as reflected in the parameters to be measured and the requirements of the analytical laboratory being used. Sample volume must be sufficient for all analyses, including laboratory QA/QC and any repeat analyses used for verification.

Laboratory personnel should be contacted for the sample volume required to complete all analyses. Individual, minimum composite portions should be 25 to 100 milliliters, with a total composite volume of 2-4 gallons. Larger volumes may be necessary if samples are to be separated into aliquots or if bioassay tests are to be conducted.

Volume requirements for individual analyses range from 25 ml for pH and volatile organic determinations to 1,000 ml or more for BOD, oil and grease, settleable matter, and temperature determinations. The inspector should always collect more than the minimum sample volume to allow for spillage and laboratory reruns. Appendix D lists minimum volume requirements for various pollutant parameters.

2.10 SAMPLE PRESERVATION AND HOLDING TIMES

| Prese | ervation techniques ensure that the sample remains representative of the wastestream at the time of |
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| collection. | Since most pollutants in the samples collected are unstable (at least to some extent), this instability |
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requires that the sample be analyzed immediately or that it be preserved or fixed to minimize changes in the pollutant concentrations between the time of collection and analysis. Because immediate analysis is not always possible, most samples are preserved regardless of the time of analysis.

However, problems may be encountered when 24-hour composite samples are collected. Since sample deterioration can take place during the compositing process, it is necessary to preserve or stabilize the samples during compositing in addition to preserving aggregate samples before shipment to the laboratory. Preservation techniques vary depending on the pollutant parameter that is to be measured; therefore, familiarity with 40 CFR Part 136 (see Appendix E) is essential to ensure proper preservation techniques. It is important to verify that the preservation techniques for one parameter do not affect the analytical results of another in the same sample. If this is the case, two discrete samples should be collected and preserved accordingly.

Sample preservation techniques consist of refrigeration, pH adjustment, and chemical fixation. Refrigeration is the most widely used technique because it has no detrimental effect on the sample composition and does not interfere with any analytical methods. Refrigeration requires that the sample be quickly chilled to a temperature of 4°C, which suppresses biological activity and volatilization of dissolving gases and organic substances. This technique is used at the start of sample collection in the field and during sample shipment, and continued until the sample it received in the laboratory for analysis. Sample temperature should be verified and recorded by the inspector. This is particularly important if the analytical results are to be used in an enforcement action.

In addition to preservation techniques, 40 CFR Part 136 indicates maximum holding times. Times listed are the maximum times between sample collection and analysis that are allowed for the sample to be considered valid. The wastewater becomes a sample upon combination of the last aliquot. At that point, holding time limitations begin. A detailed list of preservation methods and holding times appears in Appendix E of this module. These sample preservation procedures and holding times were selected by EPA because they retard sample degradation and minimize monitoring costs by extending holding times as long as possible.

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2.11 SAMPLE DOCUMENTATION

Because sampling reports may be used in enforcement proceedings, the inspector must keep a precise record of sample collection and data handling. All field records containing these data must be signed by the inspector at the time of collection. If required, the following information should also be documented in the field records.

- Unique Sample or Log Number All samples should be assigned a unique identification number. If
 there is a serial number on the transportation case, the inspector should add this number to his/her
 field records.
- Date and Time of Sample Collection Date and time (including notation of a.m. or p.m.) of sample collection must be recorded. In the case of composite samples, the sequence of times and aliquot size should be noted.
- Source of Sample, Including Facility Name and Address This may be obtained from the sample request form. A narrative description and/or diagram referring to the particular site where the sample was taken should be included.
- Name of Sampling Personnel The name(s) and initial(s) of the person(s) taking the sample must be indicated. For a composite sample, the name(s) of the person(s) installing the sampler and the name(s) of the person(s) retrieving the sample must be included.
- Sample Type Each sample should indicate whether it is a grab or composite sample. If the sample is a composite, volume and frequency of individual samples should be noted.
- <u>Preservation Used</u> Any preservatives (and the amount) added to the sample should be recorded. The method of preservation (e.g., refrigeration at 4°C) should be indicated.
- Analysis Required All parameters for which the sample must be analyzed at the laboratory should be specified.
- <u>Field Analysis</u> Field measurements must be recorded at the time that the analysis is completed. Examples of analysis which must be conducted and recorded in the field include pH, temperature, dissolved oxygen, chlorine residual, and sulfites.
- Flow If flow is measured at the time of sampling, the measurement must be recorded.
- <u>Production Rates</u> Information on products manufactured and production rates should be included since many effluent limitations are based on production rates.

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- <u>Date, Time, and Documentation of Sample Shipment</u> The shipment method (i.e., air, rail, or bus) as well as the shipping papers or manifest number should be noted.
- Comments Comments refer to all relevant information pertaining to the sample or the sampling site. Such comments include the condition of the sample site, observed characteristics of the sample, environmental conditions that may affect the sample, and problems encountered in sampling.

2.12 SAMPLE IDENTIFICATION AND LABELING

At the time that a sample is collected, a waterproof, gummed label or tag should be attached to the sample container. This label is necessary to prevent misidentification of samples since it provides the laboratory with relevant information for sample analysis, such as:

- Name of the sample collector
- Sample identification number
- Date and time of sample collection
- Location of sample collection
- Preservatives used.

Sample seals should be used to protect the sample's integrity from the time it is collected to the time it is opened in the laboratory. The seal should also contain the collector's name, the date and time of sample collection and a sample identification number. Information on the seal must be identical to the information on the label. In addition, the seal must be attached so it must be broken to open the sample container. Example sample identification labels are provided in Appendix H. Caution should be observed to assure that glue on sample seals and tag wires do not contaminate samples, particularly those containing volatiles and metals.

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2.13 SAMPLE PACKAGING AND SHIPPING

After the samples are properly labeled, they should be placed in a transportation case along with the chain-of-custody record form, pertinent field records, and analysis request forms. (Chain-of-custody procedures are covered below.) Glass bottles should be wrapped in foam rubber, plastic bubble wrap, or other material to prevent breakage during shipment. The wrapping can be secured around the bottle with tape. Samples should be placed in ice or a synthetic ice substitute that will maintain sample temperature at 4°C throughout shipment. Ice should be placed in double-wrapped watertight bags to ensure the water will not drip out of the shipping case. Metal or heavy plastic chests make good sample transportation cases. Filament tape wrapped around each end of the ice chest ensures that it will not open during transport. Sampling records can be placed in an envelope and taped to the transportation case to avoid getting them wet in case either a sample or ice bag leaks. Shipping containers should also be sealed to prevent tampering.

Most samples will not require any special transportation precautions except careful packaging to prevent breakage and/or spillage. If the sample is shipped by common carrier or sent through the U.S. mail, it must comply with DOT Hazardous Materials Regulations (49 CFR Parts 171-177). Air shipment of hazardous materials samples may also be covered by requirements of the International Air Transport Association (IATA). Before shipping a sample, the inspector should be aware of, and follow, any special shipping requirements. Special packing and shipping rules apply to substances considered hazardous materials as defined by IATA rules. Wastewater samples are not generally considered hazardous materials (see Footnote Number 3 in Appendix E).

2.14 CHAIN-OF-CUSTODY PROCEDURES

Once a sample has been obtained and collection procedures are properly documented, a written record of the chain of possession of that sample must be made. "Chain-of-custody" refers to the documented account of changes in possession that occur for a particular sample or set of samples. The chain-of-custody record allows an accurate step-by-step recreation of the sample path, from origin through analysis. Some of the information that needs to be addressed in chain-of-custody is:

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- Name of the person collecting the sample
- Sample ID numbers
- Date and time of sample collection
- Location of sample collection
- Name(s) and signature(s) of all persons handling the samples in the field and in the laboratories.

To ensure that all necessary information is documented, a chain-of-custody form should be developed. An example of such a form used by EPA is found in Appendix I. Chain-of-custody forms should be preprinted on carbonless, multipart paper so all personnel handling the sample receive a copy. All sample shipments must be accompanied by the chain-of-custody record while a copy of these forms should be retained by the originator. In addition, all receipts associated with the shipment should be retained. Carriers typically will not sign for samples; therefore, seals must be used to verify that tampering has not occurred during shipment.

When transferring possession of samples, the transferee must sign and record the date and time on the chain-of-custody record. In general, custody transfers are made for each sample, although samples may be transferred as a group, if desired. Each person who takes custody must fill in the appropriate section of the chain-of-custody record.

Typically, the chain-of-custody for a sample is as follows:

- Sampling Personnel The person(s) who takes possession of the sample as soon as it is collected
- <u>Laboratory Personnel</u> <u>Laboratory personnel</u>, whether from agency laboratories or from an independent laboratory, will be responsible for the sample from analysis through disposal.

In addition, permit and/or compliance group should receive a copy of the completed chain-of-custody form, particularly if the sample results are to be used for enforcement purposes.

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Chain-of-custody records are critical if analytical data are to be used in an enforcement proceeding because they allow such data to be introduced as evidence without testimony of the persons who made the record. Therefore, it is important that all chain-of-custody records be complete and accurate. To maintain the sample's integrity, chain-of-custody records must show that the sample was properly collected, preserved, and analyzed, and was not tampered with. Since it is not possible to predict which violations will require legal action, it should be assumed that all data generated from sampling will be used in court.

2.15 SPECIAL SAMPLING REOUIREMENTS

In general, most samples are taken using similar techniques. However, the inspector should be aware that certain parameters require special precautions in sample collection, preservation, and handling.

2.15.1 Bacteriological Sampling

Bacteriological sampling should always be a grab sample collected in a sterilized container, according to Standard Methods for the Examination of Waster and Wastewater. A 125 ml or larger sample container should be used to provide a minimum sample volume of 100 ml and adequate mixing space. Unlined caps or ground glass tops should be used to ensure complete sterilization of the container's closure. Bottles and caps must be thoroughly cleaned with detergent and hot water and a final deionized water rinse should be performed prior to use. All traces of detergent must be removed. A test for bacteriostatic or inhibitory residues is described in Standard Methods for the Examination of Water and Wastewater.

When sampling water containing residual chlorine, a dechlorinating agent such as sodium thiosulfate should be added to the sample bottle prior to sterilization in an amount to provide an approximate concentration of 100 milligram per liter (mg/1) in the sample. This can be accomplished by adding 0.1 ml of a 10 percent solution of sodium thiosulfate to a 125 ml sample bottle. The dechlorinating agent neutralizes any residual chlorine and will prevent further reaction between bacteria and chlorine.

| For | bacteriological san | pling, the contain | er must be kep | pt unopened | until the momer | nt that the | sample is |
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| collected. | During sampling, | the bottle's lower | part should be | e held with the | he mouth of the | bottle faci | ng the |

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direction of the current. The stopper or cap should be protected from contamination during sampling and must be replaced immediately after the sample has been taken. The inspector should fill the sample bottle to within one to two inches of the top. S/he should not rinse the bottle with the sample. The inspector should never collect the sample in an unsterilized sample container and then transfer it to a sterile container.

2.15.2 Radiological Sampling

Polyethylene, polyvinyl chloride, or teflon containers are recommended for collecting radioactive samples because these containers are less adsorbent than glass or metal containers. Since radioactive elements are often present in submicrogram quantities, a large fraction of the elements may be lost by adsorption on container or glassware surfaces used in analysis. This loss may, in turn, cause a loss of radioactivity and possibly contaminate subsequent samples due to reuse of inadequately cleaned containers. Glass bottles are also more susceptible to breakage during handling than plastic containers. The standard preservation technique for radiological sampling is acidification to a pH of less than 2.0 with HNO₃. However, there are some exceptions, and the inspector should contact the laboratory before the sample is collected to find out what these are. Prior to sampling, the area should be surveyed with a beta-gamma survey instrument, such as a Geiger-Mueller meter. If radiation levels above instrument background are detected, the inspector should consult a radiation safety specialist to determine appropriate safety procedures.

2.15.3 Metals Sampling

New plastic or glass containers should be used for metals sampling. If previously used containers are used for metals sampling, they should be washed with 1+1 nitric acid and rinsed with redistilled water. Samples should be preserved with nitric acid at collection time to keep metals in solution and prevent them from plating out on the container wall. Approximately 5 ml of concentrated, redistilled HNO₃ should be added per liter of sample to reduce pH to below 2.0. If only dissolved metals are to be measured, the sample should be filtered through a 0.45 membrane filter prior to acidification.

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2.15.4 Volatiles Sampling

Analysis of volatile organic substances requires a 40 ml glass sample vial, sealed with a teflon-coated septum seal. The sample must be collected so there are no air bubbles in the container after the screw cap and septum seal are applied. Because it is difficult to completely fill the 40 ml bottle directly from the wastestream, a larger glass bottle that has been appropriately cleaned may be used to grab the sample from the wastestream; the inspector should then transfer the sample to the 40 ml vial. The sample must be poured into the vial very slowly to minimize aeration of the sample. If the sample is known to contain residual chlorine, 10 mg of sodium thiosulfate must be added to the empty vial first.

2.15.5 Oil and Grease Sampling

Sampling for oil and grease is unique because the pollutant is immiscible. Oil tends to adhere to the sampling device; therefore, an oil and grease sample must always be a grab sample taken in a glass container. A teflon insert should be included in the glass sample container's lid. However, if teflon is not available, aluminum foil extending out from under the lid may be used. Grab samples for analysis of oil and grease (or other immiscible pollutants) should not be transferred from the sampling container and must be analyzed separately to avoid pollutant loss. The sample must be preserved by adding H₂SO₄ to reduce the pH to less than 2.0 and then chilled to 4°C.

2.15.6 Cyanide Sampling

Cyanide is very reactive and unstable. Because of its nature, cyanide must be analyzed as soon as possible after collection. The sample must be taken in a polyethylene or glass bottle. If the sample cannot be analyzed immediately, it must be preserved after collection with NaOH pellets or a strong NaOH solution to raise the pH of the sample to 12.0 or above. If residual chlorine is present, 0.6 grams (g) of ascorbic acid is added to the sample container. A preserved cyanide sample has a maximum holding time of 14 days. However, the maximum holding time is reduced to 24 hours when sulfide is present. A lead acetate paper spot test can be done in the field to determine the presence of sulfide. Sulfide can be removed by adding

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cadmium nitrate powder to the sample. If cadmium nitrate is added, the sample must be filtered prior to adding the NaOH. Preserved samples must be stored in a closed, dark bottle at 4°C.

2.15.7 Organics and Pesticides

Although conventional sampling practices should be followed for these parameters, there are several special considerations. First, the sample bottle must not be prerinsed with the sample before collection. Second, if grab samples of these parameters are taken, the samples must always be collected in amber glass containers, one liter to one gallon volume. Composite samples must be collected in refrigerated glass containers through teflon tubing. Third, if the sample can not be extracted within 72 hours of collection, the sample's pH may need to be adjusted with sodium thiosulfate. Table II of 40 CFR Part 136 (which is excerpted in Appendix E) should be consulted to determine the specific preservation method for each group of organic compounds and pesticides.

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3. ANALYTICAL METHODS FOR ONSITE ANALYSIS

Proper analytical methods are extremely important to a successful sampling program. The inspector should consult such reference materials as 40 CFR Part 136, EPA's <u>Methods for Chemical Analysis of Water and Wastes</u>, and <u>Standard Methods for the Examination of Water and Wastewater</u> before beginning any analytical tests.

Generally, pollutant parameter values should be determined by one of the standard analytical methods shown in Appendix E. The inspector usually does not analyze the actual collected sample. The exception to this is analyzing a sample for parameters that cannot be preserved. Measurements of these parameters should be taken at the beginning of the sampling period so that, if violations or problems are identified, additional measurements or information may be obtained during the remainder of the inspection. The most common parameters for which field measurements are conducted are temperature, Dissolved Oxygen (DO), pH, and chlorine residual. Field analysis of each of these parameters is highlighted below.

- <u>Temperature</u> Temperature determinations can be made with any good grade mercury filled or dial-type Celsius thermometer. The dial-type thermometer is preferred over the glass type for field work because of its durability and ease of reading. All temperature measuring devices must be calibrated periodically with a precision thermometer traceable to the National Bureau of Standards.
- <u>DO</u> The electrode method is predominantly used for onsite DO determinations. The sample size for this type of determination is 300 ml. Most DO probes are temperature-sensitive and have temperature compensation built in. The DO meter must be calibrated onsite in accordance with the manufacturer's specifications before any DO measurements are made.
- pH pH determinations are often conducted during a sampling inspection. The inspector should arrange to have a pH meter available. The pH meter must be properly calibrated by the use of two buffers prior to each set of pH measurements. The inspector should be aware of conditions in the wastestream that may cause inaccurate readings. For example, oil and grease may interfere with readings (cause a sluggish response by coating the electrodes).
- Chlorine Residual Chlorine in an aqueous solution is unstable and the concentration will decrease rapidly. Exposure to sunlight or other strong light, or agitation will accelerate chlorine reduction; therefore, analysis should begin immediately after sampling. A field colormetric kit such as the

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HACH DRIOO is an approved EPA procedure if the instrument is properly calibrated using standards to develop a calibration curve.

• Conductivity - Specific conductance is a useful method to approximate the total amount of inorganic dissolved solids. Conventional conductivity devices consist of two or more platinum electrodes separated by a test solution. The major disadvantage with this type of system is the possibility of polarization or poisoning (fouling) of the electrodes. Conductivity systems based on the measurement of inductance or capacitance are also available. The electrodes in these systems are insulated by a layer of glass or other insulating material. System response is less rapid, but problems with fouling and polarization are eliminated. Temperature is important in conductivity measurements. For example, the conductivity of salt water increases 3 percent per degree at 0°C, and only increases 2 percent per degree at 25°C. Therefore, it is necessary to record temperature with conductivity measurements or to adjust the temperature of the samples prior to making conductivity measurements. Most conductivity meters have a manual temperature compensation feature.

The inspector should always remember that when analysis is performed in the field, each piece of equipment should be checked prior to leaving for the sampling site. The inspector should verify that the equipment is in working order and calibrated and that batteries are properly charged before leaving on the inspection.

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4. AUTOMATIC SAMPLERS

A wide variety of automatic samplers are available commercially. Most have the following five interrelated subsystem components:

- Sample Intake Subsystem The sample intake gathers representative samples from the sampling stream. The intake is usually the end of a plastic suction tube. The inside diameter of this tube should be at least 1/4 inch, which is large enough to lessen chances of clogging but small enough to maintain velocity and to avoid solids settling. It should also be resistant to physical damage from large objects in the flow stream. Nonleaching tygon tubing is most often used; however, teflon tubing must be used when sampling for priority pollutants. The end of this tubing should be fixed so that its sampling location can be maintained throughout the sampling period. The automatic sampler should provide for the line purging after each sample is drawn to prevent contamination of subsequent samples.
- Sample Gathering Subsystem Automatic samplers provide one of three basic gathering methods:
 - Mechanical Mechanical gathering subsystems are usually built into place and include devices such as cups on cables, calibrated scoops, and paddle wheels with cups. Although these systems may obstruct the stream flow, they also take into account site-specific considerations, such as a very high sampling lifts and wide or extremely deep channel flows. Because of the mechanical nature of the system. These units require periodic inspection and maintenance.
 - Forced Flow Forced-flow gathering subsystems are often built into place as permanent sampling facilities; thus, like the mechanical gathering subsystems, they may obstruct the stream flow. They also require periodic inspection and maintenance. However, forced flow subsystems have the advantage of being able to sample at great depths. In addition, because this gathering system uses air pressure to transport the sample, it may be ideal for sample collection in potentially explosive environments.
 - Suction Lift The suction lift is the most widely used type of sample gathering subsystem due to its versatility and minimal affect on flow patterns. Suction lifts are limited to 25 vertical feet or less because of internal friction losses and atmospheric pressure. As with all suction devices, when the pressure or a liquid that contains dissolved gases is reduced, the dissolved gases tend to pass out of the solution. Because the gases leaving the surface have entrained suspended solids, the liquid's surface layer becomes enhanced with suspended solids. To minimize the concentration effect, at least 100 ml per sampling unit should be collected.

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- Sample Transport Subsystem The sample is usually transported from the sample intake to the collection bottle by a plastic tube referred to as the sample transport subsystem. The tubing should be at least 1/4 inch inside diameter to maintain adequate flow and to prevent plugging. The tubing should be sized so that a velocity of at least two feet per second can be maintained. Care should be exercised to avoid sharp bends, kinks, and twists in the transport line. Automatic samplers usually provide a line purge after each sample is collected to ready the line for the next sample transfer. The inspector should provide clean transport tubing for each new sample site to prevent sample contamination.
- Sample Storage Subsystem The sample storage subsystem can accommodate either a single large collection bottle or a number of smaller collection bottles. The total sample volume storage capability should be at least 2 gallons (7.6 liters); some samplers have a capacity as great as 5 gallons. To preserve the samples, storage subsystems should also be large enough to provide space for ice to chill the sample after collection. In addition, preservatives may be added to the sample bottle(s) prior to sample collection. Samples with individual bottles for discrete collection are usually equipped with a cassette which rotates to fill the bottle at the time of sampling. As previously mentioned, whether large composite or discrete samples are collected, it is necessary to use collection bottles made of the appropriate materials.
- Controls and Power Subsystem The automatic samplers most widely used have encapsulated solid state controls. This minimizes the effect of the highly unfavorable environments that may be encountered in the field, such as high humidity and corrosiveness. These units are also sealed so they may be used with minimum risks in potentially explosive environments. In addition, sealed units protect the controls if the sampler is accidentally submerged. Samplers operating from a power supply are more reliable than battery operated models; however, field conditions often prohibit the use of a power supply. The control units allow selection of time or flow-compositing method, or continuous sampling method.

Several factors should be considered in selecting automatic sampling equipment. Among these are:

(1) convenience of installation and maintenance; (2) equipment security; and (3) cold weather operation.

Sampling equipment should always be handled carefully and maintained in accordance with the manufacturer's instructions. Most equipment failures are caused by careless handling and poor maintenance. Equally important is equipment security, specifically when sampling is done as part of an enforcement proceeding.

Manhole locations where battery-operated equipment may be installed and the cover replaced will aid in maintaining security. If sampling equipment must be left unattended, the sampler should be provided with a lock or seal which, if broken or disturbed, would indicate that tampering had occurred.

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| Use of automatic samplers during cold weather presents problems with freezing. Sampler malfunctions |
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| and frozen intake lines are quite common. These problems may be handled by using heat tape or placing the |
| sampler inside a thermostatically controlled, electrically heated enclosure. In the absence of special equipment, |
| freezing may be prevented by installing the sampler in a manhole or wet well or by wrapping the sampler with |
| eight or nine inches of insulation and wind protection. Also, the sampler should be positioned well above the |
| effluent stream so that the tubing runs in a taut, straight line to prevent liquid from pooling. Criteria for |
| selecting automatic sampling equipment are listed in Appendix J. |

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5. FLOW MEASUREMENT

5.1 IMPORTANCE OF FLOW MEASUREMENT

Pollutant limits in a NPDES permit are usually specified as a mass loading. To determine a mass loading and thereby evaluate compliance with permit limits, it is necessary for the inspector to obtain accurate flow data. "Flow measurement" is the commonly used term for this process. In addition to verifying compliance with permit limits, flow measurement serves to:

- Provide operating and performance data on the wastewater treatment plant
- Compute treatment costs, based on wastewater volume
- Obtain data for long-term planning of plant capacity.

This section briefly describes flow measurement. For a more detailed discussion, the inspector should refer to two other EPA guidance manuals, the 1988 NPDES Compliance Inspection Manual and the 1981 NPDES Compliance Flow Measurement Manual.

5.2 OPEN CHANNEL FLOW

Open channel flow, where the flow occurs in conduits that are not full of liquid, is the most prevalent type of flow at NPDES-regulated discharge points. Partially full pipes that are not under pressure are classified as open channels as well. Open channel flow is measured using both primary and secondary devices. These devices are explained below.

5.2.1 Primary Devices

| Primary devices are calibrated, hydraulic structures installed in the channel so flow measuren | nents can be |
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| obtained by measuring the depth of liquid at a specific point in relationship to the primary device. | Weirs and |
| flumes are examples of primary devices. | |

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The most common type of weir consists of a thin, vertical plate with a sharp crest that is placed in a stream, channel, or partially filled pipe. Figure 5-1 shows a profile of a sharp-crested weir and indicates the appropriate nomenclature. Four common types of sharp-crested weirs are shown in Figure 5-2. The crest is the upper edge of the weir to which water must rise before passing over the structure. The depth of water above the crest of the weir is termed the "head." To determine flow rate, the inspector must measure the hydraulic head. The rate of flow over a weir is directly related to the height of water (hydraulic head) above the crest. To measure the hydraulic head, a measuring device is placed upstream of the weir at a distance of at least four times the head. To approximate the head, the inspector can measure at the weir plate. However, this value will provide only a rough estimate of flow.

The flume is an artificial channel constructed so the wastestream flows through it. The wastestream's flow is proportional to the depth of water in the flume and is calculated by measuring the head. A flume is composed of three sections: (1) a converging upstream section; (2) a throat or contracted section; and (3) a diverging or dropping downstream section. The two principal types of flumes are the Parshall Flume and Palmer-Bowles Flume.

Figure 5-3 presents a sketch of the Parshall Flume, identifying its different parts and indicating capacities. In the Parshall Flume, the floor level of the converging section is higher than the floor of the throat and diverging section. The Flume operates on the principle that when water flows through a constriction in the channel, a hydraulic head is produced that is proportional to the flow. The Parshall Flume is good for measuring open channel waste flow because if is self-cleaning. Sand or suspended solids are unlikely to affect the devices's operation.

A Palmer-Bowles Flume, which may or may not have a constriction, has a level floor in the throat section and is placed in a pipe for approximately the length of the pipe's diameter. The depth of water above the raised step in the throat is related to the discharge rate. The head should be measured a distance (d/2) upstream of the throat where d is the size (width) of the flume. The height of the step is usually unknown until

| | | | |
|--------|-------------|------|------|
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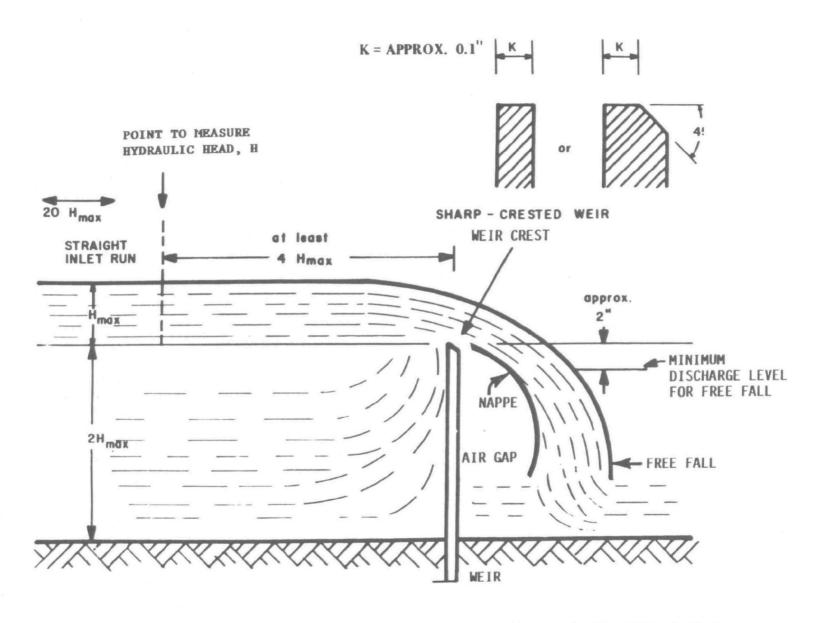
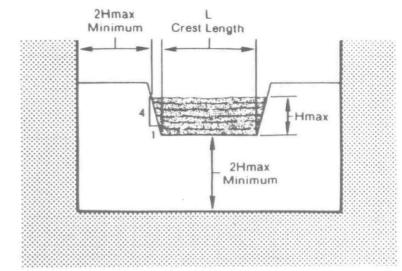
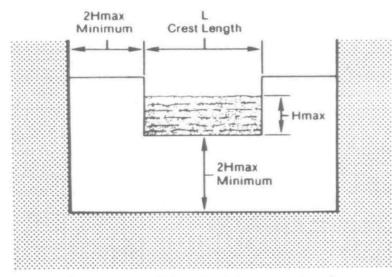


FIGURE 5-1. PROFILE AND NOMENCLATURE OF SHARP-CRESTED WEIRS

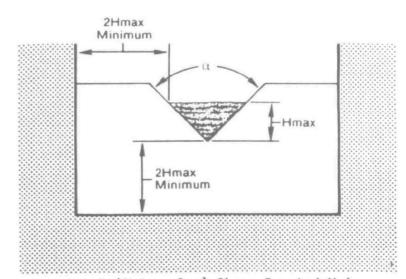
Suppressed (Without End Contractions)
Rectangular Weir



Trapezoidal (Cipolletti) Sharp-Crested Weir



Contracted (With End Contractions)
Rectangular Weir



V-Notch (Triangular) Sharp-Crested Weir

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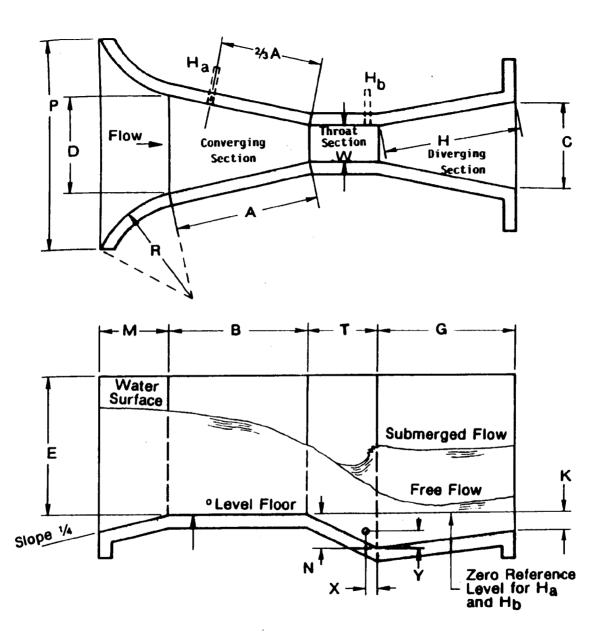


FIGURE 5-3. DIMENSIONS AND CAPACITY OF THE PARSHALL MEASURING FLUMES FOR VARIOUS THROAT WIDTHS

| ı | w | , | | λ | 2 3 | Α | | В | | c | | D | £ | | | т | | G | | H | K | | ı _ | N | | P | | R | x | Y | Capa | city |
|-----|-----|-----|------|----------------|-----|-------|----|-------------------|----|-------|-----|-----------------|------------------|-----|-----|-----|-----|-----|-----|------|-----|-----|-----|----------------|-----|-----------------|-----|-----|-----|-------|-------|-------|
| Ft | : | In. | Ft. | In. | Ft. | In. | Ft | . In. | Ft | . In. | Ft. | In. | Ft. | In. | Ft. | In. | Ft. | In. | Ft. | in. | In. | In. | Ft. | In. | Pt. | In. | Ft. | In. | In. | In. | Mini- | Max 1 |
| ٥ | | 3 | 1 | 6 3 | 1 | 14 | 1 | 6 | 0 | 7 | ٥ | 103 | $1-1\frac{1}{2}$ | 0 | 0 | 6 | 1 | ٥ | 1 | 5 32 | 1 | 0 | ٥ | $2\frac{1}{4}$ | ٥ | o | 0 | 0 | 1 | 1 1/2 | 0.03 | 1.9 |
| ۰ | 4 | 6 | 2 | $\frac{7}{16}$ | 1 | 4 2 6 | 2 | 0 | 1 | 31/2 | 1 | 3 8 | 2 | 0 | 1 | 0 | 2 | 0 | 0 | 0 | 3 | 1 | 0 | 41/2 | 2 | $11\frac{1}{2}$ | 1 | 4 | 2 | 3 | 0.05 | 3.9 |
| | | 9 | 2 | 105 | 1 | 111 | 2 | 10 | 1 | 3 | 1 | 105 | 2 | 6 | 1 | 0 | 1 | 6 | 0 | 0 | 3 | 1 | 0 | 41/2 | 3 | $6\frac{1}{2}$ | 1 | 4 | 2 | 3 | 0.09 | 8.9 |
| 1 | 1 | 0 | 4 | 6 | 3 | 0 | 4 | 47 | 2 | 0 | 2 | $9\frac{1}{4}$ | 3 | 0 | 2 | 0 | 3 | 0 | 0 | 0 | 3 | 1 | 3 | 9 | 4 | $10\frac{3}{4}$ | 1 | 8 | 2 | 3 | 0.11 | 16.1 |
| 1 | | 6 | 4 | 9 | 3 | 2 | 4 | 77 | 2 | 6 | 3 | 43 | 3 | 0 | 2 | 0 | 3 | 0 | 0 | 0 | 3 | 1 | 3 | 9 | 5 | 6 | L | 8 | 2 | 3 | 0.15 | 24.6 |
| 2 | | 0 | 5 | 0 - | 3 | 4 | 4 | 107 | 3 | 0 | 3 | $11\frac{1}{2}$ | 3 | 0 | 2 | a | 3 | ٥ | Q | 0 | 3 | 1 | 3 | 9 | 6 | 1 | ì | 8 | 2 | 3 | 0.42 | 33.1 |
| 3 | , | 0 | 5 | 6 | 3 | 8 | 5 | 43/4 | 4 | 0 | 5 | 1 7 8 | 3 | 0 | 2 | 0 | 3 | 0 | 0 | 0 | 3 | 1 | 3 | 9 | 7 | 3 1 | 1 | 8 | 2 | 3 | 0.61 | 50.4 |
| 1 | | 0 | 6 | 0 | 4 | 0 | 5 | 10 <u>5</u> | 5 | 0 | 6 | 41/4 | 3 | 0 | 2 | 0 | 3 | 0 | 0 | 0 ` | 3 | 1 | 6 | 9 | 8 | $10\frac{3}{4}$ | 2 | 0 | 2 | 3 | 1.3 | 67.9 |
| 5 | | 0 | 6 | 6 | 4 | 4 | 6 | 41/2 | 6 | 0 | 7 | 6 5 | 3 | 0 | 2 | 0 | 3 | 0 | 0 | 0 | 3 | 1 | 6 | 9 | 10 | 11/4 | 2 | 0 | 2 | 3 | 1.6 | 85.6 |
| 6 | | 0 | 7 | 0 | 4 | 8 | 6 | 10 <mark>3</mark> | 7 | 0 | | 9 | 3 | 0 | 2 | 0 | 3 | 0 | 0 | 0 | 3 | 1 | 6 | 9 | 11 | $3\frac{1}{2}$ | 2 | . 0 | 2 | 3 | 2.6 | 103.5 |
| 7 | | 0 | 7 | 6 | 5 | 0 | 7 | $4\frac{1}{4}$ | 8 | 0 | 9 | $11\frac{3}{8}$ | 3 | 0 | 2 | 0 | 3 | 0 | 0 | 0 | 3 | 1 | 6 | 9 | 12 | 6 | 2 | 0 | 2 | 3 | 3.0 | 121.4 |
| | | 0 | 8 | 0 | 5 | 4 | 7 | 101 | 9 | 0 | 11 | 13/4 | 3 | 0 | 2 | 0 | 3 | 0 | O | 0 | 3 | ı | 6 | 9 | 13 | 81 | 2 | G | 2 | 3 | 3.5 | 139.5 |
| ۱۰, | Equ | als | 1 cu | . ft | peı | sec. | | | | | | | | | | | | | | | | | | | | | | | | | | |

| I | V | Width of flume throat. | G | Axial length of diverging section. |
|---|----------------|--|---|--|
| I | ٨ | Length of side wall of converging section. | H | Length of side wall of the diverging section. |
| 1 | $\frac{2}{3}A$ | Distance back from end of crest to gage point. | K | Difference in elevation between lower end of flume and crest. |
| 1 | В | Axial length of converging section. | H | Length of approach floor. |
| | С | Width of downstream end of flume. | N | Depth of depression in throat below crest. |
| | D | Width of upstream end of flume. | P | Width between ends of curved ving walls. |
| - | E | Depth of flume. | R | Radius of curved ving vall. |
| | T | Length of flume throat. | X | Horizontal distance to H _b gage point from low point in throat. |
| | | | Y | Vertical distance to $H_{\rm b}$ gage point from low point in throat. |
| | | | | |

FIGURE 5-3. DIMENSIONS AND CAPACITY OF THE PARSHALL MEASURING FLUMES FOR VARIOUS THROAT WIDTHS (CONTINUED)

the manufacturer's data are consulted, and it is difficult to measure manually the height of water above the step at an upstream point. The dimensions of each Palmer-Bowles Flume are different. Therefore, the manufacturer's data must be consulted to establish a relationship between the head and the discharge rate.

5.2.2 Secondary Devices

Secondary devices are used in conjunction with primary devices to determine the actual flow passing the measuring point. Typically, secondary devices measure the depth of water in the primary device and convert the depth measurement to a corresponding flow, using established mathematical formulas. The output of the secondary device is generally transmitted to a recorder and/or totalizer to provide instantaneous and historical flow data to the operator. Outputs may also be transmitted to sampling systems to facilitate flow proportioning. Secondary devices can be organized into two broad classes:

- A nonrecording type with direct readout (e.g., a staff gauge) or indirect readout from fixed points (e.g., a chain, wire weight, or float)
- A recording type with either digital or graphic recorders (e.g., float in well, float in flow, bubbler, electrical, or acoustic).

5.3 CLOSED CHANNEL FLOW

Closed channel flow is normally encountered between treatment units in a wastewater treatment plant and after lift stations, where liquids and/or sludges are pumped under pressure. It is also encountered in submerged outfalls. Flow in closed channels is usually measured by a metering device inserted into the conduit. Examples of closed channel flow measuring devices are the Venturi Meter and the electromagnetic flow meter.

The Venturi Meter is one of the most accurate primary devices for measuring flow in closed channels. It is basically a pipe segment consisting of an inlet with a converging section, a throat, and a diverging outlet section, as illustrated in Figure 5-4. The water velocity is increased in the constricted portion of the inlet section which results in a decrease in static pressure. The pressure difference between the inlet pipe and the throat is proportional to the flow.

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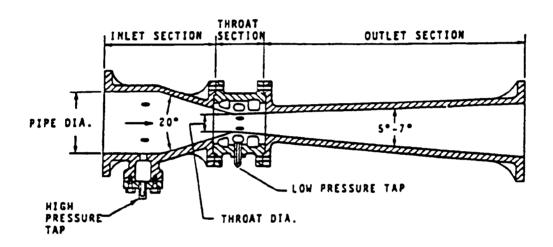


FIGURE 5-4. CONFIGURATION AND NOMENCLATURE OF VENTURIC METER

NOTES:

| at right angles through a magnethrough the field. In the case of | tic field will be pro of the electromagnet | portional to the ic flow meter, the | act that the voltage induced by a conductor moving to the velocity of that conductor as it moves eter, the conductor is the stream of water to be extromagnetic oils. A typical electromagnetic flow | | |
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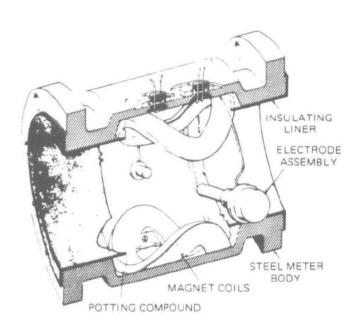


FIGURE 5-5. ELECTROMAGNETIC FLOW METER

| NOTES: | | | |
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6. QUALITY ASSURANCE/QUALITY CONTROL

Quality Assurance and quality control are tools necessary to maintain a specified level of quality in the measurement, documentation, and interpretation of sampling data. Quality Control (QC) is a set of procedures that provides precise and accurate analytical results. Quality Assurance (QA) ensures that these results are adequate for their intended purposes. QA is intended to increase confidence in the validity of reported analytical data. As an example of the distinction between quality control and quality assurance, procedures established to calibrate a piece of field equipment are QC while checking the calibration of the equipment is QA. QA checks will help the inspector determine when sample collection techniques are inadequate.

A QA program has two primary functions. First, it should continually monitor the reliability (accuracy and precision) of results reported. This function is the determination of quality. Second, QA should control quality to meet program requirements for reliability.

6.1 QUALITY CONTROL PROCEDURES FOR SAMPLING

Sampling QC begins with calibration and preventive maintenance procedures for sampling equipment. The inspector should prepare a calibration plan and documentation record for all field sampling and analysis equipment. Appendix K summarizes procedures to use for calibrating field equipment. A complete document record should be kept in a QC logbook, including equipment specifications, calibration date, calibration expiration date, and maintenance due date. Automatic samplers should be calibrated for sample quantity, line purge, and timing factor.

The quality of data resulting from sampling activities is dependent on the following major activities:

- Collecting representative samples (see Chapter 2)
- Maintaining the integrity of samples through proper handling and preservation (see Chapter 2)
- Adhering to adequate chain-of-custody and sample identification procedures (see Chapter 2).

| | · · · · · · · · · · · · · · · · · · · | |
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| NOTES: | | |

• Practicing QA techniques in the field (discussed below).

6.2 OUALITY ASSURANCE PROCEDURES FOR SAMPLING

A quality assurance program for sampling equipment and for field measurement procedures (of such parameters as temperature, DO, pH, and conductivity) is necessary to ensure data of the highest quality. A field quality assurance program should contain the following documented elements:

- Analytical methodology; special sample handling procedures; and precision, accuracy, and detection limits of all analytical methods used.
- Basis for selection of analytical and sampling methodology. For example, all analytical methodology should consist of approved procedures. Where methodology does not exist, the QA plan should state how the new method will be documented, justified, and approved for use.
- Amount of analyses for QC, expressed as a percentage of overall analyses, to assess data validity.
 Generally, the complete quality assurance program should approximate 15 percent of the overall program, with 10 and 5 percent assigned to laboratory QC and field QC, respectively. The plan should include a shifting of these allocations or a decrease in the allocations depending on the degree of confidence established for collected data.
- Procedures to calibrate and maintain field instruments and automatic samplers.
- Performance evaluation system which allows sampling personnel to cover the following areas:
 - Qualifications of personnel for a particular sampling situations
 - Determination of the best representative sampling site
 - Sampling technique, including location of the sampling points within the wastestream; the choice of grab or composite samples; the type of automatic sample; special handling procedure; sample preservation; and sample identification
 - Flow measurement, where applicable
 - Completeness of data, data recording, processing, and reporting
 - Calibration and maintenance of field instruments and equipment

| • | Use of QC samples such as duplicate, split, or spiked samples to assess the validity of data. |
|--------|---|
| NOTES: | |

• Training of all personnel involved in any function affecting data quality.

The inspector should realize the importance of implementing QA in sample collection to minimize such common errors as improper sampling methodology, poor sample preservation, and lack of adequate mixing during compositing and testing.

6.3 LABORATORY QUALITY ASSURANCE/QUALITY CONTROL

Laboratory QA/QC procedures ensure high-quality analyses through instrument calibration and the processing of control samples. Precision of laboratory findings refers to the reproducibility of results. In a laboratory QC program, a sample is analyzed independently (more than once) using the same methods and set of conditions. Precision is estimated by comparing the measurements. Accuracy refers to the degree of difference between observed values and known or actual values. The accuracy of a method may be determined by analyses of samples to which known amounts of reference standards have added.

Four specific QA procedures can be used to increase confidence in the validity of the reported analytical data: duplicate, blank, split, and spiked samples. They are described below.

- <u>Duplicate Samples</u> Separate samples taken at the same time and location using duplicate equipment or one sample pulled and separated into two aliquots for duplicate analysis at the same laboratory. Duplicate samples check for precision. These samples provide a check on sampling equipment and sampling techniques. They also indicate the representativeness of the sampling location.
- <u>Blank Samples</u> Check the contamination of chemical preservatives. A specified quantity of preservative (equal to that ordinarily added to a wastewater sample) is added to a sample of deionized water. After laboratory analysis, the value for the blank is subtracted from the sample value to obtain the actual value. In the case of automatic sampling, the deionized water must be run through the sampler prior to sample collection and then the appropriate preservative is added.
- Split Samples Allow the comparison of analytical techniques and procedures from separate laboratories.
 Samples are divided into two, or preferably three, segments for analysis in separate laboratories.
 Sampling personnel should exercise caution when splitting samples to avoid producing

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| NOTES: | | | | | | |
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| large differences in TSS. | All large discrepancies in results should be investigated and the cause |
|---------------------------|---|
| identified. | |

• <u>Spiked Samples</u> - Provide a proficiency check for the accuracy of analytical procedures. Known amounts of a particular constituent should be added to an actual sample or blanks of deionized water at concentrations where the accuracy of the test method is satisfactory. The amount added should be coordinated with the laboratory.

It is a good practice if each group of samples (or testing batch) contains at least one blank, one standard duplicate, and one spiked sample (as applicable). When a batch contains more than 10 samples, every tenth sample should be followed by a duplicate and a spike (as applicable).

NOTES:

7. SUMMARY

This module discussed the procedures and protocol used during sampling of a permittee's effluent or during observation of a permittee's self-monitoring procedures. The need for a sampling plan and for coordination with the laboratory performing analyses were stressed in order to promote consistency between inspectors gathering samples and to ensure that prerequisite laboratory requirements are met during all sampling events. The module also emphasizes the importance of using proper sample collection techniques, including the selection of an appropriate sample location and sample type, the preparation of sample containers, and the preservation, labeling, and handling of samples after collection in order to establish the validity of each sample should violations be identified that lead to enforcement actions.

The module further explained several instances in which special sampling requirements must be followed. It discussed methods used for onsite analysis of samples for pollutants that cannot be preserved and mentioned some of the concerns involved with such analyses. Finally, this module described various chain-of-custody and quality assurance procedures that should be practiced during all sampling events to ensure the accuracy, integrity, and reliability of each sample and of the corresponding analytical results. Inspectors must conduct all sampling activities on the premise that each may lead to an enforcement action.

APPENDIX A GLOSSARY

GLOSSARY

- Aliquot Portion of a sample.
- Biochemical Oxygen Demand (BOD) Amount of oxygen used in the biochemical oxidation of organic matter in a specified time, at a specified temperature, and under specific conditions. Standard test for measuring the organic strength of a wastestream.
- Blank sample Samples of deionized water with a known quantity of preservative added.
- Chain-of-custody Written record of the possession and handling of the sample, from collection through laboratory analysis, disposition of the analytical results, and disposal of the unused sample remnants.
- Composite sample Sample composed of two or more discrete samples. The aggregate sample will reflect the average water quality over the compositing or sample period.
- Confined space Space having limited means of entry or exit and subject to the accumulation of toxic or combustible gases or to a deficiency of oxygen.
- Duplicate sample Separate samples taken from the same source at the same time for analysis using identical analytical techniques by the same person.
- Grab sample Sample collected over a time period not exceeding 15 minutes. A single sample that represents the characteristics of the wastestream only at the specific time and location of collection.
- Instantaneous measurement In-situ or grab measurement for such parameters as DO, pH, temperature, and specific conductance.
- Priority pollutants A list of 126 pollutants, established by EPA, considered hazardous to the environment and to humans.
- Quality Assurance (QA) Refers to a management/administrative check on procedures and practices used during sampling and analysis that ensure the accuracy, precision, reproducibility, and representativeness of reported data.
- Quality Control (QC) Routine application of procedures to control the accuracy and precision of the sampling and analytical measurement process (as a function of quality assurance). QC of sampling procedures should include the use of duplicate, spiked, and/or split samples and sample blanks. QC of analytical procedures should include proper calibration of instruments and the use of appropriate analytical procedures.
- Spiked sample Effluent or blank sample to which a known quantity of substance has been added.
- Split sample Sample that has been divided into two or more containers for analysis by different analysts or laboratories.
- Supernatant A substance floating above or on the surface of another substance.
- Turbidity Condition in a wastestream caused by the presence of suspended matter resulting in the scattering and absorption of lightrays.

APPENDIX B REFERENCES

REFERENCES

- American Public Health Association, American Water Works Association, and Water Pollution Control Federation. Standard Methods for the Examination of Water and Wastewater. Use the most current, EPA-approved edition.
- Federal Water Pollution Control Act. 33 USC 1251 et seq., as amended by the Water Quality Act of 1987. P.L. 100-4, February 4, 1987.
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- U.S. Environmental Protection Agency. 1979. Methods for Chemical Analysis of Water and Wastes. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio. EPA-600/4-79-020.
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APPENDIX C

REVIEW QUESTIONS AND ANSWERS ON NPDES SAMPLING PROCEDURES

REVIEW QUESTIONS ON NPDES SAMPLING PROCEDURES

- 1. List at least three objectives of a sampling program.
- 2. Describe the appropriate conditions for taking a grab sample.
- 3. Describe the appropriate conditions for taking a composite sample.
- 4. Grab samples should be taken for which parameters?
- 5. List the advantages and disadvantages of using an automatic sampler.
- 6. Describe a "representative" sample location.
- 7. Where should the sample container be held when taking a bacteriological sample and why?
- 8. Oil and grease samples should be taken in what type of container?
- 9. List three sample preservation techniques.
- 10. List three factors to consider when selecting automatic sampling equipment.
- 11. What type of sample container is used for volatile organics and how is the container filled?
- 12. What is the purpose of chain-of-custody procedures?
- 13. List specific QA procedures for sampling activities.
- 14. What is the purpose of QA?

ANSWERS TO REVIEW QUESTIONS ON NPDES SAMPLING PROCEDURES

- 1. List at least three objective of a sampling program.
 - To verify compliance with effluent limitations
 - To verify self-monitoring data
 - To verify that parameters specified in the permit are consistent with wastewater characteristics
 - To support permit reissuance and revision
 - To provide basis for enforcement procedures.

[Section 1.3]

- 2. Describe the appropriate conditions for taking a grab sample.
 - Batch dischargers
 - · Constant waste conditions over a period of discharge
 - Extreme conditions exist
 - Wastestream is adequately mixed.

[Section 2.7]

- 3. Describe the appropriate conditions for taking a composite sample.
 - To determine average pollutant concentration. [Section 2.7]
- 4. Grab samples should be taken from which parameters?
 - pH
 - Temperature
 - · Oil and grease
 - Residual chlorine
 - Soluble sulfides
 - DO
 - Cyanide
 - Volatile organics.

[Section 2.7]

- 5. List the advantages and disadvantages of using an automatic sampler.
 - Advantages
 - More practical for sampling a large number of locations
 - Reduces human error in complex sampling activities
 - Reduces exposure to potentially hazardous environments
 - Requires less labor.

- Disadvantages
 - Cost of equipment
 - Maintenance requirements.

[Section 2.7]

- 6. Describe a "representative" sample location.
 - Wastestream must be adequately mixed and sample should be taken in the center of the flow, a few inches below the surface.

[Section 2.5]

- 7. Where should the sample container be held when taking a bacteriological sample and why?
 - . At the lower part of the bottle with the mouth of the bottle facing the direction of the current.
 - To avoid contamination.

[Section 2.14]

- 8. Oil and grease samples should be taken in what type of container?
 - Glass container with a teflon insert in the lid. [Section 2.14]
- 9. List three sample preservation techniques.
 - Refrigeration (metals pH < 2)
 - pH adjustment (cyanide pH > 12)
 - Chemical neutralization (tecal coliform 0.008 % Na₂S₂O₃)

[Section 2.10]

- 10. List three factors to consider when selecting automatic sampling equipment.
 - Convenience in installation and maintenance
 - Equipment security
 - · Cold weather operation.

[Chapter 4]

- 11. What type of sample container is used for volatile organics and how is the container filled?
 - 40 ml glass bottle with a teflon coated septum seal. The bottle must be filled to the top without any air bubbles.

[Section 2.14]

- 12. What is the purpose of chain-of-custody procedures?
 - To provide an accurate written record that tracks the possession of a sample from origin through analysis.

[Chapter 7]

- 13. List specific QA procedures for sampling activities.
 - Split samples
 - Blank samples
 - Duplicate samples
 - Calibration plan for sampling equipment. [Section 6.2]
- 14. What is the purpose of a QA program?
 - To ensure the integrity of a sample. [Chapter 6]

APPENDIX D

VOLUME OF SAMPLE REQUIRED FOR DETERMINATION OF THE VARIOUS CONSTITUENTS OF INDUSTRIAL WASTEWATER

Volume of Sample Required for Determination of the Various Constituents of Industrial Wastewater

(U.S. Environmental Protection Agency 1973. Handbook for Monitoring Industrial Wastewater. Technology Transfer.)

| Tests | Volume of Sample 1, in m |
|--|--|
| PHYSICAL | |
| Color and odor (2) | . following sample . 100 . 100 . 100 to 1,000 |
| Temperature (2) Toxicity (2) Turbidity (2) | . following sample . 1,000 to 20,000 |
| CHEMICAL | |
| Dissolved Gases | |
| Ammonia (3) Carbon dioxide (3), free CO ₂ Chlorine (3), free C1 ₂ Hydrogen (3), H ₂ Hydrogen sulfide (3), H ₂ S Oxygen (3), O ₂ Sulfer dioxide (3), free S ₂ | 200 200 1,000 500 500 to 1,000 |
| Miscellaneous | |
| Acidity and alkalinity Bacteria, iron Bacteria, sulfate-reducing Biochemical Oxygen Demand (BOD) Carbon dioxide, total CO ₂ (including CO ₃ , HCO ³ , and free) Chemical Oxygen Demand (COD) (dichromate) Chlorine requirement Chlorine, total residual C1 ₂ (including OC1 ³ , HOC ³ , NH ₂ C1, NHC1 ₂ , and free) | . 500 . 100 . 100 to 500 . 200 . 50 to 100 . 2,000 to 4,000 |

 $\mathbf{m}\mathbf{l}$

Volume of Sample Required for Determination of the Various Constituents of Industrial Wastewater

(U.S. Environmental Protection Agency 1973.

Handbook for Monitoring Industrial Wastewater.

Technology Transfer.)

| Tests | Volume of Sample ¹ , in |
|--|------------------------------------|
| Miscellaneous | |
| Chloroform, extractable matter | |
| Detergents | |
| Hydrazine | |
| Microorganisms | |
| Volatile and filming amines | |
| Oily matter | , |
| Organic nitrogen | |
| Phenolic compounds | |
| pH, colorimetric | |
| Polyphosphates | |
| Silica | |
| Solids, dissolved | |
| Solids, suspended | |
| Tannin and lignin | |
| Cations: | |
| Aluminum, A1 ⁺³ | . 100 to 1,000 |
| Ammonium (2), NH ₄ ⁺ | . 500 |
| Antimony, Sb ⁺³ to Sb ⁺³ | |
| Arsenic, As ⁺³ to As ⁺⁵ | . 100 to 1,000 |
| Barium, Ba ⁺² | |
| Cadmium, Cd ⁺² | • |
| Calcium, Cd ⁺² | . 100 to 1,000 |
| Chromium, Cr ⁺³ to Cr ⁺⁶ | |
| Copper, Cu ⁺² | . 200 to 4,000 |
| Iron (3), Fe^{+2} and F^{+3} | |
| Lead, Pb ⁺² | . 100 to 4,000 |
| Magnesium, Mg ⁺² | . 100 to 4,000 |
| Manganese, Mn ⁻ to Mn ⁻ | . 100 to 1,000 |
| Mercury, Ng ⁺ and Hg ⁺² | |
| Potassium, K ⁺ | |
| Nickel, Ni ⁺² | . 100 to 1,000 |

Volume of Sample Required for Determination of the Various Constituents of Industrial Wastewater

(U.S. Environmental Protection Agency 1973.

<u>Handbook for Monitoring Industrial Wastewater.</u>

Technology Transfer.)

| Tests | Volume of Sample ¹ , in a |
|---|--------------------------------------|
| Cations: | |
| Silver, Ag ⁺ | . 100 to 1,000 |
| Sodium, Na ⁺ | |
| Strontium, Sr ⁺² | |
| Tin, Sn ⁺² and Sn ⁺⁴ | |
| Zinc, Zn ⁺² | . 100 to 1,000 |
| Anions: | |
| Bicarbonate, HCO ₃ | . 100 to 200 |
| Bromide, Br | |
| Carbonate, CO ₃ ² | . 100 to 200 |
| Chloride, Cl | |
| Cyanide, CN | 25 to 100 |
| Fluoride, F1 | |
| Hydroxide, OH | |
| Iodide, I | |
| Nitrate, NO ₃ ⁻² | |
| Nitrite, NO ₂ | |
| Phosphate, ortho, PO ₄ , HPO ₄ , HPO ₄ , | |
| Sulfate, SO ₄ ² , HSO ₄ | |
| Sulfide, S ⁻² , HS | |
| Sulfite, SO, 2, HSO, | 50 to 100 |

⁽¹⁾ Volumes specified in this table should be considered as guides for the approximate quantity of sample necessary for a particular analysis. The exact quantity used should be consistent with the volume prescribed in the standard method of analysis, whenever a volume is specified.

(2) Aliquot may be used for other determinations.

⁽³⁾ Samples for unstable constituents must be obtained in separate containers, preserved as prescribed, completely filled, and sealed against all exposure.

APPENDIX E

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, HOLDING TIMES, AND TEST METHODS (EXCERPTED FROM 40 CFR PART 136)

NPDES Compliance Monitoring Inspector Training: SAMPLING

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES Source: 49 FR 43260 Friday October 26, 1984 40 CFR Part 136

| Container (1) | Preservative ^{(2),(3)} | Maximum Holding Time ⁽⁴⁾ |
|---------------|--|--|
| | | |
| P,G | Cool, 4°C 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ | 6 hours |
| P,G | Cool, 4°C 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ | 6 hours |
| | | |
| P,G | Cool, 4°C | 14 days |
| P,G | Cool, 4°C | 14 days |
| P,G | Cool, 4°C H ₂ SO ₄ to pH<2 | 28 days |
| P,G | Cool, 4°C | 48 hours |
| P,G | Cool, 4°C | 48 hours |
| P,G | None required | 28 days |
| P,G | Cool, 4°C H ₂ SO ₄ to pH<2 | 28 days |
| P,G | None required | 28 days |
| P,G | None required | Analyze immediately |
| | P,G P,G P,G P,G P,G P,G P,G P,G P,G | P,G |

NPDES Compliance Monitoring Inspector Training: SAMPLING

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES (Continued) Source: 49 FR 43260 Friday October 26, 1984 40 CFR Part 136

| | | | Maximum |
|---|--------------------------|---|---------------------|
| Parameter | Container ⁽¹⁾ | Preservative (2),(3) | Holding Time (4) |
| Color | P,G | Cool, 4°C | 48 hours |
| Cyanide, total and amenable to chlorination | P,G | Cool 4°C | 14 days (6) |
| to emormation | | NaOH to pHD12 0.6 g ascorbic acid ⁽ | 5) |
| Fluoride | P | None Required | 28 days |
| Hardness | P,G | HNO ₃ to pH<2 | 6 months |
| Hydrogen ion (pH) | P,G | None required | Analyze immediately |
| Kjeldahl and organic nitrogen | P,G | Cool, 4°C H ₂ SO ₄ to pH<2 | 28 days |
| METALS ⁽⁷⁾ | | | |
| Chromium VI | P,G | Cool, 4°C | 24 hours |
| Mercury | P,G | HNO_3 to $pH<2$ | 28 days |
| Metals except above | P,G | HNO ₃ to pH<2 | 6 months |
| Nitrate* | P,G | Cool, 4°C | 48 hours |
| Nitrate-nitrite | P,G | Cool, 4° C H_2SO_4 to pH<2 | 28 days |
| Nitrite | P,G | Cool, 4℃ | 48 hours |

NPDES Compliance Monitoring Inspector Training: SAMPLING

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES (Continued) Source: 49 FR 43260 Friday October 26, 1984 40 CFR Part 136

| | o (1) | (2).(3) | Maximum (4) |
|------------------------------|--------------------------|---|---------------------|
| Parameter | Container ⁽¹⁾ | Preservative ^{(2),(3)} | Holding Time (4) |
| Oil and grease | G | Cool, 4°C H ₂ SO ₄ to pH<2 | 28 days |
| Organic Carbon | P,G | Cool, 4°C H ₂ SO ₄ to pH<2 | 28 days |
| Orthophosphate | P,G | Filter immediately Cool, 4°C | 48 hours |
| Dissolved oxygen (probe) | G bottle & top | None required | Analyze immediately |
| Phenols | G | Cool, 4°C H ₂ SO ₄ to pH<2 | 28 days |
| Phosphorus (elemental) | G | Cool, 4°C | 48 hours |
| Phosphorus, total | P,G | Cool, 4°C H ₂ SO ₄ to pH<2 | 28 days |
| Residue, total | P,G | Cool 4°C | 7 days |
| Residue, filterable | P,G | Cool 4°C | 7 days |
| Residue, nonfilterable (TSS) | P,G | Cool 4°C | 7 days |
| Residue, settleable | P,G | Cool 4°C | 48 hours |
| Residue, volatile | P,G | Cool 4°C | 7 days |
| | | | |

NPDES Compliance Monitoring Inspector Training: SAMPLING

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES (Continued) Source: 49 FR 43260 Friday October 26, 1984 40 CFR Part 136

| Parameter | Container (1) | Preservative ^{(2),(3)} | Maximum Holding Time ⁽⁴⁾ |
|----------------------------------|---------------------------|--|--|
| Silica | P | Cool 4°C | 28 days |
| Specific conductance | P,G | Cool 4°C | 28 days |
| Sulfate | P,G | Cool 4°C | 28 days |
| Sulfide | P,G | Cool 4°C add Zinc Acetate and sodium hydroxide to pHD9 | 7 days |
| Sulfite | P,G | None required | Analyze immediately |
| Surfactants | P,G | Cool 4°C | 48 hours |
| Temperature | P,G | None required | Analyze imudiately |
| Turbidity | P,G | Cool, 4°C | 48 hours |
| ORCANIC TESTS (8) | | | |
| Purgeables Halocarbons | G, teflon-lined septum | Cool 4°C 0.008% Na ₂ S ₂ O ₃ (5) | 14 days |
| Purgeable Aromatics Hydrocarbons | G, teflon-lined septum | Cool 4°C 0.008% Na ₂ S ₂ O ₃ (5) | 14 days |
| | | HCl to pH 2 ⁽⁹⁾ | |

NPDES Compliance Monitoring Inspector Training: SAMPLING

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES (Continued) Source: 49 FR 43260 Friday October 26, 1984 40 CFR Part 136

| Parameter | Container ⁽¹⁾ | Preservative ^{(2),(3)} | Maximum Holding Time ⁽⁴⁾ |
|--|--------------------------|--|---|
| Acrolein and Acrylonitrite | G, teflon-lined septum | Cool 4°C 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ Adjust pH to 4.5 ⁽¹⁰⁾ | 14 days |
| Extractables (phenols) | G, teflon-lined cap | Cool, 4°C H ₂ SO ₄ to pH<2 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ | 7 days (until extraction) 30 days (after extraction) |
| Benzidenes (11) | G, teflon-lined cap | Cool, 4°C 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ | 7 days until extraction (13) |
| Phthalate esters (11) | G, teflon-lined cap | Cool, 4°C | 7 days until extraction, 40 days after extraction |
| Nitrosamines (11)(14) | G, teflon-lined cap | Cool, 4°C store in dark 0.008% Na ₂ S ₂ O ₃ | 7 days until extraction, 40 days after extraction(12) |
| PCBs ⁽¹¹⁾ Acrylonitrate | G, teflon-lined cap | c∞l, 4℃ | 7 days until extraction, 40 days after extraction |
| Nitroaromatics and isophorone (11) | G, teflon-lined cap | Cool, 4°C 0.008% Na ₂ S ₂ O ₃ store in the dark | 7 days until extraction, 40 days after extraction |
| Polynuclear aromatic hydrocarbons (11) | G, teflon-lined cap | Cool, 4°C 0.008% Na ₂ S ₂ O ₃ (5) store in the dark | 7 days until extraction, 40 days after extraction |

| Parameter | Container ⁽¹⁾ | Preservative ^{(2),(3)} | Maximum Holding Time ⁽⁴⁾ |
|-------------------------------|--------------------------|--|--|
| Haloethers (11) | G, teflon-lined cap | Cool, 4°C 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ | 7 days until extraction, 40 days after extraction |
| Chlorinated hydrocarbons (11) | G, teflon-lined cap | Cool, 4°C | 7 days until extraction, 40 days after extraction (12) |
| TCDD | G, teflon-lined cap | Cool, 4°C 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ | 7 days until extraction, 40 days after extraction |
| PESTICIDES TEST | | | |
| Pesticides ⁽¹¹⁾ | G, teflon-lined cap | Cool, 4°C pH 5-9 ⁽¹⁵⁾ | 7 days until extraction, 40 days after extraction |
| RADIOLOGICAL TEST | | | |
| Alpha, beta, and radium | P,G | HNO ₃ to pH<2 | 6 months |

⁽¹⁾ Polyethylene (P) or Glass (G).

⁽²⁾ Sample preservation should be performed immediately upon sample collection. For composite samples each aliquot should be preserved at the time of collection. When use of an automatic sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES (Continued) Source: 49 FR 43260 Friday October 26, 1984 40 CFR Part 136

- When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For preservation requirements, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not comply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.80% by weight or less (pH about 12.30 or less).
- (4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample stability.
- (5) Should only be used in the presence of residual chlorine. Add ascorbic acid, a few crystals at a time, until no chlorine remains. Then add 0.6 more grams of ascorbic acid for each liter of sample.
- (6) Maximum holding time is 24 hours when sulfide is present.
- (7) Samples should be filtered immediately onsite before adding preservative for dissolved metals.
- (8) Quidance applies to samples to be analyzed by GC, LC, or GC/MS for specific organic compounds.
- (9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
- (10) Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling. Optionally, all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered, then NaOH is added to pH 12.
- When the extractable analytes of concern fall within a single chemical category, the specified preservation and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote (5) (re: the requirement for thiosulfate reduction of residue chlorine) and footnotes (12) and (13) (re: the analysis of benzidine).

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES (Continued) Source: 49 FR 43260 Friday October 26, 1984 40 CFR Part 136

Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

⁽¹³⁾ If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% Na_2SO_3 .

APPENDIX F

EPA ORDER 1440.2

HEALTH AND SAFETY REQUIREMENTS FOR EMPLOYEES ENGAGED IN FIELD ACTIVITIES

ENVIRONMENTAL PROTECTION AGENCY

ORDER

1440.2

July 12, 1981

PROTECTIVE SERVICES - SAFETY

HEALTH AND SAFETY REQUIREMENTS FOR EMPLOYEES ENGAGED IN FIELD ACTIVITIES

1. PURPOSE. This Order establishes policy, responsibilities, and mandatory requirements for occupational health and safety training and certification, and occupational medical monitoring of Agency employees engaged in field activities.

2. DEFINITIONS.

- a. The term "field activities" as used in this Order means EPA program activities that are conducted by EPA employees outside of EPA administered facilities. These activities include environmental and pesticides sampling, inspection of water and wastewater treatment plants, and hazardous material spills and waste site investigations, inspections, and sampling.
- b. The term "health and safety training" means scheduled, formal or informal training courses, approved and sponsored by EPA and conducted by EPA or its contracted agents which is designed to develop, improve and upgrade the health and safety knowledge of EPA employees involved in field activities.
- c. The term "occupational medical monitoring" means surveillance over the health status of employees by means of periodic medical examinations or screening in accordance with the Agency's Occupational Medical Monitoring guidelines.
- d. The term "certification" as used in this Order means that the employee has successfully completed the minimum classroom and field training requirements for the specified level of training and the Agency has issued a certificate attesting that the employee met these requirements.

ORDER

1440.2

July 12, 1981

3. REFERENCES.

- a. 29 CFR 1910, Parts 16, 94, 96, 106, 109, 111, 134, 151, 1000, Occupational Health and Safety Standards.
- b. Executive Order 12196, Section 1-201, Sec. (k), Occupational Health and Safety Programs for Federal Employees.
- c. 29 CFR 1960.59(a), Occupational Safety and Health for the Federal Employee.
 - d. EPA Occupational Health and Safety Manual, Chapter 7(1).
 - e. EPA Training and Development Manual, Chapter 3, Par 7(b).
- f. Occupational Health and Safety Act of 1971, P.L. 91-596, Sec. 6.
 - g. EPA Order on Respiratory Protection (Proposed).
 - h. 49 CFR, Parts 100-177, Transportation of Hazardous Materials.
 - i. EPA Cider 1000.18, Transportation of Hazardous Materials.
- j. EPA Order 3100.1, Change 1, Uniforms, Protective Clothing, and Protective Equipment.
- 4. BACKGROUND. Field activities are a critical part of most EPA programs. These activities range from routine environmental reconnaisance sampling, inspections, and monitoring, to entering and working in environments with known and unknown hazards. Since protection cannot be engineered into the field working situation, the protection of personnel engaged in field activities involves training employees in safe operational procedures and the proper use of appropriate personal protective clothing and equipment.
- 5. APPLICABILITY. This Order applies to all EPA organizational units which have employees engaged in field activities.
- 6. <u>POLICY</u>. It is the policy of the Environmental Protection Agency to carry out its field activities in a manner that assures the protection of its employees.

| ORDER | 1440.2 |
|-------|---------------|
| | July 12, 1981 |

7. RESPONSIBILITIES.

- a. Assistant Administrators, Regional Administrators, Deputy
 Assistant Administrators, Laboratory Directors, and Division

 Directors. These officials are responsible within their jurisdictions for implementing the provisions of this Order and for budgeting the necessary funds for employee training and certification, personal protective clothing and equipment, and occupational medical monitoring programs.
- b. <u>Supervisors</u>. Supervisors are responsible for complying with the requirements of this Order for employee training and certification, and occupational medical monitoring programs. They will identify those employees who require training and certification, and occupational medical monitoring, and assure they receive it to comply with the provisions of this Order and will insure these requirements are properly contained in position descriptions and job postings.
- c. Employees. Employees are responsible for making known upon request from their supervisors the extent of their individual occupational health and safety training and the history of their occupational medical monitoring participation. Employees should notify their supervisor of any hazardous work situation and make suggestions for corrective measures. Employees are responsible for applying the knowledge, skills, and techniques acquired through training in a manner that will help assure their health and safety and that of fellow workers.
- d. Occupational Health and Safety Designees. The Occupational Health and Safety Designees are responsible for identifying program areas that require training and certification, and occupational medical monitoring; recommending or providing training and certification resources to meet the requirements of this Order; and maintaining records of persons receiving training and certification.
- e. Office of Occupational Health and Safety. The Director, Office of Occupational Health and Safety is responsible for establishing policy and requirements for adequate training and certification programs for field activities, developing and maintaining an occupational medical monitoring program, approving health and safety training and certification programs for employees involved in field activities, and for evaluating the results of these training and certification programs.

ORDER 1440.2
July 12, 1981

8. CSJECTIVES.

- a. Training and Certification. The objectives of the health and safety training and certification programs for employees involved in field activities are:
- (1) To assure that EPA employees are aware of the potential hazards they may encounter during the performance of field activities;
- (2) To provide the knowledge and skills necessary to perform the work with the least possible risk to personal health and safety;
- (3) To assure that Agency program goals are accomplished in as safe and healthful manner as feasible; and
- (4) To assure that EPA employees can safely disengage themselves from an actual hazardous situation which may occur during a field activity.
- b. (coupational Medical Monitoring. The objectives of the occupation: Medical Monitoring program are:
- (i) To detect any adverse effects of occupational exposure on the enloyees health and to initiate prompt corrective actions when inducated; and
- (2) To assure that employees assigned arduous or physically taxing jobs or jobs requiring unique skills are able to perform those jobs without impairing their health and safety or the health and safety of civers.
- 9. TRAINING AND CERTIFICATION REQUIREMENTS. Employees shall not be permitted to engage in routine field activities until they have been trained and certified to a level commensurate with the degree of anticipanta hazards.
- a. <u>Basic Level</u>. All employees shall be provided a minimum of 24 hours of health and safety training prior to their becoming involved in normal, routine field activities. The training shall include but not be limited to classroom instruction in all the following subject areas:

| ORDER | 1440.2 |
|-------|---------------|
| | July 12, 1981 |

- (1) Employee Rights and Responsibilities;
- (2) Nature of Anticipated Hazards:
- (3) Emergency Help and Self-Rescue;
- (4) Vehicles Mandatory Rules and Regulations;
- (5) Safe Use of Field Equipment:
- (6) Use, Handling, Storage, and Transportation of Hazardous Materials:
 - (7) Personal Protective Equipment and Clothing, Use and Care:
- and
- (8) Safe Sampling Techniques.

In addition to classroom instruction, the employee shall accompany an employee experienced in field activities and perform actual field tasks for a minimum of three days within a period of three months after classroom instruction. Employees satisfactorily completing these requirements will receive certification at the Basic Level of training from the Occupational Health and Safety Designee at the Reporting Unit.

- b. Intermediate Level. All inexperienced employees who are to work with experienced employees in uncontrolled hazardous waste and hazardous spill sites investigations or employees engaged in other activities which at a later date are determined by the Director, Office of Occupational Health and Safety, to present unique hazards requiring additional training, shall be provided a minimum of 8 hours of additional health and safety training. This training shall include (in addition to the Basic Level requirements) but not be limited to the following subject matter:
 - (1) Site surveillance, observation, and safety plan development;
- (2) Use and decontamination of totally enclosed protective clothing and equipment;
- (3) Use of field test equipment for radioactivity, explosivity, and other measurements; and
 - (4) Topics specific to other identified activities.

In addition to classroom instruction, the employee shall accompany another employee experienced in hazardous waste and spill site investigations and/or.cleanup operations and perform actual field tasks for a minimum of three days within a period of three months after classroom instruction. The employee should also be able to provide on-the-job training and instructions to inexperienced employees during normal, routine field activities (as required above). Employees satisfactorily completing these requirements will be certified at the Intermediate Level by the Occupational Health and Safety Designee at the Reporting Unit.

ORDER

1440.2 July 12, 1981

- c. Advanced Level. All employees who manage uncontrolled hazardous waste site and spill site monitoring, sampling, investigations, and cleanup operations shall be provided a minimum of 8 hours additional health and safety training. The classroom training shall include but not be limited to (in addition to the Basic and Intermediate Level requirements), instruction in the following subject areas:
 - (1) Management of restricted and safe zones;
 - (2) Rules of Handling the Press and VIP's; and
 - (3) Safe Use of Specialized Sampling Equipment.

In addition to classroom instruction, the employee shall accompany another employee with experience in managing hazardous waste and spill site investigations or cleanup operations and perform actual field tasks for a minimum of three days within a three month period after receiving classroom instruction. After satisfactorily completing these requirements, employees will receive Advanced Level certification from the Occupational Health and Safety Designee at the Reporting Unit.

d. General.

- (1) An employee may receive certification at the next higher level by completing only the additional training requirements if certified at the next lower level within the previous one-year period.
- (2) The Director, Office of Occupation! Health and Safety, may certify employees based on an evaluation of previous training, education, and experience. Recommendations for this type certification should be made to the Director by the Occupational Health and Safety Designee at the Reporting Unit.
- 10. FREQUENCY OF TRAINING. Employees at the Basic, Intermediate, and Advanced Levels shall complete a minimum of 8 hours of refresher classroom instruction annually consisting of a review of all subject areas to maintain their certification. In addition to the classroom instruction, employees shall have demonstrated by having performed actual field tasks that they have sufficient practical experience to perform their assigned duties in a safe and healthful manner.

11. RECORD OF TRAINING.

a. A record of the level of training and certification shall be maintained in the employee's official personnel file.

ORDER

1440.2

July 12, 1981

- b. The Occupational Health and Safety Designee shall maintain a roster of employee training and certification so that a schedule of annual training can be established.
- c. The Occupational Health and Safety Designee shall issue a certificate to the employee showing the level of training and certification.
- 12. OCCUPATIONAL MEDICAL MONITORING REQUIREMENTS. All employees routinely engaged in field activities which present the probability of exposure to hazardous or toxic substances, which are arduous or physically taxing, or which require the use of respiratory protective equipment shall be included in the Agency's Occupational Medical Monitoring Program. Employees should not be permitted to engage in field activities unless they have undergone a baseline medical examination (as defined in the Agency's Occupational Medical Monitoring Guidelines), which will show physical fitness and provide a base to measure any adverse effects their activities may have on these individuals.
- 13. SAVINGS PROVISION. Changes in the Act, Executive Order, or EPA and OSHA stundards and guidelines which occur after the effective date of this Order will automatically come under the purview of this Order on the effective date of the change.

Full implementation of this Order shall be within one year of its effective date.

Edward J. Hanlev

Director, Office of Management Information and Support Services

APPENDIX G LIST OF FIELD SAMPLING EQUIPMENT

LIST OF FIELD SAMPLING EQUIPMENT

- Tools
 - Multi-Tooled Jack Knife (Swiss Army Type)
 - Electrical and Duct Tape
 - Tape Measure
 - Handheld Range Finder and Level
 - Camera/Film
 - Flashlight
 - Screwdriver
 - Adjustable and Vise Grips Wrench
 - Pliers
 - Plastic Bucket
 - Nylon Cord
 - Field Notebook with Waterproof Paper
- Samplers
 - Tubing
 - Sample Bottles
 - Batteries
 - Desiccant
- Flow Measurement Devices
- Meters
 - pH Buffer
 - Chart Paper
- Sample Containers
- Coolers/Ice
- Preservatives
- Transportation Materials
 - Bubblepack Material
 - Filament Tape
 - Shipping Labels
 - Chain-of-Custody Forms
 - Water Resistent Marker/Pen
 - Analysis Request Forms

• Protective Clothing

- Hard Hat
- Safety Shoes
- Gloves
- Coveralls
- Reflective Safety Vest
- Safety Glasses/Goggles
- Rain Wear

• Safety Equipment

- First-Aid Kit
- Safety Harness and Retrieval System
- Ventilation Equipment
- Meters (Oxygen Content, Explosivity, and Toxic Gas)
- Respirator
- Self-contained Breathing Apparatus (If Appropriate)

APPENDIX H SAMPLE IDENTIFICATION LABELS

EXAMPLE SAMPLE TAG

OFFICE OF ENFORCEMENT NATIONAL ENFORCEMENT INVESTIGATIONS CENTER BUILDING 53, BOX 25227, DENVER FEDERAL CENTER DENVER, COLORADO 80025





| Projec | ct Code | Station | No. | | | N | /o/[| Day/ | Yea | 7 | | Tin | ne | | | | Desig | nate: | |
|--------------|------------|---------|--------------|--------------|------------|------------|------------|----------|-----------|---------|--------|---------|-----------|---------------|-----------------|--------|-----------|------------|------|
| | | | | | | | | | | | | | | | | | Comp |). | Grab |
| Statio | n Location | | | | | | | S | amp | olers | : (S | igna | ature |) | | | | | |
| # P | Remarks: | | Bacteriology | Mutagenicity | Pesticides | Volatile (| Priority F | Organics | Oil and C | Cyanide | Metals | Mercury | Phenolics | COD, TOC | Solids (T | вор | Analyses: | ((| |
| Lab Sample # | | | ogy | icity | is . | Organics | Pollutants | GC/MS | Grease | | | | S | XC, Nutrients | TSS) (TDS) (SS) | Anions | Ses: | Tes: No: L | ڲۣٚ |

APPENDIX I

EXAMPLE RECORD OF FIELD SAMPLE DATA AND CHAIN-OF-CUSTODY RECORD

Distribution: Original Accompanies Shipment: First Copy to Coordinator Field Files: Second Copy to Representative of Inspected Facility

NPDES Compliance Monitoring Inspector Training: SAMPLING

Accepted Declined

REGION VIII. ONE DENVER PLACE

APPENDIX J

CRITERIA FOR SELECTION OF AUTOMATIC SAMPLING EQUIPMENT

CRITERIA FOR SELECTION OF AUTOMATIC SAMPLING EQUIPMENT

- 1. Capability for AC/DC operation with adequate dry battery energy storage for 120-hour operation at 1-hour sampling intervals.
- 2. Suitability for suspension in a standard manhole while accessible for inspection and sample removal.
- 3. Total weight, including batteries, under 18 kilograms (40 pounds).
- 4. Sample collection interval adjustable from 10 minutes to 4 hours.
- 5. Capability for flow-proportional and time-composite samples.
- 6. Capability for collecting a single 9.5 liter (2.5-gallon) sample and/or collecting 400-milliliter (0.11-gallon) discrete samples in a minimum of 24 containers.
- 7. Capability for multiplexing repeated aliquots into discrete bottles.
- 8. One intake hose with a minimum inner diameter of 0.64 centimeters (0.25 inches).
- 9. Intake hose liquid velocity adjustable from 0.61 to 3 meters per second (2.0 to 10 feet per second) with dial setting.
- 10. Minimum lift of 6.1 meters (20 feet).
- 11. Explosion-proof.
- 12. Watertight exterior case to protect components in the event of rain or submersion.
- 13. Exterior case capable of being locked, including lugs for attaching steel cable to prevent tampering and to provide security.
- 14. No metal parts in contact with waste source or samples.
- 15. An integral sample container compartment capable of maintaining samples at 4°C to 6°C for a period of 24 hours at ambient temperatures ranging from -30°C to 50°C.
- 16. With the exception of the intake hose, capability of operating in a temperature range from -30°C to 50°C.
- 17. Purge cycle before and after each collection interval and sensing mechanism to purge in the event of plugging during sample collection and then to collect the complete sample.
- 18. Field repairability.
- 19. Interchangeability between glass and plastic bottles, particularly in discrete samplers, is desirable.
- 20. Sampler exterior surface painted a light color to reflect sunlight.

APPENDIX K

QUALITY CONTROL PROCEDURES FOR FIELD ANALYSIS AND EQUIPMENT

QUALITY CONTROL PROCEDURES FOR FIELD ANALYSIS AND EQUIPMENT

| <u>Parameter</u> | General | <u>Daily</u> |
|--|---|---|
| Dissolved Oxygen • Membrane Electrode | Enter the make, model, and serial and/or ID number for each meter in a logbook. | Calibrate meter using manufacturer's instructions or Winkler-Azide method. |
| | Report data to nearest 0.1 mg/l. | Check membrane for air bubbles and holes. Change membrane and KCl if necessary. |
| | | Check leads, switch contacts, etc. for corrosion and shorts if meter pointer remains off-scale. |
| Winkler-Azide method | Record data to nearest 0.1 mg/l. | Duplicate analysis should be run as a precision check. Duplicate values should agree within ± 0.2 mg/l. |
| <u>pH</u> | | |
| Electrode Method | Enter the make, model, and serial and/or ID number for each meter in a logbook. | Calibrate the system against standard buffer solutions of known pH value; (e.g., 4, 7, and 9 at the start of a sampling run). |
| · | | Periodically check the buffers during the sample run and record the data in the logbook. |
| | | Be on the alert for erratic meter |

Quarterly

Check instrument calibration and linearity using a series of at least three dissolved oxygen standards.

Take all meters to the laboratory for maintenance, calibration, and quality control checks.

response arising from weak batteries, cracked electrodes, fouling, etc.

| Parameter | <u>General</u> | Daily | Quarterly |
|--------------------------------|---|--|---|
| pH (Continued) | | | |
| • Electrode Method (Continued) | | Check response and linearity following highly acidic or alkaline samples. Allow additional time for equilibration. Check against the closest reference solution each time a violation is found. Rinse electrodes thoroughly between samples and after calibration. | |
| Conductivity | Enter the make, model, and serial and/or ID number for each meter in a logbook. | Standardize with KCl standards having similar specific conductance values to those anticipated in the samples. Calculate the cell constant using two different standards. | Take all meters to lab for maintenance, calibration, and quality control checks. |
| | | | Check temperature compensation. |
| | | | Check date of last platinizing and replatinize, if necessary. |
| | | Rinse cell after each sample to prevent carryover. | Analyze NBS or EPA reference standard and record actual vs. observed readings in the logbook. |

35-45°C.

| Parameter | <u>General</u> | <u>Daily</u> | <u>Ouarterly</u> |
|--|--|---|--|
| Residual Chlorine Amperometric Titration | Enter the make, model, and ID and/or serial number of each titration apparatus in a logbook. Report results to nearest 0.01 mg/l. | Refer to instrument manufacturer's instructions for proper operation and calibration procedures. | Biweekly: return instrument to lab for maintenance and addition of fresh, standardize reagents. |
| <u>Temperature</u> Manual | Enter the make, model, and serial and/or ID number and temperature range. | Check for air spaces of bubbles in the column, cracks, etc. Compare with a known source if available. | Biweekly: check at two temperatures against a NBS or equivalent thermometer. Enter data in logbook. |
| | All standardization shall be against a traceable NBS or NBS calibrated thermometer. Reading shall agree within ±1°C. If enforcement action is anticipated, calibrate the thermometer before and after analysis. All data shall be read to the nearest 1°C. Report data between 10 and 99°C to two significant figures. | | Temperature readings shall agree within ±1°C or the thermometer shall be replaced or recalibrated. Initially and biannually: accuracy shall be determined throughout the expected working range of 0°C to 50°C. A minimum of three temperatures within the range should be used to verify accuracy. Preferable ranges are: 5-10°C, 15-25°C, and |

| <u>Parameter</u> | General | <u>Daily</u> | <u>Ouarterly</u> |
|-----------------------------------|--|---|---|
| • Thermistors, Thermographs, etc. | Enter the make, model, and serial and/or ID number of the instrument in a logbook. All standardization shall be against a NBS or NBS calibrated thermometer. Reading should agree within ±1°C. If enforcement action is anticipated refer to the procedure listed above. | Check thermistor and sensing device for response and operation according to the manufacturer's instruction. Record actual vs. standard temperature in logbook. | Initially and biannually: accuracy shall be determined throughout the expected working range of 0°C to 50°C. A minimum of three temperatures within the range should be used to verify accuracy. Preferable ranges are: 5-10°C, 15-25°C, and 35-45°C. |
| Flow Measurement | Enter the make, model, and serial and/or ID number of each flow measurement instrument in a logbook. | Install the device in accordance with the manufacturer's instructions and with the procedures given in owner's manual. | Annually: affix record of calibration (NBS, manufacturer) to the instrument log. |
| Automatic Samplers | Enter the make, model, and serial and/or ID number of each sampler in a logbook. | | Check intake velocity vs. head (minimum of three samples), and clock time setting vs. actual time interval. |