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NPDES Compliance Inspection Video Workbook: Sampling Wastewater at a Wastewater Treatment Facility

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SAMPLING WASTEWATER AT A WASTEWATER TREATMENT FACILITY WORKBOOK

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FOREWORD

PURPOSE OF WORKBOOK

In the fulfillment of their responsibilities, compliance inspectors are often required to conduct sampling at wastewater treatment facilities. The purpose of the *Sampling Wastewater at a Wastewater Treatment Facility* workbook is to teach compliance inspectors proper sampling procedures.

This workbook was prepared to be used in conjunction with the video, "Sampling Wastewater at a Wastewater Treatment Facility." This workbook will provide supplemental training information to augment the information contained in the video and will provide questions and answers throughout to test the user's knowledge. Answers to the questions are included in Appendix D. In addition, an exercise is included at the end of the workbook to allow users to design a sampling plan. Although this workbook was designed to accompany the video, it can also be used as an independent training resource.

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CHAPTER 1
INTRODUCTION

Facilities that discharge wastewater to receiving waters of the United States must apply for and maintain a National Pollutant Discharge Elimination System (NPDES) permit for that discharge. The NPDES permit specifies discharge limitations and self-monitoring requirements including sampling location, pollutant parameters to be monitored, monitoring frequency, type of sample required to be collected for each pollutant parameter, and reporting requirements.

PURPOSE OF CONDUCTING A COMPLIANCE SAMPLING INSPECTION

The purpose of a Compliance Sampling Inspection (CSI) is to collect samples of a facility's permitted discharge to verify compliance with the NPDES permit, evaluate the facility's self-monitoring data, and collect data for potential enforcement actions.

NPDES Permit Compliance Verification

Samples collected during the inspection and subsequently analyzed provide data that, when compared to the facility's permit discharge limits, verify the facility's compliance or noncompliance at the time of the sampling event. The sampling and analysis must be conducted according to proper procedures to ensure that the resulting analytical data will accurately represent the discharge at the time of sample collection.

Facility Self-Monitoring Evaluation

The NPDES permit program relies on permittee self-monitoring to provide discharge data to allow verification of compliance. The validity of this compliance verification depends on the validity of the self-monitoring data that is reported to the Control Authority. Therefore, the data provided must accurately represent the facility's wastestream and must be reported correctly. Correct reporting depends upon the exact transferral of data from other records and reports (e.g., laboratory bench sheets, operational logs) to the Discharge Monitoring Report (DMR). A review and comparison of the facility's self-monitoring DMR information with reports, logs, and flow data will allow the inspector to evaluate whether the permittee is correctly reporting the self-monitoring data.

Enforcement Action Data

Because facilities that violate the conditions of their NPDES permits are subject to enforcement action, the thoroughness and accuracy of the compliance inspection are paramount. The compliance inspector should observe the permitted sample location to certify that self-monitoring samples are collected at the point designated by the permit and should review the self-monitoring sampling techniques and sample handling procedures to ensure that they are proper. A review of self-monitoring data can identify suspect information, indicating that fraud or falsification may have occurred. The inspector should not voice this concern to facility personnel because it could hinder the facility's cooperation in the collection of additional information. The inspector should obtain copies of suspect DMRs and all corresponding reports, logs, and data. These materials will be invaluable in later enforcement proceedings.

Data obtained from compliance sampling can also be used as supplemental information in an enforcement proceeding. Sampling data are only useful in support of an enforcement action, however, if the inspector has followed the correct sampling, preservation, and chain-of-custody procedures and the laboratory has used the appropriate analytical methodology.

CHAPTER 2
PRE-INSPECTION PREPARATION

To conduct an effective sampling inspection, the compliance inspector must be properly prepared. This chapter presents information and guidance to be used by a compliance inspector in preparing for an inspection including developing a sampling plan. The overall success of a sampling inspection begins long before the inspector enters the treatment plant. Thorough preparation identifies critical needs and issues before the actual sampling event and allows the inspector to address these issues early on.

Developing a sampling plan allows the inspector to become familiar with the plant and its processes before arriving onsite. The sampling plan ensures that the inspector has all of the necessary equipment, knows what parameters and locations to sample, knows how to properly sample the given parameters, and is aware of the necessary safety precautions.

FACILITY AND PLANT PROCESS INFORMATION REVIEW

One of the first steps in inspection preparation and sampling plan development is to review relevant file information. This information may include the following:

- NPDES permit and permit application
- Inspection reports
- Discharge Monitoring Reports (DMRs)
- Correspondence between the permittee and Control Authority
- Information on enforcement actions.

NPDES Permit/Permit Application

A review of the facility's NPDES permit will identify the designated sampling location. This information will allow the inspector to ensure that samples collected during the inspection are collected at the monitoring location used by the permittee. If the permittee does not sample at the permit-designated location, the inspector may choose to collect two sets of samples; the first set at the required sampling point and the second set at the location routinely sampled by the permittee.

The permit should also identify the required parameters for sampling by the facility. The inspector most likely will sample for the same parameters. The permit will also identify type of sample to be collected (grab, composite, or continuous) for each pollutant, and the frequency at which each parameter is to be monitored.

The inspector should also review the facility's permit application, which will often provide additional information about the facility, such as the geographic location, plant processes, and location of the discharge point.

Inspection Reports

Review of previous inspection reports will identify processes used at the facility and will indicate which units are in service. The reports often describe the wastewater treatment processes and identify Operation and Maintenance (O&M), laboratory, and other issues. Safety concerns may be noted which allows the inspector to adequately prepare for those needs. The reports may contain data of parameters sampled during the inspections. Some inspection reports contain photographs of the facility, which are helpful to inspectors in becoming familiar with the plant before the actual inspection.

Discharge Monitoring Reports

A review of the DMRs will provide information on the facility's self-monitoring results. DMRs normally include the average and maximum level at which a parameter, such as daily flow, was discharged during the reporting period. A DMR review will allow the inspector to determine facility compliance, noncompliance parameters and the frequency of noncompliance.

Correspondence

Inspectors should review the facility's related correspondence file to understand the interaction between the permittee and the Control Authority. Correspondence will often indicate process modifications and facility upgrades and will sometimes shed light on the facility's attitude toward noncompliance. If the facility conducts in-house sample analyses, the correspondence file may reveal laboratory problems. This file may also contain updated information on the replacement of personnel.

Enforcement Actions

If any enforcement proceedings have been pursued against the facility, the actions should be reviewed to determine whether the facility is under any special requirements or

monitoring conditions. The enforcement action may also identify additional parameters for which the inspector should collect samples. The inspector may be required to ascertain the facility's compliance status with current enforcement action requirements; therefore, a thorough review of the enforcement history is essential.

SAMPLING PLAN CRITICAL INFORMATION

A review of the sampling techniques will prepare the inspector for successful sample collection. The inspector should review its appropriate sampling methods for each parameter to be monitored. The sampling plan is a guide for inspectors to identify all sampling needs and can be used as a checklist to ensure that all items needed for a successful sampling event will be available onsite. The following information should be included in the sampling plan:

- Parameters to be sampled
- Required sample containers and caps
- Sample container cleaning requirements
- Sample type
- Sample volume required
- Sampling location(s)
- Preservation requirements
- QA/QC and chain-of-custody
- Safety precautions
- Sample transport procedures
- Sample analysis.

Parameters

The sampling plan should identify each of the parameters to be sampled (e.g., metals, conventionals) and the equipment that will be needed. As indicated previously, samples should be collected for each parameter regulated by the facility's NPDES permit.

Sample Containers

The type of sample container and cap required for each sample parameter should be listed in the sampling plan. These requirements are found in 40 *CFR* Part 136. A summary of those requirements is included in Appendix A. Sample containers must be made of chemical-resistant material that does not react with or contaminate samples. Sample containers consist of clear glass, amber colored glass, and polyethylene (plastic). Sample container lids may be plastic or plastic with teflon septums. Teflon-lined lids are required for volatile organics

samples. Glass containers are required for samples such as oil and grease, phenols, and some priority pollutants. Amber colored bottles should be used when sampling for parameters that break down or oxidize when exposed to sunlight (for example, cyanide or phenols). For parameters that may be collected in either glass or plastic containers, the plastic container is preferred because it is less likely to break. Preplanning for sample containers and lids will result in the correct type being available at the time of sampling.

Cleaning Procedures

The sampling plan should include the specific cleaning requirements for the sample containers and equipment. Specific cleaning procedures depend on the parameters to be sampled and may require the use of water, various chemicals, sanitizers, detergents, and acids. Certain analytical methods specify particular cleaning procedures. All sample containers and equipment must be cleaned as required prior to use.

Sample Types

The proper sample type to be collected for each parameter should be recorded in the sampling plan. Use of the correct sample type helps ensure a representative sample is collected. There are two types of samples: grab and composite. Parameters that are not amenable to compositing (e.g., pH, temperature, dissolved oxygen, chlorine, purgeable organics, oil and grease, coliform bacteria, and others specified in the NPDES permit) should be collected as grab samples.

Sample Volumes

The sampling plan should note the sample volume required for each parameter and ensure that adequate containers will be available for onsite sample collection. The sample volume information is normally available from the laboratory that will conduct the analyses. The necessary volume will depend on the type and number of analyses needed. Sufficient sample must be collected to perform the required analyses and the laboratory Quality Assurance/Quality Control (QA/QC) measures. The volume of sample required for the specific parameters to be sampled can be found in Appendix B.

Sampling Location

The sampling plan should include the sampling location for each parameter to be sampled. Required sampling locations are given in the facility's NPDES permit. The permit may require that samples be collected at the influent and/or effluent, before and/or after

disinfection, immediately following the final process, or at the receiving waters. Samples required at both the influent and effluent help to determine the loading to the plant and percent of pollutant removal. The permit may require that specific samples be collected before chemical disinfection if the sample constituent can be altered by the disinfectant. The sampling location must allow the collection of a sample representative of the entire flow. Samples should be collected where the flow is turbulent and well mixed. They should be collected to ensure that surface scum and settled sludge are not included. Samples should be collected from the middle of the waste stream with the collection bottle below the surface and the opening facing upstream. By listing the sample location in the sampling plan, the inspector can become familiar with the required location for compliance sampling and ascertain that the facility is collecting samples at the designated locations.

Sample Preservation

Sample preservation ensures that the sample parameter constituents do not change between the time of collection and the time of analysis. Basically there are three methods of sample preservation; cooling, pH adjustment, and chemical fixation. Sample preservation methods for wastewater parameters are found in 40 *CFR* Part 136. The compliance inspector should review and incorporate the parameter-specific preservation information into the sampling plan and should become familiar with the preservation method for each parameter. The necessary preservation chemicals and ice (to maintain sample temperature at or below 4°C) should be listed in the sampling plan to ensure their inclusion with the equipment taken to the facility. The maximum holding time of the sample parameters, between collection and analysis, should also be listed in the sampling plan and can be found for specific parameters in 40 *CFR* Part 136. Appendix A includes a list of required sample preservation methods and maximum allowable holding times.

QA/QC/Chain-of-Custody

The sampling plan should list sampling QA/QC procedures and sample chain-of-custody requirements; both are essential to the validity of the inspection data. Sampling QA/QC requirements include (1) the use of blanks, (2) that representative samples be collected, (3) that sample collection records be maintained, (4) that samples be properly preserved, and (5) that chain-of-custody procedures be implemented.

The laboratory QA/QC requires that a number of duplicate, split, blank, and spiked samples be collected and analyzed and that approved sample analytical methods be used. The purpose of QA/QC is to ensure that the samples have not been unintentionally altered through

the accidental contamination of the samples. Quality control blanks include trip blanks, field blanks, and equipment blanks. Trip blanks are prepared at the beginning by pouring reagent water into a sampling container. This sample stays with the other sample containers throughout the sampling event to check for contamination that might have occurred during transport to and from the sampling site. Field blanks are prepared at the site by pouring reagent water into prepared sample bottles and handling them as all the other samples are handled. These blanks indicate whether contamination occurred during sample contamination. Equipment blanks are used to detect contamination during sampling. They are prepared by running reagent water through a clean piece of equipment and pouring it into a prepared sample bottle. QC also requires that a number of duplicate, split, and spiked samples be analyzed for the specific parameters. Quality assurance requires that records be maintained of sample collection, that approved analytical methods be used.

Chain-of-custody forms are used to document sample collection information, the names of all persons handling the sample, and the times the sample changed hands. Use of chain-of-custody forms ensures that the samples remain under the direct observation or control of the individuals handling the samples. The chain-of-custody form must accompany the samples and contain the signature of each individual who assumes custody of the samples.

Safety

The sampling plan should list the safety precautions to be taken at the facility and identify all personal safety equipment to be taken to the facility (for example, gloves, goggles, steel-toed shoes, long sleeves, hard hat).

Sample Transport

The sampling plan should identify the method of sample transport to the laboratory. Samples may be transported by the inspector or through a common carrier. The sample transportation should be evaluated to guarantee the maximum allowable holding time will not be exceeded and that samples will be shipped in compliance with DOT regulations. If samples are to be shipped overnight, the inspector should make certain that such a service is available at or near the wastewater treatment facility.

Sample Analysis

The compliance inspector must ensure the correct analytical technique will be used for each sample parameter. To achieve sample quality assurance the technique must be conducted precisely as directed in 40 CFR Part 136. Many approved 40 CFR Part 136 procedures are

contained in *Standard Methods for the Analyses of Water and Wastewater*. Any deviation from the approved methods undermines the validity of the sample and reduces its value as a tool for NPDES permit compliance verification, a facility self-monitoring evaluation, and a tool for enforcement action.

COORDINATION WITH THE ANALYTICAL LABORATORY

The inspector should plan and coordinate with the laboratory that will be analyzing the samples. This may involve obtaining coolers from the laboratory and tamper-proof seals, mailing labels, and shipping labels needed to transport samples to the laboratory as quickly as possible. If the compliance inspector does not have the necessary sampling containers, they can usually be supplied by the laboratory. The laboratory should ship the sample containers to the inspector along with the needed preservatives. The inspector should discuss with the laboratory parameters to be sampled, required sample size, number of samples for each parameter, and specific sample preservation and holding requirements. The laboratory should be contacted at an early date to be sure the necessary supplies are available at the time of the inspection.

During discussions with the laboratory, delivery and analysis of the samples should be coordinated. The samples should be delivered as quickly as possible since all samples must be analyzed within the required parameter holding time. The laboratory should be informed of the planned date of the sample transport to ensure adequate personnel will be available to analyze the samples in a timely manner.

COORDINATION WITH THE FACILITY

If the inspection is to be announced, the facility should be notified in advance to ensure that strategic personnel will be present to answer questions and conduct the visit. Small wastewater treatment plants often have a single operator with the ability to answer the vital questions. If the compliance inspector arrives on a day when that operator is involved elsewhere, the information such as sampling point, sampling technique, and location of files and report data may be unavailable to the inspector.

If the inspection is to be unannounced, the inspector must inform all involved personnel, including other government agencies, that the facility is not to be contacted. The element of surprise is beneficial in allowing samples to be collected under normal operating conditions.

EQUIPMENT PREPARATION

In preparation for a successful sampling event, the compliance inspector must be sure that all the necessary sampling equipment is present and in proper working order. Each item of equipment should be checked and calibrated, if applicable. The inspector may verify the equipment calibration after setup at the sampling site.

The batteries for automatic composite samplers should be recharged in preparation for the sampling event. It is wise to include one extra battery in case during field setup a battery is found to be too weak. Inspectors should also include additional tubing for the sampler. Dippers for collecting grab samples should be included in the supplies to be taken to the sample site. Inspectors should review facility records to determine whether the sampling location is accessible with a long handle dipper or whether a sampler will need to be lowered by a rope to the point of sampling.

The sample container and cap requirements should be evaluated during inspection planning for type, quantity, and size. As previously noted, sample container requirements for specific parameters can be found in 40 *CFR* Part 136. During the preparation for the sampling event, the inspector should collect the proper sample containers and caps for all sampling requirements. Extra containers and caps should be included in case of loss or breakage.

The inspector should ensure that the sampling containers and sampling equipment have been properly cleaned to prevent sample contamination. Sampling containers and equipment that are not properly cleaned may contain residue of samples previously collected. The residue can spike the facility's sample with constituents not present in the wastestream or falsely increase the level of contaminants that are present. The contamination of the samples will result in invalid data, which are not usable for evaluating the facility or for enforcement support.

The specific sample container cleaning requirements depend on the type of pollutant being sampled. Directions for the proper cleaning procedures can be found in the EPA's *Handbook for Analytical Quality Control in Water and Wastewater Laboratories*. The tubing used with a composite sampler also requires cleaning according to an approved procedure, or replacement with new approved tubing. Analytical equipment should also be calibrated.

The compliance inspector should use a field log during the sampling event to record vital site and sample information. A log book should be included with the supplies. A field log book should be a bound volume from which pages can be neither removed nor replaced.

The inspector must ensure all sample labels and/or seals are prepared. The labels may be either stick-on or attachable with clear tape. When preparing the labels, the inspectors should use only pens with waterproof ink. To be prepared for the sampling event, the inspector should add extra labels, waterproof pens, and clear tape to the equipment taken to the sampling site.

As discussed previously, preservation chemicals will be needed to prepare specific sample parameters for holding until analyzed. The preservation chemicals stabilize the sample so that the constituents do not change and the analysis data accurately reflect the sample source at the time of collection. The necessary sample parameter-specific chemical preservation information can be found in 40 *CFR* Part 136 or from the laboratory.

Reagent water will be needed for the sample and equipment blanks that will be included with the samples for analyses. The laboratory should be able to provide reagent water for this purpose.

In preparation for the sampling event, the compliance inspector should assess the possible safety needs at the facility and collect the necessary safety equipment. General safety equipment collected should include steel-toe boots, gloves, safety goggles or glasses, hard hats, and hearing protection. Specific safety equipment will depend upon the individual facility and parameters to be sampled.

The following miscellaneous items need to be included in the inspection preparation: coolers, ice, bubble wrap, and sealable bags. These items will be used in sample preservation and transport to the laboratory. If the inspector does not have access to sample coolers, the laboratory can probably supply them.

QUESTIONS

1. In preparation for a sampling event, a compliance inspector should review the facility's NPDES permit. Which of the following items are normally contained in the permit?

   A. Facility name and permit number
   B. The discharge monitoring limitations
   C. The permit effective date
   D. Special requirements and responsibilities
   E. All of the above

2. The facility DMR should be reviewed during sampling preparation. If available, how many months of data should be reviewed?

   A. One month
   B. Three months
   C. Six months
   D. Twelve months
   E. Forty-eight months

3. In the process of preparing for a successful sampling event, which of the following should the inspector include in the sampling plan?

   A. Parameters to be sampled, sample container cleaning requirements
   B. Preservation requirements, facility and laboratory information
   C. Sample analyses methods, sample chain-of-custody
   D. Safety precautions, a list of sampling equipment and miscellaneous items

4. The facility to be sampled and inspected must always be notified at least 2 weeks in advance to ensure that they are properly prepared for the inspection and that the necessary staff will be present to answer questions.

   A. True
   B. False

5. Which of the following describes an appropriate field log book used by a compliance sampling inspector?

   A. A loose leaf notebook
   B. A book completed in pencil to allow corrections to be made
   C. A notebook containing advance information to be completed during the inspection
   D. A record that remains in the control of the inspector at all times

6. List at least three pieces of safety equipment that should be taken for use during the inspection.

   _____
   _____

_____

7. During preparation for the sampling inspection, the sample containers and caps should be reviewed for which of the following?

   A. Type
   B. Quantity
   C. Size
   D. All of the above
   E. None of the above

CHAPTER 3
ONSITE ACTIVITIES

PRE-INSPECTION MEETING

The compliance inspector should meet with the facility staff and discuss what the inspection will entail. During the opening discussion, the inspector should determine the sampling location used by the facility personnel. The inspector should then have the plant staff identify that sampling location.

Sampling Location Evaluation

To verify the facility self-monitoring program, it is very important that the inspector thoroughly evaluate the sampling locations. The NPDES permit description of the designated sampling location should be compared to the actual location. If the location is not the designated point for sample collection, the inspector should question facility staff to determine how and why the location was selected.

The inspector must determine whether the designated sampling location will produce a representative sample. A representative sample, when analyzed, will produce data that present the actual level of the contaminants in the entire wastestream at the moment the sample was collected.

A representative location for the collection of an influent sample is a point preceding any sidestreams that are returned to the influent from the treatment plant processes. A representative location for the collection of an effluent sample is a point that includes all discharged process wastestreams. The location must have a well-mixed flow to ensure that constituents of the wastestream are uniform throughout the discharge. The flow at the sample point should be turbulent to keep the solids in suspension. The turbulence will also ensure that scum does not form on the water surface and that debris will not be present. The sample should represent the overall discharge and must not include scum, sediment, or debris. The sample should be collected from the middle of the flow with the container opening facing upstream. Sampling techniques should be compared to the approved methodology to ensure sample validity for compliance evaluation and enforcement needs.

As a result of the NPDES-designated sample location evaluation, the inspector may determine that the location will not provide a representative sample. The inspector should collect samples at the designated location but should also collect samples at a location determined to have a discharge that will supply a representative sample.

NPDES-DESIGNATED SAMPLE TYPE

The NPDES permit designates the sample type, either grab or composite, that is required for each parameter. If composite samples are required, the inspector should collect the aliquots manually or install an automatic sampler at the sample location and ensure that it is operating properly. The inspector should return the following day to collect the composite sample.

Grab Samples

Grab samples are collected manually over a period that does not exceed 15 minutes. Normally, the sample is collected as quickly as the flow has filled the sample container or dipper. The sample is collected by placing the sample container directly into the flow or by collecting a portion of the flow in a dipper and then filling the sample container from the dipper. Grab samples provide an instantaneous measurement of the flow constituents at the time of sample collection. Grab samples are appropriate when the wastestream flow and constituents have little variation and for parameters whose constituent levels change rapidly. Grab samples are required for specific sample parameters, such as pH, temperature, dissolved oxygen, oil and grease, and bacteria. The parameter to be sampled determines whether a grab sample is required and whether the sample must be collected directly into the sample container (e.g., oil and grease).

The collection of a fecal coliform sample entails several specific considerations. The sample container must remain capped until the time of sample collection to keep contaminants, which might cause a false high, out of the container. If the sample will be collected from a chlorinated wastestream, sodium thiosulfate, a dechlorinating agent, must be added to the sample container prior to sample collection. Sodium thiosulfate, which may be in a liquid or powder form, will halt the disinfection action of the chlorine. While wearing gloves, the inspector must collect the sample directly into the sterile sample container. The direct collection of the sample will keep extraneous sources of coliform from being introduced. The sample container should not be overflowed since this will result in a loss of the sodium thiosulfate. Finally, a head space of air must be left in the sample container to aid in mixing the sample prior to analysis.

Composite Samples

Composite samples can be collected manually, as four or more discrete grab samples, at equal time intervals and volumes. When a manual composite is collected, the flow must be recorded at the time of collection and the individual samples proportioned to the flow when the aliquots are composited. Most compliance inspectors use an automatic composite sampler, which collects a sample at a selected volume and time interval. Composite samples may be flow proportional so that when the flow increases or decreases, the volume of sample collected increases or decreases accordingly. Composite samples may also be proportioned to flow by increasing or decreasing the frequency of sample collection as the flow increases or decreases. Composite samples are normally collected over 24 hours and therefore give an accurate portrayal of a wastestream that varies in flow and constituents.

Time composite samples are normally collect into a single large-volume container at specified intervals of time. The samples are acceptable for wastestreams that remain relatively uniform in both their constituents and their flow rate during the sample collection.

COMPOSITE SAMPLER

Setup

The inspector will set up the composite sampler at the representative sample location. The sampler should be checked to be certain the batteries have a sufficient charge to operate the sampler for the planned sampling period. The automatic sampler must be programmed to collect a sample volume that is sufficient for conducting the analyses. However, the sample volume should not cause the sample container to overflow. The inspector will also set the desired time interval between sample collection (e.g., 15 minutes, 30 minutes, 60 minutes). A sample cycle should be initiated to collect a trial sample to be sure the sampler is functioning properly and the volume collected is adequate. It is advised that, when possible, the inspector remain at the site to observe whether the first sample is collected at the appropriate time interval.

The inspector may wish to place a tamper-proof seal on the composite sampler and the sample line to be sure the sample is not intentionally altered during collection.

Cooling

Ice should be added to the automatic sampler to cool the samples to 4°C during collection. An automatic sampler that has a light colored case is preferred for sampling during the summer to increase the lasting ability of the ice.

Sample Removal

Manually collected aliquots should be well mixed and then transferred to a single composite container. To reduce the chance of contamination and loss of constituents, inspectors should fill parameter sample containers directly from the composite. The composite sample must be well mixed before the individual parameter containers are filled to ensure that a representative sample is placed in each sample container.

Automatic samplers that have a single container for the collected aliquots require the composite to be well mixed when the individual parameter sample containers are filled. Aliquots collected in an automatic sampler with discrete bottles are normally combined as a composite. The composite may consist of all or a portion of the discrete aliquots. The parameter sample containers are filled from the composite sample. If the inspector notes that one of the discrete aliquots is unusual, the inspector should record the aliquot characteristics and collection time in the field log. The inspector may choose to have the unusual aliquot analyzed individually rather than as a portion of the composite.

SAMPLE IDENTIFICATION

Samples must be positively identified from the instant of collection to the recording of the analytical data to ensure the exact identity of each sample. During the sampling event, the inspector should maintain a field logbook that contains all pertinent sample information. The inspector should identify each sample with a label that will remain with that sample until the laboratory logs the sample in and performs the analysis. The sample information in the field logbook must correspond with that on the sample container labels.

Field Log

An inspector's logbook is a field record of the sampling inspection and the samples collected. The log should be concise and contain observations and findings of the inspection. Information on unusual samples should be documented. The inspector must also record sample collection data. The log should contain the sample identification number, date and time of sample collection, name of the person(s) collecting the sample, sample parameters, specific

location of the sample collection, and preservatives added. Additionally, the log can be used to record sample transportation information.

Sample Labeling

The sample container must be properly labeled to ensure positive sample identification at all times. Multiple samples, in their containers, will be stored together, and the sample label is the only certain means of identification. A properly completed and attached label will quickly and positively identify each individual sample. The information should be recorded with a waterproof pen on a waterproof label which is attached to the container, not the cap. Adhesive labels may be used, or clear tape can be placed over the label attaching it to the sample container. Each sample container label should be properly completed with all of the following vital information: sample identification number, date and time of sample collection, name of the person collecting the sample, sample parameter or analyses requested, preservative added, and sample site location.

SAMPLE PRESERVATION

Purpose

The purpose of sample preservation is to protect the sample constituents so that the analytical data will successfully portray the wastestream at the time of collection. Preservation maintains the sample integrity during the time between sample collection and analysis at the laboratory.

Methods

Sample preservation, as directed in 40 *CFR* Part 136, can be achieved through the following methods: cooling, pH adjustment, and chemical fixation. The sample parameter will determine the preservation method to be used. Many parameters are preserved by using a combination of the methods. Sample preservation should be preformed immediately upon sample collection. Appendix A includes 40 CFR Part 136 preservation methods.

Preservation through cooling requires that the sample be cooled rapidly to 4°C and then maintained at or below that temperature until analyzed. Cooling is normally accomplished by placing the samples in a cooler with ice. When a composite sample is collected, the individual aliquots must be cooled as required during the time of collection. If aliquots are collected manually, they must be stored and cooled in a refrigerator or in ice. If an automatic composite sampler is used, the inspector should add ice to the sampler unit case. Samples stored in a

refrigerator must be maintained at the required temperature. The majority of the wastewater parameters, unless analyzed immediately, require cooling to at least 4°C for preservation.

Many parameters require pH adjustment to preserve the sample until analysis. The adjustment may be achieved through addition of an acid (e.g., sulfuric, hydrochloric, nitric) to reduce the pH, or addition of an alkaline (e.g., sodium hydroxide) to raise the pH. The adjustment is not accomplished by the addition of a specified volume of acid or alkaline, but through the addition of a sufficient quantity to bring the sample to the required pH for the preservation to occur. Samples receiving an acid addition usually require the pH to be adjusted to < 2. Samples receiving an alkaline addition require adjustment to various pH levels, depending upon the parameter.

Samples such as fecal coliform and Winkler Dissolved Oxygen (DO) require immediate chemical fixation to maintain the sample until an analysis for the parameter is conducted. Proper chemical fixation will maintain the sample parameter constituent at the current level.

All of the sample parameters specified in 40 *CFR* Part 136 have a maximum allowable holding time after proper sample preservation. The holding time depends on the specific parameter and therefore may vary from as short as a few hours to as long as months. The holding time is considered to be the maximum time the sample can be preserved and still reproduce an accurate picture of the wastestream at the time the sample was collected. Samples should always be analyzed as soon as is possible. Chlorine residual, pH, DO, and temperature and must be analyzed immediately.

CHAIN-OF-CUSTODY

A chain-of-custody form is a paper trail of the vital information concerning the sample. It identifies who has had possession of the sample and what has been added to it. The information recorded on a chain-of-custody form provides legal documentation of the sample handling. Numerous samples may be tracked on a single chain-of-custody form.

Procedures

The inspector should complete a chain-of-custody record that contains information about the inspector, the sample, and the laboratory. The form should contain the following information: sample identification numbers, date and time of sample collection, sample types and locations, sampler name(s), preservatives used, number of containers, analyses requested,

date and time the samples are relinquished and by whom, and date and time the samples are received in laboratory and by whom.

An additional procedure for ensuring the integrity of a sample is to place a tamper-proof seal over the container and cap. The seal should contain the sample ID number, the date and time of sample collection, and the initials of the person collecting the sample.

An example of a completed chain-of-custody form is provided in Appendix C to assist the inspector in the proper method of completing a chain-of-custody form and to illustrate the type of information requested.

SAMPLE TRANSPORTATION

To prepare the samples for shipping, the inspector should be sure the sample containers are properly labeled and tightly sealed. Glass sample containers should be wrapped in bubble wrap to prevent breakage. The containers should be placed in a sealed waterproof bag in a cooler. Sufficient ice must be added to the cooler to maintain the 4°C temperature during the transportation to the laboratory. The chain-of-custody form should be placed in a separate sealed bag in the cooler on top of the samples and ice. The cooler should be secured with several bands of tape. The address should then be attached to the top of the cooler. The inspector must be careful to use a shipping source that will get samples to the laboratory within the prescribed holding time.

If the inspector personally transports the samples to the laboratory, he or she must first prepare the samples and place them in a cooler in a manner similar to that described above. The inspector must sign off when the samples are released into the custody of the laboratory personnel who in turn sign to document their receipt of the samples. The inspector must be certain that the samples will be delivered to the laboratory quickly so that the analyses can be conducted within the prescribed holding times. The samples should be kept secure during the transport. The vehicle should be locked at all times when the inspector is not present.

QUESTIONS

1. During the onsite sampling, the compliance inspector should compare the NPDES permitted sampling point to the:

   A. Permit application
   B. Normal facility sampling point
   C. QA/QC plan
   D. Previous inspection sampling report

2. A representative sample should portray the sample source. The factors to review to determine whether the sample location will provide a representative sample are:

   A. That the flow is well-mixed
   B. That the flow is turbulent and the solids are suspended
   C. That the location does not have a concentration of scum, sediment, or debris
   D. All of the above

3. When collecting a sample, the inspector should hold the container mouth so that it is:

   A. Facing upstream
   B. Facing downstream
   C. Facing the side of the flow
   D. Facing the surface of the flow

4. Name three of the parameters required to be collected as a grab sample?

   _____
   _____
   _____

5. When conducting an evaluation of the effluent sampling location, the inspector should:

   A. Compare the normal facility sampling location to the NPDES-designated location
   B. Determine whether the sampling location will provide a representative sample
   C. Collect samples from the designated location
   D. Collect samples after all discharge sidestreams
   E. All of the above

6. What are the two types of samples designated by the NPDES permit?

   _____
   _____

7. Fecal coliform samples have special sampling considerations. Which of the following is not a special consideration?

   A. The container cap must not be removed until the sample collection
   B. The sample must be collected directly into the container
   C. The sample must be analyzed immediately
   D. Sodium thiosulfate must be used to halt the disinfection by chlorine
   E. The sample container must not be overflowed

8. When a composite sampler is set up, the inspector must not:

   A. Program the sample volume
   B. Program the time interval
   C. Add ice to the case

D. Inform the facility personal of the time they should remove and refrigerate the composite

9. Which of the following sample preservation methods is most often required?

A. Cooling
B. pH adjustment
C. Chemical fixation
D. Holding time
E. None of the above

10. Preservation through cooling requires the sample to be maintained at:

A. 4°F
B. 20°F
C. 4°C
D. 20°C

11. Sample preservation by the pH adjustment method requires a specified volume of acid or alkaline be added to the sample.

A. True
B. False

12. Which of the following cannot be preserved by chemical fixation?

A. Fecal coliform
B. Chlorine residual
C. Winkler dissolved oxygen

13. Which of the following actions is not required in preparing samples for shipping?

A. The cooler containing the samples must be placed in a sealed box
B. The cooler must contain ice to keep the samples cool
C. The sample containers should be in a sealed bag
D. The cooler should be sealed with tape

CHAPTER 4
OTHER CONSIDERATIONS

SELF-MONITORING VERIFICATION

During the sampling inspection, one of the inspector's most important responsibilities is to evaluate the facility's self-monitoring program. This evaluation is to determine whether the facility data submitted to the Control Authority accurately represent the discharge. The inspector should evaluate the sampling location to determine whether it is as designated in the NPDES permit and whether samples collected are representative. The facility's sampling technique, sample preservation, and sample storage should also be evaluated. The inspector should evaluate the analytical techniques if the sample analyses are conducted onsite. The accuracy of reporting the laboratory data should be verified.

FLOW MEASUREMENT ACCURACY CONFIRMATION

Most NPDES permitted facilities are required to report the average daily flow and the monthly maximum flow. Also, many NPDES permits require that parameters such as BOD_5 and total suspended solids be reported in pounds discharged per day. The reported data will be incorrect if the flow measurement is inaccurate. Therefore, the flow measuring equipment should be inspected and the primary flow measuring device reading compared with the secondary device readout. The variation in the measurement between the two devices should be less than 10 percent.

RECORDS INSPECTION

To evaluate the permittee's self-monitoring, the inspector must review several types of records. The records should be maintained by the facility for a minimum of 3 years and should be readily available to the inspector.

Laboratory

The laboratory records should be reviewed to evaluate whether currently approved analytical methods are used, whether the analysis method is recorded, and whether samples are analyzed within the allowable holding time. The laboratory records should identify the date and time of sample analyses and the initials of the individual conducting the analyses. The mathematical calculations performed by the laboratory staff should be randomly reviewed to

evaluate whether the calculations are performed correctly. The laboratory bench sheet data should be compared to the submitted DMRs.

The sampling equipment should be inspected for proper operation and cleaning. The laboratory equipment should be inspected for proper operation, calibration, and temperature. Laboratory chemicals, such as pH buffers, should be checked to be sure that the expiration date has not been exceeded.

Discharge Monitoring Reports

The DMRs should be reviewed to ensure that the submitted data correspond to the laboratory analyses data. Normally, a review of the most recent 12 months of reports is sufficient. Also, the inspector should check to see whether any additional analyses are conducted, but are not reported as required. The DMRs should also be reviewed to ensure that they are properly completed and that monitoring is conducted at the required frequency and for all required parameters. The sample type (i.e., grab, composite) should be compared to the NPDES permit requirement.

Sampling

The permittee should have records that document the facility's sampling information. The inspector should review these records for sampling location, date and time of sample collection, and the sample parameter collected.

Chain-of-Custody

The facility should maintain chain-of-custody records for the NPDES permit-required sample parameter analyses. The completed forms should be maintained at the facility even if laboratory analyses are performed offsite, the laboratory should return a copy of the chain-of-custody record to the facility with the sample data.

Quality Control

If possible, the laboratory procedures and data should be reviewed to determine whether QC is being conducted properly. The laboratory should be analyzing split, duplicate blank, and spike samples at a rate dependent upon the number of parameter analyses performed.

QUESTIONS

1. Which one of the following is not a purpose of a compliance inspection?

   A. To diagnose the cause of the facility's compliance problems
   B. To verify compliance with the NPDES permit
   C. To evaluate the facility's self-monitoring
   D. To collect enforcement data

2. During a normal sampling inspection, the inspector will not review which of the following records?

   A. Laboratory
   B. DMRs
   C. Sampling
   D. Maintenance
   E. Chain-of-custody

CHAPTER 5
SAMPLING CONSIDERATIONS EXERCISE

You are to perform a CSI at a WWTP which is required by its NPDES permit to monitor Biochemical Oxygen Demand (BOD), Total Suspended Solids (TSS), Total Residual Chlorine (TRC), Oil and Grease (O&G), pH, Dissolved Oxygen (DO), fecal coliform, copper, and trichloroethlene (TCE). The facility is required to collect composite samples for BOD, TSS, and copper. The facility's sampling point does not require entry into a confined space. To perform this sampling you need to develop a sampling plan that includes the following:

1. Based on the information provided in 40 CFR Part 136 (see Appendices A and B) complete the following table by indicating necessary sample types, sample containers, sample volumes, and preservation techniques for each pollutant parameter to be sampled.

Pollutant to Sample	Sample Type	Sample Container	Minimum Sample Volume	Preservative
BOD				
TSS				
TRC				
O&G				
pH				
DO				
Fecal				
Copper				
TCE				

2. Indicate parameters that have to be analyzed onsite.

3. Identify the sampling equipment and protective clothing needed to take to the site.

CHAPTER 6
ADDITIONAL GUIDANCE/REFERENCES

A copy of required sample preservation, sampling containers, and allowable holding times as in 40 *CFR* Part 136 guidance is found in Appendix A. Sample volumes to be collected for different pollutants and described in Appendix B.

Inspectors should refer to the following references for further guidance:

U.S. Environmental Protection Agency. September 1994. "Interim Final NPDES Compliance Inspection Manual."

U.S. Environmental Protection Agency. August 1990. *NPDES Compliance Inspector Training, Sampling*.

APHA, AWWA, and WEF. *Standard Methods for the Examination of Water and Wastewater*. (Use most current approved edition.)

Code of Federal Regulations. Title 40, Part 136. Office of the Federal Register.

U.S. Environmental Protection Agency. September 1981. *NPDES Compliance Flow Measurement Manual*.

U.S. Environmental Protection Agency. 1979b. *Methods for Chemical Analysis of Water and Wastes*. EPA-600/4-79-020.

U.S. Environmental Protection Agency. 1982. *Handbook for Sampling and Sample Preservation of Water and Wastewater*. EPA-600/4-82-029

U.S. Environmental Protection Agency. March 1980. *Collection and Analysis of Purgeable Organics Emitted from Wastewater Treatment Plants*. EPA-600/2-80-017.

APPENDIX A

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES (EXCERPT FROM 40 *CFR* PART 136 TABLE 11)

Appendix A

Required Containers, Preservation Techniques, and Holding Times
(Excerpt from 40 *CFR* Part 136, Table II)

Parameter	Container[1]	Preservative[2,3]	Maximum Holding Time[4]
BACTERIAL TESTS			
Coliform, fecal and total	P,G	Cool, 4°C 0.008% $Na_2S_2O_3$[5]	6 hours
Fecal streptococci	P,G	Cool, 4°C 0.008% $Na_2S_2O_3$[5]	6 hours
INORGANIC TESTS			
Acidity	P,G	Cool, 4°C	14 days
Alkalinity	P,G	Cool, 4°C	14 days
Ammonia	P,G	Cool, 4°C H_2SO_4 to pH<2	28 days
Biochemical oxygen demand	P,G	Cool, 4°C	48 hours
Biochemical oxygen demand, carbonaceous	P,G	Cool, 4°C	48 hours
Bromide	P,G	None required	28 days
Chemical oxygen demand	P,G	Cool, 4°C H_2SO_4 to pH<2	28 days
Chloride	P,G	None required	28 days
Chlorine, total residual	P,G	None required	Analyze immediately
Color	P,G	Cool, 4°C	48 hours

Appendix A

Required Containers, Preservation Techniques, and Holding Times
(Excerpt from 40 *CFR* Part 136, Table II)
(Continued)

Parameter	Container[1]	Preservative[2,3]	Maximum Holding Time[4]
Cyanide, total and amenable to chlorination	P,G	Cool, 4°C NaOH to pH>12 0.6 g ascorbic acid[5]	14 days[6]
Fluoride	P	None required	28 days
Hardness	P,G	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months
Hydrogen ion (pH)	P,G	None required	Analyze immediately
Kjeldahl and organic nitrogen	P,G	Cool, 4°C H_2SO_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°C	48 hours
Oil and grease	G	Cool, 4°C HCl, H_2SO_4 to pH<2	28 days
Organic carbon	P,G	Cool, 4°C HCl, H_2SO_4 to pH<2	28 daysOrthophosphate phosphorusP,GFilter immediately Cool, 4°C48 hours
Dissolved oxygen Probe Winkler	 G bottle & top G bottle & top	 None required Fix onsite and store in the dark	 Analyze immediately 8 hours
Phenols	G	Cool, 4°C H_2SO_4 to pH<2	28 days

Required Containers, Preservation Techniques, and Holding Times
(Excerpt from 40 *CFR* Part 136, Table II)
(Continued)

Parameter	Container[1]	Preservative[2,3]	Maximum Holding Time[4]
Phosphorus (elemental)	G	Cool, 4°C	48 hours
Phosphorus, total dissolved	P,G	Cool, 4°C H_2SO_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
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 plus sodium hydroxide to pH >9	7 daysSulfiteP,GNone requiredAnalyze immediately
Surfactants	P,G	Cool, 4°C	48 hours
Temperature	P,G	None required	Analyze immediately
Turbidity	P,G	Cool, 4°C	48 hours
ORGANIC TESTS[8]			
Purgeable halocarbons	G, teflon-lined septum	Cool, 4°C 0.008% $Na_2S_2O_3$[5]	14 days
Purgeable aromatic hydrocarbons	G, teflon-lined septum	Cool, 4°C 0.008% $Na_2S_2O_3$[5] HC1 to pH2[9]	14 days

Appendix A

Required Containers, Preservation Techniques, and Holding Times
(Excerpt from 40 *CFR* Part 136, Table II)
(Continued)

Parameter	Container[1]	Preservative[2,3]	Maximum Holding Time[4]
Acrolein and acrylonitrile	G, teflon-lined septum	Cool, 4°C 0.008% $Na_2S_2O_3$[5] Adjust pH to 4-5[10]	14 days
Phenols[11]	G, teflon-lined cap	Cool, 4°C	7 days until extraction 40 days after extraction
Benzidenes[11]	G, teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[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 0.008% $Na_2S_2O_3$[5] Store in the dark	7 days until extraction; 40 days after extractionPolychlorinated biphenyls (PCBs)[11]G, teflon-lined capCool, 4°C7 days until extraction; 40 days after extraction
Nitroaromatics and isophorone[11]	G, teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[5] 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% $Na_2S_2O_3$[5] Store in the dark	7 days until extraction; 40 days after extraction
Haloethers[11]	G, teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[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
2,3,7,8-tetrachlorodibenzo-p-dioxin[1]	G, teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
PESTICIDES TEST			
Organochlorine pesticides[11]	G, teflon-lined cap	Cool, 4°C pH 5-9[15]	7 days until extraction; 40 days after extraction

Appendix A

Required Containers, Preservation Techniques, and Holding Times
(Excerpt from 40 *CFR* Part 136, Table II)
(Continued)

Parameter	Container[1]	Preservative[2,3]	Maximum Holding Time[4]
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 chemical 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 chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting are completed.

Appendix A

Required Containers, Preservation Techniques, and Holding Times
(Excerpt from 40 *CFR* Part 136 Table II)
(Continued)

3	When any sample is to be shipped by common carrier or sent through the Unites States mail, 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 the preservation requirements of this Table, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: hydrochloric acid (HC1) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and sodium hydroxide (NaOH) in water solutions at concentrations of 0.08% by weight or less (pH about 12.3 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 be 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 and has received a variance from the Regional Administrator under § 136.3(e). 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 that this is necessary to maintain sample stability.
5	Should only be used in the presence of residual chlorine.
6	Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments to determine whether 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.
7	Samples should be filtered immediately onsite before adding preservative for dissolved metals.
8	Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific organic compounds.
9	Samples receiving no pH adjustment must be analyzed within 7 days of sampling.
10	The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
11	When the extractable analytes of concern fall within a single chemical category, the specified preservation and maximum holding times should be observed for optimum safeguarding 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 between 6 and 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 residual chlorine) and footnotes 12 and 13 (re: the analysis of benzidine).
12	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.

Appendix A

Required Containers, Preservation Techniques, and Holding Times
(Excerpt from 40 *CFR* Part 136 Table II)
(Continued)

[13]	Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
[14]	For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to between 7 and 10 with NaOH within 24 hours of sampling.
[15]	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 B

REQUIRED SAMPLE VOLUMES

Appendix B

Volume of Sample Required for Determination of the Various Constituents of Industrial Wastewater (Associated Water and Air Resource Engineers, Inc. 1973 *Handbook for Monitoring Industrial Wastewater*. USEPA Technology Transfer.)

Tests	Volume of Sample, (1) ml
PHYSICAL	
Color and Odor(2)	100 to 500
Corrosivity(2)	Flowing sample
Electrical conductivity(2)	100
pH, electrometric(2)	100
Radioactivity	100 to 1,000
Specific gravity(2)	100
Temperature(2)	Flowing sample
Toxicity(2)	1,000 to 20,000
Turbidity(2)	100 to 1,000
CHEMICAL	
Dissolved Gases:	
Ammonia,(3) NH(3)	500
Carbon dioxide,(3) free $C0_2$	200
Chlorine,(3) free Cl_2	200
Hydrogen,(3) H_2	1,000
Hydrogen sulfide,(3) H_2S	500
Oxygen,(3) O_2	500 to 1,000
Sulfur dioxide,(3) free SO_2	100
Miscellaneous:	
Acidity and alkalinity	100
Bacteria, iron	500
Bacteria, sulfate-reducing	100
Biochemical oxygen demand (BOD)	100 to 500
Carbon dioxide, total $C0_2$ (including $C0_3$GG, $HC0_3$G, and free)	200
Chemical oxygen demand (dichromate)	50 to 100
Chlorine requirement	2,000 to 4,000
Chlorine, total residual Cl_2 (including OClG, HOCl, NH_2Cl, $NHCl_2$, and free)	200
Chloroform-extractable matter	1,000
Detergents	100 to 200
Hardness	50 to 100
Hydrazine	50 to 100
Microorganisms	100 to 200
Volatile and filming amines	500 to 1,000
Oily matter	3,000 to 5,000
Organic nitrogen	500 to 1,000
Phenolic compounds	800 to 4,000
pH, colorimetric	10 to 20
Polyphosphates	100 to 200
Silica	50 to 1,000
Solids, dissolved	100 to 20,000
Solids, suspended	50 to 1,000
Tannin and lignin	100 to 200

Appendix B

Volume of Sample Required for Determination of the Various Constituents of Industrial Wastewater (Continued)

Tests	Volume of Sample, (1) ml
Cations:	
Aluminum, Al^{+++}	100 to 1,000
Ammonium,(3) $NH4^{+}$	500
Antimony, Sb^{+++} to Sb^{+++++}	100 to 1,000
Arsenic, As^{+++} to As^{+++++}	100 to 1,000
Barium, Ba^{++}	100 to 1,000
Cadmium, Cd^{++}	100 to 1,000
Calcium, Ca^{++}	100 to 1,000
Chromium, Cr^{+++} to Cr^{++++++}	100 to 1,000
Copper, Cu^{++}	200 to 4,000
Iron,(3) Fe^{++} and Fe^{+++}	100 to 1,000
Lead, Pb^{++}	100 to 4,000
Magnesium, Mg^{++}	100 to 1,000
Manganese, Mn^{++} to $Mn^{+++++++}$	100 to 1,000
Mercury, Hg^{+} and Hg^{++}	100 to 1,000
Potassium, K^{+}	100 to 1,000
Nickel, Ni^{++}	100 to 1,000
Silver, Ag^{+}	100 to 1,000
Sodium, Na^{+}	100 to 1,000
Strontium, Sr^{++}	100 to 1,000
Tin, Sn^{++} and Sn^{++++}	100 to 1,000
Zinc, Zn^{++}	100 to 1,000
Anions:	
Bicarbonate, HCO_3G	100 to 200
Bromide, BrG	100
Carbonate, CO_3GG	100 to 200
Chloride, ClGG	25 to 100
Cyanide, CnG	25 to 100
Fluoride, FlG	200
Hydroxide, OHG	50 to 100
Iodide, IG	100
Nitrate, NO_3G	10 to 100
Nitrite, NO_2GG	50 to 100
Phosphate, ortho, PO_4GG, HPO_4GG, H_2PO_4G	50 to 100
Sulfate, SO_4GG, HSO_4 100 to 1,000	
Sulfide, SGG, HSG 100 to 500	
Sulfite, SO_3GG, HSO_3G 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; containers must be completely filled and sealed against air exposure.

APPENDIX C

EXAMPLE CHAIN-OF-CUSTODY FORM

U.S. ENVIRONMENTAL PROTECTION AGENCY
Environmental Services Division

CHAIN OF CUSTODY RECORD

REGION VIII, ONE DENVER PLACE
999 18TH STREET
DENVER, CO 80202-2413

PROJ. NO.	PROJECT NAME							NO OF CON-TAINERS							REMARKS
SAMPLERS: *(Signature)*															
STAT. NO	DATE	TIME	COMP	GRAB	STATION LOCATION										

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

Split Samples
[ ] Accepted **Signature**

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

APPENDIX D

QUIZ ANSWERS

ANSWERS

Chapter 2

1. E
2. D
3. A
4. B
5. D
6. Gloves, hard hats, steel-toe boots, hearing protection, safety goggles
7. D

Chapter 3

1. B
2. D
3. A
4. pH, temperature, DO, oil and grease, bacteria
5. E
6. Grab, composite
7. C
8. D
9. A
10. C
11. B
12. D
13. A

Chapter 4

1. A
2. D

SAMPLING EXERCISE ANSWERS

1.

Pollutant to Sample	Sample Type	Sample Container	Minimum Sample Volume (ml)	Preservative
BOD	Composite	P,G	100	Cool to 4°C
TSS	Composite	P,G	50	Cool to 4°C
TRC	Grab	P,G	200	None
O&G	Grab	G	3,000	Cool to 4°C HCl, H_2SO_4 to pH<2
pH	Grab	P,G	100	None
DO	Grab	G	500	None
Fecal	Grab	P,G	100	Cool to 4°C 0.008%$Na_2S_2O_3$
Copper	Composite	P,G	200	HNO_3 to pH<2
TCE	Grab	G, teflon-lined cap	500	Cool to 4°C

P = Plastic
G = Glass

2. TRC, pH, and DO must be analyzed onsite.

3. Sampling Equipment

Composite sampler
Battery
Sampler tubing
Ice
pH meter
pH buffers
Deionized water
DO meter
TRC meter
Bucket/Dipper
Sample preservatives
Sample containers
Sample bottle labels/seals
Sample logbook
Chain-of-custody forms
Shipping labels
Security tape
Cooler chest
Packing material
Analysis request forms
Waterproof pen

Protective Clothing

Safety shoes
Hard hat
Safety glasses or goggles
Hard hat
Gloves