

What's In This Chapter?

1. Common equipment used in a water laboratory
2. How to use the meniscus to properly measure a liquid volume
3. The basic laboratory techniques of pipetting and titration
4. Common laboratory safety procedures and techniques
5. The three different types of samples
6. A description of a good sample location
7. Which tests are best determined from grab samples
8. The hold time and temperature for biological samples
9. The three cardinal rules to sampling.
10. When in the daily flow cycle chlorine residuals should be taken
11. The basic procedure for
 - Alkalinity
 - Chlorine residual
 - pH
 - Fluoride
 - Bacteriological sampling
12. The function and use of QAQC
13. Proper recordkeeping procedures

Key Words

- Fecal Coliform
- NFR
- QAQC

Introduction

Level of Material

The material in this lesson is presented at a beginning level. There has been no assumption that the reader is familiar with water laboratory equipment or procedures. Only a general discussion is provided for routine laboratory testing. There has been no attempt to provide step-by-step procedures for the tests discussed in this lesson. The goal is to provide the user with a general understanding of the common laboratory procedures used in a typical water laboratory in a small treatment plant.

Reference Text

There are numerous reference books available that provide step-by-step procedures for all of the laboratory tests that are normally conducted by an operator. Contact EPA, the American Water Works Association or the local state regulatory agency for information on these books.

Commercial Laboratories

Many small systems prefer to utilize a commercial laboratory to perform the testing that is required by ADEC. However there are lab tests that help an operator control their treatment process (process control testing). A basic understanding of both types of testing can be useful for the entry level operator.

Content

This lesson contains the following sections:

- Equipment
- Laboratory techniques
- Laboratory safety
- Sampling procedures
- Types of samples
- Testing
- **QA/QC¹**
- Record keeping

¹ **QA/QC** - Quality Assurance - Quality Control - The formal process of assuring the laboratory is performing testing in the most accurate and precise manner possible in order to comply with specified regulations.

Equipment

Limitations

A typical laboratory could contain a wide variety of special equipment. The following discussion provides information on only that equipment discussed in the test procedures provided in this lesson.

Hardware

Water Bath Incubator

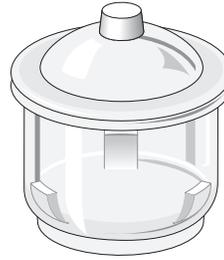
The incubator required to perform the fecal coliform test must be capable of maintaining a temperature of 44.5°C within 0.2°C. A water bath incubator is frequently used. This device looks like a rectangular metal basin. Water is placed inside the basin. The temperature is maintained by a heating element at the bottom of the basin and a lid that keeps the heat from escaping. This type of incubator is capable of maintaining the strict temperature requirement for the fecal coliform test.

Drying Oven

A small oven that looks like a rectangular metal box is used to maintain the proper temperature to dry filters for the suspended solids and total solids tests.

Desiccator

A desiccator is a glass or plastic device that looks similar to a salad bowl with a lid. The device is used to allow heated samples to cool without taking on moisture from the air. To prevent moisture from entering the samples a material called desiccant is placed in the bottom of the desiccator. This material absorbs moisture.



Desiccator

Fecal Filter Apparatus

A special stainless steel or plastic funnel and filter holder are used to run the **fecal coliform**² test. The bottom of this device is mounted in a rubber stopper that will fit into a filter flask. The top of the funnel secures to the bottom by a 1/4 turn twist or a magnet.



Funnel

² **Fecal Coliform** - The Fecal coliform group of bacteria is a bacterial indicator of contamination. This group has as one of its primary habitats the intestinal tract of human beings. Coliforms also may be found in the intestinal tract of other warm-blooded animals. Also called E. Coli or Escherichia Coli.

Autoclave

In order to sterilize the equipment needed to perform bacteriological testing an autoclave is needed. This device allows the development of high temperatures and pressures needed to kill all microorganisms. An autoclave is very similar to a pressure cooker.

Glassware

Volumetric Flasks

In order to measure large volumes of liquid accurately, a volumetric flask is used. These devices have only one measurement point. This point is a line etched on the neck of the flask. Various sizes are available.

Pipettes

In order to measure small volumes of liquids accurately a pipette is used.

Burettes

In order to deliver precise volumes of fluid at a controlled rate a long glass tube called a burette is used. This tube is fastened into a stand and filled from the top. The flow is controlled by a small valve on the bottom.

Graduated Cylinders

Graduated cylinders are glass or plastic cylinders that have a scale marked to the nearest milliliter etched onto their sides. Graduated cylinders are used to measure sample volumes. While fairly accurate, they are not as accurate as volumetric flasks.

Beakers

Beakers are glass or plastic containers used in a laboratory. While they have a scale on the side that indicates their volume they are the least accurate of all of the glassware. They are used to hold samples, mix solutions and other general tasks in the lab.

Erlenmeyer Flasks

A special flask called an Erlenmeyer flask is used to titrate samples. (See titration discussion). These flasks have a large flat bottom and taper to a narrow top to prevent spilling when the contents are mixed.

Vacuum Flask

Vacuum flask are used in the fecal coliform and suspended solids test. The vacuum flask is made like an Erlenmeyer flask except there is a connection near the top of the flask for a vacuum source.

Gooch Crucible

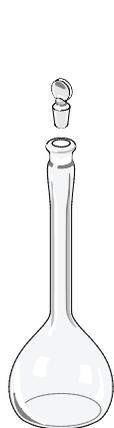
One of the suspended solids test procedures requires the use of a ceramic device called a Gooch Crucible. This device is funnel shaped with small holes in the lower end. The Gooch is placed in a rubber holder in a vacuum flask and a glass fiber filter is placed inside.

Buchner Funnel

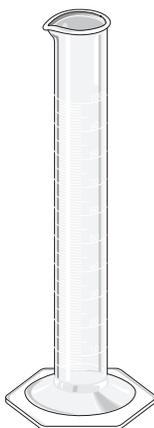
The Gooch crucible is too small to allow filtration of a large volume sample that contains a high concentration of solids. To measure suspended solids a buchner funnel is used. This is a large ceramic or plastic funnel with holes in a plate that has been formed inside the funnel. A glass fiber filter is placed on the plate and the funnel placed in a rubber holder in a vacuum flask.

Petri Dish

A petri dish is a small flat glass or plastic dish with a lid. Media and bacteriological samples are placed in the petri dish which is placed in an incubator. There are a wide variety of petri dish sizes available.



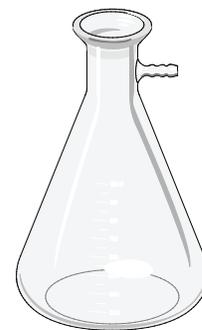
Volumetric flask



Graduated cylinder



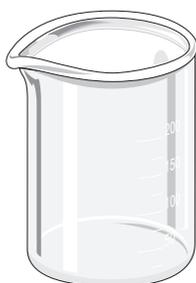
Erlenmeyer flask



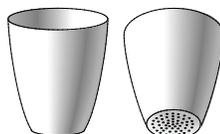
Vacuum flask



Pipette



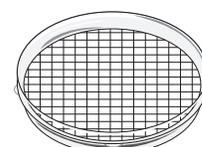
Beaker



Gooch crucible



Buchner funnel



Petri dish

Basic Laboratory Techniques

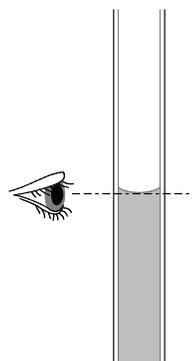
In order to understand the following laboratory testing procedures it is necessary to explain three basic laboratory techniques. These are pipetting, titrating and reading liquid volumes.

Pipetting

Liquid is drawn into a pipette by use of a vacuum placed on the top of the pipette. This vacuum is commonly created with a rubber bulb that is placed over the end of the pipette. The flow of liquid out of the pipette is controlled by placing the end of one finger over the opening at the end of the pipette. While this sounds easy it takes considerable practice to be able to perform this task with high accuracy.

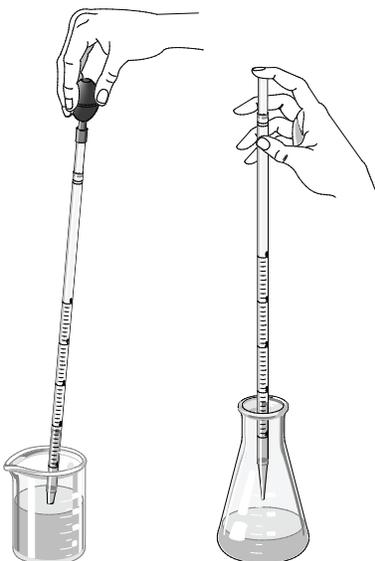
Reading volumes

When a fluid is placed in a glass measuring device such as a graduated cylinder, volumetric flask, pipette or burette the quantity must be recorded. Most fluids adhere to the side walls of a glass container. In doing this the surface becomes concaved. This concaved surface is called a meniscus. To properly read the level of fluid in a glass device, read along the bottom of the meniscus.

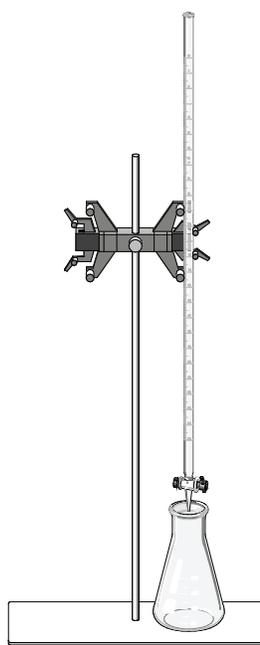


Titrating

Titration is performed in many of the test procedures. This process requires a burette and erlenmeyer flask. A sample and some chemical are placed in the flask. A second chemical is placed in the burette. When the two chemicals mix a color is developed or they cause a color to disappear. The normal procedure is to twirl the flask while allowing a controlled rate of flow from the burette to enter the flask. When a specific color point is reached the volume used is determined from the burette and used to calculate the concentration in the flask.



Pipetting



Titration technique

Laboratory Safety

Preventing Hazards

The laboratory is not necessarily a dangerous place. However, when the laboratory is used by inexperienced and/or careless operators accidents can easily happen. The basic tests covered in this manual requires a minimum of laboratory experience and are not highly hazardous as long as basic safety precautions are observed.

Proper procedures and techniques can be placed into two general groups:

1. Personal protection
2. Proper handling of chemicals and bacteriological materials.

Personal Protection

The most important aspect of laboratory safety is to protect yourself and others around you.

Eye Protection

Eyes must be protected from splashing chemicals, chemical dust, high pressure water and broken glass. Protection is provided by wearing chemical safety glasses or goggles anytime the operator is performing laboratory tests, handling glassware or chemicals.

Safety Equipment

Each laboratory should be equipped with the following safety equipment:

- Fire extinguisher - dry chemical or CO₂
- Fully equipped first-aid cabinet
- Fire blanket
- Chemical eye-wash

Operators should be trained in the use of this equipment and know its location.

Pipetting

When transferring chemicals or measuring a sample with a pipette, always use a pipette fill bulb. Never suck the solution into the pipette with your mouth.

Handling Samples

Samples should not be handled with bare hands. Use disposable latex gloves to protect against infections.

Wash-Up

To prevent the transfer of chemicals to the eyes and the transfer of disease, wash hands with hot water and soap after handling sewage samples.

Eating

To prevent accidental intake of chemicals or ingesting pathogenic organisms, never use laboratory glassware for serving food or drink. Never smoke in a laboratory. Never eat or drink in a laboratory.

Labels

Containers of chemicals received from a manufacturer have the proper labeling. When a chemical is transferred to a storage bottle the following information must be placed on the label:

- Name of chemical
- Chemical formula - if known
- Concentration
- Date
- Initials of the person preparing the container

In addition it is desirable to place an **NFR**³ label on the container. If the container is removed from the treatment plant the NFR label must be on the chemical.

³ **NFR** - National Fire Rating System
This system provides the users of chemicals with a four diamond placard that indicates the health concern, flammability, and reactivity levels of the chemical.

Handling Chemicals and Samples

The operator may be required, as part of the job, to handle a variety of hazardous materials. It is beyond the scope of this manual to discuss all of the chemicals an operator may encounter in the lab. Caution: chemicals which are handled improperly, or randomly mixed together can produce heat, toxic fumes or even explode. In order to properly handle, control and dispose of these materials it is important that the operator review the Material Safety Data Sheets (MSDS) for each chemical used with care. The following is a brief and general discussion of handling precautions. However, this information does not substitute for the MSDS.

MSDS

A current MSDS must be on-site for each chemical that is in the laboratory. It is the responsibility of the employer to see that each person who may use a chemical is aware of the location of the MSDS and has been trained in how to read the MSDS.

Corrosive Chemicals

Acids

Operators may handle sulfuric, hydrochloric, nitric and glacial acetic acids. All acids are classified as corrosive and may cause burns to the skin, if contacted.

Spills

The specifics on how to handle a spill are found in the MSDS. However, in general the spill may be controlled by dilution with large and immediate quantities of water or neutralized with sodium bicarbonate. The spill area should then be cleaned and dried.

Contact with Skin

Contact with acids can quickly cause severe burns to the skin. The area should be immediately washed with large quantities of cool water and neutralized with sodium bicarbonate.

Pipetting

As with other materials, acids should only be pipetted using a pipette bulb.

Diluting with Water

When diluting an acid with water, always add the acid to the water. Never add the water to the acid.

Bases

Operators and laboratory technicians may handle sodium hydroxide, potassium hydroxide, and ammonium hydroxide. These are all strong bases that are corrosive to clothing and can cause burns to the skin. First aid is provided by immediately washing the area with large quantities of cool water.

Infectious Material

While it is unlikely that an operator would contract a disease while handling water or wastewater samples, the possibility does exist. An operator can prevent disease by following a few simple rules.

Open Wounds

Untreated water samples may contain bacteria, viruses and protozoa that can cause disease. One of the methods of contacting these microorganisms is through breaks in the skin. Keep wounds covered by wearing disposable latex gloves when handling all samples.

Ingestion

When handling fecal coliform plates it is easy to transfer microorganism to the hands and then transfer them to your mouth while eating or smoking. Therefore, wash your hands thoroughly after handling samples, even if latex gloves are worn.

Clothing

In the performance of routine laboratory work the operator can easily collect pathogenic microorganisms onto their clothes. These microorganisms can then be transferred to the home. Therefore, change clothing at the plant and wash up before going home. If a shower is available, it is a good idea to shower and change clothes before leaving the plant.

Sampling Procedures

Must Be Representative

Sampling is often the most neglected technique in laboratory control testing. A sample must accurately represent the body of water intended for study. Although a test may be performed carefully and accurately, the result is meaningless if the sample is not representative of the water source from which it was taken.

Obtaining Representative Samples

Over the years operators and technicians have developed specific sampling techniques and procedures that, when used properly, provide representative samples. These are called the principles of sampling.

Principles of Sampling

Three Cardinal Rules

Water samples are taken from a wide variety of locations under many different conditions. Sampling sites should be selected to meet the requirements of the information desired. Sampling methods should be carefully considered. Regardless of the site or method chosen, there are Three Cardinal Rules that apply to all samples. They are cleanliness, documentation and preservation.

Cleanliness

All containers including caps and measuring devices with which the sample comes in contact must be cleaned. Process control samples should be taken in containers washed in soap and water. Total coliform and fecal coliform samples must be taken in a special sterilized container.

Documentation

Water sample labels should note:

- The type of sample
- Source the sample is collected from
- Location of sampling point
- The date and hour sampled
- Name of sampler
- The temperature of the sample
- Recent weather conditions
- Flow at time of sampling

Location Expanded

A sample is only a representation of the conditions at the point of sampling. For example, the conditions in a sedimentation basin may vary greatly from one end of the basin to the other. Therefore, it is imperative that the exact basin sample location be included in the documentation. For example, we would write “east end of basin two feet back of overflow weir.” We would not write “sample taken from sedimentation basin.”

It is often helpful to identify standard sample sites within the plant and distribution system with a number or other identifying title. The exact description of that sample point can be detailed on a sample site plan or standard sampling plan.

Preservation

Samples may contain living organisms which continue to grow unless the life processes are slowed by lowering temperatures or halted by addition of chemicals. In addition, chemical degradation can also occur if samples are not properly preserved and stored prior to testing.

Preservation Methods

The correct form of preservation must be practiced, and will vary with the type of sample. In general, samples containing living organisms (bacteriological samples) may be preserved up to 6 hours if refrigerated at 4°C. Chemical samples may need to be stored out of the light or have a specific chemical added.

Chain of Custody

If a sample is sent off site for analysis, a proper chain of custody procedure should be followed. A chain of custody provides a paper trail that documents each person that handles the sample from sampling to disposal. This procedure ensures proper quality control of the sample when data validation is necessary.

Other Sampling Considerations

Representative Location

Samples must be selected from a location that is representative of the conditions. Typically, this is a location where the flow is well mixed. Dead ends and corners of tanks and basins are not usually representative of the entire flow.

Number and Volume

In order for the sampling to be representative, there must be the proper number and volume of samples collected.

Large Particles

Large particles should be excluded from the sample. They are not representative of the sample stream.

Deposits and Growths

Deposits and growths that have accumulated at the sampling site must be avoided in the sample. This includes slime or algae growing on sample lines and faucets.

Aseptic Conditions

The process of maintaining the quality of a bacteriological sample is called aseptic handling. This means avoiding contamination from skin, clothing, equipment, water, and adjacent surfaces.

Data

Unless the proper data is recorded with the sample the sample is not valid.

Mixing

Always mix the sample before removing a portion.

Time Frame

Samples should be tested as soon as possible – always within the permissible time interval after sampling.

Summary

A good sample location is one where the flow is well mixed, has easy access and the flow is representative of the overall conditions.

Types of Samples

There are three types of samples collected by plant operators:

1. Grab samples
2. Composite samples
3. Proportional composite samples

Each type of sample has its proper function.

Grab Samples

A “grab” sample is a sample that is taken at one particular time. Grab samples are taken because they are required or because there is a lack of time to collect composite samples. For some tests, grab samples are preferred.

Use of Grab Samples

Tests such as residual chlorine, dissolved oxygen, and pH are determined from grab samples because they cannot be preserved and a grab sample is the most representative.

Sampling and Flow - Chlorine

If only one chlorine residual sample can be taken per day, it is best to take this sample at peak flow. Chlorine residuals are normally at their lowest during peak flow. This is because; even with flow proportional chlorinators the feed rate is not increased at an exact proportion to flow. If at all possible chlorine residual samples should be taken at minimum and at peak flows. This will provide the operator with a view of the residual ranges found in the system. The required minimum CT must be maintained in the distribution system to ensure proper disinfection has taken place. The minimum CT is calculated based on chlorine residuals at peak flow when there is the lowest contact time in the system.

Sampling and Flow - Total Coliform

Because total coliform numbers in the water system are related to chlorine levels it is best to sample for total coliform at the same time that samples are collected for chlorine residuals.

Sampling for pH

In order for pH readings to be representative of the plant conditions they should be taken at maximum and minimum flows. If, due to time constraints, they can only be taken once a day, then they should be taken at the same time each day.

Composite Samples

A composite sample is a series of grab samples poured together to make one sample. The simplest type of composite sample consists of grabs of equal volume and is applicable only to situations of uniform flow.

Use of Composite Samples

A composite sample is only representative if the flow at the point of sampling remains constant throughout the sampling period. The sampling period is typically 24 hours but may be more or less depending on the constituent to be tested for.

Flow Proportional Composite Samples

In proportional composite samples, the volume of each portion is adjusted to the flow at the time the portion is collected. All portions are mixed together to produce a final sample representative of the flow during that particular collection period. Composite samples are representative of the character of the flow over a period of time. The effects of intermittent changes in strength and flow are eliminated. The portion collected should be obtained with sufficient frequency to obtain average results.

Required Lab Testing

Requirements

The Alaska Department of Environmental Conservation (ADEC) monitoring summary specifies which lab tests will be required of a specific system. This section covers the some of the common tests an operator may perform. All water systems are required to perform and report specific laboratory tests.

The following material is a discussion and description of the listed tests, no intent for step-by-step instruction is implied. Always follow the instructions for the specific test kit used.

Water systems must use EPA-approved analytical methods when analyzing samples to meet federal monitoring requirements or to demonstrate compliance with drinking water regulations. Approved methods are listed in the Code of Federal Regulations after publication in a final rule or as part of an expedited approval. Approved methods are developed by EPA, other government agencies, universities, consensus methods organizations, water laboratories, and instrument manufacturers.

Analytical Method

An analytical method is a procedure that determines the concentration of a contaminant in a water sample. Analytical methods generally describe:

- How to collect, preserve, and store the sample.
- Procedures to concentrate, separate, identify, and quantify contaminants present in the sample.
- Quality control criteria the analytical data must meet.
- How to report the results of the analysis.

In general, an analytical method:

- Is applicable to routine analyses of samples.
- Is suitable for measuring the drinking water contaminant in the concentration range of interest.
- Provides data with the necessary accuracy and precision to demonstrate compliance or meet monitoring objectives in a wide variety of drinking waters.
- Includes instructions for all aspects of the analysis from sample collection to data reporting.
- Incorporates appropriate quality control criteria so that acceptable method performance is demonstrated during the analysis of samples.

Sampling

For Screw-cap Bottles

To collect water samples using screw-cap sample bottles, use the following procedures:

- Label the bottle with the site number, date, and time.
- Remove the cap from the bottle just before sampling. Avoid touching the inside of the bottle or the cap. If you accidentally touch the inside of the bottle, use another one.
- Leave a 1-inch air space. Do not fill the bottle completely (so that the sample can be shaken just before analysis). Recap the bottle carefully, remembering not to touch the inside.
- Fill in the bottle number and/or sample site number on the appropriate label. This is important because it tells the lab which bottle goes with which site.
- If the samples are to be analyzed in the lab, place them in the cooler for transport to the lab.

For Whirl-pak® Bags

To collect water samples using Whirl-pak® Bags, use the following procedures:

- Label the bag with the site number, date, and time.
- Tear off the top of the bag along the perforation above the wire tab just prior to sampling. Avoid touching the inside of the bag. If you accidentally touch the inside of the bag, use another one.
- Fill the bag no more than 3/4 full!
- Pull on the wire tabs to close the bag. Continue holding the wire tabs and flip the bag over at least 4-5 times quickly to seal the bag. Don't try to squeeze

the air out of the top of the bag. Fold the ends of the wire tabs together at the top of the bag, being careful not to puncture the bag. Twist them together, forming a loop.

- Fill in the bag number and/or site number on the appropriate label. This is important! It is the only way the lab knows which bag goes with which site.

Total Alkalinity

Alkalinity is a measure of the capacity of water to neutralize acids. The total alkalinity analysis involves titration. In this test, titration is the addition of small, precise quantities of sulfuric acid (the reagent) to the sample until the sample reaches a colorimetric end point corresponding to a specific pH (known as an endpoint). The amount of acid used corresponds to the total alkalinity of the sample.

Sampling and Storage

Collect samples in clean plastic or glass bottles. Fill completely and cap tightly.

Avoid excessive agitation or prolonged exposure to air. Samples should be analyzed as soon as possible after collection but can be stored at least 24 hours by cooling to 4 °C (39 °F) or below. Warm to room temperature before analyzing.

Equipment

For total alkalinity, use a portable test kit, such as a Hach model AL-DT using a Digital titrator. Digital titrators have counters that display numbers. A plunger is forced into a cartridge containing the reagent by turning a knob on the titrator. As the knob turns, the counter changes in proportion to the amount of reagent used. Alkalinity is then calculated based on the amount used. Digital titrators allow for much more precision and uniformity in the amount of titrant that is used.

Procedure

Follow the procedures instructions for specific brand and model test kits you use. The following is a general procedure for the Hach test kit.

1. Collect the sample

2. Measure total Alkalinity:

Select the sample volume and Sulfuric Acid (H_2SO_4) Titration cartridge corresponding to the expected alkalinity concentration as mg/L calcium carbonate (CaCO_3) from table in the kit instructions.

1. Insert a clean delivery tube into the sulfuric acid titration cartridge and attach the cartridge to the titrator body.
2. Hold the titrator, with the cartridge tip pointing up, over a sink. Turn the delivery knob to eject air and a few drops of titrant. Reset the counter to 0 and wipe the tip.
3. Use a graduated cylinder to measure the sample volume from the table in the kit instructions. Transfer the sample into a clean 250-mL Erlenmeyer flask. Dilute to about the 100-mL mark with deionized water, if necessary.
4. Add the contents of one Phenolphthalein Indicator Powder Pillow and swirl to mix.
5. If the solution turns pink, titrate to a colorless end point. Place the delivery tube tip into the solution and swirl the flask while titrating with sulfuric acid.

Record the number of digits required. Note: If the solution is colorless before titrating with sulfuric acid, the Phenolphthalein (P) Alkalinity is zero; proceed with step 8.

6. Calculate: Digits Required x Digit Multiplier = mg/L CaCO₃ P Alkalinity
7. Add the contents of one Bromcresol Green-Methyl Red Indicator Powder Pillow to the flask and swirl to mix.
8. Continue the titration with sulfuric acid to a light greenish blue-gray (pH 5.1), a light violet-gray (pH 4.8), or a light pink (pH 4.5) color, as required by the sample composition; see kit instructions. Record the number of digits required.
9. Calculate: Total Digits Required x Digit Multiplier = mg/L as CaCO₃ Total Alkalinity.

Note: Carbonate, bicarbonate and hydroxide concentrations may be expressed individually using the relationships shown in kit instructions.

Note: Milligrams equivalent/L Alkalinity = mg/L as CaCO₃ ÷ 50.

Perform an Accuracy Check

This accuracy check should be performed when interferences are suspected or to verify analytical technique.

1. Snap the neck off an Alkalinity Standard Solution Voluette® Ampul, 0.500 N.
2. Use a TenSette® Pipet to add 0.1 mL of standard to the sample titrated in Steps 6 or 9.
3. Resume titration back to the same end point. Record the number of digits needed.
4. Repeat, using two more additions of 0.1 mL. Titrate to the end point after each addition.
5. Each 0.1 mL addition of standard should require 25 additional digits of 1.600 N titrant or 250 digits of 0.1600 N titrant. If these uniform increases do not occur, refer to kit instructions on the Accuracy Check and Standard Additions.

Interferences

Highly colored or turbid samples may mask the color change at the end point. Use a pH meter to identify the endpoints for these samples.

Chlorine may interfere with the indicators. Add one drop of 0.1 N Sodium Thiosulfate to eliminate this interference.

Chlorine Residual (Free Residual – DPD) Color Comparator Method

The amount of free chlorine present in a distribution system is important because these chlorine molecules provide additional protection against waterborne diseases, should contamination enter the distribution system.

DPD Method

The DPD method is the only acceptable procedure for measuring free chlorine residual in water distribution systems. N,N-diethyl-p-phenylenediamine (DPD) is added to a sample and, through a series of reactions, a chemical is produced that is

red in color. The color intensity correlates to the residual chlorine concentration. The sample color is compared to colors on a color wheel to determine chlorine residual.

Storage of Samples

Chlorine in solution is very reactive and very unstable. Because of this it is impossible to preserve a sample for residual chlorine. Any sample taken for residual chlorine analysis must be tested immediately. According to EPA, this means the sample must be tested within fifteen minutes of collection.

There are also other concerns when sampling for residual chlorine. Exposure to sunlight and sample agitation reduces the chlorine to ineffective forms. Additionally, a dirty sample collection bottle, whether glass or plastic, can create a chlorine demand. All these interferences will give you lower residual chlorine values than what may actually be present in the field. All of these interferences can also be avoided with proper sample collection and handling.

Equipment

Hach Model #CN - 66 or comparable Wallace & Tiernan or LaMotte comparators

Reagents

DPD Free Chlorine Powder Pillow - Hach #14070-99

Special Instructions

Each brand of color comparator has its own peculiarities. This procedure will be for the HACH comparator. Special instructions will be added in the right hand column of each step for the other comparators.

Procedure - Free Chlorine Residual

1. Clean comparator glass cells

Periodically wash the cells with hot soapy water and a soft test tube brush. Rinse thoroughly with distilled water. Let the cells drain dry.

2. Rinse glass cells with sample

Do this each time the cells are used. The rinse will remove any remaining chemical and accumulated dust.

3. Fill left sample tube

Place 5 mL of sample in the left-hand cell/tube. This is referred to as the blank. The purpose of this step is to compensate for any color or turbidity in the sample.
Note: The Wallace & Tiernan comparator calls for 15 mL of sample. The LaMotte does not use a blank.

4. Fill right sample tube

Place 5 mL of sample in the right-hand cell/tube. This is the portion of the sample to be tested.

5. Add reagents to tube

Add contents of powder pillow to right-hand cell. DPD tablets may be substituted for the powder pillow.

Note: Be sure the reagent pillow has DPD Free printed on it. Do not use DPD Total!

6. Insert rubber stoppers

These will prevent loss of sample during mixing.

Note: Rubber stoppers are a possible source of contamination. Gentle swirling should achieve adequate mixing, making the stoppers unnecessary.

7. Stir by swirling the test cell

Do not place thumb or finger on the top of the test cell - this could contaminate the sample.

8. Wait 30 seconds

Read color within one minute.

9. Adjust the comparator wheel

Hold the comparator up to a light source and look through the viewing windows in the back of the comparator. Rotate the color wheel until the color in the test window matches the color in the blank window.

With the LaMotte, move the test cell until it matches the color in one of the vials.

10. Read the residual

Read the test results on the scale at the bottom of the comparator. The residual will be indicated mg/L.

11. Record the results

The results should be recorded as mg/L of free chlorine residual.

12. Rinse the test cells

Rinse each tube twice with distilled water, invert the tubes, and place in the comparator to dry. If there is no distilled water, rinse with sample water, invert, and allow the cells to dry.

Note: DPD powder comes in small foil envelopes rather than plastic pillows.

However, they are still referred to as pillows. These envelopes are impervious to sunlight, thus extending their shelf life over the pillows.

Chlorine Residual (Free Residual 0-2 mg/L - DPD) Spectrophotometer

The DPD method is the only acceptable procedure for measuring free chlorine residual in water distribution systems. N,N-diethyl-p-phenylenediamine (DPD) is added to a sample and, through a series of reactions, a chemical is produced that is red in color. The color intensity correlates to the residual chlorine concentration. In this test, a spectrophotometer is used to more accurately measure this intensity of the red color instead of a color wheel.

Equipment

HACH Pocket Colorimeter II Chlorine Test Kit (Cat. No. 5870000) Borosilicate glass sample container with screw-on top.

Reagents

DPD Free Chlorine Powder Pillow - HACH # 1407099.

Procedure

1. Fill a 10 -mL cell with sample

2. Press the POWER key

The arrow should indicate the low range channel (LR). For residuals in the 0 to 2.00 mg/L range, the instrument should be in low (LO) range. This allows the result to be read in the hundredths. To change modes, consult owners manual.

3. Calibrate the meter

Remove the meter cap. Place the blank in the cell holder with the diamond mark facing the keypad. Fit the meter cap over the cell compartment to cover the cell.

Note: Wipe excess liquid and finger prints off sample cells.

Press ZERO/SCROLL. The display will show “- - -” then “0.00”. Remove the blank from the cell holder.

4. Fill a second 10-mL cell

Fill a second 10-mL cell to the 10-mL line with sample.

5. Add DPD reagent

Add the contents of one DPD Free Chlorine Powder Pillow to the sample cell (the prepared sample).

6. Cap and shake

Firmly screw cap onto sample bottle and shake gently for 20 seconds.

Note: Shaking dissipates bubbles that may form in samples with dissolved gases.

Note: A pink color will develop if chlorine is present.

7. Place sample in instrument

Carefully wipe the outside of the sample bottle to remove any liquid, dirt, and/or fingerprints. Arrange the sample bottle so the white diamond is facing the operator. Within one minute after adding DPD reagent, place the mixed sample into the cell holder. *Note:* Accuracy is not affected by undissolved powder.

8. Place light shield

Place the instrument cap over the sample. The cap should fit snugly, with the smooth side of the cap facing the operator.

9. Read and record

Press READ/ENTER. The instrument will show “- - -” followed by the results in mg/L chlorine. *Note:* If the sample temporarily turns yellow after reagent addition, or if the display shows over range, dilute a fresh sample and repeat the test. A slight loss of chlorine may occur because of the dilution. Multiply the result by the appropriate dilution factor.

Fluoride Residual - Low Range (0-2 mg/L)

Fluoride is fed into water distribution systems as a means of reducing dental cavities in children.

Method

The method described in this procedure is for the HACH Fluoride Pocket Colorimeter II with SPADNS 2 (Arsenic-free) Kit.

Storage of Samples

Since fluoride is very stable, samples for testing can be stored for seven (7) days providing that the sample is cooled to 4°C. The storage container should be polypropylene or borosilicate glass. Warm samples to room temperature before analysis.

Equipment

Fluoride Pocket Colorimeter II with SPADNS 2 (Arsenic-free) Kit (Cat. No. 2513100)

Polypropylene or borosilicate glass sample container with screw-on top.

Reagents

- Distilled water
- Accuvacs Non-Arsenic SPADNS Reagent ampuls or SPADNS 2 (Arsenic-free) Fluoride Reagent Solution, 1 L

Ampuls or Pipets?

Fluoride test may be conducted using accuvac© ampuls or pipets. This procedure is for SPADNS AccuVac® Method* USEPA Accepted.

Procedure

1. Press the POWER key

The arrow should indicate channel 2.

Note: See instructions for information on selecting the correct range channel.

2. Collect sample

Collect at least 40 mL of sample in a 50-mL beaker. Fill another 50-mL beaker with at least 40 mL of deionized water. *Note:* The sample and water should be the same temperature (± 1 °C).

3. Fill the Ampuls

Fill a SPADNS Fluoride AccuVac Ampul with sample. Fill another SPADNS Fluoride AccuVac Ampul with deionized water (the blank).

Note: Keep the tip of the ampul immersed until the ampul fills completely.

4. Mix the Ampuls

Quickly invert the ampuls several times to mix.

Note: Wipe off any liquid or fingerprints.

5. Wait 1 minute

6. Read the Blank

Place the blank in the cell holder and cover the blank with the instrument cap.

7. Press ZERO/SCROLL

The display will show “- - -” then 0.0. Remove the blank from the cell holder.

8. Read the Sample

Place the prepared sample in the cell holder and cover the sample cell with the instrument cap.

9. Press READ/ENTER

The display will show “- - -”, followed by results in mg/L fluoride (F⁻). Note: If the instrument shows a flashing 2.2 (over range), dilute the sample with an equal volume of water and repeat the test. Multiply the result by 2. **Important:** The primary MCL for Fluoride is 4.0 and the secondary MCL is 2.0.

Accuracy Check - Standard Solutions Method

Use a 1.00 mg/L fluoride standard solution in place of the sample. Perform the procedure as described above.

Safety Concern

Older reagents used in previous Hach test kits contained sodium arsenite. Final solutions remaining after the test contained arsenic in sufficient concentration to be regulated as a hazardous waste for Federal RCRA. Operators should use the newer arsenic-free reagents.

pH

pH is a measure of hydrogen ion (H⁺) concentration and is generally used to describe a system as being acidic or basic. It is not to be confused with alkalinity or acidity, which are entirely different tests. pH measurements are taken at various points throughout a treatment plant, and any abnormal readings can be an indication of a change in water quality.

Sampling

You must measure the pH within 2 hours of the sample collection. This is because the pH will change due to the carbon dioxide from the air dissolving in the water, which will tend to lower the pH of the sample.

Equipment

pH Meters

A pH meter measures the electric potential (millivolts) across an electrode when immersed in water. This electric potential is a function of the hydrogen ion activity in the sample. Therefore, pH meters can display results in either millivolts (mV) or pH units.

A pH meter consists of a potentiometer, which measures electric current; a glass electrode, which senses the electric potential where it meets the water sample; a reference electrode, which provides a constant electric potential; and a temperature compensating device, which adjusts the readings according to the temperature of the sample (since pH varies with temperature). The reference and glass electrodes are frequently combined into a single probe called a combination electrode.

There is a wide variety of meters, but the most important part of the pH meter is the electrode. Buy a good, reliable electrode and follow the manufacturer's instructions

for proper maintenance. Infrequently used or improperly maintained electrodes are subject to corrosion, which makes them highly inaccurate.

pH “Pocket Pals” and Color Comparators

pH “pocket pals” are electronic hand-held “pens” that are dipped in the water and provide a digital readout of the pH. They can be calibrated to one pH buffer (lab meters, on the other hand, can be calibrated to two or more buffer solutions and thus are more accurate over a wide range of pH measurements).

Color comparators involve adding a reagent to the sample that colors the sample water. The intensity of the color is proportional to the pH of the sample. This color is then matched against a standard color chart. The color chart equates particular colors to associated pH values. The pH can be determined by matching the colors from the chart to the color of the sample.

Procedure

The procedure for measuring pH is the same whether it is conducted in the field or lab. If you are using a “pocket pal” or color comparator, follow the manufacturer’s instructions. Use the following steps to determine the pH of your sample if you are using a meter.

1. Rinse the electrode

Rinse the electrode well with deionized water.

2. Place the electrode

Place the pH meter or electrode into a 7.0 buffer solution. Read and record the pH and adjust the pH meter to read 7.0.

3. Rinse the electrode

Rinse the electrode well with deionized water.

4. Place the electrode

Read and record the pH of the sample. Rinse the electrode well with deionized water

5. Place the electrode

Place the pH electrode into pH 7.0 buffer solution. The probe should be continuously soaked in the buffer which has the pH value closest to the suspected pH of the sample to be measured, typically 7.0 for most water treatment plants.

Note: Although the pH buffer or sample should be well mixed, excessive agitation can trap extra CO₂ and lower the pH of the solution being tested. Samples containing large amounts of dissolved CO₂ must be measured quickly since the CO₂ can escape into the atmosphere.

Turbidity - (Nephelometric Method)

Description

Turbidity is an expression of the optical properties of water which cause light to be scattered and absorbed rather than be transmitted in a straight path. The measurement of light scattered at a 90 degree angle is performed with a nephelometer. As the turbidity increases, the amount of light scattered will increase.

Sources of Turbidity

This turbidity is usually caused by finely divided suspended matter such as clay, silt, plankton and other organic and inorganic material.

Relationship to TSS

Attempts to correlate turbidity to suspended solids is impractical due to the fact that turbidity is related to particle size, shape and refractive index, as well as quantity.

Application

The procedure outlined below is general and can be applied to several brands of nephelometers. Be sure to read carefully the manufacturer's operation manual for your particular instrument.

Equipment

Several nephelometers and turbidimeters have been approved by the USEPA. Manufacturers include Hach, HF Instruments and Turner Designs.

Reagents

Due to the precision necessary for this instrument, it is recommended that standards be purchased rather than prepared. One standard must be on hand for each range used. These purchased standards are called Primary Standards.

Standards purchased after 1992 are very stable and will need to be replaced only when the glass shows any visual sign of scratching. Under normal use these standards should be replaced once a year.

Procedure

1. Calibrate the instrument

Be sure to check the manufacturer's instruction for warm-up time and calibration. A separate standard must be used to calibrate each scale used.

2. Collect sample

Collect a representative sample in a clean container. Samples may be stored up to 24 hours in the dark. The sample should be thoroughly mixed by shaking 15 times through a one-foot arc. The air bubbles should be allowed to dissipate before testing. Fill a sample cell to the line (about 15 mL), taking care to handle the sample cell by the top. Cap the cell.

3. Prepare the sample cell

Wipe the cell with a soft, lint-free cloth to remove water spots and fingerprints. Apply a thin film of silicone oil. Wipe with a soft cloth to obtain an even film over the entire surface.

4. Press: I/O

The instrument will turn on. Place portable instruments on a flat, sturdy surface. Do not hold the instrument while making measurements.
Insert sample cell

5. Insert the sample cell in the instrument cell compartment so the diamond or orientation mark aligns with the raised orientation mark in front of the cell compartment. Close the lid.

6. Select range to be measured

Select manual or automatic range selection by pressing the RANGE key. The display will show AUTO RNG when the instrument is in automatic range selection.

7. Signal averaging

Select signal averaging mode by pressing the SIGNAL AVERAGE key. The display will show SIG AVG when the instrument is using signal averaging. Use signal average mode if the sample causes a noisy signal (display changes constantly)

8. Press: READ

The display will show - - - - NTU, then the turbidity in NTU. Record the turbidity after the lamp symbol turns off. NTU = Nephelometric Turbidity Units

9. Discard sample and clean sample cell

Cold Water Problems

When the water is cold the heat from the turbidimeter may cause condensation to develop on the outside of the glass or gas bubbles to form on the inside of the glass. Either condition will give a false, high turbidity reading. It may be necessary to either read the results quickly or allow the sample to warm slightly before proceeding.

Bacteriological Sampling**Description**

Sampling of water distribution systems for bacterial contamination is an essential procedure for determination of water safety. It is, therefore, essential that the proper techniques be used to eliminate the possibility of contamination of the sample while it is being collected. In addition, a chlorine residual test must be performed at the same time as collecting a bacterial sample in order to comply with the Disinfectant/Disinfection Byproduct Rule.

Equipment

On-Site Testing When collecting samples for on-site testing, use sterilized borosilicate glass and wide-mouth bottles with ground glass stoppers and with a minimum capacity of 120 mL.

When to Sample

It is best to sample at the first of the month. This allows a chance for a second sample, should the first one be lost or damaged in shipping.

Mailing

When mailing samples the following sterilized container are acceptable:

- Heat-resistant polypropylene bottle with plastic screw-on top with 120 mL capacity.
- Borosilicate glass bottle with plastic screw-on top with 120 mL capacity.
- Polypropylene plastic “whirl pack” with 125 mL capacity.

A bacteriological sample that is mailed must get to the laboratory within 30 hours after collection or the laboratory will not test the sample.

Insulation

Samples mailed or shipped by plane must be insulated and protected from damage and freezing.

Special Instructions

Elimination of Chlorine Residual

Sample bottles and “whirl packs” prepared in commercial laboratories will have sodium thiosulfate in the bottle to eliminate chlorine in the sample. The sodium thiosulfate will appear as a white powder, crystal or clear liquid. This material should not be rinsed from the container.

Do Not Reuse Containers

If, for some reason, the sample could not be shipped, dump the sample but do not reuse the bottle.

Procedure

1. Select a sample site

Sample sites should be representative of the system. There are two types of sample locations:

- Those that are identified on the official sample plan.
- O & M sampling points, which include raw water, reservoirs, dead ends, low points in the system and new lines.

2. Select sample point

Routine sample points should have been identified as part of the development of the official sampling plan. The best sample points are faucets approximately 30 inches above the ground or from inside faucets that have none of the characteristics listed below. Sample points to be avoided are:

- Drinking fountains
- Lawn faucets
- Hoses
- Kitchen faucets
- Leaky faucets
- Aerators
- If a faucet with an aerator must be used, the aerator should be removed.

3. Sanitize faucet

Wipe the outside of the faucet with a mild chlorine solution.

4. Allow water to run 5 minutes

Or wait for a sufficient time to allow water from the distribution system to enter the sampling point.

5. Adjust the flow so that there will be no splashing

Splashing could cause some of the sodium thiosulfate to be displaced and could cause contamination to drip into the container.

6. Open container

Remove the lid or open the whirl pack. Keep the lid or stopper pointed down. Do not touch the inside of the container. Do not blow into the pack while open.

7. Fill the container

One inch of head space (air) should be left in sample bottles and 2 inches in the whirl pack. This improves mixing of the sample at the laboratory. A minimum of 100 mL is necessary for each sample. Since the container holds 120 mL, leaving an air space will still provide sufficient actual sample volume.

8. Seal container

Replace lid on bottles, pull wires of Whirl bag to flatten the top of the bag and whirl the bag over three times. Fold the wires over the bag.

9. Turn water “Off”

10. Pack for shipping

The container should be insulated to maintain the temperature of the sample. If shipping is delayed, refrigerate the sample. If the sample cannot be shipped on the same day it was collected, then discard and resample.

11. Collect a Chlorine Residual Sample

Collect a sample and test for free chlorine residual. Record the results.

Record Sample Data

Standard Sampling

The containers used by commercial and state laboratories are supplied with a standard sample data form. Completely fill out all portions of the form.

Essential Data

When a form is not available, as in a O & M sample, record the following information:

- Public water system number
- Sources of water, ground, surface and name of stream or lake, if surface
- Time collected
- Date sample collected
- Sample location
- Name of person collecting sample
- Was the water chlorinated?
- If the sample is mailed, time and date of mailing. If shipped by plane, date and flight number.

Copy of Data

When shipping a sample to a state or commercial laboratory, keep and file a copy of the data form that was sent with the sample.

Repeat Sample

When a positive coliform bacteriological sample is received Repeat Samples must be collected and tested in order to determine if contamination is actually present.

Procedure

Four (4) Repeat Samples must be collected. These Repeat Samples must be collected within 24 hours of the time the notification of the positive sample was received by the utility.

Where to Sample

1. One sample must be collected from the same tap used for the positive sample.
2. One sample within five (5) customer connects upstream of the original sample location.

3. One sample within five (5) customers connections downstream of the original sample location.
4. One sample from any other location.

Same Day Collection

All Repeat Samples must be collected on the same day.

When There is Only One Sample Point

If only one sampling point exists in the system then you may:

- Collect one Repeat Sample a day for four (4) consecutive days or,
- Collect one 400 mL sample on one day from the tap.

Following Month

During the month following a positive coliform sample five (5) routine samples must be collected from the system.

Removal From The Record

There are three ways of invalidating a coliform positive sample from a routine sample.

- A coliform positive sample may be removed from the utility records,(called invalidated) if all repeat samples taken from the original coliform positive tap are coliform positive and all repeat samples taken from the original tap are negative. This indicates a domestic plumbing problem.
- ADEC may invalidate a sample if the laboratory establishes that lab error caused the positive result.
- The department may also invalidate a positive result if the department's representative determines that the positive result was from a situation not representative of the water quality in the distribution system. You must work closely with the local ADEC Drinking Water Program representative in these situations.

Evaluating Coliform Testing Results

Violation

The water system is in violation of the drinking water regulations if any one of the following occurs:

- If the system fails to submit the required number of samples during any one month or if required repeat samples are not collected.
- A violation exists if the system receives more than one total coliform positive sample.

Acute Health Risk

Fecal Coliform

A test that indicates the presence of fecal contamination from warm-blooded animals including humans is the test for specific coliform bacteria called a fecal coliform or E. coli.

If a routine or repeat sample is total coliform positive, the laboratory will analyze the total coliform positive culture medium to determine if fecal coliform or E. coli are present.

When a system receives more than one positive coliform sample and the confirmation test for *E. coli* is also positive, the system is said to have an acute risk to human health.

Public Notification

General For all violations and situations, immediately consult with ADEC when you learn of the violation or situation. However must issue a public within the required time frame, even if you are unable to contact the primacy agency.

Non-Acute Violation

When a system has an MCL violation of a total coliform rule the public must be notified. This notification must take place within 30 days of the notification from the laboratory that a sample tested positive. This would occur if both the routine sample and repeat samples were positive to total coliform.

Acute Violation

When an acute violation is received public notification must take place within 24 hours of the receipt of the notification from the laboratory that a violation has occurred. This would occur if a water system had any:

- fecal coliform or *E. coli* positive REPEAT sample or
- has a fecal coliform or *E. coli* positive ROUTINE sample followed by a total coliform positive REPEAT sample, or
- failure to test for fecal coliform or *E. coli* when any repeat sample tests positive for coliform.

Quality Assurance/Quality Control

Quality assurance and quality control is often referred to as QA/QC. This term refers to a program that includes methods and procedures used in the lab to guarantee the validity of the numbers reported on monitoring reports. A QA/QC program is a required component of laboratory operation and may be part of the general procedure of the laboratory. These QA/QC programs outline specific procedures and activities required to meet federal quality control requirements and also to meet some states' laboratory accreditation processes.

Examples

Some examples of quality assurance/quality control tests include:

- Checking or calibrating lab thermometers to NIST15 standard thermometers
- Running duplicates of tests to determine if results are repeatable
- Checking or calibrating turbidimeters to NIST standards

Value of QA/QC

QA/QC tests are not only required, they are a good way to check and assure the validity of test results. Lab tests take a tremendous amount of time and energy to perform. Lab results are a basis for future construction and planning. Test results need an assurance and some controls to determine their accuracy.

Recordkeeping

Required monitoring

There are many levels of recordkeeping and criteria for both preparing and storing records. The following are a few basic considerations that can apply to all recordkeeping systems.

- **Blue Ink**
Keep all records in blue ink.
- **Do Not Alter**
Records should never be altered by erasing or white-out, but incorrect numbers should be crossed out and initialed by the operator.
- **Initials**
Initials or signatures should accompany all recorded readings. A listing of all initials or signatures should be on file on the premises.
- **Record Retention**
All records should be kept for at a specific period time. The time required for record retention depends on the type of constituent. For example, bacteriological tests must be kept for 5 years. Lead and Copper tests must be kept for 10 years.

Process Control Records

While these records are not influenced by the regulations as much as the water quality records, all records should be neatly prepared, signed, and kept on site for a reasonable length of time.

Conclusion

Relationship to Sample

The overall bottom line of laboratory testing is that the test and its results are no better than the sample, or the actual portion of water that is tested. It is critical that the sample be taken and the tests run on a sample that represents the proper overall objective.

Sample Types

If a “snapshot” of what is happening at a specific moment is desired, a grab sample should be collected and the appropriate test performed. Composite sample should only be taken if required by ADEC.