DOCUMENT RESUME

ED 221 399 SE 039 211

AUTHOR	Carnegie, John W.
TITLE	Sampling. Operational Control Tests for Wastewater Treatment Facilities. Instructor's Manual [and] Student Workbook.
INSTITUTION	Linn-Benton Community Coll., Albany, Oreg.
SPONS AGENCY	Office of Water Program Operations (EPA), Cincinnati, Ohio. National Training and Operational Technology Center.
PUB DATE	Nov 81
GRANT	EPA-900953010
NOTE	42p.; Slide/tape program which accompanies this module is also available from Linn-Benton Community College.
AVAILABLE FROM	Linn-Benton Community College, 6500 S.W. Pacific Blvd., Albany, OR 97321 ($1. student workbook, $2. instructor's guide).
EDRS PRICE	MF01 Plus Postage. PC Not Available from EDRS.
DESCRIPTORS	Instructional Materials; *Laboratory Procedures; Postsecondary Education; *Sampling; Teaching Guides; *Training Methods; *Waste Water; Water Quality; *Water Treatment

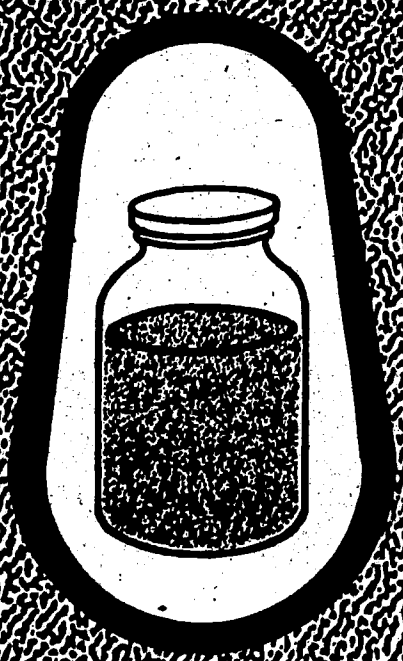
ABSTRACT

A brief overview of the basic concepts and philosophies for sampling water and waste water systems is presented in this module. The module is not intended to specify sampling procedures, frequencies, or locations for specific treatment facilities but rather to outline those general procedures which should be followed when sampling under most circumstances. The instructor's manual contains a statement of instructional goals, lists of instructor/student activities and instructional materials, lecture outline, narrative of the slide/tape program used with the module, overhead transparency masters, and student worksheet (with answers). The student workbook contains objectives, list of references, worksheet, and procedures demonstrating how to select a sample site and to collect and preserve a sample for the more common operational control tests. No special prerequisite skills are needed before the module is started. (Author/JN)

Operational Control Tests
for Wastewater Treatment Facilities

Sampling

Instructor's Manual



Linn-Benton Community College
Albany, Oregon

SAMPLING

Written By:
John W. Carnegie, Ph. D.
Linn-Benton Community College
Albany, Oregon

Instructional Design:
Priscilla Hardin
Corvallis, Oregon

Project Management:
John W. Carnegie, Ph. D.
Linn-Benton Community College
Albany, Oregon

Project Officer:
Lynn S. Marshall
United States Environmental Protection Agency
National Training and Operational Technology Center
Cincinnati, Ohio

Developed Under
EPA Grant #900953010
November, 1981

SAMPLING

CONTENTS

INSTRUCTIONAL GOALS

Upon completion of this lesson the student should be able to select a sample site and to collect and preserve a sample for the more common operational control tests.

INSTRUCTOR ACTIVITY

For best results follow this sequence:

1.	Review objectives with the students.	5 minutes
2.	Assign students the reading of the material in the Student Workbook.	10 mintues
3.	View and listen to the slide/tape program.	17 minutes
4.	Discuss major points listed below and in lecture outline. Show examples of sampling equipment.	15 minutes
5.	Assign worksheet.	10 minutes
6.	Correct worksheet.	5 minutes
7.	Demonstration and/or sampling "tour".	20 minutes

OTHER ACTIVITIES:

Since this is not really a test procedure it is difficult to practice the technique. However, a discussion of the major points that follow will reinforce the concepts of sampling:

1. Stress the importance of good sampling. "The best analytical test is no better than the sample that was collected".
2. Discuss the selection of sample sites. Encourage students to use their own plants as examples.
3. Discuss time and frequency of sampling in regard to objectives.
4. Show and discuss various sizes and types of sample containers and manual samplers.
5. Discuss storage and preservation. Point out table of preservation procedures in Student Supplementary Materials. Also refer to Standard Methods.

Using overheads in Appendix A, go over sample calculations for ml of sample to collect for flow proportional composite samples.

If a plant is close, take the group on a walking tour of the process units, stopping to point out good places to sample and areas to avoid. Encourage students to volunteer solutions.

If possible, demonstrate or have available for display automatic sampling equipment. Manufacturer's representatives are often available to show their equipment.

STUDENT ACTIVITY

1. Read objectives.
2. Read procedures material.
3. Listen to and view slide/tape program.
4. Take part in class discussion.
5. Complete worksheet.
6. Take part in tour of plant.

INSTRUCTIONAL MATERIALS LIST

1. Instructor's Guide for Sampling
2. Student Workbook for Sampling
3. Procedures Manual for Operational Control Tests
4. 35mm projector
5. Cassette tape player with automatic synchronization
6. Overhead projector
7. Projector screen
8. Examples of sample jars and samplers
9. Copy of "Standard Methods"
10. Automatic sampling equipment (optional)

LECTURE OUTLINE

SAMPLING

I. What is a sample?
   A. A small representative of the whole.
II. Why do you want to sample?
   A. To satisfy permit requirements.
   B. Operational control of plant or individual systems.
   C. Permanent records for expansion.
III. What is a representative sample?
IV. What kind of samples are there?
   A. Grab
   B. Composite
   C. Flow Proportional
V. Where do you take samples?
   A. Always first ask "What do I want to know?"
   B. Established routine points
      1. Influent/effluent
      2. Basins
      3. Identify points
      4. Special situations
   C. Some general rules:
      1. In a well mixed area
      2. Avoid sediment and floating material
VI. How do you take a sample?
   A. General
      1. Sample containers
         a. adequate size
         b. type
         c. clean or sterilized
         d. pre-treatment
      2. Manual - dippers

3. Automatic
   a. portable
   b. built-in

B. Grab Sample

C. Composite

D. Flow Proportional

E. Records to keep during sampling

F. Preservation and storage times

8

SAMPLING

NARRATIVE

Slide #

1. This lesson on sampling covers sampling in general, but gives specific information related to sampling for operational control of wastewater treatment systems.

2. The lesson was written by Dr. John W. Carnegie and Priscilla Hardin did the instructional design. Dr. Carnegie was also the project director.

3. In this lesson we will discuss "What is a sample?", why take samples, and look at the factors that affect representative samples.

4. A sample is a small part of a larger quantity taken to study the characteristics and properties of the larger quantity. A sample must be representative; that is, the sample must resemble the larger quantity as closely as possible. Since it is not possible to test the whole volume of water in a treatment system, representative samples must be collected and analyzed.

5. Why do we want to sample? Samples must be taken from plant effluents for analysis to assure compliance with discharge permits.

6. Samples are taken throughout the plant to help in the operational control of the system and individual treatment components.

7. Samples are also taken throughout the plant for analysis by a wide range of tests to build and maintain permanent records which may be used to justify expansion or process modification.

8. The factors which affect representativeness are: type of sample, sample site selection, sampling frequency, sampling devices, collection techniques, sample handling, preservation and identification.

9. Let's take a closer look at each of these factors starting with sample type.

10. There are three basic types of samples: grab, composite, and flow proportional composite.

11. A grap sample is one sample taken at a particular place at a particular time, and really represents only that place and time.

12. Grab samples are useful where the composition of the source is relatively constant. Grab samples taken at intervals and analyzed individually can be used to monitor fluctuations.

13. If a source is large, such as a lagoon, grap samples taken at different locations can be used to determine uniformity of the source.

14. Composite samples are grab samples mixed together and analyzed as one. They may be a set of grab samples collected at the same point but at a different time.

15. Or they may be a set collected at different points at about the same time. In either case the composite sample is useful for observing average concentrations and, therefore, represent a savings in laboratory time and expense.

16. Flow proportional samples are composite samples made up of grab samples whose volume is dependent upon plant flow at the time of sampling. Since composites are most commonly used for averaging, more statistical weight must be given to samples taken during high flow periods. This is accomplished by using larger volume for higher flow time than for lower flow times.

17. The selection of the sampling site will influence the results of sampling.

18. It is important to ask yourself, "What do I want to learn from the sample?" before selecting a site. Different objectives will mean different sample site selections.

19. Probably the most common objective is to monitor process status and treatment efficiency for routine operations. For these purposes sample sites should be established which are used routinely. Establish routine sample sites and identify and mark them clearly. Routine sample sites might include plant influent and effluent, basins, sludge lines, and before and after individual unit process.

20. Special situations arise where your objectives may require special sample points not routinely used. Describe the special sampling points clearly on your sheet so that the results can be interpreted accurately.

21. Regardless of the site selected, be sure to choose a well mixed and representative area

22. and be sure to avoid scum and deposits.

23. The time and frequency that a sample is taken is also critical. The objectives must be considered in determining the time and frequency of sampling.

24. For example, grab samples used for chlorine residual should be taken at peak flow.

25. Grab samples used for determination of DO, pH, and fecal coliforms, for example, should be taken at minimum and maximum flow.

26. Flow proportional composite samples should be composited over a 24 hour period taking a grab once per shift or as often as once per hour depending on the specific objectives for sampling.

27. Let's turn our attention to sampling devices. The equipment used to collect samples falls into two categories: manual and automatic.

28. Manual devices consist of a container of some sort that can be put into the source in a safe and convenient manner to obtain the desired volume. Such devices may be homemade or commercial. They may be attached to a pole or a rope. Special devices are available to allow sampling below the liquid surface. Regardless of the type, they should be clean. Samples for most operational tests can be collected in metal or plastics. Some tests, for metals or organics, may require glass sampling containers.

29. Automatic sampling devices may be built into the plant or portable. Automatic samples are timed pumping devices that deliver selected volumes at selected times to composite sample containers. The composite may be stored near the sample site or in some cases, pumped to the laboratory refrigerator. Portable units store the sample in the device.

30. Automatic samplers save time and money, but have inherent problems because they are automatic. Long delivery lines and poor storage are two potential problems.

31. Now let's take a look at collection techniques; how would you go about actually collecting a sample.

32. First, you must have a sample container to transport your sample from the sample site to the laboratory. Three items should be considered here: type, size and cleanliness.

33. For most purposes, plastic sample jars with wide mouth plastic caps are acceptable. However, if sterilization is required or if tests such as organic analysis are to be run, glass may be required. Check the specifications for sample collection for each analysis you intend to run.

34. Obviously, you must have enough volume to perform the analysis. Jar tests may require several liters, while a gas chromatograph may only require a fraction of a milliliter. Generally, a couple liters is adequate and samples of less than 100 ml are not recommended.

35. Most samples may be collected in thoroughly washed and distilled-water rinsed containers. For bacteriological analysis, however, sample containers must also be sterilized. Occasionally, preservation chemicals must be added to sample containers before collecting the sample, as is done with sodium thiosulfate prior to collection of chlorinated effluent for biological analysis.

36. A convenient sample carry tray or box will make the process easier and avoid spills and breakage.

37. So, away we go, to collect a grap sample. Let's take a sample from an aeration basin at a routine sampling site.

38. Here's the site and the sampler.

39. Make sure the sampler is clean. Rinse it a couple of times if necessary.

40. Lower the sampler into the basin, being careful to avoid foam or other floating devices.

41. Unless you have added some chemicals to the sample containers, it is a good idea to rinse them once with some sample.

42. Then go ahead and fill the container, but leave a little air space so the lab technician can mix the sample prior to running the analysis.

43. Rinse the sampler and replace it.

44. Be sure the sample container is clearly identified.

45. There are several other items to check and record before we're done. The date, time, location, temperature, and weather conditions should be recorded. On site measurements such as dissolved oxygen, pH, and temperature should also be made and recorded at this time, if they are needed. Any unusual conditions observed should also be noted.

46. The procedure for collecting a flow proportional composite sample is the same as for a grab except you must calculate and collect a specific volume. In order to do this you must know 4 things:

47. First, you need to know the total amount of sample desired over the entire composite sampling period.

48. Second, you need to know the number of sampling times desired over the composite sampling period.

49. Third, you need to know the flow rate at the time of sampling.

50. And finally, you need to know the average daily flow rate.

51. The volume to be collected at any one of the sampling times is then calculated by this formula:

$$\text{ml of sample to collect} = \frac{\text{Total amount of sample ml} \times \text{Flow rate at sampling, MGD}}{\text{Number of sampling times} \times \text{Ave. flow rate, MGD}}$$

Be sure to use the same units for flow and volume in both cases. Refer to the student materials for an example problem.

52. After making this calculation proceed with the collection as before. The individual samples may be stored in a refrigerator and mixed just prior to analysis or mixed immediately after collection by adding to a larger container in the refrigerator.

53. Automatic sampling requires careful selection of collection site, frequency and volume. Be sure to secure the inlet tubing so that it stays in the proper position.

54. If you are using a portable unit, check the power source, the sampling frequency and volume settings; make sure the sample container is clean and properly positioned in the sampler and that the discharge tube is in the container. It is a good idea to run the sampler on manual to make sure everything is okay.

55. If a built-in system is being used, check the pump periodically and check the sampling frequency and volume settings. Check the sample container for cleanliness. Check that the refrigerator is cooling and that the discharge tube is in the container.

56. Now, we must consider handling and preservation.

57. Samples may be preserved by refrigeration or the addition of chemicals. Standard Methods is your best reference for preservation techniques.

58. The best rule is to take the sample to the lab immediately and do the analysis as soon as possible.

59. If in doubt, always store the sample in the refrigerator.

60. In this lesson we have learned that a sample is a representative of a large part of the plant.

61. We have stated that samples are taken to meet discharge permit requirements, to aid in operational control, and to establish permanent records.

62. We looked at all of the factors affecting the collection of a good sample.

63. And we have summarized some suggestions for preservation and handling of samples. Remember: Your lab test results are only as good as your sampling technique.

APPENDIX A

Use the overheads 1, 2, and 3 to explain the calculation for desired sample volume with flow proportional composite samples. Work slowly through the sample problems on the overheads. Stress the following points:

1. You must decide ahead of time how much total composite volume you need and how many grab samples you will take to create the composite.

2. The flow rate at sampling is instantaneous flow at the time of sampling.

3. Average flow rate is average daily flow rate.

4. Units must be consistant; i.e. use ml for volumes, MGD for flows.

Assign a second problem if time permits. Suggested problem: (use overhead #4)

You wish to collect 2 liters over a 12 hr period sampling every 2 hours. The average flow rate is 8.0 MGD. How many ml would you collect at the second sampling time if the flow was then 5.5 MGD?

SOLUTION:

$$\text{Vol. to Collect} = \frac{2000 \text{ ml} \times 5.5 \text{ MGD}}{6 \times 8.0 \text{ MGD}}$$

$$= 229.2 \text{ ml or } 230 \text{ ml}$$

FLOW PROPORTIONAL COMPOSITE

$$\text{VOL. to collect} = \frac{\text{TOTAL VOL.}}{\text{No. of samples}} \times \frac{\text{FLOW, at sampling}}{\text{AVG. FLOW}}$$

SAMPLE PROBLEM #1

IF:

You need 2500 ml total sample volume

You collect 4 samples

The avg. flow rate is 2.5 MGD

The flow at sampling is 3.0 MGD

FIND:

Volume of sample to collect

$$\text{VOL., to collect} = \frac{\text{TOTAL VOL.}}{\text{No. of samples}} \times \frac{\text{FLOW, at sampling}}{\text{AVG. FLOW}}$$

$$\text{VOL., ml} = \frac{2500\ \text{ml}}{4} \times \frac{3.0\ \text{MGD}}{2.5\ \text{MGD}}$$

$$= 750\ \text{ml}$$

SAMPLE PROBLEM #2

You wish to collect 2 liters over a 12 hr. period sampling every 2 hrs. The average flow rate is 8.0 MGD. How many mi would you collect at a sample time if the flow was 5.5 MGD?

SAMPLING

WORKSHEET

Directions: Place an "X" by the best answer or answers. In some cases there may be more than one correct answer.

1. A sample is:
   a) X a small part of a larger quantity.
   b) X representative of the sampled body.
   c) X representative of the whole only at the point and time of sampling.
   d) ____ always taken in a quiet, deep part of the basin.
   e) ____ None of the above.

2. Which of the following is <u>not</u> a good reason for sampling?
   a) ____ To satisfy permit requirements.
   b) ____ To aid in operational control.
   c) X To fill in the blanks in the data sheet.
   d) ____ To provide background data for plant expansion.
   e) ____ All of the above are good reasons.

3. The three major types of samples are:
   a) X flow proportional composite
   b) X composite
   c) ____ bucket
   d) X grab
   e) ____ portable

4. The three cardinal rules for sampling, CAP, stand for:
   a) ____ caution, action, preservation.
   b) X cleanliness, accuracy, preservation.
   c) ____ cleanliness, accountability, precision.
   d) ____ consideration, activity, purpose
   e) ____ control, accuracy, perseverance.

5. Two types of automatic samplers are:

a) X portable.

b) _____ grab.

c) _____ bucket.

d) X built-in to plant.

e) _____ suction.

6. Samples should generally be refrigerated as soon after collection as possible at:

a) _____ 0° C.

b) _____ 0° F.

c) X 3° - 4° C.

d) _____ 10° - 15° C.

e) _____ -25° C.

7. The best reference for recommended preservation methods is:

a) X Standard Methods.

b) _____ Sacramento State Manual.

c) _____ Linn-Benton Water/Wastewater Lab Manual

d) _____ WPCF MOP 11

e) _____ Plant O and M Manual

8. Place an "X" by those items which should appear on a good sample label.

a) X air temperature

b) X water temperature

c) X sampler's initial

d) X date

e) _____ MLSS

f) X time

g) X sample location

h) _____ BOD

i) _____ fecal coliform count

9. A plant has an average daily flow of 2.5 MGD. You wish to collect a flow proportional sample of 3 liters total volume at 8 times throughout the day. The flow at one of the times of sampling was 2.0 MGD. What should your sample volume be for that time?

a)_____ 30 ml

b)_____ 3 liters

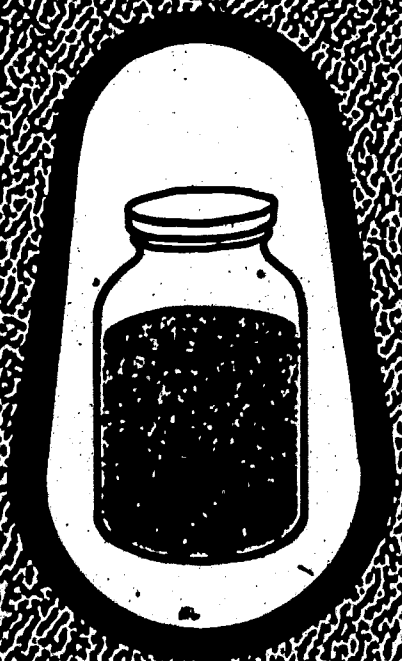
c)_____ 470 ml

d)___X___ 300 ml

e)_____ 150 ml

Operational Control Tests

for Wastewater Treatment Facilities



Sampling

Student Workbook

Linn-Benton Community College
Albany, Oregon

SAMPLING

Written By:
John W. Carnegie, Ph. D.
Linn-Benton Community College
Albany, Oregon

Instructional Design:
Priscilla Hardin
Corvallis, Oregon

Project Management:
John W. Carnegie, Ph. D.
Linn-Benton Community College
Albany, Oregon

Project Officer:
Lynn S. Marshall
United States Environmental Protection Agency
National Training and Operational Technology Center
Cincinnati, Ohio

Developed Under
EPA Grant #900953010
November, 1981

SAMPLING

CONTENTS

INTRODUCTION

This is a brief over-view of the basic concepts and philosophies for sampling in water and wastewater systems. This module is not intended to specify sampling procedures, frequencies, or locations for specific treatment facilities, but rather to outline those general procedures which should be followed when sampling under most all circumstances.

OBJECTIVES

Upon completion of this module you should be able to do the following:

1. Define the term sample.
2. Recall the reason why you might want to sample.
3. Recall the three major types of samples.
4. State the three cardinal rules for sampling.
5. Identify two types of automatic samplers.
6. Calculate the volume of sample required at any time to make a flow proportional composite sample.
7. Recall the recommended refrigeration temperature.
8. Give the best reference for preservation methods.
9. List the items that should appear on a sample label.

PREREQUISITE SKILLS

None

REFERENCE LIST

Standard Methods for the Examination of Water and Wastewater, 15th edition, American Public Health Assn, American Water Works Assn. and Water Pollution Control Federation, Washington, D.C., 1981.

Methods for Organic Pesticides in Water and Wastewater, EPA, Cincinnati, 1971.

Manual of Methods for Chemical Analysis of Water and Wastes, EPA-625/6-74-003, Cincinnati, 1974.

National Handbook of Recommended Methods for Water - Data Acquisition, Office of Water Data Coordination, USGS, U.S. Department of the Interior, Reston, VA, 1977.

SAMPLING

INTRODUCTION

Sampling is a critical part of water testing. It also 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 is performed carefully and accurately, the result may be incorrect and meaningless if the sample is taken in such a way as to be nonrepresentative of the water source from which it was taken.

PRINCIPLES OF SAMPLING

Water and wastewater samples are routinely 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, the following principles will apply. The Three Cardinal Rules for sampling are:

1. CLEANLINESS

   of all containers; including caps, and measuring devices with which the sample comes in contact.

2. ACCURACY

   of all records. The sample label should note the type of sample, sources, location of source point, the date and hour sampled, name of sample taker, the temperature of the sample, and recent weather conditions.

3. 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. Chemical degradation can also occur if samples are not properly preserved.

Other items of importance:

1. SELECT REPRESENTATIVE SAMPLING LOCATIONS.

   The sample should be taken where the flow is well mixed.

2. OBTAIN AN ADEQUATE NUMBER AND VOLUME OF SAMPLES.

3. EXCLUDE ~~LARGE~~ PARTICLES

which may be in the body of water.

4. NO DEPOSITS, GROWTHS OR FLOATING MATERIAL

that may have accumulated at the sampling point should be included.

5. ASEPTIC HANDLING OF BACTERIOLOGIC SAMPLES

Avoid contamination from skin, clothing, equipment, water, and adjacent surfaces.

6. RECORD NECESSARY SAMPLING DATA.

7. ALWAYS MIX THE SAMPLE

before removing a portion.

8. SAMPLES SHOULD BE TESTED AS SOON AS POSSIBLE -

always within the permissible time interval after sampling.

TYPES OF SAMPLES

1. GRAB SAMPLES

A "grab" sample consists of a portion of the flow taken at one particular time. Grab samples are taken because they are required or because there is a lack of time to catch composite samples. For some tests, grab samples must be used. Tests such as residual chlorine, dissolved oxygen, and pH are determined from grab samples as a portion of the flow which cannot be mixed. For some tests grab samples can be used because the quality of the component to be sampled remains uniform for a period of a day or longer. An example is a digester sample.

2. 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.

3. 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 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. Biochemical oxygen demand, settleable solids, and suspended solids tests are usually run on composite sample. The effects of intermittent changes in strength and flow are eliminated. The portion collected should be obtained with sufficient frequency to obtain average results. The rate of flow must be measured when each portion is taken and the volume of the portion adjusted to the flow at the particular time of sample. Samples may be composited either by mechanical samplers or by hand.

Use the following formula to determine the volume of sample to be taken at each sampling interval to obtain a weighted composite sample.

$$\frac{\text{Total Sample Volume}}{\text{\# of Sampling Times}} \times \frac{\text{Flow Rate at Sampling}}{\text{Average Flow Rate}} = \text{ml Sample at Sampling Times}$$

SAMPLE PRESERVATION

Samples should be chilled to 3° - 4° C. immediately. This is particularly true for the biochemical oxygen demand test and all biological tests. Samples for certain tests may require some type of chemical preservative. It is not possible to preserve samples for other tests such as dissolved oxygen and temperature. Before proceeding on an analytical test, check the sample preservation method as recommended in Standard Methods.

SAMPLING DATA

No sample should be tested unless it has been properly labeled and fully documented on the sampling data sheet. The record should contain sufficient information to provide positive identification of the sample at a later date, as well as the name of the sample collector, the date, time, and exact location the sample was taken from, the water temperature, and any data that may be useful; such as, weather, air temperature, water level, stream flow, etc. Fix sampling locations by detailed descriptions. These descriptions should be clear enough for other persons to follow.

SAMPLE PRESERVATION *

Complete and unequivocal preservation of samples, either domestic sewage, industrial wastes, or natural waters, is a practical impossibility. Regardless of the nature of the sample, complete stability for every constituent can never be achieved. At best, preservation techniques can only retard the chemical and biological changes that inevitably continue after the sample is removed from the parent source. The changes that take place in a sample are either chemical or biological. In the former case, certain changes occur in the chemical structure of the constituents that are a function of physical conditions. Metal cations may precipitate as hydroxides or form complexes with other constituents; cations or anions may change valence states under certain reducing or oxidizing conditions; other constituents may dissolve or volatilize with the passage of time. Metal cations may also adsorb onto surfaces (glass, plastic, quartz, etc.), such as, iron and lead. Biological changes taking place in a sample may change the valence of an element or a radical to a different valence. Soluble constituents may be converted to organically bound materials in cell structures, or cell lysis may result in release of cellular material into solution. The well known nitrogen and phosphorus cycles are examples of biological influence on sample composition.

Methods of preservation are relatively limited and are intended generally to (1) retard biological action, (2) retard hydrolysis of chemical compounds and complexes and (3) reduce volatility of constituents.

Preservation methods are generally limited to pH control, chemical addition, refrigeration, and freezing. Table 1, shows the various preservatives that may be used to retard changes in samples.

*Manual of Methods for Chemical Analysis of Water and Wastes, EPA - 625/6-74-003, Cincinnati, 1974.

TABLE 1

Preservative	Action	Applicable to:
$HgCl_2$	Bacterial Inhibitor	Nitrogen forms, Phosphorus forms
Acid (HNO_3)	Metals solvent, prevents precipitation	Metals
Acid (H_2SO_4)	Bacterial Inhibitor	Organic samples (COD, oil & grease organic carbon)
	Salt formation with organic bases	Ammonia, amines
Alkali (NaOH)	Salt formation with volatile compounds	Cyanides, organic acids
Refrigeration	Bacterial Inhibitor	Acidity-alkalinity, organic materials, BOD, color, odor, organic P, organic N, carbon, etc., biological organism (coliform, etc.)

In summary, refrigeration at temperatures near freezing or below is the best preservation technique available, but it is not applicable to all types of samples.

The recommended choice of preservatives for various constituents is given in Table 2. These choices are based on the accompanying references and on information supplied by various Regional Analytical Quality Control Coordinators.

TABLE 2

RECOMMENDATION FOR SAMPLING AND PRESERVATION OF SAMPLES ACCORDING TO MEASUREMENT (1)

Measurement	Vol. Req. (ml)	Container	Preservative	Holding Time(6)
Acidity	100	P, G(2)	Cool, 4°C	24 Hrs.
Alkalinity	100	P, G	Cool, 4°C	24 Hrs.
Arsenic	100	P, G	HNO_3 to pH <2	6 Mos.
BOD	1000	P, G	Cool, 4°C	6 Hrs.(3)
Bromide	100	P, G	Cool, 4°C	24 Hrs.
COD	50	P, G	H_2SO_4 to pH <2	7 Days
Chloride	50	P, G	None Req.	7 Days
Chlorine Req.	50	P, G	Cool, 4°C	24 Hrs.
Color	50	P, G	Cool, 4°C	24 Hrs.
Cyanides	500	P, G	Cool, 4°C NaOH to pH 12	24 Hrs.
Dissolved Oxygen				
Probe	300	G only	Det. on site	No Holding
Winkler	300	G only	Fix on site	No Holding

TABLE 2 (Continued)

Measurement	Vol. Req. (ml)	Container	Preservative	Holding Time(6)
Hydrolyzable	50	P, G	Cool, 4°C H_2SO_4 to pH <2	24 Hrs.(4)
Total	50	P, G	Cool, 4°C	24 Hrs. (4)
Total, Dissolved	50	P, G	Filter on site Cool, 4°C	24 Hrs.(4)
Residue				
Filterable	100	P, G	Cool, 4°C	7 Days
Non-Filterable	100	P, G	Cool, 4°C	7 Days
Total	100	P, G	Cool, 4°C	7Days
Volatile	100	P, G	Cool, 4°C	7 Days
Settleable Matter	1000	P, G	None Req.	24 Hrs.
Selenium	50	P, G	HNO_3 to pH <2	6 Mos.
Silica	50	P only	Cool, 4°C	7 Days
Specific Conductance	100	P, G	Cool, 4°C	24 Hrs. (5)
Sulfate	50	P, G	Cool, 4°C	7 Days

xi

TABLE 2 (Continued)

Measurement	Vol. Req. (ml)	Container	Preservative	Holding Time(6)
Sulfide	50	P, G	2 ml zinc acetate	24 Hrs.
Sulfite	50	P, G	Cool, 4°C	24 Hrs.
Temperature	1000	P, G	Det. on site	No Holding
Threshold Odor	200	G only	Cool, 4°C	24 Hrs.
Turbidity	100	P, G	Cool, 4°C	7 Days

1. More specific instructions for preservation and sampling are found with each procedure as detailed in this manual. A general discussion on sampling water and industrial wastewater may be found in ASTM, Part 23, p. 72-91 (1973).

2. Plastic or Glass

3. If samples cannot be returned to the laboratory in less than 6 hours and holding time exceeds this limit, the final reported data should indicate the actual holding time.

4. Mercuric chloride may be used as an alternate preservative at a concentration of 40 mg/l, especially if a longer holding time is required. However, the use of mercuric chloride is discouraged whenever possible.

5. If the sample is stabilized by cooling, it should be warmed to 25°C for reading, or temperature correction made and results reported at 25°C.

6. It has been shown that samples properly preserved may be held for extended periods beyond the recommended holding time.

TABLE 2 (Continued)

Measurement	Vol. Req. (ml)	Container	Preservative	Holding Time(6)
Fluoride	300	P, G	Cool, 4°C	7 Days
Hardness	100	P, G	Cool, 4°C	7 Days
Iodide	100	P, G	Cool, 4°C	24 Hrs.
MBAS	250	P, G	Cool, 4°C	24 Hrs.
Metals				
Dissolved	200	P, G	Filter on site HNO_3 to pH <2	6 Mos.
Suspended			Filter on site	6 Mos.
Total	100		HNO_3 to pH <2	6 Mos.
Mercury				
Dissolved	100	P, G	Filter HNO_3 to pH <2	38 Days (Glass) 13 Days (Hard Plastic)
Total	100	P, G	HNO_3 to pH <2	38 Days (Glass) 13 Days (Hard Plastic)

TABLE 2 (Continued)

Measurement	Vol. Req. (ml)	Container	Preservative	Holding Time(6)
Nitrogen				
Ammonia	400	P, G	Cool, 4°C H_2SO_4 to pH <2	24 Hrs.(4)
Kjeldahl	500	P, G	Cool, 4°C H_2SO_4 to pH <2	24 Hrs.(4)
Nitrate	100	P, G	Cool, 4°C H_2SO_4 to pH <2	24 Hrs.(4)
Nitrite	50	P, G	Cool, 4°C	24 Hrs. (4)
NTA	50	P, G	Cool, 4°C	24 Hrs.
Oil & Grease	1000	G only	Cool, 4°C H_2SO_4 to pH <2	24 Hrs.
Organic Carbon	25	P, G	Cool, 4°C H_2SO_4 to pH <2	24 Hrs.
pH	25	P, G	Cool, 4°C Det. on site	6 Hrs.(3)
Phenolics	500	G only	Cool, 4°C H_3PO_4 to pH <4 1.0 g $CuSO_4$/l	24 Hrs.
Phosphorus				
Ortho-phosphate, Dissolved	50	P, G	Filter on site Cool, 4°C	24 Hrs.(4)

SAMPLING

WORKSHEET

Directions: Place an "X" by the best answer or answers. In some cases there may be more than one correct answer.

1. A sample is:
   a)_____ a small part of a larger quantity.
   b)_____ representative of the sampled body.
   c)_____ representative of the whole only at the point and time of sampling.
   d)_____ always taken in a quiet, deep part of the basin.
   e)_____ None of the above.

2. Which of the following is <u>not</u> a good reason for sampling?
   a)_____ To satisfy permit requirements.
   b)_____ To aid in operational control.
   c)_____ To fill in the blanks in the data sheet.
   d)_____ To provide background data for plant expansion.
   e)_____ All of the above are good reasons.

3. The three major types of samples are:
   a)_____ flow proportional composite
   b)_____ composite
   c)_____ bucket
   d)_____ grab
   e)_____ portable

4. The three cardinal rules for sampling, CAP, stand for:
   a)_____ caution, action, preservation.
   b)_____ cleanliness, accuracy, preservation.
   c)_____ cleanliness, accountability, precision.
   d)_____ consideration, activity, purpose
   e)_____ control, accuracy, perseverance.

5. Two types of automatic samplers are:

a)_____ portable.

b)_____ grab.

c)_____ bucket.

d)_____ built-in to plant.

e)_____ suction.

6. Samples should generally be refrigerated as soon after collection as possible at:

a)_____ 0° C.

b)_____ 0° F.

c)_____ 3° - 4° C.

d)_____ 10° - 15° C.

e)_____ -25° C.

7. The best reference for recommended preservation methods is:

a)_____ Standard Methods.

b)_____ Sacramento State Manual.

c)_____ Linn-Benton Water/Wastewater Lab Manual

d)_____ WPCF MOP 11

e)_____ Plant O and M Manual

8. Place an "X" by those items which should appear on a good sample label.

a)_____ air temperature

b)_____ water temperature

c)_____ sampler's initial

d)_____ date

e)_____ MLSS

f)_____ time

g)_____ sample location

h)_____ BOD

i)_____ fecal coliform count

9. A plant has an average daily flow of 2.5 MGD. You wish to collect a flow proportional sample of 3 liters total volume at 8 times throughout the day. The flow at one of the times of sampling was 2.0 MGD. What should your sample volume be for that time?

a)_____ 30 ml

b)_____ 3 liters

c)_____ 470 ml

d)_____ 300 ml

e)_____ 150 ml