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Collection of Waste Water Sample and Preservation Techniques

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Abstract

The aim of sampling is to collect a representative sample. Representative sample means a sample in which relative proportions or concentration of all pertinent components will be the same as in the material being sampled. The sample volume shall be optimal small enough that it can be transported and large enough for analytical purposes. Because of the increasing importance on verifying the accuracy and representatives of data, greater emphasis is placed on proper sample collection, tracking and preservation techniques.

Introduction

When samples are collected from a river, stream, lakes, and reservoirs observed results may vary with seasonal stratification, diurnal variations, rainfall, runoff, wind depth, stream flow, and distance from each shore. Selection of the number and distribution of sites at which samples should be collected depends on study objectives, stream characteristics, available equipment, and other factors. If equipment is available, take an integrated sample from top to bottom in the middle of the main channel of the stream or from side to side at mid-depth. If only grab or catch samples can be collected, preferably take them at various points of equal distance across the stream; if only one sample can be collected, take it in the middle of the main channel of the stream and at mid-depth. Always prohibit eating, drinking, or smoking near samples, sampling locations, and in the laboratory. Keep sparks, flames, and excessive heat sources away from samples and sampling locations. If flammable compounds are suspected of known to be present and samples are to be refrigerated, use only specially designed *explosion-proof* refrigerators. Label adequately any sample known or suspected to be hazardous because of flammability, corrosivity, toxicity, oxidizing chemicals, or radioactivity, so that appropriate precautions can be taken during sample handling, storage, and disposal.

Collection of Wastewater Sample Types of Samples

1. Grab Samples

Grab samples are single samples collected at a specific spot at a site over a short period of time (typically seconds or minutes). Thus, they represent a "snapshot" in both space and time of a sampling area. Discrete grab samples are taken at a selected location, depth, and time. Depth integrated grab samples are collected over a predetermined part of the entire depth of a water column, at a selected location and time in a given body of water.

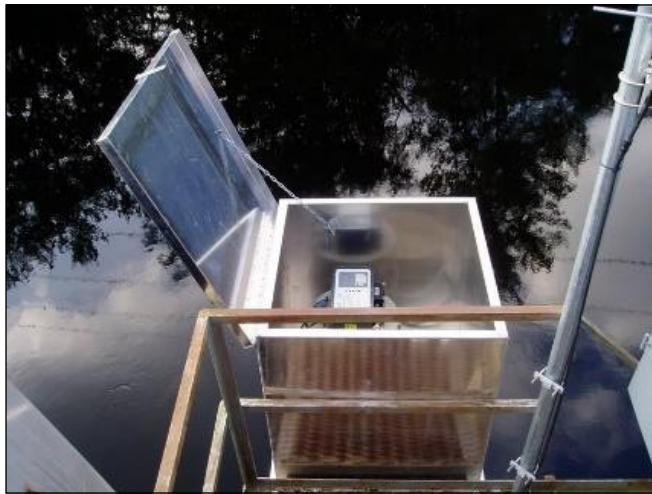


Figure 1: Grab samples



Figure 2: Catch samples

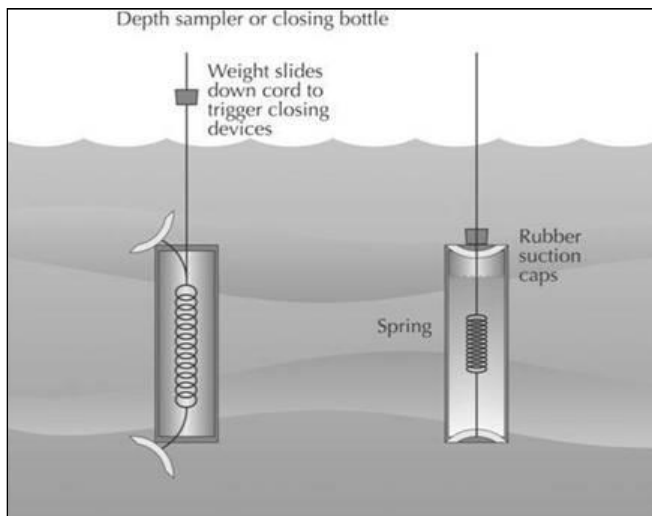


Figure 3: Composite samples

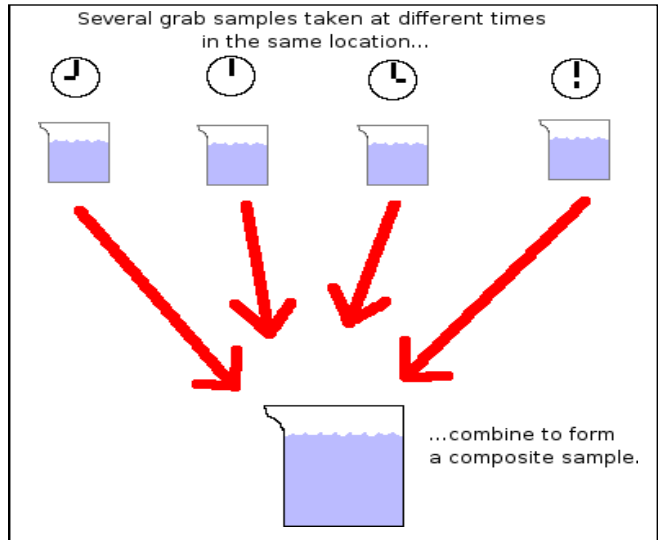


Figure 4: Integrated samples

2. Composite Samples

Composite samples should provide a more representative sampling of heterogeneous matrices in which the concentration of the analytes of interest may vary over short periods of time and/or space. Composite samples can be obtained by combining portions of multiple grab samples or by using specially designed automatic sampling devices.

Advantages of composite samples include reduced costs of analyzing a large number of samples, more representative samples of heterogeneous matrices, and larger sample sizes when amounts of test samples are limited. Disadvantages of composite samples include loss of analyte relationships in individual samples, potential dilution of analytes below detection levels, increased potential analytical interferences, and increased possibility of analyte interactions. In addition, use of composite samples may reduce the number of samples analyzed below the required statistical need for specified data quality objectives or project specific objectives.

Do not use composite samples with components or characteristics subject to significant and unavoidable changes during storage. Analyze individual samples as soon as possible after collection and preferably at the sampling point. Examples are dissolved gases, residual chlorine, soluble sulfide, temperature, and pH. Changes in components such as dissolved oxygen or carbon dioxide, pH, or temperature may produce secondary changes in certain inorganic constituents such as iron, manganese, alkalinity, or hardness.

3. Integrated (discharge-weighted) Samples

For certain purposes, the information needed is best provided by analyzing mixtures of grab samples collected from different points simultaneously, or as nearly so as possible, using discharge-weighted methods such as equal width increment (EWI) or equal discharge-increment (EDI)

procedures and equipment. An example of the need for integrated sampling occurs in a river or stream that varies in composition across its width and depth. To evaluate average composition or total loading, use a mixture of samples representing various points in the cross-section, in proportion to their relative flows. The need for integrated samples also may exist if combined treatment is proposed for several separate wastewater streams, the interaction of which may have a significant effect on treatability or even on composition.

Sampling Methods

1. Manual Sampling

Manual sampling involves minimal equipment but may be unduly costly and time-consuming for routine or large-scale sampling programs. It requires trained field technicians and is often necessary for regulatory and research investigations for which critical appraisal of field conditions and complex sample collection techniques are essential.

2. Automatic Sampling

Automatic samplers can eliminate human errors in manual sampling, can reduce labor costs, may provide the means for more frequent sampling, and are used increasingly. Be sure that the automatic sampler does not contaminate the sample. For example, plastic components may be incompatible with certain organic compounds that are soluble in the plastic parts or that can be contaminated (e.g., from phthalate esters) by contact with them. Program an automatic sampler in accordance with sampling needs.

3. Sorbent Sampling

Use of solid sorbents, particularly membrane-type disks, is becoming more frequent. These methods offer advantages of rapid, inexpensive sampling if the analytes of interest can be adsorbed and desorbed efficiently and the water matrix is free of particulates that plug the sorbent.

Sample Containers

Containers typically are made of plastic or glass, but one material may be preferred over the other. For example, silica, sodium, and boron may be leached from soft glass but not plastic, and trace levels of some pesticides and metals may sorb onto the walls of glass containers. Thus, hard glass containers are preferred. For samples containing organic compounds, do not use plastic containers except those made of fluorinated polymers such as polytetrafluoroethylene (PTFE).

Use glass containers for all organics analyses such as volatile organics, semi-volatile organics, pesticides, PCBs, oil and grease. Some analytes (e.g., bromine-containing compounds and polynuclear aromatic compound) are light sensitive;

collect them in amber – colored glass containers to minimize photodegradation.

Sample Volumes

Collect a 1-L sample for most physical and chemical analyses. For certain determinations, larger samples may be necessary. Do not use samples from the same container for multiple testing requirements (e.g., organic, inorganic, radiological, bacteriological, and microscopic examinations) because methods of collecting and handling are different for each type of test. Always collect enough sample volume in the appropriate container in order to comply with sample handling, storage, and preservation requirements.

Sample Storage and Preservation

1. Nature of sample changes

Certain cations are subject to loss by adsorption on, or ion exchange with, the walls of glass containers. These include aluminium, cadmium, chromium, copper, iron, lead, manganese, silver and zinc, which are best collected in a separate clean bottle and acidified with nitric acid to a pH below 2.0 to minimize precipitation and adsorption on container walls.

Temperature, pH and dissolved gases (oxygen, carbon dioxide) may play major roles for characteristics of water. Many organic compounds are sensitive to changes in pH and/or temperature resulting in reduced concentrations during storage. Changes in the pH-alkalinity-carbon dioxide balance may cause calcium carbonate to precipitate, decreasing the values for calcium and total hardness.

Iron and manganese are readily soluble in their lower oxidation states but relatively insoluble in their higher oxidation states; therefore, these cations may precipitate or they may dissolve from sediment, depending on the redox potential of the sample. Microbiological activity may affect the nitrate-nitrite ammonia content, phenol or BOD concentration, or the reduction of sulfate. Residual chlorine is reduced to chloride. Color, odor, and turbidity may increase, decrease, or change in quality. Sodium, silica, and boron may be leached from the glass container. Hexavalent chromium may be reduced to trivalent chromium.

Zero head-space is important in preservation of samples with volatile organic compounds and radon. Avoid loss of volatile materials by collecting sample in a completely filled container. Achieve this by carefully filling the bottle so that the top of the meniscus is above the top of the bottle rim. It is important to avoid spillage or air entrapment if preservatives such as HCl or ascorbic acid have already been added to the bottle. After capping or sealing the bottle, check for air bubbles by inverting and gently tapping it; if one or more air bubbles are observed then, if practical, discard the same and repeat refilling the bottle with new sample until no air bubbles are observed (this cannot be done if the bottle contained preservatives before it was filled).

Preservation Techniques

- To minimize the potential for volatilization or biodegradation between sampling and analysis, keep samples as cool as possible without freezing. Preferably pack samples in crushed or cubed ice or commercial ice substitutes before shipment. Avoid using dry ice because it will freeze samples and may cause glass containers to break. Dry ice also may affect a pH change in samples. Keep composite samples cool with ice or arefrigeration system set at 4 °C during compositing. Analyses the samples as quickly as possible on arrival at the laboratory. If immediate analysis is not possible, preferably store at 4 °C.

- pH adjustment in water samples:** The pH should be brought down to below 2 by adding few drops of nitric acid. This is done to prevent loss of cations by adsorption, or precipitation or ion exchange with the walls of glass containers. These include aluminum, cadmium, chromium, copper, iron, lead, manganese, silver, and zinc to minimize precipitation and adsorption on container walls.



Figure 5: Refrigerator

- Methods of preservation are relatively limited and are intended generally to retard biological action, retard hydrolysis of constituents. Preservation methods are limited to pH control, chemical addition, the use of amber and opaque bottles, refrigeration, filtration, and freezing.

- In-situ measurement in water:** Changes in temperature, pH, oxidation-reduction may occur quickly and hence determine temperature, oxidation-reduction potential, and dissolved gases *in situ*; pH, specific conductance, turbidity and alkalinity immediately after collection.

- Analyses before maximum holding time:** Samples should not be stored over prolonged periods even if proper

storage is followed. They should be analyzed for the desired parameter before the prescribed maximum holding time.

Table 1: Maximum holding time for sample preservation

pH, EC, turbidity, DO	In-situ measurement
Conductivity	24 hrs (without refrigeration) 28 days (with refrigeration)
Turbidity	In-situ preferred, but can store upto 48 hours
Hardness	7 days (without acidification to pH 2) 6 months (if acidified to pH 2)
Acidity and alkalinity	Within 24 hours desirable 14 days maximum permissible
BOD	6 hours 48 hours (with proper storage)
COD	7 days 28 days with proper storage
Coliforms	6 hours

Conclusion

Water sampling, the process of taking a portion of water for analysis or other testing, *e.g.*, drinking water to check that it complies with relevant water quality standards, or river water to check for pollutants, or bathing water to check that it is safe for bathing, or intrusive water in a building to identify its. At best, sample preservation only slows the biological and chemical changes that inevitably continue after the sample is collected. Methods of preservation are intended to retard biological action, retard hydrolysis of chemical compounds and complexes, and reduce volatility of constituents. Sample analysis cannot be always carried out immediately or within a short time after sampling; therefore, the sample preservation is necessary to inhibit the reactions in the sample until it is analyzed.

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