PROCESSING OF WATER SAMPLES A5.

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Sample processing forms a continuum with sample collection (NFM 4) and involves the compositing, subsampling (splitting), filtration, solid-phase extraction, preservation, and shipment of samples. Samples are most vulnerable to sampling artifacts, contamination, incorrect chemical treatment, and mislabeling during sample processing. Samples must be processed as soon as possible after collection.[3]

Sample processing: the measures taken to prepare and preserve a water sample as or after it is collected and shipped for laboratory analysis.

GENERAL INFORMATION 5.0

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How samples are processed depends on the targeted analytes and the intended use of the data. Processing procedures for some analytes might require modification of standard processing procedures, as described in section 5.6. **Equipment components must be made of materials that (1) will not contribute or sorb target analytes to or from the water sample, and (2) can withstand cleaning solutions**.

[3]Consult NFM 4 for collection of water samples, and in addition, NFM 1 for field preparations, NFM 2 for equipment selection, NFM 3 for equipment cleaning, NFM 6 for field measurements, NFM 7 for biological indicators, NFM 8 for bottom-material samples, and NFM 9 for field safety.

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PREPARATORY PROCEDURES 5.0.1

Use of the procedures described in this section will help to avoid mistakes and preserve sample integrity. Protocols that are applicable to most sampling efforts for surface water are described in detail in Horowitz and others (1994). Koterba and others (1995) describe the protocols for ground-water sampling that were designed for the National Water-Quality Assessment (NAWQA) Program; these protocols are generally applicable to the routine collection of ground-water samples. Field personnel are responsible for being familiar with any specific sampling protocols that might be required for their studies and programs, especially those that differ from the routine procedures covered by this field manual. For example, field procedures, bottle type, and sample preservation requirements differ for samples collected as part of the USEPA Drinking Water Program (National Water Quality Laboratory Technical Memorandum 97.05[4]).

- To minimize delays in sample processing, calibrate field instruments (NFM 6), and set up processing equipment and supplies in the work area before collecting the sample.
- Clean-sampling procedures are recommended as a general practice when processing raw samples, particularly those for analysis of trace levels of inorganic and organic analytes.
- **Clean-sampling procedures such as Clean Hands/ Dirty Hands techniques (NFM 4) are required when collecting samples to be filtered for analysis of trace elements** (Office of Water Quality Technical Memorandum 94.09; Horowitz and others, 1994; Koterba and others, 1995).

[4]The technical memorandums referenced in this manual are available on the World Wide Web; see "Selected References and Internal Documents" for memorandum titles, dates, and the Web Site address.

- **When using Clean Hands/Dirty Hands techniques:**
  - Designate the Clean Hands (CH) person and the Dirty Hands (DH) person before field work begins (table 4-2 in NFM 4).
  - **CH duties**: Has the only contact with the sample bottle; transfers sample from sampler to splitter; filters, extracts, and preserves sample.
  - **DH duties**: Operates sampling equipment and manages any contact with potential sources of contamination (for example, the churn carrier and pumps).
  - **CH and DH**: Both must wear appropriate disposable, powderless gloves (vinyl, latex, or nitrile for inorganic work; latex or nitrile for organic work).
- **Check sample-designation codes and processing requirements for each sample.** Requirements depend on program and laboratory protocols, study objectives, and data-quality requirements. Laboratory codes and processing requirements are summarized in Appendixes A5-A, B, and C.
  - **Organic analytes**. Identify the bottle requirement by checking the sample designation code (see in-text table below and Appendix A5-A). Use only containers that arrive clean, baked, and capped. **Discard any bottles that arrive uncapped**.
  - **Inorganic and radiochemical analytes**. Identify the bottle requirement by checking the sample designation code (see in-text table below and Appendixes A5-B and A5-C). For example, samples to be acidified must be collected in bottles that arrive from the laboratory acid rinsed and capped; **discard any acid-rinsed bottles that arrive uncapped**. Prerinse all bottles used for nutrients, major-ion, and trace-element samples with deionized water (DIW) before sampling. Field rinse bottles with the water to be sampled, if a field rinse is specified (section 5.0.3 and Appendixes A5-B and A5-C).

Common organic-compound sample-designation codes for the National Water Quality Laboratory of the U.S. Geological Survey

[Refer also to Appendix A5-A. ml, milliliters; °C, degrees Celsius]

Sample designation code	Bottle description and sample preservation
VOC	40-mL amber glass vials, laboratory cleaned and baked, for analysis of volatile organic compound sample (VOC or VOA); sample chilled to or below 4°C without freezing. Some programs require chemical treatment.
GCC	1-L amber, glass bottle, laboratory cleaned and baked, for various types of pesticides and organic-compound samples other than VOCs; sample chilled to or below 4°C without freezing.
TOC, DOC	125-mL amber glass bottle, laboratory cleaned and baked, for total (TOC) or dissolved (DOC) organic carbon; sample chilled to 4°C or below without freezing.

Common inorganic-constituent sample-designation codes of the National Water Quality Laboratory of the U.S. Geological Survey

[Refer also to Appendix A5-B and A5-C. mL, milliliter; <, less than; °C, degrees Celsius; L, liter]

Sample designation code	Bottle description and sample preservation
RA, FA	250-, 500-, or 1,000-mL polyethylene bottles, acid-rinsed, capped, to be filled with raw (RA) or filtered (FA) samples and acidified with nitric acid to pH <2.
RU, FU	250-, 500-, or 1,000-mL polyethylene bottles, uncapped, to be filled with untreated raw (RU) and filtered (FU) samples.
FCC	125-mL polyethylene bottles, uncapped, to be filled with filtered (FCC, brown bottle) sample for nutrient analysis and chilled to or below 4°C without freezing.
WCA, FCA	125-mL polyethylene bottles, uncapped; to be filled with raw (WCA, uncolored bottle) or filtered (FCA, brown bottle) sample for nutrient analysis, treated with sulfuric acid, and chilled to or below 4°C without freezing.
RAM, FAM	250-mL glass bottles, acid-rinsed, capped, to be filled with raw (RAM) or filtered (FAM) sample for mercury analysis, and treated with nitric acid/potassium dichromate solution.
FAR	1-L polyethylene bottles, acid rinsed, capped, to be filled with filtered (FAR) samples for radiochemical analysis and treated with nitric acid to pH <2.

- **Clean equipment and supplies as directed in NFM 3.**
  - **Organic analytes.** All containers arrive precleaned and baked from the laboratory. **Do not prerinse or field rinse these glass bottles or vials.** Samples to be analyzed for organic compounds are hereafter referred to as organic-compound samples.
  - **Inorganic analytes.** Prerinse bottles with DIW and store half filled with DIW. This procedure is required for all FA samples with target analytes at parts-per-billion (ppb) concentrations, and is recommended for all samples to be analyzed for inorganic constituents (hereafter referred to as inorganic-constituent samples) that also require field-rinsed bottles.
- **Set up a clean work area** at the field site for sample processing. (An appropriate area includes, for example, a mobile laboratory, a water-quality field vehicle (NFM 2), or clean space in a building near the sampling site.)
  - Protect the area from airborne sources of contamination such as dust, vehicle emissions, and vapors from inorganic chemicals and organic solvents.
  - Spread sheeting over the area where samples are to be processed. For inorganic-constituent samples, use plastic sheeting. For organic-compound samples, use aluminum foil.
- **Prevent direct contact with potential source(s) of contamination.**
  - Exclude airborne particulates by processing samples onsite in processing and preservation chambers.
  - Handle anoxic samples rapidly and under an inert gas atmosphere (NFM 4.0.3).
  - Keep hands gloved and away from potential sources of contamination while processing samples. While filling the sample bottle, the sample must not come in contact with gloved hands.
- **Keep sample-processing equipment covered** with a clean, noncontaminating material when not in use; keep sample bottles capped and covered or bagged.

SEQUENCE FOR PROCESSING SAMPLES 5.0.2

The order of sample collection, processing, and preservation for specific analytes should be determined before beginning field work and adhered to consistently. The recommended sequence for sample collection and processing is based on logistics for maintaining sample integrity and differs for ground-water and surface-water sampling. The recommended sequence can be modified, depending on the types of samples to be collected and on data objectives. In general, process samples in the order indicated on table 5-1.

- For ground-water sampling, the amount of well purging might affect concentrations of VOCs measured in the ground-water samples (Gibs and Imbrigiotta, 1990). Therefore, VOC samples are collected first.
- When sampling either surface water or ground water for inorganic analyses,
  - Filter trace-element samples first, as prescribed and explained in section 5.2 and in Horowitz and others (1994).
  - Next, filter nutrient, major ion, and other inorganic-constituent samples having concentrations that will not be appreciably affected as nominal pore size of the filter media decreases.
  - Filter the alkalinity sample (NFM 6) with the other anions.
- When composite samples of surface water are processed, samples for analysis of organic compounds usually are processed first and are taken from a noncontaminating compositing device separate from that for inorganic-constituent samples, unless a cone splitter is used (section 5.1).

Table 5-1. Recommended sequence for processing samples

1. Organic compounds—Raw (wholewater or unfiltered) samples first, followed by filtered samples. **Do not field rinse bottles.** Chill immediately a. Volatile organic compounds (VOCs). b. Pesticides, herbicides, polychlorinated biphenyls (PCBs) and other agricultural and industrial organic compounds.
2. Total organic carbon (TOC), dissolved organic carbon (DOC),[1] and suspended organic carbon (SOC). Chill immediately.
3. Inorganic constituents, nutrients, radiochemicals, isotopes: **For ground water,** filtered samples first, followed by raw samples. **For surface water,** raw samples first, followed by filtered samples. (Field rinse each bottle, as required.) a. Trace metals. b. Separate-treatment constituents (such as mercury, arsenic, selenium) and major cations. c. Major anions, alkalinity, and nutrients. Chill nutrients immediately. d. Radiochemicals and isotopes. (Bottle-rinse, filtration, and preservation requirements depend on analysis to be performed (section 5.6 and Appendix A5-C.)[2])
4. Radon and chlorofluorocarbons.[2] Do not rinse bottle.
5. Microorganisms (NFM 7).

[1]TOC and DOC samples can be collected whenever most appropriate for the specific field operation.
[2]Radon and chlorofluorocarbon and most isotope samples are collected outside of the processing chamber.

5.0.3 FIELD RINSING OF BOTTLES USED TO CONTAIN SAMPLES FOR ANALYSIS OF INORGANIC CONSTITUENTS

Most polyethylene sample bottles and only those glass sample bottles that are designated for analysis of inorganic constituents (inorganics bottles) are field rinsed as described in table 5-2. Check Horowitz and others (1994) and the laboratory requirements (summarized in Appendixes A5-B and A5-C) for more detailed discussions of field rinsing. **The field-rinse water normally is the same as the water that will fill the sample bottle: use wholewater sample for raw (unfiltered) samples and filtrate for filtered samples.**

- If the volume of sample obtained for processing is limited, DIW of the appropriate quality may be substituted as the rinse solution for the first two of the three required rinses.
- Wear disposable, powderless gloves while processing samples.

Check analyte requirements before field rinsing bottles. For example, DO NOT field rinse glass bottles that are designated for analysis of organic compounds.

Table 5-2. Directions for field rinse of bottles used to contain samples for inorganic-constituent analysis

[DIW, deionized water; mL, milliliters]

Bottle Preparation
• If bottles were previously rinsed and half-filled with DIW[1], discard DIW and rinse once only with the water to be sampled. Use filtrate for filtered samples and wholewater for raw samples.
• If bottles were not prerinsed with DIW, rinse twice with DIW onsite, followed by one field rinse with the water to be sampled (use only 25-mL filtrate for bottle rinse for the filtered sample[1,2]).
Field-Rinse Technique
1. Put on disposable, powderless gloves.
2. Fill sample bottle about 1/10 full of rinse water. Cap bottle.
3. Shake the bottle vigorously to rinse all interior surfaces.
4. Discard rinse water by swirling the solution out of the bottle.
5. Shake off adhering droplets.

[1]Required for filtered trace-element samples (Horowitz and others, 1994).
[2]Refer to section 5.2.1.A for detailed guidance relating to surface-water and ground-water samples.