

Techniques of Water-Resources Investigations

Book 9

Handbooks for Water-Resources Investigations

**National Field Manual
for the Collection of
Water-Quality Data**



**Chapter A3.
CLEANING OF
EQUIPMENT FOR
WATER SAMPLING**

Revised 2004

Edited by Franceska D. Wilde



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Foreword

The mission of the Water Resources Discipline of the U.S. Geological Survey (USGS) is to provide the information and understanding needed for wise management of the Nation's water resources. Inherent in this mission is the responsibility to collect data that accurately describe the physical, chemical, and biological attributes of water systems. These data are used for environmental and resource assessments by the USGS, other government agencies and scientific organizations, and the general public. Reliable and quality-assured data are essential to the credibility and impartiality of the water-resources appraisals carried out by the USGS.

The development and use of a *National Field Manual* is necessary to achieve consistency in the scientific methods and procedures used, to document those methods and procedures, and to maintain technical expertise. USGS field personnel use this manual to ensure that the data collected are of the quality required to fulfill our mission.

(signed)

Robert M. Hirsch
Associate Director for Water

Techniques of Water-Resources Investigations

Book 9

Handbooks for Water-Resources Investigations

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CLEANING OF EQUIPMENT FOR WATER SAMPLING **A3.**

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Chapter A3.

CLEANING OF EQUIPMENT FOR WATER SAMPLING

Revised 2004

Edited by Franceska D. Wilde

ABSTRACT

The *National Field Manual for the Collection of Water-Quality Data* (*National Field Manual*) describes protocols and provides guidelines for U.S. Geological Survey (USGS) personnel who collect data used to assess the quality of the Nation's surface-water and ground-water resources. Chapter A3 describes procedures for cleaning the equipment used to collect and process samples of surface water and ground water and procedures for assessing the efficacy of the equipment-cleaning process. This chapter is designed for use with the other chapters of this field manual.

Each chapter of the *National Field Manual* is published separately and revised periodically. Newly published and revised chapters will be posted on the USGS page "National Field Manual for the Collection of Water-Quality Data." The URL for this page is <http://pubs.water.usgs.gov/twri9A/> (accessed September 20, 2004).

INTRODUCTION

As part of its mission, the U.S. Geological Survey (USGS) collects data needed to assess the quality of our Nation's water resources. The *National Field Manual for the Collection of Water-Quality Data* (*National Field Manual*) describes protocols (requirements and recommendations) and provides guidelines for USGS personnel who collect those data on surface-water and ground-water resources. Chapter A3 describes procedures for cleaning the equipment used to collect and process samples of surface water and ground water and procedures for assessing the efficacy of the equipment-cleaning process.

The *National Field Manual* is Section A of Book 9 of the USGS publication series "Techniques of Water-Resources Investigations" and consists of individually published chapters. Chapter numbers are preceded by an "A" to indicate that the report is part of the *National Field Manual*. Chapters of the *National Field Manual* are referred to in the text by the abbreviation "NFM" followed by the chapter number (or chapter and section number). For example, NFM 4 refers to chapter 4, "Collection of Water Samples," and NFM 4.1 refers to the section on surface-water sampling methods.

PURPOSE AND SCOPE

The *National Field Manual* is targeted specifically toward field personnel in order to (1) establish and communicate scientifically sound methods and procedures, (2) provide methods that minimize data bias and, when properly applied, result in data that are reproducible within acceptable limits of variability, (3) encourage consistent use of field methods for the purpose of producing nationally comparable data, and (4) provide citable documentation for USGS water-quality data-collection protocols.

The equipment-cleaning procedures presented in this chapter are adequate for routine environmental conditions. A modification of the cleaning procedures might be required, for example, in order to decontaminate equipment adequately after sampling at sites where analyte concentrations are large. Modifications to the standard procedures described in this chapter must be quality controlled and documented.

REQUIREMENTS AND RECOMMENDATIONS

As used in the *National Field Manual*, the terms **required** and **recommended** have USGS-specific meanings.

Required (require, required, or requirements) pertains to USGS protocols and indicates that USGS Office of Water Quality policy has been established on the basis of research and (or) consensus of the technical staff and has been reviewed by water-quality specialists and District¹ or other professional personnel, as appropriate. Technical memorandums or other documents that define the policy pertinent to such requirements are referenced in this manual. Personnel are instructed to use required equipment or procedures as described herein. Departure from or modifications to the stipulated requirements that might be necessary to accomplish specific data-quality requirements or study objectives must be based on referenced research and good field judgment and must be quality assured and documented in permanent and readily accessible records.

Recommended (recommend, recommended, recommendation) pertains to USGS protocols and indicates that USGS Office of Water Quality policy recognizes that one or several alternatives to a given procedure or equipment selection are acceptable on the basis of research and (or) consensus. References to technical memorandums and selected publications pertinent to such recommendations are cited in this chapter to the extent that such documents are available. Specific data-quality requirements, study objectives, or other constraints affect the choice of recommended equipment or procedures. Selection from among the recommended alternatives should be based on referenced research and good field judgment, and reasons for the selection should be documented. Departure from or modifications to recommended procedures must be quality assured and documented in permanent and readily accessible records.

¹**District** refers to a water-data collecting organizational unit of the USGS located in any of the States or Territories of the United States.

FIELD MANUAL REVIEW AND REVISION

This is version 2.0 of chapter A3, "Cleaning of Equipment for Water Sampling," dated April 2004. The version number and date appear in the footer of each page. Each chapter of the *National Field Manual* is reviewed and revised periodically to correct any errors and incorporate technical advances.

Comments on the NFM, and suggestions for updates or revisions, should be sent to nfm-owq@usgs.gov. Newly revised and reissued chapters or chapter sections are posted on the World Wide Web on the USGS page "National Field Manual for the Collection of Water-Quality Data." The URL for this page is <http://pubs.water.usgs.gov/twri9A/> (accessed September 20, 2004). This page also contains a link to the NFM "Comments and Errata" page that chronicles revisions to each chapter.

ACKNOWLEDGMENTS

The information included in the original and revised versions of chapter 3 of the *National Field Manual* is based on the work of Sandstrom (1990), Horowitz and others (1994), Shelton and Capel (1994), and Koterba and others (1995). Credit for the production of this report belongs chiefly to the original editors, including D.B. Radtke, Jacob Gibs, and R.T. Iwatsubo.

The editors wish to thank and pay tribute to those who have been responsible for technical review and who contributed to the accuracy, quality, and usability of this report: S.L. Lane, R.W. Lee, and T.D. Oden. Appreciation also is extended to the following colleague reviewers who helped to improve this report: H.D. Ardourel, B.A. Bernard, K.K. Fitzgerald, D.S. Francy, S.R. Glodt, V.J. Kelly, S.W. McKenzie, S.K. Sando, C.A. Silcox, and W.R. White. In addition, the editor is indebted to I.M. Collies and L.J. Ulibarri for their editorial and production assistance.

Special thanks go to T.L. Miller and S.K. Sorenson, whose encouragement and faith in this project have been instrumental to its achievement.

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CLEANING OF EQUIPMENT FOR WATER SAMPLING **A3.**

USGS policy requires that equipment for water samples be properly cleaned before contacting the sample and that the effectiveness of the cleaning procedures be quality controlled (Sandstrom, 1990; Horowitz and others, 1994; Koterba and others, 1995). Additional specific and more stringent requirements than the standard procedures described in this chapter may be required to meet specific project or program needs.

The goal of equipment cleaning is to help ensure that the equipment is not a source of foreign substances that could affect the ambient concentrations of target analytes in samples or otherwise affect sample chemistry. Standard procedures are described in this chapter for when, where, and how to (1) clean equipment constructed of various materials and (2) collect equipment blanks and field blanks for quality control.

Equipment cleaning (decontamination): Applying cleaning solutions to the external and internal surfaces of equipment or using other nondestructive procedures (such as steam cleaning) to remove foreign substances that could affect the concentrations of analytes in samples.

Space commonly is dedicated in a workplace laboratory for equipment cleaning and for storage of cleaning supplies. In this report this work space can include the Field Service Unit or other dedicated workspace.

Document the use of standard USGS equipment-cleaning and quality-control procedures.

- ▶ Clean all sample-collection and sample-processing equipment before use.
 - Manufacturing residues must be removed from new equipment.
 - Dust and any other foreign substances must be removed from equipment that has been in storage or transport.
 - Equipment must be decontaminated immediately after use.
- ▶ Prevent cross contamination between sampling sites by rinsing equipment with deionized water (DIW) while equipment is still wet from sampling, and then clean equipment as prescribed in this chapter before transporting it to the next site.
- ▶ Do not substitute a "sample-water field rinse" for the equipment-cleaning procedures described in this chapter.
- ▶ Collect equipment blanks and field blanks for quality control of the cleaning procedure. The frequency of collecting blanks normally is based on study objectives and site conditions. **However, a minimum of one equipment blank per year is required for each piece of equipment.**

You must adhere to any additional or specific protocols mandated by the study or program for which the data are being collected.

SUPPLIES FOR EQUIPMENT 3.1 CLEANING

By D.B. Radtke, A.J. Horowitz, and
M.W. Sandstrom

The supplies commonly used to clean sample-collection and sample-processing equipment are listed in table 3–1. Cleaning supplies are to be stored in a contaminant-free cabinet. Follow safety instructions regarding the storage of chemical reagents (NFM 9).

Before gathering the cleaning supplies, check the construction materials (for example, metal, glass, or plastic) of washbasins and other cleaning items relative to the samples to be collected.

- ▶ **For analysis of inorganic constituents**—Basins, brushes, and other items used for cleaning should be constructed of a suitable nonmetallic material, preferably uncolored or white polypropylene, polyethylene, or other plastic. **Do not use cleaning agents or items that might leach or sorb metals if the equipment to be cleaned will be used for samples to be analyzed for trace elements.**
- ▶ **For analysis of organic compounds**—Basins and other cleaning items can be constructed of metal, glass, or plastic materials. Stainless steel is recommended for methanol use. **Do not use cleaning agents or items that might leach, sorb, or leave residues of organic substances that could bias or interfere with the analysis.**

CAUTION: Refer to Material Safety Data Sheets (MSDS) before handling any chemicals.

- **Wear safety gloves, glasses, and apron when working with corrosive and oxidizing solutions.**
- **Work in a well-ventilated area.**

Table 3-1. Supplies for cleaning equipment used for water-sampling activities

[ACS, American Chemical Society; DIW, distilled/deionized water; $\mu\text{S}/\text{cm}$, microsiemens per centimeter at 25 degrees Celsius; PBW, pesticide-grade blank water; VPBW, volatiles and pesticide-grade blank water (nitrogen-purged); IBW, inorganic-grade blank water; L, liter; cm, centimeter; NAWQA, National Water-Quality Assessment; TPC, total particulate carbon; TPN, total particulate nitrogen; DOC, dissolved organic carbon; NFM, *National Field Manual*; PVC, polyvinyl chloride]

Item	Description and Comments
Acid solution ¹	Hydrochloric: ACS trace-element grade (5 percent by volume in DIW). Nitric: ACS trace-element grade (10 percent by volume in DIW).
Aluminum foil	Organics only: Heavy duty, for work surfaces and equipment.
Bags, plastic or fluorocarbon polymer	Sealable bags with uncolored closure strips, various sizes. Recyclable trash bags are recommended for large equipment storage.
Noncolored plastic sheeting	Clean sheeting used to provide a clean work surface.
Brushes and sponges	Uncolored; plastic components needed for inorganic work.
Distilled/deionized water (DIW)	Maximum specific electrical conductance, 1 $\mu\text{S}/\text{cm}$ (usually District produced; Office of Water Quality Memorandum 92.01). Must not be used as a substitute for blank water.
Office-produced organic-grade deionized water	Usable only as a cleaning solution and only as specified in the text. Must not be used to substitute for PBW or VPBW. ²
Detergent	Nonphosphate laboratory soap (for example, Liquinox TM).
Gloves, disposable	Powderless, nitrile, assorted sizes.
Inorganic-grade blank water (IBW) ²	Blank water with certificate of analysis prepared and (or) quality assured by the analyzing laboratory, and is required for collecting inorganic blank samples.
Jerricans or carboys	For waste solutions and as neutralization container. Neutralization container: 25- to 30-L, polyethylene, wide-mouth, with layer of marble chips. Methanol waste container: Appropriate for flammable liquid.
Methanol ³	ACS pesticide-grade. Methanol is the organic solvent in common use for cleaning organic-compound sampling equipment and is mandated for the NAWQA Program. Study requirements might dictate use of a different ACS pesticide-grade solvent.
Neutralization materials	Marble landscape chips (1- to 2-cm chips recommended). ⁴
Pesticide-grade blank water (PBW); volatile-grade blank water (VPBW) ²	Blank water prepared and (or) quality assured by the analyzing laboratory; required for collecting blank samples as follows: PBW for pesticide analysis; VPBW for volatile compounds analysis and pesticide analysis; and either PBW or VPBW for TPC, TPN, and DOC analyses.
Safety equipment and guidelines (NFM 9)	For example, Material Safety Data Sheets (MSDS), safety glasses, chemical spill kit, apron, emergency phone numbers.

Table 3-1. Supplies for cleaning equipment used for water-sampling activities—*Continued*

Item	Description and Comments
Standpipes for submersible pump	Plastic, glass, or other suitable material; for example, pipette jars or capped PVC casing; one standpipe labeled for blank water and one each for each cleaning solution. (Do not use PVC for methanol.)
Tap water	If quality is questionable, substitute DIW. Tap water is more effective for initial and rapid removal of detergent residue.
Tissues	Laboratory grade, lint free, various sizes (for example,
	Kimwipes™).
Washbasins	One washbasin dedicated per cleaning solution; white or uncolored
	for containing acid or DIW solutions. Plastic, nonleaching. (Stainless steel is recommended for methanol.)
Wash bottles - polyethylene or	Labeled to indicate contents (for example, ACID, DIW, TAP).
fluorocarbon (dispenser or squeeze)	Fluorocarbon polymer needed for methanol, PBW, VPBW, and IBW.

¹Hydrochloric acid is required if analyzing for nitrogen species; otherwise, nitric acid is acceptable.

²USGS personnel can order IBW, PBW, and VPBW from One Stop Shopping.

³Methanol is extremely flammable and potentially explosive, emits noxious fumes, and is absorbed through the skin. Observe safety practices when handling methanol or other organic solvents.

⁴Agricultural limestone, soda ash, baking soda, and crushed shells are not recommended (Horowitz and others, 1994).

11/2005 - Add a row to table 3-1:

Disinfectant - Hypochlorite (0.525 percent) wipes (commercially available), dilute bleach solution (0.005 percent, see text), or methyl or ethyl alcohol solution.

Note: Refer to NFM 7.1 for disinfection requirements when sampling for microbial organisms.

CAUTION: Methanol is extremely flammable and potentially explosive, emits noxious fumes, and is absorbed through the skin. Observe safety practices when handling methanol or other organic solvents.

- **Wear safety gloves, glasses, and apron.**
- **Work in a well-ventilated area and away from an open flame or sparks.**
- **Make sure that all electrically powered equipment is grounded; alternating current equipment must have a ground-fault interrupter.**
- **Inspect electrical wiring for cuts, breaks, or abrasions where the metal wire is exposed.**
 - **Exposed wires can cause sparks if a short to ground occurs.**
 - **Replace faulty wires—do not rely on fixing with electrical tape.**

GENERAL CLEANING PROCEDURES 3.2

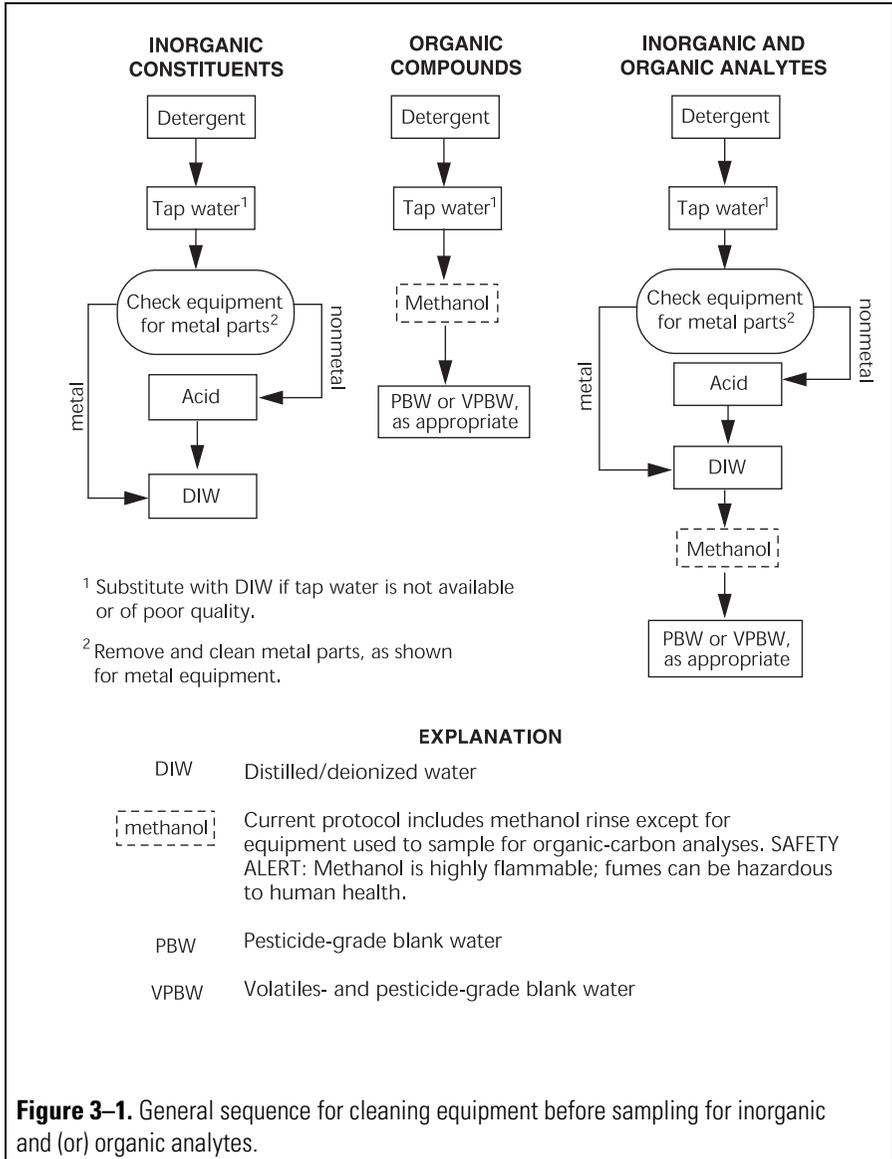
By A.J. Horowitz and M.W. Sandstrom

Equipment should be cleaned in an area protected from airborne or other sources of contamination. Procedures to remove contaminants to concentrations below the targeted method-detection levels can vary, depending on the cleaning supplies used, the type of equipment being cleaned, the solubility and concentration of contaminant(s), and the length of time equipment is exposed to contaminant(s). **Examine equipment-blank and field-blank data to determine whether adjustments to the cleaning protocol are needed** (section 3.4).

The cleaning procedure to be used depends on the type(s) of water samples that will be collected and processed. Figure 3–1 summarizes the sequence of cleaning procedures for equipment used to collect samples for inorganic and (or) organic analytes (Sandstrom, 1990; Horowitz and others, 1994; and Koterba and others, 1995).

- ▶ **Inspect equipment for stains, cuts, or abrasions. Replace parts as needed.**
 - Replace chipped or cracked glassware.
 - Replace bent sampler nozzles or samplers with bent fins (surface-water samplers).
 - Replace tubing if mold, mildew, or imbedded sediment cannot be removed.
 - Replace cracked or severely crimped O-rings.
 - Repair pump intakes and anti-backsiphons that have loose or missing screws.
 - Check the flow manifold and sample tubing to ensure that valves and quick-connect fittings are in good working order; repair or replace as necessary to eliminate any problems.
 - Cover exposed metal surfaces on surface-water samplers with "plasti-dip."

- ▶ **Rinse equipment with DIW directly after use while equipment is still wet, then use cleaning procedures.**
- ▶ **Place cleaned equipment in doubled storage bags.**



Do not allow collection and processing equipment to sit uncleaned in a field vehicle or elsewhere between field trips.

INORGANIC-CONSTITUENT SAMPLING EQUIPMENT 3.2.1

Cleaning of equipment used to collect and process water for analysis of inorganic constituents involves a five-step workplace-laboratory procedure or a five-step field-site procedure. (These procedures do not apply to field-measurement instruments—see NFM 6.) These procedures are effective for cleaning equipment exposed to water containing concentrations of as much as 50,000 µg/L of iron, 5,000 µg/L each of manganese and zinc, 400 µg/L of copper, 125 µg/L of cobalt, and large concentrations of the other trace elements (Horowitz and others, 1994).

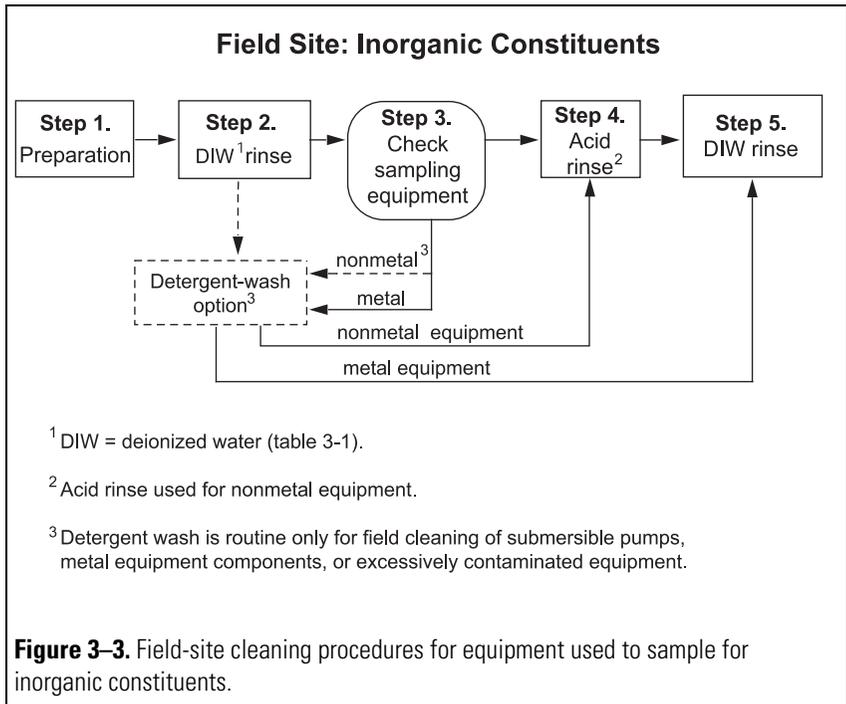
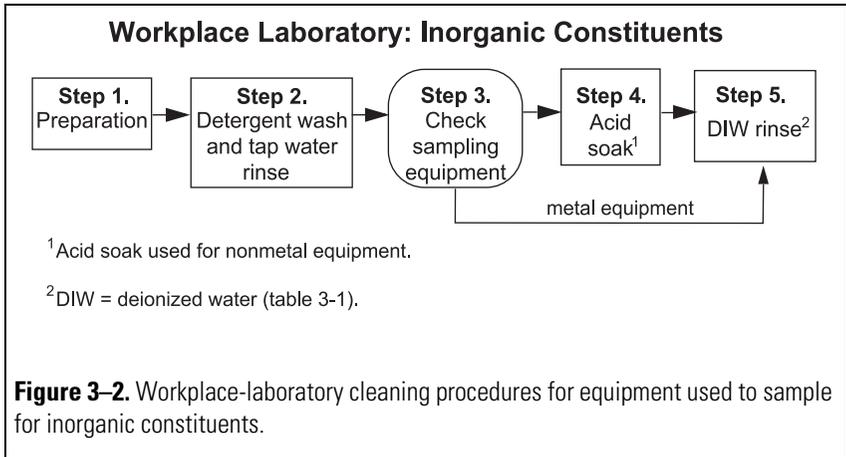
Equipment should be cleaned periodically in the workplace laboratory, where complete disassembly is more practical and more thorough procedures are possible. Compared to cleaning at the field site, cleaning procedures carried out in the workplace laboratory involve longer exposure of equipment to cleaning solutions, more frequent changes of cleaning solution, and greater volumes of rinse water.

- ▶ To minimize field cleaning of equipment between sampling sites, preclean a separate set of equipment for each site.
- ▶ If individual or dedicated sets of equipment for each field site are not available or cannot be precleaned, clean the equipment onsite and process additional field blanks during each field trip (Horowitz and others, 1994; Koterba and others, 1995).
- ▶ Return excessively contaminated equipment to the workplace laboratory for rigorous cleaning before reuse.
- ▶ After cleaning, document completion of and any modifications to the cleaning procedures. Note that procedure modifications must be sanctioned by the data-collection program and be consistent with study data-quality objectives.

Standard procedures for workplace-laboratory and field-site cleaning of equipment used to collect and process samples for analysis of inorganic constituent are described below and summarized in figures 3–2 and 3–3. Not all the steps listed apply to all equipment, however. For example,

- ▶ Omit detergent step when cleaning plastic bags for surface-water samplers.
- ▶ Omit acid step when cleaning submersible pumps or other equipment constructed of stainless steel or other metallic material (for example, the original polyethylene churn with a metal-spring spigot).
- ▶ Omit detergent and acid steps when cleaning sample bottles.

Be sure to check the specific procedures for sample bottles and other selected equipment listed in section 3.3 before proceeding with the workplace-laboratory and field-site procedures. Wear powderless laboratory gloves during the cleaning procedures, changing to a fresh pair of gloves with each change in procedure. Minimize the length of time that the gloves are in contact with any cleaning or rinse solution.



To clean equipment used when sampling for inorganic constituents:

Step 1. Preparation at the workplace laboratory or field site (figs. 3–2 and 3–3).

- a. Prepare a contaminant-free space for cleaning and drying the cleaning supplies and sample-collection and sample-processing equipment.
 - i. Gather the cleaning supplies, the equipment to be cleaned, and the plastic bags or other material with which to wrap the cleaned equipment. Check table 3–1 for the cleaning supplies needed.
 - ii. Place clean plastic sheeting over the work surface.
 - iii. Put on disposable, powderless gloves², a laboratory coat or apron, and safety glasses.
 - iv. Prepare the detergent solution, using a nonphosphate, laboratory-grade detergent.
 - **Workplace laboratory** (fig. 3–2). Use 0.1- to 2-percent solution, volume-to-volume (v/v), using a higher concentration for dirtier equipment.
 - **Field site** (fig. 3–3). Use 0.1- to 0.2-percent solution, v/v. Do not use greater than 0.2-percent solution for field cleaning.
 - v. Prepare the acid solution, using a 5-percent v/v dilution of ACS trace-element-grade hydrochloric acid (HCl) in DIW.
 - **Add the acid to the water**, not water to acid (NFM 9).
 - If nitric acid (HNO₃) will be used, prepare a 10-percent solution (v/v) of ACS trace element-grade acid in DIW.
 - vi. Label each washbasin, standpipe, and wash bottle to indicate the solution it will contain.
 - vii. Unwrap the equipment to be cleaned and discard the storage bags. Change gloves.

²Refers to laboratory gloves that are nonpowdered and intended for disposal after one use. Glove materials must be appropriate for the work to be carried out and the solutions and equipment to be contacted. For example, nitrile gloves are rated as resistant to the acids and solvents commonly encountered in water-quality field work.

Be aware of possible allergic reactions to latex or other glove materials; discontinue use immediately if an allergic reaction occurs.

- b. Clean the items used to clean the equipment.
 - i. Fill washbasins and (or) standpipes with the nonphosphate detergent solution. Put wash bottles, scrub brushes, and other small items used for cleaning into a washbasin. **Soak for 30 minutes.**
 - ii. Scrub interior and exterior sides of basins and standpipes with soft scrub brushes. Fill wash bottles with a soapy solution and shake vigorously.
 - iii. Rinse all items thoroughly with tap water to remove detergent residue. No detergent bubbles should appear when fresh tap water is agitated in the basin, standpipe, or wash bottle.
 - iv. Rinse washbasins with DIW.
 - v. Pour 5-percent HCl (or 10-percent HNO₃) solution into washbasins, standpipes, and wash bottles. Soak for 30 minutes. **Do not soak items with metal parts (exposed or hidden) in an acid solution.**
 - vi. Discard used acid solution into a neutralization container containing a bottom layer of marble chips (Step 4d).
 - vii. Rinse washbasins, standpipes, and wash bottles with DIW. Dispose of DIW using directions in Step 4d.
- c. Disassemble sample-collection and sample-processing equipment. Change gloves.
 - Submersible pumps should be disassembled periodically for workplace cleaning, but they are not usually disassembled for field cleaning.
 - Processing and preservation chamber frames should be cleaned periodically using workplace-laboratory cleaning procedures. Field cleaning is needed only if the cover is slipped over the frame instead of being clipped to the inside of the frame.

Step 2. Detergent wash and tap water rinse—Workplace laboratory (fig. 3–2).

- a. Place small equipment parts into washbasin labeled for detergent and fill with a 0.1- to 2-percent solution of nonphosphate laboratory detergent. The amount of detergent depends on the hardness of the tap water and the degree to which the equipment is dirty or contaminated.
- b. Soak equipment and tubing for 30 minutes: fill tubing with solution and keep submerged.
- c. Scrub exterior and interior of equipment surfaces to the extent possible, using a firm sponge or soft brush to remove any adhering material such as oil and grease, sediment, algae, and chemical deposits. Pay particular attention to grooves and crevices, O-rings, nozzles, and other spaces where inorganic or organic materials might be trapped. Change gloves.
- d. Rinse equipment thoroughly with warm tap water to remove detergent residue. Equipment rinsing is completed when no soap bubbles appear after the rinse water is agitated. Change gloves.

Step 2. DIW rinse and detergent-wash option—Field site (fig. 3–3).***For the DIW rinse:***

- a. Rinse equipment and tubing with DIW. Pay particular attention to removing material from grooves and crevices, O-rings, nozzles, and places where materials might be trapped. Note that equipment should already have had one DIW rinse directly after contact with sample water and before the equipment had a chance to dry.
- b. Change gloves. Proceed to field detergent-wash option only for metal equipment components or for equipment that has become excessively contaminated.

For the detergent-wash option:

A field detergent wash is used for between-site cleaning of submersible pumps, metal components of equipment, or for equipment that has become greasy or otherwise coated and requires detergent to remove foreign materials; specific instructions for submersible pumps are given in section 3.3.10.

- a. Place small equipment, tubing, and parts into basin labeled “detergent” and fill with a 0.1- to 0.2-percent detergent solution. Soak for about 10 minutes, or keep equipment assembled and circulate the solution through pump tubing for 5 to 10 cycles.
- b. Scrub equipment surfaces with a firm sponge or soft brush to remove any adhering material such as oil and grease, sediment, algae, or chemical deposits. Pay particular attention to grooves and crevices, O-rings, nozzles, and other places where inorganic materials might be trapped. Change gloves.
- c. Rinse equipment thoroughly with tap water to remove detergent residue. Use DIW if tap water is unavailable or is suspected of having a quality so poor as to contaminate the equipment. If necessary, use a wash bottle filled with DIW or tap water to rinse hard-to-reach places; pump tap water through assembled equipment for five or more tubing volumes. Equipment rinsing is complete when no soap bubbles appear after agitating the rinse water. If nonmetal equipment has been detergent-washed, go to Step 4.
- d. Place equipment into acid-solution washbasin. Change gloves.

Step 3. Check equipment—Workplace laboratory and field site (figs. 3–2 and 3–3).

- Nonmetal equipment or equipment with removable metal parts: remove any metal parts and go to Step 4.
- Metal equipment components or excessively contaminated equipment: use the detergent-wash option at the field site (step 2) and then go to Step 5, DIW rinse.

Step 4. Acid soak/rinse—Workplace laboratory and field site (figs. 3–2 and 3–3).

For equipment constructed primarily of glass or fluorocarbon polymer or some other plastic, soak (workplace laboratory) or rinse (field site) in a 5-percent (v/v) HCl solution to remove any remaining organic films and inorganic deposits.

TECHNICAL NOTE: A 10-percent (v/v) HNO₃ solution can be used instead of HCl if samples to be collected with the equipment will not be analyzed for nitrogen species.

CAUTION: Wear safety glasses and other protective apparel when working with acids.

- a. Place nonmetal equipment and tubing into the washbasin labeled “acid solution.”
- b. **Workplace laboratory.** Fill basin with dilute HCl solution (see TECHNICAL NOTE above). Soak equipment and tubing for 30 minutes. Carefully swirl the acid solution several times during the 30-minute soak to enhance removal of mineral encrustations.
- c. **Field site.** Using a wash bottle filled with 5-percent HCl solution (see TECHNICAL NOTE above), rinse exterior of equipment and tubing. Pump acid solution through the equipment and tubing, using a peristaltic pump.
- d. Carefully pour or pump the used acid solution into a neutralization container with marble chips covering the bottom (table 3–1). Do not reuse the acid solution.
 - Do not fill the neutralization container more than three-fourths full of acid solution.
 - Ventilate container and workspace to allow for safe escape of carbon dioxide gas during dissolution of marble chips.
 - Check the solution pH periodically using narrow range pH indicator strips. Neutralization is complete when the solution pH is greater than 6.0 or the original DIW pH.
 - Discard the neutral solution, as appropriate.
 - Rinse the container with tap water but retain any undissolved marble chips. Replenish chips to form a layer on the bottom of the neutralization container.

Step 5. DIW rinse—Workplace laboratory or field site (figs. 3–2 and 3–3).

- a. Place equipment into the cleaned washbasin labeled DIW. Change gloves.
- b. **Workplace laboratory.** Rinse exterior and interior of each piece of equipment and tubing thoroughly with DIW and place on a clean surface to dry or into a clean IBW washbasin if blank samples will be collected to quality control the cleaning procedures.
- c. **Field site.** Pump DIW through equipment.
- d. Pour or discharge DIW rinse water into neutralization container. Change gloves.
- e. Continue DIW rinsing until rinse-water pH is greater than 6.0 or the original DIW pH.
- f. Allow equipment to air dry in an area free from potential airborne contaminants.

Storage of clean equipment

- ▶ Place dry, clean equipment inside doubled plastic bags. For small equipment, parts, and tubing, use sealable plastic bags.
- ▶ Place the churn splitter into doubled plastic bags and then place churn splitter inside of the churn carrier.

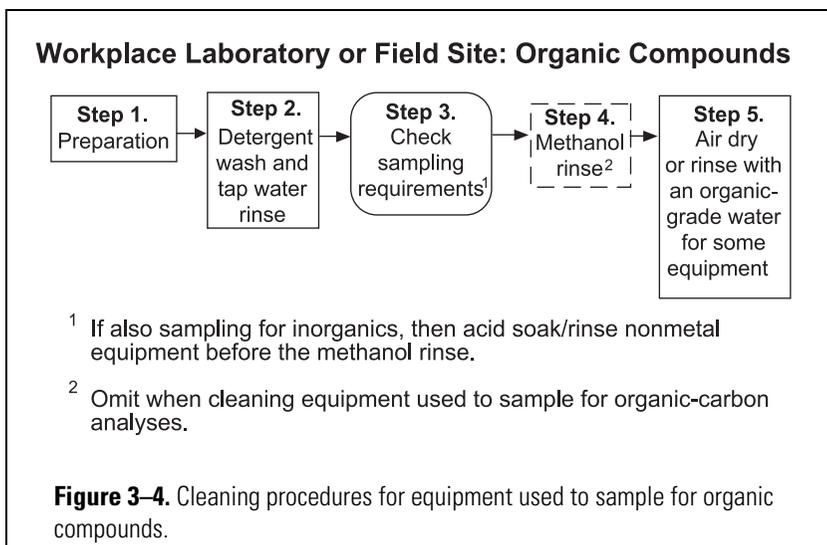
Clean equipment at the sampling site while equipment is still wet and before leaving for the next site.

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ORGANIC-COMPOUND SAMPLING EQUIPMENT 3.2.2

Nearly identical procedures are used in the workplace laboratory and at the field site to clean equipment used to sample for organic compounds. The workplace laboratory provides an environment in which equipment can be cleaned over an extended time using greater volumes of cleaning and rinsing solutions than in the field. The five-step cleaning procedure summarized in figure 3–4 is described in this section. If inorganic constituents also will be sampled for, check the sequence of cleaning solution to be used as shown in figure 3–1 before proceeding.

- ▶ Preclean a separate set of equipment for each site in order to avoid field cleaning of equipment between sampling sites. Always rinse equipment with DIW directly after use, however.
- ▶ If individual or dedicated sets of equipment for each field site are not available or cannot be precleaned, field clean equipment before moving to the next sampling site and process additional field blanks for each field trip (Koterba and others, 1995).
- ▶ Collect additional field blanks after cleaning equipment that was exposed to high levels of contamination (NFM 4) and before the equipment is reused for environmental sampling.



Standard procedures for workplace-laboratory and field-site cleaning of equipment used to collect and process samples for organic-compound analysis are described below and summarized in figure 3–4. Not all the steps listed apply to all equipment, however. For example,

- ▶ **Omit any cleaning procedure for sample bottles for organic compounds** (for example, pesticides, volatiles, and total particulate or dissolved organic carbon). Bottles for organic analyses that arrive from the laboratory should be capped and ready for use and should not be rinsed by field personnel. Discard these bottles if received uncapped.

- ▶ **Omit the methanol rinse when cleaning the equipment used to collect and process samples for total particulate carbon, particulate organic carbon, and dissolved and suspended organic carbon (TPC, POC, DOC).** If equipment (such as a submersible pump) has been in contact with methanol or other organic solvent and must be used for TPC, POC, or DOC sampling, then flush the equipment with copious quantities of sample water before collecting the sample; collection of a blank sample for DOC quality control is recommended.

Before proceeding with the workplace-laboratory and field-site procedures, be sure to check the specific procedures that are listed in section 3.3 for cleaning selected equipment. Also check for cleaning procedures that are specific to the needs of your study (for example, the NAWQA Program permits no deviation from the methanol-cleaning protocol).

To clean equipment used when sampling for organic compounds:

Step 1. Preparation (fig. 3–4).

- a. Prepare a contaminant-free space for cleaning and drying the cleaning supplies and sample-collection and sample-processing equipment.
 - i. Gather the cleaning supplies, the equipment to be cleaned, and clean storage bags and aluminum foil with which to wrap the cleaned equipment. (Check table 3–1 for the cleaning supplies needed.)
 - ii. Cover the cleaning area with aluminum foil.
 - iii. Put on disposable, powderless gloves,³ a laboratory coat or apron, and safety glasses. **Gloves provide protection from direct contact with solvents only for a limited period of time.**
 - iv. Prepare the detergent solution, using nonphosphate laboratory-grade detergent. A 0.1- to 0.2-percent (v/v) solution is normally of sufficient strength, unless equipment is very oily or greasy. **Do not use greater than a 0.2-percent solution for field cleaning.**
- b. Clean the items to be used to clean the equipment.
 - i. Label each washbasin, standpipe, and wash bottle with a black waterproof marker to indicate the solution it will contain.
 - ii. Follow Steps 2–5, listed below, to clean the washbasins, standpipes, wash bottles, and other items to be used for equipment cleaning.
- c. Disassemble sample-collection and sample-processing equipment. Submersible pumps should be disassembled periodically for workplace cleaning but usually are not disassembled for field cleaning.

³Refers to laboratory gloves that are nonpowdered on and intended for disposal after one use. Glove materials must be appropriate for the work to be carried out and the solutions and equipment to be contacted. Use nitrile or other solvent-resistant gloves when cleaning with organic solvents. **Be aware of possible allergic reactions to latex or other glove materials; discontinue use immediately if an allergic reaction occurs.**

Step 2. Detergent wash and tap water rinse (fig. 3–4).

- a. Place small equipment parts into washbasin labeled for detergent. Fill washbasin with a 0.2-percent solution of nonphosphate, laboratory-grade detergent. (The specific concentration of detergent solution depends on how contaminated the equipment might be and on the hardness of the tap water.) Change gloves.
 - **Workplace laboratory.** Soak equipment in detergent solution for 10 to 30 minutes.
 - **Field site.** Rinse equipment exterior and interior with detergent solution.
- b. Scrub the exterior and interior of equipment surfaces to the extent possible, using a firm sponge or soft brush to remove any adhering material such as oil and grease, sediment, algae, or chemical deposits. Pay particular attention to removing material from areas where particulate materials might be trapped, such as grooves and crevices, O-rings, and nozzles.
- c. Place equipment into tap water washbasin.
- d. Rinse equipment thoroughly with tap water to remove detergent residue. Use an organic-grade water (PBW, VPBW, or workplace-produced) if tap water is unavailable or is of a quality so poor as to contaminate the equipment. If necessary, use a wash bottle filled with organic-grade water or tap water to rinse hard-to-reach places. Equipment rinsing is complete if no detergent bubbles appear when rinse water is agitated. Change gloves.

Step 3. Check sampling requirements (fig. 3–4).

- a. If samples will be collected for organic analysis only, go to Step 4.
- b. If samples will be collected for inorganic analysis in addition to organic analysis, follow the procedure for the acid wash and DIW rinse before proceeding with the methanol rinse (see figs. 3–1 and 3–4).

Step 4. Methanol rinse⁴ (fig. 3–4).

- a. Use gloves that are chemically resistant to any solvent being used. Place cleaned equipment into a clean stainless steel or organic-solvent-resistant washbasin. Methanol-rinse area must be outside of the field vehicle and away from the sample-processing site. **Sample-collection, processing, and preservation areas must remain free of solvent vapors.**
- b. Use pesticide-grade methanol (or appropriate organic solvent) dispensed from a methanol fluorocarbon-polymer wash bottle (workplace laboratory) or pumped through tubing (field site) (see **TECHNICAL NOTE** below).
 - i. Rinse equipment exterior and interior with a minimum amount of methanol.
 - ii. Rinse only the interior (not the exterior) of pump tubing with methanol if collecting samples for analysis of organic compounds such as pesticides.
Exception: A methanol rinse of pump tubing is not recommended if TPC, POC, or DOC samples will be withdrawn through that tubing.
 - iii. Place equipment components and tubing on a clean aluminum foil surface. If also collecting samples for analysis of metals, use clear plastic sheeting instead of aluminum foil.
 - iv. Pour or discharge used methanol (or other organic solvent) into an appropriate waste container for flammable liquids (Water Resources Discipline Memorandum 94.007). Change gloves. Dispose of gloves used for methanol rinse appropriately.

CAUTION: Use methanol or other organic solvents sparingly and work under a fume hood or in a well-ventilated area, away from where an open flame or sparks can occur. Wear safety gloves, glasses, and apron.

⁴The methanol rinse is used to remove organic contaminants from equipment and represents standard procedure for all USGS studies. The NAWQA Program mandates use of the methanol rinse with no exceptions.

TECHNICAL NOTE: Rinse equipment with dichloromethane or hexane if the methanol rinse is not sufficient to clean equipment contaminated with excessive concentrations of hydrophobic organic compounds. If rinsing with dichloromethane or hexane, use pesticide-grade solutions, wear nitrile gloves, and use only on dry equipment (dichloromethane and hexane are not soluble in water). Do not rinse equipment with any organic solvent if equipment will be used for TPN, POC or DOC samples.

Step 5. Air dry equipment or rinse with organic-grade water (fig. 3–4).

- a. Allow methanol-rinsed equipment to air dry in an area free from dust and potential airborne contaminants (place an aluminum foil tent loosely over the drying equipment; if also collecting samples for metals analysis, use lint-free, nonabrasive wipes instead of aluminum foil).
- b. If it is not practical for the methanol to evaporate from the interior of equipment components or sample tubing, either
 - dry by blowing clean, filtered, inert gas through equipment; or
 - rinse methanol from equipment with pesticide-grade or volatile-grade blank water, dispensed from a wash bottle or pumped with a valveless fluid metering pump. This rinse water should be collected and disposed of properly, according to the regulations in effect for your locality (consult your safety officer).

Storage of clean equipment

Cover all equipment orifices with aluminum foil or fluorocarbon polymer bags, then place equipment into sealable storage bags. Isolate equipment used to collect trace-element samples from aluminum foil.

SPECIFIC PROCEDURES 3.3 FOR CLEANING SELECTED TYPES OF EQUIPMENT

By A.J. Horowitz, M.W. Sandstrom, and F.D. Wilde

The equipment-cleaning steps described in sections 3.2.1 and 3.2.2 apply to most, but not all, equipment. This section describes the cleaning procedures needed for specific equipment for which the general protocols are modified or do not apply, or for which more detailed instructions might be useful. Always wear appropriate disposable, powderless gloves throughout each cleaning procedure, and change gloves with each change of cleaning solution (see section 3.2).

INORGANIC-SAMPLE 3.3.1 BOTTLES

Bottles for samples to be analyzed for inorganic constituents include translucent colorless polyethylene, opaque brown polyethylene, and transparent glass bottles. Translucent polyethylene bottles that were acid rinsed at the laboratory should arrive capped with colorless, translucent plastic caps. Glass bottles for samples for mercury analysis also are acid rinsed and should arrive capped.

- ▶ **Discard acid-rinsed bottles that are received uncapped.**
- ▶ The more rigorous cleaning procedure that follows is required for bottles that will contain samples to be analyzed for trace elements. This bottle-cleaning procedure also is recommended for bottles that will contain samples to be analyzed for major ions and nutrients.

Before leaving for the field, clean polyethylene and glass sample bottles, including acid-rinsed bottles, as described in the steps that follow:

1. Put on powderless, nitrile gloves.
2. Fill each bottle about one-quarter full of DIW and cap.
3. Shake vigorously and decant DIW.
4. Repeat the DIW rinse (Steps 2 and 3 above) two more times.
5. Following the last rinse, fill each bottle half full with DIW and cap the bottle.
6. Rinse exterior of bottle with DIW and dry with lint-free laboratory tissue.
7. Store bottles in doubled plastic bags.

CHURN SPLITTERS 3.3.2

The plastic (non-fluorocarbon) churn splitter is used primarily for samples to be analyzed for inorganic constituents, whereas the fluorocarbon churn splitter can be used to process samples for organic-compound analysis as well as for inorganic constituents (NFM 2).

When using the detergent wash/tap water rinse for the churn splitter—Workplace-laboratory procedure (fig. 3–2, Step 2):

1. Fill churn splitter through the top (using a funnel, if necessary) with detergent solution.
2. Soak for 30 minutes.
3. Scrub interior and exterior surfaces with a soft brush, taking care not to abrade the surface.
4. Pay particular attention to cleaning the paddle and the area around the spigot.
5. Make sure the churn spigot opening and funnel are free of sediment, including fine particulates (clay), organic matter, and stains.
6. Drain some of the cleaning solution through the spigot before discarding the remaining solution.
7. Fill churn splitter about one-third full with tap water; swirl and shake the churn vigorously to remove detergent residues. Allow tap water to pass through the spigot.
8. Repeat rinse procedure until no bubbles remain in rinse water after the water is agitated.

When using the acid rinse for the churn splitter—Workplace-laboratory or field-site procedures (figs. 3–2 and 3–3, Step 4):

1. Using a 1-L wash bottle filled with a 5-percent HCL or 10-percent HNO₃ acid solution (table 3–1), rinse the entire inside surface of the churn, allowing the acid solution to drain through the spigot. **Exception:** Do not use this step if using a plastic churn splitter with a metal sprig in the spigot.
2. Decant acid solution into the neutralization container.

When using the DIW rinse for the churn splitter—Workplace-laboratory or field-site procedures (figs. 3–2 and 3–3, Step 5):

1. Fill the churn splitter with DIW to about one-third full.
2. Swirl the DIW vigorously and pour it out of the top of the churn into the neutralization container.
3. Repeat the fill-and-swirl procedures of 1 and 2 above at least twice, checking the pH of the DIW after each swirl with narrow-range pH indicator strips.
4. **Pass a portion of the DIW through the spigot only after the DIW pH equals or is greater than either 6.0 or the pH of the DIW before acidification. Pour the rest of the DIW into the neutralization container.**

When using the methanol rinse (fig. 3–4, Step 4):

1. Rinse only the fluorocarbon churn with methanol. Do not methanol rinse the plastic churn splitter.
2. Using a fluorocarbon wash bottle filled with pesticide-grade methanol (table 3–1), rinse the entire inside surface of the churn, allowing the methanol to drain through the spigot and into an appropriate container designated for methanol waste.

For storage of a cleaned churn splitter—Workplace-laboratory or field-rinse site procedures:

1. Package a clean, dry churn splitter in two new plastic bags and loosely tie or secure with a nonmetal clip. If a churn splitter must be packaged while wet, use within 1 to 3 days and (or) keep chilled to prevent bacterial growth.
2. Place entire package into the churn carrier.

CONE SPLITTERS 3.3.3

The fluorocarbon-polymer cone splitter (NFM 2) is appropriate for splitting samples for inorganic or organic analyses. When cleaning the cone splitter (Office of Water Quality Technical Memorandum 97.03), pay particular attention to removing foreign material from threaded and hard-to-access parts. Field cleaning can be minimized by having separate, precleaned cone splitters available for each site and by keeping a supply of clean tubes to replace the used tubes for each site to be sampled.

When inorganic constituents will be analyzed in samples processed through the cone splitter:

Workplace laboratory. Follow the steps as described for figure 3–2.

Field site. Referring to figure 3–3:

1. Prepare the field site as described in section 3.2.1. Put on disposable, powderless gloves.
2. Rinse the splitter thoroughly with DIW.
3. Inspect the cone splitter. If it looks dirty, is suspected of being contaminated, or was allowed to dry between field sites without a thorough DIW rinse, or if the splitter will be used for sampling both inorganic and organic analytes, use the detergent-wash option. Change gloves.
4. Acid rinse by passing 1 L of 5-percent HCl solution through the cone splitter. Collect used acid solution into a neutralization container. Change gloves.
5. Rinse the cone splitter with at least 3 L of DIW. Collect the rinse solution into a neutralization container. Change gloves.
6. Allow the cone splitter to dry and then store in a clean plastic bag. Seal the bag and store in a second plastic bag or plastic storage container for transport to the next site. A cone splitter that is packaged into bags while wet should be used within 1 to 3 days and (or) kept chilled to prevent bacterial growth.

When organic compounds will be analyzed in samples processed through the cone splitter (fig. 3–4):

Workplace Laboratory. Follow the steps described for figure 3–4.

Field Site.

1. Prepare site as described in section 3.2.2. Put on appropriate disposable, powderless gloves; if a solvent will be used, select gloves that will withstand contact with the solvent.
2. Detergent wash and rinse equipment as described for figure 3–4.
3. Check equipment and sampling requirements. If splitter will also be used for inorganic sampling, follow acid-rinse directions before rinsing with methanol or other organic solvent.
4. Proceed with the methanol (or other organic solvent) rinse, if required (section 3.2.2). (The methanol rinse is required for the NAWQA Program, for example). A methanol-rinsed cone splitter, however, must not be used if the cone splitter will contact samples for analysis of TPC, POC, or DOC.

For storage of a cleaned cone splitter:

1. Allow the cone splitter to air dry.
2. If the cone splitter will not be used to process samples for inorganic constituents at the next site, wrap the spigot and other orifices in aluminum foil.
3. Place the cone splitter into a clean plastic bag and seal.
4. Store in a second plastic bag or plastic storage container for transport to the next site.

If a cone splitter must be packaged while wet, use within 1 to 3 days and (or) keep chilled to prevent bacterial growth.

FILTRATION EQUIPMENT 3.3.4

Filtration equipment includes disposable filtration devices and various reusable plate-filter and pressure-filter assemblies. Cleaning procedures for these types of equipment are described below.

DISPOSABLE FILTRATION DEVICES 3.3.4.A

Disposable filter units have a one-time use for processing samples to be analyzed for inorganic and carbon constituents, but must be rinsed with IBW or VPBW⁵ before use to remove any manufacturing residues and condition the filter membrane. The filtration device can be prerinsed in the workplace laboratory rather than at the field site as long as the device is kept chilled and used in less than 1 day. After filtering the sample, discard the filter unit and clean or replace the sample-delivery tubing (section 3.3). The procedure below describes sufficient cleaning of the filter for analysis of inorganic constituents at the low parts-per-billion (ppb) concentration level (Horowitz and others, 1994).

Rinse the disposable filtration device before use as follows:

1. Put on disposable, powderless gloves. Use Clean Hands/Dirty Hands techniques described in NFM 4. Remember: the Dirty Hands team member performs operations that are outside of the processing chamber and the Clean Hands team member performs operations that are inside the chamber.
2. In a processing chamber, remove the filter unit from any protective bags. Attach pump tubing to the inlet connector of the filter unit, keeping the tubing as short as possible. **When using the large-capacity capsule filter, make sure the direction of flow through the filter unit matches the direction-of-flow arrow on the filter housing.**

⁵If samples for DOC analysis will be filtered, then rinse the disposable unit with VPBW instead of IBW.

3. Pump IBW (or VPBW⁵) through the filter unit. Discharge the rinse water through a sink funnel or to a toss bottle.
 - Use 1 L of IBW (or VPBW⁵) to rinse the large-capacity⁶ ($\geq 600 \text{ cm}^2$ Effective Filter Area (EFA)) filter.
 - Use 100–50 mL of IBW (or VPBW⁵) to rinse the small-capacity⁷ (19.6 cm^2 EFA) (disc) filter.
 - Operate the pump at a low speed.
 - Hold the filter unit so the discharge end is pointing up at an acute angle from the horizontal plane. (This expels any trapped air; do not allow water to spray onto chamber walls.)
4. Remove tubing from the IBW (or VPBW) reservoir and continue to operate the pump in the forward, mid-range speed position to drain as much as possible of the IBW (or VPBW) that remains in the filter unit. While the pump is operating, shake the capsule filter to help remove any entrained IBW (or VPBW).
5. Detach the filter unit from the peristaltic pump tubing, put into a clean, sealable plastic bag, and store chilled until ready for use.

3.3.4.B PLATE-FILTER ASSEMBLIES AND IN-LINE FILTER HOLDER

To clean filtration equipment used for samples to be analyzed for inorganic or organic analytes, consult sections 3.2.1 and 3.2.2, respectively. Use Clean Hands/Dirty Hands techniques, as appropriate (NFM 4). The aluminum plate-filter assembly and the fluorocarbon (PFA) in-line filter holder are used most commonly for filtering samples for analysis of non-volatile organic compounds, such as pesticides. The plastic 144-mm-diameter plate-filter assembly no longer is in common use; if used, it should be cleaned by using the procedures prescribed in section 3.2.1 and by following the guidelines below and in figure 3–3. See section 3.3.4.C for information about cleaning filtration equipment used for carbon (TPC, PIC, and DOC) samples.

⁶For example, Pall-Gelman AquaPrep™ 600 or Whatman Inc. Polycap™ ground-water capsule filters.

⁷For example, Pall-Gelman AquaPrep™ sampling device.

- ▶ **Preclean in the workplace laboratory** one filtration device per site to be sampled, if possible, in order to save the time that would be needed to clean the plate-filter assembly during the field effort.
- ▶ **During the detergent wash and (or) DIW rinse**, pay particular attention to grooves and crevices, O-rings, and support structures for the filtration device, where sediment or organic matter might be lodged. Detergent wash and DIW rinse the pressure valve.
- ▶ **Remove and discard the used filter membrane or disc (filter) at the field site**; rinse the filtration device immediately with DIW while still wet from filtering the sample, even if clean equipment is available for the next site.

Always remove the used filter media from the plate-filter assembly before cleaning and storage.

When field cleaning the aluminum plate-filter or PFA in-line filter-holder assembly, use the general cleaning instructions in section 3.2.2 for figure 3–4, as follows:

1. Inspect the filtration assembly for damage or excessive contamination and replace if necessary.
2. Wearing disposable, powderless gloves, prepare the area to be used for cleaning the plate-filter assembly by lining the table or counter surface with aluminum foil.
3. Disassemble the filter assembly and carefully remove the used glass-fiber filter media to avoid spilling any of the filter cake. Place used filter into a sealable plastic bag, seal the bag, and put aside for disposal. Place components of the filtration assembly and tubing into a washbasin for detergent. Change gloves.

4. Detergent wash by using a 0.1- to 0.2-percent nonphosphate-detergent solution. Scrub each component of the filtration assembly with a soft brush to remove any adhering material such as oil and grease, sediment, algae, or chemical deposits. Pay particular attention to grooves and crevices, O-rings, and support structures for the filter disc, where particulate materials might be lodged. Pump detergent solution through tubing. Place components of the filtration assembly onto a clean, aluminum-foil-covered surface.
5. Discard detergent solution from basin, rinse basin with tap water, and place components of the filtration assembly into the basin. Change gloves.
6. Rinse each component thoroughly to remove detergent residue, paying particular attention to grooves and crevices. Use a wash bottle filled with DIW or tap water to rinse hard-to-reach places. Place rinsed components onto a dry section of clean aluminum foil or basin.
7. Change to solvent-resistant gloves. Place components of the filtration assembly into a clean, solvent-resistant washbasin.
8. Rinse the filtration assembly components with pesticide-grade methanol or an equivalent grade for other organic solvents while outside of the field vehicle and downwind of sampling activity. Do not methanol rinse any tubing or filtration assembly to be used for collecting or processing samples for TPC, PIC, or DOC analysis (see section 3.3.4.C). **The instructions for the methanol rinse apply also for use of any other organic solvent.**
 - a. Dispense methanol from a fluorocarbon-polymer wash bottle. Rinse all sample-contacting surfaces of filtration-assembly components and tubing over a solvent-resistant basin or waste container. **Methanol-laced rinse water must be collected into an appropriate waste container designed for flammable liquids.**
 - b. Place methanol-rinsed equipment components onto a clean aluminum foil surface to air dry. (Cover equipment components loosely with an aluminum foil tent, to protect from airborne contaminants.)
9. Reassemble the filtration assembly. Wrap nozzles with aluminum foil and seal the filtration assembly in plastic bags. Double bag for transport or for long-term storage.

PRESSURE-FILTRATION AND VACUUM-FILTRATION APPARATUS 3.3.4.C

The cleaning procedures described in section 3.2.2 for figure 3–4 must be modified for the pressure- and vacuum-filtration equipment used to filter for samples to be analyzed for TPC, PIC and DOC (DOC samples also may be filtered through the disposable devices described in section 3.3.4.A). The gas-pressurized filtration assembly for processing carbon samples is constructed of either stainless steel or fluorocarbon-polymer material. Vacuum-filtration equipment consists of a polypropylene flask and a polysulfone filter funnel.

- ▶ C-Flex tubing and glass graduated cylinders also are used. **Do not bring the pressure-filter assembly in contact with methanol or other organic solvent or organic-solvent vapors.**
- ▶ **Do not clean the pressure-filter assembly with detergent.** **Exception:** see Step 3 below.
- ▶ In general, workplace-produced organic-grade water that is prepared by being passed through appropriate columns to remove organic compounds is of adequate purity for cleaning this equipment; PBW or VPBW also can be used. Workplace-produced organic-grade water, however, must not be used instead of PBW or VPBW for preparing blank samples.

When cleaning carbon-sample filtration equipment:

1. Wearing disposable, powderless gloves, disassemble the filtration assembly before it dries and place the components and other carbon-related equipment into a clean washbasin. Change gloves.

2. Using workplace-produced organic-grade water, thoroughly rinse the equipment components and place into a washbasin or onto a clean surface. Generally, these steps should be sufficient to field clean the pressure-filter assembly.
 - If necessary, use a soft-bristled toothbrush to remove sediment, chemical deposits, and other foreign material from threaded components, gaskets, O-rings, support screens, grooves, and nozzles. Take care not to scratch or mar inner surfaces.
 - Rinse the filtration equipment thoroughly with workplace-produced organic-grade water, or PBW, or VPBW.
3. If the pressure- or vacuum-filtration assembly or other carbon-sample equipment is very dirty or contaminated, clean as follows:
 - a. Disassemble and soak components for at least 1 hour in a 0.1-percent solution of nonphosphate laboratory-grade detergent.
 - b. Clean with a soft-bristled toothbrush, as described above in step 2.
 - c. Rinse repeatedly with workplace-produced organic-grade water, being sure to remove all traces of detergent.
4. Place all components of the pressure-filter assembly onto aluminum foil or other clean surface and allow to air dry thoroughly under a protective aluminum-foil tent.
5. Reassemble equipment components in preparation for sample filtration, wrapping nozzles and other equipment apertures with aluminum foil. Seal in a storage bag.

Do not use methanol or other organic solvents on the equipment used to filter samples for organic-carbon analyses.

SAMPLE TUBING 3.3.5

Cleaning procedures are described below for the tubing and nozzles used with peristaltic and valveless metering pumps. Cleaning procedures for submersible pump tubing are described in section 3.3.10.B. Wear appropriate, disposable, powderless gloves throughout the cleaning process, changing gloves with each change in cleaning solution (section 3.2).

- ▶ Preclean the number of tubing sections needed at each site in the workplace laboratory rather than recleaning tubing in the field, in order to save time during field work. Place into doubled plastic bags and store tubing dry or store wet tubing chilled to prevent bacterial growth. If bacterial growth is present, reclean tubing before use.
- ▶ Use disposable tubing if possible, especially at contaminated sites, to avoid the cleaning process and prevent the possibility of cross contamination.

When using workplace-laboratory or field-site procedures for cleaning plastic (including fluorocarbon-polymer) tubing for inorganic-constituent samples:

Follow the general sequence of procedures summarized in figures 3–2 or 3–3.

1. Pump 1 L of 5-percent HCl solution through the tubing, discharging the used acid solution into a neutralization container. Pinch and release tubing near tubing outlet while pumping the acid through to ensure that all interior surfaces are acid rinsed.
2. Pump 2 L of DIW through tubing, using the pinch-and-release method. Discharge used DIW to an acid-neutralization container, and check that the rinse-water pH is greater than 6.0 or the original DIW pH.
3. Discard neutralized solutions appropriately.
4. Clean stainless steel connections or metal tubing using detergent-wash and tap water/DIW-rinse procedures.

When using workplace-laboratory or field-site procedures for cleaning tubing for organic-compound samples:

Follow the general sequence of procedures described for figures 3-1 and 3-4. Proceed with the methanol rinse after the detergent wash and tap water rinse, unless the tubing will be in contact with samples for organic-carbon analysis. If samples also will be collected for inorganic-constituent analysis, however, acid rinse the nonmetallic tubing and components after the detergent wash/tap water rinse and before continuing with the methanol rinse.

1. Pump 1 L of nonphosphate, laboratory-grade detergent solution through tubing, followed by sufficient tap water or DIW to remove detergent residue. Pinch and release tubing near tubing outlet while pumping the solution to ensure that all interior surfaces are cleaned.
2. Place discharge end of tubing from peristaltic or valveless metering pump over methanol waste container.
 - Pass one tubing volume of methanol through the same pump system used for filtration, using the same pinch-and-release method.
 - Short sections of tubing can be held over the waste container while dispensing the methanol from a fluorocarbon-polymer wash bottle instead of pumping the methanol through the tubing.
 - **Do not methanol rinse tubing to be used for samples for TPC or DOC analysis.**
3. Store tubing in doubled plastic bags.

CAUTION: Do not use methanol around equipment that can create electrical sparks (see section 3.3.10.B).

PROCESSING AND PRESERVATION CHAMBERS AND FLOWTHROUGH CHAMBERS 3.3.6

Processing and preservation chambers are used to protect samples from atmospheric contamination generally are portable and are assembled at the field site. Large, clear plastic bags usually are clipped to the inside of the frame rather than stretched over the frame. Plastic clips are used to hold the cover tightly in place.

The flowthrough chamber, used when monitoring ground-water field measurements, is connected inline to the pump sampler. The flowthrough chamber should be kept free of sediment and dirt or deposits on the chamber walls. Air dry and store the chambers in sealable plastic bags.

When cleaning the processing and preservation chambers:

Workplace laboratory. Clean the frame of portable chambers in the workplace with detergent solution, then rinse thoroughly with tap water and dry and store in plastic bags.

Field site. Frames require regular cleaning after each use at a site if chamber covers are stretched over the outside of the frame rather than being clipped to the inside of the frame.

1. Discard the used bag.
2. Rinse the chamber frame with DIW and wipe dry with a lint-free cloth or tissue.
3. Replace chamber cover only when the next samples are ready to be processed.
4. If the processing chamber is a fixed installation (not portable), clean out any spilled sample water, solid materials, or wash solutions, and swab down the inside using DIW and lint-free laboratory tissue.
5. Use detergent solution followed by a thorough tap water or DIW rinse if a spill has contaminated the chamber.
6. Store chamber frames in plastic bags.

When cleaning the flowthrough chamber:

1. Clean the flowthrough chamber in the workplace laboratory with detergent solution and rinse thoroughly with tap water, followed by DIW. **Do not use acid solution or methanol.**
 2. If the flowthrough chamber needs to be field cleaned, remove measurement sensors and clean with a dilute detergent solution; rinse thoroughly with tap water followed by DIW. If using the flowthrough cell of a multiparameter instrument, follow the manufacturer's instructions for cleaning the cell.
-

3.3.7 RADON SAMPLER

To clean radon samplers:

1. Soak radon samplers in a detergent solution for 10 minutes.
 2. Rinse thoroughly with tap water to remove detergent residue, followed by three to five rinses with DIW.
 3. Air dry the radon sampler and store in doubled plastic bags. **Do not use methanol on radon sampling equipment.**
-

3.3.8 WELL TAPES

Always inspect and clean well tapes after each use. Procedure 1 below describes general cleaning instructions for electric and steel tapes that are used to measure water levels in monitor or observation wells that do not have fecal contamination. Procedure 2 below describes disinfection instructions for well tapes that are used to measure water levels in wells susceptible to fecal contamination, including public-supply and domestic wells.

- ▶ Before using these procedures, check with the well owner or wellfield project manager to determine whether site restrictions would be compromised by the use of any of the cleaning substances described.
- ▶ Check the instructions provided by the manufacturer before using a cleaning or disinfection agent on your electric tape other than laboratory detergent and water.
- ▶ The term "electric tape" is used here to include the sensor and other wetted parts of electronic instruments that are used to measure water levels in wells.
- ▶ Spread clean plastic sheeting on the surface over which the tape will be cleaned, to prevent the tape from contacting the ground or other potential sources of contamination.

PROCEDURE 1. This cleaning procedure contains two options for cleaning well tapes used in monitor or observation wells **that are not susceptible to fecal contamination**, and therefore do not require disinfection. Option A is the standard procedure for cleaning well tapes when no oily substance is present on the water table. Option B describes how to clean oil residues from the well tape. Inspect the well tape before starting the cleaning procedure to determine whether Option A or Option B should be used.

Option A: Cleaning an electric or steel tape where no oily residue is noticeable on the tape:

When using an electric tape and sensor equipment, follow the manufacturer's instructions for equipment care and cleaning. Steps 1 through 3 can be used in the absence of manufacturer's instructions.

1. Wash the tape with a nonphosphate, 0.1- to 2-percent laboratory detergent solution (for example, Liqui-Nox⁸), using a soft cloth or a soft brush.
2. Rinse the tape thoroughly with DIW or tap water to remove all traces of the detergent solution.

⁸Liqui-Nox and Detergent 8 are products of Alconox, Inc. Reference to these products is for descriptive purposes only and does not imply endorsement by the U.S. Geological Survey.

3. Dry the electric or steel tape with a clean, soft cloth, and rewind the tape onto the reel. Place the tape into a clean plastic bag for transport and storage.

Recommendation: If the tape will be stored for a month or more, put a drying agent such as a silica-gel packet into the plastic bag before sealing the bag.

Option B: Cleaning an electric or steel tape that is coated with oil:

When cleaning an electric tape, check with the manufacturer before exposing the tape to a solvent. This procedure should be carried out away from the well site.

1. Wearing solvent-resistant disposable gloves, prepare a nonphosphate, laboratory detergent solution (for example, 10-percent Liqui-Nox or 3-percent Detergent 8).
2. Use a soft brush, a clean terry cloth, or a sponge that is saturated with the detergent solution to remove oil from the wetted portion of the tape.
3. If an oily residue persists, use a clean cloth wetted with a solvent (such as a 10-percent naphtha solution or a 70-percent ethanol, methanol, or acetone solution) and wipe down the oily portions of the tape. Allow the tape to air dry in a well-ventilated area.
4. Using tap water or DIW, thoroughly rinse the detergent from the tape, and then dry the tape with a clean cloth.
5. Rewind the tape onto the reel and place it into a clean plastic bag for storage and transport.

Recommendation: If the tape will be stored for a month or more, put a drying agent such as a silica-gel packet into the plastic bag before sealing the bag.

Caution: Solvents mentioned above are flammable, explosive, and produce noxious fumes. Store these solvents in appropriate solvent-resistant containers that can be tightly capped and that are clearly labeled with its contents and hazards. A Material Safety Data Sheet (MSDS) must be displayed in the vicinity where the solvent is stored. Always wear disposable, solvent-resistant (for example, nitrile) gloves when working with solvents or other chemical substances. Do not leave the solvent in the sun or in a hot vehicle.

PROCEDURE 2. A well tape should be disinfected when it is being used to measure water levels in public-supply or domestic wells, or in wells susceptible to fecal contamination from other human or animal operations. **Begin Procedure 2 with a well tape that has been cleaned with a detergent solution, as described above in Procedure 1.**

Disinfecting an electric or steel tape:

1. **Select a disinfectant: either a chlorine bleach solution (described below) or a methyl or ethyl alcohol solution. If using bleach, prepare a dilute 50 mg/L (0.005 percent) solution of common household chlorine bleach (1 mL of bleach to 900 mL water⁹). If using an electric tape, check with the manufacturer before exposing the tape and related equipment to a solvent.**
 - a. Label a polyethylene sample bottle as "Well-Tape Disinfectant" and record the date of preparation, using an indelible marker. The bleach solution should be prepared fresh for each day of use (NFM 7.3).
 - b. Fill the bottle with the dilute disinfectant solution. Cap the bottle tightly, and double-bag it in a ziplock plastic bag for transport.
2. At the well site, put on disposable gloves. Wet a clean cloth with the disinfectant solution and use it to wipe down the section of the tape that was submerged in the well water.
3. Rinse the tape thoroughly with DIW or tap water. Using another clean cloth, wet and wipe the surface of the well tape through the entire chalked and wetted sections of the tape. Be thorough when rinsing bleach solution from a well tape; prolonged exposure of the tape to chlorine bleach can damage the tape.
4. Using a clean dry cloth, dry the steel tape thoroughly to prevent it from rusting.
5. Rewind the tape onto the reel and place it into a clean plastic bag for storage and transport.

Recommendation: If the tape will be stored for a month or more, put a drying agent such as a silica-gel packet into the plastic bag before sealing the bag.

⁹Prepare a 0.02 percent (200 mg/L) solution if pH is less than 6 or greater than 8 (NFM 7.3).

3.3.9 SURFACE-WATER SAMPLING EQUIPMENT

Disassemble surface-water samplers for cleaning and follow the sequence of procedures described in section 3.2 and figures 3–2, 3–3, or 3–4, as appropriate.

When using workplace-laboratory procedures for cleaning surface-water samplers:

1. Periodically disassemble samplers for workplace-laboratory cleaning. **Discard the bag sampler bag after one use**—do not attempt to scrub or detergent wash the used bag. Prepare cleaning solutions, cleaning equipment, and cleaning area as described in section 3.2.
2. Soak components in detergent solution for 30 minutes. Put on appropriate disposable, powderless gloves. Scrub components with a soft brush or sponge and rinse thoroughly (section 3.2.1 or 3.2.2). Change gloves.
3. Check the sequence of cleaning procedures (fig. 3–1).
 - a. If the sampler is used for sampling inorganic constituents, soak each nonmetallic component in a 5-percent trace-metal-grade HCl solution for 30 minutes, followed by copious rinsing with DIW (section 3.2.1). **Acid rinse only nonmetal parts.** Change gloves.
 - Acid must not contact the metal collar on the DH-81 sampler.
 - Make sure that the nozzle is unscrewed from the cap.
 - b. If the sampler is used for collecting organic-compound samples, rinse each component with pesticide-grade methanol dispensed from a fluorocarbon-polymer wash bottle and allow to air dry (section 3.2.2). **Do not methanol rinse tubing or components that will contact TPC or DOC samples.** Change gloves.
4. If collecting an equipment blank (section 3.4), change gloves and rinse each component with the appropriate blank water before collecting the blank sample.

5. Reassemble the sampler. If the sampler is dedicated to sampling for organic compounds, double wrap the sampler nozzle in aluminum foil. Place the sampler into double plastic bags and seal for storage and transport.

When using field-site procedures for cleaning surface-water samplers:

1. Unwrap precleaned washbasins (one for each cleaning solution to be used).
2. Disassemble the used sampler into its component parts (bottle, cap, nozzle) so that all of the pieces can be thoroughly wetted with the various rinses. **Discard the previously used bag-sampler bag** (do not attempt to clean the bag for reuse).
3. Wearing appropriate disposable gloves, thoroughly rinse the sampler components with DIW. Use a stream of DIW from the wash bottle, if required.
4. Check whether target analytes are inorganic constituents, organic compounds, or both. Review figure 3–1 for the appropriate cleaning sequence.
 - a. If a sampler will be used for collecting samples for analysis of inorganic constituents only, change gloves and:
 - i. Thoroughly rinse the sampler components with tap water or DIW.
 - ii. Acid rinse nonmetallic components over a container using a stream of dilute acid solution from the appropriate wash bottle, if required.
 - iii. Thoroughly rerinse the sampler components with DIW over the same washbasin, if possible (see section 3.2.1). Change gloves.
 - iv. Place each component on a clean, plastic surface. Pour used acid solution and DIW rinse water into neutralization container.
 - v. Check the pH of the rinse solution in the neutralization container. Discard when solution pH is greater than 6.0 or the original DIW pH. Change gloves.

- b. If a sampler will be used for collecting samples for analysis of organic compounds only, change gloves and:
 - i. Detergent wash, then rinse sampler components thoroughly with tap water or DIW until agitated rinse water produces no more suds. Change to solvent-resistant gloves.
 - ii. Rinse sampler components with pesticide-grade methanol (section 3.2.2), collecting the used methanol into an appropriate container for safe storage until appropriate disposal is arranged. Omit this methanol rinse if sampler will be used for collection of TPC or DOC samples.
 - iii. Place each component on a clean, aluminum-foil-covered surface to air dry and cover loosely with an aluminum foil tent, if airborne contaminants are a concern. Change gloves.
- c. If sampler will be used for collecting samples for both organic and inorganic analyses, change gloves and:
 - i. Proceed with a detergent wash and thorough tap water and (or) DIW rinse.
 - ii. Acid rinse and DIW rinse nonmetallic components, as described above, discarding used solutions appropriately. Change to solvent-resistant gloves.
 - iii. Rinse with methanol, as described above, except for equipment used to collect TPC or DOC samples.
 - iv. Place cleaned items on a clean plastic surface to air dry.
5. Reassemble sampler. If the sampler is dedicated to sampling for organic compounds, double-wrap sampler nozzle in aluminum foil. Place sampler into doubled plastic bags for storage and transport.

Do not use methanol or other organic solvents on equipment used to collect organic-carbon samples.

GROUND-WATER SAMPLING 3.3.10 EQUIPMENT

Ground water is sampled with nonpumping samplers (such as bailers, syringe samplers, and the Kemmerer sampler) and with pumping samplers (such as peristaltic and valveless metering pumps and submersible pumps) (NFM 2.1.2). Workplace-laboratory cleaning procedures are used before a sampler is used for the first time, after the sampler has been in long-term storage, and whenever the sampler has become excessively contaminated. Field-site cleaning procedures are used after sampling at a field site and before proceeding to the next sampling site. Caveats and modifications that apply to the general workplace-laboratory and field-site cleaning procedures (section 3.2) are described in this section. The cleaning procedures used should be documented on field forms.

The rinse with methanol, or other organic solvent, is appropriate only for samplers being used to collect samples for organic-compound analysis. **Solvents are never used to clean equipment when sampling for TPC or DOC.** Dispose of used methanol and all other cleaning solutions appropriately.

TECHNICAL NOTE: Sampler components made of fluorocarbon-polymer plastic generally can withstand a solvent rinse with methanol. Check with the manufacturer before using an organic solvent on pump components constructed of any other plastic material.

3.3.10.A BAILERS AND OTHER NONPUMPING SAMPLERS

Workplace-laboratory procedure. Clean nonpumping samplers in a designated area of the workplace laboratory. Follow the procedures described for figures 3–2 and 3–4, as appropriate for equipment used to sample for inorganic constituents or organic compounds, respectively.

Field-site procedure. Follow the field-site cleaning procedures described for figures 3–3 and 3–4, as appropriate for equipment used to sample for inorganic constituents or organic compounds, respectively.

- ▶ Rinse the outside of the sampler with DIW directly after use.
- ▶ After filling the sampler with each cleaning solution, shake the sampler vigorously and drain solution through the bottom-emptying device, spigot, or nozzle of the sampler.
- ▶ If the sampler looks very dirty or is contaminated, disassemble and clean sampler components using the workplace-laboratory procedure.

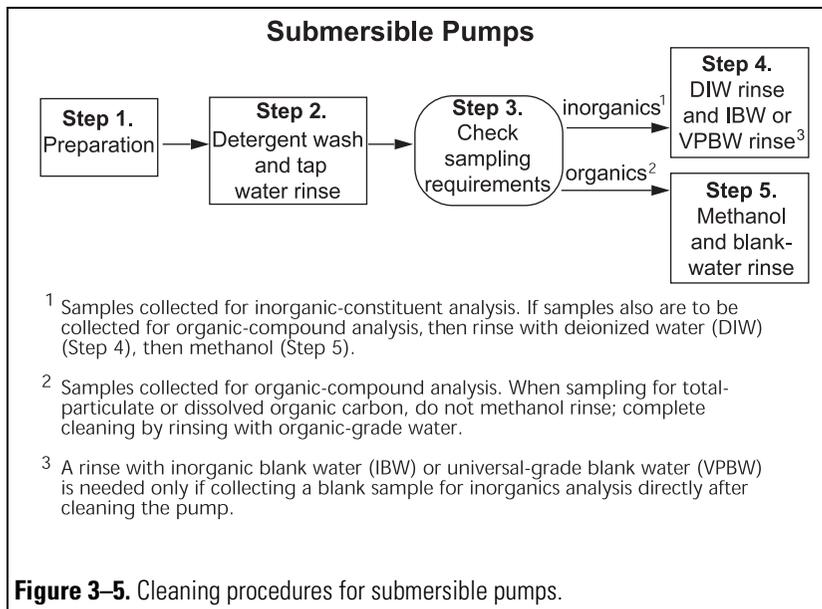
SUBMERSIBLE PUMPS 3.3.10.B AND SUBMERSIBLE-PUMP TUBING

The general sequence shown in figure 3–5 is appropriate for cleaning most submersible pumps. The field-site cleaning procedure (described below after the workplace-laboratory procedure) is sufficient for routine cleaning of the pump in most cases. Collection of blank samples for quality control must be included as a standard protocol for every study in order to document and ensure the efficacy of the cleaning procedure for the field conditions encountered.

- ▶ Fluorocarbon-polymer tubing used to collect water containing large concentrations of volatile organic compounds (VOCs) can be difficult to clean adequately.
 - Collect additional blanks if VOC concentrations in the last sample collected through the tubing were greater than 500 µg/L.
 - Pump tubing should be replaced rather than cleaned if VOC concentrations in the last sample exceeded about 700 µg/L.

- ▶ Most submersible pumps have a stainless steel casing and other metal parts and should not be acid rinsed.
 - To clean pumps that are excessively contaminated, a dilute acid rinse followed by copious water rinsing can be used occasionally without damaging the pump.
 - Repeated rinsing with dilute acid solution can pit or corrode the pump's stainless steel surface. If the surface appears dulled, then the pump should not be used for collecting trace-metal samples.

- ▶ Lubrication water inside water-lubricated pumps (for example, the Grundfos RediFlo2™) can become contaminated and potentially result in contamination of subsequent samples. Replace the lubrication water with VPBW after sampling and when cleaning the pump. Follow manufacturer's instructions.



Workplace-laboratory pump-cleaning procedure:

Use workplace-laboratory procedures about once a year and more frequently if results of the pump blank or other information indicate that the pump is contaminated.

Step 1. Preparation.

- a. Wearing appropriate gloves, prepare several gallons of a laboratory-grade nonphosphate detergent solution (about 0.1 or 0.2 percent, v/v; use up to 2-percent solution for excessively contaminated pump systems).
- b. Preclean washbasins and standpipes (section 3.2).
- c. Place pump into sink or waste basin and scrub exterior surfaces with soft brush and detergent solution; rinse thoroughly with tap water.
- d. Disassemble the pump and place components into a detergent-solution washbasin.

Step 2. Detergent wash and tap water rinse pump components and tubing.

- a. Soak pump components in the detergent solution for 30 minutes.
- b. Clean pump components with soft sponge or brush.
- c. Rinse thoroughly with tap water.
- d. Raise discharge end of tubing above the rest of the tubing. Using a peristaltic or valveless fluid metering pump, fill the pump tubing with fresh detergent solution until solution rises to the end of the tubing. Plug the tubing end(s).
- e. After 30 minutes remove plug from discharge end of tubing and flush detergent solution from tubing by pumping copious amounts of tap water through the tubing. Change gloves.

Step 3. Check sampling requirements.

- If pump will be used for collecting samples for inorganic-constituent analysis, then reassemble the pump and go to Step 4.
- Complete Step 4 if pump will be used for collecting samples for analysis of both inorganic and organic analytes before proceeding to Step 5.
- If the pump will be used for collecting samples for organic-compound analyses only, then go to Step 5.

Step 4. DIW rinse.

- a. Place pump components into DIW washbasin and dispense DIW from a wash bottle to thoroughly rinse all pump components.
- b. Using a peristaltic pump and appropriate clean tubing, pump DIW through the sample tubing to rinse.
- c. Reassemble pump and connect pump tubing. Change gloves.
- d. If collecting equipment blanks to verify that the pump has been adequately cleaned (section 3.4):
 - i. Rinse a clean standpipe dedicated to blank water with blank water.
 - ii. Insert pump into blank-water standpipe only after pump exterior has been rinsed with blank water or air dried after the methanol rinse.
 - iii. Pour IBW into the standpipe and pump at least one tubing volume to waste before collecting the blank sample.

Step 5. Rinse with methanol followed by a blank water rinse.

- a. Wearing latex or nitrile gloves, put pump components into solvent-resistant washbasin.
- b. Working under a fume hood, dispense methanol (or appropriate solvent) from a fluorocarbon-polymer wash bottle to rinse each pump component and the exterior pump casing. Collect the used solvent into a nonflammable container for storage until disposal.
 - **Do not reuse methanol or other solvents.**
 - **Work under a fume hood, if possible, or in a well-ventilated area outside of the workplace laboratory, as methanol fumes can contaminate other equipment.**
 - **Do not methanol rinse organic-carbon sampling equipment.**
- c. Place methanol-rinsed components on a clean, aluminum foil surface and allow the pump components and casing to completely air dry before reassembling the pump (see section 3.2.2). If also collecting samples for metals analysis, then use clean plastic sheeting instead of aluminum foil.
- d. Using a valveless fluid metering pump and fluorocarbon-polymer tubing, pump about 2 L of methanol through sample tubing and discharge to the methanol waste container.
- e. Reassemble the pump and connect the pump tubing. Change gloves and dispose of the methanol-contaminated gloves appropriately.
- f. Pour organic-grade water (PBW or VPBW) into a clean PBW/VPBW standpipe. Insert pump and pass about two tubing volumes of organic-grade blank water (PBW or VPBW) through the pump and tubing to waste.

CAUTION: Pumping methanol or other flammable solvents through an electrical pump system could be dangerous in the event of sparks. Methanol emits noxious fumes and is absorbed through the skin. Wear a mask, safety glasses, and other protective apparel to protect yourself when working with organic solvents.

Field-site cleaning procedure for submersible pumps and pump tubing:

Step 1. Preparation.

- a. Preclean the standpipes (one standpipe for each cleaning solution to be used, as described in 3.2.1). The standpipes need to be of sufficient height to supply necessary head for proper pump operation. Separate standpipes are designated for detergent solution and tap water rinse, DIW rinse, methanol rinse, and blank water (IBW/PBW/VPBW). Double-bag each cleaned standpipe for transport to the field site.
- b. Estimate the volumes of cleaning solutions and blank water that will be needed for the field effort (refer to fig. 3–6).
- c. Prepare the volumes of cleaning solutions needed for the field effort, using appropriate bottles for short-term storage and transport.

To estimate the volume of storage (V_s) in tubing of a set of pump-reel and extension tubing (in gallons)^{1,2}:

$$V_s = [(L_p \times C_p) + (L_e \times C_e) + V_{sp}] \times C_{sp}$$

where,

V_s is volume of storage in tubing, in gallons

L_p is length of pump-tubing segment being cleaned, in feet

L_e is length of extension tubing, in feet

C_p (or C_e) = 0.023 liter per foot for a 3/8-inch inside-diameter (ID) tubing
or = 0.041 liter per foot for a 1/2-inch ID tubing

V_{sp} is volume of solution needed to fill standpipe to minimum level
required to operate pump, in liters³

C_{sp} = 0.264 gallon per liter.

Examples

Given:

1. L_p - sample-wetted tubing segment is 100 feet for a pump-reel system that has a 1/2-inch ID tubing;
2. L_e - two, 10-foot, 3/8-inch-ID pieces of extension tubing, one running from pump-reel outlet to sample collection chamber, and another running from chamber back to pump-reel (return-flow tubing to standpipe); and
3. V_{sp} - minimum volume³ of solution required in standpipe to operate pump is 0.8 liter.

To estimate the volume of detergent solution needed for the detergent wash cycle:

$$V_s = [(100 \times 0.041) + (20 \times 0.023) + 0.8] \times 0.264 = 1.4 \text{ gallons}$$

The volume of workplace-produced deionized water needed to displace detergent solution and the volume of laboratory-produced organic-grade blank water needed to displace 2 liters of methanol just pumped into a system, ideally, would each be estimated to equal V_s ^{1,2}.

¹Estimate assumes no mixing of two solutions and ignores potential for detergent to adhere to tubing walls. Outflow from the discharge end of tubing should be checked for sudsing to determine that detergent has been removed.

²Estimate assumes no mixing at interface of two solutions and ignores potential for methanol to adhere to tubing walls. It is recommended that an additional 0.1 gallon (~ 0.4 liter) of blank water (pesticide-grade blank water or volatile-grade blank water) be used for each 10 feet of tubing to remove methanol residues from sample-wetted sections of tubing. Thus in the example above, another 1.1 (= (100 + 10) x (0.1/10)) gallons (4.2 liters) of blank water would be pumped from the system. This implies a total of about 2.5 (= 1.4 + 1.1) gallons (9.6 liters) of blank water would be used to remove methanol from the equipment setup.

³The minimum volume corresponds to the level of solution in the standpipe, which, if maintained, allows pump to operate without introducing air through the pump intake. Once this level is reached, remove pump, and measure this volume.

Figure 3–6. Estimation of cleaning-solution volumes for standpipe, pump, and pump tubing. [From Koterba and others, 1995, table 24.]

Step 2. Detergent wash and tap water rinse.

- a. Put on disposable, powderless gloves. Rest pump in a washbasin or pail partially filled with detergent solution and clean exterior of pump and tubing with a soft brush. Rinse thoroughly with tap water. (DIW can be used instead of tap water, but is less efficient in detergent removal and requires a greater volume of water than tap water.)
- b. Place pump into standpipe, add detergent solution to level above pump intake, and route the intake and discharge ends of pump tubing to the standpipe.
- c. Begin pumping:
 - i. Record the pumping rate.
 - ii. Record the time it takes to fill the sample tubing.
 - iii. Calculate the time it takes for a segment of solution to complete one cycle (fig. 3–6).
- d. Circulate detergent solution for about three cycles through the tubing and back to the standpipe. If possible, pump detergent solution through tubing at alternating high and low speeds, and (or) introduce air segments between aliquots of the detergent solution to increase cleaning efficiency.
- e. Remove the discharge end of tubing from the standpipe and pump about two tubing volumes of detergent solution to waste, adding fresh solution to the standpipe as needed. Remove pump from standpipe.
- f. Rinse detergent from standpipe with tap water until sudsing stops.
- g. Rinse pump exterior with tap water. Place rinsed pump into the tap water/DIW standpipe; add tap water/DIW to level above pump intake. Begin pumping through sample tubing. Do not recirculate rinse water, but add water as needed to maintain water level above pump intake. Continue for five or more tubing volumes. Direct rinse water to waste, away from the vicinity of the wellhead and sampling area and (or) contain as required for disposal.
- h. Collect rinse water into a small bottle and stop the pump. Shake the bottle—if sudsing is observed in the rinse water, continue the rinse procedure until no suds appear in the rinse water. Change gloves.

Step 3. Check sampling requirements.

- If a pump will be used to collect samples for inorganic-constituent analysis, go to Step 4.
- Complete Step 4 if a pump will be used to collect samples for analysis of both inorganic and organic analytes and then go to Step 5.
- If a pump will be used to collect samples for organic-compound analysis only, go to Step 5.

Step 4. DIW rinse.

A separate DIW rinse is not required if DIW was substituted for tap water.

- a. Use a clean DIW-dedicated standpipe (not the tap water standpipe) and rinse the standpipe with DIW. Rinse pump exterior with DIW. Place pump into the DIW standpipe and add DIW to level above pump intake. Change gloves.
- b. Start pumping DIW. Rinse DIW through sample tubing without recirculating, using about three tubing volumes of DIW. Keep the DIW level above pump intake.
- c. If collecting field blanks to verify that the pump has been adequately cleaned (section 3.4):
 - i. Change gloves. Rinse clean blank-water standpipe with IBW (or VPBW). Rinse pump exterior with blank water.
 - ii. Place pump into the standpipe and add IBW (or VPBW) to cover the pump intake.
 - iii. Turn on pump and displace any water residing in the pump and tubing. Continue pumping IBW (or VPBW) for one tubing volume before collecting the blank sample.

Step 5. Methanol rinse.¹⁰

Make certain that the pump or other nearby electrically powered equipment is grounded, the power cord is intact, and potential sources of sparks do not exist before rinsing pump with methanol.

TECHNICAL NOTES:

– Inspect the integrity of the seals and O-rings on the pump-motor/pump-body housing. Water inside the motor housing may indicate that methanol vapors could enter the motor. Direct-current motors inherently spark because of the commutator ring. AC motors might spark if the insulation is frayed or burnt on the motor windings or any associated wiring.

– If flammable liquids are required for cleaning electrical pump systems, use extreme caution. Vapors from solvents such as methanol can ignite if a disruption in the motor lead-insulation system occurs in the vapor-enriched zone. (Ignition from a spark from an AC induction-type motor in good operating condition is not a concern if rated as using the National Electrical Code (NEC) at Class 1, Group D.¹¹)

- a. Wear latex or nitrile gloves, safety glasses, and apron. Work in a well-ventilated area outside of the field van and downwind of the sampling area.
- b. Place pump into a clean, dedicated, solvent-resistant standpipe and route discharge end of sample tubing to a methanol waste container. Add methanol solution to level above pump intake.
- c. Pump about 2 L of methanol through the sample tubing into the methanol waste container, keeping the level of solution above the pump intake. The operator should stand back from the pump as a safety precaution in the event that an electrical spark ignites the methanol. Carefully pour any unused methanol that is in the standpipe into a methanol waste container. Let the methanol in the standpipe evaporate to dryness. Change gloves.

¹⁰Reminder to NAWQA Program personnel: the methanol rinse is mandated for cleaning equipment to be used to collect samples for analysis of all organic compounds other than organic carbon.

¹¹NEC Class 1; Group D: Areas in which flammable gases or vapors may be present in the air in sufficient quantities to be explosive; atmospheres such as acetone, alcohol, ammonia, benzene, benzol, butane, gasoline, hexane, lacquer solvent vapors, naphtha, natural gas, propane, or gas or vapors of equivalent hazard (Cole-Parmer Instrument Company, 1997).

- d. Rinse pump exterior with organic-grade water and place pump into the standpipe. Add organic-grade water to the standpipe to push the methanol out of the tubing and into the methanol waste container. Pump at least an additional 0.1 gallon (about 0.38 L) of organic-grade water through the system for every 10 ft (about 3.05 m) of methanol-wetted tubing. Discharge this water to the methanol waste container.

TECHNICAL NOTE: The recommended organic-grade water is PBW or VPBW (designated for blank samples). Workplace-produced organic-grade water might not be of adequate purity, especially after being stored, and its use requires collection of additional blank samples for quality control (see section 3.4).

- e. Repeat (d) above with blank water (PBW or VPBW) pumped from a blank-water standpipe if blank samples will be collected for analysis of organic compounds.

A methanol rinse is most safely accomplished under a laboratory hood or in another well-ventilated, clean environment.

Storage of the cleaned submersible pump and tubing:

1. Place pump into two clean, noncontaminating storage bags and tie the bags shut.
2. Cover the pump reel and tubing with doubled plastic bags or sheeting for transport to the next site.

For long-term storage (longer than 3 days), the pump and exterior and interior of the tubing must be dry before being placed into plastic bags. Tubing can be dried by blowing filtered air or filtered (inert) gas through the tubing. If tubing cannot be dried, store chilled to prevent bacterial growth. If bacterial growth has occurred, reclean before use.

QUALITY CONTROL FOR EQUIPMENT-CLEANING PROCEDURES 3.4

By A.J. Horowitz, M.W. Sandstrom, and
F.D. Wilde

Quality-control samples are required for any sampling and analysis program. Without quality-control information, the quality of the environmental data collected can be neither evaluated nor qualified. If the user has no means of knowing the associated errors, the data cannot be interpreted properly.

The purpose for obtaining quality-control (QC) samples following equipment cleaning is to ensure that the equipment and the procedures used for cleaning the equipment do not contaminate or otherwise affect the environmental samples that were or will be collected. The QC sample used to assess the adequacy of cleaning procedures before field work commences is called the equipment blank. QC sampling guidelines for microbiological sampling differs and can be found in NFM 7.

- ▶ **Blank water.** Blank water is used to develop specific types of QC samples (National Water Quality Laboratory Memorandum 92.01). The water is a solution that is free of analyte(s) of interest at a specified detection level. USGS personnel are required to use blank water that has been analyzed and certified to be of a specific grade and composition.
 - Use IBW, PBW, or VPBW (nitrogen-purged) to collect blank samples for analysis of inorganic constituents.
 - Use PBW to collect blank samples for analysis of pesticides. (Do not use PBW when collecting samples for VOC analysis.)
 - Use VPBW, which is nitrogen-purged, to process blank samples for analysis of VOCs. VPBW also is suitable as a blank sample for pesticide and inorganic-constituent analyses.
 - Use PBW or VPBW as the quality-control sample for total-particulate carbon and dissolved organic-carbon analyses (TPC and DOC). This cannot be strictly documented as a blank sample because neither PBW nor VPBW is certified to be free of organic carbon.

- **Equipment blank.** An equipment blank is blank water that is processed under controlled conditions in the workplace laboratory by being passed sequentially through each component of the sample processing and collection equipment. **An equipment blank represents an entire sampling system (fig. 3–7) and is required:**

- Annually.
- When a cleaning procedure is followed for the first time.
- When new equipment will be used for the first time.

To fulfill equipment-blank requirements:

1. Allow enough time in the study workplan to collect the annual equipment blank, complete laboratory analyses, and review analytical results before field work for the study commences.
2. Process the annual equipment blank in a clean, controlled environment in the workplace laboratory, after the equipment has been cleaned using workplace-laboratory procedures.
3. Analyze the annual equipment-blank data before collecting and processing the first water-quality sample of either the fiscal year or the study.
 - If the equipment-blank data indicate that the equipment does not introduce contaminants that will bias study results, then sampling can proceed.
 - If the equipment-blank data indicate unacceptable concentrations of analytes of interest, then the cause must be identified and the equipment or cleaning procedures must be changed or modified before sampling can proceed.

Plan ahead: Assess equipment-blank data before environmental samples are collected.

- ▶ **Field blank.** The field blank is blank water that is processed at the field site by being passed sequentially through each component of the equipment being used to collect environmental samples. The procedure for processing the field blank, like the equipment blank, can also result in a set of sequentially collected blank samples (fig. 3–7) (Horowitz and others, 1994). Other types of blank samples also are collected at the field site (NFM 4). **At least one field blank per sampling run is recommended; the numbers and distribution of QC samples depend on study objectives, the target analytes, and site conditions.**
 - Process field blanks through clean equipment.
 - If equipment is used at several sites during a field trip, process a field-equipment blank after the last sample has been collected and again after the equipment has undergone the prescribed field-cleaning procedures.
 - If multiple sets of workplace-cleaned equipment are used during a field trip, process a field blank at any site during the course of the trip. In this case, the blank must be processed before sampling to avoid contaminating the blank with residues from an environmental sample.
 - Process field blanks onsite and under the same conditions as the environmental sample.

Before filling the QC sample bottle with the appropriate blank water:

1. Check that sample bottles are clean, are the correct type, and are labeled correctly.
2. Check the certificate of analysis for the lot of blank water to be sure that it is appropriate for quality control of target analytes.
3. Record the date and lot number of the IBW, PBW, and (or) VPBW used and of the preservative used. To the extent possible, use preservative from the same lot number for an entire sampling trip for both the environmental and quality-control samples.
4. Rinse sample bottles for inorganic constituents three times with a small quantity of the blank water.

Use the following strategy for QC data collection and analysis:

1. For inorganic-constituent samples, initially send only the final equipment-blank sample for the routine inorganic blank-sample analysis or for inorganic analytes targeted by the study.
 - Archive the remaining sequentially processed blank samples (fig.3-7) until the inorganic-constituent analysis of the equipment-blank sample has been received.
 - Do not archive blank samples for organic-compound analysis.
2. Check the analytical results for the equipment blank and field blanks as soon as possible and before the next field trip.
 - If analytical results indicate that the equipment is clean within acceptable limits, the equipment may be used for field work without additional testing or analysis.
 - **Use of equipment is not recommended if analysis of the equipment blank sample indicates greater than acceptable analyte concentrations.**
3. Additional QC data collection and (or) analysis is required if the equipment blank has greater than acceptable analyte concentrations.
 - **For inorganic-sample analysis.** Submit the rest of the sequential blank samples for laboratory analysis and use the analytical results from the sequential blank samples to identify potential source(s) of contamination. Modify equipment-cleaning procedures if contamination can be remedied by a change in cleaning procedure. Repeat collection of equipment blanks until the blank data verify that the equipment is suitable for use.
 - **For organic-sample analysis.** Modify the equipment cleaning procedure if the source of contamination is known or suspected and contamination can be remedied by a change in cleaning procedure. If the source of contamination is not known, reclean equipment using workplace-laboratory procedures and collect and analyze blanks for each part of the sampling system that could be a source of contamination. Repeat collection of equipment blanks until the blank data verify that the equipment is suitable for use.

The equipment blank is the last sample of a set of sequentially processed blanks collected in the workplace laboratory, and documents the suitability of the equipment for the samples that are to be collected and analyzed. **Field blanks** are collected in the field in the same manner as the equipment blank, but they document the effectiveness of the field-cleaning procedures plus any ambient contamination.

- **Surface water:** collect the series of five sequential blank samples listed below for routine surface-water sampling.
- **Ground water:** collect the source-solution blank (Sample 1) and either a sampler blank (Sample 2) or pump blank (Sample 4), depending on the type of sampling device being used along with the filter blank (Sample 5).

Sample 1. Source solution (SS)

SS blank Put on disposable gloves. Pour the IBW, PBW, or VPBW directly into appropriate SS blank-sample bottle.¹ Add chemical treatment and (or) chill, as required for the analytes of interest.

Sample 2. SS + Sampler

Sampler blank Bottle or bag sampler: Fill sampler container with SS; attach sampler cap and nozzle; decant sample into blank-sample bottle through the nozzle. Preserve sample (add chemical treatment and (or) chill) as required (NFM 5).

Bailer or thief sampler: Fill sampler with SS; install bottom-emptying device; empty sample into blank-sample bottle through the bottom-emptying device. Preserve sample, as required.

Submersible or nonsubmersible pumps: Go to Sample 4 (Pump blank).

Sample 3. SS + Sampler + Splitter²

Splitter blank If a cone or churn splitter is used, decant remainder of the SS into sampler container, and then through splitter (through nozzle or bottom-emptying device). Refill sampler container with SS to fill churn with 3 to 5 liters of water. Alternatively, pour enough SS from samplers through cone splitter to fill splitter-blank bottle. Collect SS into blank-sample bottle through churn spigot or cone-splitter exit port(s). Preserve sample, as required.

Sample 4. SS + Sampler + Splitter + Pump

Pump blank Nonsubmersible pump (peristaltic, vacuum, or valveless metering pump): Secure intake end of clean pump tubing into churn splitter or into a subsample split with the cone splitter. Pump some sample to waste to rinse tubing, and fill pump-blank bottle directly from the discharge end. Preserve sample, as required.

Submersible pump: Place pump in blank-water standpipe and fill standpipe with enough SS to cover pump intake and allow for drawdown. Start pump at low pumping rate, discharge 0.5 liter of SS to waste, then fill blank-sample bottle with SS. Preserve sample, as required.

Sample 5. SS + Sampler + Splitter + Pump + Filter

Filter or equipment blank Pump SS through a prerinse filtration assembly (plate filter or capsule filter); pump the first aliquot to waste and then pump SS directly into the blank-sample bottle. Preserve sample, as required.

¹Process the source-solution blank in the protected environment of the workplace laboratory only, not in the field (NFM 4).

²For ground-water quality control: A splitter blank is included if a cone splitter is used; a standpipe blank commonly is collected if a submersible pump is used.

Figure 3–7. Sequence of sample collection to obtain the equipment blank.

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CONVERSION FACTORS AND ABBREVIATIONS

CONVERSION FACTORS

Multiply	By	To obtain
centimeter (cm)	0.3937	inch
square centimeter (cm ²)	.1550	square inch
meter (m)	3.281	foot
milliliter (mL)	0.06102	inch ³ or cubic inch
liter (L)	0.2642	gallon
microgram (μg)	3.53 x 10 ⁻⁸	ounce

Temperature: Water and air temperature are given in degrees Celsius (°C), which can be converted to degrees Fahrenheit (°F) by use of the following equation:

$$^{\circ}\text{F} = 1.8(^{\circ}\text{C}) + 32$$

ABBREVIATIONS

DIW	deionized water
DOC	dissolved organic carbon
HCl	hydrochloric acid
HNO ₃	nitric acid
IBW	inorganic-grade blank water, laboratory-certified free of trace elements and other inorganic constituents
μg/L	micrograms per liter
μS/cm	microsiemens per centimeter at 25°C
MSDS	Material Safety Data Sheet
NFM	<i>National Field Manual for the Collection of Water-Quality Data</i>
NAWQA	National Water-Quality Assessment Program of the U.S. Geological Survey
NWQL	National Water Quality Laboratory of the U.S. Geological Survey
PBW	pesticide-grade blank water, certified free of pesticide organic compounds by the NWQL

ABBREVIATIONS—*Continued*

PIC	particulate inorganic carbon
PFA	perfluoroalkoxy
POC	particulate organic carbon
PVC	polyvinyl chloride
QC	quality control
SS	source solution
TOC	total organic carbon
TPC	total particulate carbon
TPN	total particulate nitrogen
TWRI	Techniques of Water-Resources Investigations
URL	Uniform Resource Locator
USGS	U.S. Geological Survey
VOC	volatile organic compound
VPBW	volatile- and pesticide-grade blank water, purged with nitrogen gas and certified free of volatile and pesticide compounds by the NWQL. VPBW also can be used instead of inorganic blank water (IBW).
v/v	volume to volume

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TECHNICAL MEMORANDUMS OF THE U.S. GEOLOGICAL SURVEY, WATER DISCIPLINE

Office of Water Quality, National Water Quality Laboratory, and Water Resources Discipline numbered memorandums are available electronically on the Internet through the USGS Web site at <http://water.usgs.gov/admin/memo/> (accessed March 4, 2004).

Office of Water Quality

Memo No.	Title	Date
qw 92.01	Distilled/Deionized Water for District Operations	Dec. 20, 1991
qw 97.03	Protocols for Cleaning a Teflon Cone Splitter to Produce Contaminant-Free Subsamples for Subsequent Determinations of Trace Elements	Feb. 7, 1997

National Water Quality Laboratory (NWQL)

Memo No.	Title	Date
92.01	Technology Transfer—Availability of Equipment Blank Water for Inorganic and Organic Analysis	Mar. 25, 1992

Water Resources Discipline

Memo No.	Title	Date
wrđ 94.007	Safety--Storage, Transportation, Handling and Disposal of Methyl Alcohol	Dec. 3, 1993