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

Edited by
F.D. Wilde, D.B. Radtke, Jacob Gibs, and R.T. Iwatsubo

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Foreword

The mission of the Water Resources Division 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 and scientific agencies, and the general public. Reliable and objective 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 data collected are of the quality required to fulfill our mission.

Robert M. Hirsch
Chief Hydrologist

Techniques of Water-Resources Investigations

Book 9
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Chapters of Section A: National Field Manual for the Collection of Water-Quality Data

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[1]**Bold type indicates published chapters and chapter sections, and** shaded type **indicates chapters and chapter sections that are in preparation.**

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Chapter A3. CLEANING OF EQUIPMENT FOR WATER SAMPLING

***Edited by* Franceska D. Wilde, Dean B. Radtke, Jacob Gibs, and Rick T. Iwatsubo**

ABSTRACT

The *National Field Manual for the Collection of Water-Quality Data (National Field Manual)* provides protocols and 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 water samples and 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 announced on the USGS Home Page on the World Wide Web under "New Publications of the U.S. Geological Survey." The URL for this page is <http://water.usgs.gov/lookup/get?newpubs>.

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 data on 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.

The *National Field Manual* is Section A of Book 9 of the USGS publication series Techniques of Water-Resources Investigations (TWRI). Each chapter of this manual is published as a separate report. Chapter numbers are preceded by an “A” to indicate that the report is part of the *National Field Manual*. Other chapters and sections of other chapters of the *National Field Manual* are referred to in this report by the abbreviation "NFM" and the specific chapter and (or) section number. For example, general information on field measurements of ground water is covered in section 6.0.2 of Chapter A6, "Field Measurements," and would be cited as NFM 6.0.2.

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 documented and quality controlled.

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 a specific USGS Office of Water Quality (OWQ) 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 District[1] or other professional personnel, as appropriate. Technical memorandums or other unpublished documents that define the policy pertinent to such requirements are cited in this chapter. Personnel are instructed to use required equipment or procedures as described in this chapter. 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.

Recommended (recommend, recommended, or 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. 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.

[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

Chapters of the *National Field Manual* will be reviewed, revised, and reissued periodically to correct any errors, incorporate technical advances, and address additional topics. Please send comments or corrections to NFM-QW, USGS, 412 National Center, Reston, VA 20192 (or send electronic mail to nfm-owq@usgs.gov). Information regarding the status and any errata of this and other chapters can be found at the beginning of the electronic version of each chapter, located in the Publications Section of the following website: http://water.usgs.gov/lookup/get?owq.

Newly published and revised chapters will be announced on the USGS Home Page on the World Wide Web under “New Publications of the U.S. Geological Survey.” The URL for this page is <http://water.usgs.gov/lookup/get?newpubs>.

ACKNOWLEDGMENTS

The information in this chapter of the *National Field Manual* is based principally on the work of Sandstrom (1990), Horowitz and others (1994), Shelton (1994), and Koterba and others (1995).

The editors wish to thank and pay tribute to R.W. Lee and S.W. McKenzie, who were responsible for final technical review and who contributed to the accuracy, quality, and usability of this report. We would like to express appreciation to the following colleague reviewers for helping to improve this report: H.D. Ardourel, B.A. Bernard, K.K. Fitzgerald, D.S. Francy, S.R. Glodt, V.J. Kelly, S.L. Lane, S.K. Sando, C.A. Silcox, and W.R. White. The editors are indebted to I.M. Collies, C.M. Eberle, B.B. Palcsak, and Chester Zenone for their valuable editorial contributions, and to C.T. Mendelsohn, L.E. Menoyo, and A.M. Weaver, whose production assistance was instrumental in maintaining the quality of the report.

Special thanks go to T.L. Miller, whose encouragement and faith in this project has been instrumental to its achievement; and to D.A. Rickert and J.R. Ward, who provided the support needed to produce a national field manual for water-quality studies.

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 cleaning procedures be quality controlled (Sandstrom, 1990; Horowitz and others, 1994; Koterba and others, 1995). 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 or chemistry of target analytes in samples. Standard procedures are described in this chapter for when, where, and how to clean equipment constructed of various materials and to collect equipment blanks and field blanks for quality control. Space is commonly dedicated in an office 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 office space.

Equipment cleaning (decontamination): Applying cleaning solutions to the 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.

- 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.
  - Substances adhering to equipment from previous sampling must be removed.
- Prevent cross contamination between sampling sites by rinsing equipment with deionized water (DIW) while equipment is still wet, and then clean equipment as prescribed in this chapter before transporting it to the next site.
- Do not substitute field rinsing with sample water for the equipment-cleaning procedures described in this chapter.
- Collect equipment blanks and field blanks for quality control. A minimum of one equipment blank per year is required for each piece of equipment. The frequency of collecting blanks normally is based on study objectives and site conditions.

To help prevent sample and site contamination, be sure to use properly cleaned equipment.

SUPPLIES FOR EQUIPMENT CLEANING 3.1

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 such as 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 if methanol will be used. 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 appropriate safety gloves, glasses, and apron when working with corrosive and oxidizing solutions.
- When using chemicals, 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; µS/cm, microsiemens per centimeter at 25 degrees Celsius; PBW, pesticide-grade blank water; VBW, volatiles and pesticide-grade blank water; IBW, inorganic-grade blank water; L, liter; cm, centimeter; TOC, total organic carbon; DOC, dissolved organic carbon; SOC, suspended organic carbon; NFM, *National Field Manual*; PVC, polyvinyl chloride; IBW, inorganic-grade blank water]

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 Noncolored plastic sheeting	Sealable bags with uncolored closure strips, various sizes. Recyclable trash bags are recommended for large equipment storage. 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 µS/cm (usually District produced; Office of Water Quality Memorandum 92.01).
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 VBW.[2]
Detergent	Nonphosphate laboratory soap (for example, Liquinox™).
Gloves, disposable	Powderless, noncolored vinyl, latex, or nitrile (latex or nitrile for use with methanol), assorted sizes.
Inorganic-grade blank water (IBW)[2]	Blank water with certificate of analysis prepared and (or) quality assured by the analyzing laboratory. IBW is required for 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	ACS pesticide grade. Methanol is the organic solvent in common use for equipment cleaning, but study requirements might dictate use of a different ACS pesticide-grade solvent.
Neutralization materials	Marble landscape chips (1- to 2-cm chips recommended).[3]
Pesticide-grade blank water (PBW)[2]; volatile-grade blank water (VBW)[2]	Blank water prepared and (or) quality assured by the analyzing laboratory; required for collecting blank samples as follows: PBW for pesticide analysis; VBW for volatile compounds analysis and pesticide analysis; and either PBW or VBW for TOC, DOC, and SOC 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.)
Tapwater	If quality is questionable, substitute DIW. Tapwater is more effective for initial and rapid removal of detergent residue.
Tissues	Laboratory grade, lint free, various sizes (for example, Kimwipes™).
Washbasins	One washbasin for each cleaning solution; white or uncolored. Plastic, nonleaching. (Stainless steel is required for methanol.)
Wash bottles (dispenser or squeeze)	Labeled to indicate contents (for example, ACID, DIW, TAP). Fluorocarbon polymer needed for methanol, PBW, VBW, and IBW.

[1]Hydrochloric acid is required if analyzing for nitrogen species; otherwise, nitric acid is acceptable.
[2]PBW and VBW can be obtained from the USGS National Water Quality Laboratory (NWQL). IBW can be obtained from the USGS Quality of Water Service Unit.
[3]Agricultural limestone, soda ash, baking soda, and crushed shells are not recommended (Horowitz and others, 1994).

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.

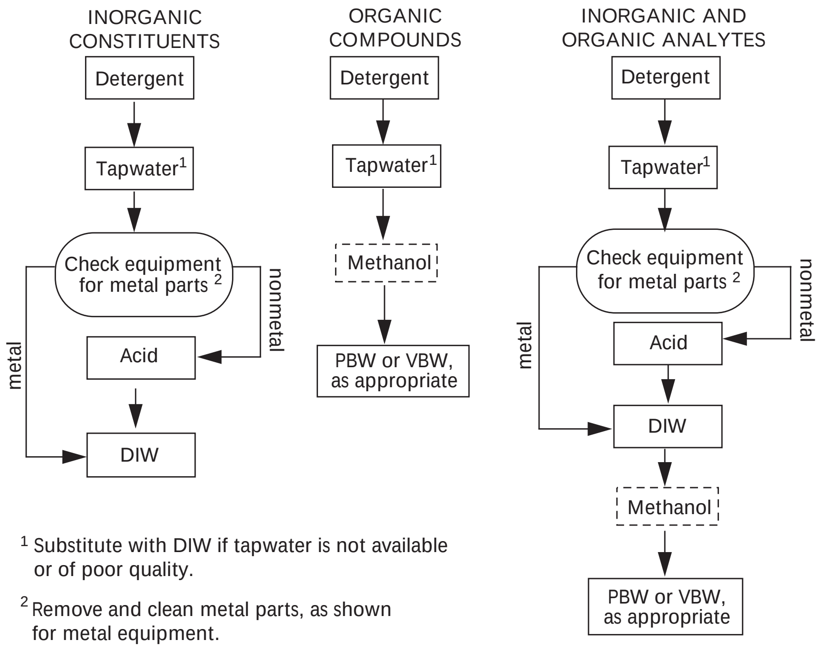
CLEANING PROCEDURES 3.2

***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 antibacksiphons 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.
  - – Recoat chipped surface-water samplers with epoxy paint or "plasti-coat." Such samplers must be recoated before use.



EXPLANATION

DIW	Distilled/deionized water
methanol (dashed box)	Current protocol includes methanol rinse for most types of equipment except that used with samples for organic-carbon analyses. However, this protocol is under review. SAFETY ALERT: Methanol is highly flammable; fumes can be hazardous to human health.
PBW	Pesticide-grade blank water
VBW	Volatiles- and pesticide-grade blank water

Figure 3-1. General sequence for cleaning equipment before sampling for inorganic and (or) organic analytes.

- ▶ Rinse equipment with DIW directly after use while equipment is still wet and before cleaning procedures are implemented.
- ▶ Place cleaned equipment in doubled storage bags.

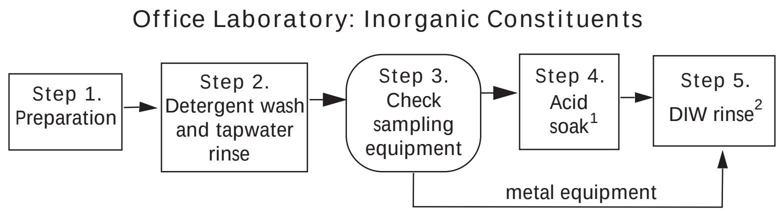
Do not allow collection and processing equipment to sit uncleaned in a field vehicle or elsewhere between field trips.

CLEANING OF EQUIPMENT USED TO SAMPLE FOR INORGANIC CONSTITUENTS 3.2.1

Cleaning of equipment used to collect and process water for analysis of inorganic constituents involves a five-step office-laboratory procedure or a five-step field-site procedure. 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). The cleaning procedures are summarized in figures 3-2 and 3-3. (These procedures do not apply to field-measurement instruments—see NFM 6.)

Equipment should be cleaned periodically in the office 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 office laboratory involve longer exposure of equipment to cleaning solutions, more frequent change of cleaning solution, and greater volumes of rinse water.

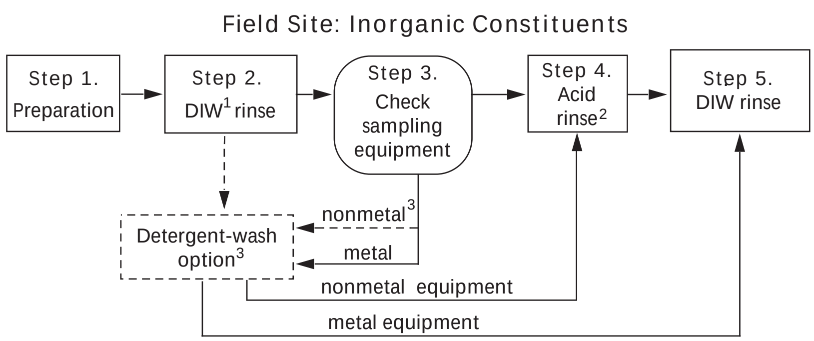
- ▶ To minimize field cleaning of equipment between sampling sites, preclean a separate set of equipment for each site.



[1] Acid soak used for nonmetal equipment.

[2] DIW = deionized water (table 3-1).

Figure 3-2. Office-laboratory cleaning procedures for equipment used to sample for inorganic constituents.



[1] DIW = deionized water (table 3-1).

[2] Acid rinse used for nonmetal equipment.

[3] Detergent wash is routine only for field cleaning of submersible pumps, metal equipment components, or excessively contaminated equipment.

Figure 3-3. Field-site cleaning procedures for equipment used to sample for inorganic constituents.

- 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 office laboratory for rigorous cleaning before reuse.
- After cleaning, document completion of and any modifications to the cleaning procedures.

Equipment-cleaning procedures for inorganic constituents

Standard procedures for office-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, the churn-splitter spigot, or other equipment constructed of stainless steel or other metallic material.
- 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 office-laboratory and field-site procedures.

Step 1. Preparation at the office 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 gloves[2], a laboratory coat or apron, and safety glasses.

   iv. Prepare the detergent solution, using a nonphosphate, laboratory-grade detergent.

   - Office 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.

   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 (HNO_3^-) 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. Use a black waterproof marker.

   vii. Unwrap the equipment to be cleaned and discard the storage bags. Change gloves.

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.

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[2]Refers to laboratory gloves that are nonpowdered on the inside 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, vinyl gloves are appropriate for most sampling activities but not when working with methanol or other organic solvents.

iii. Rinse all items thoroughly with tapwater to remove detergent residue. No detergent bubbles should appear when fresh tapwater is agitated in the basin, standpipe, or wash bottle.

iv. Rinse washbasins with DIW.

v. Pour 5-percent HCl (or 10 percent HNO_3^-) 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 office cleaning, but they are not usually disassembled for field cleaning.
- Processing and preservation chamber frames should be cleaned periodically using office-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 tapwater rinse—Office 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 tapwater 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 tapwater 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 3.3.9.

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 materials might be trapped. Change gloves.

c. Rinse equipment thoroughly with tapwater to remove detergent residue. Use DIW if tapwater 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 tapwater to rinse hard-to-reach places; pump tapwater 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—Office 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: go to Step 2, detergent-wash option at the field site and then to Step 5, DIW rinse.

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

For equipment constructed primarily of glass or fluorocarbon polymer or some other plastic, soak (office 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) HNO_3^- 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. Office 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 tapwater but retain any undissolved marble chips. Replenish chips to form a layer on the bottom of the neutralization container.

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

a. Place equipment into the cleaned washbasin labeled DIW. Change gloves.

b. Office 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

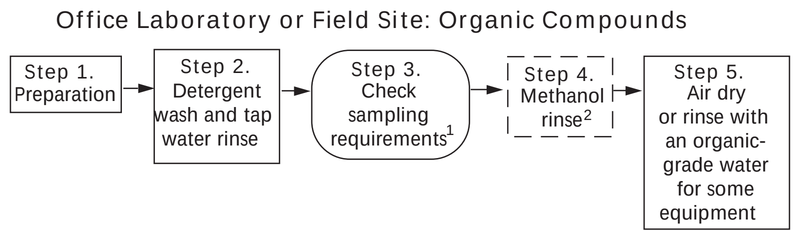
- ▶ Place dry, clean equipment inside doubled plastic bags. For small equipment, parts, and tubing, use sealable plastic bags.
- ▶ Place the churn splitter and funnel into doubled plastic bags and then place churn splitter inside of the churn carrier.

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

CLEANING OF EQUIPMENT USED TO SAMPLE FOR ORGANIC COMPOUNDS 3.2.2

Nearly identical procedures are used in the office laboratory and at the field site to clean equipment used to sample for organic compounds. The office 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.



[1]If sampling for inorganics also, acid soak/rinse nonmetal equipment before the methanol rinse.

[2] Omit when cleaning equipment used to sample for organic carbon analyses.

Figure 3-4. Cleaning procedures for equipment used to sample for organic compounds.

Equipment-cleaning procedure for organic compounds

Standard procedures for office-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. Bottles for organic analyses arrive from the laboratory capped and ready for use and should not be rinsed by field personnel. Discard bottles if received uncapped.
- ▶ Omit the methanol rinse when cleaning the equipment used to collect and process samples for total, dissolved, and suspended organic carbon (TOC, DOC, SOC). If equipment (such as a submersible pump) that has been in contact with methanol or other organic solvent must be used for TOC, DOC, or SOC sampling, flush the equipment with copious quantities of sample water before collecting the sample; collection of a blank sample for DOC quality control is recommended.

Be sure to check the specific procedures for selected equipment listed in section 3.3 before proceeding with the office-laboratory and field-site procedures.

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 or fluorocarbon polymer sheeting.

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 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 office cleaning but usually are not disassembled for field cleaning.

[3]Refers to laboratory gloves that are nonpowdered on the inside 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, vinyl gloves are appropriate for most sampling activities but not when working with methanol or other organic solvents. Use solvent-resistant gloves when cleaning with organic solvents. Latex or nitrile disposable, powderless gloves are appropriate when using methanol.

Step 2. Detergent wash and tapwater 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 tapwater.) Change gloves.

- Office 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 inorganic or organic materials might be trapped, such as grooves and crevices, O-rings, and nozzles.

c. Place equipment into tapwater washbasin.

d. Rinse equipment thoroughly with tapwater to remove detergent residue. Use an organic-grade water (PBW, VBW, or office-produced) if tapwater 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 tapwater 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 rinse[4] (fig. 3-4).

a. Change to 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.

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.

b. Use pesticide-grade methanol (or appropriate organic solvent) dispensed from a methanol fluorocarbon-polymer wash bottle (office 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 interior of pump tubing with methanol.

   - Do not rinse exterior of pump tubing with methanol.
   - Do not rinse pump tubing with methanol or any organic solvent if TOC, DOC, or SOC samples will be withdrawn through that tubing.

[4]Current (1998) cleaning protocol dictates the use of methanol to remove contaminants from equipment to be used to collect samples for analysis of organic compounds.

iii. Place equipment components and tubing on a clean aluminum foil surface.

iv. Pour or discharge used methanol (or other organic solvent) into an appropriate waste container for flammable liquids (Water Resources Division Memorandum 94.007). Change gloves. Dispose of gloves used for methanol rinse appropriately.

TECHNICAL NOTE: Rinse 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 TOC, DOC, or SOC 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).

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.

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 FOR CLEANING SELECTED TYPES OF EQUIPMENT 3.3

***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. Wear appropriate disposable, powderless gloves throughout each cleaning procedure, changing gloves with each change in cleaning solution and as described in section 3.2.

INORGANIC-SAMPLE BOTTLE CLEANING PROCEDURES 3.3.1

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.
- A cleaning procedure is required for bottles that will contain samples to be analyzed for trace elements and 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, vinyl 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.

3.3.2 CHURN SPLITTER CLEANING PROCEDURES

Plastic churn splitters are used primarily for samples to be analyzed for inorganic constituents (NFM 2). Avoid the need to field-clean the churn splitter by using a separate, precleaned churn splitter at each field site to be sampled, if possible.

When using the detergent wash/tapwater rinse for the churn splitter—Office-laboratory procedure (fig. 3-2, Step 2):

1. Fill churn splitter through the funnel 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 spigot 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 through the funnel splitter about one-third full with tapwater; swirl and shake churn vigorously to remove detergent residues. Allow tapwater 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—Office-laboratory or field-site procedures (figs. 3-2 and 3-3, Step 4):

1. Do not allow acid solution to contact the outside of churn splitter, or the churn spigot.

2. Do not pass acid solution through the spigot.

3. Decant acid solution by pouring out of the top of the churn into the neutralization container.

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

1. Fill the churn splitter through the funnel 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.

For storage of a cleaned churn splitter—Office-laboratory or field-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.

3.3.3 CONE SPLITTER CLEANING PROCEDURES

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:

Office 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 deionized water.
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 deionized water. 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):

Office 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 inorganics 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).
    - Do not use any organic solvent if the cone splitter will contact samples for analysis of TOC, DOC, or SOC.
    - If samples processed through a splitter will be analyzed for TOC, DOC, or SOC, rerinse the splitter thoroughly to completely remove residues from the detergent wash. Use PBW, VBW, or other organic-grade water for the final rinse if complete methanol evaporation is impractical. If the cone splitter will not be used to process samples for inorganic constituents at the next site, wrap nozzle and other orifices in aluminum foil.

For storage of a cleaned cone splitter:

1. Allow the cone splitter to air dry.
2. Place the cone splitter into a clean plastic bag and seal.
3. 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.

3.3.4 FILTRATION EQUIPMENT CLEANING PROCEDURES

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

3.3.4.A Disposable Capsule Filter Cleaning Procedure

The disposable capsule filter has a one-time use for processing samples to be analyzed for inorganic constituents but must be cleaned before use. The filter can be prerinsed in the office laboratory instead of at the field site as long as it is kept chilled and used in less than 1 day. After filtering the sample, clean or replace the sample-delivery tubing and discard the capsule filter. The cleaning procedure described below comprises sufficient cleaning of the filter for analysis of inorganic constituents at the parts-per-billion (ppb) concentration level (Horowitz and others, 1994).

To clean the disposable capsule filter, pump 1 L of DIW to the filter through precleaned tubing (section 3.3.5) as follows (refer to NFM 5.2.1.A for additional instructions):

1. 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. Put on disposable, powderless gloves.

2. In a processing chamber, remove the capsule filter from the protective bags. Attach pump tubing to the inlet connector of the capsule filter, keeping the tubing as short as possible. Make sure the direction of flow through the capsule filter matches the direction-of-flow arrow on the side of the filter.

3. Pump 1 L of DIW through the capsule filter; discharge waste rinse water through a sink funnel or to a toss bottle.
   - Operate the pump at a low speed.
   - Hold the capsule filter so the arrow is pointing up at an acute angle from the horizontal plane. (This expels trapped air from the capsule; do not allow water to spray onto chamber walls.)
4. Remove tubing from the DIW reservoir and continue to operate the pump in the forward, mid-range speed position to drain as much of the DIW that remains in the capsule filter as possible. While the pump is operating, shake the capsule filter to help remove any entrained DIW.
5. Detach the capsule filter from the peristaltic pump tubing, put into a clean, sealable plastic bag, and store chilled until ready for use at the next site.

Plate-Filter Assembly Cleaning Procedure 3.3.4.B

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).

- ▶ Preclean in the office laboratory one plate-filter assembly 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 filter, where sediment or organic matter might be lodged. Detergent wash and DIW rinse the pressure valve.
- ▶ Remove and discard the used filter at the field site; rinse the filter assembly immediately with DIW while still wet from filtering the sample, even if a clean filter assembly is available for the next site.

When field cleaning the plastic plate-filter assembly:

1. Disassemble the plate-filter assembly inside the processing chamber while it is still wet from the sample water and while wearing disposable, powderless gloves.

   a. Remove the used filter media carefully to avoid spilling any of the filter cake.

   b. Place the filter media into a sealable plastic bag. Seal and pass the bag out of the chamber. Change gloves.

2. DIW rinse all components of the plate-filter assembly, including the exterior and interior of the tubing and the pressure valve, dispensing the DIW from a wash bottle. Pay particular attention to grooves and crevices, O-rings, and support structures for the membrane filter, where inorganic or organic materials might be lodged. Change gloves.

3. Inspect the plastic plate-filter assembly. Use the detergent-wash option described in figure 3-3 (Step 2) if the filter assembly looks dirty, is suspected of being contaminated, or was allowed to dry after use without first rinsing thoroughly with deionized water.

4. Reassemble the plate-filter assembly, reattaching the piece of tubing to the outlet of the filter assembly and placing the discharge end of the tube through the drain or disposal funnel in the bottom of the processing chamber to the acid-neutralization container. Reconnect the filter assembly to the peristaltic pump with the sample tubing. Change gloves.

5. To acid rinse the plate-filter assembly, pump 1 L of 5-percent HCl solution (or 10-percent HNO_3^- solution) through the plate-filter assembly. Check that the acid solution is being discharged into the acid-neutralization container. Alternately squeeze and release the tubing at the outlet to force the acid solution to cover and rinse all interior surfaces of the filtration assembly. (Be careful not to force tubing from the outlet by squeezing tubing for too long.)

6. To DIW rinse the plate-filter assembly, pump 2 L of deionized water through the assembly, using the same squeeze-and-release method described above in 5 for the acid rinse. Ensure that all the rinse water is being discharged to the acid-neutralization container. After confirming that the pH of the acid rinse solution is greater than 6.0 or the original pH of the DIW, appropriately discard solutions from the neutralization container.

7. For storage, place the cleaned plate-filter assembly and tubing into clean double bags for temporary storage until use at the next site. If wet when bagged, store for no longer than 24 hours and (or) chill to prevent bacterial growth. The filter assembly must be dry if stored for more than 24 hours.

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

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

1. Inspect the aluminum (or stainless steel) plate-filter 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 remove the used glass-fiber filter media carefully to avoid spilling any of the filter cake. Place used filter media into a sealable plastic bag, seal the bag, and put aside for disposal. Place components of the plate-filter 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 filter 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 glass-fiber filter, where inorganic or organic materials might be lodged. Pump detergent solution through tubing. Place components of the plate-filter assembly onto a clean, aluminum-foil-covered surface.
5. Discard detergent solution from basin, rinse basin with tapwater, and place components of the plate-filter 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 tapwater to rinse hard-to-reach places. Place rinsed components onto a dry section of clean aluminum foil or basin. Change gloves. If the assembly will be rinsed with

methanol or other organic solvent, change to disposable, solvent-resistant gloves, and place components of the filter assembly into a clean, solvent-resistant washbasin.

7. Rinse plate-filter assembly components with pesticide-grade methanol or an equivalent grade for other organic solvents. Do not methanol rinse any tubing or filtration assembly to be used for collecting or processing samples for TOC, DOC, or SOC analysis. The instructions for the methanol rinse apply also for use of any other organic solvent. Rinse the equipment with methanol while outside of the field vehicle and downwind of sampling activity.

    a. Dispense methanol from a fluorocarbon-polymer wash bottle. Rinse all sample-contacting surfaces of filter-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, if concerned about airborne contaminants.)

8. Reassemble the plate-filter assembly. Wrap nozzles with aluminum foil and seal filter assembly in plastic bags. Double bag for transport or for long-term storage.

3.3.4.C Pressure-Filter Assembly Cleaning Procedure

The cleaning procedures described in section 3.2.2 for figure 3-4 do not apply to the filtration assembly used for samples to be analyzed for DOC and SOC. The filtration assembly for processing organic-carbon samples is a gas-pressurized apparatus constructed of either stainless steel or fluorocarbon-polymer material.

- ▶ Do not bring the pressure-filter assembly in contact with methanol or other organic solvent or organic-solvent vapors.
- ▶ In general, office-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 VBW also can be used. Office-produced organic-grade water, however, must not be substituted for blank samples.

▶ Do not clean the pressure-filter assembly with detergent. Exception: see Step 3 below.

When using office-laboratory or field-site cleaning procedures for cleaning the pressure-filter assembly:

1. Wearing disposable, powderless gloves, disassemble the pressure-filter assembly before it dries and place components into a clean washbasin. Change gloves.

2. Using office-produced organic-grade water, thoroughly rinse the pressure-filter assembly and place it into a washbasin or onto a clean surface. Generally, these steps are 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 when scrubbing.

   - Rinse the pressure-filter assembly thoroughly with office-produced organic-grade water or PBW or VBW.

3. If the pressure-filter assembly is very dirty or contaminated, clean as follows:

   a. Disassemble and soak assembly for at least 1 hour in a 0.1-percent solution of nonphosphate laboratory-grade detergent.

   b. Scrub with a soft-bristled toothbrush, as described above in 2.

   c. Rinse repeatedly with office-produced organic-grade water, being sure to remove all traces of detergent.

4. Place all components of the pressure-filter assembly onto aluminum foil and allow to air dry thoroughly under a protective aluminum foil tent.

5. Reassemble the pressure-filter assembly, wrap nozzles in aluminum foil, and seal in a storage bag.

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

3.3.5 SAMPLE TUBING CLEANING PROCEDURES

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 3.3.9.B. Wear appropriate, disposable, powderless gloves throughout the cleaning process, changing gloves with each change in cleaning solution as indicated throughout section 3.2.

- ▶ Preclean the number of tubing sections needed at each site in the office 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 office-laboratory or field-site procedures for cleaning plastic (including fluorocarbon-polymer) sample tubing used for samples to be analyzed for inorganic constituents, follow the general sequence of procedures described for figures 3-2 or 3-3, and those described for filtration assemblies (section 3.3.4).

To summarize the key steps for 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 same 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 tapwater/DIW rinse procedures.

When using office-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 tapwater rinse. If samples also will be collected for inorganic-constituent analysis, however, acid rinse nonmetallic tubing and components after the detergent wash/tapwater rinse and before continuing to the methanol rinse. When cleaning sample tubing:

1. Pump 1 L of nonphosphate, laboratory-grade detergent solution through tubing, followed by sufficient tapwater 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 TOC, DOC, or SOC analysis.
3. Store tubing in doubled plastic bags.

> CAUTION: Do not use methanol around equipment that can create electrical sparks (see section 3.3.9.B).

3.3.6 PROCESSING AND PRESERVATION CHAMBERS AND FLOWTHROUGH CHAMBER CLEANING PROCEDURES

Processing and preservation chambers 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. When the bag is clipped to the inside, it is not necessary to field clean the chamber frame.

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:

Office laboratory. Clean the frame of portable chambers in the office with detergent solution, then rinse thoroughly with tapwater 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 clipped to the frame.

1. Discard the used bag.
2. Wipe the chamber frame with DIW.
3. Replace chamber cover only when the next samples are ready to be processed.
4. If the processing chamber is a fixed installation, clean out any spilled sample water, solid materials, or wash solutions, and swab down the inside using deionized water and lint-free laboratory tissue.
5. Use detergent solution followed by a thorough tapwater 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 office laboratory with detergent solution and rinse thoroughly with tapwater, 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 tapwater followed by DIW.

RADON SAMPLER CLEANING PROCEDURE 3.3.7

Soak radon samplers in a detergent solution for 10 minutes and rinse thoroughly with tapwater to remove detergent residue; follow with three to five rinses with DIW. Do not use methanol. Air dry the radon sampler and store in doubled plastic bags.

3.3.8 SURFACE-WATER SAMPLER CLEANING PROCEDURES

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 office-laboratory procedures for cleaning surface-water samplers:

1. Periodically disassemble samplers for office-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 shown in figure 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 TOC, DOC, or SOC 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 it 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 tapwater 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 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 tapwater 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.

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 tapwater 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, if needed, as described above.

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 SAMPLER CLEANING PROCEDURES 3.3.9

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). Office-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 office-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 under review and appropriate only for samplers being used to collect samples for organic-compound analysis. Solvents are never used to clean equipment when sampling for TOC, DOC, or SOC. 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.

3.3.9.A Cleaning of Bailers and Other Nonpumping Samplers

Office-laboratory procedure. Clean nonpumping samplers in a designated area of the office 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 office-laboratory procedure.

3.3.9.B Cleaning of Submersible Pumps 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 office-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 last sample collected through the tubing were greater than 500 µg/L.

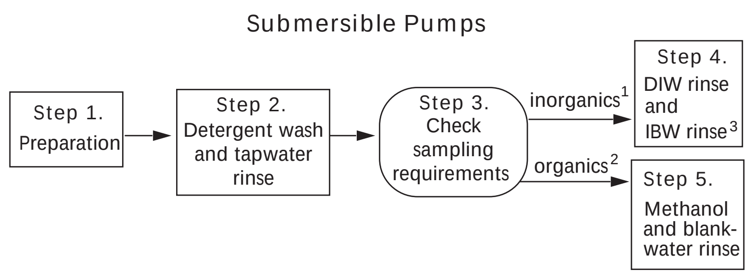
– Pump tubing should be replaced rather than cleaned if VOC concentrations in last sample exceeded about 700 µg/L.

▶ 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, the pump must not be used for collecting trace-metal samples.

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



[1] Samples collected for inorganic-constituent analysis. If samples are also to be collected for organic-compound analysis, rinse with deionized water (DIW) (Step 4), then methanol (Step 5).

[2] Samples collected for organic-compound analysis.
When sampling for total, dissolved, or suspended organic carbon, do not methanol rinse; complete cleaning by rinsing with organic-grade water.

[3] A rinse with inorganic blank water (IBW) is needed only if collecting a blank sample for inorganics analysis directly after cleaning the pump.

Figure 3-5. Cleaning procedures for submersible pumps.

Office-laboratory pump-cleaning procedure:

Use office-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 tapwater.

d. Disassemble the pump and place components into a detergent-solution washbasin.

Step 2. Detergent wash and tapwater rinse pump components and tubing.

a. Soak pump components in the detergent solution for 30 minutes.

b. Scrub pump components with soft sponge or brush.

c. Rinse thoroughly with tapwater.

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 tapwater through the tubing. Change gloves.

Step 3. Check sampling requirements.

— If pump will be used for collecting samples for inorganic-constituent analysis, 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, 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 blank water followed by a methanol rinse.

a Change to 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 office laboratory, as methanol fumes can contaminate other 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).

d. Using a valveless fluid metering pump and fluorocarbon-polymer tubing, pump about 2 L of methanol through sample tubing and 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 an organic-grade water (PBW or VBW) into a clean PBW/VBW standpipe. Insert pump and pass about two tubing volumes of organic-grade blank water (PBW or VBW) 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 tapwater rinse, DIW rinse, methanol rinse, and blank water (IBW/PBW/VBW). 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.

The volume of storage in tubing, V_s, of a set of pump-reel and extension tubing can be estimated[1,2] as follows:

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

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 liters[1]
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 volume[1] 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 office-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].

---

[1]Estimate 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.

[2]Estimate 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.

[3]The 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 tapwater rinse.

a. Put on disposable, powderless gloves (usually vinyl). 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 tapwater. (DIW can be substituted for tapwater, but is less efficient in detergent removal and requires a greater volume of water than tapwater.)

b. Place pump into standpipe, add detergent solution to level above pump intake, and route intake and discharge end 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 tapwater until sudsing stops.

g. Rinse pump exterior with tapwater. Place rinsed pump into standpipe; add tapwater/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 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 tapwater.

a. Use a clean DIW-dedicated standpipe, not the tapwater standpipe, and rinse with DIW. Rinse pump exterior with DIW to remove any detergent residue. 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 3 tubing volumes of DIW. Keep the DIW level above pump intake.

c. Collect DIW rinse water in a clean bottle, shake, and check for suds. Continue to DIW rinse until rinse water is free of suds.

d. 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. Rinse pump exterior with blank water.

   ii. Place pump into the standpipe and add IBW to cover the pump intake.

   iii. Turn on pump and displace any water residing in the pump and tubing. Continue pumping IBW for one tubing volume before collecting the blank sample.

Step 5. Methanol rinse.

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.[5])

a. Change to latex or nitrile gloves. Wear 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 sample tubing into methanol waste container, keeping the level of solution above 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 put any unused methanol from bottom of standpipe into methanol waste container. Let methanol in the standpipe evaporate to dryness. Change gloves.

[5]NEC 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 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 to the methanol waste container after used methanol is collected.

TECHNICAL NOTE: The recommended organic-grade water is PBW or VBW (supplied by NWQL for blank samples). Office-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 VBW) pumped from a blank-water standpipe if blank samples will be collected for analysis of organic compounds.

Use of methanol is not recommended as a routine procedure for field cleaning of the pump. A methanol rinse is most safely accomplished as an office-laboratory procedure.

Storage of the cleaned submersible pump and tubing:

1. Place pump into two clean, noncontaminating storage bags and close bags.
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.

▶ 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 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 VBW to process blank samples for analysis of VOCs. VBW is also suitable as a blank sample for pesticide analysis.
- Use PBW or VBW as the quality-control sample for total and dissolved organic-carbon analysis (TOC and DOC). This cannot be documented as a blank sample because neither PBW nor VBW is certified to be free of organic carbon.

- **Equipment blank.** An equipment blank is blank water that is processed under controlled conditions in the office 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 office laboratory, after the equipment has been cleaned using office-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, sampling can proceed.
   - If the equipment-blank data indicate unacceptable concentrations of analytes of interest, 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 office-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) VBW 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 office-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 office 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 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. SS blank	Source solution (SS) Put on disposable gloves. Pour the IBW, PBW, or VBW directly into appropriate SS blank-sample bottle.[1] Add chemical treatment and (or) chill, as required for the analytes of interest.
Sample 2. Sampler blank	SS + Sampler 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. Splitter blank	SS + Sampler + Splitter[2] 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. Pump blank	SS + Sampler + Splitter + Pump 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. Filter or equipment blank	SS + Sampler + Splitter + Pump + Filter Pump SS through a prerinsed 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.

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

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

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

CONVERSION FACTORS AND ABBREVIATIONS

CONVERSION FACTORS

Multiply	By	To obtain
centimeter (cm)	0.3937	inch
meter	3.281	foot
milliliter (mL)	0.06102	$inch^3$ or cubic inch
liter (L)	0.2642	gallon
microgram (µg)	3.53×10^{-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:

$$°F = 1.8(°C) + 32$$

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	*National Field Manual for the Collection of Water-Quality Data*
NWQL	National Water Quality Laboratory of the U.S. Geological Survey
OWQ	Office of Water Quality of the U.S. Geological Survey
PBW	pesticide-grade blank water, certified free of pesticide organic compounds by the NWQL
PVC	polyvinyl chloride
QC	quality control
QWSU	Quality of Water Service Unit
SOC	suspended organic carbon
SS	source solution
TOC	total organic carbon
TWRI	Techniques of Water-Resources Investigations
URL	Uniform Resource Locator
USGS	U.S. Geological Survey
VBW	volatiles-grade blank water, certified free of volatile compounds by the NWQL
v/v	volume to volume

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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 Division

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

PUBLICATIONS ON TECHNIQUES OF WATER-RESOURCES INVESTIGATIONS

The U.S. Geological Survey publishes a series of manuals describing procedures for planning and conducting specialized work in water-resources investigations. The material is grouped under major subject headings called books and is further divided into sections and chapters. For example, Section A of Book 9 (Handbooks for Water-Resources Investigations) pertains to collection of water-quality data. The chapter, which is the unit of publication, is limited to a narrow field of subject matter. This format permits flexibility in revision and publication as the need arises.

The Techniques of Water-Resources Investigations (TWRI) reports listed below are for sale by the U.S. Geological Survey, Branch of Information Services, Box 25286, Federal Center, Denver, CO 80225 (authorized agent of the Superintendent of Documents, Government Printing Office). Prepayment is required. Remittance should be sent by check or money order payable to the U.S. Geological Survey. Prices are not included because they are subject to change. Current prices can be obtained by writing to the above address. When ordering or inquiring about prices for any of these publications, please give the title, book number, chapter number, and "U.S. Geological Survey Techniques of Water-Resources Investigations." An updated list of TWRI reports can be found by accessing the World Wide Web url: http://water.usgs.gov/lookup/get?TWRI.

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