
SAMPLE PROCESSING 2.2

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Water samples must be processed as quickly as possible after collection. The equipment most commonly used for sample processing includes sample splitters, filtration units or assemblies, solid-phase extraction systems, and chambers in which samples are processed and treated with chemical preservatives. Having several available sets of cleaned processing equipment is recommended. The equipment and supplies used to process surface-water and ground-water samples are listed in section 2.4.

SAMPLE SPLITTERS 2.2.1

The collection of surface water generally results in a single composite sample. A ground-water sample generally is not composited; instead the sample is pumped directly into separate bottles for designated analyses. There are exceptions. For example, a ground-water sample can be composited when the sample is collected using a nonpumping method (bailer or thief sampler).

Once a sample has been composited, the sample is often subdivided (split) into subsamples for analysis. Each whole-water subsample should contain suspended and dissolved concentrations of target analytes that are virtually equal to those in every other subsample. Uses of the churn splitter (fig. 2-9) and the cone splitter (fig. 2-10) are discussed in sections 2.2.1.A and 2.2.1.B, respectively, and testing and comparative data between the splitters are described in Capel and others (1995, 1996) and in Office of Water Quality Technical Memorandum 97.06.

2.2.1.A Churn Splitter

The 8 or 14-L plastic churn splitter is recommended to composite and split surface-water samples for trace-element analysis (fig. 2-9). Stainless steel and glass containers are used to composite samples for organic analysis. To avoid sample contamination, do not collect or extract samples for trace-element analyses from a metal container, or samples for organic-compound analysis from a nonfluorocarbon-polymer plastic container.

The following modifications to the churn splitter and its deployment are required:

- ▶ **Modified churn spigot:**
This spigot is described in Horowitz and others (1994) and is available from the USGS Quality of Water Service Unit (QWSU) in Ocala, Fla.
 - The spigot contains a metal spring to keep the spigot valve closed when not in use. If the spigot leaks, sample can contact the spring. The spring represents a potential source of metal contamination.
 - To prevent leakage of the spigot, silicone sealant is injected inside the push-button mechanism.



Figure 2-9. Churn splitter (from Capel and Larson, 1996).

- + ▶ **Funnel assembly:** To meet requirements for trace-element sampling, a funnel assembly is inserted into a 1-in. hole drilled through the lid of the churn splitter (fig. 2-9).
 - The funnel is used when pouring whole-water samples into the churn splitter so that the churn lid can be left on, thus minimizing exposure of the composite sample to atmospheric contamination.
 - To make the funnel assembly, cut the top section (at the shoulder line) from a 1-L polypropylene sample bottle and insert into the hole drilled in the churn lid. Cut the bottom two-thirds from of a 1-L NALGENE™ or other larger diameter sampler bottle and use as a funnel cap.

- + ▶ **Churn covering:** To keep the entire churn-splitter assembly clean during sampling and prevent potential contamination, the churn splitter is placed inside two pliable, clear plastic bags (double bagged). These bags should be large enough to completely enclose the churn splitter, including funnel and churn handle, with enough excess material so that the bag openings can be gathered, folded over, and kept closed.

- + ▶ **Churn carrier:** The carrier is a white plastic container, with lid, large enough to hold the double-bagged churn splitter assembly.
 - The lid of the carrier serves both as a seal for the carrier and as a windbreak when the collected subsample is poured into the churn funnel.
 - The purpose of the carrier is to minimize contamination during transport. In exposed areas, such as bridges and roadways, the carrier can protect against atmospheric sources of contamination, particularly material from motor vehicles.

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Volume of sample needed:

- ▶ Subsamples totaling 10 L can be withdrawn from the 14-L churn for whole-water analysis, whereas subsamples totaling 5 L can be withdrawn from the 8-L churn for whole-water analysis.
- ▶ The 4 L remaining in the 14-L churn and the 3 L remaining in the 8-L churn should not be used for total, total recoverable, or suspended material subsamples because they will not be representative. However, the sample mixture remaining in either churn can be used for filtered subsamples for the determination of dissolved constituents.

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Advantages of the churn splitter:

- ▶ Simple to operate.
- ▶ Easy to clean.

Limitations of the churn splitter (see also Ward and Harr (1990), Horowitz and others (1994), and Capel and Larson (1996), and Office of Water Quality Technical Memorandum 97.06):

- ▶ Although it can be used to split samples with particle sizes $\leq 250 \mu\text{m}$ and suspended-sediment concentrations $\leq 1,000 \text{ mg/L}$, splitting accuracy becomes unacceptable for particle sizes $>250 \mu\text{m}$ and suspended-sediment concentrations $>1,000 \text{ mg/L}$.
- ▶ Sample volumes less than 3 L or greater than 13 L cannot be split for whole-water subsamples.
- ▶ Plastic (nonfluorocarbon polymer) churn splitters should not be used to composite samples for determination of organic compounds.
- ▶ Samples for bacteria determinations are not to be taken from a churn splitter because the splitter cannot be autoclaved.

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Cone Splitter 2.2.1.B

The cone splitter is a pour-through device constructed entirely of fluorocarbon polymers (fig. 2-10). The cone splitter may be used to process samples with particle sizes $\leq 250 \mu\text{m}$ and suspended-sediment concentrations $\leq 10,000 \text{ mg/L}$. Its primary function is to split the sample simultaneously into as many as 10 equal-volume samples. Some cone splitters have a 2-mm mesh screen in the reservoir funnel to retain large debris, such as leaves and twigs, that could clog or interfere with the splitting process. Below the funnel is a short standpipe that directs sample water in a steady stream, into a splitting chamber that contains a notched, cone-shaped splitting head with 10 equally spaced exit ports around its base. There should be no ridges, benches, or surfaces inside the splitting chamber that could retain material or interfere with the splitting process. The cone splitter is supported either by tripod legs or with an adjustable clamp and stand.

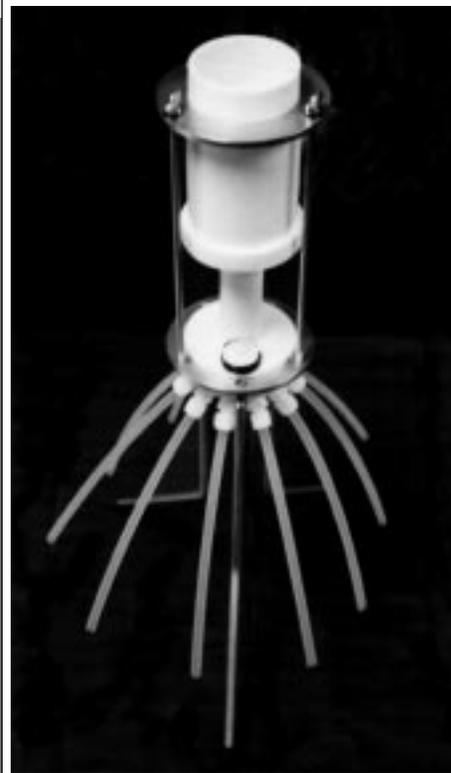


Figure 2-10. Cone splitter (from Capel and Larson, 1996).

The cone splitter is built to very close tolerances in order to achieve accurate and reliable operation. Bias to data can result from splitter imperfections or improper operation; therefore, calibration and proper use is necessary when processing samples. +

Advantages of the cone splitter

- ▶ Used to process samples with suspended-sediment concentrations from 1,000 to 10,000 mg/L.
- ▶ Samples as small as 250 mL can be split into 10 equal sub-samples.
- ▶ Samples greater than 13 L can be processed.
- ▶ Samples to be analyzed for organic compounds (except for volatile organic compounds) can be processed through the fluorocarbon polymer cone splitter.
- ▶ Samples containing sediment particles ranging in size from very fine clay and silt (1 to 10 μm) to sand-size particles (250 μm) can be split. +

Limitations of the cone splitter

- ▶ Accuracy of the volume equivalents must be verified before using a new or modified cone splitter (see instructions below).
- ▶ Splitter is awkward to operate and clean in the field.
- ▶ Sample is vulnerable to contamination from atmospheric sources or from improper operation.
- ▶ Splitting capability for sediment particles $>250 \mu\text{m}$ must be quantified.
- ▶ Samples for bacteria determinations are not to be collected with the cone splitter because the splitter cannot be adequately sterilized.
- ▶ The cone splitter must be level for proper operation. +

Before using a new or modified cone splitter, test the splitter to be used as follows (Office of Water Quality Technical Memorandum 80.17):

1. Inspect the cone splitter housing and outlet ports. They should be smooth and symmetrical without any visible burrs or chips. The cone splitter must be clean.
2. Place the cone splitter on a stable platform or bench. The splitter must be level for proper operation. Use a bull's-eye level to check leveling and recheck during use (leveling changes with movement in the field vehicle).
3. Connect 10 discharge tubes to the outlet ports. The tubes must be the same length, and as short as possible. Label the outlets from 1 to 10.
 - All tubes must be pushed in as far as possible to the machined surface in the Swagelok™ fitting in the splitter ports. The ends should be flush with the bottom of the inside of the port.
 - Tubes need only extend into the receiving containers sufficiently to prevent spillage.
 - Tubes must not extend in so far that the ends become submerged.
4. Wet the cone splitter by pouring several liters of deionized water through it.
 - Lightly tap the system to dislodge adhering water drops, then discard the water.
 - Place empty sample bottle under each outlet tube.
5. Accurately measure 3 L of deionized water into a 1-gal narrow-mouth plastic bottle.
6. Rapidly invert the 1-gal bottle over the reservoir, letting deionized water flow out as fast as possible. For proper operation, the standpipe must be discharging at its full flowing capacity.
7. After all deionized water has passed through the splitter, tap the assembly several times to dislodge adhering water drops. Check for spills and leaks. If any are observed, stop the test, correct the problem, and repeat steps 1-7.
8. Measure the volumes of the 10 subsamples carefully, within an accuracy of ± 1.0 mL. Record the volumes for each outlet on a form similar to table 2-6.

9. Repeat the test a minimum of three times. Use the same initial volume for each test.

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Calculate and document the results of the cone-splitter accuracy test as follows:

1. Referring to the example in table 2-6, calculate the mean volume of each subsample (\bar{x}) and standard deviation (S_x) for each test:

$$\bar{x} = \frac{\sum x_i}{n} \text{ and}$$

$$S_x = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}},$$

where x_i is the measured volume for each subsample and n is the number of subsamples (outlet ports).

2. Calculate the standard deviation in percent (E_x):

$$E_x = \frac{S_x}{\bar{x}} \times 100.$$

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3. Calculate the error for each subsample (E_i):

$$E_i = \frac{x_i - \bar{x}}{\bar{x}} \times 100.$$

4. Compute the mean standard error (\bar{E}_x) for the three tests and document the maximum and minimum errors (E_i) for all tests on field forms.
5. If data-quality requirements warrant, note the error patterns for individual outlets to determine which outlets produce consistent bias and label them with their mean percent bias error. Depending on the objectives of the study and how the subsamples will be used, this pattern of error may not be of concern.

A cone splitter is considered acceptable for sample processing if the mean standard error (\bar{E}_x) for the three tests is 3 percent or less, and no individual error (E_i) exceeds ± 5 percent.

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Table 2-6. Example of six cone-splitter accuracy tests using deionized water

[Modified from Office of Water Quality Technical Memorandum 80.17]

Test number	1	2	3	4	5	6	Averages							
Initial sample weight (grams)	2,499.4	2,499.5	2,499.5	2,499.5	2,499.5	2,499.4								
Outlet number	Outlet volume (x_i)	Per-cent (E_i)	Outlet volume (x_i)	Per-cent (E_i)	Outlet volume (x_i)	Per-cent (E_i)	Mean volume (\bar{x}_i)	Per-cent (E_i)						
1	248.4	-0.5	247.4	-0.9	247.8	-0.8	248.1	-0.7	247.8	-0.8	249.2	-0.2	248.4	-0.6
2	246.8	-1.2	245.6	-1.6	246.3	-1.4	248.4	-6	246.3	-1.4	246.7	-1.2	246.8	-1.2
3	249.4	-1	250.6	.4	249.8	0	251.1	.5	249.8	0	248.7	-.4	250.1	.1
4	250.7	.4	252.5	1.1	251.8	.8	251.3	.6	251.8	.8	250.5	.3	251.6	.7
5	248.1	-.6	249.8	0	249.3	-.2	249.3	-.2	250.2	.2	248.1	-.6	249.0	-.3
6	252.2	1.0	250.3	-.2	252.7	1.2	252.0	.9	252.7	1.2	250.6	-.4	251.8	.8
7	245.7	-1.6	246.2	-1.4	246.0	-1.5	246.3	-1.4	246.6	-1.3	245.9	-1.5	246.1	-1.5
8	252.7	1.2	254.2	1.8	252.9	1.3	253.3	1.4	253.1	1.3	254.6	2.0	253.5	1.5
9	248.7	-.4	247.3	-1.0	247.5	-.9	247.1	-1.1	248.2	-.6	249.5	-.1	248.0	-.7
10	253.9	1.7	252.1	.9	251.8	.8	250.6	.3	251.7	.8	253.0	1.3	252.0	1.0
Final sample weight	2,496.6	2,498.3	2,496.8	2,497.5	2,498.2	2,496.8	2,497.5	2,497.5	2,498.2	2,496.8	2,497.4	2,497.4	2,497.4	2,497.4
Sample loss	2.8	1.2	2.7	2.0	1.3	2.6	2.0	2.0	1.3	2.6	2.6	2.6	2.6	2.6
Mean weight (\bar{x})	249.7	249.8	249.7	249.8	249.8	249.7	249.8	249.8	249.8	249.7	249.8	249.7	249.8	249.8
Standard deviation (S_x)	2.7	2.7	2.8	2.3	2.5	2.7	2.3	2.3	2.5	2.7	2.7	2.7	2.7	2.7
Error percent (E_x)	1.1	1.1	1.1	.9	1.0	1.1	.9	.9	1.0	1.1	1.1	1.1	1.1	1.1

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2.2.2 PROCESSING AND PRESERVATION CHAMBERS

Processing and preservation chambers reduce the possibility of random atmospheric contamination during sample splitting, filtration, and preservation. These chambers are required for samples for trace-element determinations (Horowitz and others, 1994). The processing chamber can serve also as a collection chamber for pumped samples. There is no standard design for either fixed or portable chambers; however, to prevent contamination of inorganic samples with metals, the materials used in their construction should be either nonmetallic or completely covered by or embedded in nonmetallic material. Plastic components have been tested and do not emit volatile substances that might contaminate a VOC sample. This can be further documented by collecting an ambient blank in the chamber(s).

Fixed chambers can be enclosures permanently installed in a field vehicle for the sole purpose of sample collection, processing, or preservation. Fixed chambers must not be used as a storage area. The portable chamber illustrated is inexpensive and easily constructed with 1/2-in. white polyvinyl chloride (PVC), tubes which are used to support a clear plastic bag (fig. 2-11). The transparent bag forms a protective tent to work within while collecting, processing, or preserving samples. Another option is to purchase or construct a fixed or portable glove box. The glove box also should have no exposed metal parts. Glove boxes that can be filled with inert gas should be used for samples to be excluded from contact with atmospheric gases.

- ▶ The processing chamber sits over a fixed or portable sink lined with plastic to which a waste-disposal funnel has been attached, allowing rinse water to drain to waste. If a sink is not available, a waste bottle is placed inside the chamber to contain liquid waste and overflow. A hole can be drilled through the top crossbar of the processing chamber through which sample tubing can be inserted and a filter assembly attached.
- ▶ The preservation chamber consists only of the frame and bag cover. A suitable container placed inside the preservation chamber is needed to contain spent preservative ampoules (see NFM 5).

+ Use of separate chambers for sample processing and sample preservation can be the most convenient approach and is necessary if more than one type of chemical preservation will be used. Multiple preservation chambers can help prevent cross contamination and also save time by eliminating the need to change covers between treatments. However, when only a single preservative is needed (such as nitric acid), it can be added while the sample is in the processing chamber without a bag change, after all other samples have been removed.

Where space inside the field vehicle is extremely limited, having both a processing chamber and a preservation chamber set up at the same time might not be feasible. Under such circumstances, the processing-chamber frame may also function as a preservation-chamber frame as long as the cover is changed before sample preservation and is clipped to the chamber frame rather than supported over the frame. The preservation chamber cover must be changed every time the preservation procedure requires a change in gloves (NFM 5).



A.



B.

Figure 2-11. Example of (A) a polyvinyl chloride frame of a processing or preservation chamber, and (B) sample being processed within the chamber. (A, Photograph by B.A. Bernard; B, photograph by Jacob Gibs.)

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2.2.3 FILTRATION SYSTEMS

Filtration separates particulate substances (solid-phase and biological materials) from the solute or aqueous phase of a water sample. Water samples are filtered for analysis of inorganic constituents, organic compounds, and biological materials to help determine the environmental fate and quantify the transport of these target analytes. Detailed information about filtration can be found in Kennedy and others (1976), Ward and Harr (1990), Horowitz and others (1994), and Koterba and others (1995).

For surface water, the most common filtration system consists of a reversible, variable-speed battery-operated peristaltic pump or a metering pump that forces the whole-water sample through tubing into a filter assembly. For ground water, the sample ordinarily is pumped through a sample line directly into a filter assembly. If the sample is collected by bailer, the sample generally is emptied through a valve with fluorocarbon polymer tubing and is transferred to the filter assembly by means of a peristaltic pump. Some bailers can be directly fitted with a filter and hand-pump system.

TECHNICAL NOTE: Separation of solid from aqueous phases can be achieved by methods other than filtration, and data requirements may dictate the need for an alternative method such as centrifugation, ultracentrifugation, dialysis or lipid-membrane separation, and reverse-flow osmosis and tangential-flow filtration.

The filter assembly to be used depends on the type of target analytes, which are discussed in the following sections. Membrane filters commonly used to filter inorganic samples generally are made of cellulose nitrate, polycarbonate polymers, or polyethersulfone-based media. These filter media are not suitable for filtering samples to be analyzed for organics; glass microfiber is the media used for filtering most organic samples (silver filters are used for dissolved-organic compound samples).

A filtered sample is defined operationally by the nominal pore size of the filter media used.

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- ▶ The filter pore size selected depends on study objectives, data requirements, and industry standards.
- ▶ The standard pore sizes of filter media used by the USGS are
 - 0.7 μm for pesticides, most other organic compounds, and some bacteria (NFM 7).
 - 0.45 μm for inorganic constituents (including major ions, radiochemicals, and trace elements), some bacteria (NFM 7), and dissolved organic carbon.
 - 0.2 μm or less for trace-element samples to be analyzed for some geochemical applications and interpretive studies and for nutrient samples for which exclusion of bacteria at the 0.2- μm threshold is desirable.
- ▶ See NFM 7 for a description of filter media for biological analysis.

Inorganic Constituents 2.2.3.A

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Samples for analyses of inorganic constituents are filtered by use of either a disposable capsule-filter assembly or a plate-filter assembly (table 2-7). Construction materials of filtering systems must not be a source of sample contamination with respect to the substances for which the sample will be analyzed.

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Table 2-7. Capsule filter or plate filter requirements for processing of samples for analysis of inorganic chemical constituents

[Table modified from Horowitz and others (1994) and includes only those constituents evaluated in the experiments described in the reference]

Disposable capsule filter required ¹		Disposable capsule filter recommended; plate-filter assembly acceptable
Aluminum	Lead	Anions (chloride, sulfate)
Antimony	Lithium	Calcium
Barium	Manganese	Magnesium
Beryllium	Molybdenum	Nutrients (nitrogen, phosphorus)
Boron	Nickel	Radiochemicals
Cadmium	Silver	Silica
Chromium	Thallium	Sodium
Cobalt	Uranium	Strontium
Copper	Zinc	
Iron		

¹Requirements for surface-water sampling described in Horowitz and others (1994) are generally applicable to ground-water studies, with the caveat that study objectives and data-quality requirements must be fulfilled.

Disposable capsule filter

The protocol for filtering a sample for analysis of inorganic constituents (inorganic sample) is to use a disposable capsule filter such as the Gelman 12175 ground-water sampling capsule (fig. 2-12). Use of a disposable capsule filter eliminates the potential for contamination from a reusable filter assembly (such as the plate filter) and also eliminates time-consuming field-cleaning procedures that are required for a reusable assembly after each sample is filtered.

Horowitz and others (1994) concluded that the disposable capsule filter would not be a probable source of sample contamination for inorganic constituents, including trace elements, major ions, nutrients, stable isotopes, and radiochemicals (table 2-7) if precleaned with a minimum of 1 L of deionized water. Filter media of capsule filters are available in several nominal pore sizes in addition to the standard 0.45 μm .

Advantages of the disposable capsule filter compared to the plate-type filter:

- ▶ Capsule filters are sealed units; hence, the likelihood of contamination is reduced because the filter itself is not handled.
- ▶ Surface area of the capsule filter is roughly three times that of the 142-mm plate filter and is less subject to clogging.
- ▶ Cleaning the filter between samples is not necessary because each capsule filter is used only once and then discarded.
- ▶ QWSU provides quality-control checks of each lot of capsule filters and provides a certificate of analysis with each filter.

Do not use the disposable polyethersulfone capsule filter for organic samples.

Do not reuse filters



Figure 2-12. Disposable capsule filter (published with permission of Gelman Sciences).

Plate-filter assembly

Before 1994, the most common filtration assembly used for USGS studies for filtering inorganic samples was the nonmetallic backflushing plate-filter assembly designed to hold a 142-mm filter. Two screens support the filter media (one above and one below) and allow water flow in either direction without disruption of the membrane. A smooth-tipped plastic forceps is needed to transfer the filter media to the plate of the filter assembly. (Kennedy and others, 1976, give a detailed description of and instructions for use of the plate-filter assembly.)

- ▶ Types of plate-filter assemblies for inorganic samples include:
 - Plastic backflushing assembly (described above), available for 47-mm, 142-mm, and 293-mm filters (fig. 2-13).
 - Plastic vacuum filter assembly for 47-mm-diameter filter; used with either a hand vacuum pump or a peristaltic pump.
 - Fluorocarbon polymer filter assembly designed for 47-mm-diameter filters; can be used for in-line filtering of inorganic or organic samples by changing to the appropriate filter media.
- ▶ The plate-filter assembly is no longer recommended for routine filtration of samples for analysis of inorganic constituents, but is still used to filter samples for analysis of major ions, nutrients, stable isotopes, and radiochemicals (table 2-7).



Figure 2-13. Nonmetallic backflushing plate-filter assembly for 142-millimeter diameter filter media. (Illustration reproduced with permission of Gelman Sciences.)

Trace Organic Compounds 2.2.3.B

Filtering whole-water samples isolates suspended solid-phase substances from the aqueous phase, thus allowing separate determinations of organic compounds in each phase.

As preparation for determining organic compounds that partition onto suspended matter (hydrophobic compounds)—Filtering primarily concentrates suspended materials on the filter, enhancing extraction efficiency and lowering analytical detection limits. This is especially useful for whole-water samples with small concentrations of suspended material where large volumes of sample (4 to 40 L) must be filtered to provide an analyzable mass of suspended materials.

As preparation for determining organic compounds that are more water soluble (hydrophilic)—Filtering is used to remove suspended material, because it often contains interfering constituents that are co-extracted with target analytes.

Filtering also helps to preserve samples for organic determinations because microorganisms that could degrade compounds in the sample are removed (Ogawa and others, 1981).

Equipment needed to filter samples for determination of organic compounds is described in detail in Sandstrom (1995) and includes a positive displacement pump, an aluminum plate-filter assembly (different filter assemblies can be required, depending on the analysis to be done), the filter media, and metal forceps. All equipment and components used for filtering whole-water samples for organic determinations should be made of materials that will not contaminate the sample or sorb analytes and that are suitable for use with organic cleaning solvents. Such materials include stainless steel or aluminum, fluorocarbon polymer, glass, and nonporous ceramics (hard-fused microcrystalline alumina). Other materials can be used, but they must not introduce contaminants or cause sorptive losses, and they must be sufficiently resistant to degradation by cleaning solvents. Use of plastics, rubber, oils and other lubricants are to be avoided because they can result in sample contamination, analytical interference, and (or) sorptive losses.

Metering pump

The valveless piston metering pump consists of a pump head with a reciprocating piston driven by a 12-volt direct current (DC), variable-speed motor (fig. 2-14). It has a delivery rate of up to 500 mL per minute.

The pump head and all wetted parts are constructed of ceramic, fluorocarbon polymer, or stainless steel components, which are resistant to organic solvents. These pumps can tolerate some suspended materials in the sample being pumped, but large concentrations of suspended materials can cause excessive wear of pump parts and strain on the pump motor.

The ceramic piston and shaft of these pumps will break if motor amperage exceeds 4 amps. To avoid this, either a 4-amp DC circuit breaker should be used in-line with the pump power line or an alternating to direct current converter with 4-amp maximum output should be used.

The pump and filter assembly are connected by 1/4-in.-diameter convoluted fluorocarbon polymer tubing with appropriate fittings. The convoluted tubing does not crimp when bent, unlike straight fluorocarbon polymer tubing.



Figure 2-14. Valveless piston metering pump. (Photograph by B.A. Bernard, 1998).

Filtration assemblies

The filtration equipment described in the following list includes various types of plate-filter assemblies, one of which is used for solid-phase extraction.

- ▶ The most common plate-filter assembly consists of two machined aluminum or stainless steel plates, designed to hold a 142-mm-diameter filter, which are held together by locking bolts or a locking ring (fig. 2-15).
 - The plates have fluorocarbon polymer-coated silicone or Viton™ O-rings set in grooves to seal the filter assembly. A stainless steel screen on the lower plate supports the filter.
 - A valve is built into the upper plate to exhaust trapped air. Connectors are built into the center of the top and bottom plates so that inlet and outlet fluorocarbon polymer tubing can be attached.
 - Plate-filter assemblies are available for filters with diameters ranging from 13 to 293 mm. The size selected is determined

by the sample volume to be filtered and the concentration of suspended materials in the sample.

- ▶ In-line filter assemblies used specifically for organonitrogen sample filtering include 13-mm stainless steel or 25-mm disposable nylon membrane filter assemblies. The filter assembly is connected to the pumping system by means of appropriate tubing connectors. Further details are given in NFM 5 and in Sandstrom and others (1994).



Figure 2-15. Aluminum plate-filter assembly for 142-millimeter diameter filter media. (Photograph published with permission of GeoTech Environmental Equipment, Inc.)

64—SELECTION OF EQUIPMENT FOR WATER SAMPLING

- ▶ For solid-phase extraction (SPE), a special disposable in-line plastic filter capsule is used to filter organonitrogen herbicide samples before sending the filtrate to the NWQL. +
- Although filter assemblies made from plastic components should not be used to process water-sediment samples for organic compound determination, they have been approved for a specific SPE method.
- The housing of this capsule filter is a thin nylon membrane filter, 25 mm in diameter, with a pore size of 0.45 μm . The capsule contains polypropylene with luer lock inlet and outlet connectors.
- Filter units for organonitrogen herbicides by SPE are available from QWSU.

Filter media

Tortuous-path depth filters made of borosilicate glass fibers are used to filter most samples for organic determination because the filter materials are basically inert and can be precleaned with organic solvents or baked at 450°C for at least 2 hours. Depth filters also can process larger fluid volumes than membrane filters without clogging. Membrane filters (cellulose or polycarbonate polymers) commonly used to process samples for determination of nutrients and other inorganic constituents are not suitable for filtering samples for organic determinations, mainly because they are not resistant to organic solvents used to preclean sampling and processing equipment. Membrane filters made of silver metal with a 0.45- μm pore size are resistant to organic solvents, but they become clogged too quickly for filtering the relatively large sample volumes (1 to 5 L) often needed for determination of organic compounds. +

For organic samples (except for organonitrogen herbicides and suspended/dissolved organic carbon):

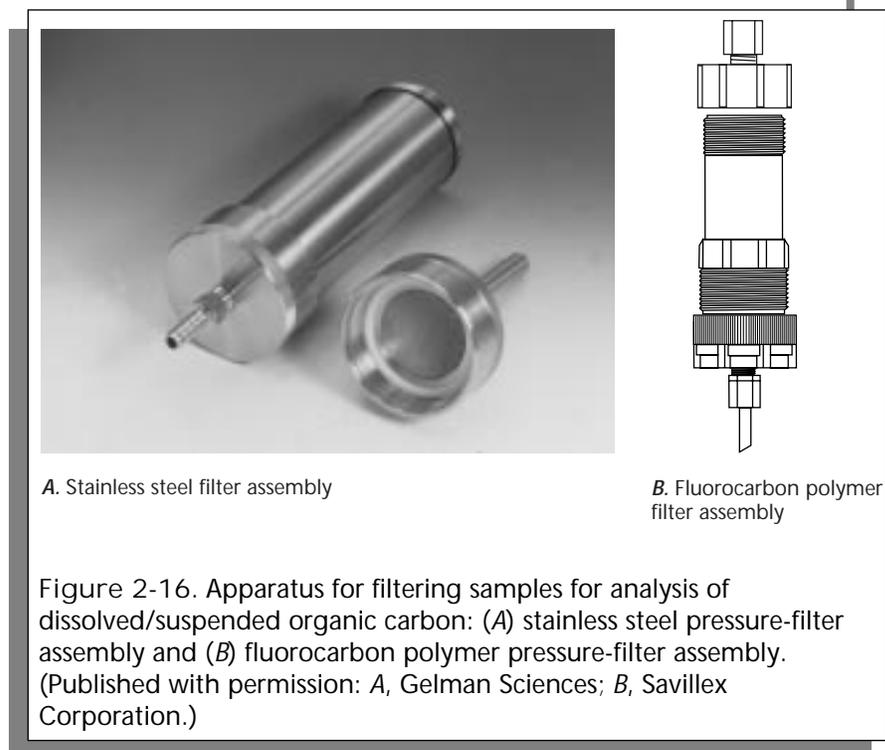
- ▶ Use glass-fiber filter with 0.7- μm nominal pore size, baked at 450°C for at least 2 hours.
- Obtain 142-mm diameter filter through QWSU. +
- Obtain filters with other diameters (0.7- μm nominal pore size and baked) through NWQL by special request.

- ▶ Use only filters without binders. (Acrylic resin binders can leach and contaminate samples, or might not be completely combusted when baked at 450°C.) Binder-free glass-fiber filters are available in various diameters ranging from 13 to 293 mm.

Dissolved and Suspended Organic Carbon 2.2.3.C

A stainless steel or fluorocarbon polymer pressure filter assembly (fig. 2-16) fitted with a 47-mm, 0.45- μm -pore-size silver membrane filter is used to separate dissolved from suspended phases of organic carbon.

- ▶ Use a hand-pressure pump, peristaltic pump, or an organic-free nitrogen gas tank fitted with clean tubing to apply pressure to filter dissolved organic carbon (DOC) from suspended organic carbon (SOC) samples (Office of Water Quality Technical Memorandum 78.06, see "Internal Documents").



66—SELECTION OF EQUIPMENT FOR WATER SAMPLING

- ▶ Maintain gas pressure applied to the filter assembly at less than 15 lb/in².
- ▶ Ensure that the gas is clean by way of gas-purveyor certification or by attaching an in-line 0.2-mm Gelman Acro™ 50 hydrophobic membrane filter disk. Do not use any other type of filter.
 - Because the sample does not contact the in-line gas-filter disk, the disk can be used to filter gas used to process multiple samples, or until the disk clogs or is contaminated.
 - Store in-line filter disk in a resealable plastic bag between uses.

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2.2.4 PUMP TUBING

Pump tubing refers to the sample lines used with peristaltic, metering, and submersible pumps. Field personnel are cautioned to evaluate possible artifacts in a sample associated with pump tubing and tubing connections.

- ▶ Tubing connectors and connections that contact the sample should be made of inert material, to the extent possible. Stainless steel connections must be the highest grade available (SS 316). If flexible copper, aluminum, or stainless steel tubing is used for CFC sampling, it should be refrigeration grade. Such fittings ordinarily are delivered with coatings of machining lubricants, which must be removed by cleaning before use.
 - Greaseless fittings can be ordered. Fluorocarbon polymer, silicone, C-Flex™ pump tubing commonly are used with portable submersible pumps and with peristaltic or metering pumps. Use of a fluorocarbon polymer material generally is recommended because fluorocarbon polymers are relatively inert with respect to many inorganic and organic analytes (table 2-8).

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- + ▶ **Silicone tubing is suitable when sampling for inorganic analytes only, and only after appropriate cleaning (see NFM 3).**
 - Measurable concentrations of silica (0.09 to 0.24 mg/L) have been detected in blank samples passed through silicone tubing (Horowitz and others, 1994). These concentrations are likely to be significant only if low-ionic-strength water is being sampled.
 - Silicone tubing was not designed for use with acids.
 - Silicone tubing is gas permeable and very sorptive of organic compounds.
- + ▶ **C-Flex™ tubing is made from a thermoplastic elastomer and is suitable for use when sampling for all inorganic analytes.**
 - C-Flex™ is relatively resistant to acid. Acid resistance is a factor because dilute hydrochloric acid is required in cleaning procedures (see NFM 3).
 - C-Flex™ is less permeable to gas than silicone tubing.

Table 2-8. Common varieties and characteristics of fluorocarbon polymer tubing

FEP (fluorinated ethylene polypropylene)
<ul style="list-style-type: none"> • Most transparent • Best abrasion resistance • High flexibility • Least expensive of the Teflon™ varieties
PFA (perfluoroalkoxy)
<ul style="list-style-type: none"> • Less transparent than FEP • Virtually nonporous (nonpermeable) • Most expensive of the Teflon™ varieties
PTFE (polytetrafluoroethylene)
<ul style="list-style-type: none"> • Least transparent; milky to white • Most flexible • Midpriced between FEP and PFA
Kynar™ (polyvinylidene fluoride)
<ul style="list-style-type: none"> • Translucent • Not very flexible • Less expensive than the Teflon™ varieties
Tefzel™ (Ethylenetetrafluoroethylene)
<ul style="list-style-type: none"> • Withstands higher pressure than Teflon™ • Most expensive

68—SELECTION OF EQUIPMENT FOR WATER SAMPLING

- ▶ **Fluorocarbon polymer tubing is recommended when sampling for most inorganic and organic analytes. Fluorocarbon polymer tubing is available in corrugated, convoluted, and straight-wall configurations (fig. 2-17).** +
 - Fluorocarbon polymer tubing premolded to the shape of the pump head is available.
 - Convoluted fluorocarbon polymer tubing is flexible and easy to handle. Attach convoluted tubing to each end of the premolded tubing.
 - Fluorocarbon polymer tubing sheathed in another plastic is available at lower cost but is not recommended because the fluorocarbon polymer twists, constricts, and cracks.

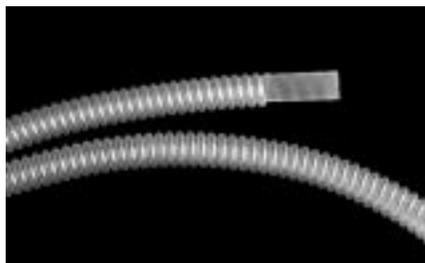
- ▶ **Polyvinyl chloride (PVC) tubing (Tygon™) is suitable for inorganic samples only, and it must be appropriately cleaned prior to contact with inorganic samples.**
 - PVC tubing can be washed with dilute acid.
 - PVC tubing has the lowest gas permeability of any peristaltic pump tubing.
 - For use with a peristaltic pump, PVC tubing has a shorter life than silicone, C-Flex™, or Norprene™. +
 - PVC tubing may leach plasticizers.

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- + ▶ Norprene™ tubing is made from a thermoplastic elastomer (a polypropylene base with USP mineral oil) and is suitable when sampling for inorganic analytes only. It must be appropriately cleaned prior to contact with samples collected for inorganic analysis.
- Norprene™ tubing can be washed with dilute acid.
 - Norprene™ tubing may leach USP mineral oil.
 - For use with a peristaltic pump, Norprene™ tubing has the longest life of any manufacturer-recommended tubing material.
 - The gas permeability of Norprene™ tubing is lower than that of silicone tubing and greater than that of PVC tubing.

For filtering ground-water samples that are pumped directly from the well to a filter assembly, C-Flex™ or fluorocarbon polymer tubing is recommended. (Nylon tubing also might be an acceptable alternative, but it has not been tested as a source of trace-element or organic compound contamination.) Silicone tubing is not recommended because it is gas permeable.

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A. Convoluted (spiral) design



B. Corrugated (parallel) design

Figure 2-17. Flexible fluorinated ethylene polypropylene (FEP) tubing: (A) convoluted design and (B) corrugated design. (Published with permission of Cole-Parmer Instrument Company: A, Copyright 1992. Cole-Parmer does not warrant this illustration to be current, accurate, or suitable for any purpose; B, Copyright 1987. Cole-Parmer does not warrant this illustration to be current, accurate, or suitable for any purpose.)

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