

## 5.2.1 INORGANIC CONSTITUENTS

Most filtration systems currently used by the USGS are appropriate for filtering wholewater samples, if the limitations of each system are taken into account. **Standard USGS procedure is to filter inorganic-constituent wholewater samples through a 0.45-micrometer ( $\mu\text{m}$ ) pore-size disposable capsule filter.** Filtration through media with pore sizes other than 0.45  $\mu\text{m}$  or with other equipment (such as tangential-flow devices) depends on the use and interpretation of the data and can yield substantially different results for trace-element concentrations.

Data-quality requirements for interpretive studies of ground-water and surface-water chemistry can dictate filtering the sample through a nominal pore size of  $\leq 0.2 \mu\text{m}$ . The quality-assurance procedures used for samples filtered through the 0.45- $\mu\text{m}$  nominal-pore-size capsule, plate, or other filtration equipment also are required for the  $\leq 0.2\text{-}\mu\text{m}$  filters. If concentrations of target analytes are analyzed at sub-parts-per-billion levels, more stringent QA/QC measures are needed. Such samples can be filtered through a plate filter or other filtration equipment (for example, a 47-mm-diameter vacuum-filter unit) as long as the equipment used is approved by the study or program, data-quality requirements are met, and additional quality-control samples are collected. For additional information on filtration artifacts, procedures, and equipment, see Kennedy and others (1976), Salonen (1979), McCarthy (1988), McCarthy and Zachara (1989), Puls and Barcelona (1989), Ward and Harr (1990), Horowitz and others (1992, 1994), Williams and others (1993), Robards and others (1994), and Koterba and others (1995).

Cleaning and conditioning of various filter media used for inorganic constituents are summarized in table 5-3. Contamination during sample filtration can be reduced by following the instructions given for cleaning, conditioning, and handling of the filter media.

**Table 5-3.** Field cleaning and conditioning procedures for media used to filter samples for inorganic-constituent analysis

[ $\mu\text{m}$ , micrometer; mL, milliliter; sample, the water to be sampled;  $\mu\text{g/L}$ , microgram per liter; mm, millimeter;  $\text{HNO}_3$ , 1 molar solution of ultrapure-grade nitric acid; HCl, 1 molar solution of ultrapure-grade hydrochloric acid; nutrients, nitrogen and phosphorus species; DIW, District- or laboratory-produced deionized water of known quality, ASTM Type-1 grade or better; IBW, laboratory-produced inorganic-grade blank water; *N*, normal; >, greater than]

Description	Filter media	Field cleaning/ conditioning	Application
Disposable capsule filter <sup>1</sup> (Polypropylene)	Polysulfone, pleated membrane, 0.45- $\mu\text{m}$ or 0.2- $\mu\text{m}$ pore size	Clean with 1,000 mL DIW and remove residual DIW <sup>2</sup>  Condition with 25 mL sample	Major ions and nutrients; trace elements with concentrations > 1 $\mu\text{g/L}$ ; radio- chemicals and isotopes
Plate filter — 142 mm (Polycarbonate or acrylic)	Cellulose nitrate, tortuous path (0.45 and 0.1 $\mu\text{m}$ are most commonly used pore sizes)	Clean with 500 mL DIW and extract residual DIW  Condition with 100 mL sample	Major ions and nutrients; trace elements if concentrations > about 100 $\mu\text{g/L}$
Cartridge or hand- pressure filter assembly—47 mm (Polypropylene or fluorocarbon polymer)	Cellulose nitrate, tortuous path (0.45, 0.2, and 0.1 $\mu\text{m}$ are most commonly used pore sizes)	Clean with 100 mL DIW and remove residual DIW  Condition with 20 mL IBW or 10 mL sample	Major ions and nutrients; trace elements with concentrations at about 1 $\mu\text{g/L}$ or greater
Cartridge or hand- pressure filter assembly—47 mm (Fluorocarbon polymer)	Polycarbonate (such as Nuclepore), direct path (0.40 and 0.1 $\mu\text{m}$ are most commonly used pore sizes)	Soak in $\text{HNO}_3$ rinse with IBW. <sup>3</sup> Remove residual IBW  Condition with 20 mL IBW or 10 mL sample	Major ions and nutrients; trace elements with concentrations at about 1 $\mu\text{g/L}$ or greater

<sup>1</sup>Example: Gelman Sciences 12175 (0.45  $\mu\text{m}$ ); 600 square-centimeter filtration area. Other disposable capsule filters are available that have different effective filtration area, media type, and media pore size.

<sup>2</sup>For trace-metal analyses at nanogram-per-liter concentration levels, first acid rinse with 500 mL of 1-*N* HCl (polysulfone membranes cannot withstand  $\text{HNO}_3$ ).

<sup>3</sup>Substitute HCl for  $\text{HNO}_3$  if sampling includes nutrients.

- ▶ Before filtering, designate one member of the processing team as Clean Hands (CH) and another member as Dirty Hands (DH) if using the CH/DH method (NFM 4).
- ▶ Wear appropriate, disposable, powderless gloves throughout the process. Vinyl gloves are adequate for inorganic-constituent sampling.
- ▶ Filter the samples within a processing chamber to minimize the possibility of contamination.

### 5.2.1.A Capsule-Filter Procedure

The capsule filter is a disposable, self-contained unit composed of a pleated filter medium encased in a plastic housing that can be connected in-line to a sample-delivery system (such as a submersible or peristaltic pump) that generates sufficient pressure (positive or negative) to force water through the filter. Filter media are available in several other pore sizes, but 0.45  $\mu\text{m}$  is the pore size used routinely for most studies at this time. The capsule filter is required for most studies when filtering samples for trace-element analysis and is recommended when filtering samples for major-ion or other inorganic-constituent analyses.

The following instructions implement Clean Hands/Dirty Hands (CH/DH) techniques and the other QA procedures that are required for trace-element samples with analyte concentrations at the parts-per-billion (ppb) level and that are recommended as good field practice for all samples.

- ▶ The DH team member performs operations that are outside of the processing chamber and the CH team member performs operations inside the chamber. DH and CH must wear appropriate disposable, powderless gloves (gloves).
- ▶ Pre-clean capsule filters (step 5 below) before leaving for the field to save field time.

**Fill bottles for filtered samples in this sequence:  
FA (trace elements) → FAM (mercury) → FA and  
FU (major ions) → FCC or FCA (nutrients) →  
FAR and all other samples.**

*To prepare the work space, sample bottles, and capsule filter:*

1. *CH/DH*: Put on one or several layers of gloves.
2. *CH*: Assemble processing chamber, attach chamber cover, and change gloves. Place capsule filter and sample bottles into chamber, and run discharge end of peristaltic pump tubing into the chamber. Open DIW<sup>7</sup> container and cover it with a plastic bag to prevent contamination from airborne particulates.
3. *CH/DH*: (*CH*) Insert intake end of peristaltic pump tubing through the plastic covering and into a I-L container of DIW.
  - a. (*DH*): Attach tubing to peristaltic pump head and pump DIW to fill tubing.
  - b. Discharge waste rinse water through a sink funnel or a toss (waste) bottle.
4. Discard DIW stored in DIW-prerinsed sample bottles. If sample bottles were not DIW-prerinsed by field personnel:
  - a. Wearing gloves, rinse off exterior of each bottle.
  - b. Pour DIW into bottle until about one-tenth full.
  - c. Cap bottle and shake vigorously about five times.
  - d. Uncap and empty bottle.
  - e. Repeat b–d of step 4 twice (for a total of three times).
  - f. Recap bottles until ready to field rinse.

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<sup>7</sup>Office of Water Quality Technical Memorandum 92.01 describes the quality required of the deionized water.

5. **Clean the capsule filter.** If the capsule filter was precleaned, go to the sections that follow on "To filter a composite sample" or "To filter a pumped sample," as appropriate. The steps below comprise sufficient precleaning of the filter for inorganic analytes at the parts-per-billion (ppb) concentration level. More rigorous precleaning procedures that include rinsing with trace-metal-grade hydrochloric acid are required for samples containing ppb concentrations of target analytes (table 5-3). **Only CH touches those portions of tubing that will be in direct contact with the DIW or capsule filter.**
- a. *CH*: In the processing chamber, remove capsule filter from protective bags.
    - Attach pump tubing to inlet connector of capsule filter, keeping tubing as short as possible. **Make sure direction of flow through capsule filter matches the direction-of-flow arrow on the side of the capsule.**
    - To help minimize aeration of the sample (usually for ground-water samples), secure a short length of clean fluorocarbon polymer tubing onto capsule filter outlet to extend into the sample bottle so the bottle can be filled from the bottom up.
  - b. *CH/DH*: Pump 1 L of DIW through capsule filter; discharge waste rinse water through a sink funnel or to a toss bottle.
    - *DH* operates the pump at a low speed.
    - *CH* inverts the capsule filter so the arrow on the housing is pointing up. (This expels trapped air from the capsule during initial filling; do not allow water to spray onto the chamber walls.)
  - c. *DH*: Remove tubing from DIW reservoir and continue to operate pump in forward mid-range speed position to drain as much of the DIW that remains in the capsule filter as possible. While pump is operating, shake capsule filter to help remove any entrained DIW.
  - d. *CH*: Detach capsule filter from peristaltic pump tubing, put it into a clean, sealable plastic bag, and place in a corner of the processing chamber until ready for use.

Filtration procedures differ somewhat, depending on how the sample is collected. If the sample is collected using discrete collection equipment, such as the surface-water bag or bottle sampler or ground-water bailer, use the procedures described below in "To filter a composite sample." If the sample is collected by pumping it directly from the source, use the procedures described below in "To filter a pumped sample." Ground-water samples usually are not collected as a composite. If samples are to

be withdrawn from a well using a bailer, consider using a bailer to which the capsule filter or other filtration device can be connected inline to the bailer bottom-emptying device. Pouring a sample from the top of the bailer into another receptacle aerates the sample and therefore is not a generally recommended procedure for processing ground-water samples.

***To filter a composite sample (generally for surface water):***

1. Field rinse peristaltic pump tubing with the water to be sampled.
  - a. *CH:* Rinse the outside of each end of the peristaltic pump tubing.
  - b. *CH:* Transfer intake end of peristaltic pump tubing into composite sample. If a churn splitter is used, transfer intake end of peristaltic pump tubing through churn funnel and reseal plastic bag around the tubing.
  - c. *DH:* Start peristaltic pump to slowly pump sufficient sample to completely fill pump tubing.
  - d. *CH:* Discard rinse water through the sink funnel or into a toss bottle or other receptacle and dispose of appropriately. Prevent water from ponding in the processing chamber.
  - e. *DH:* Stop peristaltic pump after tubing is field rinsed.
2. Field rinse capsule filter:
  - a. *CH:* Remove cleaned capsule filter from plastic bag and attach discharge end of the peristaltic pump tubing to the inlet connector on the capsule filter.
    - A clean, small plastic hose clamp may be used to secure the discharge end of the tubing to the capsule filter inlet connector.
    - Check that the direction of sample flow through the capsule filter matches the direction of the arrow on the capsule.
  - b. *DH:* Operating the pump at low speed, pump sample through the tubing to the capsule filter.
  - c. *CH:* Turn capsule filter so that the outlet is pointing up (arrow on capsule housing is pointing up) and flow of the sample forces trapped air out of the capsule filter while it is filling. **Do not let sample spray onto chamber cover.**
    - The chamber cover must be changed if sample has sprayed onto it.
    - If some water that sprayed onto the chamber cover has dripped into the sample bottle, discard the bottle, change the cover, and collect a new sample.

- d. *DH*: Stop the peristaltic pump as soon as the capsule filter is full of sample and all air in the capsule filter has been expelled.

TECHNICAL NOTE: The goal is to minimize clogging the filter medium with suspended materials by minimizing the volume of sample that will be used to field-rinse the filter.

3. Collect sample filtrate.

- a. *CH*: Check that there is a tight connection between the pump tubing and the capsule filter.

*DH*: Check that the intake tube is properly inserted in the sample and start the pump.

*CH*: Collect a maximum of 25 mL of the water to be sampled as it discharges through the filter. **Do not exceed 25 mL.**

*CH*: Field rinse a precleaned 250-mL FA bottle for trace-element sample only with sample filtrate.

*DH*: Stop the pump in time to prevent losing filtrate to waste.

*CH*: Cap bottle, shake vigorously, and then discard rinse water into appropriate receptacle.

- b. *DH*: Start pump and resume flow from pump to the filter.

*CH*: **Filter only the next 200 mL of the sample** into the trace-element FA bottle (fill to top of upper lip of standard 250-mL polyethylene bottle). Cap bottle securely and set aside for chemical treatment.

- c. *DH*: Stop the pump after the trace-element FA bottle is filled.
- d. If a filtered mercury sample is required, restart pump and repeat steps 3a–c, substituting a FAM bottle for the FA bottle.
- e. *CH*: Field rinse any remaining sample bottles for inorganic analyses. **Use no more than a total of 100 mL of filtrate per capsule filter to field rinse any remaining bottles for filtered sample.**
- f. Fill remaining bottles in the following order: (1) major cations, (2) nutrients and major anions (including alkalinity), (3) radiochemicals (Appendix A5-A), and (4) stable isotopes. Cap each bottle immediately after filling.

***To filter a pumped sample (usually ground water):***

Ground-water samples usually are withdrawn from a well by means of a submersible pump. Note that this method might be appropriate for some surface-water samples. The capsule filter or other filter assembly is connected inline with the sample tubing in order to collect samples directly from the well.

- ▶ When sampling ground water, DH should check that the turbidity values recorded at the end of purging have remained stable. Equipment changes or adjustments that disrupt sample flow can affect sample turbidity and should be avoided. If sample flow is disrupted, pump for several minutes until ambient turbidity values are reestablished.
- ▶ **Maintain a smooth, uniform flow.** Do not stop pump or divert flow from capsule filter or other filter assembly during bottle field rinse or filtration, if possible.

TECHNICAL NOTE: If using a three-way valve, changing the setting to divert the flow of sample being pumped to the filter with a submersible pump can cause air bubbles to form, can air-block the filtration equipment, and can cause changes in pumping rate that could result in increased turbidity. These effects should be avoided to preserve sample integrity; therefore, flow to the filter should not be stopped until all filtration is complete.

1. Field rinse the capsule filter with sample water:
  - a. *CH:* Ensure that the sample line is full of sample and free of bubbles; then attach the discharge end of the sample line to the inlet connector on the capsule filter.
    - Practice your technique for attaching the capsule filter to the tubing carrying flowing water so that water does not spray onto chamber walls.
    - Check that the direction of flow matches the direction of the arrow on the capsule.
  - b. *DH:* Adjust the sample flow through the sample line to the capsule filter, keeping a slow rate of flow.
  - c. *CH:* Turn the capsule filter so the outlet is pointing up (arrow on capsule housing is pointing up) and the flow of sample forces trapped air out while the capsule filter is filling.
    - Do not allow water to spray onto chamber walls.
    - The capsule filter should be full of sample. No air should be left in the capsule filter.

- d. Field rinse bottles for inorganic-constituent filtered samples with sample filtrate (section 5.0.3). Use bottles that were already rinsed three times with DIW. Determine whether the potential clogging of pores in the filter medium is of concern for your samples (see TECHNICAL NOTE below).

*CH:* Fill a 250-mL FA bottle for trace elements with 25 mL of sample filtrate; cap, shake vigorously, and discard rinse water into appropriate receptacle.

*CH:* Fill a FA bottle for trace elements with about 200 mL of sample filtrate (to top of upper lip of 250 mL bottle). Cap bottle and set aside for chemical treatment.

*CH:* If a mercury sample is required, field rinse and fill a FAM bottle using the same procedure as for the 250-mL FA bottle.

*CH:* Field rinse remaining bottles, trying to use no more than an additional 100 mL of sample filtrate.

TECHNICAL NOTE: Depending on sample turbidity and composition, the nominal pore size of filter media tends to decrease as the volume of sample passed through the filter increases because pores are clogged by sediment loading or mineral precipitation on the filter (Horowitz and others, 1994). Ground water with turbidity  $\leq 5$  NTU should not affect filter pore size appreciably. To minimize the chance of filter clogging, limit the volume of sample passed through the filter by eliminating the field rinse—be sure that you use clean bottles and fill them one after the other. For ground-water sampling, do not stop the pump during the field-rinse and sampling process.

- e. *CH:* Collect sample filtrate immediately into any remaining bottles in the following sequence (flow rate should be slow enough to avoid splashing sample out of the bottle): (1) major cations, (2) major anions and nutrients (including alkalinity sample for field titration), (3) radiochemicals (check Appendix A5-A for bottle-rinse and filtration requirements), (4) stable isotopes.
- f. *CH:* Cap each bottle immediately.

**Rinse FA, FU, FAM, FCA, and FCC bottles  
with filtered sample—not with raw sample.**

*After collecting filtered samples:*

1. *CH:* If samples require chemical treatment, place FA bottles in the preservation chamber and go to section 5.4.
2. For filtered samples that do not require chemical treatment:
  - a. *CH:* Set samples outside processing chamber.
  - b. *DH:* Check that information on the bottle label is correct and complete.
  - c. *DH:* Pack samples that require chilling in ice or refrigerate immediately.
  - d. *DH:* Pack remaining samples for shipping (section 5.5).
3. Rinse all reusable equipment with DIW immediately—before equipment dries.
  - *CH:* If equipment will be reused at another site before returning to the office, rinse immediately with DIW and field clean tubing and other sample-wetted parts of the equipment using the prescribed cleaning procedures (NFM 3).
  - *CH:* If equipment or tubing will not be reused before returning to the office, rinse immediately with DIW and store rinsed tubing and equipment in plastic bags for office or laboratory cleaning.
4. **Discard the capsule filter after filtering each sample—do not reuse.**
5. Document the filtration procedures used on field forms and in field notes.

**Use of the 0.45- $\mu$ m disposable capsule filter for trace-element samples is required for many USGS programs.**

### 5.2.1.B Plate-Filter Procedure

The filtering procedure using a 142-mm-diameter plastic plate-filter assembly is described below. The procedure remains basically the same for plate-filter assemblies of different diameters.

*Prepare and precondition plate-filter assembly:*

The following instructions pertain to either a 142-mm-diameter or a 47-mm-diameter plastic plate-filter assembly and require that the assembly components have been rigorously cleaned (NFM 3). To avoid recleaning in the field, prepare a set of filtration equipment for each well or surface-water sampling station. (Ignore Step 3 below if plate-filter assembly has been rinsed in the office.)

1. *CH:* Put on gloves. In a processing chamber, open a clean plate-filter assembly and load with the filter.
  - a. Using nonmetallic forceps, place the bottom retaining screen on the base of the filter assembly. **Do not interchange bottom and top retaining screens.**
  - b. Place the filter on top of bottom retaining screen using clean, blunt plastic or ceramic forceps. Do not touch the filter with hands (gloved or ungloved).
    - Be sure that only one filter is transferred from its original container directly to the plate of the filter assembly. Take care not to transfer the paper liner that separates each filter.
    - The filter should never be removed from the original container until each is transferred to the plate-filter assembly for use. (Exception: polycarbonate (Nuclepore) filter medium is precleaned with acid solution. If transferring one of these, hold the filter with forceps and rinse off acid with inorganic blank water (IBW) dispensed from wash bottle.)
  - c. Using forceps, place the top retaining screen on top of the filter.

TECHNICAL NOTE: If filtering sediment-laden water, a prefilter can be placed between the filter and the top retaining screen.
  - d. Dispense 10 to 20 mL of DIW from a wash bottle onto the filter.

- e. Close the plate-filter assembly by aligning the top and bottom plates and lightly tightening the plastic bolts, followed by finger tightening opposite pairs of bolts. **Overtightening can cause the plate-filter assembly to warp and leak.** Check that O-rings are in place before closing the assembly. Change gloves.
2. *DH/CH*: Pass the discharge end of the pump tubing through the hole in the side or top of the processing chamber. **Only the CH team member touches sections of tubing that will be in direct contact with the plate-filter assembly.**
    - Keep tubing as short as practical.
    - Attach a short piece of clean tubing to outlet connector of plate-filter assembly.
  3. *DH/CH*: Rinse the plate-filter assembly with DIW, using a peristaltic pump, as follows (**rinsing must be repeated each time a clogged filter is replaced with a new filter**):
    - a. *CH/DH*: Place intake end of peristaltic pump tubing into a 500-mL container of DIW. Turn pump on low speed.
    - b. *CH*: Open the air-vent valve on top of the plate-filter assembly. Tilt the filter assembly slightly to the side and squeeze the outlet tube closed to force trapped air out through the vent. Release the outlet tube. (Venting trapped air is necessary because air bubbles will reduce the effective filtering area by preventing sample from passing through the filter.)
    - c. *CH*: Close valve when top is filled with sample.
    - d. *CH*: Pump sample through the plate-filter assembly and discard this field-rinse water through the sink funnel or into the toss bottle to prevent the water from ponding in the bottom of the processing chamber.
    - e. *CH/DH*: Remove intake end of the pump tubing from the DIW container and continue to pump, draining as much of the remaining DIW from the plate-filter assembly as possible.
  4. If using a peristaltic pump to transfer the sample to the processing chamber (go to step 5 if sample delivery is with a submersible ground-water pump):
    - a. *CH*: Rinse intake end of the peristaltic pump tubing with the water to be sampled.
    - b. *CH*: Transfer intake end of the peristaltic pump tubing into the container of sample. If a churn splitter is used, transfer the intake end through the churn funnel and reseal the plastic bag around the tubing.

- c. *CH*: Remove peristaltic pump tubing from the inlet connector of the plate-filter assembly and hold the end of the tubing over the sink funnel or toss bottle.
  - d. *DH/CH*: Start the peristaltic pump in the forward position at slow speed and pump sufficient sample to fill and rinse all pump tubing. Stop the pump after the tubing is rinsed.
5. *CH*: Attach the discharge end of the peristaltic-pump or submersible pump tubing to the inlet connector of the plate-filter assembly.
    - Keep tubing as short as practical.
    - A clean, small, plastic hose clamp can be used to secure the discharge tubing to the inlet connector.
  6. *DH*: Start sample flow to the plate-filter assembly.
  7. *CH*: Vent trapped air and rinse plate-filter assembly as instructed in steps 3 b–d above.
    - If using a peristaltic pump, turn pump on low speed.
    - If using a submersible pump, maintain a slow and steady flow rate.
  8. *CH*: Rinse appropriate sample bottles once with filtrate. Filter no more than 100 mL of sample for the final rinse of all sample bottles that require rinsing.
  9. Filter samples, filling bottles in the following order, as applicable to study objectives and sample designation:
    - a. Trace elements

TECHNICAL NOTE: Study objectives and data-quality requirements govern procedures to be used if the filtered trace-element sample is to reflect concentrations of analytes in true solution (the dissolved fraction). Such interpretive studies of ground-water or surface-water chemistry commonly use  $\leq 0.1\text{-}\mu\text{m}$  filter media and plate-filter assembly or a tangential flow method of phase separation. Note that any deviation from the standard procedure for collecting filtered trace-element samples through the  $0.45\text{-}\mu\text{m}$  capsule filter must be documented and reported with the analytical results.
    - b. Major cations
    - c. Nutrients, major anions, and alkalinity sample
    - d. Radiochemicals
    - e. Isotopes

10. *CH*: If the filter medium clogs before the needed volume of water is filtered, carefully remove the filter and replace with a new filter. Repeat steps 1 through 7. Cap each bottle immediately after filling.
  11. **If samples require chemical treatment → Go to section 5.4.**
  12. *DH*: After filtration,
    - a. Check that information on the bottle label is complete and set the samples aside for shipping (section 5.5). Samples that must be chilled need to be refrigerated or packed in ice as quickly as possible and maintained at 4°C without freezing.
    - b. Disconnect and disassemble the plate-filter assembly. **Discard the used filter.**
    - c. Rinse all equipment with DIW immediately after use and before it dries. Equipment that has dried after sampling without being rinsed or cleaned needs to be cleaned vigorously with a detergent and rinsed with DIW before the next use. Nonmetallic equipment must also be acid rinsed.
    - d. Put rinsed tubing in a plastic bag for cleaning at the office laboratory.
    - e. If equipment is to be used at the next site, field clean all the equipment using the procedures described in NFM 3. Field cleaning between sampling sites is carried out while still at the sampling site.
  13. Document on field forms and in field notes any modifications to the filtration procedures used.
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