

- ▶ 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.**

Revise the above sequence to: FA (trace elements and cations) -> FAM (mercury) -> FCC or FCA (nutrients) -> FU (anions) -> FAR -> DOC 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 **1-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.
- +

+

---

<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 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

**Capsule Filter requires 2-L rinse of IBW or DIW**

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.

13. 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/major ion 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. Do not rinse the DOC bottle.**

- f. Fill remaining bottles in the following order: (1) nutrients and major anions (including alkalinity), (2) radiochemicals (Appendix A5-A), and (3) stable isotopes. Cap each bottle immediately after filling. Collect capsule-filtered dissolved organic carbon (DOC) last.

Note: Page revised 2/27/2009.

***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. Do not rinse the DOC bottle.

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) nutrients and major anions (including alkalinity sample for field titration), (2) radiochemicals (check Appendix A5-A for bottle-rinse and filtration requirements, (3) stable isotopes, (4) capsule-filtered dissolved organic carbon (DOC).
- f. *CH:* Cap each bottle immediately.

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

Note: Page revised 2/27/2009.

*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.**