5.2.1.A Capsule- and Disk-Filter Procedure

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Capsule and disk filters are disposable, self-contained units composed of a pleated or woven filter medium encased in a polypropylene or other plastic housing that can be connected inline 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.[1] Filter media are available in several pore sizes, but 0.45 µm is the pore size used routinely for most studies at this time. Capsule or disk filters (table 5.2.1.A.1) are required routinely for most studies when filtering samples for trace-element analyses and are recommended when filtering samples for major-ion or other inorganic-constituent analyses.

Table 5.2.1.A.1. Filter rinsing and conditioning procedures for capsule and disk filters used to filter samplers for inorganic-constituent analysis.

[µm, micrometer or micron; mL, milliliter; sample, the water to be sampled; µg/L, microgram per liter; mm, millimeter; HNO_3, 1-normal solution of ultrapure-grade nitric acid; HCl, 1-normal 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, certified inorganic-grade blank water; >, greater than]

Description	Filter media	Field rinsing	Filter Conditioning		Application
Capsule filter,[1] disposable (Polypropylene)	Versapor®, pleated membrane, 0.45 µm	Rinse with 2L IBW or DIW[3] (quality assured);dispel residual rinse water.	25 mL of sample if 250-mL acid-rinsed bottle	50 mL of sample if 125-mL acid-rinsed bottle	Major ions and nutrients; trace elements, radiochemicals, and isotopes
Disk filter,[2] disposable	Thermopor® membrane, 0.45-µm pore size	Rinse with 50 mL IBW or DIW (quality assured); dispel residual rinse water.	10 mL of sample if 250-mL acid-rinsed bottle	20 mL of sample if 125 mL acid-rinsed bottle	Major ions and nutrients; trace elements, radiochemicals, and isotopes

[1]Example: Pall Versapor® High Capacity (0.45 µm); 700-square-centimeter filtration area.

[2]Example: Pall AquaPrep® (0.45 µm); 19.6-square-centimeter filtration area.

[3]For trace-metal analyses at nanogram-per-liter concentration levels: Large-capacity capsule—first acid rinse with 500 mL of 1-*N* HCl; small-capacity disk—first acid rinse with 50 mL 1-*N* HCl. (Note: Some membrane media cannot withstand HNO_3.)

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 microgram-per-liter (µg/L)[2] 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 disposable, powderless nitrile gloves (gloves) when handling equipment and chemical solutions. Do not allow the water that enters the sample bottle to contact gloved (or bare) hands. After donning, rinse the gloves with deionized water (DIW) of known quality or inorganic blank water while gently rubbing together to remove any surface residue and before handling sampling equipment.

[1] The maximum operating pressure given by the Pall Corporation for the GWV large-capacity filter is 50 psi and 75 psi for the AquaPrep® disk filter

[2] Commonly referred in some scientific literature in terms of parts per billion (ppb).

► Filters (step 5 below) may be rinsed before leaving for the field to save field time, provided they are stored individually in sealed plastic bags, isolated from potential sources of atmospheric contamination, and used the same day.

Fill bottles for filtered samples in this sequence:

FA (trace elements and cations) → FAM (mercury) →

FCC or FCA (nutrients) → FU (anions) → FAR (radiochemicals) →

all other samples , then DOC (dissolved organic carbon) .

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 the capsule filter, optional hose barb adapter (adapter is not compatible with the disk filter) and sample bottles into the chamber, and insert discharge end of peristaltic pump tubing into the chamber. Open DIW container and cover it with a plastic bag to prevent contamination from airborne particulates.
3. *CH/DH*: (CH) Rinse the intake end of peristaltic pump tubing with DIW then insert it through the plastic covering and into a l-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.
5. **Rinse the capsule filter. (These procedures are generally also followed for the disposable disk filter, but using the rinsing procedures described on table 5.2.1.A.1). If the capsule filter was prerinsed →**, *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 rinsing of the filter for inorganic analytes at the parts-per-billion (ppb) concentration level. More rigorous procedures that include acid rinsing with trace-metal-grade hydrochloric acid are required for samples containing ppb concentrations of target analytes (table 5.2.1.A.1). If the capsule filter is acid rinsed, check that the pH of the DIW rinse effluent is less than 4.

- **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 the capsule filter from its protective bag.

   - Attach the optional hose barb adapter, if appropriate, and pump tubing to the inlet connector of capsule filter, keeping the tubing as short as possible. **Make sure the 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 groundwater samples), secure a short length of clean fluorocarbon polymer tubing onto the capsule filter outlet to extend into the sample bottle so that the bottle can be filled from the bottom up.

b. *CH/DH*: Pump 2 L of DIW or IBW through the capsule filter (50 mL through the disk 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 or top.)

c. *DH*: Remove tubing from the 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 the pump is operating, shake the capsule filter to help remove any entrained DIW.

d. *CH*: Detach capsule filter from the peristaltic pump tubing, place the filter 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 groundwater 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." Groundwater 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 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 groundwater samples.

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

1. Field rinse peristaltic pump tubing with sample water and without the filter attached.

   a. *Clean Hands (CH)*: Rinse the outside of the intake 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 the filling cap (or churn funnel) and reseal the plastic bag around the tubing.

      - The intake should be immersed in the composite vessel sufficiently so that air is not drawn through the tubing while pumping. **TIP**: A diagonal-cut end of the tubing will help prevent the tubing from attaching to the side of the sample container (churn) during pumping/filtering.

      - Do not allow the pump tubing to rest on the bottom of the composite vessel, to avoid drawing potentially sediment-laden water to the filter.

c. *Dirty Hands (DH)*: Start peristaltic pump to slowly pump sufficient sample to completely fill pump tubing.

d. *CH*: Direct rinse water to 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 the peristaltic pump after tubing is field rinsed.

2. Field rinse capsule filter. (Use the same general procedure if using the disposable disk filter, referring to table 5.2.1.A.1 for volume of rinsing and conditioning fluids.):

a. *CH*: Remove rinsed filter from the plastic bag and attach discharge end of the peristaltic pump tubing to the inlet connector on the capsule filter. If the optional hose barb adapter is used, attach it to the inlet of the capsule filter, then connect the pump tubing to the hose barb adapter. If an optional vacuum tube (FCCVT) container will be used for the nutrient sample, attach a second hose barb adapter to the outlet of the capsule filter.

b. Check that the direction of sample flow through the filter matches the direction of the arrow on the filter housing.

c. *DH*: Operating the pump at low speed, pump sample through the tubing to the filter.

d. *CH*: Invert the filter (arrow on filter housing is pointing up) so that the sample in the filter housing forces out any trapped air. **Prevent sample spray on the inside chamber walls and top.**

- The chamber cover must be replaced if water was sprayed on the chamber walls while preparing the filter. Replacing the cover is necessary in order to prevent droplets at the top or wall of the chamber from dripping into an open sample bottle.
- If some water that sprayed onto the chamber cover has dripped into the sample bottle, discard the bottle and fill a fresh bottle after replacing the wet chamber interior.

e. *DH*: Stop the peristaltic pump as soon as the filter housing is full of sample and all air has been expelled.

TECHNICAL NOTE 4. 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.

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3. Collect sample filtrate to fill an FA bottle for inorganic trace elements and major cations.

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

- *DH*: Check that the intake tube is properly inserted in the sample and start the pump.
- *CH*: Condition the filter and the sample bottle with the water to be sampled as it discharges through the filter. Use the volume from table 5.2.1.A.1 but **do not exceed this volume**.
- *CH*: Field rinse a prerinsed 250-mL FA bottle for trace elements and cations with 25 mL of capsule filter sample filtrate; cap, shake vigorously, and discard rinse water into an appropriate receptacle. If a 125-mL FA bottle is used, rinse with 50 mL of sample filtrate. If the disk filter is used, rinse the filter and condition the bottle with the volumes on table 5.2.1.A.1.

- ***Remember***: If droplets from water that was used when rinsing the filter were sprayed onto the chamber cover and dripped into the sample bottle, discard the bottle, change the chamber cover, and rinse and fill a fresh bottle.
- *DH*: Stop the pump in time to prevent losing filtrate to waste.
- *CH*: Cap the bottle, shake vigorously, and then discard rinse water into an appropriate receptacle.

b. *DH*: Start the pump and resume flow from the pump to the filter, taking care not to exceed 300 mL per minute.

c. *CH*: **Filter only the next 200 mL of the sample** trace-element and major cation FA bottle (fill to near the bottom of the upper lip of a standard 250-mL polyethylene bottle) or **filter only the next 125 mL of the sample** into the trace-element/major cation FA bottle (fill to the neck of a standard 125-mL polyethylene bottle). Do not allow the tip of the filter to contact the rim of the FA bottle.

d. *DH*: Stop the pump after the FA bottle is filled.

e. If a filtered mercury sample is required, restart the pump, then field rinse and fill an FAM bottle using the same procedure as for the 250-mL FA bottle. The minimum sample volume for the FAM bottle is 250 mL and the rinse volume is 25 mL.

f. *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 samples. Do not rinse the vacuum tube, DOC bottle, or any other prebaked glass sample bottles.**

g. *Follow the directions below if the optional vacuum tube container will be used instead of the FCC bottle. Otherwise skip to step h. Note: Do not use vacuum tubes beyond the expiration date. Doing so may not provide sufficient sample volume for analysis. Vacuum tubes are delivered sterile and should not be field rinsed.*

   – *CH*: Insert the narrow end of the piercing collar into the outlet hose barb adapter.

   – *DH*: Start the pump. Allow 8-10 mL of sample to flow through the piercing collar, then stop the pump.

   – *CH*: Insert the septum end of the vacuum tube into the piercing collar. Allow the tube to fill, then remove it when water ceases to enter the tube. DO NOT PUMP WATER INTO THE VACUUM TUBE. Repeat this step for the second tube. Remove the tube and discard the piercing collar. Processing of the nutrient sample with the vacuum tubes is complete.

h. Fill remaining bottles for the analysis dictated by the project sampling and analysis plan in the order below. If the vacuum tubes were used to process the nutrient sample, skip to the next appropriate container. If a DOC sample is collected, pass it through the filter last. Cap each container immediately after filling.

   1. Nutrients.
   2. Major anions (including alkalinity).
   3. Radiochemicals.

4. Stable isotopes and dissolved inorganic carbon (DIC), and ultraviolet absorbing substances (UAS).
5. Dissolved organic carbon (DOC).

To filter a pumped sample (generally applicable to groundwater):

Groundwater 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 or disk filter assembly is connected in line with the sample tubing in order to collect samples directly from the well.

► When sampling groundwater, DH should check that the turbidity values recorded at the end of well purging are 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 constant, uniform flow rate of about 500 mL per minute.** Do not stop the pump or divert flow from the capsule filter or disk filter assembly during bottle field rinse or filtration, if possible.

TECHNICAL NOTE 5. 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 airlock 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 or disk filter with sample water:
   a. *CH*: Ensure that the sample line and manifold are full of sample and free of air bubbles, then attach the discharge end of the sample line to the inlet connector on the filter housing. If the optional hose barb adapter will be used, attach it to the inlet of the capsule filter, then connect the pump tubing to the hose barb adapter. If the optional vacuum tube (FCCVT) will be used for the nutrient sample, attach a second hose barb adapter to the outlet of the filter housing.
      - Practice the technique for attaching the filter to the tubing carrying flowing water so that water does not spray onto the chamber walls or top.
      - Check that the direction of flow matches the direction of the arrow on the filter housing.
   b. *DH*: The sample flow rate through the sample line to the filter should be about 500 mL per minute.
   c. *CH*: Invert the filter (arrow on the housing is pointing up) so that the flow of sample forces trapped air out while filling the filter housing.
      - Do not allow water to spray onto the inside chamber walls or top.
      - The filter housing should be full of sample with no air inside.
      - Field rinse the filter with the water to be sampled as it discharges through the filter. Use the volume from table 5.2.1.A.1.
2. Field rinse and fill bottles for inorganic-constituent filtered samples with sample filtrate (section 5.0.3) using bottles that previously were rinsed three times with DIW. Determine whether the potential clogging of pores in the filter medium is of concern for the samples (see TECHNICAL NOTE 6 below).

Always prevent the tip of the filter housing from touching the bottle rim. When filling the sample bottle, hold it at an approximately 30-degree angle to allow the filtrate to flow down the inside wall of the bottle (this technique minimizes sample aeration).

a. CH: Field rinse a 250-mL FA bottle for trace elements and cations with 25 mL of sample filtrate; cap, shake vigorously, and discard rinse water into an appropriate receptacle. If a 125-mL FA bottle is used, rinse with 50 mL of sample filtrate. If the disk filter is used, rinse the filter and the condition bottle with the volumes on table 5.2.1.A.1.

b. CH: Fill a 250-mL FA bottle for trace elements and cations with about 200 mL of sample filtrate (near the bottom of the upper lip of the 250-mL bottle) or a 125-mL FA bottle with 125 mL of filtrate. Cap the bottle and set aside for chemical treatment.

c. CH: If a filtered mercury sample is required, field rinse and fill an FAM bottle using the same procedure as for the 250-mL FA bottle. The minimum sample volume for the FAM bottle is 250 mL and the rinse volume is 25 mL.

d. CH: Field rinse remaining bottles with no more than an additional 100 mL of sample filtrate. **Exception: Do not rinse the vacuum tube, DOC, or any other prebaked glass sample bottles.**

TECHNICAL NOTE 6. Depending on the sample turbidity and composition, the nominal pore size of the filter media tends to decrease as the volume of sample passed through the filter increases because pores are clogged due to sediment loading or mineral precipitation on the filter (Horowitz and others, 1994). Groundwater with turbidity ≤5 NTU should not affect filter pore size appreciably. Use rinsed bottles and fill them one after the other. For groundwater sampling, do not stop the pump during the field-rinse and sampling process.

e. *Follow the directions below if the optional vacuum tube container (FCCVT) will be used. Otherwise skip to step f. Note: Do not use vacuum tubes beyond the expiration date. Doing so may not deliver sufficient sample volume. Reduce the pump speed to the lowest rate possible. Vacuum tubes are delivered sterile and should not be field rinsed.*

   – *CH*: Insert the narrow end of the piercing collar into the hose barb adapter.

   – *DH*: Allow 8–10 mL of sample to flow through the piercing collar.

   – *CH*: With the tube upright, insert the septum end into the piercing collar. Remove the tube when water is about one-fourth inch from the bottom of the tube cap. Repeat this step for the second tube. DO NOT CHANGE THE PUMP SPEED until all tubes are filled. Remove the tube and discard the piercing collar. Processing of the nutrient sample with the vacuum tubes is complete.

f. *CH*: Fill remaining bottles for the analysis dictated by the project sampling and analysis plan in the order below. If the vacuum tubes were used to process the nutrient sample, skip to the next appropriate container. If a DOC sample is collected using the capsule or disk filter, pass it through the filter last. The flow rate should be slow enough to avoid sample from splashing out of the bottle. Cap each container immediately after filling.

   1. Nutrients.

   2. Major anions (including alkalinity).

3. Radiochemicals.
4. Stable isotopes and dissolved inorganic carbon (DIC), and ultraviolet absorbing substances (UAS).
5. Dissolved organic carbon (DOC).

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 the 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 and the optional piercing collar if one was used—do not reuse. The optional hose barb adapter may be removed and discarded or it may be reused after cleaning.
5. Document the filtration procedures used and lot numbers for filters, vacuum tubes, and preservatives on field forms and in field notes.

References

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