

Sterile Field Sampling Procedures for Aqueous Samples Requiring Analysis for Perchlorate

The following guidance is designed for use by field personnel collecting samples for Perchlorate in drinking, surface or ground waters for analysis by EPA Method 314.0, 314.1, 331, 6850 or 6860. The procedures are derived from EPA Method 314.1: *Determination of Perchlorate in Drinking Water Using Inline Column Concentration/Matrix Elimination Ion Chromatography with Suppressed Conductivity Detection (May 2005)*, and The DOD Perchlorate Handbook, Revision 1, Change 1 (*August 2007*), and are meant to be used as a reference. They do not address all possible conditions that may arise during sampling.

Prior to sampling, request the appropriate sterile filtering kits from the laboratory. Each kit will contain one syringe one sterile container, one non-sterile container and two sterile filters in a resealable bag. Standard sampling containers should not be used for these analyses.

Please review all 3 pages of this guidance document before sampling. You will be collecting a bulk water sample which will then be filtered using the procedure noted in the sample filtering section.

Sample Containers and Filters:

Non-Sterile Sampling Container – 125mL wide mouth polyethylene bottle

Sterile Sampling Container – 120mL sterile specimen container

Sterile Sample Filters – 26mm sterile, 0.2µm filter, individually packaged

Sterile Syringe – 60mL, sterile Luer lock disposable syringe, individually packaged

Sample Collection

Wear clean nitrile or latex gloves at all times when collecting and filtering samples for perchlorate. When handling the sterile syringe, do not touch the lower half of the syringe barrel, the sides of the plunger or the Luer connection and when handling the sterile filter, do not touch the Luer connection or the exit tip, handle only by the edges. Additionally, do not touch the lip or threads of the sterile sample container or the inside of the lid.

When sampling from a cold water tap, open the tap and allow the system to flush until the water temperature has stabilized (~3 to 5 minutes). Collect a representative sample from the flowing stream using the non-sterile container. Rinse the container three times with the sample water prior to collecting the final aliquot. When sampling from an open body of water, fill the non-sterile container with water sampled from a representative area. Rinse the container three times with the water prior to collecting the final aliquot. In either case, fill the container approximately $\frac{3}{4}$'s full.

In all cases, the bulk sample should be sterile filtered in the field following the procedures noted below. If samples cannot be field filtered, they must be delivered to the laboratory as soon as possible to allow for sterile filtering in the laboratory. In this case, samples should be collected, with headspace, in sterile sample containers (do not rinse the sterile container prior to collection), stored on ice at 4°C, and be delivered to the laboratory within 48 hours of collection. Note on the COC that the samples require sterile filtering in the laboratory.

Sample Filtering

Once representative samples are obtained in the field, they must be sterile filtered as soon as possible to remove any native microorganisms. Perchlorate is known to be susceptible to microbiological degradation by anaerobic bacteria. Samples are sterile filtered to remove microbes and stored with headspace to reduce the potential for anaerobic degradation.

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Depending upon the volume of sterile sample required, (usually 40 – 50mL is sufficient) multiple sets of sterile syringes and filters may be needed per sample. Each sterile syringe/filter set is good for approximately 50-60mL of filtered sample. If more than 60mL of volume is needed then a new sterile syringe and sterile filter must be used for each additional 60mL aliquot. All sterile filtered aliquots for an individual sample are to be collected in the same sterile sample container.

Remove a sterile syringe from its package and place the tip into the sample. Do not touch the tip or bottom of the syringe barrel. Draw up the sample until the syringe is basically full (50~60mL). Once full, turn the syringe upright. Grasp a 0.2µm sterile syringe filter package by the sides of the plastic, essentially squeezing the edges of the filter, and remove the paper cover. Do not touch the filter. While the syringe is upright, place the luer connection of the filter onto the luer connection of the syringe and twist until tight (about a ½ turn). Remove the plastic cover from the filter and turn the syringe downward.

Depress the syringe plunger gently, discarding the first 3-5mL of sample. Open a sterile sample container without touching the interior, lip, threads or inside of the lid. Using gentle pressure, pass the sample through the filter into the sample container at a rate of 2-3 drops per second. This process may take several minutes. Do not force the sample through the syringe as this may rupture the filter and any particulates or microbes trapped on the filter will be passed back into the sample. In the event that there is particulate matter in the sample, passing all of the volume through the filter may be difficult and the filter may become clogged. In this case, turn the syringe upright and remove the clogged filter. Using the same procedure as before, attach a new sterile filter to the syringe, turn the syringe downwards and discard an additional 3-5mL of sample as previously done. Continue with sample filtering. Do not let the syringe or filter make contact with the sterile sample container.

If additional sample volume is required, a new syringe and filter assembly must be used. Do not return the first syringe to the sample container to collect another aliquot. After sufficient sample volume (approximately 40 - 100mL depending on project QC requirements), has been filtered, and leaving some headspace, quickly and tightly seal the container. Label the container, place inside the resealable bag that the filter kit was in, seal, and place in a cooler on ice. Complete any related field or Chain of Custody documentation. Syringes and filters are single use items and must be discarded after each sample aliquot.

Sterile Filter Blanks: Although not required, a sterile filter blank (for each lot of syringes and filters) should be collected during sampling to evaluate whether the sterile syringe/filter set is a source of potential contamination. The sterile filter blank is collected in the field by passing reagent grade field water through a new syringe/filter setup, the same as that used for the samples. The filter blank is processed in the same manner, at the same time and general location as the samples.

Sample Shipment and Storage

Field samples must be chilled on ice during shipment and should be at 0°C - 6°C when they are received at the laboratory. Field samples stored in the lab must be held at or below 6°C until analysis, but should not be frozen.

Sample Holding Times

Samples that are stored and collected in the manner described in this guidance document may be held for a maximum of 28 days before analysis.

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Equipment:



Sterile Container



Non-Sterile Container



Sterile Filters

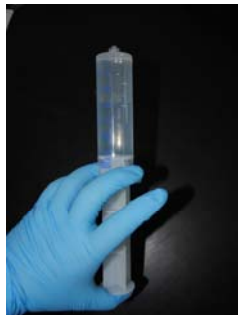


60mL Sterile Syringe

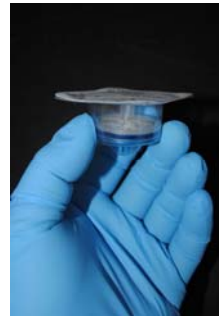
Procedure:



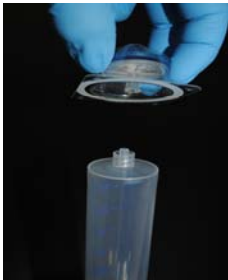
Completely fill syringe from non-sterile container



Once full, turn upright



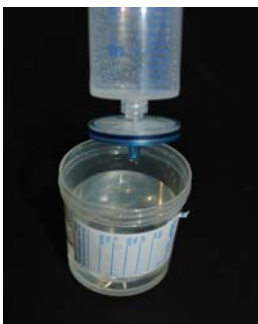
Grasp edges of filter cover, securing the filter between fingers, remove seal



Attach filter to syringe, twist until 'tight', about 1/2 turn, remove filter cover and discard



Turn syringe downwards, depress plunger gently, discarding the first 3-5mL



Gently (2-3 drops/second) push sample through filter and into sterile container



Cap sterile container, label, bag and place in cooler on ice
 Complete COC and all related field documentation

