HOC Sampling Media Preparation and Handling; XAD-2 Resin and GF/F Filters

Eric Crecelius and Lisa Lefkovitz
Pacific Northwest National Laboratory
Battelle Marine Sciences Laboratory
1529 West Sequim Bay Road
Sequim, WA 98382

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1.0 Scope and Application

This method is applicable to the preparation of sampling media used in the collection of hydrophobic organic compounds (HOCs) from water.

The dissolved HOC phase is collected on XAD-2 resin, a macroreticular resin bead that selectively scavenges HOC from other media such as water and/or air. The manufacturing process of this material results in very dirty product and a very rigorous clean-up procedure is needed to remove these potential interferences. Also, care needs to be taken when handling the resin to avoid damage of the beads which could lead to reintroduction of the original contaminants possibly bound into the beads.

Glass fiber filters are used to filter out the **?**particulate" fraction of the water. Since HOCs are preferentially bound to particulates in these media, this material needs to be isolated to determine the particulate-bound fraction of HOCs present. Again, special cleaning and handling procedures are required to obtain filters clean enough for trace level HOC analyses.

2.0 Definitions

HOC	Hydrophobic organic contaminants
GF/F	Glass fiber filter
XAD-2	Manufacturers name for a class of polymeric resin beads used to isolate HOCs from water.
LRB	Laboratory Record Book

3.0 Responsible Staff

Laboratory Supervisor. A Technical Specialist or Scientist having expertise in the principles involved with this procedure and in the use of laboratory operations in general. Responsible for ensuring that analysts are trained in the use of the instrument and that maintenance logs are being completed.

Analyst. A Technician, Technical Specialist, or Scientist assigned to utilize the instrument for actual sample analysis using this procedure. Responsible for 1) understanding the proper use of tools and solvents; 2) recording information regarding maintenance of the instrument in the appropriate logbooks; 3) reporting any significant problems with the instrument to the Laboratory Supervisor; and 4) tabulating and reporting sample data to the Laboratory Supervisor.

4.0 Procedure

4.1 XAD-2 Resin

XAD-2 resin can be obtained from a number of different vendors but is manufactured solely by Rohm and Haas. The size of the resin beads is 20-60 mesh. A rigorous clean-up procedure must be applied prior to use of the resin for collection of HOCs.

4.1.1 Apparatus and Reagents

Methylene Chloride, Acetone, Hexane, Methanol; HPLC grade or better
Glass wool/soxhlet extracted in hexane/acetone (50:50)
Amberlite XAD-2 Resin, 20-60 mesh. Rohm and Haas manufacturer

4.1.2 Resin Clean-up Method

The XAD-2 resin is cleaned in the lab by a series of solvent extractions in a large soxhlet apparatus (or in multiple set-ups). The resin is extracted sequentially for 24 hours each in methanol, acetone, hexane and methylene chloride. This is followed by sequential 4-hour extractions in hexane, acetone and methanol which cycles the resin back to a polar solvent. The methanol is then displaced from the resin by numerous rinses with organic-free water. The resin can be stored at this point in clean jars immersed in the water in a dark place for up to three months. The final four-hour hexane extract may be used for a laboratory XAD-2 blank. The last methanol rinse may be used as the starter methanol on the next XAD-2 batch.

4.1.3 QC of Resin/Is it Clean?

A portion of the resin from each clean-up batch must be tested to ensure a thorough clean-up has been performed. As noted above, the final four-hour hexane extract may be used for a laboratory XAD-2 blank. Alternatively, a representative amount of pre-cleaned resin from a given clean-up batch may be extracted using the extraction scheme to be used for the project of interest and the extract analyzed as a resin blank. The cleanliness of the resin will be evaluated on a project specific basis.

4.1.4 XAD-2 Resin Column Preparation

XAD-2 resin must be packed into a column for use as a sampling media for dissolved phase HOCs. The resin columns may be glass, stainless steel or teflon and can vary in size. This procedure is specific to glass columns with dimensions of 300 mm x 50 mm, fitted with nylon end plugs sealed with viton O-rings.

XAD-2 resin columns are prepared by first attaching one teflon adaptor with a swagelock fitting and a 3 inch length of latex tubing to one end of the glass column, and pushing a large plug of cleaned glass wool into the bottom. The column is filled about ½ full with organic free water and clean resin is poured into the column in a slurry to a final packed length of ~19.5 cm (~400 cc). The resin is packed by pumping excess water out from the bottom using a water aspirator peristaltic pump but always maintaining enough water in

the colimn to cover the resin. The column should not contain air bubbles or channels. Glass wool is added at the top to take up the space between the XAD-2 and the column threads. A solid nylon end cap with O-ring is placed on the top and then, after inverting the column and unscrewing the adaptor, the other end is capped in the same fashion.

4.1.5 Column Handling and Storage

Upon receipt of a cleaned batch of resin, the batch is named for the date of receipt and recorded in the project LRB. A copy of the chromatogram of the resulting XAD-2 resin blank that is determined for that batch is also included in the LRB. All columns are assigned individual numbers based on the resin batch number which is written in permanent marker on a piece of tape wrapped around the outside of the column. Columns are stored in a clean, cool place in the dark and can be stored up to 6 months prior to use. After sampling, columns should be stored at 4°C in the dark. There is no holding time for sampled resin columns prior to extraction.

4.2 Glass Fiber Filter

4.2.1 Apparatus and Reagents

Muffle Furnace
Al foil, heavy duty, extra wide
Whatman 293 mm GF/F 0.7µm nominal pore size glass fiber filters

4.2.2 Filter Clean-up Method

Filters are wrapped in a single layer of heavy duty aluminum foil which is sealed around the filter to create a "bag." The filter and aluminum foil are then ashed for four hours at 450°C (±20°C).

4.2.3 QC of Filter/Is it Clean?

One filter (or more, since more than a single filter may be used for a given sample) should be extracted using the extraction scheme to be used for the project of interest and the extract analyzed as a filter blank. The cleanliness of the filter will be evaluated on a project specific basis.

4.2.4 Filter Storage and Handling

Cleaned filters are stored inside of their foil bags in a clean, cool place prior to sampling. Multiple filter/ foil units can be stored in sealed polyethylene bags for storage and/or shipping. The bags containing cleaned filters from the same lot are labeled as the preparation date of filters, the initials of the technician who prepped them, the number of filters in the bag and the page number of the LRB where the preparation information is recorded.

After filters are used for sampling, they are to be folded in quarters (pie shaped) and placed in sealed ashed foil bags and stored frozen in plastic bags. There is no holding

time for storage of sampled filters prior to extraction.

4.3 Interferences

Take appropriate precautions to prevent contamination of any equipment associated with this analysis.

5.0 Data Analysis and Calculations

Not applicable.

6.0 Quality Control

6.1 Solvent Blanks. Use only HPLC grade or higher purity solvents for clean-up. Only a single lot number of each solvent should be used. A solvent blank test will be performed upon the start of a new lot number by concentrating a representative volume of solvent to 1 mL and analyzing on the appropriate analytical instrument. Cleanliness of the solvent will be determined on a project specific basis.

6.2 Resin Blank per batch. Resin used for a given project should be isolated to a single manufacturer's lot number since the original level of contamination of the resin can vary significantly with lot. Resin blanks will be analyzed per clean-up batch as specified in Section 4.1.3. Cleanliness of the resin will be determined for each new lot number on a project specific basis.

6.3 One Filter blank per batch. Filters used for a given project should be isolated to a single manufacturers lot number. Filter blanks will be analyzed per clean-up batch as specified in Section 4.2.3. Cleanliness of the filters will be determined on a project specific basis.

6.4 All results will be recorded in an LRB which is reviewed periodically by the laboratory supervisor and monthly by the project manager.

7.0 Safety

All analysts following this procedure should be aware of routine laboratory safety concerns, including the following:

7.1 Protective clothing and eyeglasses should be worn when appropriate.

7.2 Proper care must be exercised when processing samples because volatile and flammable solvents are involved.

8.0 Training Requirements

All staff preparing sampling media described above must first read this SOP and then demonstrate proficiency in the process prior to performing the work under the supervision of the laboratory manager.

9.0 References

MSL-A-006. Marine Sciences Laboratory Training.