SOP for cleaning polyurethane foam (PUF) sampling media using the Dionex ASE 350 Accelerated Solvent Extraction System

Last Revised October 20th, 2015

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Purpose

This is the standard operating procedure used to clean and prepare polyurethane foam (PUF) sampling disks for storage, deployment in the field, and use as laboratory method blanks using the Dionex ASE 350 Accelerated Solvent Extraction System. This is essentially a pre-extraction/removal of PCBs and other organic contaminants that are present on the PUF as they arrive from the factory. This process requires about 1 hour of preparation, 6.5 hours for the ASE to run, and 1.5 hours to dry and wrap the PUF after they have run through the ASE. This process needs to be started and completed in the same day.

Materials and Laboratory instruments

- Dionex ASE 350 Accelerated Solvent Extraction System
- Polyurethane foam disks
- Methanol, Pesticide Grade, A450-4, Fisher Scientific
- Hexanes, Pesticide Grade, H300-4, Fisher Scientific
- Acetone, Pesticide Grade, A40-4, Fisher Scientific
- ASE collection jars
- ASE jar septa and caps
- Large oversized tweezers
- ASE sampling cells and caps
- ASE glass fiber filters
- ASE cell plunger
- Heavy duty Aluminum foil
- One quart Ziploc bags

Cleaning the PUF

Loading the ASE cells

Before beginning, make sure there is enough combusted aluminum foil to wrap each clean PUF in after it has run on the ASE 350. If you did not do this the night before, you can do this right before beginning the ASE run. The foil needs to combust for minimum 6 hours, which is slightly less time than the ASE will need to run 19 cells.

Load 3 PUF into each ASE cell. Do not forget to place a glass fiber filter in the bottom of each cell. At this point, it is ok to use your hands (wearing nitrile gloves) to handle the PUF. However, you may find it helpful to use a pair of oversized tweezers. After all three PUF are in the cell, put on the top ASE cap.

Load the ASE cells into the ASE 350 along with as many ASE collection bottles. Since the extracts will not be saved, there is no need to triple rinse the ASE cells, collection jars, or septa caps prior to use. The extracts will be discarded.

Operating the ASE 350

Turn on the ASE 350 and open the Nitrogen gas tank (fully open the regulator) that feeds nitrogen to the ASE.

Under load method/schedule, select sequence 2, starting at line 1. Sequence 2 does not include a rinse between samples, so it is used only for sampling media cleaning. Make sure that the acetone and hexane bottles are each full before starting the cleaning run. The run parameters for Sequence 2 are as follows:

- Temperature: 100 C
- Heat Time: 5 min
- Statid Time: 5 min
- Rinse Volume: 60%
- Purge time: 200 Sec

> Note: Allow approximately 20 min for each ASE cell to run. To run 19 ASE cells, will take approximately 6.5 hrs. So start this process early on in the morning if you intend to clean a lot of PUF, and give yourself adequate time in the afternoon (~1.5 hrs) to allow for drying and wrapping the PUF in foil.

Drying the PUF

When the ASE run has finished, lay out a large piece of clean aluminum foil in the fume hood. This will be used for drying the PUF. Make sure the fume hood is completely free of chemicals before doing this.

Remove the caps from both ends of an ASE cell. With a triple rinsed ASE cell plunger, gently force out the PUF onto the clean piece of foil laid out in the fume hood. The puff will “pop” out if you do this too fast, so be sure to do this carefully. The purge at the end of the ASE run removes the vast majority of solvent on the PUF, so drying takes on a few minutes. When the PUF are dry, they will be noticeably smaller than when they came out of the ASE cell and the edges will curve up slightly. Make sure that the PUF no longer smell of solvent before wrapping them in foil.

Note: At this point, set aside one PUF from the first cell, one from the middle cell, and one from the last cell. These 3 PUF will be set aside for quality control.

Wrapping the PUF in Aluminum Foil

Using the triple rinsed tweezers, place the dry PUF into a clean piece of combusted aluminum foil. The foil should be large enough to completely wrap around the PUF nearly two times. It is not enough to just make sure the edges overlap each other. Place the PUF near the center of the foil, so that you can take one edge of the foil and fold it over so that it comes to the opposite edge of the PUF. When you fold the other edge of the foil over, there should be some excess to wrap around the edge of the PUF. Fold the sides of the foil tight, but be careful not to depress the edges of the PUF disk. With the sides folded, tightly fold the top and bottom edges over two times, about half an inch each time, to seal the foil tight.

Finally, fold the top and bottom sides down to the edge of the PUF so that the PUF is wrapped in a square of foil. Pinch the edges tight while again being careful not to deform the edges of the PUF. Place each foil-wrapped PUF into a one quart Ziploc bag. Separate the PUF that were in the first and last ASE cells (6 PUF total) from the rest of the cleaned PUF batch, and clearly indicate their position in the cleaning run (first or last) on the Ziploc bag. These will be extracted as blanks and quantified as samples for QA/QC on the batch

Labeling

With a permanent marker, clearly label each PUF with the batch ID. This will be your initials and the date the PUF were cleaned. For example, PUF cleaned by Andrew Awad on October 18, 2016 would all bare the label AA10182016. This is a necessary part of our record keeping. Clearly label the 3 PUF that were set aside during the drying with “first”, “middle”, or “last”, depending on which cell they came from, and the words “do not deploy” – these will be set aside for QA/QC.

Storage

Store all cleaned PUF in the clean media freezer in the lab (SC 1246). These PUF are now ready to be sent into the filed for sampling and field blanks, and can be stored in the freezer until needed as laboratory blanks. Clearly document in your lab notebook the batch name, the number of PUF that you cleaned, and that the PUF were stored in the clean media fridge after cleaning.

Blank PUF Quality Control and Quality Assurance

As soon as possible, extract the clean PUF that were set aside for QA/QC as laboratory method blanks. It is most efficient to run these as the first three method blanks with the next batches of field samples to be extracted and analyzed. However, if there is a reason for concern, such as increasing mass levels in the lab blanks, it may be necessary to run these samples independently of field samples in order to identify and address any issues. The important thing is that we constantly monitor the blank levels and look for any irregularities and address them as soon as they arise. Good blanks should have total PCB masses close to or below 1 ng/PUF. Make sure to quantify the congeners in the reference too, so that you know how much mass the standards contribute from impurities.