National Human Exposure Assessment Survey (NHEXAS)

Arizona Study

Quality Systems and Implementation Plan for Human Exposure Assessment

The University of Arizona
Tucson, Arizona 85721

Cooperative Agreement CR 821560

Standard Operating Procedure **SOP-BCO-L-2.0**

Title: Preparation of Filters and PUF for Field Collection of Metals and Pesticides in Air

Source: The University of Arizona

U.S. Environmental Protection Agency
Office of Research and Development
Human Exposure & Atmospheric Sciences Division
Human Exposure Research Branch

Preparation of Filters and PUF for Field Collection of Metals and Pesticides in Air

1.0 Purpose and Applicability

This standard operating procedure (SOP) describes the procedures for precleaning filters and polyurethane foam (PUF) prior to air sampling with these media. The sampling media are used for sampling indoor air, outdoor air, and personal air.

2.0 Definitions

PUF: polyether type of polyurethane foam used for collection of vapor phase organics.

3.0 References

3.1 R.G. Lewis and K.E. MacLeod; "A Portable Sampler for Pesticides and Semi-Volatile Industrial Organic Compounds," Anal. Chem., **54**; 310-315 (1982).

3.2 Interim Report by Battelle Memorial Institute, Columbus, Ohio, to U.S. EPA/R.G. Lewis on Cooperative Agreement CR 822082; "Transport and Migration of Lawn-Applied Pesticides into the Home: Spatial and Temporal Profile."

4.0 Discussion

This procedure provides cleaning methods to reduce potential background contamination of air sampling media. A Teflon-coated glass fiber filter (Pallflex T60A20) and PUF plug are used in tandem for air sampling pesticides; an all Teflon filter (Gelman Teflo; 0.2 um pore size) is used for air sampling for metals. The filters and PUF plugs used for pesticide sampling are pre-cleaned with acetone as this solvent is used for extraction of the pesticides from these matrices after air sampling. The filters used for sampling metals do not require precleaning.

5.0 Responsibilities

5.1 Battelle will be responsible for purchasing the filter and PUF material, and for cleaning these media.

5.2 Battelle will check for background contamination in the cleaned sample lots of filters and PUF plugs prior to shipment of the materials to UA. Representative PUF and filters will be extracted and analyzed per SOPs BCO-L-11.0 and BCO-L-15.0 to determine pesticide residue levels. Representative filters will be

digested and analyzed per SOPs BCO-L-3.0 and BCO-L-8.0 to determine metals residue levels.

5.3 Battelle will ship these materials to UA.

6.0 Materials and Reagents

6.1 Materials

6.1.1 Soxhlet extractors (small size, e.g. Kontes 585000-0021; and medium size e.g. Kontes 585000-0022) consisting of extractor, condenser, and 250 mL round-bottom flask.

6.1.2 Heating mantle.

6.1.3 Variac controller.

6.1.4 Tongs and filter tweezers

6.1.5 PUF plugs; open cell PUF; low density (R45 type) with density = 1.4 lb/ft^3 (0.022 g/cm^3); available pre-cut for air sampling from either University Research Glassware (Carrboro, NC) or Supelco, Inc; PUF plug size is 22 mm diameter x 76 mm length.

6.1.6 Teflon-coated glass fiber filters (Pallflex T60A20; 25 mm filters for personal and fixed site pesticide samples.

6.1.7 Vacuum desiccator (Ladd Research Industries)

6.1.8 Large Kim-Wipes (15" x 15").

6.1.9 Aluminum foil

6.1.10 Muffled wide-mouth glass jars.

6.1.11 Polyethylene Zip-lock bags, 4 mils thick (4"x 4" size and quart size).

6.1.12 Teflon tape (1/2" width).

6.1.13 Teflon filters; Gelman Teflo; 0.2 um pore size, 25 mm for personal metals samples and 0.2 um pore size, 37 mm for fixed site metals samples

6.1.14 Graduated cylinders

6.2 Reagents

6.2.1 High Purity dry N_2 tank (Battelle storeroom).

6.1.2 Boiling chips (Hengar crystals).

6.1.3 Acetone (high purity).

6.1.4 Hexane (high purity)

7.0 Procedure

7.1 Cleaning and Packaging Procedure for Pesticides Filter

7.1.1 Load 50 filters into a small Soxhlet extractor using acetone-rinsed tweezers or tongs. Make sure that the filters are below the height of the siphon tube.

7.1.2 Add the condenser. Connect the water lines of the condenser to the house water supply. Turn on the water and check that there are no leaks.

7.1.3 Add 150 mL of acetone to the 250 mL round-bottom flask and 3-4 boiling chips. Connect the flask to the extractor.

7.1.4 Add the heating mantle and Variac. Slowly turn up the heat with the Variac until the acetone boils smoothly and distills into the extractor. Extractor should fill and dump every 15-20 min. Watch one cycle and verify correct operation.

7.1.5 Extract filters for 14-16 h (overnight).

7.1.6 Turn off Variac and remove heating mantle. Allow extractor to cool about 15 min.

7.1.7 Turn off water. Remove condenser. Tilt extractor body and dump any residual solvent into the round-bottom flask

7.1.8 Unload filters from Sohxlet with acetone-rinsed tongs or tweezers onto a clean, hexane-rinsed square of aluminum foil in a laboratory hood; cover

immediately with a single layer of Kim-Wipe, and allow to dry undisturbed for about 10 min.

7.1.9 Place all filters in a pre-cleaned wide-mouth glass jar with Teflon-lined screw-cap lid, and label jar with lot number of the filter media and date extracted.

7.1.10 Wrap one layer of Teflon tape over joint between top and bottom parts of glass jar. Store at room temperature until material is shipped to UA.

7.2. Cleaning and Packaging Procedure for Pesticides PUF Plugs

7.2.1 Load 10 PUF plugs into a medium Sohxlet extractor and wedge them in against the walls of the extractor so that they are below the siphon tube. Set up 5 extractors for a batch of 50 PUF plugs.

7.2.2 Continue with set-up, extraction, and cool-down as described above in Steps 7.1.1-7.1.7, using 250 mL acetone in each 500 mL round-bottom flask.

7.2.3 Wipe inside of vacuum desiccator with acetone-moistened Kim-Wipe.

7.2.4 Unload PUF plugs from Soxhlet using acetone-rinsed tongs into the vacuum desiccator (40°C, 20" Hg vacuum, 10 mL/min of high purity dry N_2). Dry the PUF plugs in the desiccator for 1 hr.

7.2.5 Using acetone-rinsed tweezers, place each clean, dry PUF plug in a 4" x 4" polyethylene Zip-lock bag. Place 25 bags in a quart size Zip-lock bag and label with batch number. Store in a -70°C freezer until material is shipped to UA.

7.3 Cleaning Procedure for Metals Filters

7.3.1 Filters are used as received from the manufacturer. The lot number is clearly marked for tracking purposes.

7.4 Calculations

None.

7.5 Quality Control

7.5.1 Laboratory Blanks/Material Checks

7.5.1.1 After cleaning and drying 50 Pallflex filters for pesticides, remove one filter for analysis prior to shipment to the field. Extract and analyze per SOPs BCO-L-11.0 and BCO-L-15.0.

7.5.1.2 After cleaning and drying 50 PUF plugs for pesticides, remove one PUF for analysis prior to shipment to the field. Extract and analyze per SOPs BCO-L-11.0 and BCO-L-15.0.

7.5.1.3 Pesticide levels must be <0.01 μg/filter and <0.01 μg/PUF plug for shipment. If levels are greater than this, then the batch must be re-extracted and re-analyzed.

7.5.1.4 For every 50 filters for metals, remove one filter for analysis prior to shipment to the field. Digest and analyze per SOP BCO-L-3.0. Metals levels must be less than 0.01 μg/filter. If levels are greater than this, reanalyze the digestate and analyze two more filters. If two of the three samples are acceptable, use the batch; if two of the three are not acceptable, then discard the batch.

7.5.2 Field QC

7.5.2.1 For each batch of 50 PUF plugs (or filters), the following allocation will be used to assure necessary QA/QC samples.

40 PUF	indoor and outdoor air samples at 20 homes
5 PUF	personal air samples in 5 homes
2 PUF	field duplicates of indoor and outdoor air in 1 home
1 PUF	pre-shipment blank (materials suitability)
1 PUF	field blank
1 PUF	field spike

This allocation provides 10% overall QA and 5% field duplicate QA (2 out of 40) on fixed site (indoor and outdoor) samples. The handling of field blank and field spike samples is covered in SOP UA-F-14.0.

8.0 Records

8.1 The laboratory notebook assigned to NHEXAS for pesticides materials preparation will contain the record of all relevant information for the pesticide PUF plugs and filters.

(a) This record book will contain, for each batch extracted, the batch number, the date of extraction, the purchase order number for the PUF and the date received, the lot number for the filter media, and the lot number of the acetone used for extraction.

(b) This record book will also contain the pesticide levels in the pre-shipment blanks.

(c) This record book will be retained in the laboratory where these operations are performed and will be transferred to the office of the Battelle co-PI at the conclusion of the program.

8.2 The laboratory notebook assigned to NHEXAS for metals materials will contain the record of all relevant information for the filters used.

(a) This record book will contain, for each batch used, the batch number, the date of analysis, the lot number, and the lot number of reagents used for digestion.

(b) This record book will also contain the metals levels in the pre-shipment blanks.

(c) This record book will be retained in the laboratory where these operations are performed and will be transferred to the office of the Battelle co-PI at the conclusion of the program.