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Quality Systems and Implementation Plan for Human Exposure Assessment

The University of Arizona
Tucson, Arizona 85721

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Standard Operating Procedure **SOP-BCO-L-29.0**

Title: Preparation of Filters and XAD-2 for Field Collection of Pesticides and Polycyclic Aromatic Hydrocarbons (PAH) in Air

Source: The University of Arizona

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

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Preparation of Filters and XAD-2 for Field Collection of Pesticides and Polycyclic Aromatic Hydrocarbons (PAH) in Air

1.0 Purpose and Applicability

This standard operating procedure (SOP) describes procedures for pre-cleaning filters and XAD-2 prior to air sampling with these media. The sampling media are used for sampling indoor air and outdoor air.

2.0 Definitions

XAD-2: A polystyrene-divinylbenzene resin used for collection of vapor phase pesticides and PAH. XAD-2 is widely used for semi-volatile organic compound air sampling.

3.0 References

3.1 "Characterization of Polycyclic Aromatic Hydrocarbon Exposure Among Children of Low-Income Families from Inner Cities and Rural," First year report to U.S. Environmental Protection Agency/NERL on Cooperative Agreement CR 822073 from Battelle Memorial Institute, Columbus, Ohio; August 30, 1994.

3.2 "Transport and Migration of Lawn-Applied Pesticides into the Home: Spatial and Temporal Profile," Interim report to U.S. Environmental Protection Agency/ NERL on Cooperative Agreement CR 822082 from Battelle Memorial Institute, Columbus, Ohio; June 12, 1995.

4.0 Discussion

This procedure describes cleaning methods to reduce potential background contamination of air sampling media. A quartz fiber filter (Pallflex) and an XAD-2 (Supelco) backup trap are used in tandem for air sampling pesticides and PAH. Each filter is placed in an oven at 450°C and the XAD-2 resin is pre-cleaned with dichloromethane (DCM). DCM is the solvent that is used to extract the pesticides and PAH from the air matrices after sampling.

5.0 Responsibilities

5.1 Battelle will be responsible for purchasing the filters and XAD-2 resin, and for cleaning these media.

5.2 Battelle will check the background levels of pesticides and PAH in cleaned sets of filter/XAD-2 prior to shipment of these sampling modules to UA. For every 50

sets cleaned, one filter and one XAD-2 cartridge will be extracted individually and analyzed per SOPs BCO-L-15.1 and BCO-L-30.0 to determine background pesticides and PAH, respectively.

5.3 Battelle will pack the sampling modules and ship them to UA.

6.0 Materials and Reagents

6.1 Materials

6.1.1 Soxhlet extractors (small size, e.g. VWR 27613-022; and large size e.g. VWR 27613-088) including extractors, condensers, and round-bottom flasks.

6.1.2 Heating mantles.

6.1.3 Variac controllers.

6.1.4 Tongs and filter tweezers

6.1.5 XAD-2 resin (Supelco)

6.1.6 Quartz fiber filters (Pallflex)

6.1.7 Drying column (4" x 14")

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

6.1.9 Aluminum foil

6.1.10 Impactor discs (URG-2000-25K, 4 LPM, 10 μm cutpoint))

6.1.11 Teflon tape (1/2" wide).

6.1.12 Muffle oven (Blue M, Model CW7780F)

6.1.13 Petri dishes (4" diameter)

6.1.14 Filter holders (URG-2000-25AD)

6.1.15 Graduated cylinders

6.2 Reagents

6.2.1 High Purity dry N_2 cylinder (size 1A).

6.1.2 Boiling chips (Hengar crystals).

6.1.3 Dichloromethane (distilled-in-glass).

7.0 Procedure

7.1 Cleaning and Packaging Procedure for Filter/XAD-2 Sets

7.1.1 Place 100 g of pre-cleaned XAD-2 (Supelco) between two sections of glass wool in a Soxhlet extractor. Set up a second Soxhlet extractor in the hood.

7.1.2 Attach 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 800 mL of DCM to the 1000 mL round-bottom flask and 3-4 boiling chips. Connect the flask to the extractor.

7.1.4 Place the round-bottom flask in the heating mantle and set the Variac to position 65 to allow the DCM to boil smoothly and distill into the extractor. The Variac setting may vary slightly, depending on the heating mantle used in the extraction. The extractor should fill and dump approximately every 30 min. Watch one full cycle of operation and adjust if necessary.

7.1.5 Extract XAD-2 for at least 14-16 h (overnight).

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

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

7.1.8 Transfer XAD-2 (200 g) from the two Soxhlet extractors to a drying column and let the DCM drain from the column.

7.1.9 Attach the column to a cylinder (size 1A) of high purity nitrogen with Teflon tubing. The rate of nitrogen flow through the column is set so as

to agitate the bed gently to remove the residual DCM. Typically, an entire tank of nitrogen is needed to dry 200 g of XAD-2. The cleaned and dried XAD-2 is stored in a clean jar ready for packing.

7.1.10 Place quartz fiber filters in Petri dishes and empty glass cartridges in a muffle oven at 450°C for 12 h.

7.1.11 Pack each clean glass cartridge with approximately 10 g of cleanXAD-2 and place each filter in a filter holder behind a coded impactor disc. Attached the assembled filter holder to the packed XAD-2 glass cartridge and seal both ends with Teflon tape.

7.1.12 Wrap each packed sampler module first in aluminum foil, then in bubble wrap, for shipment to UA.

7.2 Calculations

None.

7.3 Quality Control

7.3.1 Laboratory Blanks/Material Checks

7.3.1.1 After cleaning, drying, and packing 50 filter/XAD-2, remove one filter/XAD-2 set for analysis of background pesticide and PAH levels prior to shipment to the field. Extract and analyze as per SOPs BCO-L-15.1 and BCO-L-30.0.

7.3.1.2 Individual target pesticide and PAH levels must be <0.05 μg per filter/XAD-2 set. If this level is exceeded, the entire batch must be re-extracted and re-analyzed.

7.3.2 Field QC

7.3.2.1 For each batch of 50 filter/XAD-2 modules, the following allocation will be used to assure the necessary fraction of QA/QC samples:

44	indoor and outdoor air samples at 22 homes
2	field duplicates of indoor and outdoor air at 1 home
2	pre-shipment blanks (one filter and one XAD-2)
1	field blank
1	field spike

This allocation provides 10% overall QA samples 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 designated for the project for the preparation of pesticides and PAH sampling materials will contain records of all relevant information for the pesticide/PAH XAD-2 modules and filters.

8.1.1 For each batch extracted, this record book will contain the batch number, the date of extraction, the date received, the lot number of the filter media, and the lot number of the DCM used for extraction.

8.1.2 The record book will also note the pesticide/PAH levels measured in the pre-shipment blanks.

8.1.3 The 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.