The Children's Total Exposure to Persistent Pesticides and Other Persistent Organic Pollutants (CTEPP) Study

Pre-cleaning Filters and XAD-2

Battelle
Columbus, OH 43201
Contract No. 68-D-99-011

Standard Operating Procedure **CTEPP-SOP-5.10**

Title: Pre-cleaning Filters and XAD-2

Source: Battelle

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

STANDARD OPERATING PROCEDURE (SOP) FOR PRE-CLEANING FILTERS AND XAD-2

Prepared by: _____ Date: _____

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1.0 Scope and Applicability

This standard operating procedure (SOP) describes the method 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 Summary of Method

This procedure provides cleaning methods to reduce potential background contamination of air sampling media. A quartz fiber filter (Pallflex) and an XAD-2 backup trap are used in tandem for air sampling of target analytes. The filters are placed in an oven at 450 °C and XAD-2 is pre-cleaned with dichloromethane (DCM).

3.0 Definition

XAD-2: XAD-2 resin is used for collection of vapor phase target analytes.

4.0 Cautions

Standard laboratory protective clothing, gloves, and eye covering is required.

5.0 Responsibilities

5.1 The project staff will be responsible for purchasing the filters and XAD-2 material, and for cleaning these media.

5.2 The project staff will check for background contamination in the cleaned sample lots of XAD-2/filters prior to shipment of the materials to the field. Representative sets of cleaned XAD-2/filters will be extracted and analyzed per CTEPP-SOP-5.12 and CTEPP-SOP-5.24 to determine residue levels of target analytes.

5.3 The project staff will pack the sampling modules and ship them to the field site.

5.4 The CTEPP Laboratory Team Leader (LTL) will oversee the cleaning operation of filters and XAD-2 resin and ensure that SOPs are followed by all project staff.

6.0 Apparatus and Materials

6.1 Materials

6.1.1 Soxhlet extractors (small size, e.g. Kontes 585000-0021; and large size e.g. Kontes 585000-0023) consisting of 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 (Supelco)

6.1.6 Quartz fiber filters (Pallflex)

6.1.7 Drying columns (4" x 14")

6.1.8 Large Kim wipes (15" x 15")

6.1.9 Aluminum foil

6.1.10 Muffle oven (Blue M, Model CW7780F)

6.1.11 Petri dishes (4" diameter)

6.1.12 Teflon tape (1/2" width)

6.1.13 Filter holders (URG-2000-25AD)

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

6.1.15 Graduated cylinders

6.1.16 Disposable latex gloves

6.1.17 Disposable cotton gloves

6.2 Reagents

6.2.1 High Purity dry N_2 tank (Battelle storeroom)

6.2.2 Boiling chips (Hengar crystals)

6.2.3 Dichloromethane (DCM); high purity

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. A total of six setups (100 g of XAD-2 per each setup) can be placed in the same 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 minutes. 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 minutes.

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 per each drying column) 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 450ºC for approximately 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. Place three barcoded labels on each packed cartridge. The barcoded labels will be generated following CTEPP-SOP-4.11.

7.2 Wrap each packed sampler module first in aluminum foil, then in bubble wrap, for shipment to the field.

8.0 Records

All records will be kept in a bound laboratory record book. The laboratory notebook designated for the project for the preparation of sampling materials will contain records of all relevant information for the XAD-2 and filters. For each batch extracted, this record book will contain the batch number, the date of extraction, the lot number of the filter media, and the lot number of the DCM used for extraction. The record book will be retained in the laboratory where these operations are performed until the conclusion of the study and will be archived in a secure room for three years after completion of the study.

9.0 Quality Control and Quality Assurance

9.1 Pre-shipment Blank Checks: After cleaning, drying, and packing filter/XAD-2 sets, remove one filter/XAD-2 set for analysis of background levels of target analytes prior to shipment to the field. Extract and analyze as per CTEPP-SOP-5.12 and CTEPP-SOP-5.24.

9.2 Field QA/QC Samples: For a typical batch of 55 filter/XAD-2 modules, the following allocation will be used to assure the necessary fraction of QA/QC samples:

50	indoor and outdoor air samples
1	field duplicates of indoor or outdoor air
1	pre-shipment blank (one filter/XAD-2)
1	field blank
1	laboratory method blank
1	laboratory fortified blank

This allocation provides 9.1% (5 out of 55) overall QA samples.

9.3 All records will be kept in a bound laboratory record book. All paper documents will be kept in the project folders. Electronic files will be stored in the sampling folders and archived on a CD ROM or a ZIP disc after the project is completed.

9.4 The laboratory record book will be reviewed and verified by the QA Officer, the LTL, and the Task Order Leader through internal field audits and quality control audits.

10.0 Reference

10.1 J. C. Chuang, C. Lyu, Y-L Chou, P. J. Callahan, M. Nishioka, K. Andrews, M. A. Pollard, L. Brackney, C. Hines, D. B. Davis, and R. Menton, "Evaluation and Application of Methods for Estimating Children's Exposure to Persistent Organic Pollutants in Multiple Media." EPA/600/R-98/164a, EPA/600/R-98/164b, and EPA/600/R-98/164c (Volume I, II, and III), 1999.