United States Environmental Protection Agency

Office of Research and Development

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

Extracting and Preparing Dust and Soil Samples for Analysis of Neutral Persistent Organic Pollutants

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

Standard Operating Procedure **CTEPP-SOP-5.14**

Title: Extracting and Preparing Dust and Soil Samples for Analysis of Neutral Persistent Organic Pollutants

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 EXTRACTING AND PREPARING DUST AND SOIL SAMPLES FOR ANALYSIS OF NEUTRAL PERSISTENT ORGANIC POLLUTANTS

Prepared by: ________________ Date: ________________

Reviewed by: _______________ Date: ________________

Approved by: _______________ Date: ________________

Approved by: _______________ Date: ________________

Approved by: _______________ Date: ________________

1.0 Scope and Applicability

This standard operating procedure (SOP) describes the method for extracting and preparing dust and soil samples for analysis of neutral persistent organic pollutants.

2.0 Summary of Method

The method for extracting and preparing either dust or soil sample for analysis of neutral persistent organic pollutants is summarized in this SOP. It covers the extraction and concentration of samples that are to be analyzed by gas chromatography/mass spectrometry (GC/MS).

3.0 Definition

3.1 Surrogate Recovery Standard (SRS): The compounds that are used for QA/QC purposes to assess the extraction and recovery efficiency obtained for individual samples. Known amounts of these compounds are spiked into the dust or soil prior to extraction. The SRSs are quantified at the time of analysis and their recoveries indicate the probable extraction and recovery efficiency for native analytes that are structurally similar. The SRSs are chosen to be as similar as possible to the native analytes of interest, but they must not interfere in the analysis.

3.2 Internal Standard (IS): The compounds that are added to sample extracts just prior to GC/MS analysis. The ratio of the detector signal of the native analyte to the detector signal of the corresponding IS is compared to ratios obtained for calibration curve solutions where the IS level remains fixed and the native analyte levels vary. The IS is used to correct for minor run-to-run differences in GC injection, chromatographic behavior, and MS ionization efficiency.

4.0 Cautions

Standard laboratory protective clothing and eye covering is required.

5.0 Responsibilities

5.1 The project staff who performs the sample extractions will be responsible for obtaining samples from the sample coordinator, entering relevant information in the extraction/preparation laboratory record books, and sending final extracts for analyses.

5.2 The CTEPP Laboratory Team Leader (LTL), the QA Officer or designee, and Task Order Leader (TOL) will oversee the sample extraction operation and ensure that SOPs are followed by all project staff.

6.0 Apparatus and Materials

6.1 Materials

6.1.1 Solid Phase Extraction (SPE) cartridges (Flurisil)

6.1.2 Heating mantles for 250 mL round-bottom flasks

6.1.3 Variac controllers

6.1.4 Clean glass wool

6.1.5 Analytical syringes

6.1.6 Wide-neck glass funnels (muffled)

6.1.7 Large Kim-wipes (15" x 15")

6.1.8 Latex gloves

6.1.9 1 dram glass vials with Teflon-lined screw caps; muffled

6.1.10 1.8 mL glass GC vials with Teflon-lined screw caps; muffled

6.1.11 Kuderna-Danish concentrators (large 24/40 3-ball Snyder condenser, 125 mL reservoir flask and 25 mL tube); (Kontes 570000)

6.1.12 Small 19/22 3-ball Snyder condensers

6.1.13 Disposable Pasteur glass pipettes (muffled and stored in clean glass jar)

6.1.14 Vortex mixer (American Scientific Products)

6.1.15 Graduated cylinders

6.1.16 Heated water bath

6.1.17 Ultrasonic bath

6.1.18 Clean sample vials

6.1.19 Quartz fiber filters (Pallflex)

6.1.20 Solid Phase Extraction (SPE) manifold (JT Baker or Supelco)

6.2 Reagents

6.2.1 Methanol (distilled-in-glass)

6.2.2 n-Hexane (distilled-in-glass)

6.2.3 Boiling chips (Hengar crystals)

6.2.4 Surrogate Recovery Standard Spiking Solution

6.2.5 Internal Standard Spiking Solution

6.2.6 Distilled, deionized water (DI water)

6.2.7 Diethyl ether (EE); distilled-in-glass

7.0 Procedure

7.1 Extraction and Concentration.

7.1.1 The dust samples are sieved and only the fine dust samples (<150 μm) are used for extraction. The visible small rocks are removed from the soil sample, and the rest of the soil sample is mixed with glass rod prior an aliquot is removed for extraction. To the extent possible, retrieve 10 to 12 samples from the same materials batch from the freezer and place each sample cartridge on the laboratory bench for 10 min to come to room temperature. Extract and analyze these samples as a batch.

7.1.2 Put on clean gloves.

7.1.3 Weight out 0.5 g of each dust or 1 to 2 g of each soil sample into a clean sample vial using a 2-place balance and record the weight in the laboratory record book. If 0.5 g of dust is not available, then weight out the amount that is available.

7.1.4 Spike 200 μL of the SRS spiking solution onto the dust or 50 μL of the SRS spiking solution onto the soil and allow the solvent to disperse before addition of the extraction solvent (~10 min.). *Note that the spiked levels may be changed and the exact spiked amounts will be recorded in the laboratory record book (LRB).*

7.1.5 Add 10 mL of 10% EE in hexane to the dust or soil, put on the cap and swirl to wet. Place up to 12 samples in a ultrasonic bath. Sonicate the samples for 15 min.

7.1.6 Transfer the sample extract to a KD evaporator tube through a clean, hexane-wetted, quartz fiber filter.

7.1.7 Repeat Steps 7.1.5 and 7.1.6.

7.1.8 Rinse the sample residue with ~5 mL of 10% EE in hexane and repeat Step 7.1.6.

7.1.9 Add 3-5 boiling chips to the KD tube/flask. Attach the large Snyder condenser to the flask. Concentrate the extract in a heated (from ~60°to ~90°C) water bath to ~10 mL.

7.1.10 Change to small Snyder condenser and concentrate the extract to 0.6-0.8 mL. Remove the KD assembly from the water bath and let it stand in the hood to cool.

7.1.11 Rinse down the insides of the flask and tube with hexane, to bring the volume to 1 mL. Vortex for ~3 s to mix. *Note that steps 7.1.12-7.1.15 are required for dust samples but not soil samples and the cleanup steps will be recorded in the LRB.*

7.1.12 Place SPE cartridges on the SPE manifold and condition each cartridge in sequence with 6 mL of 50% EE in hexane, followed by 100% of hexane. Close the valve stem on the manifold to prevent the cartridge from going dry between solvents.

7.1.13 Using a clean Pasteur pipette, transfer a sample extract to an SPE cartridge.

7.1.14 Elute the cartridge into a clean vial with 12 mL of 15% EE in hexane and 6 mL of DCM.

7.1.15 K-D concentrate the extract to 0.6 to 0.8 mL and rinse down the sides of the tube with hexane to bring the volume to 1 mL.

7.1.16 Spike the extract with 10 L of the Internal Standard spiking solution, and vortex for ~3 s to mix.

7.1.17 Transfer the extract, using a muffled disposable glass Pasteur pipette, to a clean 1.8 mL GC vial for GC/MS analysis. Label the sample vial with its respective sample ID.

7.2 Store the extract in a $\leq$-10°C freezer until GC/MS analysis.

8.0 Records

8.1 Records of the preparation of field samples, blanks, and matrix spikes will be retained in a LRB that is kept in the extraction laboratory. This LRB will record all sample preparation activities. These samples will be recorded in the LRB by field sample ID and the laboratory generated QA/QC sample will be assigned with a laboratory sample number (a unique number that combines the 5 digit LRB number-2 digit page number-2 digit line number). The date of extraction, the lot number of solvents used for extraction, and the spike level of the surrogate recovery standards and internal standards will be recorded in the LRB.

8.2 The LRB 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 A field blank, laboratory method blank, laboratory fortified blank, consist of a sample vial that will be extracted together with the field samples. The field blank analyses are performed to verify that minimal contamination occurs through sample handling during shipping and field operations. The laboratory method blank analyses are performed to verify that minimal contamination occurs through sample preparation. The laboratory fortified blank analyses are performed to verify the recoveries of analyte preparation procedures.

9.2 Field crews will be reminded to wear clean clothing and shoes, to remove all pesticide products from their residences that may contain the target analytes, and to refrain from using these materials during the field study. Field crews will also be reminded to obtain clean clothing after visiting a home where they know or suspect that these pesticides have been applied within the previous week. Cigarette smoking is not permitted during the field sampling. Field crews should store the samples in a clean environment away from any known combustion sources.

9.3 Surrogate recovery values of 50-150% in blanks, and actual samples will be deemed acceptable, and no correction to the data will be made. For recoveries less than 50% or greater than 150%, the data will be flagged. For recoveries greater than 130%, the concentration of the surrogate spiking solution will be checked against a calibration curve to determine whether inadvertent solvent loss has resulted in higher spike levels. If this has occurred, the surrogate spiking solution will be re-prepared and analyzed.

9.4 One laboratory method blank that is analyzed as a sample concurrently with a field sample set will be analyzed for typically every 50 samples processed. If significant target analyte levels (>0.1 µg) are found in the field blanks or laboratory blanks, the source of contamination must be identified and more laboratory blanks, together with additional field blanks, trip blanks, and storage blanks, will be analyzed.

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.