National Human Exposure Assessment Survey (NHEXAS)

Maryland Study

Quality Systems and Implementation Plan for Human Exposure Assessment

Emory University
Atlanta, GA 30322

Cooperative Agreement CR 822038

Standard Operating Procedure **NHX/SOP-L11**

Title: Extraction of Neutral Pesticides and PAHs from House Dust and Soil

Source: Harvard University/Johns Hopkins University

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

1. Title of Standard Operating Procedure

   Harvard University/Johns Hopkins University Standard Operating Procedures:
   L11 Extraction of Neutral Pesticides and PAHs from House Dust and Soil, Rev. 1.0

2. Overview and Purpose

   This SOP is a modification of SwRI SOP-01-17-01, "Extraction of Neutral Pesticides and PAHs from Housedust."

3. Discussion

   This SOP covers extraction of dust and soil samples at SwRI after they have been sieved and divided. For sieving and division procedures, see HSPH SOP L05 “Sieving and Division of Dust and Soil Samples.”

   This procedure is applicable to house dust samples taken using the HVS3 dust sampler for pesticide exposure monitoring, and to soil samples. This method is limited to target analytes in which efficient extraction from dust and soil has been demonstrated. Specific target analytes for each project will be specified by the client, the study protocol or the quality assurance narrative.

4. Personnel Responsibilities

   It is the responsibility of the Study Director (Project Manager) to assure that all steps described in this procedure are performed.

   It is the responsibility of the Extraction Lab Supervisor to assure that all the steps in this procedure are performed.

   It is the responsibility of study personnel to comply with all criteria described in this procedure.

5. Required Equipment and Reagents

   5.1 Equipment

   200 mL Soxhlet extractor with 500 mL flatbottom flask, condenser and heating mantle
   500 mL Kuderna-Danish (KD) concentrator with 15 mL graduated receiver,
   3-chamber Snyder column
   glass funnel
   10 mL glass pipet column
   1-cm diam. Teflon disk filters
   Teflon boiling chips, rinsed twice with hexane
   vials, 1 dr., solvent washed and oven dried with teflon lined caps
   glass wool
   volumetric pipettes, various volumes
   transfer pipettes, solvent washed and oven dried
   steam bath
   1-L glass graduated cylinder

Whatman #1 filter paper

5.2 Reagents

n-hexane, Fischer OPTIMA grade or equivalent
diethyl ether, Fischer OPTIMA grade or equivalent
acetone, Fischer OPTIMA grade or equivalent
dichloromethane, Fischer OPTIMA grade or equivalent
sodium sulfate, anhydrous, ACS reagent grade
p-Terphenyl-d_{14} stock solution
nitrogen, Zero grade or equivalent
Florisil, 200 mesh, activated Mg-Silicate, Sigma Chemical

5.3 Forms

computer with database software including the following tables as sub-tables of the main database:
Check-In
Extraction
SwRI Form 5: Extraction Laboratory Form (set up as a computerized database)

6. Procedure

6.1 Check-In

When dust and soil samples are received from the laboratory where they have been sieved and divided, a technician will log them in and check them against chain-of-custody forms according to HSPH SOP G04, SwRI SOP 01-01-01, or an equivalent system.

1. Upon receipt of samples, check sample ID numbers against chain-of-custody forms. Photocopy the chain-of custody forms. File the copies to be kept at SwRI. File the originals to go out with extracts to be analyzed for PAHs by EPA OHR-HERL.

2. Access the Sample Check-In Table (Table 1) on the computer database. Locate the ID numbers of the samples you have received and fill in the table. When Table 1 is completed for the samples received, back up the file.

Table 1 -- Check-In

Sample ID	Date Received	Initials of Technician	Storage Location

6.2 Equipment and Solution Preparation

6.2.1 Equipment Preparation

Prior to extraction and solvent preparation, all glassware, pipettes, vials and funnels will be solvent washed and oven dried according to section 4.0 of SWRI SOP 01-19-01.

Whatman #1 filter paper: Soxhlet pre-extract for 24 hours with 1:1 (v/v) dichloromethane:acetone. Dry the filters under zero nitrogen and store them in a clean glass container until used.

Teflon boiling chips: Place the boiling chips in a beaker. Use a Teflon squirt bottle of hexane to rinse the chips. Decant the hexane and repeat the process. Allow the chips to dry before using.

6.2.2 Solution Preparation

Extraction Solvent: The 6 % (v/v) diethyl ether in n-hexane extraction solvent is prepared by mixing 240 mL of diethyl ether, measured with a 1-L graduated cylinder, with 3760 mL of n-hexane.

A surrogate spiking solution is prepared to contain 40 ng/µL p-terphenyl-d14 in n-hexane. The portion of this solution to be used for an extraction will be left at room temperature for about 30 minutes before use.

Oven-dried and solvent (6 % diethylether in n-hexane) rinsed sodium sulfate:

6.3 Extraction Procedure

1. Check the database to find the mass of the pesticide/PAH fraction that was sent. Weigh approximately 2 g of the dust (or 30 g of soil) to the nearest 0.001 g. If the mass of the fraction sent is less than 2.0 g, use all of it. Enter your initials, the mass of each sample, and the date into the database (Table 2 -- Extraction). Back up the file.

Table 2 -- Extraction

Sample ID	Initials of Technician	Mass used for extraction	Extraction begun		Extraction completed		Storage location
			Date	Time	Date	Time	

2. Spike the 2 g (or 30 g) portion with 100 µL (i.e., 4 µg) of the surrogate solution and wrap it in pre-extracted Whatman #1 filter paper.

3. Add Teflon boiling chips to a 500 mL flatbottom flask and attach the flask to a 200 mL Soxhlet. Place the wrapped dust or soil portion in the Soxhlet and add 200 mL of 6% (v/v) diethyl ether in hexane through the top of the Soxhlet.

4. Fit the condenser to the top of the Soxhlet and adjust the heating mantle so that the Soxhlet siphons at a rate of 2-4 cycles/hour. Enter the time in the database and back it up.

5. Soxhlet extract the dust or soil for at least 16 hours. During the extraction period, inspect the Soxhlet from time to time to insure the siphon rate remains constant. When the extraction is complete, record the date and time in the database and back it up.

6. At the completion of the extraction period, turn the heat off and allow the extraction apparatus to cool down to the room temperature. Siphon the remaining solvent from the Soxhlet into the flask.

7. Pass the extract through a glass funnel containing oven dried and solvent-rinsed sodium sulfate and transfer it to a KD concentrator fitted with a graduated receiver with 2 times solvent rinsed teflon boiling chips and three chamber Snyder column.

8. Heat the KD apparatus on a steam bath and concentrate the extract to a volume of approximately 3 mL. Remove the apparatus from the heat and rinse the Snyder column with 2 mL n-hexane.

9. After the concentrator reaches room temperature, wipe any moisture condensate and then rinse each joint with 1 mL of n-hexane into a receiver. Disassemble the unit.

10. Quantitatively transfer the concentrated extract from the receiver to a 3 dr. vial.

11. If the initial mass of dust was at least 1.0 g, bring the final volume to 10 mL by rinsing the receiver with n-hexane. If the initial mass of dust was less than 1.0 g, bring the final volume to 5.0 mL and document in the Laboratory Notebook and database.

6.4 Cleanup Procedure

1. Plug a clean glass column (made from 10 mL serological pipet) at the narrow end with glass wool. Add 4 mL of Florisil to the column.

2. The Florisil column is preconditioned with approximately 10 mL of n-hexane.

3. 1.0 mL of the concentrated extract from section 6.3, step 9, is added to the column. Label the rest of the extract as reserve and save in a locked freezer under 4°C.

4. The extract is eluted with 20 mL of 10 % acetone (v/v) in n-hexane and collected in a 6 dr vial. The eluent is concentrated with warm water (40°C) and zero nitrogen flow to a final volume of 3.0 mL in n-hexane and quantitatively transferred to a 1 dr vial. The extract is concentrated further to a final volume of 2 mL in 6 % (v/v) diethylether in n-hexane for analysis.

5. This procedure results in an effective final volume of 20.0 mL per 2.0 g of house dust or soil. Label the extract with the project number, sample ID, date of extraction, and final volume.

6.5 Extract Log-In

1. Upon completion of the extraction process, the three extract fractions are logged-in to the extract freezer.

2. The amount of dust or soil extracted and the location of the extract are entered into the database. A flow chart of the extraction process is on file in the laboratory; any changes will be approved by the SwRI Project Manager or his designate.

3. The Project Coordinator, the Project Manager, the GC/MS Laboratory Supervisor, and the analyst are then notified by providing them with a printout of the appropriate section of the database.

6.6 Records

1. All analytical procedures are recorded in the computer database in accordance with client/contract requirements.

2. Upon completion of the extraction process, the extract is logged-in to the extract freezer according to SWRI SOP 01-01-01.

3. The amount of dust or soil extracted and the location of the extract is entered into the laboratory notebook. A flow chart of the extraction process is on file in the laboratory

4. The project manager, analytical manager, and analyst are then notified by providing them with a printout of the appropriate section of the database.

7. Quality Assurance Procedures

In addition to 10% blanks and 10% duplicates, p-terphenyl-d14 is added to all samples to monitor both GC retention time and MS output.

8. References

Harvard University/Johns Hopkins University Standard Operating Procedures:
G04 Chain-of-Custody and Sample Tracking
G05 Storage and Shipping of Samples
F04 Collection, Storage, and Shipment of House Dust Samples for Metal, Pesticide, and PAH Analysis
F05 Collection, Storage, and Shipment of Soil Samples for Metal, Pesticide, and PAH Analysis
L05 Sieving and Division of Dust and Soil Samples

SWRI SOP 01-01-01 Sample Log-In and Sample Custody
SWRI SOP 01-19-01 Log-In, Preparation and Shipment of Housedust Samples