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-L10**

Title: Extraction of Neutral Pesticides and PAHs from Air Sampling Media

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:
   L10 Extraction of Neutral Pesticides and PAHs from Air Sampling Media, Rev. 1.0

2. Overview and Purpose

   This SOP is a modification of SwRI SOP-01-17-03, "Extraction of Neutral Pesticides and PAHs from Exposure Monitoring Media."

3. Discussion

   This procedure is applicable to polyurethane foam (PUF) plugs and quartz micro-fiber filters used for pesticide and PAH exposure monitoring. It is limited to target analytes and media in which efficient extraction 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 project manager, the laboratory manager, and the analytical 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

   Soxhlet extractors, 50 mL, 100 mL, 200 mL and 500 mL with condensers
   flatbottom flasks, 500 mL, 1-L
   500 mL Kuderna-Danish (KD) concentrator with 18 mL graduated receiver,
   3-chamber Snyder column.
   glass funnel
   5-mL Glass cartridge
   1-cm dia. Teflon disk filters
   Teflon boiling chips
   vials, 1 dr., solvent washed and oven dried with teflon lined caps.
   glass wool
   volumetric pipettes, various volumes, calibrated.
   transfer pipettes, solvent washed and oven dried.
   steam bath
   1-L glass graduated cylinder

5.2 Reagents

n-hexane, Fischer OPTIMA grade or equivalent
diethyl ether, Fischer OPTIMA grade or equivalent
acetone, Fischer OPTIMA grade or equivalent
sodium sulfate, anhydrous, ACS reagent grade
p-terphenyl-d14 stock solution.
nitrogen, Zero grade or equivalent

6. Procedure

6.1 Preparation

6.1.1 Equipment Preparation

Prior to extraction and solvent preparation, all glassware, pipettes, vials and funnels will be solvent washed and oven dried according to SwRI SOP 01-04-01.

6.1.2 Solution Preparation

1. 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.

2. A surrogate spiking solution is prepared to contain 10 ng/µL p-terphenyl-d14 in n-hexane as described in SwRI SOP 01-17-02, Section 4.3. Precise surrogate spiking levels will be determined by the analytical manager.

6.2 Extraction Procedure

1. When PUF air sampling trains are received equipped with quartz micro-fiber filters, the PUF and quartz filter are extracted as a single sample.

2. The size of the Soxhlet extractor and the volume of 6 % v/v diethyl ether in n-hexane to be used varies with the sampling medium and will be determined by the extraction laboratory supervisor.
   Typical extractor size and solvent volume used for a low-volume PUF plug and filter are 100 mL Soxhlet, 150 mL solvent.

3. Teflon boiling chips are added to the flatbottom flask and the flask is attached to the appropriate Soxhlet. The sample is placed in the Soxhlet, surrogate is added, and an appropriate volume of 6 % diethyl ether in n-hexane is added through the top of the Soxhlet.

4. The condenser is fitted to the top of the Soxhlet and the heating mantle is adjusted so that the Soxhlet siphons at a rate of two to four times per minute. The siphoning rate may be greater for smaller Soxhlets.

5. The sample is Soxhlet extracted for a period of at least 16 hours. During the extraction period, the Soxhlet is inspected from time to time to insure the siphon rate remains constant. The total extraction time is recorded in the laboratory notebook.

6. At the completion of the extraction period, the remaining solvent in the Soxhlet is siphoned into the flask, the heat is removed and the extraction apparatus is allowed to come to room temperature.

7. The extract is then passed through a glass funnel containing oven dried sodium sulfate and transferred to a KD concentrator fitted with a graduated receiver and three chamber Snyder column.

8. The KD apparatus is heated on a steam bath and the extract is concentrated to a volume of approximately 15 mL.

9. The concentrated extract is concentrated under zero nitrogen in the receiver to a volume of approximately 5 mL. The extract is quantitatively transferred to a 1 dr. vial. The final volume of the extract depends upon the sample matrix and expected analyte concentration and will be determined by the analytical manager prior to extraction.

10. The extract final volume will be entered in the laboratory notebook.

6.3 Florisil Cleanup Procedure

1. The following procedure will be used after all extractions.

2. Place a clean Teflon disk filter at the 5 mL glass cartridge.

3. Add 1.0 g of Florisil to the column.

4. Precondition the Florisil column with approximately 5 mL of 10% v/v acetone in n-hexane.

5. Add 1.0 mL of the concentrated extract to the column.

6. Rinse the column with approximately 20 mL of 10% acetone (v/v) in n-hexane.

7. Collect a 20.0 mL volume of the eluent in a 3 dr. vial and concentrate it to final volume.

6.4 Records

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

2. An Extraction Report including ID numbers of samples, a flow chart of the extraction process, the amount of solvent used, the extract final volume, whether a cleanup procedure was employed, and the location of the extract is entered into the laboratory notebook.

3. Copies of the Extraction Report are provided for the Principal Investigator, HSPH Quality

Assurance Officer, project manager, analytical manager, and analyst.

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:
G05 Storage and Shipping of Samples
F02 Collection, Storage, and Shipment of Indoor and Outdoor Air Samples for Metal Analysis
L09 Preparation of Exposure Media (PUFs and Quartz Fiber Filters) for Air Samplers for Pesticide and PAH Collection

SwRI SOP 01-01-01 Sample Log-In and Sample Custody
SwRI SOP 01-04-01 Extraction Laboratory
SwRI SOP 01-17-02 Determination of Pesticides, Acid Herbicides, Phenols, and PAHs by GC/MS
SwRI SOP-01-17-03 Extraction of Neutral Pesticides and PAHs from Exposure Monitoring Media