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-L12**

Title: Extraction of Neutral Pesticides from Isopropanol Dermal Wipes

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:
   L12 Extraction of Neutral Pesticides from Isopropanol Dermal Wipes, Rev. 1.0

2. Overview and Purpose

   This SOP is a modification of SwRI SOP-10-17-08, "Extraction of Neutral Pesticides, Acid Herbicides, and Phenols from Isopropanol Wipes."

3. Discussion

   This procedure is applicable to hand and surface wipes consisting of SOF-WICK dressing sponges soaked with isopropanol. This procedure 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 and Supplies

   mechanical shaker
   Turbo-Vap Evaporator
   flatbottom flasks, 500 mL, 1-L
   Turbo-Vap Vessels, glass, 200 mL
   vials, 1 dr. and 6 dr., solvent washed and oven dried with teflon lined caps.
   volumetric pipettes, various volumes, calibrated.
   transfer pipettes, solvent washed and oven dried.

   5.2 Reagents

   n-Hexane, Fischer OPTIMA grade or equivalent
   diethyl ether, Fischer OPTIMA grade or equivalent
   sodium sulfate, anhydrous, ACS reagent grade
   p-Terphenyl-d14 stock solution
   2,4-DB, Analytical Standard Grade
   nitrogen, Zero grade or equivalent
   Carbitol, ACS Reagent Grade
   potassium hydroxide, ACS Reagent Grade
   diazald, ACS Reagent Grade

6. Procedure

6.1 Sample Storage and Preparation

Upon receipt from the field, wipe samples are logged in and checked against chain-of-custody forms according to SwRI SOP 01-01-01, or an equivalent system. Samples are stored at approximately 4°C pending extraction and are tracked according to SwRI SOP 01-01-01, or an equivalent system.

6.2 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.

6.3 Solution Preparation

1. 37 % w/v KOH solution is prepared by weighing 370 g of KOH in a 2-L glass beaker containing a magnetic stir bar. Deionized water is added to the beaker to bring it to a volume of approximately 900 mL. The solution is stirred and gently heated until the KOH is totally dissolved. The beaker is allowed to return to room temperature. The solution is then transferred to a 1-L graduated cylinder and diluted to a volume of 1.0 L. The solution is stored in a clean glass amber bottle with a teflon lined closure at room temperature.

2. 1:1 diethyl ether in n-hexane extraction solvent is prepared by mixing equal volumes of pesticides grade diethyl ether and n-hexane in a 4-L glass container.

3. Surrogate spiking solutions are prepared to contain 10 ng/µL p-terphenyl-d14 and 10 ng/µL 2,4-DB each in 10 % ether in n-hexane as described in SwRI SOP 01-17-02, Section 4.3.

6.4 Extraction Procedure

1. Terphenyl-d14 surrogate is spiked into each sample containing wipes to be analyzed for neutral pesticides.

2. If necessary, additional isopropanol is added to each sample container so that it contains excess isopropanol.

3. The sample container is then shaken for 30 min on a mechanical shaker.

4. The excess isopropanol is decanted into a 500 mL flatbottom flask.

5. A 50 mL portion of 1:1 diethyl ether in hexane is added to the container and it is shaken for an additional 1 min.

6. The ether/hexane extract is decanted and added to the isopropanol fraction.

7. The extraction is repeated with an additional 50 mL portion of ether/hexane.

8. The sponges are squeezed to remove any additional solvent and the sample container is rinsed with small portions of hexane. All solvent fractions are combined in the flask.

9. The dilute extract is transferred to a Turbo-Vap vessel and concentrated in the Turbo-Vap at a temperature of 32-40° C to a concentration of approximately 15 mL.

10. The extract is concentrated to a volume of 2 mL in 10 % ether/hexane.

11. Heavily soiled sample extracts may be submitted for additional cleanup according to Section 10 below.

12. When neutral pesticides are target analytes, approximately 1 dr. glass vial and labeled with the sample i.d., solvent and final volume.

6.5 Cleanup Procedure (Optional)

1. The following procedure is used when Florisil cleanup is deemed appropriate by the analytical manager.

2. A clean Teflon disk filter is placed at the 5 mL glass cartridge.

3. 1.0 g of Florisil is added to the column.

4. The Florisil column is preconditioned with approximately 5 mL of 10% v/v acetone in n-hexane.

5. 1.0 mL of the concentrated extract is added to the column.

6. The column is rinsed with approximately 20 mL of 10% acetone (v/v) in n-hexane.

7. A 20.0 mL volume of the eluent is collected in a 3 dr. vial and concentrated to a final volume of 1.0 mL in 10 % ether in n-hexane for analysis. A larger final volume may be appropriate in instances of extremely contaminated samples.

6.6 Sample Extract Analysis

Sample extracts are then analyzed by GC/MS according to SwRI SOP 01-17-02.

6.7 Records

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

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.

The project manager, analytical manager and analyst are then notified by providing them with a copy of the appropriate laboratory notebook page(s).

All analytical procedures are recorded in proper laboratory notebooks in accordance with client/contract requirements.

7. Quality Assurance Procedures

10% blanks and 10% duplicates will be taken.

8. References

Harvard University/Johns Hopkins University Standard Operating Procedures:
F06 Collection, Storage, and Shipment of Dermal Wipe Samples for Metal and Pesticide Analysis

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-10-17-08 Extraction of Neutral Pesticides, Acid Herbicides, and Phenols from Isopropanol Wipes

U.S. Environmental Protection Agency *Test Methods for Evaluating Solid Waste* (SW 846) Method 8150 Chlorinated Herbicides, 1986.