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-L13**

Title: Extraction of Neutral Pesticides from Drinking Water

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

Notice: *The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), partially funded and collaborated in the research described here. This protocol is part of the Quality Systems Implementation Plan (QSIP) that was reviewed by the EPA and approved for use in this demonstration/scoping study. Mention of trade names or commercial products does not constitute endorsement or recommendation by EPA for use.*

1. Title of Standard Operating Procedure

Harvard University/Johns Hopkins University Standard Operating Procedures:
L13: Extraction of Neutral Pesticides from Drinking Water, Rev. 1.0

2. Overview and Purpose

This SOP is a modification of SwRI SOP-01-17-11 "Extraction of Neutral Pesticides and PCBs in Drinking Water."

3. Discussion

This procedure is applicable for any environmental health project requiring water extraction for neutral pesticides. This procedure is applicable for the determination of target analytes whose recovery by this procedure has been validated.

4. Personnel Responsibilities

4.1 It is the responsibility of the laboratory supervisor to assure that all steps described in this procedure are performed.

4.2 It is the responsibility of the analyst to comply with all the criteria described in this procedure. Any deviations from this SOP will be documented in the laboratory notebook and reported to the project manager.

5. Required Equipment and Reagents

5.1 Equipment

kiln (600°C)
balance, precision 0.0001 g
refrigerator (4°C)
pH meter
pipettes
glassware: beakers, 250-mL Erlenmeyer flask
separatory funnel, larger than 1.0 L

5.2 Reagents

detergent: Alconox or equivalent (Alconox - a biodegradable labware detergent made by Alconox, Inc. New York, USA)
deionized water
acetone
p-terphenyl-d_{14}
10% diethyl ether in n-hexane
10N NaOH
1:1 H_2SO_4
methylene chloride (DCM)

NaCl
Na_2SO_4, anhydrous
nitrogen, dry

6. Procedure

6.1 Glassware Cleaning and Storage

All solvents used for rinses are distilled in glass (Burdick and Jackson pesticide grade or equivalent). Extremely dirty glassware is solvent washed prior to SOP glassware cleaning. All laboratory glassware is:

- Washed with hot water and detergent (Alconox or equivalent)
- Rinsed with hot water;
- Rinsed with deionized water;
- Rinsed with acetone;
- Rinsed with deionized water;
- Fired overnight in a kiln at approximately 600°C
- Stored in a contaminant free atmosphere until time of use.
- Glassware and other apparatus that will be in contact with the sample is protected to the extent possible from other contamination by utilizing laboratories which are devoted exclusively to the determination of trace organics.

6.2 Preparation of Surrogate Solution

- A stock solution for p-terphenyl-d_{14} will be prepared at concentration level of 1-2 mg/mL (precision of weighing should be ±0.0001 g). The amount weighed, the final solvent volume, the lot number of the standard, and lot numbers of all solvents will be recorded in the laboratory notebook. Each standard will be labeled with the common name of the analyte, solvent, concentration, date of preparation, and lot number.
- The lot number will be assigned as "xx-yy-zz" where xx is the notebook page number, yy is the sequential number of the standard prepared on page xx, and zz is the notebook identification. The expiration date of the stock standards will be one year from the date of preparation. Store all solutions at 4° C.
- Working surrogate solution: A working surrogate solution is prepared by diluting the stock solution in 10 % ether/hexane. The precise concentration will be determined by the analytical manager and will be documented in the laboratory notebook.

6.3 Extraction of Pesticides from Water (Separatory Funnel Technique)

6.3.1 Take 1.0L water sample, check/adjust pH to between 5.0 and 9.0 with 10N NaOH or 1:1 H_2SO_4. Add an appropriate volume of the surrogate spiking solution. The volume of surrogate to be spiked will be determined by the analytical manager.

The volume and lot number of each solution spiked will be recorded in the laboratory notebook.

6.3.2 Add 100 mL Methylene chloride (DCM) and shake vigorously for 3 minutes and then add 50 g of NaCl. Shake vigorously for 3 more minutes.

6.3.3 Let layers separate. Remove the organic layer in a 250 mL Erlenmeyer flask.

6.3.4 Re-extract the aqueous layer one more time with 100 mL DCM (don't add NaCl). Pool the organic layer in the 250 mL Erlenmeyer flask.

6.3.5 Dry the organic layer by filtering through 30.0 g anhydrous Na_2SO_4. Solvent exchange (use dry nitrogen stream) to 10% diethylether in n-hexane to final volume of 1 mL.

6.4 Quantitation

The determination of target analytes is then performed by GC/MS according to SwRI SOP 01-17-02. The concentration of analytes in the sample C_s (µg/mL) is calculated by

$$C_s = C_x \times DF \times (V_s/V_e)$$

where

C_x = concentration of the target analyte in the extract (µg/mL),
DF = dilution factor of the extract
V_s = volume of the water sample extracted (mL)
V_e = final volume of the extract.

6.5 Laboratory Notebook:

Record all the calculations, project number, lot number of all the materials including the solvents used during extraction procedure in the laboratory notebook.

Sample extracts are then logged into a freezer location and the location of the samples is entered into the laboratory notebook.

7.0 Records

All observations and procedures used for surrogate and matrix spike preparation will be entered into the laboratory notebook.

The sample ID, initial volume, final volume and lot numbers of all solvents and spike solutions used will be recorded.

Sample extracts will be logged into the freezer and the location written into notebook.

8.0 References

SwRI SOP 01-17-02	Determination of Pesticides, Acid Herbicides, Phenols, and PAHs by GC/MS
SwRI SOP-01-17-11	Extraction of Neutral Pesticides and PCBs in Drinking Water