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-L05**

Title: Sieving and Division of Dust and Soil Samples

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:
   L05 Sieving and Division of Dust and Soil Samples, Rev. 1.0

2. Overview and Purpose

   This SOP is a modification of SwRI SOP-01-19-01, "Log-in, Preparation and Shipment of Housedust Samples." Additional details on dust and soil sieving are from Que Hee et al. and Aschengrau et al., respectively. [Assuming that SwRI will do the sieving.]

3. Discussion

   This procedure is applicable to house dust samples taken using the HVS3 dust sampler, and to soil samples.

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 study personnel to comply with all criteria described in this procedure.

5. Required Equipment and Reagents

   5.1 Equipment

   gloves
   homogenizer
   disposable weigh boat or glass dish (Pyrex or equivalent) (for drying soil)
   Teflon spoon or spatula (for drying soil)
   hood
   mist respirator
   sieves: 1-mm (___ mesh) and 149-µm or 150 µm (100 mesh) for dust
   2-mm (9 mesh) and 250 µm (60 mesh) for soil brush for cleaning sieves
   balance capable of weighing to 0.001 g
   bottles: 3 dr and 6 dr, glass with Teflon lined lids
   bottles for storing samples between steps if necessary

   5.2 Forms

   Chain-of-Custody forms
   computer with database software including the following tables as sub-tables of the main database (see Table 5 at the end of this SOP for the total of these tables):
   Sample Check-In
   Weighing
   Drying
   Sieving
   Splitting

6. Procedure

6.1 Check-In

Each sample will arrive in a jar in a plastic bag with extra ID labels.

When dust and soil samples are received from the field, a technician will log them in and check them against chain-of-custody forms according to HSPH SOP G04.

1. Upon receipt of samples, check sample ID numbers against chain-of-custody forms and fill in the forms. Photocopy the chain-of custody forms. File the copies to be kept. File the originals to go out with samples to be analyzed.

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. To estimate the volume, use the marks on the sample jar. Replace the jar in the bag with its extra ID labels. Store samples in a locked freezer. When Table 1 is completed for the samples received, back up the file.

Table 1 -- Check-In

Sample ID	Date Received	Initials of Technician	Estimated Volume (1)	Storage Location

3. The Sample Custodian prepares a work order from the check-in lists upon receipt of each shipment.

4. Designated personnel from Westat will fax to the Quality Assurance Officer once a week a Sample Shipping Form showing samples shipped to SwRI.

Sample Shipping Form

Sample ID	Sample medium	Field Technician 2 initials	Date mailed	Date received	Date receipt reported to Westat

5. The Quality Assurance Officer will:
   -- verify the samples received against the Sample Shipping Form
   -- list any additional samples actually received
   -- fax a report on the sample verification to the designated Westat personnel
   -- supply a copy of the Sample Shipping Form to the Project Manager and the Data Coordinator every week after receiving samples.

6.2 Weighing

1. When ready to weigh a sample, access "Table 2 -- Weighing" on the computer database. Locate the ID number of the sample. Enter the date and your initials.

Table 2 -- Weighing

Sample ID	Date Weighed	Initials of Technician	Estimated Volume (2)	Mass of jar with sample	Mass of jar without sample	Mass of sample

2. Read the volume of dust or soil from the mark on the jar and record it in the "Estimated Volume (2)" column.

3. Weigh the sample jar and its contents without the cap with accuracy of ± 0.001 g. Record the weight in the "Mass of jar with sample" column.

4. Pour all the dust or soil into a homogenizer. Weigh the empty bottle without the cap. Record the weight in the "Mass of jar without sample" column. The computer will calculate "Mass of sample." When Table 2 is completed, back up the file.

5. If the sample is to be stored before drying (soil) or sieving (dust), pour it into a container, label, and store.

6.3 Drying -- Soil

1. When ready to dry soil, access "Table 3 -- Drying" on the computer database. Locate the ID number of the sample. Enter the date and your initials.

Table 3 -- Drying

Sample ID	Date		Initials of Technician	Mass of sample		% Moisture
	Start	Finish		Wet	Dry	

2. Wear gloves.

3. Attach a piece of clear tape near the edge of a disposable weigh boat or glass dish, and transfer the ID label from the sample jar to the tape. Transfer the soil sample onto the dish and spread it around with a Teflon spoon or spatula. Place the dish on a shelf or table near the fume hood in the laboratory to air dry overnight at room temperature. Remove gloves.

4. If the sample is to be stored before sieving, leave the soil in the dish. Slide the dish into a plastic bag without disturbing the soil. Put the extra ID labels in the bag under the dish, and seal the bag. Place it in a freezer at -12 ± 8°C.
5. When the sample is dry, weigh it (nearest 0.001 g). In Table 3, record the date under "Finish." Record the mass as "Mass of Sample: Dry." The computer will calculate the % moisture. When Table 3 is complete, back up the file.

6.4 Sieving

1. When ready to sieve a sample, access "Table 4 -- Sieving" on the computer database. Locate the ID number of the sample. Enter the date and your initials.

Table 4 -- Sieving

Sample ID	Date Sieved	Initials of Technician	Mass of sample after sieving

2. Carry out the procedure in the hood. Wear gloves and a mist respirator.

3. Sieve the sample through a coarse sieve to remove larger particles. Use the back of a gloved hand to crush larger particles. Discard the material that does not pass through the sieve.

Sieving	Sieve Size	
	Dust	Soil
Coarse	1-mm (16 mesh)	2-mm (9 mesh)
Fine	149-μm or 150 μm (100 mesh)	250-μm (60 mesh)

4. Sieve the sample through a fine sieve. Discard the material that does not pass through the sieve.

5. Weigh the fine fraction (nearest 0.001 g) and record in the "Mass of sample after sieving" column of the Sieving table. When Table 4 is completed, back up the file.

6. If the sample is to be stored before splitting, pour it into a container, label, and store.

7. Clean the sieve and the collection pan after sieving each sample. Tap the sieve on a hard surface. Use a brush to remove residual particles.

6.5 Splitting

1. When ready to split a sample, access "Table 5 -- Splitting" on the computer database. Locate the ID number of the sample. Enter the date and your initials.

Table 5 -- Splitting

Sample ID	Date Split	Initials of Technician	Mass of Fraction (to 0.001 g)			
			Metals	Pesticides/ PAHs	Storage Reserve	Field Spikes

2. Look at the equations shown in Table 6 (dust) or 7 (soil), and decide how many fractions can be made from the sample and what the masses of the fractions should be. The tables show the sample type (ST) for the fraction ID numbers.

[PBR, RAW -- I deleted field spike fraction per PBR's note, added ST number for dry weight fraction]

Table 6 -- Equations for Splitting Dust

X Mass of sample after sieving (g)	M Metals fraction ST = 22	P Pesticide/PAH fraction ST = 23	S Storage fraction ST = 24
0-0.2	X	0	0
0.2-2.2	0.2	X - 0.2	0
2.2-3	X - 2	2	0
> 3	1	2	X - 3

Table 7 -- Equations for Splitting Soil

X Mass of sample after sieving (g)	M Metals fraction ST = 32	P Pesticide/PAH fraction ST = 33	S Storage fraction ST = 34	W Dry Weight ST = 36	D Discard
< 36	???	???	???	???	???
36-86	1	30	X - 36	5	0
≥ 86	1	30	50	5	X - 86

3. Get bottles for the fractions and label them using the ID labels that came with the field sample. Make sure that the ID numbers are correct.

[SwRI: What is the mass cutoff between 3 and 6 dr bottles?]

4. Split the sample into up to four fractions (for metals analysis, pesticide/PAH analysis, dry weight, and storage reserve) according to Table 6 or 7.

5. Weigh the fractions of the sample (nearest 0.001 g) and record the masses in the computer database in "Table 5 -- Splitting."

6. Store the "Metals" fraction at room temperature. Store the other samples in a freezer.

6.6 Shipping

1. Ship the "Metals" samples to HSPH to be extracted and analyzed, according to SOP G05 "Storage and Shipping of Samples."
[SwRI's extraction supervisor, Hamed Edrisi, would be responsible for shipping these]

2. The "Pesticides/PAHs" samples will be extracted at SwRI.

3. The storage reserve fraction will be kept for later use.

6.7 Recordkeeping

Table 5 at the end of this SOP is the sum of Tables 1-4 (sideways for readability). Make sure that all data have been entered for each sample.

7. Quality Assurance Procedures

The sample tracking system will assure the identity and custody of each sample and fraction.

8. References

Aschengrau, Ann, Robert Bornschein, Merrill Brophy, et al. *Three City Urban Soil-Lead Demonstration Project: Protocols for Sampling and Analysis of Soils, Dust, and Handwipes.* Internal EPA document, October 1991.

Harvard University/Johns Hopkins University Standard Operating Procedures:
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
L02 Cleaning of Glass and Plastic Containers
L06 Extraction of Metals from Sampling Media
L11 Extraction of Neutral Pesticides and PAHs from House Dust and Soil

Que Hee, Shane S., Belinda Peace, C. Scott Clark, et al. "Evolution of Efficient Methods to Sample Lead Sources, Such as House Dust and Hand Dust, in the Homes of Children," *Environmental Research,* 38:77-95 (1985).

SWRI SOP 01-01-01 Sample Log-In and Sample Custody
SWRI SOP 01-17-01 Extraction of Neutral Pesticides and PAHs from Housedust
SWRI SOP 01-19-01 Log-In, Preparation and Shipment of Housedust Samples
SWRI SOP 01-19-03 GC/MS

Table 8 Database for Check-In, Weighing, Drying, Sieving, and Splitting of Dust and Soil Samples (sum of Tables 1-5, sideways for readability)

	Sample ID		
	Date Received		Check-In
	Initials of Technician		
	Estimated Volume (1)		
	Storage Location		
	Date Weighed		Weighing
	Initials of Technician		
	Estimated Volume (2)		
	Mass of jar with sample		
	Mass of jar without sample		
	Start	Date	Drying
	Finish		
	Initials of Technician		
	Wet	Mass of sample	
	Dry		
	% Moisture		
	Date Sieved		Sieving
	Initials of Technician		
	Mass of sample after sieving		
	Date Split		Splitting
	Initials of Technician		
	Metals	Mass of fraction	
	Pesticides/PAHs		
	Storage Reserve		