

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-F12

Title: Duplicate Sampling

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

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1. Title of Standard Operating Procedure

Harvard University/Johns Hopkins University Standard Operating Procedure:
F12 Duplicate Sampling, Rev. 1.0

2. Overview and Purpose

This SOP is designed to provide both general and specific guidance for the collection of duplicate samples by HSPH for the NHEXAS project. The purpose of duplicate sampling is to ensure that the value obtained for a sample is indeed correct within sampling and analytical limitations and conversely that sampling and analytical procedures are being followed and are working satisfactorily. In addition, the collection of duplicates over a long period of time will allow the calculation of the method's precision and limit of detection.

3. Discussion

Duplicate samples will be collected at a rate of 10% of the total number of samples to be obtained. A minimum of 2 duplicates will be taken for any set of samples totaling less than 10 (not including the duplicates). Each medium will require a different protocol for duplicate analysis. Duplicate requirements for each medium are described in the SOP for that medium. Section 6.2 of this SOP summarizes the procedures.

The duplicate samples will be collected and analyzed identically to their counterparts, in order to ensure consistency of treatment.

To help with data analysis and interpretation of duplicates as well as actual samples, both lab blanks and field blanks will be collected and analyzed. The primary analysis of the duplicates will be to calculate

- (1) the mean,
- (2) the absolute difference of each of the two duplicates from their mean, and
- (3) the relative percentage difference of each of the two duplicates from their mean.

To assess the method's precision and limit of detection, the standard deviation of the differences of all of the duplicates for a given sample type will be calculated. A plot of the absolute difference vs. the mean for each pair of duplicates will be made; this will illustrate the behavior of differences at low values as well as high values. A corresponding plot of relative difference vs. the mean for each pair of duplicates will be also made.

4. Personnel Responsibilities

- 4.1 Sampler Preparation: As specified in the appropriate SOP.
- 4.2 Field Collection: As specified in the appropriate SOP.
- 4.3 Storage: As specified in the appropriate SOP.
- 4.4 Shipment: As specified in the appropriate SOP.
- 4.5 Analysis: As specified in the appropriate SOP.

5. Required Equipment and Reagents: As specified in the appropriate SOP.

6. Procedure

6.1 Preparation for Collection: As specified in the appropriate SOP.

6.2 Sample Collection Procedure: As specified in the appropriate SOP. Summary:

Medium	Duplicate Samples	Replicate Sample Aliquots	Replicate Analyses
Air -- Indoor, Outdoor	4%. Using extra pump. Indoor: metal and pesticide/PAH samples will be taken at the same time, using one pump, to reduce burden.	None	10%
Air -- Personal	0%	None	10%
Dust	8%. Adjacent floor area (or as close as possible) after cleaning apparatus.	10% if quantities are sufficient.	10%
Soil	9%. Duplicate cores at all sampling points.	10% by splitting and analyzing composite samples.	10%
Dermal wipe	None. No mechanism has been devised.	None	10%
Water	5%. Taken like normal sample.	10%	10%
Food -- duplicate diet	None, because of respondent burden.	None	?
Food -- mini-market basket	No duplicate questionnaires will be administered.	None	
Urine	None, because of respondent burden.	10% of samples will be split.	
Blood	None, because of respondent burden.	10%. Procedures to be worked out with CDC.	

6.3 Preservation and Storage: As specified in the appropriate SOP.

6.4 Handling and Shipping: As specified in the appropriate SOP.

6.5 Laboratory Analysis: As specified in the appropriate SOP.

6.6 Data Workup: As specified in the appropriate SOP.

6.7 Sample Tracking: As specified in the appropriate SOP.

6.8 Calculations: (use a computer spreadsheet)

- 1 Calculate the absolute difference between each duplicate pair.
- 2 Calculate the average of the duplicate pair.
- 3 Calculate the relative percent difference: divide the absolute difference by 2, divide the product by the average, then multiply that product by 100%.
- 3 Prepare a plot of the absolute difference vs. the average for each duplicate pair.
- 4 Prepare a plot of the relative percent difference vs. the average for each duplicate pair.
- 5 Calculate the standard deviation of the absolute differences between each duplicate pair.
- 6 Calculate the precision: multiply the standard deviation (from step 5) by 3.

7. Quality Assurance Procedures

Another staff member will verify all computer entries and check the equations being used in the spreadsheet.

8. References

Harvard/Emory/Johns Hopkins University Standard Operating Procedures:

- G03 Identification Numbers for Samples and Forms
- G04 Chain-of-Custody and Sample Tracking
- G05 Storage and Shipping of Samples
- F01 Field Sampling -- General Information
- F02 Collection, Storage, and Shipment of Indoor and Outdoor Air Samples for Metal, Pesticide, and PAH Analysis
- F03 Collection, Storage, and Shipment of Personal Air Samples for Metal Analysis
- 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
- F06 Collection, Storage, and Shipment of Dermal Wipe Samples for Metal and Pesticide Analysis
- F07 Collection, Storage, and Shipment of Drinking or Tap Water Samples for Metal and Pesticide Analysis
- F08 Collection, Storage, and Shipment of Duplicate Diet Samples for Metal, Pesticide, and PAH Analysis
- F09 Administration and Analysis of Food Checklist and Purchase of Mini-Market Basket Food
- F10 Collection, Storage, and Shipment of Urine Samples for Metal, Pesticide, and Creatinine Analysis
- F11 Collection, Storage, and Shipment of Blood Samples for Metal, Pesticide, PAH, VOC, and Lipid Analysis

Taylor, John K., *Statistical Techniques for Data Analysis*, Lewis Publishers Inc., 1990.