# METHODS FOR SAMPLING AND ANALYZING HOUSEHOLD DUST FOR THE UNIVERSITY OF MICHIGAN DIOXIN EXPOSURE STUDY

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## Introduction

"Dioxins" or dioxin-like compounds are a family of structurally related chemicals including polychlorinated dibenzodioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and polychlorinated biphenyls (PCBs) which are all byproducts of industrial processes. The University of Michigan Dioxin Exposure Study (UMDES) was undertaken in response to concerns among the population of Midland and Saginaw Counties that dioxin-like compounds from the Dow Chemical Company facilities in Midland have resulted in contamination of soils in the Tittabawassee River flood plain and areas of the City of Midland. There is concern that people's body burdens of PCDDs, PCDFs and PCBs may be elevated because of environmental contamination. A central goal of the UMDES is to determine the factors that explain variation in serum congener levels of PCDDs, PCDFs, and PCBs, and to quantify how much variation each factor explains. House dust concentration and loading were included in the list of potentially explanatory factors to investigate. This report describes the methods utilized to sample and analyze the UMDES household dust samples and discusses their effectiveness and potential limitations. The descriptive statistics, distributions, and congener patterns of the household dust concentration and loading data have been described elsewhere.\(^1\)

### **Materials and Methods**

Respondent Selection: Five populations in Midland, Saginaw, Bay, Jackson, and Calhoun Counties, Michigan, USA were sampled using a two-stage area probability household sample design. In order to be eligible for participation in the household dust sampling portion of the UMDES, subjects had to have lived in their residence at least five years and had to be the owner of their residence. A more detailed description of the populations and respondent selection methodology is reported elsewhere.<sup>2</sup>

Sampling Strategy: Household dust sampling was conducted in the home of each eligible respondent following their consent. The sample was taken from sampling locations that presented the highest potential for human contact with household dust and dirt. The locations were generally a frequently occupied living space (e.g., living or family room) and a high traffic hallway or pathway. Samples were taken from both hard and soft surfaces with carpeted floors and area rugs being the preferred sampling surfaces. Samples were not taken of undisturbed dust in generally inaccessible areas. Sample areas within each sample location were selected based on the representativeness of the area of the location and the amount of accessible space the area provided. Furniture movement was limited to lightweight objects such as coffee tables, end-tables, and open-leg chairs or loveseats. Sofas, recliners, and other large furniture were not moved unless it was determined that the area underneath the furniture was accessible to the respondents for potential exposure. Accessible was defined as 6 inches or more vertical space from the floor to the bottom of the furniture.

Sampling Apparatus: High Volume Small Surface Samplers (HVS3s) manufactured by CS-3, Inc. (Sandpoint, ID) were used to collect the UMDES household dust samples. Each HVS3 vacuum cleaner had Teflon® coatings and gaskets and was equipped with a Teflon® coated aluminum cyclone and fine-particle filter capable of capturing

99.95% of particles above 0.3 µm aerodynamic mean diameter. The bulk dust and fine fraction samples were collected in an I-Chem certified pre-cleaned wide-mouth amber glass catch bottle and on a 10-inch diameter Pell-Gelman glass fiber type A/E filter respectively. The HVS3's suction source was a Royal® Commercial upright vacuum Model Number 1028Z manufactured by the Royal Appliance Manufacturing Company (Glenwillow, OH).

Sampling Protocol: The sampling protocol was based, with minor modifications, on the American Society for Testing and Materials (ASTM) method "Standard Practice for Collection of Floor Dust for Chemical Analysis". Adhesive tape (at least 1 meter long) was placed on the top edge of the area to be sampled. A second piece of adhesive tape was placed on the floor so that the two pieces were parallel to each other, on either side of the portion of area to be sampled, and between 0.5 and 1.5-m apart. The corners were connected to complete the rectangle. A fabric measuring tape pre-marked into 13-centimeter sections was taped across the top of the sampling area with clear packaging tape. A second pre-marked fabric tape was extended across the bottom of the sampling area and covered with clear packaging tape. This was necessary so that the HVS3 did not disturb the fabric tape during sampling. The HVS3 was placed in one of the lower corners of the sampling area and the flow rate and pressure drop were adjusted according to the floor type. Sampling was performed by moving the nozzle between the ends of the pre-marked fabric tapes. The HVS3 was moved back and forth four times on the first 13 cm strip (determined by the "blocks" marked on the fabric tape) at approximately 0.5 m/s. It was then angled over to the adjacent strip and the four double passes were repeated. Sampling continued until an adequate sample was collected or the area was exhausted. If additional sampling was required to collect enough dust, another sampling area was selected and the protocol was repeated. The sampling technicians attempted to collect a minimum of 10 grams of total dust in order to yield analytical detection limits equal to or less than 1 part per trillion (ppt).

Field Documentation: Field data sheets were completed at each sampled residence. Data collected on the sheets included the date, time, and sampling team as well as the sampling locations, indoor conditions, surface types, equipment settings, total area sampled and total sampling time. Each field data sheet also included a labeled diagram of all sampled locations. Samplers also noted whether there was a wood-burning fireplace near the sampling location, whether there were any household pets at the residence, and whether the sampling location had been flooded and if so, when.

Shipping Procedures: Samples were transported on ice to a dedicated 4° C cooler before being delivered to Vista Analytical Laboratory for analysis. Samples were shipped according to 40 Code of Federal Regulations 761.65 (i)(3) and in accordance with current and applicable United States Department of Transportation (US DOT) standards. A chain of custody form containing the study name, dust sample ID number, sample date, sample description, container type, sample matrix, and analyses required was completed and secured inside the shipping container with the dust samples. The samples were sent via overnight carrier. Upon receipt by the laboratory, the laboratory personnel completed the chain-of-custody forms, measured and recorded the internal temperature of the shipping container, and checked the respondent identification numbers on the containers to insure consistency with the chain of custody forms.

Sampler Cleaning: HVS3s and individual sampling trains were decontaminated following each use to protect against cross-contamination of household samples. HVS3s were removed from the residence and decontaminated infield by wiping down the filter holder, vacuum body, wheels, and cord with pre-soaked acetone wipes. Sampling trains were removed from the HVS3 after each use and decontaminated with distilled water, Alconox, methanol and acetone at the end of each sampling day in a secure decontamination facility.

*Sample Analysis:* The bulk dust and filter sample were combined resulting in one household dust sample per sampled residence. Analyses were performed by Vista Analytical Laboratory, Inc. (El Dorado Hills, California, USA) for the WHO designated 29 PCDD, PCDF, and PCB congeners<sup>4</sup> using US EPA methods 8290<sup>5</sup> and 1668<sup>6</sup>.

QA/QC: Equipment rinsate blanks were collected by the sampling team after every fifty samples and submitted to Vista Analytical Laboratory for laboratory analysis. The equipment rinsate blank consisted of a sample of distilled water that had passed through the HVS3 sampling train following normal cleaning procedures. Submitted blanks were analyzed for the WHO designated 29 PCDD, PCDF, and PCB congeners<sup>4</sup>. Quality control procedures for analytical services were conducted by Vista Analytical Laboratory in accordance with their standard operation procedures and the individual method requirements.

Data Analysis: A descriptive analysis of PCDD, PCDF, and PCB congener concentration and loading was performed for each of the five geographic regions. Tests for equality of these levels among the regions was performed. Upper quantiles were compared among regions in addition to comparisons of mean levels. To test for potential effects of variables such as sampled surface age, sampled surface type, presence of a wood-burning fireplace, household activities, and soil PCDD, PCDF, and PCB concentration on household dust levels, regression models for left-censored data, with survey sample weights, were employed using the entire dataset and all geographic regions. Both Stata<sup>7</sup> and SAS<sup>8</sup> statistical software packages were utilized to complete the analyses.

#### **Results and Discussion**

Results and discussion will not be available until after complete study results have been presented to the affected communities in August of 2006.

#### References

- 1. Zwica L, Knutson K, Towey T, Hedgeman E, Franzblau A, Chen Q, Lee S-Y, Sima C, Gillespie BW, Adriaens P, Demond A, Lepkowski J, Ward B, Ladronka K, Olson K, Sinibaldi J, Chang S-C, Gwinn D, Swan S, Garabrant D. *Organohalogen Comp* 2006 (forthcoming).
- 2. <u>Lepkowski J</u>, Olson K, Ward B, Ladronka K, Sinibaldi J, Franzblau A, Adriaens P, Gillespie BW, Chang S-C, Chen Q, Demond A, Gwinn D, Hedgeman E, Knutson K, Lee S-Y, Sima C, Swan S, Towey T, Zwica L, Garabrant D. *Organohalogen Comp* 2006 (forthcoming).
- 3. American Society for Testing and Materials, Standard Practice for Collection of Floor Dust for Chemical Analysis, Designation D 5438-00, Reprinted from the Annual Book of ASTM Standards, Philadelphia, PA.
- 4. Van den Berg M, Birnbaum L, Bosveld AT, Brunstrom B, Cook P, Feeley M, Giesy JP, Hanberg A, Hasegawa R, Kennedy SW, Kubiak T, Larsen JC, van Leeuwen FX, Liem AK, Nolt C, Peterson RE, Poellinger L, Safe S, Schrenk D, Tillitt D, Tysklind M, Younes M, Waern F, and Zacharewski T. *Environmental Health Perspectives* 1998;106:775.
- 5. United States Environmental Protection Agency. Method 1668, Revision A: Chlorinated biphenyl congeners in water, soil, sediment, and tissue by HRGC/HRMS. Washington, DC: Office of Water, 1999.
- 6. United States Environmental Protection Agency (US EPA). Method 8290: Polychlorinated dibenzodioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) by high-resolution gas chromatography/high-resolution mass spectrometry (HRGC/HRMS). Washington, DC: Office of Solid Waste and Emergency Response, 1994.
- 7. StataCorp. Stata Reference Manual Release 8. College Station, TX: Stata Press, 2003.
- 8. SAS Institute. SAS/STAT User's Guide Version 8. Cary, NC: SAS Institute Inc., 1999.