

QUALITY ASSURANCE METHODS DURING THE SERUM ANALYSES OF PCDD, PCDF, AND PCB LEVELS FOR THE UNIVERSITY OF MICHIGAN DIOXIN EXPOSURE STUDY

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Introduction and Study Goals

The University of Michigan Dioxin Exposure Study (UMDES) was undertaken to determine whether environmental contamination downstream of the Dow Chemical Plant in Midland, MI is contributing to the body-burden of dioxins, furans and PCBs in the surrounding population. To investigate, blood samples taken from a randomized sample of the population living within three counties surrounding the Dow Chemical plant and the Tittabawassee River were compared to a referent population of individuals living in two removed counties.

Whole blood samples obtained for this study needed to be in the range of 70 to 80 milliliters to assure that sufficient serum (20 – 30 milliliters) could be separated for the lipid and congener analyses. Duplicate samples were not obtained from study participants, so alternate methods needed to be designed for ensuring the precision and accuracy of the serum results. With help from the National Center for Environmental Health (NCEH) laboratories a protocol was designed including initial laboratory standardization and ongoing blind sample incorporation. This paper presents the methods and outcomes of these quality assurance measures.

Materials and Methods

All serum analyses were performed by Alta Analytical Laboratories using internal adaptations of EPA methods 8290 and 1668 for dioxins, furans and PCBs.^{1,2} Lipid concentrations were determined enzymatically.³ Exact methods for the serum analyses including specimen handling, processing and laboratory methods can be found in the UMDES protocol located at www.umdioxin.org.

Quality assurance methods occurred in two stages: a set of initial tests prior to the start of any fieldwork and serum sample collection, and ongoing tests while fieldwork and sample collection were active. Prior to the start of sample collection, NCEH laboratories sent Alta Analytical laboratories three, 30 milliliter, unknown serum samples for analysis. The three samples, all aliquots from the same standard reference material, were used to identify any initial problems in technique that may have caused excessive variation in results from the NCEH laboratories results. Additionally, since Alta Analytical only performed lipid determinations gravimetrically, the enzymatic lipid determination results provided by an outsourced medical contractor needed to be verified.

During fieldwork, a protocol was set up to deliver blind serum samples to Alta Analytical for ongoing quality assurance. Shipping containers, amber jars, unique study identification numbers, custody seals and chain of custody forms for 20 serum samples were sent to the NCEH labs. NCEH personnel filled the jars with approximately 30 milliliters of standard reference material 1589a,⁴ sealed and labeled the jars, then sent the frozen samples by overnight delivery to a phlebotomy contractor associated with UMDES. The UMDES phlebotomy contractor then included one blind sample for approximately every 20 study samples shipped to Alta Analytical. Care was taken to

mimic every detail of a normal study specimen. Results for these 20 samples were then forwarded on to the NCEH personnel for comparison.

Graphic comparisons of results and variation were performed using SAS version 9.1.

Results and Discussion

Results and discussion will be provided after August 15th, 2006.

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