Second Interlaboratory Comparison for the Analysis of Pu-239 in Synthetic Urine at the μBq (~100 aCi) Level - A Work in Progress.

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At the 1998 BAER conference, the authors reported on the conduct and results of the first interlaboratory comparison for the analysis of Pu-239 in synthetic urine at the μBq (~100 aCi) level. The need for Pu-239 detection capabilities at this low level was the pending implementation of a radiobioassay program by the Department of Energy for the resettlement of the Marshall Islands residents. The impetus for the first study was the desire of the Office of International Health Program, Department of Energy to evaluate and validate new technologies that may supercede the existing fission tract analysis for the analysis of Pu-239 at the uBq/L levels. Of particular interest was the possible use of mass-spectrometric detection capabilities in conjunction with radiochemical separations and concentration of Pu-239. Participants in this first study included Brookhaven National Laboratory using fission tract analysis (FTA) and Inductively Coupled Plasma Mass Spectrometry (ICP-MS), Los Alamos National Laboratory using Thermal Ionization Mass Spectrometry (TIMS) and Pacific Northwest National Laboratory using ICP-MS. The results of this first study indicated that the FTA and MS methods had the detection capability to measure Pu-239 at the uBq/L level but with moderate to substantial biases at the lower test level. Only the Brookhaven National Laboratory's ICP-MS method met the ANSI 13.30 requirements for bias and precision at all levels tested.

Following a 1999 meeting held at LANL by interested laboratories, a second interlaboratory comparison study was formulated in 2000 and implemented in 2001. This study was supported by the Department of Energy, Office of International Health and the participating laboratories of Los Alamos National Laboratory using TIMS, Lawrence Livermore National Laboratory using Accelerator Mass Spectrometry (AMS) and the University of Utah using FTA. Consistent with the first study, five test levels were to be evaluated for the analytes; blank or zero analyte and approximately 20, 50, 150 and 280 uBq/L. Five samples per testing level were provided for the non-zero analyte levels. The second study differed from the first by increasing the number of blank samples from five to eight and including Pu-240 and a uranium interference with the Pu-239 in each synthetic urine sample. The laboratories using mass-spectrometric techniques were to quantify Pu-239 and Pu-240. The natural uranium interference was added at a concentration typical of urine from a non-exposed population living near Los Alamos.

This presentation will discuss the sample preparation and verification protocols used by NIST for this study, a synoptic description of each participant's radiochemical, atom detection and data reduction protocols, and available study results. The results of a laboratory's results for each testing level will be evaluated and estimates of bias (difference) relative to the NIST analyte value and precision from the five replicate samples will be calculated. The estimated bias and precision values will be compared to the acceptable bias and precision criteria of ANSI N13.30. The detection capability of each method will be evaluated using the minimum detectable concentration equation provided in ANSI N13.30 and through other data evaluation means.