## B134 Assessing Microheterogeneity of Surrogate Post-Detonation Urban Debris (SPUD) Standard Reference Material (SRM) 4600 Using Microbeam X-Ray Fluorescence (µ-XRF)

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After attending this presentation, attendees will understand the importance of developing a well-characterized, homogeneous SRM to aid in the development of methods needed to characterize the debris expected from the aftermath of a nuclear attack.

This presentation will impact the forensic science community by discussing how the development of this SRM will facilitate the ability to analyze debris from an improvised nuclear device detonation in a city, aiding intelligence efforts in identifying the responsible parties.

Concerns over the use of nuclear or radiological materials in terrorist attacks have led to the emergence of the nuclear forensics field. This discipline focuses on best practices to evaluate such material both before and after the detonation of a nuclear device. A thorough characterization of pre- and post-detonation materials may aid intelligence efforts to attribute responsibility for an attack by identifying the fuel type, weapon design, production process, production date, and other features of the device.[1] Well-characterized SRMs and Certified Reference Materials (CRMs) are needed to ensure that nuclear forensic measurements will be sufficiently accurate and precise to be legally defensible.

Currently, there is a shortage of SRMs/CRMs needed to develop methods for analyzing pre- and post-detonation nuclear material.[1] In some cases, cost, scarcity, or quality of existing materials is problematic, whereas in other cases, a suitable material simply does not exist. Given the likelihood that a large city would be the target of an attack, efforts have been undertaken to prepare to analyze the debris expected from the detonation of an improvised nuclear device in an urban environment. The National Institute of Science and Technology has led a collaboration to develop two vitrified SPUD SRMs: SRM 4600 and SRM 4601.[2] A glass-like material was selected for its durability, control over what can be added to the mixture, straight-forward mass balance, and relative ease of homogenization. The matrix of the SRMs is a composite of materials and metals present in concrete (cement, crushed stone, and sand) and steel, two of the most common urban building materials. SRM 4600 was doped with natural depleted uranium to serve as a blank; SRM 4601 was doped with Uranium-235 (approximately 22% highly enriched uranium) to serve as a test sample.

As part of the process to certify the major and minor components of the SRMs, non-destructive µXRF measurement with Principal Component Analysis (PCA) on the resulting data was used.[3,4] Sixteen 1.5-gram samples of loose solid SRM 4600, each representing a replicate of the production lot, were prepared in XRF sample cups sandwiched between two sheets of X-ray-transparent film. To assess the homogeneity of the sample, 10,000 spectra were collected at both a single, fixed location and random locations from each of the sixteen "sandwiches." Collecting 5,000 spectra from the same location provided measurements of variability due to the instrument and systemic sources of error. The 5,000 spectra collected from random locations across the "sandwich" represent variability due to both the instrument and sample heterogeneity.

Quantifying the homogeneity of a sample is necessary to identify the effects of "nuggets," small subsamples of the bulk material enriched in certain elements.[3,4] These nuggets, which can vary in size and composition, cause the XRF data to stray from a normal distribution, precluding the use of common statistical measures of variability. PCA is able to take multiple statistical factors from the X-ray map and single location data in order to establish a detection limit for nuggets. The nominal beam diameter of the instrument used is 400µm, and a larger spot size can be simulated by averaging adjacent data points. This process will average out any imperfections present, and the averaging can be repeated iteratively until the PCA treatment indicates no detectable nuggets. From these analyses, a Minimum Sample Mass (MSM) can be calculated, which defines the smallest mass of sample needed for all elements to be homogenous. Preliminary calculations for SRM 4600 suggest an MSM of 3mg is required for reproducible measurements of element concentrations to not be susceptible to sample heterogeneity. This presentation will discuss the analysis of the 16 replicates of SRM 4600, which were analyzed by the above methodology to verify the accuracy of the preliminary MSM calculations and determine if the MSM can be decreased.

**Reference(s):**

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**Nuclear Forensics, Post-Detonation, Standard Reference Material**



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