APPLICATION OF MICRO-XRF FOR LOCATING AND EXTRACTING TRACE PLUTONIUM PARTICLES ON FILTERS AND SWIPES

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A number of spatially resolved, imaging analytical and materials science techniques are commonly used to examine plutonium. Until recently, however, Micro-X-ray fluorescence (MXRF) instrumentation had been relatively uncommon and underutilized for spatially resolved plutonium analysis and imaging. A particularly challenging application is locating and identifying plutonium particles trapped in HEPA filters for extraction and analysis by other methods. Such particles are often <1 μm in diameter. Although this is well within the resolution of scanning electron microscopy (SEM) and concurrent X-ray imaging, many of the HEPA filter samples analyzed in this study contained only a minimal amount of total plutonium, resulting in the presence of only isolated micron sized plutonium particles spread over multiple cm^2 areas. SEM instrumentation is designed primarily for sub micron to micron scale analysis and is not easily applied to imaging over multiple cm^2 areas such as the HEPA filter samples discussed here. Thus, locating particles by SEM would be extremely difficult. Further, the necessity to analyze a plutonium-contaminated sample under vacuum by SEM would result in radioactive contamination of the sample chamber, and the sample is non-conducting which would result in detrimental charging from the electron beam.

Here, the advantages of MXRF were utilized to spatially map HEPA filter samples to locate and identify plutonium particles that were then extracted for SEM morphology and other analyses. The samples were sealed inside and analyzed directly through plastic bags to avoid instrument contamination (something that is not possible when using an SEM instrument due to the short penetration depth and dispersion of the electron beam). Although a majority of the plutonium particles on these samples were too small and isolated to detect by MXRF using a 30 μm diameter polycapillary optic-collimated X-ray beam, a method was developed to successfully locate a few larger aggregates of plutonium particles (up to several 10s of μm in diameter). The technical challenges for locating plutonium particles were extreme due to various issues such as severe energy overlaps with filter impurities, poor plutonium signal-to-background due to severe source scatter, and extremely low particle density across the HEPA substrate. The instrumental techniques developed to overcome these hurdles and successfully image the particles will be presented.

Surface swipe substrates were also examined for plutonium and other residue spatial distribution using MXRF. Again, MXRF was chosen for its capability to map elements over large areas on a non-conducting surface and with better plutonium detection limits than SEM. Additionally, a larger 2 mm diameter aperture was used to restrict the X-ray beam to provide the ability to image over areas ~10 cm wide. A comparison of these very large area maps with the much higher resolution polycapillary HEPA filter maps will be discussed.