Analyzing Challenging Materials with Micro-X-ray Fluorescence

Christopher G. Worley
Los Alamos National Laboratory

Micro-X-ray fluorescence (MXRF) is routinely used to non-destructively determine most major and minor and some trace elemental constituents in radioactive materials prior to using destructive analytical methods such as mass spectrometry isotopic analysis, radiochemical methods, inductively coupled plasma spectroscopy, and matrix assay classical chemical methods. Determining the major elemental composition of radioactive materials is straightforward using MXRF, but when the sample matrix is plutonium or enriched uranium, safely handling and containing the material can be challenging. Various thin polymer films marketed to XRF analysts for liquid analysis are used to seal the sample in a liquid cell. As with liquid applications, if light elements are of interest, the film type is chosen to optimize light element X-ray transmission while ensuring the film is robust enough to avoid rupture during handling and analysis. For solids with sharp and/or protruding edges, light element analysis can be difficult.

Another obstacle with analyzing radioactive samples is the measurement medium. Many samples are analyzed at atmospheric pressure to avoid the possibility of breaching the containment film when applying vacuum, but this eliminates the ability to examine most third row elements at ppm concentrations. The MXRF instrument used in the applications to be discussed was recently retrofitted with a helium flush system to improve light element sensitivity, and examples using this system will be presented.

Other significant MXRF challenges faced in analyzing common radioactive materials include: differentiating XRF from diffraction peaks, contending with peak overlaps from the sample matrix and impurities, severe analyte signal absorption by the plutonium or uranium matrix, and the presence of radioactive decay X-ray and gamma peaks. Due to these complicating factors, examining plutonium and uranium materials by MXRF can be quite complex. However, MXRF has proven invaluable as an elemental screening tool for these materials as will be demonstrated in the examples presented.