Thin Film XRF Specimen Preparation:  
Improvements Over The Dried Spot Approach

Christopher G. Worley  
Los Alamos National Laboratory

The concentration of minor and trace analytes in plutonium metal can affect the physical properties of the metal, and wavelength dispersive X-ray fluorescence (WDXRF) is a useful method for quantifying some of these analytes. However, radioactive solutions are analyzed after dissolving the plutonium, and time-consuming steps must be taken to minimize the possibility of the liquid leaking before, during, and after analysis. Past attempts to analyze solid material involved drying drops of the radioactive solution, and with enough time-intensive specimen preparation, acceptable results were obtained. In the current work, a thin film WDXRF specimen preparation method is compared to the previous dried spot approach.

The specimen preparation method used routinely for analyzing gallium in plutonium by WDXRF entails dissolving plutonium metal and removing the plutonium with ion exchange chromatography. The gallium remaining in solution is then analyzed. Relative accuracy and precision values well below 1% can be achieved using this process. However, the specimen solution is radioactive due to the presence of residual plutonium and trace americium. Hence, the potential exists for radioactive liquid to leak during the analysis. A secondary containment film is used to minimize this risk, but avoiding having to analyze radioactive solutions would be ideal. A thin film dried specimen preparation method was developed to avoid analyzing solutions but which also provides excellent accuracy and reproducibility. In addition, both gallium and iron were quantified using this method. Improvements in reproducibility and accuracy using this thin film approach will be compared to the previous dried spotting technique.