

WASTE REDUCTION PROCESS IMPROVEMENTS IN THE ANALYSIS OF PLUTONIUM BY X-RAY FLUORESCENCE: RESULTS FROM MULTIPLE DATA SETS

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An X-ray fluorescence (XRF) method is well established for quantifying gallium in plutonium metal.¹ The currently certified method involves metal dissolution, extracting the plutonium with ion exchange solid phase extraction (SPE), and quantifying the eluate for gallium content. Because disposing transuranic (TRU) waste is expensive, waste reduction would substantially reduce costs as well as adhere to environmental management waste minimization requirements.

In the current study, several modifications were made to the established XRF method. The sample size was reduced by 50%, which decreased needed SPE acid volumes and resin by ~50%. Consequently, fewer TRU-contaminated acid containers needed to be disposed. Also, a pre-nitrated resin was used, which required approximately half the amount of nitric acid currently necessary to fully condition the resin.

In addition to these waste minimization practices, several additional cost savings and safety improvements were implemented. Ultra high quality acid (ppt impurities) is used in the currently certified method for historical reasons, but considerably cheaper trace metal grade acid (ppb impurities) was used in the modified process. Toxic hydrofluoric acid is used in the current method for historical reasons but was eliminated in the modified process. Finally, Mylar specimen containment film was replaced with more robust Kapton to reduce the possibility of film rupture during analysis of the specimen. This tougher film was also necessary due to the longer counting and X-ray beam exposure times required from reducing the sample size.

Multiple batches of previously well-characterized plutonium were analyzed over three months using this modified method, and the results were encouraging. These results and the modified process uncertainty will be presented and compared with what is achievable using the currently certified specimen preparation method.

1) C. G. Worley, *Advances in X-ray Analysis*, **2002**, 46, 369-374.