Development of Small-Scale Automated Separations for Thermal Ionization Mass Spectrometry Sample Preparation

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Plutonium isotopic analysis is performed by members of the International Atomic Energy Agency to verify international compliance with the nuclear non-proliferation treaty. Thermal ionization mass spectrometry (TIMS) analysis is currently considered by many to be the most sensitive Pu analytical method. One of the challenges associated with TIMS analysis is its susceptibility to reduced ionization efficiency caused by analyte impurities co-loaded on the filament. In order to reduce impurities from the sample matrix and those present in the reagents used during the sample purification process, sequential chemical separations are performed at ever-decreasing volume scales, with final sample purification often consisting of a single anion exchange bead and microliters of solution. Sample preparation for TIMS has previously been performed manually and has been an arduous, time consuming, and costly endeavor.

Research is being conducted in a collaborative effort between Pacific Northwest National Laboratory and Sandia National Laboratory to automate these separation procedures, with a current focus on the small-scale analyte purification steps. As part of the automated separation procedure, chromatographic separations are being developed using high specific activity ^{238}Pu (~$6x10^5$ Bq/μg), which is a useful radiometric analogue for simulating ^{239}Pu concentrations measured by TIMS in environmental samples. At present, we are developing anion exchange resin separations at the microliter volume scale. Once fully developed, the automated microliter separations may be performed in one tenth the time required for the manual microliter separations. This automation reduces the time and cost associated with high sensitivity Pu isotopic analysis.