Spring 2015

Alternative Sample Loading Preparation for Thermal Ionization Mass Spectrometry

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Recommended Citation

Husson, Scott M.; Powell, Brian; Fugate, Glenn; Locklair, David; and Mannion, Joseph, "Alternative Sample Loading Preparation for Thermal Ionization Mass Spectrometry" (2015). Chemical and Biomolecular Graduate Research Symposium. 9.

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Thermal Ionization Mass Spectrometry (TIMS) analysis is known as the “gold standard” in isotopic ratio measurements for plutonium.

Isotopic distribution of plutonium indicates the “grade” of material and can be used to determine the source; a common measurement being the $^{239+240}$Pu/$^{244}$Pu ratio.

TIMS is used widely for nuclear forensics and nuclear safeguards analyses of long-lived isotopes; due partly to ultra-low detection limits offered by TIMS (on order of ~femtograms for Pu) and unparalleled accuracy.

**What is Thermal Ionization Mass Spec.?**

- Ion Source
- Magnet sector
- Mass Analyzer
- Detector

Ionization occurs at or near the surface of a hot (~2000 °C) rhenium filament.

**Current Sample Loading Process: Bead Loading**

- Sample Purification ~ 2 - 3 Weeks
- Low HCl
- High HCl

Bead loading offers the lowest detection limit of any current TIMS sample loading method by:
- Maintaining a reducing atmosphere within the TIMS
- Producing plutonium –carbide species
- Providing an ion point source

**Cons of Bead Loading:**
- Bead loading is difficult, time-consuming, and expensive.
- SRNL reports that approximately 20% sample loss occurs from bead-loading.
- It is estimated that approximately 95% of the sample could remain in the graphitic skeleton left by the bead.

**Alternative Sample Loading Method: Thin-Film Loading**

- Goal: Simplify sample loading by pre-coating the rhenium filaments with anion-exchange material, eliminate/reduce sample loss, and improve overall efficiency.

**Production of a Thin-Film Ion Point Source**

- Bead loading provides an “ion-point source” greatly improving ion transportation into the mass spec region of the instrument relative to more dispersed ion sources.