eichrom

Expertise in Sample Preparation

Application Note: 603

# Metal Impurities in Uranium, Plutonium and Mixed Oxides

**Introduction**: Metal impurities in uranium, plutonium and mixed oxides are measured in nuclear power plant fuel, weapons and waste. ICP-AES, ICP-MS, TIMS and alpha spectroscopy are used to measure a wide variety of impurities. New methods have been developed to shorten the analysis time, decrease waste generation and improve precision of these analyses.

**The Approach**: To avoid the need to maintain gram per liter Pu and U standards for matrix matching, these new methods remove the uranium and/or plutonium from the sample prior to instrumental analysis. The approach is quite versatile, allowing U only to be removed, while Pu and other metals pass, or capturing both U and Pu together while allowing the other matrix impurities to pass and be analyzed.

For example, the specification for neptunium and plutonium in nuclear fuel is less than, 3.3 Bq/g U. Removal of the uranium from the sample matrix makes the use of alpha spectroscopy possible by eliminating uranium self adsorption and the source of an interfering spectra from $^{234}U$ which overlaps with $^{237}Np$ at 4.7 MeV.

Uranium and/or Plutonium are removed from the sample by the use of an extraction chromatographic resin, marketed under the name of UTEVA® Resin. This resin is comprised of diamyl amylphosphonate sorbed on an inert polymeric support. A nitric acid uptake curve for UTEVA Resin is shown in Figure 1. Virtually no other metals sorb on UTEVA when loaded from nitric acid.

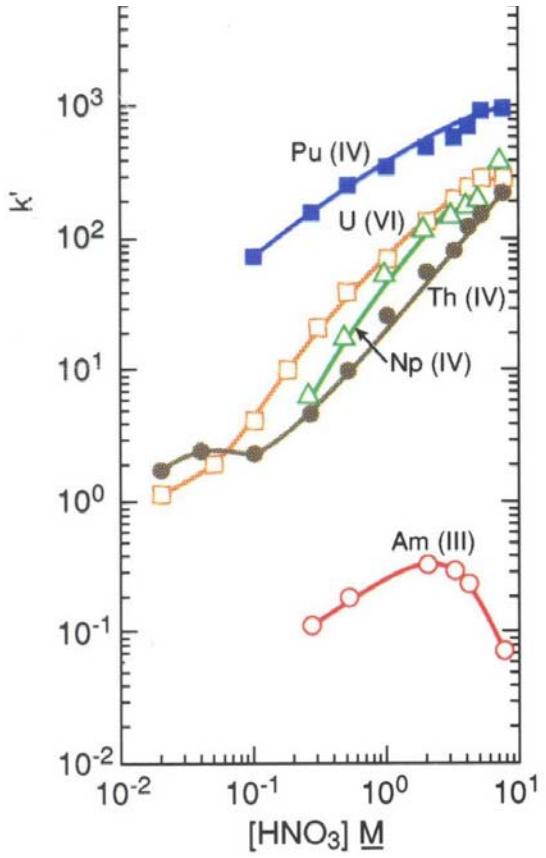


**Figure 1. UTEVA Resin Acid Dependency**

**References:**

C. Good, Third International Uranium Hexafuoride Conference, Paducah KY, (1995)
S. L. Maxwell, III, Eichrom Western Users' Group Workshop, Albuquerque, MN (2000)
M. J. Brisson, ACS Spring National Meeting, Orlando, FL (2002)

## Case History 1: Analysis of impurities in Pu/U.

A method has been developed and implemented at the Washington Savannah River site to characterize legacy U and Pu solids stored for over a quarter of a century. Analysis was required as part of a stabilization and repackaging program associated with long term storage. Over 1600 cans of material were analyzed containing various amounts of U and Pu-239. Figure 2 is a schematic of the U/Pu removal using UTEVA Resin. Uranium and plutonium samples were spiked with a variety of metals. Their ICP-MS recoveries are presented in Table 1.

**Table 1.**

| Element | % Recovery |
|---|---|
| Ag | 106 |
| Al | 101 |
| As | 88 |
| B | 89 |
| Ba | 106 |
| Be | 90 |
| Ca | NA |
| Cd | 94 |
| Ce | 108 |
| Cr | 103 |
| Cu | 106 |
| Fe | 106 |
| Ga | 101 |
| Hf | 90 |
| HG | 77 |
| K | 102 |
| La | 108 |
| Li | 101 |
| Mg | 103 |
| Mo | 101 |
| Na | 98 |
| Nb | 98 |
| Ni | 103 |
| P | 154 |
| Pb | 99 |
| Se | 87 |
| Si | 132 |
| Ta | 84 |
| V | 104 |
| W | 113 |
| Zn | 91 |
| Zr | 63 |

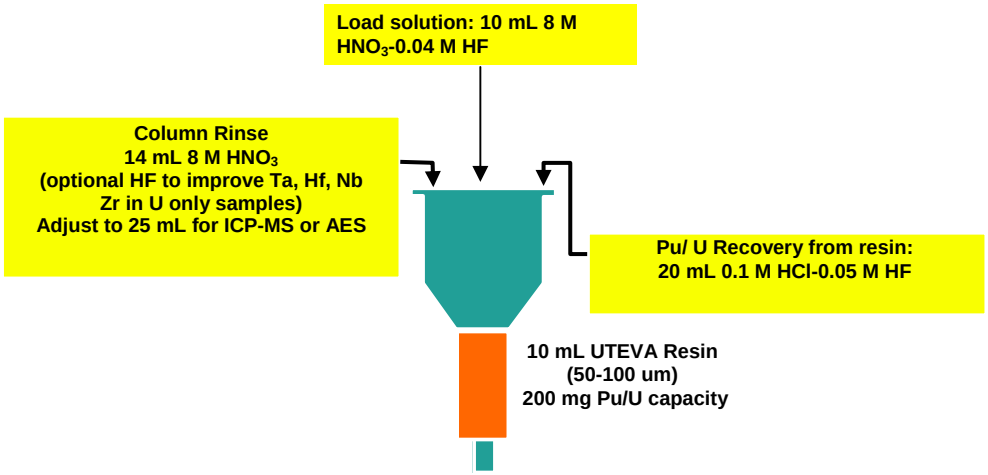


**Figure 2. U/Pu matrix removal using UTEVA Resin for trace metal analysis.**

## Case History 2: Analysis of transuranic elements in $UF_6$

A variation on the method in case 1 allows for the collection and measurement of plutonium with the trace metal fraction. This is accomplished by dissolving the uranium sample in both 2 M nitric acid and 0.1 M oxalic acid. After a 15 mL 0.1 M oxalic acid/ 2 M nitric acid rinse, the combined load and rinse are passed through one additional UTEVA column. This accomplishes the $>10^8$ decontamination factor for uranium based on a 0.5 gram sample of 4.4% enriched U.

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