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HIGH SPEED ION EXCHANGE TECHNIQUES FOR NEPTUNIUM, PLUTONIUM, AND URANIUM IMPURITY ASSAYS

by

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INTRODUCTION

Rapid, efficient ion exchange separations can be performed 10 to 15 times faster then conventional flow rates by using a modified commercial vacuum extraction system and small particle resins. At the Savannah River Plant (SRP), these techniques are being applied to make ion exchange separation techniques more rapid and thus more practical for routine laboratory applications.

APPLICATIONS

- Uranium removal prior to impurity measurements via ICP, DCAP spectrometry.
- Plutonium removal prior to impurity measurements via ICP, DCAP spectrometry.
- Purification of plutonium dissolver solutions prior to plutonium assay via coulometry.
- Purification of neptunium solutions prior to neptunium assay via ICP, DCAP via spectrometry.
- Sample cleanup prior to ion chromatography measurements on chloride, nitrate.

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EQUIPMENT

- Spe-ed Mate™-10 Vacuum Extraction System, Applied Separations, Inc., Bethlehem, PA.

  - Modified by Applied Separations with baked-on epoxy coating on stainless steel surfaces for corrosion resistance.

  - Use of 15-mL columns with polyethylene frits (20-μm pore size).

  - Plastic disposable tips to assure no contact with stainless steel and thus prevent sample contamination.

  - Applied vacuum: 8 to 12 in. mercury.

ION EXCHANGE RESINS

- Cation: AG 50 W-X12 (hydrogen form, minus 400 mesh).

- Anion: AG MP-1 (chloride, nitrate, sulfate forms, 200 to 400 mesh).

- Resins from BIO RAD, Richmond, CA.

APPLICATION DETAILS

Uranium Removal

Uranium removal prior to impurities measurements. Advantages are:

- Better uranium removal than solvent extraction, particularly when high fluoride levels are present.

- Allows recovery of elements that are not recovered with organic solvents[1] such as:

  - TBP - Tributylphosphate.
  - TOPO - Trioctylphosphine oxide.
  - TEHP - Tri-(2-ethylhexl)-phosphate.
  - DHDECMP - Dihexyl-N,N-diethylcarbamylmethylene phosphonate.

Solvent/Acid	Elements Recovered by Ion Exchange/Not by Solvent
- TBP,HNO3:	Gold, palladium, rhenium, thorium, and thallium.
- DHDECMP,HNO3:	Gold, hafnium, rare earths, and thorium.
- TOPO,HNO3:	Bismuth, gold, hafnium, molybdenum, thallium, and thorium.
- TEHP,HNO3:	Gold, palladium, rhenium, thallium, and thorium.

- Ion Exchange Scheme

A combination of HCl-based cation exchange and sulfate anion exchange.[2]

Plutonium Removal

Plutonium removal prior to impurities measurements via plasma emission spectrometry:

- Much more rapid than DC are spectrographic techniques.
- Anion exchange removal of plutonium using 8N HNO_3 after metal dissolution in HCl.
- System can be used in a glove box location.

Purification of Plutonium Dissolver Solution Prior to Assay via Coulometry

- Time savings: separation time reduced from 2 hours to 20 minutes for five samples.
- Anion exchange using 8N HNO_3 elution of impurities, elution of plutonium with 0.36 N HCl-0.01 N HF.
- Initial tests show accuracy and precision better than 0.1%.

Purification of Neptunium Solution Prior to Assay by DCAP,ICP Spectrometry

- Anion exchange using 9N HCL-0.02 N NH_4I to remove plutonium, elution of neptunium with 1 N HCl-0.1 N HF. Neptunium wavelength used: 456.0 nm.

Uranium, Americium, and Plutonium Removal Prior to Ion Chromatographic Measurements of Chloride and Nitrate

- Cation exchange removal of uranium, americium, and plutonium elution of chloride and nitrate with water.
- Allows fast sample cleanup and protection of IC columns.

REFERENCES

1. J. E. Rein, "Separations of Actinides before Metal Impurity Analyses," Los Alamos National Laboratory Report, LA-UR-86-2006 (June 1986).

2. F. W. E. Strelow and C. R. Van Zyl, "The Separation of Uranium from Other Elements by Ion Exchange Chromatography," Journal of the South African Chemical Institute," Vol. 20 (1967).