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Beryllium: Necessity for Monitoring

UGM05 - 09/12/05 - Manchester

Outline

- Beryllium, uses and properties
- Beryllium: associated problems
- Necessity for monitoring
- Problem encountered
- Dipex resin
- Effects of large quantities of Uranium
- Summary

Beryllium: uses and properties

- Discovered in 1798
  - Not widely used in Industry until 1940s and 50s
- Lighter than Aluminum, Stiffer than Steel
  - 2nd lightest metal
  - 6 times stiffer than steel
- High heat absorption
  - One pound absorbs as much heat as 5 pounds of copper
- Be Metals, Alloys, Salts and Oxides are used for a wide variety of Industries
  - Structures in high-speed aircraft (space shuttle)
  - Satellite mirrors and space telescopes
  - Golf clubs and bicycle frames
  - Neutron moderators or reflectors in nuclear reactors

Beryllium: associated problems

- Physical Problems
  - Expensive
  - Brittleness
    - Increases toxicity
- Health Hazard
  - Most Significant disadvantage for industrial use
    - Causes Chronic Beryllium Disease (CBD)
      - No known cure, can only be treated
    - Produces scaring of lung tissue
      - Chronic, may take years to develop
    - Average latency period is 10-15 years
    - 2-5 % of population Be sensitive
      - Exemple: Over 100 current and former DOE employees have CBD

Necessity for monitoring

- Workers safety
  - Air and surfaces monitoring
- Currently
  - ICP-AES or GFAA on microwave digested samples
  - sensitive to certain interferences

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Problems encountered

Interfering elements in the AES spectrum of Beryllium

Table 1. Potential Spectral Interferences for Be determination by ICP-AES[a]

Analyte	Peak (nm)	Intensity	Analyte	Peak (nm)	Intensity
Cr	312.870	15.0	Nb	313.079	2200.0
U	312.879	6.0	Ti	313.080	6.0
Zr	312.918	400.0	Ce	313.087	65.0
Nb	312.964	22.0	Th	313.107	27.0
U	312.973	15.0	Be[b]	313.107	41000.0
Zr	312.976	550.0	Tm	313.126	2300.0
Th	312.997	10.0	U	313.132	8.0
V	313.027	1020.0	Hf	313.181	20.0
OH	313.028	0.0	U	313.199	15.0
Ce	313.033	50.0	Cr	313.206	1000.0
Be[b]	313.042	64000.0	Zr	313.207	7.0
U	313.056	6.0	Th	313.226	5.0
OH	313.057	0.0	Mo	313.259	1800.0
U	313.073	0.0	Ce	313.259	30.0

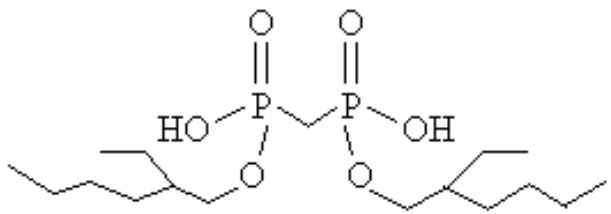
[a]As listed in Varian Plasma96 software version 1.12

[b]Commonly used peaks for beryllium determination by ICP-AES

- Beryllium lines very intense → method is very sensitive for the determination of beryllium
- Interfering lines from other elements could lead to false positives
- Positive determination of beryllium leads to shut down of operation until cleanup

Problems encountered

- Currently Interfering lines are corrected using inter element correction (IEC)
- Uranium is particularly spectrally rich
- Spectrum shifts depending on the degree of enrichment of the Uranium
- Method for removing AES interfering elements from samples desired, Uranium in particular
- Several EXC Resins evaluated for their ability to purify Be samples



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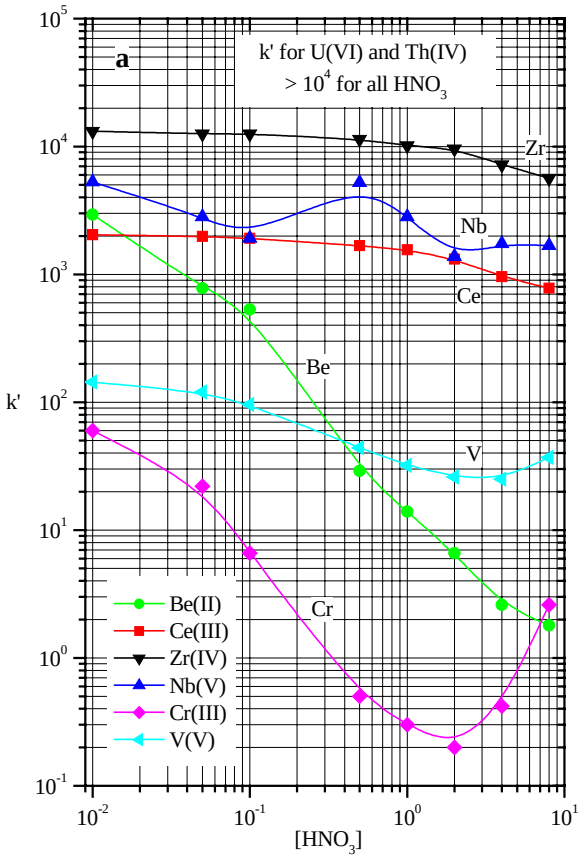

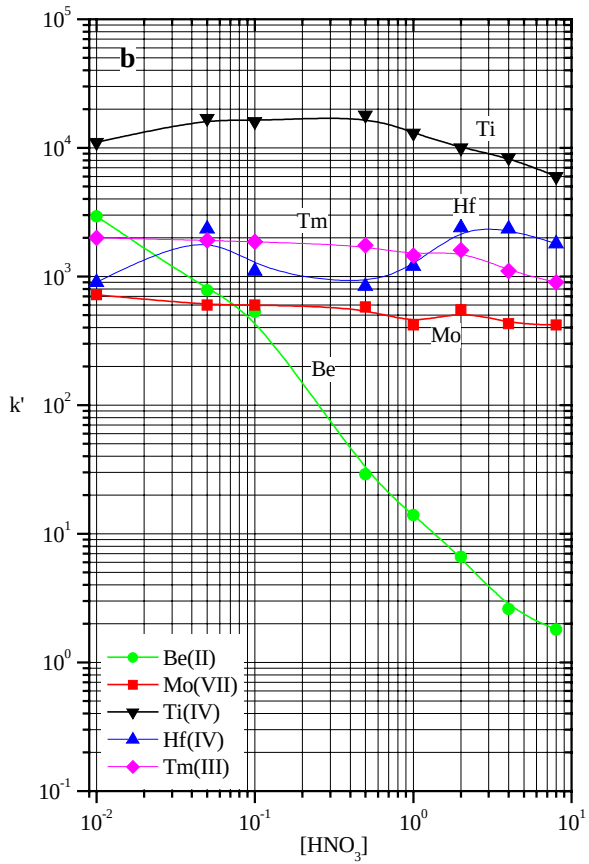

- Be retained at low acid and stripped at high acid
- Most interferences strongly retained over entire range
- Cr weakly retained over entire range
- Single column should purify Be from all interferences
- Uranium very strongly retained

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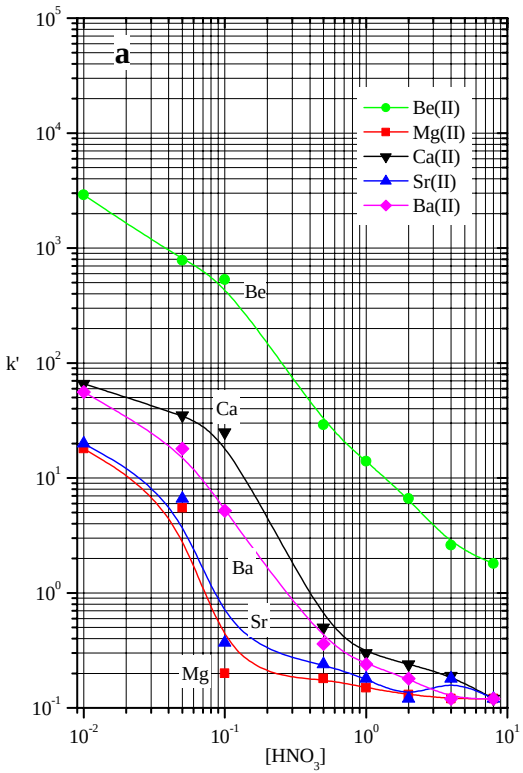
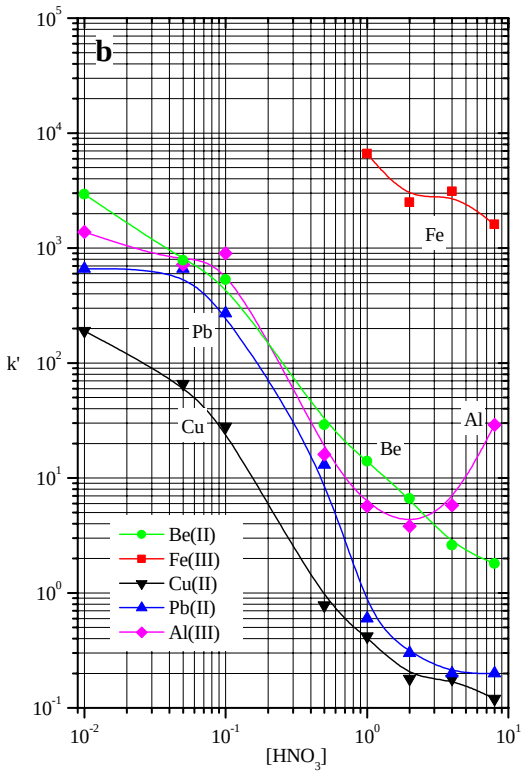
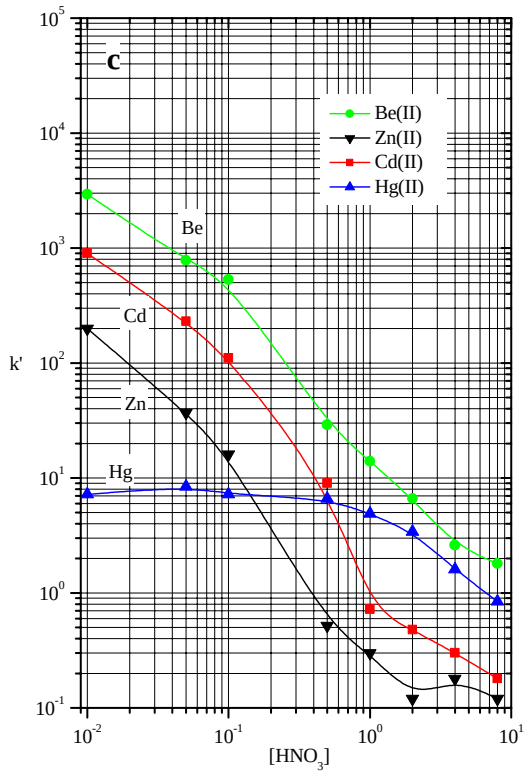
Uptake of Selected Elements on Dipex Resin







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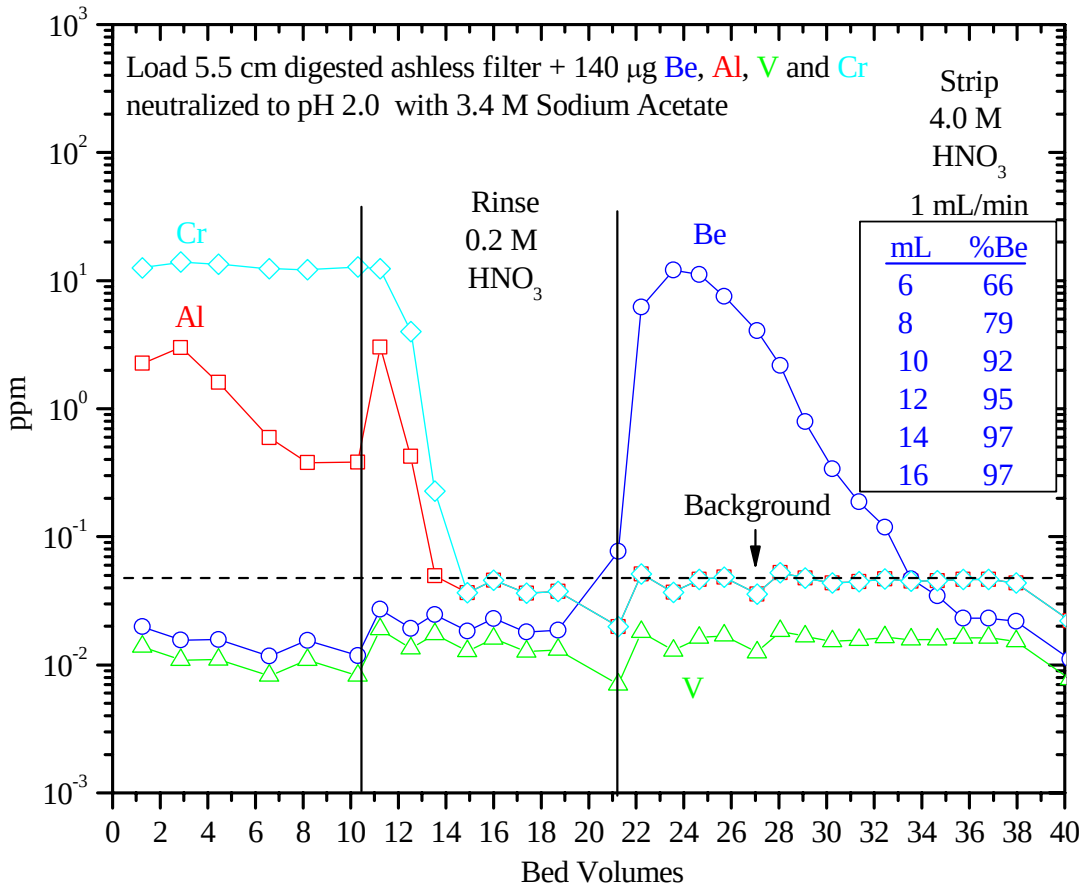
Elution of Be and Selected Elements on Dipex Resin



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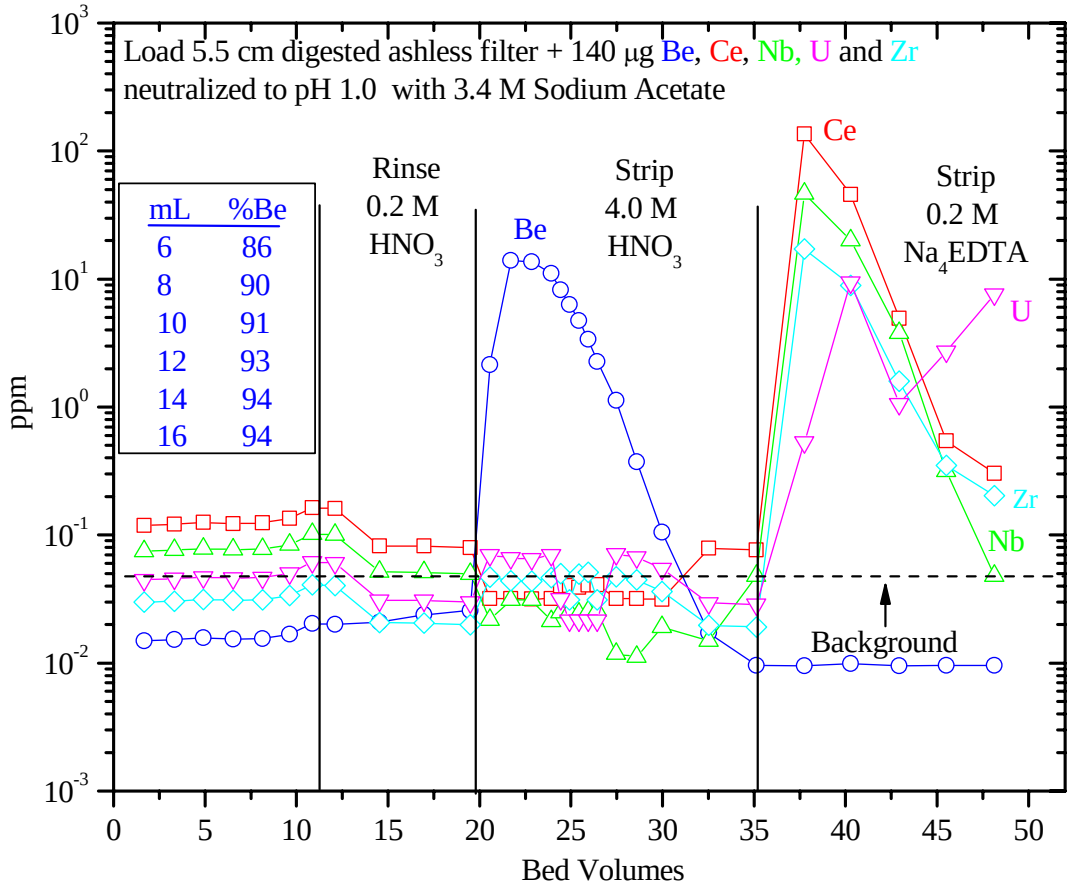

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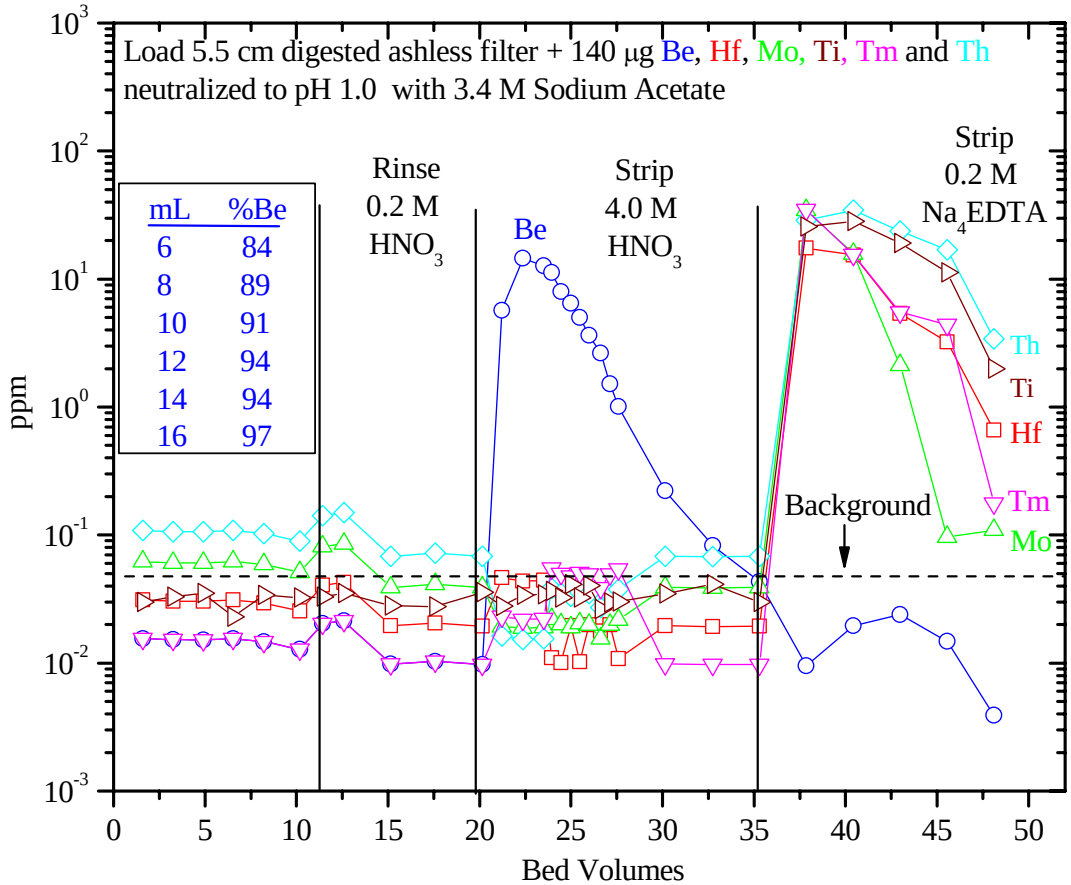

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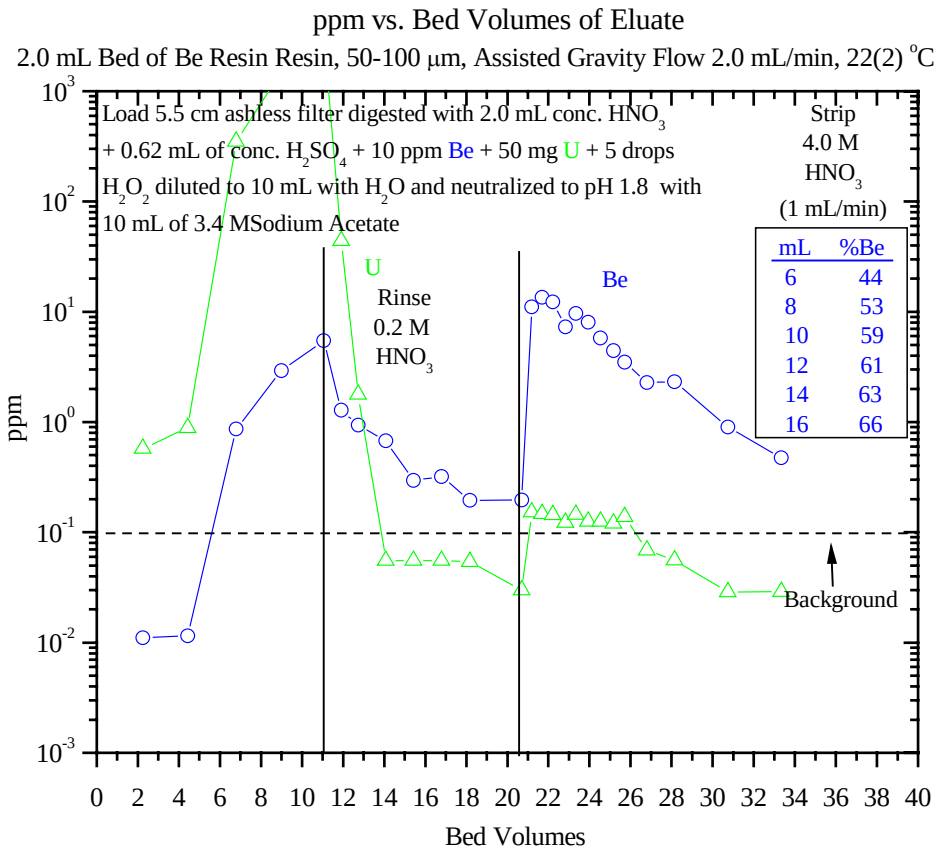
Effect of Large Quantities of Uranium



- Uranium uptake on Dipex Resin is very high
- Large amounts of Uranium could consume resin capacity and reduce Beryllium yields
- 50 mg of U reduces Beryllium yield to ~60%
- 100 mg of U reduces Beryllium yield to 29% and leads to U in Beryllium fraction

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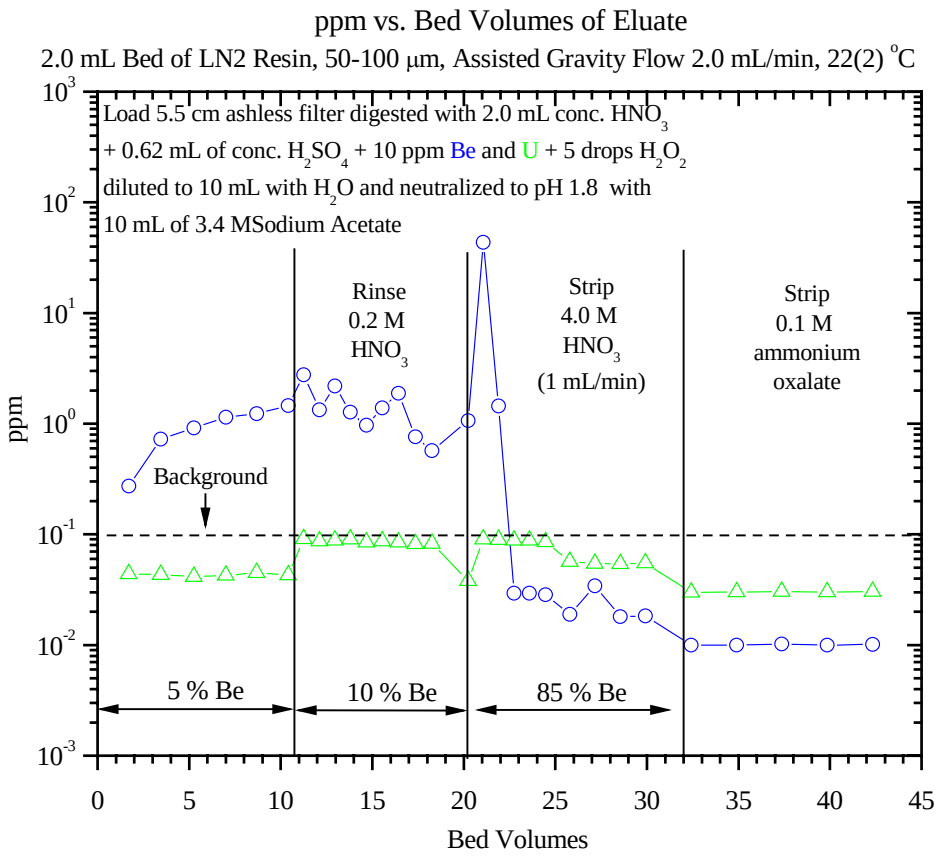
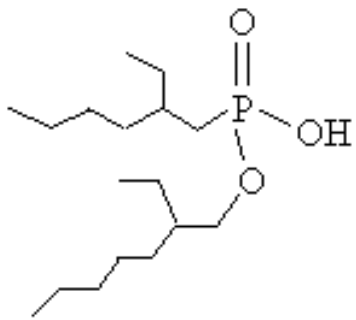


- An LN2 Resin guard column effectively separates U from Be
- LN2 resin contains a substituted phosphonic acid



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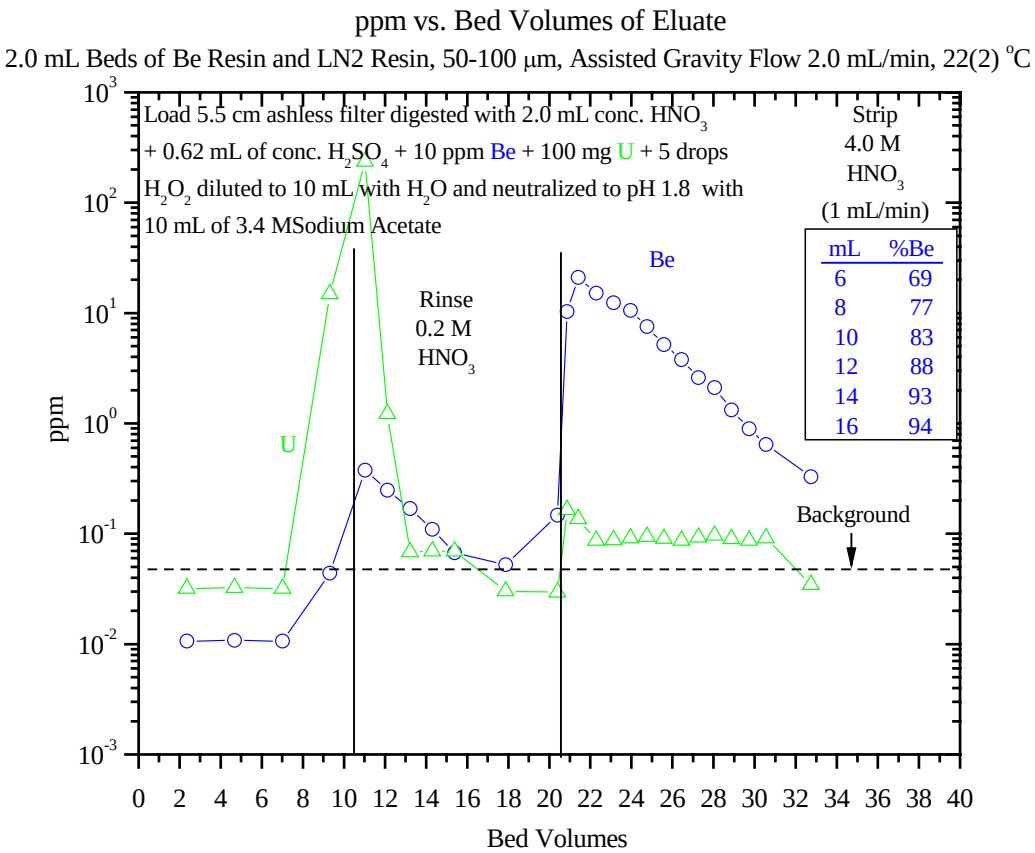

- Coupling an LN2 Resin guard column increases Uranium capacity without decreasing Beryllium yields or changing chemistry
- Over 100 mg of Uranium does not decrease Beryllium yield or produce Uranium in Beryllium fraction
- GC attached through load, rinse and strip

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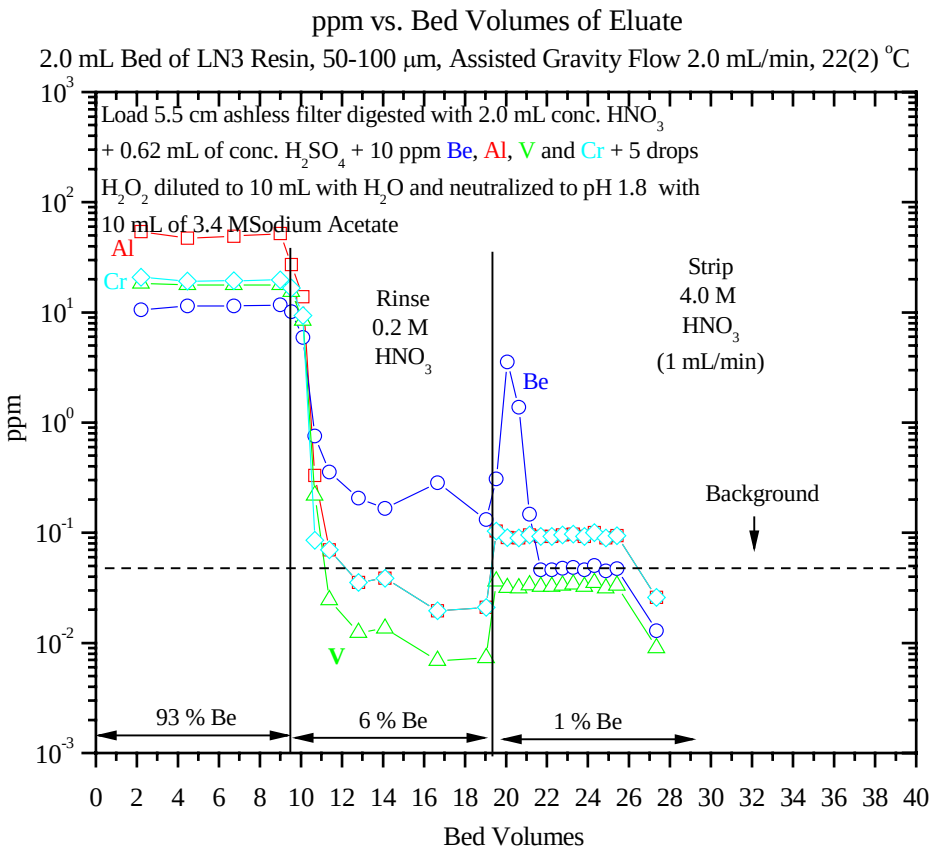
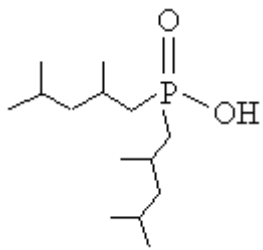
Effect of Large Quantities of Uranium



- An LN3 Resin guard column also removes U from Be and retains Be less than LN2
- LN3 resin contains a substituted phosphinic acid



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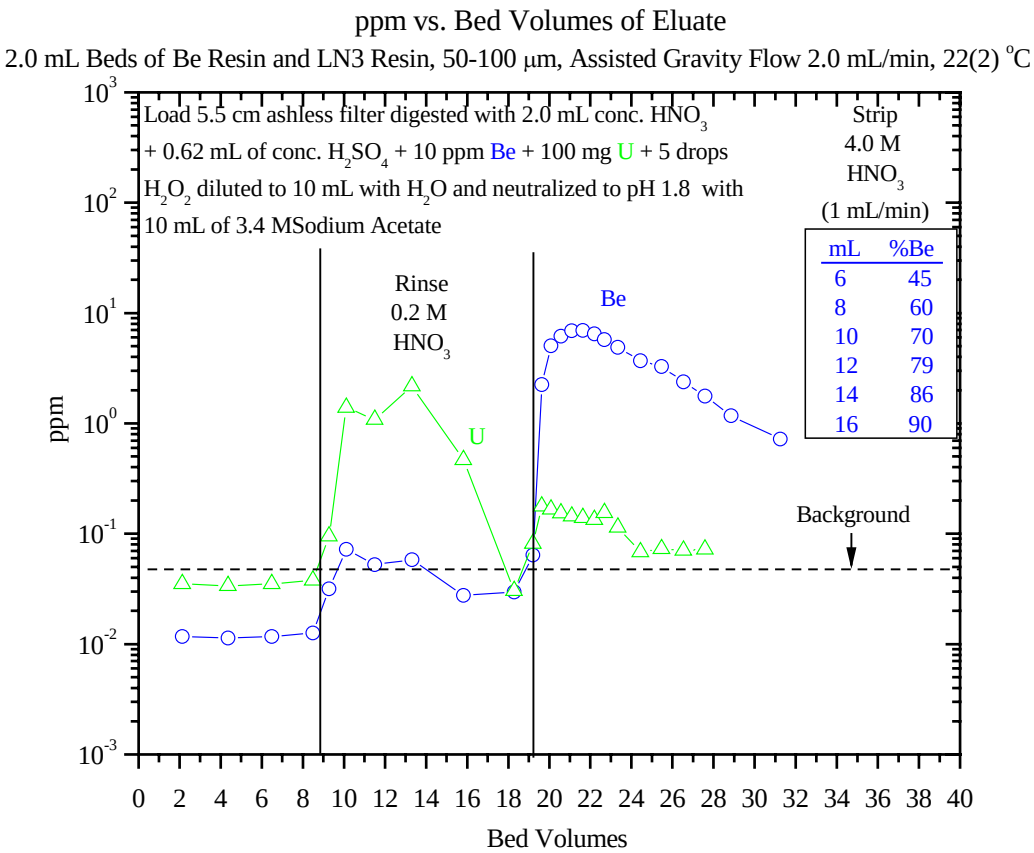
Effect of Large Quantities of Uranium



- Coupling an LN3 Resin guard column increases Uranium capacity without decreasing Beryllium yields or changing chemistry
- Over 100 mg of Uranium does not decrease Beryllium yield or produce Uranium in Beryllium fraction
- GC can be removed following rinse

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Effect of Large Quantities of Uranium

Beryllium Yields and Uranium Impurity vs mg Uranium in Load Solution[a]

	2 mL Beryllium Resin		2 mL Beryllium Resin + 2 mL LN2		2 mL Beryllium Resin + 2 mL LN3	
mg U	% Be in 12 mL[b]	µg U in Be fraction	% Be in 12 mL[b]	µg U in Be fraction	% Be in 12 mL[b]	µg U in Be fraction
0.14	90	< 1.5[c]	85	< 1.5	N/A	N/A
10	92	< 1.5	N/A	N/A	N/A	N/A
25	86	< 1.5	87	< 1.5	97	< 1.5
50	61	< 1.5	88	< 1.5	97	< 1.5
75	N/A	N/A	81	< 1.5	93	< 1.5
100	29	580	88	< 1.5	79	< 1.5

[a]Whatman filter paper spiked with 0.14 mg Be, digested with H_2SO_4/H_2O_2, and neutralized with sodium acetate to pH 1.8

[b]Beryllium Resin Strip Solution 4.0 M HNO_3

[c]Detection limit for Uranium by ICP-AES under the experimental conditions

- LN2 and LN3 Resins effectively increase the capacity for Uranium
- With LN2, the GC remains connected through the load, rinse and strip
- With LN3, the GC can be removed following the rinse

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Summary

- Efficient, reliable method for purifying Be from all ICP-AES spectral interfering elements has been found using a single column
- Method is compatible with current monitoring and sample digestion methods
- Method is robust and performs over a wide pH range
  - pH can be monitored with indicator to ensure optimal performance
- Inserting a LN2 or LN3 Resin guard column increases U capacity without changing the chemistry or significantly decreasing Be yields
- Dipex Resin currently available from Eichrom as Beryllium Resin