

PRODUCT SHEET

Actinide Resin™ (AC Resin)

Main Applications

- Preconcentration and separation of actinides
- Gross-alpha activity determination
- Separation of Beryllium

Packing

| Order N°. | Form | Particle size |
|---|--|---------------|
| AC-B25-A, AC-B50-A, AC-B100-A, AC-B200-A | 25g, 50g, 100g and 200g bottles AC Resin | 100-150 µm |
| AC-C20-A, AC-C50-A, | 20 and 50 2mL columns AC Resin | 100-150 µm |
| AC-R25-S, AC-R50-S | 25 and 50 cartridges AC Resin | 50 – 100 µm |

Physical and chemical properties

Density : 0.35 g/ml

Capacity :

| Element | Capacity (mg / g of resin) |
|---------|-------------------------------|
| Th(IV) | 86.1 |
| U(VI) | 145.7 |
| Nd(III) | 45.4 |
| Fe(III) | 32.1 |
| Ca(II) | 14.8 |
| Ba(II) | 45.0 |
| Be(II) | 0.64 |

Conversion factor D_w/k' : 1.94

Conditions of utilization

Recommended T of utilization : /

Flow rate: A grade: 0.6 – 0.8 mL/min

Storage: Dry and dark, T<30°C

For additional information see enclosed literature study

Eichrom methods

| Reference | Description | Matrix | Analytes | Support |
|----------------------|--|----------------------|-------------------------|------------------|
| ACW11 | Gross Alpha Radioactivity in Water | water | alpha emitting nuclides | bulk |
| Application note 602 | Beryllium analysis – Matrix removal for more reliable ICP-AES analysis | metal, salts, alloys | Beryllium | bulk, cartridges |

TRISKEM INTERNATIONAL

Parc de Lormandière Bât. C – Rue Maryse Bastié – Campus de Ker Lann – 35170 Bruz – France

Tel +33 (0)2.99.05.00.09 – Fax +33 (0)2.99.05.07.27 – www.triskem-international.com – email : contact@triskem.fr

SAS au capital de 40.000 euros – SIRET 493 848 972 00011 – APE2059Z – TVA intra communautaire FR65 493 848 972

09/09/15

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Actinide Resin™

The Actinide Resin™ (AC Resin, in the literature also referred to as DIPEX™ resin) is mainly used for the preconcentration and separation of actinides from acidic solutions obtained from environmental samples (soil and large volume water samples)^{(1) (2)}. The AC Resin shows a higher affinity for actinides in general and in particular a better selectivity for americium, compared to its ion exchange analogue Diphonix®. This high affinity is also used for the determination of the gross alpha activity of urine and water samples.⁽⁴⁾⁽⁵⁾

The AC Resin is composed of an inert support which is impregnated with bis(2-ethylhexyl) methanediphosphonic acid (H₂DEH[MDP]) (Figure 1).

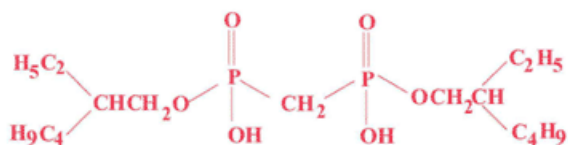


Figure 1: H₂DEH[MDP]^{(1) (2)}.

Horwitz et al.⁽¹⁾ studied the extraction characteristics of the resin in hydrochloric acid. The extraction equilibrium of Am(III) and Fe(III) is reached within 20 min, 30 min respectively, independent of the hydrochloric acid concentration (Figure 2).

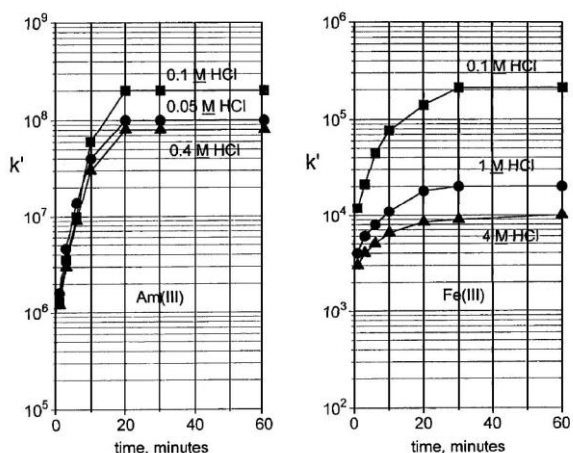


Figure 2 : Extraction kinetics of Am(III) and Fe(III) at different HCl concentrations (AC Resin, 50-100µm)⁽¹⁾.

The figures 3 and 4 show that the retention of actinides and various other elements frequently found in environmental samples (Ca, Fe,...) is very strong at pH 2 and is then diminishing more or less distinctively with increasing acid concentration. The elements shown in fig. 4

represent potential interferents for the uptake of actinides, especially at pH 1 or 2. Bi(III), Fe(III), Ti(IV), Eu(III) and Fe(II) can interfere over the whole acid range. It should be noted that Ra(II) is also very well fixed at pH 2.

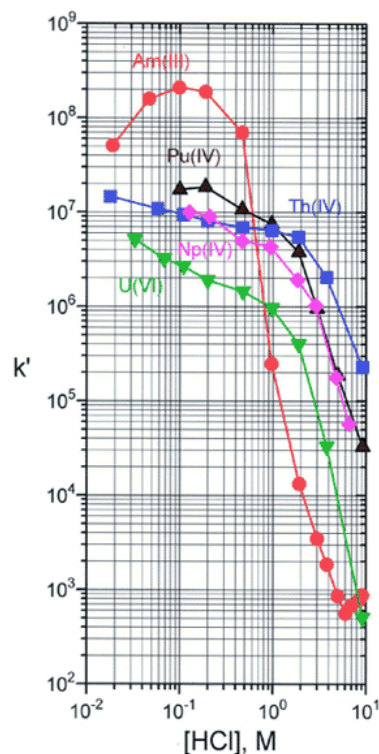


Figure 3 : k' values of actinide elements in HCl⁽¹⁾.

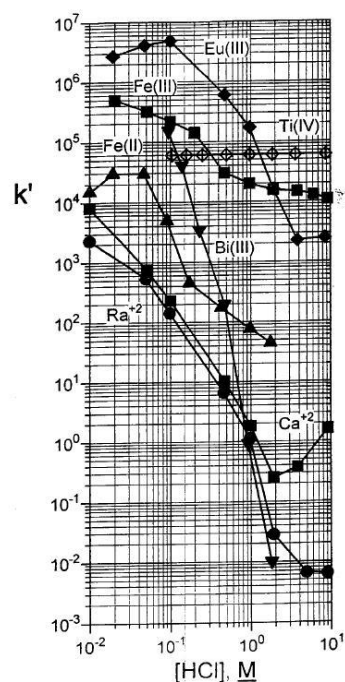


Figure 4 : k' values of various elements in HCl⁽¹⁾.

With regard to the high selectivity of the resin for Am the authors also evaluated the impact of

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several interferents, namely Ca, Al and Fe, on the Am uptake (Fig. 5). The interference shown by the elements increases in the order Fe(II)<Ca(II)<Al(III)<Fe(III). Fe(II) shows very little impact on the Am uptake, the capacity factor k' of Am(III) remains greater than $10E+3$ for all HCl concentrations and Fe(II) concentrations between 0.01M and 2M. For Ca the k' Am remains greater than 100 at Ca concentrations between 0.01M and 2M. Fe(III) concentrations equal to or higher than 0.01M on the other hand efficiently prevent the retention of Am, independent of the concentration of HCl.

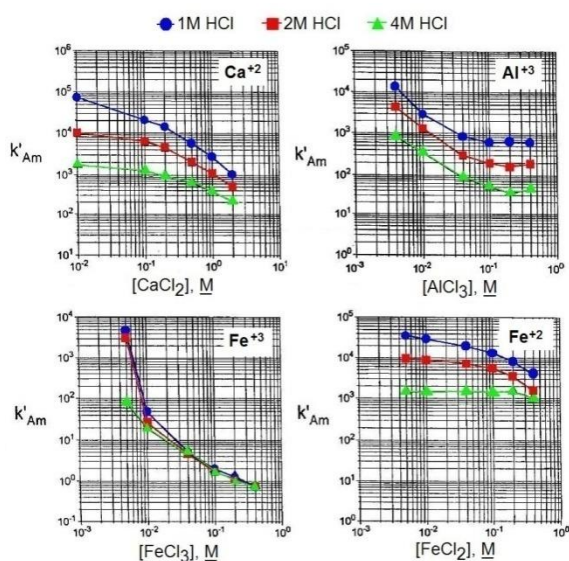


Figure 5 : k' values for Am on AC Resin in presence of Ca(II), Al(III), Fe(II) and Fe(III) in 1M, 2M and 4M HCl⁽¹⁾

The AC Resin is robust against the interference of HF which is often used for total dissolution of soil samples⁽¹⁾. The authors additionally studied the behavior of Ti(IV) since it is strongly retained on the resin ($k' \sim 6 \times 10E+4$) and can thus interfere with the retention of the actinides (Figure 4). For both of the HCl concentrations tested in the study the HF shows very little impact on the retention of the actinides. At HF concentrations greater than 1M the k' values start to diminish, but remain, even at HF concentrations as high as 4M, greater than $k' \sim 10E+3$.

The interference caused by selected anions (sulfate and phosphate) were evaluated for Np(IV) (Figure 7). The capacity factor k' Np(IV) drops from $5 \times 10E+7$ to $2 \times 10E+4$ in 1M HCl and from $10E+6$ to $2 \times 10E+4$ in 3M HCl. There is thus a distinct impact of both anions on the retention, nevertheless, the k' remains greater than $10E+4$, strong Np retention is thus ensured even at sulfate or phosphate concentrations up to 4M.

The resin is used to concentrate actinides from leached soil samples (up to 5g of soil^{(1) (2)}), water samples (up to 100L^{(1) (2) (3)}) or urine⁽⁴⁾. For aqueous samples it was found that the extraction is quantitative for resin to sample ratios up to 250mg of resin/L of sample^{(1) (2)}; Eikenberg et al. established that a ratio of 400mg of resin/L is necessary for urine samples⁽⁴⁾.

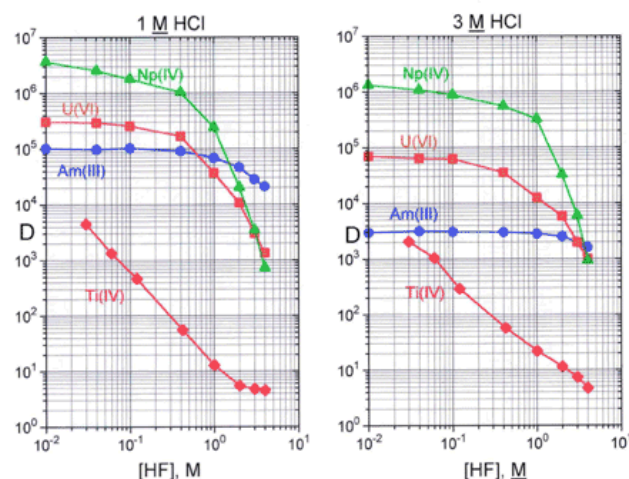


Figure 6 : k' values of different actinides on AC Resin in presence of HF in 1M and 3M HCl⁽¹⁾.

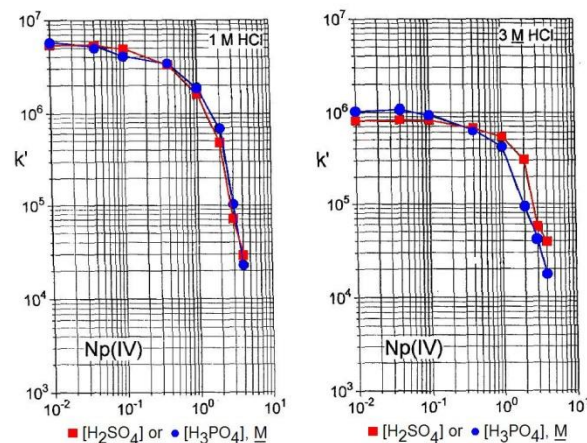


Figure 7 : k' values for Np(IV) on AC Resin in presence of H₂SO₄ and H₃PO₄ in 1M and 3M HCl⁽¹⁾.

The measurement of the actinides after their extraction can then be performed by different methods.

- In order to allow for an individual determination of the actinides the extractant (DIPEXTM), including the extracted actinides, is eluted using isopropanol. 7 to 15 mL of isopropanol are needed to quantitatively elute the extractant from 2 mL of resin (corresponding to one column). The isopropanol solution is then evaporated and the remaining extractant mineralized using 30% hydrogen peroxide and a

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sodium vanadate catalysator ⁽¹⁾. The final residue is redissolved in concentrated acid, evaporated and finally dissolved in the acid used as loading solution for the respective separation.

- Eikenberg et al. ⁽⁴⁾ also eluted the resin using isopropanol and mineralized the extractant in presence of concentrated acids (HCl or HNO₃) before transferring the dissolved residue into a scintillation vial. The authors obtained a detection limit of 1.5mBq/L with a counting time of 500 minutes and an initial sample volume of 500mL.

- In 2006 Croudace et al. ⁽³⁾ proposed a fusion based decomposition of the resin using lithium metaborate instead of wet digestion. The authors use this method in routine for the measurement of Pu and U in natural water samples. As result of the borate fusion at 1100°C they obtain a glass that is then dissolved in 8M HNO₃; Pu and U are then separated on a 1X8 type anion exchange resin followed by UTEVA resin. The chemical yields obtained using this method are in the order of >80% and the detection limit can be as low as 1mBq/L. The decomposition of the resin using this method takes only a few minutes, but the corresponding equipment and a Pt5%-Au crucible are needed.

The results obtained on the characterization of the resin and on its application to water samples were used to develop routine methods for the determination of the gross-alpha activity concentration of potable water by α/β discrimination liquid scintillation counting. The European Directive 98/83/EC imposes an effective dose of <0.1mSv/a for potable water. In order to control for this value it has been suggested to use, amongst others, the gross-alpha activity concentration as a screening parameter. In case the gross-alpha activity concentration of a sample surpasses 0.1Bq/L further analysis should be performed (activity concentration of contributing alpha emitters) and the obtained results used to calculate the effective dose of the respective water sample.

The water samples are acidified to pH 2 and a known quantity of resin is added to the sample (note: in order to assure quantitative Ra extraction the amount of resin needs to be adjusted for very Ca rich samples). The extraction of the alpha emitters is performed in batch mode under stirring for 30 min up to overnight. The resin is then filtered using a cellulose acetate filter and allowed to dry. It is then transferred, either with or without the filter, into a scintillation vial. A LSC cocktail is added and the sample is shaken vigorously. The cocktail dissolves the extractant from the resin (including the extracted cations) while the stripped

resin settles down at the bottom of the vial; the filter becomes translucent in contact with the cocktail and is thus not interfering with the subsequent LSC measurement. The described method allowed obtaining a detection limit of 30 mBq/L for a 4h counting time using a sample volume of 100 mL and its accuracy was shown by the successful participation to a OPRI intercomparison exercise ⁽⁵⁾

Kwakman ⁽⁵⁾ also tested his method on the cooling water of nuclear installations and found that numerous beta emitters were also extracted onto the resin. Studies on the use of the AC Resin for the determination of the gross alpha and beta activity are currently under way.

Finally the characterization of the resin by Horwitz et al. ⁽¹⁾ indicated that it might be possible to use it for the separation of radium and actinium. Both elements are retained at pH 1 – 2, Ra can then be eluted using 1M HCl (Figure 8).

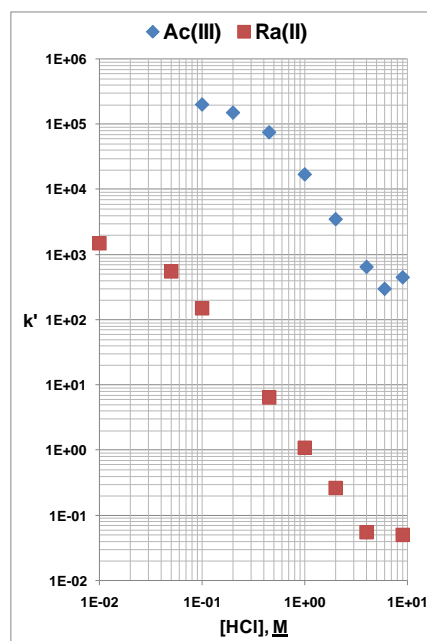


Figure 8 : k' values of Ra and Ac in HCl on AC Resin ⁽¹⁾

Another, very different, application of the AC Resin is the separation of beryllium from environmental and industrial matrices. Beryllium metal is frequently used in the nuclear industry due to its thermal and mechanical properties, as well as its capacity as neutron moderator and reflector.

Beryllium is toxic for lung tissue. The main uptake path is the inhalation of dusts or aerosols. The exposition to air with high Be concentration can lead to the acute beryllium disease. The exposition to low concentrations of Be over an extended periods can lead to the so called chronic

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beryllium disease (CBD); the symptoms can appear up to 30 years after the exposition. With respect to its toxicity its monitoring is of high importance in case of a risk of exposition.

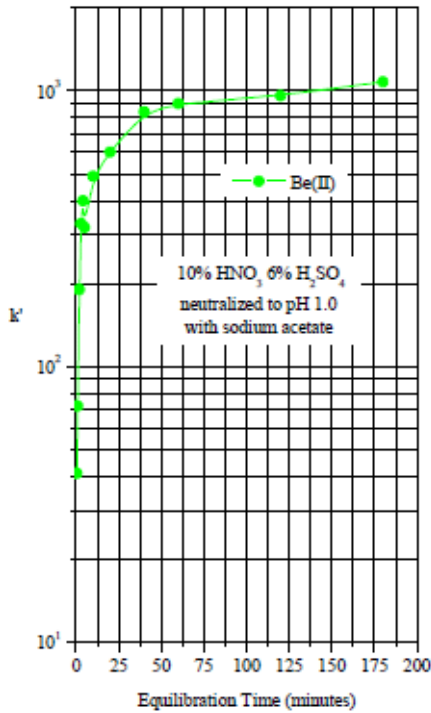


Figure 9 : Kinetic of the Be extraction onto AC Resin. Extraction conditions: Be solution 10%-HNO₃ - 6% H₂SO₄ stabilized at pH1 with sodium acetate; 22+/-2°C, 50-100µm resin.

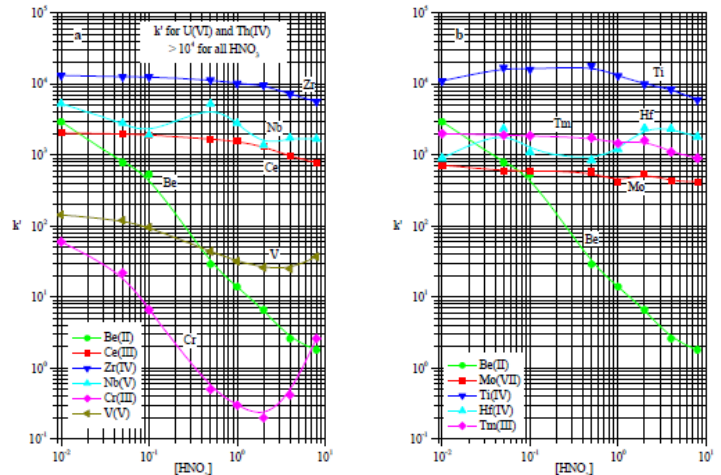


Figure 10 : k' values of elements spectrally interfering with Be in ICP-AES^{(6) (7)}.

Horwitz and McAllister^{(6) (7)} developed a fast and robust method for the determination of Be via ICP-AES in environmental samples and samples from the surveillance of industrial installations handling Be.

Figure 9 shows the extraction kinetics of Be, it indicates that the extraction equilibrium is reached after 40 minutes.

McAllister and Horwitz characterized the AC Resin not only for Be, but also for other elements that could interfere with the measurement of Be by ICP-AES (figure 10) or that could interfere with its extraction (figure 11).

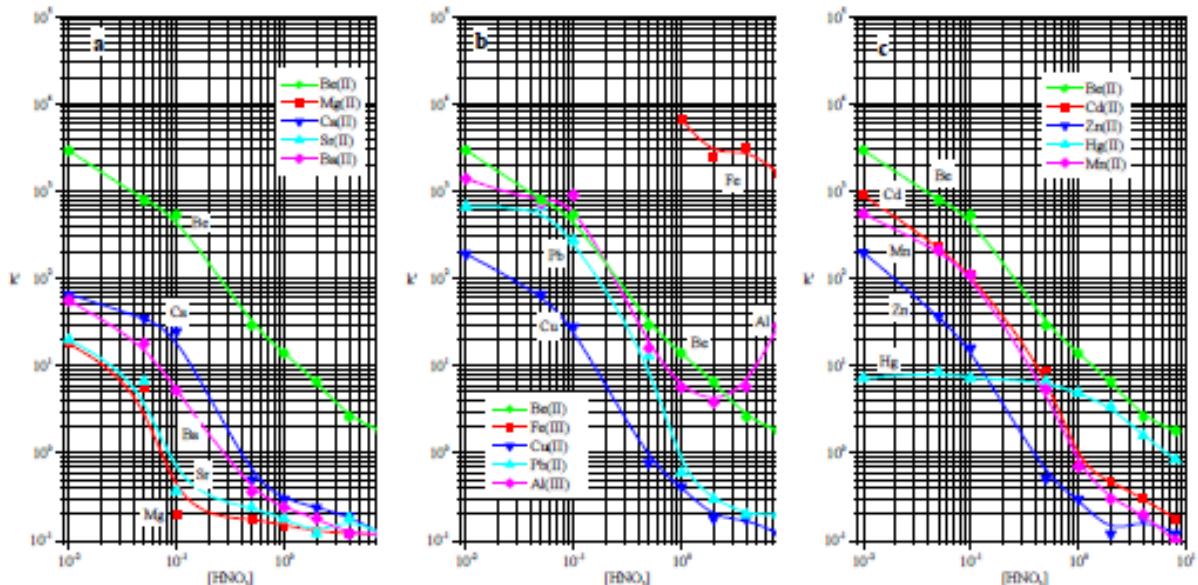


Figure 11 : k' values of elements showing a matrix interference with Be in ICP-AES^{(6) (7)}.

Be is strongly retained between pH 1 and pH 2 and can be eluted using nitric acid of a concentration greater than 1M. Zr, Nb, Ce, Ti, Hf,

Tm and Mo are strongly retained over the whole acid concentration range, these elements will not interfere with the Be measurement with ICP-AES (figure 10). Cr, Ca, Ba, Pb, Cu, Al, Cd, Mn, Zn and

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Hg are potential interferents for the retention/elution of Be on the AC Resin.

Following the obtained data the authors established a method for the separation of Be (figure 12):

The sample load solution containing $\text{HNO}_3/\text{H}_2\text{SO}_4/\text{H}_2\text{O}_2$ is stabilized at pH 1 to 2 using a 3.4M sodium acetate solution.

The resin is rinsed with 0.2M HNO_3 . Be is finally eluted with 4M HNO_3 at a flow rate of 1mL/min.

The chemical yield of the separation was reported to be greater than 90% for industrial and environmental samples. The authors applied their method amongst other to filter samples, for these they could find results in the range of 0.0428 to 0.1452 μg of Be per 100 cm^2 of filter material⁽⁹⁾.

One of the main interferences of beryllium is uranium. One method of separating Be and U is based on the use of LN2 or LN3 resin. Both resins contain phosphonyl groups which show a very high affinity for U(VI) over a large range of acid concentrations.

By using LN2 or LN3 as guard column upfront to the AC Resin they extract the U(VI) while Be passes and is then fixed on the AC Resin⁽⁶⁾ (Table 1).

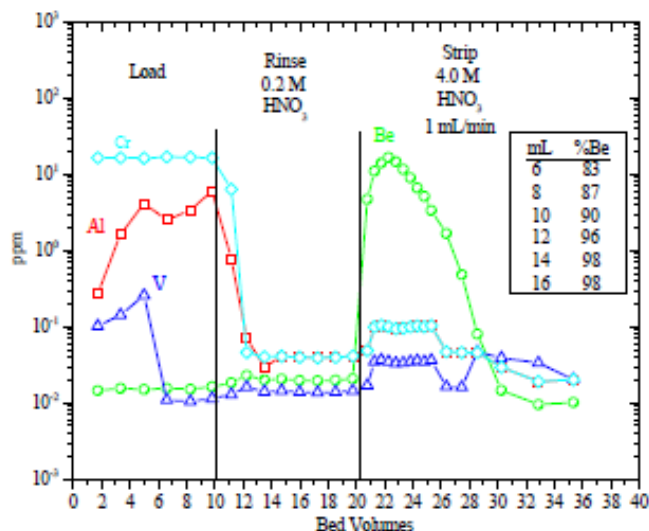


Figure 12 : Elution of Be, Al, Cr and V from a 2mL cartridge of AC Resin (50-100 μm) ; 22 \pm 1 $^\circ\text{C}$; matrix : ashless filter 5.5cm diameter spiked with 140 μg Al, Be, Cr and V – wet digested using H_2SO_4 and H_2O_2 , addition of 10mL HNO_3 and stabilization at pH 1 with a 3.4M sodium acetate solution.

| mg U | 2mL AC Resin | | 2mL AC Resin +2mL LN2 | | 2mL AC Resin +2mL LN3 | |
|------|---------------------------|--------------------------------|---------------------------|--------------------------------|---------------------------|--------------------------------|
| | % Be in 12mL ^b | μg U in Be fraction | % Be in 12mL ^b | μg U in Be fraction | % Be in 12mL ^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 |

Table 1 : Beryllium yields and uranium impurity vs mg Uranium in Load Solution

^a Whatman filter paper spiked with 0.14mg Be, digested with $\text{H}_2\text{SO}_4/\text{H}_2\text{O}_2$, and neutralized with sodium acetate to pH 1.8

^b AC resin strip solution 4.0 HNO_3

^c Detection limit for uranium by ICP-AES under experimental conditions

Bibliography

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