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A method for the separation of beryllium from spectral interfering elements in inductively coupled plasma-atomic emission spectroscopic analysis

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Abstract

A quick, simple and effective chromatographic method for the separation of beryllium from a wide range of elements is described. The elements selected comprise elements which can interfere with the determination of beryllium by inductively coupled plasma-atomic emission spectroscopy (ICP-AES) and elements which commonly occur in environmental and industrial sample matrices. Beryllium is separated from all possible ICP-AES spectral interfering elements using a single extraction chromatographic (EXC) cartridge containing an acidic chelating organophosphorus extractant, Dipex®, sorbed onto an inert polymeric substrate. The separation method has been evaluated using simulated samples generated using several different digestion methods currently employed in beryllium analyses performed in Department of Energy (DOE) facilities. Incorporating a guard cartridge containing either 2-ethyl-1-hexylphosphonic acid mono 2-ethyl-1-hexyl ester or bis(2,4,4-trimethylpentyl)phosphinic acid to selectively remove U(VI) allows the isolation of beryllium from samples containing over 100 mg of uranium without changing the load, rinse or strip conditions of the method.

Keywords: Chronic beryllium disease (CBD); Dipex®; ICP-AES; Extraction chromatography

1. Introduction

Beryllium metal, alloys and salts have been used widely in industry for decades. Beryllium is particularly valuable in specific areas of nuclear technology. Its ability to reflect neutrons and its efficiency in the production of neutrons when exposed to alpha emitters has led to its use in nuclear reactors and nuclear weapons [1]. However, the use of Be carries with it some serious health hazards, the most serious of which is chronic beryllium disease (CBD). Chronic beryllium disease is caused by the inhalation of beryllium compounds, particularly the oxide, and produces scarring of the lung tissue. The lung condition may take years (average 10–15 years) to develop symptoms [2–4]. The disease has no known cure and usually results in death. Over 100 current and former Department of Energy employees have CBD [5].

Because of the seriousness of CBD, the United States Department of Energy promulgated in 1999 the CBD prevention program, 10CFR Part 850, to protect DOE workers from exposure to Be-contaminated dust. This program requires frequent monitoring of air and possible contaminated surfaces to identify potential health risks. Therefore, there is an increased need for Be analysis in DOE facilities. The current analytical method of choice is inductively coupled plasma-atomic emission spectroscopy (ICP-AES) [6]. Samples, including air filters and surface smears, are digested using combinations of hydrochloric, hydrofluoric, nitric and/or sulfuric acid and hydrogen peroxide at elevated temperature to ensure that beryllium oxide or metallic beryllium is in an aqueous soluble state [7]. Beryllium is then determined by ICP-AES using the 313.107 and 313.034 nm lines in the atomic emission spectrum of beryllium. Although ICP-AES is an excellent method

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Table 1
Potential spectral interferences for Be determination by ICP-AES[a]

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

for the detection of beryllium because of the low detection limits achievable, a number of elements can cause spectral interferences (Table 1). Therefore, there is a need to separate Be from such elements. Because of the large number of samples that need to be analyzed, any separation must be simple, fast, and reliable.

Several methods including precipitation, ion-exchange and various colorimetric and fluorimetric techniques have been used to concentrate and/or determine beryllium in environmental samples [1]. The objective of this study was to develop a rapid and robust method for the isolation of beryllium from the wide range of matrices that could be encountered in samples currently generated in DOE facilities. Extraction chromatography has been demonstrated to be a fast and reliable method for the separation and preconcentration of a number of radionuclides and metal ions from a wide range of matrices [8]. After evaluating the uptake of beryllium and selected ICP-AES spectral interfering elements on several extraction chromatographic materials, an EXC material called Dipex®, containing a liquid diphosphonic acid extractant sorbed onto acrylic ester beads, was found to exhibit favorable metal ion retention characteristics for the isolation of beryllium [10,11]. A full characterization of the Dipex® resin for the separation of beryllium can be found in a separate publication by the authors [10]. In addition, since U(VI) has been identified as a particularly common and troublesome interference in the determination of beryllium by ICP-AES in DOE facilities [5], methods for dealing with samples containing high concentrations of U(VI) were explored.

2. Experimental

2.1. Reagents

The extraction chromatographic resin containing the Dipex® chelating organophosphorus extractant was obtained from Eichrom Technologies Inc. (Darien, IL). The Dipex® resin contains a liquid diphosphonic acid extractant, bis(2-ethylhexyl)methanediphosphonic acid (F.W. 400.41), sorbed onto Amberchrom CG-71 ms acrylic ester beads (50–100 μm). LN2 and LN3 resins were obtained from Eichrom Technologies Inc. and contain (2-ethyl-1-hexyl)phosphonic acid mono 2-ethyl-1-hexyl ester (HEH[EHP]) and bis(2,4,4-trimethylpentyl)phosphinic acid, respectively, sorbed onto Amberchrom CG-71 ms acrylic ester beads (50–100 μm). Hydrochloric, hydrofluoric, nitric and sulfuric acid solutions were prepared from trace metal grade acids (Fisher Scientific) using deionized water obtained from a Milli-Q2 water purification system. Sodium acetate, boric acid and 35% H_2O_2 were ACS grade (Aldrich). Metal ion solutions were prepared using single element atomic absorption standard solutions of 1000 or 10,000 ppm metal ion in water (Be, Nb, Cr, Mo), dilute nitric acid (U, Pb, Fe, Zr, Ce, Th, Tm, Ca) or dilute hydrochloric acid (V, Al, Ti, Hf) (Fisher Scientific).

2.2. Procedures

2.2.1. Determination of weight distribution ratios and capacity factors

The uptake of metal ions by Dipex® resin from acidic solutions was measured by contacting a known volume of solution (5.0–7.5 mL) with a known mass of resin (100–300 mg) in a borosilicate glass culture tube. Unless otherwise noted, all experiments were performed at 22(2) °C using 50–100 μm particle size resin beads. To limit the effect of consuming the capacity of the resin, the ratio of resin to aqueous phase and the metal ion concentrations were varied to produce a molar ratio of extractant to metal ion of at least 50:1. The mixing of resin and the aqueous phase was performed by intermittent agitation on a vortex mixer and by using a small magnetic stir bar rotated at a speed sufficient to keep the resin beads suspended in the aqueous phase. Following equilibration with the resin, the aqueous phase was filtered with a 0.45 μm PTFE filter to remove any resin particles.

Metal ion concentrations were determined by inductively coupled plasma-atomic emission spectroscopy on a Varian Liberty Series II sequential inductively coupled plasma-atomic emission spectrometer using the parameters listed in Table 2. In this study, beryllium was determined using the 234.861 nm peak of its atomic emission spectrum. While the 234.861 nm peak is less intense than the 313.042 and 313.107 nm peaks, the 234.861 nm peak is considerably

Table 2
Parameters employed for metal ion determination by ICP-AES

Analyte	Peak (nm)	Window (nm)	Analyte	Peak (nm)	Window (nm)
Al	396.152	0.080	Nb	316.340	0.040
Be	234.861	0.040	Pb	220.353	0.027
Ca	393.366	0.080	Th	283.730	0.040
Ce	413.765	0.080	Ti	334.941	0.080
Cr	267.716	0.040	Tm	313.126	0.040
Fe	259.940	0.040	U	385.958	0.080
Hf	277.336	0.040	V	292.402	0.040
Mo	281.615	0.040	Zr	343.823	0.040
Integration time (s)		3.00	Sample uptake delay (s)		30
Replicates		3	Pump rate (rpm)		15
Power (kW)		1	Instrument stabilization (s)		15
Viewing height (mm)		10	Rinse time (s)		30

freer of spectral interferences [6], and the intensity of the 234.861 nm peak is still sufficient for the concentrations of beryllium employed in this study. However, the beryllium separation method was developed to effectively isolate beryllium from all elements which could interfere with beryllium determination by ICP-AES using the 234.861, 313.042 or 313.107 nm lines in the beryllium atomic emission spectrum.

Preliminary experiments showed that the uptake of beryllium approached equilibrium after 40 min [10]. An equilibration time of 1 h was used in all determinations of weight distribution ratios. Weight distribution ratios (D_w) and cartridge capacity factors (k') were calculated as described in previous publications [9,10], using the following equations:

$$D_w = \frac{(A_0 - A_s)/w}{A_s/V} \tag{1}$$

$$D_v = \frac{D_w d_{extr}}{0.4} \tag{2}$$

$$k' = D_v \left(\frac{v_s}{v_m}\right) \tag{3}$$

where A_0 and A_s are the aqueous phase metal ion concentrations (parts per million) before and after equilibration, w is the weight of the resin in grams, V is the volume of aqueous phase in milliliters, d_{extr} is the density of the extractant (1.05 g/mL), 0.4 is the extractant loading in grams of extractant per gram of resin, and v_s (0.15) and v_m (0.66) are the volumes of stationary phase (extractant) and mobile phase, respectively, for a dry packed cartridge of Dipex® resin in mL per mL of resin. Duplicate experiments showed that the reproducibility of the D_w measurements was generally within 10%, although the uncertainty was somewhat higher for the highest D_w values (>10^3).

2.2.2. Determination of metal ion elution curves

A volume (10–20 mL) of solution containing 0.1–10,000 ppm of various metal ions, selected as possible spectral interferences in the determination of beryllium by ICP-AES or as possible matrix interferences in the uptake of beryllium on Dipex® resin, was loaded onto a 2 mL dry packed cartridge of Dipex® resin (50–100 μm particle size, 9 mm inner diameter, 30 mm cartridge height). The cartridge was then rinsed with dilute HNO_3 and stripped with 3–5 M HNO_3 to remove beryllium or 0.2 M tetrasodium-EDTA to remove metal ions which are strongly absorbed from high concentrations of HNO_3. Unless otherwise noted, all experiments were performed at 22(1) °C using 2 mL/min flow rates. Fractions of the eluate were collected into tared 15 mL polypropylene centrifuge tubes and diluted to 5 mL with deionized water. Metal ion concentrations in the diluted eluate solutions were then determined by ICP-AES using atomic emission lines of the highest intensity and/or having the fewest spectral interferences. The concentration of beryllium determined by ICP-AES was found to vary as the matrix of the beryllium containing solution was changed [10]. Therefore, the beryllium concentrations determined by ICP-AES were normalized to the 4.0 M HNO_3 matrix used for the ICP-AES calibration standards. In some experiments, a 2 mL dry packed cartridge of LN2 or LN3 resin was used as a guard cartridge on top of the Dipex® resin cartridge.

2.2.3. Preparation of simulated samples

Circles of ashless filter paper (5.5 cm in diameter) and GHOSTWIPES® sampling wipes were spiked with 1.4 μg–100 mg of selected metal ions and digested using combinations of hydrochloric, hydrofluoric, nitric and/or sulfuric acid and H_2O_2 as specified in various digestion protocols employed in the DOE complex [7]. Following digestion, the samples were diluted to 10 mL with deionized water and neutralized with 3.5 M sodium acetate to pH 1–4.5 (pH meter). The final volume of the solution was then brought to 20 mL with deionized water. Some samples generated using hydrofluoric acid in the digestion process were also made 0.1 M in boric acid to scavenge any traces of fluoride remaining following the digestion [12]. Some of the digestion methods evaluated did not completely digest the filter paper, leaving solid matter suspended in the final 20 mL neutralized solution. Loading samples in which the undissolved filter paper is suspended can result in reduced flow rates across the extraction chromatographic resin. Therefore, samples were centrifuged at 1500 rpm for 5 min and the supernatant decanted to separate the solution from any solid material. Samples containing undissolved solids can also be filtered. Based on beryllium mass balances (95–105%) obtained during experiments, beryllium does not appear to preferentially partition with the undissolved filter material. A thorough evaluation of digestion methods employed by the DOE complex in the determination of beryllium can be found in [7].

3. Results and discussion

3.1. Uptake of Be(II) and ICP-AES spectral interferences on Dipex® resin

Fig. 1 depicts the uptake (k', column capacity factor) on Dipex® resin versus HNO_3 of Be(II) and metal ions identified as potential spectral interferences in the determination of beryllium by ICP-AES (Table 1). A more complete evaluation of the uptake of Be(II) and potential ICP-AES spectral interferences and matrix interferences can be found in [9,10]. The data in Fig. 1, suggest that the separation of Be(II) from the potential ICP-AES interferences should be possible by loading a Dipex® resin cartridge with a solution of pH 1–2, from which Be(II), and many of the spectral interferences will be retained. The neutralized digestion solution containing any Cr present in the sample will pass through the cartridge. Rinsing the cartridge with a small volume of dilute nitric acid (0.1–0.2 M HNO_3) should remove any traces of weakly retained species and stripping the cartridge with 3–5 M HNO_3 will produce the Be, free of any of the ICP-AES spectral interfering elements.

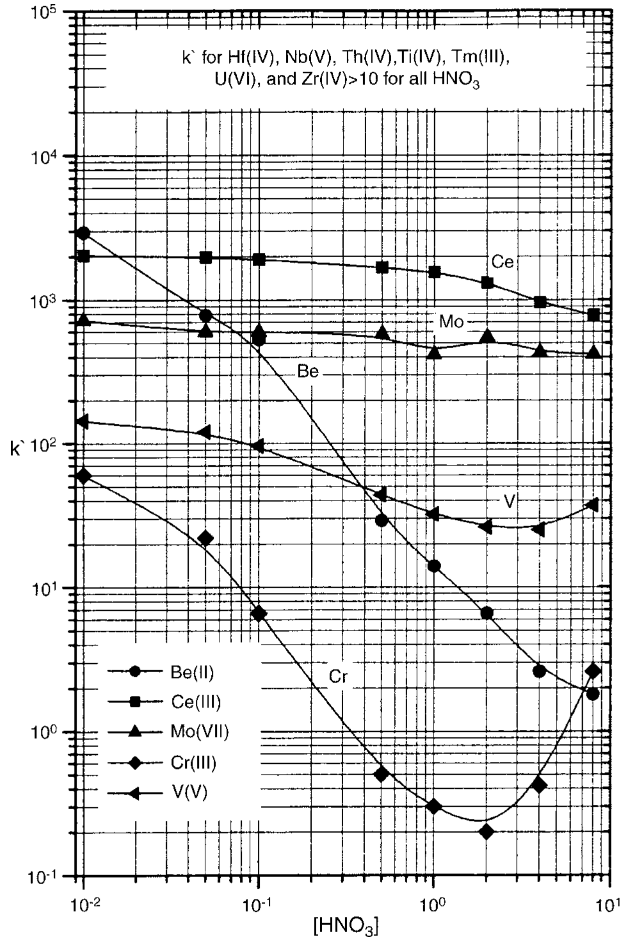

Fig. 1. k' for beryllium and potential ICP-AES spectral interferences on Dipex® resin vs. HNO_3, 1 h equilibration time, 22(1) °C, 50–100 μm particle size.

3.2. Elution curves for Be(II) and spectral and matrix interferences on Dipex® resin

3.2.1. Sulfuric acid/H_2O_2 digestion

Figs. 2–4 depict data for the elution of Be and all of the potential ICP-AES spectral interfering elements from simulated samples digested using the sulfuric acid/H_2O_2 digestion method employed in beryllium analyses at the Y-12 national security complex [5]. In each of the three figures, beryllium is effectively separated from >99% of all the potential ICP-AES spectral interfering elements and recovered in 91–98% yield in 10–12 mL of 4 M HNO_3. Following the 4 M HNO_3 strip of the cartridge, a small amount of water (2 mL) was used to rinse the HNO_3 from the cartridge and then 0.2 M Na_4EDTA was used to remove Ce(III), Hf(V), Nb(V), Mo(VII), Th(IV), Ti(IV), Tm(III), U(VI) and Zr(IV) to verify that they were present in the load solution and retained by the Dipex® resin. Previous experiments have shown that 4 M HNO_3 provides the most effective stripping of beryllium from the Dipex® resin [10]. The use of ashless filter paper or GHOSTWIPES® sampling wipes did not affect the separation or the recovery of beryllium.

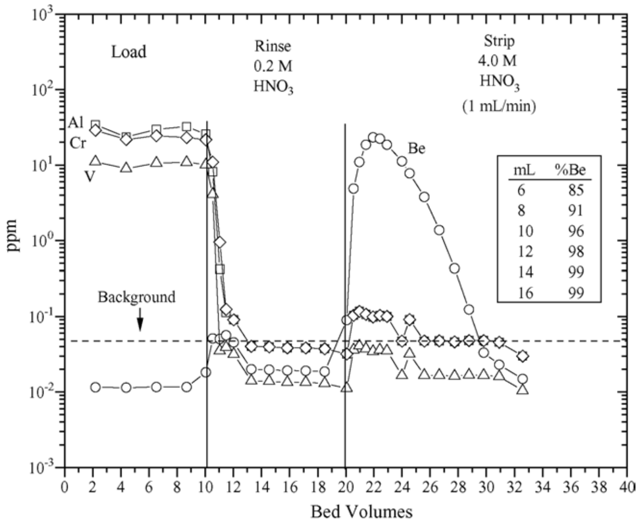

Fig. 2. Elution of Be, Al, Cr and V on a 2 mL dry packed cartridge of Dipex® resin, 50–100 μm particle size, flow rate 2 mL/min, 22(1) °C, load: 5.5 cm ashless filter spiked with 140 μg Be, Al, Cr and V, digested with H_2SO_4 and H_2O_2, diluted to 10 mL with HNO_3 and neutralized to pH 1 with 3.4 M sodium acetate.

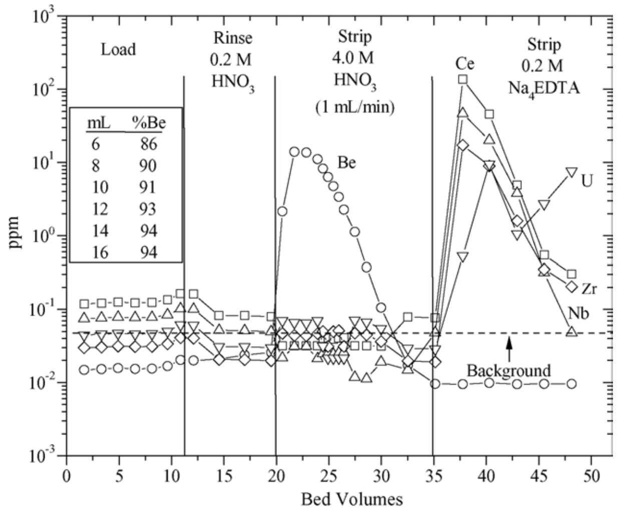

Fig. 3. Elution of Be, Ce, U, Zr and Nb on a 2 mL dry packed cartridge of Dipex® resin, 50–100 μm particle size, flow rate 2 mL/min, 22(1) °C, load: 5.5 cm ashless filter spiked with 140 μg Be, Ce, U, Zr and Nb, digested with H_2SO_4 and H_2O_2, diluted to 10 mL with HNO_3 and neutralized to pH 1 with 3.4 M sodium acetate.

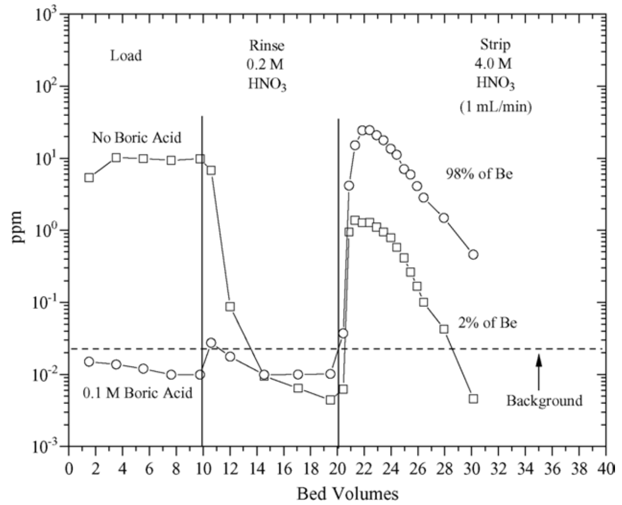

Fig. 5. Elution of Be on a 2 mL dry packed cartridge of Dipex® resin, 50–100 μm particle size, flow rate 2 mL/min, 22(1) °C, load: 5.5 cm ashless filter spiked with 140 μg Be, digested with HCl, HNO_3, HF and H_2O_2, diluted to 10 mL with deionized water and neutralized to pH 1 with 3.4 M sodium acetate, boric acid added to one sample to sequester fluoride.

3.2.2. HCl, HNO_3, HF, H_2O_2 digestion

Fig. 5 depicts the elution of beryllium on Dipex® resin from simulated samples generated using a HCl, HNO_3, HF, and H_2O_2 digestion protocol similar to those used during beryllium analyses performed by several laboratories in DOE facilities [7], with and without the addition of boric acid to scavenge fluoride. As can been seen in Fig. 5, the addition of boric acid is vital to the success of the beryllium separation method from samples containing the fluoride ion. Without the addition of boric acid to the neutralized digestion solution, residual fluoride present following the digestion complexes the beryllium [13], preventing its uptake on by the Dipex® resin.

The formation of beryllium–fluoride complexes leads to a loss of 98% of the beryllium during the load and rinse. When boric acid is added to the load solution, any traces of fluoride ion are scavenged through the formation of fluoroboric acid [12] and pass through the cartridge during the load and rinse. Beryllium is retained by the Dipex® resin cartridge and recovered in 92–97% yield in 10–12 mL of the 4 M HNO_3 strip. The elution profiles of the potential ICP-AES spectral interfering elements were also determined, but have been omitted for brevity. However, as in Figs. 2–4, when boric acid is used to scavenge fluoride ions remaining following the digestion, beryllium is effectively separated from >99% of all of the potential ICP-AES spectral interferences.

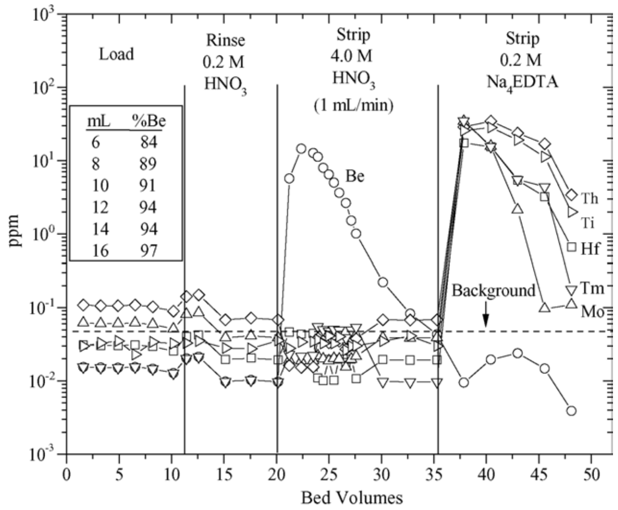

Fig. 4. Elution of Be, Th, Ti, Hf and Mo on a 2 mL dry packed cartridge of Dipex® resin, 50–100 μm particle size, flow rate 2 mL/min, 22(1) °C, load: 5.5 cm ashless filter spiked with 140 μg Be, Th, Ti, Hf and Mo, digested with H_2SO_4 and H_2O_2, diluted to 10 mL with HNO_3 and neutralized to pH 1 with 3.4 M sodium acetate.

Table 3
Capacity of Dipex® resin for selected metal ions

Metal ion	Dipex® capacity in mg/2 mL bed	mg to reduce Be yield to 90%[c]
Cr(VI)	N/A	>100
Mo(VI)	N/A	25
U(VI)	102.6[a]	25
Ti(IV)	12.5[b]	7.5
Th(IV)	60.6[a]	10
Fe(III)	22.6[a]	10
Pb(II)	53.8[b]	50
Ca(II)	10.4[a]	>100
Be(II)	0.9[b]	0.5

[a] From [9].

[b] Calculated from data in [9].

[c] 5.5 cm ashless filter, digested with H_2SO_4/H_2O_2, neutralized to pH 2 with 3.4 M sodium acetate, diluted to 20 mL.

Table 4
Beryllium yields and uranium impurity vs. mg uranium in load solution[a]

mg U	2 mL beryllium resin		2 mL beryllium resin + 2 mL LN2		2 mL beryllium resin + 2 mL LN3	
	% 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 resinstrip solution 4.0 M HNO_3.
[c] Detection limit for uranium by ICP-AES under the experimental conditions.

3.3. Capacity of Dipex® resin

Many of the metal ions which are potential ICP-AES spectral interferences in the determination of beryllium or may be present as matrix constituents are strongly retained on the Dipex® resin (Table 3). Therefore, large quantities of these metal ions could interfere with the uptake of beryllium on the Dipex® resin. The capacities of the Dipex® extraction chromatographic resin for selected metal ions have been determined previously [9] and are listed, along with the amount of these metal ions which have been observed to decrease the recovery of beryllium to <90% using a 2 mL cartridge of the Dipex® resin. If samples routinely contain high amounts of these strongly retained metal ions, larger columns of the Dipex® resin can be employed during the beryllium separation or an appropriate guard column can be used to selectively remove the problematic impurity (vide infra). Methods for separating beryllium from large quantities of impurities such as Fe, Mo, Cr and Ti are currently being explored.

3.4. Separation of beryllium from samples containing high uranium concentrations

Uranium has been identified as a particularly important impurity encountered during analysis of beryllium by the Y-12 national security complex, with samples occasionally containing up to 100 mg of uranium [5]. A method to effectively isolate beryllium from samples containing high levels of uranium was therefore explored.

Previous studies by the authors have shown that extraction chromatographic materials containing substituted phosphonic or phosphinc acids selectively retain U(VI) over Be(II) from a wide range of acidities [11]. As shown in Table 4, incorporating a 2 mL guard column of LN2 or LN3 resin, containing 40 wt% 2-ethyl-1-hexylphosphonic acid mono 2-ethyl-1-hexyl ester and bis(2,4,4-trimethylpentyl)phosphinic acid, respectively, beryllium can be effectively isolated in high yield from solutions containing up to 100 mg of U(VI) without changing the load, rinse or strip conditions of the beryllium separation method. Without the incorporation of the guard column, decreased beryllium yields are observed when greater than 25 mg of U(VI) are present in the sample.

3.5. Monitoring the pH of the load solution

Since the uptake of beryllium on Dipex® resin is poor from highly acidic solutions, a simple method of monitoring the pH of the neutralized digested filter solution loaded onto the Dipex® resin is necessary to ensure optimal performance of the beryllium separation method. The use of a pH meter would provide a very precise control of pH. However, using a pH meter could lead to cross contaminating samples and may be too time consuming given the large number of samples which need to be analyzed [5]. Therefore, the use of pH indicators (methyl violet or crystal violet [14]) or pH papers should be used to ensure that the load solution has a pH of >1. Additionally, since sodium acetate will buffer the solution to a maximum pH of 4.5, the addition of a consistent excess of sodium acetate could be a way of ensuring a load solution of pH >1, without the risk of extensive hydrolysis of the metal ion species possible if a strong base was used to neutralize the samples.

4. Summary and conclusions

A simple and effective method for the separation of beryllium from potential ICP-AES spectral interfering elements using a single extraction chromatographic column containing the Dipex® chelating organophosphorus extractant has been developed. The method can be tailored to the varied digestion protocols employed in Department of Energy facilities. Beryllium can be effectively isolated from samples containing up to 100 mg of uranium using a single 2 mL cartridge of the Dipex® resin equipped with a 2 mL guard column containing either a substituted phosphonic or phosphinic acid.

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