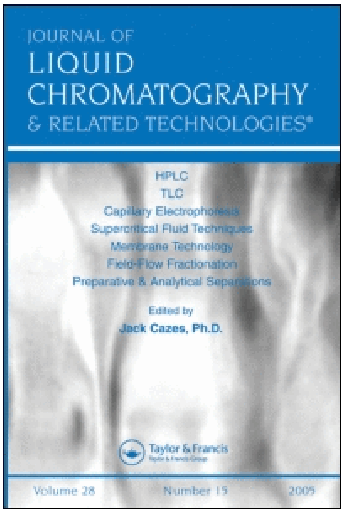

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Use of Zirconium(IV) Arsenophosphate Columns for Cation Exchange Separation of Metal Ions Interfering in the Spectrophotometric Determination of Uranium with Sodium Diethyl Dithiocarbamate

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USE OF ZIRCONIUM(IV) ARSENOPHOSPHATE COLUMNS FOR CATION EXCHANGE SEPARATION OF METAL IONS INTERFERING IN THE SPECTROPHOTOMETRIC DETERMINATION OF URANIUM WITH SODIUM DIETHYL DITHIOCARBAMATE

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ABSTRACT

A simple cation exchange method has been developed for the quantitative separation of uranium from some metal ions which generally interfere in its spectrophotometric determination using sodium diethyl dithiocarbamate as a reagent. The method requires only a single bed operation and enables a satisfactory (Error ± 5%) separation of uranium [UO_2(II)] upto 1080 µg from ten metal ions on a 2 g column of zirconium(IV) arsenophosphate cation exchanger in H(I) form.

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INTRODUCTION

Spectrophotometric methods are commonly used for the determination of metal ions, but they generally require a prior separation of interferences to improve their efficiency. Liquid-liquid extraction and ion-exchange methods are widely used for this purpose. The former methods have limitations in selecting a suitable solvent, while the latters employ mostly organic resins which are not highly selective for metal ions. Inorganic ion exchangers have an advantage in this regard over their organic counterparts in addition to their higher radiation and thermal stabilities (1). Therefore, they can be used more beneficially in separations of radiochemical and analytical importance (2,3). The present work is in continuation of our such an effort (4-12) in this direction which describes a cation exchange method for the removal of interferences generally encountered in the spectrophotometric determination of uranium (13-22),using zirconium(IV) arsenophosphate. This material has already shown earlier an excellent ion-exchange behaviour (5).

MATERIALS AND METHODS

Reagents and Chemicals

All the reagents and chemicals used in this study were of AnalaR grade obtained from the B.D.H. Poole (England) or E.Merck (Darmstadt).

Apparatus

A Bausch and Lomb Spectronic 710 spectrophotometer was used for colorimetry.

Synthesis of the Ion-Exchange Material

Zirconium(IV) arsenophosphate was prepared by the method reported earlier (5) and was heated at 200°C for an hour to obtain its modified phase useful for the present study.

Separation and Determination of Uranium

Two grams of the uniformly sized ion exchanger particles (60-100 mesh) in H^+ form were used for the column operation in a glass tube having an internal diameter of $\sim$ 0.6 cm. The column was washed thoroughly with 4M HNO_3 ($\sim$250 ml) to remove any metallic impurities and then with demineralized water (DMW). The sample solution was then loaded on the top of the column very slowly (Flow rate $\approx$ 0.2 ml/min) and the elution was made with 0.01M HNO_3 (Flow rate $\approx$ 0.5 ml/min) which removed all the metal ions except UO_2(II). The metal ions thus collected in the effluant were determined volumetrically as usual by EDTA. UO_2(II) was ultimately eluted out with 50 ml of 1M HNO_3. This fraction of the effluent was evaported to dryness and the residue dissolved in $\sim$2 ml of DMW to which were added 0.5 ml of an aqueous (2%) sodium diethyl dithiocarbamate solution and methanol to make the final volume 25 ml. Its absorbance was measured after 5 minutes at 380 nm against the reagent blank which gives the amount of UO_2(II) on the basis of a previously drawn calibration curve. The results are summarized in Tables 1 and 2 for the separation of uranium from some binary and multimineral synthetic mixtures respectively.

TABLE 1

Separation of Uranium from Some Binary Mixtures Using Zirconium(IV) Arsenophosphate Columns

Sl. No.	Separation	Amount loaded (µg)	Amount found* (µg)	% Error
1.	$Ca(II)-UO_2(II)$	200.0 Ca(II) 810.0 $UO_2(II)$	204 Ca(II) 796.5 $UO_2(II)$	+2.0 Ca(II) -1.7 $UO_2(II)$
2.	$Mg(II)-UO_2(II)$	121.5 Mg(II) 810.0 $UO_2(II)$	122.3 Mg(II) 796.5 $UO_2(II)$	+0.66 Mg(II) -1.7 $UO_2(II)$
3.	$Sr(II)-UO_2(II)$	438.0 Sr(II) 810.0 $UO_2(II)$	434.2 Sr(II) 810.0 $UO_2(II)$	-0.90 Sr(II) 0.00 $UO_2(II)$
4.	$Ba(II)-UO_2(II)$	687.0 Ba(II) 810.0 $UO_2(II)$	678.0 Ba(II) 815.4 $UO_2(II)$	-1.3 Ba(II) +0.67 $UO_2(II)$
5.	$Mn(II)-UO_2(II)$	275.0 Mn(II) 810.0 $UO_2(II)$	272.6 Mn(II) 783.0 $UO_2(II)$	-0.73 Mn(II) -3.3 $UO_2(II)$
6.	$Co(II)-UO_2(II)$	295.0 Co(II) 810.0 $UO_2(II)$	294.1 Co(II) 800.4 $UO_2(II)$	-0.34 Co(II) -1.2 $UO_2(II)$
7.	$Ni(II)-UO_2(II)$	294.0 Ni(II) 810.0 $UO_2(II)$	292.7 Ni(II) 815.4 $UO_2(II)$	-0.34 Ni(II) +0.67 $UO_2(II)$
8.	$Zn(II)-UO_2(II)$	327.0 Zn(II) 810.0 $UO_2(II)$	325.9 Zn(II) 810.0 $UO_2(II)$	-0.31 Zn(II) 0.00 $UO_2(II)$
9.	$Cu(II)-UO_2(II)$	318.0 Cu(II) 810.0 $UO_2(II)$	326.2 Cu(II) 796.5 $UO_2(II)$	+2.6 Cu(II) -1.7 $UO_2(II)$
10.	$Cr(III)-UO_2(II)$	260.0 Cr(III) 810.0 $UO_2(II)$	258.6 Cr(III) 800.4 $UO_2(II)$	-0.38 Cr(III) -1.2 $UO_2(II)$

*Mean value of five replicates.

TABLE 2

Separation of Uranium from Some Multimineral Synthetic Mixtures Using Zirconium(IV) Arsenophosphate Columns

Sl. No.	Composition of the mixture loaded (µg)	Amount of $UO_2(II)$ found* (µg)	% Error
1.	40 Ca(II), 24.3 Mg(II), 87.6 Sr(II), 137.4 Ba(II), 55 Mn(II), 59 Co(II), 58.8 Ni(II), 65.4 Zn(II), 63.6 Cu(II), 52 Cr(III), 270 UO_2(II)	270.0	0.00
2.	80 Ca(II), 48.6 Mg(II), 175.2 Sr(II), 274.8 Ba(II), 110 Mn(II), 118 Co(II), 117.6 Ni(II), 130.8 Zn(II), 127.2 Cu(II), 104 Cr(III), 540 UO_2(II)	533.3	-1.2
3.	120 Ca(II), 72.9 Mg(II), 262.8 Sr(II), 412.2 Ba(II), 165 Mn(II), 177 Co(II), 176.4 Ni(II), 196.2 Zn(II), 190.8 Cu(II), 156 Cr(III), 810 UO_2(II)	783.0	-3.3
4.	160 Ca(II), 97.2 Mg(II), 350.4 Sr(II), 549.6 Ba(II), 220 Mn(II), 236 Co(II), 235.2 Ni, 261.6 Zn(II), 254.4 Cu(II), 208 Cr(III), 1080 UO_2(II)	1027	-4.9
5.	200 Ca(II), 121.5 Mg(II), 438 Sr(II), 687 Ba(II), 275 Mn(II), 295 Co(II), 294 Ni(II), 327 Zn(II), 318 Cu(II), 260 Cr(III), 1350 UO_2(II)	1215	-10
6.	200 Ca(II), 121.5 Mg(II), 438 Sr(II), 687 Ba(II), 275 Mn(II), 295 Co(II), 294 Ni(II), 327 Zn(II), 318 Cu(II), 260 Cr(III), 810 UO_2(II)	796.5	-1.7

*Mean value of five replicates.

RESULTS AND DISCUSSION

The salient feature of the present study is to use an inorganic ion exchanger for the removal of the interfering metal ions in the spectrophotometric determination of uranium with sodium diethyl dithiocarbamate. Zirconium(IV) arsenophosphate cation exchanger has been used as an adsorbent because of its high selectivity for UO_2(II). The method is simple and rapid requiring only a single bed operation. As the results show (Tables 1 and 2), a satisfactory separation (Error range ≈ ± 5%) upto 1080 µg of UO_2(II) is achieved on a 2 g column of the ion exchanger with a standard deviation $< 0.5\%$. Even a higher amount of uranium can be separated from other metal ions if the amount of the exchanger is increased in the column. The method requires pH adjustment of the analyte as 6-7 which eliminates certain metal ions such as Ti(IV), Sb(V) and Bi(III) due to their rapid hydrolysis under such a condition. Ag(I) if present, in the sample solution can be removed by treating the solution with dil.HCl before its loading on the exchanger bed.

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