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A Rapid Method for the Extraction and Separation of Uranium from Thorium and Other Accompanying Elements Using Octadecyl Silica Membrane Disks Modified by Tri-*n*-octyl Phosphine Oxide

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ABSTRACT

A simple method for rapid and selective separation of uranyl ion from Th^{4+} and other accompanying elements by using octadecyl silica membrane disks modified with tri-*n*-octyl phosphine oxide (TOPO) in the presence of 0.5 M HNO_3 is presented. The influences of type and concentration of acid, nature of ligands, flow rates, and nature of stripping agents have been investigated. Maximum capacity of the membrane disks modified by 50 mg TOPO was found to be 938 μg of uranium. The method was applied to the recovery of uranyl ion from two different soil samples.

Key Words. Uranium separation; SPE; Octadecyl silica disks; TOPO; Spectrophotometric determination

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INTRODUCTION

Uranium, an element with the highest atomic number of any naturally occurring element, is found in granites, metamorphic rocks, lignite, monazite sands, and phosphate deposits as well as minerals such as uraninite, carnotite, and pitchblende. The concentration in phosphate rocks can be as high as 0.12 mg/g (1). It is only seldom that a given method for the determination of uranium can be applied without the preliminary separation of interfering elements.

Coprecipitation with cerous hydroxide, barium sulfate, iron hydroxide, and aluminum hydroxide (2–6); liquid–liquid extraction (LLE) with tri-*n*-octyl phosphine oxide (TOPO) (7–10]), tributyl phosphate (11–13), crown ethers (14, 15) and other ligands (16–19); ion-exchange separation (20–27); solid phase extraction (SPE) (28, 29); and supercritical fluid extraction (30) are among the most suitable reported methods for the separation of small amounts of uranium. However, most of these methods suffer from such problems as the need for large amounts of ligands, reagents, and high purity solvents; disposal of organic solvents; and long procedural time.

Metal chelating resins (adsorbed or chemically bonded chelates) have recently been found to be of great utility for the preconcentration and separation of trace metals from different matrices (31–42). The use of supports loaded with chelating agents is particularly convenient because such resins are easy to prepare although their stabilities and collection abilities may be inferior to those of chelating resins containing various functionalized groups (40). In a previous publication (41) we showed that separation of barium from alkali and alkaline earth metal ions is feasible with octadecyl silica membrane disks modified with crown ethers. The aim of this work is the development of a rapid and efficient method for the extraction and separation of uranium from associated elements using octadecyl silica membrane disks modified with TOPO. The influences of experimental parameters such as type of adsorbed ligands, concentration of different acids, type and volume of suitable eluents, and eluent flow rates on the extraction efficiency of the modified membrane disks were investigated. The method has also been extended to the separation of uranium from a large number of other elements and also from a number of real samples.

EXPERIMENTAL

Reagents

Reagent-grade dibenzo-18-crown-6 (DB18C6), tri-*n*-octyl phosphine oxide (TOPO), and arsenazo(III) (all from Merck) were used as received. Analytical-grade picric acid, oxalic acid, $UO_2(NO_3)_2 \cdot 6H_2O$, $Na_2MoO_4 \cdot 2H_2O$,

NH_4HCO_3, $NaVO_3 \cdot H_2O$, and nitrate or chloride salts of other cations (all from Merck) were of the highest purity available and were used without any further purification except for vacuum drying. All solvents and acids used were of AnalaR grade from Merck.

A stock solution of uranium (1.0 mg/mL) was prepared by dissolving proper amounts of $UO_2(NO_3)_2 \cdot 6H_2O$ in 6 M HCl in a 100-mL flask. Dilute solutions containing 100 μg/mL of U(VI) were prepared by appropriate dilution of the stock solution in doubly distilled deionized water. The standard mineral and sediment samples were obtained from the International Atomic Energy Agency (Australia).

Apparatus

All absorbance measurements were carried out on a Cary 3 Varian spectrophotometer. A Varian-20 atomic absorption spectrophotometer was used for metal ion measurements under the recommended conditions for each metal ion.

Sample Extraction

Extractions were performed with 47 mm diameter × 0.5 mm thickness 3M Empore membrane disks containing octadecyl-bonded silica (8 μm particles, 60 Å pore size) from Varian. The disks were used in conjunction with a standard Millipore 47 mm filtration apparatus.

Before extraction, each membrane was washed with 10 mL methanol, 10 mL 1 M HCl, 25 mL deionized water, and 10 mL methanol to remove all contaminants arising from the manufacturing process and environment. After drawing air through the disk for several minutes it was placed inside an oven at 65°C. Then a solution of 50 mg TOPO dissolved in 2 mL methanol was introduced onto the disk surface and allowed to penetrate inside the membrane completely. The solvent was evaporated at 65°C. The membrane disk modified by TOPO was now ready for sample extraction.

The general procedure for the extraction of uranyl ions on the membrane disk was as follows. First, the modified disk was preconditioned by passing a 10-mL portion of a 0.5 M HNO_3 solution through it. Then 10 mL of the sample solution containing 50 μg U(VI) and 0.5 M nitric acid was passed through the modified disk (flow rate = 20 mL/min). The extracted uranyl ion was then stripped from the disk using 90 mL of 0.5 M solution of NH_4HCO_3 (flow rate = 4 mL/min). Fractions were collected and completely evaporated to dryness, and the uranium content was determined by the Arsenazo(III) method (21).

RESULTS AND DISCUSSION

In some preliminary experiments which were carried out to investigate the quantitative extraction of UO_2^{2+} ions by the octadecyl silica membrane disks in the absence and presence of such potential modifying ligands as DB18C6 and TOPO, the following results were obtained. First, the plane membrane disk did not show any tendency to extract uranyl ion from sample solutions. On the other hand, the recovery data obtained by a membrane disk modified by DB18C6 in the presence of 0.01 M picric acid revealed the retention, at most, of about 40% uranium. However, it was found that a membrane disk modified by TOPO is capable of completely retaining UO_2^{2+} ions in the sample solutions (the test solutions contained 50 μg uranyl ion in 10 mL 0.5 M HNO_3).

In the next step we studied the extraction of uranyl ions by modified membrane disks from different acid media. Table 1 shows that quantitative extraction of uranyl ion is achieved from solutions containing 0.5–1 M HNO_3 or 0.5 M HCl. The extraction of uranyl ions from dilute HNO_3 solutions by alkyl esters of acid phosphoric such as tributyl phosphate (TBP) is reported to be based on the formation of a neutral complex of $UO_2(NO_3)_2 \cdot 2TBP$ (43). Thus, the possible composition of the adsorbed complex should be $UO_2X_2 \cdot 2TOPO$ ($X = NO_3^-, Cl^-$). The decreased extraction of uranyl ion from sulfuric acid media may be due to the formation of such anionic complexes as $UO_2(SO_4)_2^{2-}$ and $UO_2(SO_4)_3^{4-}$ with overall formation constants of $\beta_2 = 1.75 \times 10^4$ and $\beta_3 = 4.38 \times 10^7$, respectively (44). Further extraction of uranyl ion was then performed from 0.5 HCl or HNO_3 solutions.

It is obvious from Table 1 that by increasing the HCl concentration in the solution, the percent recovery of uranyl ion is largely reduced, most probably due to the formation of $UO_2Cl_3^-$ ion (44). Thus, we found that a solution of 6 M HCl is suitable for the successful elution of uranyl ion from the disk. The results showed that 70 mL of 6 M HCl solution is necessary for complete elu-

TABLE 1
Percent Recovery of 50 μg U(VI) with the Modified Membrane Disk Using Different Concentrations of Various Acids

Acid	Concentration (M)	% Recovery
HCl	0.5	99.1
HCl	1.0	97.2
HCl	6.0	NR[a]
HNO_3	0.5	100.0
HNO_3	1.0	100.0
HNO_3	2.0	97.3
H_2SO_4	0.05	86.9
H_2SO_4	0.2	89.1
H_2SO_4	0.5	93.5

[a] Not retained.

TABLE 2
Percent Recovery of 50 μg U(VI) from the Modified Membrane Disk Using 60 mL of Varying Concentrations of NH_4HCO_3 at Different pH Values

Concentration (M)	pH	% Recovery
0.25	8.35	70.3
0.50	8.25	91.8
0.50	7.00[a]	85.6
0.50	5.80[a]	80.4
1.00	8.02[a]	79.4
2.00	7.77[a]	28.3

[a] pH adjustments were made by addition of proper amounts of concentrated HCl.

tion of uranyl ion from the disk. However, because of the hazardous effects of HCl, we decided to examine different concentrations of ammonium bicarbonate as the eluent for adsorbed uranyl ions.

Table 2 shows that a 0.5 M NH_4HCO_3 solution at a pH 8.25 is suitable for the elution of uranyl ions from the disk. It should be noted that the carbonate ion forms a very stable anionic complex with uranyl ion, $UO_2(CO_3)_3^{4-}$, with an overall formation constant of $\beta_3 = 2 \times 10^{18}$ (44), resulting in the successful elution of the adsorbed uranyl ion from the disk. The quantitative stripping of the retained uranyl ion by the modified membrane disk was then studied by using various volumes of 0.5 M NH_4HCO_3. The results are summarized in Table 3. As seen, the elution of uranyl ion from the membrane disk is quantitative with 90 mL of 0.5 M NH_4HCO_3 solution.

The influence of flow rates of the sample and stripping solutions from the membrane disk on the retention and recovery of uranyl ion was investigated. It was found that in the 1–50 mL/min range the retention of uranyl ion by the membrane disk is not significantly affected by the sample solution flow rate. On the other hand, quantitative stripping of the uranyl ion from the disk was achieved in a flow rate range of 0.5–4 mL/min. Thus, a flow rate of 2 mL/min

TABLE 3
Percent Recovery of 50 μg U(VI) from the Modified Membrane Disk Using Varying Volumes of 0.5 M Solution of NH_4HCO_3 at pH 8.25

Volume (mL)	10	20	30	40	50	60	70	80	90
% Recovery	45	57.6	68.4	79.0	89.7	92.0	94.4	97.3	100.1

was employed in all cases for the elution of uranyl ion from the disk with 90 mL of 0.5 M NH_4HCO_3 solutions.

The influence of the amount of TOPO loaded on the membrane disk on the retention of 50 μg U(VI) was studied, and the results are shown in Table 4. As seen, the extraction of uranyl ion is quantitative when 50 mg or more TOPO is used. The maximum capacity of the disk when modified by 50 mg of TOPO was studied by passing 10 mL portions of 0.5 M HNO_3 solution containing 1000 μg of U(VI) through the disk, followed by washing the disk with 25 mL of a 0.5 M HNO_3 solution and determination of the retained metal ions spectrophotometrically. The maximum capacity of the disk was found to be 938 ± 1 μg uranium.

In order to investigate separation of uranyl ion from Th(IV), the extraction of 50 μg of Th^{4+} from 10 mL of 0.5 M HNO_3 solution by a disk modified with 50 mg TOPO was studied. The results showed that Th(IV) ions were retained completely by the membrane disk, but only 35% of the adsorbed Th(IV) was eluted by using 90 mL of 0.5 M NH_4HCO_3 solution. The Th(IV) determination was performed with the Arsenazo(III) method (45). Fortunately, the retention of Th(IV) by the modified membrane disk is negligible in the presence of 0.025 M EDTA, and it can be separated from uranyl ion quantitatively.

In order to study the selective separation of uranyl ion from its binary mixtures with diverse metal ions, an aliquot of 0.5 M HNO_3 solutions (10 mL) containing 50 μg U(VI) and milligram amounts of other cations was taken, and the recommended procedure was followed. The results are summarized in Table 5. The results clearly show that uranyl ions in the binary mixtures are retained almost completely by the membrane disk, even in the presence of up to 100 mg of the diverse ions. Meanwhile, the determination of foreign ion concentrations in the eluent revealed that, with the exception of Mo(VI), the amount of foreign ion eluted was lower than 0.7% of its initial concentration. The concentration of Mo(VI) eluted was 4.6% of its initial concentration.

In order to assess the applicability of the method to real samples, it was applied to the separation and recovery of uranyl ions from two soil samples. A proper amount of finely powdered soil sample (with a uranium content below 50 μg) was digested and dissolved as described elsewhere (43). After evaporation of acids to dryness, a proper amount of 0.5 M HNO_3 (8–9 mL) was

TABLE 4
Percent Recovery of 50 μg U(VI) by the Membrane Disks Modified with Different Amounts of TOPO

TOPO (mg)	0	5	15	25	35	43	50	75	100
% Recovery	0	31.2	53.9	70.6	80.2	89.7	100.0	100.0	100.0

TABLE 5

Separation of 50 μg U(VI) from Binary Mixtures

Foreign ion	Added as	Amount taken (mg)	% Recovery of U(VI)
Na^+	NaCl	100	98.5
K^+	KCl	100	100.0
Mg^{2+}	$Mg(NO_3)_2 \cdot 6H_2O$	100	99.1
Ca^{2+}	$CaCl_2$	100	98.2
Ni^{2+}	$Ni(NO_3)_2 \cdot 6H_2O$	100	97.4
Co^{2+}	$Co(NO_3)_2 \cdot 6H_2O$	100	99.4
Fe^{3+}	$Fe(NO_3)_3 \cdot 9H_2O$	100	95.6
Cd^{2+}	$Cd(NO_3)_2 \cdot 4H_2O$	100	100.2
Cr^{3+}	$Cr(NO_3)_3 \cdot 9H_2O$	100	102.1
Zr(VI)	$ZrO_2Cl_2 \cdot 8H_2O$	10	96.0
Zn^{2+}	$ZnCl_2$	100	96.1
Pb^{2+}	$Pb(NO_3)_2$	100	103.8
Cu^{2+}	$Cu(NO_3)_2 \cdot 3H_2O$	100	100.9
Mn^{2+}	$MnCl_2 \cdot 4H_2O$	100	100.0
Hg^{2+}	$HgCl_2$	30	98.6
V(V)	$NaVO_3 \cdot H_2O$	10	96.3
Al^{3+}	$Al(NO_3)_3 \cdot 9H_2O$	100	98.2
Mo(VI)	$Na_2MoO_4 \cdot 2H_2O$	100	99.0
Ce^{3+}	$CeCl_3 \cdot 7H_2O$	6	100.0
Ti^{3+}	1000 μg/mL solution	5	101.9
Th^{4+} [a]	1000 μg/mL solution	1	96.5

[a] In the presence of 0.02 M EDTA the tolerance limit was increased to 100 mg of Th^{4+}.

TABLE 6

Determination of Uranium in Real Samples

Sample	Certified amount	Amount found
S-7	%0.527 U_3O_8	%0.57 (3.1)[a] U_3O_8
IAEA-312[b]	16.5 μg U/g	16.9 (2.4) μg U/g

[a] Values in parentheses are RSDs based on three replicate analyses.

[b] The certified value of Th^{4+} in this sample is 91.4 μg/g.

added to dissolve the precipitate and the resulting solution was diluted to the mark in a 10-mL volumetric flask, and the recommended procedure was followed. The results of triplicate analyses (Table 6) showed that uranium recovery from soil samples S-7 and IAEA-312 was 108 and 102%, respectively.

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CONCLUSION

The proposed method has the following advantages. It can selectively separate uranyl ion from foreign cations, including Th^{4+}, even when they are present at high concentrations. The method can be applied to the separation of uranium at both trace levels and at higher concentrations. It is a simple, highly selective, and reproducible method for the separation of uranyl ion. The reproducibility of the procedure is within ±3%.

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