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Research Article

EXTRACTION AND SEPARATION OF BARIUM (II) FROM POLYETHER COMPOUNDS USING ORGANIC ACID MEDIUM

Burungale S. H*

Department of Chemistry, Yashwantrao Chavan College of Science, Karad-415124 (India)

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A B S T R A C T

A simple solvent extraction method has been developed for the separation of Barium from associated elements using polyether compounds. The separations are carried out from trinitrophenol medium. The adsorption of Barium was quantitative from 2M sodium acetate. It was separated from number of elements in binary mixtures. Most of the elements showed a very high tolerance limit and was possible to separate Barium from a number of elements in Multicomponent mixtures. Method is very simple, rapid, and selective and has good reproducibility.

INTRODUCTION

Barium has attracted considerable attention because it is one of the major constituent of fission product and also found in number of rocks. For the precise determination, it is very essential to separate barium from other elements. The determination of barium in environmental samples such as rocks, minerals, marine organisms and bone is important in terms of geochemistry and bio-chemistry. However, the environmental samples often contain large amount of calcium and other alkaline earth metals which may cause spectral interference. Therefore, several liquid liquid extraction methods. have been explored for the separation of barium. The classical methods were selective precipitation either as insoluble sulfate or chromate.

In such extractions no attempts were made to separate barium (II) from die associated elements. Some efforts were made to use crown ethers for solvent extraction separation studies of barium(II).

18-crown-6(1-2), dibenzo-18-crown-6(3), Dibenzo-24-crown-8(4) were used for the study of extraction constant and stability constant.. Attempts were made to separate barium(II) from strontium(5) and calcium(6). The separation of barium(II) from other alkaline earths was achieved but for such separation other extractants along with dibenzo-18-crown-6 and 18-crown-6 wore employed(7).

The stability of strontium and barium(II) has been reported with 18-crown-6 from pierce solution(8). The substoichiometric ion-pair extraction of barium(II) with cryptand-2.2.2 and 18-crown-6 has also been carried out using picrate as a counter anion(9). Spectrophotometric investigation of complex formation of barium(II) and calcium with aza-l5-C-5 has been carried out(10-14). From the literature survey it is clear that no systematic efforts have been made for the solvent extraction separation studies of barium(II) and its separation from the associated elements using dibenzo-24-crown-8. Such studies are reported in this chapter. The method developed is simple and has extractive capacity at trace level and offers many more advantages over the reported methods. Using the method, barium(II) can be separated very effectively from associated elements in Multicomponent mixtures. The developed method has been extended for the determination of barium(II) in number of real samples.

Experimental

Apparatus

Spectrophotometer, a digital pH meter, with glass and Calomel electrodes, els was used.

A stock solution of barium(II) (1.00 mg/ ml) was prepared by dissolving 1.90 g. of barium nitratc(AnalaR grade, BDH) in 1000 ml of distilled deionised water and standardized gravimetrically(14). A solution containing 100 μg / ml of barium (II) was prepared by appropriate dilution of the standard stock solution. All other chemicals were of AR grade and were used without further purification.

Corresponding author:* **Burungale S. H
Department of Chemistry, Yashwantrao Chavan College of Science, Karad-415124 (India)

General Procedure

To an aliquot of a solution containing 100 µg barium (II), picric acid was added so as to have a concentration of 0.00001-0.05 M in a total volume of 10 mL The solution was then transferred to a separating funnel and was equilibrated with 5 ml of 0.001 M suitable crown ether with nitrobenzene as the diluent, for 1 min on a wrist action flask shaker. The two phases were allowed to settle and separate. Barium(II) was stripped from the organic phase with 5 ml of 2.0 M nitric acid and was determined spectrophotometrically with Sulfonazo-III at 640 nm(14). The concentration of barium (II) was calculated from a calibration graph.

Extraction of Barium (II) as a function of picric acid Concentration with crown ethers

In order to ascertain the optimum concentration of picric acid required for the quantitative extraction of Barium(II), the extraction studies were performed with a range of crown ethers of 0.001 M concentration in nitrobenzene. The picric acid concentration as varied from 0.00001 to 0.05M.The results of extraction studies are shown in Table- 1 and fig-1 it is clear that amongst various crown ethers only Solutions of crown ether were prepared from Dicyclohexano-18-crown-6(DC18C6 quantitatively extraction of Barium (II) with increase in concentration from 0.001-0.05 M picric acid. extraction studies of Barium (II) was carried out with 0.001M DC-18- Crown-6 with nitrobenzene as a diluent.

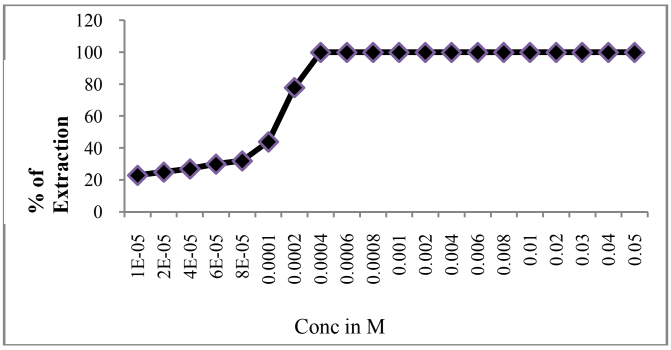

Fig 1 Extraction of Barium (II) as a function of picric acid Concentration

Extraction Mechanisms

[DC-18C6] + $Ba(NO_3)_2$ $\xrightarrow{\text{Trinitrophenol}}$ [DC-18C6 Ba]$^{2+}$.Trinitrophenolate+HNO_3

Extractant Metal salt Extractable Complex
[DC-18C6 Ba]$^{2+}$.[Trinitrophenolate]$^{2-}$ $\xrightarrow{\text{Sodium Acetate}}$ [DC-18C6] + [Trinitrophenolate]^{2}
Extractable Complex Ba(acetate)$_2$ +Na-Trinitrophenolate

The amount of Barium extracted on 0.001M DC18C6 and percentage removal calculated using the following equations

$$qe= \frac{C_o - C_e}{m} X\ V \text{ ----------------------}(1)$$

$$\%\ removal = \frac{C_o - C_e}{C_o}\ 100\text{---------------}(2)$$

Where, (mg/L) is concentration of lead before adsorption, (mg/L) is equilibrium concentration (g) adsorbent weight and V solution volume in liter.

Effect of varying Concentration of Dicyclohexano-18-crown-6

In order to ascertain the optimum concentration of DC-18-Crown-6 required for the quantitative extraction of Barium (II), the extraction studies were performed with a range of picric acid of 0.001 M with varying the concentration of DC-18- Crown-6 in nitrobenzene. The concentration of DC-18-Crown-6 was varied from 0.0000005-0.1M. The results of extraction studies are shown in Table- 2 which indicate that the extraction of Barium (II) was increased with increase in DC-18- Crown-6 concentration and was quantitative from 0.001 – 0.1M DC-18- Crown-6. Further extraction studies of Barium (II) was carried out using 5 ml of 0.001M DC-18- Crown-6 in nitrobenzene as a diluent.

Table 1 Effect of varying Concentration of Dicyclohexano-18-crown-6

DC-18- Crown-6 Conc.M	Extraction %E	Distribution D
0.0000005	08	0.087
0.000001	12	0.136
0.000005	16	0.190
0.00001	18	0.220
0.00005	28	0.250
0.0001	38	0.538
0.0005	88	7.333
0.001	100	∞
0.005	100	∞
0.01	100	∞

Concentration of Sodium acetate as Stripping Agent

After extraction of Barium (II) from 0.001 M picric acid with 0.001M DC-18- Crown-6 in nitrobenzene, it was stripped from the organic phase with various concentration of sodium acetate stripping agent. Further concentration of sodium acetate studies of Barium (II) were carried out with 1M sodium acetate. The results of stripping of Barium (II) are shown in table-4

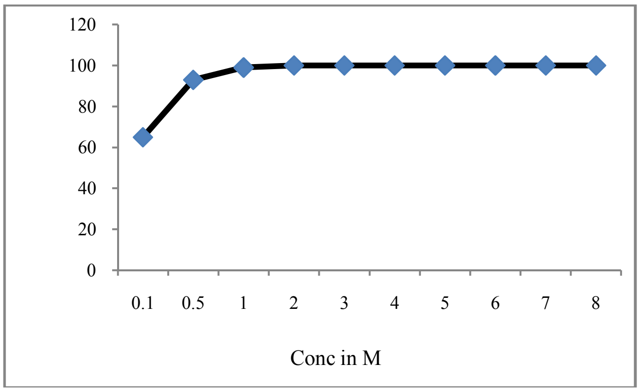

Fig 2 Concentration Sodium acetate as Stripping Agent. Barium (II) -100 µg, Crown Ether-0.001M in Nitrobenzene, 0.001M Picric Acid

Effect of various Diluents

To study the effect of diluents on the extraction of Barium (II), extractions were performed from 0.001M picric acid using 0.001M DC-18- Crown-6 in various solvents such as benzene, toluene, xylene, carbon tetrachloride, chloroform, and nitrobenzene. The phase volume ratio was maintained at unity. It was observed that the extraction of Barium (II)was 32% with benzene, 30% with toluene, 28% with xylene, 13% with carbon tetrachloride, 43% with chloroform, and was quantitatively only with nitrobenzene. Therefore studies of

Barium (II) were carried out form nitrobenzene as diluent.The results of extraction studies are shown in Table1

Table 1 Effect of various Diluents

Barium (II)-100 µg, DC-18C-6 0.001M, 0.001M Picric Acid, Strippant 1.0M sodium acetate

Diluent	Dielectric Constant	Extraction %	Distribution Ratio D
Benzene	2.28	32	0.471
Toluene	2.38	30	0.428
xylene	2.3	28	0.389
Carbon Tetrachloride	2.24	13	0.149
Chloroform	4.8	43	0.754
Tetrachloroethane	8.2	78	3.545
Tetrachloromethane	9.08	73	2.704
Dichloroethane	10.5	80	4.00
Nitrobenzene	34.8	100	∞

Effect of varying Concentration of

Barium (II)-was extracted with 10 ml of 0.001m dicyclohexano-18-crown-6 from 0.001M picric acid using nitrobenzene as a diluent. The Barium (II)-was varied from 25- 300µg per 10 ml. it was found that 10ml of 0.001M DC18C6 solution was adequate to extract Barium (II)-quantitatively up to 200µgper 10 ml of sample solution. The results are shown in table-2.

Table 2 Effect of varying Concentration of potassium (I)

DC-18C-6 0.001M, 0.001M Picric Acid, Strippant 2.0M HNO_3

Barium (II) µg	Extraction %	Distribution Ratio D
25	100	∞
50	100	∞
75	100	∞
100	100	∞
125	100	∞
150	100	∞
175	100	∞
200	100	∞
225	98	49.00
250	95	19.00
275	92	11.50
300	89	8.09

Period of Equilibration

Barium (II) was extracted with 10 ml of 0.001m dicyclohexano-18-crown-6 from 0.001M picric acid using nitrobenzene as a diluent. The period of equilibration was varied from 1-30 minutes on the wrist action flask shaker. It was observed that the extraction of Barium (II) was quantitatively in minutes of equilibration. To ensure complete extraction of Barium (II) .the period of equilibration employed was 2 minutes

Separation of Barium (II) from Binary Mixtures

Barium (II) was extracted with 10 ml of 0.001m dicyclohexano-18-crown-6 in nitrobenzene from 0.001M picric acid in the presence of a various diverse ions. The tolerance limit was set at the amount of foreign ions required to cause a ± 2% error in the recovery of potassium (I). Amongst s block cations, rubidium(I), cesium(I), strontium (II), were co-extracted along with potassium (I), strontium (II), and barium(II) showed low tolerance limit. All the s- block cations showed high tolerance limit. Amongst d block cations, showed high tolerance limit. All the s- block cations showed high tolerance limit. Most of the p- block cations showed high tolerance limit except lead (II) which showed low tolerance limit. The results of separation of Barium (II) from binary mixtures are shown in Table 3

Table 3 Separation of Barium (II) from Binary Mixtures

Ions	Added as	Tolerance Limit(mg)	Ions	Added as	Tolerance Limit (mg)
Li^+	LiCl	35	Ce^{3+}	$CeCl_3.6H_2O$	0.1
Na^+	NaCl	25	Sb^{3+}	$SbCl_3$	0.1
Rb^+	RbCl	0.5	Y^{3+}	$Y(NO_3)_3$	0.5
Cs^+	CsCl	0.1	Zr^{4+}	$Zr(NO_3)4.4H_2O$	0.3
Be^{2+}	$BeSO_4.4H_2O$	0.3	V^{4+}	$VOSO_4.4H_2O$	0.5
Mg^{2+}	$MgCl_2.6H_2O$	30	Th^{4+}	$Th(NO_3)_4$	0.5
Ca^{2+}	$CaCl_2$	15	U^{6+}	$UO_2(NO_3)_2.6H_2O$	10
Sr^{2+}	$Sr(NO_3)_2$	20	Cr^{6+}	$K_2Cr_2O_7$	0.5
Cu^{2+}	$CuCl_2.2H_2O$	25	Mo^{6+}	$(NH_4)_6Mo_7O_{24}.4H_2O$	0.5
Ni^{2+}	$NiCl_2.6H_2O$	15	W^{6+}	$Na_2WO_4.4H_2O$	30
Mn^{2+}	$MnCl_2.4H2O$	40	Cl^-	HCl	15
Zn^{2+}	$ZnCl_2$	15	Br^-	HBr	10
Pb^{2+}	$Pb(NO_3)_2$	30	SCN^-	NaSCN	75
Fe^{3+}	$FeCl_3.6H_2O$	25	ClO4	ClO_4	15
Cr^{3+}	$Cr(NO_3)_3.9H_2O$	35	$CH3COO^-$	CH_3COOH	35
Al^{3+}	$Al(NO_3)_3.9H_2O$	10	Tartrate	Tartaric acid	25
La^{3+}	$LaCl_3$	15	EDTA	EDTA	35

Separation of Barium (II) from multicomponent Mixtures

Barium (II) was extracted from 0.001M picric acid with 0.001M DC18C6. Under these set conditions there was no extraction of iron (III), uranium (VI) and molybdenum (VI) but from 8.0 M hydrochloric acid there was quantitatively extraction of iron(III) with dicyclohexano-18-crown-6, Uranium(VI) was quantitatively extracted with dibenzo-18-crown-6 from 8.0 M hydrochloric acid while molybdenum(VI) was quantitatively extracted with dibenzo-18-crown-6 from 8.0 M hydrochloric acid whereas potassium(I) was not extracted. Most of the geological samples contain iron hence its separation from other trace elements is important. The separation of iron (III), Barium (II) and other elements is achieved as follows. When a mixture containing iron(III), Barium (II) and lithium (I) was extracted with 0.001M dicyclohexano-18-crown-6 in nitrobenzene from 8.0 M hydrochloric acid, Iron was extracted quantitatively leaving behind Barium (II) and in aqueous phase. The aqueous phase after evaporating was treated with water and extracted 0.001M dicyclohexano-18-crown-6 in nitrobenzene from 0.001M picric acid. Under these set conditions Barium (II) was extracted while lithium (I) remained in the aqueous phase. Iron and Barium (II) from the respective organic phases were stripped with 2.0M nitric acid and 1M Sodium Acetate. The separation of Barium from iron and other cations was accomplished by following similar methodology table-4

Table 4 Separation of potassium (I) from multicomponent Mixtures

No	Mixture	Taken µg	Found µg	Recovery %	Extractants	Stripping agents
1	Fe(III)	100	100	100	DC18C6,8MHCl	2M HNO_3
	Ba(II)	100	100	100	DC18C6,0.01MPicric acid	1M Sodium Acetate
	Li(I)	100	100	100	Aqueous Phase	-----
2	Fe(III)	100	100	100	DC18C6,8MHCl	2M HNO_3
	Ba(II)	100	100	100	DC18C6,0.01MPicric acid	1M Sodium Acetate
	Be(II)	100	100	100	Aqueous Phase	-----
3	Fe(III)	100	100	100	DC18C6,8MHCl	2M HNO_3
	Ba(II)	100	100	100	DC18C6,0.01MPicric acid	1M Sodium Acetate
	Mg(II)	100	100	100	Aqueous Phase	-----

4	Fe(III)	100	100	100	DC18C6,8MHCl	2M HNO_3
	Ba(II)	100	100	100	DC18C6,0.01MPicric acid	2M HNO_3
	Ca(II)	100	100	100	Aqueous Phase	-----
5	U(VI)	100	100	100	DB24C8,8MHCl	2M HNO_3
	Ba(II)	100	100	100	DC18C6,0.01MPicric acid	1M Sodium Acetate
	Li(I)	100	100	100	Aqueous Phase	-----
6	U(VI)	100	100	100	DB24C8,8MHCl	2M HNO_3
	Ba(II)	100	100	100	DC18C6,0.01MPicric acid	1M Sodium Acetate
	Be(II)	100	100	100	Aqueous Phase	-----
7	U(VI)	100	100	100	DB24C8,8MHCl	1M HNO_3
	Ba(II)	100	100	100	DC18C6,0.01MPicric acid	Sodium Acetate
	Th(IV)	100	100	100	Aqueous Phase	-----
8	U(VI)	100	100	100	DB24C8,8MHCl	2M HNO_3
	Ba(II)	100	100	100	DC18C6,0.01MPicric acid	1M Sodium Acetate
	Ce(III)	100	100	100	Aqueous Phase	-----

Application to the Analysis of Barium (II) from various Synthetic Samples

The proposed method was applied for determination of Barium (II) in synthetic samples. The results are shown in table- 5

Table 5 Analysis of Barium (II) from various Synthetic Samples

Composition/µg	Barium (II) /µg	Mean/µg	Recovery/%
Ba(II)	150	149.8	99.6
K(I)	200		
Ca(II)	200		
Ba(II)	150	149.5	99.66
Na(I)	200		
Mg(II)	200		
Ba(II)	150	149.6	99.73
Fe(III)	200		
Sr(II)	200		

CONCLUSION

The important feature of the developed method is that it permits the separation of potassium (I) at trace level concentration. The concentration of crown ether required is very low. It is possible to separate Barium (II) from iron(III), lithium(I), beryllium(II), calcium(II), magnesium(II) thorium(IV), effectively.

The method was extended to the analysis of Barium (II) in various standard rock samples, blood serum and milk samples yielding excellent results. The method is simple, rapid, selective, reproducible and adds significantly to the use of crown ethers as extractants for the separation of Barium(II).

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