

Separation of Europium(III) from Other Elements Employing Solvent Extraction Technique

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A rapid and selective method has been developed for the extraction of Eu(III) with 1,2,3-benzotriazole into *n*-heptanol. The effect of various parameters on the extraction coefficient value such as effect of pH, time of equilibration, solvents, effect of various anions and cations have been evaluated.

INTRODUCTION

Europium is the rarest of the rare earth elements. It is used in phosphors, garnets, nucleonic control, refractory compounds and in lasers. Various other reagents¹⁻⁴ have been used for the solvent extraction of Eu(III). However, the extraction of Eu(III) with 1,2,3-benzotriazole for the radiochemical separation has not been reported in the literature. The present work describes a rapid and a selective method for the extraction of Eu(III) with 1,2,3-benzotriazole into *n*-heptanol. Ideal conditions for the extraction have been established.

EXPERIMENTAL

All the chemicals and reagents used were of A.R. grade. ¹⁵² + ¹⁵⁴Eu and isotope used for studying the interference of other cations were supplied by BRIT, Bombay. The stock solution of Eu(III) was prepared by dissolving A.R. grade EuCl₃ in distilled water containing 5 mL of concentrated HCl. Solution of anions was prepared by dissolving appropriate salts in distilled water and that of cations was prepared in the same way, but 5 mL of concentrated HCl was added to the solution, wherever required. The strength of these solutions was determined by the standard method.⁵ The solution of the reagent 1,2,3-benzotriazole used in the extraction studies was prepared by dissolving appropriate amount of the reagent in distilled water. The purity of the reagent was checked by determining its melting point (96°C) and it was found to be in agreement with that reported in the literature.

Gamma-emitters were counted on a single-channel pulse-height analyzer in conjunction with a 3.5 cm × 3.5 cm NaI(Tl) well-type crystal detector. Beta-emitters were counted on a thin end window type G M. counter in conjunction with a decade scaler, high voltage unit and a timer.

Extraction Procedure

In a separating funnel 1.0 mg of Eu(III) labelled with $^{152} + ^{154}\text{Eu}$ was taken. 10 mL of 1% aqueous solution of 1, 2, 3-benzotriazole was added. Volume of the aqueous phase was made to 17.0 mL with distilled water after adjusting the pH of the solution with ammonia. The aqueous phase was equilibrated with 17.0 mL of *n*-heptanol for 4.0 min. The phases were allowed to separate. A 2 mL aliquot of each phase was taken for counting on a gamma ray spectrometer at the channel corresponding to the 0.34 MeV photopeak of $^{152} + ^{154}\text{Eu}$. The extraction coefficient value (E) was calculated using the equation:

$$E = \frac{\text{Activity of 2 mL aliquot of the organic phase}}{\text{Activity of 2 mL aliquot of the aqueous phase}}$$

RESULTS AND DISCUSSION

Effect of pH, Time of Equilibration and Solvents

From Table 1 it can be seen that a maximum E value of 128 was obtained for the extraction of Eu(III) with 1,2,3-benzotriazole into *n*-heptanol at a pH of 8.5 and an equilibration time of 4.0 min

The extraction of Eu(III) into different organic solvents was ascertained and it was observed that *n*-heptanol was the best solvent for the extraction. The effects of solvents on E value followed the trend given below:

$$E = \begin{array}{cccccc} \text{n-heptanol} > \text{cyclohexanone} > \text{xylene} > \text{nitrobenzene} > \text{benzene} > \\ 128.0 & 0.54 & 0.47 & 0.21 & 0.13 \end{array}$$

$$E = \begin{array}{cccc} \text{ether} > \text{toluene} > \text{chloroform} = \text{carbontetrachloride} > \\ 0.10 & 0.06 & 0.05 & 0.05 \end{array}$$

$$E = \begin{array}{cccc} \text{ethyl acetate} = \text{1-butanol} = \text{heptane} > \text{ethyl methyl ketone} > \\ 0.02 & 0.02 & 0.02 & 0.0045 \end{array}$$

$$E = \begin{array}{cccc} \text{octanol} = \text{hexane} > \text{1-pentanol} > \text{nitromethane} > \text{amyl acetate}. \\ 0.0032 & 0.0032 & 0.0012 & 0.00067 & 0.0004 \end{array}$$

After equilibration no phase separation was observed for formaldehyde and ethylene glycol. Hence, in all further studies *n*-heptanol was used as a solvent.

Effect of Anions

The effect of various anions on the extraction coefficient value of Eu(III) revealed that 100 mg each of iodide, chloride, bromide, molybdate and bromate, 50 mg of thiourea, 10 mg each of acetate, sulphate, thiocyanate, thiosulphate, nitrite, borate and perchlorate do not decrease the extraction coefficient value. Sulphite, carbonate, EDTA, iodate, oxalate fluoride, cyanide, tartrate and citrate interfered, but their interference was removed by expelling them with concentrated HNO_3 and HCl prior to the extraction of Eu(III). The interference of chromate and dichromate was decreased by reducing it with concentrated HCl and H_2O_2 , whereas the interference of phosphate was removed by precipitating it with ammonium molybdate prior to the extraction of Eu(III).

TABLE-I
EFFECT OF pH AND TIME OF EQUILIBRATION ON THE EXTRACTION COEFFICIENT VALUE OF Eu(III) WITH 1,2,3-BENZOTRIAZOLE INTO n-HEPTANOL

pH	Extraction coefficient E	Percentage extraction %E	Time of equilibration min
2.0	0.0030	0.30	4.0
3.0	0.0034	0.34	4.0
4.0	0.0045	0.44	4.0
5.0	0.0051	0.50	4.0
6.0	0.0060	0.60	4.0
7.0	0.0090	0.89	4.0
7.5	0.1	9.09	4.0
8.0	32.0	96.96	4.0
8.5	128.0	99.22	4.0
8.5	122.0	99.18	4.0
8.5	103.0	99.03	4.0
9.0	90.0	98.90	4.0
10.0	42.0	97.67	4.0
8.5	61.0	98.38	2.0
8.5	97.0	98.97	3.0
8.5	118.0	99.16	4.0
8.5	104.0	99.04	5.0
8.5	106.0	99.06	6.0

Separation Factor

The separation factor for other elements in the extraction of Eu(III) was determined (Table 2). Of the elements studied, Ba(II), Cs(I), Zr(IV), Sb(V), Sn(II), Rb(I), Ca(II), As(IV), K(I), Ce(III), Cr(VI), Sn(IV), Re(VII) and P(V) were extracted to the extent of less than 1.0%. S(VI), Na(I), Pd(II), Mo(VI), Sb(III), Se(IV), Hf(IV), Ru(III), As(III), Ce(IV), W(VI) and Tl(I) were extracted between 1.0 to 10.0%. Pt(IV) was extracted to greater than 16%, but it was masked with 10 mg of thiosulphate. Te(IV) was extracted to more than 19% but it was removed as tellurium metal with SO₂-water. Cr(III) was extracted to more than 37% and was suppressed to below 10% by oxidising it to Cr(VI) with NaOH and H₂O₂. Ir(IV) and Fe(III) were extracted to greater than 41% and 77% respectively and they were masked with 10 mg of thiocyanate. Ag(I) and Au(III) were extracted to greater than 89%, their interference was removed by masking Ag(I) with 10 mg of thiosulphate and that of Au(III) was removed by precipitating Au(III) as gold metal with SO₂-water. Fe(II) was extracted to greater than 91% but was masked with thiocyanate. Hg(II) was extracted to greater than 95%, and its

interference was removed with 100 mg of iodide. Zn(II) and Co(II) were extracted to greater than 99%. The interference of Zn(II) was removed by precipitating Zn(II) as ZnS and rejecting the precipitate of ZnS after centrifugation, whereas the interference of Co(II) was removed by masking with hydrogen peroxide.

TABLE-2
SEPARATION FACTOR OF DIFFERENT ELEMENTS IN THE EXTRACTION OF
Eu(III) WITH 1,2,3-BENZOTRIAZOLE INTO n-HEPTANOL

S.F. greater than	Elements	
	with milligram amounts of carrier	without milligram amounts of carrier
10 ⁵	Te(IV)*, Au(III)*, Zn(II)*,	
10 ⁴	Ba(II), Cs(I), Zr(IV), Sb(V), Sn(II), Rb(I), Ca(II), As(V), K(I), Ce(III), Cr(VI), Sn(IV), Re(VII), P(V), Cr(III)*, Ir(IV)*, Fe(III)*, Fe(II)*	Ba(II), Cs(I), Zr(IV), Sn(II), Rb(I), Ca(II), As(V), K(I), Ce(III), Cr(VI), Sn(IV), Re(VII), S(VI), W(VI), Cr(III)*, Ir(IV)*, Fe(III)*, Fe(II)*,
10 ³	S(VI), Na(I), Pd(II), Mo(VI), Sb(III), Se(IV), Ru(III), As(III), Ce(IV), W(VI), Tl(I), Pt(IV)*, Ag(I)*, Hg(II)*, Co(II)*,	Sb(V), P(V), Na(I), Pd(II), Mo(VI), Sb(III), Se(IV), Hf(IV), Ru(III), As(III), Ce(IV), Tl(I), Pt(IV)*, Ag(I)*, Hg(II)*, Co(II)*.
10 ²	Pt(IV), Cr(III), Ir(IV), Te(IV).	Pt(IV), Fe(III), Te(IV).
10 ¹	Fe(III), Ag(I), Au(III), Fe(II).	Cr(III), Ir(IV), Ag(I), Au(III), Fe(II), Co(II).
10 ⁰	Zn(II), Hg(II), Co(II).	Zn(II), Hg(II).

*After treatment

1.0 mg of Eu(III) was back extracted with 1:1 HCl to the extent of greater than 99%.

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