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Solvent Extraction of Indium(III) with Alizarin into 1-Octanol

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The reagent alizarin has been used for the extraction of indium(III) into 1-octanol. The effect of various parameters on the extraction coefficient value of indium(III) has been evaluated. The stoichiometry of metal to reagent, determined by the method of substoichiometric extraction and slope ratio was found to be in the ratio of 1 : 3. Separation factor for a number of elements in the extraction of indium(III) has been evaluated.

INTRODUCTION

Alizarin[1] reacts with a large number of metal-ions like tin, gallium, aluminium, calcium, iron, zirconium, europium, etc. to form coloured lakes. A literature survey reveals that this reagent has not been applied for the extraction of indium(III). The present investigation deals with developing a rapid and selective method for the extraction of indium(III) with this reagent. ^{114m}In was used as a tracer for studying the extraction process.

EXPERIMENTAL

Chemicals and reagents: All the chemicals and reagents used were of A.R. grade. Stock solution containing 10 mg/mL of In(III) was prepared by dissolving A.R. grade Fluka make $InCl_3$ in double disttilled water containing 5 mL of concentrated HCl. The strength of the the solution was determined by the method given by Scott[2]. Solutions of cations and anions were prepared by dissolving the appropriate amount of salt in distilled water and concentrated acid wherever necessary. The strength of these solutions was estimated by standard methods given in Vogel[3]. ^{114m}In and other isotopes used for studying the elemental interference were obtained from the BRIT of BARC, Trombay, Bombay (India).

Instrumentation: Gamma-emitters were counted on a gamma-ray spectrometer in conjunction with a 3.5 cm × 3.5 cm NaI(Tl) well-type detector. Beta-emitters were counted on a thin end-window type G.M. counter in conjunction with a decade scalar, high voltage unit and timer.

Extraction Procedure: A 1-mL solution of $InCl_3$ containing 1.0 mg of In(III) labelled with ^{114m}In was taken in a 125 mL separatory funnel. 10 mL of 0.25% alizarin solution in methanol was added and volume was made with distilled water. The pH was adjusted to 8.0 with ammonia. The mixture was equilibrated with 20.0 mL of 1-octanol for an equilibration time of 3.0 min. The phases were

allowed to separate. A 2 mL aliquot of each phase was withdrawn for counting at the channel corresponding to the 0.19 MeV photopeak of ^{114m}In. The organic and aqueous phases were found to be equal after equilibration, hence 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

The extraction coefficient value indicated that maximum extraction occurred at a pH of 8.0 although the percentage extraction was found to be better than 98% over the pH range of 6.0 to 9.0 (Table 1). The E value was found to decrease in the more acidic and alkaline ranges. The extraction coefficient reached a maximum value of 98 at pH of 8.0. Determination of the effect of time of equilibration revealed that the percentage extraction was better than 98% for an equilibration time of 1.0 to 5.0 min (Table 1).

TABLE 1
EFFECT OF pH AND TIME OF EQUILIBRATION ON THE EXTRACTION COEFFICIENT VALUE OF In(III)

pH	Extraction coefficient (E)	%E	Time of equilibration min
2.0	0.009	0.89	3.0
3.0	0.020	1.96	3.0
4.0	0.050	4.76	3.0
5.0	1.850	64.91	3.0
6.0	55.000	98.21	3.0
7.0	65.000	98.48	3.0
8.0	93.000	98.94	3.0
8.0	86.000	98.85	3.0
8.0	98.000	98.99	3.0
8.5	82.000	98.80	3.0
9.0	81.000	98.78	3.0
10.0	25.000	96.15	3.0
8.0	56.000	98.25	1.0
8.0	65.000	98.48	2.0
8.0	98.000	98.99	3.0
8.0	82.000	98.80	4.0
8.0	79.000	98.75	5.0
8.0	82.000	98.80	6.0

The effect of different solvents on the E value of In(III) using alizarin was studied and 1-octanol was found to be the best solvent for the extraction. The effect of solvents on the E value followed the trend given below:

1-octanol > iso-amylalcohol > chloroform = ethylacetate > xylene >
E = 98 18 2 2 1.8
iso-butyl methyl ketone > nitromethane > heptane > benzene = nitrobenzene >
1.53 1.45 1.05 0.97 0.97
hexane > toluene > 1, 2-dichlorobenzene > cyclohexane > carbontetrachloride.
0.79 0.76 0.68 0.40 0.21

Pyridine, 1-pentanol, 1-butanol, and cyclohexanone were found to be miscible with the aqueous phase.

Interference: The effect of sodium, potassium or ammonium salts on the extraction coefficient value of In(III) revealed that 100 mg of chloride, cyanide, thiocyanate, iodide, chromate, dichromate, arsenite and sulphate; 50 mg of oxalate, bromide, carbonate, nitrate, citrate, fluoride, bromate and sulphite; 25 mg of perchlorate and phosphate; 10 mg of borate, hypophosphite, chlorate, thiosulphate and thiourea do not decrease the E value of In(III). The interference of nitrite, acetate, iodate and tartarate were removed by decomposing them with the concentrated HNO_3 and HCl, whereas tungstate was separated as tungstic acid prior to the extraction of In(III). EDTA was found to lower the E value of In(III).

Stoichiometry: The stoichiometry of the extracted species was determined by the method of substoichiometric extraction which was carried out as follows:

TABLE 2
SEPARATION FACTOR FOR VARIOUS ELEMENTS IN THE EXTRACTION OF In(III) WITH ALIZARIN INTO 1-OCTANOL

Separation factor (S.F.) grater than	Elements	
	with carrier (2 mg)	without carrier
10^4	Na(I), S(VI), P(V), Cl(I) Mo(VI), Cr (VI), As(V) Fe(III)*, Zr(IV)*, Sn(IV)*, Co(II)*, Sc(III)*, Sn(II)*.	Na(I), S(VI), P(V), Cl(I), Mo(VI), Tc(VII), Cr(VI), W(VI), As(V), Zr (IV)*, Co(II)*, Zn(II)*, Cd(II)*.
10^3	Pd(II), Re(VII), Au(III), Ir(IV), Cu(II), Cs(I), Ag(I), Hg(II), Sb(III), Sb(V), Se(IV), W(VI), Te(IV), K(I), As(III), Rb(I), Eu(III)*, Zn(II)*, Mn(II)*, Cd(II)*, Ca(II)*, Ce(III)*, Ce(IV)*, Tl(I)*, La(III)*.	Pd(II), Re(VII), Au(III), Ir(IV), Cu(II), Cs(I), Ag(I), Ba(II), Hg(II), Sb(III), Sb(V), Se(IV), Te(IV), K(I), As(III), Rb(I), Os(IV), Ca(II), Ce(III)*, Ce(IV)*, La(III)*, Tl(I)*, Eu(III)*, Mn(II)*.
10^2	Os(IV), Ba(II), Zr(IV), Pt(IV), Ca(II).	Zr(IV), Zn(II), Fe(II), Pt(IV), Ca(II).
10^1	Cd(II), Co(II), Zn(II), Fe(III), Fe(II), Ce(III), Ce(IV), Tl(I), La(III).	Cd(II), Co(II), Fe(III), Ce(III), Ce(IV), Tl(I), La(III), Mn(II).
10^0	Eu(III), Sc(III), Sn(II), Sn(IV), Mn(II).	Eu(II), Sc(III), Sn(II), Sn(IV).

* = after treatment.

Solution containing increasing amount of In(III) labelled with ^{114m}In was taken in a series of separating funnels. 12.8 mg of alizarin in 10 mL of methanol was added. The pH was adjusted to 8.0 with ammonia and the total volume was made to 30 mL with distilled water. The aqueous phase was equilibrated for 3.0 min. with 20 mL of 1-octanol. A 2 mL aliquot of the organic phase was counted on a gamma-ray spectrometer. The activity of the aliquot was plotted against the

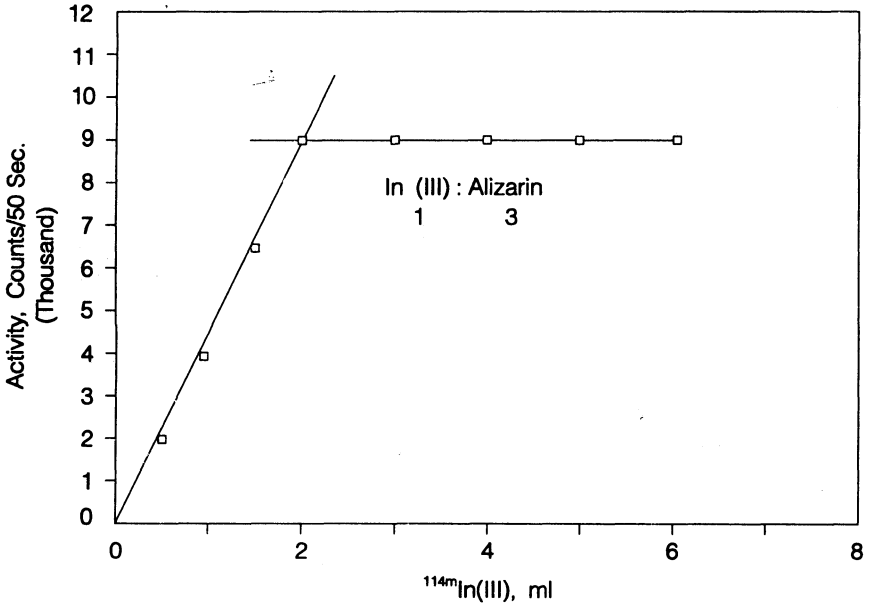

Fig. 1. Reproducibility of substoichiometric extraction

amount of In(III) taken. The graph (Fig. 1) shows that the activity increased with an increase in the In(III) concentration till it was subequivalent to the reagent added. A break was observed at the equivalence point corresponding to metal to reagent concentration in the ratio of 1 : 3.

The stoichiometry of 1 : 3 was further supported by the slope-ratio method. A graph of log HR Vs log E was a straight line with a slope of 3 (Fig. 2). The separation factor for number of elements in the extraction of In(III) was evaluated are given in Table 2. The reproducibility of the method was found to be 6.52%. 0.8 Mg of In(III) was back-extracted from 20 mL of the organic phase to better than 99% with 20 mL of 2M HCl.

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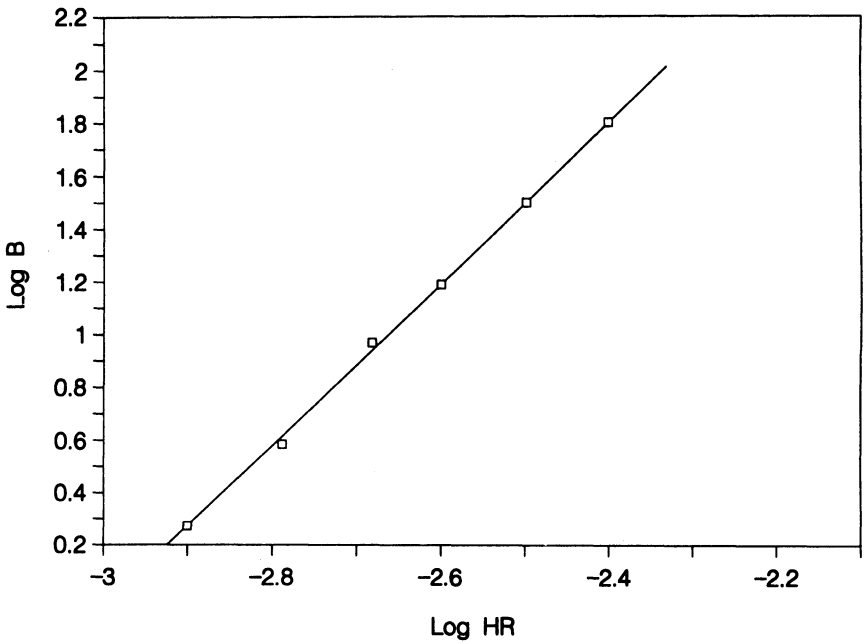

Fig. 2. Slope ratio method

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