

Solvent Extraction of Lead (II) and Its Separation from Bismuth (III)

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Solvent extraction behaviour of lead(II) with *n*-octylaniline in different organic solvents from halogen acid solutions was investigated. A scheme for separation of lead and bismuth is developed. The nature of probable extracted species has been established. The method has been applied for separating and estimating lead from synthetic mixtures. The proposed method is simple, rapid and also selective.

INTRODUCTION

High molecular weight amines are excellent extractants of ion association complexes of metals.¹⁻³ The use of *n*-octylaniline synthesized in the laboratory as an extractant for some metals was investigated.⁴⁻⁶ These studies have been extended to lead(II) which can be extracted quantitatively from its aqueous acid solutions with the reagent in different organic solvents as the diluents.

EXPERIMENTAL

All chemicals used were of A.R. grade. Stock solutions of lead and bismuth were prepared using their nitrate salts and standardized.^{7, 8}

n-Octylaniline was prepared by the method given by Pohlandt⁹ and used (% v/v) solution in chloroform. Acetic acid and 0.2 M sodium acetate were used to prepare buffer solution of pH 4.5.

To an aliquot of the solution containing 2.0 mg of lead (II), enough hydrochloric acid was added to make the concentration 2.5 M in a volume of 10 mL. Transfer the solution to 100 mL separatory funnel and shake for one minute with two 10 mL portions of 3% *n*-octylaniline in chloroform. Combine the extract and strip lead (II) from combined organic phase with two 25 mL portions of acetate buffer solution. The metal ion in aqueous phase is determined complexometrically.

RESULTS AND DISCUSSION

The extraction of lead (II) by reagent solution as a function of hydrochloric, hydrobromic and hydriodic acids concentrations was investigated. Lead (II) could be quantitatively extracted from 2.5 M hydrochloric acid, 0.3-2.0 M hydrobromic acid and 1.5 M KI + 0.5-2.5 M H₂SO₄ using two 10 mL portions of reagent in

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chloroform. Increase in reagent concentration was found to enhance the extraction. The excess reagent concentration had no significant adverse effect on magnitude of extraction. Different organic solvents were tried as diluents but only chloroform gives promising results.

Slope of $\log D$ versus \log of *n*-octylaniline concentration at fixed acid concentration was 2.0, indicating probable extracted species $2RNH_3^+ \cdot PbCl_4^{2-}$.

Effect of diverse ions

Several ions were examined for interference (Table-1) in the proposed method. The tolerance limit was set at the amount required to cause less than 1.5% recovery of the analyte. Moderate amounts of many ions are tolerable. The ions which show positive interference by being co-extracted were Tl(III), Bi(III), Al(III), Fe(II) and Fe(III), Zn(II) and Cd(II). Bismuth(III) is separated in hydrobromic acid media while Fe(II) and Fe(III) is masked with ascorbic acid, whereas ions which show negative interference by preventing the extraction of analyte were Tl(I), Mg(II), thiourea and EDTA.

TABLE-1
EFFECT OF DIVERSE IONS Pb(II), 2.0 mg; AQUEOUS PHASE, 2.5 M HCl IN VOLUME OF 10 mL; *n*-OCTYLANILINE 3%, 2 × 10 mL IN CHLOROFORM

Foreign ion	Tolerance limit (mg)	Foreign ion	Tolerance limit (mg)
Ga(III)	20	Mo(VI)	1
In(III)	20	Pt(IV)	1
Tl(III)	Co-Ext.	Pd(II)	1
Tl(I)	Int.	Os(VIII)	1
Bi(III)	Co-Ext.	Ru(III)	2
Fe(II)	10	Rh(III)	1
Fe(III)*	10	Au(III)	2
Sn(II)	5	Zn(II)	Co-Ext.
Sb(III)	10	Cd(II)	Co-Ext.
Mn(II)	15	Mg(II)	Int.
Ca(II)	20	H ₂ O ₂	1 mL
Sr(II)	10	Acetate	100
Ba(II)	10	Citrate	50
Co(II)	10	Maionate	40
Ni(II)	10	Salicylate	50
Cu(II)	10	Ascorbate	25
As(III)	15	Thiourea	Int.
Cr(VI)	10	Thiocyanate	25
Ag(I)	5	Phosphate	25
V(V)	5	Oxalate	25
Al(III)	Co-Ext.	Fluoride	50
Re(VI)	5	EDTA	Int.
W(VI)	2		

*Masked with ascorbate.

Separation of lead(II) from bismuth(III)

To an aliquot of solution containing 2.0 mg each of bismuth(III) and lead(II), add enough hydrobromic acid to make a solution 0.04 M in hydrobromic acid in a volume of 25 mL. Shake with 10 mL, 3% *n*-octylaniline chloroform. Strip bismuth(III) with two portions of 25 mL acetate buffer and determine complexometrically. Lead remaining in aqueous phase is determined complexometrically. The results of analysis are reported in Table-2.

TABLE-2
ANALYSIS AND RESULTS OF SYNTHETIC MIXTURES

Analysis No.	Bismuth (III)			Lead (II)		
	Taken	Found	Recovery	Taken	Found	Recovery
	mg	mg	%	mg	mg	%
1	2.0	1.99	99.9	2.0	2.00	100.0
2	0.5	0.49	99.9	2.0	1.99	99.9
3	1.0	0.99	99.8	1.5	1.49	99.9
4	2.0	1.99	99.9	0.5	0.49	99.8
5	1.5	1.49	99.9	1.0	1.00	100.0

Separation of lead from synthetic mixture

The extraction of lead(II) from 2.5 M hydrochloric acid into *n*-octylaniline facilitates its separation from tin, chromium, molybdenum, gold, silver, tungsten, copper, rhodium, indium, osmium, ruthenium and platinum from binary as well as multicomponent mixtures. The average recovery of lead was 99.8%.

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