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Sequential Separation of Cobalt,Nickel and Lead by using α- – Oximino aceto acetanilide benzoylhydrazone(HINABH)

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ABSTRACT

Simple, sensitive and accurate spectrophotometric method has been developed for sequential separation of Co^{II},Ni^{II} and Pb^{II} with α-oximinoacetoacetaanilidebenzoylhydrazone(HINABH). HINABH forms Yellowish brown complex with Co^{II} and yellow colored complexes with Ni^{II} and Pb^{II}.Co^{II}-HINABH complex was extracted in to n-butanol in the pH range 6.0-7.0 at λ max 425 nm., whereas Ni^{II} and Pb^{II} complexes were extracted into isoamylalcohol quantitatively in the pH range 8.5-9.5 at λ max 420nm and 410 nm respectively. HINABH complexes of Co^{II},Ni^{II} and Pb^{II} obeyed Beer's law in the range of 0.2-5.0 ppm,1-14 ppm and 2-16 ppm respectively. The method had been successfully employed for the determination of Co^{II},Ni^{II} and Pb^{II} in the synthetic mixtures and alloys.

Keywords: Spectrophotometric determination, α-oximinoacetoacetaanilidebenzoylhydrazone, cobalt, nickel and lead samples.

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INTRODUCTION

Although several methods have been reported for the spectrophotometric determination of Co^{II}, Ni^{II} and Pb^{II} [1-5], they suffer from various limitations such as delayed color development , heating time, interference from various diverse ions. In the present method HINABH has been used as an analytical reagent for the sequential separation and simultaneous determination of Co^{II},Ni^{II} and Pb^{II}.

The aim of this work is to develop a simple, fast, cost effective, and interference-free method for the determination of Co^{II},Ni^{II} and Pb^{II} in presence of each other that would be useful in industries or pharmaceutical laboratory.

MATERIALS AND METHODS

Apparatus

All glassware's used for experimental purpose were made up of Pyrex or corning glass. The burette, pipette and standard flasks were calibrated by the method described by Vogel[6].

Instruments

pH meter: A digital pH meter was used for pH measurements. The pH meter was calibrated by employing the buffer solutions of pH 4.0, 7.0 and 9.2.

Spectrophotometer: The absorption measurements were carried out on a Bausch and Lomb spectronic-20, using 1cm matched glass cell. The spectrophotometer was calibrated by measuring the absorption spectra of potassium chromate in KOH solution and that of potassium permanganate in sulphuric acid solution.

Preparation of experimental solution

The stock solution of cobalt(5000 ppm) was prepared by dissolving 5.92 g of $CoSO_4.7\ H_2O$ in 250 cm^3 of distilled water containing 5 cm^3 of con.H_2SO_4 and standardized gravimetrically[7].

The stock solution of Nickel(1000 ppm) was prepared by dissolving 1.12 g of $NiSO_4.6\ H_2O$ in 250 cm^3 of distilled water containing 5 cm^3 of con.H_2SO_4 and standardized gravimetrically[8].

The stock solution of lead(II) was prepared(5000ppm) by dissolving 2.0 g of lead nitrate in distilled water containing 3 cm^3 of con.$HNO_3$and was diluted to 250 cm^3 with distilled water and was standardized gravimetrically by salicylaldoxime method. HINABH was synthesized by the reported method [9]. The buffer solutions of pH 6.0 pH 9.0 were used for maintaining pH. A 0.5% HINABH solution was prepared by dissolving weighed amount in dimethylforamide and then diluting up to the mark.

Preparation of foreign ion solutions

The solutions of cations and anions were prepared by dissolving their A.R. grade salts in distilled water or dilute acids as required. The solutions of various anions were prepared by dissolving their A.R. grade sodium, potassium or ammonium salts in distilled water.

Procedure for sequential separation and spectrophotometric determination of **Co^{II}, Ni^{II} and Pb^{II} using HINABH as a reagent.**

To an aliquot containing cobalt, nickel and lead, 1cm3 of 0.5% HINABH, 2 cm3 of buffer solution of pH 6.0 were added. The final volume was made up to 10 cm3 with distilled water. The cobalt complex was extracted quantitatively into10 cm3 of n-butanol and the amount of cobalt was determined [10]. The organic phase containing Ni and Pb was shaken for 2 minutes with 10 cm^3 of 0.1N HCl for back extraction of nickel. Both the aqueous phases obtained at the time of first extraction and after back extraction were combined. After concentration, to the combined aqueous phase containing Ni and Pb , 1 cm3 of 0.5 % HINABH,2cm3 of buffer solution of pH 9.0 and excess of $SO4^{2-}$were added. The contents were diluted to 10 cm3 with distilled water.Ni(II) HINABH was extracted with 10cm3 of isoamyl alcohol and the absorbance was measured at 420 nm.[11]

The aqueous phase was evaporated to dryness. The residue was dissolved in 7 cm3 distilled water and 1 cm3 of 0.5% HINABH and 2cm3 of buffer solution of pH 9.0 were added. Lead was extracted into 10 cm3 of isoamyl alcohol. The absorbance of organic phase was measured against a reagent blank at 410 nm.

RESULTS AND DISCUSSION:

Beer's law, Sandell Sensitivity, Regression analysis and Stability of the complexes

Beer's law is obeyed in the concentration range of 0.2-5.0 ppm for cobalt,1-14 ppm for nickel and 2-16 ppm for lead respectively. The molar absorptivities and Sandell sensitivity of extracted species were 4980 $dm^3\,mol^{-1}cm^{-1}$ and 0.01185 ug $ml^{-1}cm^{-2}$ for Co, and 2169$dm^3\,mol^{-1}cm^{-1}$ and 0.024 ug $ml^{-1}cm^{-2}$for Ni and 4264 $dm^3\,mol^{-1}cm^{-1}$ and 0.0518 ug $ml^{-1}cm^{-2}$ for Pb .Regression coefficient of correlation values of cobalt, nickel and lead were 0.999, 0.997 and 0.986 respectively.(Figures1-3) The complexes of Co,Ni and Pb were stable for72 hrs ,48 hrs and 52 hrs. respectively.

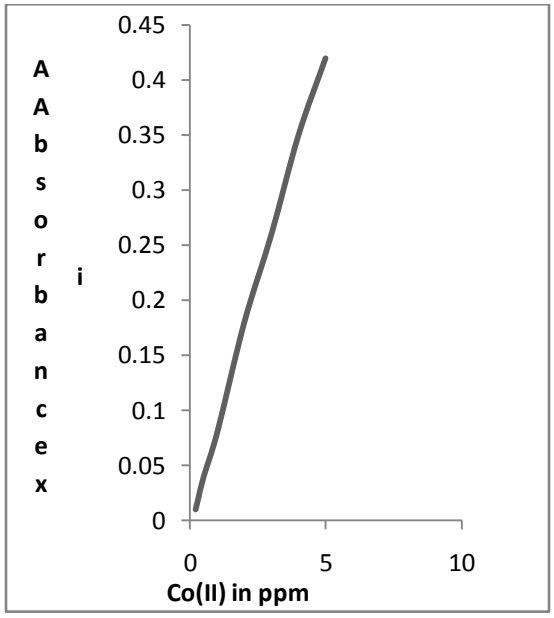

Figure 1.Calibration curve of Co(II) with HINABH

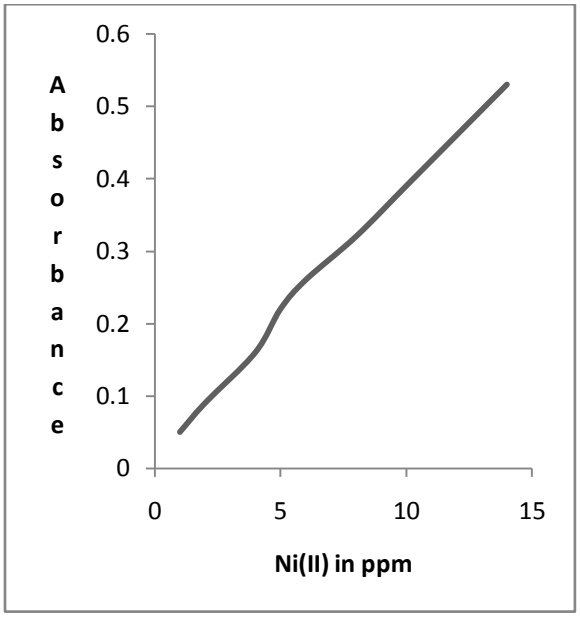

Figure 2.Calibration curve of Ni(II) with HINABH

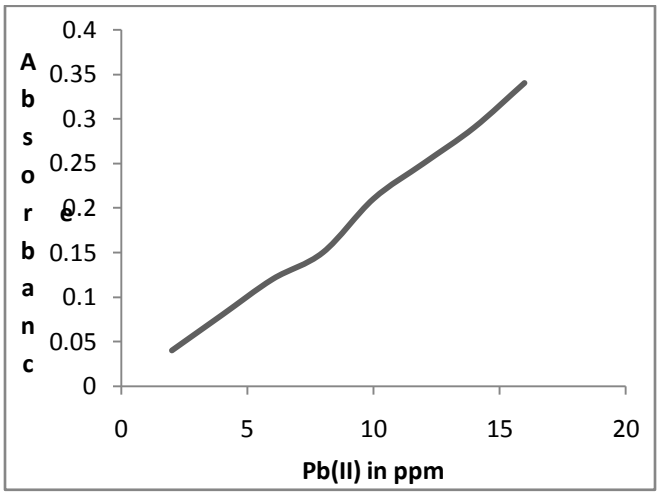

Figure .3.Calibration curve of Pb(II) with HINABH

Interference studies:

For the interference studies,20 mg/ml of anions and 10.0 mg/ml of were added individually for determination of 50 ug of Co, 100 ug of Ni and Pb.Table I Shows the tolerance limit of foreign ion to cause an error of not more than ±2% recovery of Co^{II},Ni^{II} and Pb^{II}.

Table 1 Interference Studies

Tolerance limit (ug ml^{-1})	Effect of diverse ions on CoII	Effect of diverse ions on NiII	Effect of diverse ions on PbII
20000	SCN^-,F^-,Cl^-,Br^-,I^-,SO_4^{--} ,IO_3^-,BrO_3^-,NO_2^-,NO_3^- ,$S_2O_7^{--}$,ClO_3^-	SCN^-,F^-,Cl^-,Br^-,I^-,SO_4^{--} ,IO_3^-,BrO_3^-,NO_2^-,NO_3^- ,$S_2O_7^{--}$,ClO_3^-	SCN^-,F^-,Cl^-,Br^-,I-,SO_4^{--},IO_3^- ,BrO_3^-,NO_2^-,NO_3^-,$S_2O_7^{--}$,ClO_3^-

	,Tartarte,Oxalate,Acetate	,Tartarte,Oxalate,Acetate	,Tartarte,Oxalate,Acetate
18000		Urea	-
15000	-	-	IO3-
10000	Ca^{2+},Cd^{2+},Zn^{2+},Li^{+},Mg^{2+} ,Mn^{2+},Bi^{3+}, W^{6+},Ba^{2+}	Ca^{2+},Cd^{2+},Zn^{2+},Li^{+},Mg^{2+}, Mn^{2+},Bi, $^{3+}$, W^{6+}	Ca^{2+},Cd^{2+}, Li^{+},Mg^{2+},Mn^{2+}, $UO2^{2+}$,Hg^{2+},Al^{3+},Co^{2+},Ba^{2+}, $Mo^{6+}Ru^{3+}$,Se^{4+}, Bi, $^{3+,}W^{6+}$
8000	Mn^{2+}, Al^{3+}	Al^{3+}	Citrate
5000	Ag^{+},Zr^{Iv}	Ag^{+}	Oxalate,$HPO4^{-}$
4000	-	-	Zn^{2+}
1000	Hg^{2+}, Mo^{6+},Cr^{3+}	Citrate	
500		-	V^{5+}, Cr^{3+}
100		Cr^{3+}	-

EDTA was masked with calcium chloride.Cu(II) was reduced to Cu(I) with sodium metadisulphite. Fe(II) and Fe(III) were masked with tartarate .Pd(II) was separated in the acidic range(0.1 N H2SO4).Co(II) was separated sequentially for determination of Nickel, Co(II) was masked with citrate for determination of Pb(II).PdII was separated in the acidic range and interference due to Pb(II) was removed with sulphate.

Determination of Co^{II}, Ni^{II} and Pb^{II} in synthetic mixtures and alloy

Synthetic mixtures containing CoII, NiII and PbII were prepared and amount was each was determined sequentially. Real samples containing Co, Ni and Ni, Pb were analyzed simultaneously in presence of each other as per the developed procedure.

The alloy sample (10-100mg) containing Ni,Pb, which was spiked with Co was taken in a beaker. To this 10 ml of aquaregia was added. The solution was evaporated to dryness. The residue was dissolved in 8 cm^3 of con.HCl and the solution was diluted and filtered. Masking agents were used whenever required. The filtrate was diluted with distilled water and Co, Ni,Pd were analyzed by the method developed. [Table2]

Table2.Sequential separation and spectrophotometric determination of Co,Ni and Pb

Amount of metal taken(µg/cm^3)			**Amount of metal found(µg/cm^3)**			***R.S.D**		
Co	Ni	Pb	Co	Ni	Pb	Co	Ni	Pb
50	100	20	49.30	101.00	20.50	0.68	0.57	0.98
40	60	30	40.60	80.70	32.50	1.64	0.41	1.27
20	40	40	19.30	39.00	43.00	1.73	1.48	1.26
10	30	50	10.70	28.70	51.50	1.53	1.16	0.82

Alloy sample	**Amount of metal present(µg/cm^3)**			**Amount of metal found µg/cm^3 ±SD)**		
(ITA Lab.)	Co	Ni	Pb	Co	Ni	Pb
**Phosphorusbronze(1436)	5.0	7.8	4.0	5.6±0.61	7.4 ± 0.34	7.4 ± 0.34
Phosphorusbronze(1408)	4.0	8.2	8.0	3.8±1.21	7.9±0.51	8.13± 0.31

* Average of 5 determinations, ** Average of 3 determinations

CONCLUSION

The proposed method for the sequential separation and determination of Co^{II}, Ni^{II} and Pb^{II}.is simple, rapid, sensitive and has the advantage of enabling a wide range of determination without the need for delayed color development or heating. The satisfactory applicability of the proposed procedure to the determination of Co^{II},Ni^{II} and Pb^{II} in various samples shows the utility of the method because of the low cost of the instrument, ease of handling, lack of need for consumables, and almost no maintenance .

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