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Liquid-liquid extraction and spectrophotometric determination of Co(II) with N,N'bis (O-hydroxy acetophenone) ethylene diimine derivative as an analytical reagent

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Abstract

A new analytical reagent N,N'bis (O-hydroxy acetophenone) ethylene diimine (HAPED) is proposed for the extraction and spectrophotometric determination of Co(II). The Reagent was synthesized and characterized by IR, NMR, elemental analysis as well as Mass spectrometry. HAPED reacts with Cobalt to give dark yellow colored complex which can be quantitatively extracted into Chloroform at pH 2.6. The organic extract shows maximum absorption at 400 nm where absorption due to similarly prepared reagent blank is negligible. The beer's law is followed in the concentration range 1-10 µg/ml of Co(II). The molar absorptivity and sandell's sensitivity of Co(II) -HAPED complex are 3195.26 Lit $mol^{-1}cm^{-2}$ and 0.0185 mg/cm^2 respectively. The proposed method is rapid, sensitive, reproducible, accurate and has been satisfactory applied for determination and separation of Co(II) in commercial mixtures, pharmaceutical samples and alloys.

Keywords: Cobalt (II), Spectrophotometric determination, HAPED reagent
Introduction

The importance of Cobalt in animal nutrition has lead to the development of procedures for its determination in minute amounts in soils, plants and animal tissues. Metal is used in corrosion resistant alloys and magnets. Cobalt is biologically more compatible with human systems. Cobalt is also used in electroplating. Present method is highly sensitive, selective, simple, rapid, and accurate than reported methods in the literature. Method can be effectively applied for determination of iron in synthetic mixtures, pharmaceutical samples and alloys.

There are certain reagents applied for determination of iron by solvent extraction method [2-5]. Atomic Absorption Spectroscopy [5-8] is often lacking in sensitivity and affected by matrix conditions of samples such as salinity. Extractive methods [9-10] are highly sensitive but generally lacks in simplicity.

Spectrophotometry is essentially a trace analytical technique and is one of the most powerful tools in chemical analysis. A wide variety of reagents have been proposed for the spectrophotometric determination of Co(II) . The extractive spectrophotometric analysis enables to separate desired metal ion, which is to be estimated in presence of other metal from samples. In the present work a novel analytical reagent N,N'bis(O-hydroxy acetophenone) ethylene diimine (HAPED), was used for the extractive spectrophotometric determination of Co (II). Developed method can be employed for efficient determination of Cobalt at µg level. The results of analysis obtained were compared with those obtained by known methods.

Experimental

Instruments: A Shimadzu 2100 UV-Visible spectrophotometer with 1.0 cm quartz cell was used for absorbance studies. An Elico LI-120 digital pH-meter was used for pH adjustment.

Synthesis of Reagent: The reagent was synthesized by O-hydroxy acetophenone and ethylene diamine in methanol in 2:1 molar proportions are mixed in Round Bottom Flask. Shake the flask for 10 to 15 minutes. Immediately dark yellow colour solid is obtained which is poured in ice cold water. The solid obtained is separated by filtration and washed with cold water and the product is recrystallised from ethanol. The yield was about 90%. It is then characterized and used for extractive spectrophotometric determination of Co (II). A stock solution of reagent with concentration 0.1% was prepared in methanol. The scheme of reaction is as shown in (Fig.1).

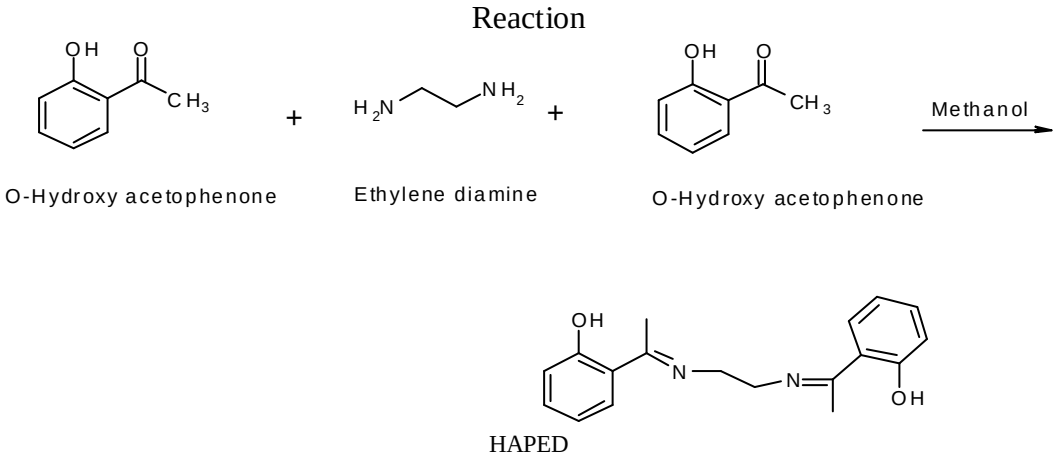

Fig.1 Synthesis of Reagent N,N'bis(O-hydroxy acetophenone) ethylene diimine (HAPED)

Preparation of stock solution

A weighed quantity of Cobalt Nitrate was dissolved in double distilled water Containing dilute Nitric acid and then diluted to desired volume by double distilled water. The solution was then standardized by Nitroso R-salt method.

Recommended procedure

Mix 2 cm^3 aqueous solution containing 1-100mg of Cobalt and 1cm^3 of 0.1% methanolic solution of reagent in 25 cm^3 beaker. Adjust the pH of the solution to required value with buffer solution Make the final volume 10 cm^3. Transfer the solution into 125 cm^3 separating funnel and equilibrate for 1min with 10 cm^3 Chloroform. Allow the two phases to separate and measure the absorbance of organic phase containing the complex at 400 nm against reagent blank

Preparation of calibration plot

The calibration curve was prepared by taking known amount of Cobalt which was treated as described in the procedure. A graph of absorbance against concentration was prepared (Fig.2). The concentration of the unknown iron solutions is determined from the calibration plot.

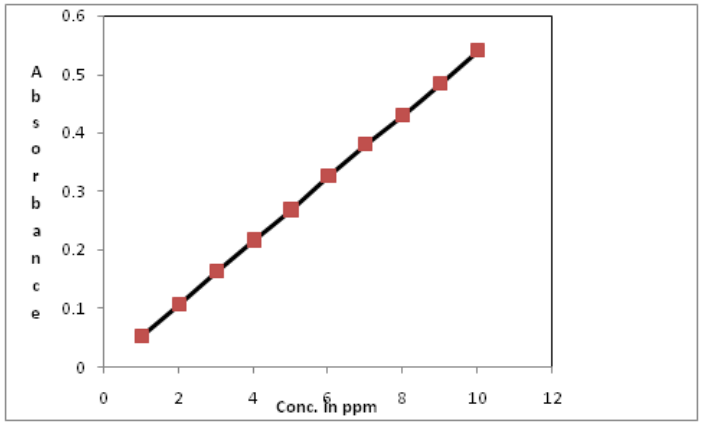

Fig.2 Calibration Plot of Co (II) in mg/ml against Absorbance

Composition of the extracted species

The composition of the extracted species was determined by using the Job's continuous variation method and verified by mole ratio method and slope ratio method. These methods show that the composition of Co (II): HAPED regent is 1: 2 (Fig. 3).

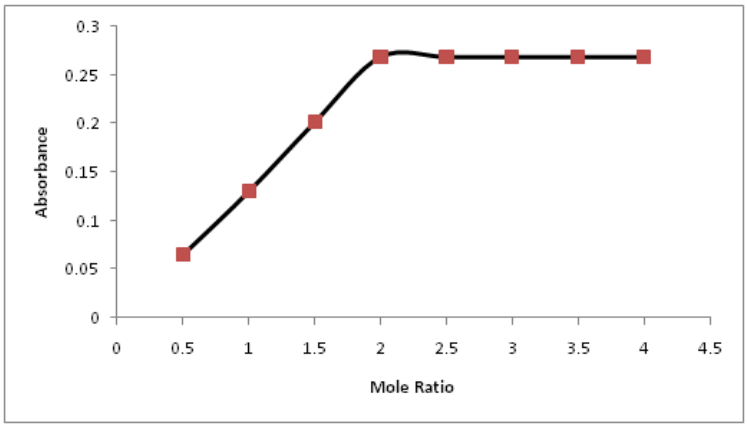

Fig.3 Mole Ratio Method

Effect of foreign ions

The effect of diverse ions on the Cobalt (II) determination was studied, in presence of a definite amount of a foreign ion. Various cations anions were investigated in order to find the tolerance limit of these foreign ions in the extraction of Cobalt (II) (Table 2). The tolerance limit of the foreign ion was taken as the amount required causing an error of not more than 2% in recovery of Cobalt (II). The ions which interfere in the spectrophotometric determination of iron were masked by using appropriate masking agents (Table 3).

Comparison between reagents

Various reagents were investigated by the earlier researchers for removal of Cobalt (II). The proposed reagent N,N'bis(O-hydroxy acetophenone) ethylene diimine (HAPED) is found more superior as that of reported regents and are presented in (Table 5).

Applications

The present method was applied for determination of amount of Cobalt(II) in various samples of alloys, commercial mixtures, injection vial, tablets. The results obtained were in well agreement with standard methods, Table- 4. Every result is average of independent determinations.

Table 1

Sr. No.	Different parameters Studied	observation
1	Solvent	Chloroform
2	pH	2.6
3	Equilibrium time	1 min.
4	Stoichiometry M:L	1:2
5	95% confidence limit	±0.1532
6	Reagent Conc.	0.1%
7	Volume of Rgt.	2ml
8	Average of 7 determination	9.957
9	Stability of the complex	37 hrs.

Table 2 Effect of foreign ions

S. No.	Interfering ions	Tolerance limit
1	SO_4^-,SO_3^-, CN^-, I^-, Cl^-, phosphate, Tartarate BrO_3^-, ClO_3^-, IO_3^-, NO_2^-, Br^-, NO_3^-,	15
2	Oxlate	12
3	Acetate,	06
4	Mg(II), Mo(VI), Ce(IV), Ca(II), As(III), W(VI), Cd(II)	10
5	Al(III), Bi(III),	10
6	Ag+,K+, Na+,	6
7	Fe(II), Ni(II), Mn(II), Cd(II), Pd(II),Cu(II), EDTA	Interfere strongly

Table-3: Effect of masking agent

Sr. No.	Interferring Ions	Masking Agents
1	Cd(II)	Thiourea
2	Pd(II)	Thiourea
3	Ni(II)	DMG

4	Cu(II)	Sodium thiosulphate
5	EDTA	Boiled with conc.HNO_3
6	CN^-	Boiled with conc.HNO_3 And formaldehyde

Table 4 Applications

Sr. No.	Sample	Standard method	Present method
1	Steel alloy	9.2%	9.0%
2	High Speed steel	9.64µg	9.63µg
3	Vitamin B-12 Injection	50	48
4	Ni(100) + Co(100)	100ppm	99.50ppm
5	Ni(50) + Pd(50)+Co(50)	50ppm	49.32ppm

Table 5 Comparison between regents

Sr./ Ref. No.	Reagent	Remark
1	Isonitroso Dimedone	Fe^{+3},Cu^{+2},Mo^{+6},V^{+5} Interfere
2	Nitroso –R-Salt	Cu, Ni interfere
3	1-(2-Pyridyl –Azo)-2-naphthol	Ni interfere
4	2 – Theonyl trifluro acetone	Cr^{+3},Ni^{+2},Fe^{+2}, Mn^{+2} Interfere
5	Picramineazo-Diamino pyridine	$Fe^{+3,}Al^{+3}$ Masked with EDTA
6	4-(5- Chloro-2-pyridylazo) - m-phenelene diamine	Fe^{+3},Cr^{+3},W^{+6} Interfere
7	2-Pyridyl -2-thienyl β- ketoximes	Pd^{+2},Pt^{+4} Interfere
8	Pyrazine Carboxamide	Pb^{+2}, Bi^{+3}, Hg^{+2} Interfere

Conclusion

The results obtained show that the newly developed method in which the reagent N,N'bis(O-hydroxy acetophenone) ethylene diimine (HAPED)was synthesized, can be used for quantitative estimation of Co (II). The proposed novel reagent is found to be more effective over reported reagent from earlier investigators. The proposed method is simple, rapid and requires less volume of organic solvent. The method is also precise, less time consuming and easily employed anywhere as does not require sophisticated instruments.

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