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Development of a new analytical method for extractive spectrophotometric determination of Co(II) using 4-[({4-[n-(3-oxo-1,2-oxazolidin-4-yl)carboximidoyl] phenyl}methylidene) amino]-1,2-oxazolidin-3-one (OOCPMAO) as a reagent

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ABSTRACT

Cobalt is extracted quantitatively at pH 6.4 using 4-[({4-[N-(3-oxo-1,2-oxazolidin-4-yl)carboximidoyl] phenyl}methylidene) amino]-1,2-oxazolidin-3-one (OOCPMAO) as a reagent. The absorption maxima was obtaind at 425 nm. The coloured complex was then extracted in chloroform and determined by spectrophotometer. The calibration plot was found between 1 to 10 ppm. The molar absorptivity of colored species was found to be 4.9359 X 10^3 Lit mol^{-1} cm^{-1} and the sensitivity of the method as defined by Sandell's was 8.391 X 10^{-5} $\mu g / cm^2$. The stability of complex was up to 72 hours. 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, it was 1:2. The method has been applied to analysis of cobalt in superalloys, paints, varnishes, and inks. The result was competable with earlier known methods.

KEYWORDS

Cobalt;
Spectrophotometer;
Solvent extraction;
OOCPMAO.

INTRODUCTION

The review of the above literature survey reveals that Cobalt forms chelate complexes with many organic reagents which are bonded through N, O and/S atoms. Out of the reagents used for the extraction & spectrophotometric determination of Co (II), many reagents suffer limitations as of, interference by other ions, Lengthy and tedious procedure for reagent preparation, Requirements of surfactants, the stability of complex with time, Solvent is not convenient to use, Complex formation requires more time. Survey of applications of Cobalt leaves a scope for as many reagents as can be applied for the extraction and spectrophotometric determination of Cobalt.

The present work describes a spectrophotometric method for quantitative estimation of Co (II) with OOCPMAO.

EXPERIMENTAL

Apparatus: Glassware used in the present investigations was all made up of Borosil. The burettes pipettes and standard flasks were calibrated In accordance with method described in voge1[1]. An analytical balance of Metteler Toledo (modelAG-285) used for weighing the samples. pH study was carried out by using pH meter of make Labindia PICO. All measurements of absorption spectra were made on one cm silica cells or 1 cm glass cells. The spectrophotometer[2] used was UV-Varian 50 probe. The calibration of the spectrophotometer was checked measuring absorption spectrum of 0.004% solution of potassium chromate in 0.05 M potassium hydroxide solution and also with 0.0058% solution of potassium per magnate in 1M sulphuric acid. The observed spectrum was in good agreement with the spectrum reported in the literature[3].

RESULTS AND DISCUSSION

pH –study

The extraction of cobalt with OOCPMAO was carried out over the pH range of 1 to 11. It reveals that 99 % of the metal is extracted into organic phase in the pH range 8.5-9.0. It was found that complex[4] does not form below pH 4.0. Since absorbance shown by the solution in pH range 1 to 4 was negligible, the analytical work for the estimation of cobalt was carried out at pH 6.4. (Figure 1)

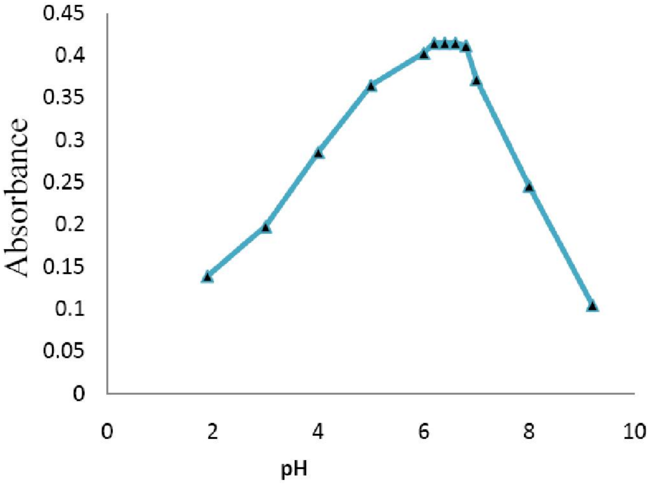

Figure 1

Absorbance maxima

The absorption spectrum[5] of Co – OOCPMAO complex in chloroform shows the maximum absorbance at λ = 425 nm. The absorbance due to the reagent at this wavelength was negligible. Hence, 425 nm was selected for the absorbance measurement in spectrophotometric determination of Co (II) against the reagent blank.

Solvent study

Various solvents were tried to determine the maximum extraction of cobalt. Chloroform was found to be the most suitable solvent as it showed the highest extraction. The extraction of cobalt varied from maximum to minimum for the solvents in the order of chloroform >carbon tetrachloride > n-butanol > benzene > ethyl acetate > toluene > xylene > iso-amyl alcohol >ether> pet ether.

Calibration curve

Different amounts of the cobalt (ii) from 1 to 10 μg/ cm^3 were extracted quantitatively under optimum experimental conditions and the chloroform extract was measured at 425 nm against reagent blank. The plot of absorbance against concentration of cobalt gave a straight line indicating that the beer's law is obeyed in this range. The molar absorptivity of colored species was found to be 4.9359 x 10^3 lit mol^{-1} cm^{-1} and the sensitivity of the method as defined by sandell's was 8.391 x 10^{-5} μg / cm^2.

Equilibration time

The absorbance by co – oocpmao complex was checked by varying the time of equilibration from 30 sec to 5 min. It was observed that equilibration time of 2 min was found to be optimum for complete extraction of 5 μg cobalt.

Stability of complex with time

The study of stability of complex with variation in time showed that the complex was stable up to 72 hours after which absorbance decreases slowly.

Effect of reagent concentration

The effect of variation in the concentration of OOCPMAO in the range of 0.1 to 3.0 cm^3 of 0.1% OOCPMAO on the extraction and color development was tried. It was found that 1 cm^3 of 0.1% OOCPMAO was sufficient for complete extraction and colour development Hence for subsequent studies 1 cm^3 of 0.1 % OOCPMAO was employed.

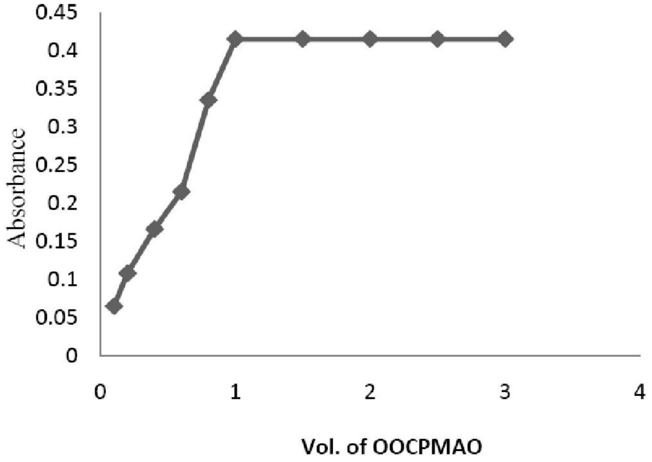

Figure 2

Nature of the extracted species

The absorption spectra for the chloroform extracts of the aqueous solution containing metal : OOCPMAO in the molar concentration ratio 1 : 1, 1 : 2, 1:3, and 1:4, indicated that under the experimental conditions, the nature of absorption spectrum remains identical irrespective of the reagent concentration with no change in the value of λ_{max}. However, the absorbance increases as the molar reagent concentration increases with respect to the metal concentration. The composition of the extracted species was determined by using the Job's continuous variation method[6] and verified by mole ratio method[7] and slope ratio method. Job's continuous variation method shows a sharp maxima at 0.33 mole fraction of cobalt indicating that the colored complex extracted into chloroform was formed by reaction of Co (II) and OOCPMAO in the ratio of 1:2,the plot of absorbance against mole ratio of OOCPMAO : Co (II) shows a sharp break corresponding to mole ratio 1:2, which supports the composition of the extracted species as Co- OOCPMAO.

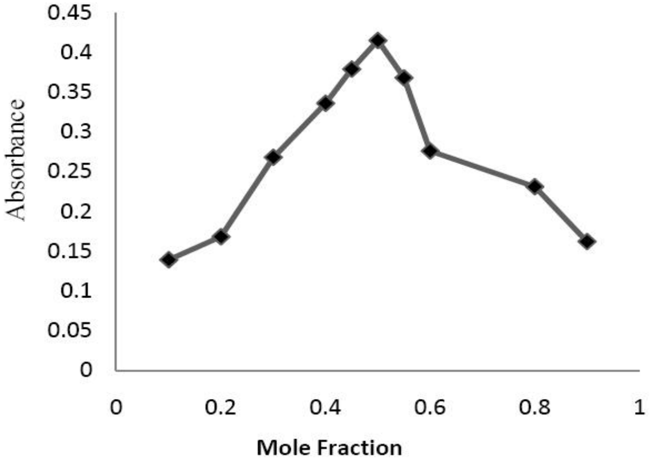

Figure 3

Interference study

The effect of diverse ions on the cobalt (II) determination was studied, in presence of a definite amount of a foreign ion. Various cations and anions were investigated in order to find the tolerance limit of these foreign ions in the extraction of cobalt. The tolerance limit of the foreign ion was taken as the amount required causing an error of not more than +2% in the recovery of cobalt (II).

TABLE 1 : Masking agents used to remove interference

Sr. No	Interfering Ion	Masking agent added
1	Cu(II)	Sodium thiosulphate
2	Ag(I)	Potassium iodide
3	Cd(II)	Potassium iodide
4	Pb(II)	Sodium thiosulphate
5	Mn(II)	Sodium fluoride
6	Ce(IV)	Sodium fluoride
7	Cr(II)	Ammonium acetate
8	Citrate	Sodium molybdate
9	Tartarate	Sodium molybdate

Precision, accuracy and sensitivity

The precision and accuracy of the spectrophotometric method were tested by analyzing the solutions containing known amount of cobalt. Average of ten

TABLE 2 : Determination of Co (II) using OOCPMAO from different samples

Sr. No.	Sample	Amount of Co (II)	
		Standard method	Present method
I	Cobalt alloys		
1	Cobalamine	69.2%	69.8%
2	Steel	9.56%	9.55%
II	Vegetable Oil	0.0018%	0.0017%
III	Synthetic mixture		
1	Co(10)+Zn (10)	9.98ppm	9.96ppm
2	Co (10) + Mo (10)	9.98ppm	9.96ppm
3	Co(10) +Mg(10)	9.98ppm	9.96ppm
IV	Pharmaceutical Samples		
1	Surbex –T	0.327mg	0.326mg
2	Vitamin B12	50mg	49mg

determinations with 5 μg Co in 10 cm^3 solution varies between 50 μg and 50.0896 μg at 95% Confidence limit.

CONCLUSION

The results obtained show that the newly developed method in which the reagent OOCPMAO used can be effectively used for quantitative extraction and estimation of Co(II) from aqueous media. The proposed method is quick and requires less volume of organic solvent. The results show good agreement with the standard methods. The method is very precise, faster and simpler than other methods.

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