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NOTE

Micellar Spectrophotometric Determination of Cobalt in the samples of Alloys and Vitamins with N-(2′-thiazolyl)-2-hydroxybenzamide

S. KHATURIA, M. CHAHAR, K.P. SONI and N. BHOJAK*
Postgraduate Department of Chemistry, Dungar College, Bikaner-334 001, India

In the present investigation, potential applicability of a novel reagent N-(2′-thiazolyl)-2-hydroxybenzamide (NTHB) for the spectrophotometric determination in micellar medium has been reported. The presence ot a non-ionic surfactant Triton X-100 has not only enhanced the solubility and stability of the cobalt^{2+}-NTHB complex, but a marked increase in the absorption intensity has been observed. The best results were obtained in the pH range 6–7 and Beer's law is obeyed up to 0.95 ppm with optimum concentration range 0.11–0.85 ppm for the accurate determination of cobalt at λ_{max} 424 nm. Sandell's sensitivity and molar extinction coefficient have been found to be 0.0010 $\mu g/cm^2$ and 6.3×10^4 mol^{-1} cm^{-1} respectively. Effect of time and diverse ions on the absorption intensity have also been studied. The method is selective and successfully been applied to determine cobalt in the samples of alloys and vitamins.

Key Words: Cobalt, N-(2′-thiazolyl)-2-hydroxybenzamide (NTHB), Triton X-100.

Amide group containing reagents have been used as potential extractants for the separation and determination of many metal ions[1–4]. N-(2′-thiazolyl)-2-hydroxybenzamide (NTHB) has been synthesized by the reaction of 2-aminopyridine with salicylic acid. It is a weak acid and is found to be soluble in alcohol, acetone and in chlorinated hydrocarbons such as chloroform. It also dissolves in alkaline aqueous media but is practically insoluble in water at pH less than 7. In the presence of surfactants (above CMC) its solubility has been found to be increased significantly. Further, a marked increase in absorption intensity has also been observed. This phenomenon of micellar solubilization with increasing absorption intensity has been used to develop a novel and selective spectrophotometric method for determination of cobalt(II) in alloys and vitamins.

Stock solution of N-(2′-thiazolyl)-2-hydroxybenzamide NTHB (0.1 M) was prepared by dissolving its requisite amount in ethanol. The three surfactants used are Triton X-100 solution (1.8×10^{-2} M, 100 CMC), sodium dodecylsulphate (SDS) (0.50 M, 75 CMC) and hexadecyltrimethyl ammonium bromide (HTAB) (9.2×10^{-2}M, 100 CMC). Acetate buffers prepared by mixing 0.2 M sodium

acetate and 0.2 M acetic acid were used to maintain the pH in acidic medium. Borate buffers were prepared by mixing boric acid and sodium hydroxide and were used to maintain the pH in alkaline medium. All spectra were recorded as per recommended procedure.

Recommended procedure: In 25 mL standard flasks, add 2.5 mL of buffer solution of pH 6.0, a suitable aliquot of cobalt, 2.5 mL 1.8×10^{-3} M triton X-100 solution and 0.5 mL 5×10^{-3} M NTHB solution. Dilute the solution to volume with double distilled water and then record the absorbance at λ_{max} 424 nm. For finding the precision and accuracy, repeat the experiments five times each.

Absorption spectra of solutions containing cobalt-NTHB complex in micellar medium were recorded against reagent blanks by adjusting the pH in the range of 2.5–10.0. The absorbance was found to increase with increase in pH up to 6.0 and remained constant up to pH 7.5; thereafter it started decreasing. For further studies acetate buffer of pH 6.0 was selected. Absorption spectra of the cobalt-NTHB complex were recorded in the presence of different surfactants, nonionic triton X-100, cationic HTAB and anionic SDS. Maximum absorbance was found to be with triton X-100. Effect of varying concentration of NTHB was also studied by increasing the concentration of NTHB, at a fixed metal ion concentration and triton X-100 concentration and at pH 6.0. It was observed that for maximum complexation, 6 times molar excess of NTHB was required. However, in subsequent studies, 10 times molar excess of NTHB was maintained. The influence of amount of ethanol on absorbance of the Co-NTHB complex was investigated because NTHB was used in ethanolic medium. Absorbance was found to decrease slightly when ethanol concentration was increased from 0.5% to 12% v/v. In all other experiments, ethanol concentration was maintained at 2% v/v. Beer's law was found to be valid from 0.11 to 0.95 ppm of Co^{2+} ions and the optimum range for the accurate determination of Co^{2+} is recommended as 0.11–0.85 ppm. The complex is found to be reasonably stable over a period of approximately 90 min during which the analysis can easily be completed. The complex solution however is unstable when stored for prolonged periods and should be prepared fresh before analysis. The effect of various ions on the absorption intensity of the cobalt-NTHB complex has been investigated following the recommended procedure. The order in which reagent, surfactant, metal ion and buffer solution were mixed has no influence on absorbance intensity. However, in some cases a precipitate appears, particularly when iron and nickel ions are found in matrices above 10 ppm. It dissolves by the addition of surfactant solution. In order to avoid any complication, the following order, *i.e.*, buffer solution, metal solution, surfactant solution and reagent solution, has been preferred.

Determination of cobalt in alloys: Alloy solution was prepared as per reported procedure.[5] An aliquot was taken out from this solution and the amount of cobalt was determined as per recommended procedure (Table-1).

Determination of cobalt in vitamins: One capsule of the vitamin tablet was transferred into a 100 ml Erlenmeyer flask. It was digested several times with nitric acid and sulphuric acid. Most of the acid was evaporated and the rest was neutralized with 1% NaOH solution; finally the total volume was made up with

double distilled water. An aliquot was taken out from this solution and the amount of cobalt was determined as per recommended procedure (Table-2).

TABLE 1
ANALYSIS OF COBALT IN ALLOYS†

Name of sample	Certified value (%)	Observed value (%)	RSD (%)
JSS 654-7 Stainless steel	0.45	0.42	0.92
JSS 655-4 Stainless steel	0.28	0.25	0.80
JSS 607-6 Stainless steel	4.27	4.17	1.15

†Each value is the mean of five simultaneous determinations.

TABLE 2
ANALYSIS OF COBALT IN VITAMINS‡

Sample present (μg/mL)	Amount of cobalt present (μg/mL)	Vitamin found (μg/mL)	Recovery (%)
1	22	20.3	92
2	23	21.4	93
3	21	19.3	92

‡Each value is the mean of five simultaneous determinations.

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REFERENCES

1. M.L. Grayeski and J.K. Devasto, *Anal. Chem.*, **29**, 791 (1982).
2. H. Hirose, K. Hiratani, K. Kasuga, K. Saito, T. Koike, E. Eimura and Y. Nagawa, *J. Chem. Soc., Dalton Trans.*, 2679 (1992).
3. K.S. Patel and R.K. Mishra, *Talanta*, **29**, 791 (1982).
4. B.S. Garg, N. Bhojak, P. Dwivedi and V. Kumar, *Transition Met. Chem.*, **24**, 463 (1999).
5. K.P. Soni, N. Bhojak, M. Chahar and S. Khaturia, *Asian J. Chem.*, **15**, 1030(2003).

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