April 2006 Volume 2 Issue 2

Trade Science Inc.

Analytical CHEMISTRY

An Indian Journal

Full Paper

ACAIJ 2(2), 2006, [45-47]

Extractive Spectrophotometric Determination Of Cobalt(II) With 1-(Benzimidazol-2-Yl) Ethanone Thiosemicarbazone

K.Syamasundar
Department of Chemistry,
JNTU College of Engineering,
Kukatpally, Hyderabad- 500072 (INDIA)

M.Adharvana Chari, D.Shobha
Department of Chemistry,
JNTU College of Engineering,
Kukatpally, Hyderabad- 500072 (INDIA)
E-mail: drmacj@yahoo.co.uk

Received: 25th July, 2005
Accepted: 4th August, 2005
Web Publication Date: 5th April, 2006

ABSTRACT

1-(Benzimidazol-2-yl) ethanone thiosemicarbazone is used as a chromophoric reagent for the extraction and spectrophotometric determination of Cobalt (II). Cobalt (II) forms 1:2 complexes with the reagent at pH 9.0. The 1:2 metal complex has λ_{max} value at 388 nm. The Cobalt (II) thiosemicarbazone complex is stable for 72 h, with molar absorptivity of 5.89 x 10^3 lit mol^{-1} cm^{-1} and Beer's law is obeyed in the range of 0.3 to 6.2 ug/ml. The effect of various foreign ions is also studied. The stability constant of the complex is determined.The method is applied for the determination of Cobalt(II) in medicinal samples and the results are compared with atomic absorption spectrophotometer.

KEYWORDS

Cobaltous chloride;
1-(Benzimidazol-2-yl) ethanone thiosemicarbazone;
Spectrophotometric;
determination;
Medicinal samples;
AAS.

INTRODUCTION

Thiosemicarbazones are important sulfur donar ligands. Various thiosemicarbazones have been reported in literature[1] for the spectrophotometric determination of Cobalt (II). The use of thiosemicarba zones in inorganic analysis has been reviewed by Singh et al[2]. These reagents find wide range of applications in medicine[3] The different thiosemicarba zones are employed for the spectrophotometric determination of different metal ions[4-7] due to the ability of thiosemicarbazones in forms intense coloured complexes with various metal ions. Benzimidazoles are also used in the inorganic analysis. Among the different thiosemicarbazones 1-(Benzi midazol-2-yl) ethanone thiosemicarbazone (BET) is sensitive and new reagent which is used by us for the spectrophotometric determination of Cobalt (II) in medicinal samples.

EXPERIMENTAL

Analytical grade chemicals were used in studies. The pH measurements were made by using digital

Full Paper

pH meter Model DPH-500. The absorbance measurements were made by using U.V.visible recording spectrophotometer Model U-3210, Hitachi Corporation, Tokyo, Japan. Z-6100 (Hitachi, Japan) type of AAS is used for the determination of cobalt metal.

Reagents

Cobalt (II) solution

About 4.04 g cobaltous chloride hexahydrate ($CoCl_2 \cdot 6H_20$) is dissolved in distilled water and made up to one litre to get stock solution. Lower concentrations of Co (II) solutions are prepared by diluting the stock solution with double distilled water. The stock solution is standardized volumetrically by using oxine[8].

BET solution

The reagent was prepared by refluxing methanolic 1-(Benzimidazole-2-yl) ethanone and aqueous thiosemicarbazide in CH_3COOH medium using the procedure used by Chari et al[9] (m.p. 220°C). 1 $x10^{-2}$ M solution is used in the studies. The metal ion concentration is 5 x 10^{-4} M.

Buffer solutions

Buffer solutions were prepared by adopting the standard procedures[10]. The various solutions employed in the preparation of buffer solutions were 1 M HCl and 1 M CH_3COONa (pH 1.0-3.0), 2 M CH_3COOH and CH_3COONa (pH 3.5-6.0), 0.2 M CH_3COOH and 1 M CH_3COONa (pH 7.0), 2 M NH_4Cl and 2 M NH_4OH (pH 8-12).

Preparation of sample solutions

Medicinal sample

Neurobion and ZB LAC ($B_2+B_6+B_{12}$) medicines are analysed for the determination of cobalt. A known weight of a medicinal sample is treated with a few ml of 0.3N H_2SO_4 in a small beaker. The contents are heated on a hot plate untill the fumes of H_2SO_4 cease to evolve. The contents are diluted with a little distilled water and filtered through whatman No.41 filter paper in to 100 ml standard flask and is made up to the mark with distilled water. This solution is used for further studies.

General Procedure

Different aliquots[11] of 10 ml solutions each containing constant volumes of 5.0 ml buffer pH (9.0), 2 ml of 2 $x10^{-2}$ M reagent and varying volumes of cobalt (II) solution containing 10 to 100 μg are prepared. The solutions are shaken twice with two 5 ml portions of $CHCl_3$ for 60 seconds. The organic phases are dried and absorbances are taken at 388 nm against reagent blank. A linear plot passing through the origin is obtained when absorbance is plotted against the amount of the metal ion.

RESULTS AND DISCUSSION

The absorption spectra of the BET and its cobalt complex are recorded in the wavelength range 300 to 500 nm individually against buffer and reagent blank respectively. The reagent showed the negligible absorbance and cobalt (II) complex showed absorbance at 387 nm. Job's and mole ratio methods are applied for the determination of composition of the complex.

The cobalt metal forms complex with BET in 1:2 ratio (M:L) under the experimental conditions. The complex colour is stable for 72 h, and absorbance remains constant in the pH range 8.0 to 9.5. The studies are carried out at pH 9.0 using ammonical buffer. A ten fold molar excess of the reagent is used to obtain maximum colour intensity. The method obeyed Beer's law in the range of 0.3 to 6.2 μg/ml. The molar absorptivity of the cobalt (II) complex is found to be 5.89 x 10^3 lit mol^{-1} cm^{-1}. The stability constant of the complex is 4.942 x 10^{16}.

Effect of foreign ions

The effect of foreign ions in the determination of 5.893 μg/ml of cobalt (II) was studied. The tolerence limits of other anions and cations are represented in TABLE 1.

Applications

Known quantity of ZB-LAC is treated with HCl and diluted to with water into 100ml volumetric flask and used for the spectrometric determination.

Cobalt present in medicinal samples was determined by simple known procedure. Into 25ml sepa-

TABLE 1: Tolerence limit of diverse ions in the determination of cobalt (II). Amount of Co (II) = 5.893 µg/ml pH=9.0

Foreign ion	Tolerance limit (µg/ml)	Foreign ion	Tolerance limit (µg/ml)
Ag(I)	Interferes	Bromide	160
Al(III)	8	Carbonate	240
Ba(II)	549	Chloride	71
Ca(II)	200	Citrate	2
Cr(III)	10	DMG	1
Cu(I)	1	EDTA	Interferes
Cd(II)	1	Fluoride	12
Fe(III)	5	Iodied	180
Hg(II)	60	Nitrite	92
K(I)	390	Nitrate	124
Mg(II)	150	Oxalate	616
Mn(II)	142	Phosphate	1500
Mo(VI)	40	Sulphite	288
Na(I)	460	Sulphate	280
Ni(II)	24	Thiourea	152
Pb(II)	6	Thiocynate	23
V(V)	Interferes		
Zn(II)	1		

rating funnel containing 5ml buffer and 2 ml of 2 x 10^{-2} M reagent solution, known quantity of medicinal sample is added. The resulting solution is shaken twice or thrice with 10ml of $CHCl_3$ for 60 seconds. The organic phase absorbance is measured at 388 nm against reagent blank. The amount of cobalt (II) present in sample is computed from the predetermined calibration plot. The results are compared with AAS method and shown in TABLE 2.

TABLE 2: Medicinal sample analysis for the determination of Cobalt (II)

Sample	Amount of Cobalt (II) found (ppm)		
	BET Method	AAS Method	Difference
1. ZB-LAC (B_2+B_6+B_{12})	27.0	27.3	0.3
	27.2	27.4	0.2
	27.2	27.2	0.0
	27.4	27.4	0.0
Average	27.2	27.3	0.1
2. Neurobion	14.10	14.40	0.3
	14.20	14.30	0.1
	13.80	14.60	0.8
	14.70	14.70	0.0
Average	14.20	14.50	0.3

ACKNOWLEDGMENT

The authors thank to the Principal, JNTU College of Engg, Hyderabad for providing the laboratory facilities.

REFERENCES

[1] B.K.Reddy, C.Jaya Kumar, L.S.Sarma, A.Varada Reddy; Asian J.Chem., **9**, 487 **(1997)**.
[2] R.B.Singh, B.S.Garg, R.P.Singh; Talanta, **25**, 619 **(1978)**.
[3] K.Butler; US Patent, 382(3), 266 **(1968)**.
[4] K.H.Reddy, D.V.Reddy; Indian J.Chem., **22**, 824 **(1983)**.
[5] A.Admann, B.Filerat; Chim.Acta Turc., **3**, 51 **(1975)**.
[6] A.Varada Reddy et al.; Asian J.Of Chem., **9**, 218 **(1997)**.
[7] A.V.Reddy, Y.K.Reddy; Talanta, **33**, 617 **(1986)**.
[8] A.I.Vogel; 'A Text Book of Quantitative Analysis', Longmans, 309, 329, 389, 479 **(1962)**.
[9] K.Syamasundar, M.Adharvana Chari; J.Indian Chem. Soc., **78**, 32 **(2001)**.
[10] D.D.Perrin, Boyd Dempsey; 'Buffers for pH Metal Ion Control', Chapman and Hall, London, 128-134 **(1974)**.
[11] A.V.Reddy et al; Asian J.Chem., **9**, 491 **(1997)**.