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Extractive and Spectrophotometric Determination of Cobalt (II) Using 2-(5- Bromo-2- Oxoindolin-3-Ylidene) Hydrazinecarbothioamide as Analytical Reagent

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ABSTRACT

2-(5-bromo-2-oxoindolin-3-ylidene) hydrazine carbothioamide, [5-Bromoisatin thio semi carbazone (HBITSC)] extracts Cobalt (II) quantitatively (99.5%) into n-amyl alcohol. The n-amyl alcohol extract shows an intense peak at510nm (λ max). Beer's law is obeyed over the Co (II) concentration range 1.0 – 5.0ug mL^{-1}. The molar absorptivity is 4125.7 L mol^{-1} cm^{-1} at 510 nm and Sandel's sensitivity is 14.28 ng cm$^{2\ -1}$. The composition of extracted species found to be 1:2 (Co: HBITSC) by Job's continuous variation and Mole ratio method. Interference by various ions has been studied. The proposed method has been applied for the determination of Co (II) in pharmaceutical samples.

Keywords: HBITSC, Cobalt, Spectrophotometry, Molar Absorptivity.

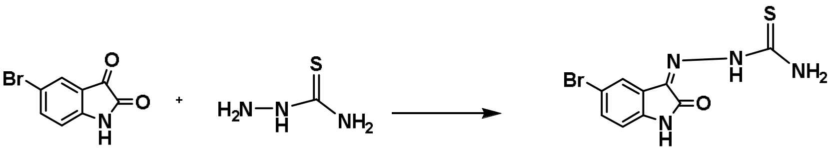
INTRODUCTION

Various reagents [1-23] are available for the spectrophotometric determination of cobalt. A solvent extraction is becoming important separation technique in chemistry. During the past two decades, considerable attention has been paid to the chemistry of the metal complexes of Schiff bases containing nitrogen and other donor atoms and is also used as an efficient analytical reagent in trace analysis of some metal cations [1-23]. 2-(5- bromo-2- oxoindolin-3-ylidene) hydrazine carbothioamide, (HBITSC) has been used for the development of extractive spectrophotometric methods for the determination of nickel (II) [11] and palladium (II) [12].In the present communication, we describe the extractive spectrophotometric determination of Co(II) with 2-(5- bromo-2- oxoindolin-3-ylidene)hydrazine carbothioamide [HBITSC)].

MATERIALS AND METHODS

ELICO - SL 159 spectrophotometer with optically matched quartz or glass cells of 1cm path length were used for absorbance measurements. An ELICO LI-127 pH meter was employed for pH measurements.

Synthesis of 2-(5-Bromo -2-Oxoindolin-3-ylidene)Hydrazine Carbothioamide (HBITSC): Schiff base ligand HBITSC was synthesized by refluxing equimolar amount of ethanolic solution of 5-Bromoisatin with thiosemicarbazide for 4 - 5 h. On cooling the reaction mixture, a sharp yellow crystal product separated out (80%, yield) which was collected by filtration. The resulting HBITSC was recrystallized using aqueous ethanol as the procedure recommended by Vogel [13]. The product was characterized by elemental and spectral analysis.



5-bromoindoline-2,3-dione

(*Z*)-2-(5-bromo-2-oxoindolin-3-ylidene)hydrazine-1-carbothioamide

hydrazinecarbothioamide

Its solution was prepared in Dimethylformamide (DMF). A stock solution of Co (II) was prepared by dissolving cobalt acetate in water containing dilute acetic acid and it was standardized by thiocyanate – pyridine method gravimetrically [14]. Working solutions of Co (II) were made by suitable dilutions. All other reagents used were of AR grade and all the solutions were prepared in doubly distilled water.

Extractive Spectrophotometric Determination of Co (II): To an aliquot of aqueous solution containing 10.0 to 50.0 µg of Co (II) 3.0 cm^3 of borax buffer solution of pH 9.2 and 1mL of 1% solution of HBITSC prepared in DMF added. The volume of solution was made up to 10 mL with distilled water and then equilibrated for 1 min with 10 ml of n-amyl alcohol and the phases were allowed to separate. The n-amyl alcohol extract was collected in a 10 mL measuring flask and made up to the mark with n-amyl alcohol. The absorbance of n-amyl alcohol extract was measured at 510 nm against a reagent blank prepared under identical conditions. The Cobalt content of the sample solution was determined from calibration curve. To study the effect of other ions, the respective foreign ions were added to aqueous phase before the extraction and adjustment of pH.

Determination of Co (II) in catalyst sample: Catalyst sample of cobalt (0.10 g) was dissolved by boiling with 10 mL of aquaregia. The solution was evaporated to dryness and the residue was dissolved in 5 mL of 1M HCl filter and resulting solution was diluted to 250 mL with distilled water. An aliquot (1mL) of this solution was analyzed for Co (II) by the procedure as described earlier.

RESULTS AND DISCUSSION

Co (II) could be extracted quantitatively (99.5%) by HBITSC into n- amyl alcohol from an aqueous solution of pH 8.5 to 9.5 and in the presence of 3mL of buffer solution (0.1 M Borax solution) of pH 9.2 and 1 mL of 1% solution of HBITSC prepared in DMF. Solvents used for extraction of Co (II) can be arranged on the basis of their extraction coefficient values as n- amyl alcohol > n-butyl alcohol > ethyl acetate > benzyl alcohol > chloroform > carbontetrachloride > toluene > nitrobenzene > xylene > chlorobenzene. N-amyl alcohol was found to be the best extracting solvent hence; it was selected for the extraction throughout the work. The n- amyl alcohol extract of Co-HBITSC complex showed an intense peak at 510nm [Fig - 1]. The absorbance due to the reagent is negligible at this wavelength, so the absorption measurements were taken at this wavelength. The result shows that the system confirmed to Beer's law at this wavelength over a Co (II) concentration range of 1.0-5.0 µg ml^{-1} [Fig -2]. The molar absorptivity of the extracted complex on the basis of Co (II) content was calculated to be 4125.7 L $mole^{-1}$ cm^{-1}. It was found that 1ml of 1% solution of HBITSC prepared in DMF was sufficient to extract 50 µg of Co (II). The colour of the n- amyl alcohol extract was found to be stable at least 12 h at room temperature.

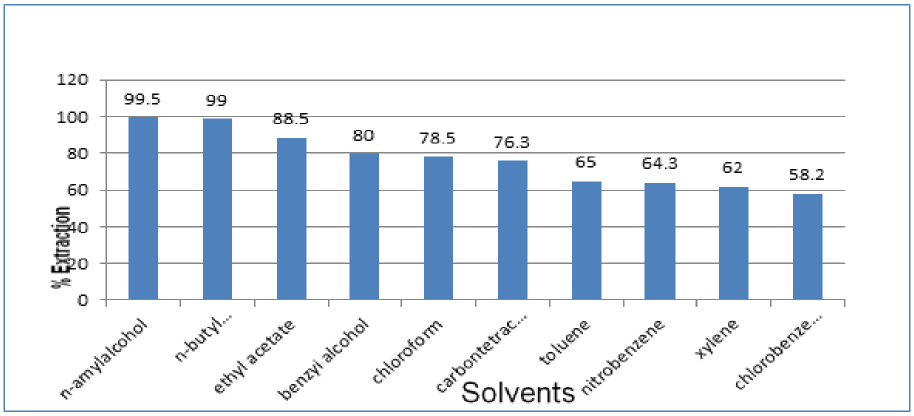


Fig. 1 Percentage Extraction of CO (II) into Various Organic Solvents

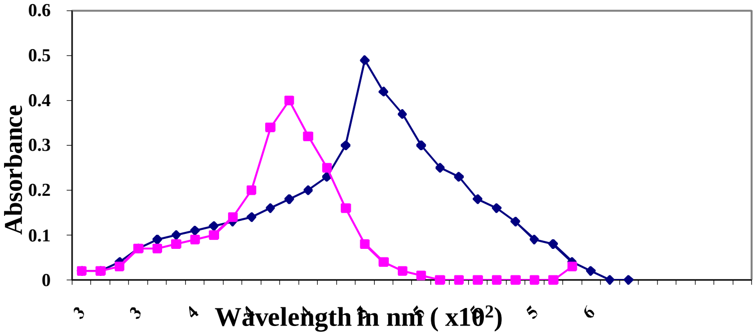


Fig 2. Solution A: Absorbance spectra of HBITSC, Solution B: Absorbance spectra of Co –HBITSC Complex

Effect of other ions: Co (II) (30µg) was determined in the presence of various ions. The following ions in the amount indicated, did not interfere in the spectrophotometric determination of Co(II) (30µg):

a) 10mg each of Li(I), Ca(II), Mg(II), Zn(II), Sr(II), Cd(II)
b) 0.1 mg each of Pt(IV), Ru(III), Ir(IV), Os(IV), Pd(II),Rh(III),Cr(III),Cu(II),Ni(II)
c) 20 mg each of - chloride, bromide, nitrate, thiocyanate, acetate .

Interference due to iodide, thiosulphate and EDTA was removed by boiling solution with concentrated HNO_3 before the adjustment of pH.

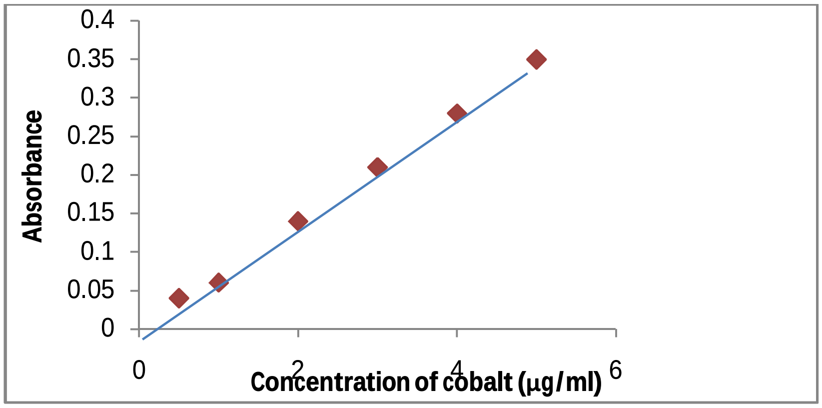


Fig 3: Calibration Curve for Co (II)

Composition of the Extracted Complex: The composition of the extracted complex was found to be 1:2 (Co: HBITSC) by Job's continuous variation and Mole ratio methods [Fig 4 and 5].

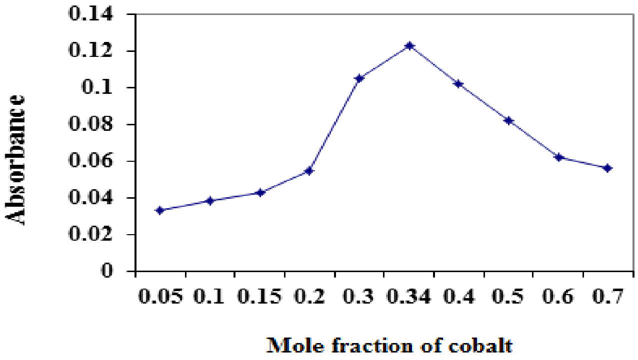


Fig.4 Job's Continuous Variation Method

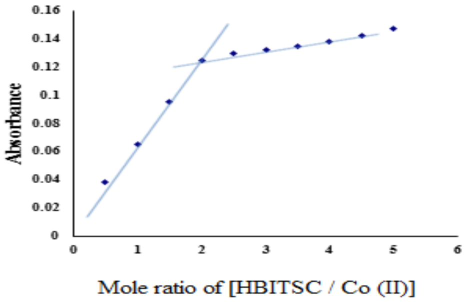


Fig. 5 Mole ratio of [HBITSC / Co (II)]

APPLICATIONS

Precision, Accuracy, Sensitivity and Applications of Method: The precision and accuracy of the method were tested by analyzing the solution containing a known amount of Co (II) following the recommended procedure. The average of 10 determination of 20 µg of Co (II) in 10 cm^3 solutions was 20.3µg, which is varied between 20.6449 and 19.955 at 95% confidence limit. Standard deviation and Sandell's sensitivity of the extracted species is found to be 0.4827and 14.2814.28 ng cm^{-2} respectively. The proposed method has been applied for the determination of Co (II) in catalyst sample. The result of the analysis of the sample was comparable with those obtained by the standard method [14] for Co (II) (Table 1).

Table1. Determination of Cobalt (II) in catalyst sample

Sample	Co found in present Method (%)	Co found in reported method (Nitroso R method) [14] (%)
Cobalt-alumina catalyst	7.45	7.47

* Results are the average of three independent determinations.

CONCLUSIONS

The extractive spectrophotometric determination of Co (II) was successfully carried out. The method is simple and reliable. Experimental conditions for maximum extraction was determined and applied for the analysis of catalyst sample to determine the cobalt content. The results of the analysis of the sample were comparable with those obtained by the NitrosoR salt method [14] for Co (II) (Table 1).

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