New Spectrophotometric Method for Determination of Cadmium

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Abstract: The reagent 2-hydroxy-4-*n*-butoxy-5-bromopropiophenone thiosemicarbazone (HBBrPT) has been used for the determination of Cd(II) by using spectrophotometric method. The reagent HBBrPT gave an intense yellow colour with Cd(II) solution in basic medium. The maximum absorbance was observed at 440 nm, in basic buffer solution (pH 10.00). The molar absorptivity and Sandell's sensitivity of Cd(II)-HBBrPT complex were 4035 mol⁻¹ cm⁻¹ and 0.02765 µg cm⁻² respectively. The stability constant of 1:2 Cd(II)-HBBrPT complex was 8.46x10⁶. The effect of various iron was also studied.

Keywords: 2-Hydroxy-4-*n*-butoxy-5-bromopropiophenone thiosemicarbazone, Spectral study, Spectrophotometric determination, HBBrPT complex.

Introduction

Thiosemicarbazone are known as analytical reagents¹⁻⁶. The reagents is formed by the condensation of thiosemicarbazide carbonyl compound. Thiosemicarbazones are also found to have biological activity. These compounds contain an azomethine for their reactivity with number of transition metal ions, which form coloured complexes. Further the metal complexes formed with these reagents are of great medicinal value in the treatment of diseases like influenza, protozoa, small pox and certain kinds of tumor⁷. These compounds are known for their antitubercular activity⁸. Metal chelate of there reagent inhibit tumor growth and increase the activity of some drugs⁹. In the treatment of cancer the active species is the metal chelate of thiosemicarbazone¹⁰. Metal chelates of these compounds are used as pesticides¹¹ and fungicides^{12, 13} in agriculture.

Experimental

Preparation of HBBrPT

The reagent 2-hydroxy-4-*n*-butoxy-5-bromopropiophenone thiosemicarbazone was prepared by simple condensation of 2-hydroxy-4-*n*-butoxy-5-bromopropiophenone with thiosemicarbazide by adopting the standard procedures. The structure of the compound is given below (I).

HBBrPT

Scheme 1. 2-Hydroxy-4-*n*-butoxy-5-bromopropiophenone thiosemicarbazone.

The structure has been established based on IR, NMR and Mass spectra. The melting point of HBBrPT is 108-109 °C.

Solution preparation

Buffer solutions were prepared using HCl, CH₃COOH and CH₃COONa in acidic medium and NH₄OH, NH₄Cl in basic medium.

Preparation of metal solution and reagent solution

Cd(II) solution was prepared using analytical reagent grade cadmium nitrate. The amount of Cd(II) in this solution was determined volumetrically using EDTA¹⁴. Appropriate quantity of 2-hydroxy-4-*n*-butoxy-5-bromopropiophenone thiosemicarbazone was dissolved in benzene for making 0.0005 M reagent solution.

An aliquot of the solution containing 5.62-16.86 µg/mL of Cd(II), 10 mL of basic buffer solution of the pH 10 and 1 mL 0.0005M 2-hydroxy-4-*n*-butoxy-5-bromopropiophenone thiosemicarbazone was taken in 25 mL volumetric flask were made upto the mark with distilled water. The absorbance of this solution was measured at 440 nm against reagent blank.

Shimadzu 160A UV-visible spectrophotometer (Japan) equipped with 1 cm quartz cell was used in these investigations for making absorbance measurements. A pH meter ELICO L 1-120 (ELICO, Hyderabad) was used to make pH measurements.

Results and Discussion

Cd(II) reacts with 2-hydroxy-4-*n*-butoxy-5-bromopropiophenone thiosemicarbazone in basic pH to give yellow coloured species. The colour reaction of Cd(II) with the reagent is instantaneous even at room temperature. The absorbance of yellow coloured species at a wavelength corresponding to maximum absorbance *i.e.* 440 nm remains constant at least one hour. Studies on the effect of pH on the absorbance revealed that the maximum colour was formed in a solution of pH 10.00. A ten-fold excess of the reagent was adequate for the complete colour development. Addition of excess of the reagent has no adverse effect on absorbance. The order of addition of various components shows no effect on absorbance values. Physicochemical and analytical characteristic of Cd(II)-HBBrPT data shown in Table1. The studies relating to the effect of Cd(II) related that a linear relationship exists between metal ion concentration and absorbance in the range 5.62-16.86 µg/mL, the molar-absorptivity and sandell's sensitivity are 4035 mol⁻¹ cm⁻¹ and 0.02765 µg cm⁻² respectively.

Table 1. Physicochemical and analytical characteristic of Cd(II)-HBBrPT.

Characteristics	Results
pH	10
λ max, nm	440
Colour	Yellow
Molar absorptivity, $\text{L mole}^{-1}\text{cm}^{-1}$	4035
Sandal's stativity, $\mu\text{g cm}^{-2}$	0.02765
Beer's law validity upto $\mu\text{g/mL}$	28.10
Opt. Conc. Range, $\mu\text{g/mL}$	5.62-16.86
Stability constant(k)	8.46×10^6
Standard derivation(S)	0.299
ΔG° , k.cal	-12.462

As the metal ion Cd(II) forms the coloured complex with the reagent, an attempt was made to determine the composition and the stability constant of the complex. The method of Vosburgh and Cooper's¹⁵ showed that only one complex is formed. To determine the stoichiometry of complex, Job's method¹⁶ related data is shown in Figure 1 and mole ratio method¹⁵ is shown in Figure 2 were conducted to make these determinations.

Figure 1. Job's method (Cd(II)-HBBrPT Complex). **Figure 2.** Mole-ratio method (Cd(II)-HBBrPT Complex).

It is noticed that Cd(II) forms a stable yellow colored 1:2 (metal: ligand) complex with 2-hydroxy-4-*n*-butoxy-5-bromopropiophenone thiosemicarbazone. The stability constant of the complex was found to be 8.46×10^6 .

The effect of various ions on the determination of Cd(II) was studied to find the tolerance levels of this foreign iron the present method the results are presented in Table 2.

Table 2. Interference of diverse ions.

Cations	Tolerance limit, ppm	Anions	Tolerance limit, ppm
Co ⁺²	10	Br ⁻	500
Pb ⁺²	100	Cl ⁻	100
Cr ⁺⁶	5	I ⁻	50
Th ⁺²	50	NO ₃ ⁻	500
Sm ⁺²	100	CH ₃ COO ⁻	10
Na ⁺	500	SO ₄ ⁻²	500
K ⁺	500		
Al ⁺³	10		
Ag ⁺	50		
Pd ⁺²	100		

Applications

The complex formed between cadmium and 2-hydroxy-4-*n*-butoxy-5-bromopropiophenone thiosemicarbazone is stable and can be used for analysis. The method has been applied for the analysis of synthesized mixtures containing Cd(II). The related data is shown in Table 3.

Table 3. Analysis of synthetic mixture.

Amounts taken, µg	Found*, µg
250	250.57
310	310.19
190	191.87

*Average value of three determinations

Conclusions

Cd(II) forms a 1:2 stable yellow colored complex with 2-hydroxy-4-*n*-butoxy-5-bromopropiophenone thiosemicarbazone. This complex was used for the determination of cadmium in microgram quantities. The stability constant of the complex is 8.46×10^6 . The molar absorptivity and Sandell's sensitivity are $4035 \text{ mol}^{-1} \text{ cm}^{-1}$ and $0.02765 \text{ µg cm}^{-2}$ respectively. The method has been applied for the analysis of cadmium in synthesized mixtures and also in alloys.

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