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Research Article

2-HYDROXY-4N-BUTOXY-5-BROMO PROPIOPHENONE OXIME AS AN ANALYTICAL REAGENT FOR COPPER(II)

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ABSTRACT

2-Hydroxy-4n-butoxy-5-bromo propiophenoneoxime (HBBPO) was developed as a new analytical reagent for extraction and spectrophotometric determination of Cu(II).InpH3.0-10.0the reagent gives brown coloured precipitate with Cu(II). Spectrophotometric methods revealed that the stoichiometry of the complex is 1:2(metal:ligand). Beer's law is obeyed from 31.77 µg/ml to 254.16 µg/ml of Cu(II). Molarabsorptivity and Sandell's sensitivity at 650 nm were found to be1.4×10^{2} $Lmol^{-1}cm^{-1}$ and0.45µg/cm^{2}respectively. The stability constant of Cu(II)-HBBPOcomplexisfoundtobe$6.05x10^{8}$. Gibb'sfreeenergy change forcomplex formation reaction wasfound to be -12.18Kcal/mol. The reagent canbe used forthe analysisof brass and bronze.

Keywords: Oxime,2-Hydroxy-4n-butoxy-5-bromo propiophenoneoxime, HBBPO.

INTRODUCTION

Inthecurrent scenario of analytical chemistry, many reagents are widely available for gravimetric and spectrophotometric determination of metalions. They includeo-hydroxyketoxime[1-3], phenyl hydrazones, thiosemicarbozones[4], chalconeoximes[5] etc .In this work, we report the use of 2-Hydroxy-4n-butoxy-5-bromo propiophenoneoxime (HBBPO) as a gravimetric reagent for Cu(II). Spectrophotometric methods have been used to confirm the stoichiometry of the complex and to determine the stability constant of the complex. There agent is used to determine copper in brass.

EXPERIMENTAL

Spectrophotometric measurements were made on a Systronics UV/VIS spectrophotometer (model-118) using 10mm glass cells. All the pH measurements were done onSystronic pH meter (model-324) and buffer solution of required pH were obtained using sodium acetate-acetic acid & hydrochloric acid-sodium acetate buffers of suitable concentration.

Synthesis of 2-Hydroxy-4n-butoxy-5-bromo propiophenoneoxime (HBBPO)

Respropiohenone was prepared from resorcinol, propionic acid,anhydrous zinc chloride. 2-hydroxy-4n-butoxypropiophenone (HBP) has prepared by reported method6. 2-hydroxy-4n-butoxy-5-bromo propiophenone (HBBP) was prepared by Bromination7of HBP. The oxime of HBBP was prepared bysodium acetate method. It was crystallised from ethanol, colourless needle like crystals were obtained, with m.p. of 64 □ 1oC.(N found = 4.43 %, calculated =4..68 %,). The oxime is soluble in solvents like ethanol, acetone, Carbontetrachloride, chloroform etc.

Stock solution

Stock solution of Cu(II) (0.005M) was prepared by dissolving $CuSO_4 \cdot 5H_2O$ in distilled water and was used after standardization with EDTA. Stock solution of HBBPO (0.01M) was prepared by dissolving oxime in 70 % aqueous ethanol.

Effect of diverse ions

In gravimetric determination of copper (31.77mg) at pH5, it was found that Ba^{+2},Ca^{+2},Sr^{+2}, Mg^{+2},Al^{+3}, Zn^{+2}, Cd^{+2} and common anions Cl^{-},Br^{-},NO_3^{-},SO_4^{2-} did not interfere. At this pHPd^{+2},Co^{+2} , Ni^{+2}, Mn^{+2}, Fe^{+3} interfere seriously.

Spectrophotometric studies

The absorbance spectrum of Cu-HBBPO complex in chloroform showsmaxima at 410nm and 650nm, but the measurements were carried out at 650 nm using solvent blank because at this wavelength reagent shows negligible absorption. The sensitivity of the method as defined by Sandell is 0.19 µg/cm^2 at 650nm.

Beer's law is obeyed between the range 31.77 –254.1 ppm of Cu(II).

Validity of Beer's law

The Cu(II)-HBBPO complex in chloroform obeys from 31.77 to 254.16 ppm of Cu(II).Beyond this concentration the absorbance plot showed deviation from linearity. The molar absorptivity of the complex obtained from absorbance data is found to be 1.4x10^{2}Lmol^{-1}cm^{-1} at 650nm. The Sandell was found 0.45µg/cm^2 of Cu(II)at 400nm.

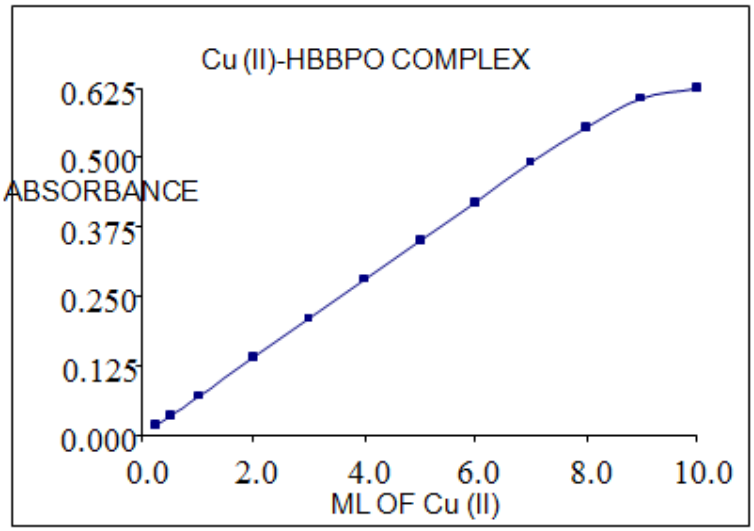


Fig. 1: Validity of Beer's law

Table: Physico-chemical data of Cu(II)- HBBPO

Characteristics	Results
Beer's law Limit (ppm)	31.77 – 254.16
Opt. Conc. Range (ppm)	63.54 –222.39
Molar absorptivity, ε (L mole-1 cm-1)	140
Sandell's sensitivity (µg/cm2)	0.45
Stability constant (K)	6.05 x 10^8
ΔG0 (k.cal)	-12.18
M:L	1:2

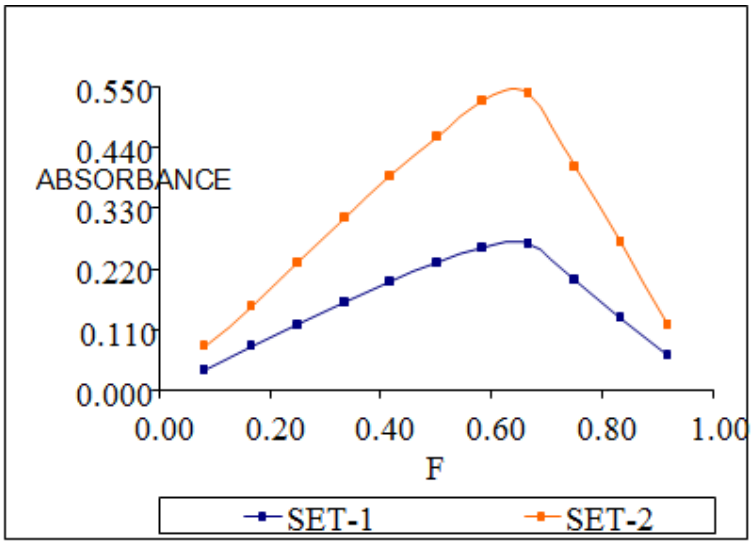
Stoichiometry and stability constant of complex

Thestoichiometry of Cu(II)-HBBPOcomplexwasdeterminedby(1)Job'smethod[8] of continuous variation and (2) Yoe and Jones mole-ratio method[9].Both the methods gave the metal:ligand ratio of 1:2. The stability constant of the complex was calculated using the following formula :

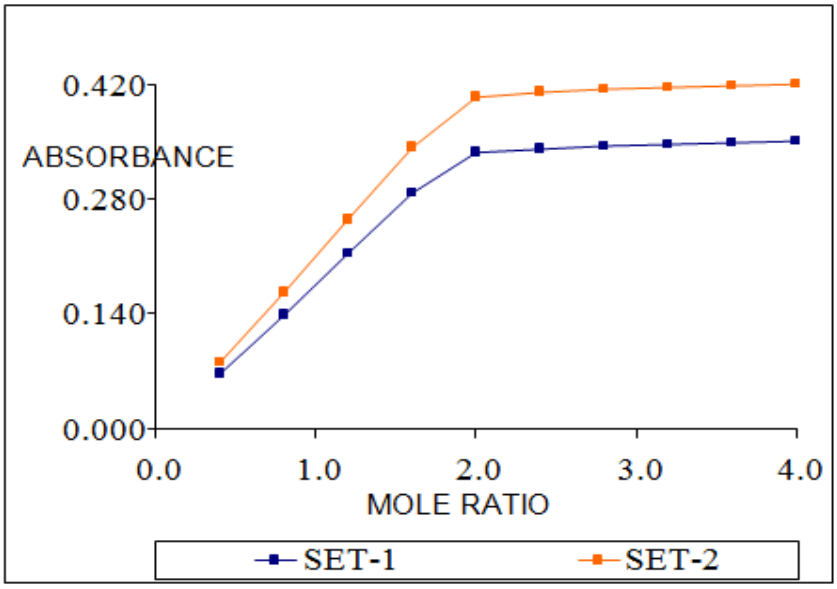
$$K= (1- \alpha)/4C^2\alpha^3$$

Where,α = (Em-Es)/Em,(Em is the maximum absorbance found from graph and Esis the absorbance at stoichiometric molar ratio of the reagent in the complex),and it was found tobe6.05 x10^{8}from Ks value.Gibb'sfreeenergychangeforcomplexfor

mationreactionwascalculatedanditsvaluewas found -12.18Kcal/molat 30 ^{0}C. The graphs were shown below:



Plots of Job's method of continuous variation for determination of M:Lratio
SET-1: 0.01M Cu(II),0.01MHBBPO;pH=5.0;λ=650nm
SET-2: 0.02M Cu(II),0.02MHBBPO;pH=5.0;λ=650nm
Fig. 2: Job's Method Cu(II)-HBBPO Complex



Yoe and Jone's mole ratio method for determination of M:Lratio
SET-1 : 0.0025MCu(II),0.010MHBBPO;pH=5.0; λ=650nm
SET-2 : 0.0030MCu(II),0.012MHBBPO;pH=5.0;λ=650nm
Fig. 3: Mole Ratio Method Cu(II)-HBBPO Complex

Determination of copperin brass

Exactly0.5041gmof brass was taken dissolvedinnitricacid (1:1).The excess nitricacidwas boiled offandthesolution was dilute to100mlwithdistilled water. Take 2 ml diluted solution of the sample in a separating funnel and add 8 ml of distilled water in to it. Add 20 ml of 5.0 pH buffer solution and 10 ml of 0.01 M HBBPO solution and stir it for two to three minutes. Allow the mixture settle for five minutes and then take organic layer in cell for spectrophotometric measurements. Zn(II)and other trace metals did not interfere at this pH. The experiment was repeated three times. Cu(found):66.66 %,Cu(reported): 67.00 %

The same reagent has been taken for the determination of the other transition metal ions.

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