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Extractive spectrophotometric determination of Nickel(II) with 2-hydroxy-4*n*-butoxy-5-bromo acetophenone oxime

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ABSTRACT

Nickel(II) react with 2-Hydroxy-4n-butoxy-5-bromo acetophenone oxime (HBBAO) to give dark green precipitates, which are extractable into chloroform layer. They exhibits λ_{max} at 400 nm and 600 nm and obey Beer's law in the concentration range 29.36-234.84 µg/ml. Job's method for continuous variation, Yoe and Jones' mole ratio method and the slope ratio method show metal:ligand ratio in complex to be 1:2. The stability constant of the complex is found to be 5.49 X10^7. The effect of foreign ions has also been investigated in the determination of metal. The reagent has also been found to give quite satisfactory results for Ni(II) present in synthetic mixtures.

INTRODUCTION

Ketones in which –OH group is suitably placed with >C=O group also act as good chelating agents. Therefore, salicylaldoxime[1-3], resacetophenone oxime[4], ortho-hydroxy acetophenone oxime and its derivatives[5-6] etc. have been used for the spectrophotometric and gravimetric determination of Nickel and other transition metal ions. In the present work the use of 2-hydroxy-4n-butoxy-5-bromo acetophenone oxime (HBBAO) as analytical reagent for Ni(II) has been described.

MATERIALS AND MTHODS

Experimental

Instruments : Spectrophotometric measurements were made with a Systronics UV/VIS spectrophotometer (model-118) using 10mm glass cells. All pH measurements were made with Systronic pH meter (model-324).

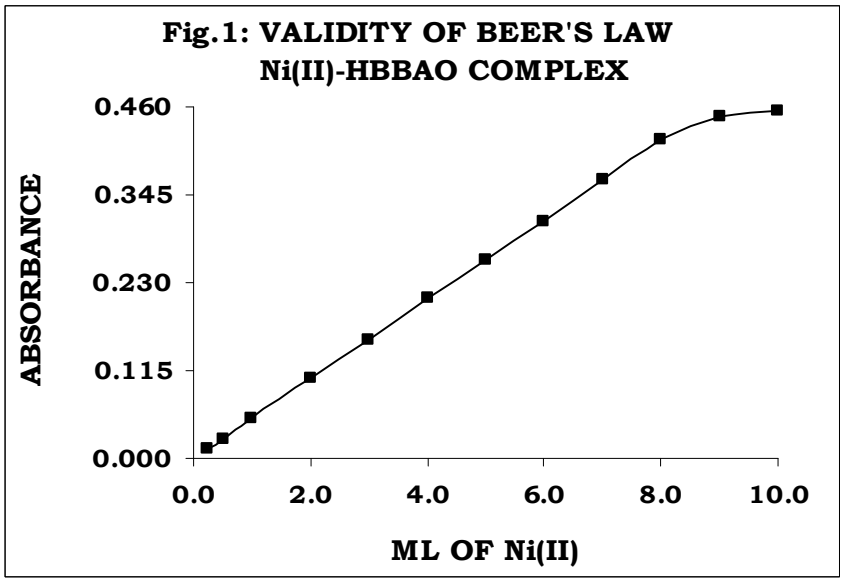
Reagent (HBBAO) : 2-hydroxy-4n-butoxy acetophenone (HBA) was prepared from resacetophenone following method of Eijkmann et al.[7] using n-butyl bromide and anhydrous potassium carbonate in acetone for 8 hrs. 2-hydroxy-4n-butoxy-5-bromo acetophenone (HBBA) has been prepared by Bromination[8] of HBA. The oxime of HBBA was prepared by sodium acetate method. The reagent when recrystallised from ethanol was obtained in the form of colourless needle like crystals with m.p. of 68 ± 1^{o}C, with M.W. 301.9 (cal. for $C_{12}H_{16}O_3NBr$). The reagent is insoluble in water but soluble in alcohol, acetone, benzene, chloroform, carbon tetrachloride etc. The elemental analysis and spectral analysis of the compound confirm its structure.

Stock solution : Stock solution of $NiCl_2.6H_2O$ (0.1M) was prepared by dissolving the pure salt in distilled water containing few drops of hydrochloric acid. The amount of Ni(II) in this solution was determined with EDTA method[9].

Spectrophotometric procedure : When an alcoholic solution of HBBAO was added to 0.01M aqueous metal ion solution, dark green precipitates of complex were obtained in the pH range 1-10. They were soluble in non-polar solvents like chloroform, benzene, carbon tetrachloride and toluene etc. Therefore, the complex was directly extracted in chloroform layer for extractive spectrophotometric determination of Ni(II).

RESULTS AND DISCUSSION

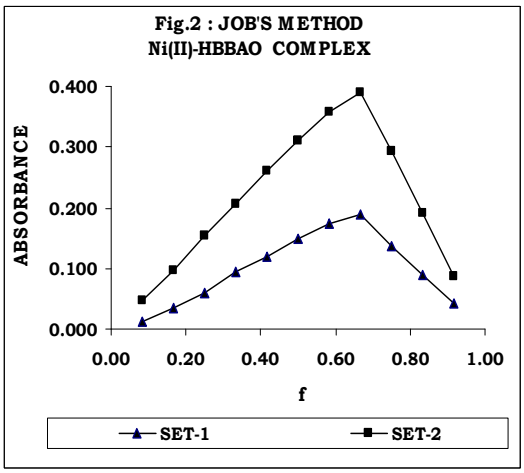
Optimum pH and selection of Wavelength : The absorbance is dependent upon the wavelength used. The absorbance measurement of Ni(II)-HBBAO complex shows a maxima at 400 nm and 600 nm. As the interference due to the reagent appeared to be negligible at wavelength of 600 nm was selected for the present work. On the studying the effect of pH, it was found that maximum complex formation takes place at pH 8.0. Hence, all the measurements were done at pH 8.0 and wavelength 600 nm.

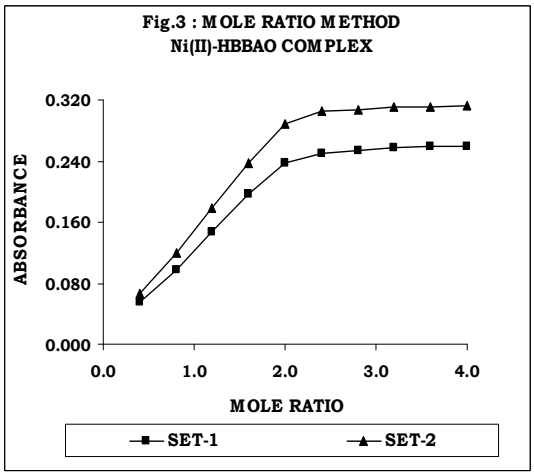


Validity of Beer's law and optimum concentration range : The graph (Fig.1) obeys Beer's law in the range of 29.36-234.84 µg/ml of Ni(II). At higher concentrations the plot shows a

negative deviations from linearity. The optimum concentration range for the complex in organic layer is found to be 88.07-234.84 µg/ml. The molar absorptivity and Sendell sensitivity were calculated to be 104 L mol^{-1} cm^{-1} and 0.56 µg cm^{-2}, respectively at 600 nm.

Stoichiometry and Stability Constant of the Complex : The stoichiometry of the Ni(II)-HBBAO complex was studied by (i) Job's method of continuous variation[10] (Fig.2) (ii) Yoe and Jones mole ratio method[11] (Fig.3). Both the method gave the M:L ratio of 1:2.



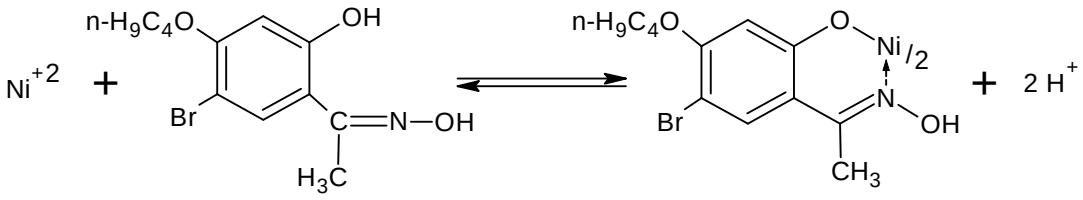


The stability constant of the complex was calculated from the job's method and the mole ratio method are given in Table-1. From the table, the average value of stability constant may be taken as 5.49×10^{7} and free energy of formation was –10.73 kcal/mole at 30°C.

TABLE-1 stability constant of Ni(II)–HBBAO complex at 30°C

Method employed	Em	Es	α	K (n=1)
Mole ratio method				
Set-I	0.260	0.237	0.08846	5.27×10^{7}
Set-II	0.312	0.288	0.07692	5.63×10^{7}
Job's Method				
Set-I	0.210	0.189	0.10000	5.63×10^{7}
Set-II	0.418	0.391	0.06459	5.42×10^{7}
Mean K_s	-	-	-	**5.49×10^{7}**

IR Spectral Studies : The IR spectrum of reagent and complex revealed that the –OH (stretch) band of 3404 cm^{-1} for the reagent disappears when the complex is formed i.e., the complex formation takes place through the N of oximino group and O- of the 2-hydroxy group. Based on above data the Ni(II)-HBBAO complex can be assigned the following structure.



Effect of foreign ions on the precipitation of Nickel : The interference due to the presence of other ions on the determination nickel ions as Ni(II)-HBBAO complex has also been studied. A difference of more than ± 2 % in the absorbance value has been considered as interference. According to this criterion, the tolerance limits of various ions, expressed in µg, for a solution containing 1174.20 µg Ni(II) are as follows.

up to 100000 µg	:	Na^{+}, K^{+}, NH_4^{+}, Cl^{-}, NO_3^{-} , SO_4^{-2} , CH_3COO^{-}
up to 10000 µg	:	Ca^{+2}, Ba^{+2}, Sr^{+2}, Mg^{+2}, Al^{+3}, Zn^{+2}, Cd^{+2}, $S_2O_3^{-2}$, MoO_4^{-}, citrate, tartrate, oxalate
up to 1000 µg	:	Cr^{+3}, UO_2^{+2}, Pd^{+2}
up to 100 µg	:	Ni^{+2}, Co^{+2}, Mn^{+2}, Fe^{+3}
up to 10 µg	:	EDTA

Determination of Nickel in synthetic mixtures : In absence of real samples microgram quantities of nickel in some synthetic mixtures were also determined. The result are shown in Table-2.

Table-2 Analysis of Nickel in various synthetic mixtures

Sr. No.	Composition (µg)	Nickel found (µg)
1.	Ni (88.06), Zn (65.39), Sr (87.62)	87.91, 88.03, 88.00
2.	Ni (44.03), NH_4 (45.10), Ca (40.08)	44.01, 44.94, 44.03
3.	Ni (58.71), Na (34.50), Al (40.47)	58.70, 58.70, 58.68

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