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Research Article

Direct and Derivative Spectrophotometric Determination of Nickel (II) using 2,4-DimethoxyBenzaldehyde IsonicotinoylHydrazone (DMBIH)

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ABSTRACT

Hydrazone compounds are established as selective and sensitive analytical reagents for the determination of transition meatals.2,4-Dimethoxy benzaldehyde isonicotinoyl hydrazone has been synthesized ,charactrized and its analytical application investigated . It react with Nickel (II) in aqueous solution at pH 9.0 and at room teamperature to form a yellow coloured M:L(1:1) complexes with absorpition maximum at 410 nm and molar absorptivity 5.92×10^4 ($l.mol^{-1}.cm^{-1}$) Beers law is obed in the range of 0.1467 to 1.760 µg/ml of Ni(II).The newly developed methods have been successfully applied to determination of Nickel (II) in alloy samples.

INTRODUCTION

Metal ions in general and transition metals in particular are found to play an important role in industry, agriculture, plant nutrition, biological activities of living beings and medicine. Nickel is essential for the active synthesis of urease in plant cells and of various hydrogenases in bacteria.In several species of higher plants, including jack beans (*Canavalia* sp.), soybeans (*Glycine max*), rice (*Oryza sativa*), and tobacco (*Nicotiana tabacum*),nickel is required for effective urea metabolism and urease synthesis (Kasprzak 1987; Sigel and Sigel 1988). . Most metallic nickel produced is used to manufacture stainless steel and other nickel alloys with high corrosion and temperature resistance .These alloys are used in ship building, jet turbines and heat elements, cryogenic installations, magnets, coins, welding rods,electrodes, kitchenware, electronics, and surgical implants; other nickel compounds are used in electroplating,battery production, inks, varnishes, pigments, catalysts, and ceramics The potential analytical applications of hydrazone derivatives have been reviewed by Singh et al[1] . Hydrazones are important class of known analytical reagent [2-8] . Derivative spectrophotometry is a very useful approach for determining the concentration of single component in mixtures with overlapping spectra as it may eliminate interferences

EXPERIMENTAL

Spectrophotometric measurements were made in a Schimadzu 160A microcomputer based UV-Visible spectrophotometer equipped with 1cm quartz cells, an ELICO LI-120 digital pH meter for pH adjustments and Sartorius electronic balance was used for weighing. All reagents used were of AR grade unless otherwise stated. All solutions were prepared with doubly distilled water. Stock solution of nickel(II) (1×10^{-2} M) was prepared by dissolving 0.2377 g of $NiCl_2$. $6H_2O$ (AR GSC) in double distilled water and made up to the mark in a 100-ml volumetric flask. A few drops of diluted hydrochloric acid were added to avoid hydrolysis before dilution of stock solution. The resulting solution was standardized gravimetrically[1].

The reagent 2,4-Dimethoxy benzaldehyde isonicotinoylhydrazone (DMBIH) was prepared by simple condensation of 1 mole of 2,4-Dimethoxy benzaldehyde with 1 mole of isonicotinoylhydrazone (Fig.1).

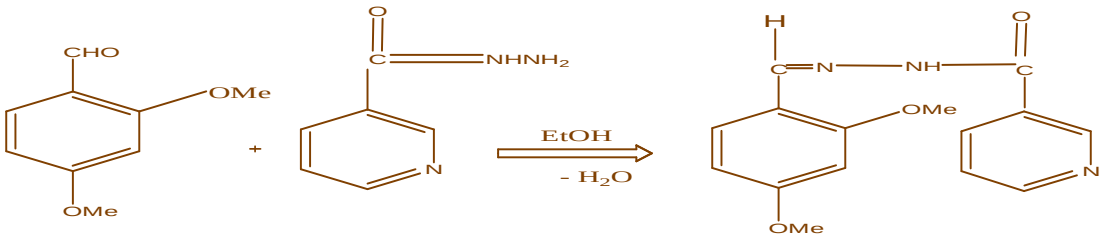

Fig. 1: Structure of 2,4-Dimethoxy benzaldehyde isonicotinoyl hydrazone

The reagent solution (0.01M) was prepared by dissolving 0.2853 g of the compound (DMBIH) was transferred in to a 100-ml volumetric flask. and diluted up to the mark with DMF solvent to get $1x10^{-2}$ M concentration of DMBIH solution

Reaction with metal ions

The reactions of some important metal ions were tested at different pH values. The samples were prepared in 10 ml standard volumetric flasks by adding 3 ml of buffer(pH 1.0-11), 0.5 ml of metal ion($1x10^{-3}$M) and 0.5 ml of ($1x10^{--2}$) DMBIH solutions. The solution mixture was diluted up to the mark with distilled water. The absorbance was measured in 200-800 nm range against reagent blank. The results are summarized in Table 1.

Table 1

Metal ion	pH	λ max (nm	Molar absorptivity (Lmol-1cm-1) x 10^4
W(VI)	8.0	394	3.43 $x10^4$
V(V)	6.0	396	3.13 $x10^4$

Recommended procedure

Direct spectrophotometry : An aliquot of the solution containing 0.1467 to1.7607 µg/mL of nickel (II), 3ml of buffer solution (pH 9.0) and 0.5 ml of $1x10^{-2}$M DMBIH reagent were taken in a 10ml standard volumetric flask and the solution was diluted up to the mark with distilled water. The absorbance of the solution was recorded at 410 nm in a 1.0 cm cell against corresponding reagent blank prepared in the same way but without nickel (II) metal solution. The absorption spectra of DMBIH and its Ni (II) complex under the optimum conditions are shown in Figure 2. The Ni (II)-DMBIH complex shows the maximum absorbance at 410 nm, where the reagent blank does not absorb appreciably.

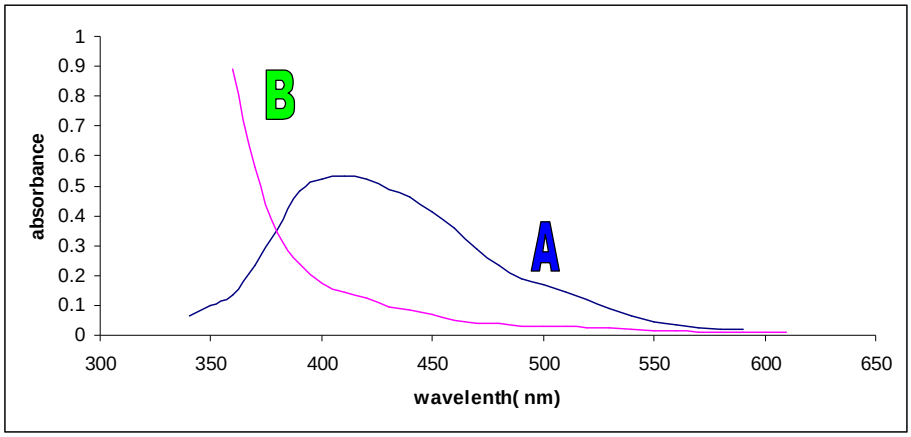

Fig. 2: Absorption spectra of a) **Ni(II)-DMBIH complex vs reagent blank b) DMBIH vs buffer blank**

First derivative spectrophotometric determination of Ni(II)

For the above solution, first-order derivative spectrum was recorded with a scan speed having a degree of freedom 9, in the wavelength range from 300-800 nm. The derivative peak height was measured by peak zero method at 466 nm.The peak height was plotted against the amount of Nickel (II) to obtain the calibration curve. Wavelength values are plotted against absorbance and presented in Fig-3.

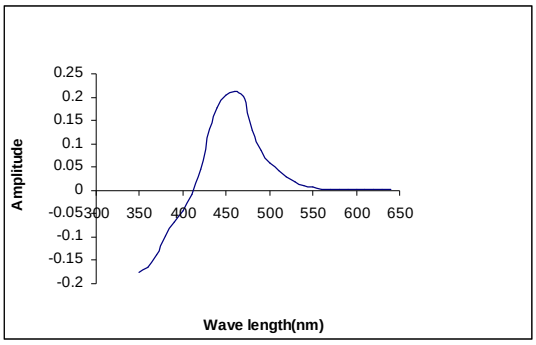

Fig. 3: First derivative spectrum of [Ni (II)-DMBIH] vs reagent

The typical first order derivative spectra of Ni(II)-DMBIH shown in Figure :4 respectively. This shows that, the derivative amplitude measured at 466 nm for Ni-DMBIH were found to be proportional to the amount of Ni(II) metal ion.

RESULTS AND DISCUSSION

2,4-Dimethoxy benzaldehyde isonicotinoylhydrazone (DMBIH) reagent is a blend of a carbonyl compound and a hydrazine. The reagent solution is stable for 48 hrs. in alkaline medium. The ligand presumably coordinates the metal ions to give a neutral water soluble complex.

Determination of Nickel (II) using DMBIH

Nickel (II) reacts with DMBIH in alkaline medium to give bright yellow coloured water-soluble complex.The colour reaction between Nickel (II) and DMBIH are instantaneous even at room temperature in the pH range 8.5-9.5. The absorbance of the bright yellow coloured species remains constant for more than 3 hrs. The maximum colour intensity is observed at pH 9.0. A 10-fold molar excess of reagent is adequate for full colour development. The order of addition of buffer solution, metal ion and reagent has no adverse effect on the absorbance. The complex formation reaction between Nickel (II) and DMBIH has been studied in detail based on the composition of the complex as determined by using Job's and molar ratio methods. Important physico-chemical and analytical characteristics of Nickel (II) and DMBIH are summarized in Table-2.

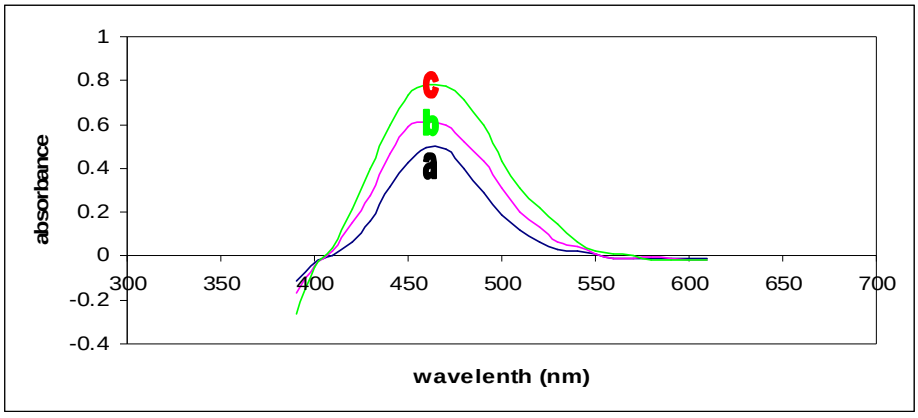

Fig. 4: First order Beer's law derivative spectra of [Ni(II)-DMBIH] complex a: 0.5869 µg/ml of Ni(II); b: 0.8803 µg/ml of Ni(II); c: 1.1738 µg/ml of Ni(II)

Table 2: Physico-chemical and analytical characteristics of Nickel (II)

Characteristics	Results
λ_{max} (nm)	410
pH range (optimum)	8.5-9.5
Mole of reagent required per mole of metal ion for full colour development	10- folds
Molar absorptivity ($l.mol^{-1}.cm^{-1}$)	$5.92x\ 10^4$
Sandell's sensitivity ($\mu g.cm^{-2}$)	0.00099
Beer's law validity range (µg/ml)	0.1467 – 1.7607
Optimum concentration range (µg/ml)	0.293-1.6140
Relative standard deviation (%)	1.36
Composition of complex (M:L) obtained in Job's and mole ratio method	1: 1
Stability constant of the complex	$7.12x\ 10^7$

Effect of Diverse ions

The effect of various diverse ions in the determination of Nickel (II) was studied to find out the tolerance limit of foreign ions in the present method. The tolerance limit of a foreign ion was taken as the amount of foreign ion required to cause an error of ±2 % in the absorbance or amplitude. The results are given in table-3.

Table 3: Tolerance limit of foreign ions in the determination of 0.73366 µg/ml of Nickel(II)

Ion added	Tolerance limit µg/ml	Ion added	Tolerance limitµg/ml
Iodide	1690	La^{3+}	206
Urea	480	Ba^{2+}	189
Thiocynate	534	Ag^{+}	58
Bromide	839	Co^{2+}	64
Thiourea	565	Zn^{2+}	85
Nitrate	489	W^{6+}	21
Tetraborate	1490	Zr^{4+}	14
Acetate	563	Ce^{4+}	59
Phosphate	891	* Cu^{2+}	1.32
Chloride	420	**Hg^{2+}	1.67
Fluoride	356	Mo^{6+}	2.67
Na^{+}	463	Fe^{3+}	1.73

* Masked with 852 µg/ml of phosphate
**Masked with 234 µg/ml of Ascorbic acid

APPLICATIONS

Preparation of alloy sample

1 g of the alloy sample was dissolved in a mixture of 2 ml of concentrated hydrochloric acid and 10 ml of concentrated nitric acid. The solution was evaporated to a small volume. 5ml of 1:1 sulfuric acid was added and the solution wasevaporated to dryness. The residue left over was extracted with 15 ml of water and the solution was diluted to 100 ml with double distilled water. This serves as the stock solution.The stock solution was appropriately diluted to prepare working solutions. The results are given in table-4.

Table 4: determination of Ni(II) in alloy samples

Alloy sample	Composition	Nickel(II) (%)		Error (%)
		Certified	Found	
BCS 364	Cu = 80.00%; Sn = 9.35%; Pb = 9.25%; Ni = 0.28%;Sb = 0.18%; Zn = 0.13%; As = 0.065%; P = 0.056%;Al= 0.002%; Si = 0.003%	0.28	0.25	0.107
Gun metal	Zn = 1.37%; Sn = 9.22%; Cu = 87.95%; Pb= 1.13%;Fe = 0.01%; P = 0.07%; Ni = 0.24%	0.24	0.22	0.090
Monel 400	Ni = 63.01 %; C= 0.15%; S = 0.0024%; Mn = 0.07%;Si = 0.50%; Fe = 2.50%; Cu = 31.00%;	63.01	61.90	0.017

CONCLUSION

From the above discussion, it can be concluded that DMBIH is a potential reagent for the derivative spectrophotometric determination of Ni (II). It is very easy to synthesize DMBIH, a novel class of reagent. The present derivative method is simple and rapid with out the need for heating or extraction compared to other reagents that were used for the spectrophotometric determination of Nickel (II). Ni(II) forms yellow coloured complexes with 2,4-Dimethoxy benzaldehyde iso nicotinoyl Hydrazone(DMBIH).This complex is used for the determination of Ni(II) in microgram quanitites.The method has been applied for the analysis of Ni(II)in alloy samples.

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