

# A Comparative study of Nickel (II) using 4-Hydroxy 3,5 dimethoxy benzaldehyde 4-hydroxy benzoyl hydrazone and Cinnamaldehyde 4-hydroxy benzoylhydrazone in presence of micellar medium by Spectrophotometry

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**Abstract:** Two simple, sensitive, rapid and selective spectrophotometric methods have been developed for the determination of Nickel (II) by using newly synthesized reagents 4-Hydroxy 3,5 dimethoxy benzaldehyde-4-hydroxybenzoylhydrazone (HDMBHBH) and Cinnamaldehyde 4-hydroxy benzoylhydrazone (CMHBH) in presence of neutral surfactant TritonX-100 (5%) (micellar medium). Nickel (II) forms a yellow coloured water-soluble complex with HDMBHBH and CMHBH-in the pH range 9.0-12.0. The Ni (II)-HDMBHBH complex shows maximum absorbance at  $\lambda_{max}$  408 nm in the pH range 8.0-9.0 and Ni (II)-CMHBH shows at  $\lambda_{max}$  400 nm in the pH range 8.0-9.0. At these wavelengths ( $\lambda_{max}$ ), the complex shows maximum absorbance while the reagent blanks shows negligible absorbance. Hence, analytical studies were carried out at  $\lambda_{max}$  408 nm at pH 9.0 for HDMBHBH and 400 nm at pH 9.0 for CMHBH against reagent blanks. Beer's law is obeyed in the range 0.058-0.586  $\mu\text{g ml}^{-1}$  and the optimum concentration range from ringbom plot is 0.117-0.528  $\mu\text{g/ml}$  for Ni (II)-HDMBHBH, and 0.146-1.467  $\mu\text{g ml}^{-1}$ , 0.293-1.320 for Ni (II)-CMHBH. The molar absorptivity and Sandell's sensitivity for the coloured solution were found to be  $3.22 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$  and 0.0018  $\mu\text{g. cm}^{-2}$  for HDMBHBH,  $2.17 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$  and 0.0027- $\mu\text{g. cm}^{-2}$  for CMHBH respectively. The interference effects of various diverse ions have been studied. Ni (II) forms 1:1 complex with HDMBHBH and CMHBH stoichiometry with stability constant  $5 \times 10^6$  for HDMBHBH and  $8.17 \times 10^5$  for CMHBH. The standard deviation in the determination of 0.293- $\mu\text{g ml}^{-1}$  of Nickel (II) is 0.003 for HDMBHBH and 0.002 for CMHBH. The Relative standard deviation is 2.17 % for HDMBHBH and 3.33 % for CMHBH. First and second order derivative spectroscopic methods was developed at  $\lambda_{max}$  450 nm and 485 nm for HDMBHBH and at 460 nm and 482 nm for CMHBH, for the determination of Nickel (II) which is more sensitive than the zero order method. The developed method has been employed for the determination of Nickel (II) in edible oils, plant materials and in alloy samples. The results are in good agreement with the certified values.

**Keywords:** Comparative study of Nickel (II), Spectrophotometry, Edible oils, plant materials, alloy samples, micellar medium, Triton x-100 (5%), HDMBHBH, CMHBH.

## INTRODUCTION

The potential analytical applications of hydrazones and its derivatives have been reviewed by Singh *et al* [1]. Hydrazones are important class of known analytical reagents. Due to analytical potentialities of hydrazones herein we report the synthesis, characterization and analytical properties of reagents 4-Hydroxy-3, 5-dimethoxybenzaldehyde-4-hydroxybenzoylhydrazone (HDMBHBH) and Cinnamaldehyde 4-hydroxy benzoylhydrazone (CMHBH). As the light of the above herein we report the direct and derivative spectrophotometric methods for determination of Nickel (II) using HDMBHBH and CMHBH in edible oils, plant materials and in alloy samples. Derivative spectrophotometry is a very useful approach for determining the concentration of single component in mixtures with overlapping spectra as it may eliminate interferences.

Nickel is remarkably versatile metal ion in biological chemistry. Nickel is abundant in lithosphere and biosphere so that natural deficiency does not occur. This may be the reason why nickel was not found to be essential for many years.

Nickel is used in many industrial and consumer products, including stainless steel, magnets, coinage, and special alloys. It is also used for plating and as a green tint in glass. Nickel is pre-eminently an alloy metal, and its chief use is in the nickel steels and nickel cast irons. It is also widely used for many other alloys, such as nickel brasses and bronzes, and alloys with copper, chromium, aluminum, lead, cobalt, silver, and gold. Because of its permanence in air and its inertness to oxidation, it is used in coins, for plating iron, brass, etc.

Nickel plays numerous roles in biology. In fact urease (an enzyme which assists in the hydrolysis of urea) contains nickel. A nickel-tetrapyrrole coenzyme, F430, is present in the methyl coenzyme-M reductase which powers methanogenic archaea. Other Nickel containing enzymes include a class of super oxide dismutase and a glyoxalase.

Although Nickel is proved essential trace element for several organisms, it is an environmental carcinogen. It is very toxic to most plants and moderately toxic to mammals. Metallic nickel is especially important in the hydrogenation of vegetable oils to produce vanaspati and is a good methanation catalyst for the production of methane from carbon monoxide and hydrogen.

For the determination of Nickel (II), at microgram quantities, a number of hydrazone derivatives and several analytical techniques such as AAS, ICP-AES, ICP, X-Ray fluorescence spectroscopy and UV-visible spectrophotometry were

employed. Among them, spectrophotometric methods are preferred because they are cheaper and easy to handle.

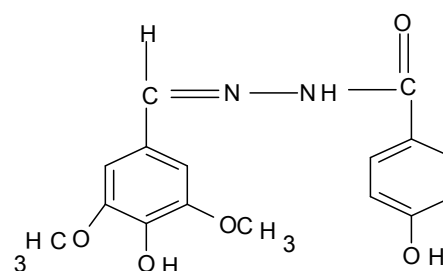
Hydrazones and its derivatives reacts with many metal ions forming colour complexes and act as chelating agents. In general the technique of solvent extraction is widely used in the spectrophotometric determination of metal ions. However, organic solvents such as benzene and chloroform are often carcinogenic, toxic and cause environmental pollution. It is significant to develop a method which does not involve solvent extraction. A micellar solution has the ability to enhance the stability of metal complex and has been utilized as a medium for the spectrophotometric determination of the metal chelate [2-5]

## MATERIALS AND METHODS

The absorbance and pH measurements were made on a Shimadzu UV-visible spectrophotometer (Model UV-160A) fitted with 1.0 cm Quartz cells and Elico digital pH meter (Model LI 120) respectively. Suitable settings for derivative were as follows. The spectral band length was 5 nm, the wavelength accuracy was 0.5 nm with automatic wavelength correction and the recorder was a computer controlled thermal graphic printer with a cathode ray tube and one degree of freedom in the wavelength range 300 – 800 nm.

The reagents (HDMBHBH and CMHBH) were prepared by the Sah and Daniels [6] procedure

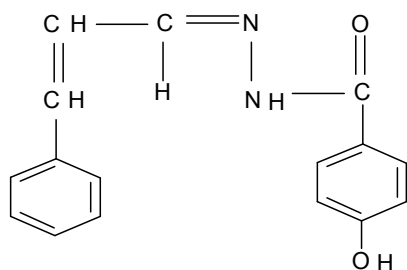
**Preparation of HDMBHBH:** 1.82 g of 4-hydroxy 3, 5 dimethoxy benzaldehyde (I) and 1.52 g of 4-Hydroxy benzhydrazide (II) were dissolved in sufficient volume of Carbinol and the mixture is refluxed for 4 hours. The contents were allowed to cool and the product was separated by filtration. A crude sample (yield 80%) is obtained (C<sub>16</sub>H<sub>16</sub>N<sub>2</sub>O<sub>5</sub>). The resultant product is recrystallised twice from hot methanol. Pure light greenish coloured crystals of 4-Hydroxy 3, 5 domethoxy benzaldehyde 4-hydroxy benzoylhydrazone (HDMBHBH) (III) (m.p. 292-294°C.) was obtained.



**Structure of HDMBHBH**

**Preparation of CMHBH:**

1.32 ml of Cinnamaldehyde (I) and 1.52 g of 4-Hydroxy benzhydrazide (II) were dissolved in sufficient volume of methanol and the mixture is refluxed for 4 hours. The contents are allowed to cool and the product was separated by filtration. A crude sample (yield 80%) is obtained ( $C_{16}H_{13}O_2N_2$ ). The resultant product is recrystallised twice from hot methanol. Pure light yellowish crystals of Cinnamaldehyde-4-hydroxy benzoylhydrazone (CMHBH) (III) (m.p. 242-244°C.) was obtained.

**Structure of CMHBH**

The mass spectrum of HDMBHBH and CMHBH shows that molecular ion peak at  $m/z$  317.1 ( $M+1$ ) and 267 ( $M+1$ ) respectively.

The structures of HDMBHBH and CMHBH were confirmed based upon above IR, NMR and mass spectral data

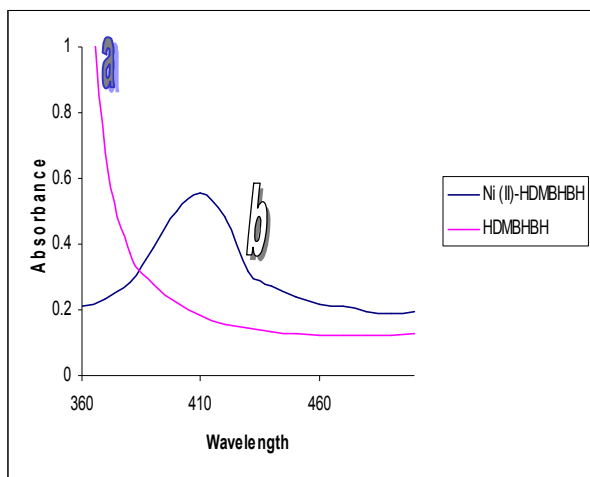
A 0.01M solutions of HDMBHBH and CMHBH in Dimethyl formamide (DMF) were employed in the present studies. The reagent (HDMBHBH and CMHBH) solutions (0.01M) were prepared by dissolving suitable quantity (0.316 g for HDMBHBH and 0.264 for CMHBH) of the compound in 100 ml of dimethyl formamide. The reagent solution is stable for 6 hours in presence of micellar medium. The working solutions were prepared daily by diluting the stock solution to an appropriate volume. All other chemicals used were of analytical grade.

**RESULTS AND DISCUSSION**

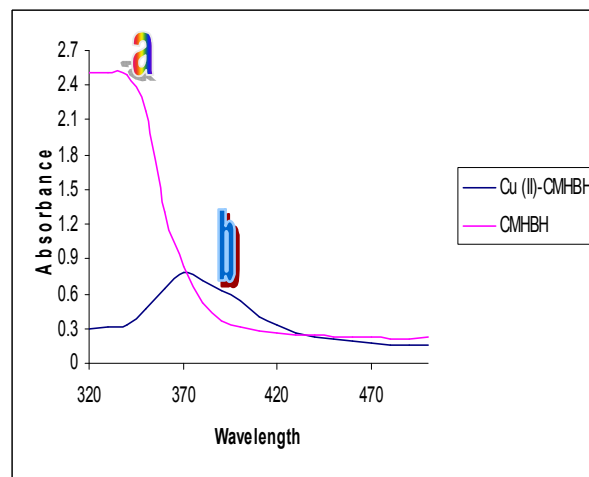
The reagents 4-Hydroxy 3, 5-dimethoxy benzaldehyde 4-hydroxy benzoylhydrazone (HDMBHBH) and Cinnamaldehyde 4-hydroxy benzoylhydrazone (CMHBH) were easily synthesized as any other Schiff base reagent. In basic medium, the ligands presumably coordinates the metal ions as di-anion to give neutral complexes. The absorption spectra of reagents (HDMBHBH, CMHBH) and Copper (II) complex under the optimum conditions are shown in Figure1&2.

**Table: 1.Spectral data of Reagents (HDMBHBH and CMHBH)**

Assignments	IR Spectral bands( $cm^{-1}$ ) of		Assignments	The $^1H$ NMR (300 MHz) spectrum of in DMSO solvent	
	HDMBHBH	CMHBH		HDMBHBH	CMHBH
$\nu$ NH stretching	3529	3452	NH	11.54 (S, 1H)	$\delta$ 11.57(S,1H)
$\nu$ OH stretching	3075	3218-3092	OH (phenolic)	10.13 (S, 1H)	10.74 (S, 1H)
$\nu > C=O$ stretching	1642	1620	N=C-H	8.1 (S, 1H)	8.19-8.22 (S, 1H)
$\nu C=N$ stretching	1609	1577	ArH (pyridine ring)	7.77 – 7.80 (D, 2H)	7.77 – 7.80 (D,2H)
			ArH (aldehydic)	6.84 –6.96 (M,4H)	7.60 – 7.62 (D,2H)
			-OCH <sub>3</sub> (2 Methoxy)	6.63 (S, 6H)	

**Fig-1 Absorption spectra of**

(a)HDMBHBH Vs Buffer blank  
 (b) Ni (II)-HDMBHBH Vs Reagent blank  
 HDMBHBH - $2 \times 10^{-3}$  M (0.5 ml)  
 Ni (II)- $2 \times 10^{-4}$  M (0.5 ml)  
 pH 9.0 ( 3.0 mL )  
 Triton-X-100 (5%)-0.5 ml

**Fig-2 Absorption spectra of**

(a) CMHBH Vs Buffer blank  
 (b) Ni (II)-CMHBH Vs Reagent blank  
 CMHBH- $5 \times 10^{-3}$   
 Ni (II)- $5 \times 10^{-4}$  M (0.5 ml)  
 pH 9.0 (3.0 mL )  
 Triton-X-100 (5%)-1.0 ml

### Effect of reagent (HDMBHBH and CMHBH) concentration

A 10-fold molar excess of reagent (HDMBHBH and CMHBH) was necessary for complex and constant colour development. Excess of the reagent has no effect on the absorbance of the complex. The absorbance of the complex solution was found independent of the order of the addition of the reagents.

### Effect of time Time stability of the coloured solution

The absorbance of the solution was measured at different time intervals to ascertain the time stability of the colour of the complex. The colour reaction between Nickel (II) with HDMBHBH and CMHBH was found to be instantaneous at room temperature. A

slow decrease in absorbance was observed for the coloured species after 10 min. The stability of the complex was increased by adding neutral surfactant Triton X-100 (5%) and the colour remained stable for more than 6 hours.

### The effect of surfactants

The effect of various surfactants such as Triton X-100, Sodium dodecyl benzene sulphonate (SDBS) and Cetyltrimethyl ammonium bromide (CTAB) on the absorption profiles of the system has been investigated and presented in Table 2. In presence of Triton X-100 (5%) both complex were more stable and exhibited maximum absorbance, hence Triton X-100 (5%) has been selected for further studies.

**Table: 2. Influence of different surfactants**

Surfactant	Absorbance of Ni (II)-HDMBHBH complex at $\lambda_{\max}$ 408 nm	Absorbance of Ni (II)-CMHBH complex at $\lambda_{\max}$ 400 nm
None	0.325	0.328
Triton X-100 (5%)	0.565	0.404
CTAB (5%) (Cationic)	0.413	0.341
SDBS (5%) (Anionic)	0.433	0.335

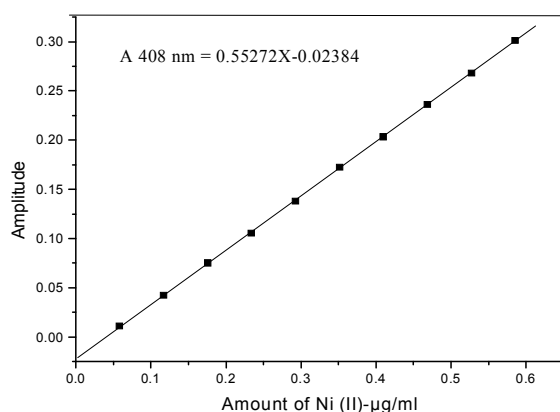
**Table: 3. Optical characteristics, precision and accuracy data.**

Characteristics	Results	
	HDMBHBH	CMHBH
$\lambda_{\max}$	408 nm	400 nm
pH range	8.0-12.0	8.0-12.0
Optimum pH range	8.0-9.0	8.0-9.0
Mole of reagent required per mole of metal ion for full colour development	10 (folds)	10 (folds)
Molar absorptivity ( $L \cdot mol^{-1} \cdot cm^{-1}$ )	$3.22 \times 10^4$	$2.17 \times 10^4$
Sandell's sensitivity ( $\mu g/cm^2$ )	0.0018	0.0027
Beer's law validity range ( $\mu g/ml$ )	0.058-0.586	0.146-1.467
Optimum concentration range ( $\mu g/ml$ )	0.117-0.528	0.293-1.320
Composition of complex (M: L) obtained in Job's and molar ratio method	1:1	1:1
Stability constant of the complex	$5 \times 10^6$	$8.17 \times 10^5$
Standard deviation in the determination of 0.317 $\mu g/ml$ (HDMBHBH) and (CMHBH) of Nickel (II) - for ten determinations.	0.003	0.002
Relative standard deviation (%)	2.17	3.33
Regression coefficient	0.99995	0.99996
Detection limit ( $\mu g/ml$ )	0.009	0.006
Determination limit ( $\mu g/ml$ )	0.027	0.018

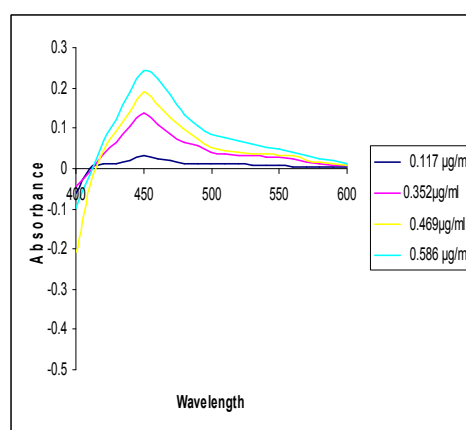
### Optical characteristics, precision and accuracy data.

Beer's law limits, molar absorptivity and Sandell's sensitivity values, regression equation and correlation coefficients for Nickel (II)-HDMBHBH and Nickel (II)-CMHBH complex were compared and presented in Table 3. From this it was noticed that in derivative spectra the peak position shift towards higher wavelengths and Beer's law range was also improved compared to zero order method.

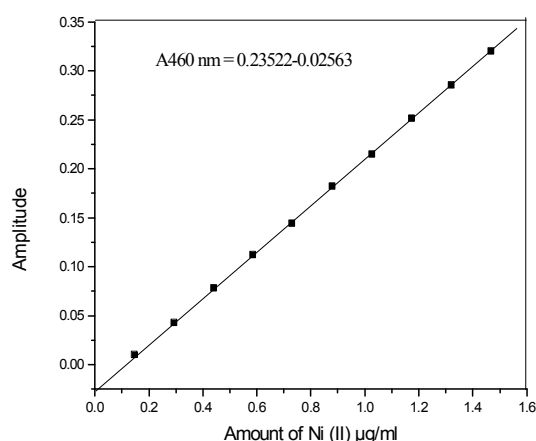
The calibration graph follows the straight-line equation  $Y = aX + b$ ; where  $c$  is the concentration of the solution,  $Y$  is measured absorbance or peak height and  $a$  and  $b$  are constants. By substituting the Ni (II)-HDMBHBH complex experimental data in the above equation, the calibration equations were calculated as  $\lambda_{\max} 408 \text{ nm} = 0.55272X - 0.02384$  for zero order data and  $\lambda_{\max} 450 \text{ nm} = 0.45459X - 0.02242$  For first derivative data and  $\lambda_{\max} 485 \text{ nm} = 0.70344X - 0.02552$  For second order derivative data which gives the straight lines.



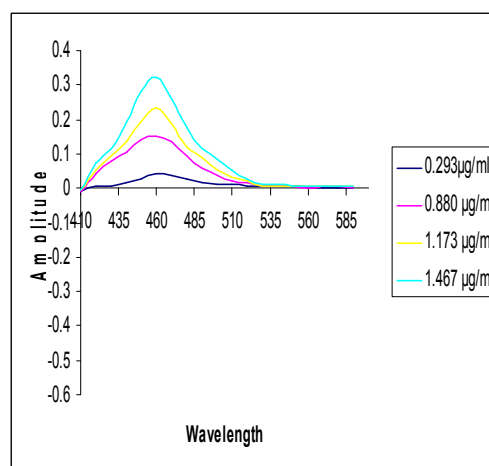
Ni (II)-HDMBHBH - Beer's law  
Zero order  
HDMBHBH- $2 \times 10^{-3}$  M (0.5 ml)  
Ni (II) - $2 \times 10^{-5}$  M (0.5 ml)  
pH --9.0 (3.0 ml)  
Triton-X-100 (5%)-0.5 ml  
 $\lambda_{\max}$ -----408 nm



Ni (II)-HDMBHBH - Beer's law  
first order derivative spectra  
HDMBHBH- $2 \times 10^{-3}$  M (0.5 ml)  
Ni (II) - $2 \times 10^{-5}$  M (0.5 ml)  
pH --9.0 (3.0 ml)  
Triton-X-100 (5%)-0.5 ml  
 $\lambda_{\max}$ -----450 nm



Ni (II)-CMHBH beer's law  
 Zero order  
 CMHBH  $-5 \times 10^{-3}$  M (0.5 ml)  
 Ni (II)  $-5 \times 10^{-5}$  M (0.5 ml)  
 pH  $-9.0$  ( 3.0 mL )  
 Triton-X-100 (5%)-1.0 ml  
 $\lambda_{\text{max}}$ -----400 nm



Ni (II)-CMHBH beer's law  
 first order derivative spectra  
 CMHBH  $-5 \times 10^{-3}$   
 Ni (II)  $-5 \times 10^{-5}$  M (0.5 ml)  
 pH 9.0 (3.0 mL )  
 Triton-X-100 (5%)-1.0 ml  
 $\lambda_{\text{max}}$  -----460 nm

By substituting the Cu (II)-CMHBH complex experimental data in the above equation, the calibration equations were calculated as  $\lambda_{\text{max}} 400 \text{ nm} = 0.36885 - 0.04877$  for zero order data and  $\lambda_{\text{max}} 460 \text{ nm} = 0.23522 - 0.02563$  for first derivative data,  $\lambda_{\text{max}} 482 \text{ nm} = 0.27507 - 0.02386$  for second derivative data which gives the straight lines.

#### Zero order method:

The developed spectrophotometric methods proposed in the present studies were applied for the determination of Nickel (II) in edible oils, plant materials and in alloy samples. The results are in good agreement with the certified values

To a 10 ml standard flask containing 3 ml of buffer of pH 9.0 and 0.5 ml of Reagent, a suitable aliquot of the sample solution was added and the contents were diluted to 10 ml with distilled water and its absorbance was measured at  $\lambda_{\text{max}}$  against the reagent blank. The amount of Nickel (II) present in

these samples was computed from a pre-determined calibration plot.

#### Estimation of Nickel in Edible oils

A 50 g of the sample was digested in 40 ml of conc.  $\text{HNO}_3$ , heated on a water bath and shaken vigorously until fine emulsion was formed. The heating was continued with the gradual addition of 40 ml of 6% Hydrogen peroxide. The aqueous phase was then transferred in to the beaker with the help of separating funnel. The extraction was repeated thrice with further addition of 20 ml of Conc.  $\text{HNO}_3$  and 20 ml of 6% Hydrogen peroxide. The extracts were evaporated to dryness. The residue was dissolved in minimum amount of 1M HCl and transferred into a 50-ml volumetric flask and diluted to the mark with distilled water. The suitable aliquots of the above sample were analyzed by recommended procedure for the determination of Nickel (II). The results obtained were compared with AAS method.

**Table: 4. Determination of Nickel (II) in Edible oils**

Oil sample	AAS Method	Amount of Ni (II) found ( $\mu\text{g/ml}$ )											
		HDMBHBH						CMHBH					
		Zero order	Error (%)	D1	Error (%)	D2	Error (%)	Zero order	Error (%)	D1	Error (%)	D2	Error (%)
Sun flower oil	0.66	0.64	-1.56	0.65	-1.53	0.65	-1.53	0.62	-6.45	0.63	-4.76	0.65	-1.53
Ground nut oil	0.42	0.41	-2.43	0.41	-2.43	0.41	-2.43	0.40	-5.0	0.41	-2.43	0.41	-2.43

\*Average of best three determinations among five determinations

**Table: 5. Determination of Nickel (II) in Alloy samples**

Alloy Sample	AAS Method	Amount of Copper (II) %											
		HDMBHBH						CMHBH					
		Zero order	Error (%)	D1	Error (%)	D2	Error (%)	Zero order	Error (%)	D1	Error (%)	D2	Error (%)
Monel 400b	63.01	62.97	-0.06	62.99	-0.01	62.99	-0.01	62.94	-0.11	62.99	-0.03	63.0	-0.01
BCS-CRM 38Ta	41.90	41.87	-0.07	41.88	-0.04	41.89	-0.02	41.78	-0.28	41.86	-0.09	41.89	-0.02

\* Average of best three determinations. (a). Ni 63.01%; C 0.15% ;S 0.0024%; Mn 0.07% ; Si 0.5%; Fe 2.5%;Cu 31.0% (b). Ni 41.9%; Fe 36%;Cr 12-46%; Mo 5.83%; Ti 2.95%; Se 0.28%; Al 0.29%; Co 0.21%; Mn 0.08%; Cu 0.092%

### Application to alloys

Alloy material (0.25 g) was dissolved in concentrated HCl (15 ml) by warming. A little concentrated nitric acid (1 ml) was added and slowly evaporated to dryness. The residue was dissolved in 10 ml of 1M HCl and the resulting solution was concentrated to ca. 5 ml, diluted to ca. 50 ml with distilled water, filtered and made up to 100 ml. Suitable aliquots of the sample were analyzed for the determination of Nickel (II) by following the recommended procedure.

### 3. Preparation of plant samples

Freshly collected samples were cleaned and dried for one hour in open air protecting from mineral contamination. The dry samples were finely powdered in a mortar. The powdered material was brought in to solution by wet ashing method according to the procedures given in the literature. The suitable aliquots of the above sample were analyzed and the results are presented in table 6.

### Precision and accuracy

The precision and accuracy of the proposed methods were studied by analyzing (10 replicates) 0.293  $\mu\text{g/ml}$  of Nickel (II) and the RSD values were found to be less than 3.33 %.

### Effect of diverse ions

In order to assess the analytical potential of the proposed methods, the effects of some diverse ions which often accompany Nickel (II) were examined by carrying out the determination of 0.293  $\mu\text{g/ml}$  (HDMBHBH) and 0.733  $\mu\text{g/ml}$  (CMHBH) Nickel (II) in the presence of foreign ions. An ion was considered to interfere in the determination if the absorbance observed differed by more than  $\pm 2\%$  from that for Nickel (II) alone. The results presented in Table 7 indicate that many metal ions and anions do not interfere in the determination of Nickel (II). However, Cu (II) and Fe (III) interfere seriously as they readily form coloured species with HDMBHBH and CMHBH. The tolerance limit of Cu (II), Fe (III) was enhanced by using masking agents of fluoride, phosphate presented in Table 7.

**Table: 6. Determination of Nickel (II) in Plant materials**

Plant materials	AAS method	Amount of Copper (II) %											
		HDMBHBH						CMHBH					
		Zero order	Error (%)	D1	Error (%)	D2	Error (%)	Zero order	Error (%)	D1	Error (%)	D2	Error (%)
Eucalyptus leaves	1.063	1.061	-0.18	1.062	-0.09	1.062	-0.09	1.058	-0.47	1.06	-0.28	1.062	-0.09
Pisum sativum (Hulls)	2.051	2.049	-0.09	2.049	-0.09	2.049	-0.09	2.048	-0.14	2.05	-0.04	2.05	-0.04
Mangifera indica leaves	2.226	2.224	-0.08	2.225	-0.04	2.225	-0.04	2.222	-0.18	2.225	-0.04	2.225	-0.04
Azadirachta indica leaves	1.527	1.523	-0.26	1.525	-0.19	1.525	-0.19	1.522	-0.32	1.525	-0.19	1.525	-0.19

**Table: 7 Tolerance limits of diverse ions in the determination of 0.293 and 0.733 µg/ml Nickel (II).**

Ion Added	HDMBHBH Tolerance Limit (µg/ml)			CMHBH Tolerance Limit (µg/ml)		
	Zero order	D1	D2	Zero order	D1	D2
Iodide	1649	1903	1903	1269	1395	1522
Urea	480	540	600	300	360	480
Thiocyanide	581	581	581	349	349	349
Bromide	860	958	958	639	719	860
Thiourea	532	685	685	304	456	456
Nitrate	434	558	620	496	620	620
Tetra borate	1474	1474	1474	246	246	246
Acetate	540	540	540	295	413	413
Phosphate	854	854	854	474	474	474
Chloride	390	461	532	177.5	284	355
Tartarate	875	1125	1250	625	750	875
Citrate	1324	1702	1892	946	946	946
Flourude	322	322	322	227	227	227
Na <sup>+</sup>	483	529	575	230	299	299
Sn <sup>+2</sup>	95	118	118	35.61	59.35	94.96
La <sup>+3</sup>	208	236	236	69.45	111.2	111.2
Ba <sup>+2</sup>	164	205	205	68.66	109.86	109.86
Hg <sup>+2</sup>	28	34	38	20.09	20.09	20.09
W <sup>+6</sup>	183	220	220	91.92	126.28	126.28
Zr <sup>+4</sup>	18	18	18	2.73	4.56	6.38
Bi <sup>+3</sup>	626	668	689	208.98	229.87	250.77
Ce <sup>+4</sup>	56	70	70	7.0	11.20	14.0
Fe <sup>+3</sup> *	1.65	1.65	1.65	2.0	2.0	2.0
Cu <sup>+2**</sup>	1.27	1.27	1.27	1.90	1.90	1.90
Li <sup>+2</sup>	5.0	5.0	5.0	1.0	1.0	1.0
Mo <sup>+6</sup>	2.87	2.87	2.87	4.7	4.7	4.7
Pd <sup>+2</sup>	3.19	4.2	4.2	5.32	6.38	6.38



**Table: 8. masking agents for interfering ions of HDMBHBH and CMHBH**

HDMBHBH	CMHBH
*Masked by Fluoride 322 µg/ml	*Masked by Fluoride 227 µg/ml
*Masked by Phosphate 854 µg/ml	**Masked by Citrate 946 µg/ml

**Table 9. Comparison of spectrophotometric methods for the determination of Nickel (II)**

Reagent	pH	$\lambda_{\max}$ (nm)	Molar absorptivity (L mol <sup>-1</sup> cm <sup>-1</sup> )	Beer's law Range µg/ml	Reference
Oxamidoxime	3.0-5.0	233	21.0 x10 <sup>3</sup>	1-30 ppm	7
Furyl- $\alpha$ -dioxime	Acidic	435	14.5 x10 <sup>3</sup>	1.0-20.0 ppm	8
Benzil- $\alpha$ -dioxime	8.0-11.4	275	12.0 x10 <sup>3</sup>	1.0-20.0 ppm	9
Formaldoxime	>12.0	473	18.4 x10 <sup>3</sup>	Up to 2 ppm	10
Nicotinamidoxime	11.0	575	4.2 x10 <sup>3</sup>	0.3-10 ppm	11
Resacetophenone oxime	8.0-11.0	595	0.13 x10 <sup>3</sup>	6-1500	12
4-tetra-Butylcyclohexane-1,2-dione dioxime	7.0	386	4.1 x10 <sup>3</sup>	0.55-17 ppm	13
2-Hydroxy-5-methyl propiophenone oxime	0.1-10	590	0.15 x10 <sup>3</sup>	0.55-17 ppm	14
Iso vanillin thiosemicarbazone	9.0	400	6.4 x10 <sup>3</sup>	2.0-20.0 ml/g	15
Biacetylmonoxime –4-phenyl-2-thiosemicarbazone	5.2-10.0	375	17.1 x10 <sup>3</sup>	0.5-2.5 ppm	11
Phenanthrene quinone monosemicarbazone	8.5	460	6.4 x10 <sup>3</sup>	2.0-20.0 ml/g	16
4-Hydroxy 3,5dimethoxy benzaldehyde 4-hydroxy benzoylhydrazone (HDMBHBH)	9.0	408	3.22 x 10 <sup>4</sup>	0.058-0.586	Present method
Cinnamaldehyde 4-hydroxybenzoylhydrazone (CMHBH)	9.0	400	2.171x10 <sup>4</sup>	0.146-1.467 µg/ml	

## CONCLUSION

The proposed methods are simple, accurate and have advantages over the reported methods which suffer from interference by large number of ions or require either heating or extraction or are less sensitive.

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