RESEARCH ARTICLE

Determination of Ni(II) in Alloy by Spectrophotometric Method with a new Chromogenic Reagent (IDPBS)

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ABSTRACT

A new chromogenic reagent 4-((imidazole-2-yl)diazenyl)-N-(pyrimidin-2-yl)benzenesulfonamide was synthesized by the reaction between imidazole and Sulphadiazine as azo reagent and used as new spectrophotometric method with highly sensitive to determined Nickel (II). The reagent reacts with nickel(II) in aqueous solution at pH= 7 to form dark- brown colored complex. The reagent and its complex were identified by Fourier transform infrared (FT-IR), Uv-visible,^{1}HNMR, and ^{13}CNMR spectroscopies and investigate the formula and charge of the prepared complex by molar ratio and molar conductivity measurements. The complex shows λmax at 486 nm with a molar absorptivity 0.3299 x 10^{4} L.mo1^{-1}.cm^{-1} and Sandell's sensitivity 0.0177µg.cm^{-2}.Beer's law is obeyed in the concentration range (0.5–7.6µg.mL^{-1}) of Ni (II) with excellent linearity depicted by correlation coefficient value of 0.9995 with a detection limit of 0.1927µg.ml^{-1}. Recovery and relative error values of precision and accuracy of method were found to be R.S.D.% = 0.39221, Re=98.7% and Erel = -1.3% . The nature of the complex showed that (M:L) ratio was 1:2. This method was applied for the determination of Ni(II) in alloy due to Sensitive, accurate, and rapid spectrophotometric method. The results obtained were compared with the flame atomic absorption spectrometry method, and results are in conformity.

Keywords: Azo dye, Interferences and masking agents, Nickel (II) determination and Spectrophotometry, Thermodynamic.

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INTRODUCTION

Nickel (II) is a solid white metal, where it is made by its strength, ductility, heat resistance and corrosion, and essential element for the activation of urease enzyme and have an important role in biological systems.[1,2] Additionally, Ni has potentially harmful effects on health. Exposure to Ni can cause eczema and some allergic reactions on the skin. Furthermore,[3,4] compounds of this element may be carcinogenic. Considering the mentioned effects, accurate and precise determination of this element becomes significant.[5,6] Thus it is clear that the determination of Nickel, at trace level, in human blood and urine samples is of great significance from the public health and environmental point of view.[7-10]

Nickel is one of the important alloying elements for steel and cast iron. A Nickel metallic enzyme is an essential micronutrient[11,12] for plants that arose from ureases. A literature survey indicated that several spectrophotometric methods.[13] However, the spectrophotometric method still has the advantages of simplicity and of not requiring expensive or complicated test equipment. This has led to the development of a wide variety of spectrophotometric methods for the determination of Nickel were reported for the determination of Nickel (II) by using various chromogenic reagents., In views of this separation and determination of Nickel from associated elements is indispensable. Therefore, it is very important to develop a sensitive, rapid, and economical method for the quantitative determination of its trace amount in various samples of environmental importance.[14]

Several analytical techniques have been monitored for the determination of trace level Nickel(II), it includes atomic absorption spectrometry, inductively coupled plasma emission spectrometry[15] and X-Fluoresces spectrometry were reviewed. A few reagents are available for the spectrophotometric determination of nickel(II),[16,17] Nickel(II) was determination in Human blood and Urine at pH=8 The linear range of the calibration graph was between 5 and 500ng.mL^{-1} with limit of detection (LOD) 2.33ng.mL^{-1}. Relative standard deviations (RSD) were 2.33%.[18]

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The aim of this method is to prepare the new chromogenic reagent and uses to the determination of trace amount of Nickel (II) in the alloy sample as a new spectrophotometric method. The reagent and its complex identified by many techniques and compared the results of the determination of nickel (II) with results obtained from the flame atomic absorption.

EXPERIMENTAL

Materials and Methods

Reagent and Solutions

All analytical reagents and solutions used in preparation are in excellent purity.

Instrumentation

All techniques that are used in the characteristic study of the synthesized new ligand and their complexes are :

a- UV-Vis.6100 PC Double beam Spectrophotometer, EMC LAB, Germany.
b- FT-IR spectrophotometer 8400S, Shimadzu, Japan.
c- pH-meter, InoLab ,WTW,135i, Germany.
d- Conductivity meter, Digital, Inolab, Germany.
e- Melting point, SMP30, Sturt, England.
f- Balance BL 2105, Sartorius, Germany.
g- Mova 400MHZ,^{1}HNMR,^{13}CNMR spectrophotometer

Synthesis of New Ligand (IDPBS)

The ligand 4-(imidazole-2-yl) diazenyl)-N-(pyrimidin-2yl) benzenesulfonamide (IDPBS) was prepared via diazotization from the reaction of sulfadiazine with imidazole, with appropriate conditions and additions to the process of azo and conduct interaction with a snowy environment because of the interaction of exothermic. The resultant precipitate yield was 78.12% from dark brown crystals and melting point found to be (169-171ºC) as show in Scheme 1.

Preparation of Buffer Solution

The buffer solution was prepared by dissolving (0.7708g, 0.01mole) of ammonium acetate in 1000mL of distilled water to obtained different (pH)by the addition of concentrated ammonia solution or concentrated acetic acid and prepare a wide range of acidic functions between (pH = 4-12).

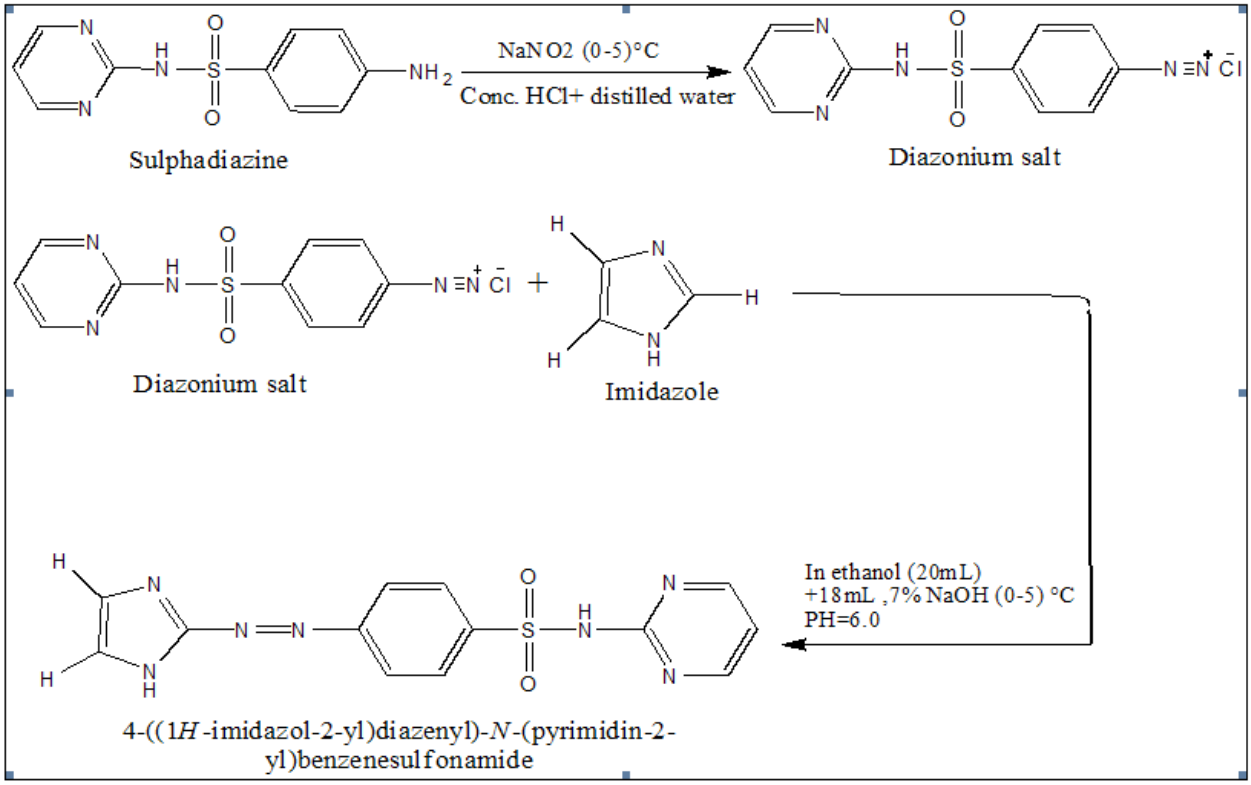
Synthesis of Divalent Nickel (II) Complex

The metal complexes were prepared by molar ratio (1:2) (M:L) by dissolved (1mmol) of the metal chloride salts in 10ml of buffer solution in optimal acidic function. Chlorides were selected as good leaving groups and to prevent interference with negative ions, and for ease of use in the buffer solution with optimum acid function and (2mmol) in 20mL of ethanol absolute of ligand, the precipitate was washed and recrystallized with ethanol absolute and dried in air Table 1 show the physical properties of IDPBS and its Ni-IDPBS Complex.

RESULTS AND DISCUSSION

Absorption Spectra

The absorption spectra of reagent (IDPBS) and the complex Ni(II)-IDPBS were scanned against methanol as blank, the maximum absorption peaks of the ligand was at (344nm),



Schem1:synthesis of 4 - (imidazole-2-yl) diazenyl) -N- (pyrimidin-2yl)benzenesulfonamide (IDPBS) ligand

Table 1: The physical properties of azo ligand and its Ni-IDPBS complex

No.	Molecular formula	Color	m.p (°C)	Yield (%)	Am $Ohm^{-1}mol^{-1}cm^2$			Time reaction
					DMF	Ethanol	DMSO	
1	$C_{13}H_{11}N_7O_2S$	Dark brown	169-171	78.12	-	-	-	4 hour
2	$[Ni(C_{13}H_{11}N_7O_2S)_2Cl_2]$	Brown	194-196	69.37	2.7	3.8	8.5	-

The maximum absorption of Ni^{2+} complex was at 486nm after the optimal application conditions, as shown in Figures 1 and Table 2.

Optimization of Reaction Conditions

pH effect

A series of Nickel ions solutions in(1×10^{-4}M) with ligand(IDPBS) were prepared at pH=4-12, and measure the absorbance for each solution at the maximum wavelength of Nickel complex. The results showed that the best pH = 7 in 25ºC.[19] as shown in Table 3, Figure 2.

Effect of the Concentration of Ligand

A series of solutions were prepared by mixing of $1x10^{-4}$M of Nickel(II) ion solution with different concentration ranging of ligand (IDPBS) from ($0.5x10^{-4}$-$4x10^{-4}$M), the acidic function was adjusted at pH=7 and measure the absorbance against water and ethanol as a blank solution. The results show that the optimal concentration for ligand was ($2x10^{-4}$M) as shown in Table 4 and Figure 3, which shown that the effect of ligand (IDPBS) concentration.

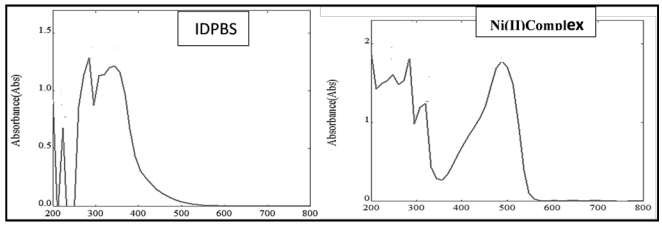


Figure 1: UV-Vis. Spectrum for IDPBS ligand and its Ni-IDPBS complex

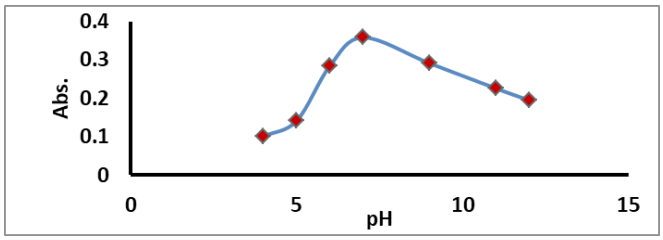


Figure 2: Effect of pH of solution on complex formation

Effect of Time

The effect time in the absorbance of ion complex in (1×10^{-4}M)of Ni(II) ion and (2×10^{-4} M) of a reagent which formed at optimal conditions, the results of the follow-up that the complexities are almost constant for 48hour a period has been established 15 minutes to complete the interaction where. The results of this study showed that the prepared ligand could be used to estimation of Nickel similarly to other reagents that were used to estimate other ions spectrally[20] show in Figure 4.

Effect of the Sequences of Addition

Method summarized in Table 5 was followed to study the effect of sequences of addition under optimum conditions.

From results illustrated in Table 5 the third-order addition was adopted due to the maximum absorbance obtained.

Effect of Temperature

The temperature may affect the stability of complex, in (1×10^{-4}M)of Ni(II) ion and (2×10^{-4} M) of reagent for that, the

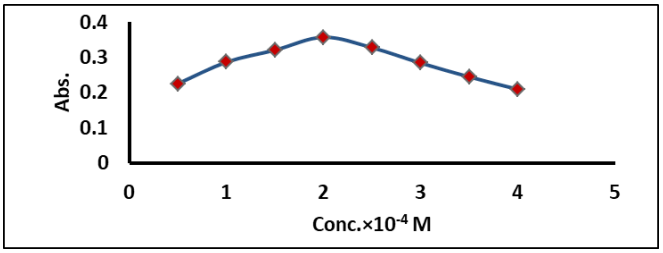


Figure 3: Effect of ligand concentration

Table 2: The electronic transitions for IDPBS ligand and Ni-IDPBS Complex

The molecular formula of ligand	λ(nm)	Wavenumber (cm^{-1})	Transition type
$C_{13}H_{11}N_7O_2S$	344	29069	n→π*
	284	35211	π→π*
	224	44642	π→π*
$[Ni(C_{13}H_{11}N_7O_2S)_2Cl_2]$	486	20576	CT
	316	31645	n→π*
	275	36364	π→π*

Table 3: Effect of acidic function pH for the formation

Metal ion with (IDPBS)	pH/Abs.								
	4	5	6	7	8	9	10	11	12
Ni(II)	0.101	0.142	0.285	0.3568	0.316	0.291	0.254	0.225	0.194

Table 4: The best concentration of ligand equivalent to Ni^{2+} in complex

C concentration of ligand (M)×10^{-4}	0.5	1	1.5	2	2.5	3	3.5	4
Complex of Ni^{2+}, λmax =486nm	0.2253	0.2866	0.3207	0.3568	0.3274	0.2838	0.2445	0.2093

Table 5: Effect of Sequence of addition on the formation of Ni^{2+}complex with IDPBS ligand at pH=7

Sequence of addition number	*Sequence of addition*	*Abs.*
1	M+PH+L	0.2758
2	L+M+PH	0.3174
3	M+L+PH	0. 3568

effect of temperature (5–50)°C was studied under optimum conditions, where the results showed that the Nickel(II) complex absorbance remained almost constant at (15–30°C) as in Figure 5.

Construction of Calibration Curve

A serious of different concentrations were prepared of metal ion with concentrations ranging from (0.5-8.8µg/mL) and ($2x10^{-4}$M) from the ligand and complete the volume to the mark with absolute ethanol, where the results showed that the calibration curve follows the Lambert Beer's law for a range of concentrations (0.5-7.6µg/mL). This shows that the method is highly sensitive and can be used to estimate metals within low concentrations as in Table 6 and Figure 6.

Determination of stoichiometry and formation constant

Mole ratio method, in addition to Job`s method of continuous variations and slope analyses, were chosen to study the

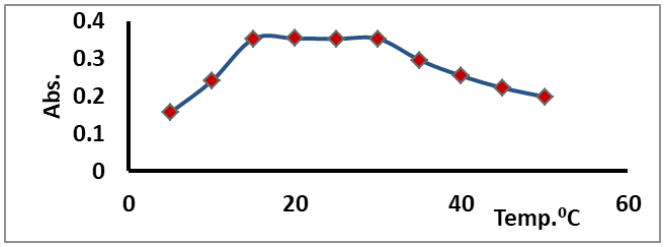


Figure 5: Effect of temperature on the stability of the complex.

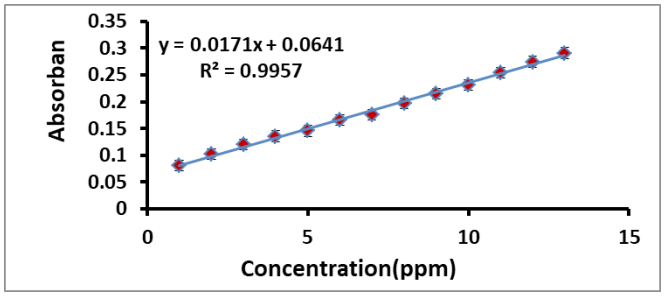


Figure 6: Calibration curve of Nickel(II) complex

Table 6: The calibration curve of metal complexes

λ_{max} (nm)	486
Beer's Law limit (µg/mL)	0.5-7.6
Molar Absorptivity ($Lmol^{-1}cm^{-1}$)	0.3299×10^{4}
Sandell's Sensitivity (µg cm^{-2})	0.0177
Limit of Detection(µg/mL)	2.4866×10^{-2}
Limit of Quantification (µg/mL)	8.2803×10^{-2}
Regression Equation	y=0.0563x+0.0297
Slope	0.0563
Correlation coefficient (R)	0.9995

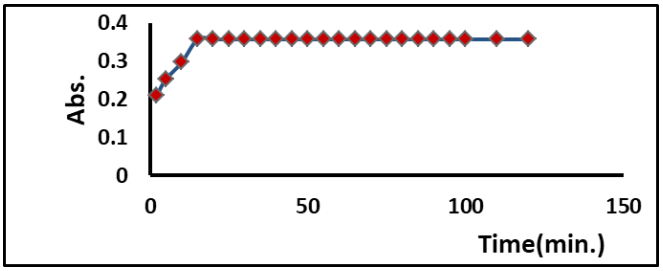


Figure 4: Effect of time on the stability of the Nickel(II) complex

composition of the complex formed, results illustrated. Their methods indicated that the ratio of metal ion to reagent molecules (M:L) was (1:2) at pH = 7.

Job method

A solution of different volume and equal concentration of Ni(II) ion with the ligand (IDPBS) were mixed, as in Figure 7.

Mole ratio method ligand

They are carried out by mixing a known concentration of Ni(II) ion (5.8 µg/mL) with an increasing concentration of (IDPBS) as Figure.8.

Slope Analysis

The analytical slope of the previous results was extracted by dividing the slope resulting from the calibration curve study to find the best concentration of the Nickel-metal (0.0563) on the slope produced by studying the effect of the reagent concentration in finding the best concentration of the reagent (0.1125), The output is (0.5004) This is evidence that the ratio between the metal and the ligand = 0.5004, one mole of copper is bound with tow mole of ligand (IDPBS) to form a complex.

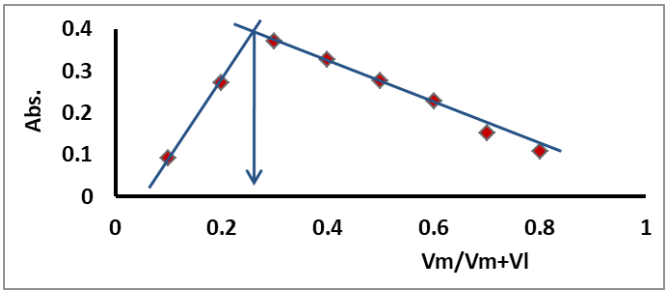


Figure 7: Continuous variation method of Nickel (II) complex

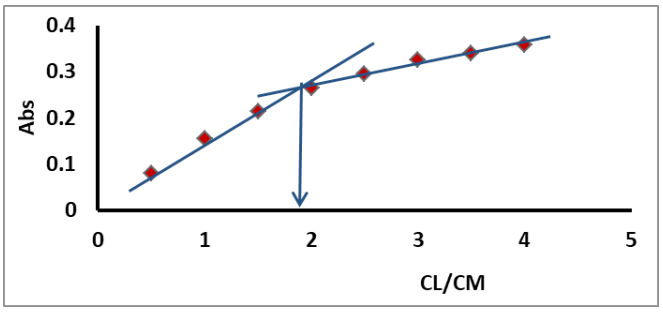


Figure 8: Mole ratio curve of Nickel (II) complex.

Table 7: Value of stability constant for Ni(II) complex.

Complex	*Am*	*As*	*α*	$K_{sr.}$	$K_{inst.}$	*Log* K_{st}
$[Ni(IDPBS)_2Cl_2]$	0.2683	0.3202	0.1621	$4.918x10^{9}$	2.033×10^{-10}	9.691

Study the Stability of the Complex

Mole ratio method was used to determine the stability constant of the colored complex depending on the equilibrium reaction for the complex. Calculations illustrated in Table 7.

$M^{+n} + nL^{-} \rightarrow MLn$

$K_{\text{stability constant}} = [ML^{+n}]/[M^{+n}][L^{-}]^{n}$

$\alpha = A_m - A_s / A_m$

Where A_m is the greatest absorption and A_s is absorption at the stoichiometry

Thermodynamic Function of the Complex

Thermodynamic functions ΔH, ΔG, and ΔS were calculated; results were illustrated in Table 8 and Figure 9.

A negative value of enthalpy explained that the reaction was exothermic for that, it can be noted by decreasing the temperature the possibility of complex formation will be increased, in addition to that the reaction was spontaneous according to the negative sign of free energy.[21,22] The stability of the complex was confirmed due to the value of entropy which approaches to zero(less random and spontaneous).

Effect of foreign ions

A definite amount of cations and anions were used as a foreign ions to study the possibility of the interferences with the determination of Ni(II) ion, results explained in Table 11.

Some ions were selected to study the effect of the interferences with Ni(II) ion (Table11), it was found that some of the ions increased the absorbance while the others decreased the absorbance, this due to the competition of this ions with Ni(II) to form the complex with the ligand which decreased the competition and increased the sensitivity of this method towards Ni(II) ion, where was used as a masking reagent to eliminate the effect of the interference by returning the absorption values to the original calculated output without interference.[23]

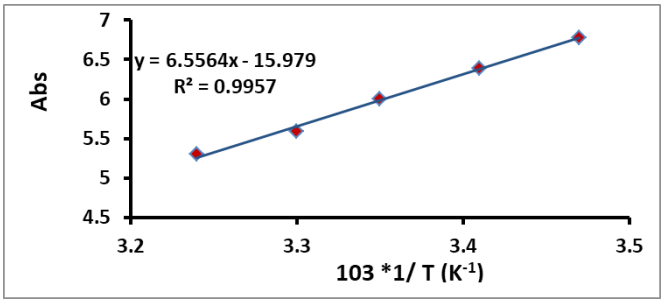


Figure 9: Relationship of Log K_{st} and 1/T of Ni(II) complex

Table 8: The effect of temperature on the thermodynamic function for Ni(II) complex.

T(K)	1/T (K^{-1})	Log. K	-ΔH (K.J/mole)	-ΔG (K.J/mole)	ΔS - (K.J/mole. K)
288	0.00347	6.7753	125.53	37.35	306.18
293	0.00342	6.3867		35.84	306.62
298	0.00335	5.9972		34.21	306.44
303	0.00330	5.5921		32.43	307.27
308	0.000324	5.3024		31.26	306.07

Table 9: Values of standard deviation, % standard deviation.

Comp. of ion	Conc. Of ion(M)	S.D	R.S.D%
Ni(II)	0.1×10^{-4}	0.0014	0.3921
	0.5×10^{-4}	0.0011	0.3081
	0.8×10^{-4}	0.009	0.2521

Table 10: The relative percentage error and the pre-processing ratio of ligand complexes.

Complex ion	Analytical value ($mol.L^{-1}$)	d	$\%E_{real}$	%Re
Ni(II)	0.987×10^{-4}	-0.013×10^{-4}	-1.3	98.7

Study of FTIR spectra for Ligand and Complex

Figure 10 and Table 12 explained the FTIR study and the absorption frequencies for reagent and the Ni-IDPBS.[24,25]

Magnetic Resonance Spectrometer

^{1}H NMR Spectra of Ligand (IDPBS)

The 1H NMR Spectra of ligand shows the chemical displacements of aromatic and elliptical protons, The single band at (13.1873 ppm) belong to one proton of an amine group (NH) in imidazole cyclic, the three single bands at (7.0459, 7.0566and 7.0655 ppm) due to the protons of (H1), the multiple bands at (7.9737ppm to 7.9912ppm) belongs to nine protons of (H2), while the band at (2.500 ppm) related to (DMSO d^{6}),[26,27] as in Figure 11.

^{13}CNMR Spectra of Ligand (IDPBS):

The Figure 12 shows the ligand spectrum showing the signals of the carbonate ring of benzene and the carbon group of

Table 11: Effect of foreign ion on the determination of Ni(II) ion.

Foreign ions	5.8µg/mL Absorbance after addition of ions	d	E%	%Re
Cation				
Without Cation	0.3568	-----	-----	----
Cu^{+2}	0.2506	-0.106	-29.76	70.23
Co^{+2}	0.3462	-0.011	-2.97	97.02
Cd^{+2}	0.3094	-0.047	-1.31	98.69
Pd^{+2}	0.3745	0.018	4.96	104.96
Mn^{+2}	0.3891	0.032	9.05	109.05
Fe^{+2}	0.3275	-0.029	-8.21	91.78
Zn^{+2}	0.2487	-0.108	30.29	69.70
Anion				
Without anion	0.3568	---	---	---
CO_3^{-2}	0.3274	-0.029	-8.23	91.76
SO_4^{-2}	0.3385	-0.018	-5.12	94.87
$C_2O_4^{-2}$	0.3196	-0.037	-9.42	90.76
CH_3COO^{-2}	0.3318	-0.025	-7.01	92.98
NO_2^{-1}	0.3273	-0.029	-8.26	91.73
SCN^{-1}	0.3452	-0.021	-3.25	96.74
Cl^{-1}	0.3407	-0.016	-4.51	95.48
Masking agent				
Without masking agent	0.3568	---	---	---
Ascorbic acid	0.2764	-0.0804	-1.117	98.82
Potassium Thiocyniate	0.2877	-0.0691	-19.36	80.63
EDTA-Na_2	0.1364	-0.2204	-61.77	38.23
Thiourea	0.1682	-0.1886	-52.85	47.14
Potassium chloride	0.2493	-0.1075	-30.12	69.87
Tartaric acid	0.3526	-0.0042	-0.324	99.67
Salicylic acid	0.2557	-0.1011	-28.33	71.66

Table 12: Typical FT-IR absorption frequencies for reagent and complexes

Compound	*N-H*	*N=Nv*	*S=Ov*	*M-Nv*
Ligand IDPBS	3206(m)	1467(w)	1294(s)	---
Ni(II)complex	3215(S)	1406(w)	1265(s)	466(m)

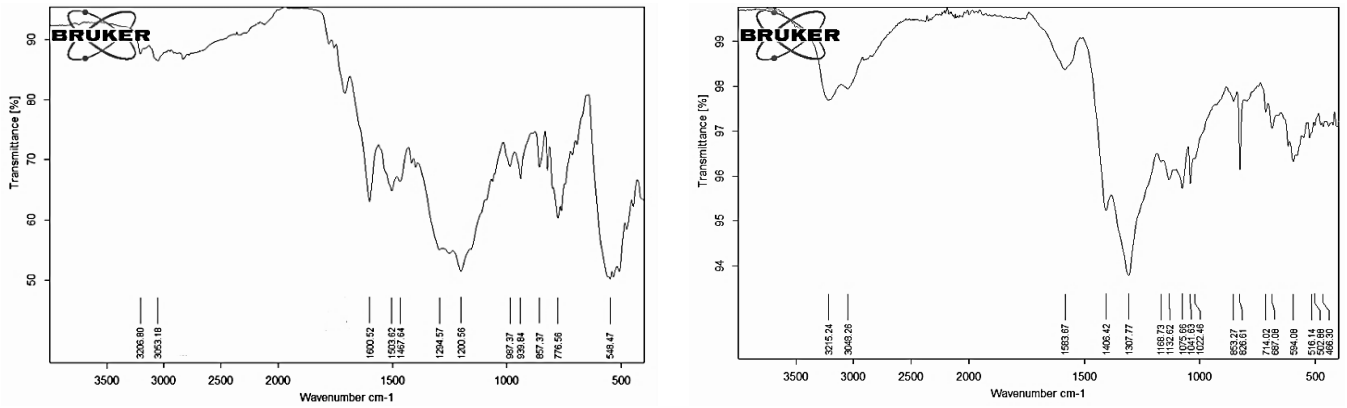


Figure 10: FTIR spectrum of IDPBS and Ni-IDPBS Complex

imidazole cyclic , The single band at (115.6486 ppm) belong to one Carbone of imidazole cyclic, the single band at (122.4811 ppm) belong to one Carbone of benzene (C2), the multiple bands at (145.4675 ppm to 158.3548ppm) belongs to carbon aromatic rings [26] The single band at (39.5200 ppm) related to (DMSO d^6), TMS, Figure 12.

Precision

The precision of the analytical method was determined by calculating the amount of standard deviation (S.D) and the relative standard deviation (%R.S.D) of the complex and optimal conditions[28] in Table 9.

Accuracy

The accuracy means the approximation of the practical value from the theoretical value so that the results are accurate and precise. The accuracy of the analytical method is calculated by using the percentage relative error and the pre-processing ratio of the previously prepared complexes as in table 10 . The results show that this method used to estimate the metals using ligand (IDPBS) is high accuracy.[29]

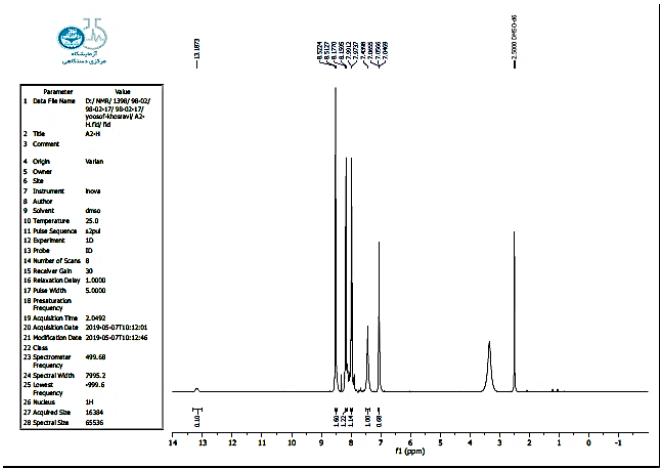


Figure 11: ^{1}H NMR Spectra of ligand (IDPBS)

%Ereal = d/μ×100

μ = Analytical value

d = Analytical value - theoretical value

Suggested Structure of the Complex

A suggestion of the complex structure, as shown in Figure 13, is due to FT-IR spectra, and the stoichiometry obtained from job, slope analysis and mole ratio methods.

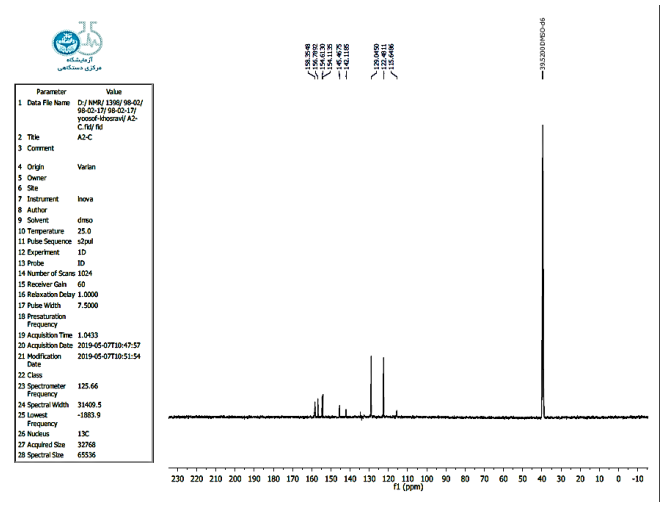


Figure 12: ^{13}CNMR Spectra of ligand (IDPBS)

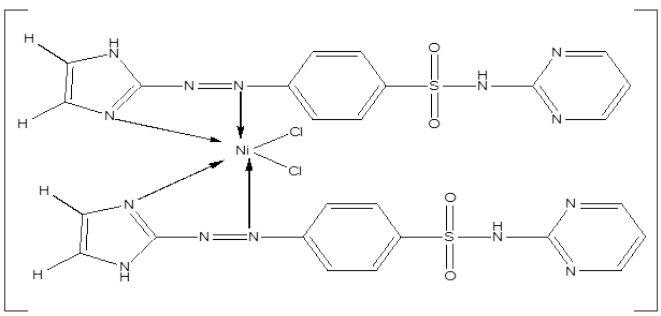


Figure 13: The suggested structures of Nickel(II) Complex

Table 13: The percentages of Nickel determine by ligand in Nickel alloy using atomic absorption method and spectrophotometric method

		By Spectrophotometric			*By Flame atomic absorption*		
Sample	*Content%*	*method %*	*%E_{rel}*	*%Re*	*%*	*%E_{rel}*	*%Re*
Alloy Nickel	30	28.6	-1.4	98.6	27.2	-2.8	97.2

Application

The prepared ligand was used as a reagent for determined of Nickel in alloy, which contains(Ni=30%, Cu=55%, Zn=15%) spectrophotometrically, and the results were compared to these were obtained by flame atomic absorption method as in Table 13.

We can observe the compatibility between the spectral method and the atomic absorption method data, so we can conclude that the spectral method is the most widely used in the estimation of nickel in different samples with precision, selectivity and high sensitivity (Table 13).

CONCLUSION

The proposed spectrophotometric method is simple, highly sensitive, and selective for the determination of Ni (II) in alloy samples. The limit of detection of the proposed method is superior when compared to the reported method. The method has an additional advantage over reported method owing to its complexing reagent employed in the present method i.e., 4-((imidazole-2-yl)diazenyl)-N-(pyrimidin-2-yl) benzenesulfonamide economical and easy to prepare in an ordinary laboratory. The proposed method is highly sensitive due to the stabilization of colored complex for more than 48 hours formed by interactions of the metal ion with newly synthesized reagent low reagent consumption, elimination of the analytical error, less interference, and statistical analysis which made the method to be more sensitive and selective.

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