

Spectrophotometric Determination of Micro Amount of Nickel (II) Using an (Azo) Derivative in the Presence of Surfactant, Study of Thermodynamic Functions and Their Analytical Application

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Abstract

It is characterized as a nickel spectrophotometric approach that is very sensitive and substantially interference-free. For the study of Co (II), Mn (II), and other metals in this complex, the imidazole azo ligands (DPIDA) were produced and Mn (II), Co (II), and other metals in this complex were analyzed. Ni (II), "Cu" (II), Zn (II), Cd (II) and Hg (II) ions are among the most important azo ligands in the area of coordinated chemistry. First, molarity and ¹HNMR spectroscopy were carried out. To explain their conductivity, we must take into account the fact that they have octahedral geometry with a bidentate ligand coordinated by an imidazole (N3) and an azo group (O)-atom. In addition, the thesis examined the use of Reagent was employed in binary water solution to conduct spectroscopy with nickel (II) ion, and it was discovered that the reagent is difficult to the ion and has the maximum sensitivity. 530.55 nm (pH = 8) is the absorption wavelength. A dynamic nickel-pair reagent with more than 24 hours of stability was found in the concentration range of (0.1–2.5 g/mL) by Beer's law. masking agent and concentration all have an impact. Stoichiometry with molar reagent-metal ratios as well as the effects on the absorption of cations and anions, as well as the effect of ionic strength and temperature were explored in these studies (the constant changes). According to the mallard equation, the molar absorptiveness of metal reagent (1:2) is (2.4x10⁴ L. mol⁻¹. cm⁻¹). It is possible to detect (0.004 microns/ml) and quantify (0.627 microns/ml) using the same detection and quantification limit values. This dynamic nickel pair was more stable, with a more soluble molecular conductivity and a lower melting point (0.1–2.5 g/mL) in the range of pH conformance (0.1–2.5 g/mL). FT.IR. and UV-Vis spectroscopy methods have been used to classify both compounds. Comparing free reagent spectra to bathochromic transition (UV Vis), it can be seen that the approach used to compute nickel per cent deviation is very precise and accurate (RSD per cent). the relative error (E per cent) deviated from the standard deviation (SD) (4.011 per cent, 0.803 per cent). Temperature also had a role in determining the thermodynamic functions (H, G, S). With great accuracy and precision, the method was applied to a variety of environmental and industrial models.

Keywords: Azo dye (DPIDA), Nickel (II), Spectrophotometry

1. Introduction

As a consequence of their wide variety of scientific and technological applications.,[1] [2], Azo dyes are the most common kind of colourant in use today. Coupling reagents are used to diazotize aromatic moieties to create azo dyes with one or more azo groups coupled to one or more aromatic moieties. High-level dyes are used.[3], This kind of dye is often used in textiles and cosmetics. the ligand [4]. Additionally, since surfactants include a balance of hydrophilic and hydrophobic components, their application is altered...[5], Neutral molecules and ionic micelles commonly interact toward the diffusion-controlled limit in an aqueous solution.. [6], with a rate constant of 108-101o dm³ mol⁻¹ s⁻¹[7]. Environmentalists are concerned about heavy metal contamination in natural rivers and soils because of the potential harm it may do to living things. Some household animals and plants were once considered

to need nickel to grow and thrive...[8], and it's a vital component in both business and the natural world. [9], When Zerner found in 1975 that urease was a nickel enzyme, it was regarded as a biologically important metal...[10], Among the transition metals, nickel is especially hazardous. On the contrary, nickel and its compounds are well-known to cause major health issues. A simple and sensitive approach for identifying nickel (II) toxicity, such as lung cancer, has been developed by researchers. [11]. Nickel-eczema, a skin disorder, may also be caused by nickel. [12]. The ability to work in a variety of environments is consequently vital. [13]..The vibrant solutions of these compounds have long captivated inorganic and analytical chemists...[14]. UV-Vis spectrophotometry techniques are frequently employed because of their simplicity and speed. In comparison to other metals, nickel is quite safe. Although nickel is absorbed by acidic foods after cooking, [15], They pose no danger to human health.

Because nickel is not easily absorbed by the human body, it does not pose a threat. Even while respiratory tract neoplasia is common among nickel refinery workers, this metal has also been discovered to have carcinogenic properties. [16]. Inductively coupled plasma emission and mass spectrometry have all been used to find traces of nickel in various samples. (II) [17], Nickel (II) was measured using the X-fluoresce spectrometry method. [18]. Many reagents are available for this task. Nickel (II) trace detection in alloy samples will be improved by the development of an entirely new chromogenic reagent. The reagent and its complex were proven to be nickel (II) using a variety of procedures, and the findings were compared using flame atomic absorption data.

2. Experimental

Preparation of (DPIDA) ligand

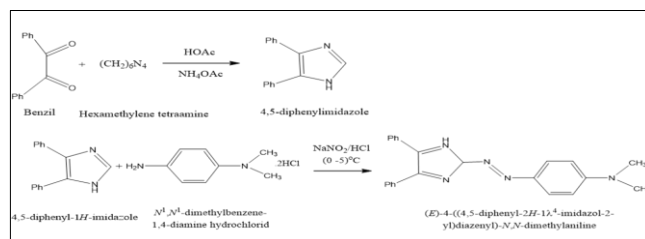
A / preparatory work done on raw materials (4,5 Diphenyl imidazole)

It was prepared using the alpha Di carbonyl condensation method, which involved ammonia, aldehydes, and ice acetic acid, and the precursor was made by combining (2.1 g, 0.01 mole) of benzene with both (0.015 moles, 21) of the following: hexamethyl tetraamine and amino acetate in an ice-cold glass flask (100 ml). The mixture was raised to room temperature and cooled to room temperature before the mixture was resuspended in an ice-cold glass flask. Adding ammonium hydroxide precipitated the imidazole derivative, which was then washed with distilled water to remove any excess base and dried in the air before being recrystallized from the ethanol to obtain on a white precipitate and measured for its melting point (229-230 C) and yield. After cooling to a Baker's capacity of 1 L, the solution was transferred and diluted by adding (400 ml) of distilling water (75 per cent).

B / N 1, N 1 dimethyl benzene 1,4 diamine dihydrochloride 4,5 diphenyl imidazole coupling reaction Sodium nitrate solution was prepared by dissolving 0.70 gm of sodium nitrate in 10 ml of distilled water drop by drop with stirring, and the diazonium salt was formed as a result of the gradual addition of one ml of hydrochloric acid to this solution, which was cooled in an ice bath at 0.5 °C. To make 4,5diphenyl imidazole, a solution of imidazole derivative and sodium hydroxide was dissolved in 25 ml of alcohol, and the orange precipitate was formed after the addition of the solution, which was filtered and dried, and then recrystallized from ethanol with a yield of 71% as shown in the diagram.

3. Materials and Methods

In this article, for example. The preparation of all analytical reagents and solutions is carried out with the utmost care.



Scheme 1: The ligand (DPIDA) is prepared.

Preparation of the Standard Solutions

1-Nickel (II) solution (1000 g/mL) [NiCl₂(H₂O)₆] was dissolved in 100 mL of distilled water to provide a solution of (0.220 g) in the solution.

2-The sodium hydroxide solution (0.1M) was made by adding 100 mL of distilled water to 0.4 g of sodium hydroxide and mixing well.

3-Acid solution (0.1M): diluting (0.4 mL) concentrated hydrochloric acid (38 percent, 1.19 g/ml) in 50 ml pure water. 4-The DPIDA reagent solution (0.001 M) After dissolving the requisite weight (0.091g) in absolute ethanol and increasing the amount to (250mL) with ethanol, the mixture was ready to use.

Interferences

Cation's solution of (Cd²⁺, Hg²⁺, Fe³⁺, Ba²⁺, pb²⁺, Cr³⁺, Co²⁺) ions (0.1mg/mL) were prepared by dissolving (0.163 grams) of CdCl₂, (0.221 grams) of HgCl₂, (0.226grams) of FeCl₃.6H₂O, (0.190grams) of BaCl₂, (0.403 grams) of CoCl₂.6H₂O, (0.512 grams) of CrCl₃.6H₂O in 100 milliliters of distilled water for each

Preliminary Study

DPIDA (0.001M) was added to the test tube, and then drops of hydrochloric acid (0.1 M) and NaOH (0.1 M) were added to one part of the mixture to study the effect of the acidic function, while drops of HCl (0.1 M) or NaOH (0.1 M) were added to the other part to see if the acidic function had any effect on the precipitation. The acidic media created the colour clearly, but the basic medium faded the colour somewhat.

4. Results and Discussion

Nickel (II) complex formed at (pH= 8) has an absorption maximum of (max= 530.5 nm) compared to (max= 476 nm) for the solution reagent. This indicates that the formation of the complex is accompanied by a marked increase in absorbance and a bathochromic shift of approximately (75nm) optimization of variables.

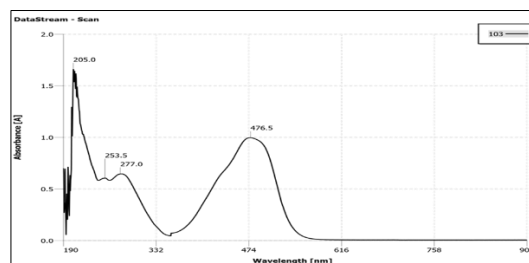


Figure 1: - The reagent spectrum (DPIDA)

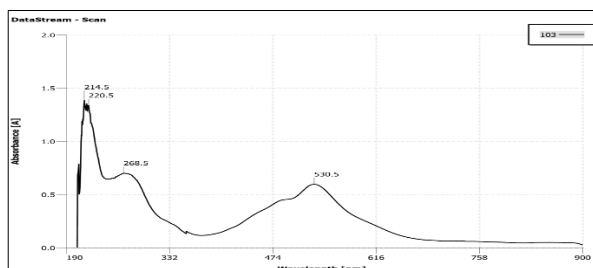


Figure 2: - The spectra of Ni (II) complex with (DPIDA) reagent.

The effect of adding surfactants (micelle medium)

As a result of the use of surfactants, the development of novel techniques for determining specific ions was greatly improved. Many researchers have examined the significance of the creation of (metal, ligand) complexes in the systems including the (Micellar) system, and the impact of adding (Triton X-114) was explored as a possible way to stabilize the complex. Triton X-100 and Tween 80, with varying amounts of nickel (II) absorbance, are presented in table (1).

Concentration of surfactant (v/v %)	Absorption values of Ni complex in (TWEEN-80)	Absorption values of Ni complex in (TRITON-114)	Absorption values of Ni Complex in (TRITON-100)
1%	0.299	0.177	0.259
2%	0.463	0.201	0.321
3%	0.411	0.243	0.347

Optimization of Reaction Conditions

Effects of pH: Reagent (DPIDA) was buffered at several pH ranges from (1 to 10) using HCl (0.1M)/NaOH (0.01M), and the final pH of each solution was recorded by pH-meter. The absorbance was measured at (570 nm, 20 Co) at 20°C for each buffered solution.

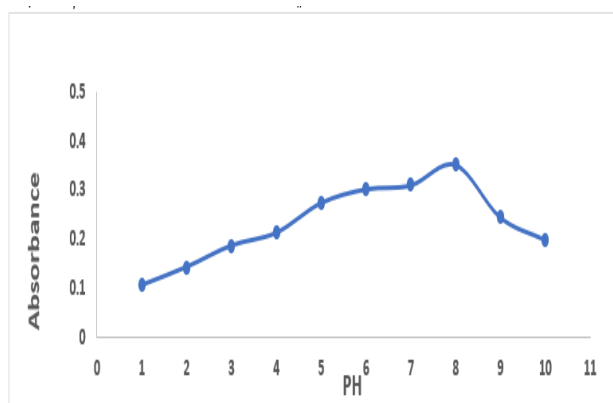


Figure 3: - Effect of pH value

However, when the pH climbed from (1.0 to 8.0) in Figure (3), the absorbance increased gradually, but fast (above pH 8.0), As pH rises, so does the nickel complex solution absorbance. This may be due to an increased sensitivity at this pH.

Complexity stability is affected by the passage of time. Reagent and ion reactions are shown in table 1 utilizing the optimal circumstances, and these findings indicate the composition of mercury (II) complex and stay stable (in terms of absorption values) for 90 minutes after the experiment started. Using this reagent to measure mercury parasitically is encouraged by the findings of this investigation.

The reagent's impact size

At (pH=8), the absorbance of a mercury complex was shown to rise with increasing reagent concentration in the table (2).

Volume Conc. of L. × 10 ⁻³	0.5	1.0	1.5	2.0	2.5	3.0
Abs.	0.267	0.351	0.395	0.455	0.382	0.341

The sequence has an impact

Table (3) shows the sequence of additions used to analyze the reaction content in a complex absorbance, and the results are as follows:

Sequence of Number	Sequence of Addition	Abs. of Cu Complex
1	M+L+PH	0.461
2	L+M+PH	0.457
3	M+PH+L	0.449

Nickel (M), ligand (L), and pH (function of hydrogen ion) table (3) show that the first arrangement is the best one, while the other sequence gives a decrease in absorbance of complex that may be returned to the effect of acid, base inions with metal, so the addition of the first sequence was dependent on this method to determine the nickel ion complex.

The temperature has an effect

Complex formation was studied as a function of temperature change. Results from this investigation demonstrate that the complex's absorption values peak and give the highest colour intensity at the temperature of (30 C0), beyond which the absorption values decline, presumably owing to the complex's instability. Assembling and heating the complex at a temperature of no more than 30 degrees Celsius

Temperature	Absorbance of Ni complex At 1µg /ml
10	0.451
20	0.445
30	0.441
40	0.437
50	0.435
60	0.431

Effect of Time

Findings in Table 1 reveal that ion-reagent reactions may continue for 90 minutes under optimal circumstances, and these results reflect the composition of the complex and stay stable in terms of absorption values.

Using this reagent to measure nickel parasitically is encouraged by the findings of this investigation.

Table 1: The effect of time

Time/Min.	Abs.
1	0.447
10	0.452
20	0.459
30	0.461
40	0.458
50	0.461
70	0.463
100	0.459
24h	0.451
48 h	0.445

Calibration Curve

Figure 3 shows the calibration curve of the nickel-ion complex, and Table 4 displays the analytical results for determining nickel ions by employing reagents. Beers law were observed in the concentration range of (0.1–2.5 g/ml) with the absorptivity (2.4x, 10⁻⁴.L.mol⁻¹. cm⁻¹) (DPIDA).

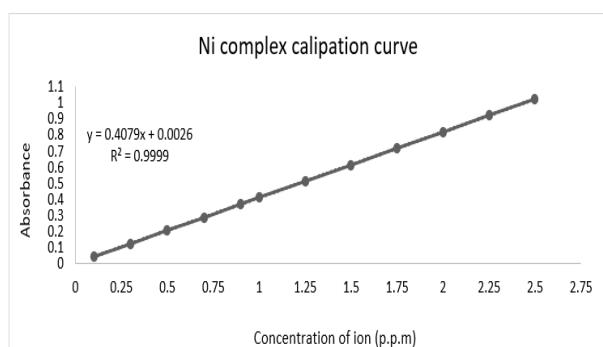


Figure 4: Calibration curve for spectrophotometric nickel determination (II).

Determination of Stoichiometry and Formation Constant

1. The Mole Ratio approach

With the mole ratio technique and the addition of Job's method, the nickel (II) ion (1.994 x 10⁻⁵ M) was studied with the rising composition of the complex produced, and the results were displayed in concentration from the reagent (DPIDA) (1.994x10⁻⁵ - Figs 7 and 8). There was a ratio of 13.958x10⁻⁵M between nickel ion metal (M) and reagent molecules (L), which formed a complex (metal:L) with the reagents at pH=8...

Table 5: - Nickel analytical data (II)

Analytical Data	Value
linear equation	Y=0.407x
Linear range [M]	0.1- 2.5
Detection limit(μg/mL) ^a	0.004
Limit of quantification (μg/mL) ^b	0. 627
Molar Absorptivity (Lmol ⁻¹ cm ⁻¹)	2.4×10 ⁴
Correlation coefficient	0.9989
λ _{max}	530nm
Temp.	30°C
Time	90 min
Color of product	Dark violet
a Limit of detection (LOD)= (SD/S) *3.3 b (Limit of quantification) LOQ= (SD/S) *10 where SD is standard	

deviation, S is the slope of the calibration curve

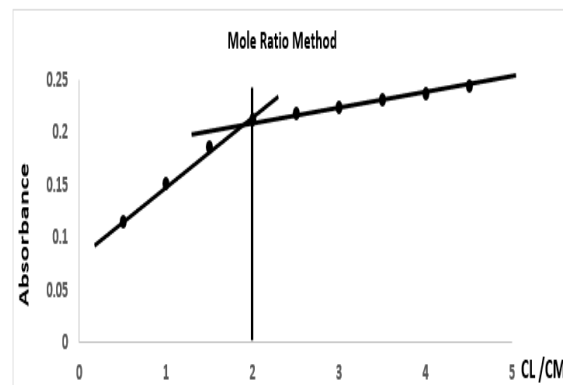
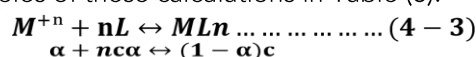


Figure 5: - Mole ratio method

The mole ratio approach was used to estimate the stability constant of the colored complex concerning the equilibrium reaction for the complex. There are examples of these calculations in Table (6).



$$K = \frac{[ML_n]}{[M^{+n}][L]^n} \dots \dots \dots (5 - 3)$$

$$K = \frac{\alpha^n}{(1 - \alpha)^n c^n} \dots \dots \dots (6 - 3)$$

$$K = \frac{1 - \alpha}{n^n \alpha^{n+1} c^n} \dots \dots \dots (7 - 3)$$

$$\alpha = \frac{Am}{Am + As} \dots \dots \dots (8 - 3)$$

Am is maximal absorption and (As) is absorption at stoichiometry, as seen in the figure

Table 6: - The stability constant value of complex

Complex	Am Value	As Value	degree of dissociation α	Stability Constant K
[Ni (DPIDA) ₂]	0.245	0.231	0.130	5.8 ×10 ⁶

Table (6) shows that the complex is quite stable, allowing the ligand (DPIDA) to be used in the spectrum estimate of nickel ions.

2- Job's method in this method mixture of different volumes of the solution in equal concentration (1x10⁻³ M) from both ion (Ni⁺²) and ligand were mixed.

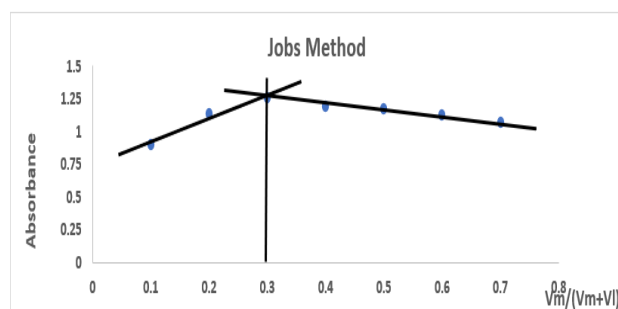


Figure 6: - Job's method of continuous variations.

There were four different kinds of buffer solutions tested to see whether the type of buffer solution affected the absorbance of the nickel (II) complex and the difference in absorption values of the nickel (II) ion complex with the reagent was noted (DPIDA)

The effect of adding buffer solutions

using optimal conditions and the results are shown in Table (7).

Table 7: - The effect of adding buffer solutions	
Type of buffer solution	Absorbance of Ni complex
Phosphate	0.425
Citric acid	0.169
Acetate	0.143
Ascorbate	0.416
Absorbance value without addition buffer solution of Ni complex = 0.465	

To achieve high sensitivity and accuracy, the acidic

function was limited to using an acid or base only, according to the results of the experiment. The nickel (II) complex and the presence of the buffer solution absorption was less than the absorption obtained with the use of hydrochloric acid and dilute sodium hydroxide.

Effect of ionic strength

There was a study done to see how the addition of various concentrations of constant volume Na₂SO₄ and NaNO₃ affected the complex solution as indicated in Table (8).

Table 8: - The effect of ionic strength solutions					
Added salt	Molar Concentration Of added salt	Absorption after adding salt	Added salt	Molar Concentration Of added salt	Absorption After Adding salt
NaNO3	0.0005	0.504	Na2SO4	0.0005	0.583
	0.005	0.565		0.005	0.536
	0.05	0.488		0.05	0.543
	0.5	0.549		0.5	0.413
Absorption before adding salt to the Ni complex at 570 nm = 0.461					

Effect of Foreign Ions

Some cation and anion solutions employed as

foreign ions were introduced to the iron solution to examine the interference impact,

A-The cation effect B- The inions effect

Table 9: - The effect of Cation					
Interfering ion	Ion salt formula	5 µg/ml		10 µg/ml	
		The absorption Values after adding the ions	Mistake Percentage E%	The absorption Values after Adding the ions	Mistake Percentage E%
---	---	0.461		0.461	
Fe ³⁺	FeCl ₃ .6H ₂ O	0.813	76.351 %	0.956	107.375 %
Cu ²⁺	CuCl ₂ .6H ₂ O	0.001	- 99.782 %	0.001	- 99.783 %
Cr ³⁺	CrCl ₃ .6H ₂ O	0.295	- 36.008 %	0.937	103.253 %
Co ²⁺	CoCl ₂ .6H ₂ O	0.043	- 90.067 %	0.001	- 99.783 %
Ba ²⁺	BaCl ₂ .2H ₂ O	0.873	89.370 %	0.921	99.783 %
Hg ²⁺	HgCl ₂	0.036	- 92.192 %	0.077	- 83.297 %
Cd ²⁺	CdCl ₂	0.092	- 80.043 %	0.021	- 95.444 %

Table 10. Accuracy and precision studies.					
Interfering ions	Formula Structure of ions	µg /ml10		40µg/ml	
		The absorption values after adding the ions	Mistake percentage E%	The absorption values after adding the ions	Mistake percentage E%
—	—	0.461	—	0.461	—
Cr ₂ O ₇ ²⁻	K ₂ Cr ₂ O ₇	0.346	- 24.945 %	0.534	15.835 %
SCN ¹⁻	KSCN	0.663	43.817 %	0.725	57.266 %
CO ₃ ²⁻	K ₂ CO ₃	0.777	68.546 %	0.679	47.288 %
SO ₄ ²⁻	K ₂ SO ₄	0.884	91.757 %	0.891	93.275 %
IO ₃ ²⁻	KIO ₃	0.820	77.874 %	0.919	99.349 %
CrO ₄ ²⁻	K ₂ CrO ₄	0.779	68.980 %	0.803	74.186 %
Br ¹⁻	KBr	0.752	63.123 %	0.933	102.386 %

Ni (II) ion interferences were studied using a few ions that were selected for this purpose (Table 9). Due to the rivalry between these ions and Ni (II) to form the complex with the ligand, it was found that some of the ions enhanced absorbance while others lowered absorbance, which lessened competition and improved the sensitivity of this approach towards the

ion Ni (II). Ni-specific and sensitive reactions were observed (II). Masking agents may be used to test the selectivity of a response.

Precision and Accuracy of the Method Described.

Recoverability and relative standard deviation (RSD

per cent) were used to measure the implemented method's accuracy and precision. In the table, recovery and RSD percentages were shown (11).

Table (11) shows that the devised approach was accurate, with a relative standard deviation of 0.4%.

Conc. of present [M]	Conc. of found [M]	RSD%	Recovery%	Error%
1.703×10^{-5}	1.635×10^{-5}	4.239	78.31	21.69
2.555×10^{-5}	2.357×10^{-5}	1.830	97.37	2.73

Table 12 shows that temperatures only have a little impact on the stability of the complex.

T (C)	T(K)	α	$k \times 10^6$
10	283.15	0.115	8.823
15	288.15	0.121	7.602
20	293.15	0.129	6.607
25	298.15	0.138	4.858
30	303.15	0.144	4.328

The stability constant for the [Ni (DPIDA)2] complex is affected by temperature...Stability constants for Ni (II) complexes as a function of temperature are shown in Table 11.

DPIDA was used to study the stability constant of Ni (II) at varying temperatures ranging from (10-35) oC. Tables show the outcomes of the study (12)

The Complex's Thermodynamics

Data on the thermodynamic functions H, G and S were obtained, as shown in Fig. 6 and Table 7. (12).

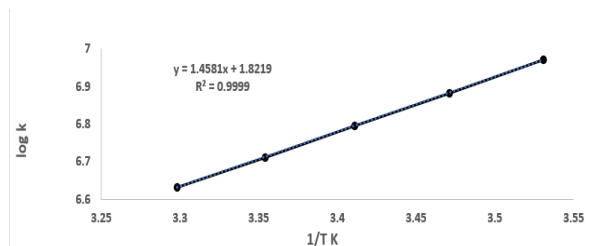


Figure7: - Relation between Log K and 1/T values for nickel (II) complex.

Table 13: - The effect of temperature on thermodynamic function for nickel (II) complex					
T(K)	(1/T)	Log K	ΔH Kj. Mol ⁻¹	ΔG Kj. Mol ⁻¹	ΔS Kj. Mol ⁻¹ .k ⁻¹
283.15	3.531	6.970	-27.916	- 37.646	0.0343
288.15	3.470	6.882		- 37.954	0.0348
293.15	3.411	6.795		- 38.272	0.0353
298.15	3.354	6.712		- 38.163	0.0343
303.15	3.298	6.630		- 38.511	0.0349

The exothermic nature of the reaction was shown by the negative value of enthalpy, which can be seen by lowering the temperature. The creation of complexes will rise, as well as the spontaneous reaction based on the negative sign of the free energy

Because the entropy of the complex approaches zero, it is safe to assume that it is stable (less random and spontaneous).

Study of FT-IR Spectra for Ligand and Complex

Figs 8, 9 and Table 10 explain the FT-IR study and the absorption frequencies for complex and the reagent (DPIDA).

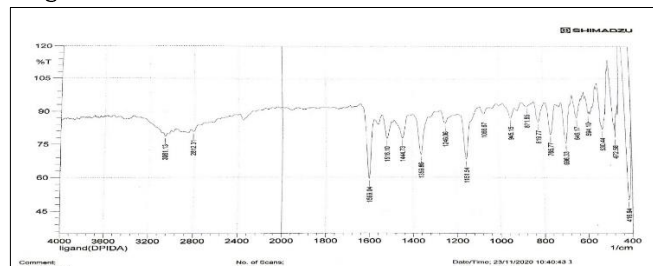


Figure 8: - FT-IR spectrum of ligand

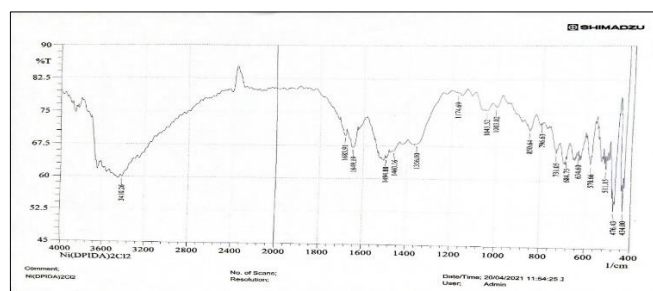


Figure 9: - FT-IR spectrum of the complex.

Table 14: - FT-IR absorption frequencies for reagent and the reagent (DPIDA)

Compound	DPIDA	[Hg (DPIDA)2Cl2]
(N-H)	3061 m	3410 s
(C-H) Ar	2812 m	2974 w
(N=N)	1444 m	1460 m
(C=N)	1599 m	1649 m
(C=C)	1516 m	1529 m
(C-O)	1246 m	1251 s
(M-O)	480 s
(M-N)	597 s

S=strong, M=medium, W=weak

The Suggested Figure for the Complex

The FT-IR spectra and the stoichiometry obtained from the Job and Mole ratio methods suggest a complex structure as depicted in figure (10).

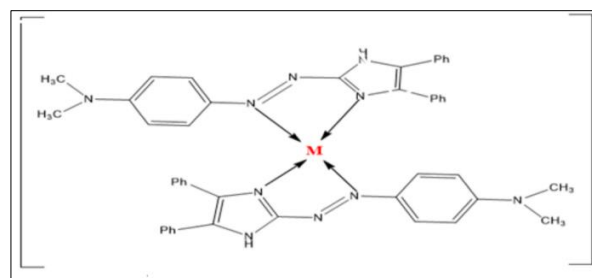


Figure 10: Suggested structure for the complex [M(DPIDA)2]

5. Conclusion

Nickel has been quantified in several samples using a highly sensitive approach, as well as a low-cost method (II). Tests in the lab and the field have shown that the new approach can accurately measure Ni (II). In the tests, nickel (II) was quantified in a broad

variety of samples. Several analytic characteristics show that this approach may be utilized to accurately and reliably identify Ni (II).

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