Spectrophotometric Determination of Micro Amount of Zinc (II) using Sodium 4-((4,5-Diphenyl-Imidazol-2-Yl) Diazenyl)-3-Hydroxynaphthalene-1-Sulfonate (SDPIHN) In the Presence of Surfactant, Thermodynamic Functions and Their Analytical Applications Are Studied

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Abstract
Anew easy, quick, highly selective and spectrophotometric sensitivity technique has already been expanded find out zinc (II) ion by using sodium 4-((4,5-diphenyl-imidazol-2-yl) diazenyl)-3-hydroxynaphthalene-1-sulfonate (SDPIHN) as chromogenic reagent (ligand) in presence of non-ionic surfactant (Triton x-100) at (PH=10.5) to form a dark brown complex in wavelength (599 nm). At the specified PH, the compound remained stable for four hours. In the concentration range (1µg/mL-12µg/mL), Beer's low is observed, along with molar absorptivity($0.2229x10^4$L.mol^{-1}.cm^{-1}) and Sandal's sensitivity (0.0293 µg/cm^2). (0.227 µg/mL) and (0.749 µg/mL), respectively, were the limits of detection (LOD) and Limit of quantification (LOQ). (Mole ratio technique, job's method) were used to investigate the stoichiometry of complexes, and the two approaches suggested that the metal to reagent ratio is (1:2). The reagent concentration's absorption effect's value, surfactant concentration, PH, reaction duration, temperature, addition sequences, ionic strength, masking agent, and the influence of many parameters such as affect cations and anions, among others. Thermodynamic functions were also computed by looking at temperature's influence on the reaction, which was referred to as an endothermic reaction. As part of the complex solid's creation, researchers investigated some of its physical properties, like solubility, molar conductivity, and melting point. The zinc complex generated was examined using UV-visible rays, and the new method's precision and accuracy were assessed using Relative Standard Deviation (RSD%) and Relative Error (E%).

1. Introduction

Azo dyes are one of the most significant compounds because they are extremely effective at repelling metal ions from the periodic table[1]. The Azo chemical formula is R-N=N-R', where (-N=N-) is the Azo group and R or R' might be aryl or alkyl compounds. Azo compounds with the formula (R-N=N-R) are known as Azo fibrous group, and they are not widely used in various fields because they are unstable due to their rapid decomposition into nitrogen, whereas Azo compounds with the formula (Ar- N=N-Ar) are known as aromatic Azo compounds, and they are more stable due to their active donor (N=N) [2-5]. The interaction of heterocyclic Azo compounds with metal ions results in chelatic coordination complexes that are of interest in analytical and biological chemistry [6-9]. Azo compounds have a strong pharmacological activity, and when delivered as a metal complex, the medicine becomes extremely effective[10].

Zinc is one of the microelements required for optimal human cell activity [11]. It can function as a catalyst, causing several enzymes involved in the production and structure of DNA, RNA, proteins, and hormones to become active[12]. Zinc is required during the division, growth, and regeneration of cells[13]. It also defends cells against free radical damage [14]. Zinc is the second most prevalent trace element in the human body, trailing only iron. Zinc is one of the few transition elements, including Mn, Fe, Co, Cu, Zn, and Mo, that are necessary for human physiology, along with the first and second series metals Na, K, Mg, and Ca. Immune function can be harmed by a lack of zinc [15, 16]. Zn(II) metal core complexes usually have a four-coordination number and a tetrahedral geometry. Zn, as a late transition metal with full d valence electrons, may form a stable 18-electron complex with its ligands utilizing a 4-coordination number [17].

Surfactants are substances that reduce the surface tension between two liquids or a liquid and a solid. Liquids have a property called surface tension. It is the amount of labor or energy necessary to raise the surface area of a liquid due to intermolecular forces.

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The proportion of molecules present at a liquid's surface to those present in the liquid's bulk is determined by their concentration. At low concentrations, surfactants linger on the liquid's surface. The surface tension of water decreases as more surfactant is added. As the surface becomes clogged with surfactants, more molecules form micelles. This concentration is known as the critical micelle concentration (CMC) [18, 19].

The creation of the complex between zinc metal and the Azo-linked Schiff base reagent is explained in this study as a simple and sensitive spectrophotometric approach for the measurement of trace quantities of zinc (II) (DPH). Elemental analysis and infrared, UV–Vis spectroscopy were used to analyze the reagent and its complex. The effect of certain analytical parameters on complex formation, such as pH and reagent concentrations, was investigated.

2. Experimental

Preparation of (SDPIHN) ligand

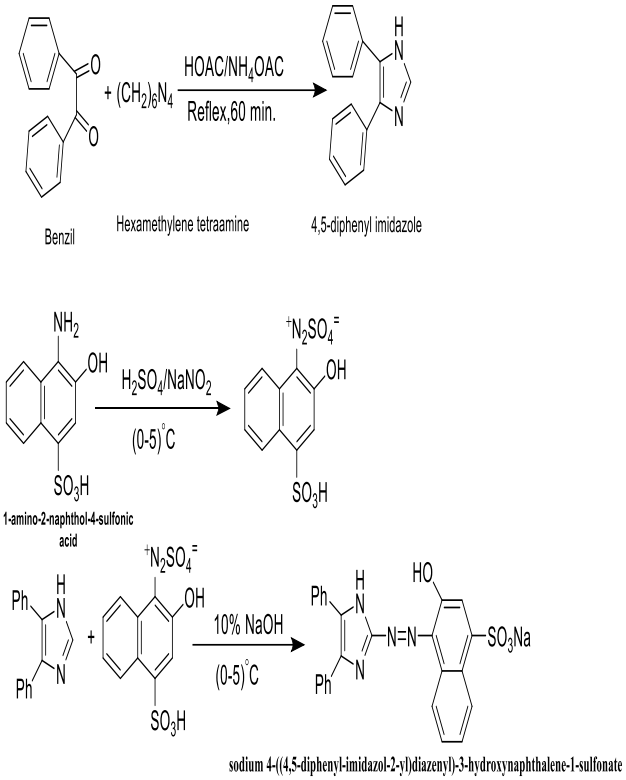
A-preparation of (4,5 Diphenyl imidazole)

(4.2g, 0.02mol) of benzil and (1.4g,0.0099 mole) of hexamethylene tetraamine were mixed in a 250mL round flask with (12g,0.155 mole) of ammonium acetate, after that comes (80mL) of ice acetic acid, and the sublimation of the solution with continued stirring for an hour, then transfer the solution after cooling it to a Baker´s capacity (1 L) and dilute by adding (400 mL) of distilling water , followed by sodium hydroxide precipitation of the imidazole derivative to clear the residue, followed by filtration and rinsing with distilled water. The formed precipitate was dried and tested for melting point. (229 - 230 C°) and the yield ratio was (93 %).

B-Synthesis of Ligand Sodium 4-((4,5-diphenyl-imidazol-2-yl) diazenyl)-3-hydroxynaphthalene-1-sulfonate (SDPIHN).

By dissolving (3.588 g, 0.02 mole) of 1-amino-2-napthol-4-sulfonic acid in a solution made by combining (4 mL) sulfuric acid with (50 mL) distilled water. The diazonium salt pairs were used to make the new imidazole liquor. Cool the mixture to 0ºC, then drop in a solution of sodium nitrite (1.38 g, 0.02 mole) dissolved in (10 mL) distilled water, stirring constantly and keeping the temperature beneath 5ºC, before allowing the solution to stay for 15 minutes to complete the nitrogenation process.

Cool to 0ºC and leave for half an hour after dissolving (3.3 g.) of the imidazole derivative in 50 mL ethanol with 50 mL NaOH. This imidazole derivative solution was gradually added to the salt solution while it was being cooled to 0ºC. The solution was stained a dark red color, and it was then left at 0ºC for 2 hours. The acidity function (PH=6) was then obtained by adding dilute sulfuric acid (50mL,1M) to it drop by drop to generate a reddish-orange precipitate. The precipitate was filtered and rinsed with pure water to remove the sodium chloride formed during the pairs and neutralization procedures, then dried and recrystallized with ethanol to produce the in its pure form and the yield ratio was(57%).



Scheme 1: Preparation of (SDPIHN) ligand

Preparation of Standard Solutions

Zinc (II) solution (1000 µg/mL): made by dissolving (0.455 g) of [Zn (NO3)2].3H2O in 100 mL distilled water.

Sodium hydroxide solution (0.1M):100 mL distilled water was added to (0.4 g) sodium hydroxide to make this solution.

Hydrochloric acid solution (0.1M): 0.40 mL concentrated hydrochloric acid (38 %, 1.19 g/mL) was diluted in 50 mL distilled water to make this solution.

Reagent solution (SDPIHN): was made by dissolving the required weight (0.1g) in absolute ethanol and (0.1g) filling the remaining capacity to (100mL) with ethanol.

Interferences

Cations solution of (Cd^{2+} ,Ni^{2+}, Fe^{3+} , Cu^{2+} , Pb^{2+},Cr^{3+},Co^{2+},Mg^{2+},$Hg^{2+}$and Ag^{+}) ions were prepared by dissolving (0.219g) of $Cd(NO_3)_2.4H_2O$, (0.495g) of $Ni(NO_3)_2.6H_2O$, (0.723g) of $Fe(NO_3)_3.9H_2O$, (0.380g) of $Cu(NO_3)_2.3H_2O$,(0.159g) of $Pb(NO_3)_2$,(0.493g) of $Co(NO_3)_2.6H_2O$, (0.769g) of $Cr(NO_3)_3.9H_2O$,(1.055g) of $Mg(NO_3)_2.6H_2O$,(0.170g) of $Hg(NO_3)_2.H_2O$,(0.157g) of $AgNO_3$ respectively in 100 mL distilled water for each.

3. Results and Discussion

The absorption spectra of the reagent and zinc (II) complex are shown in figures 1 and 2. The reagent solution spectra has an absorption maximum of (λ max=463 nm), while the zinc (II)

complex formed at (pH= 10.5) has an absorption maximum of (λ max= 599nm), indicating that the complex formation is accompanied by a significant increase in absorbance and a bathochromic shift of approximately (136 nm).

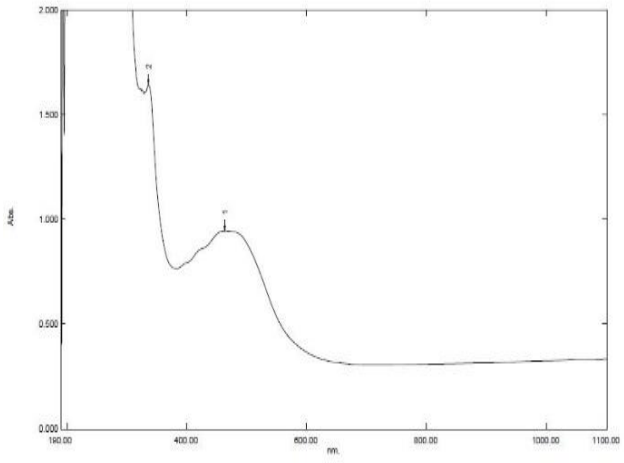


Figure 1: The spectrum of reagent [SDPIDHN]

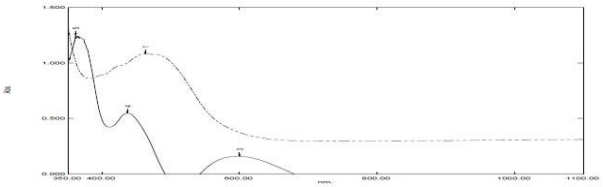


Figure 2: the spectrum of Zn(II) complex with [SDPIHN] reagent

The Effect of Adding Surfactants

Surfactants have a major impact on spectrophotometry and chromatography, particularly in the development of new ion estimation methods. Numerous studies have emphasized the importance of the development of (metal-ligand) complexes in systems including the system, for reasons such as the creation of the complex may be more stable in these systems, therefore the effect of adding (Triton X-100) was investigated and (Triton X-114), (Tween-80), and (Sodium Dodecyl Sulfate) in the absorbance of zinc (II), as shown in table (1).

Table 1: - Absorption values of zinc complex in different mediums Absorption of Zn complex without addition =0.114				
Concentration of surfactant (v/v %)	Absorption values of Zn complex in (Tween-80)	Absorption values of Zn complex in (Triton-114)	Absorption values of Zn complex in (Triton x-100)	Absorption values of Zn complex in (Sodium Dodecyl Sulfate)
10%	0.116	0.115	0.117	0.111
20%	0.118	0.117	0.122	0.085
30%	0.120	0.119	0.126	0.061

Optimization of Reaction Conditions

1-Effect size of reagent

As revealed in the experiment, the influence of reagent volume on zinc complex absorbance is presented in Table (2). The absorbance increased as the reagent concentration was increased, according to the results.

Table 2 : Effect of reagent volume							
Volume of reagent(mL)	0.5	1	1.5	2	2.5	3	3.5
Abs.	0.055	0.116	0.168	0.246	0.304	0.322	0.311

2-Effect of surfactant (Tx-100) volume

As revealed in the experiment, the influence of reagent volume on zinc complex absorbance is displayed in Table (3).

Table 3: The effect of (Tx-100) volume					
Volume of (Tx-100) (mL)	0.25	0.5	1	1.5	2
Abs	0.336	0.337	0.331	0.328	0.326

3-Effect of PH value

The absorbance was measured at (599nm) at 20ºC using a standard amount of zinc (II) and reagent (SDPIHN) buffered at several pH-ranges from (1 to 10.5) using HCL(0.1M)/ NaOH (0.1M), and the final pH of each solution was calculated with a pH-meter.

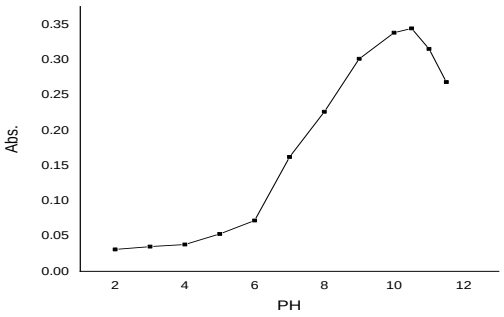


Figure 3: Effect of PH value

The absorbance of the zinc complex solution grew steadily as the pH increased from 2.0 to 10.5, but rapidly declined above pH 10.5. The higher absorbance of the zinc complex solution under these circumstances could be attributed to an increase in the reagent's sensitivity at this PH value.

4-Effect of Time

Table (4) shows the reagent's follow-up interaction with the ion under optimal conditions. These findings reflect the composition of the zinc (II) complex, which is stable (in terms of absorption values) 4 hours after the experiment started.

Table 4: Time effect									
Time(min.)	1	10	20	30	60	120	180	240	1440
Absorbance	0.342	0.343	0.343	0.343	0.343	0.342	0.341	0.338	0.315

5-Effect of Temperature

The influence of temperature variation on the development of complexes was investigated. Table (5) illustrates the findings of this study, which reveal that at the temperature of (30 C0). The complex's absorption values reach their peak and produce the best color intensity; however, the absorption values thereafter diminish, possibly due to a decrease in the complex's stability.

Table 5: Temperature effect						
Temperature C°	10	20	30	40	50	60
Abs.	0.324	0.342	0.361	0.280	0.244	0.201

6-Effect of Sequence

The six addition arrangements were used to investigate the sequence of the reaction content in a complex absorbance. The findings are summarized in table (6)

Table 6: Sequence effect		
Sequence of number	Sequence of addition	Abs. of Zn complex
1	M+L+Tx-100+PH	0.341
2	M+ Tx-100+L+PH	0.332
3	Tx-100+M+L+PH	0.301
4	L+M+Tx-100+PH	0.287
5	M+PH+L+Tx-100	0.326
6	M+L+PH+Tx-100	0.205

Tx-100 = triton x-100, pH = hydrogen ion functions
M = zinc ion, L = ligand
The results in table (6) demonstrate that the first sequence is the best, but the other sequence results in a drop in complex absorbance, which might be due to the impact of acid, base inions with metal, therefore the first sequence was used to decide the zinc ion complex in this approach.

7- Ionic Strength Effect

The sodium nitrate and sodium sulfate salts were prepared at different concentrations ranging from (0.0005-o.5 M) for each salt in order to demonstrate the influence of ionic strength on the absorption of zinc(II) complex after adding (1ml) from solutions of these salts to complex zinc(II).

Table 7: Ionic strength effect					
Adding salt	Conc.(M) of add salt	Abs.	Added salt	Conc.(M)of add salt	Added salt
Na_2SO_4	0.5	0.330	$NaNO_3$	0.5	0.304
	0.05	0.322		0.05	0.305
	0.005	0.293		0.005	0.271
	0.0005	0.288		0.0005	0.279

According to the data provided in table (7), all the concentrations listed in the table have no effect on the absorption value.

Calibration Curve

In the concentration range (1–12 µg/mL), beer's low obeyed. The molar absorptivity is (0.222×104 L mol-1 cm-1) and the absorbance of zinc ion complex was found to be linear based on the metal concentration. Figure (4) depicted the zinc ion calibration curve, while table (8) supplied analytical data for quantifying zinc ion using reagents (SDPIHN).

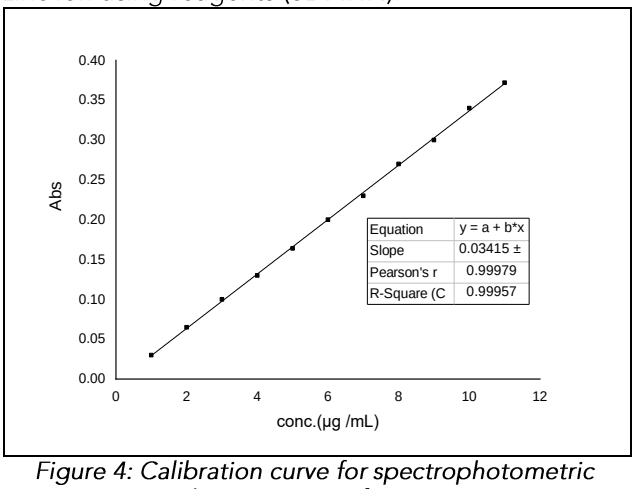


Figure 4: Calibration curve for spectrophotometric determination of zinc (II)

Table 8: Data collected for zinc (II) analysis	
Analytical Data	Value
Molar Absorptivity ($L.mol^{-1}.cm^{-1}$)	0.2229×10^4
Slope	0.0341
Linear equation	Y=0.0341X
Linear range(µg/mL)	(1-12)
Sandal sensitivity ($µg/cm^{2)}$	0.0293
Detection limit (µg/mL)	0.2271
Limit of quantification (µg/mL)	0.7496
Linearity coefficient (R^2)	0.9996
Correlation coefficient (r)	0.9998
λ_{max}	599nm
Color of product	Dark brown

Stoichiometry And Formation Constant Estimation

To investigate the composition of the complex produced, the mole ratio method was combined with Job's method of continuous variations. At pH=10.5, both approaches revealed that the metal ion to reagent ratio (M: L) was (1:2).

1-Job's Method

In this procedure, a mixture of various identical concentrations of the ion (Zn^{2+}) and the ligand were generated in different amounts of solution ($1x10^{-4}$ M).

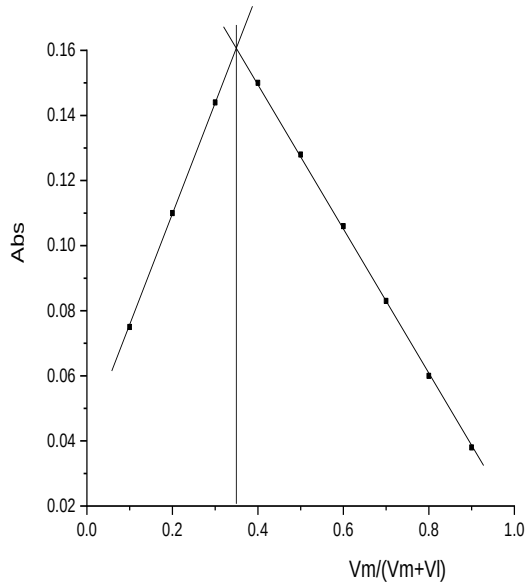


Figure 5: Job`s method

2-Mole Ratio Method

The approach indicate that zinc ion forms a (1:2) complex (M-L) with reagent by utilizing a known and constant concentration of zinc (II) ion (6.127× 10 -4 M) with increasing concentration of reagent (SDPIHN) (1.53x10-4 -18.381x10-4M).

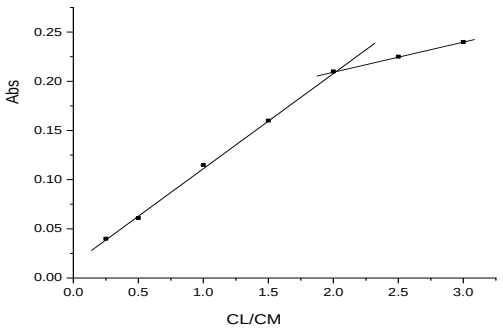


Figure 6: Method of Mole Ratio

The stability constant was calculated using the mole ratio technique from the equilibrium reaction of the colored complex. The calculated findings are displayed in the table below (9).

$$M^{+n} + nL \leftrightarrow MLn$$

$$\alpha + nc\alpha \leftrightarrow (1-\alpha)\, c$$

$$K=\frac{[MLn]}{[M^{+n}][L]}$$

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

$$K=\frac{1-\alpha}{n^n\alpha^{n+1}c^n}$$

$$\alpha=\frac{Am-As}{Am}$$

Where (As) is absorption at the stoichiometric concentration and (Am) is maximum absorption.

Table 9. The value of the complex stability constant				
Complex	(Am) Value	(As) Value	degree of dissociation (α)	Stability Constant (K)
[Zn (SDPID)$_2$]	0.240	0.210	0.125	5.967×10^8

The results in Table (9) show that the complex is stable, allowing the ligand (SDPIHN) to be used in the estimation of zinc ions' spectrum.

The impact of temperature on the stability constant of the [Zn (SDPIHN)$_2$] complex

At temperatures ranging from (10-30) C0, the values of the Zn (II) stability constant with the reagent (SDPIDHN) were investigated. Table (10) displays the results

Table 10. The impact of temperatures on the stability constant of Zn (II)complex.			
T (C^o)	T (K)	α	K×10^8
10	283.15	0.208	1.172
15	288.15	0.227	0.880
20	293.15	0.250	0.639
25	298.15	0.276	0.458
30	303.15	0.290	0.387

The data in Table (10) showed that temperature had only a minor impact on the stability of the complex.

Complex's Thermodynamic Function

Thermodynamic functions ΔG, ΔH and ΔS were determined, and the results are shown in Figure (7) and Table (11).

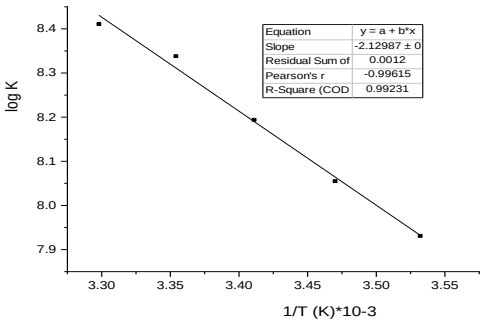


Figure 7: Relationship between Log K and 1/T values of zinc(II) complex.

Table 11: The impact of temperature on thermodynamic function for zinc (II) complex.					
T(K)	1/T×10^{-3}(K^{-1})	log K	ΔH	ΔG (K.J/mole)	ΔS (K.J/mole .K)
283.15	3.532	7.931	40.764	-42.942	0.2956
288.15	3.470	8.055		-44.383	0.2954
293.15	3.411	8.194		-45.932	0.2957
298.15	3.354	8.338		-47.537	0.2961
303.15	3.298	8.411		-48.757	0.2953

When the enthalpy value is positive, the reaction is endothermic, which may be seen by increasing the temperature. Because of the negative sign of free energy, the complex formation will be decreased, and the reaction will be spontaneous.

Effect of Foreign Ions

To explore the interference effect, a specific concentration of different cation and anion solutions, which were utilized as foreign ions, were mixed with zinc solution.

A-The cations effect

B- The inions effect

Table 12:The Cations effect

Foreign/ions	Cation's formula structure	(10µg/mL) Abs. after addition of Cations	Error%
Cd^{2+}	$Cd(NO_3)_2.4H_2O$	0.209	38.88
Ni^{2+}	$Ni(NO_3)_2.6H_2O$	0.321	6.14
Cr^{3+}	$Cr(NO_3)_3.9H_20$	0.255	25.43
Co^{2+}	$Co(NO_3)_2.6H_2O$	0.402	-17.54
Fe^{3+}	$Fe(NO_3)_3.9H_2O$	0.167	51.16
Hg^{2+}	$Hg(NO_3)_2$	0.088	74.26
Pb^{2+}	$Pb(NO_3)_2$	0.190	44.44
Mg^{2+}	$Mg(N0_3)_2.4H_2O$	0.294	14.03
Ag^{+}	$AgNO_3$	0.392	-14.61
Cu^{2+}	$Cu(NO_3)_2.3H_2O$	0.266	22.22
Absorbance without interferences = 0.342			

Table 13: The inions effect

Foreign ions	Formula structure of Anions	(906µg/0.5mL) Absorption after addition of Anions	E%	(453µg/0.25mL) Absorption afteraddition of Anions	E%
SO_4^{2-}	K_2SO_4	0.250	26.87	0.290	15.20
Br^{1-}	KBr	0.440	-28.64	0.470	-37.42
SCN^{1-}	KSCN	0.206	39.76	0.280	18.12
$IO3^{1-}$	KIO_3	0.111	67.54	0.201	41.22
CrO_7^{2-}	K_2CrO_7	0.314	8.18	0.320	6.43
CO_3^{2-}	K_2CO_3	0.390	-14.03	0.340	0.58
CN^{1-}	KCN	0.190	44.44	0.137	59.94
Absorbance without interferences = 0.342					

The results obtained in the above tables show that most of the ions have an effect on the absorption values of the zinc (II) complex; depending on the nature of the added ions, some of these ions increased absorbance while others decreased absorbance; this was due to competition of these ions with Zn(II) to form the complex with the ligand, which decreased competition and increased the sensitivity of this method towards Zn(II) ion. The reaction for Zn (II) was both selective and sensitive. By using appropriate agents of omission and substitution, the ability to be discerning in response can be validated.

Effect Masking agents

For the determination of the zinc(II) complex, the optimal masking agent is limited. This investigation was done by adding (1mL) from each masking agent, as stated in table (14), to see the effect of the competitive process between them and the reagent in interacting with the interfering ions and withholding them .

Table 14: The effect of masking agent on zinc (II) complex absorption

Masking agent (0.1M)	Abs of Zn(II)complex
Without Masking agent	0.342
Thiourea	0.503
Ascorbic acid	0.195
Na2EDTA	0.165
Citric Acid	0.366
KCl	0.243
Na2HPO4	0.092
Formal dehyde	0.280

Table (14) shows that (Citric acid) has no influence on the absorption of the produced complex, indicating that it could be utilized as a masking agent.

Use of a best masking agent to determine the zinc (II) complex in the presence of cat ions interference

The best masking agent (Citric acid) described in table (15) was used to obtain the best estimate of the zinc (II) complex in the presence of interfering cationic ions.

Table 15: The effect of masking agent in the presence of cat ions on zinc(II) complex absorption.

Foreign ions	Absorption after addition cat ion (10µg/ml) and addition masking agent (0.1M)	Relative Error (E%)
Cd^{2+}	0.344	-0.580
Ni^{2+}	0.340	0.584
Cr^{3+}	0.343	-0.290
Co^{2+}	0.348	-1.750
Fe^{3+}	0.339	0.870
Hg^{2+}	0.341	0.290
Pb^{2+}	0.345	-0.870
Mg^{2+}	0.349	-2.040
Ag^{+}	0.338	1.160
Cu^{2+}	0.346	-1.160

The absorption values of the zinc (II) complex in the presence of interfering cations when adding a better

masking agent, absorbance values near to the absorbance values before adding the interference, can be shown in table (15).

Statistical treatment of the results

By making three series of zinc ion solution with varied concentrations, reading the absorption three times for each concentration, and then treating the results to determine the value of (RSD %), as shown in table (16), the relative standard deviation was accepted as a measure of precision.

Table 16: Calculation the value of Relative Standard Deviation

Zinc added [M]	Abs. of Zn (II) complex	RSD%
4.588×10^{-5}	0.101, 0.102, 0.0.104, 0.105	1.772
7.640×10^{-5}	0.165, 0.168, 0.171, 0.166	1.579
1.830×10^{-4}	0.375, 0.381, 0.378, 0.376	0.700

Sensitivity of spectrometric method in determination zinc (II) ion

The sensitivity of the method employed to determine zinc in this way was indicated by the expression limit of detection. The results showed that this spectroscopic method can estimate the lowest concentration of zinc ion to be (3.475×10-6 M), indicating that the method is successful in determining zinc (II) ion.

Determination of the melting point of the complex:

The melting point of the zinc complex ranged from (261-263 C0), while the reagent's melting point was (219-223 C0). The discrepancies in melting point values at different degrees of disintegration confirm the complex's complexity and reveal the partial weight gain of the complex relative to the detector's partial weight.

Measurement of Conductivity

The complex's molecular conductivity was measured at room temperature and under ideal conditions, and it was found to be (0.170 µS/cm). Because conductivity is a measure of an electrolyte solution's ability to charge electricity through the migration of ions under the influence of an electric field, the complex is clearly non-ionic and uncharged.

FT-IR Spectra of Ligand and Complexes

Look at the figures to better understand the FT-IR study results and the absorption frequencies of the complex and ligand.

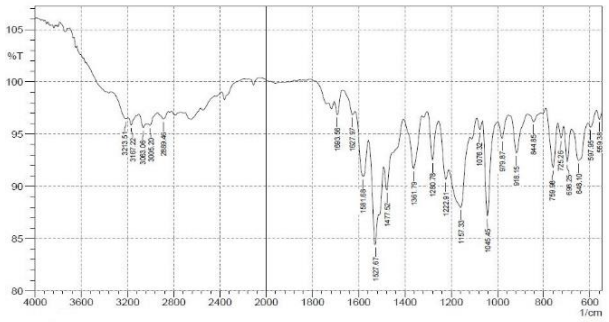


Figure 8: Reagent FT-IR Spectroscopy

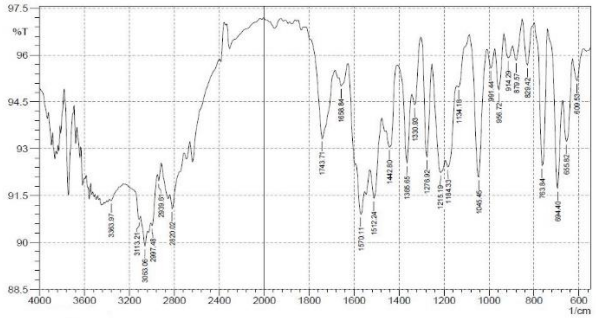


Figure 9: complex FT-IR Spectroscopy

Application

The proposed approach was used to measure zinc (II) in medication samples using zinc as an active ingredient. The sample was prepared using an acid digestion (HCl) method and then added to a reagent (SDPIHN) to determine the zinc concentration in the sample; the findings are provided in table (16).

Table 16: Result of application for zinc(II) in a drug

Abs. of a drug	Conc. Zinc (II) present (µg/ml)	Conc. Zinc (II) found (µg/ml)	Recovery%	Error%
0.350	10	10.07	99.304	0.696

4. Conclusion

The analytical method for determining zinc (II) in several samples has been devised, and it is simple, low cost, quick, and accurate. The reagent can detect zinc (II) in a variety of materials, according to the findings. Identification, detection limit, and precision are examples of analytical metrics that show this approach can determine Zn (II). The approach is suitable for spectrophotometric analysis because it produces a dark brown zinc complex.

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