Determination and Identification of Nickel(II) Spectroscopy in Alloy Samples Using Chromogenic Reagent (HPEDN)

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Abstract. A modern and easy procedure performed using the spectrophotometric method and estimating of Ni (II) in alloy samples. Using a ligand 1-((4-(1-(2-hydroxyphenylimino)ethyl)phenyl)diazene)naphthalene-2-ol (HPEDN) and a synthesis of the azo-schiff reaction by reaction p-aminoacetophenone with 2-naphtol in first step and reaction of product with 2-aminonaphthol, Ligand and its complex were identification by using UV-Vis. Spectrophotometry (326nm for ligand (HPEDN), 516nm for complex(Ni-HPEDN)), FT-IR, ¹H-NMR, and ¹³C-NMR. The optimal conditions in the complex composition were studied at pH=9, temperature 25°C, and time 15min. The best concentration was found for Ligand (2×10⁻⁴ M), Under optimum conditions concentration ranged from (1.1-7.1) µg/mL of Nickel(II) were obeyed Beer`s Law, with Molar Absorptivity 0.2648×10⁴L.mol⁻¹.cm⁻¹. Limit of Detection(LOD) and Limit of Quantification(LOQ)were 0.3928 and 1.3082 µg.mL⁻¹, respectively. The stoichiometric composition of the chelate is 1:2 (Ni:HPEDN). This method is Sensitive, accurate and rapid spectrophotometric method. The results obtained were compared with flame atomic absorption spectrometry method and results are in conformity.

Keywords: Determination of nickel(II), Alloy sample, UV-Vis spectrophotometry, Calibration

1. Introduction

Nickel is a silvery white metal with a light golden appearance. It is one of the widespread elements in the earth and it is very important industrial and biological mineral nickel is apparently essential for proper liver function. It helps in the production of prolactin, which plays an important role in the production of milk for the mother. It also helps in absorbing the iron necessary for the body and thus protects against anemia. On the other side, some of nickel compounds are carcinogenic. Ni(II) is a somewhat toxic element compared with other metals. It is known that inhalation of Ni and its compounds can lead, to serious problems, including respiratory system cancer. Moreover, Ni can cause a skin disorder known as nickel eczema and allergic reaction, and certain Ni compounds may be carcinogenic. The researcher Ali were able to estimate the Trace Amounts of Nickel Ions in Human Blood and Urine spectrophotometrically by using an organic reagent 2-aminopyrimidine on nanocellulose. 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%. The aim of this work, is to synthesis and characterization of Azo-Schiff base ligand, and then prepared of its complex with transition Ni(II) and suggested the geometrical structures of Azo-Schiff base complex and his estimate in Alloy samples after applying the best conditions.

2. Experimental

2.1 Materials

All solvents and chemical Reagents used in this study have high purity equipped by B.D.H, Fluka and Merck companies without any additional purification processes.

2.2 Instrumentation

All techniques that were used in characteristic study of the synthesized ligand and its complexes were a-UV-6100 PC Double beam Spectrophotometer (EMCLAB, Germany), FT-IR Spectrometer, Bruker Tensor II in range 500-4000cm⁻¹, Germany, pH-meter (InoLab, WTW, 135i, Germany); Conductivity meter (Digital, InoLab, Germany); Melting point (SMP 30, Stuart, England); Balance BL 2105 (Sartorius, Germany); Mova 400 MHZ, 1HNMR, 13CNMR spectrophotometer.

2.3.1 Synthesis of new ligand (HPEDN)

The ligand 1-((4-(1-(2-hydroxyphenylimino)ethyl)phenyl)diazene)naphthalene-2-ol (HPEDN) was prepared according to the procedure described by Esraa et.al as shown in scheme1. below.
Scheme 1. Pathway of synthesis of 1-((4-(1-(2-hydroxyphenylimino)ethyl)phenyl)diazenyl)naphthalene-2-ol (HPEDN) ligand

2.3.2. Preparation of divalent Nickel(II) complex:
The Nickel complex was prepared by the molar ratio (1:2) (M:L) by dissolving (1 mmol) of the Nickel chloride salt (NiCl₂·6H₂O) in 10 mL of buffer solution in pH=9. and (2 mmol) in 20 mL of ethanol absolute of ligand, the mixture solution of metal ligand was heated at (50–60) °C for 1 h, the precipitate was washed and recrystallized with ethanol absolute and dried in air. Table 1 show the physical properties of ligand and its complex

Table 1. Physical properties of (HPEDN) and (Ni-HPEDN).

| No. | Molecular formula | Color | Yield (%) | Am/Ohm | DMSO | Ethanol | DMF | Reaction Time |
|-----|------------------|-------|-----------|--------|-------|---------|-----|--------------|
| 1   | C₂₄H₁₉N₃O₂       | Reddish Brown | 178-180 | 72.51  | -     | -       | 4   |
| 2   | [Ni(C₂₄H₁₀N₃O₂)₂(H₂O)₂] | Light Brown | 179-181 | 71.93  | 15.7  | 9.1     | 10.5| 1h           |

3. RESULTS AND DISCUSSION:

3.1. UV-Visible Spectrophotometry
UV-Vis spectrum of the solid nickel complex solution dissolved in ethanol showed the appearance of absorption peaks. It is observed that there is a clear difference in the (λ max) values of the ligand (HPEDN) and the complex (Ni-HPEDN) formed with a red shift towards a higher wavelength, as shown in Fig. 1 and Table 2.
3.2. Study the Optimal Conditions for the Formation of the (Ni-HPEDN)

1. Study the effect of pH

A group of 10 ml vials were taken in which 1 ml of nickel ion solution was placed in a concentration \((1\times10^{-3} \text{M})\) and complete the volume with the buffer solution by changing the value of the range \(\text{pH}=4-12\) and the concentration \((1\times10^{-4} \text{M})\) was prepared. The ligand solution was prepared by taking 1ml from the ligand solution \((1\times10^{-3} \text{M})\) and complete the volume with absolute ethyl alcohol, so it became the final concentration \((1\times10^{-4} \text{M})\). And then mixing the 1mL of the Nickel ion with 1mL of the ligand and adjusting the acid function with the extent of the absorption record at the greatest wavelength of the nickel, we notice in \(\text{pH}=9\) an increase in the absorbance values and then it returns to decline due to the formation of unstable complexes or the precipitation of nickel ions \(^{17}\) as shown in Fig. 2.

![Fig2. Effect of pH value in complex (Ni-HPEDN).](image)

2. Study the effect of ligand concentration

Study the effect of the change of the ligand concentration on the formation of the complex it was found that the absorption values of the complex increased to a peak at addition \((2\times10^{-4} \text{M})\), the reason is due to pushing the reaction between nickel and ligand ion towards the formation of the complex \((\text{Ni-HPEDN})\) and giving the best color intensity. As the concentration of the reagent continues to increase, the absorption values begin to decrease and the cause may be due to the sufficiency of the coordinate field of ions with the ligand or, the reagent is not completely dissolved in solvent. as shown in Fig. 3, which shown that the effect of ligand (HPEDN) concentration.

![Fig 3. Effect of ligand concentration](image)

3. Effect of time in stability of (Ni-HPEDN).

The reaction of the ligand with Nickel was monitored during a period of time ranging from \(2-120\) minute) to know the stability of the formed complex and the period was established as \((15\) minute) a preferred time to complete the interaction between the Nickel solution with the ligand (HPEDN) \(^{18}\) as shown Fig. 4.

![Fig 4.](image)
4. Effect of Temperature in stability of (Ni-HPEDN).

It appears from Figure 5 that the Nickel complex absorption values reach their peak and give the best color intensity at temperatures ranging from (15-30)°C then the absorption values decrease with increasing temperatures. The reason is due to a decrease in the stability of the Nickel complex or the result of its dissociation at high temperatures.

5. Effect of the order of addition in the composition of the (Ni-HPEDN) complex

Three additions were adopted to know the extent of their effect on the absorption of nickel complex. The first addition was used to estimate the nickel element due to the competition of the negative ions of the acid and the base to bond with the metal, which leads to a decrease in the absorption values as Table 3.

| No. | Sequence of addition | Abs. (Ni-HPEDN) |
|-----|---------------------|-----------------|
| 1   | Ni to HPEDN to pH=9 | 0.2736          |
| 2   | HPEDN to Ni to pH=9 | 0.2403          |
| 3   | Ni to pH=9 to HPEDN | 0.1834          |

6. Study the construction of calibration curve

The figure 6 shows that the nickel complex with ligand is subject to the Law of Beer Lambert’s in the range (1.1-7.1 ppm), we note that the molar absorption coefficient is of a high value. The method is high sensitivity and can be used to estimate nickel within low concentrations.
The stoichiometry of the nickel complex was found using three methods:

**a. Method of proportionality**
To find the ratio of the metal ion to the ligand, use a fixed and known concentration of the nickel ion (5.8 ppm) with different concentrations of ligand (0.5×10⁻⁴ - 4×10⁻⁴ M), and the results of the study show that the ratio is (1:2) (M:L) as shown in Fig. 7.

**b. Method of Job**
Use different sizes of the metal ion ranged (0.5-4 mL) and different sizes of the ligand ranged (0.5-4 mL) to find the ratio of the metal ion to the ligand and found that the ratio is (1:2) (M:L) as shows Fig. 8.

**c. Study of slope analysis**
The analytical slope obtained from dividing the slope obtained from the study of the calibration curve by the slope obtained from the study of the effect of the reagent concentration and the output indicates the ratio of the metal ion to the ligand as Table 5.

**8. Study the stability calculation of the (Ni-HPEDN) complex**

\[ M^{2+} + nL \rightarrow ML_n \quad \text{(1)} \]

\[ \alpha c \quad \text{nac} \quad (1-\alpha)c \]
\[ \alpha = \frac{Am - As}{Am} \quad \text{---------------- (2)} \]

\[ K_{\text{inst}} = \frac{\text{[ligand]}}{c(1-\alpha)} \quad \text{------------------(3)} \]

\[ K_{\text{st}} = \frac{1}{K_{\text{inst}}} \quad \text{------------------------(4)} \]

Table 6. Absorption values (As) and (Am) as well as values of each (\( \alpha \)) and (Kst) and (Kinst) of the Nickel complex.

| Metal ion | As Value | Am Value | \( \alpha \) | \( K_{\text{inst}} \) mol L\(^{-1} \) | \( K_{\text{st}} \) mol L\(^{-1} \) | \( \text{Log } K_{\text{st}} \) |
|-----------|----------|----------|----------------|----------------|----------------|----------------|
| Ni(II)    | 0.2636   | 0.3144   | 0.1615         | 2.014\times10^{-10} | 4.965\times10^{9} | 9.695         |

From the results of molar ratios show the stability of the nickel complex by calculating the degree of dissociation and stability of constant.

9. Study the determination of thermodynamic functions to complexes formation

Thermodynamic function \( \Delta H^\circ \), \( \Delta G^\circ \) and \( \Delta S^\circ \) were calculated, results were illustrated in table 7 and Figure 9.

Table 7: The effect of temperature on thermodynamic function for Ni-HPEDN

| Metal ion complex | T(K) | \( 1/(T(K^{-1}) \) | Log \( K_{\text{st}} \) | \( -\Delta G^\circ \) KJ mol\(^{-1} \) | \( -\Delta H^\circ \) KJ mol\(^{-1} \) | \( -\Delta S^\circ \) KJ mol\(^{-1} \) K\(^{-1} \) |
|-------------------|------|-----------------|----------------|----------------|----------------|----------------|
| [Ni(C\(_2\)H\(_{24}\)N\(_3\)O\(_2\))\(_2\)(H\(_2\)O)\(_2\)]   | 288  | 0.00347         | 6.7753         | 37.35          | 125.53         | 306.18         |
|                   | 293  | 0.00342         | 6.3867         | 35.84          | 306.62         | 306.44         |
|                   | 298  | 0.00335         | 5.9972         | 34.21          | 307.27         | 307.27         |
|                   | 303  | 0.00330         | 5.5921         | 32.43          | 308.07         | 308.07         |
|                   | 308  | 0.00324         | 5.3024         | 31.26          | 308.07         | 308.07         |

\[ y=5.9002X-13.503 \]
\[ R^2=0.9995 \]

Fig.9. Relationship of \( \text{Log } K_{\text{st}} \) and \( 1/T \) of Ni(II) Complex

9. 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 in table 8.

Table 8: Values of standard deviation, percentage standard deviation.

| Comp. of ion | Conc. Of ion(M) | S.D   | R.S.D% |
|--------------|-----------------|-------|--------|
| Ni(II)       | 1.7             | 0.0015| 0.3921 |
|              | 4.1             | 0.0013| 0.3081 |
|              | 6.7             | 0.0011| 0.2521 |

10. Accuracy

The accuracy means the approximation of the practical value from the theoretical value so that the results are accuracy and precision. The accuracy of analytical method is calculated by using the
percentage relative error and the pre-processing ratio of the previously prepared complexes as in table 9. The results show that this method used to estimate the metals using ligand (HPEDN) is highly accuracy.

\[ \%E_{\text{real}} = \frac{d}{\mu} \times 100 \]

\( \mu = \) Analytical value
\( d = \) Analytical value - theoretical value

determining value - theoretical value

| Complex ion | Analytical value (mol.L\(^{-1}\)) | d | \(E_{\text{real}}\) | Re% |
|-------------|---------------------------------|---|-----------------|-----|
| Ni(II)      | 1.7                             | 0.07 | 4.1          | 104.1 |
|             | 4.1                             | 0.08 | 1.9          | 101.9 |
|             | 6.7                             | -0.1 | -1.4         | 98.6  |

Table 9. The relative percentage error and the pre-processing ratio of ligand complex.

Effect of foreign ions.

Definite amount of cations and anions were used as a foreign ions to study the possibility of the interferences with determination of Ni(II) ion, results explained in table (10).

| Interference ion | Complex Ni-HPEDN | Withou | Ni\(^{2+}\) | Co\(^{2+}\) | Cd\(^{2+}\) | Pd\(^{2+}\) | Mn\(^{2+}\) | Fe\(^{2+}\) | Zn\(^{2+}\) |
|------------------|-----------------|--------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
|                   | Cation         |        |             |             |             |             |             |             |             |             |
| 1.9 ppm Abs.      | 0.2865         | 0.2652 | 0.3074      | 0.1708      | 0.2927      | 0.2663      | 0.2916      | 0.3748      |             |
| d                | -              | -0.021 | 0.0209      | -0.115      | 0.0062      | -0.021      | 0.0051      | 0.0883      |             |
| E%               | -              | -7.43  | 7.29        | -40.38      | 2.23        | -7.05       | 1.78        | 32.88       |             |
| Re%              | -              | 92.56  | 107.29      | 59.61       | 102.23      | 92.94       | 101.78      | 132.88      |             |
| 4.4 ppm Abs.      | 0.2865         | 0.2694 | 0.2953      | 0.2016      | 0.3061      | 0.2773      | 0.2947      | 0.3602      |             |
| d                | -              | -0.017 | 0.0088      | -0.0849     | 0.0196      | -0.0092     | 0.0082      | 0.0737      |             |
| E%               | -              | -5.96  | 3.07        | 29.63       | 6.841       | -3.211      | 2.862       | 25.72       |             |
| Re%              | -              | 94.04  | 103.07      | 70.36       | 106.84      | 96.78       | 102.86      | 125.72      |             |
| 6.9 ppm Abs.      | 0.2865         | 0.2735 | 0.2807      | 0.2193      | 0.2974      | 0.2952      | 0.2818      | 0.3309      |             |
| d                | -              | -0.013 | -0.0058     | -0.0672     | 0.0109      | 0.0087      | -0.0047     | 0.0444      |             |
| E%               | -              | 4.53   | 2.24        | -23.45      | 3.81        | 3.036       | -1.64       | 15.49       |             |
| Re%              | -              | 95.46  | 97.97       | 76.54       | 103.81      | 103.03      | 98.35       | 115.49      |             |
| The determination metal ion | Interference ion | 2.34ppm |             |             |             |             |             |             | 5.27ppm     |

Table 10. Effect of foreign ion on the determination of Ni(II) ion.
A group of positive and negative ions was chosen to know the extent of the interaction of these ions with the chosen ions within the study. We note an increase or decrease in the absorption values with respect to the positive ions due to the competition between the positive ions and the ions chosen within the study. The decrease in absorption in the presence of negative ions is due to its behavior as masking agents\(^{22,23}\) and was chosen Ascorbic acid as a masking agent.

12. Study of FT-IR spectra for ligand and complex

Figures 10 and Table 11 explained the FT-IR study and the absorption frequencies for Ligand and the complex Ni-HPEDN\(^{24}\).

| metal ion | Abs. | d | E% | Re% | Abs. | d | E% | Re% |
|-----------|------|---|----|-----|------|---|----|-----|
| Ni(II)    | 0.2773 | - | - | - | 0.2773 | - | - | - |
| CO\(_3\)\(^{-2}\) | 0.2581 | -0.11 | -4.78 | 95.22 | 0.2603 | -0.32 | -6.13 | 93.86 |
| SO\(_4\)\(^{-2}\) | 0.2297 | -0.14 | -6.31 | 94.89 | 0.2584 | -0.35 | -6.81 | 93.18 |
| C\(_5\)O\(_4\)\(^{-2}\) | 0.2504 | -0.138 | -5.70 | 94.35 | 0.2486 | -0.22 | -4.34 | 95.66 |
| CH\(_3\)COO\(^{-}\) | 0.2485 | -0.132 | -5.68 | 94.32 | 0.2557 | -0.31 | -5.79 | 94.21 |
| NO\(_2\)\(^{-}\) | 0.2586 | -0.113 | -4.84 | 95.16 | 0.2518 | -0.27 | -5.19 | 90.81 |
| SCN\(^{-}\) | 0.2692 | -0.068 | -2.92 | 97.08 | 0.2675 | -0.18 | -3.53 | 96.46 |
| The determined metal ion | Masking agent (0.01M) | Abs. | d | %E\(_{\text{real}}\) | %Re |
| Ni(II) | Ascorbic acid | 0.2778 | 0.0104 | 0.18 | 100.18 |
| | Potassium Thiocyanate | 0.1452 | -2.7625 | -47.63 | 52.37 |
| | EDTA-Na\(_2\) | 0.0784 | -4.1597 | -71.72 | 28.28 |
| | Thiourea | 0.2134 | -1.3363 | -23.04 | 76.96 |
| | Potassium chloride | 0.1893 | -1.8403 | -31.73 | 68.27 |
| | Tartaric acid | 0.1508 | -2.6453 | -45.61 | 54.39 |
| | Salicylic acid | 0.0865 | -3.9908 | -68.81 | 31.93 |
Table 11: Typical FT-IR absorption frequencies for HPEDN and Ni-HPEDN

| Compound      | νO-H (cm⁻¹) | νC=N (cm⁻¹) | νN=N (cm⁻¹) | νM-O (cm⁻¹) | νM-N (cm⁻¹) |
|---------------|-------------|-------------|-------------|-------------|-------------|
| Ligand HPEDN  | 3454 (s)    | 1666 (m)    | 1413 (s)    | ---         | ---         |
| Ni(II) complex| 3372 (m)    | 1673 (m)    | 1461 (s)    | 577 (w)     | 469 (m)     |

13. ¹H NMR and ¹³C NMR Spectra of Ligand (HPEDN)

The ¹H NMR spectra of ligand shows the chemical displacements of aromatic and aliphatic protons. The single band at (9.8491 ppm) belong to one proton of hydroxyl group (OH), the three single bands at (2.5895, 2.6189 and 2.6471 ppm) due to the protons of methyl group (CH₃), the multiple bands at (6.7574 ppm to 8.4526 ppm) belongs to nine protons of aromatic rings, while the band at (2.500 ppm) related to (DMSO d₆). Proton NMR spectra of the ligand are shown in Fig. 11. ¹³C NMR spectra of the ligand, appearance of single band at (39.520 ppm) belong to one carbon of methyl group (CH₃), the single band at (155.3790 ppm) belong to one carbon of azomethine (–C=N–), the multiple bands at (108.6402 to 145.3493 ppm) belongs to 22 of carbon aromatic rings, as shown in fig. 12.

Fig. 11. ¹H NMR spectra of ligand (HPEDN)
14. The suggested figure for the Ni-HPEDN complex

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 and molar electrical conductivity.

![Complex Structure](image)

**Fig. 13. suggested figure for the Ni-HPEDN complex**

[Di aqua bis(1-((4-(1-(2-hydroxyphenylimino)ethyl)phenyl)diazenyl)naphthalene-2-ol)Nickel ion(II)]

15. 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 those where obtained by flame atomic absorption method as in table 12 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 12. The percentages of Nickel determine by ligand in Brass Nickel alloy using atomic absorption method and spectrophotometric method

| Sample   | Content% | By Spectrophotometric method % | E_{rel}% | Re% | By Flame atomic absorption % | E_{rel}% | Re% |
|----------|----------|--------------------------------|----------|-----|-------------------------------|----------|-----|
| Alloy Nickel | 30      | 31.5                           | 1.5      | 101.5 | 28.7                         | -1.3     | 98.7 |
4. Conclusion

1- The way in which this study is fast, economical, and good, and to follow simplified conditions in preparing the ligand(HPEDN) through traditional nitrogen with some modifications

2- The possibility of using ligand(HPEDN) in estimating micrograms of nickel, as it has a colored complex with peak absorption peaks.

3- The proposed method is highly sensitive due to the persistence of the colored compound for more than 48 hours formed by metal ion reactions with a newly synthesized reagent, low reagent consumption, elimination of analytical error, reduced interference and statistical analysis which made the method to be more sensitive and selective.

5-Acknowledgments

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