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Synthesis, Characterization, Antibacterial, and Extraction Studies of Dioxouranium (Uo^{22+}) Ofn, N'-[(4, 4'-Biphenyl Bis (Methylidene))] Di Nicotinic Hydrazine

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Abstract- New Hydrazonic Schiff base was synthesized by reaction Nicotinic hydrazide with Biphenyl-4,4'-Dicarbal-dehyde have been characterized by elemental analyses IR, mass and $^1\text{H-NMR}$ spectral data. Hydrazoic Schiff base have been studied by liquid-liquid extraction towards the metal ion $\text{Uo}(\text{II})$ from aqueous phase to organic phase. behave as a good extractant ligand for uranyl ions $\text{Uo}(\text{II})$ from aqueous solution, and the study of conditions of extraction shows that the optimum pH values for extraction was (pH= 5.2), so the suitable concentration of $\text{Uo}(\text{II})$ ions in aqueous solution which is giving highest distribution ratio (D) was (1x10-4M), in addition the optimum shaking time to reach the equilibria was (60 min.), as well as the stoichiometric study by using three methods shows the structure of $\text{Uo}(\text{II})$ ions complex with ligand was 1 : 1 (metal : ligand), in addition the study of organic solvent effect appear there is no straight line relation between the distribution ratio for extraction of $\text{Uo}(\text{II})$ ions and dielectric constant for organic solvents but this study demonstrate there is an effect for the structure of organic solvent used, thermodynamic study shows the complexation reaction an endothermic. The new Schiff Bases was studied for antibacterial activities against (Bacillus subtilis and Staphylococcus aureus) are Gram positive and (Salmonella typhi and Escherichia coli) are Gram negative. The compound ligand was exhibited a variable activity of inhibition on the growth of the bacteria.

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SYNTHESIS, CHARACTERIZATION, ANTIBACTERIAL, AND EXTRACTION STUDIES OF DIOXOURANIUM Uo^{22+} OFN, N'-[4,4'-BIPHENYL BIS (METHYLDENE)] DI NICOTINIC HYDRAZINE

Strictly as per the compliance and regulations of :



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I. INTRODUCTION

Schiff bases are widely studied and used in the fields of organic synthesis and metal ion complexation [1, 2] for a number of reasons: their physiological and pharmacological activities [3-5] their use in ion selective electrodes [6-11] in the determination of heavy metals ions in environmental samples [12] and in the extraction of metals ions [13,14] and their many catalytic applications (e.g. for epoxidation of olefins, alkene cyclopropanation [15,16] trimethylsilyl-lycyanation of ketones [17] asymmetric oxidation of methyl phenyl sulfide enantioselective-epoxidation of silylenol [18] and ring-opening Polymeri-

zation of lactide [19]. Hydrazones are special group of compounds in the Schiff bases family. They are characterized by the presence of (C=N-N=C). the presence of two inter-linked nitrogen atoms was separated from imines, oximes, etc. hydrazone Schiff bases of acyl, aryl and heteroacroyl compounds have additional donor sites like C=O. The additional donor sites make them more flexible and versatile. This versatility has made hydrazones good polydentate chelating agents that can form a variety of complexes with various transition and inner transition metals and have attracted the attention of many researchers. Various hydrazones are obtained depending on the experimental conditions; which have application as biologically active compounds [20] and as analytical reagents [21]. As biologically active compounds, hydrazones find applications in the treatment of diseases such as anti-tumor [22] tuberculosis [22] leprosy and mental disorder [23]. Tuberculostatic activity is attributed to the formation of stable chelates with transition metals present in the cell. Thus many vital enzymatic reactions catalyzed by these transition metals cannot take place in the presence of hydrazones [24,25]. Hydrazones also act as herbicides, insecticides, nematocides, rodenticides and plant growth regulators.

Uranium exists in various concentrations in all parts of the environment, in certain types of rockse.g. Some types of granites, and various mineral deposits. There are also some artificial contributions of uranium to the environment e.g. from mineral exploration, emissions from the nuclear industry, the combustion of coal and other fuels, and the use of phosphate fertilizers that contain uranium.[26].1 Natural uranium is one of the elements that can be transferred to the plants or accumulated under the influence of microbial processes including oxidoreduction, production of complexing compounds, and biosorption. In this way, plants and fungi are directly involved in the absorption, accumulation of major and trace elements including radionuclides. In natural and also in disturbed environments, accumulation of uranium has been observed in plants, mushrooms and fungal mycelium. [27] The contents have been observed in mycelia by

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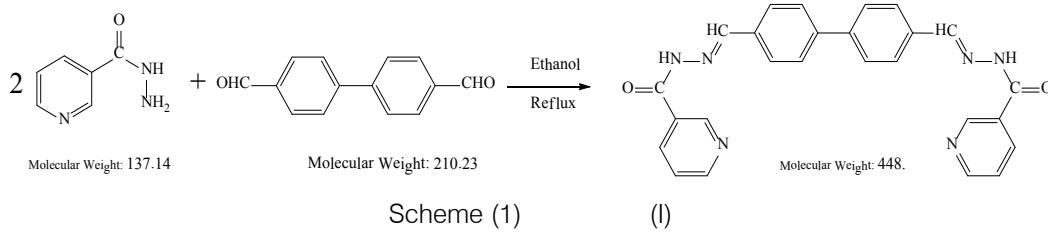


coupling the microscopic and spectrometric methods. The complex compounds formed between uranyl ions and different complexing agents are of great interest due to their possibilities to be applied in extraction of metallic ions from ores, extraction of uranium from solutions of low concentration treatment of the radioactive waters and in environmental chemistry. It is known the fact that the uranyl ions have a big complexing capacity in the presence of different organic and inorganic compounds. Behavior of uranium in solution is very interesting. Usually, uranium is found in form of uranyl ions very stable in both solution and solid state. From the structural point of view, the uranyl ions has in its center the uranium atom with the oxidation state (VI), and the two atoms of oxygen form two double bonds $\text{O}=\text{U}=\text{O}$ with an angle of 180° between them (linear, stable structure), fact demonstrated by Duval [28] and Frankuchen [29] (in his studies over the uranyl and sodium acetate). The ligands coordinate equatorially to the uranium atom and in a perpendicular plane on the plane of the $\text{O}=\text{U}=\text{O}$ bonds, coordination numbers being between 4 and 7 depending on the chemical surrounding.[30-32] The Schiff bases are chelating agents used to get different complexes with transitional metals. They form complexes compounds with different metallic ions in the 1/1 ratio form 3 very stable chelate cycles. Taking into account the high capacity of the Schiff bases to coordinate metallic ions, these are the intensively utilized as complexing agents to extract some. Metals from various media. The uranyl ions form complex compounds with numerous Schiff bases showing a strong affinity for ligands with oxygen and nitrogen-bearing ligands, a series of such complexes being already studied. [33-36].

II. EXPERIMENTAL

a) Reagents and Apparatus

All the chemicals used were of AnalaR grade and procured from Sigma-Aldrich and Fluka. Metal salts were purchased from E. Merck and were used as received. Distilled water was used in the extraction experiments. The solvents were saturated with each other before use in order to prevent volume changes of the phases during extraction. The C, H, and N were



b) Procedure of Extraction

The extraction properties of the Schiff base ligand (I) was investigated under liquid-liquid phase and neutral condition using transition metal picrates UO_{22+} (II)

analyzed on a Carlo-Erba 1106 elemental analyzer. The IR spectrum was recorded on Jusco 300 instrument in KBr pellets. ^1H NMR spectra of ligands in CDCl_3 solution were recorded on a Bruker DT-400 MHz spectrometer, and chemical shifts are indicated in ppm relative to tetra methyl silane. Mass spectra were recorded using a KRATOS MS50TC spectrometer.

AA 929 Unicam Spectrometer was used for FAAS measurements with an air-acetylene flame. A pH meter (Metrohm-691pH Meter) was also used. All extraction experiments were performed by using a mechanical flask agitator in 50 cm^3 stoppered glass flasks, M.P Apparatus Digital (32-300 $^\circ\text{C}$). The thermo gravimetric analysis (TGA) was carried out in a dynamic nitrogen atmosphere ($20\text{ mL}\cdot\text{min}^{-1}$), with a heating rate of $10\text{ }^\circ\text{C min}^{-1}$ using Shimadzu TGA-50H thermal analyzers.

c) Synthesis of N,N' -[(4,4'-biphenyl bis (methylidene))] di nicotinic hydrazide

The hot ethanolic solution (30mL), of 4, 4'-biphenyl aldehyde (1 mmol, 0.210 g), and a hot ethanolic solution (30mL), of nicotinic hydrazide (2mmol, 0.274 g), were mixed slowly with constant stirring. Containing a few drops of concentrated HCl. The reaction mixture was heated to reflux for 4hrs, where white precipitate was formed scheme (1). The solid obtained was collected and recrystallized from mixture DMF, EtOH (9: 1) as white crystals. A yellow colored precipitate was washed with water, ethanol, CHCl_3 and diethyl ether, respectively, and dried under vacuum over P_2O_5 .

White; Yield: (80 %), m. p= $285\text{ }^\circ\text{C}$, Empirical formula ($\text{C}_{26}\text{H}_{20}\text{N}_6\text{O}_2$), M. Wt: (448 g)

Mass spectrum (m/z): 448. (Figure 1).

UV-Vis: $\lambda_{\text{max}} = 360\text{ nm}$. (Figure 4).

IR (KBr disk): 3248.13 cm^{-1} (N-H), 3062.96 cm^{-1} (C-H), aromatic, 1651.07 cm^{-1} (C=O), 1600.92 cm^{-1} (C=N). (Figure 2).

$^1\text{H-NMR}$ (CDCl_3 -400MHz) $\delta = 12.0957$ (s, 2H, CO-NH-), 9.093 (s, 2H, $\text{CH}=\text{N}$), 7.591 - 8.979 (m, 16H, Ar), 2.508 -2.677(DMSO, H_2O). (Figure 3).

as substrates and measuring by UV-vis measurements the amounts of metal picrate in the aqueous phase before and after treatment with the compounds. About 10 mm of $2 \times 10^{-5}\text{ M}$ aqueous picrate solution and 10

mm of 1×10^{-3} M solution of ligand in CHCl_3 , CH_2Cl_2 , C_6H_6 were vigorously agitated in a stoppered plastic tube with a mechanical shaker for 2 min, then magnetically stirred in a thermo stated water bath at 25 °C for 1 h, and finally left standing for an additional 30 min. The concentration of the picrate ion remaining in the aqueous phase was then determined spectrophotometrically. Blank experiments showed that no picrate extraction occurred in the absence of ligand. Transition metal picrate were prepared by successive addition of 1×10^{-2} M metal nitrate solution to 2×10^{-5} M aqueous picric acid solution and shaken at 25 °C for 1 h. This metal picrate $\text{UO}_2(\text{II})$ was measured by UV-vis using maximum wavelength 352 nm. For each combination of host and metal picrate, the picrate extraction was conducted on three different samples and the average value of percent picrate extracted, with a standard deviation, was calculated. In the absence of host, a blank experiment, no metal ion picrate extraction was detected. The extractability was calculated by using the equation below:

$$\text{Extractability (\%)} = \left(\frac{A_0 - A}{A_0} \right) \times 100$$

Where A_0 is the absorbance in the absence of ligand. A denotes the absorbance in the aqueous phase after extraction. [37].

d) Biological Activity

The prepared compounds were tested for their antimicrobial activity against four species of bacteria (*Bacillus subtilis*, *Escherichia coli*, *Staphylococcus*

aureus, *Salmonella typhi*) using filter paper disc method [38]. The screened compounds were dissolved individually in DMSO (dimethyl sulfoxide) in order to make up a solution of 50, 100, and 200 µg/ml concentration for each of these compounds. Filter paper discs (Whitman No.1 filter paper, 5mm diameter) were saturated with the solution of these compounds. The discs were placed on the surface of solidified Nutrient agar dishes seeded by the tested bacteria. The diameters of inhibition zones (mm) were measured at the end of an incubation period, which was 24 h at 37°C for bacteria. Discs saturated with DMSO are used as solvent control. Ciprofloxacin 100 µg/ml was used as reference substance for bacteria. [38].

III. RESULT AND DISCUSSION

a) Synthesis

The prepared organic compound (I) was synthesized by the condensation of 4,4'-biphenyl aldehyde with nicotinic hydrazide in the molar ratio 1:2 in absolute ethanol. The reaction proceeded smoothly, producing the corresponding Schiff bases in good yield. The ligand is soluble in common organic solvent but insoluble in water. The structures of the ligand was elucidated by elemental analyses, MS, FTIR, electronic absorption, and ^1H - NMR spectra, which help in elucidating their empirical formula (Table 3.1).

b) Elemental analyses of Schiff base

The results of elemental analyses Schiff base, as shown in Table (3.2), are in good agreement with those required by the proposed formulae

Table 3.1 : Color, molecular weight and melting point of Schiff base (I)

| Schiff base | Color | M. Wt | Melting point °C | λ_{max} nm | Yield % | Crystallization Solvent |
|-------------|-------|-------|------------------|---------------------------|---------|-------------------------|
| I | White | 448 | 285 | 360 | 80 | DMF, EtOH |

Table 3.2 : Elemental analysis data of Schiff base (I)

| Schiff base | Elemental analysis Calculated (Found %) | | | | |
|-------------|---|-------------|---------------|-------|-------------|
| | C | H | N | S | O |
| I | 69.34 (69.63) | 4.56 (4.49) | 19.11 (18.74) | ----- | 6.99 (7.14) |

c) IR Spectra

The IR Spectral data are shown in table (3.3) of the prepared Schiff base. The band at 1600.92 cm^{-1} is attributed to imine group (-HC=N-) for (I) (Fig 2). The band in the spectra at $1579.8 - 1420.4\text{ cm}^{-1}$ is due to (C=C) of aromatic rings. while the band at $2858.5 - 2933.5\text{ cm}^{-1}$ are attributed to (C-H alkanes) for (I). Also,

the band at 3062.96 cm^{-1} are attributed to (C-H ar). While the band at 3248.13 cm^{-1} are attributed to (N-H), and the band at 1651.07 cm^{-1} are attributed to (C=O). However, in the IR spectra of Schiff bases this band (C=O) disappears and a new vibration band for azo methane (-HC=N-) is observed at 1600.92 cm^{-1} , indicating that complete condensation takes place. [39-40].

Table 3.3 : IR spectral data (cm^{-1}) of Schiff base (I)

| Schiff base | $\nu(\text{C}=\text{C})$ | $\nu(\text{HC}=\text{N})$ | $\nu(\text{C}=\text{O})$ | $\nu(\text{C}-\text{H})$ aliph | $\nu(\text{C}-\text{H})$ arom | $\nu(\text{N}-\text{H})$ |
|-------------|--------------------------|---------------------------|--------------------------|--------------------------------|-------------------------------|--------------------------|
| I | 1579.8 - 1420.4 | 1600.92 | 1651.07 | 2858.5-2933.5 | 3062.96 | 3248.13 |

d) $^1\text{H-NMR}$ Spectra of Schiff base (I)

The data of $^1\text{H-NMR}$ Spectra of prepared Schiff base is shown in table (3.4). The $^1\text{HNMR}$ spectra of (I) ligand in d6-DMSO (Fig 3) shows a singlet signal at 9.093 ppm assigned to the protons ($\text{HC}=\text{N}-$) groups of the ligand, while the singlet signal at 12.0957 ppm assigned to the protons (CO-NH) group of the ligand (I). The multiple signals 8.979 - 7.591 ppm are due to the

aromatic protons. The other obtained values for $^1\text{H-NMR}$ chemical shifts of the compound is given in the experimental section.

The $^1\text{HNMR}$ spectral data of the new compound. These data are in good agreement with those previously reported for similar compound. These results strongly suggest that the proposed compound has been formed. [39-40].

Table 3.4 : $^1\text{H-NMR}$ Spectra of Schiff bases (I)

| Schiff base | Chemical Shifts δ ppm | | |
|-------------|------------------------------|----------------------|------------------|
| | C-H aromatic | $\text{CH}=\text{N}$ | $-\text{CO-NH}-$ |
| I | 8.979 - 7.591 (m,16H) | 9.093 (s,2H) | 12.0957 (s,2H) |

IV. RESULTS AND DISCUSSION

The complexation reaction between the ligand (I) and Uo(II) ion to produce the ion pair complex have to extracted to the organic phase, this complexation reaction of necessity behave among many optimum condition to reach equilibria and giving stable ionpair complex.

a) Effect of pH

Extracted of 10 ml of Uo(II) ion in aqueous phase at different pH (3 –12) by using (10 ml) of (1×10^{-4} M) ligands dissolved in chloroform, after that shaking the two layers for suitable time and separate this two layers and determined the remainder of Uo (II) ion in aqueous phase and Uo (II) ion transfer to the organic

phase as complex by stripping method or by difference method according to general procedure, afterward determine the distribution ratio (D) and percentage of extraction ($E_{\text{ex}}\%$) at each pH, the results in Table (3.5), and Fig. (5) Shows the optimum of pH extraction was ($\text{pH}_{\text{ex}} = 5.2$). At pH less than optimum value effect to protonated ligand to occupy the pair of electron and then can't coordinate strongly with Uo (II) ion and give less stable ion pair complex and minimize the distribution ratio (D) and percentage of extraction ($E_{\text{ex}}\%$), also at pH values more than optimum value effect to decrease distribution ratio (D) and percentage of extraction ($E_{\text{ex}}\%$) by reason of formation ion pair complex contain anion (OH^-) this complex more soluble in aqueous phase and less extracted to organic phase and sovereignty the dissociation equilibria.

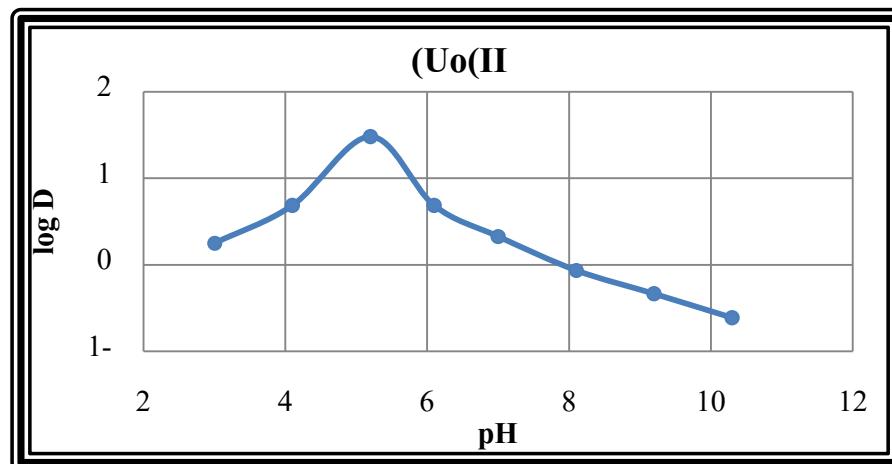
Figure 5 : pH effect on the extraction of Uo (II) ion

Table 3.5 : Effect of pH on the extraction of Uo (II) ion

| pH | UO(II) | | |
|------|--------|-------|------|
| | log D | D | E% |
| 3 | 0.252 | 1.786 | 64.1 |
| 4.1 | 0.686 | 4.848 | 82.9 |
| 5.2 | 1.481 | 30.25 | 96.8 |
| 6.1 | 0.686 | 4.848 | 78.9 |
| 7 | 0.329 | 2.135 | 62.1 |
| 8.1 | -0.063 | 0.866 | 46.4 |
| 9.2 | -0.335 | 0.462 | 31.6 |
| 10.3 | -0.613 | 0.244 | 19.6 |
| 11.1 | 0 | 0 | 0 |

b) *Effect of Shaking Time*

Extraction of Uo (II) ion from (10 ml) aqueous phase at (pH =5.2) by using (10 ml) of organic phase of (1×10^{-4} M) ligand dissolved in chloroform, after shaking the two phases at different times, separate the two phases and determine the concentration of Uo (II) ion remainder in aqueous phase and the concentration of Uo(II) ion transfer to organic phase as ion pair complex by previous spectrophotometric method and then determine distribution ratio (D) and percentage of

extraction ($E_{ex}\%$). The results in Table (3.6) and Fig. (6) demonstrate that the optimum shaking time was (60 min.) to reach the equilibria of complexation reaction and giving higher distribution ratio (D), the shaking time less than optimum shaking time not allow to reach for equilibria and giving low distribution ratio (D), but at shaking time more than optimum time effect to favor the dissociation step on the complexation and minimize the distribution ratio (D) depend on the kinetic energy of reaction.

Table 3.6 : Effect of shaking time on extraction of Uo (II)ion

| Shaking Time | UO ⁺² | | |
|--------------|------------------|-------|-------|
| | Log D | D | E% |
| 10 | 0.037 | 1.088 | 52.11 |
| 20 | 0.197 | 1.573 | 61.14 |
| 30 | 0.417 | 2.613 | 72.32 |
| 40 | 0.712 | 5.15 | 84.64 |
| 50 | 0.878 | 7.547 | 88.29 |
| 60 | 1.318 | 20.79 | 95.41 |
| 70 | 0.999 | 9.99 | 90.90 |
| 80 | 0.777 | 5.99 | 85.69 |
| 90 | 0.535 | 3.429 | 77.42 |
| 100 | 0.343 | 2.205 | 68.92 |
| 110 | 0.085 | 1.217 | 54.89 |
| 120 | 0.029 | 1.070 | 51.69 |

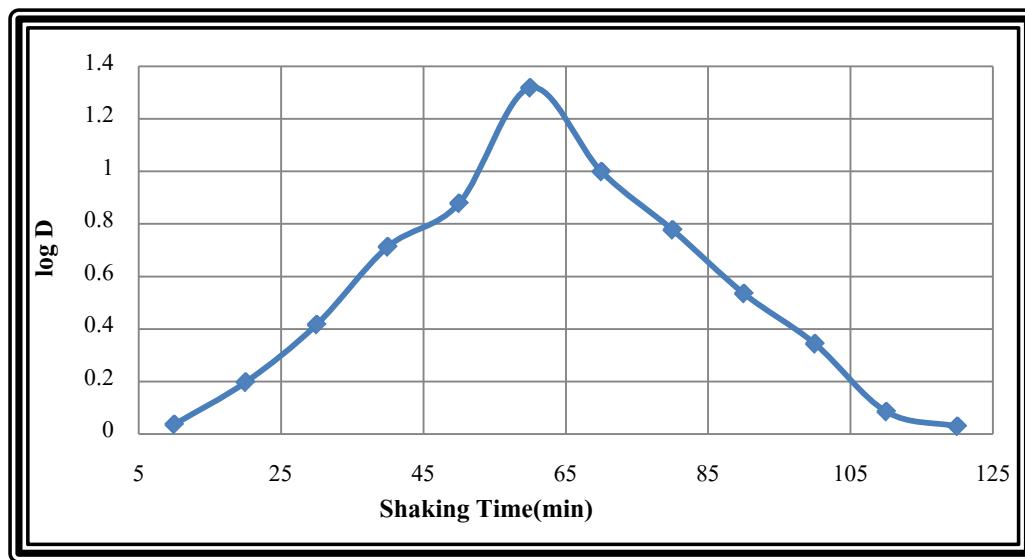


Figure 6 : Effect of shaking time on the extraction of Uo (II) ion

c) *Stoichiometry by Slope Analysis Method*

To knowing the more probable structure of Uo (II) ion pair complex produced, extracted (1×10^{-4} M) Uo (II) ion from (10 ml) aqueous phase at ($\text{pH} = 5.2$) by using (10 ml) of ligand solution dissolved in chloroform at different concentration (2×10^{-5} M – 3×10^{-4} M), after shaking the two layer for (60 min.), separate these layers and determine the remainder quantity of Uo (II) ion in

aqueous phase and transfer quantity of Uo (II) ion to organic phase as complex, after that determine distribution ratio (D) and percentage of extraction ($E_{\text{ex}}\%$) by previous method (spectrophotometric method). Afterward plot $\log D$ against $\log [L]$. The results in Table (3.7) and Fig. (7) shows from the slope of straight line that the more probable structure of ion pair complex extracted was 1: 1(metal : ligand).

Table 3.7 : Slope analysis method for extraction of Uo (II) ion

| Log [L] | Uo(II) | | | slope |
|---------|--------|-------|-------|-------|
| | D | E% | | |
| -4.698 | 0.639 | 38.98 | 1.352 | 1.352 |
| -4.522 | 1.387 | 58.11 | | |
| -4.397 | 1.932 | 65.89 | | |
| -4.301 | 2.911 | 74.43 | | |
| -4.097 | 6.109 | 85.93 | | |
| -4 | 18.58 | 94.89 | | |
| -3.698 | 18.03 | 94.75 | | |
| -3.522 | 17.87 | 94.70 | | |

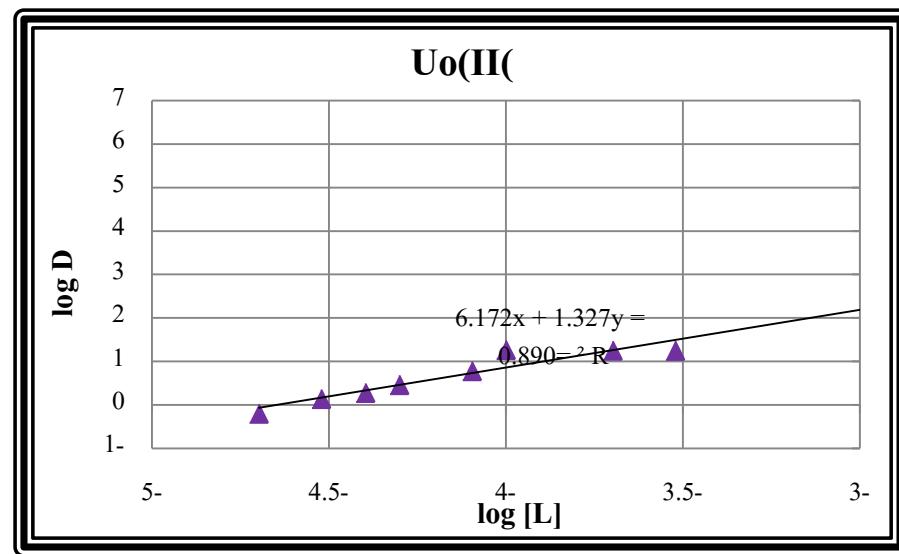


Figure 7 : Slope analysis method for extraction of Uo (II) ion

d) *Organic Solvent Effect*

Solvent extraction method take care of organic extraction method; according to this concept extracted (1×10^{-4} M) Uo (II) ion in (10 ml) aqueous phase at (pH= 5.2) by (10 ml) organic phase contain ligand dissolved in different organic solvents at concentration of (1×10^{-4} M), after shaking for (60 min) separate the two layers and determine the remainder Uo (II) ion in aqueous phase and Uo (II) ion transfer to organic phase as Uo (II) complex, according to previous method, afterward calculate the distribution ratio (D) and percentage of

extraction ($E_{\text{ex}}\%$). The results in Table (3.8) shows there is not any linear relation between distribution ratio (D) and dielectric for organic solvents used in extraction method of Uo (II) ion, Fig(8) as well as this results reflect the effect of organic solvent structure on the extraction method and without any effect for polarity of organic solvent, and reflect the effect on ion pair complex extracted to be contact ion pair (tight ion pair) or solvophobic solvent used to separate ion pair (loose ion pair).

Table 3.8 : Organic solvents effect on the extraction of Uo (II) ion

| Solvents | ϵ | Uo(II) | |
|------------------|------------|--------|------|
| | | D | E % |
| Dichloro methane | 9.08 | 5.211 | 83.9 |
| Chloroform | 5.708 | 22.81 | 95.8 |
| Benzene | 2.804 | 1.770 | 63.9 |

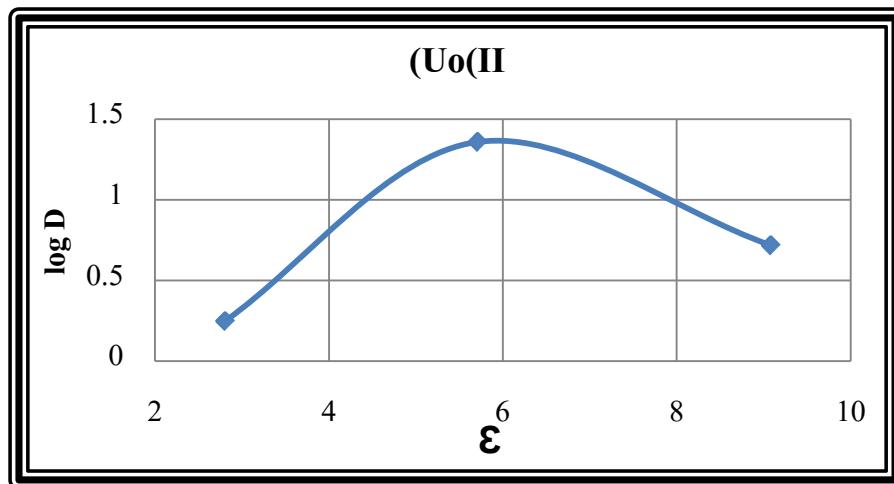


Figure 8 : Effect of solvents organic on the extraction of Uo (II) ion

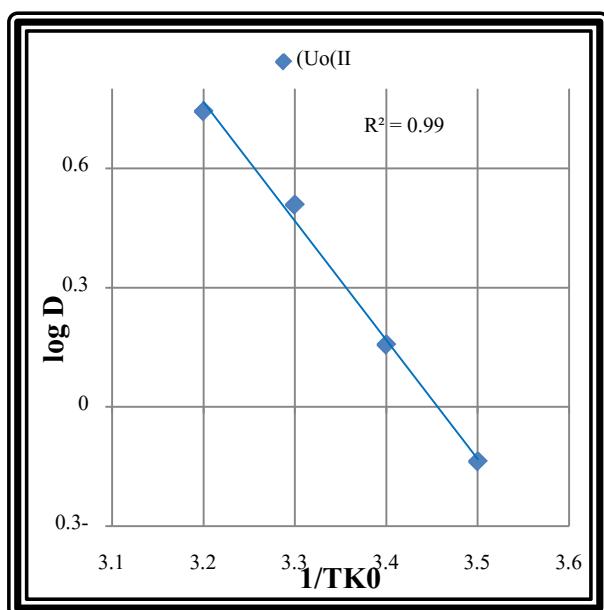
e) Effect of Temperature

Extraction of $\text{Uo}(\text{II})$ ion in (10 ml) aqueous phase at ($\text{pH} = 5.2$) by using (10 ml) of (1×10^{-4} M) ligand dissolved in chloroform at different temperature (10 – 40°C), after shaking the two layers for optimum time and separate the two layers, determine distribution

ratio (D) according to the general procedure. The results in Table (3.9) and Fig. (9) illustrate the complexation reaction between ligand and $\text{Uo}(\text{II})$ ion was Endothermic reaction, that is mean the coordination association between ligand and $\text{Uo}(\text{II})$ ion increase with temperature increasing.

Table 3.9 : Temperature effect on the extraction of $\text{Uo}(\text{II})$ ion

| T.C° | T.K° | 1/Tx10 ⁻³ K° | Uo(II) | | |
|------|------|-------------------------|--------|------|-------|
| | | | Log D | D | E % |
| 10 | 283 | 283 | 0.389 | 2.45 | 71.02 |
| 20 | 293 | 293 | 0.905 | 8.04 | 88.93 |
| 30 | 303 | 303 | 1.391 | 24.6 | 96.09 |
| 40 | 313 | 313 | 2.093 | 124 | 99.2 |

Figure 9 : Temperature effect on the extraction of $\text{Uo}(\text{II})$ ion

After calculated the thermodynamic data for extraction method to $\text{Uo}(\text{II})$ ion by using the below relation:

Using the below relation:

$$K_{\text{ex}} = \frac{D}{[\text{M}^{\text{n}+}]_{\text{aq}} [\text{L}]_{\text{org}}} \quad (4.1)$$

Table 3.10 : Thermodynamic parameters of Ligand from extraction of $\text{Uo}(\text{II})$ ion

| Cation | K_{ext} | ΔS_{ext} $\text{J K}^{-1} \text{mol}^{-1}$ | ΔG_{ext} kJ mol^{-1} | ΔH_{ext} kJ mol^{-1} |
|------------------------|--------------------|--|---|---|
| $\text{Uo}(\text{II})$ | 15.5×10^9 | 542.56 | -61.06 | 108.76 |

V. BIOLOGICAL ACTIVITY

During the last two or three decades, attention has been increasingly paid to the synthesis of Schiff bases which exhibits various biological activities including antibacterial, fungicidal, tuberculostatic and plant growth regulative properties [41]. It was judicious

to investigate the synthesis of various new types of Schiff base and studied their antibacterial activity against four strains of bacteria (*Bacillus subtilis*, *Escherichia coli*, *Staphylococcus aureus*, *Salmonella typhi*). The concentrations used for the screened compounds are 50, 100, and 200 $\mu\text{g}/\text{ml}$. Ciprofloxacin was used as reference standard while DMSO as control

and inhibition zones are measured in mm. The new compounds were tested against one strain each of a gram positive and two gram negative. The test results presence in Table (3.11), a new compound was active against tested and another compounds are no active.

All compounds are no active where used 50, 100 $\mu\text{g}/\text{ml}$ but active in the concentrations 200 $\mu\text{g}/\text{ml}$ see table (3.4).

Table 3.11 : Effect of new azo Schiff bases on the growth of tested bacteria (conc.200 $\mu\text{g}/\text{ml}$)

| Shiff base | Bacteria | | | |
|---------------|--------------------|------------------|---------------|-----------------|
| | Gram negative | | Gram positive | |
| | <i>B. subtilis</i> | <i>S. aureus</i> | <i>E.coli</i> | <i>S. typhi</i> |
| I | 20 mm | 18 mm | 17 mm | 19 mm |
| Control | 00 mm | 00 mm | 00 mm | 00 mm |
| Ciprofloxacin | 20 mm | 20 mm | 20 mm | 20 mm |

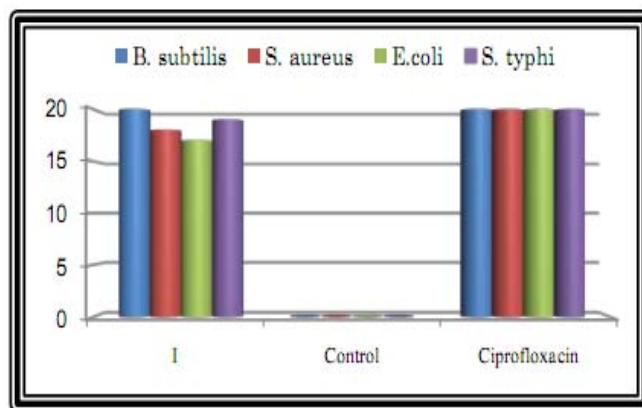


Figure 9 : Antibacterial activity (Gram positive, negative) of synthesized compounds

VI. CONCLUSION

1. The (I) compound is new and was prepared for the first time.
2. The new compound was identified by ^1H NMR, IR, LC-MS spectral methods.
3. The compound showed a high draw ratio of copper ion at pH = 5.2 and the best organic stores is chloroform and that there is no linear relationship between the polarity of the shops and the distribution ratio (D).
4. That shaking a time more than a time best, which represents the time required to complete the process complexity and give a complex stable lead to lower values ratios distribution D and this would lead to the possibility of a reflection of interaction and increase the speed of interaction back and eventually dismantle the complex and perhaps a few Side reactions that may lead to the occurrence of the phenomenon of the three layers (Three layers) for the analysis of solutions and this is what requires us to comply with a time of shaking out the best.
5. That the rate of ion correlation between copper and composite studied by Molar (cation: connected (1:1) between the ligand and the cation.
6. Calculated some parameters associated with extraction, such as constant extraction as well as parameters thermodynamic namely, enthalpy change (ΔH^*), entropy change (ΔS^*), and Gibbs free energy change (ΔG^*).
7. proven data study the effect of different temperatures on the recovery operations that the formation of complexes between ligand and cation studied endothermic (Endothermic) , as indicated negative values of energy free to spontaneous reactions, as also indicated positive values of entropy system that many random.
8. The prepared compound has been biologically screened i.e. studying their effects against two gram-positive, two gram-negative bacteria. The results show that their activities were found to vary from moderate to very strong.

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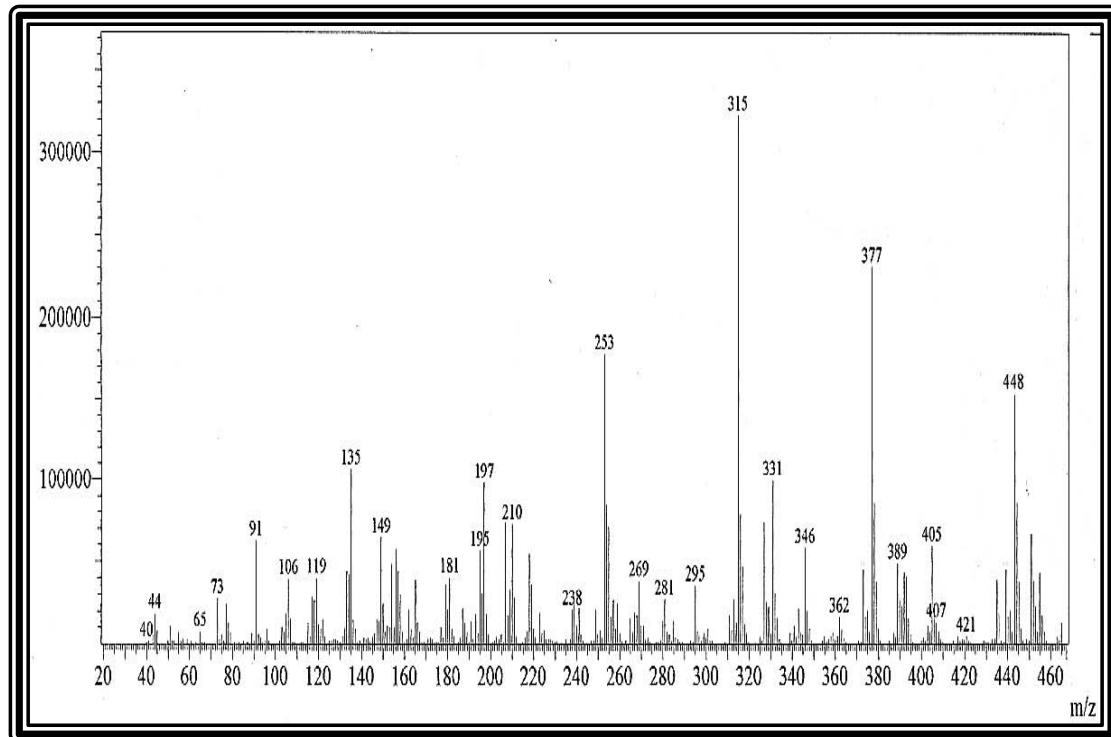


Figure 1 : MS spectra of ligand (I)

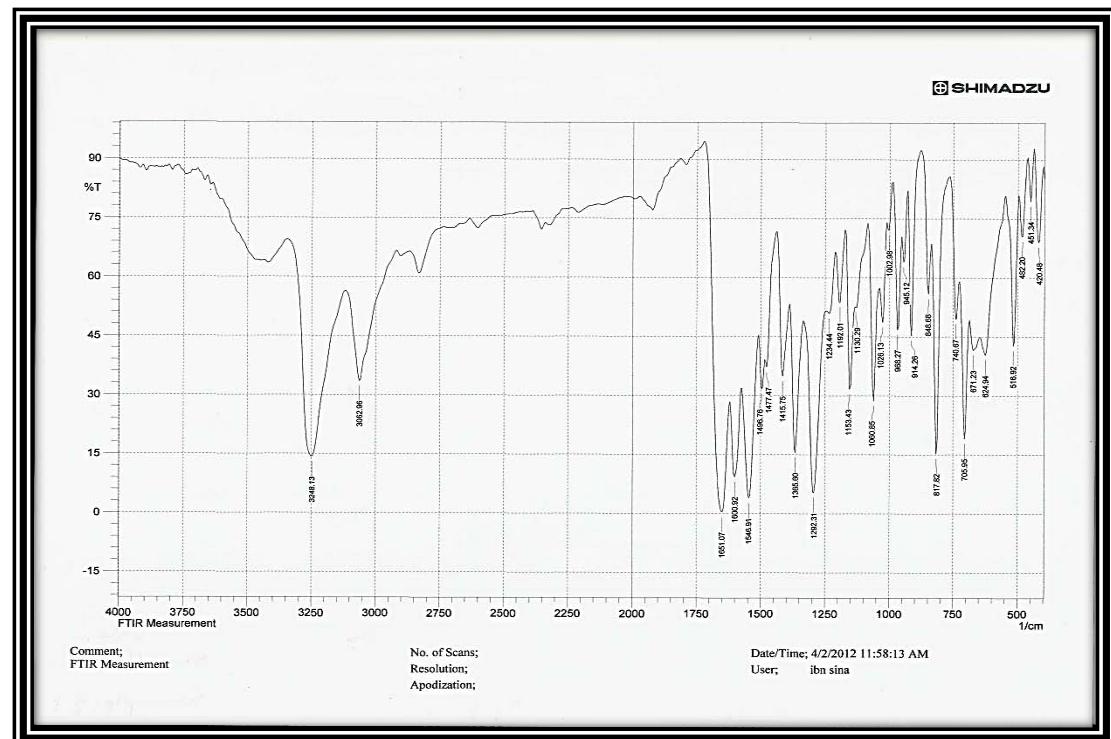


Figure 2 : IR spectra of ligand (I)

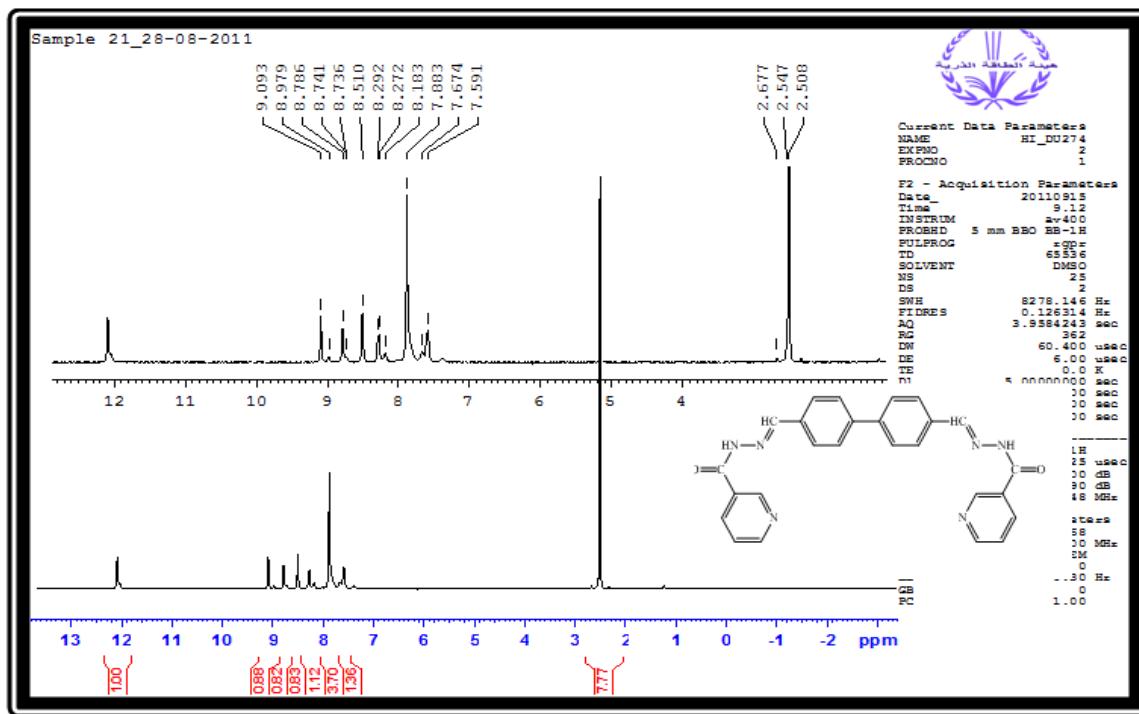


Figure 3 : ^1H -NMR spectra of ligand (I)