



# INDOLE–AZO LIGANDS (PREPARATION, SPECTRAL ANALYSIS, THERMAL STUDIES, BACTERIAL RESISTANCE STUDY)

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**Abstract:** As a result of the development in modern studies in the field of polymers, wood, glass and ceramics have been dispensed with and replaced with polymers manufactured from indole and azo due to their low density, physical properties, strength, hardness, ease of preparation and molding as required. The azo compounds entered the composition of some polymers, and this increased their importance in several fields, and spread widely in the field of various chemical industries. The indole-containing polymers with the azo group are known as large molecules with a group (N=N) in their side chain, which are characterized by high stability prepared from the multiple condensation reactions of diamine compounds with carbonyl compounds. As a result of the development in modern research in the field of polymers, wood, glass and ceramics have been dispensed with and replaced with polymers manufactured from indole and azo due to their low density, physical properties, strength, hardness, ease of preparation and molding as required. Indole-Azo ligands screened with (Uv. Vis, FT.IR, Mass) – Analysis besides to Thermal studies, bacterial resistance study.

**Keywords:** indole, azo, chalcone, ligand, dentate, amine, diazo

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## INTRODUCTION

The azo compounds entered the composition of some polymers, and this increased their importance in several fields [1,2], and spread widely in the field of various chemical industries. The indole-containing polymers with the azo group are known as large molecules with a group (N=N) in their side chain, which are characterized by high stability prepared [3-5] from the multiple condensation reactions of diamine compounds with carbonyl compounds [6,7]. And the azo compounds, which include indole compounds, are among the widest and most important azo compounds commercially and biologically and in the manufacture of some pharmaceutical drugs, amino acids and other antibiotics. The diagnostic means known as infrared spectroscopy [8-10]. The azo compounds are characterized by giving products with good proportions and high stability, and are characterized by ease of purification and deep colors that suffer displacements in wavelengths when they are consistent with the elements [11-14]. The indole-azo reagents have biological effectiveness due to the presence of the indole ring, as well as chemically effective because they possess important groups such as azo (-N=N-) that help them in coordination with different metal ions [15-17]. Accordingly, the study of indole ligands and its derivatives gained a wide [18-22] space in the field of

biological [23-26], analytical and industrial chemistry [27-30], and they were used in medicine [31, 32] as anti-cancer reagents [33-35].

## EXPERIMENTAL PART

Spectrophotometric devices were used in the analytical, spectroscopic and physical measurements of the prepared ligands, which were characterized by high accuracy from Kashan University, which was represented by the analysis below to demonstrate the structures of the synthesized ligands, as well as the chemical materials were prepared with extraordinary purity.

### Synthesis of Indole-Chalcone Ligand {1}

Formal indole (0.01 mole) countered with 3-chloro-acetoaniline (0.01 mole) in occurrence of (basic solution 5% NaOH) in rotation step, then separation, desiccating, manifestation with absolute ethanol to Indole-Chalcone Ligand {1} appreciative to studies [4,10].

### Synthesis of Indole-Azo Ligand {2}

Indole-Chalcone Ligand {1} (0.01 mole) solvated in (3 ml) of (HCl) with solution of sodium nitrite in azotation step at ice temperature, followed by coupling step with (0.01 mole) of p-methyl phenol, then separation, desiccating to yield Indole-Azo Ligand {2} appreciative to studies [4,10].

### Synthesis of Indole-Aminothiazine Ligand {3} :

Indole-Azo Ligand {2} (0.01 mole) countered with thiourea (0.01 mole) in occurrence of (acidic medium of HCl), then separation, desiccating, manifestation with absolute ethanol to Indole-Aminothiazine Ligand {3} appreciative to studies [4,10].

**Synthesis of Indole-Methylthiazine Ligand {4}:**

Indole-Azo Ligand {2} (0.01 mole) countered with thioacetamide (0.01 mole) in occurrence of (acidic medium of HCl), then separation ,desiccating ,manifestation with absolute ethanol to Indole- Methylthiazine Ligand{4} appreciative to studies [4,10] .

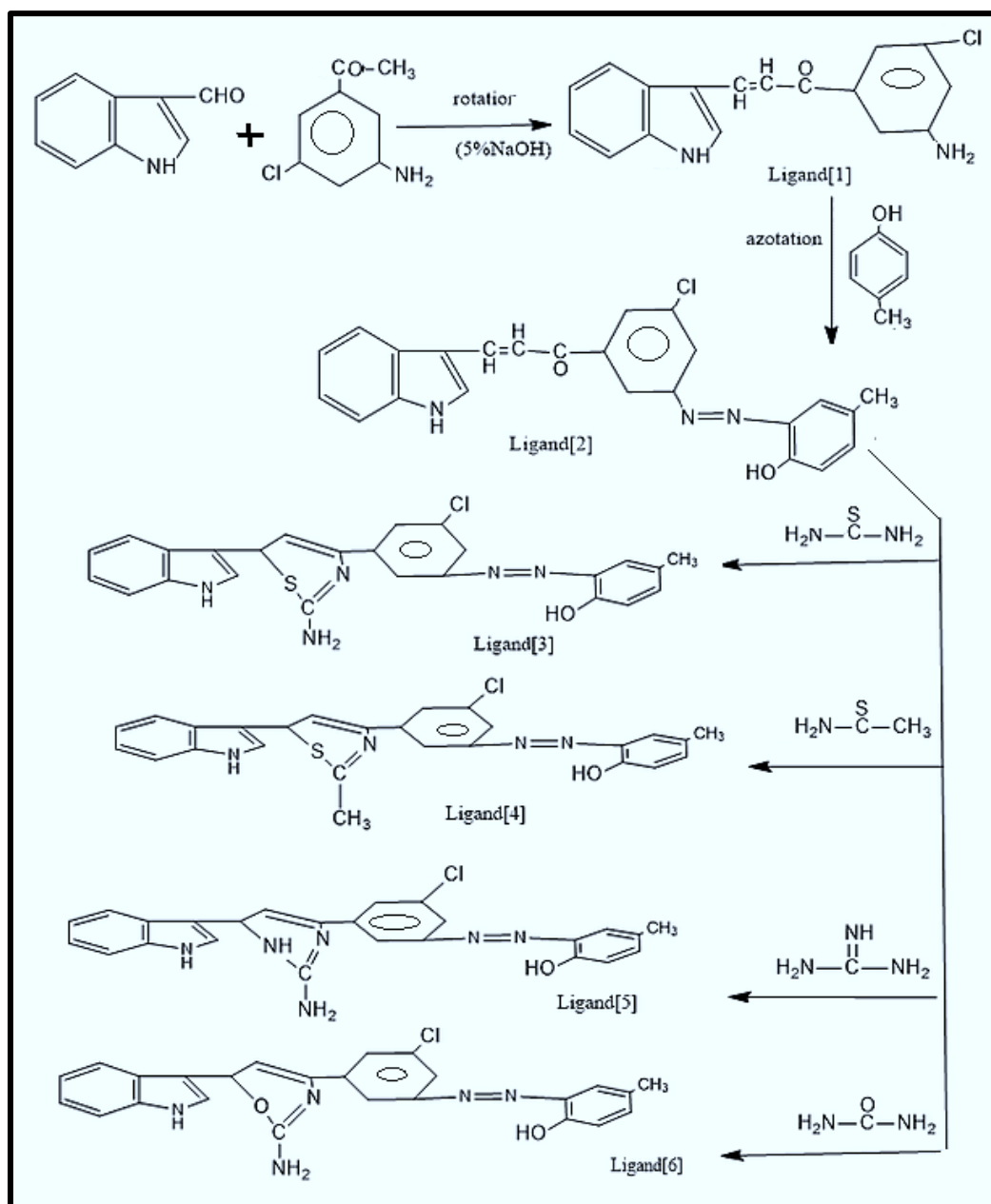
**Synthesis of Indole-Aminodiazine Ligand {5}**

Indole-Azo Ligand {2} (0.01 mole) countered with guanidine (0.01 mole) in occurrence of (acidic medium of HCl), then

separation ,desiccating ,manifestation with absolute ethanol to Indole- Aminodiazine Ligand{5} appreciative to studies [4,10] .

**Synthesis of Indole-Amino oxazine Ligand {6}**

Indole-Azo Ligand {2} (0.01 mole) countered with urea (0.01 mole) in occurrence of (acidic medium of HCl), then separation ,desiccating ,manifestation with absolute ethanol to Indole- Amino oxazine Ligand{6} appreciative to studies [4,10].



Configuration.1: Synthesis of Indole-Azo Ligands{1-6}

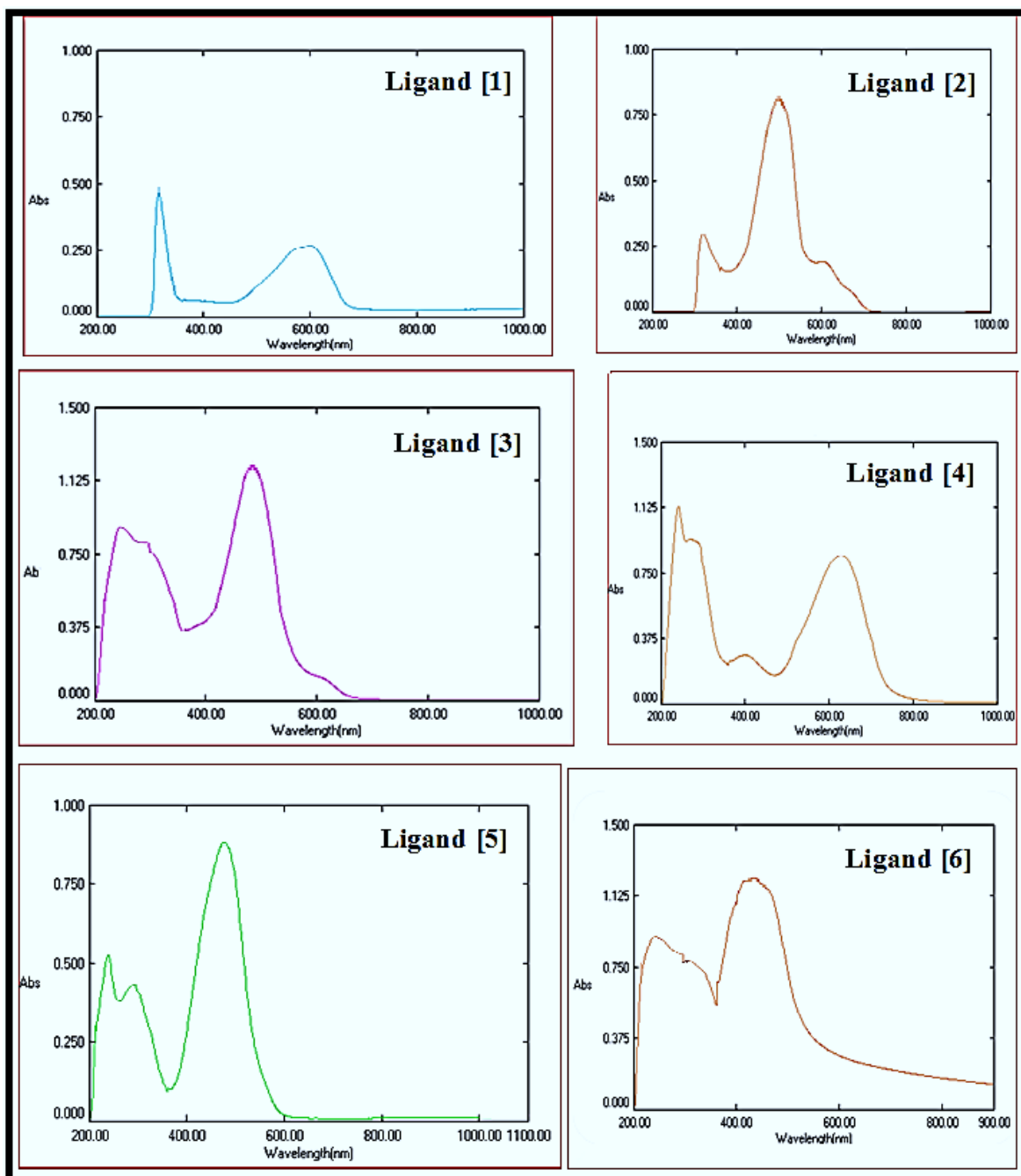
## RESULTS AND DISCUSSION

The solid ligands were characterized by using infrared (IR) and UV-Vis spectroscopy. The (IR) spectra of these ligands were recorded in the form of hard disks with potassium bromide (KBr) at the range (4000-400  $\text{cm}^{-1}$ ) and (UV-Vis) measurements were performed for the prepared ligands solutions dissolved in ethanol at a concentration ( $1 \times 10^{-4}$ ) molar using quartz cells with optical path length (1 cm), then the diamond spectrum of some ligands was measured to prove

their chemical composition, then the thermal curves of the prepared ligands were measured.

### Ultraviolet-Visible Spectroscopy

The measurements of optimal concentrations of the prepared ligands and UV-Vis spectroscopy were performed to scan the spectra of the ligands to arrive at calibration curves to find out the maximum wavelengths of the ligands using a device (UV-Visible Spectrophotometer-(Shimadzu - UV -1700).

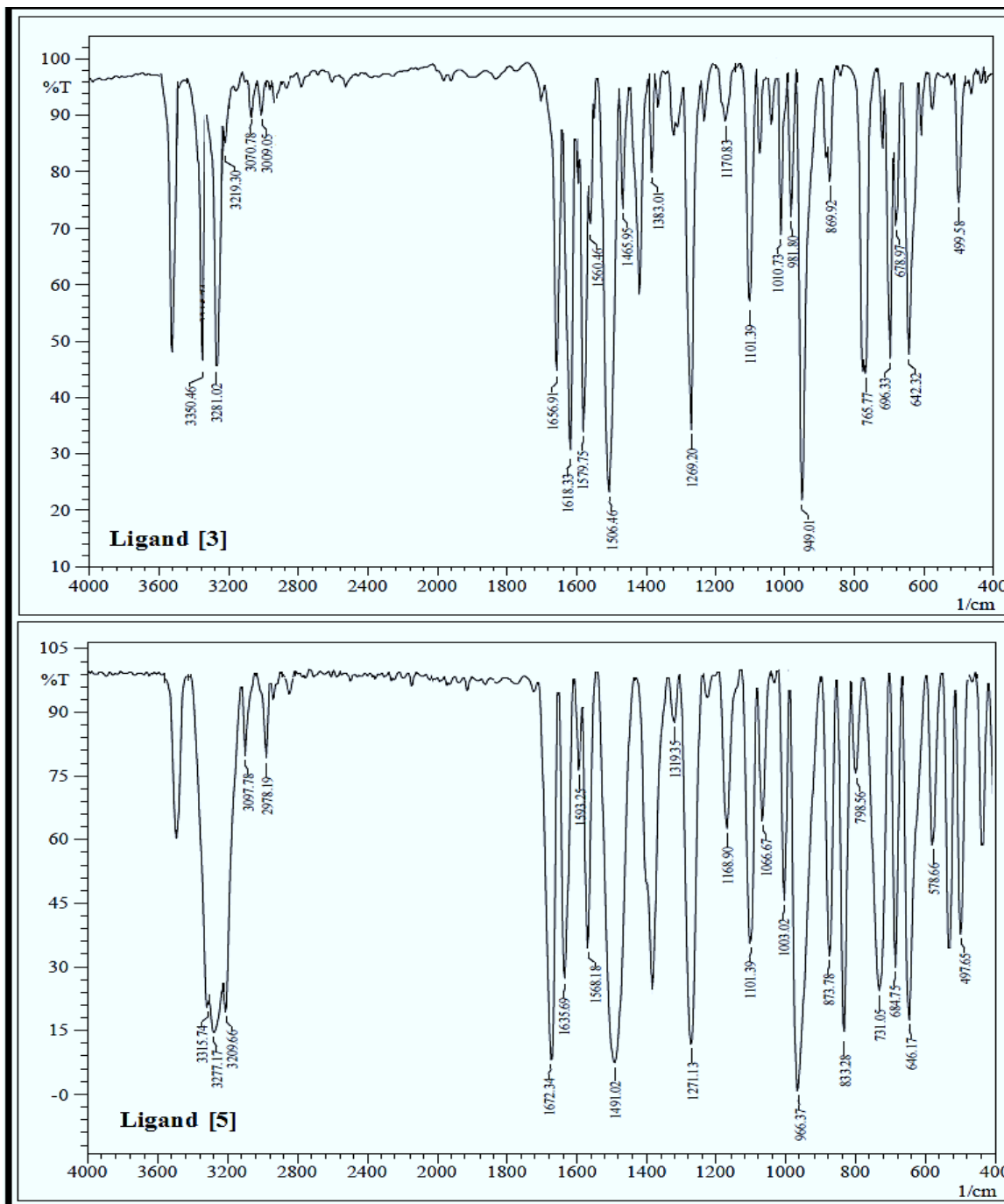


Configuration.2: UV.Vis of Indole-Azo Ligands{1-6}

### FT-IR- Revealing

Infrared spectroscopy represents one of the important diagnostic methods in confirming the structures of the prepared ligands by shifting some frequencies with absorptions of the active groups and their appearance strongly and with sharp and strong frequencies that indicate the formation of azo, chalcone groups and azo compounds as a result of the cyclic closure process of chalcone like bands at (3240 -3284)cm<sup>-1</sup> in all

synthesized ligands respectively for amine group in indole cycle., Also appearance bands at (1422-1508) cm<sup>-1</sup> for (-N=N-) Azo group in all synthesized ligands respectively, while appearance frequencies at [(3340 , 3465) to (3362-3480)] cm<sup>-1</sup> for (-NH<sub>2</sub>) amine groups in ligands {1, 3, 5, 6}, all spectral revealing approving to investigation reference [14], some of spectra:

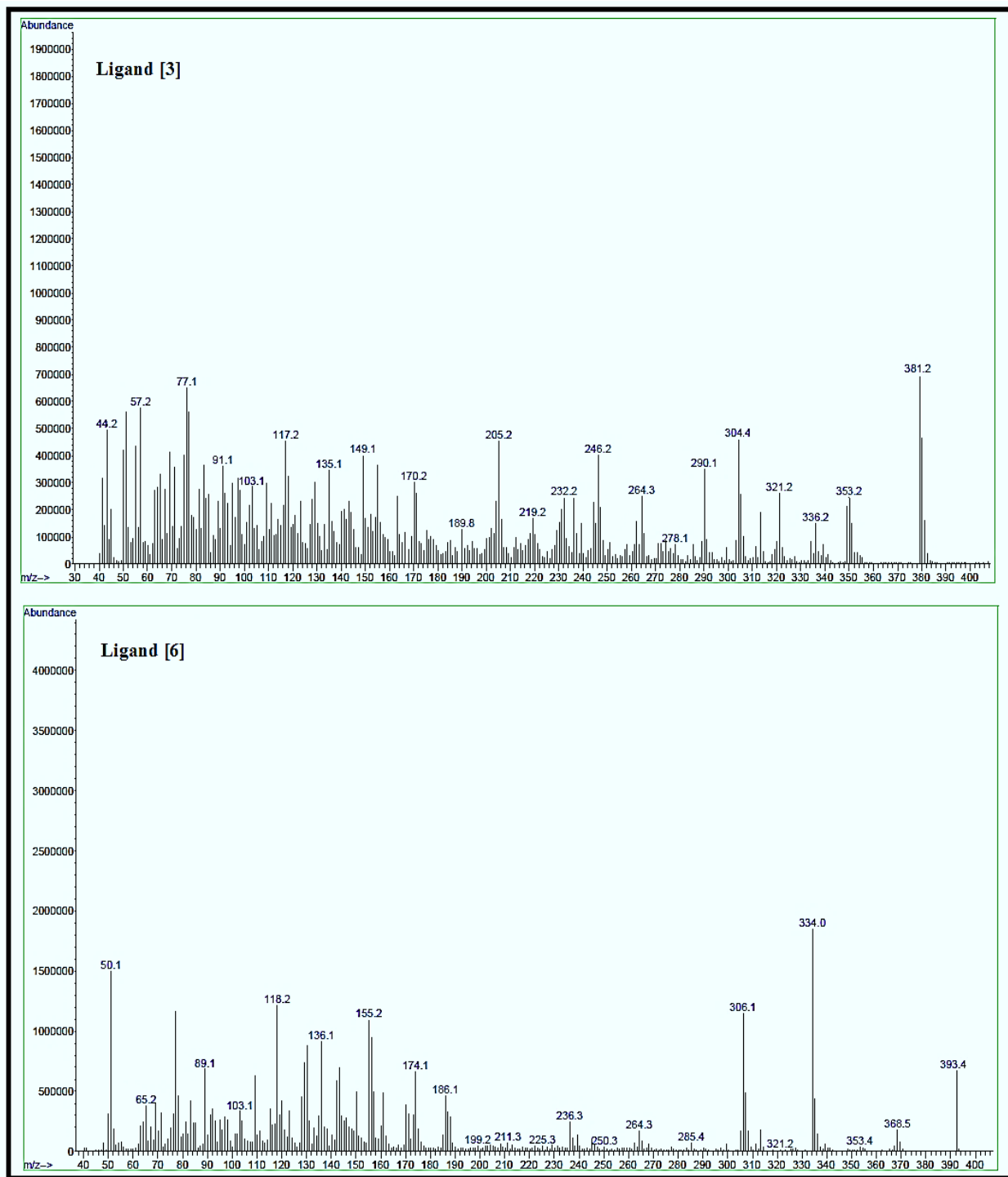


Configuration.3: I.R of Indole-Azo Ligands{3 ,5}

**Mass – Revealing**

The revealing of Indole-Azo Ligands contributed in another indication of prepared ligands {1-6} that acted by fractions of

practical groups in matching molecular weight., all spectral revealing approving to investigation reference [14] , some figures(4):

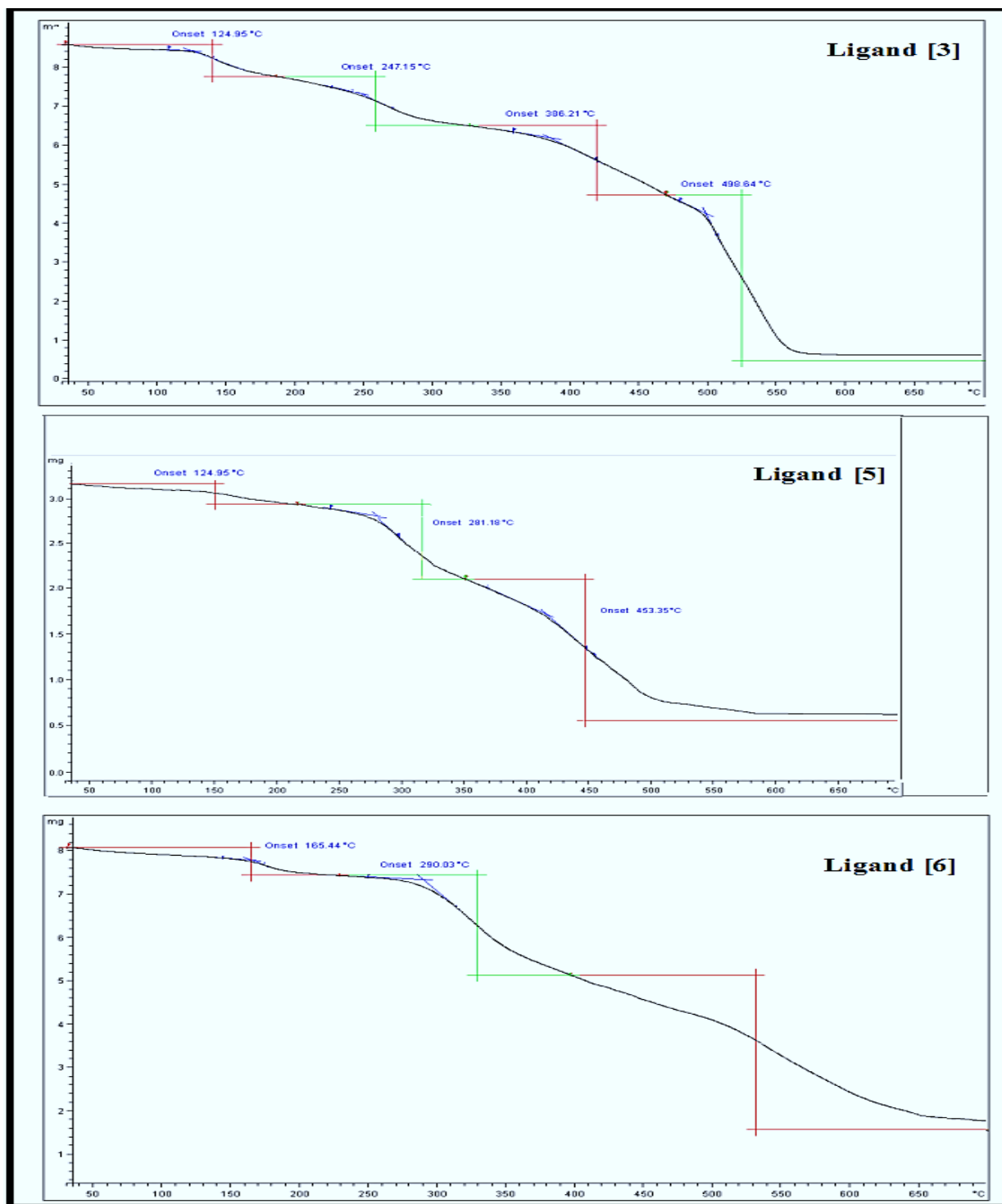


**Configuration.5 : Mass –Spect. of Indole-Azo Ligands{3 ,6}**

### Thermal Analysis of Ligands

The thermal behavior of the indole ligands prepared using TGA and DTG technique was studied in an inert atmosphere

of nitrogen gas with a wide range of temperatures ranging from (39-900) C° to 10 C. °min-1 :

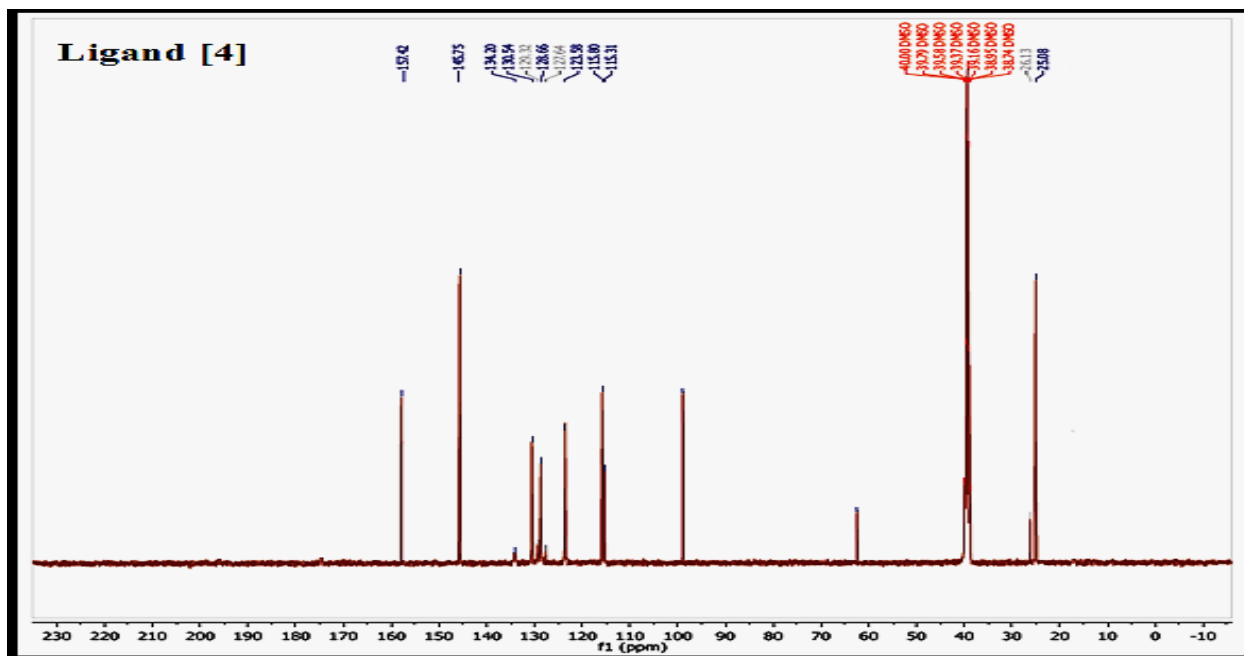


Configuration.6 : Thermal Diagram of Indole-Azo Ligands{3 ,5 ,6}

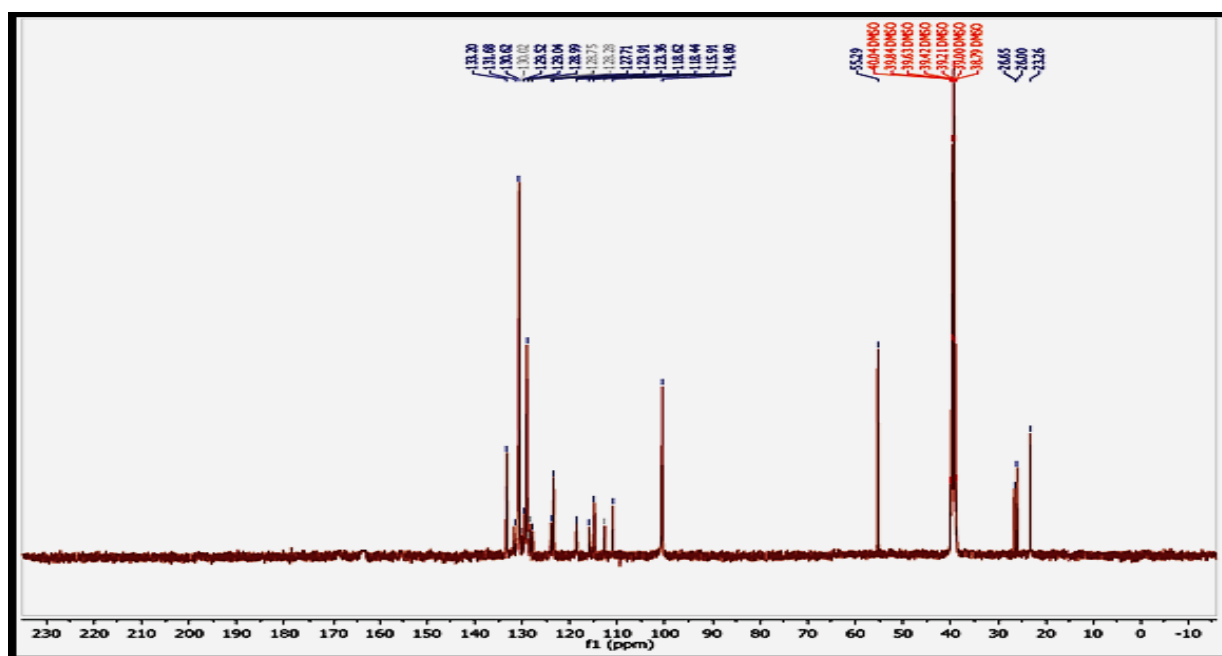
### C.NMR- Revealing

The resonance spectra represents one of the important diagnostic process in confirming the structures of the prepared ligands by appearance of signals for carbon atoms in spectrum of the active groups and their appearance strongly that indicate the formation of azo, chalcone groups and azo compounds as a result of the cyclic closure process of

chalcone like signal at  $\delta$  (99.93) for (S- C=N) endocycle of thiazine cycle , signal at  $\delta$  (25.36) for (CH<sub>3</sub>) carbon of methyl group in ligand {4}, But in ligand {6} gave signal at  $\delta$  (96.76) for (O-C=N) endocycle of Oxazine cycle and signal at  $\delta$  (23.33) for (CH<sub>3</sub>) carbon of methyl group , all spectral revealing approving to investigation reference [14], some of spectra:



Configuration.7 : C.NMR of Indole-Azo Ligand { 4 }



Configuration.8 : C.NMR of Indole-Azo Ligand{ 6 }

**Bacterial Resistance Study**

Screening the biological activity of Indole ligands and the extent to which they can be used in the medical field through their effect on inhibiting the growth of forms of pathogenic bacteria, Kinds of bacteria is Gram positive, symbolized concluded (Staphylococcus aureus , Streptococcus pneumonia ), and the another classification is Gram negative, symbolized by dint of (E.Coli ) on (three concentrations :25, 40 , 55 micro gram) depending on literatures [6, 15] .All terms mean: (+) : inhibition (2-6) mm , (++) : inhibition (7-10) mm, (+++) : inhibition (11-16) mm.

**Table.1:** Impact of the confrontation of Indole Ligands on Bacteria in (55 micro gram)

| Indole Ligands | Staphylococcus aureus | Streptococcus pneumonia | Escherichia. Coli |
|----------------|-----------------------|-------------------------|-------------------|
| Indole {1}     | +                     | +                       | +                 |
| Indole {2}     | ++                    | ++                      | +                 |
| Indole {3}     | +++                   | +++                     | +++               |
| Indole {4}     | +++                   | +++                     | +++               |
| Indole {5}     | +++                   | +++                     | +++               |
| Indole {6}     | ++                    | ++                      | ++                |

**CONCLUSIONS**

The azo compounds are characterized by giving products with good proportions and high stability, and are characterized by ease of purification and deep colors that suffer displacements in wavelengths when they are consistent with the elements., All ligands gave good stability in Thermal curves.

**Conflict of interest**

The authors declare that there is no conflict of interest.

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None

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