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Tripodal Schiff Base Compounds Containing Triazole and Mn^{3+} and Fe^{3+} Complexes: Synthesis, Characterization, and Investigation of Their Spectral Properties

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Highlights:

- Triazole
- Characterization
- Spectral Properties

Keywords:

- Schiff Bases
- Tripodal Compounds
- Metal Complex

ABSTRACT:

In this study, it was aimed to synthesize and elucidate the structure of tripodal Schiff bases, which are used in the fields of materials science, chemistry, and pharmacology, and especially for the synthesis of chemical sensor materials due to their very good tendency to form complexes with metals. For the synthesis of new Schiff base tripodal compounds and their complexation with metals; Tripodal compound was synthesized as a result of the reaction of 2-hydroxy-4-(prop-2-yn-1-yloxy) benzaldehyde and N1, N1-bis(2-aminoethyl) ethane-1,2-diamine. The obtained compounds were complexed with Fe^{3+} and Mn^{3+} metals. The structures of synthesized tripodal ligands and metal complexes were tried to be elucidated using IR, UV-vis, Photoluminescence, ^{1}H-NMR, ^{13}C-NMR spectroscopic methods.

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INTRODUCTION

The formation of tripodal complexes is directly related to the magnitude of the internal hole, the hardness of the compound, the donor automata and the counterion's desire to form complexes (Leniec et al., 2007; Vigato and Tamburini, 2004). These structures are designed to synthesize [1/1], [1/2] and extra metal ion complexes. Tripodal Schiff base materials attract attention owing to their easy synthesis and multidentate structure that is willing to bind with d- and f-metals (Schilf et al., 2004). Tripodal compounds attract attention with their unique structural properties as well as special physical and chemical properties. Tripod metal complexes, which are considered very important in terms of their chemical structures, are used in areas such as modeling metalloproteins and catalyzing the hydrolysis of activated phosphate esters (He et al., 1999). Structures containing imidazole in their structure are the focus of attention due to their biological importance in different metalloproteins and exclusively heme proteins (Nath et al., 2010). Tripodal hexadentate Schiff base materials, consisting of condensation of a multifunctional amine, such as imidazolecarboxaldehyde and tris(2-aminoethyl) amine (tren), are known for their good metal binding ability with imine and aprotic imidazole N contributors. The presence of ionizable protons on the imidazole rings of some imidazolecarboxaldehyde precursors and the substituted imidazolecarboxaldehyde vanguards confers the possibility of synthesizing transition metal complexes and the spins of metal atoms and protons. ligands in different combined oxidation states (Brewer et al., 2005; Kaur and Baral, 2014; Nath et al., 2010). Tripodal Schiff base ligands, which contain three imidazole groups in their structure, are known as first compound to exhibit spin-cross over (SCO) attitude for FeII and FeIII oxidation states, and the network structure based on imidazole-imidazolate hydrogen bonds causes contacts between SCO sites. such structures can be attached to the Fe atom via the imidazole ring (Brewer et al., 2005; Ikuta et al., 2003; Nath et al., 2010). Since they have the ability to mimic the location of metal ions in biological activities, studies on polydentate ligands are given importance. Polydentate ligands, with their cyclic, linear, branched and tripod structures, form a bonding basis to metals in different coordinations (Gelinsky et al., 2002; Kaur and Baral, 2014). Cyclic ones have high selectivity but slow metal binding. Linears, on the other hand, have low selectivity, while bonding with metal is quite fast. Tripodals have their advantages in both types. Due to both their advantageous status and easy synthesis, studies have been especially focused on Tripodal Schiff bases. In addition, tripodal Schiff bases have managed to stand out among such compounds as they have applications such as chemosensor and fluorosensing, chelation therapy for the action of metal overload, clinical diagnosis and chemotherapy (Grazina et al., 2009; Kaur and Baral, 2014; Quinn et al., 2010) . It has been reported that tripodal structures form uncharged complexes with high thermodynamic permanence and kinetic inertia, by means of their ability to form complexes with trivalent metal ions such as Fe(III), Cr(III) and Al(III). Fe (III) complexes of tripodal structures are reported to have great potential in the treatment of iron overload (Kaur and Baral, 2014). Many studies have been carried out on metal complexes with Schiff base ligands. Mn complexes, one of the prominent ones among these complexes, have managed to be one of the most interesting structures owing to their amusing biochemistry and conspicuous magnetic properties. Manganese complexes have been the focus of attention due to their applications in the agricultural sector or their involvement in catalyst and organic synthesis of different compounds (Li et al., 2011; Salehzadeh et al., 2021; Tasiopoulos et al., 2004). Molecular design of tripodal compounds provides a suitable basis for improving stability, selectivity, and complexation tendency. The sensor property of a tripodal structure is directly related to the stiffness of its extensions and the cavity diameter (Ballester et al., 1999; Fan et al., 2002). The excess of chelating effect in tripodal compounds increases the bonding desire. Large volumes of tripodal structures are effective on the desire to bond with metal. Such compounds have had the opportunity to take their place in supramolecular chemistry in terms of the development of tripodal sensors due to their stated properties (Berrocal et al., 2000; Reinoso-García et al., 2005).

In this study, 2-hydroxy-4-((1-(4-methoxyphenyl)-1H-1,2,3-triazol-4) ends of N1, N1-bis(2-aminoethyl) ethane-1,2-diamine. A new tripodal ligand was designed and prepared by binding-yl) methoxy) benzaldehyde and 4-((1-(4-chlorophenyl)-1H-1,2,3-triazol-4-yl) methoxy)-2-hydroxybenzaldehyde. Complexation of synthesized tripodal compounds with Fe^{3+} and Mn^{3+} metals was investigated.

MATERIALS AND METHODS

Materials

Chemicals used in synthesis and analysis were attained from corporate firms (Aldrich or Merck). The structure of the synthesized imine bond-containing tripodal compound was characterized using spectroscopic methods for instance FT-IR, ^{1}H NMR and ^{13}C NMR. In the structural analysis of the compound; FT-IR (ATR) measurements, Perkin Elmer Spectrum 400 Spectrophotometer, light absorption properties, Hitachi U3900h Spectrophotometer UV-vis spectrophotometer, determination of emission characteristics HORIBA Jobin Yvon Triax 550 CCD, Photoluminescence Spectrophotometer, structure determination of the molecule, ^{1}H magnetic and ^{13}C NMR (NMR) Bruker AVANCE III 400 Mhz NMR Spectrophotometer and the melting point of the compound was obtained using the Elektrothermal LTD 9200 instrument. All of the materials used within the scope of the study were dissolved in spectrophotometric grade containers and analyses were performed using a 1 cm optically pathed quartz cuvette.

Experimental procedure

During the synthesis of tripodal compounds containing imine bonds; First, 1.11 mmol of 2-Hydroxy-4-(prop-2-yn-1-yloxy) benzaldehyde was dissolved in ethanol in a balloon. It was refluxed under reflux until the dissolution of the substance was complete. 0.37 mmol, N1, N1-bis(2-aminoethyl) ethane-1,2-diamine was added to the prepared solution and the mixture was refluxed for 48 hr. Eventually the period, resulting mixture was kept at room conditions to crystallize and a yellow crystal was obtained. At the synthesis stage of complex materials, tripodal compounds and metals were taken at a ratio of 1/1 and refluxed in ethanol for 24 hours. The resulting-colored solids were filtered off and dried at room temperature. (Figure 1).

RESULTS AND DISCUSSION

Structural characterization of the material was carried out by spectroscopic methods such as ^{1}H-NMR, ^{13}C-NMR, UV-vis, and FT-IR analysis. The spectroscopic results meet the expectations about the compounds and confirm the structures.

Tripodal Schiff base materials encompass three imidazole groups "A" and "B" were synthesized, then "A1" "A2" complex structures from "A" ligand and "B1" and "B2" complexes from "B" ligand were synthesized with Fe^{3+} and Mn^{3+} metals.

6,6',6"-((1E,1'E,1"E)-((nitrilotris(ethane-2,1-diyl))tris(azaneylylidene))tris(methaneylylidene)) tris(3-((1-(4-methoxyphenyl)-1H-1,2,3-triazol-4-yl)methoxy)phenol) **(A):$C_{57}H_{57}N_{13}O_{9}$. Yield**: %88, **Color**: Orange- Red solid.

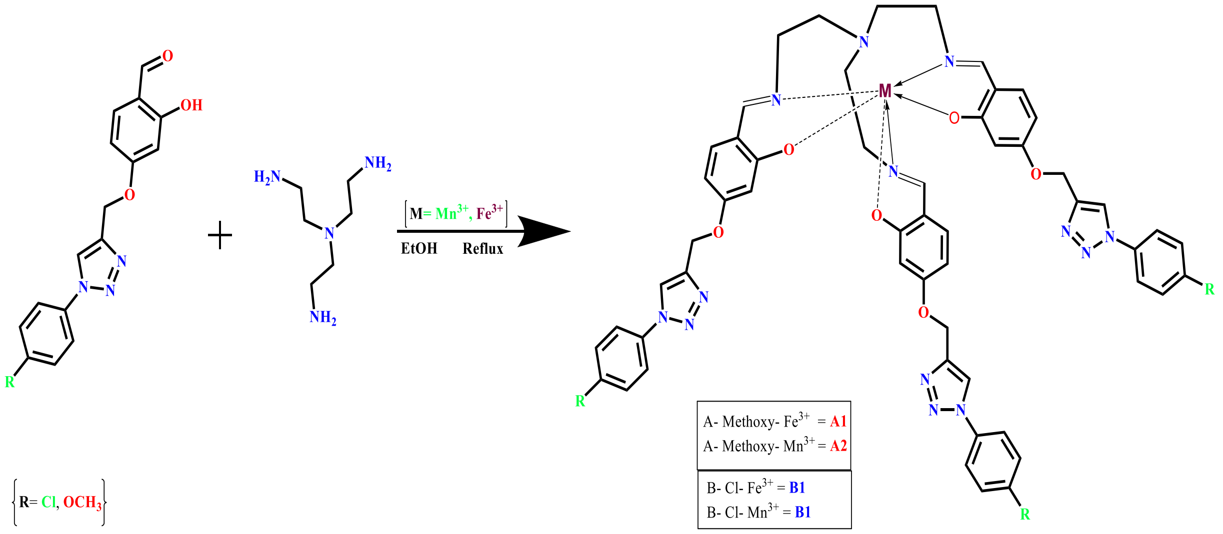

Figure 1 Synthesis steps of ligand and metal complexes

A1 complex: Yield: %79, **Color**: Dark green solid.

A2 complex: Yield: %81, **Color**: Dark red solid.

6,6',6"-((1E,1'E,1"E)-((nitrilotris(ethane-2,1-diyl))tris(azaneylylidene))tris(methaneylylidene)) tris(3-((1-(4-chlorophenyl)-1H-1,2,3-triazol-4-yl)methoxy)phenol)(**B**):**$C_{54}H_{48}Cl_3N_{13}O_6$**. **Yield**: %85, **Color**: yellow solid.

B1 complex: Yield: %77, **Color**: Tile color solid.

B2 complex: Yield: %73, **Color**: Dark red-black solid.

FT-IR spectra of Ligands and Complexes

When the FT-IR spectrum of "A" compound is examined; FT-IR data (ν, cm^{-1}): 3000- 3300 (OH), 2839- 2925 (C-H) aliphatic, ~3100 (CH_2), 1614 (C=N), 1160 (C-N), 1500-1516 (C=C), 1251 (C-O), 826,798 (C-H) aromatic. FT-IR Spectra of “A” ligand, “A1” and “A2” complex structures are given in Figure 2.

When the FT-IR spectrum of the "B" compound is examined; FT-IR data (ν, cm-1): 3000- 3300 (OH), 2835 - 2916 (C-H) aliphatic, ~3100 (CH2), 1614 (C=N), 1162 (C-N), 1501-1516 (C=C), 1246 (C-O), 800,824 (C-H) aromatic. FT-IR Spectra of “B” ligand, “B1” and “B2” complex structures are given in Figure 3.

In the FT-IR spectra of the ligands, stretch vibrations of azomethine bond were observed to shift to lower wavenumbers. This situation is thought to be caused by the coordination of the nitrogen atoms of azomethine to the metal centres. It is observed that the (C=N) band seen at 1614 cm-1 in compound A shifted to 1613 cm-1 in compound A1 and 1611 cm-1 in compound A2. similarly, the (C=N) band seen at 1614 cm-1 in compound B shifted to 1613 cm-1 in compound B1 and 1610 cm-1 in compound B2. The peak belonging to the (OH) group, which is seen around 3000 cm-1 in the FT-IR spectra of A and B ligands, is expected to disappear in complex structures formed as a result of the interaction of these ligands with metal ions. In addition, weak peaks in the range of 400-600 cm-1 after the complexation process formed by reaction with metals indicate that the targeted compounds were synthesized successfully.

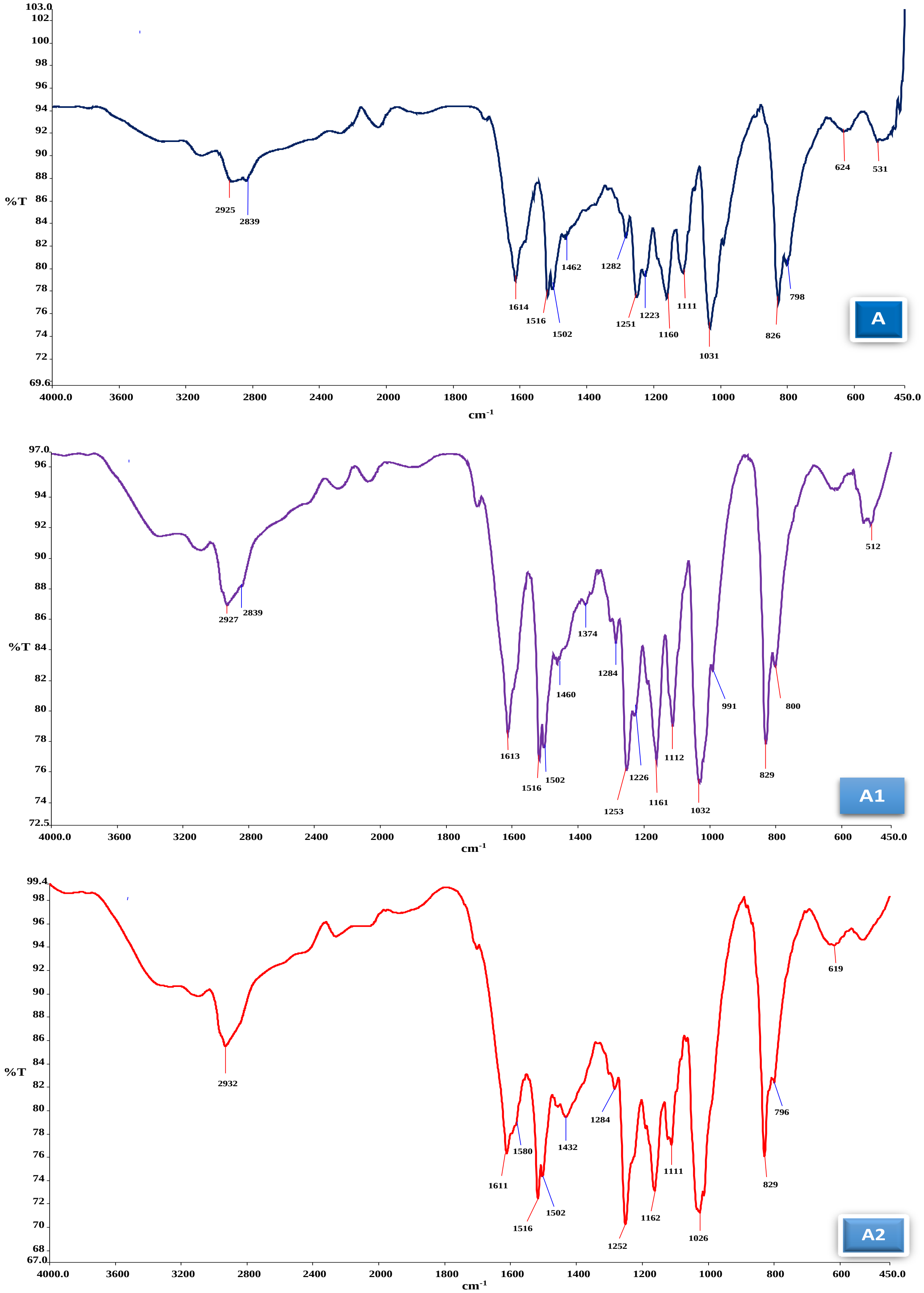

Figure 2 FT-IR spectrum of compounds “**A**, **A1** and **A2**”

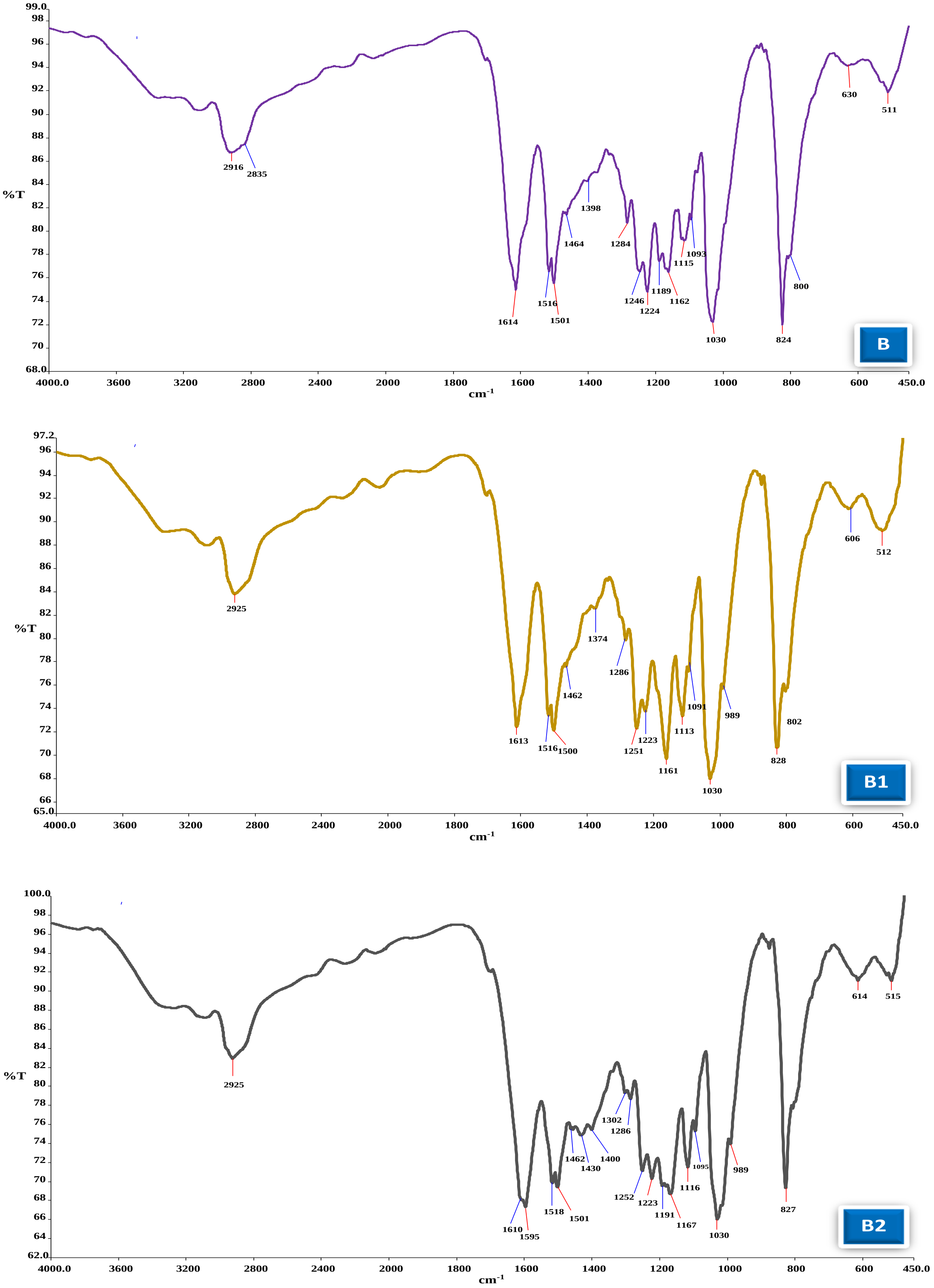

Figure 3 FT-IR spectrum of compound “**B, B1** and **B2**”.

UV-vis spectra of Ligands and Complexes

When the UV-vis spectrum of the A ligand, A1 and A2 complexes is examined, respectively Aλmax= 260 and 272 nm, A1λmax= 260 and 272 nm with 315-370 nm range and A2λmax= 260 and 270 nm with 315-370 nm range, values have been reached. UV-vis Spectra of A ligand, A1 and A2 complex structures dissolved in methanol are given in Figure 4.

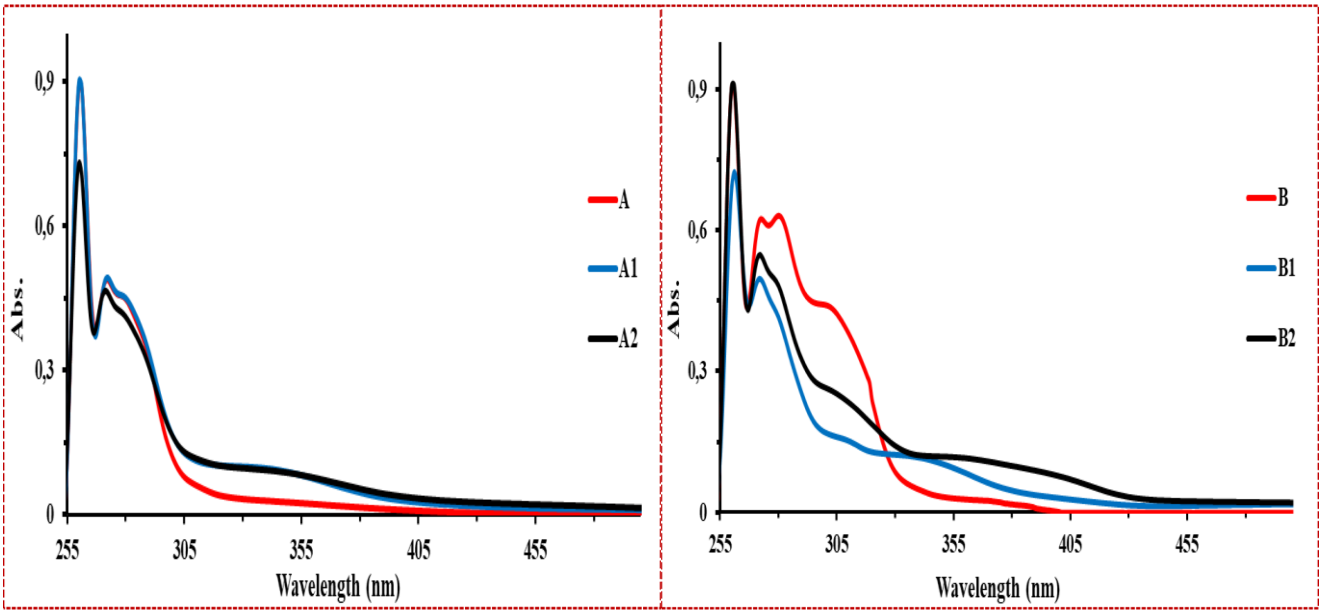

Figure 4 UV-vis spectrum of ligands and complexes.

In the UV-vis Spectra of ligands and complexes, the bands seen at 260 nm represent aromatic π-π* transitions and the bands seen at 270-272 nm represent n-π* transitions. It can also be attributed to the charge transfer transitions of the broad band Fe^{3+} and Mn^{3+} metals, which complex structures show in the range of 315-370 nm.

Photoluminescence spectra of Ligands and Complexes

The UV-vis absorbance values of the ligand and complex structures were considered, and the emission bands obtained as a result of this excitation are shown in Figure 5.

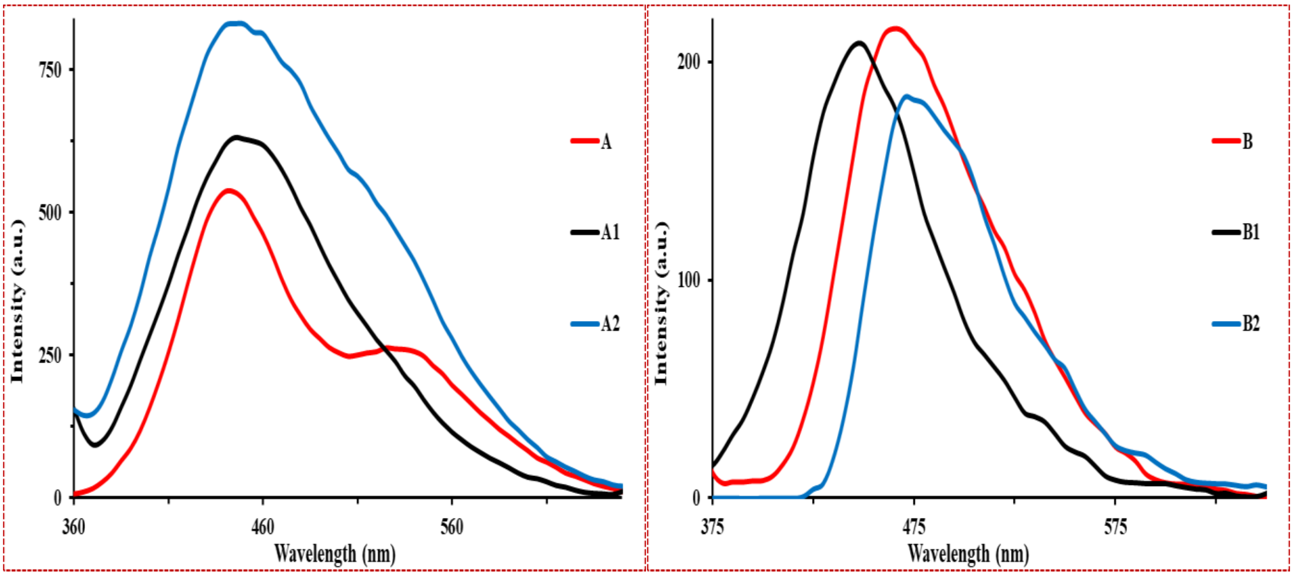

Figure 5 Photoluminescence spectra of Ligands and Complexes.

Ligand "A" showed a strong emission band at 440 nm and a broad and weak emission band in the range 505-560 nm. In the A1 and A2 complex structures, the emission band of the ligand at 440 nm widened even more, showing a stronger emission band around 450 nm with a redshift tendency. Similarly, the broad and strong emission band of the "B" ligand at 465 nm turned into the weaker emission band in the "B1" and "B2" complex structures. also, the "B1" complex showed a broad emission band at 450 nm, showing a blue-shifting tendency relative to the "B" ligand. The "B2" complex structure, on the other hand, showed an emission band at 470 nm, showing a blue-shifting tendency compared to the "B" ligand.

^{1}H and ^{13}C NMR Spectra of ligand 'A' and 'B'

^{1}H and ^{13}C NMR spectra of ligand '**A**' are given in Figure 6.

In the **^{1}H NMR** spectrum (*^{1}H NMR (400 MHz, DMSO) δ 14.00, 8.85, 8.12, 7.80, 7.78, 7.13, 6.88, 6.86, 6.47, 6.33, 6.31, 5.21, 3.54, 3.46, 3.39, 2.88, 2.81, 1.08*); the signal observed at δ1.08 ppm is the signal of protons belonging to the ($-N-CH_2$) group. The signal observed in the range of δ2.81-3.54 ppm is the signal of protons belonging to the ($-CH_2-N-$) group. The signal observed in the δ3.39-3.46 ppm range is the signal of protons belonging to the (CH_3-O-) methoxy group. The signal of the protons of the ($-CH_2-O-$) group bound to the imidosal ring is the singlet signal observed at δ5.21 ppm. The signals seen in the range of δ6.31-7.80 ppm are the signals of the protons in the rings. The signal of the azomethine (-HC=N-) group proton is seen at δ8.85 ppm. The signal belonging to the proton of the (-N-C=C-) group of the triazole ring is the singlet signal seen at δ8.12ppm. The signal of protons belonging to the phenolic (-O-H) group is seen at δ14.00 ppm.

In the **^{13}C-NMR** spectrum (*^{13}C NMR (101 MHz, DMSO) δ 167.81, 165.42, 162.38, 159.67, 143.87, 134.00, 130.49, 123.41, 122.29, 114.98, 112.19, 106.47, 102.68, 61.49, 56.06, 55.28, 19.02*); The signals at δ19.02 ppm are the signals belonging to the "C" atoms of the azomethine ($-CH_2-N-$) group. Signals belonging to the methoxy (CH_3-O-) group are seen at δ55.28 ppm. The signals at δ56.06 ppm are the signals belonging to the "C" atoms of the ($N-CH_2-$) group. The signal of the "C" atom belonging to the methoxy ($-O-CH_2-$) group is seen at δ61.49 ppm. The signals seen at δ102.68, 106.47, 112.19, 114.98, 123.41, 130.49, 134.0 ppm are those of ring carbons. The signals seen at δ122.29, 143.87 ppm are the signal for the carbon of the triazole ring. The signals at δ159.67 ppm are the signals belonging to the "C" atom of the azomethine (-HC=N-) group. Signals for the carbon atoms of the (-O-C-) group attached to the aromatic ring are seen at δ162.38, 162.42, 167.81 ppm.

^{1}H and ^{13}C NMR spectra of ligand '**B**' are given in **Figure 7**.

In the **^{1}H NMR** spectrum (*^{1}H NMR (400 MHz, DMSO) δ 14.01, 8.99, 8.12, 7.95, 7.92, 7.68, 6.88, 6.85, 6.46, 6.31, 6.29, 5.23, 3.54, 2.81*); the signal observed at δ2.81 ppm is the signal of protons belonging to the ($-N-CH_2$) group. The signal observed at δ3.54 ppm is the signal of protons belonging to the ($-CH_2-N-$) group. The signal observed at δ5.23 ppm is the signal of protons belonging to the (CH_3-O-) methoxy group. The signals seen in the range of δ6.29-7.95 ppm are the signals of the protons in the rings. The signal belonging to the proton of the (-N-C=C-) group of the triazole ring is the singlet signal seen at δ8.12ppm. The signal of the azomethine (-HC=N-) group proton is seen at δ8.99 ppm. The signal of the protons of the phenolic (-O-H) group is located at δ14.00 ppm.

In the **^{13}C-NMR** spectrum (*^{13}C NMR (101 MHz, DMSO) δ 168.10, 165.38, 162.67, 143.87, 135.64, 133.99, 133.49, 130.20, 123.41, 122.56, 111.97, 106.26, 102.68, 61.27, 55.25, 54.65*); the signals at δ54.65 ppm are the signals belonging to the "C" atoms of the azomethine ($-CH_2-N-$) group. The signals at δ55.52 ppm are the signals belonging to the "C" atoms of the ($N-CH_2-$) group. The signal of the "C" atom belonging to the methoxy ($-O-CH_2-$) group is seen at δ61.27 ppm.

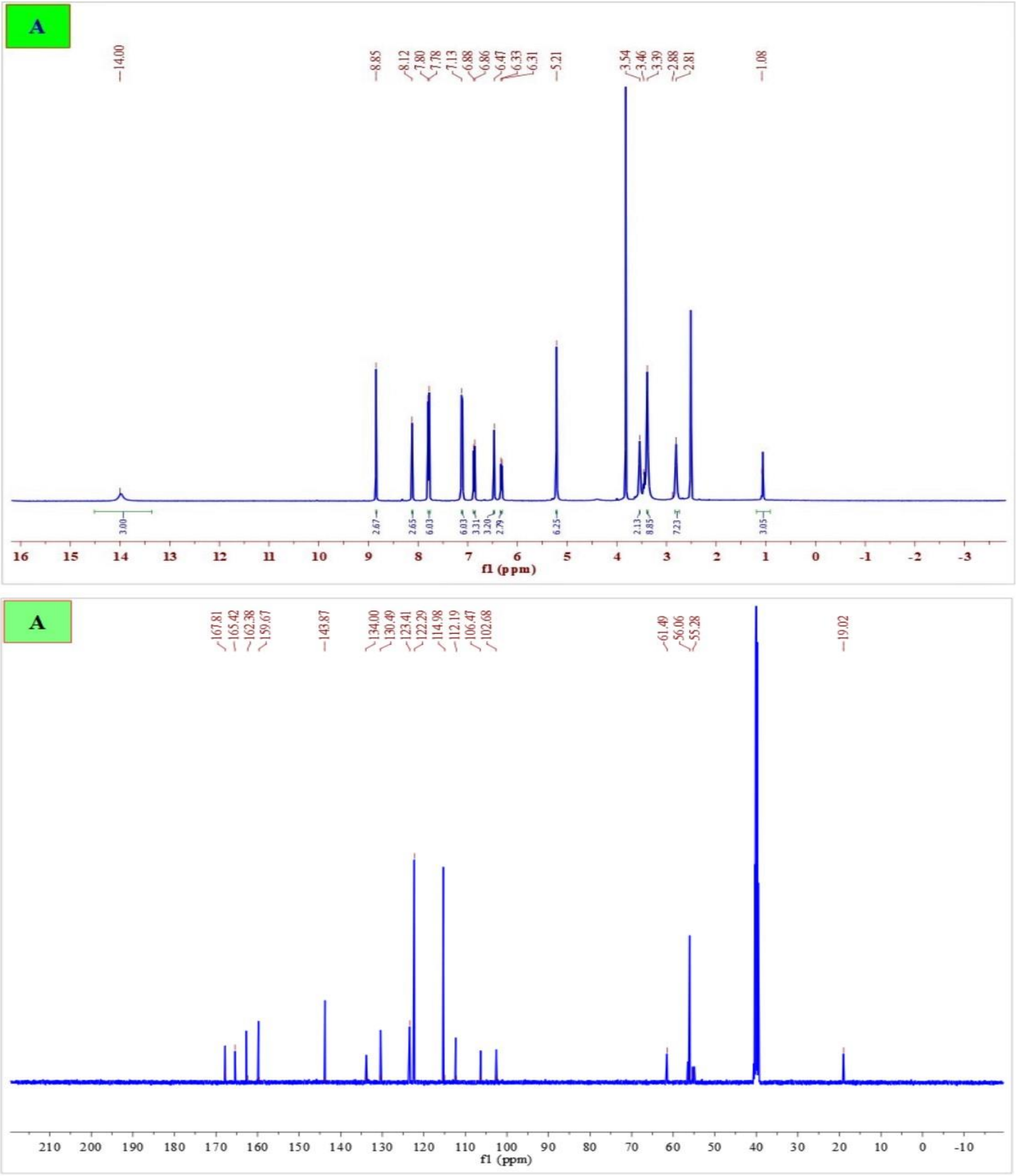

Figure 6 ^{1}H(^{13}C) NMR spectra of compound "**A**".

The signals seen at δ123.41, 143.87 ppm are the signals for the carbon of the triazole ring. The signals seen at δ δ102.68, 106.26, 122.56, 130.20, 133.49, 133.99, 135.64 ppm are those of ring carbons. The signals at δ162.67 ppm are the signals belonging to the "C" atom of the azomethine (-HC=N-) group. Signals for the carbon atoms of the (-O-C-) group attached to the aromatic ring are seen at δ168.10. Signals for the carbon atoms of the (-C-OH) group attached to the aromatic ring are seen at δ165.38 ppm.

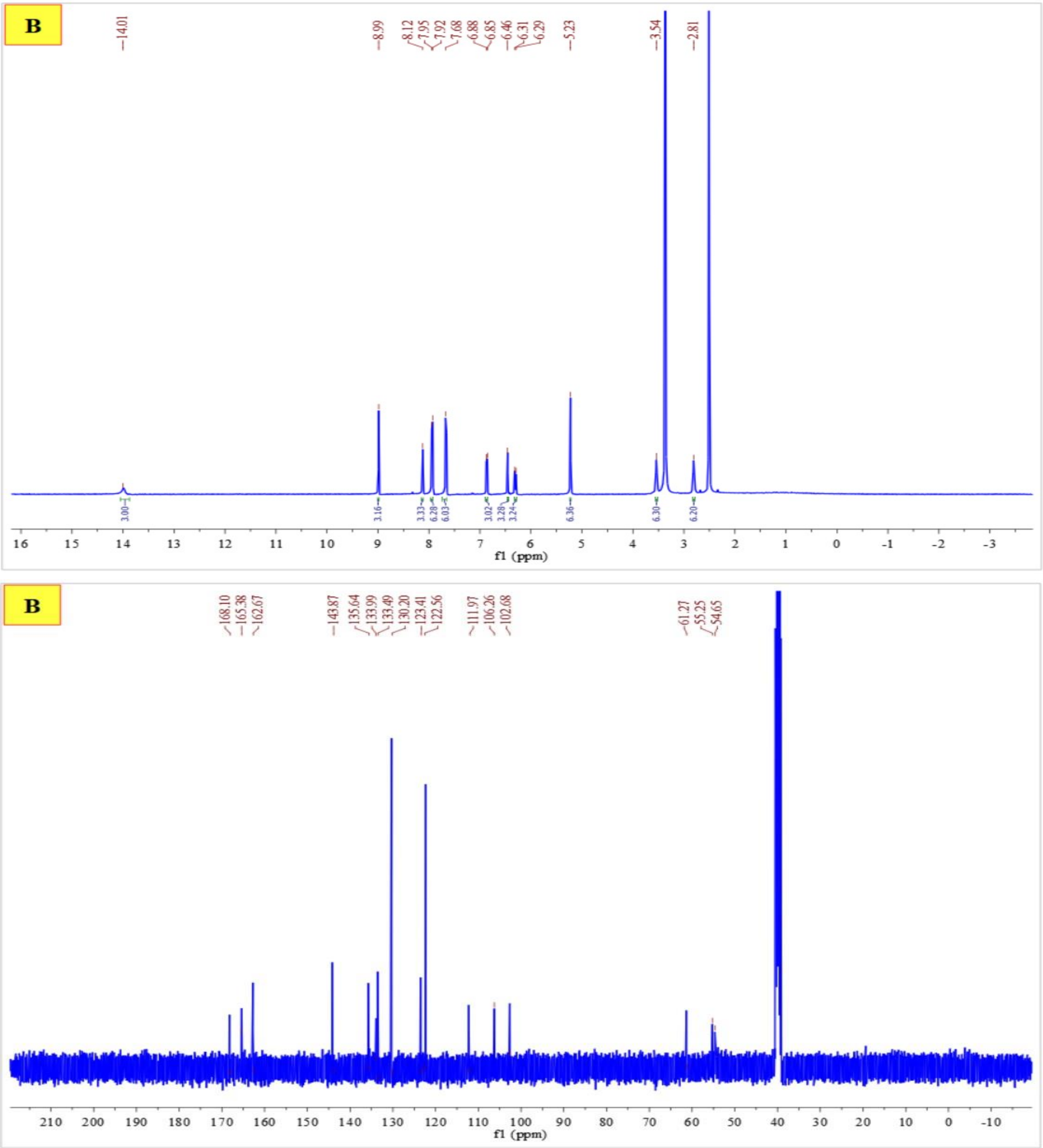

Figure 7 ^{1}H(^{13}C) NMR spectra of compound "B".

CONCLUSION

Fluorescent tripodal Schiff base compounds containing triazole group and (-C=N-) imine bond and complex structures formed as a result of the interaction of these compounds with metals were synthesized by the method in accordance with the literature. The structure of Schiff base and metal complexes was elucidated by IR, UV-vis, Photoluminescence, ^{1}H-NMR, ^{13}C-NMR spectroscopy. According to the FT-IR results, while the vibrational band belonging to the azomethine ν(-C=N-) group, which indicates the formation of trisymine, was seen at 1614 cm^{-1} in the ligands, these bands shifted to 1610-1613 cm^{-1} as a result of the complex formation. These shifts are a sign of the successful realization of complex structures. According to the structure elucidation analyses, Schiff base was found to coordinate to metal ions as a potentially polydentate ligand. Absorption values of ligand and complex structures obtained by UV-vis spectroscopy were recorded. The photoluminescence values of substances excited with the achieved absorption wavelengths were recorded. Structure illumination studies show that the ligand and complex structures intended to be synthesized can be successfully synthesized.

Some derivatives of tripodal Schiff gain importance in coordination chemistry, biochemistry, dyestuffs, plastics industry, pharmaceutical chemistry, electronics industry, and agriculture as pesticides in many fields. Due to its many usage areas, research on this subject continue. Complexing with transition metals is important in the field of bioinorganic chemistry as well as coordination chemistry. With the knowledge of the antitumor and antiviral effects of these materials, it has begun to take its place in the pharmacological field. It has become possible to obtain most heterocyclic compounds that are difficult to synthesize. The tripodal Schiff base ligand, which is very important for these reasons, was synthesized and its complex structure was obtained with some transition metals, and the structure illumination study was successfully carried out.

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Conflict of Interest

The article authors declare that there is no conflict of interest between them.

Author's Contributions

The authors declare that they have contributed equally to the article.

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