ISSN 2063-5346

Synthesis and Characterization of new ligand with (E)-5-((4-dimethylamino)-1,3,4-thiadiazole-2-thiol sometransition metal complexes

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Article History: Received: 01.02.2023 Revised: 07.03.2023 Accepted: 10.04.2023

Abstract

New ligand (E)-5-((4-dimethylamino)-1,3,4-thiadiazole-2-thiol sometransition metal complexes Cr (III), Ni (II), and Co (II) were characterized by magnetic sensitivity measurements, and in addition to spectroscopy, the FTIR and 1H-NMR spectra were cluster-compatible. Bidirectional coordinates across the nitrogen atom in the oxadiazole ring and the nitrogen atom in the amino group. This view is also supported by the emergence of a band corresponding to metallic nitrogen and vibrations at 454-688 cm-1 and 314-466 cm-1 in the complexes, respectively. The tetrahedral and square planar geometry of the complexes, the Cr (III) complex showed the octahedral geometry and the Co (II) and Ni (II) complex showed a square plane geometry with a prepared ligand.

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DOI:10.31838/ecb/2023.12.s1-B.418

1.Introduction

Heterocyclic Compounds

Heterocyclic compounds are cyclic compounds in which the ring contains at least one atom of an element other than carbon. In homogeneous cyclic compounds such as benzene, all the atoms that make up the ring are of one type, so they are called homocyclic compounds, and few Non-carbon elements are capable of forming homogeneous rings, and examples of heterocyclic compounds that do not contain the carbon element Pentamethyl-Pentaristol When the cyclic structure contains atoms of different elements, these compounds are known as heterocyclic compounds. Often such compounds contain carbon atoms with one or more other elements. The most common of these elements are oxygen, nitrogen, sulfur, and less commonly. The elements mercury, arsenic, lead, phosphorous and other elements are included, as shown in Figure (2-1) ((1).

Figure 1-2: Some heterocyclic compounds

Five-membered aromatic nitrogen cycles have been potential targets of research by several research groups due to their interesting biological activities and medicinal properties[1]. Of these, the 1,2,4-triazole scaffold forms the central part of many therapeutically active compounds such as antimicrobials[2], analgesics[3], antivirals antioxidants[4]. and anti-inflammatory drugs[5]. and anticancer agents[6]. In addition, 1,3,4-Thiadiazole and 1,3,4-Oxadiazole are also important classes of azoles with important biological properties as there are many examples in the literature including antifungals[7, 8]. and anti-inflammatory drugs. .[9, 10]., antimicrobials[11, 12]., antivirals[13, 14]. and anticancer drugs[15].

In addition, several studies have shown that clubbing of two or three heterocyclic units can significantly stimulate antimicrobial activities[16-18]. In addition, Schiff's rules have been the subject of numerous studies due to their broad spectrum of biological activities[19]. In addition, Schiff's azomethane base bonds, as attractive binding units that can link two drugs together to generate an innovative dual-function drug, have quickly become one of the most challenging and attractive topics in the literature. Drug design to build new bioactive molecules drug. Based on all of the above considerations, in addition to an extension of our studies on developments of new azole antimicrobial agents[20, 21]. We have revealed the synthesis of heterozygous ring systems with predicted antimicrobial and antiproliferative activities, except for 1,3,4-thiazole with 1,2,4-triazole, 1,3,4-thiazole, 1,3,4-oxadiazole and / Or slit a chef base into a frame work.

2.Materials and Methods

All chemicals and solvents used were chemically pure and commercially available, and all inorganic salts used chloride.

2.1.Physical Measurement

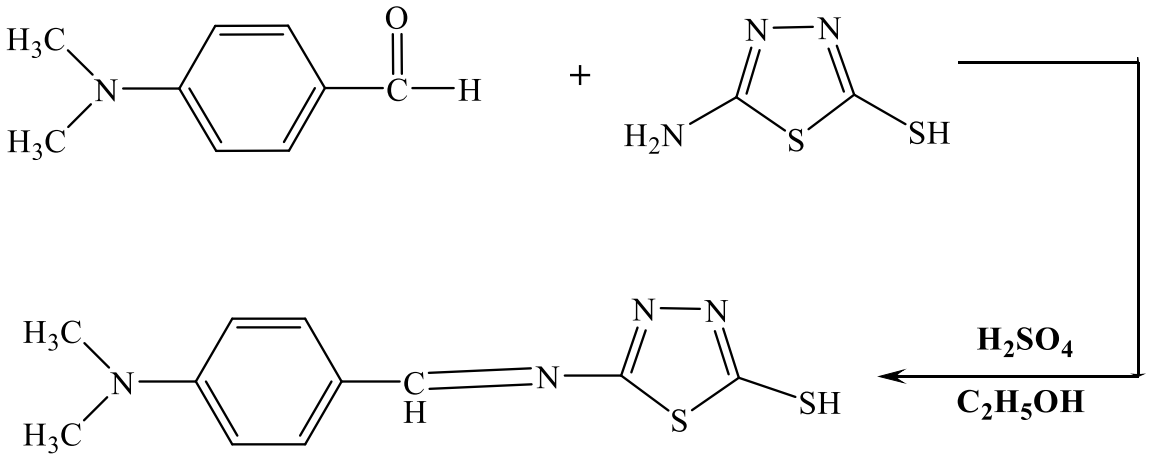
The melting point or decomposition temperature of all metal bonds and compounds prepared in the SMP31 melting model was observed. FTIR spectra were recorded in the range (250-4000) cm-1 as KBr disk using a Shimadzu FTIR spectrophotometer (model: Infrared Convergence, Shimadzu). NMR spectra were acquired using the Burker DXR AL500 system (500MHz). Mass spectra were obtained using (Network Mass Selective Detector 5973).

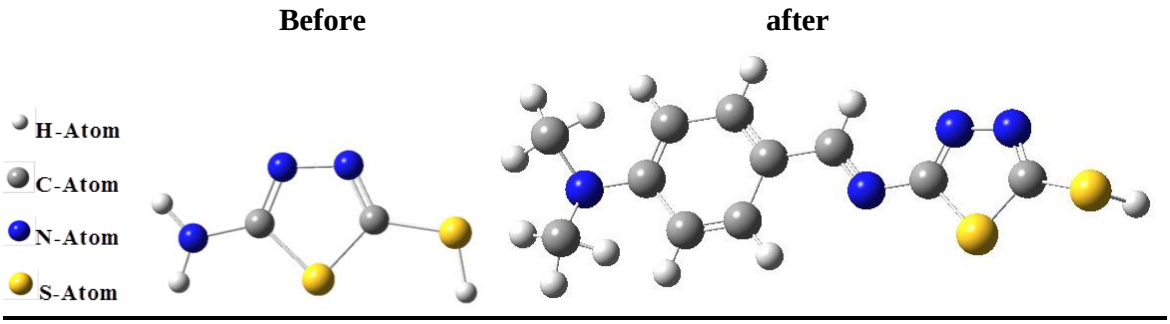
2.2.Preparation of the ligand

1-Put (6.9 g) of **P-N,N-dimethyl amino benzaldehyde** in a (100 ml) cycle and add (10 ml) of **ethanol**, then add three drops of **ice acetic acid** with the man for (5 minutes) and dissolve (5 g)rams of

amine in (10 ml) of **ethanol** in a baker's speed of (25 mg). After the amine dissolves it is added to The rhubarb that contains the first mixture, in batches, let the reaction last (10 hours).

2- (0.299g) of lycand prepared in the first way was mixed with (0.266 g) of **$CrCl_3.6H_2O$** salt dissolved in (10 ml) of **ethanol** and left for an hour.





MN), (υ SH), (υ C=N) imine,(υ C-s-C) , (N-C), **asy and** structure movement bands respectively, as shown below (table1). New bands were formed attributed to the coordinated (CBr) bonds and appeared at the region (536,578,536) cm^{-1}. This indicates that the coordinate occurred through the (N), (Br) and (S) atoms.

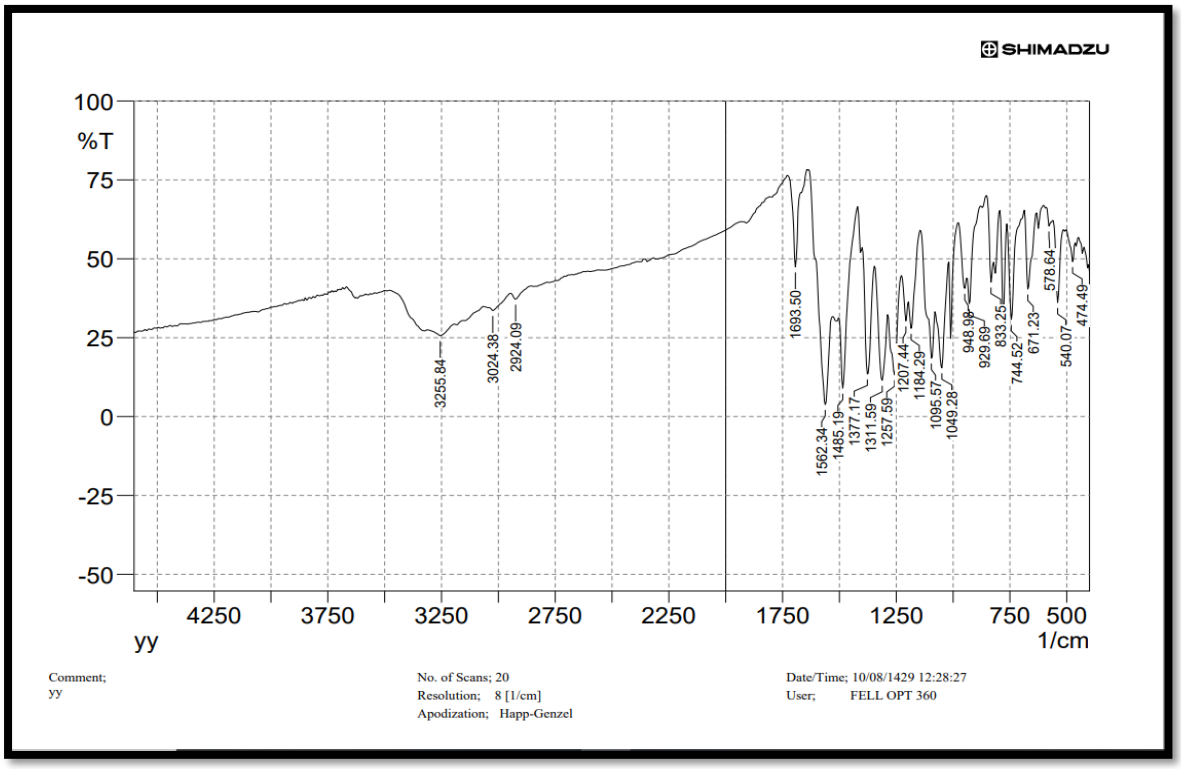
3. Result and Discussion

3.1. FT-IR spectra

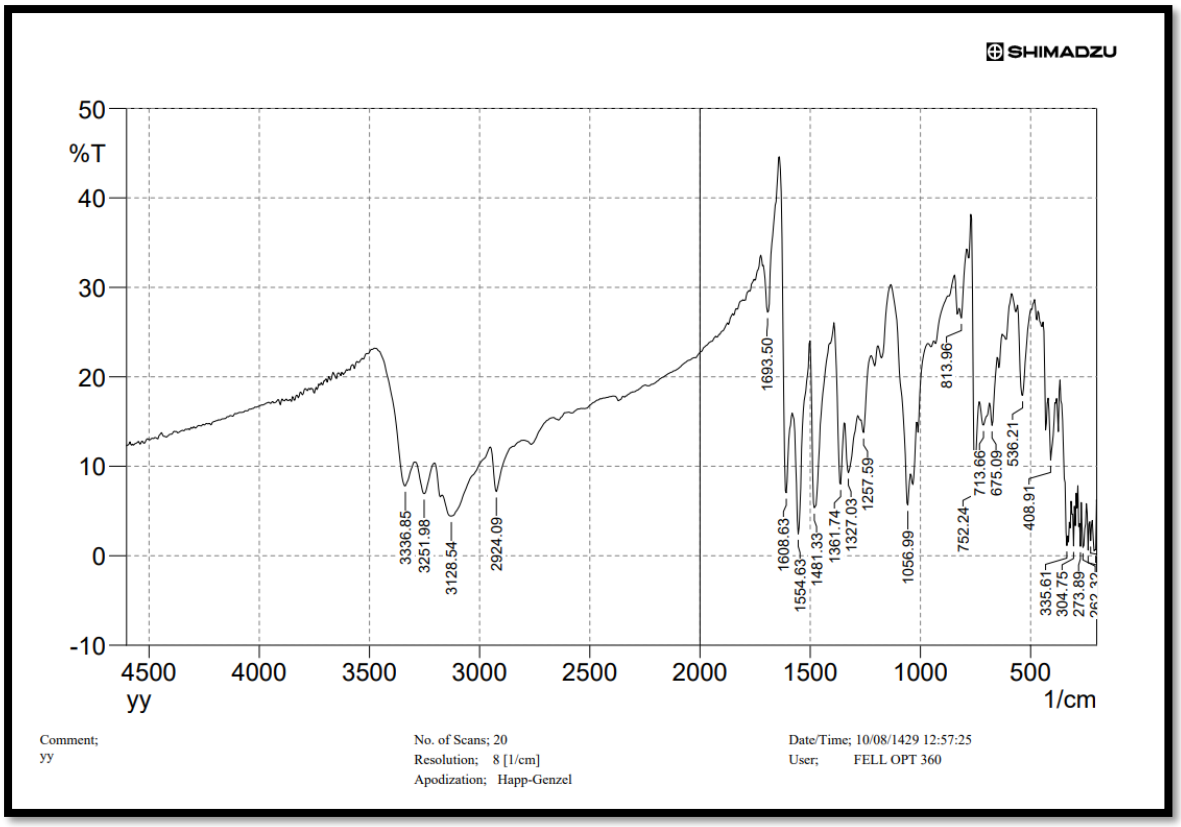
FT-IR of the synthesized ligand and its complexes were carried out KBr disc to ligand and CsI for complexes. The free ligand exhibited six major bands at (2924.09) cm^{-1}, (1608) cm^{-1},(1481) cm^{-1},(1327) cm^{-1}, which are attributable to (υ

Table 2. IR frequencies (cm^{-1}) of the compound and their metal complexes

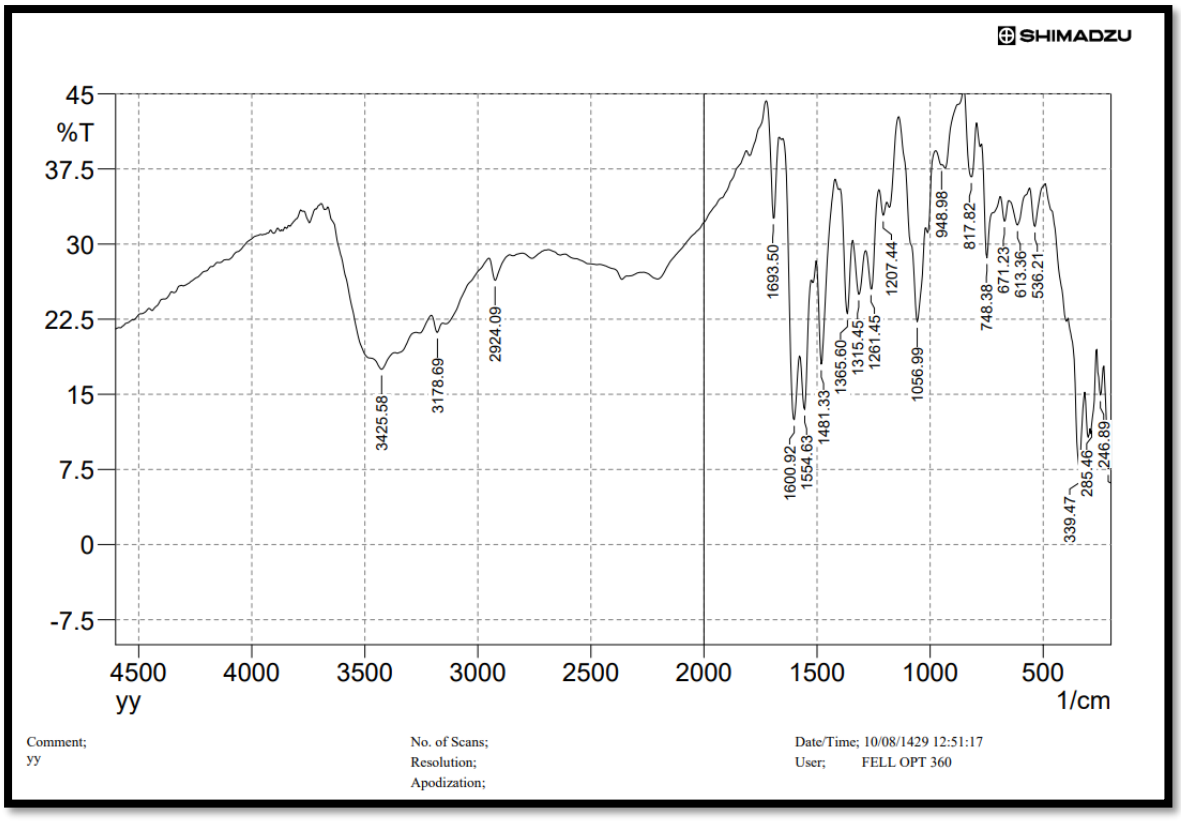
M-N	S-H	C=N) υ(imine cm^{-1}	C-S-C)υ (cm^{-1}	N-C	C=C
$C_9H_6N_3BrS_2$	2924.09	1608	1481	1327	1693
$[Cr(L_4)Cl_3]$	2924	1663	1485	1311	1554
$[Co(L_4)Cl_2]$	2924	1693	1481	1315	1554



Infrared spectrum of the



Infrared spectrum of the



Infrared spectrum of the

The mass spectrum of the ligand exhibits a molecular ion peak $[M]^{+\cdot}$at 300 m/z ,the ligand spectra shows fragments at (298 ,268, 239, 220.29, 207, 193) m/z respectively as shown in figure (2).The mass spectrum of the complex $[Co(L)Cl_2]$ shows a molecular ion peak $[M]^{+\cdot}$at(298) m/z which is equivalent to molecular mass of the complex .This complex shows another a fragment ion peak with loss

The mass spectrum of the complex $[Ni(L)Cl_2]$ shows a molecular ion peak $[M]^{+\cdot}$(298) m/z which is equivalent to molecular mass of the complex. This complex shows another a fragment ion peak with loss of chlorine atom at.

The mass spectrum of the complex $[Cr(L)Cl_3]$ shows a molecular ion peak $[M]^{+\cdot}$ (298) m/z which is equivalent to molecular mass of the complex .This complex shows another a fragment ion peak with loss of chlorine atom at. The

mass spectra of the complexes shown in figures (1).

Table 3 : The main peaks for fission in electron impact mass spectrum of

Chemical formula	**m/z**
$[C_{11}H_{12}N_4S_2]$	264
$[C_9H_7N_3S_2]$	221
$[C_{10}H_{13}N_3S]$	207
$[C_8H_7N_3S]$	177
$[C_2H_2S_2N_2]$	118
$[C_7H_6N]$	104
$[C_7H_8]$	92
$[C_6H_5]$	77
$[C_6H_6]^{4\cdot}$	66
$[C_4H_3]^{5\cdot}$	51

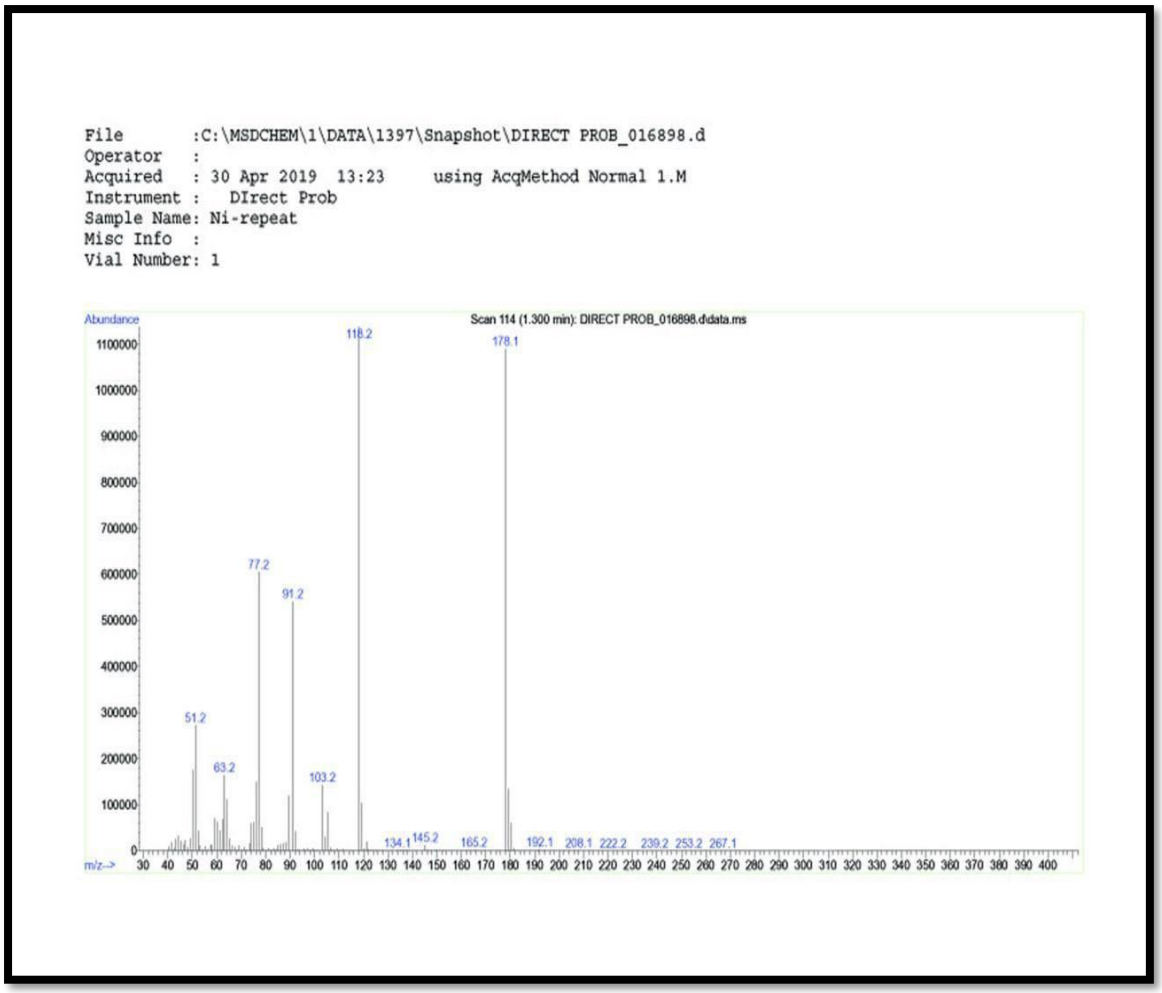
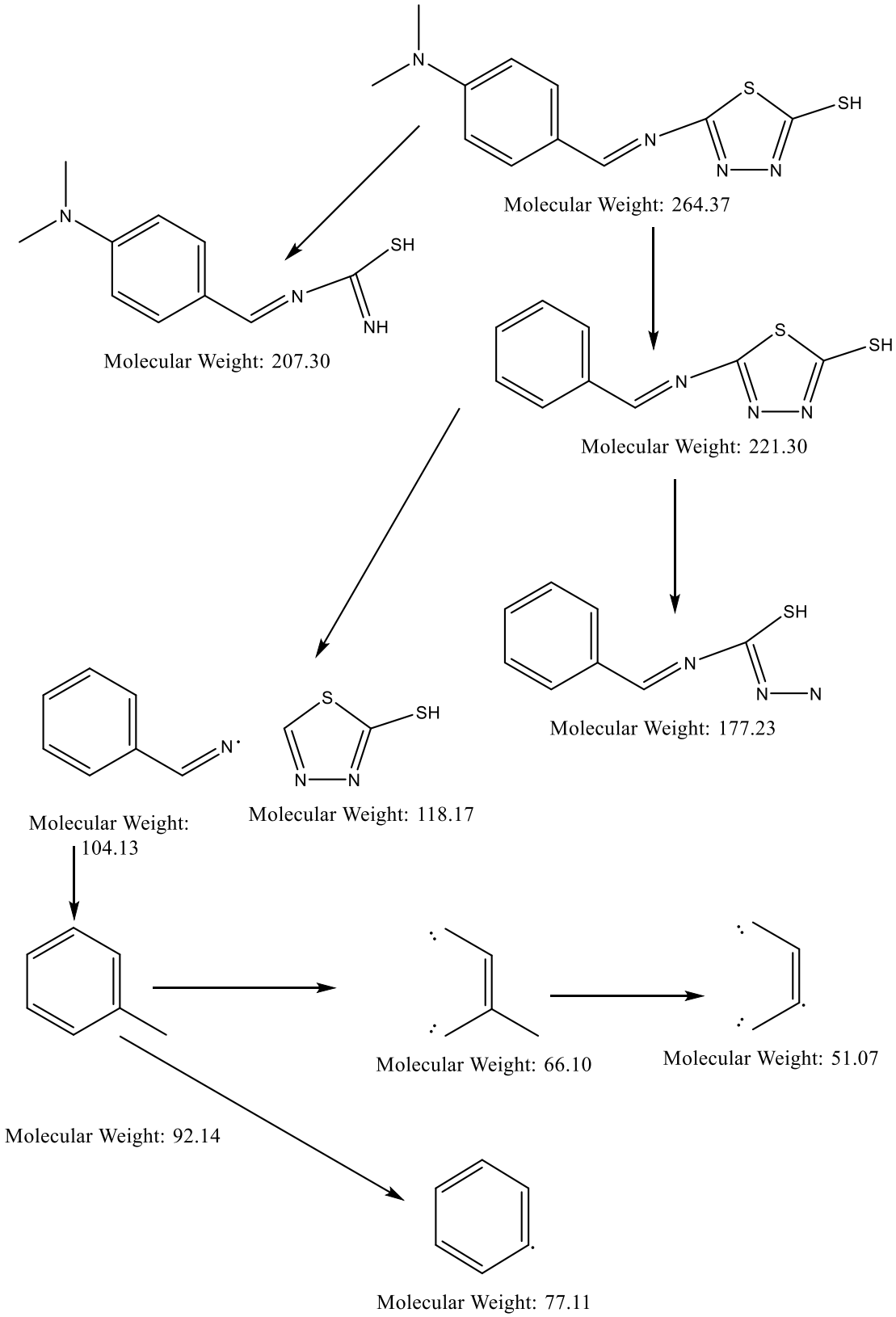


Figure 3.mass spectra of L



Scheme 1. Mass fragmentation of ligand

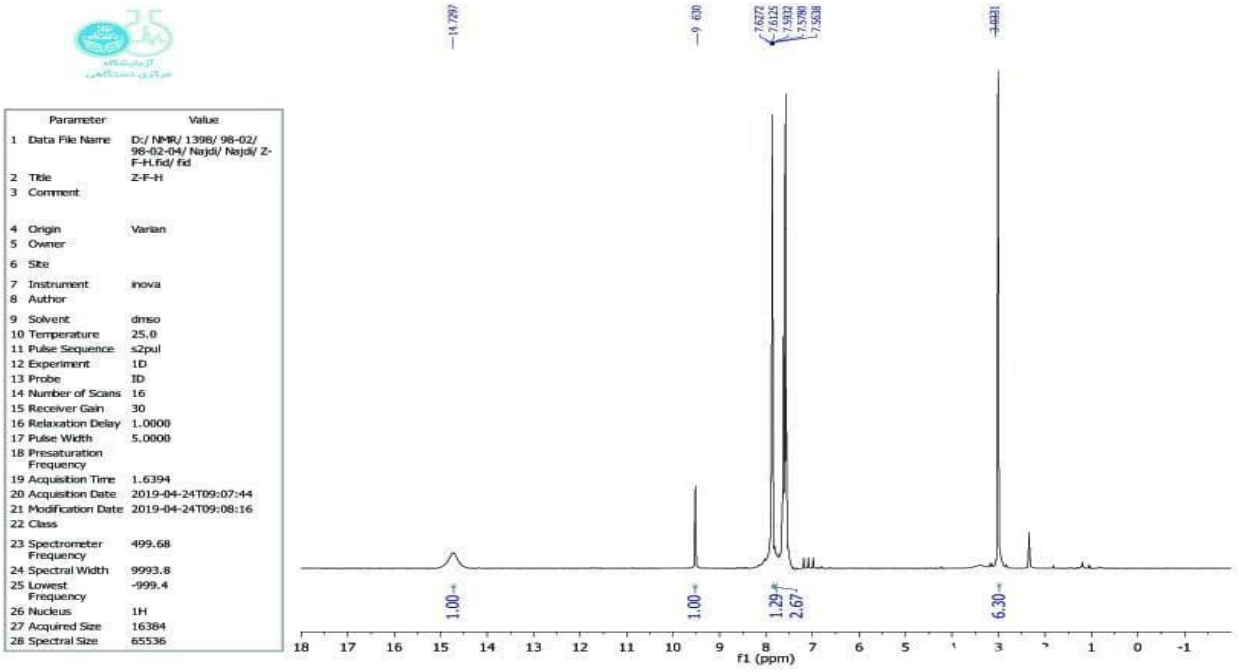


Table 4: The alignment of H[1]NMR spectrum (500 MHz, DMSO-d6,) ligand

Chemical shift δ(ppm)	Integration	Multiplicity
14.72	H	singlet
9.63	H	singlet
7.63-7.56	4H	doplete
3.03	6H	singlet

Nuclear Magnetic Resonance

The ^{1}H-NMR spectra of ligand exhibit a 14.72 ppm(1H,s,SH) 9.63ppm (H,s,NH) armatic protons, 7.63 -7.56ppm(4H,s,CH) ,3.03ppm(6H,d,CH_3) 2.5(s, DMSO). The proton NMR of the ligand shown in figure (1). ^{1}H-NMR(DMSO-d^6) spectral information was given extra support for the proposition of the structure.

Table 5 : The values of molar electrical conductivity at (M 10^{-3}) a concentration of several types of electrolytes

Electrolyte Am ($S.cm^2 .mol^{-1}$)		Non Electrolyte		Solvent	
1:4	1:3	1:2	1:1		
480	360	240	120	3.5×10^{-4} - 0.04×10^{-4}	Water
160	120	70-90	35-45	0-20	Ethanol
290-330	220-260	150-180	75-95	0-20	Nitro methane
500	340-420	220-300	120-160	0-30	Methyl cyanide
300	200-240	130-170	65-90	0-30	Di methyl formide
-	-	70-80	30-40	0-20	Di methyl sulphoxide

The magnetic properties of a compound can be determined from the electron configuration and the size of its atoms. Since magnetism is generated by electron spin, the number of unpaired electrons in a given compound indicates how magnetic the compound is. In this section, the magnetism of d-block elements (or transition metals) is evaluated. These compounds tend to have a large number of unpaired electrons the table indicates the values of electromagnetism at focus (M 10^{-3}) concentration of several types of electrolytes.

Table 6 : The values of molar conductivity and magnetic moment of ligand complexes in a solvent at a temperature of 298 K in D M S O

Complex Number	FURMULA	Am ($S.cm^2.mol^{-1}$	نسبة الالكتروليت	العزم المغناطيسي المؤثر
1		14	Non Electrolyte	3.8
2		19	Non Electrolyte	4.1

The table 6 above indicates the values of molar A_M conductivity and the effective magnetic moment$\boldsymbol{\mu}$**eff** of the ligand complexes L_1 in a solvent **DMSO** and at a concentration $\mathbf{10^{-3}}$ at a temperature 298K in a solvent **DMSO**.

Compliance with Ethical Standards statements

I. Ethical approval:

The manuscript is written in original and all the data, results pertaining to this manuscript are original according to the research performed. The authors followed academic integrity and have not copied any content/results from another source.

II. Funding details (In case of Funding):

The authors of this manuscript did not receive any funding to perform the present research

III. Conflict of interest

The authors of the study do not have any conflict of interest

IV. Informed Consent:

The authors of the manuscript agrees to publish this research in the journal if it's considerable by the editors of the journal. The authors provide full consent for reviewing and publishing this manuscript.

V. All the authors of this study contributed equally in terms of performing the research as well as in preparing the manuscript. All the authors of the study followed the guidelines of the corresponding author. Any query/suggestion related to the manuscript can be reached to the corresponding author

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