Global Journal of Science Frontier Research: B
Chemistry
Volume 15 Issue 3 Version 1.0 Year 2015
Type : Double Blind Peer Reviewed International Research Journal
Publisher: Global Journals Inc. (USA)
Online ISSN: 2249-4626 & Print ISSN: 0975-5896

Synthesis, Spectroscopic, Thermodynamic and Biological Activity Studies of Schiff Base and Metal Complexes Derived from 2-[1H-Pyrrol-2-Ylimino Methyl]- 5-Phenyl-1,3,4-Oxadiazole

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GJSFR-B Classification : FOR Code: 030306p

Strictly as per the compliance and regulations of :

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Abstract- New metal complexes of the ligand (HL) 2-[1H-Pyrrol-2-ylimino methyl]- 5-phenyl-1, 3,4-oxadiazol with the metal ions Co(II), Ni(II) and Cu(II), were prepared in alcoholic medium. The Schiff bases were condensed by using [Pyrrolcarboxaldehyde] with[2-amino-5-(phenyl-1, 3,4-oxadiazole] in alcoholice medium. As the Schiff base prepared was tridentate ligand, it was used for forming complexes with Co^{+2}, Ni^{+2}, Cu $^{+2}$ and Zn^{+2} ions of type M $(HL)_2$. All the synthesized Schiff base and their metal complexes were characterized by FTIR Spectroscopy, Electronic Spectroscopy, Elemental Analysis, Magnetic Susceptibility Measurements, Thermal Analysis , 1H-NMR Spectra, and Mass Spectra. The Activation Thermodynamic Parameters, such as ΔE*, ΔH*, ΔS* and ΔG*were calculated from the TGA curve using Coats-Red fern method. From the spectral measurements, structures for the complexes were proposed. Preliminary in vitro tests for antimicrobial activity showed that all prepared compounds displayed good significant activity to *Staphylococcusaureus, Escherishia coli, Pseudononasaeroginosa and Cndidaalbicans.*

Keywords: schiff base, microwave synthesis, thermodynamic parameters, biological activity.

I. Introduction

Increasing physiological importance of donor organic compounds including nitrogen and oxygen have active roles played by certain coordinated metal ions. These compounds have magnificent characteristics in synthesizing and studying structural aspects of metal complexes with some oxygen and nitrogen donor ligands.

Literature survey reveals that out of various isomers particularly 1,3,4-oxadiazole derivatives exhibit wide range of biological activities. Various methods for the synthesis of 1,3,4-oxadiazole have also been reported[1-4]. These biological activities are probably due to the presence of the –N=C–O group [5]. Pyrrole, oxadiazol and its derivatives form an important class of organic compounds due to their chemical structure and biological activities as analgesic, antipyretics and anti-inflammatory [6]. Even the simplest Pyrrole derivatives are widely used for analgesic medicines. Pyrroles are efficient extractants of metal ions and they have potential to form different types of coordination compounds. In addition, a pyrrolecan form a variety of Schiff bases and are reported to be superior reagents in biological, clinical and analytical applications [8,7]. The present study confines itself to study the metal complexes of Schiff bases encompassing some new metal ions such as Co(II), Ni (II) , Cu (II) and Zn (II), derived from Pyrrole and 2-amino-5-(2-hydroxy-phenyl-1,3,4-oxadiazole. Preparation, characterization and antibacterial activity of above metal complexes with this Schiff bases are reported here. Where, HL is a Schiff base of 2-amino-5-(2-hydroxy-phenyl-1,3,4-oxadiazolre acting with Pyrrole along with complexes with some oxygen and nitrogen donor ligands [9]. From the TGA curves recorded for the successive steps in the decomposition process of these ligands and complexes, it was possible to determine the following characteristic of the thermal parameters for each reaction step: initial point temperature of decomposition (T_i) stands for initial temperature point at which TG curve starts deviating from its base line. Final point temperature of decomposition (T_f) at which TG curve returns to its base line. Peak temperature, i.e. temperature of maximum rate of weight loss: the point obtained from the intersection of tangents to the peak of TG curve, whereas (DM) stands for the mass loss at the decomposition step which is the amount of mass that extends from the point T_i up to the reaction end point T_f on the TG curve, i.e. the magnitude of the ordinate of a TG curve. The material released at each step of the decomposition is identified by attributing the mass loss (Dm) at a given step to the component of similar weight calculated from the molecular formula of the investigated complexes, comparing with literatures of relevant compounds considering their temperature. This may assist identifying the mechanism of reaction in the decomposition steps taking place in the complexes under question. Activation energy (E) of the composition

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step: the integral method used is the Coats-Redfern equation [10]. For reaction order $n \neq 1$or $n = 2$, which when linearised for a correctly chosen n yields the activation energy from the slop:

$$\log\left[\frac{1-(1-\alpha)^{1-n}}{T^2(1-n)}\right] = \log\frac{ZR}{qE}\left[1-\frac{2RT}{E}\right] - \frac{E}{2.303RT} \quad \ldots\ldots n \neq 1$$

$$\log.\left[\frac{-\log.(1-\alpha).}{T^2}\right] = \log.\left[\frac{AR}{\beta E}(1-\frac{2RT}{E}\right] - \frac{E}{2.303RT} \quad \ldots\ldots n = 1$$

$\Delta S^* = 2.303R\ [\text{Log}\ (Ah/K\ T_{max})]$, $\Delta H^* = E - RT_{max}$, $\Delta G^* = \Delta H^* - T_{max}\ \Delta S^*$where: α = fraction of weight loss, T = temperature (°K), n = order of reaction, A or Z = pre-exponential factor, R = molar gas constant, E = activation energy and q = heating rate. Order of reaction (n) is the one for which a plot of the Coats-Redfern expression gives the best straight line among various trial values of n that are examined relative to that estimated by the Horovitz-Metzger method [11].

II. EXPERIMENTAL

All chemicals used were of reagent grade (supplied by either sigma Aldrich or fluka) and used as supplied. The FTIR spectra in the range (4000-400) cm^{-1} cut were recorded as KBr disc on FTIR.4200 Jasco Spectrophotometer. The UV-Visible spectra were measured in ethanol using Shimadzu UV-Vis. 160 A-Ultra-violet Spectrophotometer in the range (200-1000) nm. Magnetic Susceptibility measurements for complexes were obtained at room temperature using (Magnetic Susceptibility Balance) Jhonson Mattey Catalytic Systems Division. Gall encamp M.F.B600.010 F melting point apparatus were used to measure the melting point of all the prepared compounds. Elemental microanalysis was carried out using CHNO Elemental Analyzer Model 5500 Carlo-Elba Instruments (Italy).

a) *Synthesis of [2-amino-5-(2-hydroxy-phenyl-1,3,4-oxiadiazole]*[12] *[L]*

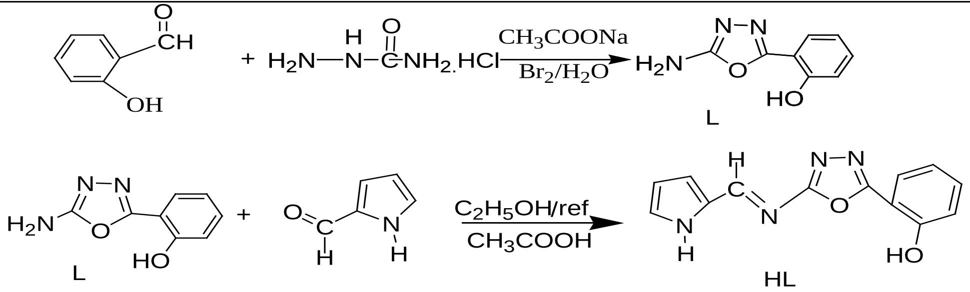
A. Synthesis Semicarbazone: Semicarbazide Hydrochloride (0.1M) and sodium acetate (0.2M) were added and dissolved in 15-20mL of distilled water placed in flat-bottomed flask. In a separate beaker containing required Salicylaldehyde (0.1M) was dissolved in free alcohol. This ethanolic aromatic aldehyde solution was added slowly to the solution of semicarbazide hydrochloride. The precipitate, which was separated, was filtered, dried and recrystallized from 95% hot ethanol.

B. Semicarbazone (0.1M) and sodium acetate (0.2M) were dissolved in 300-400 mL of glacial acetic acid with continuous stirring. Bromine (7 mL in 50 mL of GAA) was added slowly to it. The solution was stirred for an hour and then poured on crushed ice the resulting solid was separated, dried and recrystallizedfrom hot ethanol (95%). Thetable (1)showsC.H.N.S analysis of the yielded.

b) *-2-[1H-Pyrrol-2-ylimino methyl]- 5-phenyl-1,3,4-oxadiazol*[13] *[HL]*

Method (1): A mixture of equal molar amounts (0.05 mol.) of both appropriate [Pyrrolcarboxaldehyde] and the [2-amino-5-(2-hydroxy-phenyl-1,3,4-oxiadiazole], in absolute ethanol (25 ml) with (3) drops of glacial acetic acid was refluxed (4)hours. The reacted mixture was then allowed to cool at room temperature, and the precipitate was filtered, dried, and recrystallized from mixture (methanol and ethanol) (1:1) to give yellow powder.

Method(2): A mixture of equal molar amounts (0.05 mol.) of appropriate [Pyrrole-2-carbaldehyde] and the (2-amino-5-(2-hydroxy-phenyl-1,3,4-oxadiazole), were ground with a mortar, mixed, dried and subjected to microwave irradiation 280W for (10) minutes, after completion the reacted mixture was cooled to room temperature, then the solid obtained was recrystallized twice from mixture (methanol and ethanol) (1:1) to get yellow powderyield. The table (1) shows C.H.N.S analysis of the yielded.



Scheme 1

III. PREPARATION OF COMPLEXES

Method(1) : An ethanol solution of the metal salts of Co(II), Ni (II), Cu (II) and Zn (II) was added to an ethanolic solution of (HL) in 1:1 (metal : ligand) molar ratios. After stirring for 2 hours with heating 70 °C, crystalline colored precipitates formed cooling at room temperature, the resulting solids were filtered off, washed with distilled water, dried and recrystallized from ethanol and dried at 50 °C.

Method(2) : An ethanol solution of the metal salts of Co(II), Ni (II), Cu (II) and Zn (II)were added to an ethanolic solution of (HL) in 1:1 (metal : ligand) molar ratios. Then it was put in ultrasonic bath heated to 65^{0}C After 50 minutes crystalline colored precipitates formed, cooling at room temperature, the resulting solids were filtered off, washed with distilled water, dried and recrystallized by using ethanol and dried at 50 ^{0}C. Thetable (1) shows C.H.N.S analysis of the yielded.

IV. RESULT AND DISCUSSION

The synthetic procedure of Schiff base ligand is presented in Scheme 1. Then, the divalent transition metal ions *viz.*, Co (II), Ni (II), Cu(II) and Zn(II) reacted with the ligand. The composition of the complexes formed in solution has been established by mole ratio and job methods. In both cases, the results reveal (2:2) metal to ligand ratio yielded the corresponding metal chelates. Shows the decomposition point, color and electronic absorption bands for ligand and complexes are shown in Table (1, 2). The bands are classified into three distinct groups: The intermolecular transitions appeared in the UV region, charge transfer from ligand to metal, and d-d transitions appeared in the UV-Visible region.

a) *[2-amino-5-(2-hydroxy-phenyl-1, 3,4-oxiadiazole] [L]*

The reaction of Semi-carbazide Hydrochloride with Salicylic aldehyde in presence of sodium acetate/Br_2 afforded 2-amino-5-phenyl-1, 3,4-oxiadiazole [11]. The structural assignment of the product was based on its melting point and spectral (FT-IR and UV/Vis.) data as shown in table (1). The FT-IR spectrum of compound (L) exhibited significant two bandshaving the range (3402–3213)cm^{-1} which could be attributed to asymmetric and symmetric stretching vibrations of the NH$_2$ group. Stretching vibration band of (OH) occurs in the (3428) cm^{-1}. Besides this, band ranges at about (1475-1423 cm^{-1}) due to cyclic (C = N) stretching is also observed. Other bands occurring at (1518 cm^{-1}) and (1484 cm^{-1}) are due to the (N-H) bending and (C-N) stretching vibrations, respectively [14].

b) *[2-[(1H-Pyrrol-2-ylimino methyl)]- 5-(2-hydoxy-phenyl)-[1,3,4-oxiadiazol] [HL]*

The FT-IR spectra shows the disappearance of the two absorption bands due to (-NH2) stretching of amino oxadiazole [HL] showed all the suggested bonds for olefin (C-H), (C=C) aromatic, endocyclic (C=N) and exocyclicimine group. All the prepared compounds (Schiff bases) exhibited the stretching band near the region (1475-1423) cm^{-1}, this is due to the (=N-N=C-) cyclic group; 3426 cm^{-1} (υ OH Stretching), a band at 3155 cm^{-1} attributed to NH stretching (pyrrole ring), 1595cm^{-1} (υ C=N Stretching of amine), 1229 cm^{-1}, 1468 cm^{-1} (Characteristic bands of oxazole ring).All the spectral data for other compounds are listed in table (2).

^{1}H-NMR spectrum of compounds [HL], shows the following characteristic chemical shift, (CDCL3-d$_6$) ppm. The four aromatic ring protons of phenyl (δ 6.95 - 7.55) (s, 4H, Ar), and three pyrrole ring appeared at (δ 6.00 – 7.45) ppm, the signal at (δ 8.78)was attributed to (N=C-H) proton (azomethine). Beside the signal at (δ 9.56) ppm, was attributed to (N-H) proton, δ=11.95(s, 1H, OH),δ 1.584(organic solvent).

The positive ion mass spectral analysis of (HL) observed at m/z 255. (M+1) (Figure5), confirms the theoretical molecular weight i.e. 254.1.

Table 1 : The characterization data of the prepared compounds

Compound Formula,	Yield %	C	H	N	O	Cl	M
L $C_8H_7N_3O_2$	70	54.60 (54.24)	3.82 (3.98)	23.39 (23.72)	18.19 (18.06)	---	--
HL C13H10N4O$_2$	81	61.74 (61.41)	3.91 (3.96)	22.15 (22.04)	12.65 (12.59)	---	--
$[Co_2\ (HL)_2.4(H_2O)]Cl_2$	72	40.94 (40.81)	3.22 (3.16)	14.85 (14.64)	16.91 (16.73)	9.13 (9.27)	14.95 (15.40)
$[Ni_2\ (HL)_2.4(H_2O)]\ Cl_2$	67	40.96 (40.83)	3.12 (3.16)	14.99 (14.65)	16.57 (16.74)	9.20 (9.27)	15.16 (15.35)
$[Cu_2\ (HL)_2.4(H_2O)]Cl_2$	65	40.49 (40.32)	3.11 (3.12)	14.31 (14.47)	16.69 (16.53)	9.03 (9.15)	16.37 (16.41)
$[Zn_2\ (HL)_2.4(H_2O)]Cl_2$	55	40.32 (40.13)	3.17 (3.11)	14.25 (14.40)	16.22 (16.45)	9.23 (9.11)	16.94 (16.81)

Table 2 : some physical data electronic spectra for ligands and complexes in DMF

Symbol	Dec. Point ^{0}C	Conductivity $ohm^{-1}cm^{2}mol^{-1}$	MagneticMoment (B(B.M)	Color	Absorption Bands (nm)	AssignedTransition
L	244	-	-	White-yellow	209	$\pi \rightarrow \pi^*$
					285	$n \rightarrow \pi^*$
HL	285	--	-	yellow	215	$\pi \rightarrow \pi^*$
					390	$n \rightarrow \pi^*$
Co(II)	295d	146	4.78	Pale -Red	775	$^{4}T_{1}g \rightarrow {}^{4}A_{2}g$
					545	$^{4}T_{1}g^{(F)} \rightarrow {}^{4}t_{1}g^{(p)}$
					350	Charge Transfer
Ni(II)	300d	136	3.22	Pale green	230	$\pi \rightarrow \pi^*$
					290	$n \rightarrow \pi^*$
					375	Charge Transfer
					650	$^{3}A_{2}g \rightarrow {}^{3}t_{1}g^{(p)}$
					940	$^{3}A_{2}g \rightarrow {}^{3}t_{1}g^{(F)}$
Cu(II)	300d	139	1.92	Light Brawn	225	$\pi \rightarrow \pi^*$
					290	$n \rightarrow \pi^*$
					370	Charge Transfer
					425, 610,640	$^{2}B_{1}g \rightarrow {}^{2}B_{2}g$
Zn(II)	300d	161	Dia	Purple White	315	Charge Transfer

V. Infrared Spectral Analysis of Metal Complexes

The infrared spectra of the ligands show (υO-H) (weakly H-bonded) at 3429cm^{-1} and (υN-H) at 3151 cm^{-1}. The absence of this band in all the metal complexes indicates the removal of a proton of hydroxyl group of benzene ring and a proton of NH group of pyrrole ring during the chelation. The sharp intense band at 1593 cm-1 in the ligands can be assigned to υC=N (azomethine). A shift ∆ υ = 7-15 cm^{-1}) in υC=N (azomethine) is observed upon the coordination indicating that the nitrogen of azomethine group is involved in coordination. All the complexes show broad band in the region(3285-3378) cm^{-1} which may be assigned to (υ O-H) of coordinated water [15]. To account for the octahedral stereochemistry of the metal complexes, the coordination of two water molecules is expected.

The bands at 561cm^{-1}in Co(II) complexes, 579 cm^{-1} in Ni(II) complex , 588 cm^{-1} in Zn(II) complex and 587cm^{-1} in Cu(II) complex may be due to metal-nitrogen stretching vibration[16,17]. In the free ligand, the band at 1606 cm^{-1} is assigned to the stretching of C=N(oxazole ring). On complexes, this band was shifted to a lower frequency region. This shift is probably due to the lowering of bond order of the carbon-nitrogen bond resulted in forming complexes of the metal ion to the ligand through nitrogen in (υC=N) compared to its respective ligands. This suggests that the nitrogen atom of the ring has not participated in the chelation. However, in water containing chelates, this band is observed as a broad band. This may be due to coupling of the bending mode of coordinated [18].

Table 3 : Infrared data of Ligand and its metal complexes (cm^{-1})

Symbol	ν(C=N)	ν(N-H) pyrrol	ν(C-N=N-C)	ν(M-O)	ν(H2O)	ν(O-H)	ν(M-N)
HL	1594(s)	3155	1475-1423	-	-	3426	-
Co(II)	1610(s)	-----	1490-1429	477(s)	3275	-	561(s)
Ni(II)	1617(s)	-----	1485-1430	444(s)	3281	-	579(s)
Cu(II)	1603(s)	-----	1484-1427	437(s)	3241	-	588(s)
Zn(II)	1611(s)	-----	1460-1430	447(s)	3255	-	587(s)

VI. Thermal Analysis

To understand thermal decomposition process, the Schiff base and its metal complexes were examined by thermo gravimetric analysis in the temperature range of 35–700 ºC. The obtained thermo analytical results from TGA curves for all these compounds which are given in table (4). The decomposition was completed at 693 ºC for all the complexes. The data from the thermo gravimetric analyses indicated that the decomposition of the complexes (three steps) and the ligand proceeds in (two) steps. The final decomposition products were metal oxide mixture formed above 598 ºC for the metal[19].

Table 4 : Thermodynamic parameters of the ligands and metal complexes

Sample (step)	T.range ºC	N	R^2	T_{max} ºK	Ea K.J mol-1	Δ H^* KJ mol^{-1}	$ZSec^{-1}$ $x10^5$	Δ S^* J $mol^{-1}K^{-1}$	Δ G^* KJ mol^{-1}
HL(1)	37-300	1	0.99	512.79	170.4906	166.2344	1.7762	-34.0617	183.700
HL(2)	300-700	0.9	0.99	780.77	-7.87418	-14.3548	4.82	-354.698	262.5828
Co(1)	37-368	0.9	0.99	476.4	31.57044	27.61632	7.25	-289.758	165.6572
Co(2)	368-467	0.9	1	643.09	-6.84804	-12.1857	5.6	-351.864	214.094
Co(3)	368-700	0.9	0.99	766.6	-6,58243	-13.0282	6.35	-352.37	260.6221
Ni(1)	37-150	0.9	0.99	384	19.3764	16.1892	0.00381	-312.457	136.1727
Ni(2)	150-390	0.9	0.99	507	36.5	32.2919	0.017	-283.109	175.828
Ni(3)	390-700	0.9	0.99	775.12	-11.19	-17.6235	35.4	-257.205	259.2532
Cu(1)	37-180	0.9	0.99	423	125.9	46.49	7.68	-222.79	135.39
Cu(2)	180-395	0.9	0.99	554	54.214	121.04	0.5357	-100.31	169.81
Cu(3)	395-700	0.9	1	726.35	12.696	-5.734	4.91	-359.81	306.7
Zn(1)	37-190	0.9	0.99	480.8	23.71	19.72	$1.5x10^{-4}$	-321.9	174.52
Zn (2)	190-450	0.9	0.99	550	98.17	92.77	258.69	-205.2	226.16
Zn (3)	450-700	0.9	0.99	819	-10.14	-17.78	$3.7x10^{-6}$	-358.3	311.69

VII. Biological Activity

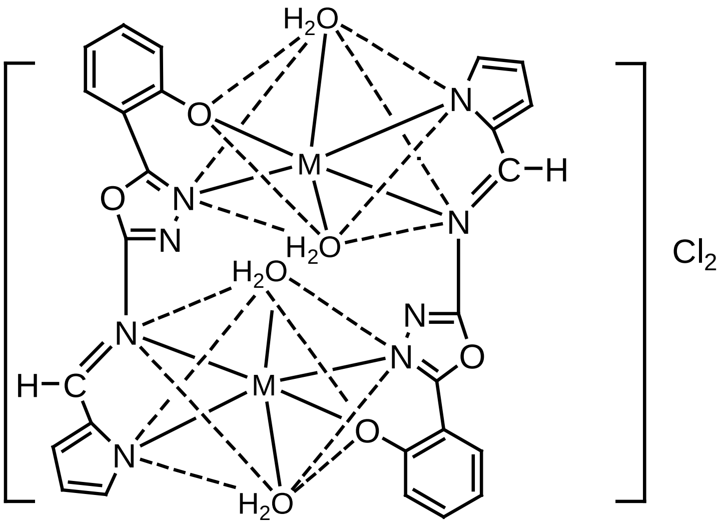
With a view to explore the possibility of obtaining biologically useful complexes that contain 1,3,4- oxadizole and pyrrole ring system, such a biological activity encourages us to prepare some new series of compunds containing the above mentioned unit. The antimicrobial activity of these compounds was determined by the agar diffusion method[20]. These types of bacteria Staphylococcus aureus, Escherishia coli, Pseudononasaeroginosa and Cndidaalbicans were used to show the biological activities of the ligand and its complexes. In this method, a standard 5mm diameter sterilized filter paper disc impregnated with the compound. Then, (1 mg per 1 ml of acetone) was placed on an agar plate seeded with the test organism. The plates were incubated for 24 hours at 37 ºC. The zone of inhibition formed was measured in mm and are represented by (+), (+ +) and (+ + +) depending upon the diameter and clarity, as presented in table (5).The preliminary screening results reveal that the compound contained thiadizole and pyrrolecomplexes exhibits highest antibacterial activity against Escherishia coli.

Table 5 : Antibacterial activity of the prepared compounds

Symbol	*Staphylococcus aureus*	*Escherishia coli*	*Pseudononas aeroginosa*	*Cndidaalbicans*
HL	+	+ + +	+	+
Co(II)	+	+ + ++	++	++
Ni(II)	+	+ + +	++	+
Cu(II)	++	+ ++	+++	++
Cu(II)	++	+ +	+++	++

Note (-) = no inhibition, (+) = (5-10) mm, (+ +)=(11-20) mm, (+ + +) = more than (20)mm

From the FTIR Spectroscopy, electronic spectroscopy, Elemental analysis, magnetic susceptibility measurements, thermal analysis suggest the structure.



[M2L2.4H2O]Cl2 ,M= Co (II), Cu (II), Zn (II)

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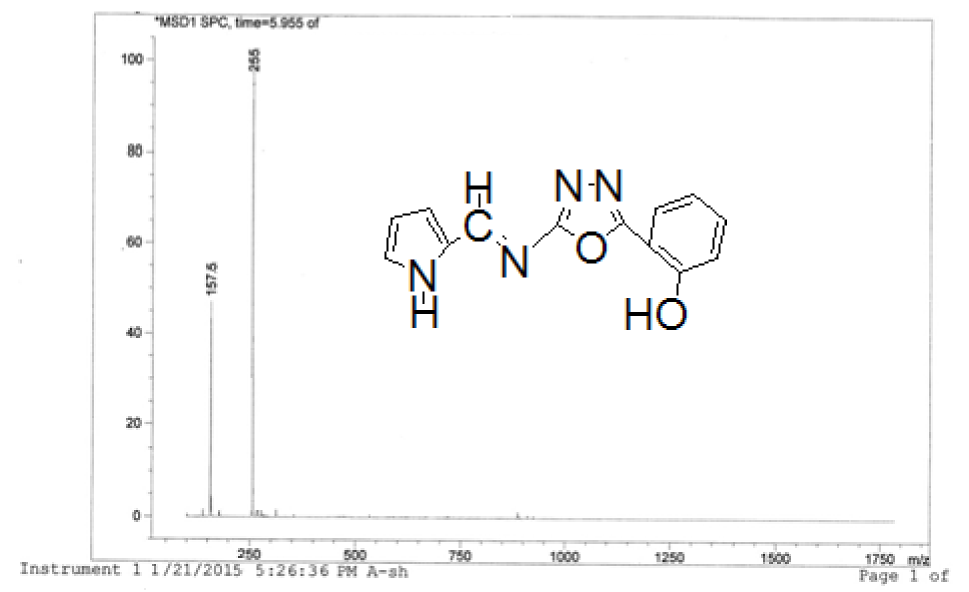
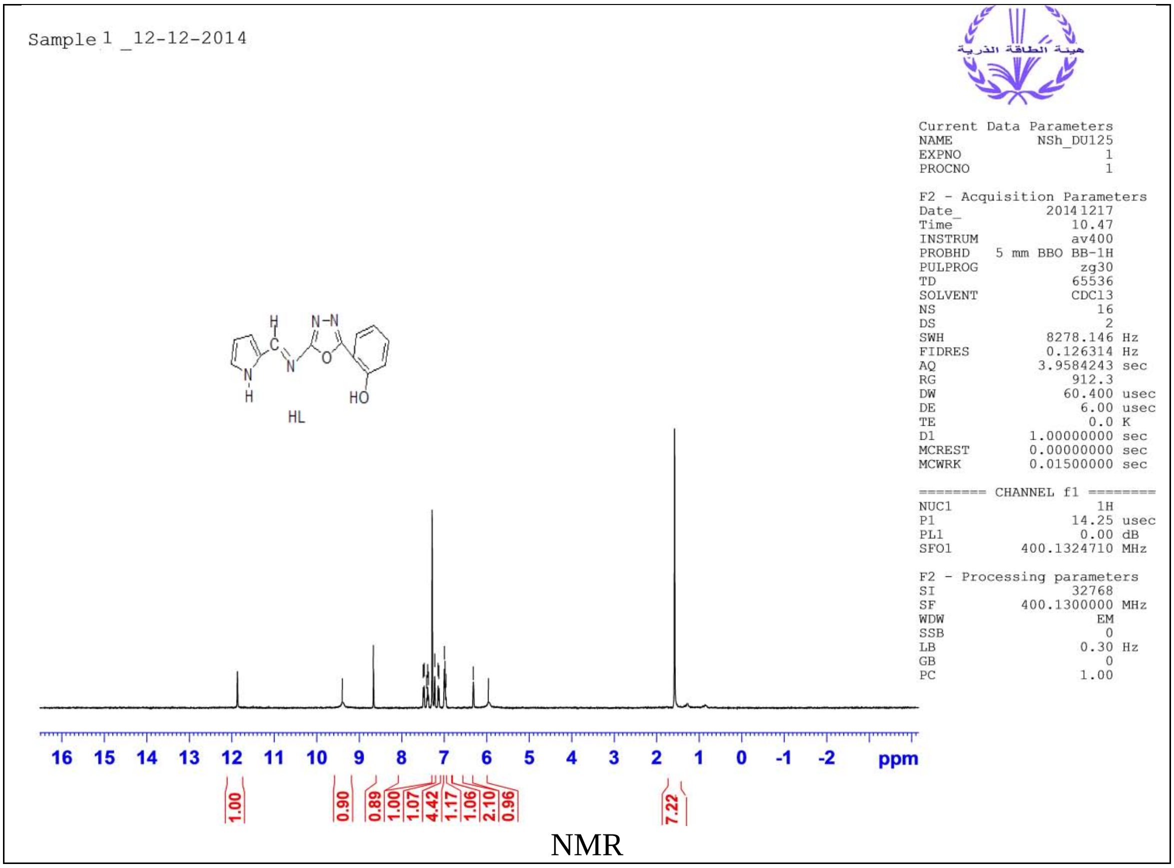


LC-MS



```
Current Data Parameters
NAME             NSh_DU125
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20141217
Time                 10.47
INSTRUM              av400
PROBHD   5 mm BBO BB-1H
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8278.146 Hz
FIDRES            0.126314 Hz
AQ               3.9584243 sec
RG                   912.3
DW                  60.400 usec
DE                    6.00 usec
TE                     0.0 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.25 usec
PL1                   0.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                   32768
SF             400.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

NMR