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Synthesis and Characterization of Some New 1,3-Oxazepine Derivatives Containing Pyrazolone Moiety *Via* [2+5] Cycloaddition Reaction

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Abstract:

This work included synthesis of some new 1,3-oxazepine derivatives starting from 4-Amino-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-one [1]. Firstly, compound [1] was converted to the corresponding imine derivatives [2] and [3] through condensation reaction with each 4-Hydroxy-3-methoxybenzaldehyde and 2-Hydroxy-3-methoxybenzaldehyde, respectively, in presence of glacial acetic acid in absolute ethanol. Imine derivatives [2] and [3] were then introduced in [2+5] cycloaddition reaction with each Phethalic anhydride, 3-Nitrophthalic anhydride and Maleic anhydride, respectively, in dry benzene to give 1,3-oxazepine derivatives [4-9]. These new derivatives might have some biological activity.

The structures of all prepared compounds were confirmed by C.H.N. elementary analysis and FT-IR spectra. Also ^{1}H-NMR spectroscopy was used to identify structure of one new compound.

الخلاصة:

تضمن هذا العمل تحضير مشتقات 1،3-اوكسازبين جديدة من المركب 4-أمينو-1،5-ثنائي مثيل-2-فنيل-1H-بايرازول-3(H2)- ون [1]. في البداية، تم تحويل المركب [1] الى مشتقي الأيمين المقابلين [2] و[3] من خلال تفاعل تكاثف مع كل من 4-هيدروكسي-3-ميثوكسي بنزالديهايد و2-هيدروكسي-3-ميثوكسي بنزالديهايد، على التوالي، بوجود حامض الخليك الثلجي كعامل مساعد في الايثانول المطلق. بعد ذلك تم ادخال مشتقات الايمين المحضرة [2] و[3] في تفاعل الاضافة الحلقية [2+5] مع كل من انهدريد الفثاليك و3-نتروانهدريد الفثاليك و انهدريد الماليك، على التوالي، في البنزين الجاف فتم الحصول على مشتقات 3،1-اوكسازبين [4-9]. ان هذه المشتقات الجديدة قد تمتلك فعالية بايولوجية.

شخصت تراكيب جميع المركبات المحضرة بوساطة التحليل الكمي الدقيق للعناصر (C.H.N.) وأطياف الاشعة تحت الحمراء. كما تم استعمال مطيافية الرنين النووي المغناطيسي للبروتون في تشخيص مركب واحد من المركبات الجديدة.

Introduction:

[2+5] Cycloaddition reaction is recently used for the synthesis of 1,3-oxazepine ring[1-4]. This reaction differs from the classical methods that were used to synthesis oxazepines, since it is not limited and produces various 1,3-oxazepine ring derivatives[5,6]. This reaction passes through cyclic transition state involves imine group as two-membered component and cyclic anhydrides as five-membered component[7,8]. Oxazepine derivatives showed various biological activities such as antibacterial[9]and inhibitors for some enzymes action[10]. Some of oxazepine derivatives are used in another applied fields[11].

Pyrazole derivatives play a vital role in many biological processes and synthetic drugs[12]. Pyrazolones are the most important derivatives of pyrazole[13]. Pyrazolone derivatives exhibit a wide variety of potentially useful applications including biological, clinical and pharmacological[14,15].

Experimental:

1. Materials

All materials have been used as provided from commertial supplier (BDH) except benzene which was purified:{4-Amino-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-one}, 4-Hydroxy-3-methoxybenzaldehyde, 2-hydroxy-3-methoxybenzaldehyde, Absolute ethanol, Glacial acetic acid, Phthalic anhydride, 3-Nitrophthalic anhydride, Maleic anhydride, Benzene and Ethyl acetate.

2. Apparatus

1. Melting points were determined by stuart melting point apparatus.
2. Elemental analysis measured on E.A.300, Euro, Italy, 2003-AL-albayt University (Jordan).
3. FT-IR spectra were recorded on FT-IR 8400s, schimadzu- spectrophotometer and using KBr discs-Kerbala University.
4. ^{1}H-NMR spectra were recorded on J 10631 C:\ Burker \ TOPSPIN 500 MHZ using tetramethyl silane as internal standard and DMSO as solvent. Measurements were made at Tahran University (Iran).

3. Preparation Methods

Synthesis of 4-(4-Hydroxy-3-methoxybenzylideneamino)-1,5-dimethyl-2- phenyl -1H-pyrazol-3(2H)-one [2]

4-Amino-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-one [1] (0.203g, 0.001mol) was dissolved in absolute ethanol (10mL), then 4-Hydroxy-3-methoxybenzaldehyde (0.152g, 0.001mol) was dissolved in absolute ethanol (10mL). Then, a drop glacial acetic acid was added to the aldehyde solution which was then added drop wise to the amine solution under reflux with stirring on water bath at 70ºC for 2hrs. Then, the mixture was allowed to cool down to room temperature. The coloured precipitate was filtered and recrystallized from ethanol, Yield 79%, M.p. 176-178ºC.

Synthesis of 4-(2-Hydroxy-3-methoxybenzylideneamino)-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-one [3]

Imine derivative [3] was prepared by using the same procedure which was used for the prepare of compound [2] with the following modifications: 2-Hydroxy-3-methoxybenzaldehyde (0.203g, 0.001mol) instead of 4-Hydroxy-3-methoxybenzaldehyde, Yield 74%, M.p. 172-174ºC.

Synthesis of 4-(1,5-Dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)-3-(4-hydroxy-3-methoxyphenyl)-3,4-dihydrobenzo[e][1,3]oxazepine-1,5-ione [4]

Imine derivative [2] (0.337g, 0.001mol) and Phthalic anhydride (0.148g, 0.001mol) were dissolved in dry benzene (20mL). The reaction mixture was refluxed with stirring on water bath at 75ºC for 5hrs, the mixture was then allowed to cool down to room temperature, the coloured precipitate was filtered and recrystallization from ethyl acetate, Yield 69%, M.p. 142-144ºC.

Synthesis of 4-(1,5-Dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)-3-(4-hydroxy-3-methoxyphenyl)-6-nitro-3,4-dihydrobenzo[e][1,3]oxazepine-1,5-dione [5]

Oxazepine derivative [5] was prepared by using the same procedure which was used for the prepare of compound [4] with the following modifications: 3-Nitrophthalic anhydride (0.193g, 0.001mol) instead phthalic anhydride, Yield 72%, M.p. 151-153ºC.

Synthesis of 3-(1,5-Dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)-2-(4-hydroxy-3-methoxyphenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione [6]

Oxazepine derivative [6] was prepared by using the same procedure which was used for the prepare of compound [4] with the following modifications: Maleic anhydride (0.980g, 0.001mol) instead phthalic anhydride, Yield 65%, M.p. 134-136ºC.

Synthesis of 4-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)-3-(2-hydroxy-3-methoxyphenyl)-3,4-dihydrobenzo[e][1,3]oxazepine-1,5-ione [7]

Imine derivative [3] (0.337g, 0.001mol) and phthalic anhydride (0.148g, 0.001mol) were dissolved in dry benzene (20mL). The reaction mixture was refluxed with stirring on water bath at 75ºC for 5hrs, the mixture was then allowed to cool down to room temperature, the coloured precipitate was filtered and recrystallized from ethyl acetate, Yield 62%, M.p. 128-130ºC.

Synthesis of 4-(1,5-Dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)-3-(2-hydroxy-3-methoxyphenyl)-6-nitro-3,4-dihydrobenzo[e][1,3]oxazepine-1,5-dione [8]

Oxazepine derivative [8] was prepared by using the same procedure which was used for the prepare of compound [7] with the following modifications: 3-Nitrophthalic anhydride (0.193g, 0.001mol) instead of phthalic anhydride, Yield 71%, M.p. 139- 141ºC.

Synthesis of 3-(1,5-Dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)-2-(2-hydroxy-3-methoxyphenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione [9]

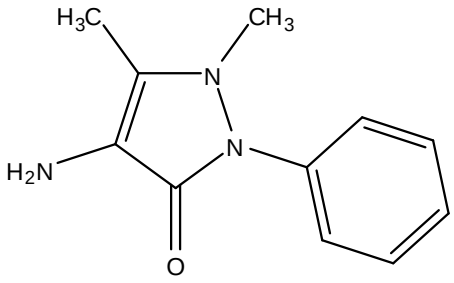
Oxazepine derivative [9] was prepared by using the same procedure which was used for the prepare of compound [7] with the following modifications: Maleic anhydride (0.980g, 0.001mol) instead of Phthalic anhydride, Yield 60%, M.p. 119-121ºC.

Table (1): Melting points, percent yields and (C.H.N.) analysis of the prepared compounds (2-9)

Comp. No.	Formula	M.Wt.	(M.P.)°C	Yield%	C.H.N. analysis					
					Calculated			Found		
					C%	H%	N%	C%	H%	N%
[2]	$C_{19}H_{19}N_3O_3$	337	176-178	79	67.65	5.63	12.46	67.33	5.82	12.66
[3]	$C_{19}H_{19}N_3O_3$	337	172-174	74	67.65	5.63	12.46	67.40	5.72	12.59
[4]	$C_{27}H_{23}N_3O_6$	485	142-144	69	66.80	4.74	8.65	67.03	4.39	9.01
[5]	$C_{27}H_{22}N_4O_8$	530	151-153	72	61.13	4.15	10.56	60.88	4.11	10.81
[6]	$C_{23}H_{21}N_3O_6$	435	134- 136	65	63.44	4.82	9.65	63.30	5.03	9.38
[7]	$C_{27}H_{23}N_3O_6$	485	128- 130	62	66.80	4.74	8.65	66.97	4.50	8.98
[8]	$C_{27}H_{22}N_4O_8$	530	139 -141	71	61.13	4.15	10.56	61.01	4.29	10.75
[9]	$C_{23}H_{21}N_3O_6$	435	119 -121	60	63.44	4.82	9.65	63.25	4.71	9.44

Results and Discussion:

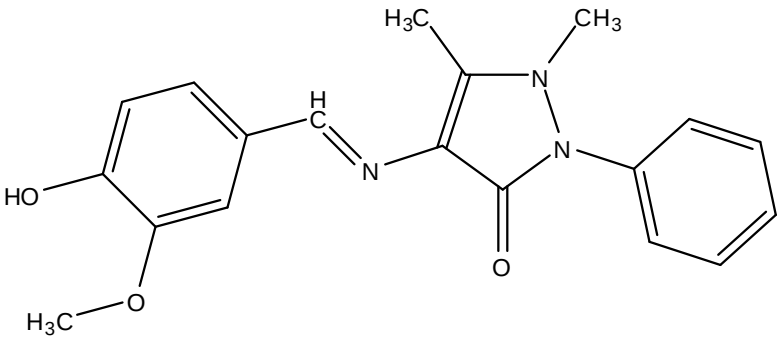
The following scheme shows the synthetic plane of this work:



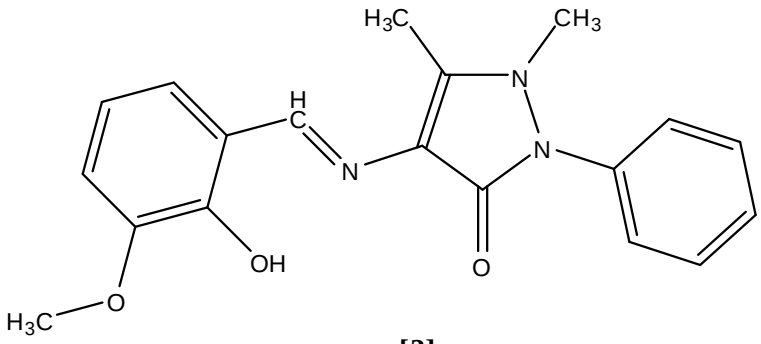
[1]

4-hydroxy-3-methoxybenzaldehyde, abs.EtOH, ref. 2hrs

2-hydroxy-3-methoxybenzaldehyde, abs.EtOH, ref. 2hrs



[2]



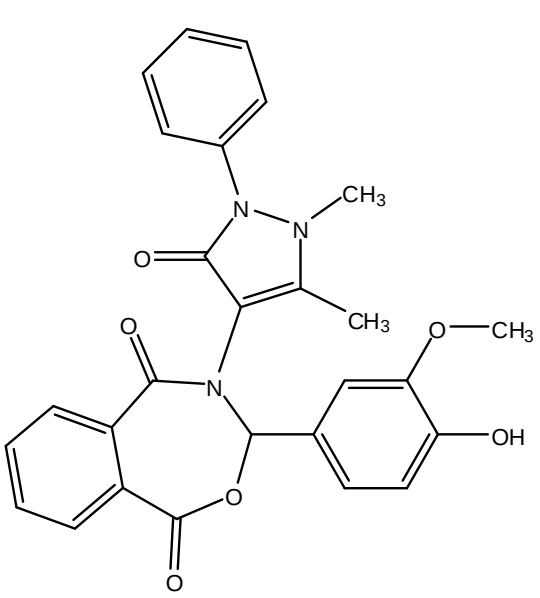
[3]

[2]

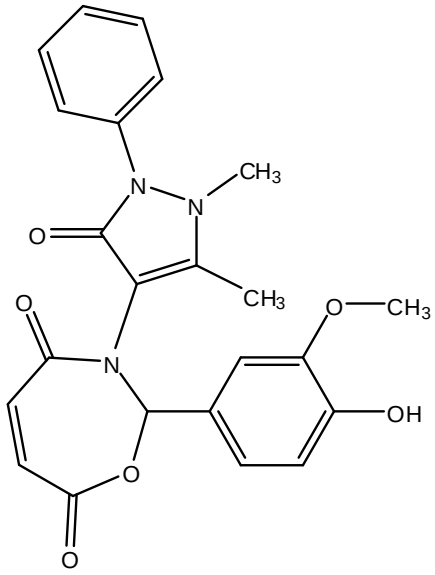
phthalic anhydride
dry benzene, ref. 5hrs

3-nitrophthalic anhydride
dry benzene, ref. 5hrs

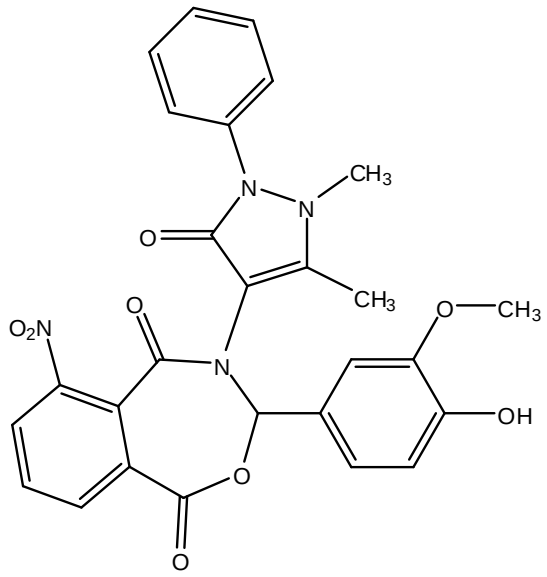
maleic anhydride
dry benzene, ref. 5hrs



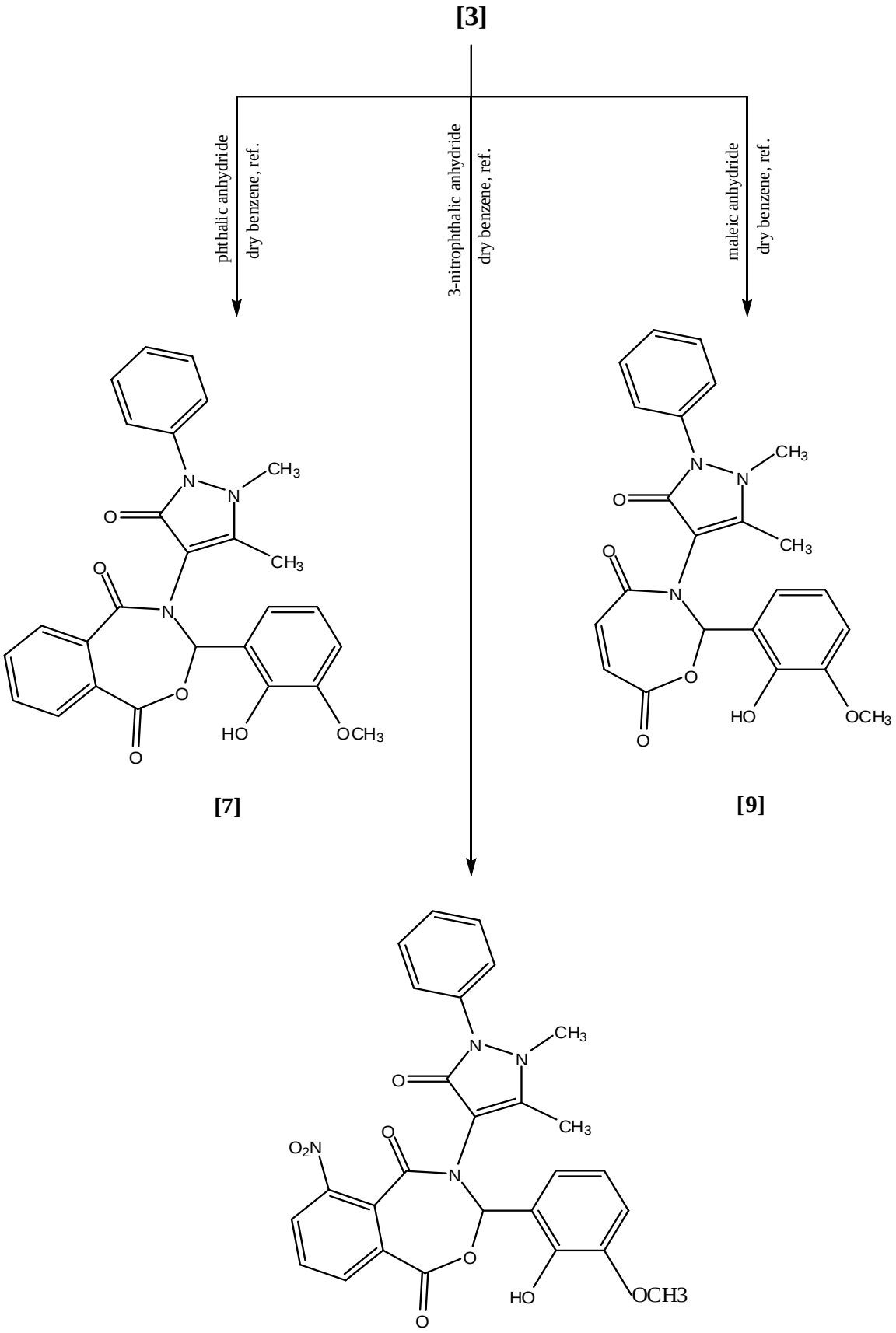
[4]



[6]

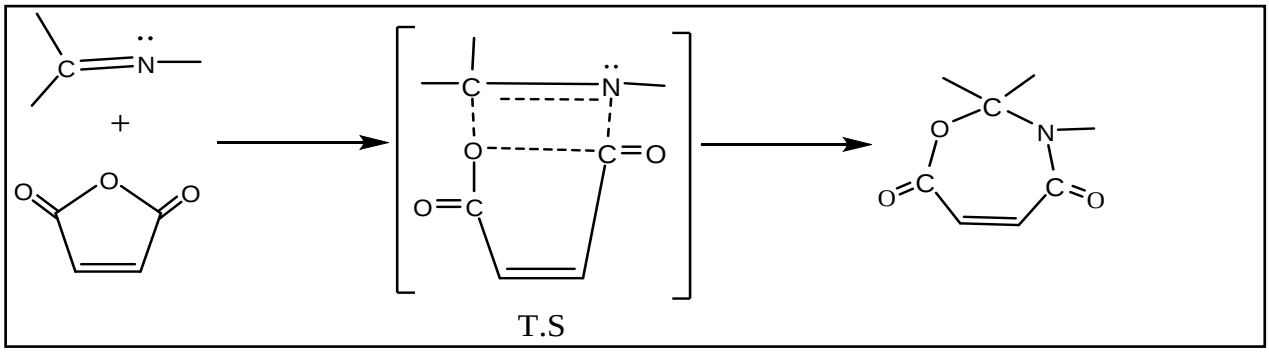


[5]



Scheme (1): ***Reactions proceeding***

The reaction of 4-Amino-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-one [1] with each 4-Hydroxy-3-methoxybenzaldehyde and 2-Hydroxy-3-methoxybenzaldehyde in presence of glacial acetic acid as catalyst resulted the formation of Schiff bases [2] and [3], respectively. The FTIR spectra of compounds [2] and [3], Figures 3 and 4 respectively, table (2), showed disappearance of the two strong absorption bands at 3420cm^{-1} and 3300cm^{-1} were due to asymmetric and symmetric stretching vibrations of (-NH_2) group, respectively, and appearance of strong band at 1626cm^{-1} and medium band at 1629cm^{-1} attributed to the υ(C=N), respectively. The weak and medium bands at 1730cm^{-1} and 1718cm^{-1} attributed the υ(C=O) of ketone, respectively. The other absorption bands for compounds [2] and [3] were listed in table (2). The elementary analysis (C.H.N.) of compounds [2] and [3], table (1), showed nearness between the calculated and found values. The reaction of the prepared imines [2] and [3] with each Phthalic anhydride, 3-Nitrophthalic anhydride and Maleic anhydride in dry benzene as solvent afforded 1,3-oxazepine derivatives [4-9]. Reaction of imines with these cyclic anhydrides was classified as [2+5] cycloaddition reaction which proceeds *via* a single transition state involves the two atoms of imine group and five atoms from cyclic anhydride to give seven-membered heterocycle as in the following scheme: [3,4]



Scheme (2): ***Approximate transition state geometry for addition of maleic anhydride to imine group***

The elementary analysis (C.H.N.) of the prepared 1,3-oxazepine derivatives [4-9], table (1), showed nearness between the calculated and found values. The FT-IR spectra of oxazepine derivatives [4-6], figures (5-7) respectively, table (2), showed disappearance of the sharp strong absorption band at 1626cm^{-1} attributed to the υ(C=N) and appearance of the following characteristic absorption bands:

Compound [4]: The medium absorption band at 1680cm^{-1} attributed to the υ(C=O) for lactam in oxazepine ring[16]. The medium absorption band at 1730cm^{-1} attributed to the stretching vibrations of (C=O) groups for lactone and ketone in oxazepine and pyrazole rings, respectively, due to the vibration coupling[16]. The medium band at 1631cm^{-1} attributed to the υ(C=C) inside pyrazole ring. The other absorption bands were listed in table (2).

Compound [5]: The weak absorption band at 1680cm^{-1} attributed to the υ(C=O) for lactam in oxazepine ring[16]. The strong band at 1730cm^{-1} attributed to the stretching vibrations of (C=O) groups for lactone and ketone in oxazepine and pyrazole rings, respectively, due to the vibration coupling[16]. The two medium absorption bands at 1545cm^{-1} and 1309cm^{-1} attributed to the asymmetic and symmetric stretching vibrations of (-NO_2) group[16]. The medium band at 1647cm^{-1} due to the υ(C=C) inside pyrazole ring. The other absorption bands were listed in table (2).

Compound [6]: The strong and medium absorption bands at 1693cm^{-1} and 1751cm^{-1} attributed to the υ(C=O) for lactam and lactone in oxazepine ring, respectively[16]. The sharp strong band at 1722cm^{-1} attributed to the (C=O) for ketone in pyrazole ring. The medium band at 1629cm^{-1} due to the υ(C=C) inside oxazepine ring. The weak band at 1660cm^{-1} due to the υ(C=C) inside pyrazole ring. The other absorption bands were listed in table (2).

The FT-IR spectra of oxazepine derivatives [7-9], figures (8-10) respectively, table (2), showed disappearance of the sharp strong absorption band at 1629cm^{-1} attributed to the υ(C=N) and appearance of the following characteristic absorption bands:

Compound [7]: The sharp strong band at 1678cm^{-1} attributed to the υ(C=O) for lactam in oxazepine ring[16]. The medium band at 1728cm^{-1} attributed to the stretching vibrations of (C=O) groups for lactone and ketone in oxazepine and pyrazole rings, respectively, due to the vibration coupling[16]. The weak band at 1635cm^{-1} attributed to the υ(C=C) inside pyrazole ring. The other absorption bands were listed in table (2).

Compound [8]: The sharp strong band at 1668cm^{-} and the sharp medium band at 1750cm^{-1} attributed to the stretching vibrations of (C=O) groups for lactam and lactone in oxazepine ring, respectively[16]. The medium band at 1710cm^{-1} attributed to the υ(C=O) of ketone in pyrazole ring. The medium band at 1626cm^{-1} due to the υ(C=C) inside pyrazole ring. The strong and medium bands at 1521cm^{-1} and 1310cm^{-1} attributed to the asymmetric and symmetric stretching vibrations of (NO_2) group[16,17]. The other absorption bands were listed in table (2).

Compound [9]: The sharp strong band at 1674cm^{-1} attributed to the υ(C=O) for lactam in oxazepine ring[16]. The sharp strong band at 1726cm^{-1} attributed to the stretching vibrations of (C=O) groups for lactone and ketone in oxazepine and pyrazole rings, respectively, due to the vibration coupling[16]. The medium band at 1624cm^{-1} due to the υ(C=C) inside pyrazole ring. The other absorption bands were listed in table (2).

^{1}H-NMR spectrum of compound [9] figure (11) showed the following characteristic chemical shifts (MeOD, ppm):

The singlet signal at δ 2.433 attributed to protons of methyl group (a) which is bonded with carbon-carbon double bond.

The singlet signal at δ 3.132 attributed to the protons of methyl group (b) which is bonded with nitrogen.

The singlet signal at δ 3.837 attributed to protons of methoxy group (c).

The singlet signals at δ 6.123 and 6.629 attributed to protons of double bond (d) and (d_1) respectively, the signals appeared singlet may be due to *cis*- conformation which leads to decrease value of their coupling constant.

The doublet signal at δ 6.832-6.849 attributed to the aromatic proton (e).

The doublet signal at δ 6.950 -6.966 attributed to the aromatic proton (f).

The triplet signal at δ 7.176 -7.210 attributed to the aromatic proton (g).

The asymmetrical sexplet pattern at δ 7.354-7.424 resulting from interaction of the triplet signal of proton (h) and the doublet signal of protons (i) in addition of the singlet signal of proton (C-H) in oxazepine ring (m).

The triplet signal at δ 7.505-7.536 attributed to the aromatic protons (j).

The singlet signals at δ 9.456 and 9.518 attributed to the phenolic (O-H) proton (k).

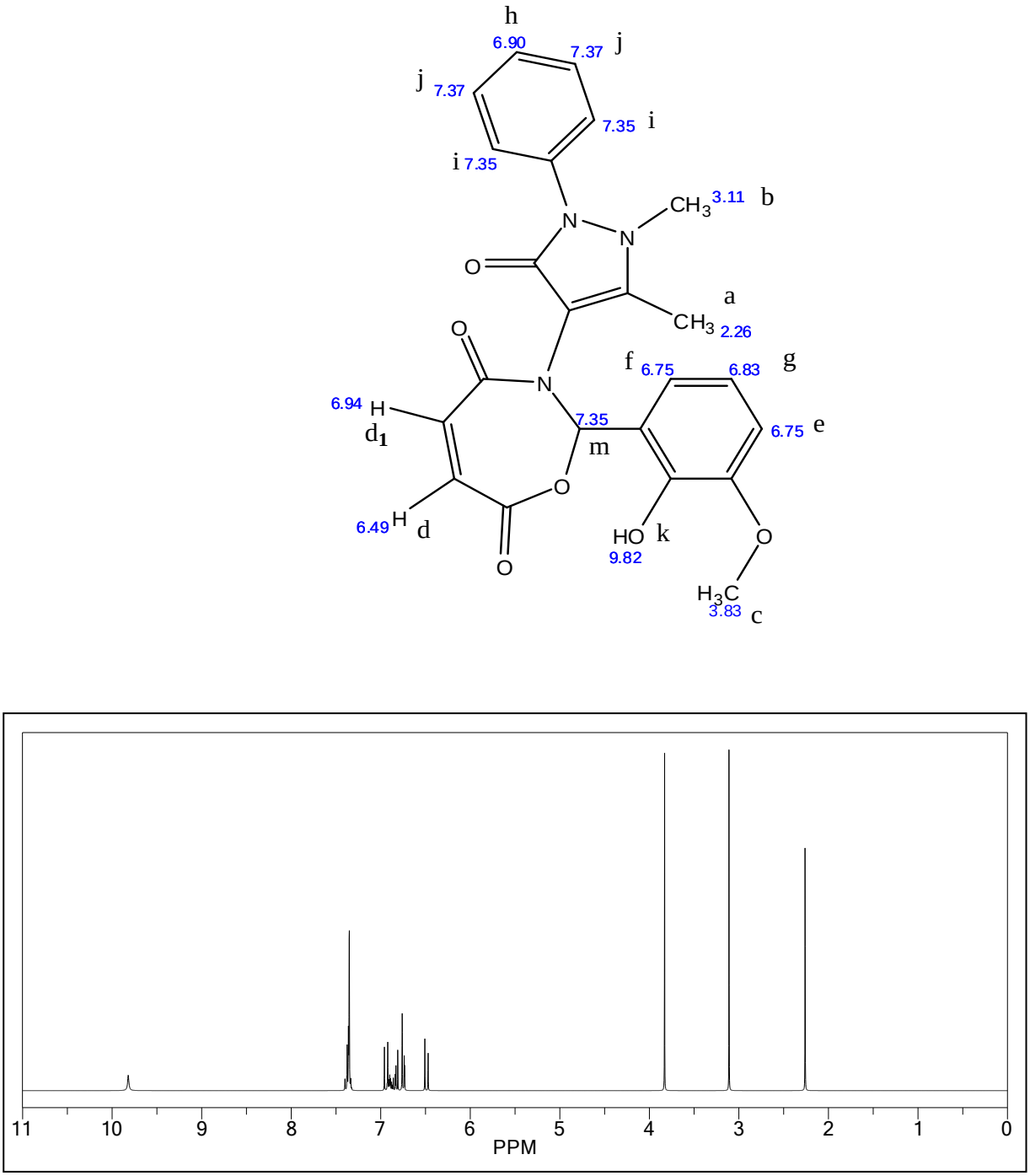


Fig.1: ***Theoretical ^{1}H-NMR spectrum of compound***

Table(2): FTIR Data of the prepared compounds [1-9] in cm^{-1}

Comp No.	υ O-H	υ NH_2	υ C-H arom.	υ C-H aliph.	Overtone bands	υ C=O	υ C=N	υ C=C	υ C=C arom.	υ NO_2	δ C-H aliph.	δ O-H in plane	δ C-H arom. in plane	υ C-O	δ C-H arom. o.o.p.	δ O-H o.o.p.
[1]	---	3420(s) 3300(s)	3170(m) 3066(m) 3010(w)	2895(s)	1805(w) 1876(w) 1960(w)	1716 (w) ketone	---	---	(s)1579 1498(w)	---	1435(m) 1356(s)	---	1271(s) 1182(s) 1105(s)	---	912(m) 839(m) 754(s)	---
[2]	(sp)3640 3589(sp) Free(O-H) 3500-3217(sp) bonding(O-H)	---	(m)3146 3086(m) 3020(w)	2978(w) 2918(w) 2837(w) 2764(w)	1797(w) 1870(w) 1950(w) 2013(w)	1730 (w) ketone	1626 (s)	---	(m)1581 1512(s)	---	1450(w)	1386(s)	(s)1286	1207(s) asym.ether 1030(s) sym.ether 1128(s) (phenol)	966(s) 868(m) 819(s) 63(s)7	700(s)
[3]	3650,3590(sp) Free(O-H) 3533-3210(sp) bonding(O-H)	---	(m)3178 3090(w)	2995(s) 2937(w) 2837(m) 2795(w) 2733(m)	1793(w) 1855(w) 1946(w)	1718 (m) ketone	1629 (s)	---	1577(s) 1516(s)	---	1420(w) 1350(w)	1379(s)	1290(s) 1257(m)	1209(s) asym.ether 1031(s) sym.ether 1134(s) (phenol)	968(m) 918(w) 866(m) 815(m) 759(s)	694(s)
[4]	3660,3600(sp) Free(O-H) 3524-3200(sp) bonding(O-H)	---	60(w)30	2970(w) 2890(w) 2840(w) 2740(w)	1810(w) 1890(w) 1980(w)	1680(m) lactam 1730(m) lactone +ketone	---	1631 (m)	1577(s) 1506(s)	---	interacted with (O-H) δ in plane	1388(s)	(s)1280	1210(w) asym.ether 1026(m) sym.ether 1134(s) (phenol)	(w)659 918(m) 870(w) (w)815 (w)765	690(s)
[5]	3660,3600(sp) Free(O-H) 3560-3230(sp) bonding(O-H)	---	(m)3160 3105(m) 3047(w) 3003(m)	2964(w) 2931(w) 2880(w) 2840(w) 2770(w) 2720(w)	1790(w) 1840(w) 1905(w) 1967(w)	1680(w) lactam 1730(s) lactone +ketone	---	1647 (m)	1610(m) 1577(s) 1500(s) 1462(s)	1545(m) asym. 1309(m) sym.	1423(m) 1350(s)	1386(s)	(s)3127	1213(s) asym.ether 1030(s) sym.ether 1130(m) (phenol) 1159(m) 1070(m) lactone	966(m) 910(m) 869(s) 825(s) 3(s)77	668(s)

[6]	3640,3599(sp) Free(O-H) 3540-3210(sp) bonding(O-H)	---	(w)3122 3082(w)	2968(w) 2940(w) 2890(w) 2820(w) 2800(w)	1790(w) 1853(w) 1890(w) 1930(w) 1965(w)	1693(s) lactam 1751(m) lactone 1722(s) ketone	---	1660 (w) 1629 (m)	1610(w) 1573(s) 1521(s) 1502(w) 1446(s)	---	1350(s)	1383(m)	1271(s)	1219(s) asym.ether 1026(s) sym.ether 1136(m) (phenol) 1165(w) 1090(w) lactone	866(s) 821(s) 761(s) 729(w)	694(s)
[7]	3589(sp) Free(O-H) 3550-3200(sp) bonding(O-H)	---	(w)3147 3099(w) 3055(m) 3005(w)	2945(w) 2830(w) 2752(w)	1788(m) 1850(w) 1970(w)	1678(s) lactam 1728(m) lactone +ketone	---	1635 (w)	(s)1580 1500(m)	---	1425(w)	1352(m)	1292(s)	1211(s) asym.ether 1030(s) sym.ether 1125(w) (phenol) 1153(m) 1075(w) lactone	968(w) 873(s) 820(w) 69(m)7	730 (w)
[8]	3640(sp) Free(O-H) 3527-3200(sp) bonding(O-H)	---	(m)3149 3086(m) 12(s)30	2958(m) 2870(w) 2820(w) 2770(w)	1793(w) 1850(w) 1905(w) 1971(m)	1668(s) lactam 1750(w) lactone 1710(m)ketone	---	1626 (m)	(w)1595 1565(w) 1471(m)	1521(s) asym. 1310(m) sym.	1419(m) 1350(m)	1370(w)	1269(m)	1215(s) asym.ether 1030(s) sym.ether 1132(m) (phenol)	(s)689 (s)691 868(m) 825(w) 779(m) 750(w)	694 (s)
[9]	3640,3574(sp) Free(O-H) 3520-3252(sp) bonding(O-H)	---	(w)3150 3120(w) 3090(w) 3057(w) 3005(w)	2964(w) 2908(w) 2840(w) 2820(w)	1786(w) 1855(w) 1970(w)	1674(s) lactam 1726(s) lactone +ketone	---	6241 (w)	(m)1575 1521(m) 1470(w)	---	1440(w)	1386(m)	1300(m) 1255(w)	1207(m) asym.ether 1030(s) sym.ether 1120(s) (phenol)	970(s) 918(m) 821(s) 63(s)7	694 (s)

sp=sharp, w=weak, m=medium, s=strong, o.o.p.= out of plane

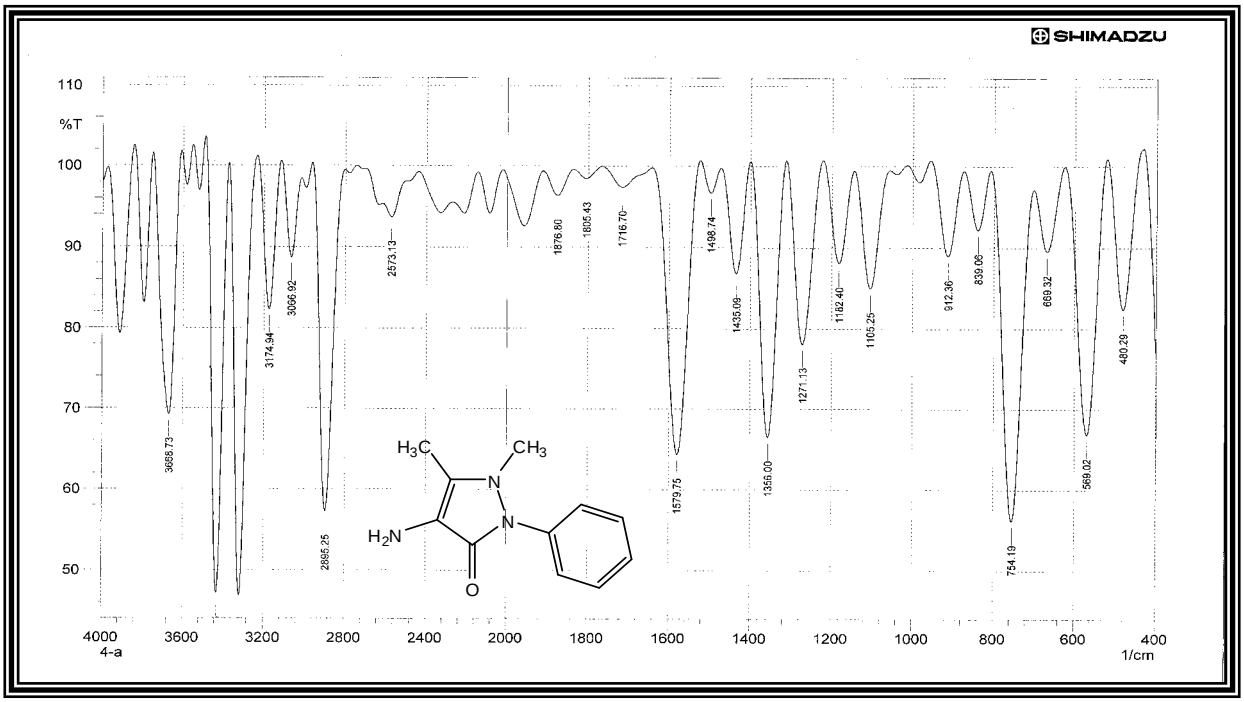


Fig. 2: ***FT-IR spectrum of compound [1]***

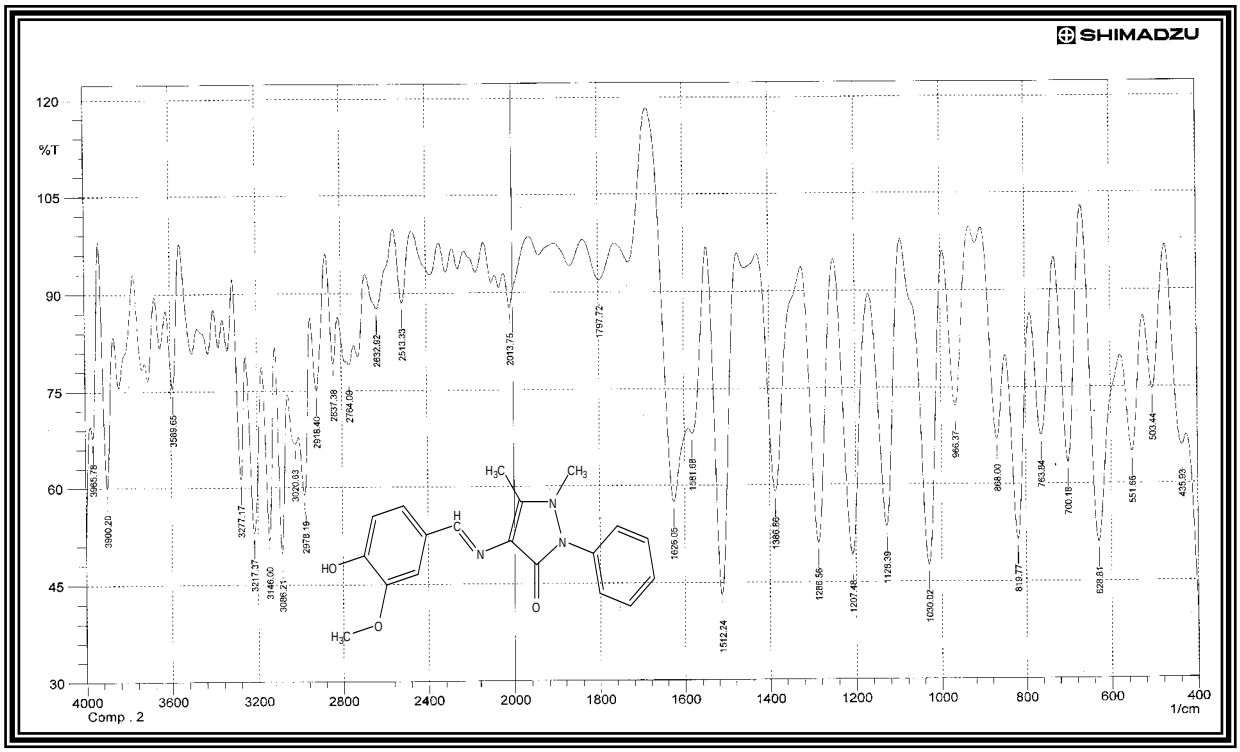


Fig. 3: ***FT-IR spectrum of compound [2]***

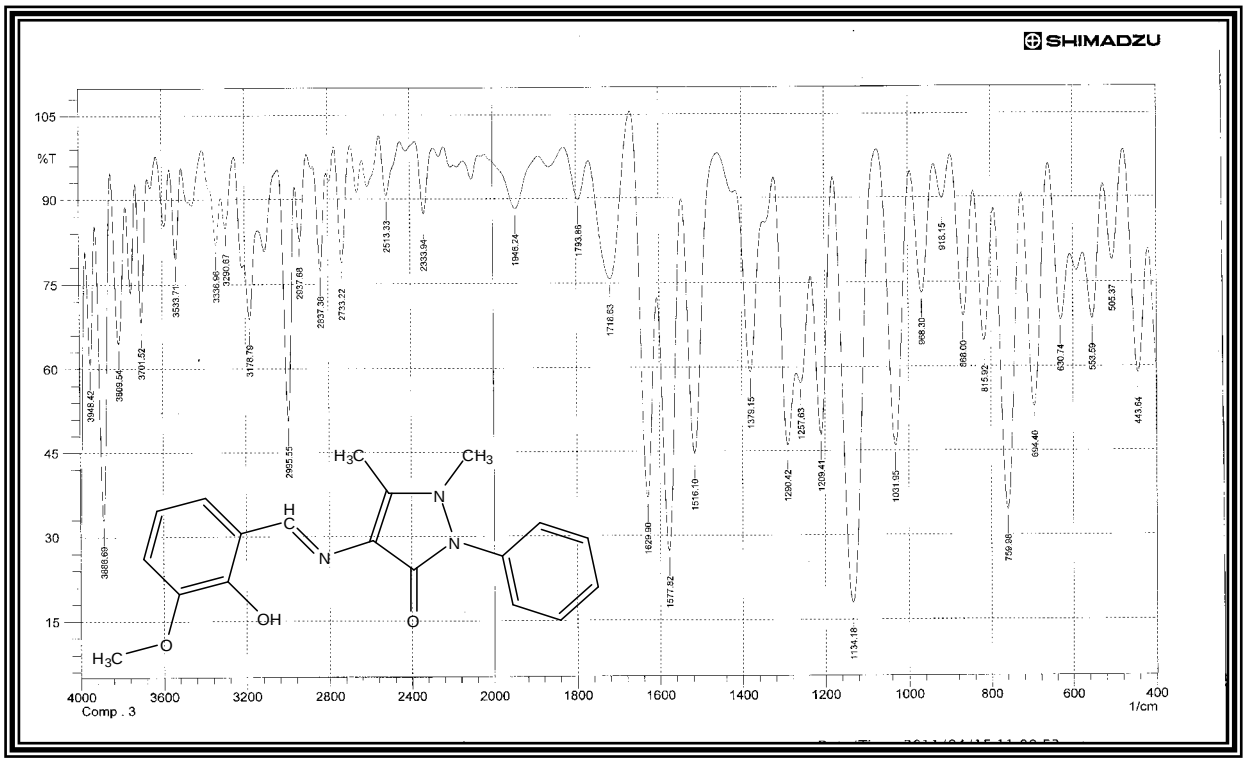


Fig. 4: ***FT-IR spectrum of compound [3]***

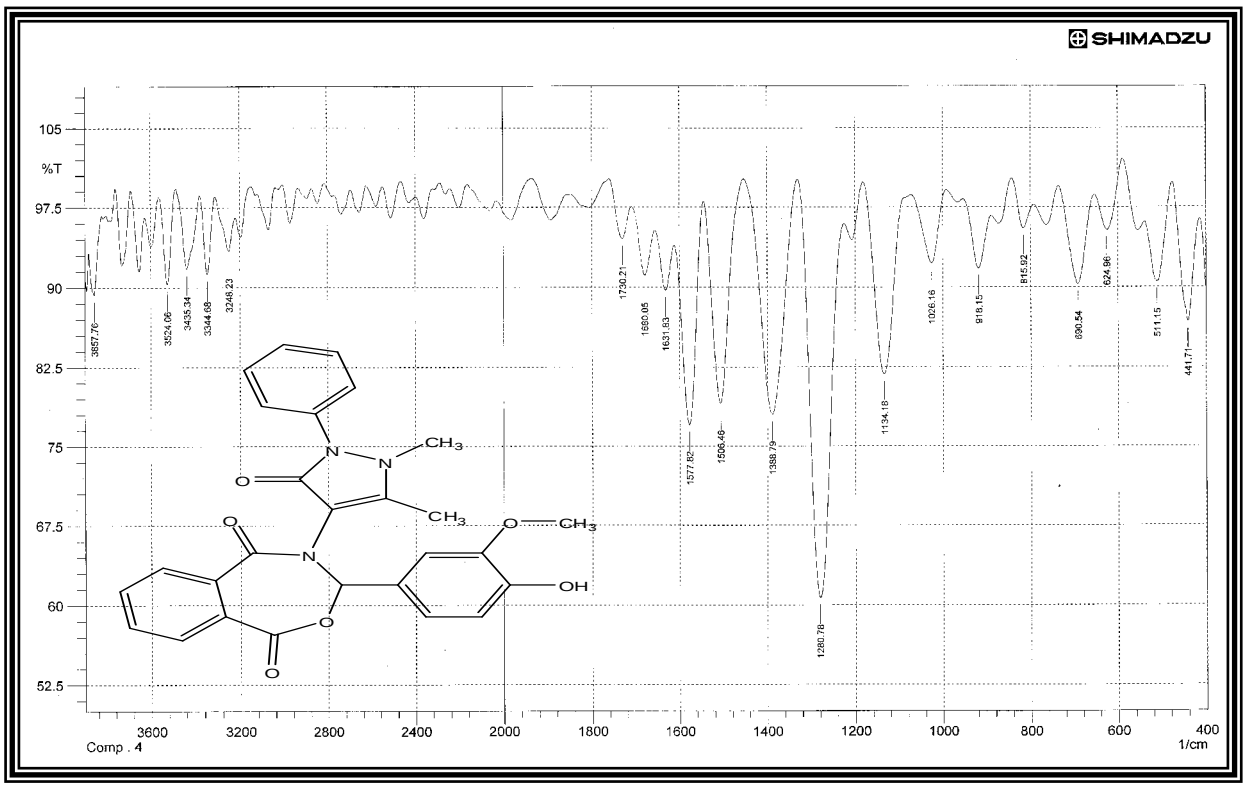


Fig. 5: ***FT-IR spectrum of compound [4]***

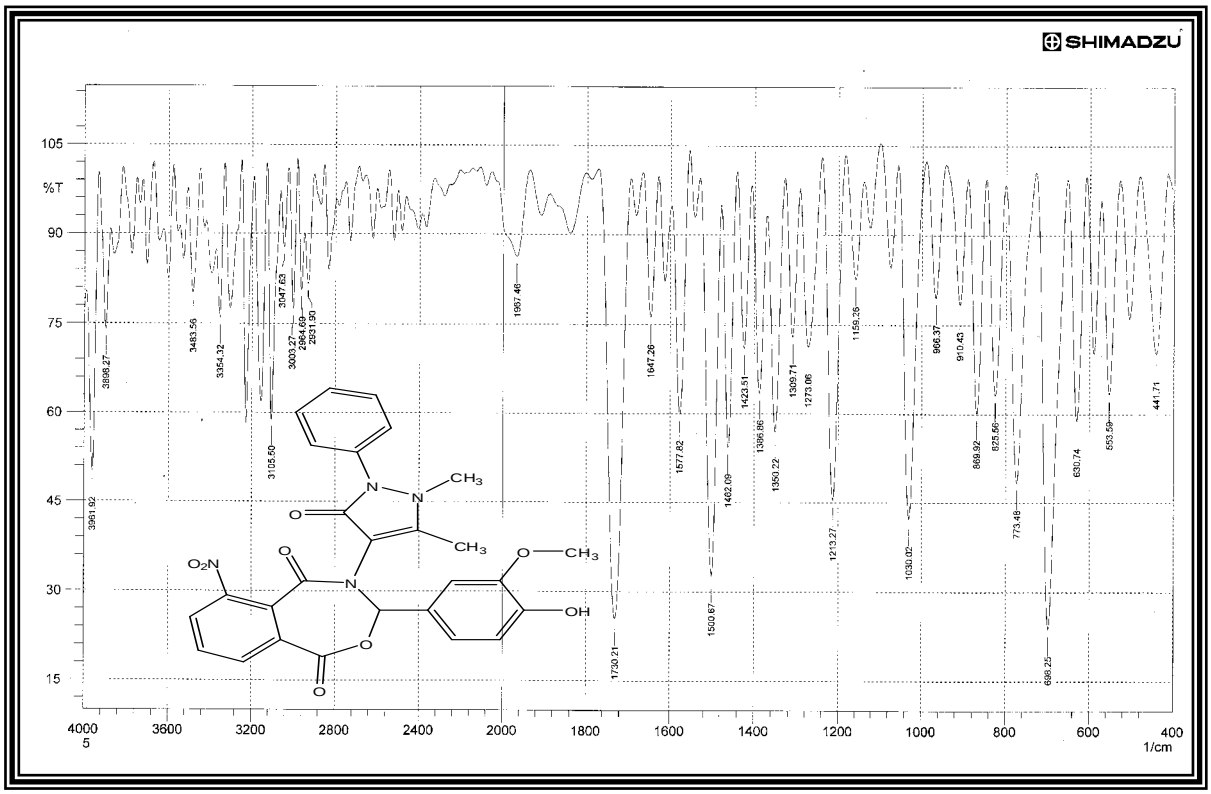


Fig. 6: ***FT-IR spectrum of compound [5]***

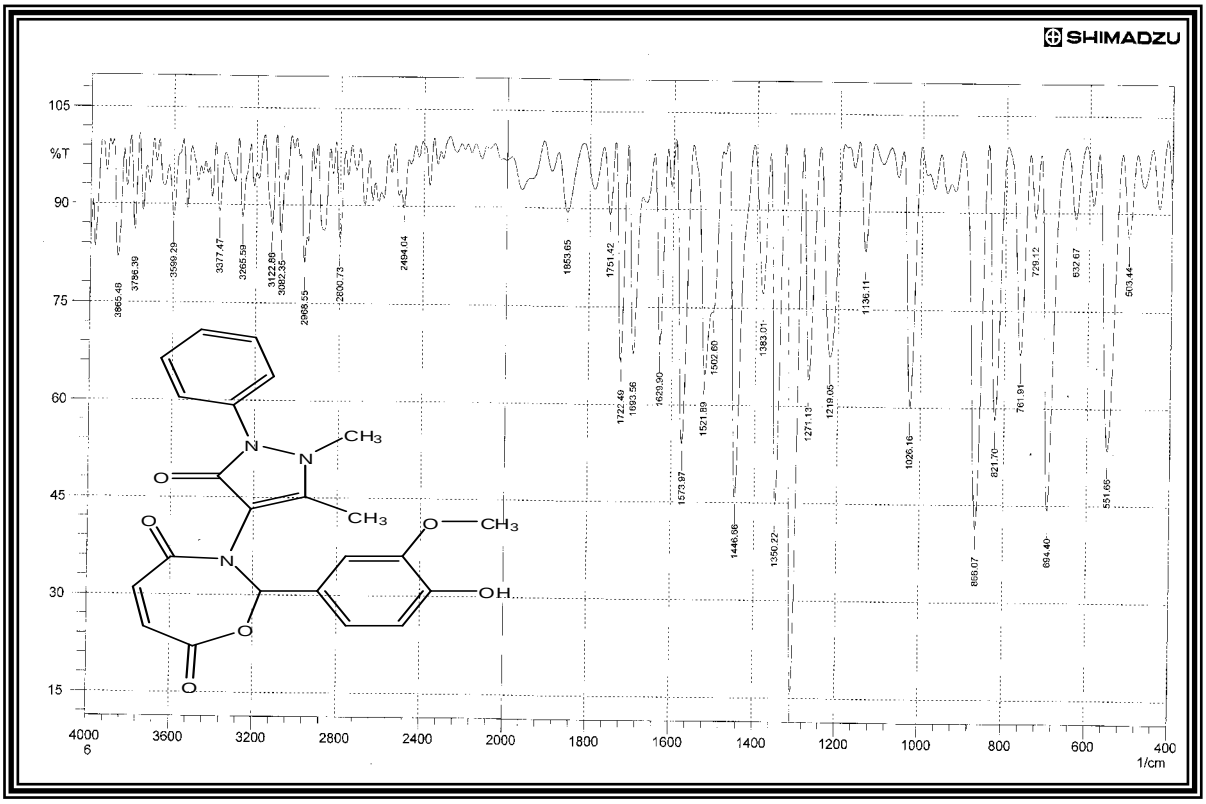


Fig. 7: ***FT-IR spectrum of compound [6]***

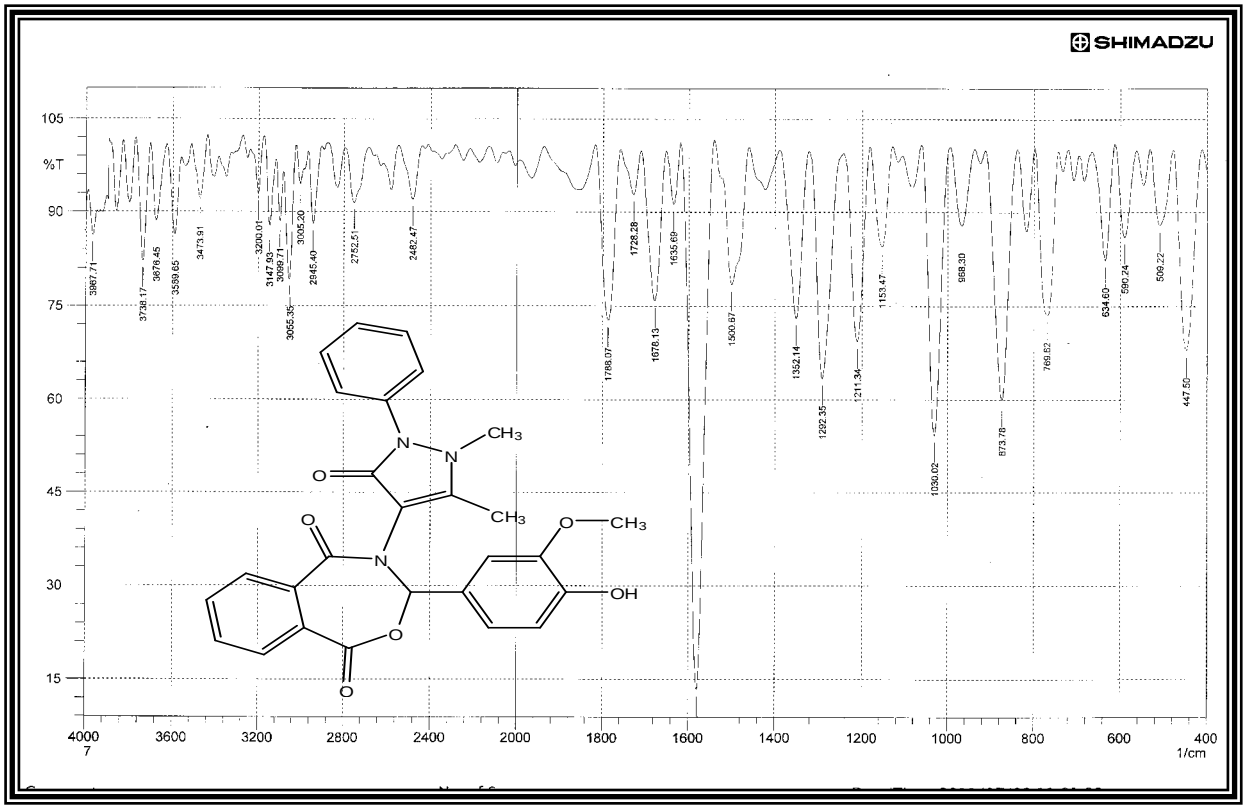


Fig. 8: ***FT-IR spectrum of compound [7]***

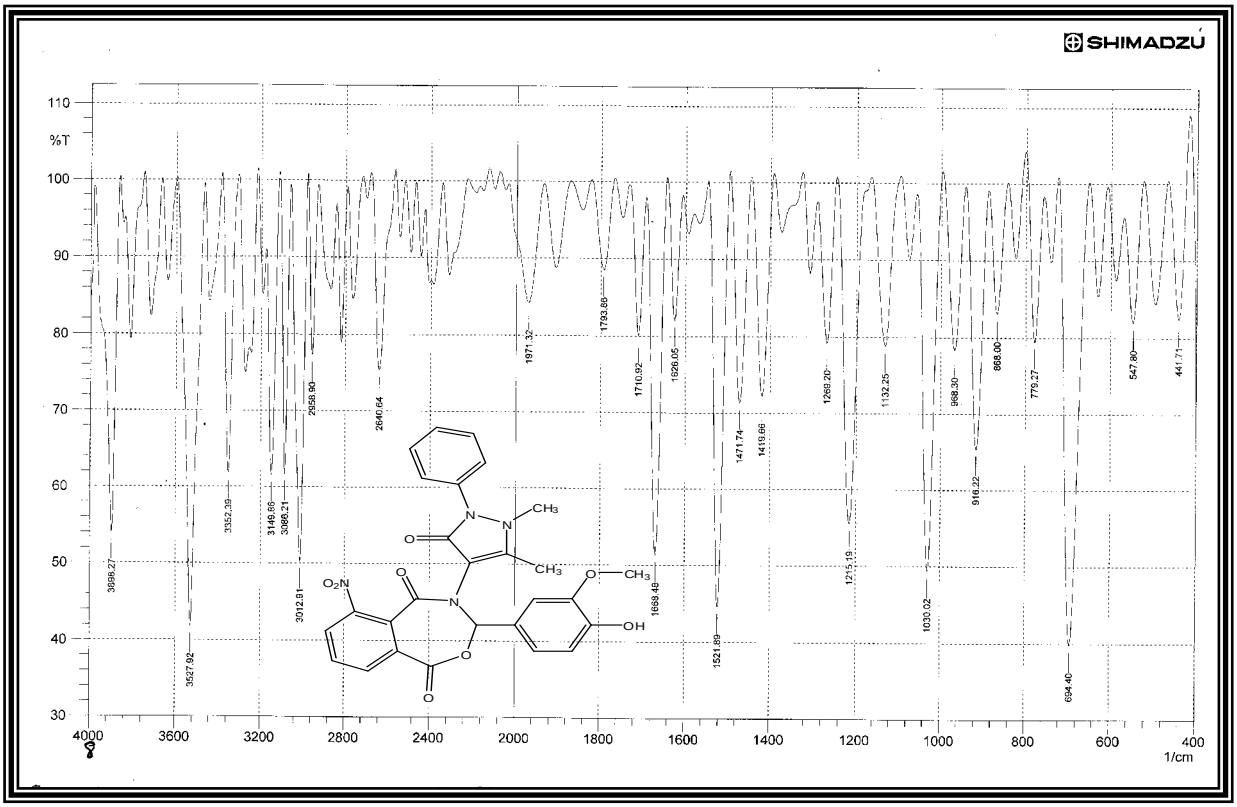


Fig. 9: ***FT-IR spectrum of compound [8]***

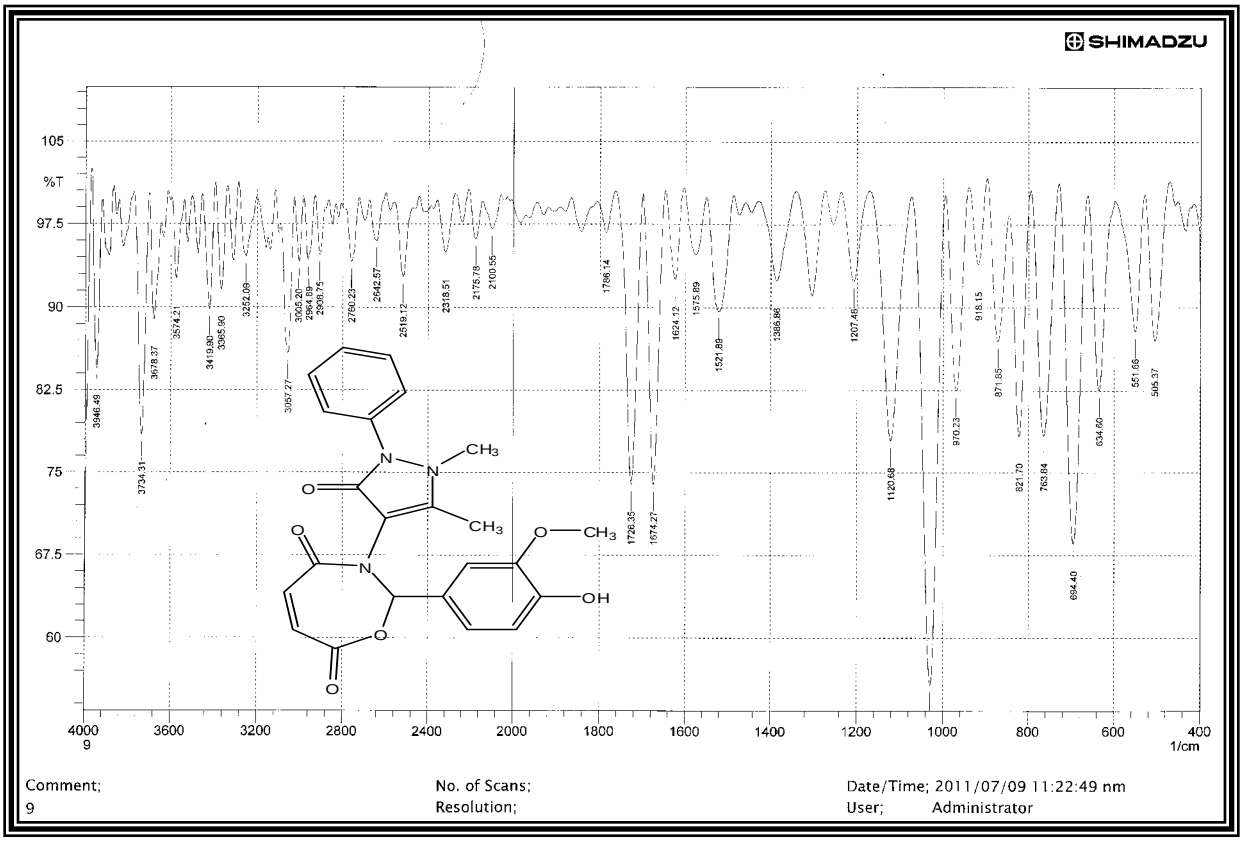
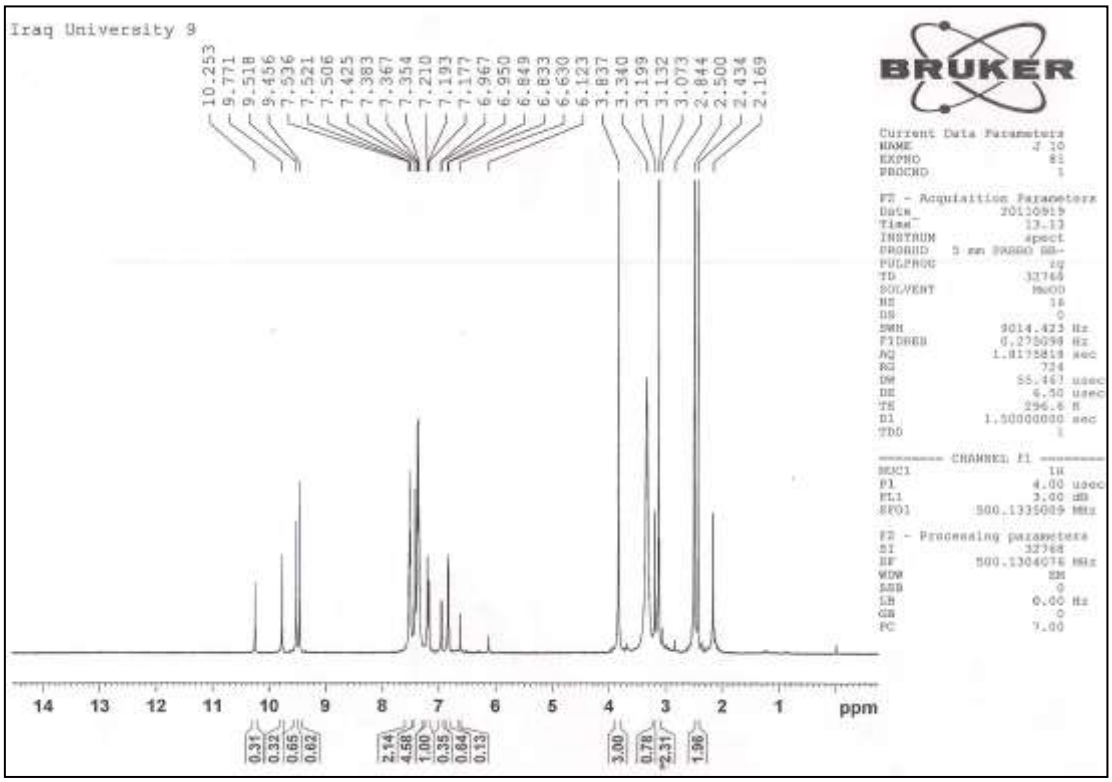
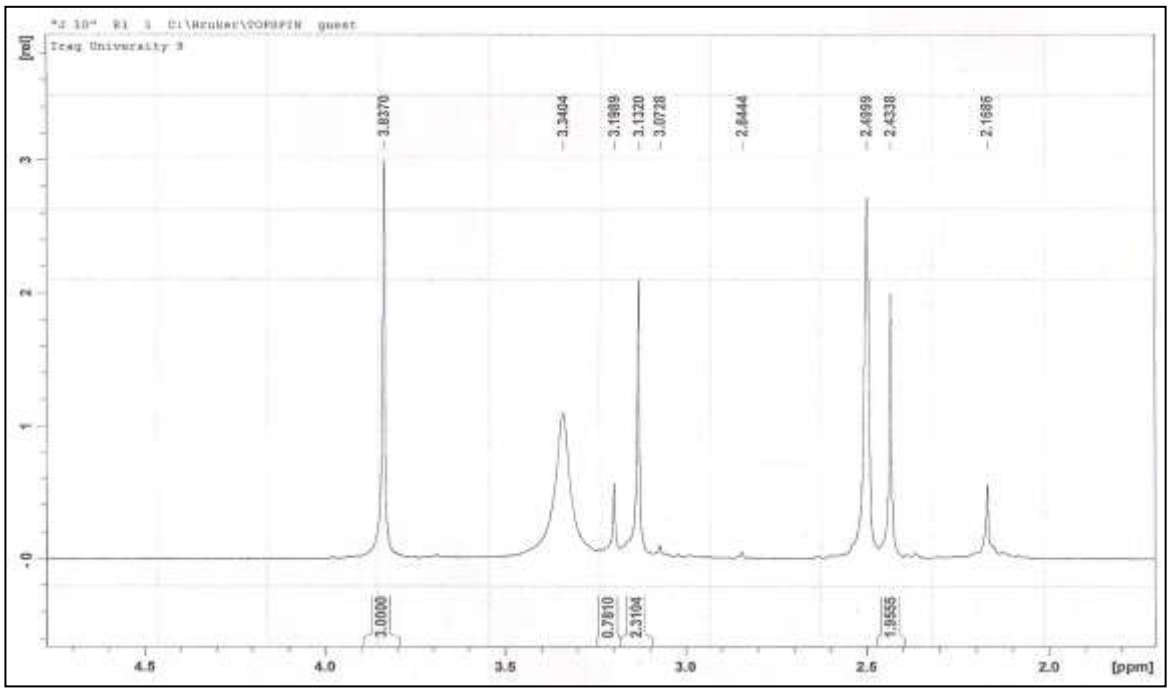
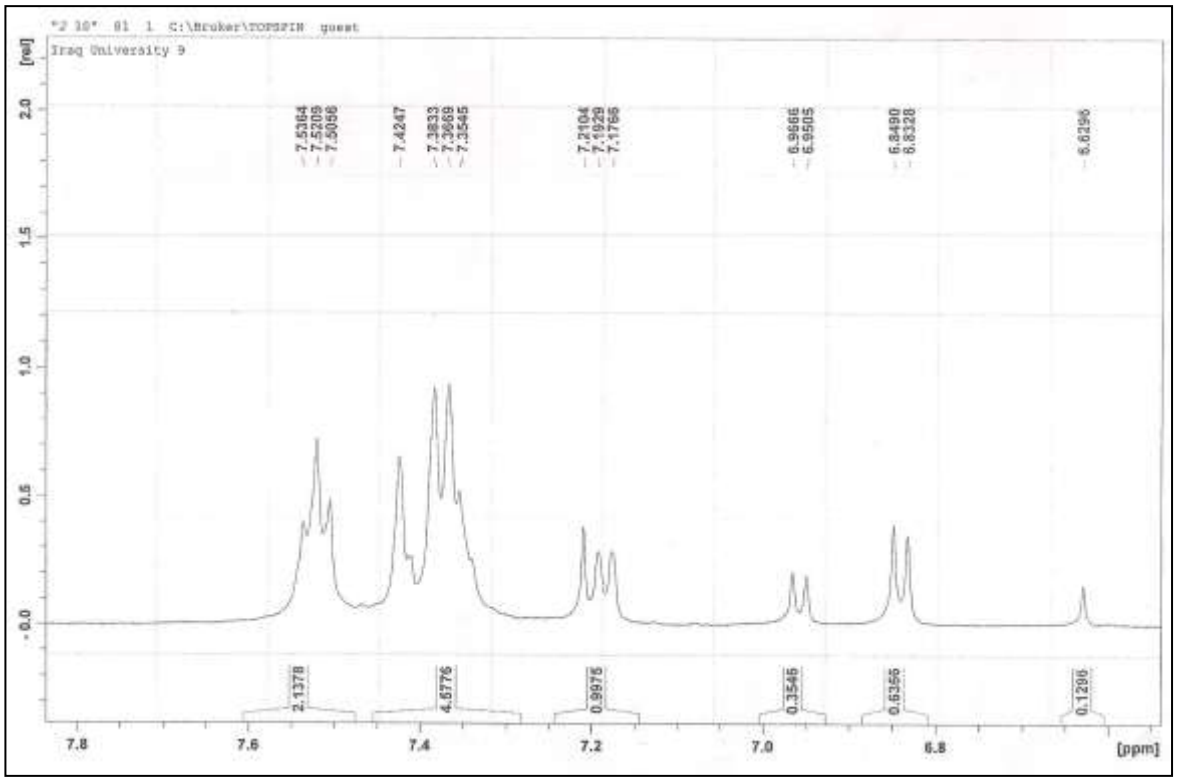


Fig. 10: ***FT-IR spectrum of compound [9]***




Iraq University 9
3.8370
3.3404
3.1989
3.1320
3.0728
2.8444
2.4999
2.4338
2.1686
3.0000
0.7810
2.3104
1.9558
[ppm]


Iraq University 9
7.5364
7.5209
7.5056
7.4247
7.3833
7.3669
7.3545
7.2104
7.1929
7.1766
6.9666
6.9505
6.8490
6.8328
6.6296
2.1378
4.5776
0.9975
0.3546
0.6366
0.1296
[ppm]

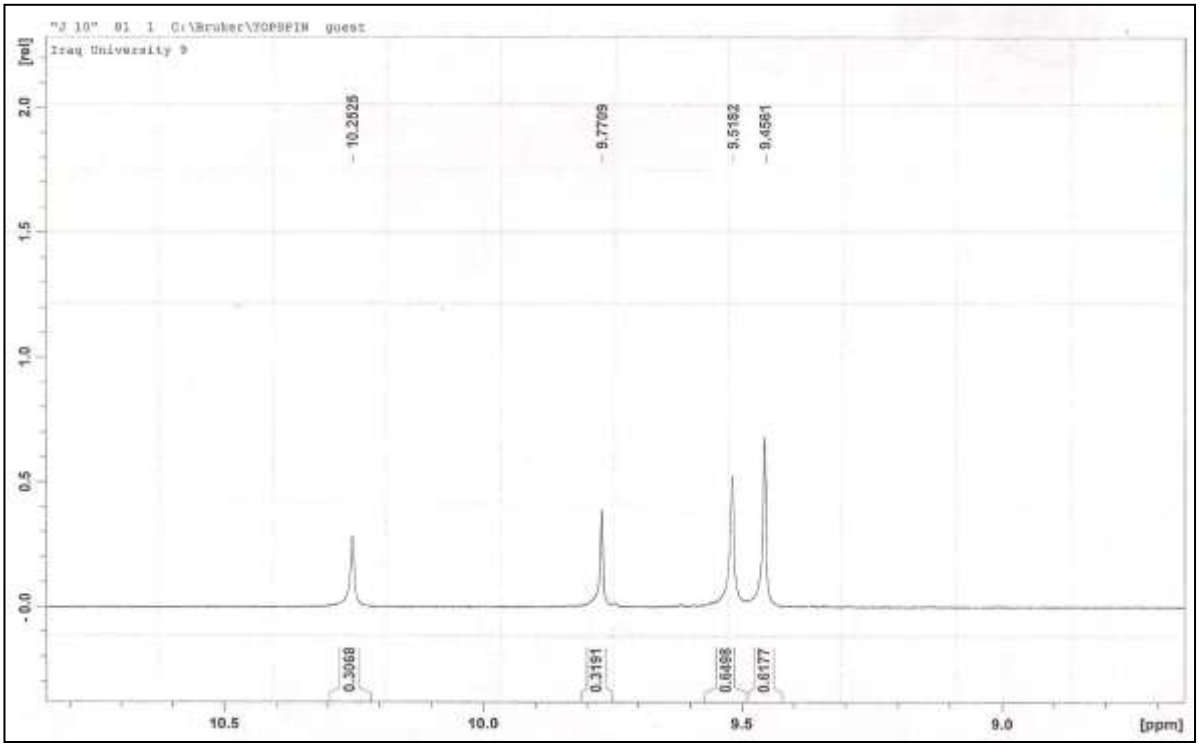


Fig. 11: ***^{1}H -NMR spectrum of compound [9]***

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