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Using A pericyclic Reactions for The Synthesis of New 1,3-Oxazepine Compounds From New Imines

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Abstract:

In this research new1,3-oxazepine derivatives[6-11] were prepared starting from new Schiff bases [4] and [5]. 5,5'-{[3-(Methoxycarbonyl)-4-oxocyclohexa-2,5-dienylidene]methylene}bis(2-methoxybenzoate) [2] was prepared by reaction of 5,5'-[(3-carboxy-4-oxocyclohexa-2,5-dienylidene)methylene]bis(2-hydroxybenzoic acid) [1] with dimethyl sulphate in presence of anhydrous sodium carbonate in dry acetone. The trihydrazide derivative [3] was obtained from treatment of triester derivative [2] with hydrazine monohydrate in absolute ethanol. Reaction of the trihydrazide derivative [3] with each Furfural and Salicyladehyde, respectively, in presence of glacial acetic acid as catalyst in absolute ethanol resulted the formation of new triimine derivatives [4] and [5], respectively. Treatment of the resulting imines [4] and [5] with each maleic anhydride, phthalic anhydride and 3-nitrophthalic anhydride, respectively, under cycloaddition reactions conditions produced new tri(1,3-oxazepine) derivatives [6-8] and [9-11] respectively. These new imines and 1,3-oxazepine derivatives may be used as antibiotics.

The structures of all prepared compounds were confirmed by C. H. N. elementary analysis and FT-IR spectra.

الخلاصة:

تم في هذا البحث تحضير مشتقات 1،3- اوكسازيبين جديدة [6-11] من قواعد شيف جديدة [4] ، [5] ،حضر 5،'5-{[3-(ميثوكسي كاربونيل)-4-أوكسوسايكلو هكسا-5,2-دايينيليدين]مثيلين}بس(2-ميثوكسي بنزوات) [2] من تفاعل 5،'5-[(3-كاربوكسي-4-أوكسوسايكلو هكسا-5,2-دايينيليدين)مثيلين]بس(2-هيدروكسي حامض البنزويك) [1] مع كبريتات ثنائي المثيل بوجود كاربونات الصوديوم اللامائية في الأسيتون الجاف. تم الحصول على مشتق ثلاثي الهيدرازيد [3] من معاملة مشتق ثلاثي الاستر [2] مع الهايدرازين المائي في الايثانول المطلق. ان تفاعل المشتق ثلاثي الهايدرازيد [3] مع كل من الفورفورال والسالساليديهايد، على التوالي، بوجود حامض الخليك الثلجي كعامل مساعد في الايثانول المطلق أعطى مشتقات ثلاثي الايمين جديدة [4] و[5] على التوالي. إن معاملة الايمينات الناتجة [4] و[5] مع كل من انهدريد الماليك، انهدريد الفثاليك، 3-نايتروانهدريد الفثاليك، على التوالي، تحت شروط تفاعلات الاضافة الحلقية اعطت مشتقات ثلاثي (1،3-أوكسازيبين) جديدة [6-8] و[9-11] على التوالي. ان مشتقات الايمين والأوكسازيبين الجديدة هذه قد تستعمل كمضادات حيوية.

شخصت تراكيب جميع المركبات المحضرة بوساطة تحليل نسب العناصر (C. H. N.) وأطياف الأشعة تحت الحمراء.

Introduction:

Pericyclic reaction is concerted processe pass-through a single cyclic transition state structure involving simultaneously breaking and formation of bonds[1].The kinetic studies for pericyclic reactions showed that the rates of these reactions do not change with changing polarity of the solvent, so these reactions never take place via generation of intermediate[1,2]. The kinetic studies also showed that the rates of these reactions are neither increased in presence of free radicals initiators nor decreased in presence of retardants, so pericyclic reactions never take place via generation of free-radicals[1,2]. A pericyclic reaction includes changing in bonding relationship which occurs as continuous concerted reorganization of electrons, Furthermore the cyclic transition state must correspond to an arrangement of the participating orbitals that can maintain a bonding interaction between the reaction components throughout the course of the reaction[2,3]. 1,3-Oxazepine is unsaturated seven-membered hetrocycle containing oxygen atom in position (1), nitrogen atom in position (3) in edition of five carbons Oxazepine derivatives showed various

biological activities such as antibacterial[4] and inhibitors for some enzymes action[5]. Some of oxazepine derivatives are used in another applied fields[6].

For a long time, the synthesis of 1,3- and 1,4-oxazepine rings was based on two limited classical types of reactions, the first reaction is called Valence-bond isomerization which is carried out via irradiation of polyarylpyridine N-oxides. This irradiation results in ring expansion to 1,3-oxazepine in high yield and some deoxygenation to the parent amines[7] . The second reaction is called Enamines condensation which is carried out by reaction of Erythro 1,2-diphenyl-2-phenylaminoethanol with dimethylacetylene dicarboxylate in methanol at room temperature to give a mixture of the Michael adduct and tetrahydro-1,4-oxazepin-7-one[8]. Recently, cycloaddition reaction, which is a type from a pericyclic reactions is used to synthesis of 1,3-oxazepine ring[9-12]. This type of reactions is not limited and gives various 1,3-oxazepine ring derivatives. The type of cycloaddition reaction that used to synthesis of 1,3-oxazepine ring was classified as (2+5) → 7 cycloaddition reaction in which two atoms of imine group as two-membered component was added to five-membered component such as maleic or phthalic anhydrides to give a seven-membered heterocycle[13,14].

Schiff base or imine compounds containing active (C=N-) group. Imines are prepared via acid-catalysed condensation reaction between aromatic aldehydes or ketones and primary amines[15-18]. Mechanism of reaction was well known[19]. Due to the great flexibility and diverse structural aspects, a wide range of Schiff bases have been synthesized and their complexation behavior studied[20,21]. Furthermore Schiff bases are reported to show a variety of interesting biological actions, including antibacterial[22], antifungal[23], anti mouse hepatitis virus (MHV)[24], inhibition of herpes simplex virus type 1 (HSV-1) and adenovirus type 5 (Ad 5)[25], anticancer[26], anti mosquito larvae[27] and herbicidal activities[28]. Also, Schiff bases are important intermediates for the synthesis of some bioactive compounds such as β-lactams[29].

The target of this research is synthesis of new structures containing more than one bioactive imine group and 1,3-oxazepine ring which probably have some biological activities.

Experimental:

General

1) The solvents and liquid reagents were purified when it was necessary; the solid materials were also dried under reduced pressure when it was necessary.
2) T.L.C were performed on pre-coated sheets with 0.25 mm layer of Silica Gel GF254 of Merck company, the detection was followed by oxidation with iodine or H_2SO_4 in ethanol (60%) followed by heating .
3) Evaporating of solvents by using Buchi vacuum rotary evaporator type 160.
4) Melting points (M.P.) were determined by Stuart melting point apparatus.
5) Elemental analysis measured on E.A.300, Euro- Vector, Italy, 2003-AL-albayt University (Jordan).
6) FT-IR spectra were recorded on FT-IR 8400_S, Schimadzu-Spectrophotometer and using KBr discs-kerbala university.

Preparation Methods

Synthesis of Dimethyl 5,5'-{[3-(methoxycarbonyl)-4-oxocyclohexa-2,5-dienylidene]methylene}bis(2-methoxybenzoate) [2]

5,5'-[(3-carboxy-4-oxocyclohexa-2,5-dienylidene)methylene]bis(2-hydroxy-benzoic acid) [1] (2.11g, 0.0050mole) was dissolved in (25mL) of dry acetone, then anhydrous sodium carbonate (0.53g, 0.0050mole) was added and the mixture was left with stirring at room temperature for 20min., then dimethyl sulphate (3.15g, 0.0250mole) was added and the mixture was refluxed with stirring at 50ºC for 24hrs., the solvent was then removed by evaporation and the product was extracted from the mixture by addition a solution of saturated sodium

bicarbonate in distilled water and ethyl acetate (4×25mL). The organic layer was dried with anhydrous magnesium sulphate and removed by evaporation, recrystallized from ethanol, yield 82%, M.P.190-192ºC.

Synthesis of 5,5'-{[3-(hydrazinecarbonyl)-4-oxocyclohexa-2,5-dienylidene] methylene}bis(2-methoxybenzohydrazide) [3]

Amixture of triester derivative [2] (2g, 0.0040mole) and hydrazine hydrate (0.60g, 0.0120mole) in absolute ethanol (20mL) was refluxed with stirring on water bath at 75ºC for 6hrs. The hydrazide was precipitate on cooling, filtered off and recrystallized from ethanol,T.L.C. (ethanol:pet.ether) (1:3), R_f=0.59,yield 78%, M.P.185ºC.

Synthesis of 5,5'-{[3-(2-(furan-2-ylmethylene)hydrazinecarbonyl)-4-oxo-cyclohexa-2,5-dienylidene]methylene}bis[N-(furan-2-ylmethylene)-2-methoxy-benzohydrazide] [4]

Trihydrazide derivative [3] (0.984g, 0.002mole) was dissolved in absolute ethanol (15mL), then Furfural (0.576g, .006mole) containing two drop of glacial acetic acid was dissolved in absolute ethanol (15mL) and then added dropwise. The reaction mixture was refluxed 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 washed well with cold ethanol, T.L.C. (ethanol:pet.ether) (1:1), R_f=0.64, yield 77%, M.P. 246-248ºC.

Synthesis of 5,5'-{[3-(2-(2-hydroxybenzylidene)hydrazinecarbonyl)-4-oxo-cyclohexa-2,5-dienylidene]methylene}bis[N-(2-hydroxybenzylidene)-2-methoxy-benzohydrazide] [5]

Trihydrazide derivative [3] (0.984g, 0.002mole) was dissolved in absolute ethanol (15mL), then 2-Hydroxybenzaldehyde (0.732g, 0.006mole) containing two drop of glacial acetic acid was dissolved in absolute ethanol (10mL) and then added dropwise. The reaction mixture was refluxed 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 washed well with cold ethanol, T.L.C. (methanol:benzene) (1:2), R_f=0.69,yield 75%, M.P. 260-262ºC.

Synthesis of 5,5'-{[3-(2-(furan-2-yl)-4,7-dioxo-1,3-oxazepin-3(2H,4H,7H)-yl carbamoyl)-4-oxocyclohexa-2,5-dienylidene]methylene}bis{N-[2-(furan-2-yl)-4,7-dioxo-1,3-oxazepin-3(2H,4H,7H)-yl]-2-methoxybenzamide} [6]

Schiff base derivative [4] (0.363g, 0.0005mole) and Maleic anhydride (0.147g, 0.0015mole) were dissolved in (20mL) of dry benzene. The reaction mixture was refluxed with stirring on water bath at 75ºC for 4hrs., The mixture was then allowed to cool down to room temperatue, the coloured precipitate was filtered and recrystallized from dioxan, T.L.C. (methanol:benzene) (1:1), R_f=0.55. yield 65%, M.P. 310ºC.

Synthesis of 5,5'-{[3-(3-(furan-2-yl)-1,5-dioxobenzo[e][1,3]oxazepin-4(1H,3H,5H) -ylcarbamoyl)-4-oxocyclohexa-2,5-dienylidene]methylene}bis{N-[3-(furan-2-yl)-1,5-dioxobenzo[e][1,3]oxazepin-4(1H,3H,5H)-yl]-2-methoxybenzamide} [7]

Schiff base derivative [4] (0.363g, 0.0005mole) and Phthalic anhydride (0.222g, 0.0015mole) were dissolved in (20mL) of dry benzene. The reaction mixture was refluxed with stirring on water bath at 75ºC for 4hrs., The mixture was then allowed to cool down to room temperatue, the coloured precipitate was filtered and recrystallized from dioxan, T.L.C. (methanol:benzene) (1:1), R_f=0.6,yield 68%, M.P.331ºC.

Synthesis of 5,5'-{[3-(3-(furan-2-yl)-6-nitro-1,5-dioxobenzo[e][1,3]oxazepin-4(1H,3H,5H)-ylcarbamoyl)-4-oxocyclohexa-2,5-dienylidene]methylene}bis{N-[3-(furan-2-yl)-6-nitro-1,5-dioxobenzo[e][1,3]oxazepin-4(1H,3H,5H)-yl]-2-methoxy-benzamide} [8]

Schiff base derivative [4] (0.363g, 0.0005mole) and 3-Nitrophthalic anhydride (0.2895g, 0.0015mole) were dissolved in (20mL) of dry benzene. The reaction mixture was refluxed with stirring on water bath at 75ºC for 4hrs., .The mixture was then allowed to cool down to room temperatue, the coloured precipitate was filtered and recrystallized from dioxan, T.L.C. (methanol:benzene) (1:2), R_f=0.62. yield 71%, M.P.342ºC.

Synthesis of 5,5'-{[3-(2-(2-hydroxyphenyl)-4,7-dioxo-1,3-oxazepin-3(2H,4H,7H)-ylcarbamoyl)-4-oxocyclohexa-2,5-dienylidene]methylene}bis{N-[2-(2-hydroxy-phenyl)-4,7-dioxo-1,3-oxazepin-3(2H,4H,7H)-yl]-2-methoxybenzamide} [9]

Schiff base derivative [5] (0.402g, 0.0005mole) and Maleic anhydride (0.147g, 0.0015mole) were dissolved in (20mL) of dry benzene. The reaction mixture was refluxed with stirring on water bath at 75ºC for 4hrs., The mixture was then allowed to cool down to room temperatue, the coloured precipitate was filtered and recrystallized from ethanol, T.L.C. methanol:benzene) (1:3), R_f=0.73, yield 67%, M.P. 347ºC.

Synthesis of 5,5'-{[3-(3-(2-hydroxyphenyl)-1,5-dioxobenzo[e][1,3]oxazepin-4 (1H, 3H,5H)-ylcarbamoyl)-4-oxocyclohexa-2,5-dienylidene]methylene}bis{N-[3-(2-hydroxyphenyl)-1,5-dioxobenzo[e][1,3]oxazepin-4(1H,3H,5H)-yl]-2-methoxy benzamide} [10]

Schiff base derivative [5] (0.402g, 0.0005mole) and Phthalic anhydride (0.222g, 0.0015mole) were dissolved in (20mL) of dry benzene. The reaction mixture was refluxed with stirring on water bath at 75ºC for 4hrs. The mixture was then allowed to cool down to room temperatue, the coloured precipitate was filtered and recrystallized from ethanol, T.L.C. methanol:benzene) (1:3), R_f=0.78,yield 70%, M.P. 363ºC.

Synthesis of 5,5'-{[3-(3-(2-hydroxyphenyl)-6-nitro-1,5-dioxobenzo[e][1,3] oxazepin-4(1H,3H,5H)-ylcarbamoyl)-4-oxocyclohexa-2,5-dienylidene]methylene} bis{N-[3-(2-hydroxyphenyl)-6-nitro-1,5-dioxobenzo[e][1,3]oxazepin-4(1H,3H,5H)-yl]-2-methoxybenzamide} [11]

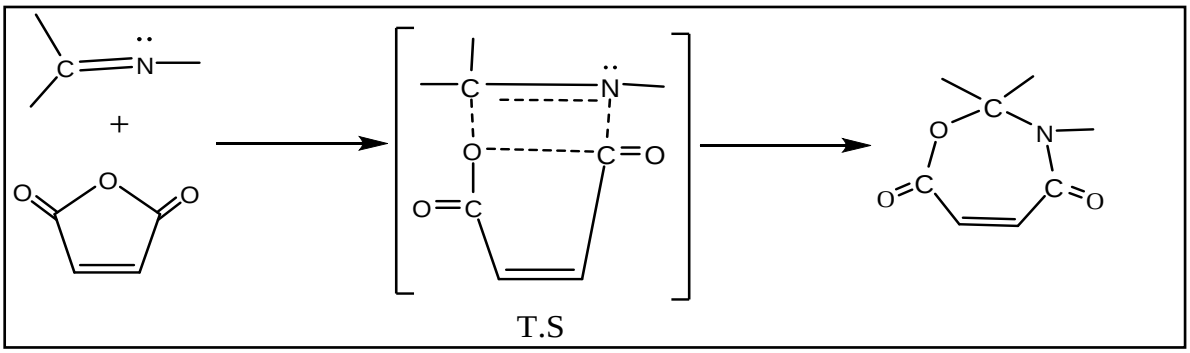
Schiff base derivative [5] (0.402g, 0.0005mole) and 3-Nitrophthalic anhydride (0.2895g, 0.0015mole) were dissolved in (20mL) of dry benzene. The reaction mixture was refluxed with stirring on water bath at 75ºC for 4hrs., The mixture was then allowed to cool down to room temperatue, the coloured precipitate was filtered and recrystallized from ethanol, T.L.C. methanol:benzene) (1:4), R_f=0.8, yield 69%, M.P. 376ºC.

Table (1): Melting points, percent yields and (C.H.N.) analysis of the prepared compounds (3-11)

Comp. No.	M.F.	M.Wt.	(M.P.)°C	Yield%	C.H.N. analysis					
					Calculated%			Found%		
					C	H	N	C	H	N
[3]	$C_{24}H_{24}N_6O_6$	492	185	78	58.53	4.87	17.07	57.81	4.33	16.23
[4]	$C_{39}H_{30}N_6O_9$	726	246-248	77	64.46	4.13	11.57	63.82	3.89	12.01
[5]	$C_{45}H_{36}N_6O_9$	804	260-262	75	67.16	4.47	10.44	67.25	4.72	10.11
[6]	$C_{51}H_{36}N_6O_{18}$	1020	310	65	60.00	3.52	8.23	59.81	3.77	7.90
[7]	$C_{63}H_{42}N_6O_{18}$	1170	331	68	64.61	3.58	7.18	64.83	3.88	6.84
[8]	$C_{63}H_{39}N_9O_{24}$	1305	342	71	57.93	2.98	9.65	66.39	3.22	9.78
[9]	$C_{57}H_{42}N_6O_{18}$	1098	347	67	62.29	3.82	7.65	62.07	4.14	7.47
[10]	$C_{69}H_{48}N_6O_{18}$	1248	363	70	66.34	3.84	6.73	66.44	3.61	7.02
[11]	$C_{69}H_{45}N_9O_{24}$	1383	376	69	59.87	3.25	9.10	60.23	2.77	8.87

Results and Discussion:

The tricarboxylic acid [1] was reacted with dimethyl sulphate to give the corresponding triester derivative [2], which was converted to the corresponding trihydrazide derivative [3] via the reaction with hydrazine monohydrate. The triimine derivatives [4] and [5] were obtained by reaction of amino groups in trihydrazide derivative [3] with carbonyl group for each furfural and salicylaldehyde, respectively. This reaction called condensation reaction because it is accompanied with releasing H_2O molecule. Mechanism of this reaction was well-known[19]. The tri(1,3-oxazepine) ring derivatives [6-11] were obtained via (2+5) cycloaddition between imine groups for Schiff base [4] and [5] as two-membered components and each maleic anhydride, phthalic anhydride and 3-nitrophthalic anhydride, respectively. These cycloaddition reactions proceed via a cyclic transition state as shown in the following scheme[2,3]:



Scheme(1): Approximate transition state geometry for maleic anhydride addition to imine group

The structures of all prepared compounds were shown in scheme (2). All new compounds were characterized by elementary analysis (C. H. N.) which showed nearness between the calculated and found values as shown in Table (1) and FT-IR spectra as shown in Table (2). FT-IR spectrum of compound [1], tricarboxylic acid, showed appearance of the following characteristic absorption bands: the two weak absorption bands at 3556cm^{-1} and 3425cm^{-1} attributed to the stretching vibrations of phenolic and carboxylic acid hydroxyl groups, respectively. The absorption band at 3059cm^{-1} attributed to the υ(C-H) aromatic of benzene ring. The two strong absorption bands at 1882cm^{-1} and 1655cm^{-1} attributed to the υ(C=O) of ketone and carboxylic acid groups, respectively. The medium absorption band at 1438cm^{-1} due to the υ(C=C) aromatic of benzene

rings. The strong absorption band at 1205cm^{-1} attributed to the υ(C-O) of phenol and carboxylic acid. The absorption band at 775cm^{-1} due to the δ(C-H) aromatic out of plane. The absorption band at 678cm^{-1} attributed to the δ(O-H) out of plane.FT-IR spectrum of compound [2], triester derivative, showed disappearance of the weak absorption bands 3556 cm^{-1} and 3425cm^{-1} of phenolic and carboxylic acid hydroxyl groups and appearance of the following characteristic absorption bands: the strong absorption band at 1676cm^{-1} attributed to the υ(C=O) of ester groups. The weak absorption band at 2931cm^{-1} due to the υ(C-H) aliphatic of (-CH_3) group. The weak absorption at 1350cm^{-1} due to the δ(C-H) aliphatic. The absorption band at 1290cm^{-1} attributed to the asymmetric streaching vibration of (C-O) of ester group. The two absorption bands at 1217cm^{-1} and 1116cm^{-1} attributed to the asymmetric and symmetric stretching vibrations of ether group (C-O-C). The sharp strong absorption band at 1797cm^{-1} attributed to the υ(C=O) of ketone. FT-IR spectrum of compound [2] also showed appearance of another important absorption bands shown in Table (2). FT-IR spectrum of compound [3], trihydrazide derivative, showed disappearance of the strong absorption band at 1676cm^{-1} due to the υ(C=O) of ester groups and appearance of strong absorption band at 1633cm^{-1} attributed to the υ(C=O) of hydrazide groups. Also disappearance of the absorption band at 1290cm^{-1} due to asymmetric stretching vibration of esteric (C-O). FT-IR spectrum of compound [3] also showed appearance of two absorption bands at 3553cm^{-1} and 3491cm^{-1} attributed to the asymmetric and symmetric stretching vibrations of (-NH_2) groups, respectivly. The absorption band at 3201cm^{-1} attributed to the υ(N-H) of hydrazide groups.The absorption band at 1828cm^{-1} due to the υ(C=O) of ketone. FT-IR spectrum of compound [3] also showed appearance of another important absorption bands shown in Table (2).

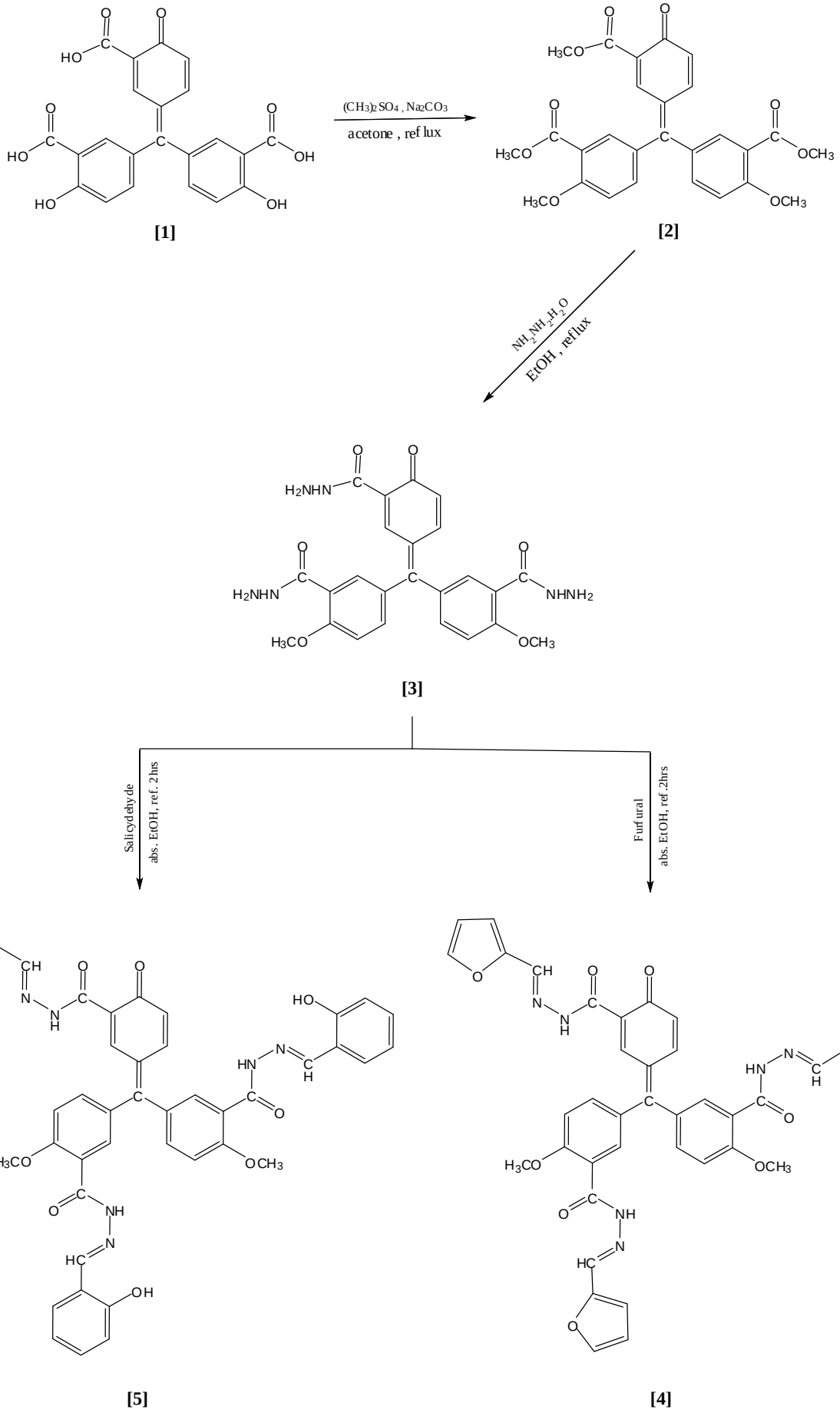
FT-IR spectrum of imine derivative [4] showed disappearance of the two absorption bands at 3553cm^{-1} and 3491cm^{-1} attributed to the asymmetric and symmetric stretching vibrations of (-NH_2) group, respectively and appearance of strong broad characteristic absorption band at 1640cm^{-1} attributed to the υ(C=N), this band appeared broad due to the interaction with υ(C=O) of amide group[30]. Furthermore, FT-IR spectrum of compound [4] showed appearance of another identical absorption bands found in Table (2). FT-IR spectrum of imine derivative [5] showed disappearance of the two absorption bands at 3553cm^{-1} and 3491cm^{-1} attributed to the asymmetric and symmetric stretching vibrations of (-NH_2) group, respectively and appearance of the following characteristic absorption bands the strong band at 1620cm^{-1} attributed to the υ(C=N), while υ(C=O) of amide group appeared as medium absorption band at 1680cm^{-1}. The three sharp absorption bands at 3460cm^{-1}, 3370cm^{-1} and 3290cm^{-1} attributed to the υ(O-H) due to the intermolecular hydrogen bonding[30,31]. Also appearance of medium absorption band at 1199cm^{-1} due to υ(C-O) of phenol. FT-IR spectrum of compound [5] also showed appearance of another identical absorption bands found in Table (2). FT-IR spectrum of 1,3-oxazepine derivative [6] showed disappearance of the strong broad band at the range (1660-1630)cm^{-1} attributed to the υ(C=N) and appearance of the following characteristic absorption bands: the strong absorption band at 1666cm^{-1} attributed to the υ(C=O) of lactam in 1,3-oxazepine ring[30,31]. The strong absorption band at 1730cm^{-1} attributed to the υ(C=O) of lactone in 1,3-oxazepine ring[30,31]. FT-IR spectrum of compound [6] also showed appearance of another identical absorption bands found in Table (2). FT-IR spectrum of 1,3-oxazepine derivative [7] showed disappearance of the strong broad band at the range (1660-1630)cm^{-1} attributed to the υ(C=N) and appearance of the following characteristic absorption bands: the medium band at 1674cm^{-1} attributed to the υ(C=O) of lactam in 1,3-oxazepine ring[30,31]. The strong band at 1739cm^{-1} attributed to the υ(C=O) of lactone in 1,3-oxazepine ring[30,31]. Furthermore, FT-IR spectrum of compound [7] showed appearance of another identical absorption bands found in Table (2). FT-IR spectrum of 1,3-oxazepine derivative [8] showed disappearance of the strong broad band at the range (1660-1630)cm^{-1} attributed to the υ(C=N) and appearance of the following characteristic absorption bands: the strong band at 1660cm^{-1} due to the υ(C=O) of lactam in 1,3-oxazepine ring[30,31]. The medium absorption band at 1745cm^{-1} attributed to the υ(C=O) of lactone in 1,3-oxazepine ring[30,31]. The two strong absorption bands at 1537cm^{-1} and 1298cm^{-1} attributed to the asymmetric and symmetric stretching vibrations of (-NO_2) group[30,31]. Also appearance strong absorption band at 902cm^{-1} due to the

stretching vibrations of (C-NO_2) bond is good evidence for the structure given to the product[31]. FT-IR spectrum of compound [8] also showed appearance of another identical absorption bands found in Table (2). FT- IR spectrum of 1,3-oxazepine derivative [9] showed disappearance of the strong absorption band at 1615cm^{-1} attributed to the υ(C=N) and appearance of the following characteristic absorption bands: the strong absorption band at 1765cm^{-1} attributed to the υ(C=O) of lactone in 1,3-oxazepine ring[30,31]. The strong absorption band at 1650cm^{-1} attributed to the υ(C=O) of lactam in 1,3-oxazepine ring[30,31]. FT-IR spectrum of compound [9] also showed appearance of another absorption bands found in Table (2). FT-IR spectrum of 1,3-oxazepine derivative [10] showed disappearance of the strong absorption bond at 1615cm^{-1} due to the υ(C=N) and appearance of the following characteristic absorption bands: the strong absorption band at 1732cm^{-1} attributed to the υ(C=O) of lactone in 1,3-oxazepine ring[30,31]. The strong absorption band at 1642cm^{-1} due to the υ(C=O) of lactam in 1,3-oxazepine ring[30,31]. FT-IR spectrum of compound [10] also showed appearance of another identical absorption bands found in Table (2). FT-IR spectrum of 1,3-oxazepine derivative [11] showed appearance of the following characteristic absorption bands: the strong absorption band at 1710cm^{-1} attributed to the υ(C=O) of lactone in 1,3-oxazepine ring[30,31]. The strong absorption band at 1620cm^{-1} attributed to the υ(C=O) of lactam in 1,3-oxazepine ring[30,31]. The two medium absorption bands at 1564cm^{-1} and 1330cm^{-1} attributed to the asymmetric and symmetric stretching vibrations of (-NO_2) group[30,31]. Beside this, appearance of strong absorption band at 852cm^{-1} due to the stretching vibrations of δ(C-NO_2) bond is good evidence for the structure given to the product[31]. FT-IR spectrum of compound [11] also showed appearance of another identical absorption bands found in Table (2).

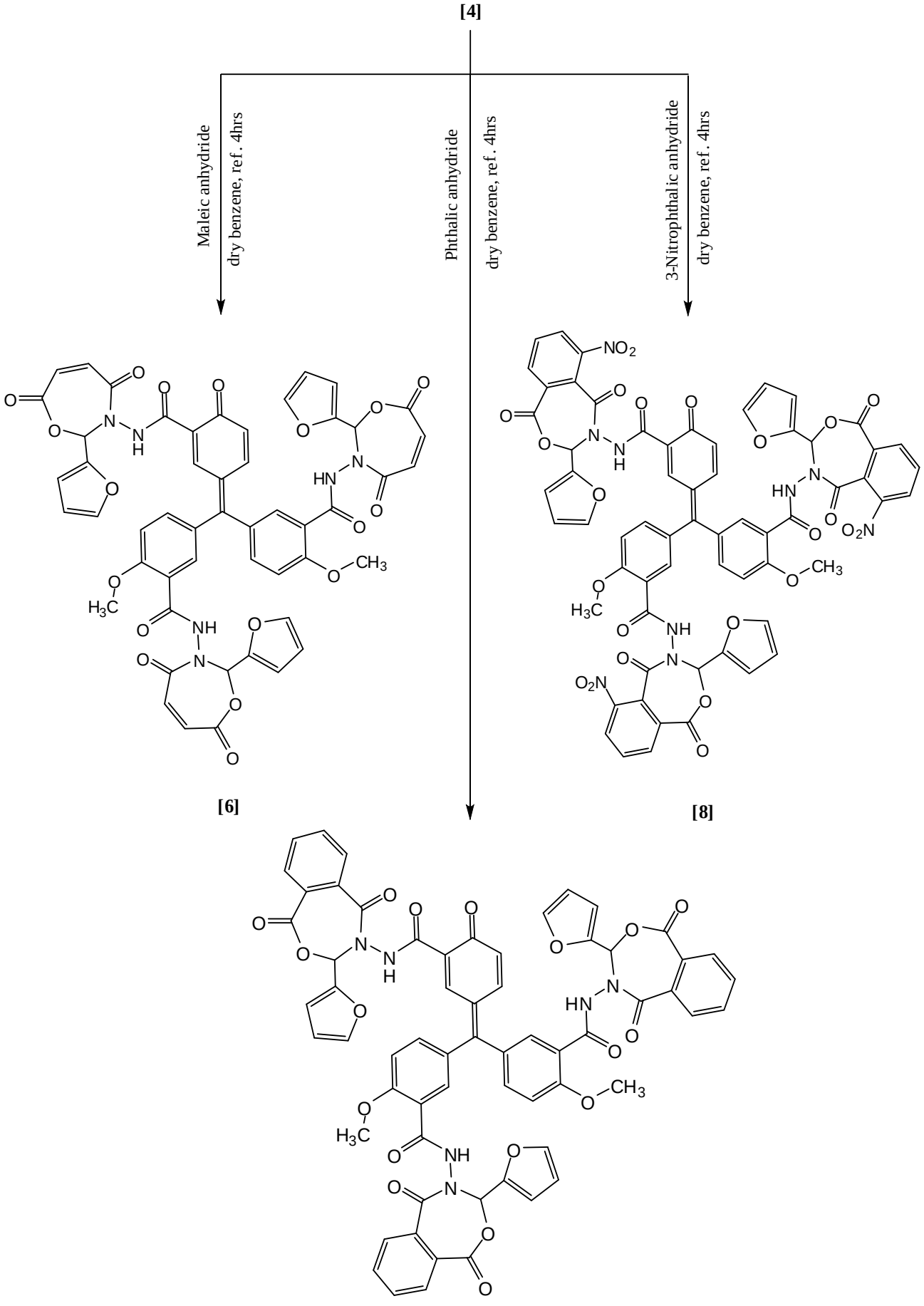
sp=sharp, w=weak, m=medium, s=strong, o.o.p.= out of plane

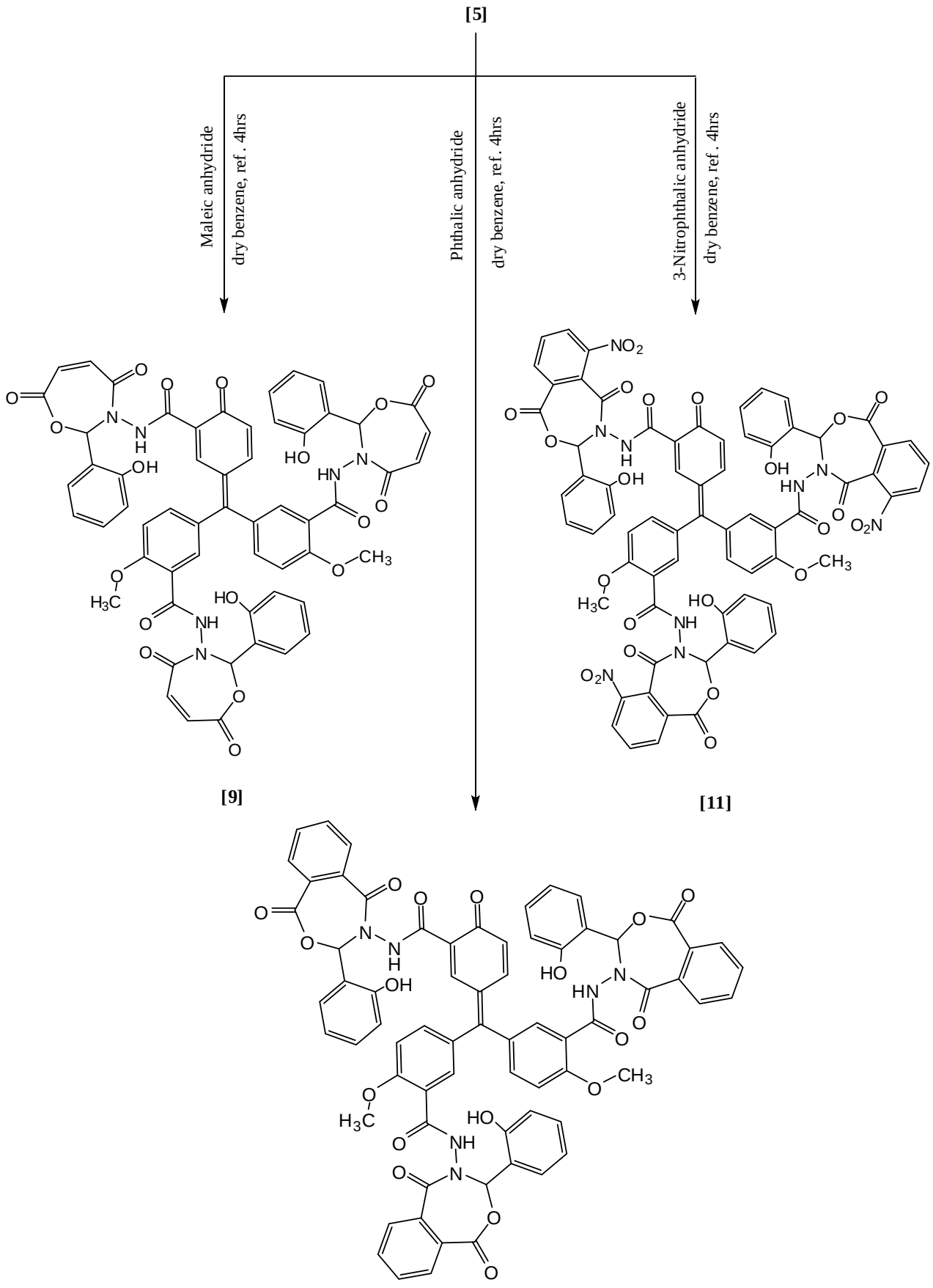
Table(2): FT-IR Data of the prepared compounds [1-11] in cm^{-1}

Comp No.	υ / O-H	υ / NH_2	υ / N-H	υ / C-H arom.	υ / C-H aliph.	υ / C=O	υ / C=N	υ / C=C arom.	υ / NO_2	/ δ C-H aliph.	υ / C-O	δ / C-H arom. o.o.p.
[1]	**3556(sp) 3425(sp)**	**---**	**---**	**3059(w)**	**---**	**1882(s) ketone 1655(s) acid**	**---**	**1438(m)**	**---**	**---**	**1205(s)**	**775(m)**
[2]	**---**	**---**	**---**	**3150(w)**	**2931(m)**	**1797(s) ketone 1676(s) ester**	**---**	**1610(m) 1454(m)**	**---**	**1350(w)**	**1290(w) asym. ester 1217(m) asym. ether 1116(w) sym. ether**	**966(s)**
[3]	**---**	**3553(m) 3491(m)**	**3201(m)**	**3070(w)**	**2950(s)**	**1828(m) ketone 1633(s) hydrazide**	**---**	**1456(s)**	**---**	**1373(s)**	**1234(m) asym. ether 1109(s) sym. ether**	**813(m)**
[4]	**---**	**---**	**3309(m) 3240 (m)**	**3066(w) 3040(w)**	**2925(w) 2889(w)**	**1830(m) ketone 1640(s) amide**	**1640(s)**	**1458(s)**	**---**	**1359(s)**	**1215(s) asym. ether 1115(s) sym. ether**	**950(s) 823(s) 738(s)**
[5]	**3460(sp) 3370(sp) 3290(sp)**	**---**	**3190(m)**	**3120(w) 3030(w)**	**2920(w) 2850(w)**	**1844(m) ketone 1680(m) amide**	**1615(s)**	**1475(s)**	**---**	**1369(m)**	**1265(s) asym. ether 1136(m) sym. ether 1199(m) phenol**	**970(s) 746(s)**
[6]	**---**	**---**	**3310(m) 3240(m)**	**3140(w) 3061(w)**	**2947(m) 2860(w) 2800(w)**	**1826(s) ketone 1730(s) lacton 1666(s) lactam**	**---**	**1600(s) 1539(m) 1460(s)**	**---**	**1359(m)**	**1298(s) lactone 1203(s) asym. ether 1126(s) sym. ether**	**941(s) 835(s) 750(s)**
[7]	**---**	**---**	**3320(w) 3230(w) 3190(w)**	**3130(w) 3061(w)**	**2980(w) 2940(w) 2870(w)**	**1797(m) ketone 1739(s) lactone 1674(m) lactam**	**---**	**1595(s) 1537(m) 1455(s)**	**---**	**1356(s)**	**1300(s) lactone 1205(s) asym. ether 1130(s) sym. ether**	**940(s) 880(m) 810(s) 750(s)**
[8]	**---**	**---**	**3350(w) 3300(w) 3250(w)**	**3140(w) 3064(w)**	**2976(w) 2910(w) 2850(w)**	**1824(s) ketone 1745(s) lactone 1660(s) lactam**	**---**	**1599(s) 1456(s)**	**1537(s) 1298(s)**	**1352(s)**	**1255(m) lactone 1205(s) asym. ether 1120(s) sym. ether**	**750(s)**
[9]	**3510(sp) 3350(sp) 3284(sp)**	**---**	**3173(w)**	**3072(w)**	**2935(w)**	**1844(m) ketone 1765(s) lactone 1650(s) lactam**	**---**	**1530(s) 1490(m)**	**---**	**1435(w) 1384(s)**	**1235(s) lactone 1255(s) asym. ether 1074(s) sym. ether 1190(s) phenol**	**933(s) 745(s)**
[10]	**3470(sp) 3370(sp) 3284(sp)**	**---**	**3200(w)**	**3150(w) 3130(w)**	**2985(w) 2910(w)**	**1840(m) ketone 1732(s) lactone 1642(s) lactam**	**---**	**1560(w) 1485(s)**	**---**	**1375(w)**	**1240(s) lactone 1267(s) asym. ether 1100(m) sym. ether 1180(m) phenol**	**972(s) 744(s)**
[11]	**3400 3300 3263(sp)**	**---**	**3200(w)**	**3111(w) 2999(w)**	**2930(w) 2800(w) 2710(w)**	**1826(s) ketone 1710(s) lactone 1620(s) lactam**	**---**	**1481(s)**	**1564(m) 1330(m)**	**1425(w) 1375(m)**	**1235(s) lactone 1269(s) asym. ether 1150(m) sym. ether 1197(s) phenol**	**970(s) 750(s)**

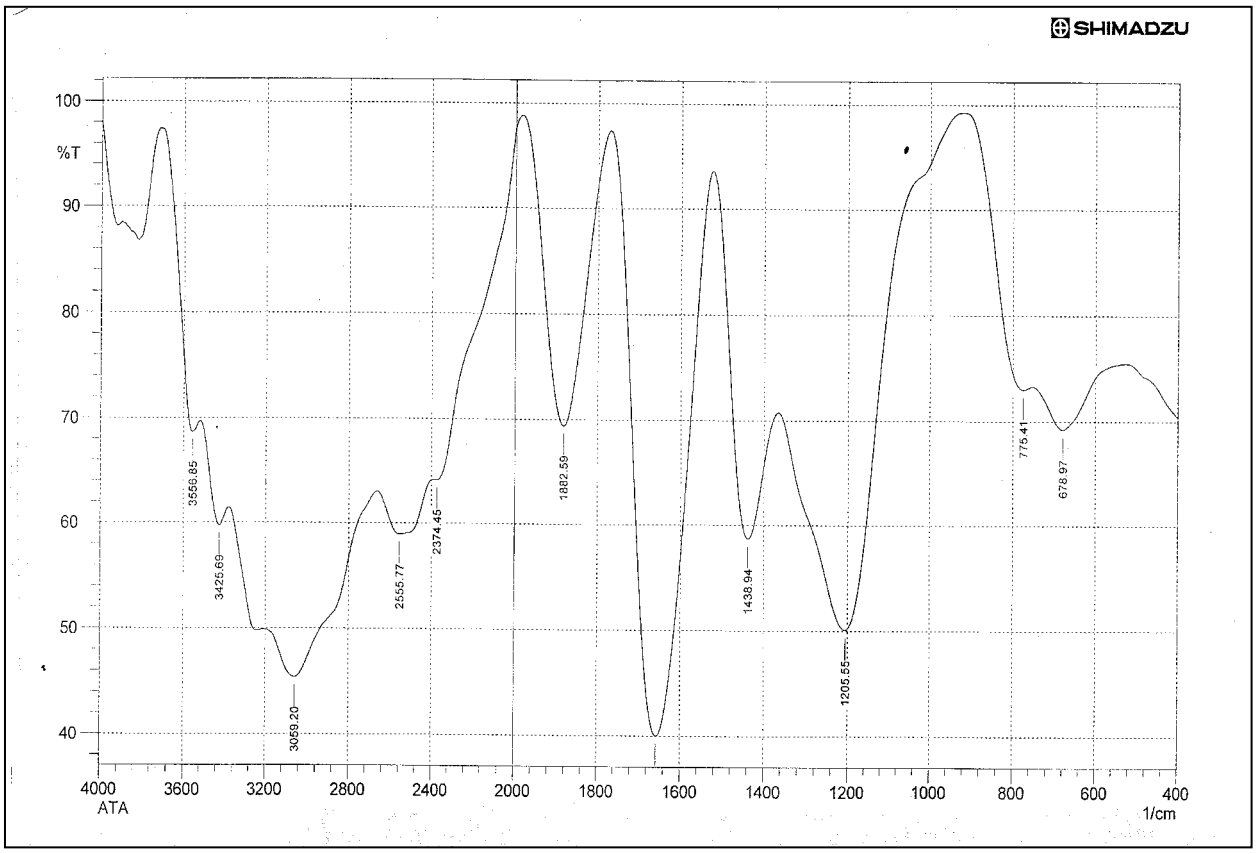

(CH3)2SO4 , Na2CO3
acetone , reflux
NH2NH2.H2O
EtOH , reflux
Salicydehyde
abs. EtOH, ref. 2 hrs
Furfural
abs. EtOH, ref. 2hrs

[1] **[2]** **[3]** **[5]** **[4]**

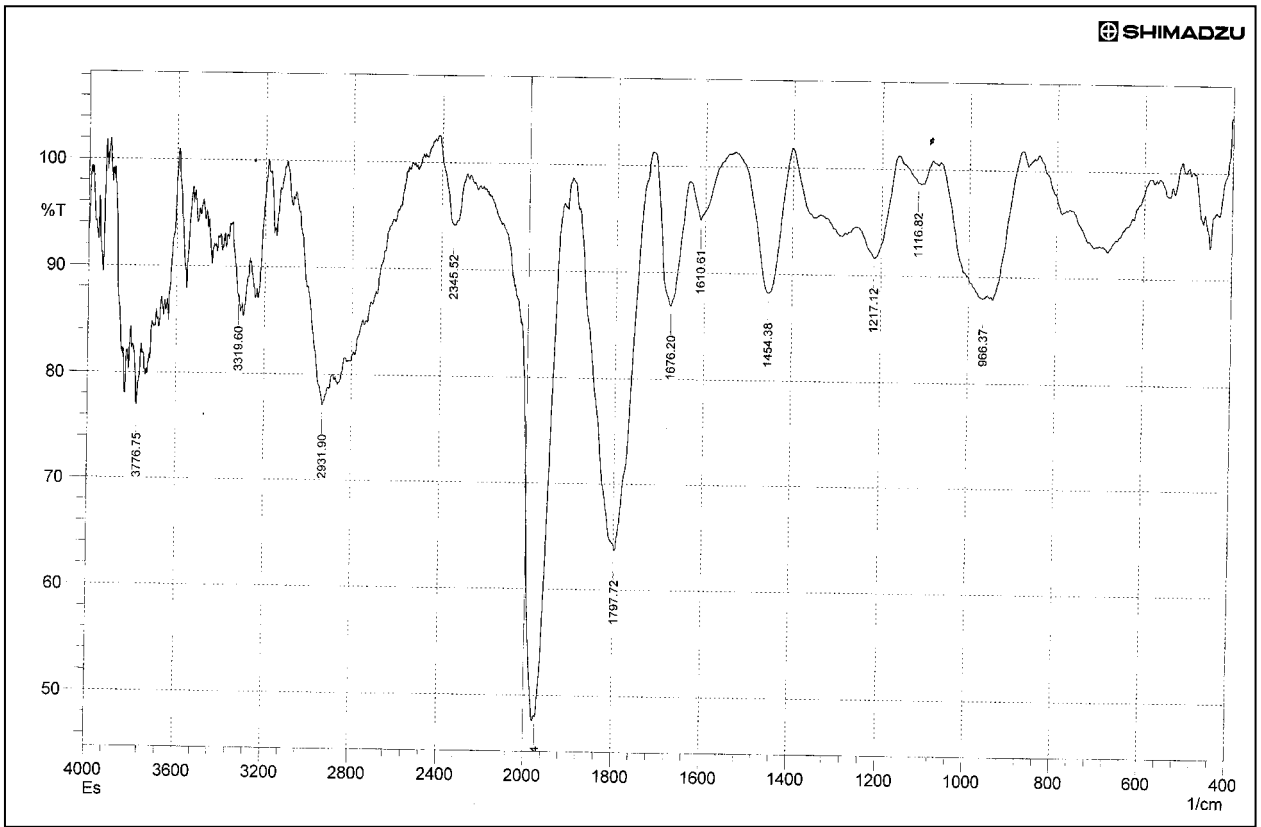

[4]
Maleic anhydride
dry benzene, ref. 4hrs
Phthalic anhydride
dry benzene, ref. 4hrs
3-Nitrophthalic anhydride
dry benzene, ref. 4hrs
[6]
[8]
[7]



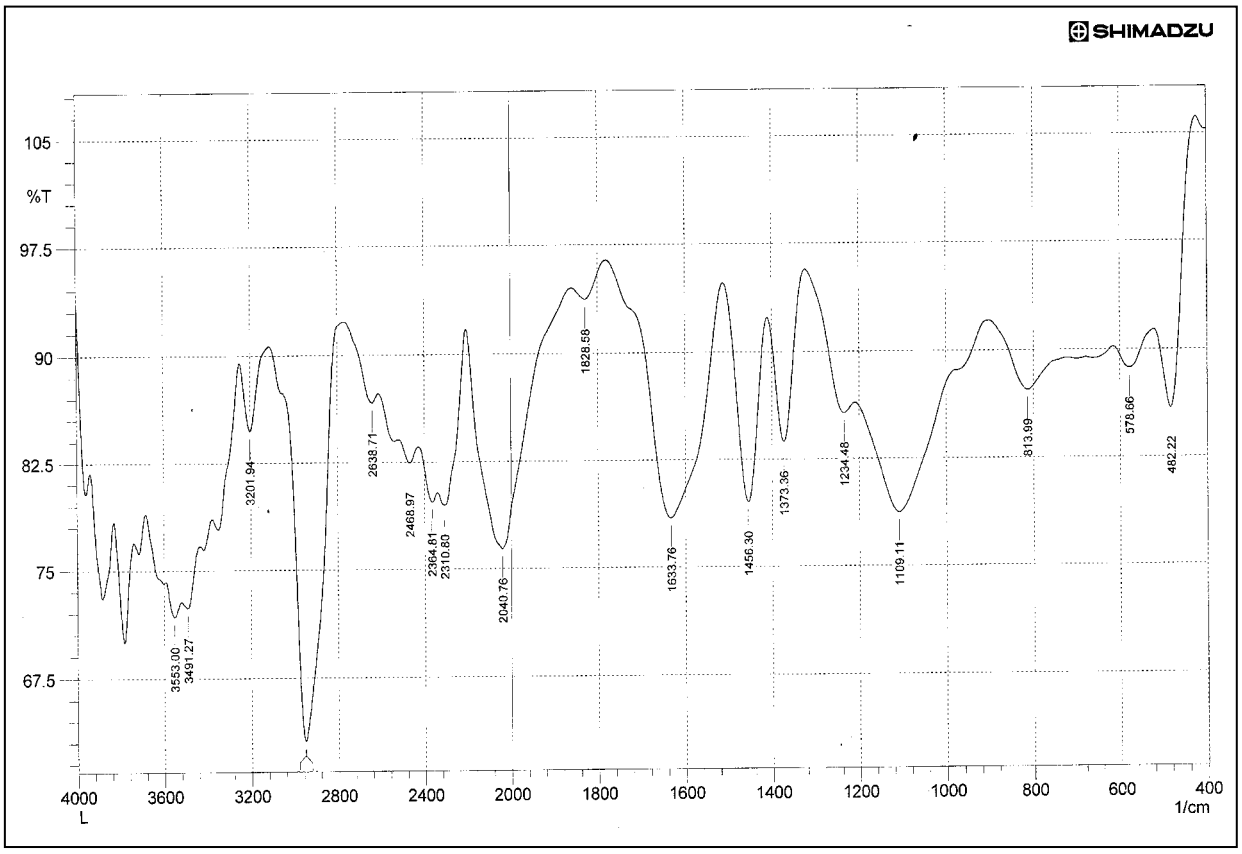
Scheme (2): Reactions proceeding



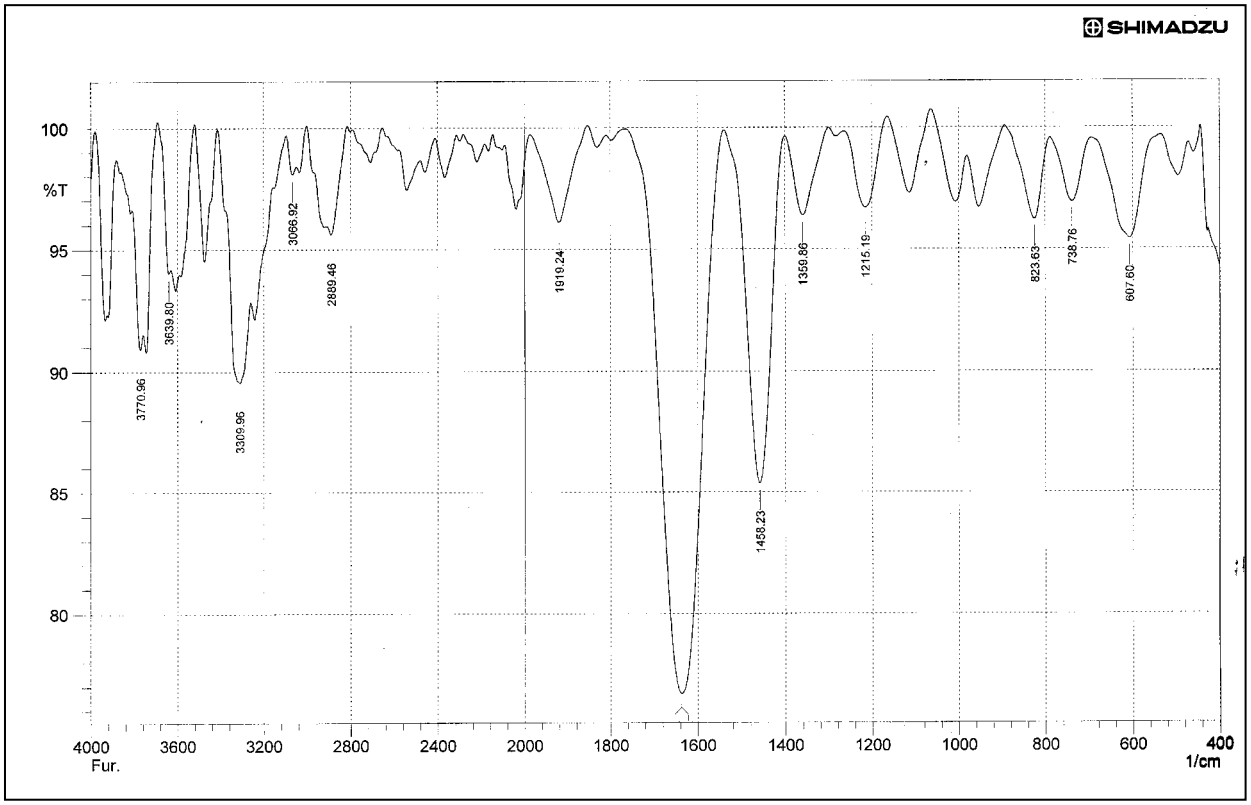
FT-IR spectrum of compound [1]



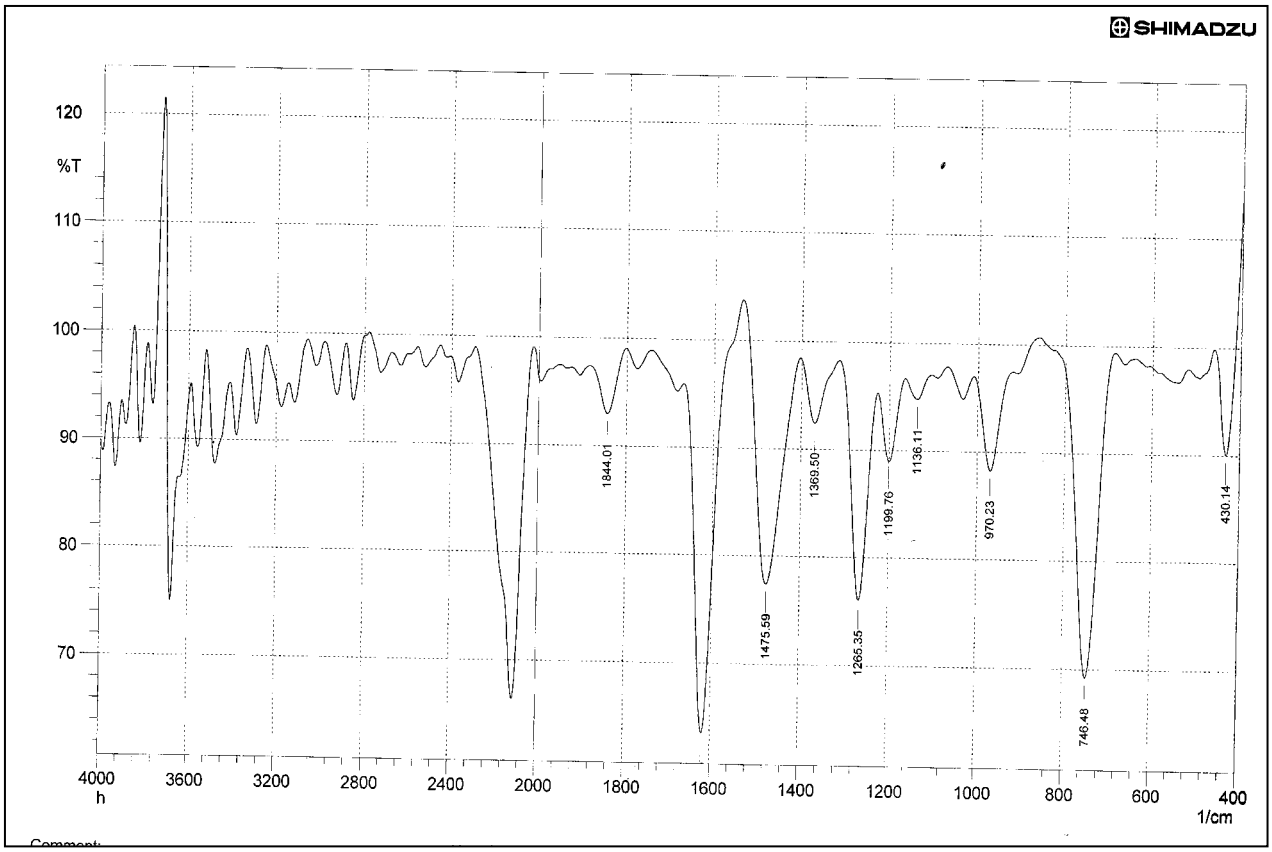
FT-IR spectrum of compound [2]



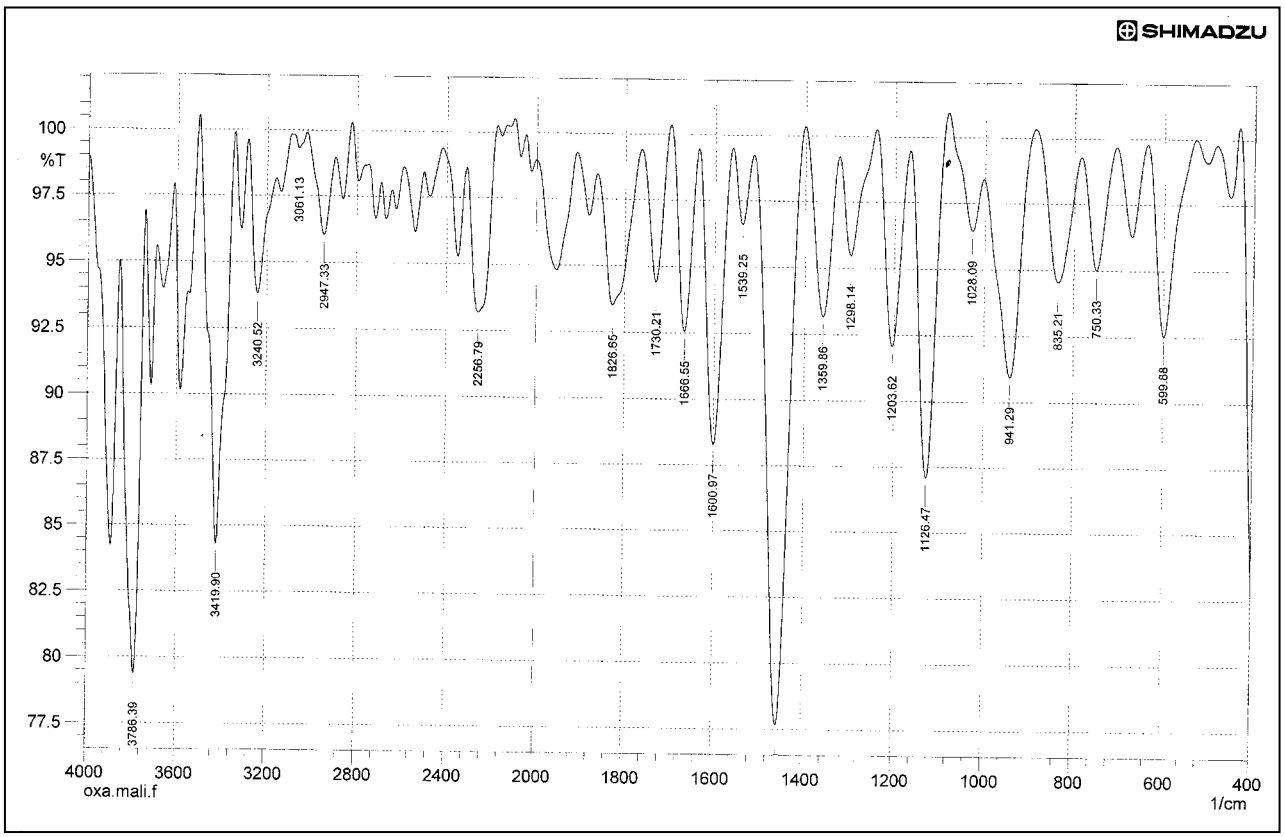
FT-IR spectrum of compound [3]



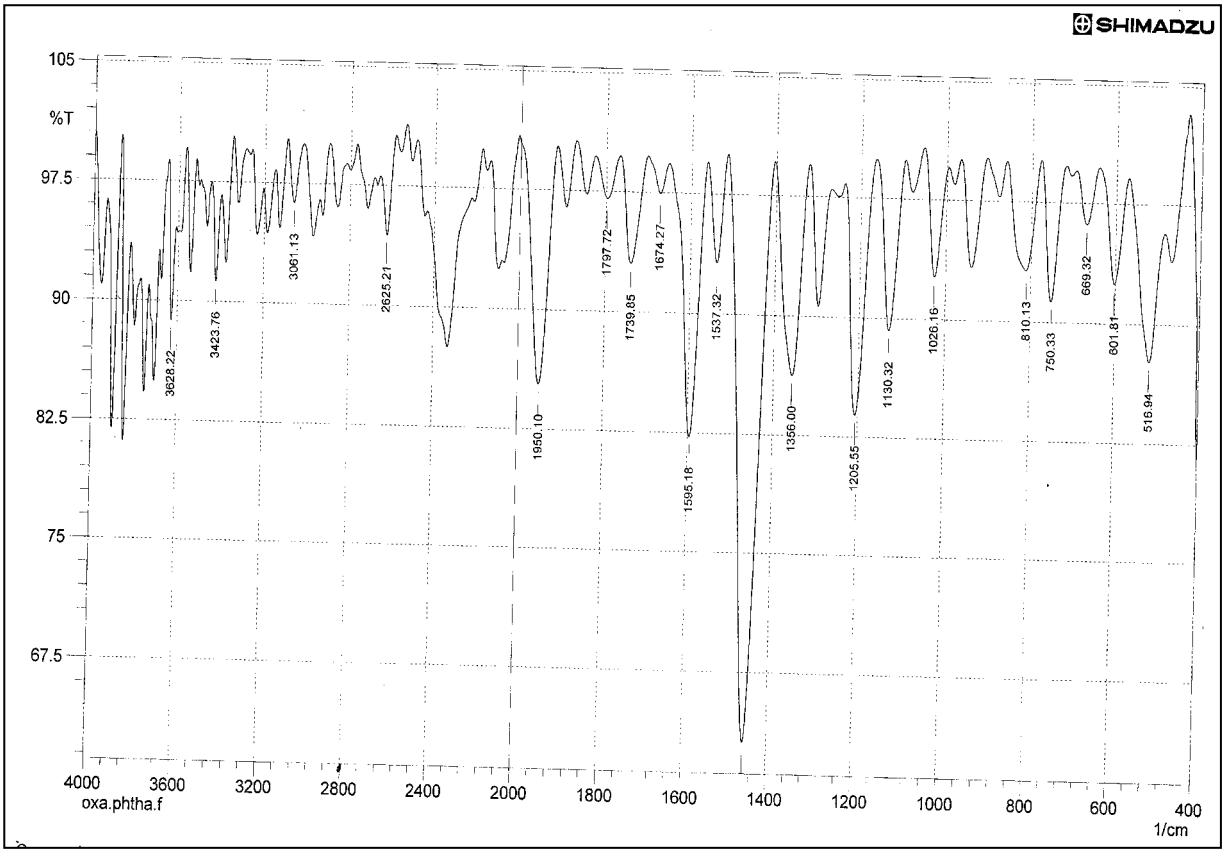
FT-IR spectrum of compound [4]



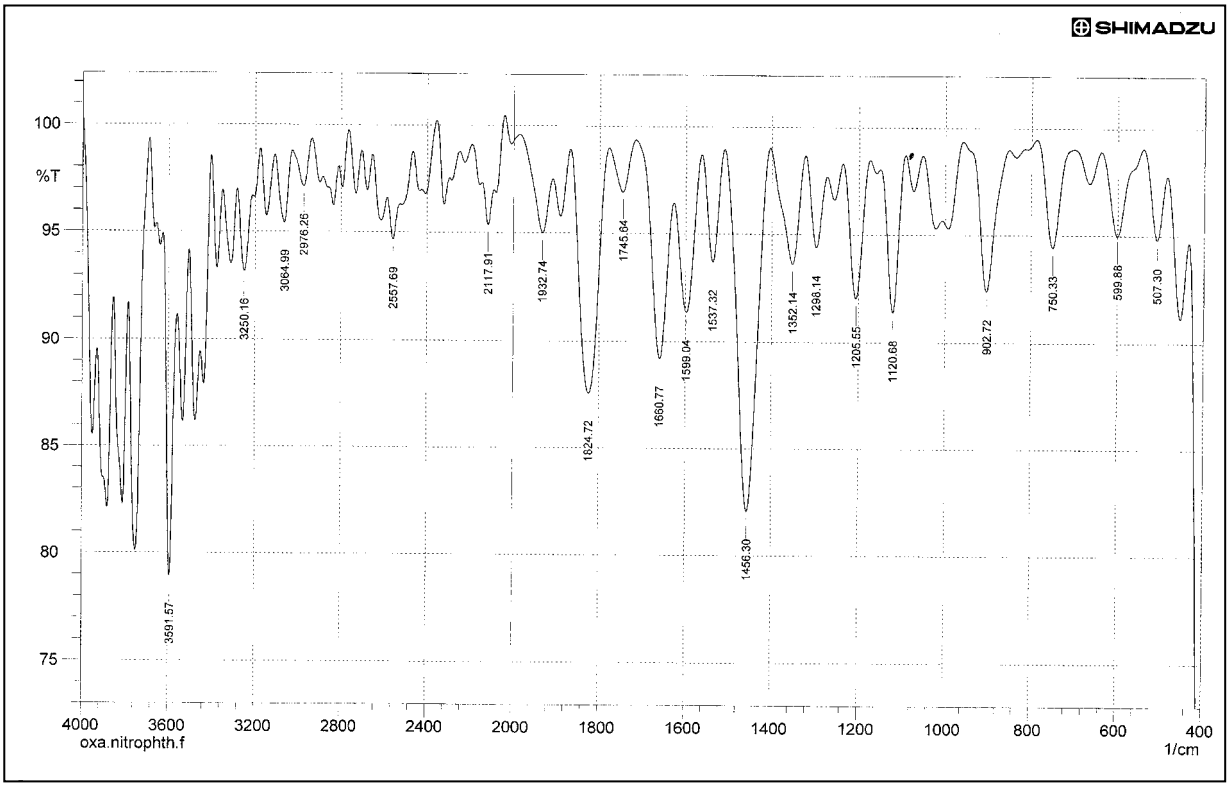
FT-IR spectrum of compound [5]



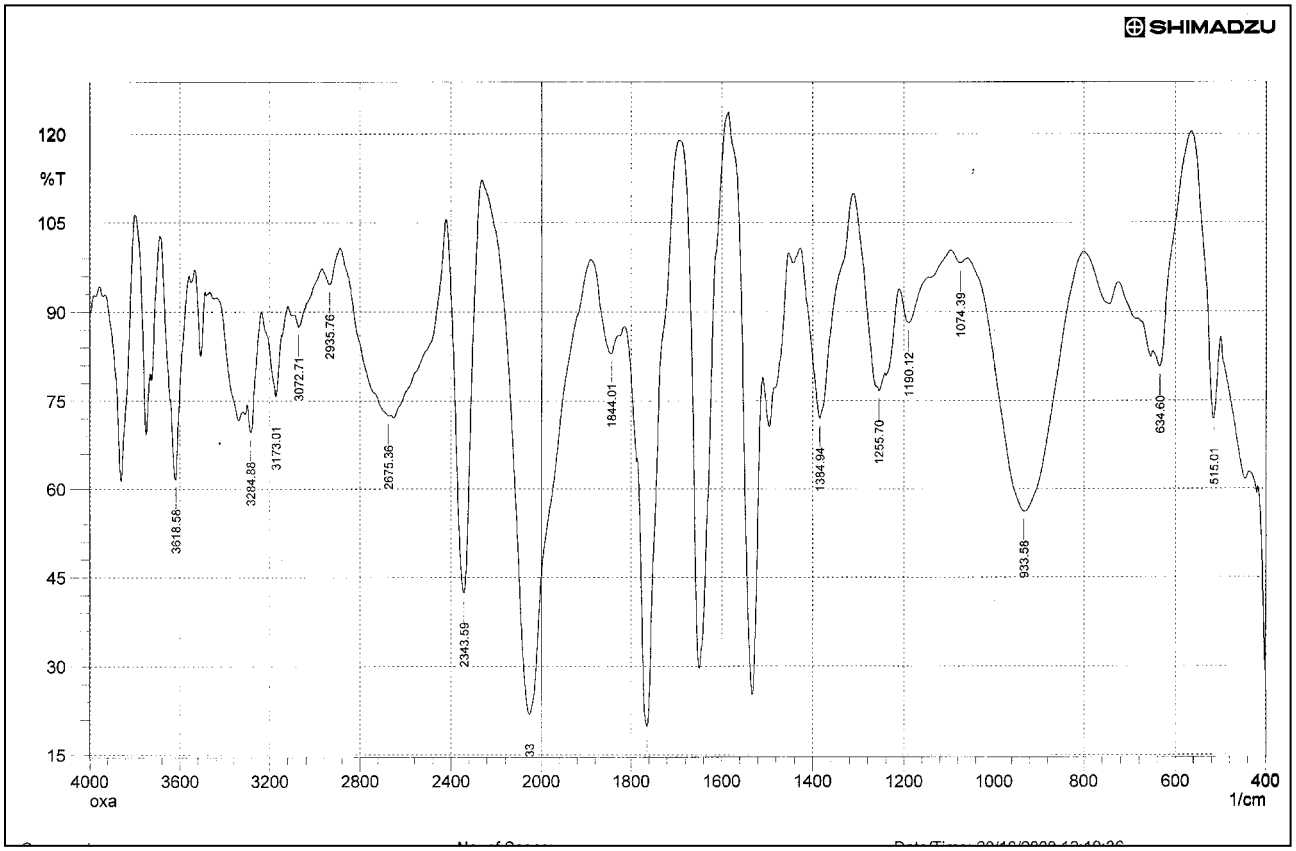
FT-IR spectrum of compound [6]



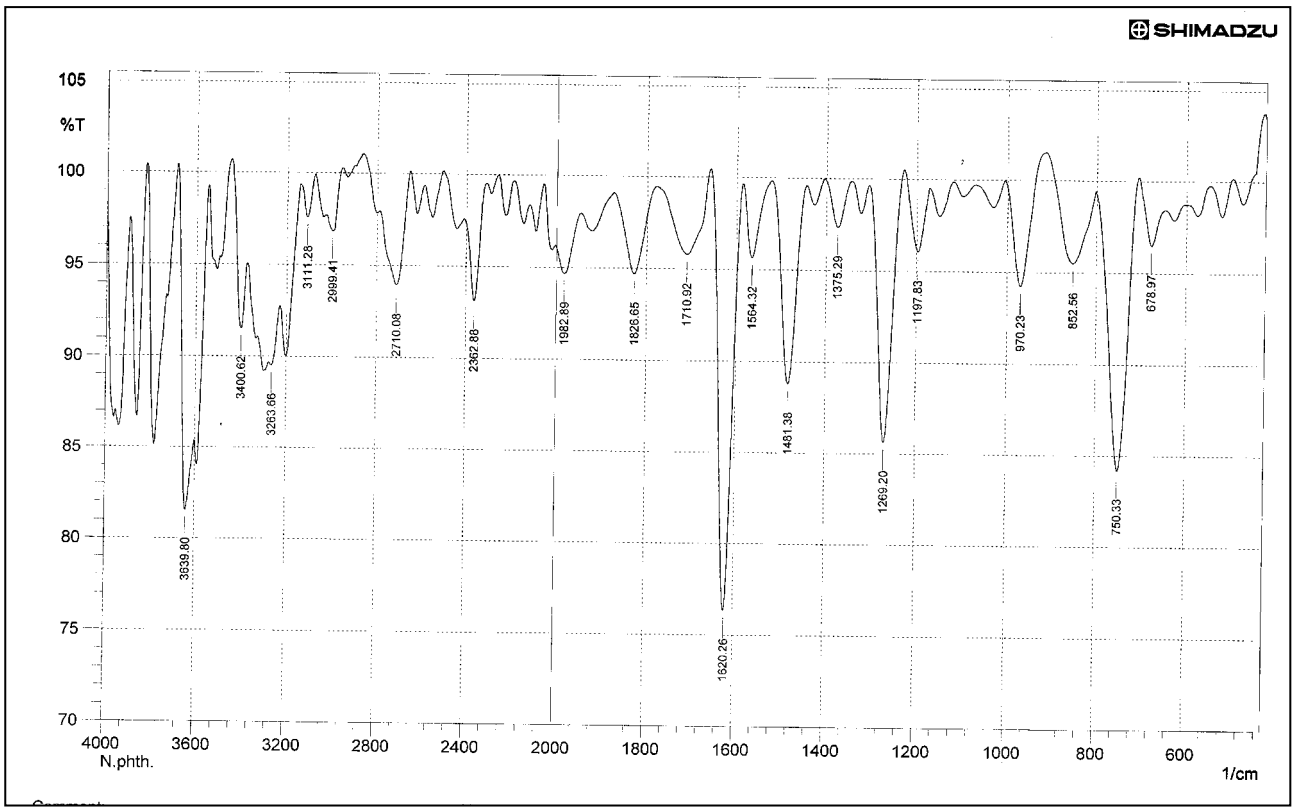
FT-IR spectrum of compound [7]



FT-IR spectrum of compound [8]



FT-IR spectrum of compound [9]



FT-IR spectrum of compound [11]

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