Synthesis of Novel 1, 3 -Oxazepine Compounds from New Azo Schiff bases Containing Thiadiazole Moiety

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Abstract

In this work new Azo imines [3-4] and 1,3-oxazepine derivatives [5-10] were prepared starting from azoaldehyde derivative 2-Hydroxy-5-(5-thiole-[1,3,4] -thiadiazol-2-ylazo)-benzaldehyde [2]. Azoaldehyde derivative [2] was prepared via coupling reaction between phenoxide anion of ***o***-Hydroxy benzaldehyde [1] and 5-thiole-1,3,4-thiadiazol-2-yl diazonium chloride.The new imines [3] and [4] were prepared by reaction of azoaldehyde derivative [2] with each primary amines 2-Amine-1,3,4-thiadiazole -5- thiol and 5-(Benzylthio)-1,3,4-thiadiazole-2- amine in presence of glacial acetic acid as catalyst in absolute ethanol , respectively. The new 1,3-oxazepine derivatives [5-10] were obtained from treatment of each new Schiff bases derivatives [4] and [5] with each maleic anhydride , phthalic anhydride and 3-Nitrophthalic anhydride ,respectively , in dry benzene.These new prepared compounds might have some biological activity.

All new derivatives were characterized by (C.H.N.) elementary analysis and FT-IR spectra.

الخلاصة:-

تم في هذا البحث تحضير مشتقات ايمينات [3-4] و 1،3- اوكسازيبين [5-10] جديدة من مشتق الازوالديهايد 2- هيدروكسي -5-(5-مركبتو-[4,3,1]- ثايادايازول-2-يل ازو) - بنزالديهايد [2] .حضر مشتق الازوالديهايد [2] من خلال تفاعل الازدواج ما بين ايون الفينوكسايد لمركب ***o*** - هيدروكسي بنزالديهايد و 5 - مركبتو-4,3,1 - ثايا دايازول - 2- يل دايازونيوم كلورايد . حضرت قواعد شيف الجديدة [3] و[4] عن طريق تفاعل مشتق الازوالديهايد [2] مع كل من 2- امينو -4,3,1- ثايادايازول-5- ثايول و5-(بنزايل ثايو)- 4,3,1-ثايادايازول-2- امين .على التوالي ، بوجود حامض الخليك الثلجي كعامل مساعد في الايثانول المطلق.تم الحصول على مشتقات 1،3 – اوكسازيبين الجديدة [5-10] من معاملة كل من مشتقات قواعد شيف المحضرة [3] و [4] مع كل من انهدريد الماليك و انهدريد الفثاليك و 3-نترو انهدريد الفثاليك ،علي التوالي ،في البنزين الجاف. أن هذة المشتقات الجديدة قد تمتلك فعالية حيوية . شخصت جميع المشتقات الجديدة بوساطة التحليل الكمي الدقيق للعناصر (C.H.N.) واطياف الاشعة تحت الحمراء.

Introduction

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(1) . 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(2). Recently, cycloaddition reaction, which is a type from a pericyclic reactions is used to synthesis of 1,3-oxazepine ring(3-6). 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(7-9).

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(10) and inhibitors for some enzymes action(11). Some of oxazepine derivatives are used in another applied fields(12).

Schiff bases or imines are prepared via acid-catalysed condensation reaction of aromatic aldehydes or ketones with primary amines (13,14).Azo Schiff bases are prepared by reaction of azoaldehydes with primary amines (15) . Mechanism of Schiff base formation was well known (13,14).Various azo Schiff bases derivatives were prepared and some of them showed biological activity such as anticancer (16) antiviral (17),antifungal (18) , antibacterial (19) and anticonvulsant (20) .Thiadiazoles have a variety of potential biological activities (21,22) , therefore a large number of thiadiazole derivatives have been prepared (23,24) .Many synthesis of 1,3,4-thiadiazoles proceed from thiosemicarbazide or substituted thiosemicarbazide (25,26) .

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) TLC were performed on pre-coated sheets with 0.25 mm layer of Silica Gel GF254 of the Merck company, the detection was followed by coloring 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.
6) FT-IR spectra were recorded on FT-IR 8400_S, Schimadzu-Spectrophotometer and using KBr discs.

Preparation Methods:

Synthesis of 2-Hydroxy-5-(5-mercapto-[1,3,4] -thiadiazol-2-ylazo)-benzaldehyde [2] (27)

2-Amino-1,3,4-thiadiazole-5-thiol (2g ,0.015mole) was dissolved in a solution of conc. hydrochloric acid (16m**L**) and distilled water (10m**L**). The mixture was cold at (0ºC) in an ice bath . a solution of sodium nitrite (1.3g ,0.0188 mole) in (10m**L**) of distilled water was added dropwise with stirring to the mixture, the temperature of the ice bath was controlled between (0-5ºC) . A solution of (1.834g ,0.015 mole) of *O*-Hydroxybenzaldehyde [1] in (15m**L**) of (10%) sodium hydroxide solution was prepared and cold to (5ºC) by immersion in an ice bath. The salicyldehyde solution was stirred vigorously ,the cold diazonium salt solution was added very slowly to the salicyldehde solution , a red colour developed and red crystals soon separated .when all the diazonium salt solution was added , the mixture was allowed to stand in an ice bath for 30 min.with occasional stirring. The solution was filtered , washed well with distilled water then with alittle alcohol , recrystallized from ethanol and dried upon filter paper,yield 66%,M.P.178-180ºC.

Synthesis of 4-((Z)-(5-mercapto-1,3,4-thiadiazol-2-yl)diazenyl)-2-((Z)-(5-mercapto-1,3,4-thiadiazol-2-ylimino)methyl)phenol [3]

Azobenzaldehyde derivative [2](0.532g , 0.002 mole) was dissolved in absolute ethanol (15 mL) containing a drop of glacial acetic acid ,then 2-Amino-1,3,4-thiadiazole-5-thiol (0.266 g ,0.002 mole) was dissolved in absolute ethanol (15 mL) and added dropwise . The reaction mixture was refluxed with stirring on a water bath at (70 ^{o}C) for 2hrs.T.L.C. (ethanol:pet.ether)(1:1) ,R_f = 0.63.Then the mixture was allowed to cool down to room temperature , the coloured precipitate was filtered and recrystallized from ethanol ,yield 68% , M.P.220-222ºC.

Synthesis of 2-((Z)-(5-(benzylthio)-1,3,4-thiadiazol-2-ylimino)methyl)-4-((Z)-(5-mercapto-1,3,4-thiadiazol-2-yl)diazenyl)phenol [4]

Azobenzaldehyde derivative [2](0.532g , 0.002 mole) was dissolved in absolute ethanol (15 mL) containing a drop of glacial acetic acid ,then 5-(butylthio)-1,3,4-thiadiazol-2-amine (0.378 g ,0.002 mole) was dissolved in absolute ethanol (15 mL) and added dropwise . The reaction mixture was refluxed with stirring on a water bath at (70 ^oC) for 2hrs.T.L.C. (ethanol:pet.ether)(1:1) ,R_f = 0.59.Then the mixture was allowed to cool down to room temperature , the coloured precipitate was filtered and recrystallized from ethanol ,yield 70% , M.P.160-162ºC.

Synthesis of (Z)-2-(2-hydroxy-5-((Z)-(5-mercapto-1,3,4-thiadiazol-2-yl)diazenyl)phenyl)-3-(5-mercapto-1,3,4-thiadiazol-2-yl)-2,3-dihydro-1,3-oxazepine-4,7-dione [5]

A mixture of imine derivative [3](0.381 g, 0.001 mole) and maleic anhydride (0.098 g ,0.001 mole) in dry benzene (20 m**L)** was refluxed on a water bath at (75ºC) for 4hrs. T.L.C. (benzene:methanol) (3:1) R_f = 0.70. , then the mixture was allowed to cool down to room temperature . The resulting solid crystals were filtered and recrystallized from dioxan, yield 65% , M.P.180-182 ºC.

Synthesis of (Z)-3-(2-hydroxy-5-((5-mercapto-1,3,4-thiadiazol-2-yl)diazenyl)phenyl)-4-(5-mercapto-1,3,4-thiadiazol-2-yl)-3,4-dihydrobenzo[e][1,3]oxazepine-1,5-dione [6]

A mixture of imine derivative [3](0.381g, 0.001 mole) and phthalic anhydride (0.148g ,0.001mole) in dry benzene (20 m**L)** was refluxed on a water bath at (75ºC) for 4hrs. T.L.C. (benzene:methanol) (3:1) R_f = 0.74. , then the mixture was allowed to cool down to room temperature . The resulting solid crystals were filtered and recrystallized from dioxan, yield 71% , M.P.174-176 ºC.

Synthesis of (Z)-3-(2-hydroxy-5-((5-mercapto-1,3,4-thiadiazol-2-yl)diazenyl)phenyl)-4-(5-mercapto-1,3,4-thiadiazol-2-yl)-6-nitro-3,4-dihydrobenzo[e][1,3]oxazepine-1,5-dione [7]

A mixture of imine derivative [3](0.381 g, 0.001 mole) and 3-Nitro phthalic anhydride (0.193 g ,0.001mole) in dry benzene (20 m**L)**, was refluxed on a water bath at (75ºC) for 4hrs. T.L.C. (benzene:methanol) (3:1) R_f = 0.79. , then the mixture was allowed to cool down to room temperature . The resulting solid crystals were filtered and recrystallized from dioxan, yield 66% , M.P.200-202 ºC.

Synthesis of (Z)-4-(5-(Benzylthio)-1,3,4-thiadiazol-2-yl)-3-(2-hydroxy-5-((5-mercapto-1,3,4-thiadiazol-2-yl)diazenyl)phenyl)-3,4-dihydrobenzo[e][1,3]oxazepine-1,5-dione[8]

A mixture of imine derivative [4](0.437 g, 0.001 mole) and maleic anhydride (0.098 g ,0.001mole) in dry benzene (20 m**L)** was refluxed on a water bath at (75ºC) for 4hrs. T.L.C. (benzene:methanol) (3:1) R_f = 0.63 then the mixture was allowed to cool down to room temperature . The resulting solid crystals were filtered and recrystallized from dioxan, yield 70%, M.P.280-282 ºC.

Synthesis of 8-(5-Benzylsulfanyl-[1,3,4]thiadiazol-2-yl)-7-[2-hydroxy-5-(5-mercapto-[1,3,4]thiadiazol-2-ylazo)-phenyl]-7,8-dihydro-6-oxa-8-aza-benzocycloheptene-5,9-dione[9]

A mixture of imine derivative [4](0.437 g, 0.001 mole) and phthalic anhydride (0.148 g ,0.001mole) in dry benzene (20 m**L)** was refluxed on a water bath at (75ºC) for 4hrs. T.L.C. (benzene:methanol) (3:1) R_f = 0.67 , then the mixture was allowed to cool down to room temperature . The resulting solid crystals were filtered and recrystallized from dioxan, yield 69% , M.P.190-192 ºC.

Synthesis of (Z)-4-(5-(Benzylthio)-1,3,4-thiadiazol-2-yl)-3-(2-hydroxy-5-((5-mercapto-1,3,4-thiadiazol-2-yl)diazenyl)phenyl)-6-nitro-3,4-dihydrobenzo[e][1,3]oxazepine-1,5-dione [10]

A mixture of imine derivative [4](0.437 g, 0.001 mole) and 3-Nitrophthalic anhydride (0.193 g ,0.001mole) in dry benzene (20 m**L)** was refluxed on awater bath at (75ºC) for 4hrs. T.L.C. (benzene:methanol) (3:1) R_f = 0.77 , then the mixture was allowed to cool down to room temperature . The resulting solid crystals were filtered and recrystallized from dioxan, yield 70%, M.P.153-155 ºC.

Table (1): Melting points, percent yields and (C.H.N.)analysis of the prepared compounds (2-10)

Comp. No.	M.P ºC	Yield %	M.F	M. Wt g/mole	C.H.N analysis					
					Calculated%			Found%		
					C	H	N	C	H	N
[2]	178-180	66	$C_9H_6N_4O_2S_2$	266	-	-	-	-	-	-
[3]	220-222	68	$C_{11}H_7N_7OS_4$	381	34.64	1.83	25.72	35.02	2.11	26.01
[4]	160-162	70	$C_{18}H_{13}N_7OS_4$	471	45.85	2,76	20.80	46.16	3.17	21,11
[5]	180-182	65	$C_{15}H_9N_7O_4S_4$	479	37.57	1.87	20.45	37.19	2.13	20.16
[6]	174-176	71	$C_{19}H_{11}N_7O_4S_4$	529	43.10	2.08	18.52	42.81	1.83	18.77
[7]	200-202	66	$C_{19}H_{10}N_8O_6S_4$	574	39.72	1.74	19.51	39.49	1.95	19.34
[8]	280-282	70	$C_{22}H_{15}N_7O_4S_4$	569	46.39	2.63	17.22	46.66	2.89	16.09
[9]	190-192	69	$C_{26}H_{17}N_7O_4S_4$	619	50.40	2.74	15.83	50.67	2.61	16.06
[10]	153-155	70	$C_{26}H_{16}N_8O_6S_4$	664	46.98	2.40	16.86	46.71	2.33	17.04

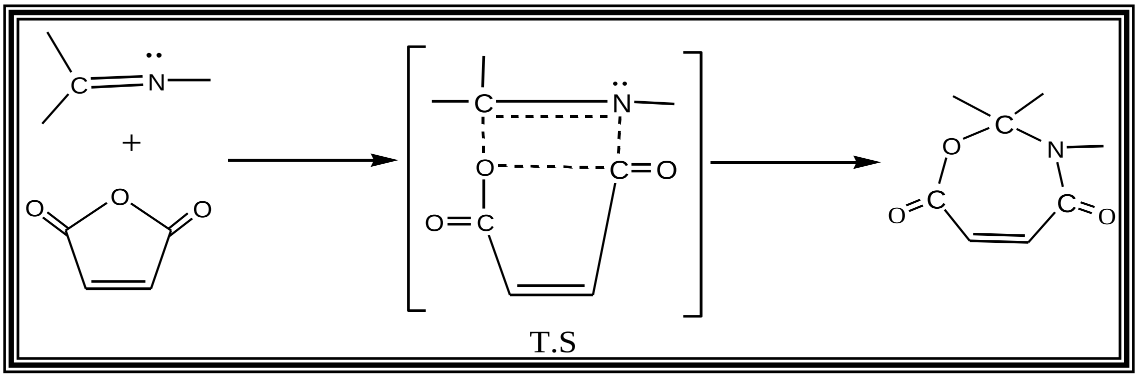
Results and Discussion

2-Amino-1,3,4-thiadiazole-5-thiol was converted to the corresponding diazonium salt which was directly converted to the coupling product ,azoaldehyde derivative [2] ,via coupling reaction with 2-Hydroxy benzaldehyde [1]dissolved in (10%v/v) sodium hydroxide solution(27). FT-IR spectrum of compound [2], azoaldehyde derivative, showed appearance of weak absorption band at 1425 cm^{-1}atributed to the υ (N=N). Also ,disappearance of the two absorption bands at 3392cm^{-1} and 3279 cm^{-1} attributed to the (-NH_2) stretching frequencies (14,28) and appearance of four sharp absorption bands at (3653-3520) cm^{-1}atributed to the υ(free O-H). The spectrum also showed appearance of four bands at (3443-3220) cm^{-1}due to the υ (bonding O-H).The weak band at 3160 cm^{-1} attributed to the υ(N-H).The two absorption band at 3074 and 3000cm^{-1} due to the υ(C-H) aromatic. The bands at 2880cm^{-1} and 2760 cm^{-1} due to the υ(C-H) aliphatic of aldehyde group (-CHO). The weak band at 2630 cm^{-1} due to the υ (C-S).Furthermore, the absorbtion band at 1674cm^{-1} due to the υ (C=O) of aldehyde group, we notice that the value was shifted to the lower frequency due to the intramolecular hydrogen bonding with ***Ortho*** hydroxy group(29). The strong absorption band at 1545 cm^{-1} due to the υ (C=N) of the thiadiazole ring. The absorption band at 1480cm^{-1} due to the υ($C\overset{\cdots}{-}C$) of aromatic ring. The absorption bands at 968,883,833 and 759 cm^{-1} were due to the δ(C-H) aromatic out of plane . FT-IR spectrum also showed a strong band at 1240cm^{-1} was attributed to the υ(C=S) of thion tautomeric form.The spectrum also showed appearance of medium band at 1292 cm^{-1} due to the δ(O-H) in plane .Schiff bases [3] and [4] have been prepared through condensation reaction between azo aldehyde derivative [2] and each primary amines 2-Amine-1,3,4-thiadiazole -5- thiol and 5-(Benzylthio)-1,3,4-thiadiazole-2- amine respectively , in presence of small amount from glacial acetic acid as catalyst in absolute ethanol .

FT-IR spectrum of imine derivative [3] showed disappearance of the absorption band at 1674cm^{-1} attributed to the υ(C=O) of aldehyde group,also disappearance of the doublet absorption band at (3392 cm^{-1},3279 cm^{-1}) attributed to the asymmetric and symmetric stretching vibrations of (-NH_2) group ,respectively and appearance of strong absorption band at 1614cm^{-1} attributed to the stretching vibration of exocyclic imine group (C=N) .

FT-IR spectrum of imine derivative [4] showed disappearance of the absorption band at 1674cm^{-1} attributed to the υ(C=O) of aldehyde group ,also disappearance of the doublet absorption band at (3392 cm^{-1},3279 cm^{-1}) attributed to the asymmetric and symmetric stretching vibrations of (-NH_2) group ,respectively and appearance of strong absorption band at 1640 cm^{-1} attributed to the stretching vibration of exocyclic imine group (C=N) .

1,3-Oxazepine derivatives [5-10] have been prepared by using a pericyclic reaction type [2-5] cyclo addition reaction between imine group in compounds [3-4] as two membered component and maleic , phthalic and 3-Nitro phthalic anhydrides as five membered components to give seven - membered 1,3-oxazepine ring. [2-5] cycloaddition reaction is a concerted process proceeds via a single cyclic transition state and thus there is no intermediate in the process .Mechanism of 1,3-oxazepine ring formation has been shown in the following scheme:



Scheme(1):Mechanism of* a pericyclic reaction type (2+5) cycloaddition reaction *between imine group and maleic anhydride

FT-IR spectrum of 1,3-oxazepine derivative [5] showed disappearance of the strong band at 1614cm^{-1} attributed to the stretching vibration of exocyclic imine group (C=N) and appearance of two strong absorption bands at1737 cm^{-1} and1695cm^{-1} attributed to the υ(C=O) for lactone and lactam structures inside1,3-oxazepine ring ,respectively.

FT-IR spectrum of 1,3-oxazepine derivative [6] showed disappearance of the strong band at 1614cm^{-1} attributed to the stretching vibration of exocyclic imine group (C=N) and appearance of two strong absorption bands at1740 cm^{-1} and1685cm^{-1} attributed to the υ(C=O) for lactone and lactam structures inside1,3-oxazepine ring ,respectively.

FT-IR spectrum of 1,3-oxazepine derivative [7] showed disappearance of the strong band at 1614cm^{-1} attributed to the stretching vibration of exocyclic imine group (C=N) and appearance of two strong absorption bands at 1745 cm^{-1} and 1689cm^{-1} attributed to the υ(C=O) for lactone and lactam structures inside1,3-oxazepine ring ,respectively.

FT-IR spectrum of compound [7] also showed appearance of two strong absorption bands at 1560cm^{-1} and 1345cm^{-1} attributed to the asymmetric and symmetric stretching vibrations of (-NO_2)

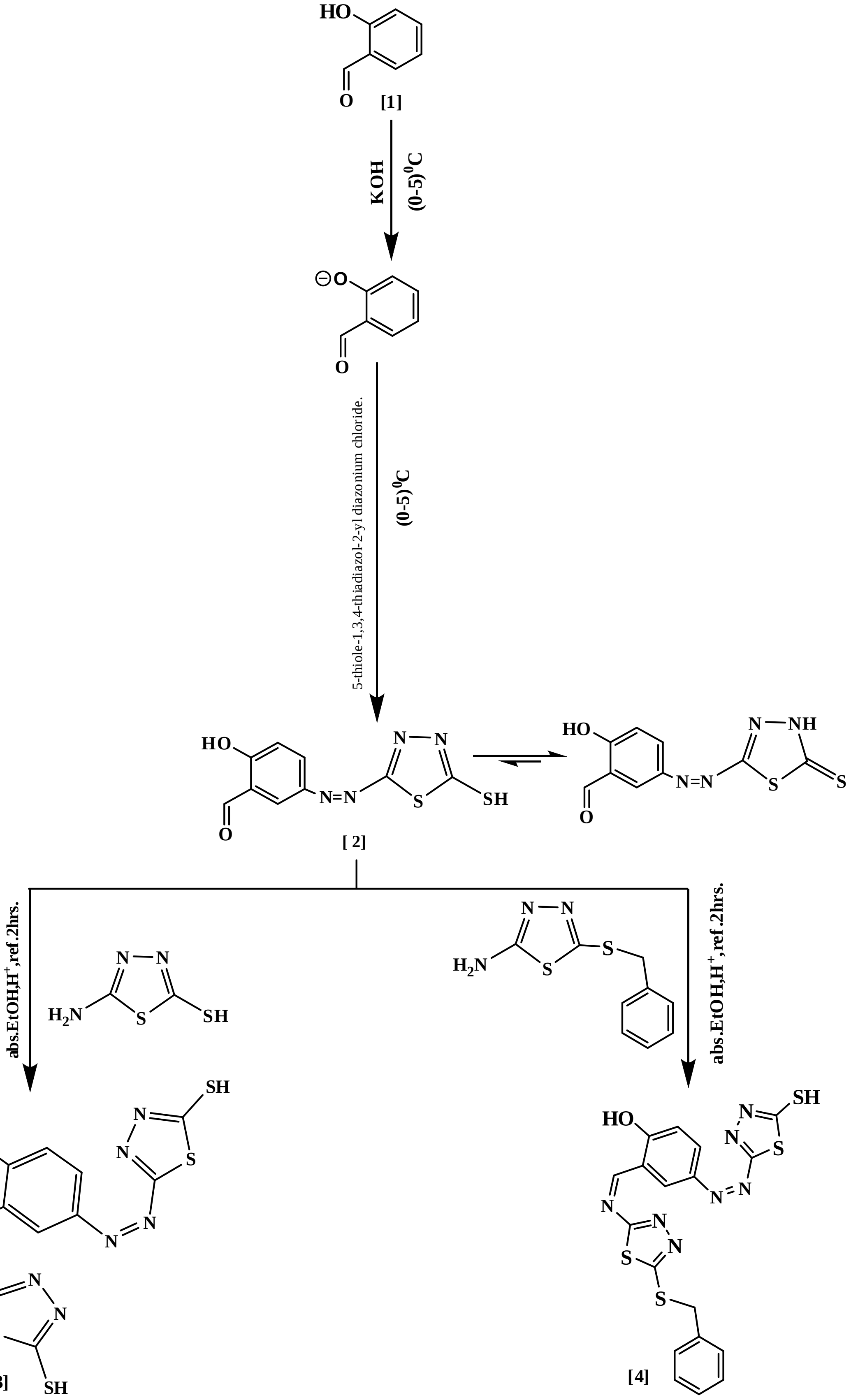
group(30). Also appearance of strong absorption band at 877cm^{-1} due to the stretching vibration of (C-NO_2) bond is good evidence for the structure given to the product(31).

FT-IR spectrum of 1,3-oxazepine derivative [8] showed disappearance of the strong band at 1640cm^{-1} attributed to the stretching vibration of exocyclic imine group (C=N) and appearance of two strong absorption bands at 1730 cm^{-1} and 1708cm^{-1} attributed to the υ(C=O) for lactone and lactam structures inside1,3-oxazepine ring ,respectively.

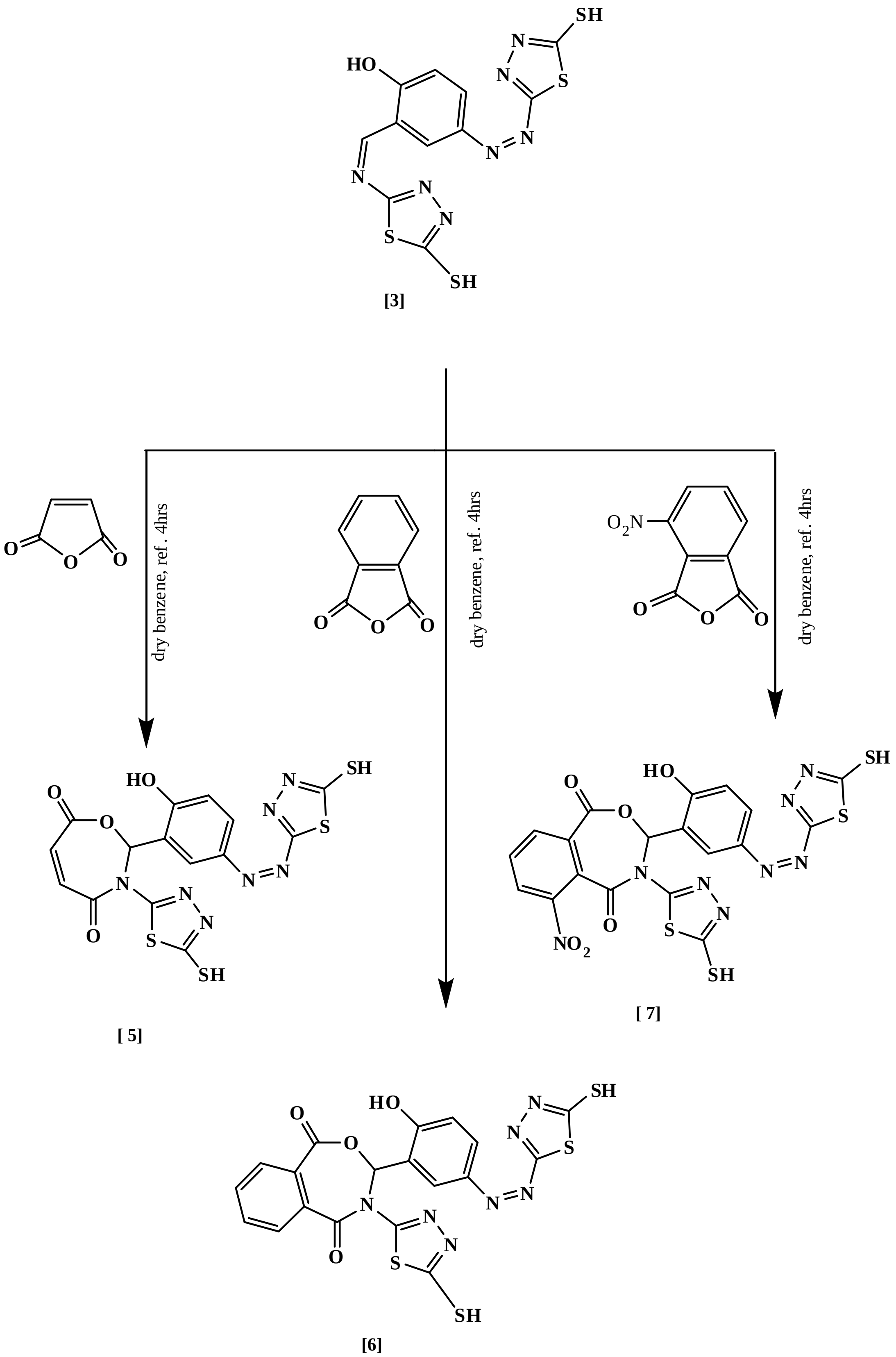
FT-IR spectrum of 1,3-oxazepine derivative [9] showed disappearance of the strong band at 1640 cm^{-1} attributed to the stretching vibration of exocyclic imine group (C=N) and appearance of two strong absorption bands at 1720 cm^{-1} and 1670 cm^{-1} attributed to the υ(C=O) for lactone and lactam structures inside1,3-oxazepine ring, respectively.

FT-IR spectrum of 1,3-oxazepine derivative [10] showed disappearance of the strong band at 1640cm^{-1} attributed to the stretching vibration of exocyclic imine group (C=N) and appearance of two strong absorption bands at 1780 cm^{-1} and 1701cm^{-1} attributed to the υ(C=O) for lactone and lactam structures inside1,3-oxazepine ring ,respectively.

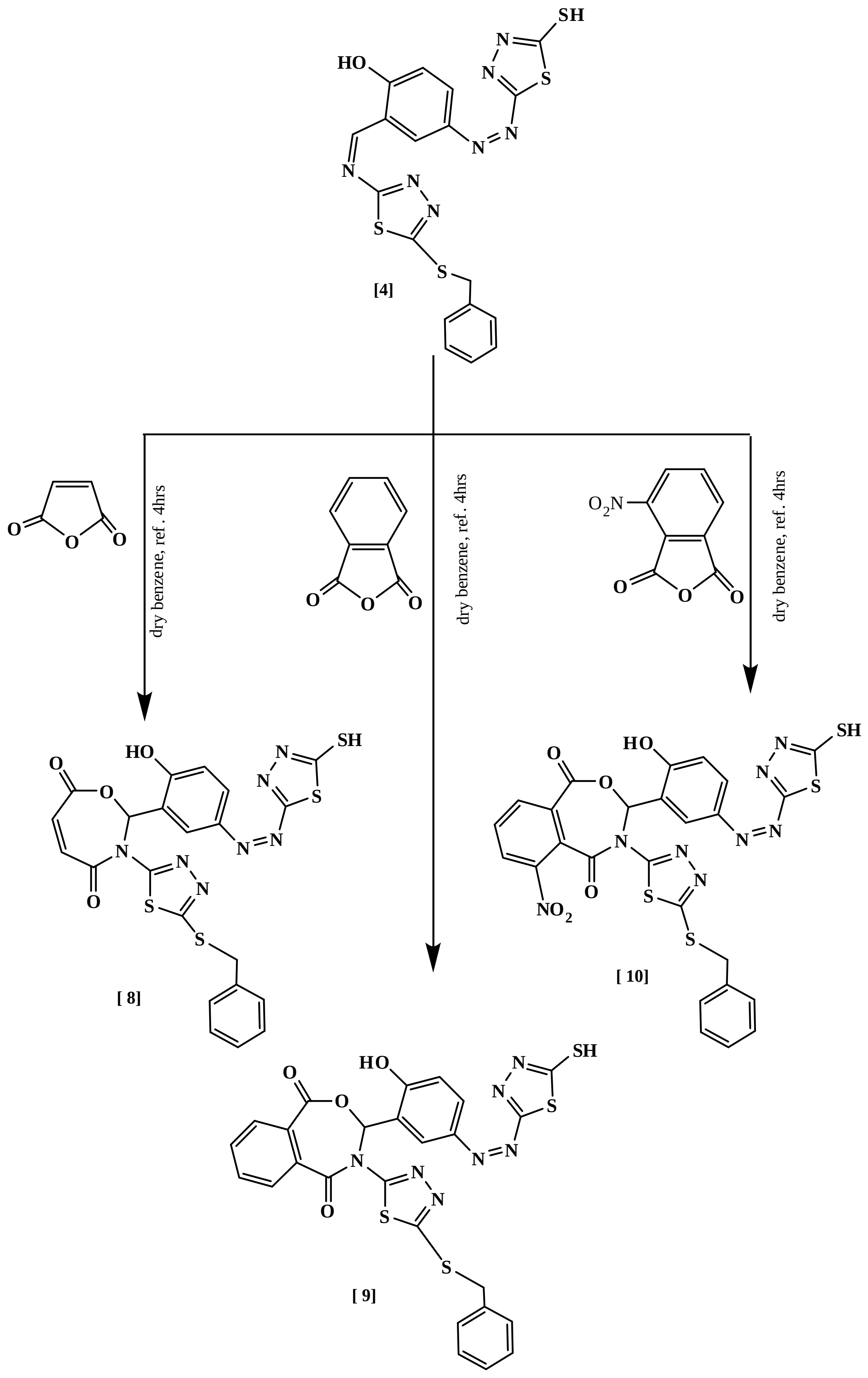
FT-IR spectrum of compound [10] also showed appearance of two strong absorption bands at 1560cm^{-1} and 1335cm^{-1} attributed to the asymmetric and symmetric stretching vibrations of (-NO_2) group(29). Also appearance of strong absorption band at 866cm^{-1} due to the stretching vibration of (C-NO_2) bond is good evidence for the structure given to the product.



Scheme (2): Reactions proceeding



Scheme (3): Reactions proceeding

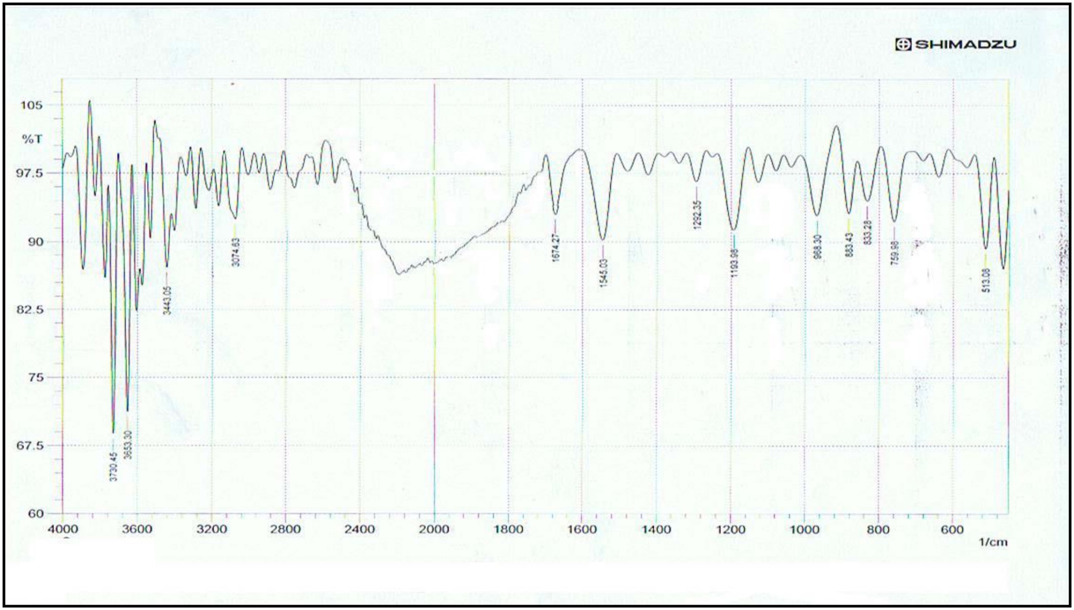


Scheme (4): Reactions proceeding

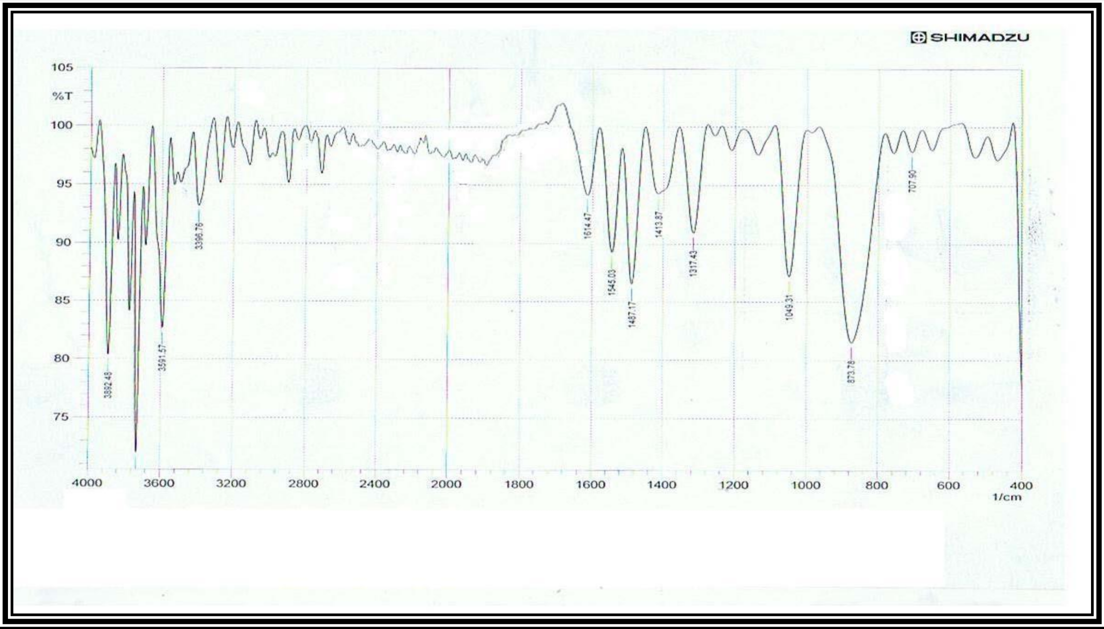
Table (2): FT-IR Data of the prepared Compounds [2-10] in cm^{-1}

Comp. no.	υ(-OH)	υ(-NH$_2$)	υ(N-H)	υ(C-H) arom.	υ (C-H) aliph.	υ(S-H)	υ(C=O)	υ(C=N)	υ $(C \overset{\text{----}}{\text{—}} C)$ arom.	δ (O-H) in plane	υ(N=N)	υ(C-O)	υ(C=S)	υ(C-H) arom. o.o.p.	υ(C-NO$_2$)
[2]	3520-3653(sp) free(OH) 3220-3443(sp)bonding (OH)	-	3160(w)	3000(w) 3074(m)	2760-2940 (w)	2550(w)	1674(s)	1545(s) endo	1480(m)	1292(m)	1425(w)	1070(m) 1035(w) phenol	1193 (s)	968(s) 883(s)	-
[3]	3591(sp) free(OH) 3260-3560(sp)bonding (OH)	-	3200(w)	3000(w) 3100(w) 3140(m)	2680-2980 (w)	2500(w)	-	1614(s) exo 1545(s) endo	1487(s)	1317(s)	1413(w)	1250(w) 1130(w)	1205 (m)	833(s) 759(s)	-
[4]	3520-3600(sp) free(OH) 3260-3400(sp)bonding (OH)	-	3200(w)	3072(w) 3132(w)	2700-2980 (w)	2530(w)	-	1640(s) exo 1543(s) endo	1600(w) 1498(s)	1321(s)	1423(w)	1240(w) 1136(m)	1209 (s)	889(s) 819(s) 758(s)	-
[5]	3498-3600(sp) free(OH) 3240-3420(sp)bonding (OH)	-	3195(w)	2993(w) 3060 (w) 3120(w)	2720-2940 (w)	2560(w)	1737(s) lactone 1995(s) lactam	- 1546(s) endo	1485(s)	1315(s)	1430(w)	1220 1070 Phenol 1060 lactone	1160 (m)	981(m) 945(w) 877(s)	-
[6]	3529-3640(sp) free(OH) 3260-3500(sp)bonding (OH)	-	3240(w)	3000(w) 3150(w)	2720-2940 (w)	2560(w)	1740(s) lactone 1685(s) lactam	- 1540(s) endo	1485(s)	1315(s)	1423(w)	1139(br) Phenol+lactone	1200 (w)	870(s) 829(m) 775(w)	
[7]	3600(sp) free(OH) 3200-3520(sp)bonding (OH)	-	3160(w)	3000(w) 3040(w) 3100(w)	2665-2960 (w)	2561(w)	1745(s) lactone 1689(s) lactam	- 1535(s) endo	1491(s)	1315(s)	1421(w)	1140(br) Phenol+lactone	1220 (w)	830(m) 790(w)	877(s)
[8]	3533-3650(sp) free(OH) 3200-3443(sp)bonding (OH)	-	3140(w)	3000(w) 3070(w) 3100(w)	2725-2930 (w)	2560(w)	1730(s) lacton 1708(s) lactam	- 1535(s) endo	1489(s)	1311(s)	1430(w)	1210(m) 1100(m) Phenol 1070(m) lactone	1175 (m)	856 (s) 810(s) 760(m)	
[9]	3525-3610(sp) free(OH) 3200-3427(sp)bonding (OH)	-	3160(w)	3000(w) 3040(w) 3080(w) 3120(w)	2677-2960 (w)	2677(w)	1720(s) lacton 1670(s) lactam	- 1533(m) endo	1490(s)	1309(s)	1420(w)	1226(m) 1141(m) Phenol 1090(m) lactone	1180 (s)	966(s) 915(s) 761(m)	
[10]	3500-3610(sp) free(OH) 3200-3477(sp)bonding (OH)	-	3190(w)	3000(w) 3050(w) 3100(w)	2840-2900 (w)	2545(w)	1780(s) lactone 1701(s) lactam	- 1545(s) endo	1490(s)	1313(s)	1425(w)	1240(m) 1163(m) Phenol 1080 lactone	1210 (w)	840(w) 810(w) 785(w) 760(w)	866(s)

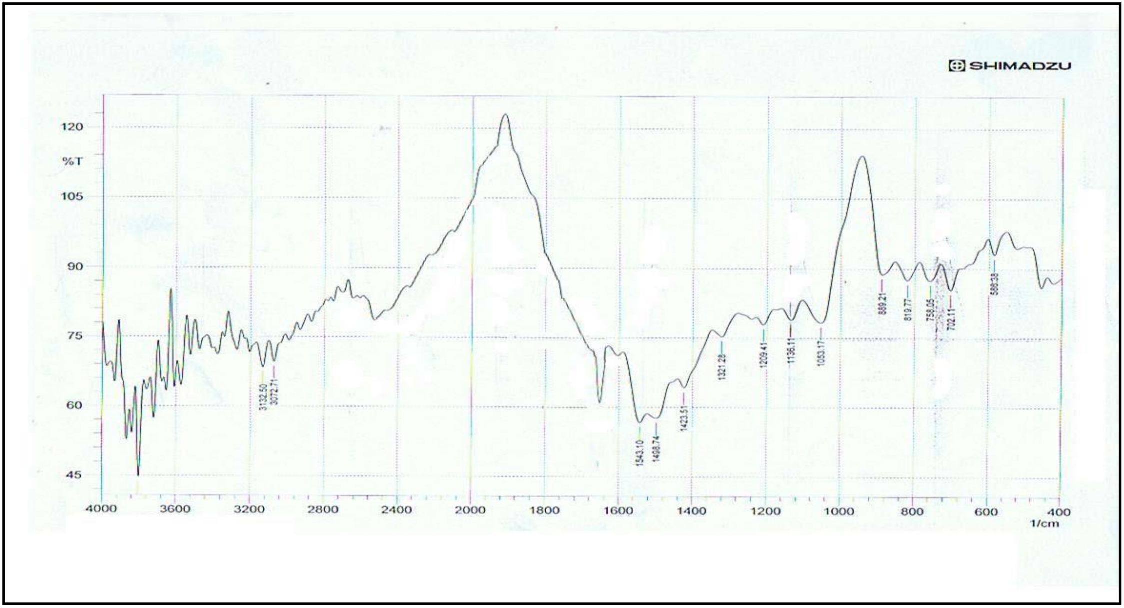
s=strong, w=weak, m=medium, sp=sharp, br=broad, o.o.p.= out of plane



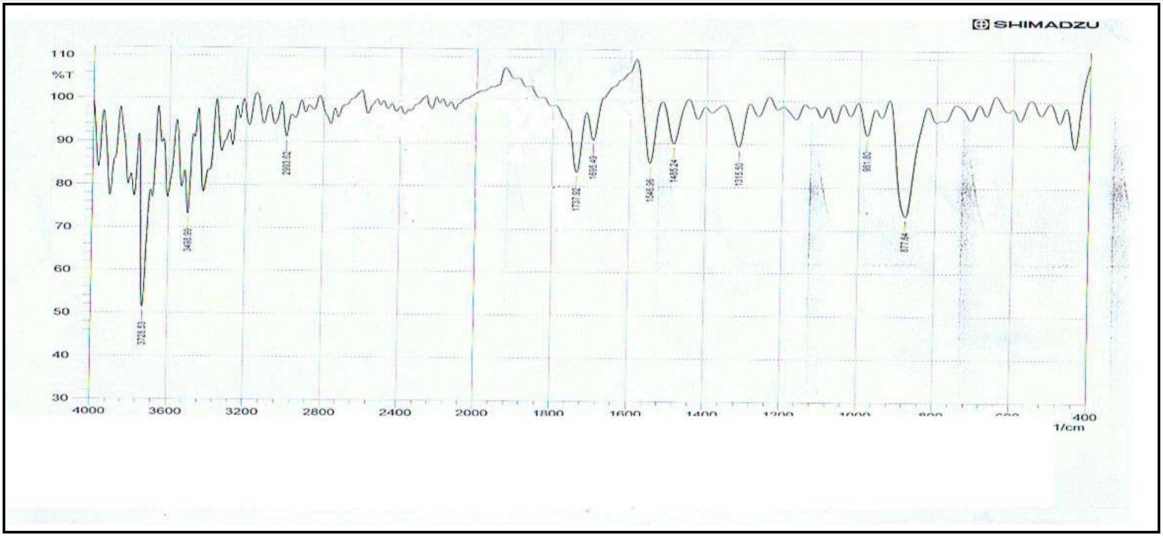
FT-IR spectrum of compound [2]



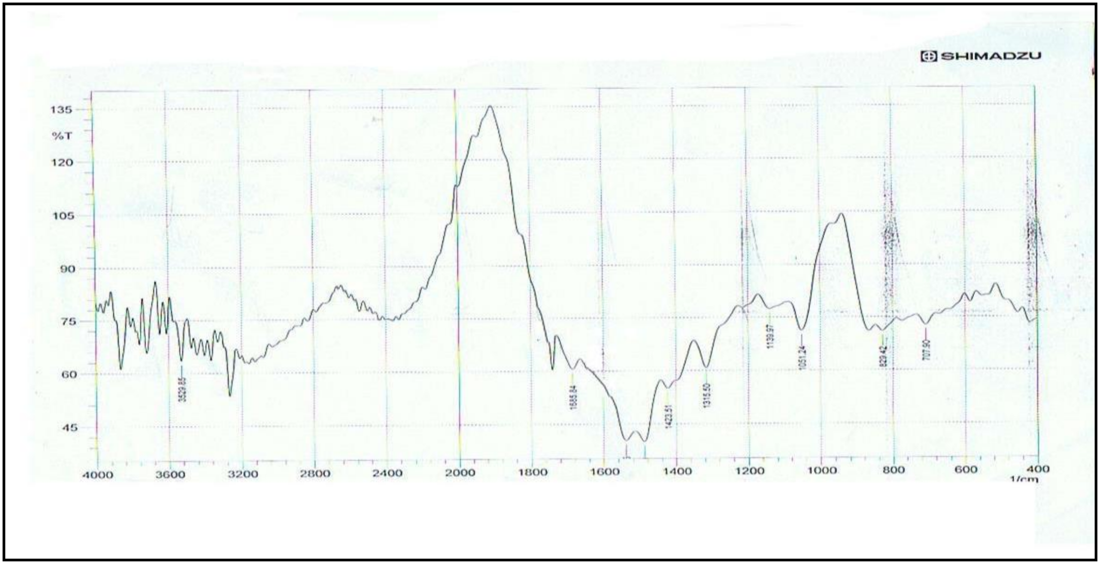
FT-IR spectrum of compound [3]



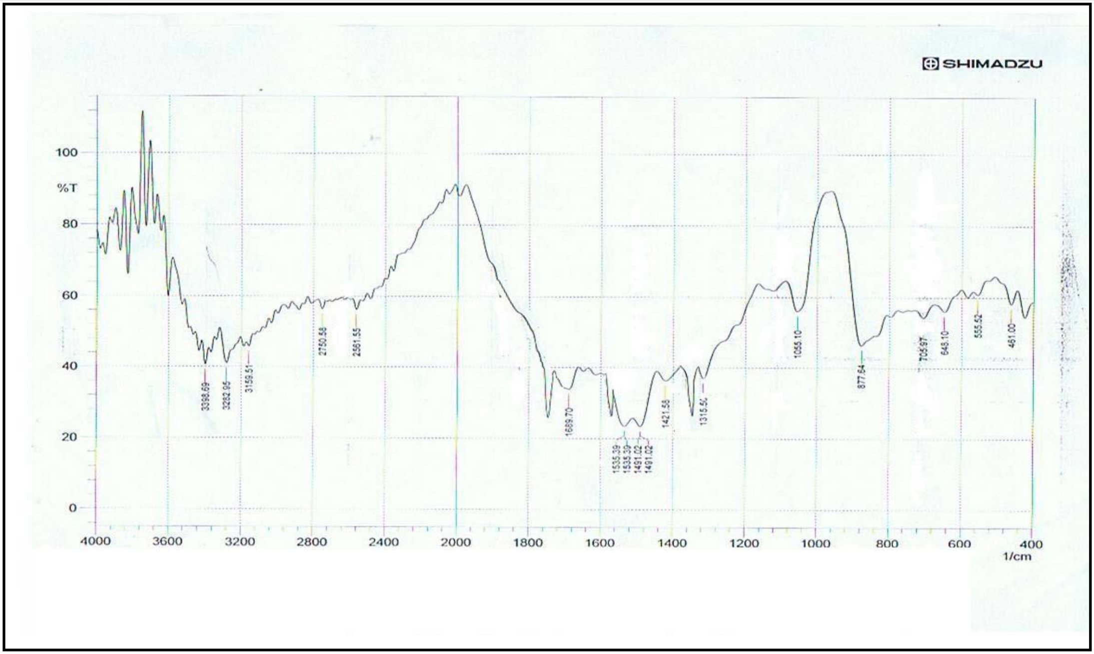
FT-IR spectrum of compound [4]



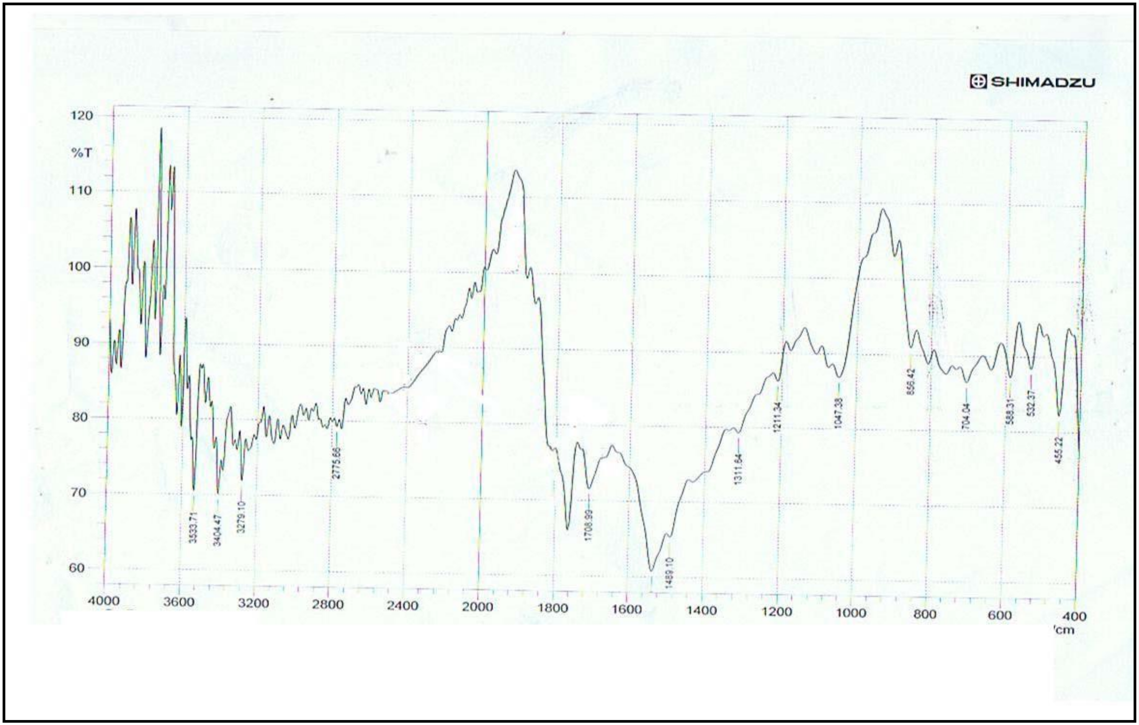
FT-IR spectrum of compound [5]



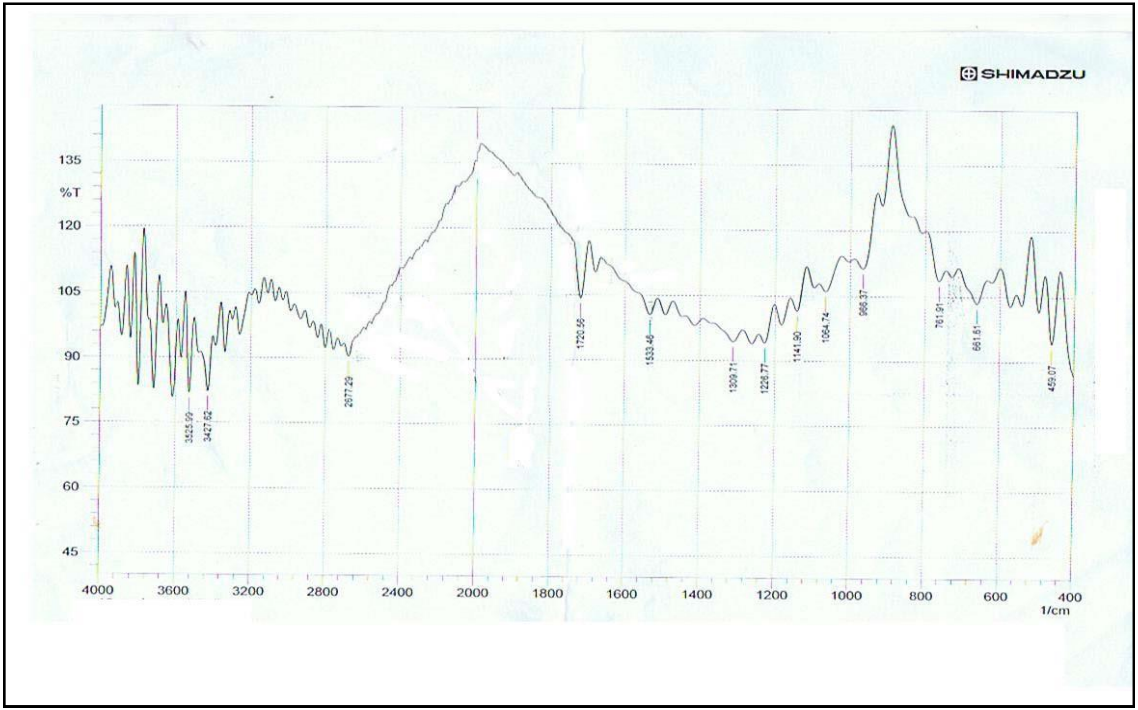
FT-IR spectrum of compound [6]



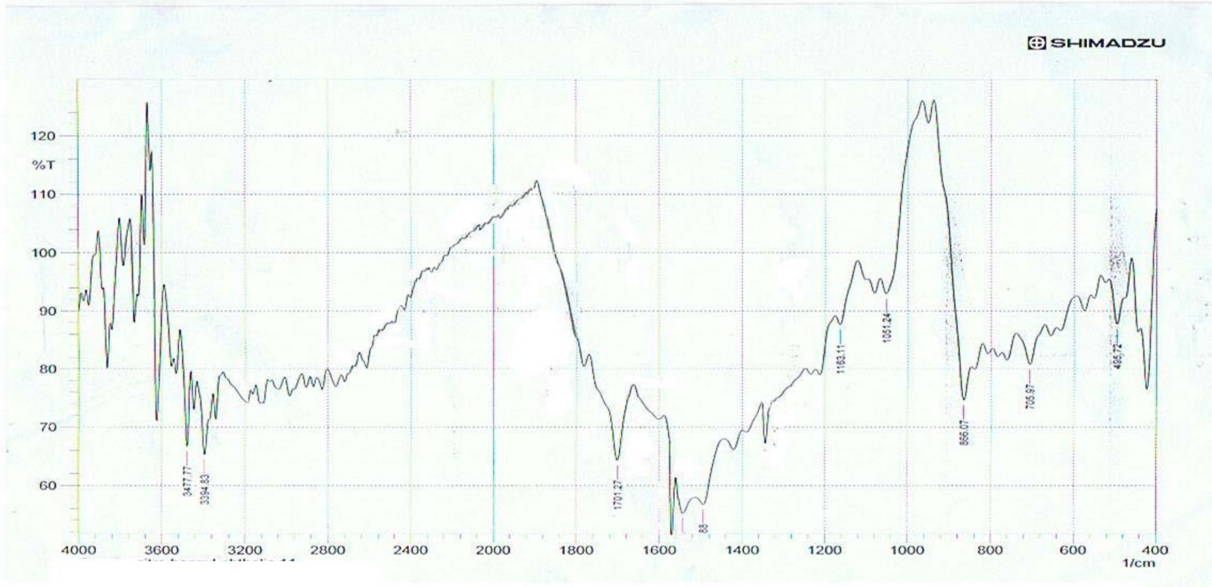
FT-IR spectrum of compound [7]



FT-IR spectrum of compound [8]



FT-IR spectrum of compound [9]



FT-IR spectrum of compound [10]

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