

Synthesis & Characterization of Oxazinan and 5-oxa-7-aza-spiro[2,5] octane from reaction of Dibenzylidene with malonic anhydride and 5-oxa-spiro[2,3] hexane-4,6-dione.

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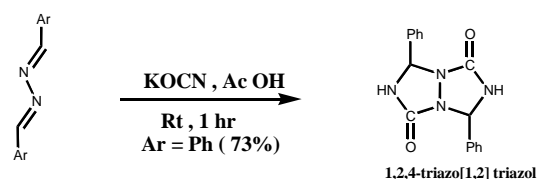
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ABSTRACT:

Di Benzylidenes were prepared by condensation of 1,2-diamino benzene with *o*-hydroxy benzaldehyde. These dibenzylidenes when treated with one equivalent of malonic anhydride or 5-oxo-spiro[2,3]hexane-4,6-dione in dry benzene give 6-membered heterocyclic ring system of 3-{2-[(2-Hydroxy-benzylidene)-amino]-phenyl}-2-(2-hydroxy -phenyl)-[1,3]oxazinane-4,6-diones (**1-3**) or 7-{2-[(2-hydroxy-benzylidene)-amino]-phenyl}-6-(2-hydroxy-phenyl)-5-oxa-7-aza-spiro[2.5]octane-4,8-diones (**7- 9**) But when two equivalents of malonic anhydride or 5-oxo-spiro[2,3]hexane-4,6-dione were used and under sam conditions compounds (**4-6** , **10-12**) were obtained .

INTRODUCTION:

The six-membered heterocyclic ring system: 1, 3-oxazine has already been reported and thoroughly reviewed in the literature ^[1-4]. The reaction of dibenzylidene with some olefins and acetylenes proceeds via a so-called "Crisscross" ^[5] pathway. In 1917 Bailey et al ^[6,7] reported a new reaction of aromatic dibenzylidene with two equivalents of cyanic acid, thiocyanic acid ^[8] and isocyanate, leading to unexpected products.



Further this "Crisscross" addition is generally used for the reaction of azines of aromatic aldehydes with various electron-deficient olefins when the double bond is terminal (e.g. alkyl acrylate, acrylonitrile) ^[9,10] or in which allylic substituents do not sterically hinder the reaction (e.g. maleic anhydride^[11] and sulfolen ^[12] 1,4-benzodiazpines have been the object of

intense studies since the early 1960s because of their values in psychotherapy . An impressive number of synthetic routes have thus been described ^[13] . Recently the attention has been concentrated on the synthesis of analogs having heterocyclic in place of the benzene ring and on compounds having additional fused heterocyclic rings.

This reaction of maleic anhydride with aromatic aldazines is related to the same reaction carried out in our laboratory. So under relatively severe conditions (150C, 20hr,xylene) , the reaction leads to fused bicyclic products via abis (3+2) cycloaddition, while under milder conditions (80C,2hr,benzene) the reaction leads to a 7-membered heterocyclic ring system via a (5+2)→7 cycloaddition^[14] Imines and N-acyl imines react with diketene to give tetrahydro-1,3-oxazine-4-ones ^[15-16]

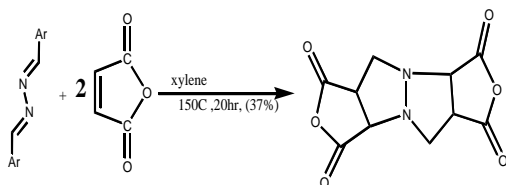
N-acyl imines undergo [4+2] cycloaddition with both C=C- and hetrodienes. For instance, isolable bis(trifluoromethyl)acyl imine, reacts with 2,2-dimethylethylene to give 1,3-oxazine.

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N-acyl immonium ions have been the most commonly used dienes to effect [4+2] cycloaddition as 4π components with substituted 1,3-butadienes. It is found that N-acylimines or immonium ions that are capable of tautomerization undergo intramolecular Diels-alder reaction to give dihydro-1, 3-oxazines [17].

The reaction of N-benzylidene 1, 5-dimethyl-2-phenylpyrazolonamines (Schiff bases with Cyclopentane -1,1-dicarboxylic anhydride to give 2-aryl-3-3(1,5-dimethyl-2-phenylpyrazolo)-1-(5) spirocyclopentyltetra hydro-1,3-oxazine-4,6-diones [18]



EXPERIMENTAL:-

Melting points were recorded on Gallenkamp melting point apparatus and were uncorrected. Their H-NMR spectra were recorded with BRUKER – AC – 200MHZ FT-NMR and elemental analysis was carried out in Mutah University on perkin-Elmre 2400 CHN Elemental analyzer. IR spectra were recorded on PYE UNICUM Sp-300 spectrophotometer in KBr, and Uv-visible spectra were recorded (in ethanol) on Schimasu Reco-160 spectrophotometer .

2-({4-[(2-hydroxy-benzylidene)-amino]-phenylimino}-methyl)-phenol:-

General method:-

To a solution of appropriate diamino benzene (3.8 g, 0.05 mol) in absolute ethanol (30 ml) was added 2-hydroxy benzaldehyde (0.05 or 0.1 mol) and the mixture was refluxed for 2 hours . After cooling, the mixture was filtered and the solid recrystallized from ethanol to give the title compound (A, B, C)

3-{2-[(2-Hydroxy-Benzylidene)-amino]-phenyl}-2-(2-hydroxyl-phenyl)- [1,3]oxazinane-4,6-dione:-

General method:-

A mixture of appropriate 2-({4-[(2-hydroxy-benzylidene)-amino]-phenylimino}-methyl)-phenol (0.01 mol) and oxetane – 2,4-dione (0.86 g , 0.01 mol) in dry benzene (20 ml) was heated for 3 hours of $80\text{ }^{\circ}\text{C}$, then the solvent evaporated and the product was collected and crystallized from THF (**1-3**) (table 1) .

This experiment was repeated using the same amounts of the reactants to obtain other derivatives.

7-(4-Dimethylamino-phenyl)-6-{4-dimethylamino-phenyl}-4,8-dioxo-5-oxa-7-aza- Spiro[2.5]oct-7-yl]-phenyl}-5-oxa-6-aza-spiro[2.5]octane-4,8-dione:-

General method:-

A mixture of appropriate 2-({4-[(2-hydroxy-benzylidene)-amino]-phenylimino}-methyl)-phenol (0.01 mol) and 5-oxa-spiro[2.3] hexane – 4,6-dione (1.12 g , 0.01 mol) in dry benzene (20 ml) was heated for 3 hours of $80\text{ }^{\circ}\text{C}$, then the solvent removed and the yellow crystalline solid was collected and crystallized from dioxin (**7-9**) (table 1) .

This experiment was repeated using the same amounts of the reactants to obtain other derivatives.

Results and Discussion:-

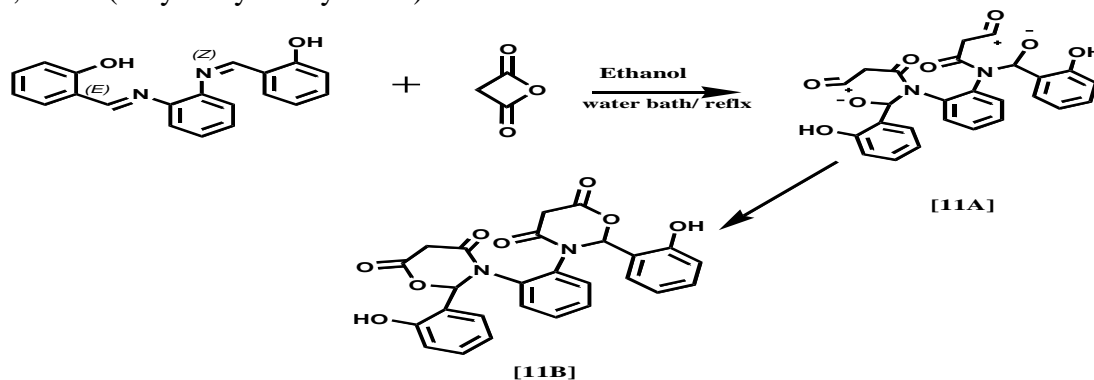
It is known that Schiff bases react smoothly with acid chlorides and anhydrides to give the corresponding addition products.^[15-17]

Appropriate Schiff bases (A, B, C) were prepared by condensation of diamino benzene with 2- hydroxy benzaldehyde. The reaction was followed by appearance of stretching bands for (C=N) at $1600 - 1610\text{ cm}^{-1}$ combined with the disappearance of

bands at $1670\text{-}1680\text{ cm}^{-1}$ and $3400\text{-}3650\text{ cm}^{-1}$ for OH and NH_2 moieties respectively in their IR spectra. In the present work the reactions of Schiff bases (A,B,C) with oxetane – 2,4-dione or 5-oxa-spiro[2.3] hexane – 4,6-dione were carried out as shown in (Scheme 2A and 2B).

However, treatment of Schiff bases with oxetane – 2,4-dione with (N,N-Bis-(2-hydroxy-benzylidene)-

benzene-1,2-diamine. to gives the dipolar intermediate [11A] which collapses to the 6- membered heterocyclic ring system.[11B] is presented. This reaction was followed by disappearance of absorption bands at ($1600\text{-}1610\text{ cm}^{-1}$) for C=N stretch and appearance of new absorption bands at ($1360\text{-}1380\text{ cm}^{-1}$) for C-N and ($1680\text{ -}1700\text{ cm}^{-1}$) for carbonyl group of lacton and lactam.



Scheme (1)

It is impressive to note that the two absorption band at ($1800\text{-}1950\text{ cm}^{-1}$) in the IR spectra of pure malonic anhydride and 2-Oxa-spiro[3.4] octane-1,3-dione (anhydride) have disappeared when the anhydride became part of the 6-membered ring system of the 2-(2-hydroxy-phenyl)-3-{2-[2-(2-hydroxy-phenyl)-4,6-dioxo-[1,3]oxazinan-3-yl]-ethyl}-[1,3]oxazinan-4,6-dione and 6-(4-Dimethylamino-phenyl)-7-{2-[2-(4-Dimethylamino-phenyl)-4,6-dioxo-[1,3]oxazinan-3-yl]-ethyl}-5-oxa-7-aza-spiro[2.5]octane-4,8-dione.

The new absorption bands of the (C=O) group in the IR spectra of the addition products [11B] appear at ($1670\text{-}1700\text{ cm}^{-1}$), this attributed to the fact that the structures of the addition products are combination of the lacton-lactam structure.

The UV spectra of 2-(2-hydroxy-phenyl)-3-{2-[2-(2-hydroxy-phenyl)-4,6-dioxo-[1,3]oxazinan-3-yl]-ethyl}-[1,3]oxazinan-4,6-dione and 6-(4-

Dimethylamino-phenyl)-7-{2-[2-(4-Dimethylamino-phenyl)-4,6-dioxo-[1,3]oxazinan-3-yl]-ethyl}-5-oxa-7-aza-spiro[2.5]octane-4,8-dione show absorption maxima at ($240\text{-}310\text{ nm}$), and at ($310\text{-}445\text{ nm}$) due to charge transfer of the aryl group and the cyclic 6-membered structure [11B]. 2-(2-hydroxy-phenyl)-3-{2-[2-(2-hydroxy-phenyl)-4,6-dioxo-[1,3]oxazinan-3-yl]-ethyl}-[1,3]oxazinan-4,6-dione and 6-(4-Dimethylamino-phenyl)-7-{2-[2-(4-Dimethylamino-phenyl)-4,6-dioxo-[1,3]oxazinan-3-yl]-ethyl}-5-oxa-7-aza-spiro[2.5]octane-4,8-dione are identified by their m.p.s, elemental analysis (table 1), IR spectra (table 2) and UV spectra (table 3). It is noticeable that the values of C-Hstr. (benzylic) absorption bands are rather high. This is in fact explained by the shift toward longer wavelength, that takes place when the benzylic carbon is linked to three electron-withdrawing groups, phenyl, O and N in the title compounds.

The reaction of 2-Oxa-spiro [3.4] octane-1, 3-dione with various Schiff bases is a sort of cycloaddition reaction. Cycloaddition is a ring formation that results from the addition of bonds to either δ or π with formation of new δ bonds. This class of reactions and its reverse encompasses a large number of individual types. Huisgen^[18] has formulated a useful classification of diverse cycloaddition in terms the

number of the new δ bond. The ring size of the product, and the number of atoms in the components taking part in the cycloaddition. This cycloaddition reaction is classified as a 2 + 5-7, and it is the first cycloaddition of this type, although in principle, one would predict that the butadienyl cation might add to an olefin through a $(4n+2)$ transition state to yield the cyclohexenyl cation^[19].

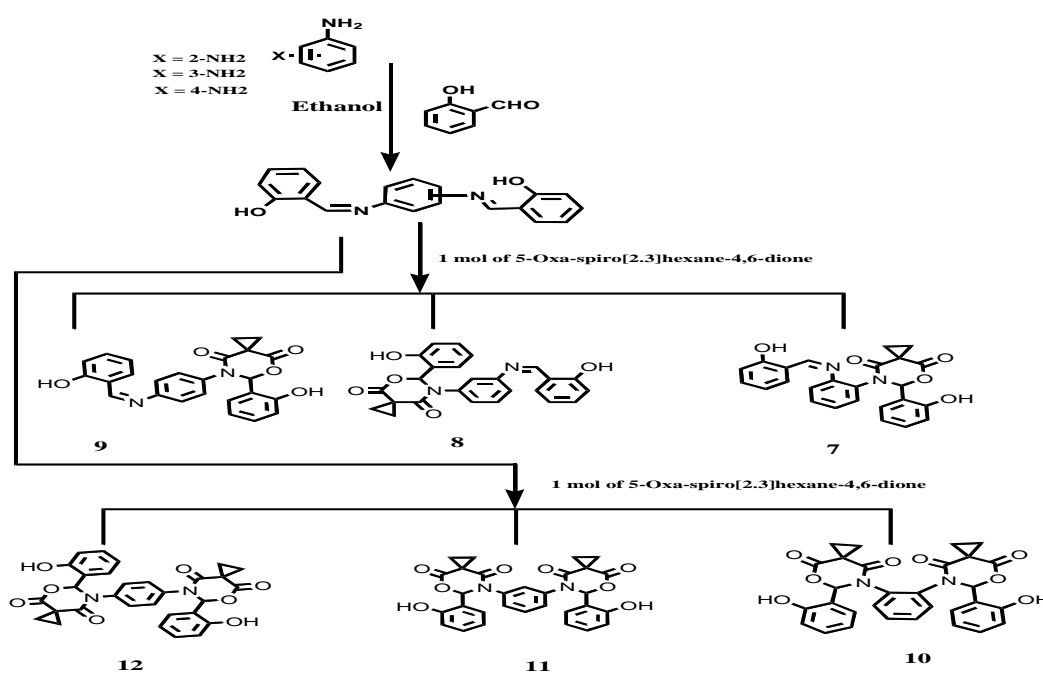
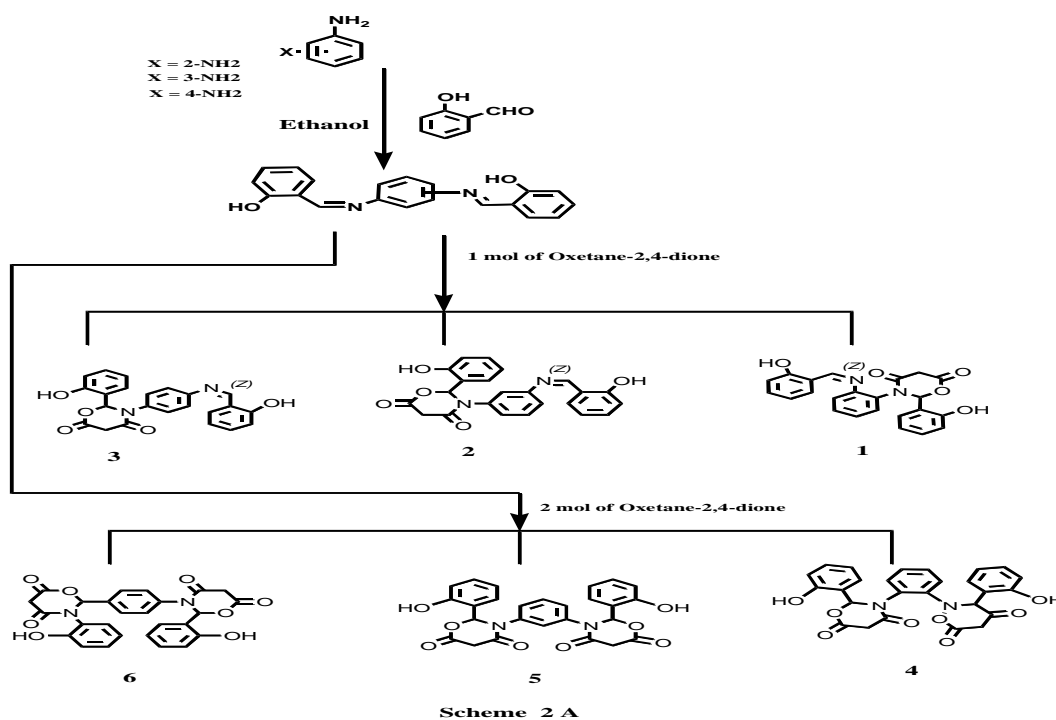


Table (1): Melting points, Yield, M.F, Elemental analyses of 2-({X-[(2-Hydroxy-benzylidene)-amino]-phenylimino}-methyl)-phenol and 3-{X-[(2-Hydroxy-benzylidene)-amino]-phenyl}-2-(2-hydroxy-phenyl)-[1, 3] oxazinane-4, 6-dione.

X= 2, 3, 4

No.	M.P/C ^o	Yield%	M.F	Calc.			Found		
				C%	H%	N%	C%	H%	N%
1	162-164	67	C ₂₃ H ₁₈ N ₂ O ₅	68.65	4.51	6.96	68.78	4.59	6.82
2	155-157	78	C ₂₃ H ₁₈ N ₂ O ₅	68.65	4.51	6.96	68.61	4.63	6.79
3	160-162	70	C ₂₃ H ₁₈ N ₂ O ₅	68.65	4.51	6.96	68.70	4.55	6.80
4	177-179	81	C ₂₆ H ₂₀ N ₂ O ₈	63.93	4.13	5.74	64.05	4.26	5.66
5	210-212	80	C ₂₆ H ₂₀ N ₂ O ₈	63.93	4.13	5.74	64.09	4.20	5.59
6	169-171	77	C ₂₆ H ₂₀ N ₂ O ₈	63.93	4.13	5.74	64.00	4.22	5.63
7	137-139	75	C ₂₅ H ₂₀ N ₂ O ₅	70.08	4.71	6.54	70.20	4.79	6.46
8	160-162	69	C ₂₅ H ₂₀ N ₂ O ₅	70.08	4.71	6.54	70.16	4.82	6.42
9	183-185	78	C ₂₅ H ₂₀ N ₂ O ₅	70.08	4.71	6.54	70.21	4.75	6.47
10	189-191	75	C ₂₆ H ₂₀ N ₂ O ₈	63.93	4.13	5.74	63.99	4.21	5.63
11	178-180	80	C ₂₆ H ₂₀ N ₂ O ₈	63.93	4.13	5.74	64.00	4.15	5.60
12	155-157	88	C ₂₆ H ₂₀ N ₂ O ₈	63.93	4.13	5.74	64.02	4.12	5.57
13	148-150	66	C ₂₅ H ₂₀ N ₂ O ₅	66.66	4.48	5.18	66.73	4.50	5.00
14	172-174	63	C ₂₅ H ₂₀ N ₂ O ₅	66.66	4.48	5.18	66.80	4.44	4.98
15	167-169	72	C ₂₅ H ₂₀ N ₂ O ₅	66.66	4.48	5.18	66.60	4.53	5.12

Table (2) : The major IR absorption (cm⁻¹) of 2-({X-[(2-Hydroxy-benzylidene)-amino]-phenylimino}-methyl)-phenol and 3-{X-[(2-Hbenamino]phenyl)-2-(2-hydroxy-phenyl)-[1,3]oxazinane-4,6-dione.

X= 2,3,4

No.	O-H str. phenol	C-H str. Benzylic	C=O str. Lactam-Lacton	C=N str. Imine	C=C str. Aromatic	C-O str. Lacton	C-H bend Aromatic
1	3440	3400	1680	1600	1580	1335	785
2	3400	3400	1690	1600	1570	1330	780
3	3430	3380	1680	1610	1580	1340	760
4	3445	3370	1690	1600	1590	1330	780
5	3435	3380	1680	1600	1590	1330	775
6	3400	3400	1690	1605	1580	1310	760
7	3400	3390	1690	1600	1575	1320	780
8	3430	3380	1700	1610	1575	1320	780
9	3430	3210	1710	1590	1570	1310	800
10	3450	3370	1685	1600	1590	1340	785
11	3440	3400	1690	1610	1580	1310	775
12	3420	3380	1680	1605	1570	1320	760
13	3450	3390	1680	1600	1575	1340	780
14	3445	3390	1685	1610	1580	1330	785
15	3435	3370	1690	1600	1570	1330	760

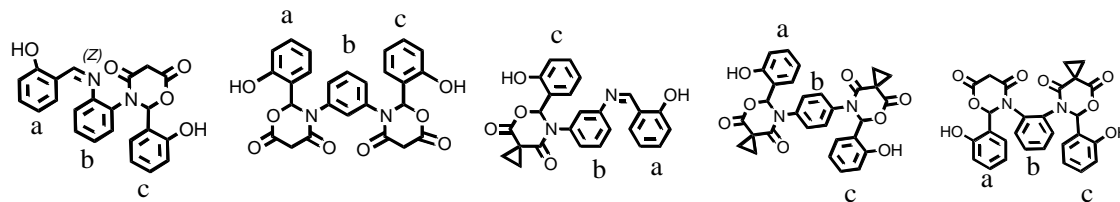
Table (3) : The UV-Visible absorption maxima (in ethanol) of 2-({X-[(2-Hydroxy-benzylidene)-amino]-phenylimino}-methyl)-phenol and 3-{X-[(2-Hydroxy-benzylidene)-amino]-phenyl}-2-(2-hydroxy-phenyl)-[1,3]oxazinane-4,6-dione.

X= 2, 3, 4

No	UV-Visible absorption maxima. λ (nm)
1	358, 310, 276, 226
2	386, 310, 266, 221
3	389, 320, 276, 225
4	377, 342, 310, 266, 226
5	385, 330, 312, 266, 229
6	360, 315, 289, 245, 224
7	358, 330, 308, 290, 230, 223
8	357, 320, 300, 267, 236, 225
9	360, 340, 312, 296, 247, 221
10	354, 300, 278, 227
11	353, 300, 280, 225
12	352, 305, 269, 230
13	380, 320, 300, 258, 228
14	379, 310, 260, 229
15	385, 311, 270, 245, 220

No.	Schiff baseName	Structure
A	2-({2-[(2-hydroxy-benzylidene)-amino]-phenylimino}-methyl)-phenol	<p>Vol.4(2)2007</p>
B	2-({3-[(2-hydroxy-benzylidene)-amino]-phenylimino}-methyl)-phenol	
C	2-({4-[(2-hydroxy-benzylidene)-amino]-phenylimino}-methyl)-phenol	

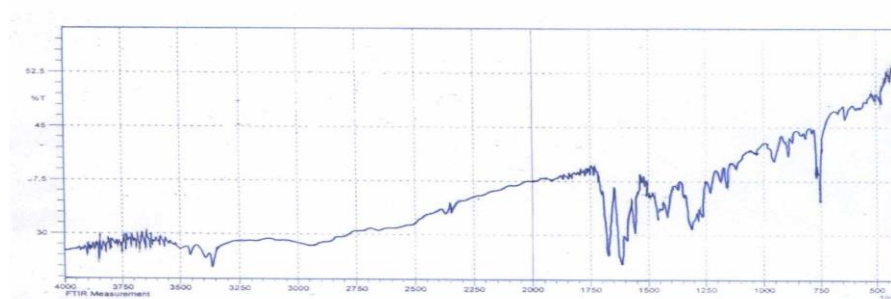
Table (4): ¹H.N.M.R Spectrophotometer of 2-({X-[(2-Hydroxy- benzylidene)-amino]-phenylimino}-methyl)-phenol and 3-{X-[(2-Hydroxy- benzylidene)-amino]-phenyl}-2-(2-hydroxy-phenyl)-[1, 3] oxazinane-4,6dione.



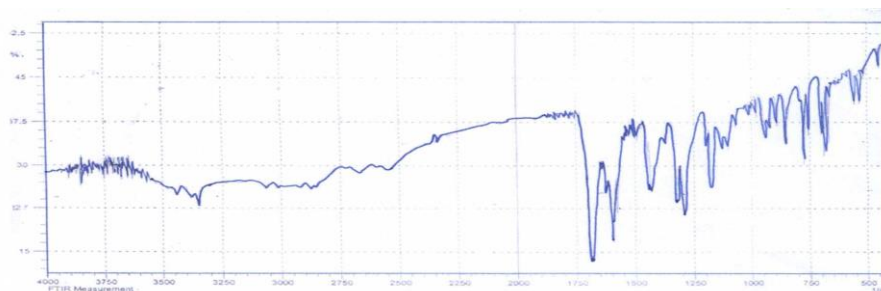
* Chemical shift= δ

** By using DMSO-d₆ as solvent

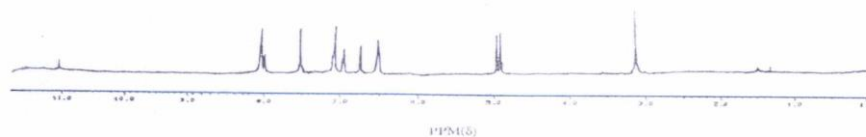
Comp.	OH	Ring A	Ring B	Ring C	H ₁	H ₂	H ₃	H ₄	H ₅	H ₆
1	4.9, 4.8	6.8, 7.0, 6.9, 6.9	7.4, 7.1, 7.4, 7.1	7.0, 6.7, 7.0, 6.68	8.26	3.1	-	7.22	-	-
5	4.8, 4.8	6.7, 7.0, 6.65, 7.1	7.3, 7.0, 7.2, 7.0	7.2, 6.7, 7.1, 6.64	7.28	3.1	-	8.1	3.09	-
8	4.6, 4.7	6.75, 7.1, 7.0, 7.05	7.1, 7.2, 7.2, 7.3	7.1, 6.6, 7.1, 6.9	8.11	-	-	7.20	0.98	0.95
12	4.8, 4.7	6.60, 7.0, 6.80, 7.0	7.1, 7.2, 7.15, 7.23	6.60, 7.0, 6.80, 7.0	7.30	8.9	8.9	8.9	8.9	7.30
13	4.9, 4.9	6.8, 6.7, 6.9, 6.5	7.0, 6.6, 7.1, 6.9	6.6, 6.8, 6.7, 6.9	7.3	3.12	-	8.85	8.9	7.3



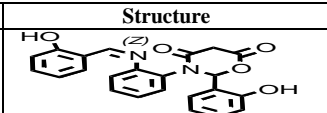
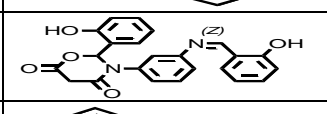
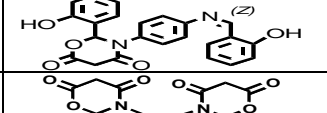
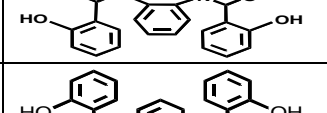
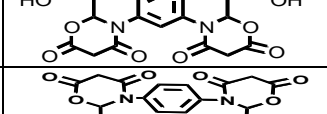
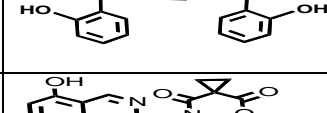
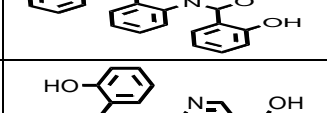
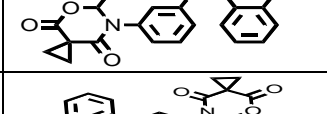
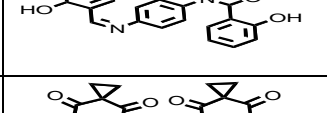
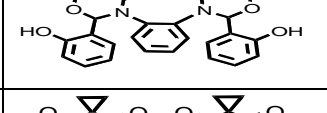
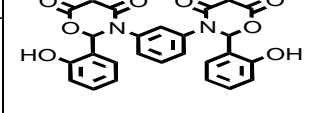
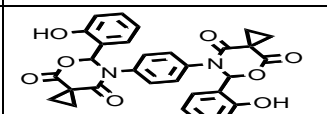
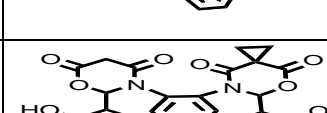
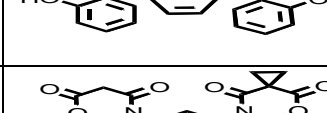
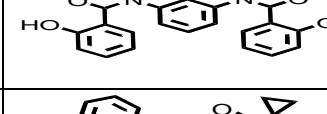
IR spectrum of 3-{3-[(2-hydroxy-benzylidene)-amino]-phenyl}-2-(2-hydroxy-phenyl)-[1, 3] oxazinane-4, 6-dione (**2**).



IR spectrum of 6-(2-hydroxy-phenyl)-7-(2-[6-(2-hydroxy-phenyl)-4,8-dioxo-5-oxa-7-aza-spiro[2.5]oct-7-yl]-phenyl)-5-oxa-7-aza-spiro[2.5]octane-4, 8-dione (**5**).



¹H NMR spectrum of 2-(2-hydroxy-phenyl)-3-{3-[2-(2-hydroxy-phenyl)-4,6-dioxo-[1,3]oxazinane-3-yl]-phenyl}-[1,3]oxazinane-4, 6-dione (**5**).

No.	Name	Structure
1	3-{2-[(2-Hydroxy-benzylidene)-amino]-phenyl}-2-(2-hydroxy-phenyl)-[1,3]oxazinane-4,6-dione	
2	3-{3-[(2-Hydroxy-benzylidene)-amino]-phenyl}-2-(2-hydroxy-phenyl)-[1,3]oxazinane-4,6-dione	
3	3-{4-[(2-Hydroxy-benzylidene)-amino]-phenyl}-2-(2-hydroxy-phenyl)-[1,3]oxazinane-4,6-dione	
4	2-(2-Hydroxy-phenyl)-3-{2-[2-(2-hydroxy-phenyl)-4,6-dioxo-[1,3]oxazin-3-yl]-phenyl}-[1,3]oxazinane-4,6-dione	
5	2-(2-Hydroxy-phenyl)-3-{3-[2-(2-hydroxy-phenyl)-4,6-dioxo-[1,3]oxazin-3-yl]-phenyl}-[1,3]oxazinane-4,6-dione	
6	2-(2-Hydroxy-phenyl)-3-{4-[2-(2-hydroxy-phenyl)-4,6-dioxo-[1,3]oxazin-3-yl]-phenyl}-[1,3]oxazinane-4,6-dione	
7	7-{2-[(2-Hydroxy-benzylidene)-amino]-phenyl}-6-(2-hydroxy-phenyl)-5-oxa-7-aza-spiro[2.5]octane-4,8-dione	
8	7-{3-[(2-Hydroxy-benzylidene)-amino]-phenyl}-6-(2-hydroxy-phenyl)-5-oxa-7-aza-spiro[2.5]octane-4,8-dione	
9	7-{4-[(2-Hydroxy-benzylidene)-amino]-phenyl}-6-(2-hydroxy-phenyl)-5-oxa-7-aza-spiro[2.5]octane-4,8-dione	
10	6-(2-Hydroxy-phenyl)-7-{2-[6-(2-hydroxy-phenyl)-4,8-dioxo-5-oxa-7-aza-spiro[2.5]oct-7-yl]-phenyl}-5-oxa-7-aza-spiro[2.5]octane-4,8-dione	
11	6-(2-Hydroxy-phenyl)-7-{3-[6-(2-hydroxy-phenyl)-4,8-dioxo-5-oxa-7-aza-spiro[2.5]oct-7-yl]-phenyl}-5-oxa-7-aza-spiro[2.5]octane-4,8-dione	
12	6-(2-Hydroxy-phenyl)-7-{4-[6-(2-hydroxy-phenyl)-4,8-dioxo-5-oxa-7-aza-spiro[2.5]oct-7-yl]-phenyl}-5-oxa-7-aza-spiro[2.5]octane-4,8-dione	
13	6-(2-Hydroxy-phenyl)-7-{2-[2-(2-hydroxy-phenyl)-4,6-dioxo-[1,3]oxazin-3-yl]-phenyl}-5-oxa-7-aza-spiro[2.5]octane-4,8-dione	
14	6-(2-Hydroxy-phenyl)-7-{3-[3-(2-hydroxy-phenyl)-4,6-dioxo-[1,2]oxazin-2-yl]-phenyl}-5-oxa-7-aza-spiro[2.5]octane-4,8-dione	
15	7-(2-Hydroxy-phenyl)-6-{4-[2-(2-hydroxy-phenyl)-4,6-dioxo-[1,3]oxazin-3-yl]-phenyl}-5-oxa-6-aza-spiro[2.5]octane-4,8-dione	

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تحضير ودراسة الصفات الفيزيائية لبعض مركبات الاوكسازينانات 5- اوكسو- 7- آزا-سبيرو [2و5] اوكتان من تفاعل ثنائي بنزليدين مع انهيدريد المالونيك 5- اوكسا- سبيرو [2و3] هكسان -4و6-دايون

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الخلاصة:

تم تحضير ثنائي بنزليدين (قواعد شيف) من تكاثف 1 و 2- ثنائي أمينو بنزين مع O-هيدروكسي بنزليدهايد، وعند مفاعلة هذه القواعد مع مول واحد من انهيدريد المالونيك و انهيدريد 5-اوكسو – سبيرو – [2,3] هكسان -4,6-دايون في البنزين الجاف كمذيب تم الحصول على نظام حلقي غير متجانس (سداسي الحلقة) وعند مفاعلة قواعد شيف هذه مع مولين من الانهيدريدات أنفة الذكر أعطى نظام حلقي غير متجانس (بحلقتين سداسيتين). و شخصت المركبات المحضرة باستخدام بعض الطرق الطيفية (IR, UV, ¹H NMR), و التحليل الدقيق للعناصر،