

SYNTHESIS & CHARACTERIZATION OF OXAZEPINE AND PYRROLIDIDES FROM REACTION OF N, N', N''-TRIS-(4-DIMETHYLAMINO-BENZYLIDENE)-[1,3,5]TRIAZENE-2,4,6-TRIAMINE WITH MALEIC,SUCCINIC ANHYDRIDE AND 1H-PYRROLIDENE.

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ABSTRACT: N,N',N''-Tris-(4-dimethylamino-benzylidene)-[1,3,5]triazene-2,4,6-triamine were prepared by condensation of [1,3,5]Triazene-2,4,6-triamine (Melamine) with o-4-dimethylamino-benzaldehyde. These Schiff-bases were reacted with one equivalent of Maleic,Succinic anhydride in absolute ethanol to give 7-membered heterocyclic ring system of 3-{4,6-Bis-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]oxazepine-4,7-dione. Addition of two equivalents of Maleic,Succinic anhydride gave of 8-{4-[(4-dimethylamino-benzylidene)-amino]-6-[4-dimethylamino-phenyl]-4,7-dioxo-4,7-dihydro-[1,3]oxazepine-3-yl}-[1,3,5]triazin-2-yl}-7-(4-dimethylamino-phenyl)-7,8-dihydro-6-oxa-8-aza-benzocycloheptene-5,9-dione. i.e, two distant 7-membered rings. Which were reacted with pyrrolidine to give anilid-pyrrolidine derivatives of maleic and Succinic.

The synthesized compounds were confirmed by their IR, ¹H NMR, UV, spectra and C.H.N. analysis.

. Key words: Synthesis & Characterization of Oxazepine

INTRODUCTION

The synthesis of 2-phenyl -1,3-oxazepine ⁽¹⁾ and the discovery of the central nervous system (CNS) activity of 1,4-benzodiazepine ⁽²⁾ by irradiation of 4-phenyl-2-oxa-3-aza bicyclo [3,2,0] hepta-3,6- dione, encouraged the chemists to look for other ways to build up the 7-membered heterocyclic ring system. One of these ways which was discovered recently , involves direct addition of maleic anhydride to the (N=C) double bond of Schiff bases ,a

number of 2,3-diaryl -2,3-di hydro-1,3-oxazepine-4,7-dione and 2-aryl-3-(1,5-dimethyl-2-phenyl pyrazolonyl)-2,3-dihydro-1,3-oxazepine-4,7-diones were prepared and characterized ^(3,4).

This reaction of maleic anhydride with aromatic aldazines is related to the same reaction carried out in our laboratory. Under relatively severe conditions (150C, 20hr,xylene) , the reaction leads to fused bicyclic products via abis (3+2)

cycloaddition, while under milder conditions (80°C, 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^(5,6).

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

EXPERIMENTAL:-

Melting points were recorded with Gallenkamp Melting point Apparatus and were uncorrected. Elemental analysis were carried out with perkin-Elmer, 2400; CHN Elemental Analyzer. IR spectra were recorded with PYE UNICAM sp-300 Infrared Spectrophotometer in KBr. Their ¹H-NMR spectra were recorded with BRUKER-AC-200MHZFT-NMR in mutha University. UV-Visible spectra were recorded (in ethanol) with Shimadzu Rec-160 spectrophotometer.

Preparation of N, N', N''-Tris-(4-dimethylamino-benzylidene)-[1, 3, 5] triazine-2, 4, 6-triamine.

N, N', N''-Tris-(4-dimethylamino-benzylidene)-[1,3,5]triazene-2,4,6-triamine were prepared by condensation of [1,3,5]Triazine-2,4,6-triamine (Melamine) with 4-dimethylamino-benzaldehyde. To a solution of 0.05 mole of (Melamine) in 30 ml of water was added 0.05 mole or 0.1 mole of 4-dimethylamino-benzaldehyde and refluxed. 2hr. Whereby a yellow crystalline solid separated out. The solid was filtered and recrystallized from ethanol

Preparation of 3-{4,6-Bis-[(2-Hydroxy-benzylidene)-amino]-[1,3,5]triazin-2-yl}-2-(2-Hydroxy-phenyl)-2,3-dihydro-[1,3]oxazepine-4,7-dione.

In a (100ml) round bottom flask equipped with double surfaced condenser fitted with Calcium chloride guard tube, was placed a mixture of 0.01 mole of N, N', N''-Tris-(4-dimethylamino-benzylidene)-[1,3,5]triazene-2,4,6-triamine and 0.01 mole of maleic anhydride in 20 ml of absolute ethanol. The reaction mixture was refluxed in a water bath for 2 hr. The solvent was removed and the resulting solid was recrystallized from THF.

This experiment was repeated using Succinic anhydride in order to obtain other 1, 3-oxazepine.

Attempted hydrolysis of 3-{4-Amino-6-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]Oxazepine-4,7-dione.

a) A mixture of 0.005 mole of 3-{4-Amino-6-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]Oxazepine-4,7-dione. and (10ml) of 10% NaOH solution was refluxed in a water bath for (20 min), then left to cool to (10°C) and acidified with 2M.HCl, whereby a crystalline solid separated out. The solid was filtered and recrystallized from THF. The product was shown to be the original starting substance (11).

b) In another experiment. 0.005 mole of 3-{4-Amino-6-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]Oxazepine-4,7-dione was mixed

with (1) 20 ml of distilled water, (2) 20 ml of 2M.HCl, (3) 20 ml of 10% NaOH solution and left at room temperature overnight . After isolation, the recovered product in each case was shown to be the unreacted starting compound.

Preparation of N-{{4-[(3-Dimethylamino-benzylidene)-amino]-6-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}}-(4-dimethylamino-phenyl)-pyrrolidene-1-yl-methyl]-succinamic acid.

To a mixture of 0.005 mole of 3-{{4-[(4-Dimethylamino benzylidene)-amino]-6-[(3-Dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]Oxazepine-4,7-dione suspended in dry THF, was added an excess (0.03 mole) of dry pyrrolidine . After 10 min of stirring the mixture at room Temperature, a clear solution was obtained. The solution was refluxed at (65C°) in water bath for (45min) than left to room temperature and separated product was filtered , washed twice with (5ml) portion of

dry THF and recrystallized from dioxane.

Several other derivatives were obtained following the same procedure.

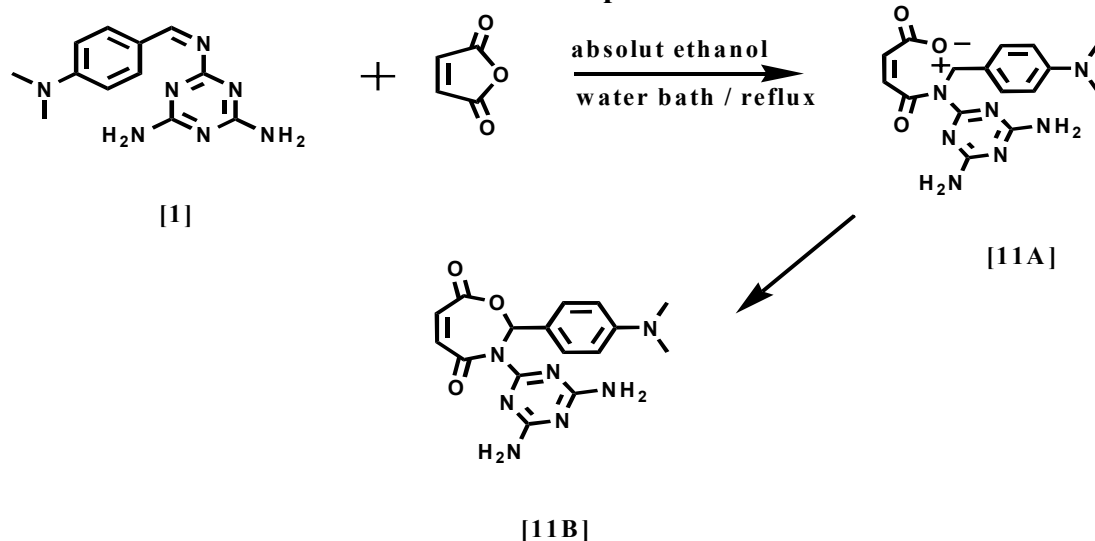
Discussion:-

Schiff bases (A, B, C) are prepared by condensation of [1, 3, 5] Triazine -2, 4, 6-triamine with 4-Dimethylamino-benzaldehyde to give (N),N,N-Di and N,N,N-Tri-(4-Dimethylamino-benzylidene)-[1,3,5]Triazine-2,4,6-triamine. The reaction is followed by the appearance of (N=CH) absorption band at (1600-1610) cm⁻¹ the disappearance of both (C=O) absorption band at (1670-1685) cm⁻¹ and (-NH₂) absorption bands at (3400, 3650) cm⁻¹ in their IR spectra (4).

Schiff bases (A, B,C) are identified by their m.ps. Elemental analysis (table-1) ,IR spectra (table-2) , and UV-Visible spectra (table-3).

It is known that Schiff bases react smoothly with Maleic and Succinic anhydrides to give the corresponding addition products (1-12).

In this paper, the reaction of the cyclic anhydride (maleic anhydride) with Schiff bases (A, B,C) can be presented as follows:



In this reaction, the nitrogen atom of the Schiff base attack one of the two (C=O) groups of anhydride yielding the dipolar intermediate (2) which collapses to the neutral species (11B) which is a combination of ω -lactone and ω -lactam in a 7-membered ring.

The reaction is followed by the disappearance of (N=C) absorption band at (1600-1610) cm^{-1} , and the appearance of the absorption bands of expected groups in the IR spectra of 3-(4,6-Diamino-[1,3,5]triazin-2-yl)-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]Oxazepine-4,7-dione (11).

The (C=O) group in the IR spectra of the addition products, 1,3-oxazepine-4,7-diones and 2-aryl-3-methyl-5,6-dihydro-7H-pyrrolo[1,2-d][1,4]benzodiazepine-6-ones⁽⁷⁻⁹⁾ is absorbed in the same region (1670-1700) cm^{-1} . This conforms the assigned 7-membered ring system structure. The cycloaddition reaction is classified as 2+5 \rightarrow 7, and it is the first cycloaddition of this type, although in principle, one would predict that the pentadienyl cation might add to an olefin through a (4n+2) transition state to yield the cycloheptenyl cation⁽¹⁰⁻¹³⁾.

Structure [11B] is a combination of both lactone and lactam in a 7-heterocyclic ring. This is indicated by the appearance of the characteristic (C=O) (lactone/lactam) absorption band at (1660-1680) cm^{-1} in their IR spectra. Furthermore, structure (11) still maintains the (cis-CH=CH) double bond of maleic anhydride as indicated by the absorption band at (1600-1610) cm^{-1} .

Furthermore, the UV-Visible spectra of Oxazepine derivatives show absorption maxima at (240-350) nm due to charge transfer of

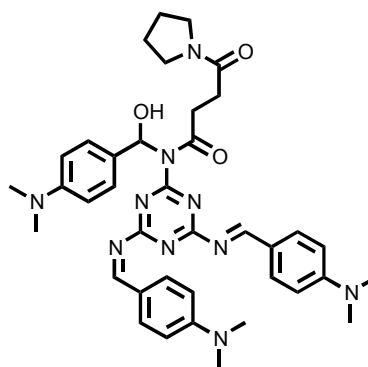
the cyclic 7-membered lactone-lactam combined structure [3], and positive Br_2/CCl_4 and KMNO_4 tests.

Structure [3A] is unlikely, because of the high strain associated with 4-membered ring system (β -lactone ring), particularly when it is fused to another relatively small ring (γ -lactam ring). In addition, Structure [3A] is expected to show the IR absorption band of C=O (β -lactone) at 1750 cm^{-1} and of C=O (γ -lactam) at 1650 cm^{-1} .

However; the lack of these absorption bands and the appearance of cis CH=CH absorption band in the IR spectrum of the lactone-lactam addition product [3] is an indicative evidence against, the structure [3A].

Structure [3B] which can be proposed for these products, results from the (2+cycloaddition of the reactants. The evidences against this structure came from the fact that the cycloaddition (2+2) reaction takes place under the influence of light and it is not expected under thermal condition.

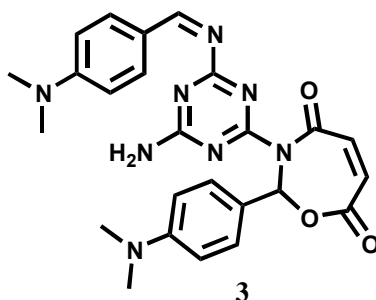
In order to avoid reclosure, the original title compounds (π) are treated with pyrrolidine to give the open-chain anilide-pyrrolidide derivatives of acid [5C]



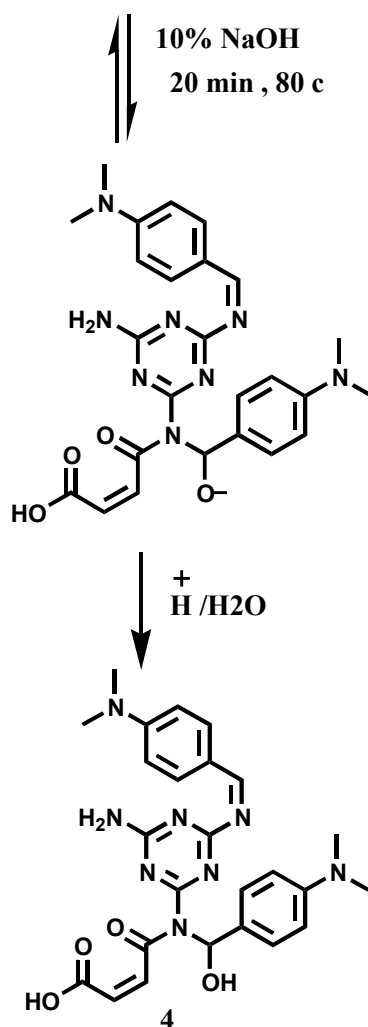
Apparently, this reaction involves an acyl-oxygen cleavage of

the ω -lactone ring, while N-C=O linkage is unaffected under these condition. Since non of the two nitrogen atoms in the resulting products carries hydrogen, where as reclosure to the cyclic diamide is not expected.

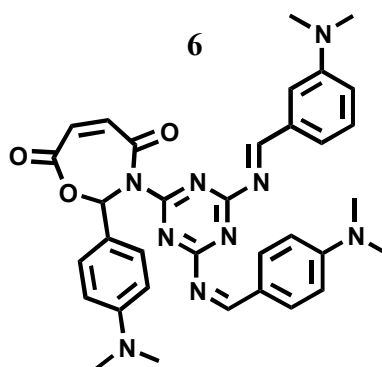
Male 4-oxo -4-pyrrolidine-1-yl-but-2-enoic acid (4, 6-diamino-[1, 3, 5] triazin-2-yl)-[(4-dimethylamino-phenyl)-hydroxyl-methyl]-amide are identified by their m.ps. elemental analysis (table-5), IR spectra (table-7), $^1\text{H-NMR}$ spectra (table-8) and UV-Visible spectra (table-9).



3-{{4-Amino-6-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]oxazepine-4,7-dione



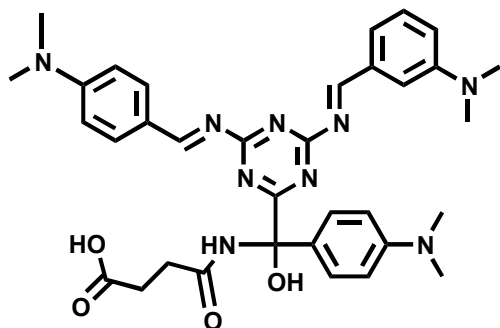
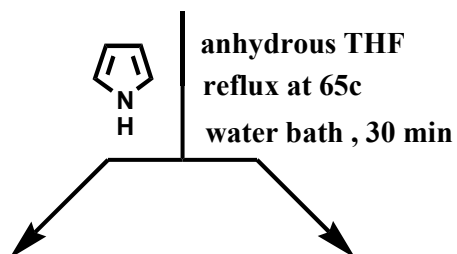
3-{{4-Amino-6-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}}-[(4-dimethylamino-phenyl)-hydroxy-methyl]-carbamoyl}-acrylic acid



6

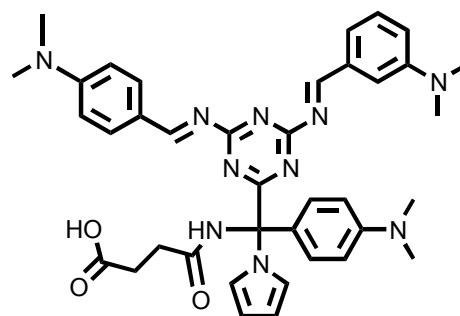
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3-{4-[(4-Dimethylamino-benzylidene)-amino]-6-[(3-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]oxazepine-4,7-dione



5C

N-[4-[(3-Dimethylamino-benzylidene)-amino]-6-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl]-4-(dimethylamino-phenyl)-hydroxy-methyl]-succinamic acid



5D

N-[4-[(3-Dimethylamino-benzylidene)-amino]-6-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl]-4-(dimethylamino-phenyl)-pyrrol-1-yl-methyl]-succinamic acid

Scheme 4

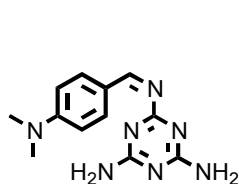
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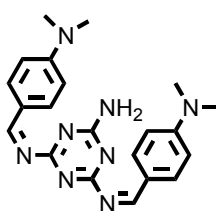
Table (1) : Melting point, percentage yield, molecular formula and element analysis of Schiff-bases (A,B,C)

Comp.	M.P/C°	Yield%	M.F	Calc.			Found		
				C	H	N	C	H	N
A	196	77	C ₁₂ H ₁₅ N ₇	56.02	5.88	38.11	55.87	6,02	37.92
B	185	79	C ₂₁ H ₂₄ N ₈	64.93	6.23	28.84	64.77	6.35	28.63
C	164	68	C ₃₀ H ₃₃ N ₉	69.34	6.40	24.26	69.11	6.55	24.03

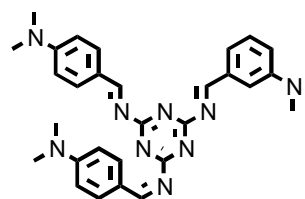
Table (2): The major IR absorptions (cm⁻¹) of Schiff-bases (A,B,C)



A



B



C

Comp.	NH ₂ str. amine	C-H str. Aromatic	C-H str. Alkane	C=N Imine	C=C str. Aromatic	C-H bend Alkane
A	3420,3270	3070	2875	1620	1575,1525	1475,1400
B	3450,3290	3060	2870	1620	1580,1540	1470,1380
C	-	3065	2885	1615	1590,1550	1470,1410

* as KBr disc

Table (3) : The UV-Visible absorption maxima λ/nm of Schiff-bases (A,B,C)

compound	UV-Visible absorption maxima λ/nm
A	380,300, 275, 223
B	360.320,275,226
C	300,265.230.223

Table (4) : Some physical properties and C.H.N. analyses of compound (1-12).

Comp.	m.p/C°	Yield%	Colour	M.F	Calc.			Found		
					C	H	N	C	H	N
1	222	68	orange	C ₁₆ H ₁₇ N ₇ O ₃	54.08	4.82	27.59	54.12	4.90	27.31
2	215	71	yellow	C ₁₆ H ₁₉ N ₇ O ₃	53.77	5.36	27.44	53.54	5.28	27.29
3	198	70	yellow	C ₂₅ H ₂₆ N ₈ O ₃	61.72	5.39	23.03	61.56	5.25	22.97
4	177	78	Brown	C ₂₅ H ₂₈ N ₈ O ₃	61.46	5.78	22.94	61.32	5.66	22.78
5	186	83	Brown	C ₂₉ H ₂₈ N ₈ O ₆	59.58	4.83	19.17	59.50	4.99	19.02
6	203	85	Brown	C ₂₉ H ₃₂ N ₈ O ₆	59.17	5.48	19.04	59.03	5.56	18.87
7	244	74	orange	C ₂₉ H ₃₀ N ₈ O ₆	59.38	5.15	19.10	59.22	5.28	18.92
8	220	77	orange	C ₃₄ H ₃₅ N ₉ O ₃	66.11	5.71	20.41	65.94	5.79	20.289
9	219	67	orange	C ₃₄ H ₃₇ N ₉ O ₃	65.90	6.02	20.34	65.87	6.20	20.13
10	211	63	yellow	C ₃₈ H ₃₇ N ₉ O ₆	63.77	5.21	17.61	63.57	5.34	17.50
11	199	66	orange	C ₃₈ H ₃₉ N ₉ O ₆	63.59	5.48	17.56	63.47	5.50	17.36
12	175	60	yellow	C ₃₈ H ₄₁ N ₉ O ₆	63.41	5.74	17.51	63.33	5.75	17.39

Table (5) : Some physical properties and C.H.N. analyses of compound (25-40).

Comp.	m.p/C°	Yield%	Colour	M.F	Calc.			Found		
					C	H	N	C	H	N
25	222	67	orange	C ₂₀ H ₂₆ N ₈ O ₃	56.33	6.14	26.27	56.27	6.04	26.06
26	235	70	yellow	C ₂₀ H ₂₈ N ₈ O ₃	56.06	6.59	26.15	55.94	6.50	26.00
27	209	61	Brown	C ₂₉ H ₃₅ N ₉ O ₃	62.46	6.33	22.61	62.20	6.21	22.48
28	211	83	Brown	C ₂₉ H ₃₇ N ₉ O ₃	62.24	6.66	22.52	62.11	6.60	22.39
29	220	80	yellow	C ₃₃ H ₃₇ N ₉ O ₆	60.45	5.69	19.23	60.31	5.49	19.20
30	189	74	yellow	C ₃₃ H ₃₉ N ₉ O ₆	60.26	5.98	19.17	60.25	6.03	19.02
31	175	66	yellow	C ₃₇ H ₄₆ N ₁₀ O ₆	61.14	6.38	19.27	60.99	6.40	19.15
32	168	69	orange	C ₃₇ H ₄₈ N ₁₀ O ₆	60.97	6.64	19.22	60.86	6.70	19.03
33	193	69	orange	C ₃₇ H ₅₀ N ₁₀ O ₆	60.80	6.90	19.16	60.71	7.01	19.01
34	162	63	Brown	C ₃₈ H ₄₄ N ₁₀ O ₃	66.26	6.44	20.33	66.11	6.39	20.14
35	205	60	Brown	C ₃₈ H ₄₆ N ₁₀ O ₃	66.07	6.71	20.28	66.00	6.69	20.03
36	200	59	Brown	C ₄₂ H ₄₆ N ₁₀ O ₆	64.11	5.89	17.80	64.01	6.00	17.67
37	182	80	orange	C ₄₂ H ₄₈ N ₁₀ O ₆	63.94	6.13	17.75	63.88	6.25	17.65
38	174	72	orange	C ₄₆ H ₅₅ N ₁₁ O ₆	64.39	6.46	17.96	64.20	6.54	17.82
39	215	62	orange	C ₄₆ H ₅₇ N ₁₁ O ₆	64.24	6.68	17.92	64.10	6.72	17.82
40	199	77	yellow	C ₄₆ H ₅₉ N ₁₁ O ₆	64.09	6.90	17.87	63.96	7.03	17.66

Table (6): IR Spectral data of Compounds (1-12).

Compound	N-H str. amine	C-H str. Olefine	C=O str. Lacton,lactam	C=C str. Olefine	C=C str. Aromatic	C=N str.	C-O str. lacton	C-H bend Aromatic
1	3450, 3200	3150	1670	1600	1580,1560	1430	1330	1020,770
2	3440, 3290	3180	1670	1600	1580,1540	1435	1320	1010,870
3	3440, 3180	3170	1678	1620	1580,1560	1450	1320	1030,920
4	3435, 3210	3150	1675	1610	1580,1555	1440	1330	1055,930
5	3430, 3200	3160	1680	1600	1580,1565	1430	1300	1070,780
6	3430, 3200	3160	1675	1600	1580,1565	1450	1305	1080,770
7	3435, 3210	3150	1680	1610	1585,1560	1440	1320	1070,780
8	-	3110	1680	1620	1590,1560	1430	1325	1080,770
9	-	-	1675	-	1585,1550	1435	1325	1080,780
10	-	3120	1660	1615	1580,1555	1345	1320	1085,900
11	-	3115	1670	1610	1575,1560	1340	1330	1080,880
12	-	-	1675	-	1580,1560	1345	1310	1070,900

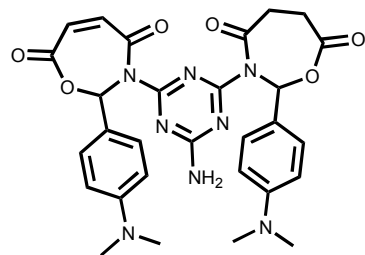
* as KBr disc

Table (7): IR Spectral data of Compounds (25-40).

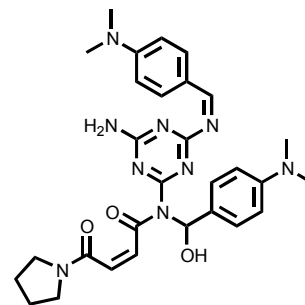
Compound	O-H str. Alcohol	C-H str. Olefine	C=O str. amide	C=C str. Olefine	C=C str. Aromatic	C=N str.	C-O Alcohol
25	3480	3140	1680	1600	1580,1490	-	1360
26	3455	-	1670	-	1590,1520	-	1355
27	3480	3150	1670	1610	1580,1510	1445	1365
28	3460	-	1660	-	1590,1480	1450	1350
29	3450	3160	1680	1605	1585,1530	-	1350
30	3440	3160	1650	1600	1590,1520	-	1335
31	3470		1665	1620	1580,1540	-	1330
32	3450	3140	1685	1615	1570,1510	-	1360
33	3480	-	1675	-	1580,1480	-	1350
34	3460	3160	1670	1610	1590,1480		1350
35	3490	-	1685	-	1590,1510	1445	1345
36	3480	3160	1670	1620	1590,1485	1435	1330
37	3470	-	1675	-	1590,1480	1440	1345
38	3460	3140	1680	1620	1590,1480	1430	1355
39	3450	3160	1680	1610	1590,1520	1445	1350
40	3450	-	1670	-	1585,1530	1430	1365

*** as KBr disc**

Table (8): ¹H.N.M.R Spectrophotometer of compounds (1, 5, 8, 28, 32, and 37)



*Chemical shift = δ

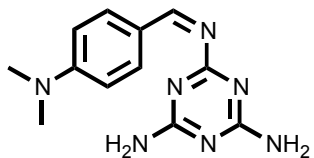
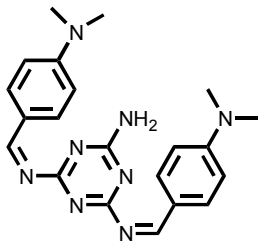
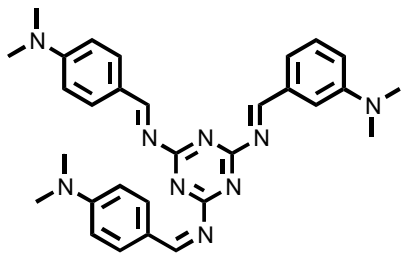


** By using DMSO-d₆as solvent

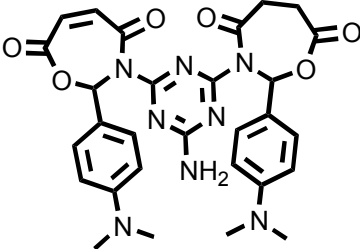
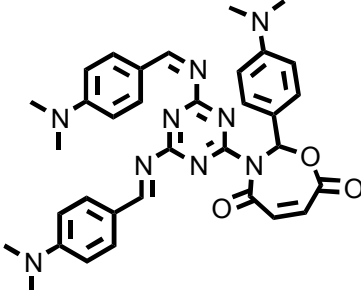
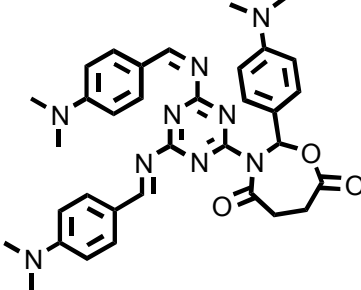
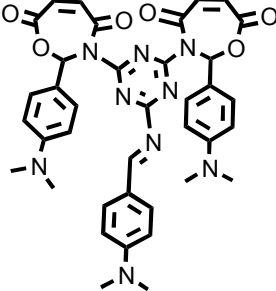
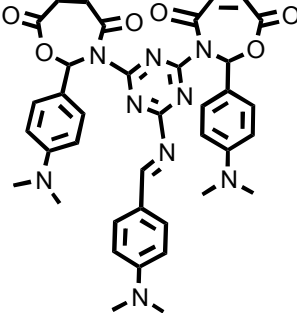
Comp.	NH ₂	<u>CH₂-C-H₂</u>	<u>H-C=C-H</u>	N-CH ₃	N=CH	<u>O-H</u> Alcohol	<u>H-C</u> Aromatic	Pyrrolidine ring			
								H ₂	H ₃	H ₄	H ₅
1	3.80	-	6.4,6.4	5.0		----	6.5-8.1	---	---	---	---
5	3.85	-	6.5,6.5	5.0		----	6.5-7.9	---	---	---	---
8	-	-	6.5,6.5	4.95	7.90	----	6.6-8.0	---	---	---	---
28	3.9	2.3	6.4,6.4	4.95	7.80	2.1	6.5-7.8	3.3	1.5	3.3	1.5
32	3.85	2.35	6.5,6.5	5.0		2.0	6.5-8.0	3.4	1.4	3.4	1.4
37	-	2.4	6.4,6.4	5.0	7.85	2.0	6.6-7.9	3.3	1.5	3.3	1.5

Table (9): The UV-Visible absorption maxima λ /nm of compounds (1-12) and (25-40).

compound	UV-Visible absorption maxima λ /nm of Oxazepine	Comp.	UV-Visible absorption maxima λ /nm of anilid - pyrrolidides
1	320,300,266,230,221	25	329,261,245,221
2	315,255,243,229	26	319,258,238,223
3	333,265,251,243,223	27	320,255,238,220
4	325,278,239,224	28	315,267,240,226
5	329,269,241,236,222	29	314,262,242,228
6	335,300,265,237,220	30	309,266,240,222
7	350,270,230,220	31	385,310,270,254,222
8	325,265,245,225	32	359,310,268,248,229
9	345,290,260,230,224	33	370,300,260,245,225
10	375,268,259,234,222	34	355,320,268,240,225
11	340,280,240,228	35	355,277,252,244,226
12	365,340,275,247,226	36	349,305,267,247,223
		37	359,300,280,254,224
		38	344,295,266,242,228
		39	370,276,245,236,228
		40	350,289,266,245,227

No.	Schiff base Name	Structure
A	N-(4-Dimethylamino-benzylidene)-[1,3,5]triazine-2,4,6-triamine	
B	N,N'-Bis-(4-dimethylamino-benzylidene)-[1,3,5]triazine-2,4,6-triamine	
C	N,N'-Bis-(4-dimethylamino-benzylidene)-N''-(3-dimethylamino-benzylidene)-[1,3,5]triazine-2,4,6-triamine	

No.	Name	Structure
1	3-(4,6-Diamino-[1,3,5]triazin-2-yl)-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]oxazepine-4,7-dione	
2	3-(4,6-Diamino-[1,3,5]triazin-2-yl)-2-(4-dimethylamino-phenyl)-[1,3]oxazepane-4,7-dione	
3	3-{4-Amino-6-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]oxazepine-4,7-dione	
4	3-{4-Amino-6-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]oxazepan-4,7-dione	
5	3-{4-Amino-6-[2-(4-dimethylamino-phenyl)-4,7-dioxo-[1,3]oxazepine-3-yl]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]oxazepine-4,7-dione	
6	3-{4-Amino-6-[2-(4-dimethylamino-phenyl)-4,7-dioxo-[1,3]oxazepan-3-yl]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]oxazepan-4,7-dione	

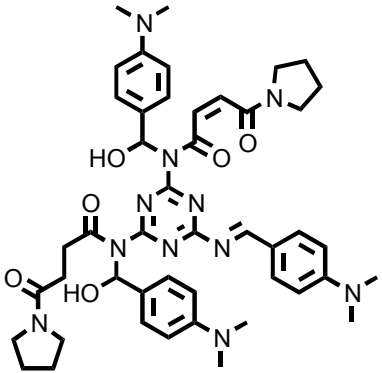
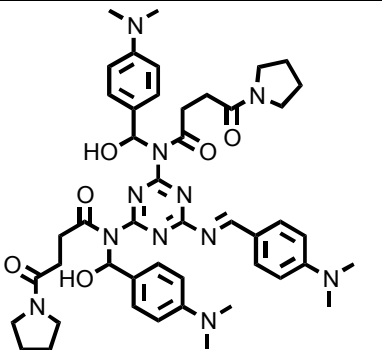
7	3-{4-Amino-6-[2-(4-dimethylamino-phenyl)-4,7-dioxo-[1,3]oxazepine-3-yl]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]oxazepan-4,7-dione	
8	3-{4,6-Bis-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-[1,3]oxazepine-4,7-dione	
9	3-{4,6-Bis-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-[1,3]oxazepane-4,7-dione	
10	3-{4-[(4-Dimethylamino-benzylidene)-amino]-6-[2-(4-dimethylamino-phenyl)-4,7-dioxo-[1,3]oxazepine-3-yl]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]oxazepine-4,7-dione	
11	3-{4-[(4-Dimethylamino-benzylidene)-amino]-6-[2-(4-dimethylamino-phenyl)-4,7-dioxo-[1,3]oxazepan-3-yl]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]oxazepine-4,7-dione	

12	3-{4-[(4-Dimethylamino-benzylidene)-amino]-6-[2-(4-dimethylamino-phenyl)-4,7-dioxo-[1,3]oxazepan-3-yl]-[1,3,5]triazin-2-yl};-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]oxazepan- 4,7-dione	
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No.	Name	Structure
25	4-Oxo-4-pyrrolidin-1-yl-but-2-enoic acid(4,6-diamino-[1,3,5]triazin-2-yl)-[(4-dimethylamino- phenyl)-hydroxy-methyl]-amide	
26	<i>N</i> -(4,6-Diamino-[1,3,5]triazin-2-yl)- <i>N</i> -[(4-dimethylamino-phenyl)-hydroxy-methyl]-4-oxo-4-pyrrolidin-1-yl-butamide	
27	4-Oxo-4-pyrrolidin-1-yl-but-2-enoic acid {4-amino-6-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-[(4-dimethylamino-phenyl)-hydroxy-methyl]-amide	
28	<i>N</i> -{4-Amino-6-[(4-dimethylamino-benzylidene)- amino]-[1,3,5]triazin-2-yl}- <i>N</i> -[(4-dimethylamino-phenyl)-hydroxy-methyl]-4-oxo-4-pyrrolidin-1-yl-butamide	
29	4-Oxo-4-pyrrolidin-1-yl-but-2-enoic acid {4-amino-6-[2-(4-dimethylamino-phenyl)-4,7-dioxo-4,7-dihydro-[1,3]oxazepin-3-yl]-[1,3,5]triazin-2-yl}-[(4-dimethylamino-phenyl)-hydroxy-methyl]-amide	

<p>30</p>	<p><i>N</i>-{4-Amino-6-[2-(4-dimethylamino-phenyl)-4,7-dioxo-4,7-dihydro-[1,3]oxazepin-3-yl]-[1,3,5]triazin-2-yl}-<i>N</i>-[(4-dimethylamino-phenyl)-hydroxy-methyl]-4-oxo-4-pyrrolidin-1-yl- butyramide</p>	
<p>31</p>	<p>4-Oxo-4-pyrrolidin-1-yl-but-2-enoic acid {4-amino-6-[(4-dimethylamino-phenyl)-hydroxy-methyl]- (4-oxo-4-pyrrolidin-1-yl-but-2-enoyl)- amino}-[1,3,5]triazin-2-yl}-[(4-dimethylamino-phenyl)-hydroxy-methyl]-amide</p>	
<p>32</p>	<p>4-Oxo-4-pyrrolidin-1-yl-but-2-enoic acid {4-amino-6-[(4-dimethylamino-phenyl)-hydroxy-methyl]- (4-oxo-4-pyrrolidin-1-yl-but-2-enoyl)- amino}-[1,3,5]triazin-2-yl}-[(4-dimethylamino-phenyl)-hydroxy-methyl]-amide</p>	
<p>33</p>	<p><i>N</i>-{4-Amino-6-[(4-dimethylamino-phenyl)-hydroxy-methyl]- (4-oxo-4-pyrrolidin-1-yl-but-2-enoyl)- amino}-[1,3,5]triazin-2-yl}-<i>N</i>-[(4-dimethylamino-phenyl)-hydroxy-methyl]-4-oxo-4- pyrrolidin-1-yl-but-2-enoic acid</p>	
<p>34</p>	<p>4-Oxo-4-pyrrolidin-1-yl-but-2-enoic acid {4,6-bis-[(4-dimethylamino-benzylidene)-amino]- [1,3,5]triazin-2-yl}-[(4-dimethylamino-phenyl)-hydroxy-methyl]-amide</p>	

<p>35</p>	<p><i>N</i>-{4,6-Bis-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-<i>N</i>-[(4-dimethylamino-phenyl)-hydroxy-methyl]-4-oxo-4-pyrrolidin-1-yl-but-2-enoic acid</p>	
<p>36</p>	<p>4-Oxo-4-pyrrolidin-1-yl-but-2-enoic acid {4-[(4-dimethylamino-benzylidene)-amino]-6-[2-(4-dimethylamino-phenyl)-4,7-dioxo-4,7-dihydro-[1,3]oxazepin-3-yl]-[1,3,5]triazin-2-yl}-[(4-dimethylamino-phenyl)-hydroxy-methyl]-amide</p>	
<p>37</p>	<p><i>N</i>-{4-[(4-Dimethylamino-benzylidene)-amino]-6-[2-(4-dimethylamino-phenyl)-4,7-dioxo-4,7-dihydro-[1,3]oxazepin-3-yl]-[1,3,5]triazin-2-yl}-<i>N</i>-[(4-dimethylamino-phenyl)-hydroxy-methyl]-4-oxo-4-pyrrolidin-1-yl-but-2-enoic acid</p>	
<p>38</p>	<p>4-Oxo-4-pyrrolidin-1-yl-but-2-enoic acid {4-[(4-dimethylamino-benzylidene)-amino]-6-[[4-(4-dimethylamino-phenyl)-hydroxy-methyl]-4-oxo-4-pyrrolidin-1-yl-but-2-enoyl]-amino]-[1,3,5]triazin-2-yl}-[(4-dimethylamino-phenyl)-hydroxy-methyl]-amide</p>	

39	<p>4-Oxo-4-pyrrolidin-1-yl-but-2-enoic acid {4-[4-dimethylamino-benzylidene)-amino]-6- [[(4-dimethylamino-phenyl)-hydroxy-methyl]-4-oxo- 4-pyrrolidin-1-yl-butryl)-amino]-[1,3,5]triazin-2-yl}- [(4-dimethylamino-phenyl)-hydroxy-methyl]-amide</p>	
40	<p>N-{4-[4-Dimethylamino-benzylidene)-amino]-6- [[(4-dimethylamino-phenyl)-hydroxy-methyl]- (4-oxo-4-pyrrolidin-1-yl-butryl)-amino]-[1,3,5] triazin-2-yl}-N-[(4-dimethylamino-phenyl)- hydroxy-methyl]-4-oxo-4-pyrrolidin-1-yl- butyramide</p>	

تحضير ودراسة الصفات الفيزيائية لمركبات الاوكسازيبين والبايرونديينات من
 تفاعل N,N,N- ترس (4- ثنائي مثيل امينو- بنزليدهايد) - [5,3,1] ترايازين -
 6,4,2- تراي أمين مع انهيدريدات الماليك والسكسنيك والبايرونول

عبد الله حسين كشاش، بشرى تركي مهدي

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[5 3 1] -(- 4)-N()

. -4 (A,B,C) -6 4 2-

-

. (12-1)

. (40-25)

-

1H- NMR