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SYNTHESIS & CHARACTERIZATION OF OXAZEPINE AND PYRROLIDIDES FROM REACTION OF N, Ń, Ň-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,Ń,Nӧ-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-benzlidene)-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, ^{1}H NMR, UV, spectra and C.H.N. analysis.

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)].

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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 (80C,2hr,benzene) the reaction leads to a7-membered heterocyclic ring system via a (5+2)→7 cycloaddition.[14] Imines and N-acyl imines react with diketen to give tetrahydro-1,3-oxazine-4-ones [5,6].

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.

EXPERIMENTAL:-

Melting points were recorded withGallenkamp 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 ^{1}H-NMR spectra were recorded with BRUKER-AC-200MHZFT-NMR in mutha University.UV-Visible spectra were recorded (in ethanol) with Schimadsu Recc-160 spectrophotometer.

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

N,Ń,Nӧ-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 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-benzlidene)-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,Ń,Nӧ-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(10C°) 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:-

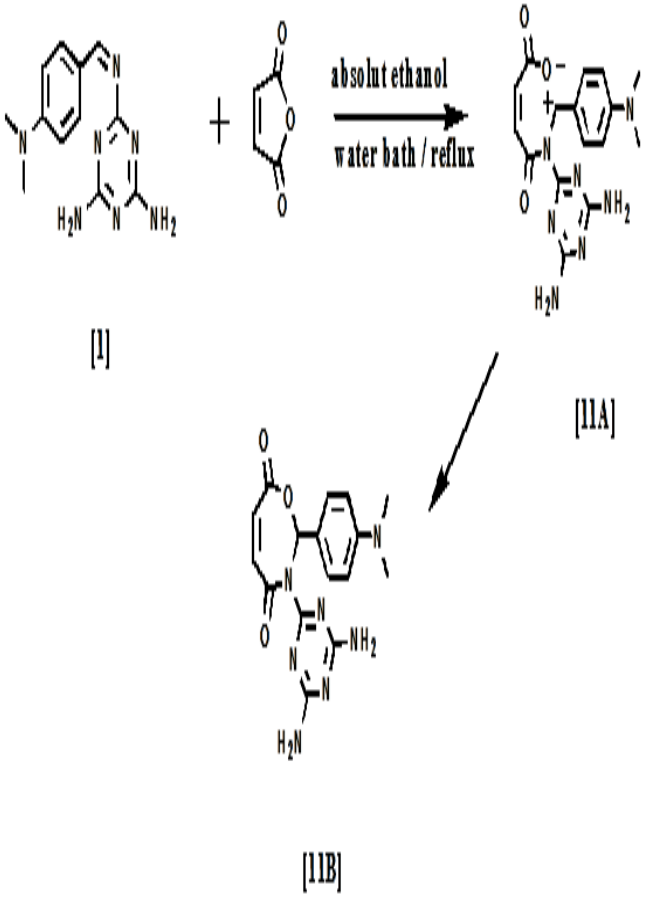
Schiff bases (A, B, C) are prepared by condensation of [1, 3, 5] Triazine -2, 4, 6-triamine with 4-Dimethylamino-benzaldehyde to give (N),N,N-Di and N,N,N-Tri-(4-Dimethylamino-benzylidene)-[1,3,5]Triazine-2,4,6-triamine.The reaction is followed by the appearance of (N=CH) absorption band at (1600-1610) cm^{-1} the disappearance of both (C=O) absorption band at (1670-1685) cm^{-1} and ($-NH_2$) 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=



[1] [11A] [11B]

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—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] .

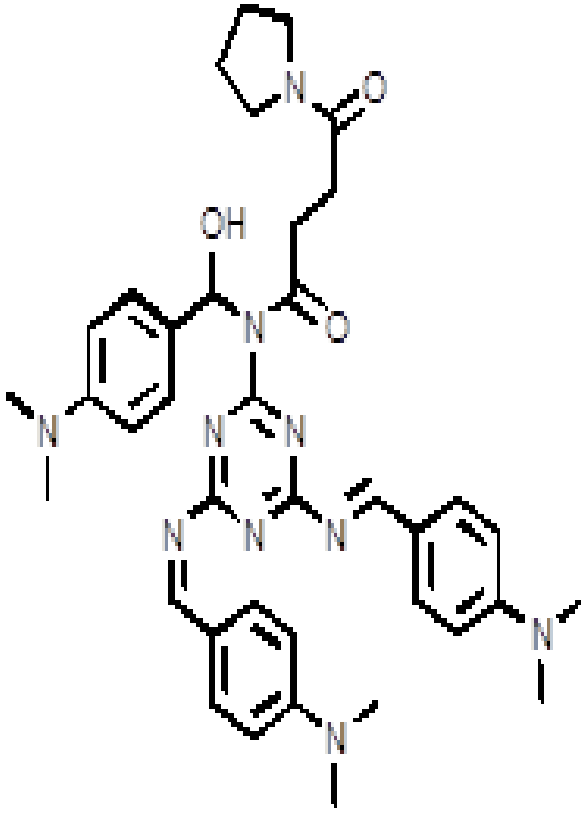
Structure [11B] is a combination of both lactone and lactam in a 7- heterocyclic ring. This is indicated by the appearance of the characteristic(C=O) (lactone/lactam) absorption band at (1660-1680) cm^{-1} in their IR spectra. Furthermore, structure (11) still maintains the (cis-CH=CH) double bond of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

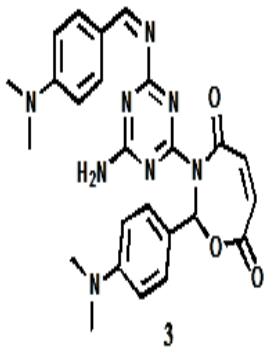
Structure [3B] which can be proposed for these products, results from the (2+cycloaddition of the reactants. The evidences against this structure came from the fact that the cycloaddition (2+2) reaction takes place under the influence of light and it is not expected under thermal condition.

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}H-NMR spectra (table-8) and UV-Visible spectra (table-9).

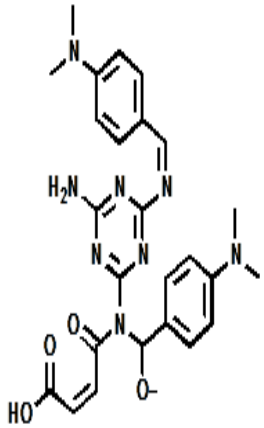


3

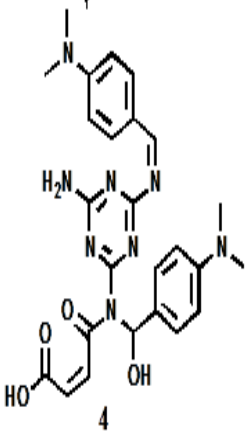
3-{4-Amino-6-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]oxazepine-4,7-dione

10% NaOH

20 min , 80 c



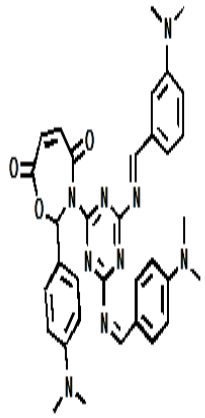
$\overset{+}{H}$ /H2O



4

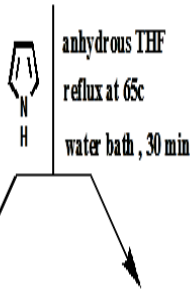
3-{{4-Amino-6-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-[(4-dimethylamino-phenyl)-hydroxy-methyl]-carbamoyl}-acrylic acid

Scheme 3



5

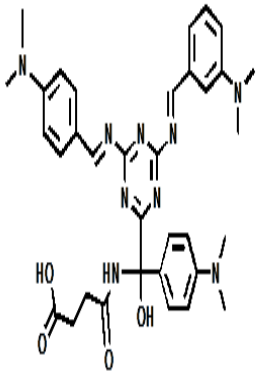
3-{4-[(4-Dimethylamino-benzylidene)-amino]-6-[(3-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]oxazepine-4,7-dione



anhydrous THF

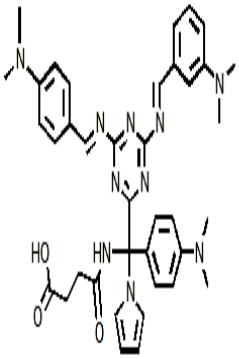
reflux at 65c

water bath , 30 min



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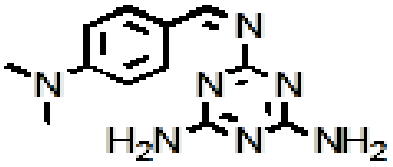
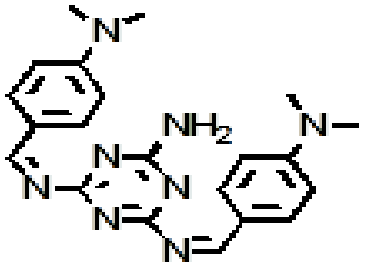
Table (1) : Melting point, percentage yield, molecular formula and element analysis of Schiff-bases (A,B,C)

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

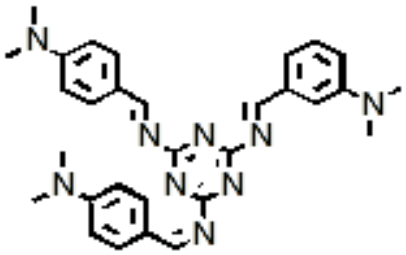
Table (2): The major IR absorptions (cm^{-1}) of Schiff-bases (A,B,C)



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

C	B
-	3450,3290
3065	3060
2885	2870
1615	1620
1590,1550	1580,1540
1470,1410	1470,1380

*** 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.		Calc.			Found		
m.p/C°							
Yield%							
Colour							
M.F		C	H	N	C	H	N
1	$C_{16}H_{17}N_7O_3$	54.08	4.82	27.59	54.12	4.90	27.31
2	$C_{16}H_{19}N_7O_3$	53.77	5.36	27.44	53.54	5.28	27.29
3	$C_{25}H_{26}N_8O_3$	61.72	5.39	23.03	61.56	5.25	22.97
4	$C_{25}H_{28}N_8O_3$	61.46	5.78	22.94	61.32	5.66	22.78
5	$C_{29}H_{28}N_8O_6$	59.58	4.83	19.17	59.50	4.99	19.02
6	$C_{29}H_{32}N_8O_6$	59.17	5.48	19.04	59.03	5.56	18.87
7	$C_{29}H_{30}N_8O_6$	59.38	5.15	19.10	59.22	5.28	18.92
8	$C_{34}H_{35}N_9O_3$	66.11	5.71	20.41	65.94	5.79	20.289
9	$C_{34}H_{37}N_9O_3$	65.90	6.02	20.34	65.87	6.20	20.13
10	$C_{38}H_{37}N_9O_6$	63.77	5.21	17.61	63.57	5.34	17.50
11	$C_{38}H_{39}N_9O_6$	63.59	5.48	17.56	63.47	5.50	17.36
12	$C_{38}H_{41}N_9O_6$	63.41	5.74	17.51	63.33	5.75	17.39
215							
71							
yellow							
198							
70							
yellow							
177							
78							
Brown							
186							
83							
Brown							
203							
85							
Brown							
244							
74							
orange							
220							
77							
orange							
219							
67							
orange							
211							
63							
yellow							
199							
66							
orange							

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

Comp.	25	26	27	28	29	30	31	32
m.p/C°	222	235	209	211	220	189	175	168
Yield%	67	70	61	83	80	74	66	69
Colour	orange	yellow	Brown	Brown	yellow	yellow	yellow	orange
M.F	$C_{20}H_{26}N_8O_3$	$C_{20}H_{28}N_8O_3$	$C_{29}H_{35}N_9O_3$	$C_{29}H_{37}N_9O_3$	$C_{33}H_{37}N_9O_6$	$C_{33}H39N_9O_6$	$C_{37}H_{46}N_{10}O_6$	$C_{37}H_{48}N_{10}O_6$
Calc. C	56.33	56.06	62.46	62.24	60.45	60.26	61.14	60.97
Calc. H	6.14	6.59	6.33	6.66	5.69	5.98	6.38	6.64
Calc. N	26.27	26.15	22.61	22.52	19.23	19.17	19.27	19.22
Found C	56.27	55.94	62.20	62.11	60.31	60.25	60.99	60.86
Found H	6.04	6.50	6.21	6.60	5.49	6.03	6.40	6.70
Found N	26.06	26.00	22.48	22.39	19.20	19.02	19.15	19.03

33	34	35	36	37	38	39	40
193	162	205	200	182	174	215	199
69	63	60	59	80	72	62	77
orange	Brown	Brown	Brown	orange	orange	orange	yellow
$C_{37}H_{50}N_{10}O_6$	$C_{38}H_{44}N_{10}O_3$	$C_{38}H_{46}N_{10}O_3$	$C_{42}H_{46}N_{10}O_6$	$C_{42}H_{48}N_{10}O_6$	$C_{46}H_{55}N_{11}O_6$	$C_{46}H_{57}N_{11}O_6$	$C_{46}H_{59}N_{11}O_6$
60.80	66.26	66.07	64.11	63.94	64.39	64.24	64.09
6.90	6.44	6.71	5.89	6.13	6.46	6.68	6.90
19.16	20.33	20.28	17.80	17.75	17.96	17.92	17.87
60.71	66.11	66.00	64.01	63.88	64.20	64.10	63.96
7.01	6.39	6.69	6.00	6.25	6.54	6.72	7.03
19.01	20.14	20.03	17.67	17.65	17.82	17.82	17.66

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

Compound	1	2	3	4	5	6
N-H str. amine	3450. 3200	3440. 3290	3440. 3180	3435. 3210	3430. 3200	3430. 3200
C-H str. Olefine	3150	3180	3170	3150	3160	3160
C=O str. Lacton.lactam	1670	1670	1678	1675	1680	1675
C=C str. Olefine	1600	1600	1620	1610	1600	1600
C=C str. Aromatic	1580.1560	1580.1540	1580.1560	1580.1555	1580.1565	1580.1565
C=N str.	1430	1435	1450	1440	1430	1450
C-O str. lacton	1330	1320	1320	1330	1300	1305
C-H bend Aromatic	1020.770	1010.870	1030.920	1055.930	1070.780	1080.770

7	8	9	10	11	12
3435. 3210	-	-	-	-	-
3150	3110	-	3120	3115	-
1680	1680	1675	1660	1670	1675
1610	1620	-	1615	1610	-
1585.1560	1590.1560	1585.1550	1580.1555	1575.1560	1580.1560
1440	1430	1435	1345	1340	1345
1320	1325	1325	1320	1330	1310
1070.780	1080.770	1080.780	1085.900	1080.880	1070.900

*** as KBr disc**

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

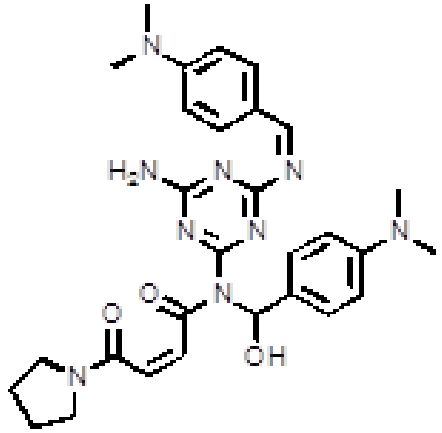
Compound
O-H str. Alcohol
C-H str. Olefine
C=O str. amide
C=C str. Olefine
C=C str. Aromatic
C=N str.
C-O Alcohol

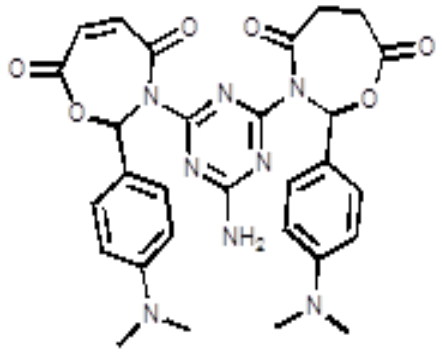
25	26	27	28	29	30	31	32
3480	3455	3480	3460	3450	3440	3470	3450
3140	-	3150	-	3160	3160		3140
1680	1670	1670	1660	1680	1650	1665	1685
1600	-	1610	-	1605	1600	1620	1615
1580,1490	1590,1520	1580,1510	1590,1480	1585,1530	1590,1520	1580,1540	1570,1510
-	-	1445	1450	-	-	-	-
1360	1355	1365	1350	1350	1335	1330	1360

33	34	35	36	37	38	39	40
3480	3460	3490	3480	3470	3460	3450	3450
-	3160	-	3160	-	3140	3160	-
1675	1670	1685	1670	1675	1680	1680	1670
-	1610	-	1620	-	1620	1610	-
1580,1480	1590,1480	1590,1510	1590,1485	1590,1480	1590,1480	1590,1520	1585,1530
-		1445	1435	1440	1430	1445	1430
1350	1350	1345	1330	1345	1355	1350	1365

*** as KBr disc**

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





Comp.	1	5	8	28	32	37
NH_2	3.80	3.85	-	3.9	3.85	-
CH_2-C-H_2	-	-	-	2.3	2.35	2.4
H-C=C-H	6.4,6.4	6.5,6.5	6.5,6.5	6.4,6.4	6.5,6.5	6.4,6.4
N-CH_3	5.0	5.0	4.95	4.95	5.0	5.0
N=CH			7.90	7.80		7.85
O-H Alcohol	----	----	----	2.1	2.0	2.0
H-C Aromatic	6.5-8.1	6.5-7.9	6.6-8.0	6.5-7.8	6.5-8.0	6.6-7.9
Pyrrolidine ring H_2	---	---	---	3.3	3.4	3.3
Pyrrolidine ring H_3	---	---	---	1.5	1.4	1.5
Pyrrolidine ring H_4	---	---	---	3.3	3.4	3.3
Pyrrolidine ring H_5	---	---	---	1.5	1.4	1.5

*Chemical shift =δ ** By using DMSO-$d_6$as solvent

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

compound	1	2	3
UV-Visible absorption maxima λ/nm of Oxazepine	320,300,266,230.221	315,255,243,229	333,265,251,243,223
Comp.	25	26	27
UV-Visible absorption maxima λ/nm of anilid - pyrrolidides	329,261,245,221	319,258,238,223	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

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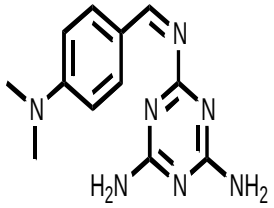
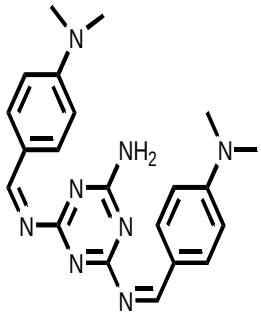
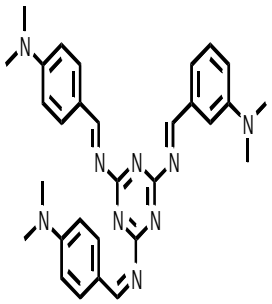
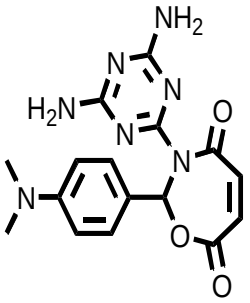
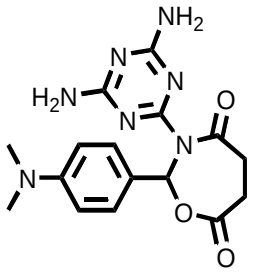
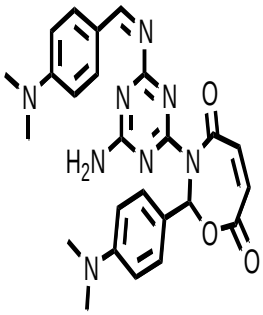
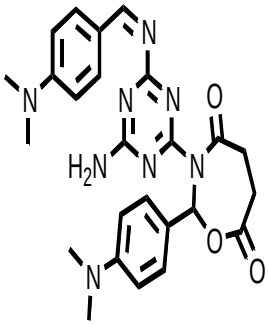
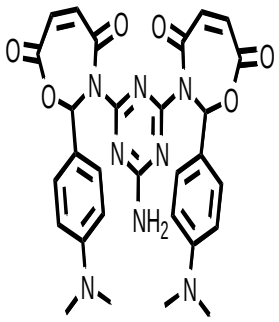
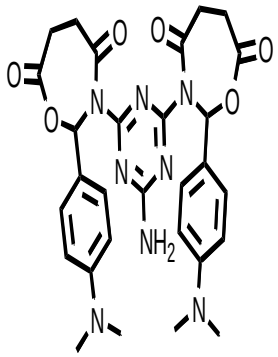
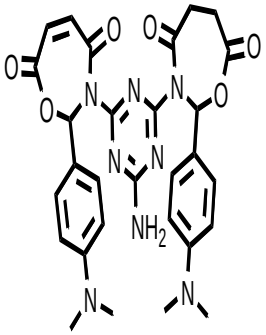
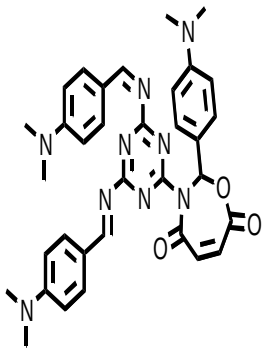
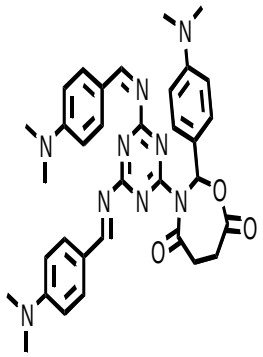
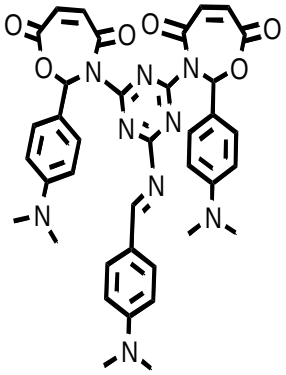
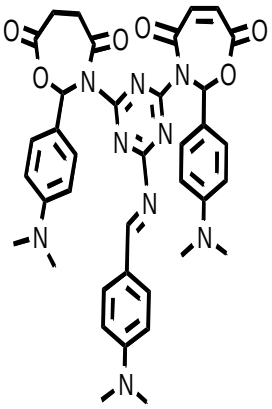
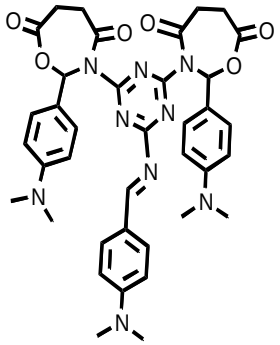
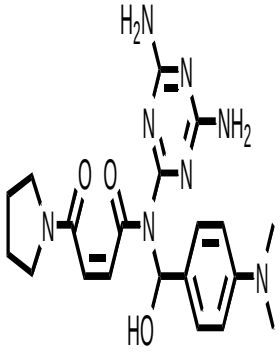
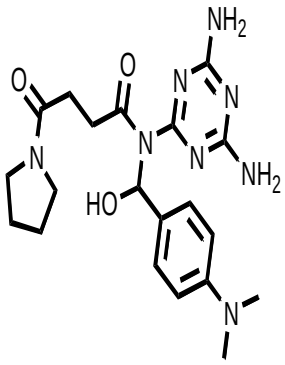
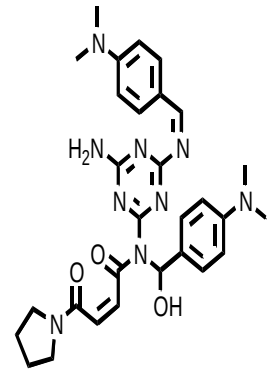
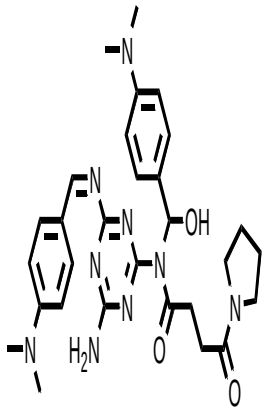
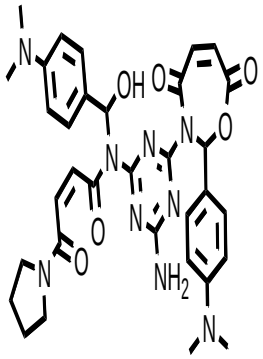
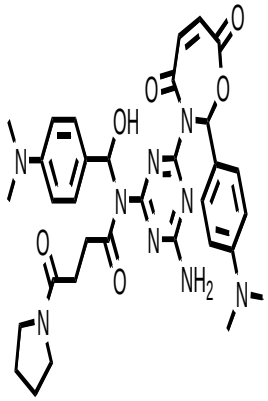
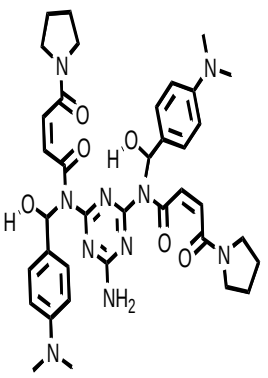
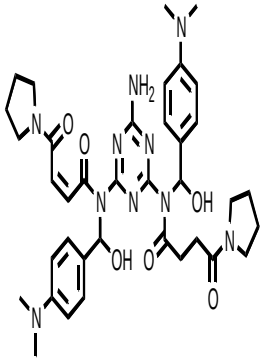
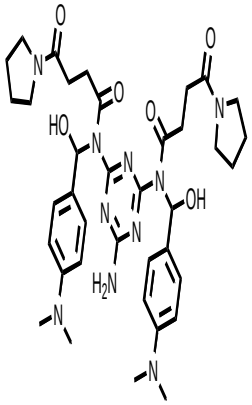
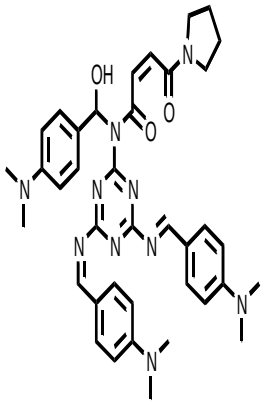
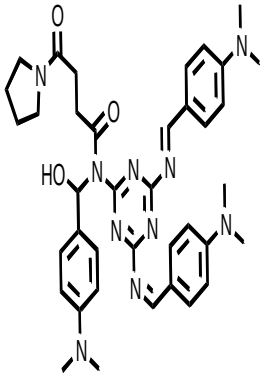
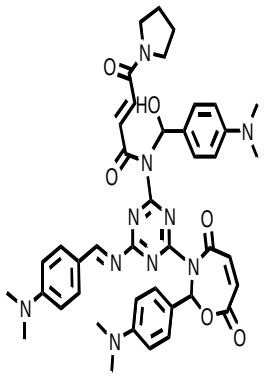
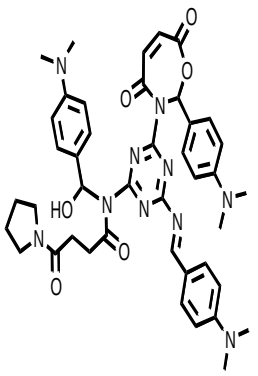
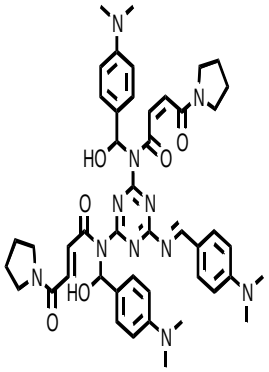
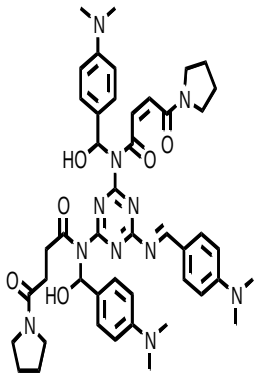
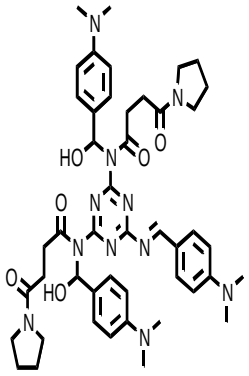
No.	Schiff base Name	Structure
A	N-(4-Dimethylamino-benzylidene)-[1,3,5]triazine-2,4,6-triamine	
B	*N,N'*-Bis-(4-dimethylamino-benzylidene)-[1,3,5]triazine-2,4,6-triamine	
C	*N,N'*-Bis-(4-dimethylamino-benzylidene)-*N''*-(3-dimethylamino-benzylidene)-[1,3,5] triazine-2,4,6-triamine	
1	3-(4,6-Diamino-[1,3,5]triazin-2-yl)-2-(4-dimethylamino-phenyl)-2,3-dihydro-[1,3]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-dimethyl amino-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-dimethyl amino-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	
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	*N*-(4,6-Diamino-[1,3,5]triazin-2-yl)-*N*-[(4-dimethylamino-phenyl)-hydroxy-methyl]-4-oxo-4-pyrrolidin-1-yl-butyramide	
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	*N*-{4-Amino-6-[(4-dimethylamino-benzylidene)- amino]-[1,3,5]triazin-2-yl}-*N*-[(4-dimethylamino-phenyl)-hydroxy-methyl]-4-oxo-4-pyrrolidin-1-yl-butyramide	
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	
30	*N*-{4-Amino-6-[2-(4-dimethylamino-phenyl)-4,7-dioxo-4,7-dihydro-[1,3]oxazepin-3-yl]-[1,3,5] triazin-2-yl}-*N*-[(4-dimethylamino-phenyl)-hydroxy-methyl]-4-oxo-4-pyrrolidin-1-yl- butyramide	
31	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	

32	4-Oxo-4-pyrrolidin-1-yl-but-2-enoic acid {4-amino-6-[[(4-dimethylamino-phenyl)-hydroxy-methyl]-(4-oxo-4-pyrrolidin-1-yl-butyryl)-amino]-[1,3,5]triazin-2-yl}-[(4-dimethylamino-phenyl)-hydroxy-methyl]-amide	
33	*N*-{4-Amino-6-[[(4-dimethylamino-phenyl)-hydroxy-methyl]-(4-oxo-4-pyrrolidin-1-yl-butyryl)-amino]-[1,3,5]triazin-2-yl}-*N*-[(4-dimethylamino-phenyl)-hydroxy-methyl]-4-oxo-4- pyrrolidin-1-yl-butyramide	
34	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	
35	*N*-{4,6-Bis-[(4-dimethylamino-benzylidene)-amino]-[1,3,5]triazin-2-yl}-*N*-[(4-dimethylamino-phenyl) -hydroxy-methyl]-4-oxo-4-pyrrolidin-1-yl- butyramide	
36	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	
37	*N*-{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}-*N*-[(4-dimethylamino-phenyl)-hydroxy-methyl]-4-oxo-4-pyrrolidin-1-yl-butyramide	

38	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-but-2-enoyl)-amino]-[1,3,5]triazin-2-yl}-[(4-dimethylamino-phenyl)-hydroxy-methyl]-amide	
39	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-butyryl)-amino]-[1,3,5]triazin-2-yl}-[(4-dimethylamino-phenyl)-hydroxy-methyl]-amide	

40	*N*-{4-[(4-Dimethylamino-benzylidene)-amino]-6-[[(4-dimethylamino-phenyl)-hydroxy-methyl]-(4-oxo-4-pyrrolidin-1-yl-butyryl)-amino]-[1,3,5] triazin-2-yl}-*N*-[(4-dimethylamino-phenyl)-hydroxy-methyl]-4-oxo-4-pyrrolidin-1-yl- butyramide	

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تحضير ودراسة الصفات الفيزياوية لمركبات الاوكسازبين والبايرولدينات من تفاعل -N,N,N- ترس (4- ثنائي مثيل امينو- بنزلديهايد) –[5,3,1] ترايازين --6,4,2 - تراي أمين مع انهيدريدات الماليك والسكسنيك والبايرول

عبدا لله حسين كشاش، بشرى تركي مهدي

الخلاصة:

تم تحضير عدد من (قواعد شيف)N – (4–ثنائي مثيل امينو–بنزايلدين)– [1و3و5] ترايزين–2و4و6–تراي أمين (A,B,C) بتكاثف الميلامين مع 4–ثنائي مثيل بنزالديهايد. فوعلت قواعد شيف هذه مع انهيدريد الماليك، انهيدريد السكسنيك فأعطت–أثنى عشر مشتقات الاوكسازبين والاوكسازبان (12–1). فوعلت الأخيرة مع البيرولدين الجاف فأعطت خمسة عشر مشتقا من الانيليد–البيروليديد لحوامض الماليئاميك ووالسكسناميك (40–25).

وتم تشخيص المركبات الناتجة بالطرق الطيفية الأشعة تحت الحمراء والأشعة فوق البنفسجية وطيف الرنين النووي المغناطيسي 1H-NMR