Synthesis and Characterization of Oxazepine and Oxazepane from reaction of 2-(2-Amino-ethylimino)-5,5-diethyl-dihydro-pyrimidine-4,6-dione and 2-[2-(5,5-diethyl-4,6-dioxo-pyrimidine-2-ylideneamino)-ethylimino]-5,5-diethyl-dihydro-pyrimidine-4,6-dione with maleic and Succinic anhydride.

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Abstract

2-(2-Amino-ethylimino)-5,5-diethyl-dihydro-pyrimidine-4,6-dione and 2-[2-(5,5-diethyl-4,6-dioxo-pyrimidine-2-ylideneamino)-ethylimino]-5,5-diethyl-dihydro- pyrimidine-4,6-dione were prepared by condensation of ethylenediamine with one equivalent and two equivalent of 5,5-diethyl-pyrimidine-2,4,6-trione. These Schiff-bases were reacted with one equivalent of maleic and succinic anhydride in absolute ethanol to give 7-membered heterocyclic ring system of 12-(2-Amino-ethyl)-3,3-diethyl-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-2,4,8,11-tetraone and were reacted with two equivalent of maleic and succinic anhydride in same solvent to give 2 (7-membered) heterocyclic ring system of 1-[2-(3,3-Diethyl-2,4,8,11-tetraoxo-7-1,5,12-triaza-spiro[5.6]dodec-12-yl)-ethyl-3,3-diethyl-7-oxa-1,5,12-triaza-Spiro[5.6] dodecane -2,4,8,11-tetraone.

The final products were identified by their melting points, elemental analysis,IR, ^{1}HNMR and UV-Visible spectra.

Introduction

5,5-diethyl barbituric acid derivatives are interesting series of heterocyclic compounds, which have been shown to be diverse pharmacological properties [1] such as antifungal [2] m antimicrobial [3] antipulsive [4] and antibacterial [5,6].

The synthesis of 2-phenyl -1,3-oxazepine [7] and the discovery of the central nervous system (CNS) activity of 1,4-benzodiazepine [8] 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 , 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 [9,10].

N-acyl ammonium 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 intermolecular Diels-alder reaction to give dihydro-1,3-oxazines [11].

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 [12]. 2-(2-Hydroxy-phenyl)-4,7-dioxo-4,7-dihydro-[1,3] oxazepine-3-carboxylic acid amide and 2-(2-hydroxy-phenyl)-3-[2-(2-hydroxy-phenyl)-4,7-dioxo-[1,3]oxazepine-3-carbonyl]-2,3-dihydro-[1,3]oxazepine-4,7-dione were synthesised from reaction of Cyclo anhydride with 1,3 bis(2-hydroxy-benzylidene)-urea (Schiff-bases) [13].

pyrylium tetrafluoroborate underwent ring expansion on treatment with excess of sodiumazide in anhydrous 1,4-dioxane to give 58-96% substituted 1,3-oxazepine.

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Furthermore, thermal rearrangement of ketovinylazirines gave substituted 1,3-oxazepines. [14]

Experimental

Melting points were recorded on Gallenkamp melting points Apparatus and were uncorrected . Elemental analysis was carried out in Mutah University on perkin-Elmre 2400 CHN Elemental analyzer . FT-IR spectra were recorded on FT-IR spectrophotometer -8400s Shimadza (KBr) and UV-Visible spectra were recorded (in ethanol) on Schimadza Reco- 160 Spectrophotometer. Their H-NMR spectra were recorded with BRUKER AC 200MHZFT NMR spectrophotometer.

Preparation of 2-(2-Amino-ethylimino)-5,5-diethyl-dihydro-pyrimidine-4,6-dione and 2-[2-(5,5-diethyl-4,6-dioxo-pyrimidine-2-ylideneamino)-ethylimino]-5,5-diethyl-dihydro- pyrimidine-4,6-dione (Schiff-base)

2-(2-Amino-ethylimino)-5,5-diethyl-dihydro-pyrimidine-4,6-dione and 2-[2-(5,5-diethyl-4,6-dioxo-pyrimidine-2-ylideneamino)-ethylimino]-5,5-diethyl-dihydro- pyrimidine-4,6-dione were prepared by condensation of one equivalent and two equivalent of ethylene diamine with one equivalent of 5,5-diethyl-pyrimidine-2,4,6-trione.

To a solution of 0.05 and 0.1 mole of ethylene diamine in 30 ml of ethanol (absolute) was added 0.05 mole of 5,5-diethyl-pyrimidine-2,4,6-trione and refluxed 2hr. Where by a yellow crystalline solid separated out . The solid was filtered and recrystallized from ethanol.

Preparation of 1-[2-(3,3-Diethyl-2,4,8,11-tetraoxo-7-1,5,12-triaza-spiro[5.6]dodec-12-yl)-ethyl-3,3-diethyl-7-oxa-1,5,12-triaza-Spiro[5.6] dodecane -2,4,8,11-tetraone.

In a 100 ml round bottom flask equipped with double surface condenser fitted with calcium chloride guard tube was placed a mixture of 0.01 mole of 2,6-Bis-(2-amino-ethylimino)-5,5-diethyl-tetrahydro-pyrimidine-4-one and 0.01mole maleic anhydride in 20 ml of ethanol absolute.

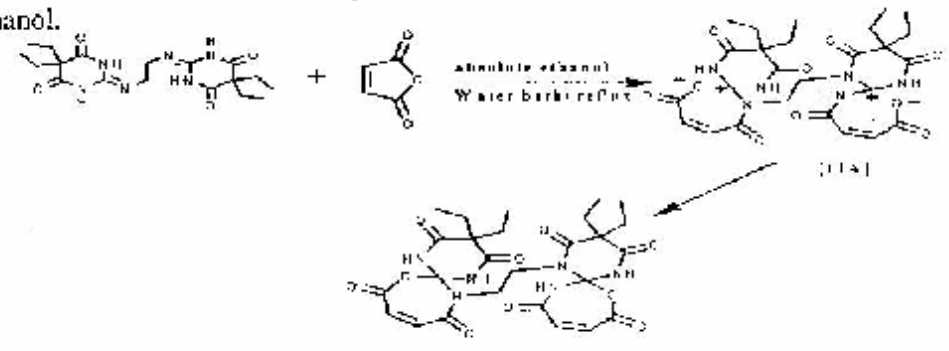
The reaction mixture was refluxed in water bath at 78C for 3hr, the solvent was then removed and the resulting solid was recrystallized from anhydrous THF.

This experiment was repeated using different of anhydride to obtain other derivatives.

Result and Discussion

It is known that Schiff bases react smoothly with acid chlorides and anhydrides to give the corresponding addition products[05-19]

In this paper, the reaction of the maleic and succinic anhydrides with 2-(2-Amino-ethylimino)-5,5-diethyl-dihydro-pyrimidine-4,6-dione and 2,6-Bis-(2-amino-ethylimino)-5,5-diethyl-tetrahydro-pyrimidine-4-one to gives the dipolar intermediate **[11A]** which collapses to the 7- membered heterocyclic ring system **[11B]** is presented.



[11A]

[11B]

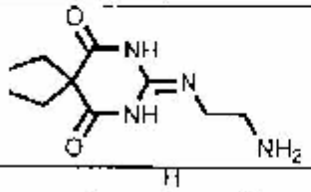
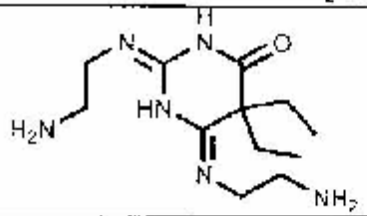
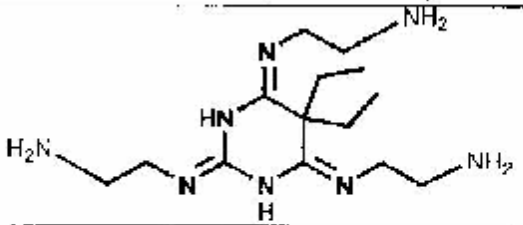
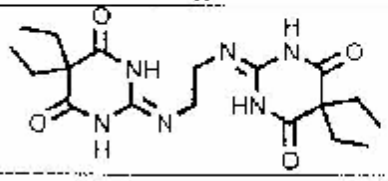
Schem (1)

This is indicated by the appearance of the characteristic C=O (lacton-lactam) absorption band at 1700cm^{-1} in the IR spectra of addition products[11B].

It is impressive to note that the two absorption bands at (1800-1950)cm^{-1} in the IR spectra of pure maleic and succinic anhydride have disappeared when the anhydride became part of the 7-membered ring system of the 12-(2-Amino-ethyl)-3,3-diethyl-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-2,4,8,11-tetraone and 1-[2-(3,3-diethyl-2,4,8,11-tetraoxo-7-1,5,12-triaza-spiro[5.6]dodec-12-yl)-ethyl-3,3-diethyl-7-oxa-1,5,12-triaza-Spiro[5.6] dodecane -2,4,8,11-tetraone.The UV-spectrum of addition products [11B] show absorption maxima at (240-320)nm , and at (310-450)nm due to charge transfer of the aryl group and the cyclic 7-membered structure [11B].

These compounds are identified by their m.ps,elemental analysis (Table 1),IR (Table 2) and UV spectra (table 3). It is noticeable that the values of C-Hstr. 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 maleic and succinic anhydride 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 [20] 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 butadiene cation might add to an olefin through a (4n−2) transition state to yield the cyclohexenyl cation [21].

No.	Schiff-Bases Name	Structure
A	2-(2-Amino-ethylimino)-5,5-diethyl-dihydro-pyrimidine-4,6-dione	
B	2,6-Bis-(2-amino-ethylimino)-5,5-diethyl-tetrahydro-pyrimidin-4-one	
C	2,4,6-Tris-(2-amino-ethylimino)-5,5-diethyl-hexahydro-pyrimidine	
D	2-[2-(5,5-Diethyl-4,6-dioxo-pyrimidine-2-ylideneamino)-ethylimino]-5,5-diethyl-dihydro-pyrimidine-4,6-dione	

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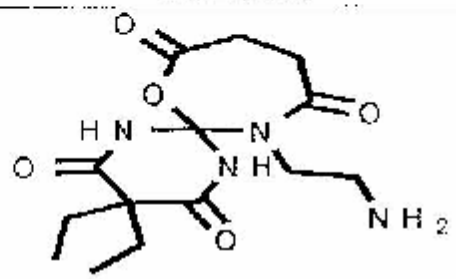
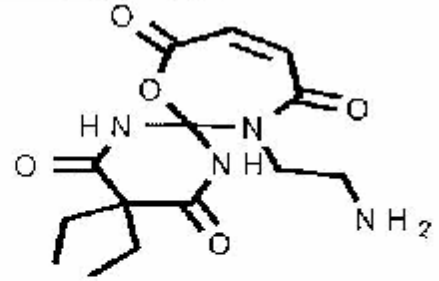
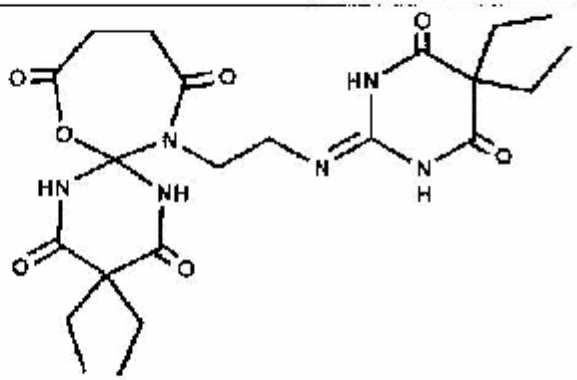
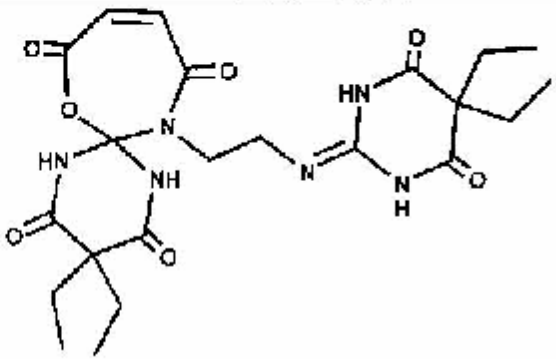
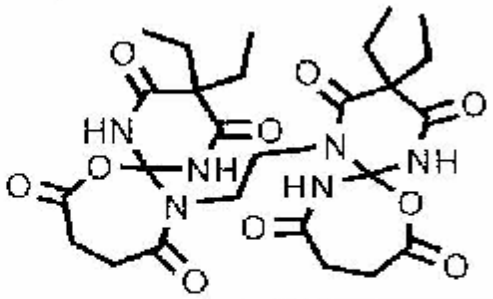
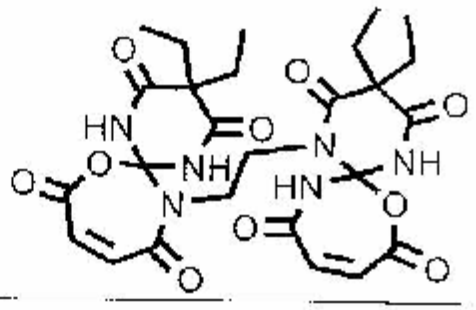
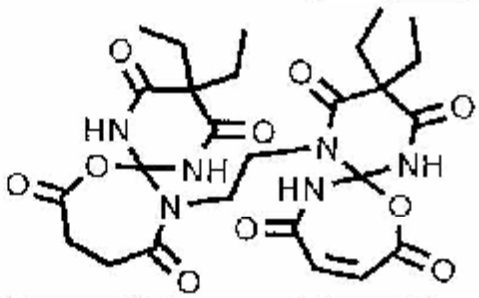
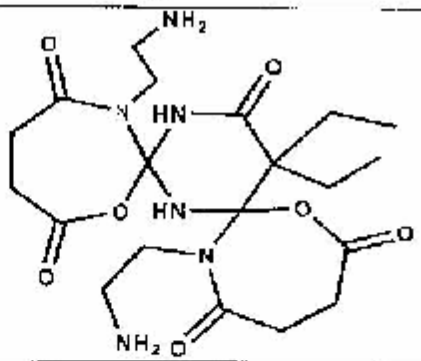
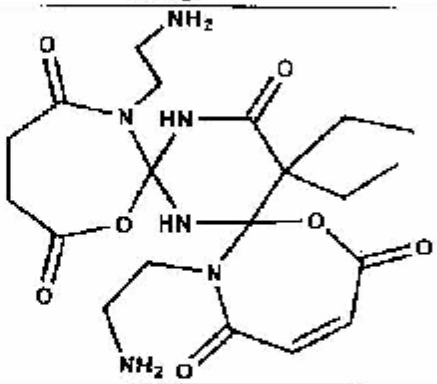
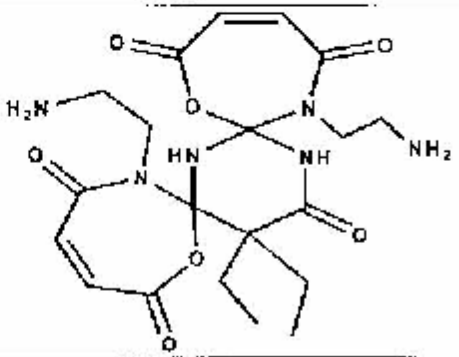
No.	Name of compounds	Structure
1	12-(2-Amino-ethyl)-3,3-diethyl-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-2,4,8,11-tetraone	
2	12-(2-Amino-ethyl)-3,3-diethyl-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-2,4,8,11-tetraone	
3	12-[2-(5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidi - ylidenecamino)-ethyl]-3,3-diethyl-7-oxa-1,5,12 triaza-spiro[5.6]dodecane-2,4,8,11-tetraone	
4	12-[2-(5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidi - ylidenecamino)-ethyl]-3,3-diethyl-7-oxa-1,5,12 triaza-spiro[5.6]dodec-9-ene-2,4,8,11-tetraone	
5	1-[2-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodec-12-yl)-ethyl]-3,3-diethyl-7 oxa-1,5,12-triaza-spiro[5.6]dodec-2,4,8,11-tetraoi	

6	1-[2-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-t spiro[5.6]dodec-9-ene-12-yl)-ethyl]-3,3-diethyl-7 1,5,12-triaza-spiro[5.6]dodec-9-ene-2,4,8,11-tetr	
7	1-[2-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-t spiro[5.6]dodec-12-yl)-ethyl]-3,3-diethyl-7-oxa-1 triaza-spiro[5.6]dodec-9-ene-2,4,8,11-tetraon	
8	6,15-Bis-(2-amino-ethyl)-18,18-diethyl-1,10-dioxa-6,8,15,16-tetraaza-dispiro[6.1.6.3] octadec-2,5,11,14,17-pentaone	
9	6,15-Bis-(2-amino-ethyl)-18,18-diethyl-1, dioxa-6,8,15,16-tetraaza-dispiro[6.1.6.3 octadec-3-ene-2,5,11,14,17-pentaone	
10	6,15-Bis-(2-amino-ethyl)-18,18-diethyl-1,10-dioxa-6,8,15,16-tetraaza-dispiro[6.1.6.3] octadec-3-ene-2,5,11,14,17-pentaone	

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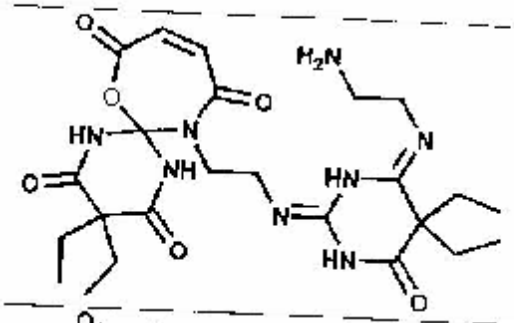
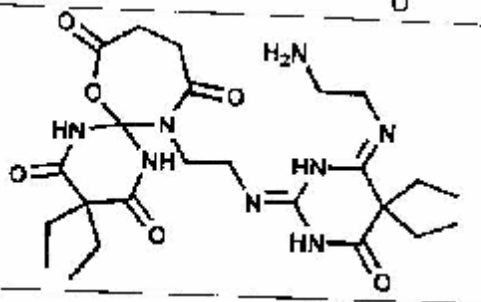
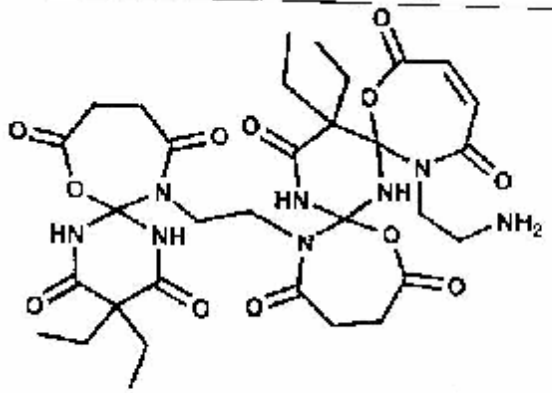
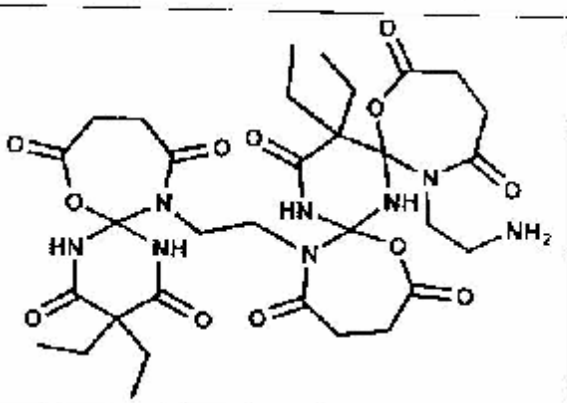
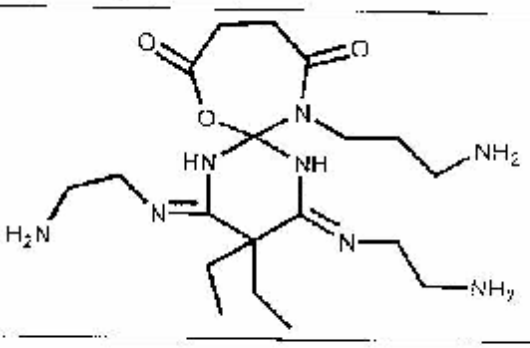
11	12-{2-[4-(2-Amino-ethylimino)-5,5-diethyl-6-oxo tetrahydro-pyrimidin-2-ylideneamino]-ethyl}-3,3 diethyl-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene 2,4,8,11-tetraone	
12	12-{2-[4-(2-Amino-ethylimino)-5,5-diethyl-6-oxo tetrahydro-pyrimidin-2-ylideneamino]-ethyl}-3,3 diethyl-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-2,4,8,11-tetraone	
13	6-(2-Amino-ethyl)-15-[2-(3,3-diethyl-2,4,8,11-tetr - oxa-1,5,12-triaza-spiro[5.6]dodec-12-yl)-ethyl] -diethyl-1,10-dioxa-6,8,15,16-tetraaza-dispiro[6. octadec-3-ene-2,5,11,14,17-pentaone	
14	6-(2-Amino-ethyl)-15-[2-(3,3-diethyl-2,4,8,11-tetr oxa-1,5,12-triaza-spiro[5.6]dodec-12-yl)-ethyl] -diethyl-1,10-dioxa-6,8,15,16-tetraaza-dispiro[6 octadec-2,5,11,14,17-pentaone	
15	12-(2-Amino-ethyl)-2,4-bis-(2-amino-ethylimino) - 3,3-diethyl-7-oxa-1,5,12-triaza-spiro[5.6] dodecane-8,11- dione	

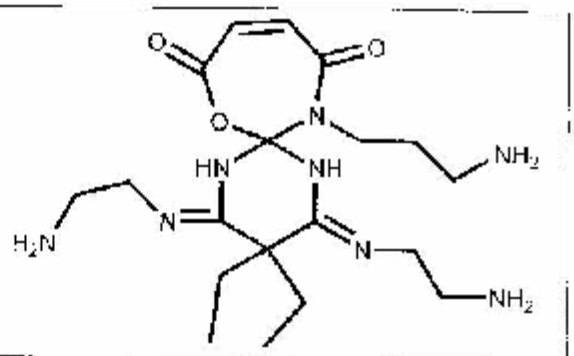
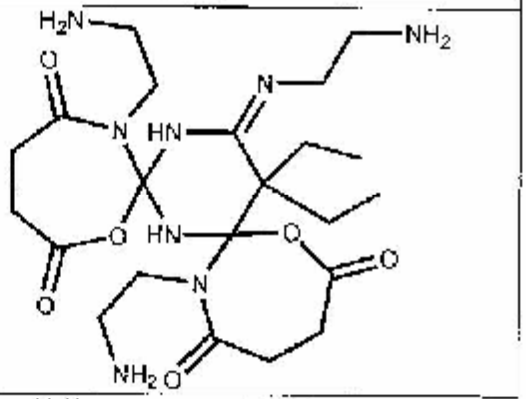
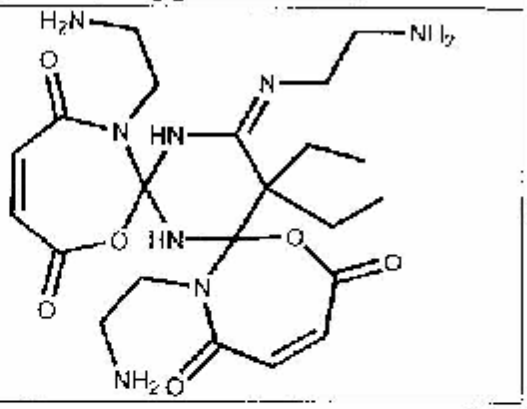
16	12-(2-Amino-ethyl)-2,4-bis-(2-amino-ethylimino) - 3,3-diethyl-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-8,11-dione	
17	6,15-Bis-(2-amino-ethyl)-17-(2-amino-ethylimino 18,18- diethyl-1,10-dioxa-6,8,15,16-tetranza-dispi [6.1.6.3] octadecane-2,5,11,14-tetraone	
18	6,15-Bis-(2-amino-ethyl)-17-(2-amino-ethylimino 18,18-diethyl-1,10-dioxa-6,8,15,16-tetraaza-dispi [6.1.6.3]octadeca-3,12-diene-2,5,11,14-tetraone	

Table (1) : Melting point, percentage yield, molecular formula and elemental analysis of 2-(2-Amino-ethylimino)-5,5-diethyl-dihydro-pyrimidine-4,6-dione - Schiff-bases

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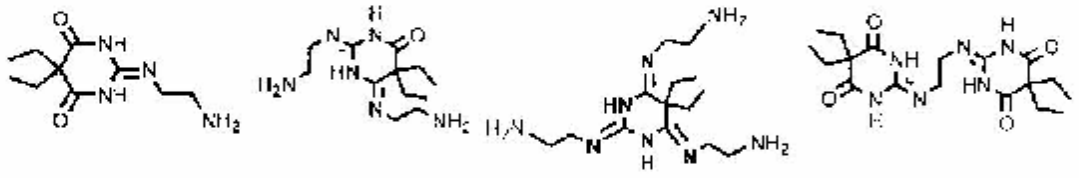
Table (2): The major IR absorptions (cm^{-1}) of 2-(2-Amino-ethylimino)-5,5-diethyl-dihydro-pyrimidine-4,6-dione -Schiff-bases



A B C D

Comp.	C-H str. Aromatic	C-H str. Alkane	C=O str	C=N Imine	C=C str. Aromatic	C-H bend Alkane
A	3060	2870	1675	1615	1570,1530	1470,1380
B	3075	2840	1680	1620	1580,1520	1480,1430
C	3050	2860	1670	1625	1590,1550	1460,1410
D	3035	2850	1690	1610	1585,1540	1480,1430

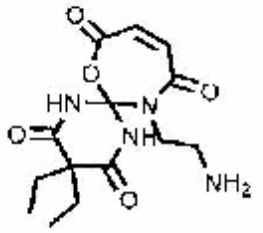
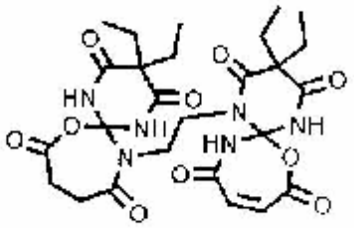
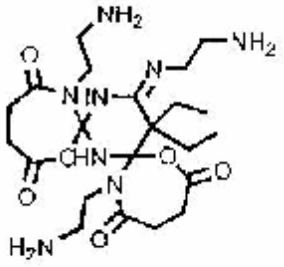
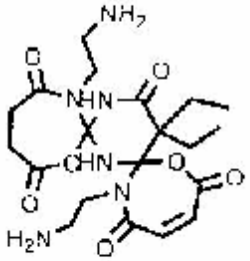
Table (3): The UV-Visible absorption maxima λ/nm of 2-(2-Amino-ethylimino)-5,5-diethyl-dihydro-pyrimidine-4,6-dione -Schiff-bases

compound	UV-Visible absorption maxima λ/nm
A	375,310,256.225,220
B	380.300,265,225
C	375,305,270,235,220
D	370,300,280,240,225

Table(4): Melting point ,percentage yield, molecular formula and elemental analysis of 1-[2-(3,3-Diethyl-2,4,8,11-tetraoxo-7-1,5,12-triaza-spiro[5.6]dodec-12-yl)-ethyl-3,3-diethyl-7-oxa-1,5,12-triaza-Spiro[5.6] dodecane -2,4,8,11-tetraone, compounds:-

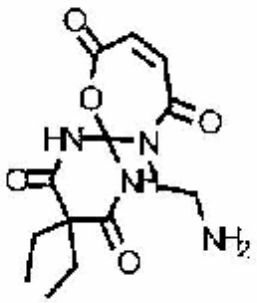
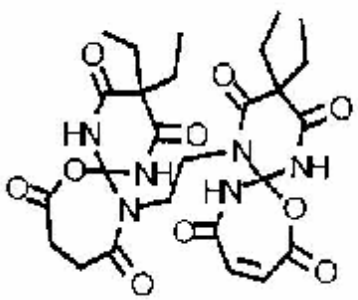
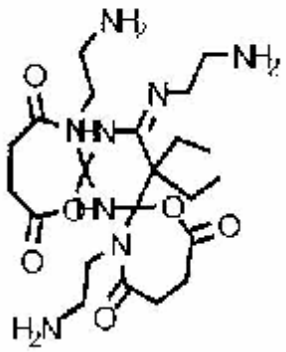
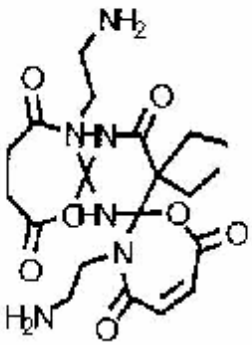
Comp	m.p/C°	Yield%	M.F	Calc. C	Calc. H	Calc. N	Found C	Found H	Found N
1	156-158	66	$C_{14}H_{22}N_4O_5$	51.52	6.79	17.17	51.43	6.84	17.05
2	177-179	60	$C_{14}H_{20}N_4O_5$	51.84	6.22	17.27	51.77	6.31	17.19
3	149-147	72	$C_{22}H_{32}N_6O_7$	53.65	6.55	17.06	53.69	6.68	17.00
4	157-155	86	$C_{22}H_{32}N_6O_7$	53.87	6.16	17.13	53.72	6.21	17.03
5	210-212	70	$C_{26}H_{36}N_6O_{10}$	52.70	6.12	14.18	52.64	6.23	14.12
6	221-223	59	$C_{26}H_{32}N_6O_{10}$	53.06	5.48	14.28	53.00	5.53	14.16
7	168-170	75	$C_{26}H_{34}N_6O_{10}$	52.88	5.80	14.23	52.80	5.94	14.11
8	144-146	70	$C_{20}H_{32}N_6O_7$	51.27	6.88	17.94	51.20	7.00	17.79
9	157-159	77	$C_{20}H_{28}N_6O_7$	51.72	6.08	18.09	51.11	6.13	18.02
10	163-165	69	$C_{20}H_{30}N_6O_7$	51.49	6.48	18.02	51.38	6.56	17.89
11	200-202	57	$C_{24}H_{36}N_8O_6$	54.12	6.81	21.04	54.05	7.00	20.96
12	211-213	68	$C_{24}H_{38}N_8O_6$	53.92	7.16	20.96	53.86	7.24	20.89
13	190-192	78	$C_{32}H_{44}N_8O_{12}$	52.45	6.05	15.29	52.40	6.15	15.18
14	189-191	80	$C_{32}H_{46}N_8O_{12}$	52.31	6.31	15.25	52.25	6.44	15.10
15	153-155	82	$C_{18}H_{34}N_8O_3$	52.66	8.35	27.30	52.57	8.42	27.21
16	160-162	74	$C_{18}H_{32}N_8O_3$	52.92	7.90	27.43	52.90	8.06	27.36
17	228-230	60	$C_{22}H_{38}N_8O_6$	51.75	7.50	21.95	51.67	7.58	21.87
18	179-181	68	$C_{22}H_{34}N_8O_6$	52.16	6.77	22.12	52.12	6.84	22.00

Table(5):The major IR absorption (cm^{-1}) of 1-[2-(3,3-Diethyl-2,4,8,11-tetraoxo-7-1,5,12-triaza-spiro[5.6]dodec-12-yl)-ethyl-3,3-diethyl-7-oxa-1,5,12-triaza-Spiro[5.6] dodecane -2,4,8,11-tetraone compounds:-

Comp	C-H str. Benzylic	C-H str. Aromatic	C=O str. Lacton.lac tam	C=C str. Olefin	C=C str. Aromatic	C-N str.	C-O str. Lacton	C-H bend. Aromatic
1	3205	3085	1680	-	1590,1550	1440	1320	1000,770
2	3210	3050	1685	1620	1580,1530	1440	1335	1045,870
3	3200	3080	1670	-	1590,1545	1445	1320	1025,860
4	3220	3080	1675	1610	1580,1550	1450	1320	1030,790
5	3230	3075	1670	-	1570,1535	1435	1315	1020,890
6	3210	3075	1670	1615	1580,1535	1430	1320	1010,860
7	3190	3060	1665	1625	1590,1540	1430	1340	1030,875
8	3195	3060	1665		1575,1535	1445	1320	1030,880
9	3220	3035	1670	1610	1590,1530	1430	1325	1030, 860
10	3200	3080	1660	1620	1585,1550	1445	1330	1040,790
11	3185	3070	1675	1610	1570,1540	1430	1325	1010,850
12	3180	3080	1680	-	1575,1550	1430	1330	1025,870
13	3200	3060	1685	1630	1590,1530	1450	1335	1055,860
14	3200	3090	1680	-	1580,1530	1430	1320	1080,890
15	3200	3065	1670	-	1590,1540	1445	1320	1020,870
16	3210	3070	1675	1610	1570,1530	1440	1310	1040,860
17	3180	3030	1665	-	1590,1550	1445	1325	1060,800
18	3200	3065	1680	1620	1570,1540	1440	1330	1020,870

Table(6) : ^{1}H.N.M.R Spectrophotometer of 1-[2-(3,3-Diethyl-2,4,8,11-tetraoxo-7-1,5,12-triaza-spiro[5.6]dodec-12-yl)-ethyl-3,3-diethyl-7-oxa-1,5,12-triaza-Spirn[5.6] dodecane -2,4,8,11-tetraone compounds:

* Chemical shift= δ

** By using DMSO d_6 as solvent

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Comp.	CH_3	CH_3-CH_2	-CH_2-CH_2-	HC=CH	-CH_2-CH_2-NH_2
1	1.1	1.92	2.4, 2.35	-	2.8, 2.6, 1.95
4	1.1	1.93	-	6.2 , 6.95	1.5 , 2.8
7	1.1	1.91	2.4 , 2.38	6.15 , 6.85	3.35 , 3.30
12	1.0	1.90	2.41 , 2.37	-	1.52 , 2.85 , 1.90
17	1.1	1.2	2.4, 2.30	-	3.3 ,2.8 , 1.5 , 2.5 ,1.90

Table(7): Uv-spectral data of 1-[2-(3,3-Diethyl-2,4,8,11-tetraoxo-7-1,5,12-triaza-spiro[5.6]dodec-12-yl)-ethyl-3,3-diethyl-7-oxa-1,5,12-triaza-Spiro[5.6] dodecane -2,4,8,11-tetraone compounds:

Compound	UV-Visible absorption maxima λ/nm
1	354,305,266,240,222
2	358,310,277,236,224
3	354,300,285,230,225
4	383,305,271,236,228
5	360,300,251,231,228
6	356,309,271,230,226
7	375,311,274,233,220
8	380,300,275,230,223
9	370,306,254,236,224
10	385,311,274,233,228
11	360,300,251,231,227
12	377,316,255,231,220
13	384,302,285,235,221
14	279,309,269,235,224
15	382,308,267,235,222
16	350,505,285,240,225
17	335,306,258,239,220
18	369,302,246,235,228

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الخلاصة

تم تحضير قواعد شيف 2 ,(2-أمينو -ثيل أمينو) 5,5 ثنائي ثيل-ثنائي هيدرو-بيرميدين-4,6-دايون و2 [2 (5,5- ثنائي ثيل-4,6-دايوكسو-بيرميدين- 2 يلدين أمينو)-ثيل امينو] ثنائي ثيل-ثنائي هيدرو -بيرميدين-4,6-دايون من تكثيف ثنائي

امينو ثيين مع مول واحد ومولين من 5,5-ثنائي ثيل بيرميدين-2,4,6-ثرايون. فوعلت قواعد شيف هذه مع مول واحد من الهيدريدات كل من المالييك والسكسنيك وتم الحصول على نظام حلقي غير متجانس (سباعي الحلقة) وعند مفاعلة قواعد شيف مع مولين من الانهيدريدات نفة الذكر أعطى نظام حلقي غير متجانس (بحلقتين سباعيتين) . وقد شخصت المركبات المحضرة بتعيين درجات انصهارها تحليل العناصر، اطياف الأشعة فوق البنفسجية واطياف الأشعة تحت الحمراء

وأطياف الرنين النووي المغناطيسي لبعض المركبات. وقد أسفرت نتائج الفحوص بالطرق المختلفة في إثبات التصنيع التركيبية للمركبات المحضرة

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