National Journal of Chemistry, 2006, Volume 23, 405-417 المجلة القطرية للكيمياء-2006-المجلد الثالث والعشرون

Synthesis and Characterization of Oxazepine and Oxazepane from reaction of (5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidein)-urea and 1,3-Bis(5,5-diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidein)- urea with maleic and Succinic anhydride.

Waleed.F. Al-Hity
Chem.Dept.College of Education for women, University of Al-Anbar
Mohammed A.Al-Hadithi
Chem.Dept.College of Science, University of Al-Anbar
Email : drwaledalhiti @yahoo.com

(NJC)

(Received on 23/1 /2006) (Accepted for publication on 19/8/2006)

Abstract

(5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidein)-urea and 1,3-Bis(5,5-diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidein)- urea were prepared by condensation of urea with one equivalent and two equivalent of 5,5-Diethyl-pyrimidine-2,4,6-trione. These Schiff-bases reacted with one equivalent of maleic and succinic anhydride in absolute ethanol to give 7-membered heterocyclic ring system of 3,3-Diethyl-2,4,8-tetraoxo-7-oxa-1,5,12-triaza-spiro [5.6] dodec-9-ene-12-carboxylic acid amide and were reacted with two equivalent of maleic and succinic anhydride in same solvent give doubled (7-membered) heterocyclic ring system of 12-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-12-carbonoyl)-3-ethyl-3-methyl-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-2,4,8,11-tetraone.

The final products were identified by there m.ps, elemental analyses,IR, ^{1}HNMR and UV-Visible spectra.

الخلاصة

تم تحضير قواعد شيف (5,5- ثنائي اثيل-4,6-دايوكسو-رباعي هيدرو- بيرميدين -2-يل أدين - ثايويوريا)- يوريا و3,1- بس(5,5- ثنائي اثيل-4,6-دايوكسو-رباعي هيدرو- بيرامادين -2-يل أدين)- يوريا من تكثيف اليوريا مع مول واحد ومولين من 5,5-ثنائي اثيل-بيرميدين-2,4,6-ترايون . فوعلت قواعد شيف هذه مع مول واحد من انهيدريدات كل من الماليك والسكسنيك وتم الحصول على نظام حلقي غير متجانس (سباعي الحلقة) وعند مفاعلة قواعد شيف مع مولين من الانهيدريدات أنفة الذكر أعطى نظام حلقي غير متجانس (بحلقتين سباعيتين) . وقد شخصت المركبات المحضرة بتعيين درجات انصهارها، تحليل العناصر، أطياف الأشعة فوق البنفسجية ،أطياف الأشعة تحت الحمراء وأطياف الرنين النووي المغناطيسي لبعض منها. وقد أسهمت نتائج التشخيص بالطرق المختلفة في إثبات الصيغ التركيبية للمركبات المحضرة .

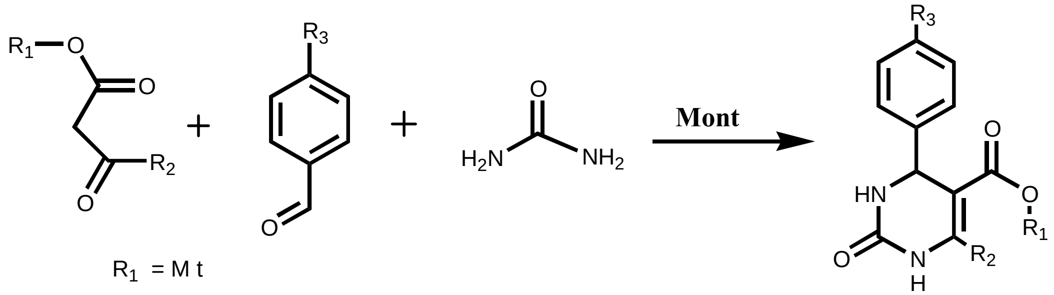
Introduction

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), 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 ,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 (9,10). 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 yields (14). 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] oxazpine-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-a,7-dione were synthesized from reaction of Cyclo anhydride with 1,3-Bis(2-hydroxy-benzylidene)-urea (Schiff-bases) (13).

However , Biginelli s[1-3] initial one-pot method of refluxing a β-keto ester , aryl aldehyde and urea with a catalytic amount of acid frequently afforded low (20-60%) yields of the desired target molecules [7]. While optimizing the reaction conditions of the Biginelli reaction, we found that treatment of β-keto ester , aryl aldehyde and urea with KSF montmorillonite in methanol afforded DHPMs in good to excellent



R1 = M t

R2 = E t

R3 = OH

2-(2-Hydroxy-phenyl)-4,7-dioxo-4,7-dihydro-[1,3] oxazpine-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-a,7-dione were synthesis from reaction of cyclo anhydride with 1,3-Bis(2-hydroxy-benzylidene)-urea (Schiff-bases) (15).

Experimental

Melting points were recorded on a 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.

Preparation of(5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidene-2-ylidene)- urea and 1,3-Bis-(5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidene-2-ylidene)- urea (Schiff-base):-

(5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidene-2-ylidene)- urea and 1,3-Bis- (5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidene-2-ylidene)- urea were prepared by condensation one equivalent and two equivalent of urea with one equivalent of 5,5-Diethyl-pyrimidine-2,4,6-trione.

To a solution of (0.05 and 0.1 mole) of urea in 30 ml of ethanol (absolute) was added o.o5 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 3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane -12-carboxylic acid amide:-

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 (5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidene-2-ylidene)- urea and 0.01mole maleic anhydride in 20 ml of ethanol absolute.

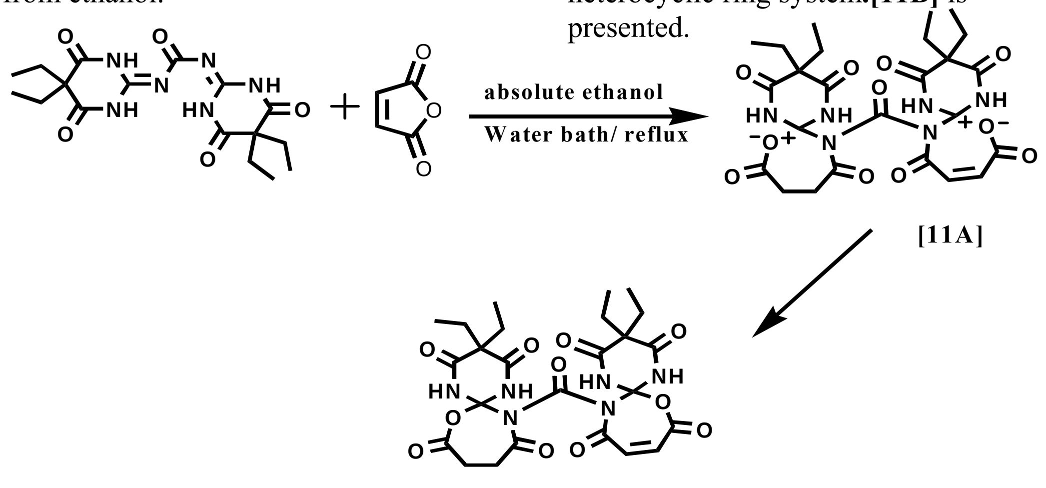
The reaction mixture was refluxed in water bath at 78C° for 3hr, the solvent was then removed and the resulting solid was recrystallized from anhydrous THF.

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

Siscussion

It is known that Schiff bases react smoothly with acid chlorides and anhydrides to give the corresponding addition products.(15-18)

In this paper, the reaction of the Maleic and Succinic anhydrides with (5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidein)- urea and 1,3-Bis(5,5-diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidein)- urea 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 band 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 3,3-Diethyl-2,4,8-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-12-carboxylic acid amide and 12-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-12-carbonyl)-3-ethyl-3-methyl-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-2,4,8,11-tetraone.

The new absorption bands of the (C=O) group in the IR spectra of the addition products **[11B]** appear at (1660-1700)cm^{-1}, this attributed to the fact that the structures of the addition products are combination of the lacton-lactam structure'**(19,20)** .

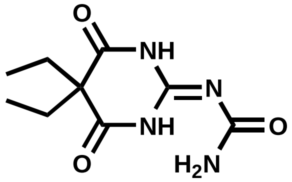
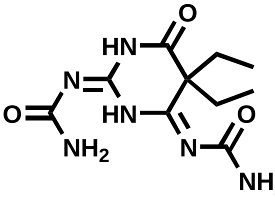
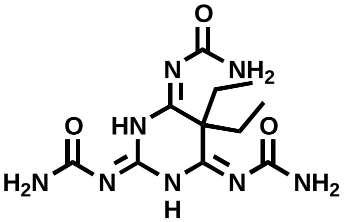
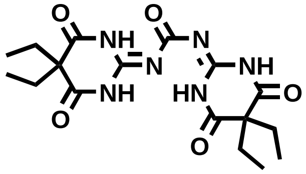
The UV spectra of 3,3-Diethyl-2,4,8-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-12-carboxylic acid amide and 12-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-12-carbonyl)-3-ethyl-3-methyl-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-2,4,8,11-tetraone. show absorption maxima at (240-310)nm , and at (310-445)nm due to charge transfer of the aryl group and the cyclic 7-membered structure **[11B]**.

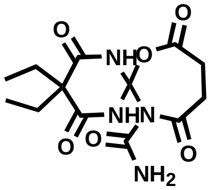
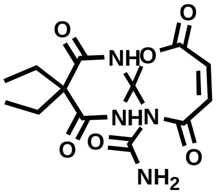
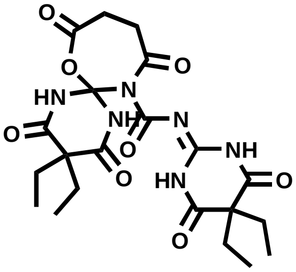
3,3-Diethyl-2,4,8-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-12-carboxylic acid amide and 12-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-12-carbonyl)-3-ethyl-3-methyl-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-2,4,8,11-tetraone compounds are identified by their m.ps,elemental analysis (table 4),IR spectra (table 5) and UV spectra (table 6). 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.

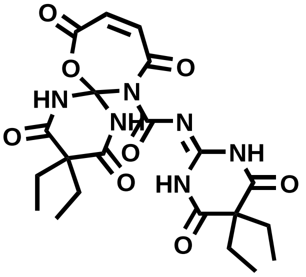
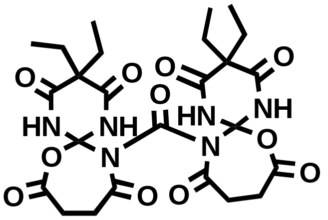
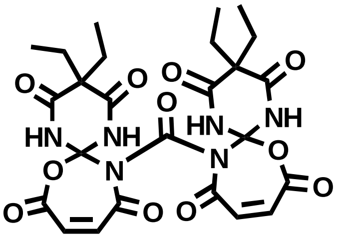
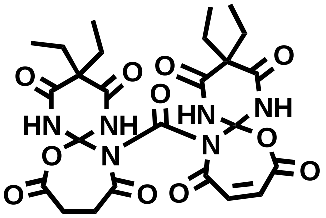
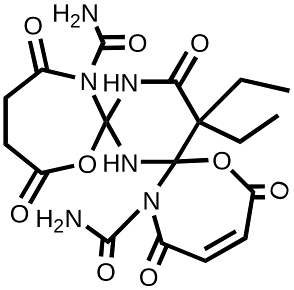
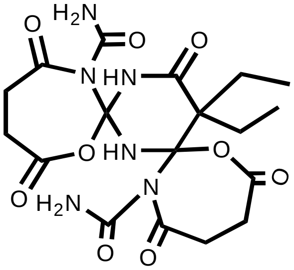
The reaction of maleic and succinic anhydride with various Schiff bases is a sort of cycloaddition reaction.Cycloaddition is a ring formation that results from the addition of bonds to either δ or π with formation of new δ bonds. This class of reactions and its reverse encompasses a large number of individual 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)**

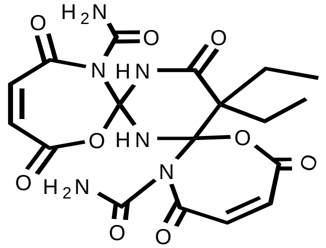
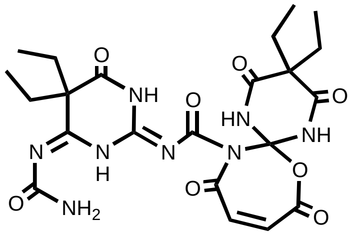
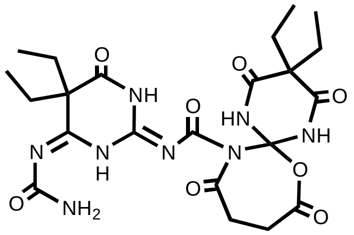
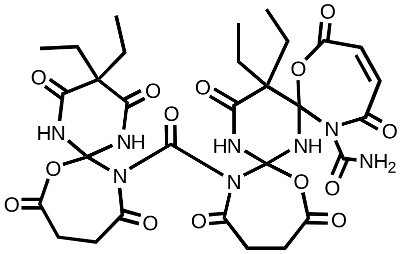
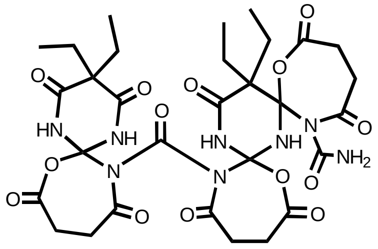
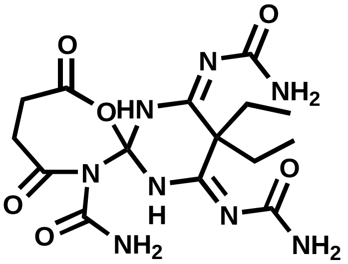
The spectrum of ^{1}H-NMR of some prepared compounds (1,2,3,4,7,8,15) , the absorbing bands at δ 0.82-7.89 (2H,t, CH_3) and δ 1.73-1.78 (2H,q , CH_2-) they belong to ethyl group . While at δ 7.80-7.96 (1H,S,NH) they belong to (C-NH-C) group. The compounds (1,3,7,8,15) show doublet bands at δ 2.43-2.45 (2H,S,CH_2) which belong to (-CH_2-CH_2-) group. Compounds (2,4,7,8) also show absorbing bands at δ 6.30-6.35 (1H,S,=CH) which represent of (-CH=CH-) group. Whereas compounds(1,2,8,15) show bands at δ 1.90-1.95 (2H,S, NH_2) which represents protons of NH_2 group.

This proves the validity of the suggested structures of the prepared compounds.

No.	Schiff-Bases Name	Structure
A	(5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidene)-urea	
B	(5,5-Diethyl-6-oxo-2-thiocarbamoylimino-tetrahydro-pyrimidin-4-ylidene)-urea	
C	(5,5-Diethyl-4,6-bis-thiocarbamoylimino-tetrahydro-pyrimidin-2-ylidene)-urea	
D	1,3-Bis-(5,5-diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidene)-urea	

No.	Name of compounds	Structure
1	3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-12-carboxylic acid amide	
2	3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza- spiro[5.6]dodec-9-ene-12-carboxylic acid amide	
3	3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-12-carboxylicacid (5,5-diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidene)- amide	

4	**3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-12-carboxylicacid(5,5-diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidene) -am ide**	
5	**12-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-12-carbonyl)-3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane**	
6	**12-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-12-carbonyl)-3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene**	
7	**12-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-12-carbonyl)-3,3-diethyl-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-2,4,8,11-tetraone**	
8	**18,18-Diethyl-2,5,11,14,17-pentaoxo-1,10-dioxa-6,8,15,16-tetraaza-dispiro [6.1.6.3] octadec-3-ene-6,15-dicarboxylic acid diamide**	
9	**18,18-Diethyl-2,5,11,14,17-pentaoxo-1,10-dioxa-6,8,15,16-tetraaza-dispiro [6.1.6.3]octadecane-6,15-dicarboxylic acid diamide**	

10	**18,18-Diethyl-2,5,11,14,17-pentaoxo-1,10-dioxa-6,8,15,16-tetraaza-dispiro [6.1.6.3]octadeca-3,12-diene-6,15-dicarboxylic acid diamide**	
11	**3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-12-carboxylic acid (4-carbamoylimino-5,5-diethyl-6-oxo-tetrahydro-pyrimidin-2-ylidene)-amide**	
12	**3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-12-carboxylic acid (4-carbamoylimino-5,5-diethyl-6-oxo-tetrahydro-pyrimidin-2-ylidene)-amide**	
13	**15-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-12-carbonyl)-18,18-diethyl-2,5,11,14,17-pentaoxo-1,10-dioxa-6,8,15,16-tetraaza-dispiro[6.1.6.3]octadec-3-ene-6-carboxylicacid amide**	
14	**15-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro [5.6]dodecane-12-carbonyl)-18,18-diethyl-2,5,11,14,17-pentaoxo-1,10-dioxa-6,8,15,16-tetraaza-dispiro[6.1.6.3]octadecane-6-carboxylic acid amide**	
15	**2,4-Bis-carbamoylimino-3,3-diethyl-8,11-dioxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-12-carboxylic acid amide**	

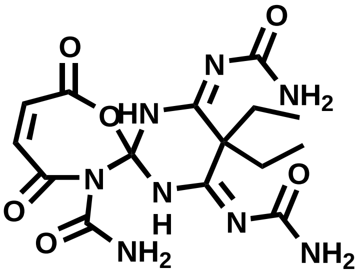
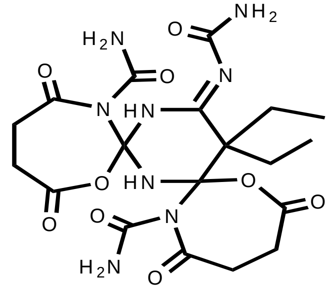
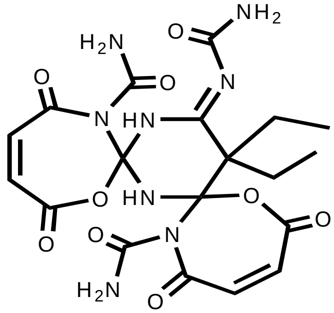
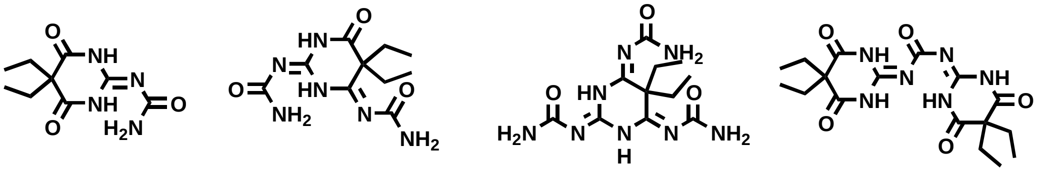
16	**2,4-Bis-carbamoylimino-3,3-diethyl-8,11-dioxo-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-12-carboxylic acid amide**	
17	**17-Carbamoylimino-18,18-diethyl-2,5,11,14-tetraoxo- 1,10-dioxa-6,8,15,16-tetraaza-dispiro[6.1.6.3] octadecane-6,15-dicarboxylic acid diamide**	
18	**17-Carbamoylimino-18,18-diethyl-2,5,11,14-tetraoxo-1,10-dioxa-6,8,15,16-tetraaza-dispiro[6.1.6.3] octadeca-3,12-diene-6,15-dicarboxylic acid diamide**	

Table (1) : Melting point, percentage yield, molecular formula and element analysis of (5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidein)- urea - Schiff-bases

Comp.	M.P/C°	Yield%	M.F	Calc.			Found		
				C	H	N	C	H	N
A	225-227	67	$C_9H_{14}N_4O_3$	47.78	6.24	24.77	47.73	6.33	24.68
B	219-221	55	$C_{10}H_{16}N_6O_3$	44.77	6.01	31.33	44.65	6.19	31.24
C	179-181	79	$C_{11}H_{18}N_8O_3$	42.58	5.85	36.11	42.46	5.93	36.05
D	173-175	63	$C_{17}H_{24}N_6O_5$	52.03	6.16	21.42	52.00	6.24	21.37

Table (2): The major IR absorptions (cm^{-1}) of (5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidein)- urea -Schiff-bases



Comp.	C-H str. Aromatic	C-H str. Alkane	C=O str amide	C=N Imine	C=C str. Aromatic	C-H bend Alkane
A	3035	2860	1680	1610	1570,1540	1470,1380
B	3060	2840	1670	1610	1570,1530	1475,1400
C	3050	2870	1690	1625	1580,1550	1470,1410
D	3045	2860	1675	1615	1585,1540	1480,1420

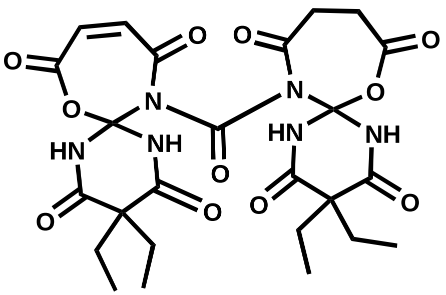
Table (3): The UV-Visible absorption maxima λ/nm of (5,5-Diethyl-4,6-dioxo-tetra hydro- pyrimidin-2-ylidein)- urea -Schiff-bases

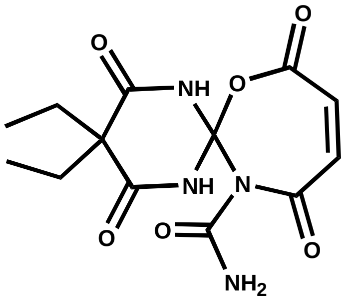
compound	UV-Visible absorption maxima λ/nm
A	370.310,275,226
B	380,305, 270, 222
C	300,268.229.223
D	370,300, 240,228

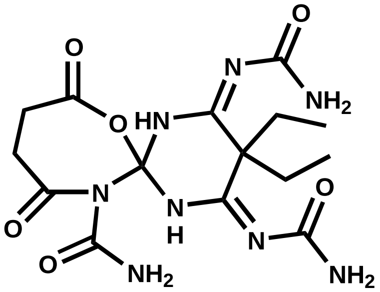
Table(4): Melting point ,percentage yield, molecular formula and elemental analysis of 3,3-Diethyl-2,4,8,11- tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6] dodecane - 12-carbothioic acid amide compounds:-

Comp	m.p/C^{o}	Yield%	M.F	Calc.			Found		
				C	H	N	C	H	N
1	164-166	70	$C_{13}H_{18}N_4O_6$	47.85	5.56	17.17	47.77	5.67	17.10
2	171-173	66	$C_{13}H_{16}N_4O_6$	48.15	4.97	17.28	48.07	5.08	17.20
3	202-204	75	$C_{21}H_{28}N_6O_8$	51.21	5.73	17.06	51.10	5.89	17.00
4	179-177	70	$C_{21}H_{26}N_6O_8$	51.43	5.34	17.13	51.32	5.42	17.05
5	189-191	68	$C_{25}H_{32}N_6O_{11}$	50.67	5.44	14.18	50.61	5.53	14.11
6	155-157	80	$C_{25}H_{28}N_6O_{11}$	51.02	4.80	14.28	50.94	4.93	14.16
7	146-148	72	$C_{25}H_{30}N_6O_{11}$	50.85	5.12	14.23	50.78	5.23	14.14
8	137-139	62	$C_{18}H_{22}N_6O_9$	46.35	4.75	18.02	46.30	4.83	17.95
9	176-178	71	$C_{18}H_{24}N_6O_9$	46.15	5.16	17.94	46.04	5.24	17.85
10	231-233	66	$C_{18}H_{20}N_6O_9$	46.55	4.34	18.10	46.45	4.46	18.02
11	211-213	83	$C_{22}H_{28}N_8O_8$	49.62	5.30	21.04	49.57	5.41	21.00
12	178-180	69	$C_{22}H_{30}N_8O_8$	49.43	5.66	20.96	49.32	5.78	20.85
13	135-137	58	$C_{30}H_{36}N_8O_{14}$	49.18	4.95	15.29	49.10	5.07	15.08
14	200-202	77	$C_{30}H_{38}N_8O_{14}$	49.05	5.21	15.25	49.00	5.36	15.19
15	183-185	76	$C_{15}H_{22}N_8O_6$	43.90	5.40	27.30	43.81	5.53	27.21
16	164-166	65	$C_{15}H_{20}N_8O_6$	44.12	4.94	27.44	44.06	5.08	27.35
17	182-184	81	$C_{19}H_{26}N_8O_9$	44.71	5.13	21.95	44.67	5.18	21.88
18	163-161	59	$C_{19}H_{22}N_8O_9$	45.06	4.38	22.13	45.00	4.51	22.09

Table(5):The major IR absorption (cm^{-1})of 3,3-Diethyl-2,4,8,11- tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6] dodecane - 12- carbothioic acid amide compounds:-

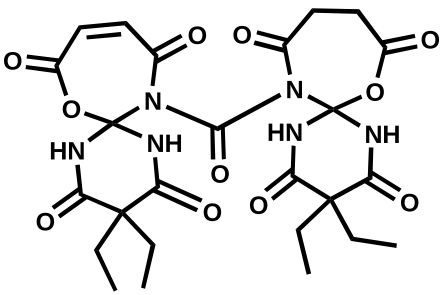


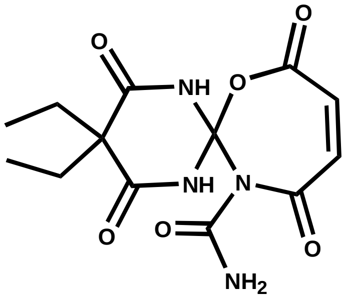


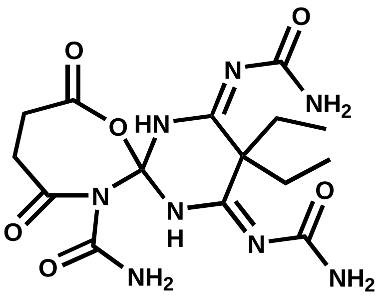


Comp	C-H str. Benzylic	C-H str. Aromatic	C=O str. Lacton,lactam	C=C str. Olefin	C=C str. Aromatic	C-N str.	C-O str. Lacton	C=S str.	C-H bend. Aromatic
1	3200	3070	1675	-	1590,1540	1445	1325	1240	1000,770
2	3210	3050	1670	1610	1570,1540	1440	1330	1235	1030,875
3	3200	3030	1680	-	1570,1530	1430	1320	1250	1010,850
4	3180	3080	1685	1625	1590,1550	1450	1310	1245	1025,870
5	3230	3065	1680	-	1570,1540	1440	1325	1240	1055,860
6	3210	3075	1670	1620	1580,1530	1440	1330	1235	1010,860
7	3190	3070	1665	1615	1575,1550	1430	1340	1250	1020,870
8	3200	3080	1665	1615	1590,1530	1445	1320	1245	1040,860
9	3220	3090	1670	-	1570,1535	1445	1325	1240	1060,800
10	3200	3060	1660	1625	1580,1535	1435	1330	1255	1020,870
11	3180	3065	1670	1615	1590,1540	1430	1320	1240	1080,890
12	3195	3080	1675	-	1575,1535	1445	1335	1230	1040,790
13	3200	3055	1665	1620	1590,1530	1430	1320	1235	1030,880
14	3210	3070	1680	-	1585,1550	1430	1320	1240	1030, 860
15	3185	3060	1685	-	1590,1545	1450	1315	1220	1045,870
16	3220	3075	1670	1615	1580,1530	1430	1320	1230	1025,860
17	3200	3080	1675	-	1580,1550	1445	1335	1245	1030,790
18	3205	3085	1680	1625	1590,1550	1440	1320	1240	1020,890

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







* Chemical shift= δ

** By using DMSO-d_6 as solvent

Comp.	$C\underline{H}_3$	CH_3-$C\underline{H}_2$	-$C\underline{H}_2$-$C\underline{H}_2$-	$\underline{H}$C=C$\underline{H}$	$N\underline{H}_2$	C-N$\underline{H}$-C
1	0.89	1.74	2.43,2.43	-	1.95	7.80
2	0.80	1.75	-	6.35,6.35	1.90	7.85
3	0.85	1.73	2.45,2.45	-	-	7.80
4	0.82	1.76	-	6.32,6.32	-	7.86
7	0.87	1.76	2.44,2.44	6.30,6.30	-	7.96
8	0.87	1.78	2.45,2.45	6.33,6.33	1.92	7.88
15	0.87	1.78	2.45,2.45	-	1.92	7.87

Table(7): Uv-spectra of 3,3-Diethyl-2,4,8,11- tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6] dodecane - 12-carbothioic acid amide compounds:-

Compound	UV-Visible absorption maxima λ/nm
1	380,302,285,235,221
2	350,306,254,236,224
3	355,300,251,231,227
4	347,302,249,233,229
5	335,301,256,239,223
6	354,309,271,230,225
7	356,505,285,240,228
8	352,300,271,236,222
9	358,310,277,236,224
10	354,311,274,233,228
11	383,320,266,230,226
12	377,310,255,231,220
13	354,300,251,231,228
14	279,309,269,235,224
15	356,311,274,233,220
16	350,505,285,240,225
17	364,300,275,230,223
18	382,308,267,235,222

References

1 - N. M AL-Janaby., M. Sc. Thesis, College of Science for girls, University of Baghdad (2003).

2 - E. Jeney and T. Zsolnai , Abt.I,Org. 193 (4), 516 (1964) . Chem. Abstr.:62, 3106 (1965).

3 – A. Motagu , Philips Brit,590, 714 (1947). Chem. Abstr,; 42, 2987 (1948).

4 - Lee ,C., R., Glashan, J., M., Martindale " The complete Drug Reference" 32nd Ed.(1999).

5- K. F. Modi, N. Krishnakunar, and H. J. Mehta, ***J. Ind. Chem. Soc.***, 1977, **LIV**, 741.

6 – V. J. Ram, U., K. Singha , and P. Y. Juru, ***Eur. J. Med. Chem.***, 1990, **25**, 533.

7- M. Toshio , K. Tsutomu , and S. Osamu, (photochemical and thermal reactions of some heterocyclic containing C=N-O and N=C-O group), ***Pure and Appl. Chem.***, 1977, **49**, 287.

8- G. W. Chessman, and S.G. Greenberg , (Synthesis of 5,6-dihydro-4H- pyrrolo[1,2-α] [1,4] benzodiazepines), ***J.Heterocyclic Chem.***, 1979, **16**, 241.

9-. H.Schiff , ***Ann,*** 1864, **131**,118.

10- J..C..Clifford, A..R.. Olaf, and M..C. Malcolm , (spectral analysis of organic compounds),2nd ed., Longman, (1972)

11- T. Takashi, T. Takeo, and S. Yashizo , (Synthesis and cycloaddition reaction of di and tri- substituted 1,3- oxazepine) Heterocycles, 11, 331-6 (1978) .

12- T. Tyoji, I. Kuniyoshi, and T. Takashi, ***Chem. Pharm. Bull***., 1987, **35** (8), 74.

13- P. Biginelli, ***Gazz. Chim. Ital.***, 1983, **23**,360.

14- H.X. Lin, X. Zhang,and L.S. Cheng, ***Chin.Chem.Lett***., 1999, **10** (11), 915.

15-W. F. Al-hiti. and M.A.Al-hadithi, (Synthesis and Characterization of

Oxazepine Oxazepane from reaction of (Schiff-bases) with maleic and Succinicanhydride., ***J.of Al-Nahrain University***, 2005, **8** (2), 27.

16- T. Tyoji, I. Kuniyoshi, and T. Takashi, ***Chem. Pharm. Bull***., 1987, **35** (8), 74

17- F. A. Hussein, O. H.Abid (synthesis of some Barbiturates via Schiff bases). ***Iraqi Journal of Chemistry***, 2000, **26**, (1), 216.

18- R. Huisgen, ***Chem. Internt***., 1968, Edit. **7,** 321.

19- M.M. Robert, and W. J. Charless, (Organic chemistry A problms : An Approach,) W.A.Bengamine,Inc,p 526 (1975)

20- F. A. Hussein, (synthesis of some Barbiturates via Schiff bases). ***Iraqi Journal of Chemistry*** , 2000, **26**,(1), 216.