

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

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ABSTRACT

(5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidein)-thiourea and 1,3-Bis(5,5-diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidein)-thiourea were prepared by condensation of Thiourea 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, succinic and phthalic 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-carbothioic acid amide and were reacted with two equivalent of maleic and succinic anhydride in same solvent give 2 (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-carbothioyl)-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 their m.p.s, elemental analyses, IR, ¹HNMR and UV-Visible spectra.

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-dihydro-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).

N-acyl immonium ions have been the most commonly used dienes to effect [4+2] cycloaddition as 4 π 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⁽⁵⁾.

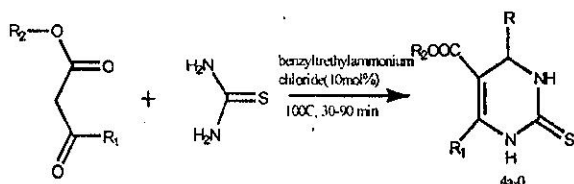
The reaction of N-Benzylidene 1,5-dimethyl-2-phenylpyrazolonamines (Schiff bases with Cyclopentane-1,1-dicarboxylic anhydride to give 2-aryl-3-(1,5-dimethyl-2-phenylpyrazolo)-1-(5) spirocyclopentyltetrahydro-1,3-oxazine-4,6-diones⁽⁶⁾.

A simple, efficient and cost-effective method has been developed for the synthesis of 3,4-dihydropyrimidin-

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2(1*H*) –ones by a one-pot three component cyclocondensation reaction of 1,3-dicarbonyl compound, aldehyde, and urea ,thiourea using benzyltriethylammonium chloride as the catalyst, under solvent-free conditions: the scope of this protocol is utilized for the synthesis of mitotic kinesin EG5 inhibitor monastrol.⁽⁷⁾



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]oxazpine-3-carbonyl]-2,3-dihydro-[1,3]oxazpine-a,7-dione were synthesis from reaction of Cyclo anhydride with 1,3-Bis(2-hydroxy-benzylidene)-urea (Schiff-bases)⁽⁸⁾.

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.

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

(5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidene-2-ylidene)-thiourea and 1,3-Bis-(5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidene-2-

ylidene)-thiourea were prepared by condensation one equivalent and tow equivalent of Thiourea with one equivalent of 5,5-Diethyl-pyrimidine-2,4,6-trione.

To a solution of 0.05 and 0.1 mole of Thiourea 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-carbothioic 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)-thiourea and 0.01mole maleic anhydride in 20 ml of Ethanol absolute.

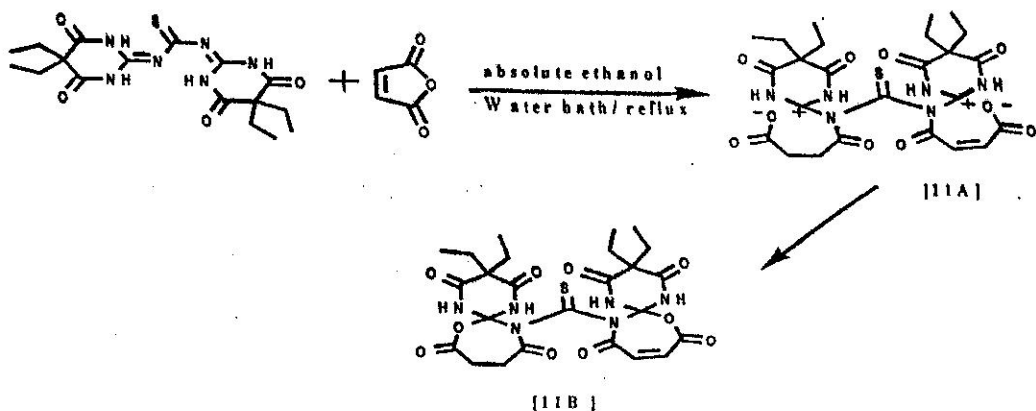
The reaction mixture was refluxed in water bath at 78C' 3hr, the solvent was then removed and the resulting solid was recrystallized from anhydrous THF.

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

DISCUSSTION:-

It is known that Schiff bases react smoothly with acid chlorides and anhydrides to give the corresponding addition products⁽⁹⁻¹²⁾

In this paper, the reaction of the Maleic and Succinic anhydrides with (5,5-Diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidein)-thiourea and 1,3-Bis(5,5-diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidein)-thiourea to gives the dipolar intermediate [11A] which collapses to the 7- membered heterocyclic ring system.[11B] is presented.



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 $(1800-1950)\text{cm}^{-1}$ 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-triazaspiro[5.6]dodec-9-ene-12-carbothioic acid amide and 12-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triazaspiro[5.6]dodecane-12-carbothioyl)-3-ethyl-3-methyl-7-oxa-1,5,12-triazaspiro[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 $(1670-1700)\text{cm}^{-1}$, this attributed to the fact that the structures of the addition products are combination of the lacton-lactam structure^(13,14).

The UV spectra 3,3-Diethyl-2,4,8-tetraoxo-7-oxa-1,5,12-triazaspiro[5.6]dodec-9-ene-12-carbothioic acid amide and 12-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triazaspiro[5.6]dodecane-12-carbothioyl)-3-ethyl-3-methyl-7-oxa-1,5,12-triazaspiro[5.6]dodec-9-ene-2,4,8,11-tetraone.

show absorption maxima at $(240-310)\text{nm}$, and at $(310-445)\text{nm}$ due to charge transfer of the aryl group and the cyclic 7-membered structure [11B]. 2-(2-Hydroxy-phenyl)-4,7-dioxo-4,7-dihydro-[1,3]oxazepine-3-carboxylic acid amide and 3,3-Diethyl-2,4,8-tetraoxo-7-oxa-1,5,12-triazaspiro[5.6]dodec-9-ene-12-carbothioic acid amide and 12-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triazaspiro[5.6]dodecane-12-carbothioyl)-3-ethyl-3-methyl-7-oxa-1,5,12-triazaspiro[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⁽¹⁵⁾ 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 ⁽¹⁶⁾.

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

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

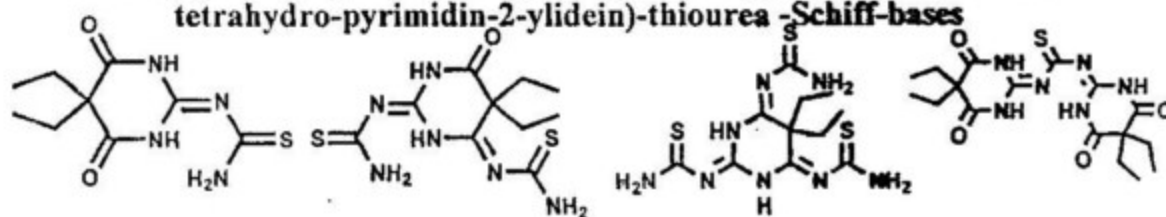
6	12-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-12-carbothiyl)-3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene	
7	12-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-12-carbothiyl)-3,3-diethyl-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-2,4,8,11-tetraone	
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-dicarbothioic 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-dicarbothioic 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-dicarbothioic acid diamide	
11	3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-12-carbothioic acid (5,5-diethyl-4-oxo-6-thiocarbamoylimino-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-carbothioic acid (5,5-diethyl-4-oxo-6-thiocarbamoylimino-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-carbothiyl)-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-carbothioic acid amide	
14	15-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-12-carbothiyl)-18,18-diethyl-2,5,11,14,17-pentaoxo-1,10-dioxa-6,8,15,16-tetraaza-dispiro[6.1.6.3]octadecane-6-carbothioic acid amide	

15	15-(3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-12-carbothioyl)-18,18-diethyl-2,5,11,14,17-pentaoxo-1,10-dioxo-6,8,15,16-tetraaza-dispiro[6.1.6.3]octadec-3-ene-6-carbothioic acid amide	
16	3,3-Diethyl-8,11-dioxo-2,4-bis-thiocarbamoylimino-7-oxa-1,5,12-triaza-spiro[5.6]dodec-9-ene-12-carbothioic acid amide	
17	18,18-Diethyl-2,5,11,14-tetraoxo-17-thiocarbamoylimino-1,10-dioxo-6,8,15,16-tetraaza-dispiro[6.1.6.3]octadecane-6,15-dicarbothioic acid diamide	
18	18,18-Diethyl-2,5,11,14-tetraoxo-17-thiocarbamoylimino-1,10-dioxo-6,8,15,16-tetraaza-dispiro[6.1.6.3]octadeca-3,12-diene-6,15-dicarbothioic acid diamide	

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

Comp	M.P/°C	Yield%	M.F	Calc.			Found		
				C	H	N	C	H	N
A	166-168	77	C ₉ H ₁₆ N ₄ O ₂ S	44.61	5.82	23.12	44.47	5.93	23.07
B	153-155	68	C ₁₀ H ₁₈ N ₄ O ₂ S	39.98	5.37	27.98	40.12	5.51	27.88
C	149-151	72	C ₁₁ H ₁₈ N ₆ S ₂	36.85	5.06	31.26	36.96	5.00	31.15
D	195-197	80	C ₁₇ H ₂₄ N ₄ O ₂ S	49.99	5.92	20.57	50.13	6.03	20.39

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



Comp.	C-H str. Aromatic	C-H str. Alkane	C=O str	C=N Imine	C=C str. Aromatic	C-H bend Alkane
A	3025	2850	1685	1620	1580,1520	1460,1350
B	3040	2860	1690	1610	1590,1540	1480,1410
C	3060	2880	1680	1615	1580,1540	1460,1420
D	3065	2870	1685	1620	1575,1550	1480,1410

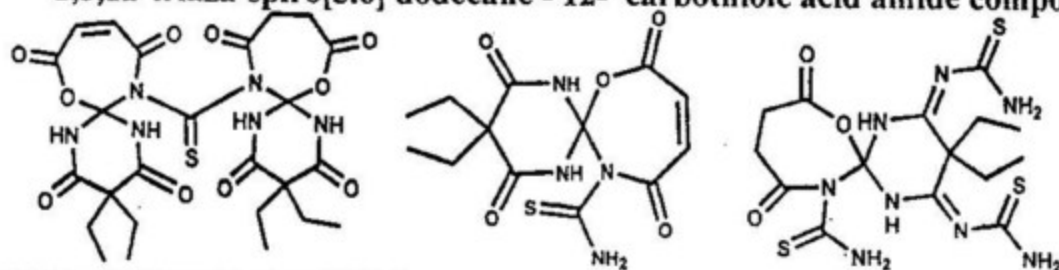
Table (3) : The UV-Visible absorption maxima λ_{nm} of (5,5-Diethyl-4,6-dioxo-tetra hydro- pyrimidin-2-ylidein)-thiourea -Schiff-bases

compound	UV-Visible absorption maxima λ_{nm}
A	380,300,266,225,220
B	370,310,275,226
C	385,305,270,231,220
D	370,300,270,245,225

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:-

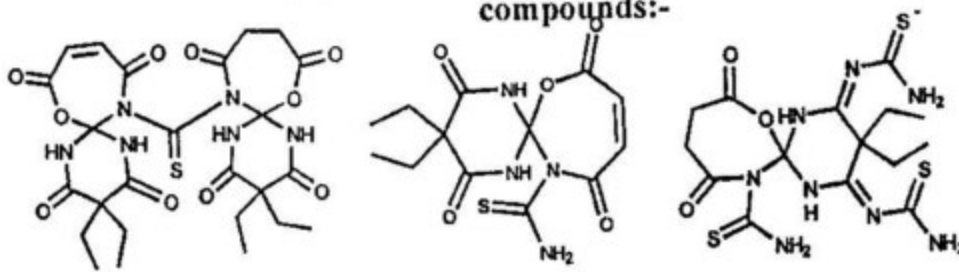
Comp	m.p/C°	Yield %	M.F	Calc.			Found		
				C	H	N	C	H	N
1	220-222	59	C ₁₂ H ₁₈ N ₄ O ₈ S	45.61	5.30	16.36	45.55	5.33	16.26
2	187-189	66	C ₁₃ H ₁₆ N ₄ O ₈ S	45.88	4.74	16.46	45.80	4.83	16.38
3	163-161	71	C ₂₁ H ₂₈ N ₄ O ₈ S	49.60	5.55	16.53	49.52	5.63	16.44
4	179-177	77	C ₂₁ H ₂₆ N ₄ O ₈ S	49.79	5.17	16.59	49.84	5.22	16.43
5	148-150	68	C ₂₅ H ₃₂ N ₄ O ₁₀ S	49.34	5.30	13.81	49.33	5.41	13.70
6	183-185	80	C ₂₅ H ₂₈ N ₄ O ₁₀ S	49.66	4.67	13.90	49.53	4.80	13.81
7	176-178	72	C ₂₅ H ₃₀ N ₄ O ₁₀ S	49.50	4.98	13.85	49.62	5.04	13.76
8	210-212	65	C ₁₈ H ₂₂ N ₄ O ₇ S ₂	43.37	4.45	16.86	43.45	4.54	16.69
9	202-204	58	C ₁₈ H ₂₄ N ₄ O ₇ S ₂	43.19	4.83	16.79	43.28	4.80	16.66
10	231-233	66	C ₁₈ H ₂₀ N ₄ O ₇ S ₂	43.54	4.06	16.93	43.59	4.13	16.89
11	190-102	62	C ₂₂ H ₂₈ N ₄ O ₈ S ₂	46.80	5.00	19.85	46.93	5.15	19.80
12	167-169	69	C ₂₂ H ₃₀ N ₄ O ₈ S ₂	46.63	5.34	19.77	46.78	5.48	19.69
13	155-157	70	C ₃₀ H ₃₆ N ₄ O ₁₂ S ₂	47.11	4.74	14.65	47.26	4.75	14.50
14	189-191	75	C ₃₀ H ₃₈ N ₄ O ₁₂ S ₂	46.99	4.99	14.61	47.10	5.08	14.55
15	171-173	76	C ₁₅ H ₂₂ N ₄ O ₇ S ₂	39.29	4.84	24.43	39.37	5.00	24.31
16	164-166	70	C ₁₅ H ₂₀ N ₄ O ₇ S ₂	39.46	4.42	24.54	39.53	4.52	24.44
17	206-208	81	C ₁₉ H ₂₄ N ₄ O ₆ S ₃	40.85	4.69	20.06	41.00	4.77	20.00
18	182-184	83	C ₁₉ H ₂₂ N ₄ O ₆ S ₃	41.15	4.00	20.20	41.25	4.12	20.16

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

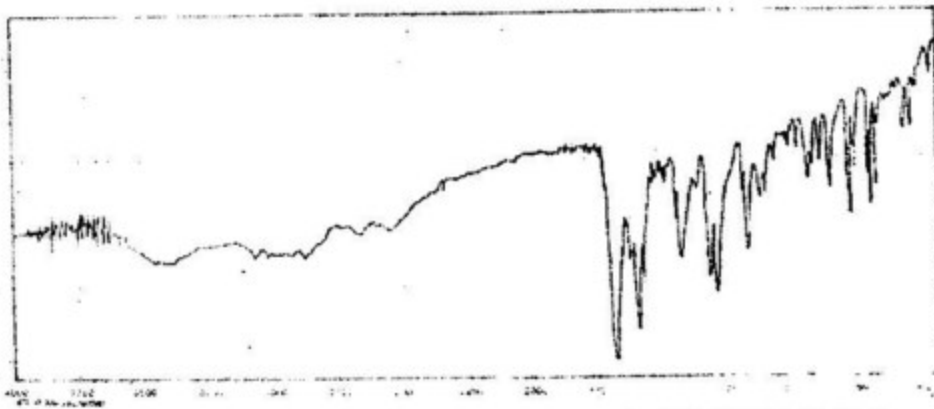
Table(6) : ¹H.N.M.R Spectrophotometer of 3,3-Diethyl-2,4,8,11- tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6] dodecane – amide compounds:-



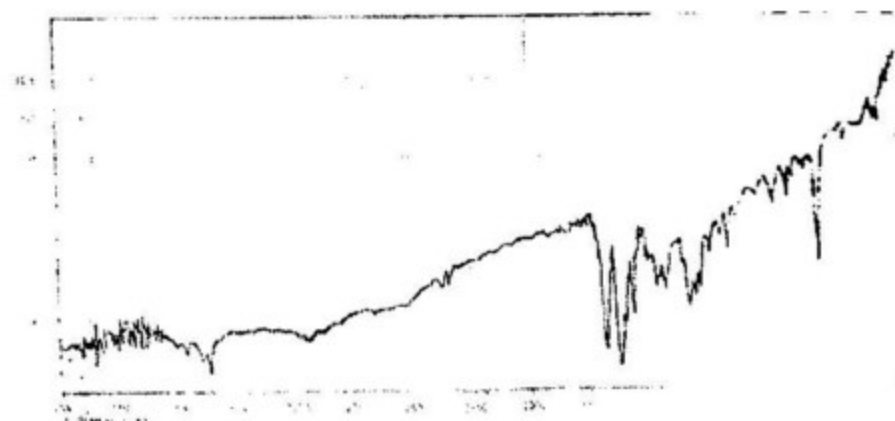
* Chemical shift= δ

** By using DMSO-d₆ as solvent

Comp.	CH ₃	CH ₃ -CH ₂	-CH ₂ -CH ₂ -	HC=CH	NH ₂	C-NH-C
1	0.86	1.75	2.44,2.44	-	1.93	7.85
2	0.85	1.77	-	6.30,6.30	1.95	7.80
3	0.88	1.76	2.43,2.43	-	-	7.85
4	0.89	1.77	-	6.32,6.32	-	7.80
7	0.85	1.79	2.42,2.42	6.31,6.31	-	7.90
8	0.88	1.75	2.44,2.44	6.32,6.32	1.90	7.80
15	0.88	1.28	2.43,2.43	-	1.95	7.90



IR spectrum of 3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-9-one-12-diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidene-amide (4)



IR spectrum of 3,3-Diethyl-2,4,8,11-tetraoxo-7-oxa-1,5,12-triaza-spiro[5.6]dodecane-9-one-12-diethyl-4,6-dioxo-tetrahydro-pyrimidin-2-ylidene-amide (4)

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تحضير وتشخيص، ودراسة الصفات الفيزيائية لمركبات الاوكسازيبين والاكسازيبان من تفاعل (5,5- ثنائي اثيل-4,6-دايوكسو-رباعي هيدرو-بيرميدين -2-يل ادين)-ثايويوريا و 3,1-بس (5,5- ثنائي اثيل-4,6-دايوكسو-رباعي هيدرو-بيرميدين -2-يل ادين)- ثايويوريا مع انهيدريدات الماليك والسكسنيك

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الخلاصة :-

تم تحضير قواعد شيف 3,1-بس (5,5- ثنائي اثيل-4,6-دايوكسو-رباعي هيدرو-بيرميدين -2-يل ادين)-ثايويوريا و 3,1-بس (5,5- ثنائي اثيل-4,6-دايوكسو-رباعي هيدرو-بيرميدين -2-يل ادين)-ثايويوريا من تكثيف الثايويوريا مع مول واحد ومولين من 5,5-ثنائي اثيل-بيرميدين-2,4,6-تريفون . 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 لفة للذكر اعطى نظام حلقي غير متجانس (بحلقتين سباعيتين) . وقد شخصت المركبات المحضرة بتعيين درجات انصهارها، تحليل العناصر، أطيف الأشعة فوق البنفسجية، أطيف الأشعة تحت الحمراء وأطيف الرنين النووي المغناطيسي لبعض منها. وقد أسهمت نتائج التشخيص بالطرق المختلفة في إثبات الصيغ التركيبية للمركبات المحضرة .