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Synthesis ,Characterization and Identification of New Oxazepine,Oxazepane and Oxazinane Compounds from reaction of Some (Z)-2-((2-hydroxybenzylideneamino)methyl)-2-(hydroxymethyl)propane-1,3-diol Derivatives with some cycloanhydrides.

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ABSTRACT

Z)-2- ((2-hydroxybenzylideneamino)methyl -2 -(hydroxy)propane -1,3-diol,(E) -4- (3-hydroxy-2,2-bis (hydroxyl methyl) propylimino)pentan-2-one, -2- ((4-(hydrazonopentane -2-ylideneamino methyl)-2 -(hydroxyl methyl)propane-1,3-diol and(Z)-2-((4-amino-1,5-dimethyl-2-phenyl-1H–pyrazol-3(2H)-ylideneamino)methyl) -2-(hydroxymethyl)propane-1,3-diol were prepared by condensation of 2-(aminomethyl)-2-(hydroxymethyl)propane-1,3-diol with 2-hydroxy benzaldehyde, pentane-2,4-dione, (E)-4-hydrazonopentan-2-one and 4-amino-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-one .These Schiff- bases were reacted with Succinic, Maleic and Malonic anhydride in absolute ethanol to give 6,7-membered heterocyclic ring system Oxazepine,Oxazepane,Oxazinan.The Schiff-bases and the final products were identified by their melting points, elemental analysis, FT-IR and UV-Visible spectra.

Introduction

The synthesis of 2-phenyl -1,3-oxazepine by irradiation of 4-phenyl-2-oxa-3-aza bicyclo[3.2.0]-hepta-3,6-diene was studied by Toshio Makai with other workers[1].The discovery of the central nervous system(CNS) activity of 1,4- benzodiazepine [2] encourage the chemists to look for more effective ways to build up the 7- membered heterocyclic ring systems from already available materials.The Six-membered heterocyclic ring system: 1,3-oxazine has already been reported and thorouly reviewed in the literature [1-4] . Maleic, arylmaleic and substituted maleic anhydrides react with trimethylsilyl azide to give 4- and 5-substituted ((oxauraciles)) dihydro-1,3-oxazine-2,6-diones [5,6].

Both 2- methoxy pyrroline and 2-methoxy piprdinine react with diketene under neutral conditions at 0c to give the corresponding, 2- methoxy dihydro 1,3-oxazine-4-ones [7,8].

One of these ways which has been discovered recently involves direct addition of maleic anhydride to the (C=N) double bond of Schiff - bases and anumber of 2,3-diaryl-2,3-dihydro-1,3-oxazepine- 4,7-diones were prepared and characterized.[5-8]

Diketene reacts with N,N-diphenyl guanidine to give the tranquilizer ketazolam and N-substituted tetrahydro-1,3-oxazine-4-one respectively [9,10]. The reaction of diketene with isocyanic acid , cyanamides and flouro sulphinyl isocyanate afforded the corresponding 1,3-oxazine-2,4-diones [11-13].

Furthermore, thermal rearrangement of ketovinylazirines gave substituted 1,3-oxazepines. [9-14]

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 undergo intermolecular Diels-alder reaction to give dihydro-1,3-oxazines [17].

The reaction of N-Benzylidene 1,5-dimethyl-2-phenylpyrazolonamines (Schiff bases with

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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 [18]. Synthesis and characterization of 8-(4-dimethyl amino-phenyl)-9-(6-R- benzothiazol-2-yl)-7-oxa-9-aza-spiro[4.5]decane-6,10-dione[19].

Extensive synthesis and testing of the barbiturates over a long time span has produced well-defined structure – activity relationships. Which have been summarized. [20] .

Materials and methods

Melting points were recorded with Gallenkamp Melting point Apparatus and were uncorrected. Elemental analysis were carried out with perkin-Elmer,2400;CHN Elemental Analyzer. FT-IR spectra were recorded on FT-IR spectrophotometer -8400s Shimadza (KBr) in Chemistry department of Education for women college AL-Anbar and UV-Visible spectra were recorded (in ethanol) on Schimadza Reco- 160 Spectrophotometer.

Preparation of (Z)-2-((2-hydroxybenzylideneamino)methyl)-2-(hydroxymethyl)propane-1,3-diol:

A mixture of 0.01 mole(1.35 g) of 2-(amino methyl)-2-(hydroxymethyl)propane-1,3-diol and 0.01 mole (1.22 g) of o-hydroxy benzaldehyde in 20 ml of absolute ethanol, was refluxed in a water bath for 1h, then left to cool in ice-water bath whereby yellow crystalline solid was precipitated. The solid was filtered, recrystallized twice from ethanol.

Preparation of (E)-4-(3-hydroxy-2,2-bis(hydroxymethyl)propan-2-one:

A mixture of 0.01 mole(1.35 g) of 2-(amino methyl)-2-(hydroxymethyl)propane-1,3-diol and 0.01 mole (1.0 g) of acetyl acetone in 20 ml of absolute ethanol, was refluxed in a water bath for 1h, then left to cool in ice-water bath whereby yellow crystalline solid was precipitated. The solid was filtered, recrystallized twice from ethanol.

Preparation of 2-((4- hydrazonopentan-2-ylideneamino)methyl)-2-(hydroxymethyl)propane-1,3-diol*:*

A mixture of 0.01 mole(2.17 g) of (E)-4-(3-hydroxy-2,2-bis(hydroxymethyl)propan-2-one and 0.01 mole (0.5 g) of hydrazine hydrate in 20 ml of absolute ethanol, was refluxed in a water bath for 1.5h, then left to cool in ice-water bath whereby yellow crystalline solid was precipitated. The solid was filtered, recrystallized twice from ethanol.

Preparation of(E)-4-((3-hydroxy-2,2-bis(hydroxymethyl)propylimino)methyl)-5-methyl-2phenyl-1H-pyrazol-3(2H)-one :

A mixture of 0.01 mole(1.35)of 2-(amino methyl)-2-(hydroxymethyl)propane-1,3-diol and 0.01 mole (2.03g)of 4-aminoanti pyrine in 20 ml of absolute ethanol, was refluxed in a water bath for 1h, then left to cool in ice-water bath whereby pink crystalline solid was precipitated. The solid was filtered, recrystallized twice from ethanol.

Preparation of 3-(3-hydroxy-2,2-bis(hydroxymethyl)propyl)-2-(2-hydroxyphenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione:

In a 100 ml round bottom flask equipped with a double surface condenser fitted with calcium chloride guard tube, was placed a mixture of 0.01mol (2.39g) of (Z)-2-((2-hydroxybenzylideneamino)methyl)-2-(hydroxymethyl)propane-1,3-diol and 0.01 mol (0.98g)of maleic anhydride in 20ml of absolute ethanol.

The reaction mixture was refluxed in water bath at 78ºC for 2hr., the solvent was then removed and the resulting solid was recrystallized from anhydrous THF. Compounds 8,11,14 preparation in same of method.

Preparation of 3-(3-hydroxy-2,2-bis(hydroxymethyl)propyl)-2-(2-hydroxyphenyl)-1,3-oxazepane-4,7-dione:

In a 100 ml round bottom flask equipped with a double surface condenser fitted with calcium chloride guard tube, was placed a mixture of 0.01mol (2.39g) of (Z)-2-((2-hydroxybenzylideneamino)methyl)-2-(hydroxymethyl)propane-1,3-diol and 0.01 mol (1.0g)of succin anhydride in 20ml of absolute ethanol. The reaction mixture was refluxed in water bath at 78ºC for 2hr., the solvent was then removed and the resulting solid was recrystallized from anhydrous THF. Compounds 9,12,15 preparation in same of method.

Preparation of (R)-3-(3-hydroxy-2,2-bis(hydroxymethyl)propyl)-2-methyl-2-(2-oxopropyl)-1,3-oxazinane-4,6-dione:

In a 100 ml round bottom flask equipped with a double surface condenser fitted with calcium chloride guard tube, was placed a mixture of 0.01mol (1.35g) (E)-4-(3-hydroxy-2,2-bis(hydroxymethyl)propylimino)pentan-2-one0.01 mol(0.86g) of malonic anhydride in 20ml of absolute ethanol. The reaction mixture was refluxed in water bath at 78ºC for 3hr., the solvent was then removed and the resulting solid was recrystallized from anhydrous 1,4-Dioxan. Compounds 10,13,16 preparation in same of method.

Results and Discussion

Schiff bases (1-4) are prepared by condensation of 2-(amino methyl)-2-(hydroxymethyl)propane-1,3-diol with salicyldehyde, acetyl acetone and 4-amino antipyrine .

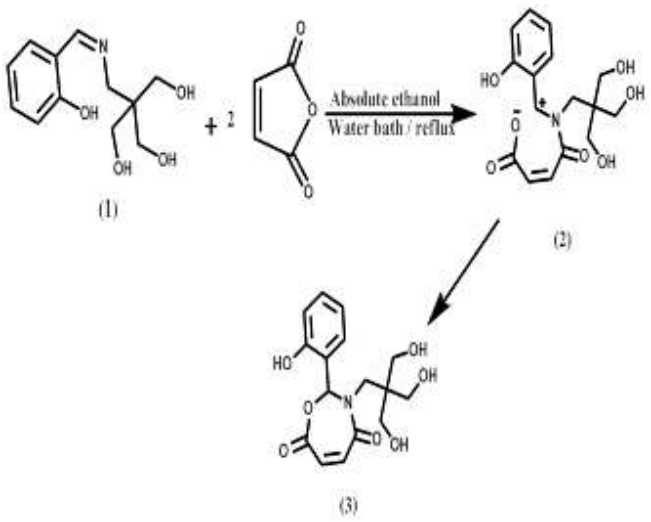
It is known that Schiff bases react smoothly with acid chlorides and anhydrides to give the corresponding addition products[21-24].

This is indicated by the appearance of the characteristic C=O (lacton-lactam) absorption band at (1665-1680)cm-1 in the FT-IR spectra of addition products (3).

It is impressive to note that the two absorption band at (1800-1950)cm-1 in the FT-IR spectra of pure Maleic ,Succinic and Malonic anhydride have diappeared when the anhydride became of the 6,7-membered ring system of the3-(3-hydroxy-2,2-bis (hydroxymethyl) propyl)-2-phenyl-1,3-oxazepane-4,7-dione,3-(3-hydroxy-2,2-bis(hydroxymethyl)propyl)-2-phenyl-1,3-oxazepine-4,7-dion and 2-(4-(dimethylamino) phenyl)-3-(3-hydroxy-2,2- bis (hydroxymethyl) propyl)-1,3- oxazinane -4,6-dione. The new absorption bands of the (C=O) group in the FT-IR spectra of the addition products (3) appear at (1665-1685)cm-1 , this attributed to the fact that the structures of the addition products are combination of the lacton-lactam structure [25]

Furthermore, the UV-Visible spectra of Oxazepine, Oxazinane derivatives show absorption maxima at(240-350)nm due to charge transfer of the cyclic 6,7- membered lactone-lactam combined structure [3]. and positive Br2/CCl4 and KMNO4 tests.

In this paper , the reaction of the Maleic, Succinic and Malonic anhydride with (1-4) Schiff bases gives the dipolar intermediate [2] which collapses to 6,7-membered heterocyclic ring system. [3] is presented.



The reaction of maleic , succinic and malonic 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.

Table (1) Melting points, yield, Molecular formula[M.F], elemental analysis of New Schiff bases(1-4)

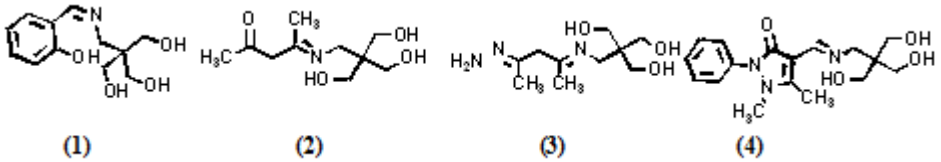


Table (2) The major FT-IR absorptions (cm-1) of New Schiff bases (1-4)

Compound	O-H str.	N-H str.	C-H str. Aromatic	C=O . str.	C=N str.	C=C str. Aromatic	C-O str.	C-H bend. Aromatic
1	3540	-	3050	-	1610	1580	1310	1010,770
2	3460	-	3070	1690	1605	1585	1320	1020,870
3	3510	3450,3310	3090	-	1620	1580	1310	1010,900
4	3470	3430,3280	3065	1680	1610	1590	1330	1020,870

Table (3) The UV-Visible absorption maxima λ/nm of New Schiff bases(1-4)

Compound	*UV-Visible absorption maxima λ/nm*
1	380,300,266.225.220
2	275,226,220
3	280,244,222
4	375,320,260,251,226

Table(4) Melting point ,percentage yield, molecular formula and elemental analysis of compounds (5-16)

Comp	*m.p/c*	*Yield%*	*M.F*	*Calc.*			*Found*		
				C	*H*	*N*	*C*	*H*	*N*
5	177-175	72	$C_{16}H_{19}NO_7$	56.97	5.68	4.15	56.68	5.55	4.01
6	190-188	66	$C_{16}H_{21}NO_7$	56.63	6.24	4.13	56.45	6.18	3.98
7	211-209	68	$C_{15}H_{19}NO_7$	55.38	5.89	4.31	55.12	5.80	4.21
8	240-238	65	$C_{14}H_{21}NO_7$	53.33	6.71	4.44	53.19	6.65	4.20
9	166-164	80	$C_{14}H_{23}NO_7$	52.99	7.31	4.41	53.04	7.16	4.23
10	170-168	71	$C_{13}H_{21}NO_7$	51.48	6.98	4.62	51.29	6.80	4.54
11	246-244	67	$C_{18}H_{25}N_3O_9$	50.58	5.90	9.83	50.41	5.74	9.70
12	188-186	70	$C_{18}H_{29}N_3O_9$	50.11	6.78	9.74	50.02	6.51	9.48
13	165-163	66	$C_{16}H_{25}N_3O_9$	47.64	6.25	10.42	47.46	6.23	10.28
14	154-152	67	$C_{20}H_{26}N_4O_6$	57.41	6.26	13.39	57.31	5.99	13.10
15	206-104	59	$C_{20}H_{28}N_4O_6$	57.13	6.71	13.33	57.03	6.52	13.08
16	258-256	57	$C_{19}H_{26}N_4O_6$	56.15	6.45	13.79	56.01	6.28	13.62

Table(5)The major IR absorption (cm-1)of compounds (5-16)

Comp.	*O-H str.*	*NH_2 str.*	*C=O str.*	*C=O str.* Lacton, lactam	*C=N str.*	*C=C str.* Olefin	*C=C str.* Aromatic	*C-O-C str.*
5	**3510,3470**	**-**	**-**	**1680**	**1430**	**1600**	**1580,1560**	**1190**
6	**3520,3480**	**-**	**-**	**1680**	**1435**	**-**	**1580,1540**	**1180**
7	**3540,3470**	**-**	**-**	**1670**	**1450**	**-**	**1580,1560**	**1170**
8	**3570**	**-**	**1710**	**1685**	**1440**	**1610**	**-**	**1200**
9	**3560**	**-**	**1700**	**1670**	**1430**	**-**	**-**	**1210**
10	**3555**	**-**	**1705**	**1675**	**1450**	**-**	**-**	**1200**
11	**3530**	**3410,3360**	**-**	**1670**	**1480**	**1610**	**-**	**1190**
12	**3560**	**3430,3320**	**-**	**1665**	**1460**	**-**	**-**	**1210**
13	**3540**	**3430,3290**	**-**	**1680**	**1450**		**-**	**1200**
14	**3550**	**3420,3280**	**-**	**1670**	**1430**	**1600**	**1580,1555**	**1190**
15	**3560**	**3415,3275**	**-**	**1670**	**1460**	**-**	**1580,1565**	**1180**
16	**3540**	**3450,3300**	**-**	**1675**	**1450**	**-**	**1580,1565**	**119**

Table (6) Uv-spectra of compounds (5-16)

Comp.	*UV-Visible absorption maxima λ/nm*
5	320,300,266,230.221
6	315,255,243,229
7	333,265,251,243,223
8	285,265,239,224
9	289,269,241,236,222
10	285,240,225,220
11	275,250,235,226
12	295,250,235,222
13	290,269,240,235,220
14	310,300,276,220.
15	325,235,223
16	330,285,261,243,223

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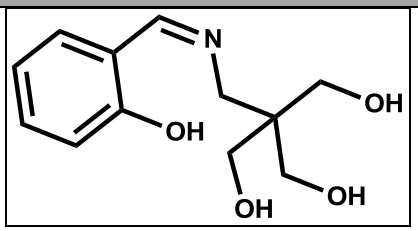
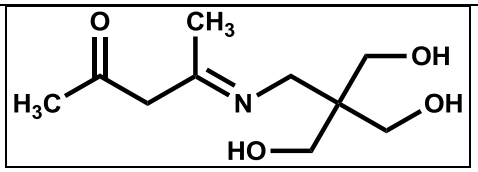
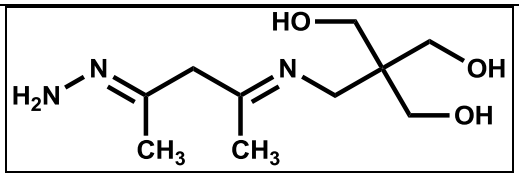
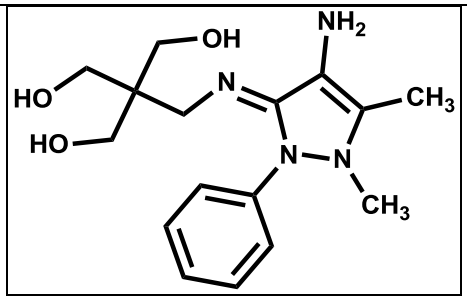
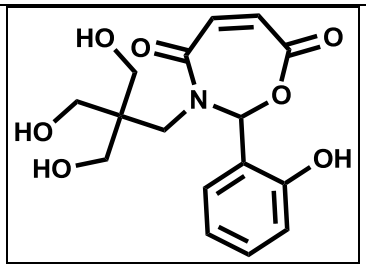
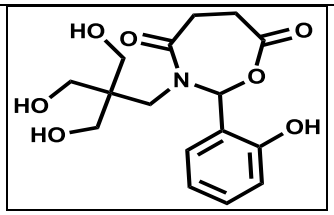
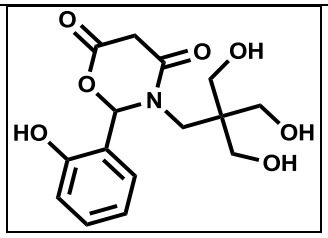
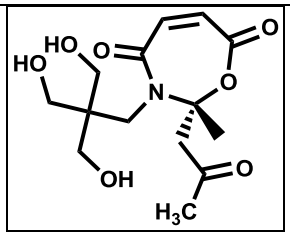
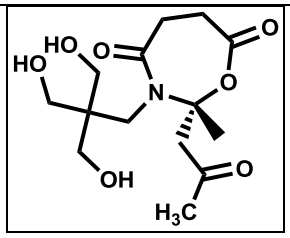
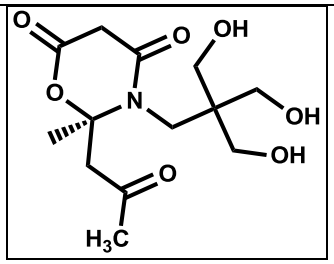
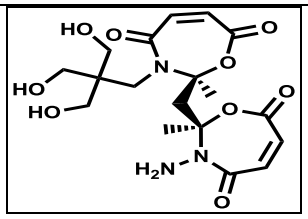
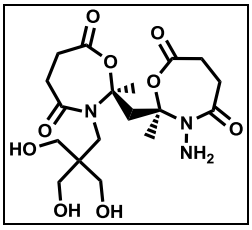
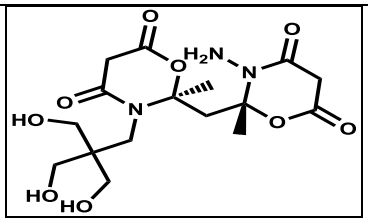
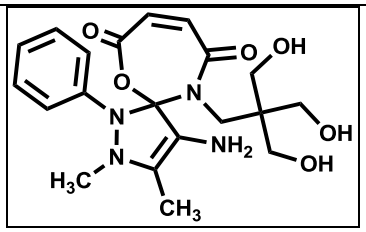
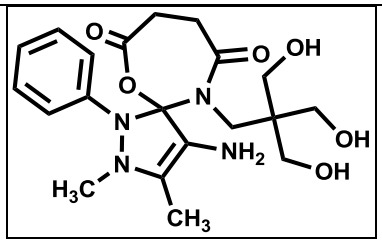
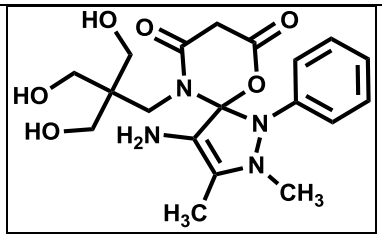
No.	Name of compounds	Structure
1	**(*Z*)-2-((2-hydroxybenzylideneamino) methyl)-2-(hydroxymethyl) propane-1,3-diol**	
2	**(*E*)-4-(3-hydroxy-2,2-bis (hydroxymethyl)propylimino)pentan-2-one**	
3	**2-((4-hydrazonopentan-2-ylideneamino) methyl)-2-(hydroxymethyl)propane-1,3-diol**	
4	**(*Z*)-2-((4-amino-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-ylideneamino)methyl)-2-(hydroxymethyl)propane-1,3-diol**	

5	**3-(3-hydroxy-2,2-bis(hydroxymethyl) propyl)-2-(2-hydroxyphenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione**	
6	**3-(3-hydroxy-2,2-bis(hydroxymethyl)propyl)-2-(2-hydroxyphenyl)-1,3-oxazepane-4,7-dione**	
7	**3-(3-hydroxy-2,2-bis(hydroxymethyl) propyl)-2-(2-hydroxyphenyl)-1,3-oxazinane-4,6-dione**	

8	**(*R*)-3-(3-hydroxy-2,2-bis(hydroxymethyl) propyl)-2-methyl-2-(2-oxopropyl)-2,3-dihydro-1,3-oxazepine-4,7-dione**	
9	**(*R*)-3-(3-hydroxy-2,2-bis(hydroxymethyl) propyl)-2-methyl-2-(2-oxopropyl)-1,3-oxazepane-4,7-dione**	
10	**(*R*)-3-(3-hydroxy-2,2-bis(hydroxymethyl) propyl)-2-methyl-2-(2-oxopropyl)-1,3-oxazinane-4,6-dione**	
11		

	(*R*)-3-amino-2-(((*S*)-3-(3-hydroxy-2,2-bis(hydroxymethyl)propyl)-2-methyl-4,7-dioxo-2,3,4,7-tetrahydro-1,3-oxazepin-2-yl) methyl)-2-methyl-2,3-dihydro -1,3-oxazepine-4,7-dione	
12	**(*R*)-3-amino-2-(((*S*)-3-(3-hydroxy-2,2-bis (hydroxymethyl)propyl)-2-methyl-4,7-dioxo-1,3-oxazepan-2-yl)methyl)-2-methyl-1,3-oxazepane-4,7-dione**	
13	**(*R*)-3-amino-2-(((*S*)-3-(3-hydroxy-2,2-bis(hydroxymethyl)propyl)-2-methyl-4,6-dioxo-1,3-oxazinan-2-yl)methyl)-2-methyl-1,3-oxazinane-4,6-dione**	
14	**2-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)-3-(3-hydroxy-2,2-bis(hydroxymethyl)propyl)-2,3-dihydro-1,3-oxazepine-4,7-dione**	
15	**2-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)-3-(3-hydroxy-2,2-bis(hydroxymethyl)propyl)-1,3-oxazepane-4,7-dione**	
16	**2-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)-3-(3-hydroxy-2,2-bis(hydroxymethyl)propyl)-1,3-oxazepane-4,7-dione**	

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تحضير وتشخيص ودراسة الصفات الفيزياوية لمركبات الآوكسازبين ،ألآوكسازبان وألآوكسينان من تفاعل قواعد شيف (Z)-2-((2-هيدروكسي بنزيلدين أمينو) مثيل)2 -((2-(Z)-هيدروكسي بنزيلدين أمينو) مثيل)-2- ((هيدروكسي) بروبان-2--1, 3 ,-دايول مع بعض الانهيدريدات الحلقية

وليد فرج حمادي خالد فاروق عبد الغفور حميد مدلول الكبيسي

الخلاصة :-

تعطي تفاعلات قواعد شيف جديدة (Z)-2-((2-هيدروكسي بنزيلدين أمينو) مثيل)-2- (هيدروكسي) بروبان-1, 3 – دايول، (Z)-2-((2-هيدروكسي بنزيلدين أمينو) مثيل)-2- (هيدروكسي مثيل) بروبان-1, 3 – دايول و (Z)-2-((4- ثنائي مثيل امينو) بنزيلدين أمينو) مثيل)-2- (هيدروكسي مثيل) بروبان-3,1- دايول مع انهيدريدات السكسنيك ، الماليك والمالونيك الحلقية مركبات سداسية و سباعية الحلقة ، الاوكسازبينات ، الاوكسازبانات،الاوكسازينات. تم التثبت من صحة التراكيب المحضرة من خلال تعيين درجات الانصهار و طيف الأشعة فوق البنفسجية – المرئية وطيف الأشعة تحت الحمراء FT-IR والتحليل العنصري الدقيق (**C.H.N.**) .