

*Synthesis and Characterization of 2-(2-hydroxy-phenyl)-3-(6-R-benzothiazol-2-yl)-2,3-dihydro-[1,3]oxazepine-4,7-dione and 2-(2-hydroxy-phenyl)-6-methyl-3-(6-R-benzothiazol-2-yl)-2,3-dihydro-[1,3]oxazepine-4,7-dione .[1]*

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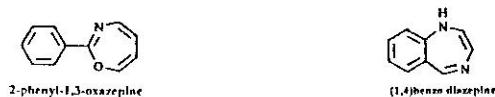
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## ABSTRACT

2-[(6-R-benzothiazol-2-ylimino)-methyl]-phenols (*Schiff bases*) were prepared by condensation of 6-R-2-amino benzothiazol with Salicyldehyde. These Schiff bases were found to react with maleic anhydride and citraconic anhydride to give 2-(2-hydroxy-phenyl)-3-(6-R-benzothiazol-2-yl)-2,3-dihydro-[1,3]oxazepine-4,7-dione and 2-(2-hydroxy-phenyl)-6-methyl-3-(6-R-benzothiazol-2-yl)2,3-dihydro-[1,3]oxazepine-4,7-dione. which were reacted with pyrrolidine to give anilid-pyrrolidine derivatives of maleic and citraconic

## INTRODUCTION

The synthesis of 2-phenyl-1,3-oxazepine<sup>(1)</sup> and the discovery of the central nervous system (CNS) activity of 1,4-benzodiazepine<sup>(2)</sup> 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 o 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<sup>(3,4)</sup>.



## EXPERIMENTAL

Melting points were recorded with Gallenkamp 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 <sup>1</sup>H-NMR spectra were recorded with BRUKER-AC-200MHZFT-NMR in mutha University.UV-Visible spectra were re-

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corded (in ethanol) with Schimadsu Recc-160 spectrophotometer.

#### **Preparation of 2-[(6-R-benzothiazol-2-ylimino)-methyl]-phenol**

Were prepared according to known procedures<sup>(3)</sup>. Tables (1-3) list m.p.s, yield. Elemental analysis,IR, and UV-spectra.

#### **Preparation of 2-(2-hydroxy-phenyl)-3-(6-R-benzothiazol-2-yl)-2,3-dihydro-[1,3]oxazepine-4,7-dione.[3] oxazepine-4,7-diones.**

In a (100ml)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-R-benzothiazol-2-ylimino)-methyl]-phenol and 0.01 mole of maleic anhydride in 10ml of dry benzene . The reaction mixture was refluxed in a water bath for 1.5 hr . The solvent was removed and the resulting solid was recrystallized from THF.

This experiment was repeated using different Schiff bases in order to obtain other 1,3-oxazepine

#### **Attempted hydrolysis of 2-(2-hydroxy-phenyl)-3-(6-R-enzothiazol-2-yl)-2,3-dihydro- [1,3]oxazepine-4,7-dione.[4]**

a) A mixture of 0.005 mole of 2-(2-hydroxy-phenyl)-3-(6-R- benzothiazol-2-yl)- 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 2-(2-hydroxy-phenyl)-3-(6-R-benzothiazol-2-yl)-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 4-Oxo-4-pyrolidin-1-yl-but-2-enoic acid [2-hydroxy -(2-hydroxy-phenyl)-methyl]- (6-R-benzothiazol-2-yl)-amide.[5]**

To a mixture of 0.005 mole of 2-(2-hydroxy-phenyl)-3-(6-R-benzothiazol-2-yl)- 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 of male 4-Oxo-4-pyrolidin-1-yl-but-2-enoic acid [2-hydroxy -(2-hydroxy-phenyl)-methyl]- (6-R-benzothiazol-2-yl)-amide were obtained following the same procedure

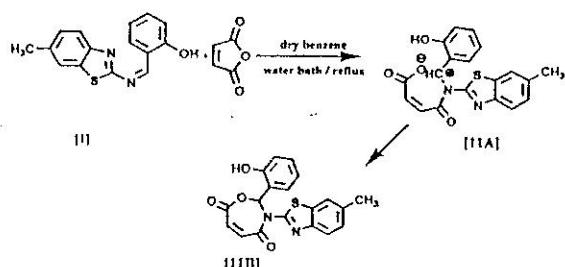
## **DISCUSSION**

Schiff bases<sup>(3)</sup> are prepared by condensation of 6-R-2-amino benzothiazol with salicyldehyde to give 2-[(6-R-benzothiazol-2-ylimino)-methyl]-phenols. The reaction is followed by the appearance of (N=CH) absorption band at (1600-1610) cm<sup>-1</sup> the disappearance of both (C=O) absorption band at (1670-1685) cm<sup>-1</sup> and (-NH<sub>2</sub>) absorption bands at (3400,3650) cm<sup>-1</sup> in their IR spectra<sup>(4)</sup>. Where: R= CH<sub>3</sub>, NO<sub>2</sub>, OCH<sub>3</sub>, Cl, Br 2-[(6-R-benzothiazol-2-ylimino)-methyl]-phenols are identified by their m.p.s., elemental analysis (table-1) ,IR spectra (table-2) , and UV-Visible spectra (table-3).

It is known that Schiff bases react smoothly with acid chlorides and an-

hydrides to give the corresponding addition products<sup>(5,6,7)</sup>.

In this paper , the reaction of the cyclic unsaturated anhydride ( maleic and citraconic) anhydride with 2-[ (6-R-benzothiazol-2-ylimino)-methyl]-phenols can be presented as follows:



In this reaction, the nitrogen atom of the Schiff base attack one of the two ( $\text{C}=\text{O}$ ) groups of anhydride yielding the dipolar intermediate (2) which collapses to the neutral species (11B) which is a combination of  $\alpha$ -lactone and  $\beta$ -lactam in a 7- membered ring.

The reaction is followed by the disappearance of ( $\text{N}=\text{C}$ ) absorption band at (1600-1610)  $\text{cm}^{-1}$ , and the appearance of the absorption bands of expected groups in the IR spectra of 2-(2-hydroxy-phenyl)-3-(6-R-benzothiazol-2-yl)- 2,3-dihydro-[1,3]oxazepine-4,7-dione , and 2-(2-hydroxy-phenyl)-6-methyl-3-(6-R-benzothiazol-2-yl)-2,3-dihydro-[1,3]oxazepine-4,7-dione[ 11]. The ( $\text{C}=\text{O}$ ) 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<sup>(8,9)</sup> is absorbed in the same region (1670-1700)  $\text{cm}^{-1}$ . 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 olefine through a (4n+2) transition state to yield the cycloheptenyl cation<sup>(10)</sup>.

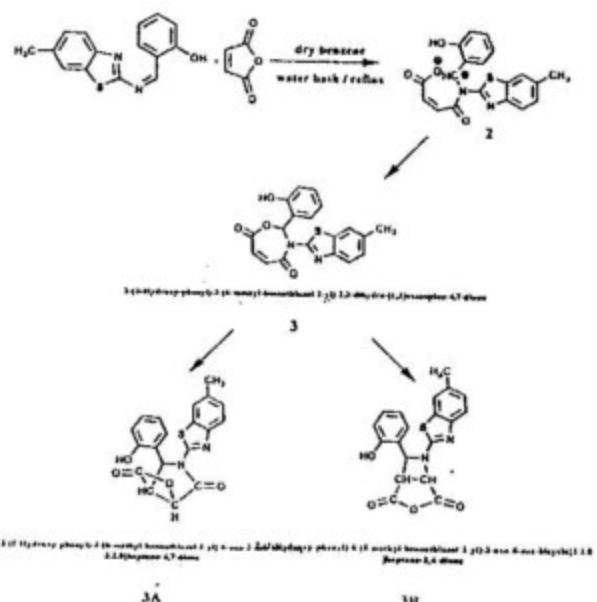
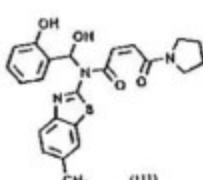
Structure [11B] is a combination of both lactone and lactam in a 7- heterocyclic ring. This is indicated by the appearance of the characteristic( $\text{C}=\text{O}$ ) (lactone/lactam) absorption band at (1660-1680) $\text{cm}^{-1}$  in their IR spectra. Furthermore, structure (11) still maintains the (*cis*- $\text{CH}=\text{CH}$ ) double bond of maleic and citraconic anhydride as indicated by the absorption band at (1600-1610) $\text{cm}^{-1}$

and positive  $\text{Br}_2/\text{CCl}_4$  and  $\text{KMNO}_4$  tests. Furthermore, the UV-Vissible spectra of Oxazepine derivitaves show absorption maxima at( 240-350)nm due to charge transfer of the cyclic 7-membered lactone-lactam combined structure [3].

Structure [3A] is unlikely, because of the high strain associated with 4-membered ring system (& - lactone ring), particularly when it is fused to another relatively small ring (  $\beta$  -lactam ring) . In addition, Structure[3A] is expected to show the IR absorption band of  $\text{C}=\text{O}$  (& -lactone) at 1750  $\text{cm}^{-1}$  and of  $\text{C}=\text{O}$  (  $\beta$  -lactam) at 1650  $\text{cm}^{-1}$ . However ; the lack of these absorption bands and the appearance of *cis*  $\text{CH}=\text{CH}$  absorption band in the IR spectrum of the lactone –lactum addition product [3] is an indicative evidence against, the structure [3A].

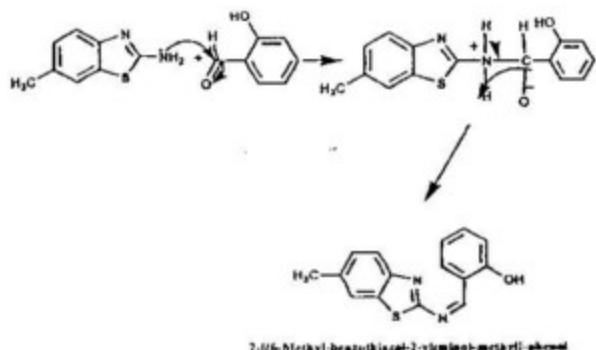
Structure [3B] which can be proposed for these products, results from the (2+2) cycloaddition of the reactants . The evidences against this structure came from the fact that the cycloaddition (2+2) reaction takes place under the influence of light and it is not expected under thermal condition . Previously , it was demonstrated that the basic hydrolysis of 2-(2-hydroxy-phenyl)-3-(6-methyl-benzothiazol-2-yl)- 2,3-dihydro-[1,3]oxazepine-4,7-dione is unsuccessful due to immediate reclosure on acidification. This reclosure is easy to achieve due to the involved  $\text{COOH}$  and  $\text{OH}$  groups within the *cis* configuration of maleic, citra-

conic acid moiety. In order to avoid reclosure the original title compounds ( $\pi$ ) are treated with pyrrolidine to give the open-chain anilide-pyrrolidide derivatives of maleic, phthalic and citraconic acid [4C] which still maintain the (cis-CH=CH) double bond configuration as evidenced by its IR absorption band at (1600-1610)  $\text{cm}^{-1}$  and by positive  $\text{Br}_2/\text{CCl}_4$  and  $\text{KMnO}_4$  tests.

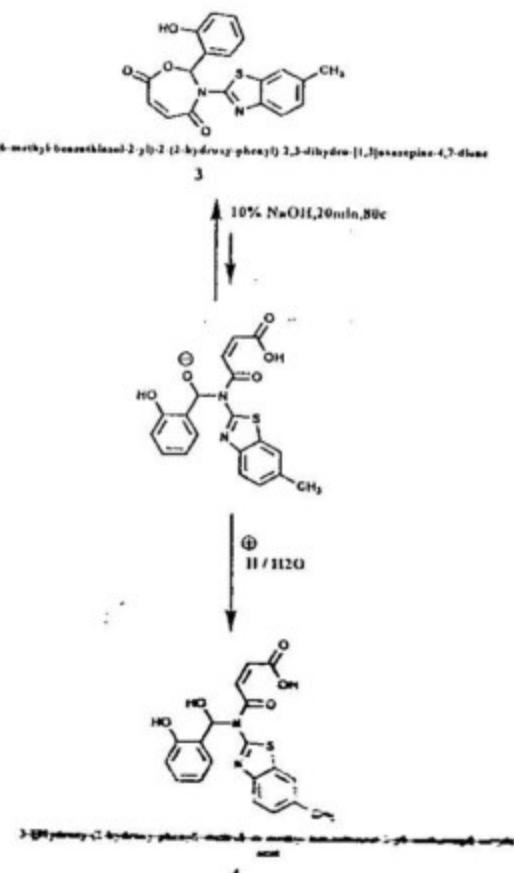


Scheme 2

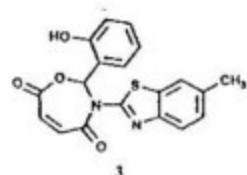
Apparently, this reaction involves an acyl-oxygen cleavage of the  $\gamma$ -lactone ring, while N-C=O linkage is unaffected under these condition. Since none of the two nitrogen atoms in the resulting products carries hydrogen, whereas reclosure to the cyclic diamide is not expected. Male 4-oxo-4-pyrrolidine-1-yl-but-2-enoic acid (6-methyl-benzothiazol-2-yl)-[hydroxy-(2-hydroxy-phenyl)-methyl]-amide are identified by their m.p.s. elemental analysis (table-6), IR spectra (table-7),  $^1\text{H-NMR}$  spectra (table-8) and UV-Visible spectra (table-9).



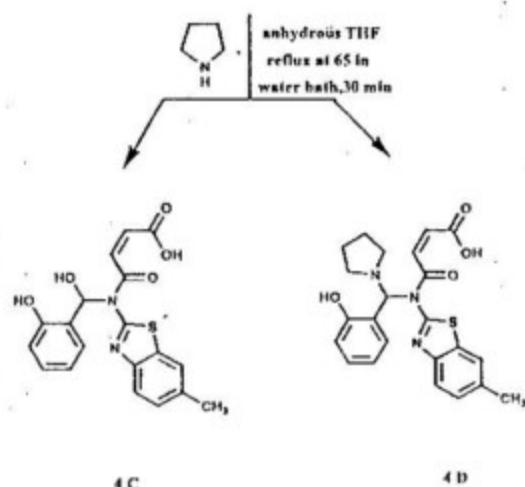
(Scheme 1)



Scheme 3

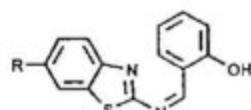


3-(6-methyl-benzothiazol-2-yl)-2-(2-hydroxy-phenyl)-3,5-dihydro-[1,3]oxazepine-4,7-dione

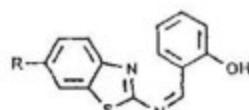


Scheme 4

Table (1): Melting point, percentage yield, molecular formula and elemental analysis of 2-[(6-R- benzothiazol-2-ylimino)-methyl]-phenol.



Compound	R	M.P./C	Yield%	M.F	Calc.			Found		
					C	H	N	C	H	N
	CH <sub>3</sub>	172	86	C <sub>11</sub> H <sub>12</sub> N <sub>2</sub> OS	67.14	4.5	10.44	67.25	4.60	10.40
	(CH <sub>2</sub> ) <sub>2</sub>	188	88	C <sub>12</sub> H <sub>14</sub> N <sub>2</sub> OS	68.06	5.00	9.92	67.99	4.89	10.04
	NO <sub>2</sub>	169	80	C <sub>12</sub> H <sub>12</sub> N <sub>2</sub> O <sub>3</sub> S	56.18	3.03	14.04	56.23	3.11	14.00
	OCl <sub>3</sub>	155	74	C <sub>12</sub> H <sub>12</sub> NO <sub>2</sub> S	63.36	4.25	9.85	63.41	4.31	9.88
	Cl	174	77	C <sub>12</sub> H <sub>12</sub> NOSCl	58.23	3.14	9.70	58.11	3.22	9.67
	Br	179	75	C <sub>12</sub> H <sub>12</sub> NOSBr	50.46	2.72	8.41	50.50	2.81	8.38

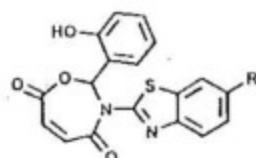
Table (2): The major IR absorptions (cm<sup>-1</sup>) of substituted 2-[(6-R- benzothiazol-2-ylimino)-methyl]-phenol.

Compound	O-H str. Phenol	C-H str. Aromatic	C-H str. Alkane	C=N Imine	C-C str. Aromatic	C-H bend Alkane	C-S str.	Others
	3456	3020	2840	1600	1590,1580,1540	1470,1360	1250	
	3460	3060	2860	1620	1590,1520,1490	1460,1415	1245	
	3450	3040	---	1610	1570,1500,1480	---	1238	1335,1530 NO <sub>2</sub>
	3439	3010	2850	1600	1580,1510,1490	1450,1420	1234	1210 C-O-C Ether
	3440	3030	---	1610	1560,1540,1480	---	1235	730 C-Cl
	3445	3050	---	1600	1580,1550,1485	---	1245	650 C-Br

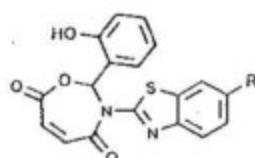
Table ( 3 ) : The UV-Vissibl absorption maxima  $\lambda/\text{nm}$  of 2-[ (6-R- benzothiazol-2-ylimino)-methyl]-phenol.

compound	UV-Vissibl absorption maxima $\lambda/\text{nm}$
	380,300,266,225,220
	370,310,275,226
	385,315,280,244,222
	375,320,260,251,226
	380,312,245,225
	345,300,270,234,223

Table ( 4 ) : Melting point,percentage yield, molecular formula and elemental analysis of 2-(2-hydroxy-phenyl)-3-(6-R- benzothiazol-2-ylimino)-2,3-dihydro-[1,3]oxazepine-4,7-dione

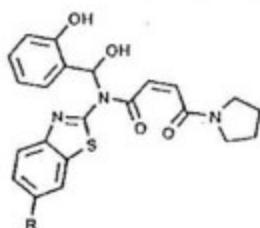


Compound	R	M.P./C	Yield %	M.F	Calc.			Found		
					C	H	N	C	H	N
1-3	CH <sub>3</sub>	192	77	C <sub>19</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> S	62.28	3.85	7.65	62.15	4.00	7.50
2-3	4,6-(CH <sub>3</sub> ) <sub>2</sub>	195	79	C <sub>20</sub> H <sub>16</sub> N <sub>2</sub> O <sub>2</sub> S	63.14	4.24	7.36	63.21	4.18	7.42
3-3	NO <sub>2</sub>	183	68	C <sub>19</sub> H <sub>12</sub> N <sub>2</sub> O <sub>2</sub> S	54.41	2.79	10.57	54.51	2.88	10.46
4-3	OCH <sub>3</sub>	170	59	C <sub>19</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> S	59.68	3.69	7.33	59.71	3.66	7.50
5-3	Cl	199	71	C <sub>19</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> SCl	55.89	2.87	7.24	56.00	2.74	7.23
6-3	Br	186	69	C <sub>19</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> SB <sub>2</sub>	50.13	2.57	6.50	50.22	2.60	6.35

Table ( 5 ) : The major IR absorptions ( $\text{cm}^{-1}$ ) of 2-(2-hydroxy-phenyl)-3-(6-R- benzothiazol-2-ylimino)-2,3-dihydro-[1,3]oxazepine-4,7-dione

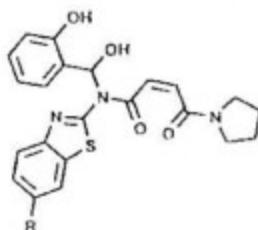
Compound	O-H str. Phenol	C-H str. Benzylic	C-H str. Olefine	C=O str. Lacton,lactum	C-C str. Olefine	C-C str. Aromatic	C-N str.	C-O str. lactum	C-H bend Aromatic	C=S str.	Others
1-3	3450	1260	1150	1670	1600	1580,1560	1440	1310	1020,770	1230	
2-3	3410	3290	3180	1670	1660	1580,1530	1445	1320	1010,870	1238	
3-3	3410	3180	3170	1678	1620	1580,1560	1450	1320	1050,920	1230	1330,1540 NO <sub>2</sub>
4-3	3435	3210	3150	1675	1610	1580,1555	1440	1330	1055,930	1280	1220, C=O,C
5-3	3430	3200	3160	1680	1600	1580,1565	1430	1300	1020,780	1245	750 Cl
6-3	3430	3200	3160	1675	1600	1580,1565	1450	1305	1080,770	1240	680 Br

Table (6) : Melting point,percentage yield, molecular formula and elemental analysis of 4-Oxo-4-pyrrolidine-1-yl-but-2-enolic acid (6-R- benzothiazol-2-yl)-[hydrosy-(2-hydroxy-phenyl)-methyl]-amide



Compound	R	M.P/C	Yield %	M.F	Calc.			Found		
					C	H	N	C	H	N
1-5	CH <sub>3</sub>	215	60	C <sub>19</sub> H <sub>23</sub> N <sub>3</sub> O <sub>4</sub> S	63.14	5.30	9.60	63.20	5.39	9.66
2-5	(CH <sub>3</sub> ) <sub>2</sub>	218	72	C <sub>21</sub> H <sub>25</sub> N <sub>3</sub> O <sub>4</sub> S	63.84	5.58	9.31	64.00	5.42	9.23
3-5	NO <sub>2</sub>	245	68	C <sub>19</sub> H <sub>23</sub> N <sub>3</sub> O <sub>4</sub> S	56.40	4.30	11.96	56.51	4.33	12.05
4-5	OCH <sub>3</sub>	215	61	C <sub>19</sub> H <sub>23</sub> N <sub>3</sub> O <sub>4</sub> S	60.91	5.14	9.27	61.03	5.07	9.38
5-5	Cl	226	60	C <sub>19</sub> H <sub>23</sub> N <sub>3</sub> O <sub>4</sub> SCl	57.70	4.40	9.18	57.77	4.53	9.28
6-5	Br	220	59	C <sub>21</sub> H <sub>25</sub> N <sub>3</sub> O <sub>4</sub> Br	52.60	4.01	8.16	52.75	4.09	8.51

Table (7): The major IR absorptions ( $\text{cm}^{-1}$ ) of 4-Oxo-4-pyrrolidine-1-yl-but-2-enolic acid (6-R- benzothiazol-2-yl)-[hydrosy-(2-hydroxy-phenyl)-methyl]-amide.



Compound	O-H str. Alcohol	O-H str. Phenol	C-H str. Olefine	C=O str. amide	C=C str. Olefine	C=C str. Aromatic	C=N str.	C-O Alcohol	C-S str.	Others
2-5,1-5,1'	3460	3310	3140	1670	1600	1580,1490	1430	1280	1230	
3-5,2-5,2'	3465	3320	3170	1670	1600	1580,1530	1430	1280	1235	
5-5,3-5,3'	3480	3320	3150	1635	1610	1520,1540	1445	1270	1230	1365,1525 NO <sub>2</sub>
2-5,4-5,4'	3500	3290	3150	1665	1605	1560,1530	1450	1290	1240	1210 C=O-C
1-5,5-5,1'	3475	3300	3160	1660	1605	1580,1530	1445	1280	1230	775 C-CI
2-5,6-5,6'	3460	3290	3160	1660	1600	1530,1550	1435	1275	1235	655 C-Br

Table (8): <sup>1</sup>H-N.M.R spectrophotometry of 2-(2-hydroxy-phenyl)-3-(6-R- benzothiazol-2-yl)-2,3-dihydro-1,3-oxazepine-4,7-dimine and 4-Oxo-4-pyrrolidine-1-yl-but-2-enolic acid (6-R- benzothiazol-2-yl)-[hydrosy-(2-hydroxy-phenyl)-methyl]-amide.



\*Chemical shift  $\delta$

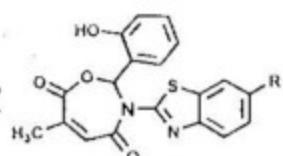
\*\* By using DMSO-d<sub>6</sub> as solvent

Comp.	H-R	H-C-C-H	O-H phenol	O-H Alcohol	H-C	Aromatic	Pyrrolidine ring			
							H <sub>1</sub>	H <sub>2</sub>	H <sub>3</sub>	H <sub>4</sub>
1-5,1'	2.4	6.4,6.4	5.0	—	—	6.5-8.1	—	—	—	—
2-5,2'	2.35,2.4	6.5,6.5	5.0	—	—	6.5-7.9	—	—	—	—
3-5,3'	3.7	6.5,6.5	4.95	—	—	6.6-8.0	—	—	—	—
4-5,4'	2.4	6.4,6.4	4.95	2.1	—	6.5-7.8	3.3	1.5	3.3	1.5
5-5,5'	2.35,2.4	6.5,6.5	5.0	2.0	—	6.5-8.0	3.4	1.4	3.4	1.4
6-5,6'	3.7	6.4,6.4	5.0	2.0	—	6.6-7.9	3.3	1.5	3.3	1.5

Table (9): The UV-Visible absorption maxima  $\lambda/\text{nm}$  of 2-(2-hydroxy-phenyl)-3-(6-R-benzothiazol-2-yl)-2,3-dihydro-  
-[1,3]oxazepine-4,7-dione and 4-Oxo-4-pyrrolidine-1-yl-but-2-enic acid (6-R-benzothiazol-2-yl)  
-[hydroxy-(2-hydroxy-phenyl)-methyl]-amide.

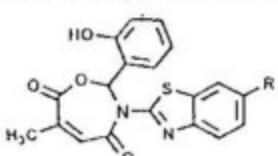
Compound	UV-Visible absorption maxima $\lambda/\text{nm}$ of oxazepine	Comp.	UV-Visible absorption maxima $\lambda/\text{nm}$ of amide - pyrrolidides
7-3	320,306,266,230,221	8-1-5-6	329,264,245,221
8-3	315,255,243,229	8-2-5-6	319,258,238,223
9-3	333,265,251,243,223	9-3-5-6	320,255,238,220
10-3	325,278,239,224	10-4-5-6	315,267,240,226
11-3	329,269,241,236,222	11-5-5-6	314,262,242,228
12-3	335,300,265,237,220	12-6-5	309,266,240,222

Table (10): Melting point, percentage yield, molecular formula and elemental analysis of 2-(2-hydroxy-phenyl)-  
benzothiazol-2-yl)-2,3-dihydro-[1,3]oxazepine-4,7-dione.



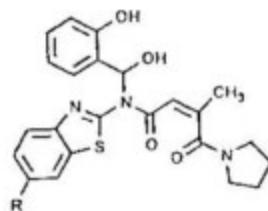
Compound	R	M.P./C°	Yield %	M.F.	Calc.			Found		
					C	H	N	C	H	N
7-3	CH <sub>3</sub>	205	77	C <sub>13</sub> H <sub>11</sub> N <sub>2</sub> O <sub>2</sub> S	63.14	4.24	7.36	63.12	4.30	7.11
8-3	(CH <sub>3</sub> ) <sub>2</sub>	237	75	C <sub>15</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> S	63.91	4.60	7.10	64.02	4.53	7.11
9-3	NO <sub>2</sub>	218	69	C <sub>13</sub> H <sub>10</sub> N <sub>2</sub> O <sub>2</sub> S	55.47	3.19	10.21	55.53	3.21	10.30
10-3	OCH <sub>3</sub>	193	58	C <sub>20</sub> H <sub>16</sub> N <sub>2</sub> O <sub>2</sub> S	60.00	4.07	7.07	60.65	4.12	7.00
11-3	Cl	199	66	C <sub>13</sub> H <sub>11</sub> N <sub>2</sub> O <sub>2</sub> SCl	56.93	3.27	6.99	57.01	3.33	7.05
12-3	Br	201	61	C <sub>13</sub> H <sub>11</sub> N <sub>2</sub> O <sub>2</sub> SBr	51.25	2.94	6.29	51.35	2.88	6.19

Table (11): The major IR absorptions ( $\text{cm}^{-1}$ ) of 2-(2-hydroxy-phenyl)-6-methyl-3-(6-R-benzothiazol-2-yl)-  
-2,3-dihydro-[1,3]oxazepine-4,7-dione



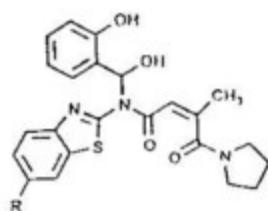
Compound	C-H str. Benzylic	O-H str. Phenol	C-H str. Olefine	C=O str. Lacton,lactum	C=C str. Olefine	C=C str. Aromatic	C=N str.	C-O str. lacton	C-H bend Aromatic	C-S str.	Others
7-3	3220	3430	3130	1680	1600	1380,1530	1440	1330	1030,880	1220	
8-3	3210	3400	3180	1670	1610	1575,1560	1435	1320	1040,870	1225	
9-3	3200	3420	3130	1670	1610	1570,1550	1440	1320	1030,920	1230	1360,1550 NO <sub>2</sub>
10-3	3200	3450	3150	1665	1600	1580,1560	1450	1330	1030,900	1220	1210 C=O-C
11-3	3210	3430	3160	1675	1600	1570,1540	1440	1335	1040,830	1230	760 Cl
12-3	3200	3420	3130	1670	1610	1580,1530	1450	1320	1040,860	1235	660 Br

Table( 12 ) melting points,percentage yield, molecular formula and elemental analysis of 3-methyl-4-Oxo-4-pyrrolidine-1-yl-but-2- enoic acid (6-R- benzothiazol-2-yl)-[hydroxy-(2-hydroxy-phenyl)-methyl]-amide.



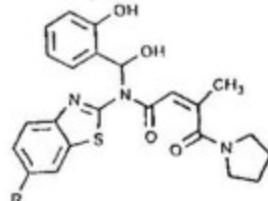
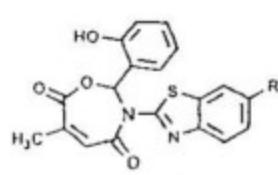
Compound	R	M.P/°C	Yield%	M.F	Calc.			Found		
					C	H	N	C	H	N
7-5	CH <sub>3</sub>	211	68	C <sub>19</sub> H <sub>22</sub> N <sub>2</sub> O <sub>3</sub> S	63.84	5.58	9.31	63.71	5.66	9.28
8-5	(CH <sub>3</sub> ) <sub>2</sub>	219	60	C <sub>21</sub> H <sub>24</sub> N <sub>2</sub> O <sub>3</sub> S	64.50	5.85	9.03	64.48	5.90	9.11
9-5	NO <sub>2</sub>	244	63	C <sub>19</sub> H <sub>22</sub> N <sub>2</sub> O <sub>3</sub> S	57.25	4.60	11.61	57.36	4.58	11.60
10-5	OCH <sub>3</sub>	214	55	C <sub>19</sub> H <sub>22</sub> N <sub>2</sub> O <sub>3</sub> S	61.65	5.39	8.99	61.69	5.30	8.95
11-5	Cl	215	61	C <sub>19</sub> H <sub>22</sub> N <sub>2</sub> O <sub>3</sub> SCl	58.53	4.70	8.90	58.56	4.78	8.82
12-5	Br	217	67	C <sub>19</sub> H <sub>22</sub> N <sub>2</sub> O <sub>3</sub> SBr	53.49	4.29	8.14	53.52	4.33	8.09

Table (13): The major IR absorptions (cm<sup>-1</sup>) of 3-methyl-4-Oxo-4-pyrrolidine-1-yl-but-2-enoic acid (6-R- benzothiazol-2-yl)-[hydroxy-(2-hydroxy-phenyl)-methyl]-amide.



Compound	O-H str. Phenol	C-H str. Olefine	C-H str. Aromatic	C=O str. amide	C=C str. Olefine	C=C str. Aromatic	C=N str.	C-O Alcohol	C-O Alcohol	Others
7-5	3350	3130	3060	1670	1690	1560,1490	1435	1380	1380	
8-5	3345	3160	3030	1675	1610	1580,1540,1470	1450	1370	1370	
9-5 <sup>a,b</sup>	3365	3175	3080	1660	1610	1580,1545,1490	1420	1390	1390	1360,1560 N(=O)
10-5	3320	3140	3050	1645	1600	1570,1520,1480	1435	1380	1380	1220 C=C-C
11-5	3340	3170	3060	1655	1690	1570,1530,1470	1440	1380	1380	730 C=C-I
12-5	3350	3175	3065	1650	1610	1580,1540	1440	1370	1370	650 C-Br

Table ( 14 ): <sup>1</sup>H,N.M.R spectrophotometry of 2-(2-hydroxy-phenyl)-6-methyl-3-(6-R-benzothiazol-2-yl)-2,3-dihydro-[1,3]oxazepine-4,7-dione and 3-methyl-4-Oxo-4-pyrrolidine-1-yl-but-2-enoic acid (6-R- benzothiazol-2-yl)-[hydroxy-(2-hydroxy-phenyl)-methyl]-amide.



\* Chemical shift =δ

\*\* By using DMSO-d<sub>6</sub>as solvent

Comp.	H-R	H-C=C-CH <sub>3</sub>	O-H phenol	O-H Alcohol	H-C Aromatic	Pyrrolidine ring			
						H <sub>2</sub>	H <sub>3</sub>	H <sub>4</sub>	H <sub>5</sub>
7-3 <sup>a,c</sup>	2.3	1.8	6.7	5.0	...	6.5-8.1	...	...	...
8-3 <sup>a,c</sup>	2.42.4	1.9	6.8	4.9	...	6.5-7.9	...	...	...
9-10-3 <sup>a,c</sup>	3.7	1.8	6.75	4.9	...	6.5-8.0	...	...	...
7-5 <sup>a,c</sup>	2.4	1.85	6.7	5.0	2.0	6.5-7.9	3.36	1.7	3.4
8-5 <sup>a,c</sup>	2.3,2.3	1.8	6.9	4.85	1.95	6.5-8.0	3.5	1.75	3.5
10-5 <sup>a,c</sup>	3.7	1.75	6.8	5.0	2.0	6.5-8.1	3.4	1.66	3.4

Table ( 15 ): The UV-Vissibl absorption maxima  $\lambda/\text{nm}$  of 2-(2-hydroxy-phenyl)-6-methyl-3-(6-R-benzothiazol-2-yl)-2,3-dihydro-[1,3]oxazepine-4,7-dione and3-methyl-4-Oxo-4-pyrrolidine-1-yl-1-but-2-enoic acid (6-R-benzothiazol-2-yl)-[hydrosy-(2-hydroxy-phenyl)- amide.

compound	UV-Vissibl absorption maxima $\lambda/\text{nm}$ of oxazepine	Comp.	UV-Vissibl absorption maxima $\lambda/\text{nm}$ of anilid - pyrrolidides
7- <sup>a</sup> -7-3-10-12	340,300,240,225	6-R-7-3-4-10	299,252,239,226
9- <sup>a</sup> -2-8-3-8-12	345,298,256,238,222	7-7-8-3-8-12	295,246,234,225
7- <sup>a</sup> -9-3-1-2-2	356,310,276,236,221	7-9-8-7-1	310,243,239,221
7-10-3-2	350,300,266,246,230	7-2-10-5-7-1	305,244,236,220
7-11-3-2	352,315,279,252,231	7-11-5-7-1	311,253,240,228
7-12-3-4-2	346,300,287,255,226	7-6-12-5-7-1	285,243,230,227

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تحضير ودراسة الصفات التركيبية (٢-(هيدروكسي-فنيل)-٣-٦-بنزوثيريازول-٢-يل)-٣-ثنائي هيدرو-١ او ٣ اوكسازبين-٤ و ٧-دابون و ٢-(٢-هيدروكسي-فنيل)-٦-مثيل-٣-(٦-معوض-بنزوثيريازول-٢-يل)-٣-ثنائي هيدرو-١ او ٣ اوكسازبين-٤ و ٧-دابون [١]

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قسم الكيمياء-كلية التربية للبنات-جامعة الانبار

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