



## Synthesis of some thiazole and phthalazine compounds from schiff bases

Mohammad S Al-Ajely\* and Azhar H Ali

Chemistry Department, College of Education, Mosul University, Mosul, Iraq

\*Corresponding Author: Mohammad S Al-Ajely, Chemistry Department, College of Education, Mosul University, Mosul, Iraq.

Received: May 20, 2019; Published: July 12, 2019

DOI: 10.31080/ASMS.2019.03.0352

### Abstract

It was known that heterocyclic compounds containing S and N have considerable importance which withdraw researchers attention due to their wide medical and biological applications. In the present investigation we have synthesized some oxadiazole derivatives ( $A_{9-15}$ ), phthalazine derivatives ( $A_{33-38}$ ) and Benzo thiazole-2-yl arylthiazol-4-one ( $A_{17-24}$ ), thiazoles ( $A_{39-41}$ ) from schiff base precursors. The thiazole compounds were cyclized using acetic anhydride, thioglycolic acid while phthalazines were cyclized using amylalcohol in HCl. The synthesized compounds were studied by IR, <sup>1</sup>HNMR spectral methods.

**Keywords:** Thiazole; Phthalazine; Schiffbases

### Introduction

Schiff base compounds achieved considerable importance due to their biological and medicinal applications [1]. Some thiazoles have prepared from schiff bases these compounds showed antibacterial activities [2]. It was found that conversion of some biologically active compounds into schiff bases will increase their effects for example isonicotinic hydrazone was found more active and more safer than the corresponding hydrerazieds [3]. Some schiff bases prepared from aromatic aldehydes and aromatic amines were found to be used as anti inflammatory agents during the last decades [4], and many other medical applications [4,5] while 1,3,4-oxadiazoles for example were extensively studied for the last decades especially in pharmaceutical aspects and other applications [5,6]. Oxadiazoles have been synthesized by numerous methods for example the compound 2-(4-(4-bromophenyl(sulfonylphenyl)-5-fluorobenzyl)1,3,4oxazole was synthesized from the reaction of <sup>1</sup>N-4-bromophenyl sulfonyl benzyl-<sup>4</sup>N-(4- fluorophenyl) thio simicarbazide with iodine or KI in ethanol. Chepa and coworkers have synthesized 1,3,4-oxadiazole derivatives [8]. Other researchers [9] have prepared oxazoles from the reaction of amino acid ester hydrazides with benzoyl chloride and cyclized the intermediate with POCl<sub>3</sub>. 1,3,4-Oxadiazoles have also prepared from succinic anhydride and some hydrazides using POCl<sub>3</sub> for cyclization [10]. In 2012 Sheet and Mohammed [11] have succeeded in synthesizing 1,3,4-oxadiazoles from oxidation of hydrazones using PbO<sub>2</sub>. Some thiazole compounds were also found to have medical application [12,13]. There are a lot of methods in the literature for the prepara-

tion of thiazole compounds among them, the cyclization of thio simicarbazides using bromo acetic acid [14]. Some of researcher have succeeded in synthesizing thiazolidine-4-one from the reaction of hydrazones with thioglycolic acid [14-16]. phthalazine compounds have also showed many medical application [17,18]. These compounds were prepared by variety of methods among them which is the most common method is the condensation of phthalic ester or anhydride with hydrazine [19]. In 2008 Basheer and Ezzat have synthesized phthalazines by treatment of amino acid hydrazones with saturated solutions of HCl [20].

### Experimental

All melting points were measured by Electrothermal melting point apparatus. All the chemical compounds were supplied by BDH, Aldrich and Fulka. The IR measurements were performed using Infrared spectrophotometer, type Tensor 27 Bruker. <sup>1</sup>NMR spectra were measured (selected compounds in DMSO-d<sub>6</sub> solvent) using Bruker 400 MHz/ Gaziosmanpasa University (Turkey). Compound 1 was prepared according to the published procedure [21].

### Synthesis of (substituted phenyl, substituted benzoyl hydrazone) ( $A_{2-8}$ )

Phenyl hydrazide (0.01 mol.) or 3-Nitrophenyl hydrazide was dissolved in 25 ml of absolute ethanol. Aldehyde or its derivatives was then added. The reaction mixture was refluxed for 2 hours, cooled, stand for 3 hour at r.t. The p.p.t was filtered off and crystallized from ethanol, Physical and IR spectral data were shown in Tables 1,2.

Comp. No.	X	Y	Molecular Formula	m.p. (°C)	Yield (%)	Colour
A <sub>3</sub>	H	H	C <sub>14</sub> H <sub>12</sub> N <sub>2</sub> O	204-207*	71	white
A <sub>3</sub>	4-Cl	H	C <sub>14</sub> H <sub>11</sub> N <sub>2</sub> OCl	164-167*	80	white
A <sub>4</sub>	2-OH	H	C <sub>14</sub> H <sub>12</sub> N <sub>2</sub> O <sub>2</sub>	160-163	65	Yellowsh white
A <sub>5</sub>	H	3-NO <sub>2</sub>	C <sub>14</sub> H <sub>11</sub> N <sub>3</sub> O <sub>3</sub>	206-208	88	white
A <sub>6</sub>	4-N(CH <sub>3</sub> ) <sub>2</sub>	3-NO <sub>2</sub>	C <sub>16</sub> H <sub>16</sub> N <sub>4</sub> O <sub>3</sub>	218-220	93	red
A <sub>7</sub>	2-OH	3-NO <sub>2</sub>	C <sub>14</sub> H <sub>11</sub> N <sub>3</sub> O <sub>4</sub>	240-244	87	Yellowsh white
A <sub>8</sub>	4-Cl	3-NO <sub>2</sub>	C <sub>14</sub> H <sub>10</sub> N <sub>3</sub> O <sub>3</sub> Cl	147-148	91	yellow

Table 1: physical data for compounds(A<sub>2-8</sub>).

Comp. No.	X	Y	IR $\nu$ cm <sup>-1</sup> (KBr)			Others
			C≡C C=N, (Ar)	C=O amide	N-H	
A <sub>2</sub>	H	H	1600 1531,1446	1642	3205	
A <sub>3</sub>	4-Cl	H	1604 1579,1488	1646	3219	690 (C-Cl)
A <sub>4</sub>	2-OH	H	1622 1609,1488	1673	3271	3443 (OH)
A <sub>5</sub>	H	3-NO <sub>2</sub>	1618 1573,1486	1664	3166	1346 sy (NO <sub>2</sub> ) 1536 asy (NO <sub>2</sub> )
A <sub>6</sub>	4-N(CH <sub>3</sub> ) <sub>2</sub>	3-NO <sub>2</sub>	1640 1614,1481	1656	3238	1364 sy (NO <sub>2</sub> ) 1524 asy (NO <sub>2</sub> )
A <sub>7</sub>	2-OH	3-NO <sub>2</sub>	1612 1575,1488	1650	3228	1347 sy (NO <sub>2</sub> ) 1525 asy (NO <sub>2</sub> )
A <sub>8</sub>	4-Cl	3-NO <sub>2</sub>	1622 1592,1474	1672	3298	751 (C-Cl) 1340 sy (NO <sub>2</sub> ) 1524 asy (NO <sub>2</sub> )

Table 2: IR. spectral data for compounds (A<sub>2-8</sub>).

### Synthesis of 2,5-diaryl-3-acetyl-1,3,4-oxadiazoles (A<sub>9-15</sub>)

Compound (A<sub>2-8</sub>), (0.003 mol) and 5 ml of acetic anhydride were mixed and refluxed for 2 hours, cooled and 50 gm. Of crushed ice was then added. The mixture was left of r.t for 24 hours. The solid precipitate was filtered and washed with water, dried at r.t. and crystallized from water. The physical properties and IR spectral data shown in Table 3,4.

2-Amino benzo thiazole (0.01 mol) was dissolved in 20 ml of abs. ethanol, substituted aldehydes (0.01 mol) was then added, 2 drops of glacial acetic acid. The reaction mixture was refluxed for 3 hours after completion of the reaction (TLC), the mixture was cooled, water was then added, the solid product was filtered off and crystallized from ethanol, the physical and spectral data were illustrated in Table 5,6.

Comp. No.	X	Y	Molecular Formula	m.p. (°C)	Yield (%)	Colour
A <sub>9</sub>	H	H	C <sub>16</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub>	61-62	58	white
A <sub>10</sub>	4-Cl	H	C <sub>16</sub> H <sub>13</sub> N <sub>2</sub> O <sub>2</sub> Cl	205-208	73	Yellowish white
A <sub>11</sub>	2-OH	H	C <sub>16</sub> H <sub>14</sub> N <sub>2</sub> O <sub>3</sub>	187-190	64	Yellowish white
A <sub>12</sub>	H	3-NO <sub>2</sub>	C <sub>16</sub> H <sub>13</sub> N <sub>3</sub> O <sub>4</sub>	100-102	77	Yellowish white
A <sub>13</sub>	4-N(CH <sub>3</sub> ) <sub>2</sub>	3-NO <sub>2</sub>	C <sub>18</sub> H <sub>18</sub> N <sub>4</sub> O <sub>4</sub>	98-101	86	Yellowish green
A <sub>14</sub>	2-OH	3-NO <sub>2</sub>	C <sub>16</sub> H <sub>13</sub> N <sub>3</sub> O <sub>5</sub>	126-129	69	Yellow
A <sub>15</sub>	4-Cl	3-NO <sub>2</sub>	C <sub>16</sub> H <sub>12</sub> N <sub>3</sub> O <sub>4</sub> Cl	95-99	72	yellow

Table 3: physical data for compounds(A<sub>9-15</sub>).

Comp. No.	X	Y	IR $\nu$ $\text{cm}^{-1}$ (KBr)					Other
			N-N	C-O-C		C=N, C $\equiv$ C (Ar)	C=O amide	
				Sym.	Asym.			
A <sub>9</sub>	H	H	1025	1063	1216	1625 1579,1449	1665	
A <sub>10</sub>	4-Cl	H	1011	1168	1293	1624 1592,1485	1651	813 (C-Cl)
A <sub>11</sub>	2-OH	H	1018	1063	1259	1624 1574,1489	1664	3446 (OH)
A <sub>12</sub>	H	3-NO <sub>2</sub>	1038	1055	1217	1628 1533,1487	1664	1348 sy (NO <sub>2</sub> ) 1533 asy (NO <sub>2</sub> )
A <sub>13</sub>	4-N(CH <sub>3</sub> ) <sub>2</sub>	3-NO <sub>2</sub>	1067	1148	1287	1595 1534,1439	1718	1173 (C-N) 1346 sy (NO <sub>2</sub> ) 1534 asy (NO <sub>2</sub> )
A <sub>14</sub>	2-OH	3-NO <sub>2</sub>	1012	1055	1261	1634-, 1491	1661	1350 sy (NO <sub>2</sub> ) 1532 asy (NO <sub>2</sub> )
A <sub>15</sub>	4-Cl	3-NO <sub>2</sub>	1018	1110	1221	1649 1607,1483	1734	743 (C-Cl) 1348 sy (NO <sub>2</sub> ) 1533 asy (NO <sub>2</sub> )

**Table 4:** IR. spectral data for compounds(A<sub>9-15</sub>)  
Synthesis of Arylidenyl 2—benzo-1,3- thiazolyl amine (A<sub>16-22</sub>).

Comp. No.	X	Molecular Formula	m.p. (°C)	Yield (%)	Colour
A <sub>16</sub>	H	C <sub>14</sub> H <sub>10</sub> N <sub>2</sub> S	92-94	74	أبيض مصفر
A <sub>17</sub>	4-N(CH <sub>3</sub> ) <sub>2</sub>	C <sub>16</sub> H <sub>15</sub> N <sub>3</sub> S	56-58	83	Deep yellow
A <sub>18</sub>	4-NO <sub>2</sub>	C <sub>14</sub> H <sub>9</sub> N <sub>3</sub> O <sub>2</sub> S	86-90	92	Yellowish white
A <sub>19</sub>	4-OCH <sub>3</sub>	C <sub>15</sub> H <sub>12</sub> N <sub>2</sub> OS	84-86	61	Yellowish green
A <sub>20</sub>	4-Cl	C <sub>14</sub> H <sub>9</sub> N <sub>2</sub> SCl	87-88	87	orange
A <sub>21</sub>	2-OH	C <sub>14</sub> H <sub>10</sub> N <sub>2</sub> OS	99-103	80	Yellowsh white
A <sub>22</sub>	2,6-diCl	C <sub>14</sub> H <sub>8</sub> N <sub>2</sub> SCl <sub>2</sub>	127-129	76	Yellow

**Table 5:** physical data for compounds(A<sub>16-22</sub>).

Comp. No.	X	IR $\nu$ $\text{cm}^{-1}$ (KBr)		Others
		C $\equiv$ C	C=C, Ar	
A <sub>16</sub>	H	1606,1445	1642	
A <sub>17</sub>	4-N(CH <sub>3</sub> ) <sub>2</sub>	1596,1487	1650	1165 (C-N)
A <sub>18</sub>	4-NO <sub>2</sub>	1596,1491	1608	1351 sy (NO <sub>2</sub> ) 1524 asy (NO <sub>2</sub> )
A <sub>19</sub>	4-OCH <sub>3</sub>	1569,1488	1601	1023 sy (C-O-C) 1253 asy (C-O-C)
A <sub>20</sub>	4-Cl	1594,1446	1603	729 (C-Cl)
A <sub>21</sub>	2-OH	1600,1448	1638	3398 (OH)
A <sub>22</sub>	2,6-diCl	1553,1435	1601	753,776 (C-Cl)

**Table 6:** IR. spectral data for compounds(A<sub>16-22</sub>).

#### Synthesis of 3-(benzothiazol-2-yl)-2- Arylthiazol -4-one (A<sub>17-24</sub>)

A compound of schiff base A<sub>16-22</sub> (0.002 mol) was mixed with (0.002) mol of thioglycolic acid in abs-ethanol (25ml), (0.136 mol) of ZnCl<sub>2</sub> anhydrous ZnCl<sub>2</sub> was then added. The reaction mixture was resluffed for 8 hours, cooled, filtered and washed with 3% sodium bicarbonate then with water and crystallized from (Dioxone - water). physical and spectral data are presented in Table 7,8.

#### Synthesis of ethyl- N - benzoyl glycinate [22] (A<sub>25</sub>)

Dry HCL gas was prepared and passed through (80 ml) of absolute ethanol till saturation. To this solution was added (0.067 mol.) N-benzoyl glycine. The mixture was refluxed at 90°C for 2 hours under dry conditions. After completion of reaction (TLC monitoring). The hot solution was added to 150 ml of water, neutralized with 3% sodium carbonate, The solid product was filtered, dried at r.t and crystallized of 75%, published mp. Is 60.5°C.

Comp. No.	X	Molecular Formula	m.p. (°C)	Yield (%)	Colour
A <sub>17</sub>	H	C <sub>16</sub> H <sub>12</sub> N <sub>2</sub> OS <sub>2</sub>	dec.188	61	white
A <sub>19</sub>	4-N(CH <sub>3</sub> ) <sub>2</sub>	C <sub>18</sub> H <sub>17</sub> N <sub>3</sub> OS <sub>2</sub>	dec.232	66	orange
A <sub>20</sub>	4-NO <sub>2</sub>	C <sub>16</sub> H <sub>11</sub> N <sub>3</sub> O <sub>3</sub> S <sub>2</sub>	191-193	70	Deep white
A <sub>21</sub>	4-OCH <sub>3</sub>	C <sub>17</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	163-165	52	white
A <sub>22</sub>	4-Cl	C <sub>16</sub> H <sub>11</sub> N <sub>2</sub> OS <sub>2</sub> Cl	178-181	64	Yellowsh white
A <sub>23</sub>	2-OH	C <sub>16</sub> H <sub>12</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	265-267	58	white
A <sub>24</sub>	2,6-diCl	C <sub>16</sub> H <sub>10</sub> N <sub>2</sub> OS <sub>2</sub> Cl <sub>2</sub>	231-233	60	white

**Table 7:** Physical data for compounds(A<sub>17-24</sub>).

Comp. No.	X	IR $\nu$ cm <sup>-1</sup> (KBr)				Others
		C-S-C	C-N	C=C C=C, Ar	C=O	
A <sub>18</sub>	H	757	1219	1572,1456	1665	
A <sub>19</sub>	4-N(CH <sub>3</sub> ) <sub>2</sub>	755	1223	1613,1504	1684	697 (C-Cl)
A <sub>20</sub>	4-NO <sub>2</sub>	762	1266	1595,1456	1711	1345 sy (NO <sub>2</sub> ) 1519 asy (NO <sub>2</sub> )
A <sub>21</sub>	4-OCH <sub>3</sub>	826	1278	1612,1440	1685	1032 sy (C-O-C) 1254 asy (C-O-C)
A <sub>21</sub>	4-Cl	758	1287	1593,1446	1636	731 (Cl)
A <sub>23</sub>	2-OH	754	1277	1601,1445	1708	3400(OH)
A <sub>24</sub>	2,6-Cl	809	1274	1578,1437	1704	753,777 (C-Cl)

**Table 8:** IR. spectral data for compounds(A<sub>18-24</sub>).

#### Synthesis of N- benzoyl glycy hydrazides [23] (A<sub>26</sub>)

Compound 25 (0.018 mol) and hydrazine hydrate (99% 0.09 mol) in 100 ml. Of abs. Ethanol were mixed and refluxed for 2 hours, cooled and excess solvent was distilled under reduced pressure. The white ppt. Was recrystallized from ethanol, mp=54-156°C, 70% yield, published mp=62.5°C.

#### Synthesis of substituted benzaldehyde N- benzoyl glycy hydrazones (A<sub>27-32</sub>)

Equi molar amounts of compound (26) and substituted aldehyde (0.005 mol.) were dissolved in 20 ml. Of ethanol (abs). The mixture was refluxed for 2 hours, cooled, The solid product was crystallized from ethanol, physical and spectral data are presented in Table 9,10.

Comp. No.	X	Molecular formula	m.p. (°C)	Yield (%)	Colour
A <sub>27</sub>	H	C <sub>16</sub> H <sub>15</sub> N <sub>3</sub> O <sub>2</sub>	181-184	87	white
A <sub>28</sub>	4-N(CH <sub>3</sub> ) <sub>2</sub>	C <sub>18</sub> H <sub>20</sub> N <sub>4</sub> O <sub>2</sub>	214-216	74	whitetoyellow
A <sub>29</sub>	4-NO <sub>2</sub>	C <sub>16</sub> H <sub>14</sub> N <sub>4</sub> O <sub>4</sub>	226-227	70	Yellowsh white
A <sub>30</sub>	4-Cl	C <sub>16</sub> H <sub>14</sub> N <sub>3</sub> O <sub>2</sub> Cl	212-214	65	white
A <sub>31</sub>	2-Cl	C <sub>16</sub> H <sub>14</sub> N <sub>3</sub> O <sub>2</sub> Cl	146-150	79	white
A <sub>32</sub>	3-NO <sub>2</sub>	C <sub>16</sub> H <sub>14</sub> N <sub>4</sub> O <sub>4</sub>	228-230	83	white

**Table 9:** physical data for compounds(A<sub>27-32</sub>).

Comp. No.	X	IR $\nu$ $\text{cm}^{-1}$ (KBr)				Others
		C=C, C $\equiv$ C Ar	C=N	C=O	N-H	
A <sub>27</sub>	H	1612,1489	1635	1685	3308	
A <sub>28</sub>	4-N(CH <sub>3</sub> ) <sub>2</sub>	1558,1495	1616	1676	3197	1180 (C-N)
A <sub>29</sub>	4-NO <sub>2</sub>	1570,1487	1602	1699	3362	1348 sy (NO <sub>2</sub> ) 1539 asy (NO <sub>2</sub> )
A <sub>30</sub>	4-Cl	1601,1489	1635	1685	3315	713 (C-Cl)
A <sub>31</sub>	2-Cl	1566,1467	1608	1685	3360	704 (C-Cl)
A <sub>32</sub>	3-NO <sub>2</sub>	1577,1487	1603	1691	3342	1348 sy (NO <sub>2</sub> ) 1523 asy (NO <sub>2</sub> )

**Table 10:** IR. spectral data for compounds(A<sub>27-32</sub>).

### Synthesis of 1-benzamideo methyl substituted phthalazines (A<sub>33-38</sub>)

A compound of (A<sub>27-32</sub>), 0.001 mol. Was dissolved in 10 ml. Of amyl alcohol saturated with dry HCL gas. The mixture was heated on steam bath for 1.5 hour and then refluxed for one hour. After completion of the reaction (TLC monitored), cooled and washed with 20% NaOH then with water and filtered. The solid product was crystallized from dioxane, physical and IR spectral data are presented in Table 11,12.

### Synthesis of 2-Aryl-3- (N- benzoyl glycy amido)-3-methyl thiazolidine -4-one (A<sub>39-41</sub>)

Equi molar amounts of compound (A<sub>33-38</sub>) and 2-mercapto propionic acid, 0.001 each were refluxed in 20 ml of absolute ethanol and 0.136 mol. Of ZnCl<sub>2</sub> (anhydrous) for 8 hours, cooled and washed with NaHCO<sub>3</sub> 3% then with water, filtered and the solid product was crystallized from ethanol, physical and IR spectral data were shown in Table 13,14.

Comp. No.	X	Molecular formula	m.p. (°C)	Yield (%)	Colour
A <sub>33</sub>	H	C <sub>16</sub> H <sub>13</sub> N <sub>3</sub> O	> 350	48	brown
A <sub>34</sub>	4-N(CH <sub>3</sub> ) <sub>2</sub>	C <sub>18</sub> H <sub>18</sub> N <sub>4</sub> O	dec.337	80	Yellowish green
A <sub>35</sub>	4-NO <sub>2</sub>	C <sub>16</sub> H <sub>12</sub> N <sub>4</sub> O <sub>3</sub>	307-308	66	yellow
A <sub>36</sub>	4-Cl	C <sub>16</sub> H <sub>12</sub> N <sub>3</sub> OCl	> 350	60	white
A <sub>37</sub>	2-Cl	C <sub>16</sub> H <sub>12</sub> N <sub>3</sub> OCl	> 350	52	white
A <sub>38</sub>	3-NO <sub>2</sub>	C <sub>16</sub> H <sub>12</sub> N <sub>4</sub> O <sub>3</sub>	193-195	71	Deep yellow

**Table 11:** physical data for compounds(A<sub>33-38</sub>).

Comp. No.	X	IR $\nu$ $\text{cm}^{-1}$ (KBr)			Others
		C=N	C=O	N-H	
A <sub>33</sub>	H	1614	1650	3417	
A <sub>34</sub>	4-N(CH <sub>3</sub> ) <sub>2</sub>	1604	1621	3406	1178 (C-N)
A <sub>35</sub>	4-NO <sub>2</sub>	1599	1599	3441	1346 sy (NO <sub>2</sub> ) 1519 asy (NO <sub>2</sub> )
A <sub>36</sub>	4-Cl	1600	1626	3385	815 (C-Cl)
A <sub>37</sub>	2-Cl	1614	1635	3383	873 (C-Cl)
A <sub>38</sub>	3-NO <sub>2</sub>	1630	1630	3437	1356 sy (NO <sub>2</sub> ) 1529 asy (NO <sub>2</sub> )

**Table 12:** IR. spectral data for compounds(A<sub>33-38</sub>).

Comp. No.	X	Molecular formula	m.p. (°C)	Yield (%)	Colour
A <sub>39</sub>	H	C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O <sub>3</sub> S	dec.327	72	yellow
A <sub>40</sub>	4-NO <sub>2</sub>	C <sub>19</sub> H <sub>18</sub> N <sub>4</sub> O <sub>5</sub> S	232-234	63	Yellowish white
A <sub>41</sub>	2-Cl	C <sub>19</sub> H <sub>18</sub> N <sub>3</sub> O <sub>3</sub> SCl	dec.352	56	white

**Table 13:** Physical data for compounds(A<sub>39-41</sub>).

Comp. No.	X	IR $\nu$ $\text{cm}^{-1}$ (KBr)				Others
		C-S-C	C=O amide	C=O lactam	N-H	
A <sub>39</sub>	H	854	1683	1695	3309	
A <sub>40</sub>	4-NO <sub>2</sub>	690	1708	1734	3373	1346 sy (NO <sub>2</sub> ) 1510 asy (NO <sub>2</sub> )
A <sub>41</sub>	2-Cl	752	1683	1712	3358	700 (C-Cl)

**Table 14:** IR. spectral data for compounds(A<sub>39-41</sub>).

## Results and Discussion

According to the previous studies heterocyclic compounds were found to have a wide range of medical and biological application [24-26] and are important for human life. For this reason we are here to discuss the preparation of new series of heterocyclic compounds derived from schiff bases.

### Synthesis of (substituted phenyl, substituted benzoyl hydrazones(A<sub>2-8</sub>))

The above compounds were synthesized from the condensation of phenyl hydrazine with some substituted benzaldehydes as mentioned in the experimental part. The IR spectra for the synthesized compounds were as follows: 1642-1673  $\text{cm}^{-1}$  for C=O amide, 1600-1640  $\text{cm}^{-1}$  for C=N stretching which sometimes appeared within the aromatic C=C region as shown in Table 2 while N-H stretching band appeared within, 3166-3299  $\text{cm}^{-1}$ .

### Synthesis of 2,5-dianyl-3-Acetyl-1,3,4-oxadiazole (A<sub>9-15</sub>)

Similar compounds have shown to have significant biological and pharmaceutical effects. These compounds were prepared by numerous methods as it was mentioned in the experimental part among them is the condensation of schiff base with acetic anhydride so we prepared these compounds using this method. The obtained compounds were characterized by the following IR absorption bands: 1651- 1734  $\text{cm}^{-1}$  for C=O and at 1595-1645  $\text{cm}^{-1}$  for C=N while C-O-C was appeared at 1055-168, 1216 -1293  $\text{cm}^{-1}$  for symm and assym. Stretching vibration. The other bands were indicated in Table 4 The <sup>1</sup>H NMR of compound(A<sub>9</sub>) showed the following signals; 1.918ppm. singlet for 3H of O=C-CH<sub>3</sub>, 2.509ppm. as singlet band related to 1H of O-C-H proton while the aromatic protons 10H appeared at 7.198- 8.479ppm.

### Synthesis of arylidene -2-benzo-1,3-thiadiazolene amine(A<sub>16-22</sub>)

The above compounds were prepared by condensation of 2-Amino benzothiazole with some substituted benzaldehydes as it was mentioned in the experimental part. These compounds were characterized by the absorption bands as indicated in Table 6: 1601 - 1650  $\text{cm}^{-1}$  for C=C aromatic in which sometimes appeared within the C=N absorption region.

### Synthesis of -3-(benzothiazol-2-yl)-2-aryl thiazoline -4- one (A<sub>17-24</sub>)

These compounds were prepared by the condensation of the schiff base (A<sub>9-15</sub>) with thioglycolic acid using anhydrous ZnCl<sub>2</sub>. The synthesized compounds were identified by IR and <sup>1</sup>H NMR, IR spectral data revealed the presence of the following absorption bands: 1636-1171  $\text{cm}^{-1}$  assigned for C=O stretch of the thiazolidone, 754 - 826  $\text{cm}^{-1}$  related for C-N stretch while C=C of the aromatic absorbed with the range of 1437-1613  $\text{cm}^{-1}$  as shown in Table 8. The <sup>1</sup>H NMR spectrum for compound(A<sub>20</sub>) showed the following resonating signals. 27ppm. for S-CH singlet, 4.01ppm belongs to 2H of CH<sub>2</sub> and doublet signed at 6.87, 6.899 for 2H Aromatic while the second signed of the aromatic proton appeared at 7.36, 7.346. Benzothiazole protons were found at 7.4-8.0ppm. Compound (23) showed resonating signal at 2.5ppm corresponds for 2H of CH<sub>2</sub> (cyclic) and the spectrum showed a multiplet signal at 7.31 - 7.43 belongs to 3H of ArH and finally a multiplet signal of 5.07-8.02ppm assigned for 4H of benzothiazole.

### Synthesis of Ethyl -n-benzoyl glycinate (A<sub>25</sub>)

This compound was prepared by esterification of N-benzoyl glycine, IR spectrum of this compound showed the following absorption bands: 1759  $\text{cm}^{-1}$  for C = O ester, 1641  $\text{cm}^{-1}$  for C = O Amide while C = C Aromatic appeared at 1493-1600  $\text{cm}^{-1}$  together with N-H at 3338  $\text{cm}^{-1}$ .

### Synthesis of N-benzoyl glyciyl hydrazides (A<sub>26</sub>)

The IR spectrum of the above compound revealed the formation of the hydrazide compound through the absorption of this group C=O at lower value and the presence of N-H band at 3194-3488  $\text{cm}^{-1}$  while the C=C appeared at 1491-1577  $\text{cm}^{-1}$ .

### Synthesis of N-benzoyl glyciyl hydrazones (A<sub>27-32</sub>)

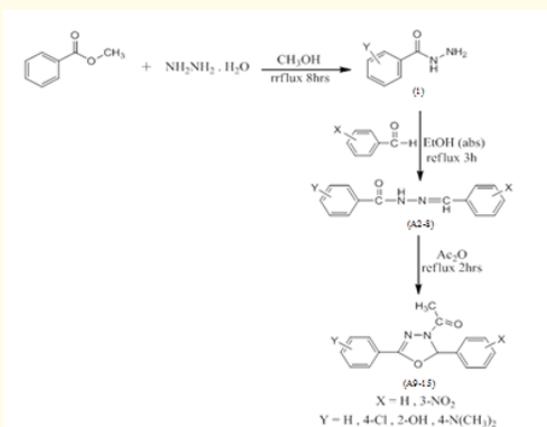
The above compounds were identified by IR studies which showed the following absorption bands; 1602-1635  $\text{cm}^{-1}$  for C=N, 1467 - 1621  $\text{cm}^{-1}$  for Aromatic C=C, 3197-3362  $\text{cm}^{-1}$  belongs to N-H while the C=O of the corresponding hydrazide appeared at 1676 - 1699  $\text{cm}^{-1}$  as indicated in Table 10.

### Synthesis of 1-Benzamido methyl substituted phthalazines (A<sub>33-38</sub>)

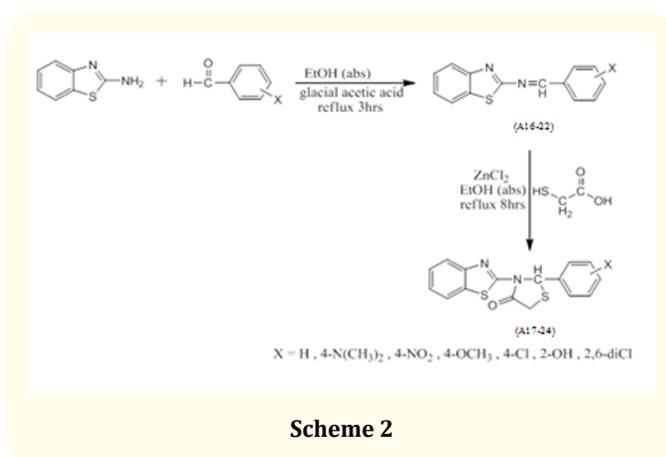
The series of compounds were appeared from the corresponding hydrazones with amyl alcohol in presence of HCL. IR spectrum of these compounds showed the following absorption bands: 1599-1630  $\text{cm}^{-1}$  for C=N, 1599-1650  $\text{cm}^{-1}$  belongs to the amide C=O while the NH appeared at 3383-3441  $\text{cm}^{-1}$  as shown in Table 12. The 1H NMR spectrum of compound [33] exhalted the following signals 8.44 ppm. for the diazine protons, 8.32 ppm for 5,8 protons of benzene ring, 8.23 ppm d of d for 6,7 protons of the benzene ring, 7.80-7.84 ppm for the phenyl ring protons adjacent to the carbonyl, NH at 5.84 ppm and 3.23 ppm singlet for the  $\text{CH}_2$ - protons.

### Synthesis of -2-Aryl-3-N-benzoylglyl amido -5-methyl thiazolidine-4-one (A<sub>39-41</sub>)

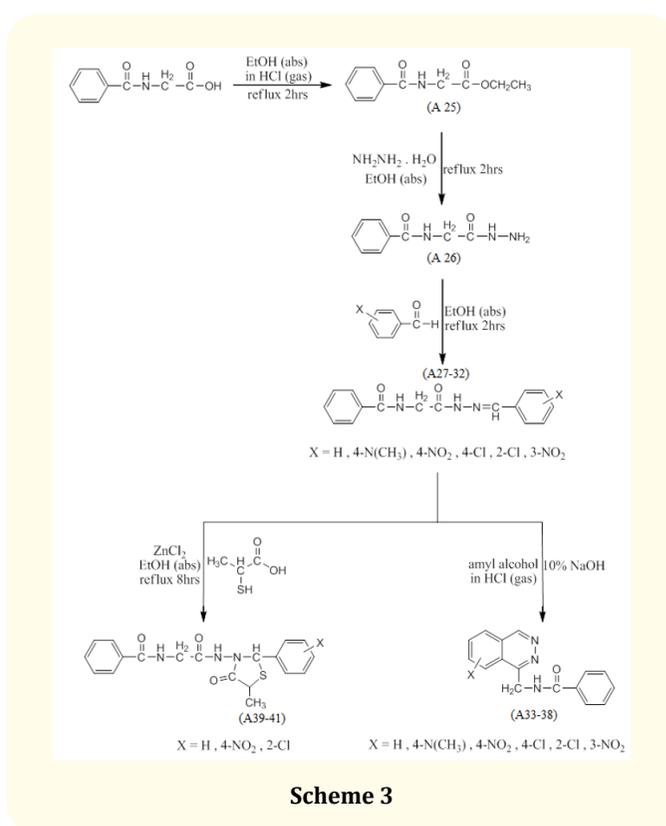
This series of compounds were prepared by the cyclization of the corresponding hydrazones using  $\text{ZnCl}_2$  in absolute ethanol the role of  $\text{ZnCl}_2$  is to catch the OH group of the thio glycolic acid and make the carbonyl of this acid available (more positive) for the nitrogen attack and cyclization. These compounds were identified by IR which showed the following absorbtion bands; 1695-1734  $\text{cm}^{-1}$  belongs to C=O of thiazole ring, C=O Amide at 1683-1708  $\text{cm}^{-1}$  while the N-H stretching appeared at 3309-3373  $\text{cm}^{-1}$  as indicated in Table 14.



Scheme 1



Scheme 2



Scheme 3

### Conclusion

According to the above results it was clear that the intermediate compounds were cyclized into the corresponding thiazoles, thiadiazoles and diazines.

## Acknowledgements

Authors would like to thanks the Iraqi ministry of higher Education and research for offering scholarship for Azhar Hussein Ali to do this work which is part of his MSc Thesis.

## Bibliography

- JR Shelton. US 2, 393, 271, 22,1946. *Chemical abstracts* 2273 (1946).
- J Salimon., et al. *Australian Journal of Basic and Applied Sciences* 4.7 (2010): 2016-2021.
- NP Buu-Hoi., et al. "Tuberculostatic hydrazides and their derivatives". *Journal of the Chemical Society* 1358 (1953).
- NA Salim. *Turkish Journal of Chemistry* 32 (2008): 229-235.
- EM Hodnett and WJ Dunn. "Structure-antitumor activity correlation of some Schiff bases". *Journal of Medicinal Chemistry* 13.4 (1970): 768-770.
- SC Naveena., et al. *Der Pharma Chemica* 3.6 (2011): 247-257.
- Shivi Bhatia and Monika Gupta. "Oxadiazole as antimicrobial agents: An overview". *Journal of Chemical and Pharmaceutical Research* 3.3 (2011): 137-147.
- C Cheptea., et al. *Cellulose Chemistry and Technology* 46.1-2 (2012): 19-25.
- JJ Kim., et al. "Synthesis and properties of photoluminescent polymers bearing electron-facilitating oxadiazole derivative side groups". *Journal of Polymer Science Part A* 40.8 (2002): 1173-1183.
- A Hussain and M Ajmal. "Synthesis of novel 1,3,4-oxadiazole derivatives and their biological propertie". *Acta Pharmaceutica* 59.2 (2009): 223-233.
- MA Sheat and OI Mohammed. *Journal of Education and Science* 25.1 (2012): 19-25.
- W Cunico., et al. "Mini-Reviews in Organic Chemistry". 5 (2008): 336-344.
- HF Roaiah., et al. *Nature and science* 8.7 (2010): 20-29.
- AZ Cesur., et al. *Acta Chimica Slovenica* 57 (2010): 355 - 362.
- BS Ramachandran., et al., *International Journal of Pharmaceutical Sciences and Research* 2.6 (2011): 1564-1568.
- S Ramachandran., et al. *World Journal of Pharmacy and Pharmaceutical Sciences* 2.1 (2013): 337-342.
- D Patel., et al. "Synthesis, characterization and biological evaluation of some thiazolidinone derivatives as antimicrobial agents". *Journal of Chemical and Pharmaceutical Research* 2.5 (2010): 84-91.
- M Napoletano., et al. "Phthalazine PDE4 inhibitors. Part 2: the synthesis and biological evaluation of 6-methoxy-1,4-disubstituted derivatives". *ACS Medicinal Chemistry Letters* 11.1 (2001): 33-37.
- AHF Abd El-Wahab., et al. *Bedair Pharmaceuticals* 4 (2011): 1158-1170.
- NR Patel., (Castle R.N. ed.), J Wiley. New York, 446 r Pharmaceuticals, 4 (2011): (1973): 1158-1170.
- HA and Basheer and NS Ezzat Tikrit *Journal of Pure Science* 13.1(2008): 166-170.
- R Saini., et al. *Journal of Chemical Research* 2.1 (2009).
- M Freifelder. *The Journal of Organic Chemistry* 31 (1966): 3875.
- HA Basheer, Ph.D.Thesis, University of Mosul. Mosul-Iraq, 56 (2000).
- N Herrera Cano., et al. Santiago AN *Journal of Agricultural and Food Chemistry* 63.14 (2015): 3681-3686.
- MS Al-Ajely and and IMShaban. *Journal of Addiction Research* 2.1 (2018).
- MS Al-Ajely. *Open Access Journal of Science* 3.2 (2019).

**Volume 3 Issue 8 August 2019**

**© All rights are reserved by Mohammad S Al-Ajely and Azhar H Ali.**