Online ISSN: 2664-2522



### Iraqi Journal of Pharmacy

Journal homepage: https://iphr.mosuljournals.com



Print ISSN: 1680-2594

Research Article:

## Synthesis and Characterization of New 1,3,4-Oxadiazole Derivatives from Sulfathiazole

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#### **Article Information**

#### Article history:

Received on: 14 April 2024 Revised on: 03 May 2024 Accepted on: 27 May 2024 Published on: 01 September 2024

#### Keywords:

Hydrazide, 1,3,4-Oxadiazole, Diazonium salt, Sulfathiazole, FT-IR spectrum

#### **Abstract**

**Background:** Oxadiazole are important heterocyclic compounds in organic chemistry. **Methods:** In this work, a series of new 1,3,4-oxadiazole moiety started from diazonium salt was synthesized. The latter (A1) was prepared form sulfathiazole compound by reacting it with sodium nitrite in an acidic medium. This was followed by its conversion to azide derivative through the reaction with sodium azide, resulting in the compound azide sulfathiazole (A2). The latter was utilized as a good starting material for the preparation of 1,2,3-triazole-4-carboxylic acid derivative (A3). The carboxylic acid moiety of which was converted to an ester derivative (A4) by reacting it with absolute ethanol in a concentrated sulfuric acid medium. The ester derivative was subsequently reacted with hydrazine hydrate to yield the hydrazide derivative (A5), which serves as the starting material for the preparation of oxadiazole compounds. The hydrazide (A5) reacts with the substituents benzoic acid under dry condition in the presence of POCl<sub>3</sub> to obtain the compounds 1,3,4-oxadiazole derivatives (A6-A12). **Conclusion:** The target compounds were obtained and confirmed by conducting an analytical study using FT-IR spectrophotometer.

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#### 1. Introduction

Oxadiazole are important heterocyclic compounds in organic chemistry, known as furodiazole, with a 5-mempering ring structure incorporating nitrogen and oxygen atoms. they have the molecular formula ( $C_2H_2N_2O$ ) and have been discovered since ancient times based on the position of

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#### How to cite:

Jassim, J., A., Nejres, A., M., Atiyah, L., G., Ibrahim, H., A., Hameed, Z., A., (2024). Synthesis and Characterization of New 1,3,4-Oxadiazole Derivatives from Sulfathiazole. Iraqi J. Pharm. 21(3), 128-135.

DOI: https://doi.org/10.33899/iraqij.p.2024.148824.1094

their two nitrogen atoms within the ring. 1,3,4-oxadiazole compounds have acquired wide range importance, especially due to their high activity in medical, pharmaceutical, biological, and organic synthetic fields [1]1,3,4-Oxadiazole derivativeshave been used in the treatment of a of a wide range of diseases such as cancer [2, 3], inflammation [4], depression [5], Alzheimer's disease [6], pulmonary tuberculosis [7], diabetes [8], blood pressure disorders [9], and as vasodilators for heart diseases [10]. Biologically, 1,3,4-oxadiazole derivatives have been using as antibacterial agents [11], antifungal agents [12], anti-viral agents [13], and anti-oxidants due to their inhibitory actions on oxidative enzymes in the humans body [14]. Industrially, the 1,3,4-oxadiazole ring has been used as building materials in the preparation of various types of polymers [15], as electron carriers in electrical circuits [16], and in the preparation of corrosions inhibitors [17]. In agriculture, 1,3,4-oxadiazole insecticides. [18]. The hydrazide undergoes nucleophilic-substitution reactions to form 1,3,4-oxadiazole derivatives by reacting with the substituent benzoic acid in the

derivatives have been used as effective presence of  $POCl_3$  as a cyclization agent . The steps for synthesizing 1,3,4-oxadiazole derivatives shown as below **scheme 1**.

**Scheme 1**. Synthesis of 1,3,4-oxadiazole derivatives

#### 2. Materials and methods

Starting material and solvents were purchased from BDH, Fluka, and Aldrich companies. The 1H-NMR spectra were recorded using a Bruker Bio Spin Gmb H spectrophotometer (400 MHz using DMSO- $d_6$  as a solvent) at the University of Gazzi Othman Basha, Turkey. The FT-IR spectra were recorded using the FT-IR spectrophotometer, and TLC (13181) silica gel with the fluorescent indicator (No. 6060) was used with the solvent system benzene: methanol in the ratio (8:2).

#### 2.1. Synthesis of sulfathiazole diazonium chloride [19] (A1)

The diazonium salt (A1) was prepared by mixing 0.007 mol (1.78 g) of sulfathiazole with 3 mL of concentrated

hydrochloric acid in an ice bath  $(0-5^{\circ}\text{C})$ . Then, a gradually added aqueous solution of sodium nitrite (0.007 mol / 0.48 g) and 10 mL of water with stirring. Stirring continues for 30 minutes, after which the formed salt is separated by filtration, dried, and the following physical constants were measured: Melting point =  $(213-211 \, ^{\circ}\text{C})$ , Percentage yield = (76%), Color = yellow.

#### 2.2. Synthesis of sulfathiazole azide [19] (A2):

An aqueous solution of sodium azide (0.007 mol / 0.45 g) is gradually added to an aqueous solution of diazonium salt (0.007 mol / 1.78 g) with continuous stirring for 20 minutes until the yellow color of the diazonium salt disappears. The reaction mixture is then allowed to settle

at room temperature for 10 minutes. The resulting precipitate is filtered, dried, and the following physical constants were obtained: Melting point = (173-171 °C) yield = 79%, purity and completion of the reaction are confirmed using Thin Layer Chromatography (TLC) with a retardation factor (Rf) value of 0.246 in a solvent system of benzene:methanol (2:8)., The following bands are observed (KBr).v(cm-1): NH(3368); N=N(2133); C=N(1537); SO<sub>2</sub>(asym 1330 and sym 1151); C-S(935).

## 2.3. Synthesis of 5-methyl-1-(4-(N-thiazol-2yl)sulfamoyl) phenyl)-1H-1,2,3-triazole-4-carboxylic acid [19] (A3):

The compound 1,2,3-triazole-4-carboxylic acid is prepared by dissolving sulfathiazole azide (A2) (0.006 mol / 1.68 g) in 30 mL of absolute ethanol, followed by the gradual addition of a solution consisting of 7 mL of sodium ethoxide and 0.8 mL of ethyl acetoacetate. The reaction mixture is heated in a water bath for 4 hours, then poured into a glass flask containing ice-cold water and neutralized using a few drops of concentrated hydrochloric acid to filter the resulting precipitate. The obtained crystals are recrystallized using absolute ethanol to yield compound A3 . The compound has the following physical properties: Molecular formula ( $C_{14}H_{13}O_4N_5S_2$ ), yield of 60%, melting point of 218-219 °C, and in TLC, an Rf value of 0.246 in a solvent system of benzene:methanol (2:8). and in the IR spectrum, the following bands are observed (KBr).v(cm-1): OH-(3509); NH-(3200); CH<sub>3</sub>-(asym 2959)and(sym 2934); C=O-(1779); C=N-(1603); C=C-(. 1456); C-O(1261); SO2(asym 1311) and (sym 1144); C-S(880).

# 2.4. Synthesis of ethyl 5-methyl-1-(4-(N-(thiazol-2-yl)sulfamoyl)phenyl)-1H-1,2,3-triazole-4-carboxylate[20] (A4):

To prepare compound A4, 0.013 mol (4.75 g) of the compound (A3) is dissolved with 30 mL of absolute ethanol, and 2 mL of  $\rm H_2SO_4$  is added as a catalyst. The reaction mixture is heated for 6 hours, then cooled and poured into a glass flask containing crushed ice-cold water and neutralized using a (10% solution of NaHCO<sub>3</sub>). The resulting precipitate is filtered, washed several times with water, dried, and the physical and spectral properties are measured. The compound has the following properties: Molecular formula ( $\rm C_{15}H_{15}O_4N_5S_2$ ), yield of 81%, melting point of 207-205 °C, and in TLC, an Rf value of 0.358 using a solvent system of benzene:methanol (2:8), and in the IR

spectrum, the following bands are observed (KBr).v(cm-1): NH-(3146); CH3-(asym 2911)and(sym 2807); C=O-(1707); C=C-(Cycl. 1593); C=N-( 1568); N=N-(1537); C-O-C-(asym1283)and(sym 1107); SO2-(asym 1319)&(sym 1152); C-S-(935).

## 2.5. Synthesis of 5-methyl-1-[4-(N-thiazole-2-yl) sulfamoyl]-1,2,3-triazole-hydrazide [20] (A5):

To prepare the hydrazide, equimolar amounts (0.005 mol) of the ester (A4) and hydrazine hydrate (80%) are mixed in a flask with 3 drops of triethylamine in 30 mL of ethanol. The reaction mixture is stirred until complete dissolution, then heated for 6 hours. After cooling, it is poured onto icecold water, neutralized with a few drops of concentrated hydrochloric acid, and the resulting precipitate is filtered. The obtained crystals are recrystallized using absolute ethanol to yield compound A5. The compound has the following physical and spectral properties: Molecular formula (C<sub>13</sub>H<sub>13</sub>O<sub>3</sub>N<sub>7</sub>S<sub>2</sub>), yield of 67%, melting point of 146-144 °C, and in TLC, an Rf value of 0.41 using a solvent system of benzene:methanol (2:8), and in the IR spectrum (KBr).v(cm-1): NH2-(3223): NH-(3111): CH3-(asym 2918) and (sym 2857); C=O-(1697); C=C(1618); C=N-(1564); N=N-(1528); SO<sub>2</sub>-(asym 1323)and(sym 1148); C-S(937).

# 2.6. Synthesis of 1-[4-(N-thiazole-2-yl)sulfamoyl phenyl]-4-(5-aryl-1,3,4-oxadiazole-2-yl)-1H-1,2,3-triazole[21] (A6-12):

Molar equivalents (0.005 mol) of the hydrazide (A5) and benzoic acid substitutes are mixed in a 100 mL glass conical flask equipped with an air condenser and a calcium chloride tube. To this mixture, 4 mL of phosphorus oxychloride (POCl<sub>3</sub>) is added, and the reaction mixture is heated for 4 hours. After cooling to room temperature, 20 mL of water is gradually added, and the mixture is poured into a glass flask containing sodium bicarbonate solution while using sunflower paper to indicate the complete of the reaction. The resulting precipitate is filtered, washing with petroleum ether (60-80), dried **scheme 2**, and the physical and spectral properties are measured, as shown in **Table** 

Scheme 2. General structure for oxadiazole derivatives compounds

Comp. No.	x	Yield (%)	M.P (°C)	Colour	Molecular formula	T.L.C Benzene:Methanol (8:2)
A <sub>6</sub>	Н	48	117-119	dark brown	C <sub>20</sub> H <sub>15</sub> O <sub>3</sub> N <sub>7</sub> S <sub>2</sub>	0.595
$A_7$	p-CH <sub>3</sub>	51	110-113	dark brown	C <sub>21</sub> H <sub>17</sub> O <sub>3</sub> N <sub>7</sub> S <sub>2</sub>	0.648
A <sub>8</sub>	o-OCH <sub>3</sub>	60	109-112	Yellow	C <sub>21</sub> H <sub>17</sub> O <sub>4</sub> N <sub>7</sub> S <sub>2</sub>	0.588
A <sub>9</sub>	P-C1	53	230-232	dark brown	C <sub>20</sub> H <sub>14</sub> O <sub>3</sub> Cl N <sub>7</sub> S <sub>2</sub>	0.637
A <sub>10</sub>	m-Cl	85	250-253*	Black	C <sub>20</sub> H <sub>14</sub> O <sub>3</sub> Cl N <sub>7</sub> S <sub>2</sub>	0.620
A <sub>11</sub>	p-NO <sub>2</sub>	78	213-216*	Brown	C <sub>20</sub> H <sub>14</sub> O <sub>5</sub> N <sub>8</sub> S <sub>2</sub>	0.612
A <sub>12</sub>	m-NO <sub>2</sub>	59	225-228	Brown	C <sub>20</sub> H <sub>14</sub> O <sub>5</sub> N <sub>8</sub> S <sub>2</sub>	0.598

**Table 1.** Physical properties of compounds(A6—12)

#### 3. Result and Discussion

The hydazide was prepared through several sequential steps starting with the synthesis of the diazonium salt by reacting sulfathiazole with sodium nitrite in the presence of concentrated hydrochloric acid at low temperature (0-5°C). The diazonium salt (A1) was then reacted with sodium azide at low temperature (5-0°C) in an aqueous medium to yield the azide derivative, sulfathiazole azide (A2), as illustrated in the above **Scheme 1**. The physical properties listed in the experimental section confirmed its formation. Additionally, infrared spectroscopy (FT-IR) spectrum Figure 1. showed distinctive peaks for the azide group at 2098 and 2133 cm-1, along with a weaker peak at 1175 cm-1, indicating the presence of the azide derivative. Thin-layer chromatography (TLC) was used to monitor the reaction by comparing the retardation factor (Rf) values of the diazonium salt and azide derivative, providing an indication of the purity of the synthesized compound, which would serve as a reactive precursor in the preparation of compound5-methyl-1-(4-N-thiazol-2-yl)sulfonamido)phenyl) --1H-1,2,3-triazole-4-carboxylic acid (A3) through its reaction with ethyl acetoacetate in a basic medium of sodium ethoxide Scheme 3.

The compound A3 formula was confirmed using spectroscopic methods, including FT-IR spectrum **Figure 2**, which exhibited distinctive peaks for the carboxyl groups at 3509 cm-1 and the carbonyl groups at 1778 cm-1, indicating its formation. Final confirmation of the compound A3 structure was obtained by measuring its 1H-NMR spectrum, which showed a highly distinctive peak for the acidic hydroxyl group at  $\delta$  ppm (s, 1H, 12.90), in addition to other characteristic peaks detailed in **Table 3**.

To prepare the hydrazide derivative (A5), compound A3 needed to be converted into the corresponding ester. Compound A3 underwent esterification by reacting it with absolute ethanol in an acidic medium of concentrated sulfuric acid, as depicted in above **Scheme 1**. The resulting ester (A4) exhibited distinctive peaks for the carbonyl group in the FT-IR spectrum Figure 3. at 1707 cm-1, along with other spectral values detailed in the experimental section. Compound A4 then underwent nucleophilic substitution reaction with hydrazine hydrate, facilitated by hydrochloric acid as an appropriate acidic catalyst, as shown in above Scheme 1. The formation of compound A5 was confirmed by its FT-IR spectrum Figure 4, which displayed distinctive absorption peaks for the primary amine groups (NH2) and the amide group (-CONH) at 3223 cm-1 and 3111 cm-1, respectively, as well as the carbonyl amide group at 1697 cm-1, in addition to other detailed peaks [22]. The 1H-NMR spectrum also showed distinctive peaks for the amine groups (NH2) and the amide group (-CONH) at δ ppm (s, 2H, 1.99) and (s, 1H, 2.61), respectively, further confirming the formation of the hydrazide derivative. Thin-layer chromatography (TLC) was used to monitor the reaction and compare the retardation factor (Rf) values, providing a clear indication of changes in the effective moieties of the compound. To prepare oxadiazole derivatives (A6-12),FT-IR given good evidence through disappearance primary amine (NH2) and amide groups (-CONH) and display band of (C-O-C) Table 2, The <sup>1</sup>H-NMR spectrum also showed disappearance of primary amine (NH2) and amide groups (-CONH) and appear A-B system due to para position in benzene ring.

<sup>\*=</sup> decompose

$$\begin{bmatrix} S \\ NH - S \\ 0 \end{bmatrix} - \begin{bmatrix} N \\ NH \end{bmatrix} = \begin{bmatrix} N \\ NH$$

**scheme 3.** General structure for oxadiazole derivatives compounds

 Table 2. Spectral data for compounds(A6--12)

Comp. No.	х	FT-IR ,ν(cm <sup>-1</sup> )								
		N-H	СН₃	C=N	C-O-C	SO <sub>2</sub>	C-S	Other		
<b>A</b> <sub>6</sub>	-H	3414	asym 2928 sym 2854	1564	asym 1427 sym 1172	asym 1377 sym 1103	785			
<b>A</b> <sub>7</sub>	p-CH <sub>3</sub>	3423	asym 2926 sym 2854	1568	asym 1427 sym 1174	asym 1379 sym 1107	759	asym 2926 sym 2854		
<b>A</b> <sub>8</sub>	o-OCH <sub>3</sub>	3431	asym 2928 sym 2850	1599	asym 1431 sym 1155	asym 1323 sym 1018	759	C-O-C asym 1290 sym 1090		
<b>A</b> 9	p-Cl	3433	asym 2922 sym 2854	1539	asym 1500 sym 1122	asym 1400 sym 1031	842	C-Cl 704		
<b>A</b> 10	m-Cl	3419	asym 2924 sym 2856	1531	asym 1429 sym 1124	asym 1350 sym 1078	844	C-Cl 713		
<b>A</b> 11	p-NO <sub>2</sub>	3414	asym 2924 sym 2854	1618	asym 1346 sym 1172	asym 1381 sym 1124	848	NO <sub>2</sub> asym 1525 sym 1284		

Table 3. The  $^1HNMR$  spectrum for compounds(A3,A5,A10 and A11)

Comp. No.	Structure	¹H-NMR , DMSO-d6, δ (ppm)
$\mathbf{A}_3$		<u>CH<sub>3</sub></u> (s,3H,3.24); <u>NH</u> SO <sub>2</sub> (s,1H,2.60); thiazole protons (d,1H, 6.91)&(d,1H,7.32); aromatic protons (AB system) (d-d,4H,7.82-8.06);OH (s,1H,12.9)
$\mathbf{A}_{5}$		$\frac{\text{NH}_2}{\text{NH}_2}$ (s,2H,2.94); NHCO-(s,1H,5.05); $\frac{\text{CH}_3}{\text{CH}_3}$ (s,3H,1.24); $\frac{\text{NH}}{\text{SO}_2}$ (s,1H,2.61); thiazole protons (d,1H,7.36)&(d,1H,7.37); aromatic protons (AB system) (d-d,4H,7.56-7.95)
${f A_{10}}$		CH <sub>3</sub> (s,3H,2.42); NHSO <sub>2</sub> (s,1H,2.61); thiazole protons (d,1H,7.36)&(d,1H,7.37); 2-aryl oxadiazole protons (m,4H,7.42-7.53); aromatic protons (AB system) (d-d,4H,7.90-7.96)
$\mathbf{A}_{11}$		$\underline{\text{CH}}_3$ (s,3H,2.60); $\underline{\text{NH}}\text{SO}_2(\text{s},1\text{H},2.61)$ ; thiazle protons (d,1H,7.33)&(d,1H,7.37); 2-aryl oxadiazole protons (AB system)(d-d,4H,7.58-7.89); aromatic (AB system) (d-d,4H,8.17-8.40)

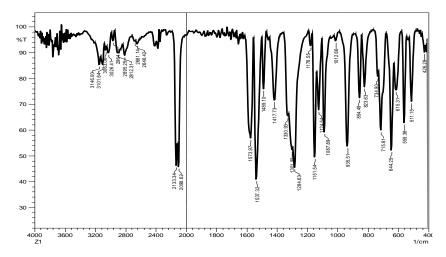


Figure 1. FT-IR Spectrum of A<sub>2</sub>

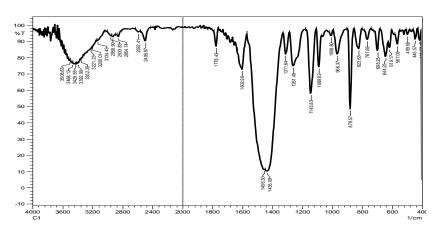


Figure 2. FT-IR Spectrum of A<sub>3</sub>

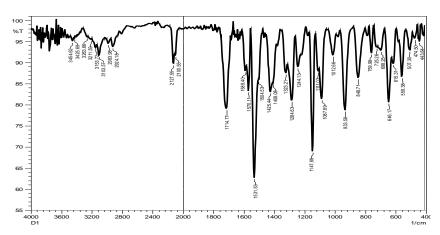


Figure 3. FT-IR Spectrum of  $A_4$ 

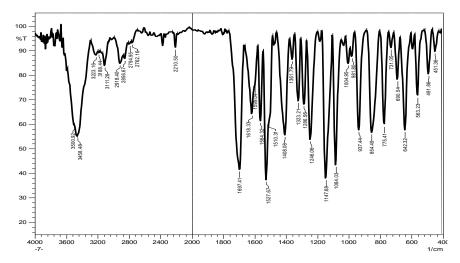


Figure 4. FT-IR Spectrum of A<sub>5</sub>

#### 4. Conclusion

This article summarizes the synthesis of 1,3,4- oxadiazole derivatives through conventional methods, starting with diazonium reactions, followed by azide cyclizations the formation of triazole containing a carboxyl group(A3), which is considered as an important fuctional group in organic chemistry and then synthesis of hydrazide from the ester and in final step the cyclization the hydrazide with substituent of benzoic acid in presence of POCl3 as cyclization agent for synthesis of 1,3,4-oxadiazole derivatives.

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#### تحضير وتوصيف مشتقات 1.3.4 -أوكساديازول الجديدة من السلفاتيازول

#### الخلاصة

المقدمة: الأوكساديازول من المركبات الحلقية غير المتجانسة المهمة في الكيمياء العضوية. الطرق: في هذا العمل تم تصنيع سلسلة جديدة من شاردة 1،3،4أوكساديازول بدأت من ملح الديازونيوم. تم تحضير الأخير (A1) من مركب السلفاثيازول بتفاعله مع نتريت الصوديوم في وسط حمضي. وتبع ذلك تحويله إلى مشتق الأزيد من خلال التفاعل مع أزيد الصوديوم، لينتج مركب أزيد سلفاثيازول (A2). تم استخدام الأخير كمادة بداية جيدة لتحضير مشتق حمض الكبريتيك تريازول-4-كربوكسيل (A3). تم تحويل جزء الحمض الكربوكسيلي منه إلى مشتق إستر (A4) عن طريق تفاعله مع الإيثانول المطلق في وسط حمض الكبريتيك المركز. تم بعد ذلك تفاعل مشتق الإستر مع هيدرازين هيدرات لإنتاج مشتق الهيدرازيد (A5)، الذي يعمل كمواد أولية لتحضير مركبات أوكساديازول. يتفاعل الهيدرازيد (A5) مع بدائله حمض البنزويك تحت الظروف الجافة بوجود POCI<sub>3</sub> للحصول على مركبات مشتقات 1,3,4-أوكساديازول (A6-A12). الاستنتاج: تم الحصول على المركبات المستهدفة وتأكيدها من خلال إجراء دراسة تحليلية باستخدام جهاز المطياف الضوئي FT-IR.

الكلمات المفتاحية: هيدر ازيد، 4،3،1 أو كساديازول، ملح الديازونيوم، سلفاثيازول، طيف الاشعة تحت الحمراء