

## Synthesis and Characterization of Some New Hydrazides and Their Derivatives

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### Abstract

New hydrazide compounds (A<sub>2</sub>) and (A<sub>9</sub>) were prepared from their corresponding esters (A<sub>1</sub>, A<sub>8</sub>). These esters were also prepared from their precursors 5-ethoxy carbonyl-(4-methoxyphenyl)-6-methyl-1,2,3,4-tetrahydropyrimidine-2-thione (A<sub>1</sub>) via multicomponent reaction type and from hippuric acid respectively. The hydrazide compounds were then allowed to react with some aldehydes forming the corresponding hydrazones (A<sub>3-7</sub>) and (A<sub>10-14</sub>). The synthesized compounds were characterized by IR, <sup>1</sup>H-NMR, <sup>13</sup>C-NMR, and Mass spectroscopies and well discussed. .

**Keywords:** Ester, Hydrazide, Hydrazone, Schiff base

## Introduction

Hydrazides are known to include an essential category of biologically active organic compounds [1-5]. These hydrazides and their condensation products were stated to possess a wide range of biological activities as well as antibacterial activity [1], tuberculostatic properties [2], HIV inhibitors [3], pesticidal [4], antifungal [5]. Some of the hydrazides and analogous hydrazones are psychopharmacological agent such as monoamine oxidase (MAO) inhibitor and serotonin antagonists [6].

Hydrazide-hydrazones compounds are not only intermediates but they are likewise very effective organic compounds in their own right. When they are used as intermediates, new derivatives can be synthesized by using the active hydrogen component of  $-\text{CONHN}=\text{CH}-$  azometine group [7-9].

A variety of methods have been used to prepare hydrazides, some of them involve the reaction of acids or their derivatives with hydrazine hydrate, anhydrides and acid chlorides, although the more popular method for the preparation of hydrazides achieved by the hydrazinolysis [10]. Hydrazone compounds as it was mentioned above due to their azo methane group activities have drawn the attention of many researchers in synthesizing numerous compounds of hydrazones by the well-known hydrazinolysis method [11,12]. These hydrazones, likewise the corresponding hydrazides, found a variety of applications in health and medicine [13, 14].

## Experimental

All reagents and chemicals were from BHD and Sigma-Aldrich companies and used without further purification. Melting points were determined with Electro thermal 9300 melting point apparatus. Infrared spectra were recorded on a Perkin-Elmer 1600 FTIR.  $^1\text{H-NMR}$ ,  $^{13}\text{C-NMR}$  spectra were recorded at 250 or 400 MHz on a Bruker AV-1400 model or Bruker AV-1250 model NMR instrument by using  $\text{DMSO-d}_6$  as a solvent. Accurate masses were obtained using a Water-Micromass LCT electrospray mass spectrometer. All the measurements were performed at the Chemistry Department, University of Sheffield, Sheffield, United Kingdom.

### Synthesis of 5-ethoxycarbonyl-4-(methoxyphenyl)-6-methyl-1,2,3,4-tetrahydropyrimidine-2-thione ( $\text{A}_1$ ) [15]:

A mixture of 4-methoxy benzaldehyde (1 mol.), ethyl acetoacetate (1 mol.), thiourea (1 mole), and some drops of sulfuric acid were heated at  $90^\circ\text{C}$  in a water bath. After completion, the mixture (monitored by TLC) was dissolved in ethanol and poured into cold water. The resulting precipitate was filtered and recrystallized from ethanol, Pale yellow solid, m.p:  $148-150^\circ\text{C}$ , yield 79%.

### Synthesis of ethyl hippurate ( $\text{A}_8$ ) [16]:

A solution of (0.1 mol.) hippuric acid or N-benzoyl glycine in (50 ml) of absolute ethanol saturated with hydrochloric acid (gas), the reaction mixture was refluxed in water bath for three hours at ( $90^\circ\text{C}$ ). After the completion of the reaction (monitored by TLC), the mixture was cooled and neutralized with sodium bicarbonate and the separated mass was filtered off. The product was recrystallized from ethanol and cooled, White solid, m.p:  $58-60^\circ\text{C}$ , yield 75%.

### Synthesis of hydrazide compounds ((A<sub>2</sub>), (A<sub>9</sub>) General procedure)<sup>[17]</sup>:

A mixture of the ester (A<sub>1</sub>) or (A<sub>8</sub>) (0.05 mol.), and hydrazine hydrate (80%), (0.10 mol.) in ethanol (20 ml) were refluxed for 2 hours. Excess ethanol was evaporated under vacuum and the precipitate product was crystallized from ethanol.

### Synthesis of hydrazone compounds ((A<sub>3-7</sub>), (A<sub>10-14</sub>) General procedure)<sup>[18]</sup>:

Equimolar quantities of hydrazide (A<sub>2</sub>) or (A<sub>9</sub>) and substituted bezaldehyde in (25 ml) of absolute ethanol were refluxed for three hours. The reaction mixture was then cooled and the solid precipitated was recrystallized from ethanol, physical and spectral data were illustrated in the following article:

## Results

#### 5-hydrazido-4-(methoxyphenyl)-6-methyl-1,2,3,4-tetrahydropyrimidine-2-thione (A<sub>2</sub>)

Orange solid, mp: 114-116 °C, Yield %: 85 %, IR spectrum,  $\nu$ , cm<sup>-1</sup>: 3175 (NH str.), 3103 (Ar-CH str.), 2992 (aliphatic-CH str.), 1660 (C=O str.), 1637 (C=S str.), 1607, 1457 (C=C str.), 1392 (CH bend). <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>),  $\delta$ , ppm: 2.3 (3H, s, CH<sub>3</sub>), 3.9 (3H, s, OCH<sub>3</sub>), 4.5 (1H, s, CH), 5.6 (1H, s, NH), 6.0 (1H, s, NH), 7.4-7.9 (4H, m, Ar-CH), 8.7 (1H, s, NH), 12.0 (2H, s, NH<sub>2</sub>). <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>),  $\delta$ , ppm: 19.48, 55.77, 59.11, 113.08, 114.84, 124.87, 137.00, 155.60, 161.48, 165.55, 171.60. High resolution mass spectrum (EI): Found: m/z 293.20 [M]<sup>+</sup>, Calculated 293.37.

#### 4-nitrophenyl-(4-methoxyphenyl-6-methyl-2-thioxo-1,2,3,4-tetrahydropyrimidin-5-yl-5-carbo) hydrazone(A<sub>3</sub>)

Yellow solid, mp: 276-278 °C, Yield %: 87 %, IR spectrum,  $\nu$ , cm<sup>-1</sup>: 3308 (NH str.), 3112 (Ar-CH str.), 2939 (aliphatic-CH str.), 1666 (C=O str.), 1628 (C=N str.), 1593, 1408 (C=C str.), 1347 (N=O str.) 1380, (CH bend). <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>),  $\delta$ , ppm: 2.4 (3H, s, CH<sub>3</sub>), 3.9 (3H, s, OCH<sub>3</sub>), 4.5 (1H, s, CH), 5.6 (1H, s, NH), 6.9-8.3 (8H, m, Ar-CH), 8.5 (1H, s, N=CH), 11.3 (1H, s, NH), 12.0 (1H, s, NH). <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>),  $\delta$ , ppm: 22.30, 56.90, 63.27, 110.01, 114.65, 123.51, 123.98, 126.33, 133.80, 140.07, 143.26, 148.79, 155.80, 161.17, 165.45, 171.22. High resolution mass spectrum (EI): Found: m/z 426.50 [M]<sup>+</sup>, Calculated 426.47.

#### 4-dimethylaminophenyl-(4-methoxyphenyl-6-methyl-2-thioxo-1,2,3,4-tetrahydropyrimidin-5-yl-5-carbo) hydrazone (A<sub>4</sub>)

Yellow solid, mp: 258-260 °C, Yield %: 73 %, IR spectrum,  $\nu$ , cm<sup>-1</sup>: 3303 (NH str.), 3065 (Ar-CH str.), 2969 (aliphatic-CH str.), 1660 (C=O str.), 1598 (C=N str.), 1557, 1445 (C=C str.), 1368 (CH bend). <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>),  $\delta$ , ppm: 2.3 (3H, s, CH<sub>3</sub>), 3.0 (6H, s, N(CH<sub>3</sub>)<sub>2</sub>), 3.9 (3H, s, OCH<sub>3</sub>), 4.5 (1H, s, CH), 5.9 (1H, s, NH), 7.2-8.1 (8H, m, Ar-CH), 8.4 (1H, s, N=CH), 11.5 (1H, s, NH), 12.2 (1H, s, NH). <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>),  $\delta$ , ppm: 21.67, 44.99, 60.12, 58.89, 108.36, 113.64, 118.68, 124.46, 126.41, 127.05, 141.78, 154.48, 155.51, 158.68, 167.33, 172.45. High resolution mass spectrum (EI): Found: m/z 424.44 [M]<sup>+</sup>, Calculated 424.54.

#### 4-chlorephenyl-(4-methoxyphenyl-6-methyl-2-thioxo-1,2,3,4-tetrahydropyrimidin-5-yl-5-carbo) hydrazone (A<sub>5</sub>)

Brown solid, mp: 132-134 °C, Yield %: 85 %, IR spectrum,  $\nu$ , cm<sup>-1</sup>: 3310-3164 (NH str.), 3102-3066 (Ar-CH str.), 2982 (aliphatic-CH str.), 1663 (C=O str.), 1601 (C=N str.),

1575, 1456 (C=C str.), 1393 (CH bend). <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>), δ, ppm: 2.2 (3H, s, CH<sub>3</sub>), 4.0 (3H, s, OCH<sub>3</sub>), 4.4 (1H, s, CH), 5.9 (1H, s, NH), 7.1-8.0 (8H, m, Ar-CH), 8.6 (1H, s, N=CH), 11.9 (1H, s, NH), 12.4 (1H, s, NH). <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>), δ, ppm: 23.31, 58.11, 63.54, 105.14, 113.17, 120.54, 123.37, 125.09, 131.15, 134.42, 142.99, 144.89, 156.01, 162.19, 165.45, 173.04. High resolution mass spectrum (EI): Found: m/z 415.80 [M]<sup>+</sup>, Calculated 415.92.

#### 4-hydroxyphenyl-(4-methoxyphenyl-6-methyl-2-thioxo-1,2,3,4-tetrahydropyrimidin-5-yl-5-carbo) hydrazone (A<sub>6</sub>)

Pale Yellow solid, mp: 136-138 °C, Yield %: 62 %, IR spectrum, ν, cm<sup>-1</sup>: 3502 (OH str.) 3335 (NH str.), 3078 (Ar-CH str.), 2976 (aliphatic-CH str.), 1669 (C=O str.), 1611 (C=N str.), 1591, 1459 (C=C str.), 1387 (CH bend). <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>), δ, ppm: 2.2 (3H, s, CH<sub>3</sub>), 2.6 (1H, s, OH), 3.8 (3H, s, OCH<sub>3</sub>), 4.8 (1H, s, CH), 5.7 (1H, s, NH), 6.9-8.3 (8H, m, Ar-CH), 8.9 (1H, s, N=CH), 11.3 (1H, s, NH), 12.0 (1H, s, NH). <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>), δ, ppm: 25.85, 62.78, 67.25, 106.09, 112.02, 122.28, 124.00, 128.2, 133.84, 135.98, 141.29, 145.02, 155.23, 163.91, 166.10, 172.34. High resolution mass spectrum (EI): Found: m/z 397.50 [M]<sup>+</sup>, Calculated 397.47.

#### 4-methoxyphenyl-(4-methoxyphenyl-6-methyl-2-thioxo-1,2,3,4-tetrahydropyrimidin-5-yl-5-carbo) hydrazone (A<sub>7</sub>)

Pale Yellow solid, mp: 126-128 °C, Yield %: 76 %, IR spectrum, ν, cm<sup>-1</sup>: 3292 (NH str.), 3028 (Ar-CH str.), 2959 (aliphatic-CH str.), 1670 (C=O str.), 1613 (C=N str.), 1601, 1474 (C=C str.), 1381 (CH bend). <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>), δ, ppm: 2.4 (3H, s, CH<sub>3</sub>), 3.0 3.4 (6H, s, OCH<sub>3</sub>), 4.9 (1H, s, CH), 5.4 (1H, s, NH), 7.0-8.5 (8H, m, Ar-CH), 9.0 (1H, s, N=CH), 11.6 (1H, s, NH), 12.4 (1H, s, NH). <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>), δ, ppm: 26.31, 51.87, 59.24, 68.59, 112.62, 115.18, 119.57, 122.84, 128.65, 133.84, 135.6, 143.29, 152.65, 154.12, 164.65, 166.65, 174.58. High resolution mass spectrum (EI): Found: m/z 411.60 [M]<sup>+</sup>, Calculated 411.50.

#### Hippuric hydrazide (A<sub>9</sub>)

White solid, mp: 154-156 °C, Yield %: 70 %, IR spectrum, ν, cm<sup>-1</sup>: 3491-3299 (NH str.), 3061 (Ar-CH str.), 2938 (aliphatic-CH str.), 1638 (C=O str.), 1630 (C=O str.), 1603, 1489 (C=C str.), 1377 (CH bend). <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>), δ, ppm: 3.8 (2H, s, CH<sub>2</sub>), 4.3 (2H, s, NH<sub>2</sub>), 7.4-7.9 (5H, m, Ar-CH), 8.7 (1H, s, NH), 11.1 (1H, s, NH). <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>), δ, ppm: 41.84, 128.70, 128.78, 131.76, 134.47, 166.95, 168.83. High resolution mass spectrum (EI): Found: m/z 194.09 [M]<sup>+</sup>, Calculated 194.09.

#### 2-hydroxy-1-naphthyl hippuryl hydrazone (A<sub>10</sub>)

Yellow solid, mp: 263-268 °C, Yield %: 70 %, IR spectrum, ν, cm<sup>-1</sup>: 3420 (OH str.), 3213 (NH str.), 3060 (Ar-CH str.), 2916 (aliphatic-CH str.), 1697 (C=O str.), 1637 (C=O str.), 1624 (C=N str.), 1595, 1481 (C=C str.), 1430 (C-H bend), 1367 (C-N str.). <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>), δ, ppm: 4.1 (2H, s, CH<sub>2</sub>), 7.3-8.3 (11H, m, Ar-CH), 8.9 (1H, s, CH), 9.3 (1H, s, OH), 11.9 (1H, s, NH), 12.8 (1H, s, NH). High resolution mass spectrum (EI): Found: m/z 348.13 [M]<sup>+</sup>, Calculated 348.14.

#### 2-hydroxyphenyl hippuryl hydrazone (A<sub>11</sub>)

Red solid, mp: 254-256 °C, Yield %: 73 %, IR spectrum, ν, cm<sup>-1</sup>: 3416 (OH str.), 3210 (NH str.), 3066 (Ar-CH str.), 2863 (aliphatic-CH str.), 1697 (C=O str.), 1628 (C=O str.), 1610 (C=N str.), 1562, 1442 (C=C str.), 1382 (C-H bend), 1356 (C-N str.). High resolution mass spectrum (EI): Found: m/z 298.280 [M]<sup>+</sup>, Calculated 298.281.

### 3-pyridiny hippuryl hydrazone (A<sub>12</sub>)

Brown solid, mp: 192.194 °C, Yield %: 65 %, IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 3309 (NH str.), 3074 (Ar-CH str.), 2962 (aliphatic-CH str.), 1682, 1629 (C=O str.), 1608 (C=N str.), 1576, 1415 (C=C str.), 1396 (C-H bend), 1352 (C-N str.). <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>),  $\delta$ , ppm: 4.4 (2H, s, CH<sub>2</sub>), 7.5-8.3 (8H, m, Ar-CH), 8.6 (1H, s, CH), 8.8 (1H, s, NH), 11.8 (1H, s, NH). <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>),  $\delta$ , ppm: 41.24, 124.42, 124.72, 128.80, 130.50, 131.81, 133.8, 134.28, 134.53, 144.39, 148.92, 167.02, 170.98. High resolution mass spectrum (EI): Found: m/z 283.11 [M]<sup>+</sup>, Calculated 283.11.

### 4-methoxyphenyl hippuryl hydrazone (A<sub>13</sub>)

Off white solid, mp: 168-170 °C, Yield %: 67 %, IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 3182 (NH str.), 3063 (Ar-CH str.), 2959 (aliphatic-CH str.), 1701, 1667 (C=O str.), 1614 (C=N str.), 1582, 1416 (C=C str.), 1392 (C-H bend), 1332 (C-N str.). <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>),  $\delta$ , ppm: 5.6 (2H, s, CH), 7.3-8.3 (8H, m, Ar-CH), (1H, s, CH), 11.9 (1H, s, NH). <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>),  $\delta$ , ppm: 41.23, 56.15, 112.29, 121.22, 122.58, 125.80, 127.72, 128.79, 131.79, 134.57, 158.04, 158.14, 166.97, 170.68. High resolution mass spectrum (EI): Found: m/z 312.135 [M]<sup>+</sup>, Calculated 312.134.

### 2,6-dichlorophenyl hippuryl hydrazone (A<sub>14</sub>)

Yellow solid, mp: 228-230 °C, Yield %: 80 %, IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 3380-3313 (NH str.), 3075-3026 (Ar-CH str.), 2965 (aliphatic-CH str.), 1682, 1644 (C=O str.), 1601 (C=N str.), 1576, 1486 (C=C str.), 1435 (C-H bend), 1395 (C-N str.), 697 (C-Cl). <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>),  $\delta$ , ppm: 3.8 (3H, s, CH<sub>3</sub>), 4.4 (2H, s, CH<sub>2</sub>), 7.0-7.9 (9H, m, Ar-CH), 8.3 (1H, s, CH), 8.7 (1H, s, NH), 11.5 (1H, s, NH). <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>),  $\delta$ , ppm: 41.19, 127.69, 128.78, 129.47, 129.90, 130.10, 130.55, 131.48, 134.3, 138.83, 142.24, 143.25, 167.02, 171.08. High resolution mass spectrum (EI): Found: m/z 350.01 [M]<sup>+</sup>, Calculated 350.04.

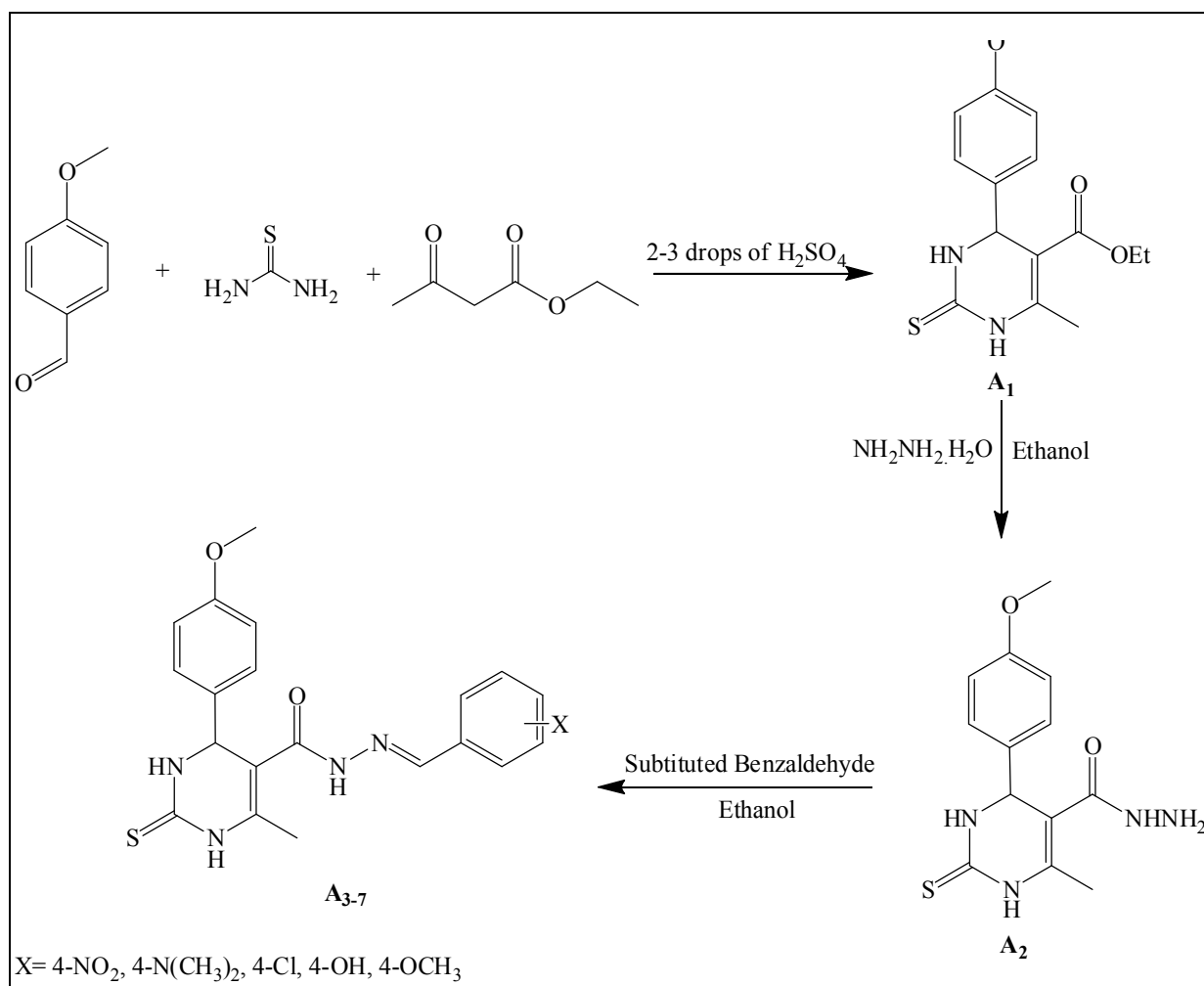
## Discussion

Hydrazides (A<sub>2</sub>) and (A<sub>9</sub>) were prepared by hydrazinolysis of their corresponding esters in absolute ethanol. The IR data of hydrazides (A<sub>2</sub>) and (A<sub>9</sub>) showed bands for N-H stretching absorbed at (3175  $\text{cm}^{-1}$ ) and (3299  $\text{cm}^{-1}$ ), the C=O bond decrease in frequencies from (1733  $\text{cm}^{-1}$ ) and (1759  $\text{cm}^{-1}$ ) in ester compounds to new values (1660  $\text{cm}^{-1}$ ) and (1638  $\text{cm}^{-1}$ ) due to the resonance in hydrazide group. The <sup>1</sup>H NMR spectrum provide a perfect confirmation to the structure formation in which the hydrazide compound showed new singlet signal at (8.7 ppm), (12.0 ppm, 2H, s, for NH<sub>2</sub>), while signals resonating at (4.3 ppm), (11.1 ppm), related to NH of compound (A<sub>9</sub>).

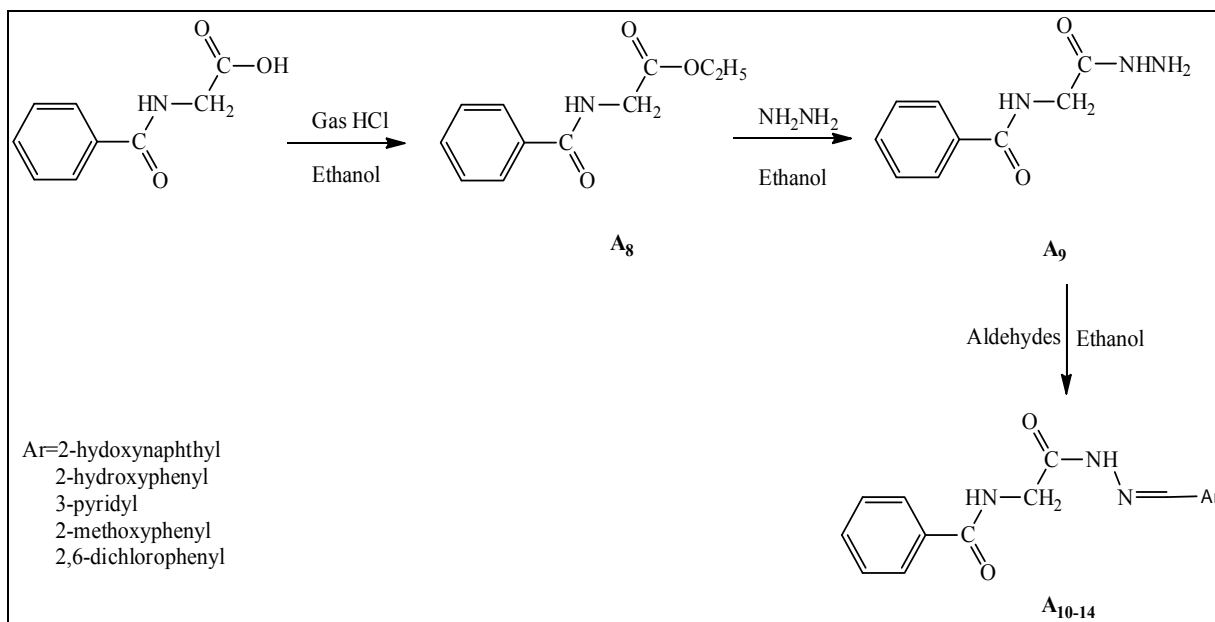
All the newly synthesized hydrazone compounds (A<sub>5-9</sub>) (scheme 1) and (A<sub>10-14</sub>) (scheme 2) showed satisfactory IR data. Strong C=O stretching band absorbed around at (1660-1670  $\text{cm}^{-1}$ ), (1628-1701  $\text{cm}^{-1}$ ) and C=N band absorbed around at (1601-1628  $\text{cm}^{-1}$ ) together with the N-H stretching bond absorbed at (3164-3335  $\text{cm}^{-1}$ ), (3182-3420  $\text{cm}^{-1}$ ) which are characteristic absorption of the hydrazone group. The <sup>1</sup>H NMR also confirms the presence of a value at (8.3-9.0 ppm) for N=CH (imine group) in hydrazone compounds, as well as the presence of both aromatic and pyrimidine ring protons as indicated within the data for these compounds. <sup>13</sup>C NMR spectra for compounds (A<sub>3-7</sub>) as it was indicated in their data presented above showed the presence of aromatic ring carbons (two equivalent carbons) for AB system resonating at (123 and 126 ppm) while pyrimidine carbons showed a value around (63 ppm) assigned to carbon attached to p-substituted ring and a value of (163 ppm) for carbon attached to methyl group while the thioxo carbon resonated within the range (167-173 ppm).

The results also revealed the presence of C=N resonating within (141-142 ppm) which is in agreement with similar published compounds [19]. Finally compounds (A<sub>10-14</sub>) showed <sup>13</sup>C-NMR signals related to aromatic of hippuric moiety at (124, 128, 131 ppm) while the aryl ring

(pyridyl ring for example) carbons appeared at (124, 134, 148 ppm) and the azo methine carbon C=N resonating within (142-144 ppm).

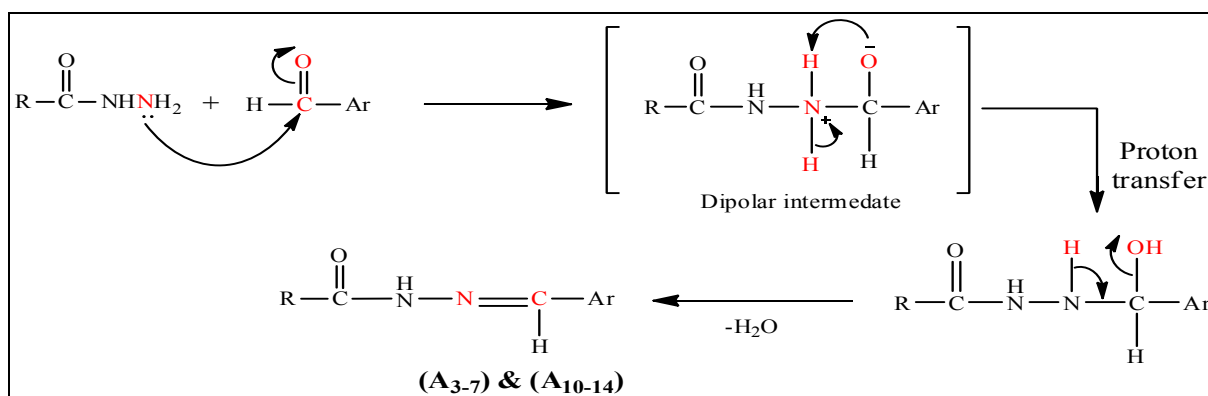


Scheme (1)



Scheme (2)

The mechanism of the formation of hydrazone compounds can be illustrated in the following scheme <sup>[20]</sup>.



Scheme (3)

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## تحضير بعض الهيدرازيدات الجديدة ومشتقاتها

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### الخلاصة

تم تحضير هيدرازيدات جديدة وتمثل المركبات (A<sub>2</sub> و A<sub>9</sub>) من استراتها المقابلة (A<sub>1</sub> و A<sub>8</sub>) وهذه الاسترات تم تحضيرها من موادها الأولية 5- ايثوكسي كاربونيل ( 4-ميثوكسي فنييل ) - 6 - مثيل -1,2,3,4- رباعي هايدرو بيريمدين - 2 - ثايون (A<sub>1</sub>) عن طريق تفاعل متعدد المكونات وكذلك من حامض الهيبيوريك على التوالي . هذه الهيدرازيدات تم مفاعلتها مع بعض الألدهايدات مكونة الهيدرازونات المقابلة (7- A<sub>3</sub>) و (14- A<sub>10</sub>). تم تشخيص المركبات المحضرة بالطرق الطيفية المتمثلة بـ IR, <sup>1</sup>H-NMR, <sup>13</sup>C-NMR و طيف الكتلة وتمت مناقشتها.

كلمات مفتاحية: أستر، هيدرازيد، هيدرازون، قواعد شيف.