Synthesis of New Heterocyclic compounds derived from Pyrazoline-5-one compound

Rafah F.Al-Smaisim*; Redha E.Al-Bayati** and Abdul Hussain K. Sharba**

*Department of pharmaceutical Chemistry and pharmacognacy, Collage of Pharmacy,
** Department of Chemistry, College of Science. University of Al-Mustansriya

Abstract
In this work new heterocyclic pyrazolin derivatives have been synthesized from diazonium chloride salt of 4-aminobenzoic acid: firstly, Azo compounds were prepared from the reaction of an ethanolic solution of sodium acetate and calculated amount of active methylene compound namely, (ethyl acetoacetate) obtain the corresponding hydrazono derivative (1). Secondly, Cyclocondensation reaction of compound (1) with hydrazine hydrate (2) in
boiling ethanol affording the corresponding pyrazoline-5-one. Then compound (2) reacted with thionyl chloride to give the corresponding acid chloride derivative(3), followed by conversion into the corresponding carboxylic acid thiosemicarbazide (4), esters (7-9), thioesters (10), (11), and amides (12-14), when treated hydrazine hydrate, thiosemicarbazide, alcohols, alkylthiol and secondary amines in dry refluxing benzene; respectively. Furthermore, 1,2,4-triazole heterocyclic ring, which might result in biologically active agents, have been prepared by refluxing thiosemicarbazide derivative (4) with sodium hydroxide solution (4%) followed acidification of the result using (10%)HCl solution. Moreover, 1, 3, 4, - thiadiazole heterocyclic ring (6) has been prepared by treatment of thiosemicarbazide derivative with concentrated sulfuric acid as cyclization agent. Finally, derivative (15) has prepared by reflux (1) with p-hydroxybenzaldehyde then the product reflux with 5-amino-1, 3, 4-thiadiazol-2-thiol to product (16) derivative. All structures of newly synthesized compounds have been characterized and identified via of their physical properties and spectral data analysis (IR, UV.)

Introduction:

Heterocyclic compounds represent an important class of biologically active molecules. Specifically, those containing the pyrazole nucleus have been shown to possess high biological activities as herbicides, fungicides, analgesics, etc[1]. Some novel pyrazole derivatives containing sulfonamide moieties as anti microbial agents, Various sulfa drugs were coupled with active methylene compounds to give various hydrazones, then novel series of pyrazoles derivatives[2]. Past few years, biologically active pyrazoles comprising fused pyrimidine moiety into the 1-position of the pyrazole ring system[3]. Moreover; reaction of azo compounds with substituted acetoacetic ester derivatives using acetic acid as solvent[4].

Materials and Methods:

Apparatus and Chemicals:

Electrothermal 9100 melting point apparatus, Perkin-Elmer 1310 infrared spectrophotometer or a Shimadzu FTIR-800, as KBr discs or thin films, UV-Visible Varian UV-Cary-100 spectrophotometers were used in this work. All the chemicals used were supplied by Merck, Fluka and BDH chemicals. The solvents were purified by distillation and dried with calcium chloride.

Experimental:

4-{{(1-(ethoxycarbonyl)-2-oxopropyl)diazenyl}benzoic acid (1)[4]

To an ice-cooled mixture of active methylene compound (ethyl acetoacetate) (0.01 mole) and sodium acetate (0.05 mole, 4.10 g) in ethanol (50 ml), was added dropwise with stirring to a cooled solution of the diazonium salt over 15 minute. The solid product was collected and recrystalized from ethanol.
4-((3-methyl-5-oxo-4,5-dihydro-1\(H\)-pyrazol-4-yl)diazynyl)benzoic acid (2)

A mixture of azo derivative (0.01 mole) and hydrazine hydrate (95 %) (0.012 mole, 0.35 g) in ethanol (30 ml) was heated under reflux for 4 hours. The reaction mixture was concentrated and the reaction product was allowed to cool. The separated product was filtered off, washed with water, and recrystallized from the appropriate solvent.

4-((3-methyl-5-oxo-4,5-dihydro-1\(H\)-pyrazol-4-yl)diazynyl)benzoyl chloride (3)

A mixture of compound (2) (0.01 mole, 2.46 g) and thionyl chloride (7 ml) was gently refluxed for 2 hours. After cooling, excess thionyl chloride was removed under reduced pressure. The product was recrystallized from benzene.

2-{4-((3-methyl-5-oxo-4,5-dihydro-1\(H\)-pyrazol-4-yl)diazynyl)benzoyl} hydrazinecarbothioamide (4)

To a solution of (3) (0.005 mole, 1.32 g) in dry benzene (25 ml), thiosemicarbazide (0.005 mole 0.45 g) was added. The mixture was refluxed for 3 hours, cooling, filtered, and recrystallized from ethanol.

4-{(4-(5-mercapto-4H-1,2,4-triazol-3-yl) phenyl)diazynyl}-5-methyl-2,4-dihydro-3H-pyrazol-3-one (5)

A mixture of (4) (0.001 mole, 0.319 g) and (4%) sodium hydroxide solution (25 ml) was refluxed for 4 hours, cooled, poured into crushed ice and acidified with dilute hydrochloric acid (10 %). The resultant precipitate was filtered, washed with water and recrystallized from ethanol.

4-{(4-(5-amino-1,3,4-thiadiazol-2-yl)phenyl)diazynyl}-5-methyl-2,4-dihydro-3H-pyrazol-3-one (6)

Compound (4) (0.001 mole, 0.32 g) was dissolved in cold concentrated sulfuric acid (10 ml) and stirred at room temperature for 24 hours. Poured into crushed ice the product was diluted and filtered, recrystallized from ethanol.

prop-2-ynyl 4-((3-methyl-5-oxo-4,5-dihydro-1\(H\)-pyrazol-4-yl)diazynyl) benzoate (7)

3-chloro-4-formylphenyl 4-((3-methyl-5-oxo-4,5-dihydro-1\(H\)-pyrazol-4-yl)diazynyl) benzoate (8)
isobutyl 4-((3-methyl-5-oxo-4,5-dihydro-1\(H\)-pyrazol-4-yl)diazynyl) benzoate (9)

To a solution of compound (2) (0.005 mole, 1.23 g) in dry benzene (25 ml), alkyl, or phenyl alcohol (0.005 mole) was added, the mixture was refluxed for 6 hours.

S-benzyl 4-((3-methyl-5-oxo-4,5-dihydro-1\(H\)-pyrazol-4-yl)diazynyl) benzenecarbothioate (10)

S-butyl 4-((3-methyl-5-oxo-4,5-dihydro-1\(H\)-pyrazol-4-yl)diazynyl) benzenecarbothioate (11)
5-methyl-4-{(4-(piperidin-1-ylcarbonyl)phenyl)diazcnayl}-2,4-dihydro-3H-pyrazol-3-one (12) 
5-methyl-4-{(4-(morpholin-4-ylcarbonyl)phenyl)diazcnayl}-2,4-dihydro-3H-pyrazol-3-one (13) 
N,N-dimethyl-4-((3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl)diazcnayl) benzamide (14)

To a solution of compound (2) (0.005 mole, 1.23 g) in dry benzene (25 ml), secondary amine (0.005 mole) was added, and refluxed for 3 hours.

4-formylphenyl 4-((3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl)diazcnayl) benzoate (15)

A mixture of compound (1) (0.01 mole, 2.46 g) was refluxed with 4-hydroxybenzaldehyde (0.01 mole, 1.22 g) on oil bath at 140–160 °C for 2 hours. The product was cooled recrystallized from the appropriate solvent.

4-{{(5-mercaptop-1,3,4-thiadiazol-2-yl)imino}methyl}phenyl  4-{{(3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-4-yl)diazcnayl}benzoate (16)

To a solution of 5-amino-1,3,4-thiadiazol-2-thiol (0.001 mole, 0.133 g) in (10 ml) of absolute ethanol, compound (15) (0.001 mole, 0.35 g) was added. The mixture was refluxed for (3 hours), cooled, filtered and recrystallized from ethanol.

<table>
<thead>
<tr>
<th>Compound Number</th>
<th>Molecular Formula</th>
<th>M.P/ °C</th>
<th>Color</th>
<th>Purification Solvent</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>C_{13}H_{14}N_{2}O_{3}</td>
<td>201-203</td>
<td>Green</td>
<td>Ethanol</td>
<td>88</td>
</tr>
<tr>
<td>2</td>
<td>C_{11}H_{10}N_{4}O_{3}</td>
<td>240-242</td>
<td>Orange-Yellow</td>
<td>Ethanol</td>
<td>68</td>
</tr>
<tr>
<td>3</td>
<td>C_{11}H_{10}ClN_{4}O_{2}</td>
<td>217 dec.</td>
<td>Deep-Green</td>
<td>Benzene</td>
<td>82</td>
</tr>
<tr>
<td>4</td>
<td>C_{12}H_{13}N_{2}O_{3}S</td>
<td>287-289</td>
<td>Brown</td>
<td>Ethanol</td>
<td>70</td>
</tr>
<tr>
<td>5</td>
<td>C_{12}H_{11}N_{2}OS</td>
<td>230-232</td>
<td>Brownish red</td>
<td>Ethanol</td>
<td>43</td>
</tr>
<tr>
<td>6</td>
<td>C_{12}H_{11}N_{2}OS</td>
<td>295-297</td>
<td>Green</td>
<td>Ethanol</td>
<td>52</td>
</tr>
<tr>
<td>7</td>
<td>C_{14}H_{12}N_{2}O_{3}</td>
<td>188-190</td>
<td>Yellow</td>
<td>Benzene</td>
<td>75</td>
</tr>
<tr>
<td>8</td>
<td>C_{15}H_{14}ClN_{4}O_{4}</td>
<td>200dec.</td>
<td>Pale Brown</td>
<td>Benzene</td>
<td>42</td>
</tr>
<tr>
<td>9</td>
<td>C_{15}H_{16}N_{3}O_{3}</td>
<td>166-168</td>
<td>Yellow</td>
<td>Chloroform</td>
<td>83</td>
</tr>
<tr>
<td>10</td>
<td>C_{18}H_{16}N_{3}O_{3}</td>
<td>177 dec.</td>
<td>Brown</td>
<td>Benzene</td>
<td>32</td>
</tr>
<tr>
<td>11</td>
<td>C_{18}H_{18}N_{3}O_{3}</td>
<td>158 dec.</td>
<td>Brown</td>
<td>Chloroform</td>
<td>59</td>
</tr>
<tr>
<td>12</td>
<td>C_{16}H_{16}N_{2}O_{2}</td>
<td>201-203</td>
<td>Brown</td>
<td>Chloroform</td>
<td>72</td>
</tr>
<tr>
<td>13</td>
<td>C_{15}H_{17}N_{2}O_{3}</td>
<td>207 dec.</td>
<td>Pale-brown</td>
<td>Chloroform</td>
<td>55</td>
</tr>
<tr>
<td>14</td>
<td>C_{13}H_{15}N_{3}O_{2}</td>
<td>185 dec.</td>
<td>Yellow</td>
<td>Chloroform</td>
<td>70</td>
</tr>
<tr>
<td>15</td>
<td>C_{18}H_{14}O_{4}N_{4}</td>
<td>320 dec.</td>
<td>Brown</td>
<td>Ethanol/water 1:1</td>
<td>56</td>
</tr>
<tr>
<td>16</td>
<td>C_{20}H_{15}N_{3}O_{3}S_{2}</td>
<td>300 dec.</td>
<td>Pale-yellow</td>
<td>Ethanol</td>
<td>68</td>
</tr>
</tbody>
</table>

Table-1: The physical properties of compounds (1-16)
Results and Discussion

For the synthesis of the target 4-aminobenzoic acid derivatives in this work, the reaction sequences are outlined in scheme (1).

Scheme (1)
Hydrazons are easily undergoing cyclocondensation reaction with hydrazine hydrate in boiling ethanol afford to the corresponding pyrazoline-5-one derivatives of p-aminobenzoic acid. Thus cyclization of azo compound with hydrazine hydrate afford the corresponding derivative (2). The IR spectrum of compound (1) shows a characteristic bands at (1735 cm\(^{-1}\)) for the carboxylic ester moiety, while bands at (1715 cm\(^{-1}\)), (1685 cm\(^{-1}\)) corresponding to the characteristic (C=O) of acetyl and carboxylic acid, respectively. The band at (1530 cm\(^{-1}\)) corresponds to the stretching vibration of the azo group, and the broad band at (2600-3200 cm\(^{-1}\)) refers to stretching vibration of hydroxyl group.

The mechanism of this cyclocondensation reaction may be outlined as follow:

\[
\begin{align*}
\text{HOOC-} & \quad \text{N=N} & \quad \text{NH}_{2} \\
\text{O} & \quad \text{O} & \quad \text{HN} \\
\text{CH} & \quad \text{R} & \quad \text{O} \\
\text{HOOC} & \quad \text{N=N} & \quad \text{N} & \quad \text{H} \\
\text{N} & \quad \text{N} & \quad \text{H} & \quad \text{CH} & \quad \text{R} & \quad \text{O} \\
& \quad \text{HOOC} & \quad \text{N=N} & \quad \text{N} & \quad \text{H} & \quad \text{H} & \quad \text{O} \\
& \quad \text{HOOC} & \quad \text{N=N} & \quad \text{N} & \quad \text{H} & \quad \text{CH} & \quad \text{R} & \quad \text{O} \\
& \quad \text{HOOC} & \quad \text{N=N} & \quad \text{N} & \quad \text{H} & \quad \text{H} & \quad \text{O} \\
& \quad \text{HOOC} & \quad \text{N=N} & \quad \text{N} & \quad \text{H} & \quad \text{CH} & \quad \text{R} & \quad \text{O} \\
\end{align*}
\]

\[
\text{HOOC} \quad \text{N=N} \quad \text{N} \quad \text{H} \quad \text{CH} \quad \text{R} \quad \text{O} \\
\text{HOOC} \quad \text{N=N} \quad \text{N} \quad \text{H} \quad \text{H} \quad \text{O} \\
\text{HOOC} \quad \text{N=N} \quad \text{N} \quad \text{H} \quad \text{CH} \quad \text{R} \quad \text{O} \\
\text{HOOC} \quad \text{N=N} \quad \text{N} \quad \text{H} \quad \text{H} \quad \text{O} \\
\text{HOOC} \quad \text{N=N} \quad \text{N} \quad \text{H} \quad \text{CH} \quad \text{R} \quad \text{O} \\
\text{HOOC} \quad \text{N=N} \quad \text{N} \quad \text{H} \quad \text{H} \quad \text{O} \\
\]

The IR spectrum of compound (2), shows the disappearance of the characteristic bands of the acetyl carbonyl group and carboxylic acid ester at (1735, 1715 cm\(^{-1}\)), and the appearance of strong bands in the (3450 cm\(^{-1}\)), attributed to (N-H) stretching vibration and the bands of (C=O) carboxylic acid appeared at (1680 cm\(^{-1}\)), pyrazolinone ring (C=O) stretching vibration appeared at (1650 cm\(^{-1}\)) and (OH)\(_{\text{st}}\) appear at (2600-3300 cm\(^{-1}\)).

The IR spectrum of compound (3) shows the disappearance of the hydroxyl group of the starting material and appearance of the new (C=O) band at (1780 cm\(^{-1}\)), for the acetyl chloride. The spectrum also shows an absorption band at (700 cm\(^{-1}\)) referring to (C-Cl) band \(^{[8]}\). The U.V. spectrum of this compound, has \(\lambda_{\text{max}}\) (MeOH) at (240 and 344 nm) responsible for (\(\pi-\pi^{*}\)).

The IR spectrum of compound (4), shows the main characteristic bands at (1220 cm\(^{-1}\)) refers to (C=S) stretching vibration, an absorption band at (1680 cm\(^{-1}\)).
cm\(^{-1}\)) for (C=O) stretching vibration which appears at (1780 cm\(^{-1}\)) in the acid chloride derivatives and at (3350) for (N-H) and (3300-3450 cm\(^{-1}\)) for (NH\(_2\)) stretching vibration. The success of the reaction has been confirmed by comparing the (C=O) absorption in the acid chloride and hydrazide derivatives.

The IR spectrum of compound (5), shows characteristic (S-H) stretching vibration as weak band at (2650 cm\(^{-1}\)) and (C=S) stretching vibration as weak band at (1233 cm\(^{-1}\)) which confirmed the tautomersim between thion and thiol\(^9\) form and an absorption band at (1640 cm\(^{-1}\)) due to (C=N) stretching vibration of triazole transition.

The IR spectrum of compound (6) shows absorption band at (1260 cm\(^{-1}\)) due to (N-N) stretching vibration and at (3300-3450 cm\(^{-1}\)) due to (NH\(_2\)) stretching vibration. The IR spectrum of compound (7) shows the disappearance of (C-Cl) stretching band and appearance of absorption band at (1730 cm\(^{-1}\)) due to (C=O) stretching vibration, appearance of (C=C-H) stretching band at (3200 cm\(^{-1}\)) and band at (2170 cm\(^{-1}\)) for (C=C ) assymetrical stretching vibration\(^9\). The success of the reaction has been confirmed by the appearance of the triple bond of the acetylenic group the thioester compounds have been synthesized by the reaction of acid chloride and RSH in refluxing dry benzene with mechanism similar to that of alcoholic ester.

The IR spectrum of compound (10) shows band at  (1690 cm\(^{-1}\)) due to (C=O) stretching vibration which had appeared at (1800 cm\(^{-1}\)) in acid chloride compound (3), band at (660 cm\(^{-1}\)) due to (C-S) stretching vibration. The IR spectrum of compound (12), shows the main characteristic bands at (1640 cm\(^{-1}\)) due to (C=O) of amide, and at (2970 cm\(^{-1}\) asy and 2880 cm\(^{-1}\) sym)\(^{10}\) for aliphatic (C-H) stretching vibration.

Compound (15) has been synthesized by treatment of compound (2) with p-hydroxybezaldehyde The IR spectrum of (15), shows the disappearance of the broad stretching band for (OH) of the carboxylic group of compound (2), and appearance of an absorption band at (1740 cm\(^{-1}\)) due to (C=O) stretching vibration of ester group, which interfered with the (C=O) stretching vibration of the aldehyde group (1725 cm\(^{-1}\)) and appearance of a weak band of (H-C=O) aldehyde in (2700 cm\(^{-1}\)).

Compound (16) was prepared by treatment of compound (15) with 5-amino-1,3,4-thiadiazol-2-thiol in absolute ethanol as a solvent. The IR spectrum of compound (16), shows absorption band at (1640 cm\(^{-1}\)) due to (C=N) stretching vibration. The U.V. spectrum of this compound, table (2) has \(\lambda_{\text{max}}\) (MeOH) at (293 and 275 nm) responsible for (\(\pi-\pi^*\))
<table>
<thead>
<tr>
<th>Compound Number</th>
<th>UV λ&lt;sub&gt;max&lt;/sub&gt; (nm)</th>
<th>v(C=O)</th>
<th>v(C≡N)</th>
<th>v(N≡N)</th>
<th>v(C-H)al</th>
<th>v(C-H)ar</th>
<th>Others</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>362, 238</td>
<td>1735(ester) 1715(acetyl) 1685 (acid)</td>
<td>1620 interfere with C=C</td>
<td>1610 interfere with C=C</td>
<td>1530</td>
<td>(2970)&lt;sub&gt;asy&lt;/sub&gt; (2850)&lt;sub&gt;sy&lt;/sub&gt; 3050</td>
<td>2600-3200 (OH)&lt;sub&gt;st&lt;/sub&gt;, 2600-3300 (O-H) Interfere with 3450(NH)&lt;sub&gt;st&lt;/sub&gt;</td>
</tr>
<tr>
<td>285, 261</td>
<td>1650(ring) Interfere with 1680(acid)</td>
<td>1580</td>
<td>(2920)&lt;sub&gt;asy&lt;/sub&gt; (2850)&lt;sub&gt;sy&lt;/sub&gt; 3090</td>
<td>2600-3300 (O-H) Interfere with 3450(NH)&lt;sub&gt;st&lt;/sub&gt;</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>344, 240</td>
<td>1780 1660(ring)</td>
<td>1550</td>
<td>(2950)&lt;sub&gt;asy&lt;/sub&gt; (2800)&lt;sub&gt;sy&lt;/sub&gt; 3050</td>
<td>700 (C-Cl)&lt;sub&gt;st&lt;/sub&gt; 3350 (N-H)&lt;sub&gt;st&lt;/sub&gt;</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>389</td>
<td>1680 Interfere with 1660 (ring)</td>
<td>1540</td>
<td>(2960)&lt;sub&gt;asy&lt;/sub&gt; (2800)&lt;sub&gt;sy&lt;/sub&gt; 3050</td>
<td>1220 (C=S)&lt;sub&gt;st&lt;/sub&gt; 3350 (NH)&lt;sub&gt;st&lt;/sub&gt; Interfere with 3300-3450 (NH)&lt;sub&gt;st&lt;/sub&gt;</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>377, 271</td>
<td>1665 (ring)</td>
<td>1555</td>
<td>(2970)&lt;sub&gt;asy&lt;/sub&gt; (2850)&lt;sub&gt;sy&lt;/sub&gt; 3080</td>
<td>1233 (C=S)&lt;sub&gt;st&lt;/sub&gt; 2650(SH)&lt;sub&gt;st&lt;/sub&gt; 3300 (NH)&lt;sub&gt;st&lt;/sub&gt;</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>356</td>
<td>1655 (ring)</td>
<td>1555</td>
<td>(2900)&lt;sub&gt;asy&lt;/sub&gt; (2800)&lt;sub&gt;sy&lt;/sub&gt; 3050</td>
<td>1260 (N-N) 3250 (NH)&lt;sub&gt;st&lt;/sub&gt; Interfere with 3300-3450 (NH)&lt;sub&gt;st&lt;/sub&gt;</td>
<td></td>
<td></td>
</tr>
<tr>
<td>395</td>
<td>1730 (ester) 1650 (ring)</td>
<td>1555</td>
<td>2950 3080</td>
<td>3200 (C=C)&lt;sub&gt;st&lt;/sub&gt; 2170 C≡C&lt;sub&gt;st&lt;/sub&gt; 3350 (NH)&lt;sub&gt;st&lt;/sub&gt;</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>310</td>
<td>1730 (ester) 1660 (ring)</td>
<td>1550</td>
<td>2970 3050</td>
<td>3270 (NH)&lt;sub&gt;st&lt;/sub&gt; 675(C-S)&lt;sub&gt;st&lt;/sub&gt;</td>
<td></td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>298</td>
<td>1730 (ester) 1660 (ring) interferes with 1680 (aldehyde)</td>
<td>1555</td>
<td>2900 3050</td>
<td>2690 (C-H) aldehyde 3350 (NH)&lt;sub&gt;st&lt;/sub&gt;</td>
<td></td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>398, 285</td>
<td>1700 1650</td>
<td>1558</td>
<td>2980 3050</td>
<td>3270 (NH)&lt;sub&gt;st&lt;/sub&gt; 675(C-S)&lt;sub&gt;st&lt;/sub&gt;</td>
<td></td>
<td></td>
</tr>
<tr>
<td>328, 217</td>
<td>1640 1650(ring)</td>
<td>1560</td>
<td>(2970) asy (2880) sy 3050</td>
<td>3350(NH)&lt;sub&gt;st&lt;/sub&gt;</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>310</td>
<td>1630</td>
<td>1625 interfere with (C=O)</td>
<td>1550</td>
<td>2950 3080</td>
<td>1160(C-O-C) 3350 (NH)&lt;sub&gt;st&lt;/sub&gt;</td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>337, 288</td>
<td>1630</td>
<td>1625 interfere with (C=O)</td>
<td>1550</td>
<td>(2950) asy (2800) sy 3050</td>
<td>3300 (NH)</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>379, 256</td>
<td>1740 (ester) 1725(amide) 1680 (ring)</td>
<td>1610</td>
<td>1550</td>
<td>(2950) asy (2800) sy 3080</td>
<td>weak 2700, 2800 (C-H)ald 3350 (NH)&lt;sub&gt;st&lt;/sub&gt;</td>
<td></td>
</tr>
<tr>
<td>16</td>
<td>293, 275</td>
<td>1730(ester) 1660 (ring)</td>
<td>1640</td>
<td>1540</td>
<td>(2900) asy (2820) sy 3050</td>
<td>weak 2550 (C-H)ald 3400 (NH)&lt;sub&gt;st&lt;/sub&gt;</td>
<td></td>
</tr>
</tbody>
</table>

*Table-2: Spectral data for compounds (1-16)*
References: