CHAPTER 2
EXPERIMENTAL

2.1 Materials and Methods

Phenyl glycine, Tyrosine, Lysine mono hydrochloride, Glutamic acid 5-Chlorosalicylaldehyde, (Purchased from Aldrich chemicals), Valine, Precoated aluminium silica plates, Methanol (E Merck chemicals), Sodiumhydroxide (Ranbaxy chemicals), 5-Nitro salicylaldehyde (Lancaster), Triethylamine, Thionylchloride, Hexane, Ethyl acetate (SD fine chemicals), Ethanol (Hayman, Germany), Silica gel (Acme’s laboratory chemicals).

2.2 Synthesis of 5 - Bromosalicylaldehyde\textsuperscript{1,2}

Bromine solution (13.0 ml) in chloroform (1:2 by weight) was added drop by drop to a solution of salicylaldehyde in 10 ml chloroform and stirred at room temperature for 1 h. Solvent removed under reduced pressure. Solid so obtained washed with ethanol and recrystallised from hot 60% ethanol. Yield : 90%, M.P. 103° - 104°C (104°C).

2.3 Synthesis of 3, 5-diiodo salicylaldehyde\textsuperscript{1}

Salicylaldehyde (15.0 gm) and sodium hydroxide (1.25N) were mixed with 115 ml of 30% solution of Na\textsubscript{2}CO\textsubscript{3}. To the resulting solution a solution of 65.0 g iodine and 150 g KI in water was made up to 175 ml was added gradually, filtered after 24 hours. The sodium compound of 3,5-diiodo salicylaldehyde crystallized on cooling. Then it was acidified with HCl and recrystallised from 60% ethanol. Yield 92 %, M.P. 107°C (108°C).
2.4 **Synthesis of amino acids methyl ester hydrochloride**³

Amino acids (50 mmole) was stirred with 50 ml of methanol at 0°C and 7.3 ml thionyl chloride (5.95 g, 50 mmole) was added and stirred for 6 hrs. Excess methanol and thionyl chloride present in the reaction mixture were removed under reduced pressure. The white solid formed was dried and stored under nitrogen atmosphere. The yield was 80 – 95%.

2.5 **Synthesis of salicylaldehyde schiff bases with aminoacids**⁴

Sodium hydroxide (10 mmol, 0.4g) was dissolved in methanol (30ml) and the amino acids (10 mmol) was added to it. The mixture was stirred at room temperature. When the mixture became homogenous a solution of salicylaldehyde/substituted salicylaldehyde (10 mmol) in ethanol was added. After two minutes the solution was evaporated to 20% of its original volume and 1 ml of acetic acid was added immediately. After two hours, yellow crystals appeared. The crystals were filtered and washed with ethanol. They were recrystallised from hot methanol to give yellow crystals. The yield range from 50-90%. Following the above procedure compounds 1-20 and 35 were synthesised and characterized by spectroscopic methods.

2.6 **Synthesis of salicylaldehyde schiff bases with aminoacid esters**⁵

Equimolar of salicylaldehyde/substituted salicylaldehyde and amino acid esters dissolved in benzene were refluxed in a Dean stark apparatus until the water was completely separated. The reaction mixture was then passed through a short silica gel column to remove the triethylamine hydrochloride. Benzene was removed under reduced pressure to obtain aldime ester. Following the above procedure compounds 21-34 and 36-39 were synthesized and characterized by spectroscopic methods.
SYNTHETIC SCHEME

Compounds 21-34 and 36-39

Compounds 1-20 and 35

R¹ = H, I

R² = H, Br, Cl, I and NO₂

R³ = CH (CH₃)₂, -CH₂-(C₆H₄-4-OH), C₆H₅

CH₂CH₂ COOH and (CH₂)₄ NH₂ HCl
# LIST OF SYNTHESIZED COMPOUNDS

<table>
<thead>
<tr>
<th>Compound No.</th>
<th>Structures</th>
<th>Compound Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td><img src="image1.png" alt="Structure 1" /></td>
<td>2-N (2-hydroxyphenyl methylene)imino-3-methyl butanoicacid</td>
</tr>
<tr>
<td>2</td>
<td><img src="image2.png" alt="Structure 2" /></td>
<td>2-N(2-hydroxyphenyl methylene)imino-3- (4-hydroxy phenyl) propanoicacid</td>
</tr>
<tr>
<td>3</td>
<td><img src="image3.png" alt="Structure 3" /></td>
<td>2-N(2-hydroxyphenyl methylene)imino pentanedioicacid</td>
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<tr>
<td>4</td>
<td><img src="image4.png" alt="Structure 4" /></td>
<td>2-N (2-hydroxyphenyl methylene)imino-6-aminohexanoicacid hydrochloride</td>
</tr>
<tr>
<td>5</td>
<td><img src="image5.png" alt="Structure 5" /></td>
<td>2-N (2-hydroxy phenyl methylene)imino-2-phenylaceticacid</td>
</tr>
<tr>
<td>6</td>
<td><img src="image6.png" alt="Structure 6" /></td>
<td>2-N(2-hydroxy-5-chlorophenyl methylene)imino-3-methyl butanoicacid</td>
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<tr>
<td>7</td>
<td><img src="image7.png" alt="Structure 7" /></td>
<td>2-N (2-hydroxy-5-chlorophenyl methylene)-imino-3-(4-hydroxy phenyl)propanoicacid</td>
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<td></td>
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<td>Formula</td>
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<tr>
<td>8</td>
<td><img src="image8.png" alt="Chemical Structure" /></td>
<td>2-N(2-hydroxy-5-chlorophenyl methylene)imino pentaedic acid</td>
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<td><img src="image9.png" alt="Chemical Structure" /></td>
<td>2-N(2-hydroxy-5-chlorophenyl methylene)imino-6-aminohexanoic acid hydrochloride</td>
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<td>10</td>
<td><img src="image10.png" alt="Chemical Structure" /></td>
<td>2-N(2-hydroxy-5-chlorophenyl methylene)imino-2-phenylacetic acid.</td>
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<tr>
<td>11</td>
<td><img src="image11.png" alt="Chemical Structure" /></td>
<td>2-N(2-hydroxy-5-bromo phenyl methylene)imino-3-methyl butanoic acid.</td>
</tr>
<tr>
<td>12</td>
<td><img src="image12.png" alt="Chemical Structure" /></td>
<td>2-N(2-hydroxy-5-bromophenyl methylene)imino-3-(4-hydroxy phenyl) propanoic acid.</td>
</tr>
<tr>
<td>13</td>
<td><img src="image13.png" alt="Chemical Structure" /></td>
<td>2-N(2-hydroxy-5-bromophenyl methylene)imino pentanedioic acid.</td>
</tr>
<tr>
<td>14</td>
<td><img src="image14.png" alt="Chemical Structure" /></td>
<td>2-N(2-hydroxy-5-bromophenyl methylene)imino-6-amino hexanoic acid hydrochloride.</td>
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<td>Chemical Structure</td>
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<tr>
<td>15</td>
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<td>16</td>
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<td>17</td>
<td><img src="image3.png" alt="Structure 3" /></td>
<td>2-N(2-hydroxy-3,5-diiodophenyl methylene)imino-3- (4-hydroxy phenyl) propanoicacid</td>
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<tr>
<td>18</td>
<td><img src="image4.png" alt="Structure 4" /></td>
<td>2-N (2-hydroxy-3,5-diiodophenyl methylene)imino pentanedioicacid</td>
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<td>19</td>
<td><img src="image5.png" alt="Structure 5" /></td>
<td>2-N(2-hydroxy-3,5-diiodophenyl methylene)imino-6-amino hexanoicacid hydrochloride</td>
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<td>20</td>
<td><img src="image6.png" alt="Structure 6" /></td>
<td>2-N(2-hydroxy-3,5-diiodophenyl methylene)imino-2-phenyl aceticacid</td>
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<td>21</td>
<td><img src="https://example.com/structure21.png" alt="Structure" /></td>
<td>Methyl-2-N- (2-hydroxyphenyl methylene)imino-3-methyl butanoate</td>
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<td><img src="https://example.com/structure22.png" alt="Structure" /></td>
<td>Methyl-2-N (2-hydroxyphenyl methylene)imino-3-(4-hydroxy phenyl) propanoate</td>
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<td><img src="https://example.com/structure23.png" alt="Structure" /></td>
<td>Methyl-2-N (2-hydroxyphenyl methylene)imino-2-phenyl ethanoate</td>
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<td><img src="https://example.com/structure26.png" alt="Structure" /></td>
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<td>Methyl-2-N (2-hydroxy-5-chlorophenyl methylene)imino-2-phenyl ethanoate</td>
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<td>28</td>
<td><img src="image" alt="Methyl-2-N(2-hydroxy-5-bromophenyl methylene)imino-3-methyl butanoate" /></td>
<td>Methyl-2-N(2-hydroxy-5-bromophenyl methylene)imino-3-methyl butanoate</td>
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<td>29</td>
<td><img src="image" alt="Methyl-2-N(2-hydroxy-5-bromophenyl methylene)imino -3-(4-hydroxyphenyl)propanoate" /></td>
<td>Methyl-2-N(2-hydroxy-5-bromophenyl methylene)imino -3-(4-hydroxyphenyl)propanoate</td>
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<td>30</td>
<td><img src="image" alt="Methyl-2-N(2-hydroxy-5-bromophenyl methylene)imino-2-phenyl ethanoate" /></td>
<td>Methyl-2-N(2-hydroxy-5-bromophenyl methylene)imino-2-phenyl ethanoate</td>
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<td><img src="image" alt="Methyl-2-N(2-hydroxy-3,5-diiodophenyl methylene)imino-3-(4-hydroxy phenyl) propanoate" /></td>
<td>Methyl-2-N(2-hydroxy-3,5-diiodophenyl methylene)imino-3-(4-hydroxy phenyl) propanoate</td>
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<td>32</td>
<td><img src="image" alt="Methyl-2-N(2-hydroxy-3,5-diodophenyl methylene)imino-6-amino hexanoate" /></td>
<td>Methyl-2-N(2-hydroxy-3,5-diodophenyl methylene)imino-6-amino hexanoate</td>
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<td>33</td>
<td><img src="image" alt="Methyl-2-N (2-hydroxy 3,5-diiodophenyl methylene)imino-2-phenyl ethanoate" /></td>
<td>Methyl-2-N (2-hydroxy 3,5-diiodophenyl methylene)imino-2-phenyl ethanoate</td>
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<tr>
<td>34</td>
<td><img src="image" alt="Dimethyl-2-N(2-hydroxy-5-nitrophenyl methylene)imino pentanedioate" /></td>
<td>Dimethyl-2-N(2-hydroxy-5-nitrophenyl methylene)imino pentanedioate</td>
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<td>35</td>
<td><img src="image" alt="2-N(2-hydroxy-5-nitrophenyl methylene)imino-3-methyl-butanoate" /></td>
<td>2-N(2-hydroxy-5-nitrophenyl methylene)imino-3-methyl-butanoate</td>
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<td>36</td>
<td><img src="image" alt="Methyl 2-N(2-hydroxy-5-nitrophenyl methylene)imino-3-methyl butanoate" /></td>
<td>Methyl 2-N(2-hydroxy-5-nitrophenyl methylene)imino-3-methyl butanoate</td>
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<td>37</td>
<td><img src="image" alt="Methyl 2-N(2-hydroxy-5-nitrophenyl methylene)imino-3-(4-hydroxy phenyl) propanoate" /></td>
<td>Methyl 2-N(2-hydroxy-5-nitrophenyl methylene)imino-3-(4-hydroxy phenyl) propanoate</td>
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<td>38</td>
<td><img src="image" alt="Methyl 2-N(2-hydroxy 3,5-diiodophenyl methylene)imino-3-methyl butanoate" /></td>
<td>Methyl 2-N(2-hydroxy 3,5-diiodophenyl methylene)imino-3-methyl butanoate</td>
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<tr>
<td>39</td>
<td><img src="image" alt="Methyl 2-N(2-hydroxy-5-nitrophenyl methylene)imino -2-phenyl ethanoate" /></td>
<td>Methyl 2-N(2-hydroxy-5-nitrophenyl methylene)imino -2-phenyl ethanoate</td>
</tr>
</tbody>
</table>
The synthesised compounds were characterized by using $^1$H and $^{13}$C NMR, IR and Mass spectroscopic methods. The details are given below.

2.7 SPECTRAL DATA OF SYNTHESIZED COMPOUNDS

(1) 2-N (2-hydroxyphenyl methylene)imino-3-methyl butanoic acid

<table>
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<th>Property</th>
<th>Value</th>
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<td>Yield</td>
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<td>M.P.</td>
<td>124 – 125°C</td>
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<td>Mol. Formula</td>
<td>C₁₂H₁₅NO₃</td>
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<tr>
<td>Mol. wt.</td>
<td>221</td>
</tr>
</tbody>
</table>

$^1$H NMR (200 MHz, CD₃OD) δ, ppm: 6.60 – 7.38 (m, 4H; C-3′, C-4′, C-5′, C-6′, Ar-H), 3.7 (d, 1H; -C-2, -CH-COOH), 2.5 (m, 1H; C-3, -CH(CH₃)₂, 1.2 (d, 6H; -CH(CH₃)₂, 11.2 (s, 1H; COOH), 8.01 (s, 1H; -CH=N-)

$^{13}$C NMR (200 MHz, CD₃OD) δ, ppm: 123.4 (C-1′), 149.2 (C-2′, -C-OH), 118.0 (C-3′), 131 (C-4′), 125.0 (C-5′), 128 (C-6′), 72.7 (−CH(CH₃)₂, 67.5 (−CH-COOH), 17.7 (−CH(CH₃)₂, 178.0 (−COOH), 162.2 (−CH=N-)

IR (KBr): 3327, 2958, 1601, 1522, 1490, 844, 755, 739 cm⁻¹

M⁺: 221

(2) 2-N(2-hydroxyphenyl methylene)imino-3- (4-hydroxy phenyl) propanoic acid

<table>
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<th>Property</th>
<th>Value</th>
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<td>Yield</td>
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<td>M.P.</td>
<td>224 – 225°C</td>
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<td>Mol. Formula</td>
<td>C₁₆H₁₅NO₄</td>
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<tr>
<td>Mol. wt.</td>
<td>285</td>
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</tbody>
</table>

$^1$H NMR (200 MHz, CDCl₃) δ, ppm: 6.7 – 7.6 (m, 8H; C-3′, C-4′, C-5′, C-6′, C-2′, C-3′, C-5′, C-6′, Ar-H), 3.82 (t, 1H; -CHCH₂), 2.9 (d, 2H; -CHCH₂), 11.3 (s, 1H; -COOH), 8.1 (s, 1H; -CH=N-)

$^{13}$C NMR (200 MHz, CDCl₃) δ, ppm: 119.2 (C-1′), 152 (C-2′, -C-OH), 117.2 (C-3′), 130.2 (C-4′), 128.3 (C-5′), 131.3 (C-6′), 70.1 (C-2, -CHCH₂), 39.3 (C-3, -CH(CH₂), -CH(CH₂), 125.5 (C-1″), 133.1 (C-2″, C-6″), 114.0 (C-3″, C-5″), 174.2 (-COOH), 163.4 (-CH=N-)

IR (KBr): 3452, 3023, 2961, 2931, 1609, 1590, 9454, 841, 795, 740, 713, 650 cm⁻¹

M⁺: 285
(3) 2-N(2-hydroxyphenyl methylene) iminopentanedioic acid

Yield : 87%
M.P. : Decomposed
Mol. Formula : C₁₂H₁₃NO₅
Mol. wt. : 251

¹H NMR (200 MHz, DMSO-d₆) δ, ppm : 6.76–7.14 (m, 4H; C-3’, C-4’, C-5’, C-6’, Ar-H), 4.0 (t, 1H; C-2, -CH-CH₂), 2.15 (m, 2H; C-3, -CH-CH₂-CH₂-), 2.23 (t, 2H; C-4, -CH₂-CH₂-COOH), 11.0 (s, 1H; -(COOH)₂), 8.13 (s, 1H; -CH=N)

¹³C NMR (200 MHz, DMSO-d₆) δ, ppm : 124.5 (C-1’), 157.8 (C-2’, C-OH), 115.8 (C-3’), 132.2 (C-4’), 121.2 (C-5’), 130.4 (C-6’) 67.2 (C-2, -CH-COOH), 30.6 (C-3, -CH-CH₂-CH₂), 30.6 (C-4, CH₂-CH₂-COOH), 177.0 (-COOH)₂, 163.7 (-CH=N)

IR (KBr) : 3449, 2970, 1640, 1560, 1646, 653 cm⁻¹

M⁺ : 250

(4) 2-N (2-hydroxyphenyl methylene) imino-6-aminohexanoic acid hydrochloride

Yield : 67%
M.P. : 198°C
Mol. Formula : C₁₃H₁₉ClN₂O₃
Mol. wt. : 287

¹H NMR (200 MHz, D₂O) δ, ppm : 6.76 – 7.46 (m, 4H; C-3’, C-4’, C-5’, C-6’, Ar-H), 4.00 (t, 1H; C-2, -CH-COOH), 2.65 (t, 2H; C-6, -CH₂-NH₂), 1.88 (m, 2H; -CH-CH₂-), 1.29 – 1.55 (m, 4H; -C-4 & C-5, -CH(CH₂)₂-CH₂-, 2.0 (s, 2H, -CH₂-NH₂), 11.0 (s, 1H; -(COOH)₂), 8.13 (s, 1H; -CH=N-)

¹³C NMR (200 MHz, D₂O) δ, ppm : 124.5 (C-1’), 157.8 (C-2’, C-OH), 115.8 (C-3’), 132.2 (C-4’), 121.2 (C-5’), 130.4 (C-6’) 67.2 (C-2, -CH-COOH), 30.5 (C-3, -CH-CH₂), 22.2 (C-4, -CH-CH₂-(CH₂)₂), 34.1 (C-5, -CH₂-CH₂-CH₂-), 42.3 (C-6’, -CH₂-NH₂) 163.7 (-CH=N), 177.0 (-COOH).

IR (KBr) : 3417, 2937, 1640, 1616, 1501, 753, 735, 704 cm⁻¹

M⁺ : 287
(5) 2-N (2-hydroxyphenyl methylene) imino-2-phenyl acetic acid
Yield: 78%
M.P.: 93 – 95°C
Mol. Formula: C_{15}H_{13}NO_3
Mol. wt.: 255

$^1$H NMR (200 MHz, CD$_3$OD) $\delta$, ppm: 6.76 – 7.14 (m, 9H; C-3', C-4', C-5', C-6', C-2'', C-3'', C-4'', C-5'', C-6'', Ar-H), 5.2 (s, 1H; CH-COOH), 11.0 (s, 1H; -(COOH)), 8.11 (s, 1H; -CH=N)

$^{13}$C NMR (200 MHz, CD$_3$OD) $\delta$, ppm: 124.5 (C-1'), 157.8 (C-2', C-OH), 115.8 (C-3'), 132.2 (C-4'), 121.2 (C-5'), 130.4 (C-6'), 178.0 (C-1, CH-COOH), 71.0 (C-2, CH-COOH), 135.0 (C-1''), 129.9 (C-2'' & C-6''), 128.9 (C-3'' & C-5''), 127.3 (C-4''), 163.7 (-CH=N-)

IR (KBr): 3427, 2978, 1635, 1454 cm$^{-1}$
M$^+$: 254

(6) 2-N(2-hydroxy-5-chlorophenyl methylene)imino-3-methylbutanoic acid
Yield: 82%
M.P.: 145-147°C
Mol. Formula: C_{12}H_{14}ClNO_3
Mol. wt.: 255

$^1$H NMR (200 MHz, CDCl$_3$) $\delta$, ppm: 6.7 – 7.46 (m, 3H; C-3', C-4', C-6', Ar-H), 3.9 (d, 1H; C-2, CH-COOH) 2.2 (m, 1H; C-3, CH(CH$_3$)$_2$), 1.0 (d, 6H, -CH(CH$_3$)$_2$), 11.0 (s, 1H; COOH), 8.13 (s, 1H; -CH=N-)

$^{13}$C NMR (200 MHz, CDCl$_3$) $\delta$, ppm: 125.9 (C-1'), 155.9 (C-2', -C-OH), 117.2 (C-3'), 132.6 (C-4'), 126.5 (C-5', -C-Cl), 130.8 (C-6'), 74.6 (C-2, CH-COOH), 26.7 (C-3, CH-(CH$_3$)$_2$, 17.1 (CH-(CH$_3$)$_2$), 177.0 (-COOH), 163.7 (-CH=N)

IR (KBr): 3417, 2960, 1650, 1513, 1270, 1223, 1165, 1120, 785, 688, 634 cm$^{-1}$
M$^+$: 255
(7) 2-N(2-hydroxy-5-chlorophenyl methylene) imino-3-(4-hydroxy phenyl)propanoic acid

Yield : 76%
M.P. : 208 – 210°C
Mol. Formula : C_{16}H_{14}ClNO_{4}
Mol. wt. : 319

^{1}H NMR (200 MHz, DMSO-d_{6}) δ, ppm : 6.5 – 7.2 (m, 7H; C-3′, C-4′, C-6′, C-2″, C-3″, C-5″, C-6″, Ar-H), 3.87 (t, 1H; -CHCH_{2}), 3.2 (d, 2H; -CH-CH_{2}), 9.63 (s, 1H; -COOH), 8.03 (s, 1H; -CH=N)

^{13}C NMR (200 MHz DMSO-d_{6}) δ, ppm : 120.4 (C-1′), 155.6 (C-2′, C-OH), 118.6 (C-3′), 132.4 (C-4′), 128.0 (C-5′, C-Cl), 130.5 (C-6′), 69.9 (C-2, CH-COOH), 38.2 (C-3, -CHCH_{2}-), 132 (C-1″), 130.0 (C-2″ & C-6″), 115.0 (C-3″ & C-5″), 156.6 (C-4″, C-OH), 172.3 (-COOH), 164.7 (-CH=N)

IR (KBr) : 3400, 2920, 1700, 1580, 1242, 1143, 792, 738, 646 cm\(^{-1}\)

M\(^{+}\) : 319

(8) 2-N (2-hydroxy-5-chlorophenyl methylene) imino pentanedioic acid

Yield : 81%
M.P. : Decomposed
Mol. Formula : C_{12}H_{12}ClNO_{3}
Mol. wt. : 253

^{1}H NMR (200 MHz, CDCl_{3}) δ, ppm : 6.7 – 7.4 (m, 3H; C-3′, C-4′, C-6′, Ar-H), 4.00 (t, 1H; C-2, CHCH_{2}-), 2.15 (m, 2H; C-3, CHCH_{2}-CH_{2}-), 2.23 (t, 2H; C-4, CH_{2}-COOH), 11.0 (s, 1H; (COOH)_{2}), 8.13 (s, 1H; -CH=N-)

^{13}C NMR (200 MHz, CDCl_{3}) δ, ppm : 125.9 (C-1′), 155.9 (C-2′, C-OH), 117.2 (C-3′), 132.6 (C-4′), 126.5 (C-5′, -C-Cl), 130.8 (C-6′), 67.2 (C-2, CH-CH_{2}-), 25.6 (C-3, -CHCH_{2}-CH_{2}-), 30.6 (C-4, CH_{2}-COOH), 177.0 (COOH)_{2}, 163.7 (-CH=N)

IR (KBr) : 3450, 2920, 1644, 1451, 1224, 1150, 1123, 638 cm\(^{-1}\)

M\(^{+}\) : 254
(9) 2-N(2-hydroxy-5-chlorophenyl methylene) imino-6-amino
hexanoic acid hydrochloride

Yield: 89%
M.P.: 167 - 168°C
Mol. Formula: C₁₃H₁₈Cl₂N₂O
Mol. wt.: 321

¹H NMR (200 MHz, D₂O) δ, ppm: 6.7 – 7.4 (m, 3H; C-3′, C-4′, C-6′, Ar-H), 3.9 (t, 1H; C-2, -CH-CH₂), 2.0 (m, 2H; C-3, CH-CH₂(CH₂)₃), 1.29 (m, 2H; C-4, -CH-CH₂-CH₂-CH₂-), 1.55 (m, 2H; C-5, -CH₂-CH₂NH₂), 2.65 (t, 2H; C-6, CH₂-NH₂), 2.0 (s, 2H; -CH₂-NH₂), 11.0 (s, 1H; COOH), 8.1 (s, 1H; -CH=N),

¹³C NMR (200 MHz, D₂O) δ, ppm: 125.9 (C-1′), 155.9 (C-2′, -C=OH), 117.2 (C-3′), 132.6 (C-4′), 126.5 (C-5′, C-Cl), 130.5 (C-6′), 65.3 (C-2, -CH=CH₂), 30.7 (C-3, -CH-CH₂-), 23.2 (C-4, -CH₂-(CH₂)₂-CH₂-), 34.1 (C-5, CH₂-CH₂-NH₂), 42.3 (C-6, CH₂-NH₂), 177.0 (-COOH), 163.4 (-C=H=N)

IR (KBr): 3217, 2940, 1678, 1656, 1567, 1468, 1263, 1154, 885, 829, 767, 698, cm⁻¹
M⁺: 323 (M+2)

(10) 2-N(2-hydroxy-5-chlorophenyl methylene)imino-2-phenylacetic acid

Yield: 88%
M.P.: Decomposed
Mol. Formula: C₁₅H₁₂ClNO₃
Mol. wt.: 290

¹H NMR (200 MHz, CDCl₃) δ, ppm: 6.7 – 7.46 (m, 8H; C-3′, C-4′, C-6′, C-2′, C-3′, C-4′, C-5′, C-6′, Ar-H), 5.29 (s, 1H; C-2, -CH-COOH), 11.0 (s, 1H; COOH), 8.11 (s, 1H; CH=N)

¹³C NMR (200 MHz, CDCl₃) δ, ppm: 125.9 (C-1′), 155.9 (C-2′, -C=OH), 117.2 (C-3′), 132.6 (C-4′), 126.5 (-C-5′, C-Cl), 130.8 (C-6′), 71.0 (C-2, -CH=COOH), 135.0 (C-1′), 129.9 (C-2′ & C-6′), 128.9 (C-3′ & C-5′), 127.3 (C-4′), 178.0 (COOH), 163.7 (-CH=N-)

IR (KBr): 3417, 2978, 1637, 1581, 1522, 1274, 1200, 1165, 729, 695 cm⁻¹
M⁺: 289
(11) 2-N(2-hydroxy-5-bromophenyl methylene) imino-3-methyl butanoic acid

Yield : 79%
M.P. : 124 - 126°C
Mol. Formula : C_{12}H_{14}BrNO_3
Mol. wt. : 300

^1H NMR (200 MHz, CD_3OD) δ, ppm : 6.65 – 7.62 (m, 3H; C-3', C-4', C-6', Ar-H), 3.9 (d, 1H; CH-CH(CH_3)_2), 2.2 (m, 1H; C-3, CH(CH_3)_2), 1.0 (d, 6H, CH(CH_3)_2), 11.0 (s, 1H; COOH), 8.1 (s, 1H; -CH=N)

^13C NMR (200 MHz, CD_3OD) δ, ppm : 126.7 (C-1'), 156.8 (C-2', C-OH), 118.0 (C-3'), 135.5 (C-4'), 115.8 (C-5', C-Br), 133.7 (C-6'), 74.6 (C-2, CH-COOH), 26.7 (C-3, CH(CH_3)_2), 17.1 (CH-(CH_3)_2), 177.0 (COOH), 163.7 (CH=N)

IR (KBr) : 3453, 2958, 1708, 1641, 1512, 1480, 1329, 1272, 1165, 875, 831 cm\(^{-1}\)
M^+ : 300

(12) 2-N(2-hydroxy-5-bromophenyl methylene) imino-3-(4-hydroxy phenyl) propanoic acid

Yield : 75%
M.P. : 105°C
Mol. Formula : C_{16}H_{14}BrNO_3
Mol. wt. : 364

^1H NMR (200 MHz, CD_3OD) δ, ppm : 6.65 – 7.62 (m, 8H; C-3', C-4', C-6', C-2'', C-3'', C-5'', C-6'', Ar-H), 4.39 (t, 1H; -CH – CH_2), 3.14 (d, 2H; -CH-CH_2), 11.0 (s, 1H, COOH), 8.2 (s, 1H; CH=N)

^13C NMR (200 MHz, CD_3OD) δ, ppm : 126.7 (C-1'), 156.8 (C-2', C-OH), 118.0 (C-3'), 135.5 (C-4'), 115.8 (C-5', C-Br), 133.7 (C-6'), 132.8 (C-1''), 129.3 (C-2'' & C-6''), 115.6 (C-3'' & C-5''), 154.5 (C-4'', C-OH), 69.9 (C-2, CH-COOH), 36.3 (C-3, -CH_2-C_6H_5), 177.0 (COOH), 163.7 (CH=N)

IR (KBr) : 3452, 3024, 1610, 1590, 1514, 1465, 1331, 1275, 1211, 893, 877, 840, 829, 767, 650 cm\(^{-1}\)

M^+ : 364
(13) 2-N(2-hydroxy-5-bromophenyl methylene) imino pentanedioic acid

Yield : 68%
M.P. : Decomposed
Mol. Formula : C_{12}H_{12}BrNO_5
Mol. wt. : 330

^1H NMR (200 MHz, CD_3OD) δ, ppm : 6.6 – 7.6 (m, 3H; C-3’, C-4’, C-6’, Ar-H), 4.0 (t, 1H; CH-CH_2-), 2.15 (m, 2H; C-3, -CH-CH_2-CH_2-), 2.23 (t, 2H, C-4, -CH_2- COOH), 11.2 (s, 1H; COOH), 8.2 (s, 1H; CH=N)

^13C NMR (200 MHz, CD_3OD) δ, ppm : 126.7 (C-1’), 156.8 (C-2, C-OH), 118.0 (C-3’), 135.5 (C-4’), 115.8 (C-5’, C-Br), 133.7 (C-6’), 67.2 (C-2, CH-CH_2), 25.6 (C-3, -CH_2-CH_2), 30.6 (C-4, CH-CH_2-CH_2-), 177.0 (COOH)_2, 163.7 (C=NH)

IR (KBr) : 3452, 2920, 1637, 1585, 1478, 1459, 1295, 1278, 1208, 780, 767, 627 cm\(^{-1}\)

M\(^+\) : 330

(14) 2-N(2-hydroxy-5-bromophenyl methylene) imino-6-aminohexanoic acid hydrochloride

Yield : 85%
M.P. : 205 - 206°C
Mol. Formula : C_{13}H_{18}BrClN_2O
Mol. wt. : 366

^1H NMR (200 MHz, D_2O) δ, ppm : 6.65 – 7.62 (m, 3H; C-3’, C-4’, C-6’, Ar-H), 4.0 (t, 1H; C-2, CH-CH_2-), 1.8 (m, 1H; C-3, -CH-CH_2-(CH_2)_2), 1.29 (m, 2H; CH-CH_2-CH_2-(CH_2)_2), 1.55 (m, 2H; CH-CH_2-CH_2-(CH_2), 2.65 (t, 2H; CH_2-NH_2), 2.0 (s, 2H; -CH_2–NH_2), 11.2 (s, 1H; COOH), 8.2 (s, 1H; CH=N)

^13C NMR (200 MHz, D_2O) δ, ppm : 126.7 (C-1’), 156.8 (C-2, C-OH), 118.0 (C-3’), 135.5 (C-4’), 115.8 (C-5’, C-Br), 133.7 (C-6’), 67.2 (C-2, CH-CH_2), 25.6 (C-3, -CH-CH_2-(CH_2)_3), 21.0 – 24.06 (C-4 & C-5), CH-CH_2 (CH_2)_2), 42.3 (C-6, CH_2NH_2), 177.0 (COOH), 162.0 (CH=N)

IR (KBr) : 3470, 2935, 1700, 1581, 1519, 1328, 1275, 1183, 1128, 735, 698 cm\(^{-1}\)

M\(^+\) : 330 (M-HCl)
(15) 2-N(2-hydroxy-5-bromophenyl methylene)imino-2-phenyl acetic acid

Yield : 89%
M.P. : 92-94°C
Mol. Formula : C_{15}H_{12}BrNO_3
Mol. wt. : 344

^1^H NMR (200 MHz, CDCl_3) δ, ppm : 6.65 – 7.62 (m, 8H; C-3′, C-4′, C-6′, C-2′′, C-3′′, C-4′′, C-5′′, C-6′′, Ar-H), 5.29 (s, 1H; C-2, -CH-COOH), 11.0 (s, 1H; COOH), 8.1 (s, 1H; CH= N)

^1^C NMR (200 MHz, CDCl_3) δ, ppm : 126.7 (C-1′), 156.8 (C-2′, -C-OH), 118.0 (C-3′), 135.5 (C-4′), 115.8 (C-5′, -C-Br), 133.7 (C-6′), 71.0 (C-2, CH-COOH), 129.9 (C-2′ & C-6′), 128.9 (C-3′ & C-5′), 127.3 (C-4′′), 178.0 (COOH), 164.0 (CH=N)

IR (KBr) : 3423, 2978, 1637, 1580, 1277, 1201, 1151, 1075, 729, 695 cm\(^{-1}\)
M^+ : 344

(16) 2-N(2-hydroxy-3,5-diiodophenyl methylene)imino-3-methyl butanoic acid

Yield : 78%
M.P. : Decomposed
Mol. Formula : C_{12}H_{13}I_2NO_3
Mol. wt. : 473

^1^H NMR (200 MHz, CDCl_3) δ, ppm : 7.82 – 7.88 (s, 2H; C-4′, C-6′, Ar-H), 3.9 (d, 1H; C-2, -CH-CH-(CH_3)\(_2\)), 2.2 (m, 1H; CH(CH_3)_2), 0.9 (d, 6H; CH(CH_3)_2), 11.0 (s, 1H; COOH), 8.23 (s, 1H; CH=N)

^1^C NMR (200 MHz, CDCl_3) δ, ppm : 127.7 (C-1′), 165.6 (C-2′, -C-OH), 86.2 (C-3′, C-I), 150.0 (C-4′), 91.6 (C-5′, -C-I), 138.2 (C-6′), 74.6 (C-2, -CH-CH-(CH_3)_2), 26.7 (C-3, -CH-(CH_3)_2), 17.1 (CH(CH_3)_2), 177.0 (COOH), 162.9 (CH=N)

IR (KBr) : 3357, 2962, 1628, 1600, 1477, 1218, 1120, 1098, 757, 629, 629 cm\(^{-1}\)
M^+ : 473
(17) 2-N(2-hydroxy-3,5-diiodophenyl methylene) imino-3-(4-hydroxy phenyl) propanoicacid

Yield : 68%
M.P. : 108-110°C
Mol. Formula : C₁₆H₁₃I₂NO₄
Mol. wt. : 537

¹H NMR (200 MHz, CDCl₃) δ, ppm : 6.68 – 7.82 (m, 6H; C-4’, C-6’, C-2’”, C-3’’, C-4’’, C-6’’, Ar-H), 4.3 (t, 1H; -CH=CH₂), 3.26 (d, 2H; C-3, -CH₃-CH₂-C₆H₄OH), 11.2 (s, 1H; COOH), 8.1 (s, 1H; CH=N)

¹³C NMR (200 MHz, CDCl₃) δ, ppm : 127.7 (C-1’), 165.6 (C-2’, -C-OH), 86.2 (C-3’, -C-I), 150.0 (C-4’), 91.6 (C-5’, -C-I), 138.2 (C-6’), 67.4 (-CH-CH₂), 36.6 (CH₂-CH₂-C₆H₄) 132.8 (C-1”), 129.3 (C-2’” & C-6’”), 115.5 (C-3’” & C-5’”), 154.5 (C-4’”, -COOH), 177.0 (COOH), 163.0 (CH==N)

IR (KBr) : 3417, 2900, 1637, 1581, 1513, 1330, 1243, 1215, 1130, 110, 665 cm⁻¹
M⁺ : 536

(18) 2-N (2-hydroxy-3,5-diiodophenyl methylene)imino pentanedioic acid

Yield : 71%
Nature : Semi solid
Mol. Formula : C₁₂H₁₁I₂NO₅
Mol. wt. : 503

¹H NMR (200 MHz, CDCl₃) δ, ppm : 7.82 (s, 2H; C-4’, C-6’, Ar-H), 4.00 (t, 1H; C-2, -CH-CH₂), 2.15 (s, 2H; C-3, CH₂-CH₂-CH₂), 2.23 (t, 2H; C-4, -CH₂-COOH), 11.0 (s, 1H; (COOH)₂), 8.13 (s, 1H; -CH=H)

¹³C NMR (200 MHz, CDCl₃) δ, ppm : 127.7 (C-1’), 165.6 (C-2’, -C-OH), 86.2 (C-3’, -C-I), 150.0 (C-4’), 91.6 (C-5’, -C-I), 138.2 (C-6’), 67.4 (C-2, CH₂-CH₂-CH₂), 30.6 (C-4; CH₂-CH₂-COOH), 25.6 (C-3, CH₂-CH₂-CH₂), 163.7 (-CH=H), 177.0 (-COOH)₂,
IR (KBr) : 3418, 2890, 1637, 1573, 1479, 738 cm⁻¹
M⁺ : 503
(19) 2-N(2-hydroxy-3,5-diiodophenyl methylene)imino-6-amino hexanoic acid hydrochloride

Yield : 71%
M.P. : 90 – 92°C
Mol. Formula : C_{13}H_{17}ClI_{2}N_{2}O_{3}
Mol. wt. : 538

{\textsuperscript{1}}H NMR (200 MHz, D_{2}O) δ, ppm : 7.82 (s, 1H; C-4′, C-6′, Ar-H), 4.00 (t, 1H; C-2, -CH-CH_{2}_), 1.88 (m, 2H; C-3, -CH-CH_{2}(CH_{2})_{3}), 1.29 (m, 2H; C-4, CH_{2}-CH_{2}(CH_{2})_{2}), 1.55 (m, 2H; C-5, CH-(CH_{2})-CH_{2}-CH_{2}), 2.65 (t, 2H; C-6, -CH_{2}-NH_{2}), 2.00 (s, 2H; CH_{2}-CH_{2}-NH_{2}), 11.2 (s, 1H; COOH), 8.06 (s, 1H; -CH=N)

{\textsuperscript{13}}C NMR (200 MHz, D_{2}O) δ, ppm : 127.7 (C-1′), 165.6 (C-2′, -C-OH), 86.2 (C-3′, -I), 150.0 (C-4′), 91.6 (C-5′, -I), 138.2 (C-6′), 67.4 (C-2, CH-CH_{2}-), 25.6 (C-3, -CH-CH_{2}-CH_{2}), 30.6 (C-4, CH_{2}-CH_{2}-), 34.2 (C-5, -CH_{2}-CH_{2}), 43.2 (C-6, -CH_{2}-NH_{2}), 177.0 (-COOH), 163.7 (-C=I)

IR (KBr) : 3443, 2990, 1638, 1415, 1221, 1130, 864 cm\(^{-1}\)

M\(^{+}\) : 502 (M-HCl)

(20) 2-N(2-hydroxy-3,5-diiodophenyl methylene)imino-2-phenyl acetic acid

Yield : 85%
M.P. : 97°C
Mol. Formula : C_{15}H_{11}I_{2}NO_{3}
Mol. wt. : 507

{\textsuperscript{1}}H NMR (200 MHz, CDCl_{3}) δ, ppm : 6.70 – 7.88 (m, 7H; C-4, C-6, C-2″, C-3″, C-4″, C-5″, C-6″, Ar-H), 5.1 (s, 1H; -CH-COOH), 10.82 (s, 1H; -COOH), 8.12 (s, 1H; -CH=N)

{\textsuperscript{13}}C NMR (200 MHz, CDCl_{3}) δ, ppm : 127.8 (C-1′), 164.2 (C-2′, -C-OH), 85.8 (C-3′, -I), 148.9 (C-4′), 92.10 (C-5′, -I), 139.0 (C-6′), 72.2 (C-2, CH-COOH), 134.1 (C-1″), 129.9 (C-2″, C-6″), 128.7 (C-3″ & C-5″), 127.4 (C-4″), 176.2 (-COOH), 163.7 (-CH=N)

IR (KBr) : 3417, 2960, 1635, 1578, 1480, 1453, 1215, 1131, 696 cm\(^{-1}\)

M\(^{+}\) : 507
(21) Methyl-2-N-(2-hydroxyphenyl methylene) imino-3-methyl butanoate

Yield : 90%
Nature : Pale yellow dense liquid
Mol. Formula : C_{13}H_{17}NO_{3}
Mol. wt. : 235

$^{1}$H NMR (200 MHz, CDCl$_3$) $\delta$, ppm : 13.2 (s, 1H; Ar-OH), 6.9 – 7.11 (m, 4H; C-3\', C-4\', C-5\', C-6\', Ar-H), 3.76 (s, 3H; -CH-COOCH$_3$), 2.39 (m, 1H, -CH(CH$_3$)$_2$), 0.9 – 1.2 (d, 6H, CH(CH$_3$)$_2$), 8.24 (s, 1H; -CH=N)

$^{13}$C NMR (200 MHz, CDCl$_3$) $\delta$, ppm : 123.2 (C-1\'), 159.6 (C-2\', C-OH), 118.7 (C-3\'), 132.5 (C-4\'), 119.2 (C-5\'), 130.7 (C-6\'), 76.3 (C-2, CH-COOCH$_3$), 171.2 (C-1, -COOCH$_3$), 52.2 (-COOC$_H$H$_3$), 31.8 (C$_H$(CH$_3$)$_2$), 19.3 (-CH(C$_H$3)$_2$, 165.3 (-CH=N)

IR (KBr) : 3350, 2978, 1700, 1581, 1519, 729, 653 cm$^{-1}$
M$^+$ : 235

(22) Methyl-2-N (2-hydroxyphenyl methylene) imino-3-(4-hydroxy phenyl) propanoate

Yield : 75%
M.P. : 129 - 130°C
Mol. Formula : C$_{17}$H$_{17}$NO$_4$
Mol. wt. : 299

$^{1}$H NMR (200 MHz, CD$_3$OD) $\delta$, ppm : 6.7 – 7.8 (m, 8H; C-3\', C-4\', C-5\', C-6\', C-2\'', C-3\'', C-5\'', C-6\'', Ar-H), 3.7 (s, 3H; C-1, COOCH$_3$), 4.2 (t, 1H; C-2, -CH-CH$_2$-), 3.26 (d, 2H; C-3, -CH-CH$_2$-), 8.07 (S, 1H; -CH=N)

$^{13}$C NMR (200 MHz, CD$_3$OD) $\delta$, ppm : 119.8 (C-1\'), 162.3 (C-2\', C-OH), 117.7 (C-3\'), 133.0 (C-4\'), 116.2 (C-5\'), 133.8 (C-6\'), 67.0 (C-2), 37.0 (C-3), 133.0 (C-1\''), 131.7 (C-2\'' & C-6\''), 128.7 (C-3\'' & C-5\'') 150 (C-4\''), 173.3 (-COOCH$_3$), 52.7 (COOCH$_3$), 162.3 (CH=N)

IR (KBr) : 3456, 2956, 1741, 1644, 1533, 764, 742 cm$^{-1}$
M$^+$ : 299
(23) Methyl-2-N (2-hydroxyphenyl methylene) imino-2-phenyl ethanoate

Yield: 78%
M.P.: 58 - 59°C
Mol. Formula: C_{16}H_{15}NO_3
Mol. wt.: 269

^{1}H NMR (200 MHz, CDCl_3) δ, ppm: 13.12 (s, 1H; C-2', Ar-OH), 6.8 – 7.4 (m, 9H, C-3', C-4', C-5', C-6', C-2'', C-3'', C-4'', C-5'', C-6'', Ar-H) 3.63 (s, 3H; COOCH_3), 3.74 (s, 1H; C-2, -CH-COOCH_3), 8.3 (s, 1H; -CH=N)

^{13}C NMR (200 MHz, CDCl_3) δ, ppm: 127.6 (C-1'), 160.9 (C-2', C-OH), 117.1 (C-3'), 132.9 (C-4'), 128.4 (C-5'), 128.9 (C-6'), 68.0 (C-2), 136.9 (C-1'') 131.8 (C-2'' & C-6'') 128.9 (C-3'' & C-5''), 127.6 (C-4''), 170.7 (-COOCH_3) 52.7 (COOCH_3), 166.8 (-CH=N)

IR (KBr): 3415, 2950, 1743, 1630, 1577, 762, 742 696 cm^{-1}
M^+: 269

(24) Methyl-2-N (2-hydroxy-5-chlorophenyl methylene) imino-3-methyl butanoate

Yield: 90%
M.P.: 76 - 77°C
Mol. Formula: C_{13}H_{16}ClNO_3
Mol. wt.: 269

^{1}H NMR (200 MHz, CDCl_3) δ, ppm: 13.13 (s, 1H; C-2', Ar-OH), 6.90 – 7.32 (m, 3H; C-3', C-4', C-6', Ar-H), 3.77 (s, 3H; -COOCH_3), 3.79 (d, 1H, C-2, -CH-CH- (CH_3)_2), 2.38 – 2.41 (m, 1H; C-3, -CH(CH_3)_2), 0.96 - 1.0 (d, 6H; CH(CH_3)_2), 8.26 (s, 1H; -CH=N-)

^{13}C NMR (200 MHz, CDCl_3) δ, ppm: 119.27 (C-1'), 159.7 (C-2', -C-OH), 118.7 (C-3'), 132.5 (C-4'), 123.3 (C-5', C-Cl), 130.7 (C-6') 171.2 (C-1, -COOCH_3), 72.1 (C-2), 31.8 (C-3), 18.1 (CH(CH_3)_2), 52.2 (-COOCH_3) 165.3 (-CH=N)

IR (KBr): 3449, 2968, 1730, 1634, 1573, 1280, 1258, 1185, 1089, 781, 645 cm^{-1}
M^+: 269
(25) Methyl-2-N (2-hydroxy-5-chlorophenyl methylene) imino -3-(4-hydroxy phenyl) propanoate

Yield : 79%
M.P. : 137 - 138°C
Mol. Formula : C_{17}H_{16}ClNO_{4}
Mol. wt. : 333

^1H NMR (200 MHz, D_{2}O) δ, ppm : 12.98 (s, 1H; C-2', Ar-OH), 6.7 – 7.5 (m, 7H, C-3', C-4', C-6', C-2''', C-3'', C-6'', Ar-H), 3.76 (s, 3H; COOCH_{3}), 4.15 (t, 1H, C-2, -CH-CH_{2}-), 3.1 (dd, 2H; C-3, -CH-CH_{2}-) 7.8 (s, 1H; -CH=N)

^13C NMR (200 MHz, D_{2}O) δ, ppm : 123.3 (C-1'), 154.7 (C-2', C-OH), 118.7 (C-3'), 132.6 (C-4'), 128.4 (C-5', C-Cl), 130.7 (C-6'), 171.2 (C-1, -COOCH_{3}), 73.1 (C-2), 39.1 (C-3), 132.6 (C-1''), 128.2 (C-2'' & C-6''), 115.4 (C-3'' & C-5''), 154.7 (C-4''), 52.5 (-COOC_{3}H_{3}), 165.6 (-CH=N).

IR (KBr) : 3453, 2900, 1737, 1645, 1592, 1279, 1248, 1230, 1214, 784, 681, 630 cm\(^{-1}\)
M\(^{+}\) : 333

(26) Dimethyl-2-N (2-hydroxy -5-chlorophenyl methylene) imino pentanedioate

Yield : 68%
M.P. : Decomposed
Mol. Formula : C_{14}H_{16}ClNO_{5}
Mol. wt. : 313

^1H NMR (200 MHz, CDCl_{3}) δ, ppm : 12.76 (s, 1H; C-2', Ar-OH), 6.9 – 7.26 (m, 3H; C-3', C-4', C-6', Ar-H_{2}), 3.76 (s, 3H; COOCH_{3}), 4.12 (t, 1H; C-2, -CH-CH_{2}), 2.37 (m, 2H; C-3, -CH-CH_{2}-CH_{2}-), 2.39 (t, 2H; C-4, -CH-CH_{2}-CH_{2}-COOCH_{3}), 3.66 (s, 3H; -COOC_{3}H_{3}) 8.3 (s, 1H; -CH=N-)

^13C NMR (200 MHz, CDCl_{3}) δ, ppm : 123.4 (C-1'), 159.4 (C-2', C-OH), 118.6 (C-3'), 132.4 (C-4'), 123.4 (C-5', C-Cl), 130.8 (C-6'), 172.8 (C-1, -COOCH_{3}), 70.0 (C-2), 28.3 (C-3), 29.8 (C-4), 172.8 (C-5, -COOCH_{3}) 52.5 (-COOC_{3}H_{3}), 166.5 (-CH=N)

IR (KBr) : 3370, 2953, 1731, 1676, 1479, 1272, 1207, 1173, 1113, 767, 702, 644 cm\(^{-1}\)
M\(^{+}\) : 313
(27) Methyl-2-N (2-hydroxy-5-chlorophenyl methylene) imino-2-phenyl ethanoate

Yield : 72%
M.P. : 102 - 103°C
Mol. Formula : C₁₆H₁₄ClNO₃
Mol. wt. : 303

$^1$H NMR (200 MHz, CDCl₃) δ, ppm: 13.13 (s, 1H; C-2', Ar-OH), 6.89 – 7.4 (m, 8H; C-3', C-4', C-6', C-2", C-3", C-4", C-5", Ar-H), 3.75 (s, 3H; COOCH₃), 5.19 (s, 1H; C-2, CH-COOCH₃), 8.2 (s, 1H; CH=N).

$^{13}$C NMR (200 MHz, CDCl₃) δ, ppm: 123.3 (C-1'), 159.5 (C-2'; C=OH), 118.7 (C-3'), 135.5 (C-4'), 119.1 (C-5'; C=Cl), 133.8 (C-6'), 170.4 (C-1, -COOCH₃), 52.7 (-COOCH₃), 74.4 (C-2), 135.5 (C-1'"), 130.8 (C-2" & C-6'"), 128.9 (C-3'" & C-5'"), 127.6 (C-4'"), 165.5 (-CH=N)

IR (KBr): 3415, 2920, 1748, 1636, 1570, 1319, 1279, 1206, 1169, 750, 697, 644 cm⁻¹
M⁺: 303

(28) Methyl-2-N (2-hydroxy-5-bromophenyl methylene) imino-3-methyl butanoate

Yield : 94%
M.P. : 81 - 82°C
Mol. Formula : C₁₃H₁₆BrNO₃
Mol. wt. : 314

$^1$H NMR (200 MHz, CDCl₃) δ, ppm: 13.17 (s, 1H; C-2', Ar-OH), 6.90 – 7.32 (m, 3H; C-3', C-4', C-6', Ar-H), 3.76 (s, 3H; COOCH₃), 3.85 (d, 1H; C-2, CH-COOCH₃), 2.36 – 2.46 (m, 1H; C-3, -CH(CH₃)₂), 0.82 – 0.97 (d, 6H; -CH(CH₃)₂), 8.4 (s, 1H; CH=N)

$^{13}$C NMR (200 MHz, CDCl₃) δ, ppm: 119.9 (C-1'), 160.1 (C-2', C=OH), 119.1 (C-3'), 133.7 (C-4'), 110.1 (C-5', C=Br), 135.3 (C-6'), 171.1 (C-1, COOCH₃), 52.1 (-COOCH₃), 72.1 (C-2), 31.8 (C-3), 18.0 (CH(CH₃)₂), 165.3 (-CH=N)

IR (KBr): 3450, 2968, 1728, 1632, 1558, 1333, 1280, 1258, 1185, 1144, 750, 626 cm⁻¹
M⁺: 315
(29) Methyl-2-N(2-hydroxy-5-bromophenyl methylene) imino –3-(4-hydroxyphenyl)propanoate

Yield : 78%
M.P. : 150 - 151°C
Mol. Formula : C_{17}H_{16}BrNO_{4}
Mol. wt. : 378

^{1}H NMR (200 MHz, CDCl_{3}) \delta, ppm : 13.05 (s, 1H; C-2', Ar-OH), 6.8 – 7.4 (m, 7H, C-3', C-4', C-6', C-2'', C-3'', C-5'', C-6'', Ar-H), 3.76 (s, 3H; COOCH_{3}), 4.12 (t, 1H, C-2), 3.27 – 3.33 (dd, 2H, C-3), 8.04 (s, 1H; -CH=N)

^{13}C NMR (200 MHz, CDCl_{3}) \delta, ppm : 128.4 (C-1'), 154.5 (C-2', C-Oh), 119.1 (C-3'), 135.4 (C-4'), 115.4 (C-5', C-Br), 133.7 (C-6'), 171.1 (C-1, -COOCH_{3}), 52.5 (-COOCH_{3}), 73.1 (C-2), 39.0 (C-3), 133.7 (C-1''), 130.7 (C-2'' & C-6''), 115.4 (C-3'' & C-5''), 154.6 (C-4'', C-Oh), 165.5 (-CH=N).

IR (KBr) : 3415, 3005, 1736, 1644, 1518, 838, 1334, 1278, 1248, 1158, 1105, 738, 617 cm\(^{-1}\)
M^+ : 377

(30) Methyl-2-N (2-hydroxy-5-bromophenyl methylene) imino-2-phenyl ethanoate

Yield : 83%
M.P. : 115 - 116°C
Mol. Formula : C_{16}H_{14}BrNO_{3}
Mol. wt. : 348

^{1}H NMR (200 MHz, CDCl_{3}) \delta, ppm : 13.14 (s, 1H; C-2', Ar-OH), 6.86 – 7.48 (m, 8H; C-3', C-4', C-6', C-2'', C-3'', C-4'', C-5'', C-6'', Ar-H), 3.76 (s, 3H; COOCH_{3}), 5.26 (s, 1H; C-2, -CH-COOCH_{3}), 8.28 (-CH=N)

^{13}C NMR (200 MHz, CDCl_{3}) \delta, ppm : 127.7 (C-1'), 160.0 (C-2', C-Oh), 119.2 (C-3'), 135.6 (C-4'), 119.5 (C-5', C-Br), 133.9 (C-6'), 170.4 (C-1, -COOCH_{3}), 52.7 (-COOCH_{3}), 74.5 (C-2), 136.5 (C-1''), 129.03 (C-2'' & C-6''), 128.7 (C-3'' & C-5''), 127.7 (C-4''), 165.5 (-CH=N).

IR (KBr) : 3415, 2920, 1748, 1636, 1568, 1318, 1278, 1206, 1169, 752, 698 cm\(^{-1}\)
M^+ : 347
(31) Methyl-2-N (2-hydroxy-3,5-diiodophenyl methylene) imino-3-(4-hydroxy phenyl) propanoate

Yield: 68%
M.P.: 77 - 78°C
Mol. Formula: C₁₇H₁₅I₂NO₄
Mol. wt.: 551

¹H NMR (200 MHz, CD₃OD) δ, ppm: 6.9 – 7.8 (m, 6H; C-4’, C-6’, C-2’”, C-3”, C-5”, C-6”, Ar-H), 3.76 (s, 3H; -CH-COOCH₃), 3.8 (t, 1H; C-2, CH-COOCH₃), 3.1 (d, 2H; CH-CH₂), 8.04 (s, 1H; -CH=N)

¹³C NMR (200 MHz, CD₃OD) δ, ppm: 119.7 (C-1’), 160.2 (C-2’, -C-OH), 119.4 (C-3’, C-I), 140.0 (C-4’), 119.4 (C-5’, C-I), 140.0 (C-6’), 170.1 (C-1, COOCH₃), 50.2 (-COOCH₃), 72.0 (C-2), 25.6 (C-3, -CH-CH₂), 129.9 (C-2” & C-6”), 128.9 (C-3” & C-5”), 157.3 (C-4”), 163.7 (CH=N)

IR (KBr): 3425, 2980, 1739, 1634, 1514, 1301, 1240, 1212, 1120, 1095, 831, 657 cm⁻¹

M⁺: 551

(32) Methyl-2-N (2-hydroxy-3,5-diiodophenyl methylene) imino-6-amino hexanoate hydrochloride

Yield: 58%
M.P.: 153-154°C
Mol. Formula: C₁₄H₁₉ClI₂N₂O₃
Mol. wt.: 552

¹H NMR (200 MHz, CDCl₃) δ, ppm: 7.82 – 7.88 (s, 2H; C-4’, C-6’, Ar-H), 3.9 (t, 1H; C-2, -CH-CH₂), 2.00 (m, 2H; C-3, CH-CH₂ (CH₂)₃), 1.29 (m, 2H; C-4, -CH-CH₂-CH₂(CH₂)₃), 1.55 (m, 2H; C-5, CH(CH₂)₂-CH₂), 2.65 (t, 2H; C-6, -CH₂-NH₂), 3.6 (s, 3H; COOCH₃), 2.0 (s, 2H; -CH₂-NH₂), 8.23 (s, 1H; -CH=N)

¹³C NMR (200 MHz, CDCl₃) δ, ppm: 127.7 (C-1’), 165.6 (C-2’, -C-OH), 86.2 (C-3’, C-I), 150.0 (C-4’), 91.6 (C-5’, C-I), 138.2 (C-6’), 65.3 (C-2, CH-CH₂), 30.8 (C-3, CH-CH₂(CH₂)₃), 22.2 (C-4, CH-CH₂-CH₂(CH₂)₃), 34.1 (C-5”, -CH₂-CH₂-NH₂), 42.3 (C-6’, -CH₂-NH₂), 172.0 (COOCH₃), 50.4 (COOCH₃), 162.7 (-CH=N)

IR (KBr): 3415, 2944, 1622, 1580, 1501, 1466, 1436, 1279, 1226, 1155, 1128, 861, 796, 656 cm⁻¹

M⁺: 551
(33) Methyl-2-N (2-hydroxy 3,5-diiodophenyl methylene) imino-2-phenyl ethanoate

Yield : 62%
Nature : Semi solid
Mol. Formula : C_{16}H_{13}I_{2}NO_{3}
Mol. wt. : 521

$^1$H NMR (200 MHz, CDCl$_3$) δ, ppm : 14.3 (s, 1H; C-2', Ar-OH), 7.26 – 7.82 (m, 7H; C-4', C-6', C-2'', C-3'', C-4'', C-5'', C-6'', Ar-H), 3.76 (s, 3H; -COOCH$_3$), 5.23 (s, 1H; C-2'), 8.06 (-CH=N)

$^{13}$C NMR (200 MHz, CDCl$_3$) δ, ppm : 127.7 (C-1'), 160.2 (C-2', C-OH), 79.5 (C-3', C-I), 129.1 (C-4'), 87.27 (C-5', C-I), 136.1 (C-6'), 170.0 (C-1, -COOCH$_3$), 52.8 (-COOCH$_3$), 73.8 (C-2), 137 (C-1''), 132.8 (C-2'' & C-6''), 130.1 (C-3'' & C-5'') 127.7 (C-4''), 164.6 (-CH=N)

IR (KBr) : 3449, 3010, 1745, 1625, 1625, 1493, 1327, 1284, 1251, 1205, 1170, 1155, 746, 692, 656 cm$^{-1}$
M$^+$ : 520

(34) Dimethyl-2-N(2-hydroxy-5-nitrophenyl methylene) imino pentanedioate

Yield : 50%
M.P. : Decomposed
Mol. Formula : C$_{13}$H$_{14}$N$_2$O$_7$
Mol. wt. : 310

$^1$H NMR (200 MHz, CDCl$_3$) δ, ppm : 7.02 – 8.38 (m, 3H; C-3', C-4', C-6', Ar-H), 3.96 (t, 1H; C-2,CH-CH$_2$), 2.39 (m, 2H; C-3, CH-CH$_2$-CH$_2$-), 2.25 (t, 2H; C-4, -CH$_2$COOCH$_3$) 3.67 (s, 6H; (COOCH$_3$)$_2$), 8.1 (s, 1H; -CH=N)

$^{13}$C NMR (200 MHz, CDCl$_3$) δ, ppm : 125.4 (C-1'), 163.9 (C-2', C-OH), 116.7 (C-3'), 127.3 (C-4'), 141.1 (C-5', C-NO$_2$), 125.5 (C-6'), 64.7 (C-2, -CH-CH$_2$), 26.2 (C-3, CH-CH$_2$-), 28.1 (C-4, -CH$_2$-CH$_2$-COOCH$_3$), 50.4 (COOCH$_3$)$_2$, 172.0 (-COOCH$_3$), 162.9 (-CH=N)

IR (KBr) : 3430, 3039, 2949, 1742, 1649, 1544, 1514, 1448, 1269, 1243, 1170, 949, 890, 80, 782, 757, 732, 670, 641 cm$^{-1}$
M$^+$ : 310
(35) 2-N(2-hydroxy-5-nitrophenyl methylene) imino-3-methyl-butanoate

Yield : 50%
Nature : Semi solid
Mol. Formula : C₁₂H₁₄N₂O₅
Mol. wt. : 266

$^1$H NMR (200 MHz, CDCl₃) δ, ppm : 7.02 – 8.38 (m, 3H; C-3′, C-4′, C-6′, Ar-H), 3.9 (d, 1H; CH-CH(CH₃)₂), 2.2 (m, 1H; CH(CH₃)₂), 1.0 (d, 6H, CH(CH₃)₂), 11.0 (s, 1H; COOH), 8.13 (s, 1H; CH=N)

$^{13}$C NMR (200 MHz, CDCl₃) δ, ppm : 125.4 (C-1′), 163.9 (C-2′, C-OH), 116.7 (C-3′), 127.3 (C-4′), 141.4 (C-5′, C-NO₂), 125.5 (C-6′), 74.6 (C-2, CH-CH(CH₃)₂), 26.7 (C-3, CH(CH₃)₂), 17.1 (CH(CH₃)₂), 176.0 (COOH), 163.7 (CH=N)

IR (KBr) : 3422, 2964, 1654, 1612, 1541, 1447, 1287, 1287, 1232, 1170, 1126, 949, 843, 984, 950, 932, 845, 631, 601 cm⁻¹

M⁺ : 266

(36) Methyl 2-N (2-hydroxy 5-nitrophenyl methylene) imino-3-methyl butanoate

Yield : 70%
Nature : Semi solid
Mol. Formula : C₁₃H₁₆N₂O₅
Mol. wt. : 283

$^1$H NMR (200 MHz, CDCl₃) δ, ppm : 6.8 – 7.5 (m, 3H; C-3′, C-4′, C-6′, Ar-H), 3.79 (s, 3H; -COOCH₃), 3.97 (d, 1H; C-2, -CH(CH₃)₂), 2.4 (m, 1H; C-3, -CH(CH₃)₂), 0.88 – 1.17 (d, 6H; -CH(CH₃)₂), 8.1 (s, 1H; -CH=N)

$^{13}$C NMR (200 MHz, CDCl₃) δ, ppm : 120.1 (C-1′), 159.04 (C-2′, C-OH), 118.4 (C-3′), 137.8 (C-4′), 140.9 (C-5′, C-NO₂), 124.2 (C-6′) 170.1 (C-1′, -COOCH₃), 52.1 (-COOCH₃), 75.0 (C-2), 31.6 (C-3), 18.9 (CH(CH₃)₂), 165.9 (-CH=N)

IR (KBr) : 3440, 2996, 1742, 1625, 1493, 1278, 1261, 1150, 1098, 973, 938, 870, 754, 729, 635 cm⁻¹

M⁺ : 283
(37) Methyl 2-N (2-hydroxy-5-nitrophenyl methylene) imino-3-(4-hydroxy phenyl) propanoate

Yield : 75%
M.P. : 208 - 209°C
Mol. Formula : C_{17}H_{16}N_{2}O_{5}
Mol. wt. : 344

\(^1\)H NMR (200 MHz, CDCl\(_3\)) \(\delta, \text{ ppm} : 6.68 – 8.38 (m, 7H; C-4', C-6', C-2'', C-3'', C-5'', C-6''), 4.35 (t, 1H; C-2, -CH-CH\(_2\)), 3.26 (d, 1H; C-3, -CH-CH\(_2\)), 3.67 (s, 3H; COOCH\(_3\)), 8.1 (s, 1H; -CH=N)

\(^13\)C NMR (200 MHz, CDCl\(_3\)) \(\delta, \text{ ppm} : 127.0\) (C-1’), 158.5 (C-2’, C-OH), 116.7 (C-3’), 130.1 (C-4’), 137.7 (C-5’, C-NO\(_2\)), 127.0 (C-6’), 71.2 (C-2, CH-CH\(_2\)), 39.0 (C-3, -CH\(_2\)-C\(_6\)H\(_5\)), 130.5 (C-1’’), 129.3 (C-2’’ & C-6’’), 115.7 (C-3’’ & C-5’’), 155.5 (C-4’’, -C-OH), 52.7 (COOCH\(_3\)), 170.0 (-COOCH\(_3\)), 165.7 (-CH=N)

IR (KBr) : 3430, 2953, 1742, 1640, 1583, 1486, 1456, 1266, 1234, 1141, 903, 837, 772, 754, 729, 635 cm\(^{-1}\)

M\(^+\) : 344

(38) Methyl 2-N (2-hydroxy 3,5-diiodophenyl methylene) imino-3-methyl butanoate

Yield : 78%
M.P. : 155 – 158°C
Mol. Formula : C\(_{13}\)H\(_{15}\)I\(_2\)NO\(_3\)
Mol. wt. : 487

\(^1\)H NMR (200 MHz, CDCl\(_3\)) \(\delta, \text{ ppm} : 7.5\) (s, 2H; C-4’, C-6’ Ar-H), 3.75 (s, 3H; COOCH\(_3\)), 3.80 (d, 1H; C-2, CH-CH-CH\(_3\)), 2.36 – 2.46 (m, 1H; C-3, -CH(CH\(_3\))\(_2\)), 0.87 – 0.97 (d, 6H; -CH(CH\(_3\))\(_2\)), 8.2 (s, 1H; -CH=N)

\(^13\)C NMR (200 MHz, CDCl\(_3\)) \(\delta, \text{ ppm} : 119.7\) (C-1’), 160.1 (C-2’, C-OH), 119.4 (C-3’, -C-I), 140.0 (C-4’), 119.4 (C-5’, C-I), 140.0 (C-6’), 170.7 (C-1, -COOCH\(_3\)), 52.2 (-COOCH\(_3\)), 72.5 (C-2), 31.7 (C-3), 19.1 (-CH(CH\(_3\))\(_3\)), 164.4 (-CH=N)

IR (KBr) : 3359, 2962, 1730, 1628, 1477, 1269, 1243, 1170, 1138, 1108, 742, 649 cm\(^{-1}\)

M\(^+\) : 487
(39) Methyl 2-N (2-hydroxy-5-nitrophenyl methylene) imino -2-phenyl ethanoate

Yield : 75%
M.P. : 112°C
Mol. Formula : C₁₆H₁₄NO₅
Mol. wt. : 264

¹H NMR (200 MHz, CDCl₃) δ (ppm) : 6.8 – 7.5 (m, 8H; C-3', C-4', C-6', C-2'', C-3'', C-4'', C-5'', C-6'', Ar-H)), 5.1 (s, 1H; -CH-COOCH₃), 3.7 (s, 3H; COOCH₃), 8.12 (s, 1H; -CH=N)

¹³C NMR (200 MHz, CDCl₃) δ, ppm : 127.7 (C-1'), 149.2 (C-2', C-OH), 117.3 (C-3'), 128.1 (C-4'), 137.2 (C-5', C-NO₂), 125.2 (C-6'), 73.2 (C-2, -CH-COOCH₃), 52.8 (-CH-COOCH₃), 170.06 (COOCH₃), 130.1 (C-1''), 129.1 (C-2'' & C-6''), 128.9 (C-3'' & C-5''), 126.5 (C-4''), 165.3 (-CH=N)

IR (KBr) : 3410, 2980, 1720, 1620, 1582, 794, 736, 643 cm⁻¹
M⁺ : 264
REFERENCES


