Chapter-4

Synthesis and Characterization of triazolothiadiazole derivatives [12a-h, 13a-f, 14a-h and 15a-j] prepared from various acid derivatives [5a-h, 6a-f, 7a-h and 8a-j]

The present chapter describe the synthesis of triazolothiadiazole derivatives i.e. (12a-h), (13a-f), (14a-h) and (15a-j) from 4-Amino-5-mercapto-4H-1,2,4-triazole (3) respectively with various acid derivatives i.e. (5a-h), (6a-f), (7a-h) and (8a-j) in presence of phosphorous oxy chloride.
4.1 EXPERIMENTAL:

Various acid derivatives (i.e. 5a-h, 6a-f, 7a-h and 8a-j) mentioned in chapter-2 on cyclization with 4-Amino-5-mercapto-4H-1,2,4-triazole (3) in presence of phosphorous oxy chloride to form corresponding triazolothiadiazole derivatives (i.e 12a-h, 13a-f, 14a-h and 15a-j). These newly prepared triazolothiadiazole derivatives (i.e 12a-h, 13a-f, 14a-h and 15a-j) were characterized by elemental content, infrared spectral data and $^1$H resonance spectral data. The research work is scanned in Scheme-4.1. Experimental procedures for the synthesis of this series of compounds have been adopted according to reported methods [1-12].

4.2 MATERIALS:

The phthalamic acid derivatives (5a-h, 6a-f, 7a-h and 8a-j) listed in table-2.10, table-2.11, table-2.12 and table-2.13 of chapter-2 have been selected for the synthesis of above said compounds. The POCl$_3$ used for synthesis was purchased from local market. The synthesis of 4-Amino-5-mercapto-4H-1,2,4-triazole (3) has already been described in Chapter-2. Other chemicals used were of A.R. grade.
4.3 **SYNTHESIS OF N-(SUBSTITUTED-PHENYL)-2-(3-(PYRIDIN-4-YL)-[1,2,4] TRIAZOLO [3,4-B] [1,3,4] THIADIAZOL -6- YL) BENZAMIDE(12A-H, 13A-F, 14A-H AND 15A-J):**

In a R.B.F., the mixture of compound (3) and phthalamic acid derivatives (5a-h, 6a-f, 7a-h and 8a-j) at stoichiometric ratio and POCl₃ (100 ml) was refluxed on wire gauze for 8 hours. The resultant reaction mixture was poured in ice water. The solution was made alkali by adding KOH the deposited solid filtered and crystallized from ethanol to yield N-(substituted-phenyl)-2-(3-(pyridin-4-yl)-[1,2,4] triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide (12a-h, 13a-f, 14a-h and 15a-j). This was obtained in 55-72% yield depending upon the nature of compound. The yields, melting points and other analytical data of all these compounds are given below. [13]

The reaction scheme was shown below,
Where, $R =$

![Scheme 4.1](image-url)
Compound - 12a

\[
\text{\begin{tikzpicture}
\begin{scope}[scale=0.5]
\draw [black, thick] (-0.5,0) -- (0.5,0);
\draw [black, thick] (0,-0.5) -- (0,0.5);
\draw [black, thick] (0.5,0) -- (1,0);
\draw [black, thick] (1,0) -- (1,1);
\draw [black, thick] (1,1) -- (0,1);
\draw [black, thick] (0,1) -- (0,0);
\draw [black, thick] (0.5,-0.5) -- (1,-0.5);
\draw [black, thick] (1,-0.5) -- (1,-1);
\draw [black, thick] (1,-1) -- (0,-1);
\draw [black, thick] (0,-1) -- (0,-0.5);
\draw [black, thick] (0.5,0.5) -- (1,0.5);
\draw [black, thick] (1,0.5) -- (1,1.5);
\draw [black, thick] (1,1.5) -- (0,1.5);
\draw [black, thick] (0,1.5) -- (0,0.5);
\draw [black, thick] (0.5,-1.5) -- (1,-1.5);
\draw [black, thick] (1,-1.5) -- (1,-2.5);
\draw [black, thick] (1,-2.5) -- (0,-2.5);
\draw [black, thick] (0,-2.5) -- (0,-1.5);
\draw [black, thick] (0.5,1.5) -- (1,1.5);
\draw [black, thick] (1,1.5) -- (1,2.5);
\draw [black, thick] (1,2.5) -- (0,2.5);
\draw [black, thick] (0,2.5) -- (0,1.5);
\end{scope}
\end{tikzpicture}}
\]

2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)-N-(4-(N-(pyrimidin-2-yl)sulfamoyl)phenyl)benzamide

<table>
<thead>
<tr>
<th>Elemental Content</th>
<th>%C</th>
<th>%H</th>
<th>%N</th>
<th>%S</th>
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<tr>
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<td>3.08</td>
<td>22.69</td>
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<td>Found</td>
<td>54.00</td>
<td>3.06</td>
<td>22.65</td>
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<th>IR Spectral characterization cm(^{-1})</th>
<th>Aromatic CH</th>
<th>-NH of Amide</th>
<th>-CO of Amide</th>
<th>-C=(=)N</th>
<th>N-N of triazole and Thiadiazole</th>
<th>-C-S not discernible</th>
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<td>3047 cm(^{-1})</td>
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<tr>
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<tr>
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<tr>
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<tr>
<td>NMR spectral Signals ((\delta), ppm)</td>
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<td></td>
<td></td>
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</tr>
<tr>
<td>4.05</td>
<td>(s, 1H, -SO(_2)NH)</td>
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<td>7.50-8.10</td>
<td>(m, 8H, Aromatic)</td>
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<tr>
<td>6.95, 8.43</td>
<td>(m, 3H, pyrimidine)</td>
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<td></td>
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<tr>
<td>7.96, 8.873</td>
<td>(m, 4H, Pyridine)</td>
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<td></td>
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<tr>
<td>9.19</td>
<td>(s, 1H, -CONH)</td>
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<td></td>
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<td></td>
</tr>
</tbody>
</table>

LC-MS data
Mass (m/z) = 556.7 (+Ve)
Figure 4.1 proton NMR spectrum of sample 12a
Figure 4.2 IR spectrum of sample 12a
Figure 4.3 LC-MS spectrum of sample 12a
Compound – 12b

![Chemical Structure of Compound 12b](image)

N-(4-(N-(5-methylpyrimidin-2-yl)sulfamoyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

<table>
<thead>
<tr>
<th>M.F.:C$<em>{26}$H$</em>{19}$N$_9$O$_3$S$_2$</th>
<th>Elemental Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>M.W.: 569.62 gm/mole</td>
<td>%C  %H  %N  %S</td>
</tr>
<tr>
<td>M.P.: 215-217°C (Uncorrected)</td>
<td>Cald. 54.82 3.36 22.13 11.26</td>
</tr>
<tr>
<td>Yil.: 72%</td>
<td>Found 54.80 3.33 22.10 11.24</td>
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<table>
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<th>NMR spectral Signals (δ, ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3045 cm$^{-1}$</td>
<td>4.03 (s, 1H, -SO$_2$NH)</td>
</tr>
<tr>
<td>3303 cm$^{-1}$</td>
<td>7.50-8.10 (m, 8H, Aromatic)</td>
</tr>
<tr>
<td>1696 cm$^{-1}$</td>
<td>8.53 (m, 2H, pyrimidine)</td>
</tr>
<tr>
<td>1325 cm$^{-1}$</td>
<td>7.97, 8.74 (m, 4H, Pyridine)</td>
</tr>
<tr>
<td>993 cm$^{-1}$</td>
<td>9.17 (s 1H, -CONH)</td>
</tr>
<tr>
<td>710-570 cm$^{-1}$</td>
<td>2.39 (s, 3H, -CH$_3$)</td>
</tr>
<tr>
<td></td>
<td>C=S not discernible</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>LC-MS data</th>
<th>Mass (m/z) = 570.8 (+Ve)</th>
</tr>
</thead>
</table>

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Figure 4.4 proton NMR spectrum of sample 12b
Figure 4.5 IR spectrum of sample 12b
Figure 4.5 LC-MS spectrum of sample12b
Compound – 12c

N-(4-(N-(2,6-dimethylpyrimidin-4-yl)sulfamoyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

M.F.: C$_{27}$H$_{21}$N$_9$O$_3$S$_2$
M.W.: 583.64 gm/mole
M.P.: 215-217°C (Uncorrected)
Yil.: 70%

<table>
<thead>
<tr>
<th>Elemental Content</th>
<th>%C</th>
<th>%H</th>
<th>%N</th>
<th>%S</th>
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</thead>
<tbody>
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<td>Found</td>
<td>55.52</td>
<td>3.60</td>
<td>21.57</td>
<td>10.07</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>IR Spectral characterization cm$^{-1}$</th>
<th>NMR spectral Signals ($\delta$, ppm)</th>
</tr>
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<tr>
<td>3043 cm$^{-1}$</td>
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<tr>
<td>3370 cm$^{-1}$</td>
<td>-NH of Amide</td>
</tr>
<tr>
<td>1684 cm$^{-1}$</td>
<td>-CO of Amide</td>
</tr>
<tr>
<td>1351 cm$^{-1}$</td>
<td>-C=N</td>
</tr>
<tr>
<td>992 cm$^{-1}$</td>
<td>-N-N of triazole and Thiadiazole</td>
</tr>
<tr>
<td>710-570 cm$^{-1}$</td>
<td>-C-S not discernible</td>
</tr>
<tr>
<td>4.06</td>
<td>(s, 1H, -SO$_2$NH)</td>
</tr>
<tr>
<td>7.50-8.10</td>
<td>(m, 8H, Aromatic)</td>
</tr>
<tr>
<td>8.38</td>
<td>(m, 1H, pyrimidin)</td>
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<td>7.96, 8.77</td>
<td>(m, 4H, Pyridine)</td>
</tr>
<tr>
<td>9.19</td>
<td>(s, 1H, -CONH)</td>
</tr>
<tr>
<td>2.31</td>
<td>(s, 3H, -CH$_3$)</td>
</tr>
<tr>
<td>2.46</td>
<td>(s, 3H, -CH$_3$)</td>
</tr>
</tbody>
</table>

LC-MS data

Mass (m/z) = 583.1 (+Ve)
Compound – 12d

N-(4-(N-(4-methoxypyrimidin-2-yl)sulfamoyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

M.F.: C_{26}H_{19}N_{9}O_{4}S_{2}
M.W.: 585.62 gm/mole
M.P.: 210-212°C (Uncorrected)
Yil.: 73%

Elemental Content

<table>
<thead>
<tr>
<th>%C</th>
<th>%H</th>
<th>%N</th>
<th>%S</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cld.</td>
<td>53.32</td>
<td>3.27</td>
<td>21.53</td>
</tr>
<tr>
<td>Found</td>
<td>53.30</td>
<td>3.25</td>
<td>21.50</td>
</tr>
</tbody>
</table>

IR Spectral characterization cm\(^{-1}\)

| 3061 cm\(^{-1}\) | Aromatic CH |
| 3377 cm\(^{-1}\) | -NH of Amide |
| 16813 cm\(^{-1}\) | -CO of Amide |
| 1350 cm\(^{-1}\) | -C=N |
| 990 cm\(^{-1}\) | -N-N of triazole and Thiadiazole |
| 710-570 cm\(^{-1}\) | -C-S not discernible |

NMR spectral Signals (δ, ppm)

| 4.09 | (s, 1H, -SO_{2}NH) |
| 7.50-8.10 | (m, 8H, Aromatic) |
| 5.87, 7.43 | (d, 2H, Pyrimidine) |
| 7.95, 8.79 | (m, 4H, Pyridine) |
| 9.18 | (s, 1H, -CONH) |
| 3.76 | (s, 3H, -OCH_{3}) |

LC-MS data

Mass (m/z) = 585.1 (+Ve)
Compound – 12e

\[
\text{N-(4-(N-(5-methoxypyrimidin-2-yl)sulfamoyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide}
\]

<table>
<thead>
<tr>
<th>M.F.:C_{26}H_{19}N_{9}O_{4}S_{2}</th>
<th>Elemental Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>M.W.: 585.62 gm/mole</td>
<td>%C    %H   %N    %S</td>
</tr>
<tr>
<td>M.P.: 218-220°C (Uncorrected)</td>
<td>Cald.  53.32 3.27 21.53 10.95</td>
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<td>Yil.: 72%</td>
<td>Found  53.30 3.25 21.50 10.90</td>
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**IR Spectral characterization cm⁻¹**

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<th>Frequency (cm⁻¹)</th>
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<td>3377 cm⁻¹</td>
<td>-NH of Amide</td>
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<td>1685 cm⁻¹</td>
<td>-CO of Amide</td>
</tr>
<tr>
<td>1358 cm⁻¹</td>
<td>-C=N</td>
</tr>
<tr>
<td>991 cm⁻¹</td>
<td>-N-N of triazole and thiadiazole</td>
</tr>
<tr>
<td>710-570 cm⁻¹</td>
<td>-C-S not discernible</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Frequency (cm⁻¹)</th>
<th>Description</th>
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<tbody>
<tr>
<td>3037</td>
<td>Aromatic CH</td>
</tr>
<tr>
<td>3377</td>
<td>-NH of Amide</td>
</tr>
<tr>
<td>1685</td>
<td>-CO of Amide</td>
</tr>
<tr>
<td>1358</td>
<td>-C=N</td>
</tr>
<tr>
<td>991</td>
<td>-N-N of triazole and thiadiazole</td>
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<tr>
<td>710-570</td>
<td>-C-S not discernible</td>
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**NMR spectral Signals (δ, ppm)**

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<thead>
<tr>
<th>Frequency (δ, ppm)</th>
<th>Description</th>
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<tbody>
<tr>
<td>4.05</td>
<td>(s, 1H, -SO₂NH)</td>
</tr>
<tr>
<td>7.50-8.10</td>
<td>(m, 8H, Aromatic)</td>
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<td>8.35</td>
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<td>7.96, 8.78</td>
<td>(m, 4H, pyridine)</td>
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<td>9.13</td>
<td>(s, 1H, -CONH)</td>
</tr>
<tr>
<td>3.85</td>
<td>(s, 3H, -OCH₃)</td>
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</table>

**LC-MS data**

Mass (m/z) = 585.1 (+Ve)
**Compound – 12f**

![Chemical Structure](image)

\[ \text{N-}(\text{4-(N-(2,6-dimethoxypyrimidin-4-yl)sulfamoyl)phenyl})-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide} \]

<table>
<thead>
<tr>
<th>Elemental Content</th>
<th>%C</th>
<th>%H</th>
<th>%N</th>
<th>%S</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Cald.</strong></td>
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<td>3.44</td>
<td>20.48</td>
<td>10.40</td>
</tr>
<tr>
<td><strong>Found</strong></td>
<td>52.63</td>
<td>3.42</td>
<td>20.45</td>
<td>10.40</td>
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</table>

**M.F.:** C_{27}H_{21}N_{9}O_{5}S_{2}  
**M.W.:** 615.64 gm/mole  
**M.P.:** 205-207°C  
(Uncorrected)  
**Yil.:** 70%

| IR Spectral characterization cm\(^{-1}\) |  
|-------------------------------|-------------------------------|
| 3069 cm\(^{-1}\) | Aromatic CH  
| 3375 cm\(^{-1}\) | -NH of Amide  
| 1681 cm\(^{-1}\) | -CO of Amide  
| 1350 cm\(^{-1}\) | -C=N  
| 993 cm\(^{-1}\) | -N-N of triazole and Thiadiazole  
| 710-570 cm\(^{-1}\) | -C-S not discernible  

| NMR spectral Signals (δ, ppm) |  
|-------------------------------|-------------------------------|
| 4.02 | (s, 1H, -SO\(_2\)NH)  
| 7.50-8.10 | (m, 8H, Aromatic)  
| 5.98 | (s, 1H, Pyrimidine)  
| 7.95, 8.72 | (m, 4H, Pyridine)  
| 9.14 | (s, 1H, -CONH)  
| 3.79 | (s, 3H, -OCH\(_3\))  
| 3.84 | (s, 3H, -OCH\(_3\))  

**LC-MS data**  
Mass (m/z) = 615.1 (+Ve)
Compound – 12g

N-(4-(N-(5,6-dimethoxypyrimidin-4-yl)sulfamoyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

<table>
<thead>
<tr>
<th>M.F.:C_{27}H_{21}N_{9}O_{5}S_{2}</th>
<th>Elemental Content</th>
</tr>
</thead>
<tbody>
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<td>M.W.: 615.64 gm/mole</td>
<td>%C   %H   %N    %S</td>
</tr>
<tr>
<td>M.P.:  224-227°C (Uncorrected)</td>
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</tr>
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<td>Yil.: 70%</td>
<td>Found 52.63 3.40 20.46 10.40</td>
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<th>NMR spectral Signals (δ, ppm)</th>
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<td>3053 cm^{-1}</td>
<td>4.06 (s, 1H, -SO_{2}NH)</td>
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<td>3371 cm^{-1}</td>
<td>7.50-8.10 (m, 8H, Aromatic)</td>
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<tr>
<td>1684 cm^{-1}</td>
<td>8.35 (s, 1H, Pyrimidine)</td>
</tr>
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<td>1357 cm^{-1}</td>
<td>7.97, 8.79 (m, 4H, Pyridine)</td>
</tr>
<tr>
<td>991 cm^{-1}</td>
<td>9.21 (s, 1H, -CONH)</td>
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<td>710-570 cm^{-1}</td>
<td>4.05 (s, 3H, -OCH_{3})</td>
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<td></td>
<td>3.87 (s, 3H, -OCH_{3})</td>
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LC-MS data

Mass (m/z) = 615.1 (+Ve)
**Compound – 12h**

N-(4-(N-(6-methoxy-2-methylpyrimidin-4-yl)sulfamoyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

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<th>Elemental Content</th>
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<td>%C</td>
</tr>
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<td>M.P.: 226-228°C (Uncorrected)</td>
<td>Cald.</td>
</tr>
<tr>
<td>Yil.: 71%</td>
<td>Found</td>
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<tr>
<th>IR Spectral characterization cm⁻¹</th>
<th>NMR spectral Signals (δ, ppm)</th>
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<tbody>
<tr>
<td>3048 cm⁻¹</td>
<td>Aromatic CH</td>
</tr>
<tr>
<td>3376 cm⁻¹</td>
<td>-NH of Amide</td>
</tr>
<tr>
<td>1683 cm⁻¹</td>
<td>-CO of Amide</td>
</tr>
<tr>
<td>1357 cm⁻¹</td>
<td>-C=N</td>
</tr>
<tr>
<td>992 cm⁻¹</td>
<td>-N-N of triazole and Thiadiazole</td>
</tr>
<tr>
<td>710-570 cm⁻¹</td>
<td>-C-S not discernible</td>
</tr>
<tr>
<td>3.98</td>
<td>(s, 1H, -SO₂NH)</td>
</tr>
<tr>
<td>7.50-8.10</td>
<td>(m, 8H, Aromatic)</td>
</tr>
<tr>
<td>5.95</td>
<td>(s, 1H, Pyrimidine)</td>
</tr>
<tr>
<td>7.95, 8.72</td>
<td>(m, 4H, Pyridine)</td>
</tr>
<tr>
<td>9.24</td>
<td>(s, 1H, -CONH)</td>
</tr>
<tr>
<td>3.78</td>
<td>(s, 3H, -OCH₃)</td>
</tr>
<tr>
<td>2.46</td>
<td>(s, 3H, -CH₃)</td>
</tr>
</tbody>
</table>

**LC-MS data**

Mass (m/z) = 599.1 (+Ve)
Where, R =

Scheme 4.2
Compound – 13a

\[
\text{N-(4-(N,N-diethylsulfamoyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo [3,4-b][1,3,4]thiadiazol-6-yl)benzamide}
\]

M.F.: $C_{25}H_{23}N_{7}O_{3}S_{2}$
M.W.: 533.63 gm/mole
M.P.: 220-222°C (Uncorrected)
Yil.: 70%

<table>
<thead>
<tr>
<th>IR Spectral characterization cm$^{-1}$</th>
<th>Elemental Content</th>
<th>NMR spectral Signals ($\delta$, ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3030 cm$^{-1}$</td>
<td>%C</td>
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<td>3343 cm$^{-1}$</td>
<td>-NH of Amide</td>
<td></td>
</tr>
<tr>
<td>1658 cm$^{-1}$</td>
<td>-CO of Amide</td>
<td></td>
</tr>
<tr>
<td>1371 cm$^{-1}$</td>
<td>-C=N</td>
<td></td>
</tr>
<tr>
<td>992 cm$^{-1}$</td>
<td>-N-N of triazole and Thiadiazole</td>
<td></td>
</tr>
<tr>
<td>710-570 cm$^{-1}$</td>
<td>-C-S not discernible</td>
<td></td>
</tr>
</tbody>
</table>

992 cm$^{-1}$

[992 cm$^{-1}$]

Aromatic CH

-NH of Amide

-CO of Amide

-C=N

-N-N of triazole and Thiadiazole

-C-S not discernible

LC-MS data

Mass (m/z) = 534.7 (+Ve)
Figure 4.6 proton NMR spectrum of sample 13a
Figure 4.7 IR spectrum of sample 13a
Figure 4.8 LC-MS spectrum of sample 13a
**Compound – 13b**

N-(4-(N-ethyl-N-phenylsulfamoyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

<table>
<thead>
<tr>
<th>M.F.:C_{29}H_{23}N_{7}O_{3}S_{2}</th>
<th>Elemental Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>M.W.: 581.67 gm/mole</td>
<td>%C   %H   %N   %S</td>
</tr>
<tr>
<td>M.P.: 252-255°C (Uncorrected)</td>
<td>Cald. 59.88 3.99 16.86 11.03</td>
</tr>
<tr>
<td>Yil.: 68%</td>
<td>Found 59.86 3.96 16.84 11.00</td>
</tr>
</tbody>
</table>

**IR Spectral characterization cm⁻¹**

<table>
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<tr>
<th>cm⁻¹</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>3032</td>
<td>Aromatic CH</td>
</tr>
<tr>
<td>3342</td>
<td>-NH of Amide</td>
</tr>
<tr>
<td>1656</td>
<td>-CO of Amide</td>
</tr>
<tr>
<td>1372</td>
<td>-C=N</td>
</tr>
<tr>
<td>993</td>
<td>-N-N of triazole and Thiadiazole</td>
</tr>
<tr>
<td>710-570</td>
<td>-C-S not discernible</td>
</tr>
</tbody>
</table>

**NMR spectral Signals (δ, ppm)**

<table>
<thead>
<tr>
<th>δ (ppm)</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.14</td>
<td>(s, 1H, -CONH)</td>
</tr>
<tr>
<td>7.50-8.10</td>
<td>(m, 13H, Aromatic)</td>
</tr>
<tr>
<td>7.95, 8.76</td>
<td>(m, 4H, pyridine)</td>
</tr>
<tr>
<td>1.17, 3.18</td>
<td>(m, 5H, -C_{2}H_{5})</td>
</tr>
</tbody>
</table>

**LC-MS data**

Mass (m/z) = 582.7 (+Ve)
Figure 4.9 proton NMR spectrum of sample 13b
Figure 4.10 IR spectrum of sample 13b
Figure 4.11 LC-MS spectrum of sample 13b
**Compound – 13c**

![Chemical Structure](image)

\[ \text{N-(4-(N,N-diphenylsulfamoyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo} \]
\[ \text{[3,4-b][1,3,4]thiadiazol-6-yl)benzamide} \]

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>M.F.</strong></td>
<td>( \text{C}<em>{33}\text{H}</em>{23}\text{N}<em>{7}\text{O}</em>{3}\text{S}_{2} )</td>
</tr>
<tr>
<td><strong>M.W.</strong></td>
<td>629.71 gm/mole</td>
</tr>
<tr>
<td><strong>M.P.</strong></td>
<td>258-260°C (Uncorrected)</td>
</tr>
<tr>
<td><strong>Yil.</strong></td>
<td>73%</td>
</tr>
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</table>

<table>
<thead>
<tr>
<th><strong>IR Spectral characterization cm(^{-1})</strong></th>
<th><strong>NMR spectral Signals (δ, ppm)</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>3065 cm(^{-1})</td>
<td>Aromatic CH</td>
</tr>
<tr>
<td>3374 cm(^{-1})</td>
<td>-NH of Amide</td>
</tr>
<tr>
<td>1682 cm(^{-1})</td>
<td>-CO of Amide</td>
</tr>
<tr>
<td>1358 cm(^{-1})</td>
<td>-C=( \text{N} )</td>
</tr>
<tr>
<td>993 cm(^{-1})</td>
<td>-N-N of triazole and Thiadiazole</td>
</tr>
<tr>
<td>710-570 cm(^{-1})</td>
<td>-C-S not discernible</td>
</tr>
<tr>
<td>3065 cm(^{-1})</td>
<td>9.21 (s, 1H, -CONH)</td>
</tr>
<tr>
<td>3374 cm(^{-1})</td>
<td>6.60-8.10 (m, 18H, Aromatic)</td>
</tr>
<tr>
<td>1682 cm(^{-1})</td>
<td>7.95, 8.78 (m, 4H, pyridine)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>LC-MS data</strong></th>
<th><strong>Elemental Content</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass (m/z) = 629.1 (+Ve)</td>
<td>%C</td>
</tr>
<tr>
<td><strong>Cald.</strong></td>
<td>62.94</td>
</tr>
<tr>
<td><strong>Found</strong></td>
<td>62.92</td>
</tr>
</tbody>
</table>
**Compound – 13d**

![Chemical Structure](image)

N-(4-(piperidin-1-ylsulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

<table>
<thead>
<tr>
<th>M.F.:C_{26}H_{23}N_{7}O_{3}S_{2}</th>
<th><strong>Elemental Content</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>M.W.: 545.64 gm/mole</td>
<td>%C       %H       %N       %S</td>
</tr>
<tr>
<td>M.P.: 220-223°C (Uncorrected)</td>
<td>Cald.    57.23  4.25  17.97  11.75</td>
</tr>
<tr>
<td>Yil.: 72%</td>
<td>Found    57.20  4.20  17.95  11.72</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>IR Spectral characterization cm(^{-1})</th>
<th>NMR spectral Signals ((\delta, \text{ppm}))</th>
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<tbody>
<tr>
<td>3037 cm(^{-1})</td>
<td>9.12 (s, 1H, -CONH)</td>
</tr>
<tr>
<td>3370 cm(^{-1})</td>
<td>7.50-8.10 (m, 8H, Aromatic)</td>
</tr>
<tr>
<td>1684 cm(^{-1})</td>
<td>7.96, 8.73 (m, 4H, pyridine)</td>
</tr>
<tr>
<td>1357 cm(^{-1})</td>
<td>1.57 (m, 2H, piperidine)</td>
</tr>
<tr>
<td>990 cm(^{-1})</td>
<td>1.51, 3.05 (m, 8H, piperidine)</td>
</tr>
<tr>
<td>710-570 cm(^{-1})</td>
<td></td>
</tr>
<tr>
<td>-Aromatic CH</td>
<td></td>
</tr>
<tr>
<td>-NH of Amide</td>
<td></td>
</tr>
<tr>
<td>-CO of Amide</td>
<td></td>
</tr>
<tr>
<td>-C=N</td>
<td></td>
</tr>
<tr>
<td>-N-N of triazole and Thiadiazole</td>
<td></td>
</tr>
<tr>
<td>-C-S not discernible</td>
<td></td>
</tr>
<tr>
<td>-Aromatic CH</td>
<td></td>
</tr>
<tr>
<td>-NH of Amide</td>
<td></td>
</tr>
<tr>
<td>-CO of Amide</td>
<td></td>
</tr>
<tr>
<td>-C=N</td>
<td></td>
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<tr>
<td>-N-N of triazole and Thiadiazole</td>
<td></td>
</tr>
<tr>
<td>-C-S not discernible</td>
<td></td>
</tr>
</tbody>
</table>

**LC-MS data**

Mass (m/z) = 545.1 (+Ve)
Compound – 13e

2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)-N-(4-(pyrrolidin-1-ylsulfonyl)phenyl)benzamide

<table>
<thead>
<tr>
<th>Elemental Content</th>
<th>%C</th>
<th>%H</th>
<th>%N</th>
<th>%S</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cald.</td>
<td>56.48</td>
<td>3.98</td>
<td>18.44</td>
<td>12.06</td>
</tr>
<tr>
<td>Found</td>
<td>56.45</td>
<td>3.95</td>
<td>18.40</td>
<td>12.04</td>
</tr>
</tbody>
</table>

**M.F.:** C_{25}H_{21}N_{7}O_{3}S_{2}
**M.W.:** 531.61 gm/mole
**M.P.:** 215-218°C (Uncorrected)
**Yil.:** 70%

**IR Spectral characterization cm\(^{-1}\)**
- 3045 cm\(^{-1}\): Aromatic CH
- 3372 cm\(^{-1}\): NH of Amide
- 1683 cm\(^{-1}\): CO of Amide
- 1355 cm\(^{-1}\): C=N
- 991 cm\(^{-1}\): N-N of triazole and Thiadiazole
- 710-570 cm\(^{-1}\): C-S not discernible

**NMR spectral Signals (δ, ppm)**
- 9.18: (s, 1H, -CONH)
- 7.50-8.10: (m, 8H, Aromatic)
- 7.98, 8.76: (m, 4H, Pyridine)
- 1.90, 3.25: (m, 8H, pyrrolidine)

**LC-MS data**
- Mass (m/z) = 531.1 (+Ve)
**Compound – 13f**

![Chemical Structure](image)

N-(4-(morpholinosulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo [3,4-b][1,3,4]thiadiazol-6-yl)benzamide

**M.F.:**C_{25}H_{21}N_{7}O_{4}S_{2}  
**M.W.:** 547.61 gm/mole  
**M.P.:** 210-212°C (Uncorrected)  
**Yil.:** 69%

<table>
<thead>
<tr>
<th>IR Spectral characterization cm(^{-1})</th>
<th>Elemental Content</th>
<th>NMR spectral Signals (δ, ppm)</th>
<th>LC-MS data</th>
</tr>
</thead>
<tbody>
<tr>
<td>3038 cm(^{-1})</td>
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<td>9.11 (s, 1H, -CONH)</td>
<td>Mass (m/z) = 547.1 (+Ve)</td>
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<tr>
<td>3373 cm(^{-1})</td>
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<td>7.50-8.10 (m, 8H, Aromatic)</td>
<td></td>
</tr>
<tr>
<td>1682 cm(^{-1})</td>
<td></td>
<td>7.95, 8.77 (m, 4H, Pyridine)</td>
<td></td>
</tr>
<tr>
<td>1357 cm(^{-1})</td>
<td></td>
<td>2.95, 3.63 (m, 8H, Morpholine)</td>
<td></td>
</tr>
<tr>
<td>992 cm(^{-1})</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>710-570 cm(^{-1})</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Aromatic CH</td>
<td></td>
<td>-C=S not discernible</td>
<td></td>
</tr>
<tr>
<td>-NH of Amide</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-CO of Amide</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-C=N</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-N-N of triazole and Thiadiazole</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Yil.:</strong> 69%</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Found** 54.80 3.85 17.88 11.70
Where, R =

\[
\begin{align*}
&\text{a)} \quad \text{R = Various substitution for } 4\text{-amino-5-(pyridin-4-yl)-4H-1,2,4-triazole-3-thiol} \\
&\text{b)} \quad \text{R = Various substitution for pthalamic acid Derivatives} \\
&\text{c)} \quad \text{R = Various substitution for N-[substituted-phenyl]-2-[3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl]benzamide} \\
\end{align*}
\]

Scheme 4.3
Compound – 14a

N-(4-((4-methylpiperazin-1-yl)sulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

<table>
<thead>
<tr>
<th>M.F.:C_{26}H_{24}N_{8}O_{3}S_{2}</th>
<th>Elemental Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>M.W.: 560.65 gm/mole</td>
<td>%C   %H   %N   %S</td>
</tr>
<tr>
<td>M.P.: 237-238°C (Uncorrected)</td>
<td>Cald. 55.70 4.31 19.99 11.44</td>
</tr>
<tr>
<td>Yil.: 68%</td>
<td>Found 55.68 4.30 19.97 11.42</td>
</tr>
</tbody>
</table>

IR Spectral characterization cm\(^{-1}\)

| 3053 cm\(^{-1}\) | Aromatic CH |
| 3315 cm\(^{-1}\) | -NH of Amide |
| 1687 cm\(^{-1}\) | -CO of Amide |
| 1362 cm\(^{-1}\) | -C=N |
| 992 cm\(^{-1}\)  | -N-N of triazole and Thiadiazole |
| 710-570 cm\(^{-1}\) | -C-S not discernible |

NMR spectral Signals (δ, ppm)

9.14 (s, 1H, -CONH)
7.50-8.10 (m, 8H, Aromatic)
7.94, 8.75 (m, 4H, Pyridine)
2.36, 3.05 (m, 8H, Piperazine)
2.24 (s, 3H, -CH\(_3\))

LC-MS data

Mass (m/z) = 561.7 (+Ve)
Figure 4.12 proton NMR spectrum of sample 14a
Figure 4.13 IR spectrum of sample 14a
Figure 4.14 LC-MS spectrum of sample 14a
**Compound – 14b**

![Chemical Structure](image)

N-(4-((4-ethylpiperazin-1-yl)sulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

<table>
<thead>
<tr>
<th>Elemental Content</th>
<th>%C</th>
<th>%H</th>
<th>%N</th>
<th>%S</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Cald.</strong></td>
<td>56.43</td>
<td>4.56</td>
<td>19.50</td>
<td>11.16</td>
</tr>
<tr>
<td><strong>Found</strong></td>
<td>56.40</td>
<td>4.52</td>
<td>19.48</td>
<td>11.23</td>
</tr>
</tbody>
</table>

**IR Spectral characterization cm⁻¹**

| 3057 cm⁻¹ | Aromatic CH
| 3313 cm⁻¹ | -NH of Amide
| 1684 cm⁻¹ | -CO of Amide
| 1369 cm⁻¹ | -C=N
| 990 cm⁻¹  | -N-N of triazole and Thiadiazole
| 710-570 cm⁻¹ | -C-S not discernible

**NMR spectral Signals (δ, ppm)**

| 9.18 | (s, 1H, -CONH)
| 7.50-8.10 | (m, 8H, Aromatic)
| 7.98, 8.74 | (m, 4H, Pyridine)
| 2.39, 3.08 | (m, 8H, piperazine)
| 1.05, 2.34 | (m, 5H, -C₂H₅)

**LC-MS data**

Mass (m/z) = 574.1 (+Ve)
Figure 4.13 proton NMR spectrum of sample 14b
Figure 4.15 IR spectrum of sample 14b
**Compound – 14c**

N-(4-((4-phenylpiperazin-1-yl)sulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

<table>
<thead>
<tr>
<th>Elemental Content</th>
<th>%C %H %N %S</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Cald.</strong></td>
<td>59.79 4.21 17.99 10.30</td>
</tr>
<tr>
<td><strong>Found</strong></td>
<td>59.77 4.20 17.97 10.28</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>IR Spectral characterization cm(^{-1})</th>
<th>NMR spectral Signals (δ, ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3063 cm(^{-1}) Aromatic CH</td>
<td>9.13 (s, 1H, -CONH)</td>
</tr>
<tr>
<td>3372 cm(^{-1}) -NH of Amide</td>
<td>6.70-8.10 (m, 13H, Aromatic)</td>
</tr>
<tr>
<td>1684 cm(^{-1}) -CO of Amide</td>
<td>7.96, 8.77 (m, 4H, Pyridine)</td>
</tr>
<tr>
<td>1355 cm(^{-1}) -C=N</td>
<td>3.15 (m, 8H, piperazine)</td>
</tr>
<tr>
<td>991 cm(^{-1}) -N-N of triazole and Thiadiazole</td>
<td></td>
</tr>
<tr>
<td>710-570 cm(^{-1}) -C-S not discernible</td>
<td></td>
</tr>
<tr>
<td></td>
<td><strong>LC-MS data</strong></td>
</tr>
<tr>
<td></td>
<td>Mass (m/z) = 622.1 (+Ve)</td>
</tr>
</tbody>
</table>
Compound – 14d

\[
\text{2-[(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)-N-(4-((4-(p-tolyl)piperazin-1-yl)sulfonyl)phenyl)benzamide}
\]

M.F.: \( C_{32}H_{28}N_{8}O_{3}S_{2} \)
M.W.: 636.75 gm/mole
M.P.: 262-265°C (Uncorrected)
Yil.: 73%

<table>
<thead>
<tr>
<th>IR Spectral characterization cm(^{-1} )</th>
<th>NMR spectral Signals (( \delta, \text{ ppm} ))</th>
</tr>
</thead>
<tbody>
<tr>
<td>3039 cm(^{-1} )</td>
<td>Aromatic CH</td>
</tr>
<tr>
<td>3373 cm(^{-1} )</td>
<td>-NH of Amide</td>
</tr>
<tr>
<td>1684 cm(^{-1} )</td>
<td>-CO of Amide</td>
</tr>
<tr>
<td>1356 cm(^{-1} )</td>
<td>-C=( N )</td>
</tr>
<tr>
<td>992 cm(^{-1} )</td>
<td>-N-N of triazole and Thiadiazole</td>
</tr>
<tr>
<td>710-570 cm(^{-1} )</td>
<td>-C-S not discernible</td>
</tr>
<tr>
<td>3039 cm(^{-1} )</td>
<td>9.13 (s, 1H, -CONH)</td>
</tr>
<tr>
<td>3373 cm(^{-1} )</td>
<td>6.60-8.10 (m, 12H, Aromatic)</td>
</tr>
<tr>
<td>1684 cm(^{-1} )</td>
<td>7.95, 8.73 (m, 4H, Pyridine)</td>
</tr>
<tr>
<td>1356 cm(^{-1} )</td>
<td>3.18 (m, 8H, piperazine)</td>
</tr>
<tr>
<td>992 cm(^{-1} )</td>
<td>2.32 (s, 3H, -CH(_3))</td>
</tr>
<tr>
<td>710-570 cm(^{-1} )</td>
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</table>

**Elemental Content**

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<th>%S</th>
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<td>4.43</td>
<td>17.60</td>
<td>10.07</td>
</tr>
<tr>
<td>60.34</td>
<td>4.40</td>
<td>17.58</td>
<td>10.05</td>
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</tbody>
</table>

**LC-MS data**

Mass (m/z) = 636.1 (+Ve)
# Compound – 14e

$$\begin{align*} &\text{N-(4-((4-(4-methoxyphenyl)piperazin-1-yl)sulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]\text{-triazolo}[3,4-b][1,3,4]\text{-thiadiazol-6-yl]benzamide} \\
&\text{M.F.: C}_{32}\text{H}_{28}\text{N}_{8}\text{O}_{4}\text{S}_{2} \\
&\text{M.W.: 652.75 gm/mole} \\
&\text{M.P.: 243-245°C} \\
&\text{(Uncorrected)} \\
&\text{Yil.: 72%} \\
\end{align*}$$

<table>
<thead>
<tr>
<th><strong>IR Spectral characterization cm(^{-1})</strong></th>
<th><strong>Aromatic CH</strong></th>
<th><strong>-NH of Amide</strong></th>
<th><strong>-CO of Amide</strong></th>
<th><strong>-C=N</strong></th>
<th><strong>-N-N of triazole and Thiadiazole</strong></th>
<th><strong>-C-S not discernible</strong></th>
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<tbody>
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<td>1683 cm(^{-1})</td>
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<td>993 cm(^{-1})</td>
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<table>
<thead>
<tr>
<th><strong>NMR spectral Signals (δ, ppm)</strong></th>
<th><strong>(s, 1H, -CONH)</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>9.17</td>
<td>(s, 1H, -CONH)</td>
</tr>
<tr>
<td>6.60-8.10</td>
<td>(m, 12H, Aromatic)</td>
</tr>
<tr>
<td>7.98, 8.72</td>
<td>(m, 4H, Pyridine)</td>
</tr>
<tr>
<td>3.16</td>
<td>(m, 8H, piperazine)</td>
</tr>
<tr>
<td>3.85</td>
<td>(s, 3H, -OCH(_3))</td>
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</table>

<table>
<thead>
<tr>
<th><strong>LC-MS data</strong></th>
<th><strong>Mass (m/z) = 652.1 (+Ve)</strong></th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th><strong>Elemental Content</strong></th>
<th><strong>%C</strong></th>
<th><strong>%H</strong></th>
<th><strong>%N</strong></th>
<th><strong>%S</strong></th>
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</thead>
<tbody>
<tr>
<td><strong>Cald.</strong></td>
<td>58.88</td>
<td>4.32</td>
<td>17.17</td>
<td>9.82</td>
</tr>
<tr>
<td><strong>Found</strong></td>
<td>58.86</td>
<td>4.30</td>
<td>17.15</td>
<td>9.80</td>
</tr>
</tbody>
</table>
Compound – 14f

N-(4-((4-(4-chlorophenyl)piperazin-1-yl)sulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

M.F.: C\textsubscript{31}H\textsubscript{25}N\textsubscript{8}O\textsubscript{3}S\textsubscript{2}Cl
M.W.: 657.17 gm/mole
M.P.: 240-242\textdegree{}C (Uncorrected)
Yil.: 70%

<table>
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<th>%C</th>
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<th>%N</th>
<th>%S</th>
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</thead>
<tbody>
<tr>
<td>Cald.</td>
<td>56.66</td>
<td>3.83</td>
<td>17.05</td>
<td>9.76</td>
</tr>
<tr>
<td>Found</td>
<td>56.64</td>
<td>3.80</td>
<td>17.03</td>
<td>9.74</td>
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<table>
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<tbody>
<tr>
<td>3032 cm\textsuperscript{-1}</td>
<td>Aromatic CH</td>
<td>-NH of Amide</td>
<td>-CO of Amide</td>
<td>-C=N</td>
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<td>3370 cm\textsuperscript{-1}</td>
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<td>1684 cm\textsuperscript{-1}</td>
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<tr>
<td>1358 cm\textsuperscript{-1}</td>
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</tr>
<tr>
<td>992 cm\textsuperscript{-1}</td>
<td></td>
<td>-N-N of triazole and Thiadiazole</td>
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</tr>
<tr>
<td>710-570 cm\textsuperscript{-1}</td>
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<td>-C-S not discernible</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>NMR spectral Signals (δ, ppm)</th>
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</thead>
<tbody>
<tr>
<td>9.13</td>
<td>(s, 1H, -CONH)</td>
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<tr>
<td>6.60-8.10</td>
<td>(m, 12H, Aromatic)</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>7.95, 8.73</td>
<td>(m, 4H, Pyridine)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3.22</td>
<td>(m, 8H, piperazine)</td>
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</table>

<table>
<thead>
<tr>
<th>LC-MS data</th>
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</thead>
<tbody>
<tr>
<td>Mass (m/z)</td>
<td>656.1 (+Ve)</td>
<td></td>
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</tbody>
</table>
Compound – 14g

N-(4-((4-(4-nitrophenyl)piperazin-1-yl)sulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

M.F.: $C_{31}H_{25}N_9O_5S_2$
M.W.: 667.72 gm/mole
M.P.: 235-237°C (Uncorrected)
Yil.: 69%

<table>
<thead>
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<td>3048 cm$^{-1}$</td>
<td>%C %H %N %S</td>
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<tr>
<td>3373 cm$^{-1}$</td>
<td>Cald. 55.76 3.77 18.88 9.60</td>
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<td>1682 cm$^{-1}$</td>
<td>Found 55.74 3.75 18.86 9.56</td>
</tr>
<tr>
<td>1356 cm$^{-1}$</td>
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<tr>
<td>990 cm$^{-1}$</td>
<td></td>
</tr>
<tr>
<td>710-570 cm$^{-1}$</td>
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</tr>
</tbody>
</table>

Aromatic CH
-NH of Amide
-CO of Amide
-C=N
-N-N of triazole and Thiadiazole
-C-S not discernible

NMR spectral Signals (δ, ppm)

<table>
<thead>
<tr>
<th>Signal</th>
<th>Assignment</th>
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<tr>
<td>9.16</td>
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<tr>
<td>7.00-8.10</td>
<td>(m, 12H, Aromatic)</td>
</tr>
<tr>
<td>7.95, 8.76</td>
<td>(m, 4H, Pyridine)</td>
</tr>
<tr>
<td>3.21</td>
<td>(m, 8H, piperazine)</td>
</tr>
</tbody>
</table>

LC-MS data

Mass (m/z) = 667.1 (+Ve)
Compound – 14h

\[
\begin{align*}
&\text{N-}(4-\text{-}(4-\text{(2,6-dichloro-4-nitrophenyl)piperazin-1-yl)sulfonyl}\text{-phenyl})-2-\text{-}(3-\text{(pyridin-4-yl)}\text{-}[1,2,4]\text{-triazolo}[3,4-b][1,3,4]\text{-thiadiazol-6-yl})\text{-benzamide}
\end{align*}
\]

M.F.: $C_{31}H_{23}N_9O_5S_2Cl_2$
M.W.: 736.61 gm/mole
M.P.: 238-240°C (Uncorrected)
Yil.: 73%

<table>
<thead>
<tr>
<th>IR Spectral characterization cm$^{-1}$</th>
<th>Elemental Content</th>
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<tbody>
<tr>
<td></td>
<td>%C %H %N %S</td>
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<tr>
<td>3062 cm$^{-1}$</td>
<td>Cald. 50.55 3.15 17.11 8.71</td>
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<td>3373 cm$^{-1}$</td>
<td>Found 50.53 3.12 17.10 8.70</td>
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<tr>
<td>1684 cm$^{-1}$</td>
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<td>1357 cm$^{-1}$</td>
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<tr>
<td>990 cm$^{-1}$</td>
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<tr>
<td>710-570 cm$^{-1}$</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

| NMR spectral Signals (δ, ppm)         |                                 |
|--------------------------------------|                                 |
| 9.19                                 | (s, 1H, -CONH)                   |
| 7.50-8.10                            | (m, 10H, Aromatic)               |
| 7.98, 8.73                           | (m, 4H, Pyridine)                |
| 3.17                                 | (m, 8H, piperazine)              |

| LC-MS data                           |                                 |
|--------------------------------------|                                 |
| Mass (m/z) = 735.0 (+Ve)             |                                 |
Where, \( R = \) 

Scheme 4.4
Compound – 15a

N-(4-((4,5-dimethyloxazol-3(2H)-yl)sulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

M.F.: C₂₆H₂₁N₇O₄S₂
M.W.: 559.62 gm/mole
M.P.: 198-200°C (Uncorrected)
Yil.: 72%

Elemental Content

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<tr>
<th></th>
<th>%C</th>
<th>%H</th>
<th>%N</th>
<th>%S</th>
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<tr>
<td>Cald.</td>
<td>55.80</td>
<td>3.78</td>
<td>17.52</td>
<td>11.46</td>
</tr>
<tr>
<td>Found</td>
<td>55.78</td>
<td>3.76</td>
<td>17.50</td>
<td>11.44</td>
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IR Spectral characterization cm⁻¹

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<tbody>
<tr>
<td>3055 cm⁻¹</td>
<td>Aromatic CH</td>
</tr>
<tr>
<td>3307 cm⁻¹</td>
<td>-NH of Amide</td>
</tr>
<tr>
<td>1665 cm⁻¹</td>
<td>-CO of Amide</td>
</tr>
<tr>
<td>1357 cm⁻¹</td>
<td>-C=N</td>
</tr>
<tr>
<td>993 cm⁻¹</td>
<td>-N-N of triazole and Thiaiazole</td>
</tr>
<tr>
<td>710-570 cm⁻¹</td>
<td>-C-S not discernible</td>
</tr>
</tbody>
</table>

NMR spectral Signals (δ, ppm)

<table>
<thead>
<tr>
<th>δ (ppm)</th>
<th>Description</th>
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</thead>
<tbody>
<tr>
<td>9.13</td>
<td>(s, 1H, -CONH)</td>
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<tr>
<td>7.50-8.10</td>
<td>(m, 8H, Aromatic)</td>
</tr>
<tr>
<td>7.95, 8.79</td>
<td>(m, 4H, pyridine)</td>
</tr>
<tr>
<td>5.85</td>
<td>(s, 2H, Oxazole)</td>
</tr>
<tr>
<td>2.22, 2.25</td>
<td>(s, 6H, -CH₃)</td>
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</tbody>
</table>

LC-MS data

Mass (m/z) = 560.8 (+Ve)
Figure 4.15 proton NMR spectrum of sample 15a
Figure 4.16 IR spectrum of sample 15a
Figure 4.17 LC-MS spectrum of sample 15a
**Compound – 15b**

N-(4-((3-methylisoxazol-2(5H)-yl)sulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

<table>
<thead>
<tr>
<th>M.F.:C_{25}H_{19}N_{7}O_{4}S_{2}</th>
<th>Elemental Content</th>
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<td>M.W.: 545.59 gm/mole</td>
<td>%C   %H   %N   %S</td>
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<td>M.P.: 212-215°C (Uncorrected)</td>
<td>Cald. 55.04 3.51 17.97 11.75</td>
</tr>
<tr>
<td>Yil.: 74%</td>
<td>Found  55.00 3.50 17.95 11.73</td>
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<table>
<thead>
<tr>
<th>IR Spectral characterization cm$^{-1}$</th>
<th>NMR spectral Signals (δ, ppm)</th>
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</thead>
<tbody>
<tr>
<td>3045 cm$^{-1}$</td>
<td>Aromatic CH</td>
</tr>
<tr>
<td>3303 cm$^{-1}$</td>
<td>-NH of Amide</td>
</tr>
<tr>
<td>1663 cm$^{-1}$</td>
<td>-CO of Amide</td>
</tr>
<tr>
<td>1361 cm$^{-1}$</td>
<td>-C=N</td>
</tr>
<tr>
<td>992 cm$^{-1}$</td>
<td>-N-N of triazole and Thiaiazole</td>
</tr>
<tr>
<td>710-570 cm$^{-1}$</td>
<td>-C-S not discernible</td>
</tr>
<tr>
<td></td>
<td>9.21 (s, 1H, -CONH)</td>
</tr>
<tr>
<td></td>
<td>7.50-8.10 (m, 8H, Aromatic)</td>
</tr>
<tr>
<td></td>
<td>7.96, 8.74 (m, 4H, pyridine)</td>
</tr>
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<td></td>
<td>4.22, 4.48 (m, 3H, Oxazole)</td>
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<td>2.24 (s, 3H, -CH$_3$)</td>
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<table>
<thead>
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<th>LC-MS data</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass (m/z) = 545.1 (+Ve)</td>
</tr>
</tbody>
</table>
Figure 4.16 proton NMR spectrum of sample 15b
Figure 4.18 IR spectrum of sample 15b
**Compound – 15c**

N-(4-((2-methyl-1,3,4-thiadiazol-3(2H)-yl)sulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

<table>
<thead>
<tr>
<th>M.F.:C\textsubscript{24}H\textsubscript{18}N\textsubscript{8}O\textsubscript{3}S\textsubscript{3}</th>
<th><strong>Elemental Content</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>M.W.: 562.65 gm/mole</td>
<td>%C     %H     %N     %S</td>
</tr>
<tr>
<td>M.P.: 237-238\textdegree C (Uncorrected)</td>
<td>Cald. 51.23 3.22 19.92 17.10</td>
</tr>
<tr>
<td>Yil.: 75%</td>
<td>Found 51.20 3.20 19.90 17.08</td>
</tr>
</tbody>
</table>

**IR Spectral characterization cm\textsuperscript{-1}**

| 3049 cm\textsuperscript{-1} | Aromatic CH |
| 3373 cm\textsuperscript{-1} | -NH of Amide |
| 1682 cm\textsuperscript{-1} | -CO of Amide |
| 1352 cm\textsuperscript{-1} | -C=N |
| 993 cm\textsuperscript{-1} | -N-N of triazole and Thiadiazole |
| 710-570 cm\textsuperscript{-1} | -C-S not discernible |

**NMR spectral Signals (δ, ppm)**

| 9.17 | (s, 1H, -CONH) |
| 7.50-8.10 | (m, 8H, Aromatic) |
| 7.97, 8.72 | (m, 4H, pyridine) |
| 3.82 | (m, 1H, thiadiazole) |
| 7.52 | (s, 1H, thiadiazole) |
| 1.43 | (d, 3H, -CH\textsubscript{3}) |

**LC-MS data**

Mass (m/z) = 562.1 (+Ve)
**Compound – 15d**

\[
\text{2-}\left(3\text{-}\left(\text{pyridin-4-yl}\right)\left[1,2,4\right]\text{triazolo}\left[3,4-b\right]\left[1,3,4\right]\text{thiadiazol-6-yl}\right)\text{-N}\left(4\text{-}\left(\text{thiazol-3(2H)-ylsulfonyl}\right)\text{phenyl}\right)\text{benzamide}
\]

<table>
<thead>
<tr>
<th>Elemental Content</th>
<th>%C</th>
<th>%H</th>
<th>%N</th>
<th>%S</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Cald.</strong></td>
<td>52.64</td>
<td>3.13</td>
<td>17.90</td>
<td>17.57</td>
</tr>
<tr>
<td><strong>Found</strong></td>
<td>52.60</td>
<td>3.10</td>
<td>17.88</td>
<td>17.54</td>
</tr>
</tbody>
</table>

**IR Spectral characterization cm\(^{-1}\):**

- 3042 cm\(^{-1}\): Aromatic CH
- 3371 cm\(^{-1}\): -NH of Amide
- 1683 cm\(^{-1}\): -CO of Amide
- 1358 cm\(^{-1}\): -C=\(N\)
- 993 cm\(^{-1}\): -N-N of triazole and Thiadiazole
- 710-570 cm\(^{-1}\): -C-S not discernible

**NMR spectral Signals (\(\delta, \text{ ppm}\)):**

- 9.14 (s, 1H, -CONH)
- 7.50-8.10 (m, 8H, Aromatic)
- 7.95, 8.79 (m, 4H, pyridine)
- 4.76 (s, 1H, thiazole)
- 5.12, 5.86 (s, 2H, thiazole)

**LC-MS data**

Mass (m/z) = 547.1 (+Ve)
**Compound – 15e**

N-(4-((4,5-dimethylisoxazol-2(5H)-yl)sulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

<table>
<thead>
<tr>
<th>Elemental Content</th>
<th>%C</th>
<th>%H</th>
<th>%N</th>
<th>%S</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Cald.</strong></td>
<td>55.80</td>
<td>3.78</td>
<td>17.52</td>
<td>11.46</td>
</tr>
<tr>
<td><strong>Found</strong></td>
<td>55.78</td>
<td>3.76</td>
<td>17.50</td>
<td>11.43</td>
</tr>
</tbody>
</table>

**IR Spectral characterization cm⁻¹**

- 3054 cm⁻¹: Aromatic CH
- 3376 cm⁻¹: -NH of Amide
- 1683 cm⁻¹: -CO of Amide
- 1357 cm⁻¹: -C=N
- 991 cm⁻¹: -N-N of triazole and Thiadiazole
- 710-570 cm⁻¹: -C-S not discernible

**NMR spectral Signals (δ, ppm)**

- 9.18 (s, 1H, -CONH)
- 7.50-8.10 (m, 8H, Aromatic)
- 7.96, 8.78 (m, 4H, pyridine)
- 4.06 (m, 1H, isoxazole)
- 5.83 (s, 1H, isoxazole)
- 1.25, 1.84 (m, 6H, -CH₃)

**LC-MS data**

Mass (m/z) = 559.1 (+Ve)
**Compound – 15f**

N-(4-((4,6-dimethylpyrimidin-1(2H)-yl)sulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

**M.F.:** \( C_{27}H_{22}N_8O_3S_2 \)

**M.W.:** 570.65 gm/mole

**M.P.:** 245-247°C (Uncorrected)

**Yil.:** 72%

<table>
<thead>
<tr>
<th>IR Spectral characterization cm(^{-1})</th>
<th>NMR spectral Signals (( \delta, ) ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3053 cm(^{-1})   Aromatic CH</td>
<td>9.14 (s, 1H, -CONH)</td>
</tr>
<tr>
<td>3376 cm(^{-1})   -NH of Amide</td>
<td>7.50-8.10 (m, 8H, Aromatic)</td>
</tr>
<tr>
<td>1684 cm(^{-1})   -CO of Amide</td>
<td>7.98, 8.77 (m, 4H, pyridine)</td>
</tr>
<tr>
<td>1352 cm(^{-1})   -C=N</td>
<td>3.23 (s, 2H, pyrimidine)</td>
</tr>
<tr>
<td>990 cm(^{-1})    -N-N of triazole and</td>
<td>5.82 (s, 1H, pyrimidine)</td>
</tr>
<tr>
<td>Thiadiazole</td>
<td>2.05, 2.24 (s, 6H, -CH(_3))</td>
</tr>
<tr>
<td>710-570 cm(^{-1}) -C-S not discernible</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Elemental Content</th>
<th>%C</th>
<th>%H</th>
<th>%N</th>
<th>%S</th>
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<tbody>
<tr>
<td>Cald.</td>
<td>56.83</td>
<td>3.89</td>
<td>19.64</td>
<td>11.24</td>
</tr>
<tr>
<td>Found</td>
<td>56.80</td>
<td>3.87</td>
<td>19.60</td>
<td>11.20</td>
</tr>
</tbody>
</table>

**LC-MS data**

Mass (m/z) = 570.1 (+Ve)
**Compound – 15g**

N-(4-(N-acetylsulfamoyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo [3,4-b][1,3,4]thiadiazol-6-yl)benzamide

M.F.: C$_{23}$H$_{17}$N$_7$O$_4$S$_2$

M.W.: 519.56 gm/mole

M.P.: 184-187$^\circ$C (Uncorrected)

Yil.: 68%

<table>
<thead>
<tr>
<th>IR Spectral characterization cm$^{-1}$</th>
<th>Elemental Content</th>
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<tr>
<td>3039 cm$^{-1}$</td>
<td>%C    %H    %N    %S</td>
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<tr>
<td>3377 cm$^{-1}$</td>
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<tr>
<td>1684 cm$^{-1}$</td>
<td></td>
</tr>
<tr>
<td>1355 cm$^{-1}$</td>
<td></td>
</tr>
<tr>
<td>993 cm$^{-1}$</td>
<td></td>
</tr>
<tr>
<td>710-570 cm$^{-1}$</td>
<td></td>
</tr>
</tbody>
</table>

- Aromatic CH
- NH of Amide
- CO of Amide
- C=N
- N-N of triazole and Thiaadiazole
- C-S not discernible

<table>
<thead>
<tr>
<th>NMR spectral Signals ($\delta$, ppm)</th>
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<tr>
<td>8.02 (s, 1H, -SO$_2$NH)</td>
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<tr>
<td>9.12 (s, 1H, -CONH)</td>
</tr>
<tr>
<td>7.50-8.10 (m, 8H, Aromatic)</td>
</tr>
<tr>
<td>7.95, 8.74 (m, 4H, pyridine)</td>
</tr>
<tr>
<td>2.05 (s, 3H, -CH$_3$)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>LC-MS data</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass (m/z) = 519.1 (+Ve)</td>
</tr>
</tbody>
</table>
**Compound – 15h**

![Chemical Structure](image)

2-(3-(pyridin-4-yl)[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)-N-(4-(pyrimidin-1(2H)-ylsulfonyl)phenyl)benzamide

<table>
<thead>
<tr>
<th>M.F.:C₂₅H₁₈N₈O₃S₂</th>
<th><strong>Elemental Content</strong></th>
<th>%C</th>
<th>%H</th>
<th>%N</th>
<th>%S</th>
</tr>
</thead>
<tbody>
<tr>
<td>M.W.: 542.59 gm/mole</td>
<td><strong>Cald.</strong></td>
<td>55.34</td>
<td>3.34</td>
<td>20.65</td>
<td>11.82</td>
</tr>
<tr>
<td>M.P.: 204-206°C (Uncorrected)</td>
<td><strong>Found</strong></td>
<td>55.32</td>
<td>3.30</td>
<td>20.62</td>
<td>11.80</td>
</tr>
<tr>
<td>Yil.: 69%</td>
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</table>

<table>
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<tr>
<th><strong>IR Spectral characterization cm⁻¹</strong></th>
<th><strong>NMR spectral Signals (δ, ppm)</strong></th>
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<tr>
<td>3051 cm⁻¹</td>
<td>Aromatic CH</td>
</tr>
<tr>
<td>3373 cm⁻¹</td>
<td>-NH of Amide</td>
</tr>
<tr>
<td>1684 cm⁻¹</td>
<td>-CO of Amide</td>
</tr>
<tr>
<td>1351 cm⁻¹</td>
<td>-C=N</td>
</tr>
<tr>
<td>990 cm⁻¹</td>
<td>-N-N of triazole and Thiadiazole</td>
</tr>
<tr>
<td>710-570 cm⁻¹</td>
<td>-C-S not discernible</td>
</tr>
<tr>
<td>3051 cm⁻¹</td>
<td>9.19 (s, 1H, -CONH)</td>
</tr>
<tr>
<td>3373 cm⁻¹</td>
<td>7.50-8.10 (m, 8H, Aromatic)</td>
</tr>
<tr>
<td>1684 cm⁻¹</td>
<td>7.98, 8.77 (m, 4H, pyridine)</td>
</tr>
<tr>
<td>1351 cm⁻¹</td>
<td>3.22 (s, 2H, pyrimidine)</td>
</tr>
<tr>
<td>990 cm⁻¹</td>
<td>4.93 (m, 1H, pyrimidine)</td>
</tr>
<tr>
<td>710-570 cm⁻¹</td>
<td>6.92, 7.52 (d, 2H, pyrimidine)</td>
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</tbody>
</table>

**LC-MS data**

Mass (m/z) = 542.1 (+Ve)
Compound – 15i

N-(4-(pyridin-1(2H)-ylsulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide

M.F.: C_{26}H_{19}N_{7}O_{3}S_{2}
M.W.: 541.60 gm/mole
M.P.: 215-217°C (Uncorrected)
Yil.: 68%

<table>
<thead>
<tr>
<th>IR Spectral characterization cm(^{-1})</th>
<th>Elemental Content</th>
<th>NMR spectral Signals (δ, ppm)</th>
<th>LC-MS data</th>
</tr>
</thead>
<tbody>
<tr>
<td>3034 cm(^{-1})</td>
<td></td>
<td>9.13 (s, 1H, -CONH)</td>
<td>Mass (m/z) = 541.1 (+Ve)</td>
</tr>
<tr>
<td>3376 cm(^{-1})</td>
<td>Aromatic CH</td>
<td>7.50-8.10 (m, 8H, Aromatic)</td>
<td></td>
</tr>
<tr>
<td>1682 cm(^{-1})</td>
<td>-NH of Amide</td>
<td>7.95, 8.73 (m, 4H, pyridine)</td>
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</tr>
<tr>
<td>1358 cm(^{-1})</td>
<td>-CO of Amide</td>
<td>3.80-6.50 (m, 6H, pyridine)</td>
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<tr>
<td>991 cm(^{-1})</td>
<td>-C=N</td>
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<td></td>
</tr>
<tr>
<td>710-570 cm(^{-1})</td>
<td>-N-N of triazole and Thiadiazole</td>
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<td></td>
</tr>
<tr>
<td></td>
<td>-C-S not discernible</td>
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<td></td>
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</tbody>
</table>

Elemental Content

<table>
<thead>
<tr>
<th>%C</th>
<th>%H</th>
<th>%N</th>
<th>%S</th>
</tr>
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<tbody>
<tr>
<td>Caled.</td>
<td>57.66 3.54 18.10 11.84</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Found</td>
<td>57.64 3.50 18.08 11.83</td>
<td></td>
<td></td>
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</tbody>
</table>
**Compound – 15j**

![Chemical Structure](image)

**N-(4-((3,4-dimethoxypyridin-1(2H)-yl)sulfonyl)phenyl)-2-(3-(pyridin-4-yl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-6-yl)benzamide**

<table>
<thead>
<tr>
<th>M.F.:C₂₈H₂₃N₇O₅S₂</th>
<th><strong>Elemental Content</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>M.W.: 601.66 gm/mole</td>
<td>%C %H %N %S</td>
</tr>
<tr>
<td>M.P.: 223-225°C (Uncorrected)</td>
<td>Cald. 55.90 3.85 16.30 10.66</td>
</tr>
<tr>
<td>Yil.: 72%</td>
<td>Found 55.88 3.83 16.26 10.64</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>IR Spectral characterization cm⁻¹</strong></th>
<th><strong>NMR spectral Signals (δ, ppm)</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>3052 cm⁻¹ Aromatic CH</td>
<td>9.19 (s, 1H, -CONH)</td>
</tr>
<tr>
<td>3378 cm⁻¹ -NH of Amide</td>
<td>7.50-8.10 (m, 8H, Aromatic)</td>
</tr>
<tr>
<td>1681 cm⁻¹ -CO of Amide</td>
<td>7.94, 8.79 (m, 4H, pyridine)</td>
</tr>
<tr>
<td>1359 cm⁻¹ -C=N</td>
<td>3.80-6.10 (m, 4H, pyridine)</td>
</tr>
<tr>
<td>993 cm⁻¹ -N-N of triazole and Thiadiazole</td>
<td>3.53, 3.82 (s, 6H, -OCH₃)</td>
</tr>
<tr>
<td>710-570 cm⁻¹ -C-S not discernible</td>
<td><strong>LC-MS data</strong></td>
</tr>
</tbody>
</table>

Mass (m/z) = 601.1 (+Ve)
4.4 RESULTS AND DISCUSSION:

The produced derivatives say, N-(substituted-phenyl)-2-(3-(pyridin-4-yl)-[1,2,4] triazolo[3,4-b][1,3,4] thiadiazol-6-yl) benzamide (12a-h, 13a-f, 14a-h and 15a-j) performed by the condensation of 4-amino-5-(pyridinyl-4-yl)-4H-1,2,4-triazole-3-thiol (3) and various phthalamic acid derivatives (5a-h, 6a-f, 7a-h and 8a-j).

Their structures were confirmed by analytical and spectral data. The C, H, N and S contents of the prepared compounds were consistent with their predicted structures as shown in Scheme-4.1 to 4.4. The IR spectra comprise the bands in the region 3200-3300 cm\(^{-1}\) for –NH of -CONH, 1350 cm\(^{-1}\) for -C=N of ring, near 990 cm\(^{-1}\) for -N-N of triazole and thiadiazole mainly and near 1695 cm\(^{-1}\) for -C=O of –CONH group.

The \(^1\)H NMR spectra of the prepared compounds (12a-h, 13a-f, 14a-h and 15a-j) show singlet around 9.2 δ ppm for 1H proton of -CONH linkage, multiplet around 7.4-8.2 δ ppm for aromatic proton and near 7.90, 8.80 δ ppm for pyridine. All other signals like aromatic proton are at their respective positions in the NMR spectrum.

The LC-MS spectrum (Figure 4.3, 4.5, 4.8, 4.11, and 4.14 and 4.17) of six samples i.e. 12a, 12b, 13a, 13b, 14a and 15a shows the peak of M\(^+\) ion at 556.7, 570.8, 534.7, 582.7, 561.7 and 560.8 which is consistent of M.W. of 12a, 12b, 13a, 13b, 14a and 15a i.e. 555.59, 569.62, 533.63, 581.67, 560.65 and 559.62 respectively. All these facts confirm the structures of 12a-h, 13a-f, 14a-h and 15a-j.
REFERENCES:


[5]. Ibrahim, D. A. “Synthesis and biological evaluation of 3,6-disubstituted [1,2,4]triazolo[3,4-b][1,3,4]thiadia zole derivatives


[7]. Amir M.; Kumar H. and Javed S. A. “Condensed bridgehead nitrogen heterocyclic system: Synthesis and pharmacological activities of 1,2,4-triazolo-[3,4-b]-1,3,4-thiadiazole derivatives of ibuprofen and biphenyl-4-yloxy acetic acid”, *Eur. J. Med. Chem.*, **2008**; 43(10), pp.2056-2066.


