6. SYNTHESIS AND CHARACTERISATION

Based on the results of virtual screening 18 novel 1,3,4-thiadiazoles were selected for synthesis. The synthetic procedure involves thermal condensation of different hetero substituted amines with carbon disulphide and hydrazine hydrate to yield hetero substituted thiosemicarbazides which on further condensation with aryl or unsaturated aryl carboxylic acids in presence of phosphoryl chloride as catalyst gave a set of 18 novel 2,5 disubstituted 1,3,4-thiadiazoles.

The synthesized compounds were recrystallised and purified by column chromatography followed by preliminary characterization (TLC, MP) and spectral characterization. The reactants used were of analar grade and were procured from reputed firms. The melting points of the compounds were determined by capillary tube method and are presented uncorrected.

The chemical profile of the reactants used in the synthesis of 1,3,4-thiadiazoles as follows:
<table>
<thead>
<tr>
<th>Property</th>
<th>1H-1,2,4-Triazol-1-Amine</th>
<th>4-Antipyrine</th>
<th>Furan-2-yl-Methanamine</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Structure</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Molecular Weight</strong></td>
<td>84.08</td>
<td>203.24</td>
<td>97.12</td>
</tr>
<tr>
<td><strong>Description</strong></td>
<td>Colourless or white</td>
<td>Light yellow</td>
<td>Colorless to yellow</td>
</tr>
<tr>
<td></td>
<td>Crystals</td>
<td>coloured crystals</td>
<td>liquid</td>
</tr>
<tr>
<td><strong>Solubility</strong></td>
<td>Soluble in ethanol,</td>
<td>Soluble in water and</td>
<td>Soluble in water</td>
</tr>
<tr>
<td></td>
<td>sparingly in chloroform</td>
<td>alcohol</td>
<td></td>
</tr>
<tr>
<td></td>
<td>and ethylacetate.</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Elemental Analysis</strong></td>
<td>C, 28.57; H, 4.80; N, 66.63</td>
<td>C, 65.01; H, 6.45; N, 20.68; O, 7.87</td>
<td>C, 61.84; H, 7.27; N, 14.42; O, 16.47</td>
</tr>
<tr>
<td><strong>Melting Point</strong></td>
<td>149-156 °C</td>
<td>107-111 °C</td>
<td>284.62 [K]</td>
</tr>
<tr>
<td><strong>Boiling Point</strong></td>
<td>200 °C</td>
<td>663.72[K]</td>
<td>436.73[K]</td>
</tr>
<tr>
<td><strong>tPSA</strong></td>
<td>53.98</td>
<td>49.57</td>
<td>35.25</td>
</tr>
<tr>
<td><strong>LogP</strong></td>
<td>-1.502</td>
<td>0.4026</td>
<td>-0.31</td>
</tr>
<tr>
<td><strong>Molar Refractivity</strong></td>
<td>2.0646</td>
<td>59.33[cm²/mol]</td>
<td>-59.83[kJ/mol]</td>
</tr>
<tr>
<td><strong>LogS</strong></td>
<td>0.378</td>
<td>-2.39</td>
<td>26.92[cm²/mol]</td>
</tr>
<tr>
<td><strong>Henry's Law</strong></td>
<td>5.31</td>
<td>4.35</td>
<td>4.6</td>
</tr>
<tr>
<td><strong>pKa</strong></td>
<td>-</td>
<td>-</td>
<td>8.765</td>
</tr>
<tr>
<td><strong>Gibbs Energy</strong></td>
<td>-</td>
<td>398.73[kJ/mol]</td>
<td>66.1[kJ/mol]</td>
</tr>
<tr>
<td><strong>Heat of Form</strong></td>
<td>-</td>
<td>125.29</td>
<td>59.83</td>
</tr>
<tr>
<td><strong>[kJ/mol]</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
## REACTANT PROFILE

<table>
<thead>
<tr>
<th>Property</th>
<th>5-Methyl isoxazole-3-Amine</th>
<th>4-(Phenyldiazenyl) Aniline</th>
<th>4-Methyl pyridin-2-Amine</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Structure</strong></td>
<td><img src="image1" alt="Structure" /></td>
<td><img src="image2" alt="Structure" /></td>
<td><img src="image3" alt="Structure" /></td>
</tr>
<tr>
<td><strong>Molecular Weight</strong></td>
<td>98.10</td>
<td>197.24</td>
<td>108.14</td>
</tr>
<tr>
<td><strong>Description</strong></td>
<td>Yellow crystalline powder</td>
<td>Colourless crystalline powder</td>
<td>Light yellow to brown flakes</td>
</tr>
<tr>
<td><strong>Solubility</strong></td>
<td>Soluble in water</td>
<td>Sparingly soluble in water</td>
<td>Freely soluble in water and DMF</td>
</tr>
<tr>
<td><strong>Elemental Analysis</strong></td>
<td>C, 48.97; H, 6.16; N,28.56;O, 16.31</td>
<td>C,73.07; H, 5.62; N, 21.30</td>
<td>C, 66.64; H, 7.46; N, 25.90</td>
</tr>
<tr>
<td><strong>Melting Point</strong></td>
<td>59-61°C</td>
<td>124°C</td>
<td>96-99°C</td>
</tr>
<tr>
<td><strong>Boiling Point</strong></td>
<td>487.14[K]</td>
<td>360°C</td>
<td>230°C</td>
</tr>
<tr>
<td><strong>tPSA</strong></td>
<td>47.61</td>
<td>50.74</td>
<td>38.38</td>
</tr>
<tr>
<td><strong>LogP</strong></td>
<td>0.46</td>
<td>3.67</td>
<td>1.1</td>
</tr>
<tr>
<td><strong>Heat of Form [kJ/mol]</strong></td>
<td>-17.87</td>
<td>251.59</td>
<td>124.47</td>
</tr>
<tr>
<td><strong>Molar Refractivity [cm³/mol]</strong></td>
<td>27.77</td>
<td>61.96</td>
<td>34.36</td>
</tr>
<tr>
<td><strong>LogS</strong></td>
<td>-0.825</td>
<td>-3.865</td>
<td>-1.36</td>
</tr>
<tr>
<td><strong>Gibbs Energy [kJ/mol]</strong></td>
<td>125.1</td>
<td>245.92</td>
<td></td>
</tr>
<tr>
<td><strong>Henry's Law</strong></td>
<td>3.14</td>
<td>6.67</td>
<td>6.95</td>
</tr>
</tbody>
</table>
## Reactant Profile

<table>
<thead>
<tr>
<th>Property</th>
<th>3,4-Dimethoxy Benzoic acid</th>
<th>2-Hydroxy-5-Sulphobenzoic Acid</th>
<th>Cinnamic Acid</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Structure</strong></td>
<td>![Structure Image]</td>
<td>![Structure Image]</td>
<td>![Structure Image]</td>
</tr>
<tr>
<td><strong>Molecular Weight</strong></td>
<td>182.17</td>
<td>217.99</td>
<td>148.16</td>
</tr>
<tr>
<td><strong>Description</strong></td>
<td>White or slightly yellow powder</td>
<td>White crystalline powder</td>
<td>White crystalline powder</td>
</tr>
<tr>
<td><strong>Solubility</strong></td>
<td>Slightly water soluble</td>
<td>Soluble in water</td>
<td>Sparingly soluble in water</td>
</tr>
<tr>
<td><strong>Elemental Analysis</strong></td>
<td>C, 59.34; H, 5.53; O, 35.13</td>
<td>C, 38.54; H, 2.77; O, 44.00; S, 14.69</td>
<td>C, 72.96; H, 5.44; O, 21.60</td>
</tr>
<tr>
<td><strong>Melting Point</strong></td>
<td>179-182°C</td>
<td>113°C</td>
<td>132-135°C</td>
</tr>
<tr>
<td><strong>Boiling Point</strong></td>
<td>302 °C</td>
<td>882.42[K]</td>
<td>300°C</td>
</tr>
<tr>
<td><strong>tPSA</strong></td>
<td>55.76</td>
<td>111.9</td>
<td>37.3</td>
</tr>
<tr>
<td><strong>LogP</strong></td>
<td>1.34</td>
<td>0.36</td>
<td>1.93</td>
</tr>
<tr>
<td><strong>Heat of Form [kJ/mol]</strong></td>
<td>-620.21</td>
<td></td>
<td>-225.61</td>
</tr>
<tr>
<td><strong>Molar Refractivity [cm³/mol]</strong></td>
<td>46.59</td>
<td>44.49</td>
<td>42.85</td>
</tr>
<tr>
<td><strong>LogS</strong></td>
<td>-1.42</td>
<td>-2.179</td>
<td>-1.896</td>
</tr>
<tr>
<td><strong>Gibbs Energy [kJ/mol]</strong></td>
<td>-435.86</td>
<td>-</td>
<td>-126.38</td>
</tr>
<tr>
<td><strong>Henry's Law</strong></td>
<td>7.81</td>
<td>10.12</td>
<td>6.28</td>
</tr>
<tr>
<td><strong>pKa</strong></td>
<td>4.069</td>
<td>2.374, 11.561</td>
<td>4.085</td>
</tr>
</tbody>
</table>
6.1 MATERIALS AND METHODS

6.1.1 SYNTHESIS\textsuperscript{245, 246}

The synthetic reaction involves thermal condensation of 0.1mM of heterocyclic amines dissolved in 50ml of ethanol and 20ml ammonia solution. Carbon disulphide (15-20ml) is added slowly with constant stirring for 15-60 minutes and allowed to stand for 1-4 hours. Sodium chloro acetate (0.1mol) and 50% hydrazine hydrate (20ml) is added to the reaction mixture warmed, filtered and evaporated to half of its volume and kept overnight. Solid obtained is filtered and recrystallised from ethanol or methanol to yield the various hetero-moieties substituted thiocarbazide.

Aliquot quantities of hetero substituted thiocarbazide 0.02mole and aromatic carboxylic acid 0.01mole were well mixed and pulverized. Fusion of both the reactants was brought about by heating the mixture at temperature 80–200 °C for 2-5 hours.

Completion of reaction was checked by means of thin layer chromatography. Recrystallisation was done with hot water, alcohol, chloroform or a mixture of solvents. The compounds got crystallized on cooling. Crystals so obtained were recrystallised and subjected to further purification by column chromatography where ever necessary (Table 11).

The IUPAC names of the synthesized compounds are

1. [5-(3,4-dimethoxyphenyl)-N-(4H-1,2,4-triazol-4-yl)-1,3,4-thiadiazol-2-amine] \textsuperscript{3205}
2. [(E)-5-styryl-N-(4H-1,2,4-triazol-4-yl)-1,3,4-thiadiazol-2-amine] \textsuperscript{3350}
3. [3-(5-((4H-1,2,4-triazol-4-yl)amino)-1,3,4-thiadiazol-2-yl)-4-hydroxybenzene sulphonic acid] \textsuperscript{3500}
4. [N-(5-(3,4-dimethoxyphenyl)-1,3,4-thiadiazol-2-yl)-5-methylisoxazol-3-amine] \textsuperscript{3650}
5. [(E)-5-methyl-N-(5-styryl-1,3,4-thiadiazole-2-yl)isoxazol-3-amine] \textsuperscript{3800}
6. [4-hydroxy-3-(5-(methylisoxazol-3-yl)amino)-1,3,4-thiadiazol-2-yl)benzene sulphonic acid] \textsuperscript{3950}
7. [5-(3,4-dimethoxyphenyl)-N-(furan-2-ylmethyl)-1,3,4-thiadiazol-2-amine] 4100
8. [(E)-N-(furan-2-ylmethyl)-5-styryl-1,3,4-thiazol-2-amine] 4250
9. [3-(5-((furan-2-ylmethyl)amino)-1,3,4-thiadiazole-2-yl)-4-hydroxybenzene sulphonic acid] 4400
10. [5-(3,4-dimethoxy phenyl)-N-(4-methyl pyridine-2-yl)-1,3,4-thiadiazol-2-amine] 4550
11. [(Z)-N-(4-methylpyridine-2-yl)-5-styryl-1,3,4-thiadiazol-2-amine] 4700
12. [4-hydroxy-3-(3-((4-methyl-pyridin-2-yl)amino)-1,3,4-thiadiazol-2-yl)benzene sulphonic acid] 4850
13. [(Z)-5-(3,4-dimethoxyphenyl)-N-(4-(phenyldiazenyl)phenyl)-1,3,4-thiadiazol-2-amine] 5000
14. [N-(4-((Z)-phenyldiazenyl)phenyl)-5-((Z)-styryl-1,3,4-thiadiazol-2-amine)] 5150
15. [(Z)-4-hydroxy-3-(5-((4-phenyldiazenyl)phenyl)amino)-1,3,4-thiadiazol-2-yl) benzene sulfonic acid] 5300
16. [4-((5-(3,4-dimethoxyphenyl)-1,3,4-thiadiazol-2-yl)amino)-1,5-dimethyl-2-phenyl-1,2-dihydro-3H-pyrazol-3-one] 5450
17. [(E)-1,5-dimethyl-2-phenyl-4-((5-styryl-1,3,4-thiadiazol-2-yl)amino-1,2-dihydro -3H-pyrazole-3-one] 5600
18. [3-(5-(((1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)amino-1,3,4-thiadiazol-2-yl)-2-yl)-4-hydroxybenzenesulphonic acid] 5750

6.1.2 PURIFICATION

Recrystallisation was done with hot water, alcohol or mixture of solvents.

Thin layer chromatography (TLC)

Stationary phase: Precoated Aluminum sheets of Alugram Silica/UV254,

Mobile phase: Chloroform and Methanol (95:5), Ethyl acetooacetate: Chloroform (9:1)

Location of spots: UV and Iodine chamber

Column chromatography

The compound under analysis was applied on to silica gel column of dimension 100cm X 3.5cm to isolate the individual compound using hexane as solvent and the
polarity was increased by chloroform followed by methanol and 100ml each of the 
fractions were collected. The obtained fractions were concentrated and monitored by TLC 
using hexane, chloroform and methanol (97:2:1) as mobile phase. The fractions which 
gave a single spot without any impurity were combined, solvent was allowed to evaporate 
and the pure product was obtained.

6.1.3 SPECTRAL ANALYSIS AND ANALYTICAL PROFILE

The synthesized compounds were characterized by Infrared spectra (IR), Nuclear 
magnetic resonance spectra ($^1$H NMR) ($^{13}$C NMR), Mass spectra and X-ray diffraction 
technique (XRD).

The IR spectrum was recorded using KBr pellets in the range of 4000-400cm$^{-1}$ on 
as Spectrum RXI- Perkin Elmer.

$^{13}$C NMR spectra and proton NMR (400MHz) spectra $^1$H & $^{13}$C NMR was 
recorded in DMSO Jeol GSX liquid state NMR spectrometer. Tetra methyl silane (TMS) 
is used as reference. Chemical shifts are reported in parts per million downfield with 
reference to internal standard.

Mass spectra were recorded on GC Clarus 500 Perkin Elmer system comprising 
an AOC-20i auto sampler and gas chromatograph interfaced to a mass spectrometer (GC- 
MS) instrument.

XRD was recorded on Rigaku Geiger-flex X-Ray Diffractometer.

The following unit gives detail description about synthesis, individual chemical 
properties of reactants, products and their analytical spectra.
6.2 RESULTS AND DISCUSSION

Figure 48: General synthetic scheme for the designed 2, 5-disubstituted thiadiazoles.
Figure 49: Proposed mechanism for synthesis of 2,5-disubstituted thiadiazoles.
Figure 50: Synthetic schemes of compounds 3205, 3350 and 3500.

Figure 51: Synthetic schemes of compounds 3650, 3800 and 3950.
Figure 52: Synthetic schemes of compounds 4100, 4250 and 4400.

Figure 53: Synthetic schemes of compounds 4550, 4700 and 4850.
Figure 54: Synthetic schemes of compounds 5000, 5150 and 5300.

Figure 55: Synthetic schemes of compounds 5450, 5600 and 5750.
Table 11: Physiochemical properties of the synthesized 1,3,4-thiadiazole compounds.

<table>
<thead>
<tr>
<th>Comp. Code</th>
<th>M.F</th>
<th>M.W (g/mol)</th>
<th>m.p (°C)</th>
<th>% Yield</th>
<th>Period (hr)</th>
<th>Rf Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>3205</td>
<td>C10H8N6O4S2</td>
<td>340.32</td>
<td>187-189</td>
<td>70</td>
<td>1.5</td>
<td>0.61</td>
</tr>
<tr>
<td>3350</td>
<td>C12H12N6O2S</td>
<td>270.31</td>
<td>201-204</td>
<td>76</td>
<td>1.7</td>
<td>0.53</td>
</tr>
<tr>
<td>3500</td>
<td>C13H10N6O4S</td>
<td>340.33</td>
<td>175-177</td>
<td>65</td>
<td>2</td>
<td>0.84</td>
</tr>
<tr>
<td>3650</td>
<td>C12H10N4O4S2</td>
<td>318.35</td>
<td>193-195</td>
<td>68</td>
<td>1.4</td>
<td>0.33</td>
</tr>
<tr>
<td>3800</td>
<td>C14H14N6O4S</td>
<td>284.33</td>
<td>211-213</td>
<td>72</td>
<td>1.3</td>
<td>0.45</td>
</tr>
<tr>
<td>3950</td>
<td>C14H12N4O4S</td>
<td>354.35</td>
<td>189-191</td>
<td>70</td>
<td>1.6</td>
<td>0.55</td>
</tr>
<tr>
<td>4100</td>
<td>C13H11N5O4S2</td>
<td>423.49</td>
<td>176-178</td>
<td>65</td>
<td>1.9</td>
<td>0.67</td>
</tr>
<tr>
<td>4250</td>
<td>C15H15N5O4S</td>
<td>389.47</td>
<td>203-205</td>
<td>60</td>
<td>2</td>
<td>0.71</td>
</tr>
<tr>
<td>4400</td>
<td>C15H15N6O4S</td>
<td>459.49</td>
<td>191-193</td>
<td>77</td>
<td>1.8</td>
<td>0.71</td>
</tr>
<tr>
<td>4550</td>
<td>C19H17N3O4S2</td>
<td>328.39</td>
<td>179-181</td>
<td>67</td>
<td>1.5</td>
<td>0.43</td>
</tr>
<tr>
<td>4700</td>
<td>C21H17N4O4S</td>
<td>294.37</td>
<td>205-207</td>
<td>71</td>
<td>1.7</td>
<td>0.64</td>
</tr>
<tr>
<td>4850</td>
<td>C21H19N4O4S</td>
<td>364.39</td>
<td>197-199</td>
<td>70</td>
<td>2</td>
<td>0.53</td>
</tr>
<tr>
<td>5000</td>
<td>C14H12N4O4S2</td>
<td>417.48</td>
<td>182-184</td>
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<td>1.4</td>
<td>0.75</td>
</tr>
<tr>
<td>5150</td>
<td>C16H16N4O4S</td>
<td>383.47</td>
<td>213-215</td>
<td>80</td>
<td>1.3</td>
<td>0.25</td>
</tr>
<tr>
<td>5300</td>
<td>C16H16N4S</td>
<td>453.49</td>
<td>169-171</td>
<td>70</td>
<td>1.6</td>
<td>0.37</td>
</tr>
<tr>
<td>5450</td>
<td>C20H15N5O4S2</td>
<td>317.36</td>
<td>185-187</td>
<td>69</td>
<td>1.9</td>
<td>0.61</td>
</tr>
<tr>
<td>5600</td>
<td>C22H19N4O4S</td>
<td>283.34</td>
<td>207-209</td>
<td>60</td>
<td>2</td>
<td>0.91</td>
</tr>
<tr>
<td>5750</td>
<td>C22H17N5S</td>
<td>353.36</td>
<td>192-194</td>
<td>79</td>
<td>1.8</td>
<td>0.43</td>
</tr>
</tbody>
</table>

Table 12: Molecular properties of the synthesized 1,3,4-thiadiazoles.

<table>
<thead>
<tr>
<th>Comp. Code</th>
<th>Boiling Point at 760mmHg</th>
<th>ΔVap (TBP) KJ/mol</th>
<th>log S</th>
<th>log D in pH</th>
<th>pKa</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1.7</td>
<td>4.6</td>
</tr>
<tr>
<td>3205</td>
<td>514.06±60</td>
<td>78.56</td>
<td>-4.381</td>
<td>3.74</td>
<td>3.77</td>
</tr>
<tr>
<td>3350</td>
<td>1027.38±28.53</td>
<td>76.90</td>
<td>-4.382</td>
<td>-0.24</td>
<td>1.25</td>
</tr>
<tr>
<td>3500</td>
<td>1034±17.21</td>
<td>76.35</td>
<td>-4.839</td>
<td>-3.52</td>
<td>-4.75</td>
</tr>
<tr>
<td>3650</td>
<td>1031.35±39.86</td>
<td>77.30</td>
<td>-4.506</td>
<td>2.74</td>
<td>2.77</td>
</tr>
<tr>
<td>3800</td>
<td>1013.47±36.32</td>
<td>75.50</td>
<td>-4.523</td>
<td>3.47</td>
<td>3.52</td>
</tr>
<tr>
<td>3950</td>
<td>1141.12±15.62</td>
<td>76.30</td>
<td>-4.994</td>
<td>-0.88</td>
<td>-3.5</td>
</tr>
<tr>
<td>4100</td>
<td>1106.84±40.81</td>
<td>85.13</td>
<td>-3.668</td>
<td>1.87</td>
<td>2.23</td>
</tr>
<tr>
<td>4250</td>
<td>1087.79±40.58</td>
<td>83.12</td>
<td>-3.987</td>
<td>2.42</td>
<td>2.78</td>
</tr>
<tr>
<td>4400</td>
<td>1049.65±17.32</td>
<td>81.35</td>
<td>-4.458</td>
<td>-1.73</td>
<td>-4.05</td>
</tr>
<tr>
<td>4550</td>
<td>1039.03±39.96</td>
<td>78.08</td>
<td>-6.231</td>
<td>-0.36</td>
<td>2.32</td>
</tr>
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<td>1020±36.46</td>
<td>76.19</td>
<td>-6.045</td>
<td>0.67</td>
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<td>-6.525</td>
<td>-2.01</td>
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<td>87.59</td>
<td>-4.988</td>
<td>5.91</td>
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<td>-7.476</td>
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<td>72.81</td>
<td>-7.497</td>
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<td>5750</td>
<td>1015±15.46</td>
<td>75.25</td>
<td>-7.973</td>
<td>-1.28</td>
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Analytical Data of (E)-5-styryl-N-(4H-1,2,4-Triazol-4-yl)-1,3,4-thiadiazol-2-amine

(3350)

Description : Crystalline yellow solid

Melting Point : 201-204°C

\[ \text{IR } \delta_{\text{KBr}}^{\text{cm}^{-1}} \] :

- 3344 cm\(^{-1}\) (s) N-H of Amine,
- 2993 cm\(^{-1}\) CH\(_3\) (s)
- 3066 cm\(^{-1}\) (s) C=C Aromatic ring,
- 1398 cm\(^{-1}\) (s) C=N
- 623 cm\(^{-1}\) (s) = C-S

\(^1\text{H NMR } (200\text{MHz, DMSO-}\delta,)\) :

- 6.165 (s, 1H, NH),
- 6.93-6.90 (d, 1H, CH=CH alkene),
- 7.70-7.73 (m, 5H, CH Aromatic),
- 8.04 (d, 2H, CII IIetero Aromatic)

\(^{13}\text{C NMR } (50\text{MHz, DMSO-d6, 25°C}) \delta:\)

- 111, 116, 127, 132-138 (C=C aromatic),
- 123.3 (CH=CH)
- 161-171 (C=N Hetero aromatic)

MS m/z :
- 270.31 (M) \(^+\) Ion Peak

XRD :
- 20 = 24, Counts: 247 (Range 0-250)
Spectra 1: IR, $^1$H NMR, $^{13}$C NMR and Mass spectrums of (E)-5-styryl-N-(4H-1,2,4-triazol-4-yl)-1,3,4-thiadiazol-2-amine (3350)

Spectra 2: XRD spectra of (E)-5-styryl-N-(4H-1,2,4-triazol-4-yl)-1,3,4-thiadiazol-2-amine (3350)
Analytical Data of (E)-5- methyl-N-(5-styryl-1,3,4-thiadiazole-2-yl)isoxazol-3-amine (3800)

Description : Yellow crystalline powder

Melting Point : 211-213°C

\[ \text{IR } \nu_{\text{KBr}} \text{ cm}^{-1} \] : 3405 cm\(^{-1}\) (s) N-H of Amine,

3254 cm\(^{-1}\) (s) C=C Aromatic ring,

2967 cm\(^{-1}\) (s) C=C alkene,

1449 cm\(^{-1}\) (s) C-H CH\(_3\),

642 cm\(^{-1}\) (s) C-S

\(^1\text{H NMR} \) (200MHz, DMSO-d\(_6\), 25°C) \( \delta \) : 6.678 (s, 1H, NH),

2.499 (s, 3H, CH\(_3\)),

7.5 (m, 3H, CH Aromatic),

7.70-7.72 (d, 1H, CH =CH)

\(^1\text{C NMR} \) (50MHz, DMSO-d\(_6\), 25°C) \( \delta \) : 20 ppm = CH\(_3\),

111-137 (C=C aromatic),

161-171 (C=N Hetero aromatic)

\( \text{MS m/z} \) : 285.12 (M)\(^+\) Ion Peak

\( \text{XRD} \) : 20 = 30, Counts: 242 (Range 0-250)
Spectra 3: IR, $^1$H NMR, $^{13}$C NMR and Mass spectrums of (E)-5- methyl-N-(5-styryl-1, 3, 4-thiadiazole-2-yl) isoxazol-3-amine (3800)

Spectra 4: XRD spectrums of (E)-5- methyl-N-(5-styryl-1, 3, 4-thiadiazole-2-yl) isoxazol-3-amine (3800)
Analytical Data (E)-1,5-dimethyl-2-phenyl-4-((5-styryl-1,3,4-thiadiazol-2-yl) amino)-1,2-dihydro-3H-pyrazole-3-one (4250)

Description : Colorless crystalline powder

Melting Point : 203-205°C

\[\text{IR } \nu_{\text{KBr}}^{\text{cm}^{-1}}\]

: 3395 cm\(^{-1}\) (s) = N-H of Amine,
1649 cm\(^{-1}\) (s) C=O
2924 cm\(^{-1}\) (s) C–C alkenes,
2149 cm\(^{-1}\) (s) C=N,
1157 cm\(^{-1}\) (s) C-N,
1339 cm\(^{-1}\) (b) CH\(_3\)
758 cm\(^{-1}\) (s) C-S

\[^1\text{H NMR} (200\text{MHz, DMSO-d6, 25°C})\delta:\]

: 2.53 (s, 3H, CH\(_3\))
9.55 (s, 1H, NH),
7.06 (d, 1H, CH Alkene),
3.84 (s, 3H, OCH\(_3\))

\[^13\text{C NMR} (50\text{MHz, DMSO-d6, 25°C})\delta:\]

: 55-CH\(_3\)
110-138(C=C aromatic),
161-171 (C=N Hetero aromatic)

\[^{\text{MS m/z}}\]

: 391.10 (M) \(^{12}\text{Ion Peak}\)

\[^{\text{XRD}}\]

: 20 = 45, Counts: 247 (Range 0-250)
Spectra 5: IR, $^1$H NMR, $^{13}$C NMR and Mass spectrums of (E)-1,5-dimethyl-2-phenyl-4-((5-styryl-1,3,4-thiadiazol-2-yl)amino)-1,2-dihydro-3H-pyrazole-3-one (4250)

Spectra 6: XRD spectrums of (E)-1,5-dimethyl-2-phenyl-4-((5-styryl-1,3,4-thiadiazol-2-yl)amino)-1,2-dihydro-3H-pyrazole-3-one (4250)
Analytical Data of (Z)-N-(4-methylpyridine-2-yl)-5-styryl-1,3,4-thiadiazol-2-amine
(4700)

Description : Colourless crystalline powder

Melting Point : 205-207°C

IR $\nu_{\text{cm}^{-1}}$ : 3424 cm$^{-1}$ (s) = N-H of Amine,
3157 cm$^{-1}$ (s) C=C Aromatic ring,
3048 cm$^{-1}$ (s) C=C alkene
847 cm$^{-1}$ (s) C=N,
1452 cm$^{-1}$ (b) CH$_3$ 675 cm$^{-1}$ (s) = C=S

$^1$H NMR (200MHz, DMSO-d$_6$) $\delta$ : 5.87 (s, 1H, NH),
7.6 - 7.7 (m, 5H, CH Aromatic),
7.4 (d, 3H, CH Hetero Aromatic),
6.61 (d, 1H, C=C)

$^{13}$C NMR (50MHz, DMSO-d$_6$) $\delta$ : 20 (CH$_3$)
119 - 143 (C=C aromatic),

MS m/z : 294.1 (M)$^+$ Ion Peak

XRD : $\theta = 5$, Counts: 242 (Range 0-250)
Spectra 7: IR, $^1$H NMR, $^{13}$C NMR and Mass spectrums of (Z)-N-(4-methylpyridine-2-yl)-5-styryl-1,3,4-thiadiazol-2-amine (4700)

Spectra 8: XRD spectrums of (Z)-N-(4-methylpyridine-2-yl)-5-styryl-1,3,4-thiadiazol-2-amine (4700)
Analytical Data of N-(4-((Z)-phenyldiazenyl) phenyl)-5-((Z)-styryl-1, 3, 4-thiadiazol-2-amine (5150)

Description : Dark Brown fine powder

Melting Point : 213-215°C

\[ \text{IR } \nu_{\text{cm}^{-1}} \]

- 3253 cm\(^{-1}\) (s) N-H
- 3050 cm\(^{-1}\) (s) C=C Aromatic ring,
- 2964 cm\(^{-1}\) (s) C=C alkene.
- 1661 cm\(^{-1}\) C=C ring stretching
- C-N (s) tertiary 1398 cm\(^{-1}\)
- 697 cm\(^{-1}\) (s) C-S

\[^1\text{H NMR (200MHz, DMSO-d6)} \delta \]:

- 6.280 (s, 1H, NH),
- 6.980 (d, 1H, CH)
- 7.7 (q, 4H, CH Aromatic),
- 8.09 (q, 4H, CH),

\[^13\text{C NMR (50MHz, DMSO-d6)} \delta \]:

- 127-138 (C=C aromatic),
- 111-116 (alkene)
- 161 (C=N aromatic)

\text{MS m/z} : 385.30 (M)\(^+\) Ion Peak

\text{XRD} : 2θ=18, count 243.
Spectra 9: IR, $^1$H NMR, $^{13}$C NMR and Mass spectrum of N-(4-((Z)-phenyldiazenyl) phenyl)-5-((Z)-styryl-1, 3,4-thiadiazol-2-amine (5150)

Spectra 10: XRD spectrums of N-(4-((Z)-phenyldiazenyl) phenyl)-5-((Z)-styryl-1,3,4-thiadiazol-2-amine (5150)
Analytical Data of (E)-N-(furan-2-ylmethyl)-5-styryl-1,3,4-thiazol-2-amine (5600)

**Description**: Brownish crystalline powder

**Melting Point**: 207-209°C

**IR**

\[ \text{cm}^{-1} \]

: 3442 cm\(^{-1}\) (s) = N-H of Amine,

2597 cm\(^{-1}\) CH\(_2\) (s)

1638 cm\(^{-1}\) (s) C=C Aromatic ring,

1384 cm\(^{-1}\) CH\(_2\) (b),

2364 cm\(^{-1}\) (s) C=N,

1324 cm\(^{-1}\) (b) CH\(_3\) 641 cm\(^{-1}\) (s) = C-S

**\(^1\text{H NMR}\) (200MHz, DMSO-d6) \(\delta\)**

: 5.87 (s, 1H, NH),

9.55 (s, 1H, OH),

7.4-7.6 (m, 5H, CH Aromatic),

7.4 (d, 3H, CH Hetero Aromatic),

7.0 (d, 1H, C=C)

3.83(d, 2H, CH\(_2\))

**\(^{13}\text{C NMR}\) (50MHz, DMSO-d6) \(\delta\)**

: 55.5 ppm = CH\(_2\),

110-123 (C=C aromatic),

148-167 (C=N Hetero aromatic)

**MS m/z**

: 283.15 (M\(^+\) Ion Peak

**XRD**

: 20 = 40, Counts: 226 (Range 0-250)
Spectra 11: IR, $^1$H NMR, $^{13}$C NMR and Mass spectra of (E)-N-(furan-2-ylmethyl)-5-styryl-1, 3, 4-thiazol-2-amine (5600)

Spectra 12: XRD spectra of (E)-N-(furan-2-ylmethyl)-5-styryl-1, 3, 4-thiazol-2-amine (5600)
Analytical Data of 5-(3,4-dimethoxyphenyl)-N-(4H-1,2,4-triazol-4-yl)-1,3,4-thiadiazol-2-amine (3205)

![Chemical Structure]

**Description**: Pale white crystalline powder

**Melting Point**: 187-189°C

**IR $\nu_{\text{cm}^{-1}}^{\text{KBr}}$**
- 3373 cm$^{-1}$ (s) $-$ N-H of Amine,
- 3195 cm$^{-1}$ (s) C=C Aromatic ring,
- 2923 cm$^{-1}$ (s) C-H methyl
- 1387 cm$^{-1}$ (s) C=N,
- 1609 cm$^{-1}$ (b) CH$_3$
- 1676.85 cm$^{-1}$ (s) C=C ring Stretching
- 657, 696 cm$^{-1}$ (s) C-S

**$^1$H NMR (200MHz, DMSO-d$_6$) $\delta$**
- 6.173 (s, 1H, NH),
- 2.502 (s, 1H, OCH$_3$),
- 7.71-7.74 (m, 3H, CH Aromatic),
- 8.060-8.053 (d, 2H, Hetero Aromatic)

**$^{13}$C NMR (50MHz, DMSO-d$_6$) $\delta$**
- 55 – OCH$_3$
- 111, 116, 127, 132-138 (C=C aromatic),
- 161-171 (C=N Hetero aromatic)

**MS m/z** : 341.55 (M)$^+$ Ion Peak
Analytical Data of 3-(5-((4H-1,2,4-triazol-4-yl)amino)-1,3,4-thiadiazol-2-yl)-4-hydroxy benzenesulphonic acid (3500)

![Chemical Structure]

Description: Fine Yellow crystals

Melting Point: 175-177°C

IR $\nu_{KBr}$ cm$^{-1}$:
- 3402 cm$^{-1}$ (s) = N-H of Amine,
- 1272.93 cm$^{-1}$ (s) C-N tertiary
- 1571 cm$^{-1}$ C=N (s)
- 642 cm$^{-1}$ (s) = C-S

$^1$H NMR (200MHz, DMSO-d6) $\delta$:
- 5.711 (s, 1H, NH),
- 7.5 (q, 4H, CH Aromatic),
- 7.3-7.4 (d, 2H, CH Hetero Aromatic),
- 8.9 (s, 1H, OH)

$^{13}$C NMR (50MHz, DMSO-d6) $\delta$:
- 152-167 (C=C Heteroaromatic)
- 110-148 (C=C aromatic),

MS m/z:
- 340.65 (M)$^+$ Ion Peak
Spectra 13: IR, $^1$H NMR, $^{13}$C NMR and Mass spectrums 5-(3, 4-dimethoxyphenyl)-N-(4H-1, 2, 4-triazol-4-yl)-1,3,4-thiadiazol-2-amine (3305)

Spectra 14: IR, $^1$H NMR, $^{13}$C NMR and Mass spectrums of 3-(5-((4H-1, 2, 4-triazol-4-yl) amino)-1, 3, 4-thiadiazol-2-yl)-4-hydroxybenzene sulphonic acid (3500)