Synthesis and Characterization of Benzoxazole Derivatives

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ABSTRACT

In view of the fact that a large number of derivatives of benzoxazole have been found to exhibit a wide variety of pharmacological activities. In the current research work, the title compounds were synthesized from 2-substituted Benzoxazole-5-carboxyhydrazide(II) on treatment with equivalent quantities of anhydrides afforded 1-[(2-substituted-1,3-benzoxazol-5-yl)carbonyl]-1,2-dihydropyridazine-3,6-dione(IIIa), of 1-[(2-substituted-1,3-benzoxazol-5-yl)carbonyl] tetrahydro pyridazine-3,6-dione(IV) and 2-[(1,3-benzoxazol-5-yl)carbonyl]-2,3-dihydrophthalazine-1,4-dione(V). The identification and characterization of all the synthesized compounds were confirmed by Elemental analysis, melting point, Thin Layer Chromatography, FT-IR, ¹H NMR and Mass Spectral data.

Keywords- Benzoxazole, Thin Layer Chromatography, FT-IR, NMR.

INTRODUCTION

The main objective of the medicinal chemistry is to synthesize the compounds that show promising activity as therapeutic agents with lower toxicity. Benzoxazoles have been reported to show a broad spectrum of biological activities. Notable among these are antihistaminic [1], antifungal[2], cyclooxygenase Inhibiting[3], antitumor[4], antiulcer[5], anticonvulsant[6], hypoglycemic[7], anti-inflammatory[8-10] and antitubercular activity[11], antibacterial activity[12]. In the present work three different heterocycles [13-20] were incorporated to benzoxazole moiety.
MATERIALS AND METHODS

All the reagents and solvents used were of laboratory grade. The melting points of synthesized compounds were determined by open capillary method and were uncorrected. The purity and homogeneity of compounds were checked using TLC technique. IR spectra[21] of compounds were recorded using KBr pellets on Perkin Elmer 337 spectrophotometer. 1H-NMR spectra[22] were recorded on Bruker Avance-300 MHz Spectrophotometer using CDCl₃ as solvent at Indian Institute of Chemical Technology(IICT), Hyderabad. Mass Spectra of the synthesized compounds were recorded on Liquid Chromatography Mass Spectrometer at Indian Institute of Chemical Technology(IICT), Hyderabad. The compounds were also subjected to C, H, N and S analysis (ThermoFinnigan) at IICT (Hyderabad).

• Synthesis and Characterization of Compounds:

1) Synthesis of 2-substituted benzoxazole-5-carboxylic acid hydrazide (II)
A mixture of a 2-substituted benzoxazole-5-carboxylic acid methyl ester IV (0.085 mol) in alcohol (150 ml) and hydrazine hydrate (99%, 0.212 mol) was heated under reflux on water bath for 8 hours. The alcohol was evaporated till 40ml remained and cooled. The product precipitated was filtered and washed with small amount of cold alcohol and followed by cold water repeatedly and dried.

The following three compounds were synthesized by using the above procedure.

a) Benzoxazole-5-carboxylic acid hydrazide (IIa)
Percentage Yield- 85%, M.P. 108-110°C, Rf-0.6 (Methanol)

b) 2-methyl-benzoxazole-5-carboxylic acid hydrazide (IIb)
Percentage Yield- 80%, M.P. 144-146°C, Rf-0.7 (Methanol)

c) 2-ethyl-benzoxazole-5-carboxylic acid hydrazide (IIc)
Percentage Yield- 77%, M.P. 138-140°C, Rf-0.6 (Methanol)

2) Synthesis of 1-[(2-substituted-1,3-benzoxazol-5-yl)carbonyl]-1,2-dihydropyridazine-3,6-dione(III)
A 11.29mmol of 1,3-benzoxazole-5-carboxyhydrazide dissolved in 20 ml of acetic acid and 11.29mmol of maleic anhydride added to the above and content refluxed for 6 hrs, reaction monitored by TLC (Methanol:EtOAc/6:4), quenched in to the ice under stirring to get the solid, filtered the solid, washed the solid with chilled water and recrystallized from minimum amount of methanol to get the white crystalline powder.

The following three compounds were synthesized by using the above procedure.

a) 1-[(1,3-benzoxazol-5-yl)carbonyl]-1,2-dihydropyridazine-3,6-dione (IIIA)
(Yield: 2.5g, 85% MP:185-187°C)

b) 1-[(2-methyl-1,3-benzoxazol-5-yl)carbonyl]-1,2-dihydropyridazine-3,6-dione (IIIB)
(Yield: 2.4g, 84% MP:190-192°C)

c) 1-[(2-ethyl-1, 3-benzoxazol-5-yl)carbonyl]-1,2-dihydropyridazine-3,6-dione (IIIC)
(Yield: 2.11g, 80% MP: 198-200°C)
3) Synthesis of 1-[(2-substituted-1,3-benzoxazol-5-yl)carbonyl]tetrahydropyridazine-3,6-dione (IV)
A 11.29mmol of 1,3-benzoxazole-5-carbohydrazide dissolved in 20 ml acetic acid and 11.29mmol of oxolane-2,5-dione added to the above and content refluxed for 8 hrs, reaction monitored by TLC (Methanol:EtOAc/6:4), quenched in to the ice under stirring to get the solid, filtered the solid, washed the solid with chilled water and recrystallized from minimum amount of methanol to get the white powder.

The following three compounds were synthesized by using the above procedure.

a) 1-[(1,3-benzoxazol-5-yl)carbonyl]tetrahydropyridazine-3,6-dione (IVa)  
(Yield: 2.25g, 78% MP: 170-172°C)

b) 1-[(2-methyl-1,3-benzoxazol-5-yl)carbonyl]tetrahydropyridazine-3,6-dione (IVb)  
(Yield: 2.3g, 80% MP: 205-207°C)

c) 1-[(2-ethyl-1,3-benzoxazol-5-yl)carbonyl]tetrahydropyridazine-3,6-dione (IVc)  
(Yield: 2.0g, 75% MP: 223-225°C)

4) Synthesis of 2-[(2-substituted-1,3-benzoxazol-5-yl)carbonyl]-2,3-dihydrophthalazine-1,4-dione (V)
A 11.29mmol of 1,3-benzoxazole-5-carbohydrazide dissolved in 20 ml of acetic acid and 11.29mmol of phthalic anhydride added to the above and content refluxed for 8 hrs, reaction monitored by TLC (Methanol:EtOAc/6:4), quenched in to the ice under stirring to get the solid, filtered the solid, washed the solid with chilled water and recrystallized from minimum amount of methanol to get the white crystalline powder.

The following three compounds were synthesized by using the above procedure.

a) 2-[(1,3-benzoxazol-5-yl)carbonyl]-2,3-dihydrophthalazine-1,4-dione (Va)  
(Yield: 2.83g, 82% MP: 220-222°C)

b) 2-[(2-methyl-1,3-benzoxazol-5-yl)carbonyl]-2,3-dihydrophthalazine-1,4-dione (Vb)  
(Yield: 2.9g, 86% MP: 220-222°C)

c) 2-[(2-ethyl-1,3-benzoxazol-5-yl)carbonyl]-2,3-dihydrophthalazine-1,4-dione (Vc)  
(Yield: 2.52g, 84% MP: 234-236°C)

Compound (IIIa)  
1-[(1,3-benzoxazol-5-yl)carbonyl]-1,2-dihydropyridazine-3,6-dione (IIIa)  
Percentage Yield- 85%, M.P. 185-187°C, RF-0.63 (Methanol:EtOAc/6:4); IR (KBr) cm⁻¹: 3320(-NH- str.), 1730 (-CO- str.), 1630 (C=N str.), 3084 (Ar–H str.), 877 (C=C bending), ¹H NMR: (CDCl₃) δ 7.71-7.85 (m, 3H), δ 8.4 (s, 1H), δ 8.8 (m, 1H), δ 2.5-2.7 (m, 2H). FAB-MS: (m/z, 100%): 257 (M+).

Compound (IIIb)  
1-[(2-methyl-1,3-benzoxazol-5-yl)carbonyl]-1,2-dihydropyridazine-3,6-dione (IIIb)  
Percentage Yield-84% M.P. 190-192°C, RF-0.67 (Methanol: EtOAc/6:4); IR (KBr) cm⁻¹: 3324(-NH- str.), 1730 (-CO- str.), 1627 (C=N str.), 3090 (Ar–H str.), 838 (C=C bending), 2900 (CH str.), ¹H NMR: (CDCl₃) δ 7.7(d, 1H), δ 7.95 (m, 1H), δ 8.8 (m, 1H), δ 2.5-2.7 (m, 2H).
Compound (IIIc)
1-[(2-ethyl-1,3-benzoxazol-5-yl)carbonyl]-1,2-dihydropyridazine-3,6-dione (IIIc)
Percentage Yield-80% M.P. 198-200°C, Rf-0.65 (Methanol:EtOAc/6:4); IR (KBr) cm⁻¹: 3325(-NH str.), 1732 (-CO str.), 1634 (C=N str.), 2890 (C–H str.), 3010 (Ar–H str.), 900 (C=C bending), ¹H NMR: (CDCl₃) δ 8.0-8.4 (m, 4H), δ 7.3-7.5 (m, 2H), δ 3.0 (q, 2H), δ 1.3 (t, 3H).

Compound (IVa)
1-[(1,3-benzoxazol-5-yl)carbonyl]tetrahydropyridazine-3,6-dione (IVa)
Percentage Yield-78% M.P. 170-172°C, Rf-0.53 (Methanol:EtOAc/6:4); IR (KBr) cm⁻¹: 3320(-NH str.), 1732 (-CO str.), 1625 (C=N str.), 3025 (Ar–H str.), 1640 (C=C str.) 3100 (CH str.), ¹H NMR: (CDCl₃) δ 7.6-7.85 (m, 4H), δ 8.4 (s, 1H), δ 2.6-2.7 (m, 4H).
Compound (IVb)
1-[(2-methyl-1,3-benzoxazol-5-yl)carbonyl]tetrahydro pyridazine-3,6-dione (IVb)
Percentage Yield-80% M.P. 205-207°C, Rf-0.55 (Methanol:EtOAc/6:4); IR (KBr) cm\(^{-1}\): 3290(-NH- str.), 1734 (-CO- str.), 1630 (C=N str.), 3084 (Ar–H str.), 1640 (C=C str.) 3000 (CH str.), \(^1\)H NMR: (CDCl\(_3\)) \(\delta\) 7.7- 7.95 (m, 3H), \(\delta\) 8.65 (m, 1H), \(\delta\) 2.60-2.70 (m, 7H).

Compound (IVc)
1-[(2-ethyl-1,3-benzoxazol-5-yl)carbonyl]tetrahydro pyridazine-3,6-dione (IVc)
Percentage Yield,75% M.P. 223-225°C, Rf-0.55 (Methanol:EtOAc/6:4); IR (KBr) cm\(^{-1}\): 3320(-NH- str.), 1738 (-CO- str.), 1626 (C=N str.), 3094 (Ar–H str.), 1654 (C=C str.), \(^1\)H NMR: (CDCl\(_3\)) \(\delta\) 8.0-8.3 (m, 4H), \(\delta\) 3.0(q, 2H) \(\delta\) 2.6-2.7 (m, 4H), \(\delta\) 1.3 (t,3H).

Compound (Va)
2-[(1,3-benzoxazol-5-yl)carbonyl]-2,3-dihydrophthalazine-1,4-dione (Va)
Percentage Yield-82% M.P. 220-222°C, Rf-0.61 (Methanol:EtOAc/6:4); IR (KBr) cm\(^{-1}\): 3390(-NH- str.), 1730 (-CO- str.), 1630 (C=N str.), 3085 (Ar–H str.), 835 (C=C str.), \(^1\)H NMR: (CDCl\(_3\)) \(\delta\) 7.6-7.9(m, 7H), \(\delta\) 8.5 (s, 1H), \(\delta\) 8.8 (m, 1H).

Compound (Vb)
2-[(2-methyl-1,3-benzoxazol-5-yl)carbonyl]-2,3-dihydrophthalazine-1,4-dione (Vb)
Percentage Yield-86% M.P. 220-222°C, Rf-0.66 (Methanol:EtOAc/6:4); IR (KBr) cm\(^{-1}\): 3390(-NH- str.), 1732 (-CO- str.), 1625 (C=N str.), 3094 (Ar–H str.), 900 (C=C bending.),3000(CH str.), \(^1\)H NMR: (CDCl\(_3\)) \(\delta\) 7.4-7.9(m, 7H), \(\delta\) 8.6 (s, 1H), \(\delta\) 8.8 (m, 1H) \(\delta\) 2.65 (s, 3H).

Compound (Vc)
2-[(2-ethyl-1,3-benzoxazol-5-yl)carbonyl]-2,3-dihydrophthalazine-1,4-dione (Vc)
Percentage Yield-84% M.P. 234-236°C, Rf-0.64 (Methanol:EtOAc/6:4); IR (KBr) cm\(^{-1}\): 3270(-NH- str.), 1730 (-CO- str.), 1620 (C=N str.), 3025 (Ar–H str.), 900 (C=C bending.), \(^1\)H NMR: (CDCl\(_3\)) \(\delta\) 7.6-8.3 (m, 8H), \(\delta\) 3.1 (q, 2H), \(\delta\) 1.28(t, 3H).

RESULTS AND DISCUSSION

- Lead Nucleus with Different substituents (R1 and R2) were summarized in Table No.1:

![5-substituted,2- substituted-1,3-benzoxazole](image_url)

Table No.1

<table>
<thead>
<tr>
<th>COMPOUND</th>
<th>R&lt;sub&gt;1&lt;/sub&gt;</th>
<th>R&lt;sub&gt;2&lt;/sub&gt;</th>
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<tr>
<td>IIIa</td>
<td>H</td>
<td>1,2-dihydropyridazine-3,6-dione</td>
</tr>
<tr>
<td>IIIb</td>
<td>CH₃</td>
<td>1,2-dihydropyridazine-3,6-dione</td>
</tr>
<tr>
<td>IIIc</td>
<td>C₂H₅</td>
<td>1,2-dihydropyridazine-3,6-dione</td>
</tr>
<tr>
<td>IVa</td>
<td>H</td>
<td>Tetrahydropyridazine-3,6-dione</td>
</tr>
<tr>
<td>IVb</td>
<td>CH₃</td>
<td>Tetrahydropyridazine-3,6-dione</td>
</tr>
<tr>
<td>IVc</td>
<td>C₂H₅</td>
<td>Tetrahydropyridazine-3,6-dione</td>
</tr>
<tr>
<td>Va</td>
<td>H</td>
<td>2,3-dihydraphthalazine-1,4-dione</td>
</tr>
<tr>
<td>Vb</td>
<td>CH₃</td>
<td>2,3-dihydraphthalazine-1,4-dione</td>
</tr>
<tr>
<td>Vc</td>
<td>C₂H₅</td>
<td>2,3-dihydraphthalazine-1,4-dione</td>
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Physical data of compound. No. IIIa-Vc were summarized in Table No.2:

**Table No.2**

<table>
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<tr>
<th>COMPOUND</th>
<th>R&lt;sub&gt;2&lt;/sub&gt;</th>
<th>Molecular Formula</th>
<th>MP(°C)</th>
<th>Yield(%)</th>
<th>Rf value</th>
</tr>
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<tbody>
<tr>
<td>IIIa</td>
<td>1,2-dihydropyridazine-3,6-dione</td>
<td>C&lt;sub&gt;12&lt;/sub&gt;H&lt;sub&gt;7&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>185-187</td>
<td>85%</td>
<td>0.63</td>
</tr>
<tr>
<td>IIIb</td>
<td>1,2-dihydropyridazine-3,6-dione</td>
<td>C&lt;sub&gt;13&lt;/sub&gt;H&lt;sub&gt;9&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>190-192</td>
<td>84%</td>
<td>0.67</td>
</tr>
<tr>
<td>IIIc</td>
<td>1,2-dihydropyridazine-3,6-dione</td>
<td>C&lt;sub&gt;14&lt;/sub&gt;H&lt;sub&gt;11&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>198-200</td>
<td>80%</td>
<td>0.65</td>
</tr>
<tr>
<td>IVa</td>
<td>Tetrahydropyridazine-3,6-dione</td>
<td>C&lt;sub&gt;12&lt;/sub&gt;H&lt;sub&gt;9&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>170-172</td>
<td>78%</td>
<td>0.53</td>
</tr>
<tr>
<td>IVb</td>
<td>Tetrahydropyridazine-3,6-dione</td>
<td>C&lt;sub&gt;13&lt;/sub&gt;H&lt;sub&gt;11&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>205-207</td>
<td>80%</td>
<td>0.55</td>
</tr>
<tr>
<td>IVc</td>
<td>Tetrahydropyridazine-3,6-dione</td>
<td>C&lt;sub&gt;14&lt;/sub&gt;H&lt;sub&gt;13&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>223-225</td>
<td>75%</td>
<td>0.55</td>
</tr>
<tr>
<td>Va</td>
<td>2,3-dihydrophthalazine-1,4-dione</td>
<td>C&lt;sub&gt;10&lt;/sub&gt;H&lt;sub&gt;10&lt;/sub&gt;N&lt;sub&gt;4&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>170-172</td>
<td>82%</td>
<td>0.61</td>
</tr>
<tr>
<td>Vb</td>
<td>2,3-dihydrophthalazine-1,4-dione</td>
<td>C&lt;sub&gt;11&lt;/sub&gt;H&lt;sub&gt;12&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>205-207</td>
<td>86%</td>
<td>0.66</td>
</tr>
<tr>
<td>Vc</td>
<td>2,3-dihydrophthalazine-1,4-dione</td>
<td>C&lt;sub&gt;12&lt;/sub&gt;H&lt;sub&gt;14&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>234-236</td>
<td>84%</td>
<td>0.64</td>
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Elemental analysis of compound. No. IIIa-Vc were summarized in Table No.3:

**Table No.3**

<table>
<thead>
<tr>
<th>compound</th>
<th>R&lt;sub&gt;2&lt;/sub&gt;</th>
<th>Molecular Formula</th>
<th>Elemental Analysis (%)</th>
<th>Elemental Analysis (%)</th>
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<tr>
<td></td>
<td></td>
<td></td>
<td>Calculated</td>
<td>Found</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>H</td>
</tr>
<tr>
<td>IIIa</td>
<td>1,2-dihydropyridazine-3,6-dione</td>
<td>C&lt;sub&gt;12&lt;/sub&gt;H&lt;sub&gt;7&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>56.04</td>
<td>2.74</td>
</tr>
<tr>
<td>IIIb</td>
<td>1,2-dihydropyridazine-3,6-dione</td>
<td>C&lt;sub&gt;13&lt;/sub&gt;H&lt;sub&gt;9&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>57.57</td>
<td>3.34</td>
</tr>
<tr>
<td>IIIc</td>
<td>1,2-dihydropyridazine-3,6-dione</td>
<td>C&lt;sub&gt;14&lt;/sub&gt;H&lt;sub&gt;11&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>58.95</td>
<td>3.89</td>
</tr>
<tr>
<td>IVa</td>
<td>Tetrahydropyridazine-3,6-dione</td>
<td>C&lt;sub&gt;12&lt;/sub&gt;H&lt;sub&gt;9&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>55.60</td>
<td>3.50</td>
</tr>
<tr>
<td>IVb</td>
<td>Tetrahydropyridazine-3,6-dione</td>
<td>C&lt;sub&gt;13&lt;/sub&gt;H&lt;sub&gt;11&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>57.14</td>
<td>4.06</td>
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<td>IVc</td>
<td>Tetrahydropyridazine-3,6-dione</td>
<td>C&lt;sub&gt;14&lt;/sub&gt;H&lt;sub&gt;13&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>58.53</td>
<td>4.56</td>
</tr>
<tr>
<td>Va</td>
<td>2,3-dihydrophthalazine-1,4-dione</td>
<td>C&lt;sub&gt;10&lt;/sub&gt;H&lt;sub&gt;10&lt;/sub&gt;N&lt;sub&gt;4&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>62.54</td>
<td>2.95</td>
</tr>
<tr>
<td>Vb</td>
<td>2,3-dihydrophthalazine-1,4-dione</td>
<td>C&lt;sub&gt;11&lt;/sub&gt;H&lt;sub&gt;12&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>63.55</td>
<td>3.45</td>
</tr>
<tr>
<td>Vc</td>
<td>2,3-dihydrophthalazine-1,4-dione</td>
<td>C&lt;sub&gt;12&lt;/sub&gt;H&lt;sub&gt;14&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>64.47</td>
<td>3.91</td>
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</table>

CONCLUSION

In the present investigation (III-Vc) compounds were synthesized. All the above compounds (IIIa-Vc) were synthesized from 2-substituted benzoxazole-5-carboxylic acid hydrazides with corresponding anhydride and using the solvent acetic acid at reflux over a period of 4-8hrs to obtain the 1-[2-substituted(1,3-benzoxazol-5-yl)carbonyl] tetrahydropyridazine-3,6-dione, 1-[2-substituted(1,3-benzoxazol-5-yl)carbonyl]-1,2-dihydropyridazine-3,6-dione and 2-[2-substituted (1,3-benzoxazol-5-yl)carbonyl]-2,3-dihydrophthalazine-1,4-dione.

The purity and homogeneity of all the synthesized compounds were confirmed by their sharp melting points (uncorrected), thin-layer chromatography.

The chemical structures were confirmed by infra-red absorption spectra of all the synthesized compounds. The aromatic Ar–H stretching for all the derivatives was found to be at the range of 3000-3200 cm<sup>-1</sup>. The presence of N-H stretching was confirmed by the peaks at the range 3290-3390 cm<sup>-1</sup>. Also some 1H-NMR spectra’s were useful for some protons in the compounds such as δ 7.30-8.0 indicates the presence of phenyl ring protons, And mass spectrum of the compounds gives mass of compounds.

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REFERENCES