

**Supplementary Information:**

**Synthesis of functionalized dihydrofurocoumarin derivatives from 3-aminoalkyl-4-hydroxycoumarin**

**Pallabi Borah\*, P. Seetham Naidu and Pulak J. Bhuyan\***

*Medicinal Chemistry Division, CSIR-North East Institute of Science & Technology,*

*Jorhat 785006, Assam, India*

**Experimental details, Melting points, IR, <sup>1</sup>H NMR (300 MHz), <sup>13</sup>C NMR, MS and analytical data and of compounds 7 and 9. Copies of <sup>1</sup>H NMR and <sup>13</sup>C NMR of 7a, 7b, 7c, 9a.**

**Experimental:**

**Representative procedure for the synthesis of compound 7:**

A mixture of 4-hydroxy-3-(phenyl-pyrrolidin-1-yl-methyl)coumarin **6a** (321 mg, 1 mmol), pyridinium salt **3a** (278 mg, 1 mmol) and DBU (152 mg, 1 mmol) in acetonitrile (10 ml) under N<sub>2</sub> atmosphere was refluxed for 3h. After completion of the reaction (monitored by TLC), the solvent was removed under reduced pressure. The product was purified by silica gel column chromatography by using 8:2 PE and ethylacetate as eluent. The product was ascertained as **7a** from various spectroscopic data. Similarly other compounds **7b-i** were synthesized and characterized.

**Compound 7a:** Yield = 298 mg (81%); mp. 167 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1711, 1745, 2868 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  4.72 (d, *J* = 4.86 Hz, 1H), 6.10 (d, *J* = 6.96 Hz, 1H), 7.19-7.96 (m, 14H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  38.17, 81.67, 93.49, 121.31, 125.24, 125.91, 126.32, 127.49 (2C), 127.81, 128.10, 128.76 (2C), 128.84 (4C), 132.66, 138.69, 140.22, 146.69, 161.91, 167.23, 186.43; MS (m/z): 369.3 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>24</sub>H<sub>17</sub>O<sub>4</sub>: C, 78.25; H, 4.38%. Found: C, 78.21; H, 4.29%.

**Compound 7b:** Yield = 225 mg (78%); mp. 159 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1746.3, 2151, 2859 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  4.56 (d,  $J$  = 4.77 Hz, 1H), 6.23 (d,  $J$  = 6.84 Hz, 1H), 7.19-7.77 (m, 9H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  39.67, 62.47, 92.31, 117.23, 121.43, 125.27, 125.69, 126.33, 127.79 (2C), 127.83, 128.11, 128.47 (2C), 140.37, 151.43, 160.29, 165.33; MS (m/z): 290.4 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>18</sub>H<sub>12</sub>NO<sub>3</sub>: C, 74.73; H, 3.83; N, 4.84%. Found: C, 74.13; H, 3.67; N, 4.61%.

**Compound 7c:** Yield = 268 mg (80%); mp. 117 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1729, 1746.3, 2842.1 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  1.46 (t,  $J$  = 4.55 Hz, 3H), 4.10 (q,  $J$  = 5.31 Hz, 2H), 4.49 (d,  $J$  = 4.14 Hz, 1H), 6.09 (d,  $J$  = 6.33 Hz, 1H), 7.21-7.81 (m, 9H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  12.37, 36.82, 58.61, 75.24, 93.29, 121.39, 125.47, 125.77, 126.46, 127.64, 127.81 (2C), 128.22, 128.63 (2C), 141.36, 149.67, 161.85, 165.89, 171.93; MS (m/z): 337.4 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>20</sub>H<sub>17</sub>O<sub>5</sub>: C, 71.42; H, 4.79%. Found: C, 71.28; H, 4.56%.

**Compound 7d:** Yield = 330 mg (82%); mp. 179 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1710.5, 1743.6, 2869 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  4.31 (d,  $J$  = 4.03 Hz, 1H), 6.01 (d,  $J$  = 6.23 Hz, 1H), 7.14-7.78 (m, 13H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  37.47, 82.19, 93.69, 121.77, 125.37, 126.71, 127.44, 128.19, 128.46 (2C), 128.69 (2C), 128.89 (2C), 129.47 (2C), 131.09, 132.17, 136.39, 138.86, 149.97, 160.08, 165.77, 186.50; MS (m/z): 403.7 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>24</sub>H<sub>16</sub>ClO<sub>4</sub>: C, 71.56; H, 3.75%. Found: C, 71.33; H, 3.60%.

**Compound 7e:** Yield = 255 mg (79%); mp. 171 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1743.2, 2150.7, 2869.5 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  4.24 (d,  $J$  = 4.45 Hz, 1H), 6.13 (d,  $J$  = 6.75 Hz, 1H), 7.23-7.75 (m, 8H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  37.56, 61.88, 92.93, 117.31, 121.45, 125.61, 126.81, 127.90, 128.15, 128.89 (2C), 129.48 (2C), 131.42, 137.51, 149.96, 161.07, 165.35; MS (m/z): 324.8 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>18</sub>H<sub>11</sub>ClNO<sub>3</sub>: C, 66.78; H, 3.11; N, 4.33%. Found: C, 66.56; H, 3.02; N, 4.21%.

**Compound 7f:** Yield = 300 mg (81%); mp. 136 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1730.3, 1744.5, 2877 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  1.39 (t,  $J$  = 4.24 Hz, 3H), 3.98 (q,  $J$  = 5.47 Hz, 2H), 4.39 (d,  $J$  = 4.08 Hz, 1H), 6.10 (d,  $J$  = 6.57 Hz, 1H), 7.21-7.73 (m, 8H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  12.41, 38.22, 58.92, 76.01, 92.49, 121.37, 125.36, 126.79, 127.18, 128.26, 128.79 (2C), 129.14 (2C), 131.29, 137.46, 149.88, 160.78, 165.24, 171.98; MS

(m/z): 371.2 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>20</sub>H<sub>16</sub>ClO<sub>5</sub>: C, 64.79; H, 4.08%. Found: C, 64.48; H, 4.01%.

**Compound 7g:** Yield = 305 mg (80%); mp. 171 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1713, 1746.6, 2878 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  2.39 (s, 3H), 4.36 (d,  $J$  = 4.75 Hz, 1H), 6.09 (d,  $J$  = 6.46 Hz, 1H), 7.14-7.81 (m, 13H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  21.71, 38.23, 82.45, 93.45, 121.33, 125.17, 126.16, 127.41, 127.87 (2C), 128.17, 128.59 (2C), 128.67 (2C), 129.19 (2C), 131.49, 133.36, 136.28, 137.81, 149.62, 161.87, 166.47, 186.31; MS (m/z): 383.5 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>25</sub>H<sub>19</sub>O<sub>4</sub>: C, 78.52; H, 4.74%. Found: C, 78.37; H, 4.37%.

**Compound 7h:** Yield = 233 mg (77%); mp. 163 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1746.3, 2149.9, 2866 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  2.31 (s, 3H), 4.32 (d,  $J$  = 4.13 Hz, 1H), 6.12 (d,  $J$  = 6.28 Hz, 1H), 7.21-7.73 (m, 8H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  21.23, 38.63, 60.23, 92.61, 117.39, 121.43, 125.30, 126.35, 127.39, 127.81 (2C), 128.16, 129.45 (2C), 133.69, 136.92, 149.59, 161.87, 166.36; MS (m/z): 304.7 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>19</sub>H<sub>14</sub>NO<sub>3</sub>: C, 75.24; H, 4.32; N, 4.62%. Found: C, 75.17; H, 4.29; N, 4.40%.

**Compound 7i:** Yield = 273 mg (78%); mp. 125 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1728.5, 1746.7, 2891 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  1.37 (t,  $J$  = 4.55 Hz, 3H), 2.35 (s, 3H), 4.11 (q,  $J$  = 5.65 Hz, 2H), 4.67 (d,  $J$  = 4.88 Hz, 1H), 6.17 (d,  $J$  = 6.96 Hz, 1H), 7.20-7.79 (m, 8H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  13.01, 21.67, 38.64, 58.46, 78.21, 93.28, 121.43, 125.20, 126.61, 127.41, 127.89 (2C), 128.17, 129.55 (2C), 134.22, 136.69, 149.91, 161.17, 166.02, 176.47; MS (m/z): 351.2 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>21</sub>H<sub>19</sub>O<sub>5</sub>: C, 71.99; H, 5.18%. Found: C, 71.59; H, 5.10%.

### **Representative procedure for the synthesis of compound 9:**

A mixture of 4-hydroxycoumarin **1a** (162 mg, 1 mmol), salicaldehyde **8a** (122 mg, 1 mmol) and pyrrolidine **5a** (70 mg, 1 mmol) was taken in a round-bottomed flask containing ethanol. Then the reaction mixture was stirred vigorously at room temperature for 3h. After completion of the reaction (monitored by TLC), the solid compound was filtered off and crude products were recrystallized from ethanol. The product **9a** was obtained in 73% yield. The structure of the compound was ascertained from the

spectroscopic data and elemental analysis. Similarly, other compounds **9b-1** were synthesized and characterized.

**Compound 9a:** Yield = 240 mg (73%); mp. 209 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1221, 1745.1, 2884.3 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  2.12 (br s, 4H), 2.93 (br s, 1H), 3.24 (br s, 1H), 3.70 (br s, 1H), 3.96 (br s, 1H), 5.31 (s, 1H), 7.26-8.08 (m, 8H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  22.79 (2C), 45.63 (2C), 50.93, 97.31, 119.38, 121.47, 122.32, 124.68, 125.21, 126.37, 127.73, 127.91, 128.11, 129.35, 149.39, 153.63, 160.09, 164.69; MS (m/z): 320.7 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>20</sub>H<sub>18</sub>NO<sub>3</sub>: C, 75.22; H, 5.37; N, 4.39%. Found: C, 75.09; H, 5.18; N, 4.31%.

**Compound 9b:** Yield = 253 mg (76%); mp. 213 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1220.9, 1747, 2897 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  1.63-1.79 (m, 6H), 2.99 (br s, 4H), 5.43 (s, 1H), 6.81-7.62 (m, 8H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  23.31, 25.72 (2C), 48.89, 51.17 (2C), 98.25, 119.37, 121.23, 122.48, 123.71, 125.67, 126.62, 127.09, 127.81, 128.13, 129.54, 148.66, 154.22, 160.01, 163.29; MS (m/z): 334.2 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>21</sub>H<sub>20</sub>NO<sub>3</sub>: C, 75.66; H, 5.74; N, 4.20%. Found: C, 75.43; H, 5.39; N, 4.11%.

**Compound 9c:** Yield = 298 mg (75%); mp. 215 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1222, 1745.4, 2864 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  2.29 (br s, 4H), 2.91 (br s, 1H), 3.20 (br s, 1H), 3.66 (br s, 1H), 3.93 (br s, 1H), 5.37 (s, 1H), 6.82-7.79 (m, 7H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  23.71 (2C), 46.37 (2C), 45.98, 98.21, 121.03, 121.97, 122.67, 123.81, 125.66, 125.91, 126.87, 127.89, 128.21, 132.49, 149.61, 158.38, 161.31, 164.29; MS (m/z): 399.2 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>20</sub>H<sub>17</sub>BrNO<sub>3</sub>: C, 60.32; H, 4.05; N, 3.52%. Found: C, 60.23; H, 4.01; N, 3.45%.

**Compound 9d:** Yield = 325 mg (79%); mp. 217 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1220.5, 1745.7, 2879.1 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  1.79-1.89 (m, 6H), 3.01 (br s, 4H), 5.41 (s, 1H), 6.91-7.69 (m, 7H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  24.71, 25.32 (2C), 48.96, 50.92 (2C), 97.31, 121.45, 121.97, 122.43, 123.75, 125.22, 125.79, 126.51, 127.23, 128.33, 132.19, 148.69, 158.45, 161.27, 164.25; MS (m/z): 413.6 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>21</sub>H<sub>19</sub>BrNO<sub>3</sub>: C, 61.18; H, 4.40; N, 3.40%. Found: C, 61.04; H, 4.32; N, 3.31%.

**Compound 9e:** Yield = 246 mg (74%); mp. 203.7 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1220.8, 1745.3, 2890 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  2.13 (br s, 4H), 2.37 (s, 3H), 2.97 (br s, 1H), 3.17 (br s, 1H), 3.68 (br s, 1H), 3.91 (br s, 1H), 5.27 (s, 1H), 6.66-7.46 (m, 7H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  21.36, 24.62 (2C), 47.23 (2C), 49.37, 96.21, 119.38, 121.22, 122.08, 123.46, 125.16, 126.69, 127.58, 128.27, 129.61, 135.24, 148.35, 156.21, 160.55, 164.41; MS (m/z): 334.2 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>21</sub>H<sub>20</sub>NO<sub>3</sub>: C, 75.66; H, 5.74; N, 4.20%. Found: C, 75.43; H, 5.59; N, 4.13%.

**Compound 9f:** Yield = 267 mg (77%); mp. 207 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1221.3, 1746.2, 2881.4 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  1.69-1.87 (m, 6H), 2.36 (s, 3H), 3.11 (br s, 4H), 5.31 (s, 1H), 6.97-7.77 (m, 7H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  21.72, 25.72 (2C), 26.01, 49.88 (2C), 50.07, 96.41, 119.44, 121.27, 121.89, 122.71, 125.69, 126.16, 127.47, 128.21, 129.45, 136.87, 149.11, 156.82, 160.18, 163.51; MS (m/z): 348.4 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>22</sub>H<sub>22</sub>NO<sub>3</sub>: C, 76.06; H, 6.09; N, 4.03%. Found: C, 76.00; H, 6.02; N, 3.99%.

**Compound 9g:** Yield = 243 mg (73%); mp. 203 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1225, 1748.2, 2896.1 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  2.23 (br s, 4H), 2.35 (s, 3H), 2.97 (br s, 1H), 3.17 (br s, 1H), 3.59 (br s, 1H), 3.88 (br s, 1H), 5.39 (s, 1H), 6.89-7.81 (m, 7H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  21.55, 24.66 (2C), 48.39, 49.35 (2C), 98.21, 119.82, 121.44, 122.36, 123.62, 127.17, 127.57, 127.93, 128.91, 129.49, 133.67, 144.11, 156.23, 160.05, 163.19; MS (m/z): 334.5 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>21</sub>H<sub>20</sub>NO<sub>3</sub>: C, 75.66; H, 5.74; N, 4.20%. Found: C, 75.41; H, 5.61; N, 4.07%.

**Compound 9h:** Yield = 264 mg (76%); mp. 207 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1220.8, 1746.4, 2889.6 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  1.70-1.84 (m, 6H), 2.35 (s, 3H), 3.07 (br s, 4H), 5.46 (s, 1H), 6.65-7.51 (m, 7H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  21.43, 25.91, 26.02 (2C), 48.61, 50.02 (2C), 98.61, 118.21, 121.41, 122.31, 124.81, 127.27, 127.46, 127.79, 128.95, 129.73, 134.40, 146.27, 156.03, 160.27, 164.19; MS (m/z): 348.2 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>22</sub>H<sub>22</sub>NO<sub>3</sub>: C, 76.06; H, 6.09; N, 4.03%. Found: C, 75.98; H, 6.01; N, 3.97%.

**Compound 9i:** Yield = 300 mg (73%); mp. 207 °C; IR (CHCl<sub>3</sub>)  $\nu_{\max}$  = 1221.7, 1747.4, 2885.3 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  2.09 (br s, 4H), 2.38 (s, 3H), 2.91 (br s, 1H), 3.11 (br s, 1H), 3.69 (br s, 1H), 3.96 (br s, 1H), 5.12 (s, 1H), 7.25-7.69 (m, 6H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>):  $\delta$  21.31, 24.17 (2C), 48.61 (2C), 49.03, 98.20, 120.07, 121.11, 122.45,

123.69, 125.51, 127.27, 127.75, 128.87, 131.47, 134.64, 145.83, 158.31, 161.28, 163.43; MS (m/z): 413.2 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>21</sub>H<sub>19</sub>BrNO<sub>3</sub>: C, 61.18; H, 4.40; N, 3.40%. Found: C, 61.05; H, 4.32; N, 3.33%.

**Compound 9j:** Yield = 332 mg (78%); mp. 211 °C; IR (CHCl<sub>3</sub>) ν<sub>max</sub> = 1220.7, 1741.6, 2877.3 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 1.73-1.85 (m, 6H), 2.39 (s, 3H), 3.10 (br s, 4H), 5.31 (s, 1H), 7.25-7.79 (m, 6H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>): δ 21.41, 25.91 (2C), 26.02, 48.88, 49.09 (2C), 98.73, 120.11, 121.23, 122.25, 123.69, 125.68, 127.31, 127.87, 128.44, 131.86, 134.14, 148.02, 159.46, 160.77, 163.87; MS (m/z): 427.1 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>22</sub>H<sub>21</sub>BrNO<sub>3</sub>: C, 61.98; H, 4.73; N, 3.29%. Found: C, 61.56; H, 4.39; N, 3.13%.

**Compound 9k:** Yield = 274 mg (79%); mp. 216 °C; IR (CHCl<sub>3</sub>) ν<sub>max</sub> = 1222, 1745.5, 2887 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 2.17 (br s, 4H), 2.36 (s, 3H), 2.38 (s, 3H), 2.87 (br s, 1H), 3.21 (br s, 1H), 3.71 (br s, 1H), 3.91 (br s, 1H), 5.39 (s, 1H), 7.21-7.77 (m, 6H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>): δ 21.37, 22.40, 24.11 (2C), 48.63 (2C), 49.11, 98.27, 118.73, 121.43, 122.10, 123.40, 127.22, 127.52, 128.78, 129.63, 133.91, 135.76, 148.45, 158.08, 160.20, 163.87; MS (m/z): 348.2 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>22</sub>H<sub>22</sub>NO<sub>3</sub>: C, 76.06; H, 6.09; N, 4.03%. Found: C, 75.59; H, 5.89; N, 3.96%.

**Compound 9l:** Yield = 292 mg (81%); mp. 218 °C; IR (CHCl<sub>3</sub>) ν<sub>max</sub> = 1223.1, 1744.6, 2878.4 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 1.68-1.75 (m, 6H), 2.33 (s, 3H), 2.35 (s, 3H), 3.09 (br s, 4H), 5.11 (s, 1H), 7.11-7.68 (m, 6H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>): δ 21.67, 22.11, 25.93 (2C), 26.07, 48.71, 49.32 (2C), 98.53, 118.30, 121.67, 121.98, 123.10, 127.13, 127.83, 128.68, 129.61, 133.53, 136.06, 146.43, 158.39, 160.29, 163.27; MS (m/z): 362.7 (M+H)<sup>+</sup>; Anal. Calcd for C<sub>23</sub>H<sub>24</sub>NO<sub>3</sub>: C, 76.43; H, 6.41; N, 3.88%. Found: C, 76.27; H, 6.29; N, 3.62%.









