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**3-(1-Phenylpropyl)-5,6,7,8,9,10,11,12-octahydro-4-hydroxy-2H-cyclodeca[b]pyran-2-one (2e)** 0.219 g (64%) of a foam: mp 163-166 °C; <sup>1</sup>H NMR (DMSO-d<sub>6</sub>) δ 10.45 (s, 1 H), 7.30 (d, *J* = 7.0 Hz, 2 H), 7.26-7.21 (m, 2 H), 7.15-7.10 (m, 1 H), 4.14 (dd, *J* = 6.1, 9.7 Hz, 1 H), 2.63-2.59 (m, 4 H), 2.24-2.18 (m, 1 H), 2.05-1.98 (m, 1 H), 1.79-1.56 (m, 4 H), 1.47-1.33 (m, 4 H), 1.23-1.13 (m, 4 H), 0.81 (t, *J* = 7.3 Hz, 3 H) ppm; <sup>13</sup>C NMR (DMSO) δ 165.0, 162.5, 159.0, 143.7, 127.6, 127.5, 125.5, 110.5, 104.3, 40.7, 28.1, 26.5, 25.3, 24.9, 24.7, 23.2, 22.1, 20.6, 20.2, 12.4 ppm; MS (EI) *m/z* 340 (M<sup>+</sup>). Anal. (C<sub>22</sub>H<sub>28</sub>O<sub>3</sub>) C, H.

**[3-[1-(5,6,7,8-Tetrahydro-4-hydroxy-2-oxo-2H-1-benzopyran-3-yl)propyl]phenyl]-carbamic acid, phenylmethyl ester (4b)** 0.231 g (12%) of yellow solid: mp 88-91 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 7.39-7.29 (m, 8 H), 7.13 (d, *J* = 7.6 Hz, 1 H), 6.73 (s, 1 H), 5.19 (s, 2 H), 4.28 (s, 1 H), 2.49-2.45 (m, 2 H), 2.24-2.22 (m, 2 H), 2.16-2.04 (m, 2 H), 1.80-1.65 (m, 4 H), 1.00 (t, *J* = 7.3 Hz, 3 H) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ 165.2, 164.0, 158.6, 153.5, 143.3, 138.6, 136.1, 130.0, 128.6, 128.4, 128.3, 122.6, 117.8, 117.4, 108.4, 105.9, 67.1, 41.4, 27.2, 24.0, 21.6, 21.5, 20.3, 12.4 ppm; MS (EI) *m/z* 433 (M<sup>+</sup>). Anal. (C<sub>26</sub>H<sub>27</sub>NO<sub>5</sub>) C, H, N.

**[3-[1-(6,7,8,9-Tetrahydro-4-hydroxy-2-oxo-2(5H)-cyclohepta[b]pyran-3-yl)propyl]phenyl]-carbamic acid, phenylmethyl ester (4c)** 0.42 g (18%) of beige solid: mp 75-78 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 7.40-7.28 (m, 7 H), 7.12 (d, *J* = 7.6 Hz, 1 H), 6.74 (s, 1 H), 6.74 (br s, 1 H), 5.19 (s, 2 H), 4.31 (t, *J* = 7.8 Hz, 1 H), 2.69-2.65 (m, 2 H), 2.42-

2.34 (m, 2 H), 2.19-2.10 (m, 1 H), 2.05-1.96 (m, 1 H), 1.76-1.73 (m, 2 H), 1.68-1.61 (m, 2 H), 1.56-1.49 (m, 2 H), 1.00 (t,  $J = 7.3$  Hz, 3 H) ppm;  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$  165.1, 164.9, 163.9, 153.2, 143.0, 138.8, 135.9, 130.2, 128.6, 128.4, 128.3, 122.5, 117.8, 117.6, 113.2, 105.5, 67.1, 41.4, 33.9, 31.4, 26.2, 24.4, 24.0, 22.2, 12.4 ppm; MS (EI)  $m/z$  447 ( $\text{M}^+$ ). Anal. ( $\text{C}_{27}\text{H}_{29}\text{NO}_5$ ) C, H, N.

**[3-[1-(5,6,7,8,9,10-Hexahydro-4-hydroxy-2-oxo-2H-cycloocta[b]pyran-3-yl)propyl]phenyl]-carbamic acid, phenylmethyl ester (4d)** 0.772 g (26%) of yellow solid: mp 78-83 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  7.38-7.20 (m, 8 H), 7.12 (d,  $J = 7.5$  Hz, 1 H), 6.71 (s, 1 H), 5.90 (br s, 1 H), 5.20 (s, 2 H), 4.31 (t,  $J = 7.8$  Hz, 1 H), 2.62-2.58 (m, 2 H), 2.43-2.33 (m, 3 H), 2.17-2.11 (m, 1 H), 2.07-2.00 (m, 1 H), 1.74-1.65 (m, 3 H), 1.58-1.25 (m, 6 H), 1.00 (t,  $J = 7.2$  Hz, 3 H) ppm;  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$  165.4, 163.8, 161.3, 142.9, 138.6, 135.8, 130.0, 128.7, 128.5, 128.3, 128.2, 122.5, 117.6, 117.4, 110.4, 105.9, 67.0, 41.2, 30.6, 29.0, 28.8, 26.1, 25.7, 23.9, 22.0, 12.2 ppm; MS (EI)  $m/z$  461 ( $\text{M}^+$ ).  
Anal. ( $\text{C}_{28}\text{H}_{31}\text{NO}_5$ ) C, H, N.

**[3-[1-(5,6,7,8,9,10,11,12-Octahydro-4-hydroxy-2-oxo-2H-cyclodeca[b]pyran-3-yl)propyl]phenyl]-carbamic acid, phenylmethyl ester (4e)** 0.286 g (15%) of yellow solid: mp 92-95 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  7.42-7.28 (m, 8 H), 7.12 (d,  $J = 7.3$  Hz, 1 H), 6.72 (s, 1 H), 5.87 (s, 1 H), 5.20 (s, 2 H), 4.33 (t,  $J = 7.8$  Hz, 1 H), 2.74-2.65 (m, 2 H), 2.50-2.45 (m, 2 H), 2.18-1.98 (m, 2 H), 1.90-1.78 (m, 2 H), 1.62-1.18 (m, 10 H), 1.01 (t,  $J = 7.3$  Hz, 3 H) ppm;  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$  165.4, 164.6, 160.5, 153.5, 142.9, 138.8,

135.9, 130.1, 128.6, 128.4, 128.3, 122.6, 117.6, 117.5, 111.0, 106.0, 67.1, 41.1, 28.9, 27.0, 25.8, 25.4, 25.3, 24.0, 22.7, 21.1, 20.7, 12.2 ppm; MS (EI)  $m/z$  489 ( $M^+$ ). Anal.

( $C_{30}H_{35}NO_5$ ) C, H, N.

**4-Cyano-N-[3-[1-(5,6,7,8-tetrahydro-4-hydroxy-2-oxo-2H-1-benzopyran-3-yl)propyl]phenyl]-2-pyridinesulfonamide (5b)** 0.080 g (56%) of white foam: mp 118-123 °C;  $^1H$  NMR ( $CDCl_3$ )  $\delta$  8.93 (s, 1 H), 8.11 (dd,  $J = 1.7, 8.2$  Hz, 1 H), 8.02 (d,  $J = 8.0$  Hz, 1 H), 7.34 (br s, 1 H), 7.24-7.16 (m, 3 H), 7.08-7.05 (m, 1 H), 4.14 (t,  $J = 7.9$  Hz, 1 H), 2.50-2.47 (m, 2 H), 2.31-2.25 (m, 2 H), 2.10-2.00 (m, 2 H), 1.80-1.70 (m, 4 H), 0.91 (t,  $J = 7.3$  Hz, 3 H) ppm;  $^{13}C$  NMR ( $CDCl_3$ )  $\delta$  163.6, 159.0, 152.5, 144.4, 141.6, 140.7, 138.0, 135.7, 129.9, 125.9, 122.9, 121.6, 120.8, 115.1, 113.0, 107.8, 105.3, 41.5, 27.2, 24.0, 21.5, 21.4, 20.3, 12.5 ppm; MS (EI)  $m/z$  465 ( $M^+$ ); HRMS (EI) calcd for  $C_{24}H_{23}N_3O_5S$  465.1358, found 465.1348.

**4-Cyano-[3-[1-(6,7,8,9-tetrahydro-4-hydroxy-2-oxo-2(5H)-cyclohepta[b]pyran-3-yl)propyl]phenyl]-2-pyridinesulfonamide (5c)** 0.178 g (55%) of white foam: mp 121-132 °C;  $^1H$  NMR ( $CDCl_3$ )  $\delta$  8.97-8.92 (m, 1 H), 8.11 (dd,  $J = 2.1, 8.1$  Hz, 1 H), 8.03 (d,  $J = 8.5$  Hz, 1 H), 7.25-7.16 (m, 3 H), 7.11-7.08 (m, 1 H), 5.97 (br s, 1 H), 4.18 (t,  $J = 7.8$  Hz, 1 H), 2.71-2.67 (m, 2 H), 2.45-2.42 (m, 2 H), 2.13-1.98 (m, 2 H), 1.85-1.54 (m, 6 H), 0.93 (t,  $J = 7.3$  Hz, 3 H) ppm;  $^{13}C$  NMR ( $CDCl_3$ )  $\delta$  164.3, 163.7, 152.4, 144.0, 141.8, 141.7, 136.0, 130.1, 129.8, 127.0, 125.7, 122.9, 121.3, 120.9, 115.1, 112.7, 104.9, 41.5, 33.9, 31.3, 26.2, 24.4, 23.9, 22.5, 12.4 ppm; MS (EI)  $m/z$  479 ( $M^+$ ). Anal.

(C<sub>25</sub>H<sub>25</sub>N<sub>3</sub>O<sub>5</sub>S) C, H, N.

**4-Cyano-[3-[1-(5,6,7,8,9,10-hexahydro-4-hydroxy-2-oxo-2H-cycloocta[b]pyran-3-yl)propyl]phenyl]-2-pyridinesulfonamide (5d)** 0.251 g (56%) of white solid: mp 214-217 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 8.86 (d, *J* = 2.0 Hz, 1 H), 8.07 (dd, *J* = 2.0, 8.1 Hz, 1 H), 7.96 (d, *J* = 8.1 Hz, 1 H), 7.15-7.10 (m, 3 H), 7.02-6.95 (m, 1 H), 4.01 (t, *J* = 7.8 Hz, 1 H), 2.59-2.55 (m, 2 H), 2.49-2.44 (m, 2 H), 2.15-1.97 (m, 2 H), 1.77-1.65 (m, 2 H), 1.65-1.52 (m, 2 H), 1.52-1.32 (m, 4 H), 0.82 (t, *J* = 7.3 Hz, 3 H) ppm; <sup>13</sup>C NMR (DMSO) δ 164.5, 162.8, 160.4, 159.0, 152.9, 145.1, 142.9, 136.2, 128.3, 123.9, 122.3, 120.2, 118.2, 115.8, 112.1, 109.8, 103.8, 40.8, 30.2, 29.1, 28.6, 25.8, 25.3, 23.1, 21.7, 12.4 ppm; MS (EI) *m/z* 493 (M<sup>+</sup>). Anal. (C<sub>26</sub>H<sub>27</sub>N<sub>3</sub>O<sub>5</sub>S) C, H, N.

**4-Cyano-[3-[1-(5,6,7,8,9,10,11,12-octahydro-4-hydroxy-2-oxo-2H-cyclodeca[b]pyran-3-yl)propyl]phenyl]-2-pyridinesulfonamide (5e)** 0.102 g (45%) of white crystals: mp 209-212 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 8.93 (s, 1 H), 8.11 (dd, *J* = 2.0, 8.1 Hz, 1 H), 8.01 (d, *J* = 8.1 Hz, 1 H), 7.23-7.16 (m, 2 H), 7.08-7.02 (m, 2 H), 5.56 (s, 1 H), 4.19 (t, *J* = 7.8 Hz, 1 H), 2.73-2.70 (m, 2 H), 2.53-2.49 (m, 2 H), 2.10-2.02 (m, 2 H), 2.00-1.78 (m, 2 H), 1.60-1.22 (m, 10 H), 0.94 (t, *J* = 7.3 Hz, 3 H) ppm; MS (EI) *m/z* 521 (M<sup>+</sup>). Anal. (C<sub>28</sub>H<sub>31</sub>N<sub>3</sub>O<sub>5</sub>S) C, H, N, S.

**Hexahydro-2H-1-benzopyran-2,4(3H)-dione (6b)** 0.94 g (56%) of white solid: mp 141-144 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 4.84-4.80 (m, 1 H), 3.54 (d, *J* = 19.4 Hz, 1 H), 3.40 (d, *J* =

19.0 Hz, 1 H), 2.60-2.53 (m, 1 H), 2.08-2.02 (m, 1 H), 1.79-1.65 (m, 4 H), 1.62-1.54 (m, 2 H), 1.44-1.40 (m, 1 H) ppm;  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$  203.0, 167.4, 74.3, 47.7, 45.6, 29.1, 23.5, 23.2, 19.7 ppm; MS (EI)  $m/z$  168 ( $\text{M}^+$ ). Anal. ( $\text{C}_9\text{H}_{12}\text{O}_3$ ) C, H.

**Hexahydro-cyclohepta[b]pyran-2,4(3H, 4aH)-dione (6c)** 0.538 g (29%) of white solid: mp 124-127 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  4.97-4.91 (m, 1 H), 3.60 (d,  $J = 18.3$  Hz, 1 H), 3.42 (d,  $J = 18.3$  Hz, 1 H), 2.64-2.58 (m, 1 H), 2.22-2.11 (m, 1 H), 2.01-1.72 (m, 5 H), 1.59-1.36 (m, 4 H) ppm;  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$  203.0, 167.2, 78.0, 52.1, 46.5, 32.1, 28.6, 27.1, 25.7, 21.3 ppm; MS (EI)  $m/z$  182 ( $\text{M}^+$ ). Anal. ( $\text{C}_{10}\text{H}_{14}\text{O}_3$ ) C, H.

**Octahydro-2H-cycloocta[b]pyran-2,4(3H)-dione (6d)** 0.071 g (7%) of white crystals: mp 110-115 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  4.84-4.78 (m, 1 H), 3.61 (d,  $J = 18.9$  Hz, 1 H), 3.40 (d,  $J = 18.9$  Hz, 1 H), 2.75-2.70 (m, 1 H), 2.14-1.97 (m, 2 H), 1.90-1.72 (m, 4 H), 1.68-1.44 (m, 6 H) ppm;  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$  204.2, 167.2, 78.2, 49.5, 46.1, 28.5, 27.3, 26.2, 24.7, 23.9, 22.1 ppm; MS (EI)  $m/z$  196 ( $\text{M}^+$ ). Anal. ( $\text{C}_{11}\text{H}_{16}\text{O}_3$ ) C, H.

**4a,5,6,7,8,8a-Hexahydro-4-hydroxy-3-(1-phenylpropyl)-2H-1-benzopyran-2-one (7b)** 0.179 g (35%) of white crystals: mp 161-164 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  7.40-7.22 (m, 5 H), 6.67 (br s, 0.4 H), 6.20 (br s, 0.6 H), 4.47-4.45 (m, 1H), 4.23 (dd,  $J = 6.6, 9.2$  Hz, 0.6 H), 4.07 (t,  $J = 7.8$  Hz, 0.4 H), 2.19-1.38 (m, 11 H), 0.98 (dt,  $J = 7.2, 7.3$  Hz, 3 H) ppm; MS (EI)  $m/z$  286 ( $\text{M}^+$ ). Anal. ( $\text{C}_{18}\text{H}_{22}\text{O}_3$ ) C, H.

**2,4a,5,6,7,8,9,9a-Octahydro-4-hydroxy-3-(1-phenylpropyl)-cyclohepta[b]pyran-2-one**

(7c) 0.029 g (15%) white solid: mp 152-157 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 7.43-7.29 (m, 3 H), 7.25-7.13 (m, 2 H), 3.49-3.41 (m, 0.5 H), 3.21 (dd, *J* = 3.2, 11.9 Hz, 0.5 H), 2.29-2.21 (m, 1 H), 2.18-2.03 (m, 1 H), 2.03-1.79 (m, 4 H), 1.79-1.54 (m, 5 H), 1.54-1.29 (m, 2 H), 1.29-1.14 (m, 1 H), 0.69 (t, *J* = 7.3 Hz, 3 H) ppm; MS (EI) *m/z* 300 (M<sup>+</sup>); HRMS (EI) calcd for C<sub>19</sub>H<sub>24</sub>O<sub>3</sub> 300.1725, found 300.1735.

**4a,5,6,7,8,9,10,10a-Octahydro-4-hydroxy-3-(1-phenylpropyl)-2H-cyclooctyl[b]pyran-2-one**

(7d) 0.126 g (48%) of white crystals: mp 179-182 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 7.43-7.15 (m, 5 H), 5.78 (s, 1 H), 4.52-4.42 (m, 1 H), 4.31-4.22 (m, 1 H), 2.43-2.33 (m, 1 H), 2.17-1.34 (m, 14 H), 1.06-0.94 (m, 3 H); MS (EI) *m/z* 314 (M<sup>+</sup>). Anal. (C<sub>20</sub>H<sub>26</sub>O<sub>3</sub>) C, H.

**4a,5,6,7,7a-Pentahydro-4-hydroxy-3-[1-(4-nitrophenyl)propyl]-cyclopenta[b]pyran-2-one**

(8a) 0.562 g (31%) of white crystals: mp 156-159 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub> & CD<sub>3</sub>OD) δ 8.26-8.20 (m, 1 H), 8.00-7.94 (m, 1 H), 7.78-7.71 (m, 1 H), 7.37 (t, *J* = 7.9 Hz, 1 H), 4.78-4.73 (m, 1 H), 4.15-4.07 (m, 1 H), 2.58-2.46 (m, 1 H), 2.22-1.62 (m, 8 H), 0.89 (dt, *J* = 2.1, 7.4 Hz, 3 H) ppm; MS (FAB) *m/z* 318 [(M+H)<sup>+</sup>]. Anal. (C<sub>17</sub>H<sub>19</sub>NO<sub>5</sub>) C, H, N.

**4a,5,6,7,8,8a-Hexahydro-4-hydroxy-3-[1-(4-nitrophenyl)propyl]-2H-1-benzopyran-2-one**

(8b) 0.63 g (24%) of yellow solid: mp 86-91 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 8.27-8.21 (m, 1 H), 8.07-8.01 (m, 1 H), 7.78-7.74 (m, 1 H), 7.47-7.40 (m, 1 H), 4.50-4.45 (m, 1 H), 4.15-4.09 (m, 1 H), 2.55-2.43 (m, 1 H), 2.32-2.03 (m, 2 H), 1.99-1.70 (m, 4 H), 1.70-1.45

(m, 4 H), 1.00-0.91 (m, 3 H) ppm.

**2,4a,5,6,7,8,9,9a-Octahydro-4-hydroxy-3-[1-(4-nitrophenyl)propyl]-cyclohepta[b]pyran-2-one (8c)** 2.5 g (60%) of yellow solid: mp 75-78 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 9.48 (s, 0.5 H), 9.32 (s, 0.5 H), 8.23-8.17 (m, 1 H), 8.04-8.00 (m, 1 H), 7.73-7.71 (m, 1 H), 7.44-7.38 (m, 1 H), 4.69-4.52 (m, 1 H), 4.18-4.10 (m, 1 H), 2.44-2.37 (m, 1 H), 2.29-1.94 (m, 2 H), 1.90-1.58 (m, 8 H), 1.56-1.38 (m, 1 H), 1.38-1.16 (m, 1 H), 0.97-0.90 (m, 3 H) ppm; MS (EI) *m/z* 345 (M<sup>+</sup>); HRMS (EI) calcd for C<sub>19</sub>H<sub>23</sub>NO<sub>5</sub> 345.1576, found 345.1590.

**4a,5,6,7,8,9,10,10a-Octahydro-4-hydroxy-3-[1-(4-nitrophenyl)propyl]-2H-cyclooctyl[b]pyran-2-one (8d)** 0.322 g (18%) of orange foam: mp 67-73 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 8.28-8.19 (m, 1 H), 8.13-8.00 (m, 1 H), 7.84-7.72 (m, 1 H), 7.50-7.38 (m, 1 H), 4.85-4.76 (m, 0.5 H), 4.50-4.41 (m, 0.5 H), 4.17-4.07 (m, 0.5 H), 3.89 (t, *J* = 4.4 Hz, 0.5 H), 3.62-3.53 (m, 0.5 H), 2.72-2.63 (m, 0.5 H), 2.57-2.48 (m, 0.5 H), 2.39-2.33 (m, 0.5 H), 2.25-2.06 (m, 1 H), 2.06-1.85 (m, 2 H), 1.85-1.33 (m, 11 H), 0.98-0.80 (m, 3 H) ppm; MS (EI) *m/z* 359 (M<sup>+</sup>); HRMS (FAB) calcd for C<sub>20</sub>H<sub>25</sub>NO<sub>5</sub>+H<sub>1</sub> 360.1811, found 360.1817.

**4-Cyano-N-[3-[1-(4a,5,6,7,8,8a-hexahydro-4-hydroxy-2-oxo-2H-1-benzopyran-3-yl)propyl]phenyl]-2-pyridinesulfonamide (9b)** 0.257 g (66%) of white solid: mp 122-125 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 9.00-8.98 (m, 0.3 H), 8.94-8.93 (m, 0.7 H), 8.14-8.09 (m, 1 H), 8.03-7.99 (m, 1 H), 7.22-7.10 (m, 3 H), 7.04-6.96 (m, 1 H), 6.09 (s, 0.3 H), 5.82 (s, 0.7 H), 4.48-4.43 (m, 1 H), 4.10-4.02 (m, 1 H), 2.22-2.11 (m, 1 H), 2.06-1.93 (m, 2 H),

1.87-1.75 (m, 1 H), 1.70-1.41 (m, 6 H), 1.41-1.19 (m, 2 H), 0.93 (t,  $J = 7.3$  Hz, 3 H) ppm; MS (FAB)  $m/z$  468 [(M+H)<sup>+</sup>]; HRMS (FAB) calcd for C<sub>24</sub>H<sub>25</sub>N<sub>3</sub>O<sub>5</sub>S + H<sub>1</sub> 468.1593, found 468.1611.

**4-Cyano-N-[3-[1-(2,4a,5,6,7,8,9,9a-octahydro-4-hydroxy-2-oxocyclohepta[b]pyran-3-yl)propyl]phenyl]-2-pyridinesulfonamide (9c)** 0.206 g (74%) of white solid: mp 163-166 °C; <sup>1</sup>H NMR (DMSO) δ 10.80 (br s, 1 H), 10.60 (br s, 1 H), 9.20-9.17 (m, 1 H), 8.61-8.53 (m, 1 H), 8.12-8.04 (m, 1 H), 7.20-6.82 (m, 4 H), 4.55-4.30 (m, 1 H), 2.45-2.38 (m, 1 H), 1.94-1.26 (m, 12 H), 0.54-0.44 (m, 3 H) ppm; MS (EI)  $m/z$  481 (M<sup>+</sup>). Anal. (C<sub>25</sub>H<sub>27</sub>N<sub>3</sub>O<sub>5</sub>S) C, H, N.

**4-Cyano-N-[3-[1-(4a,5,6,7,8,9,10,10a-octahydro-4-hydroxy-2-oxo-2H-cyclooctyl[b]pyran-3-yl)propyl]phenyl]-2-pyridinesulfonamide (9d)** 0.137 g (37%) of white crystals: mp 174-178 °C; <sup>1</sup>H NMR (CD<sub>3</sub>OD) δ 8.98 (s, 1 H), 8.31-8.29 (m, 1 H), 7.98 (d,  $J = 8.0$  Hz, 1 H), 7.14-7.13 (m, 1 H), 7.06-7.04 (m, 2 H), 6.94-6.90 (m, 1 H), 4.41-4.37 (m, 1 H), 3.88-3.81 (m, 1 H), 2.58-2.54 (m, 1 H), 2.07-1.45 (m, 14 H), 0.86-0.79 (m, 3 H) ppm; <sup>13</sup>C NMR (CD<sub>3</sub>OD) δ 174.6, 170.7, 160.5, 153.8, 147.8, 143.3, 137.5, 129.4, 126.1, 123.9, 122.8, 120.5, 116.6, 114.0, 78.8, 42.9, 42.4, 29.2, 29.0, 28.2, 25.8, 25.3, 25.0, 22.1, 13.1 ppm; MS (EI)  $m/z$  495 (M<sup>+</sup>). Anal. (C<sub>26</sub>H<sub>29</sub>N<sub>3</sub>O<sub>5</sub>S) C, H, N.

**4a,5,6,7,8,9,10,10a-Octahydro-4-hydroxy-3-(2,2-dimethyl-1-phenylpropyl)-2H-cyclooctyl[b]pyran-2-one (10)<sup>15</sup>** 0.175 g (37%) of white crystals: mp 208-211 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub> & DMSO-d<sub>6</sub>) δ 7.52 (d,  $J = 8.2$  Hz, 2 H), 7.20-7.08 (m, 3 H), 4.36-4.30 (m,

1 H), 4.12 (s, 1 H), 2.59-2.52 (m, 2 H), 1.95-1.36 (m, 12 H), 1.08-1.02 (m, 9 H) ppm; MS (EI)  $m/z$  342 ( $M^+$ ); HRMS (EI) calcd for  $C_{22}H_{30}O_3$  342.2195, found 342.2206.

**4-Cyano-N-[3-[2,2-dimethyl-1-(4a,5,6,7,8,9,10,10a-octahydro-4-hydroxy-2-oxo-2H-cyclooctyl[b]pyran-3-yl)propyl]phenyl]-2-pyridinesulfonamide (11)**<sup>15</sup> 0.071 g (68%) of white crystals: mp 182-185 °C;  $^1H$  NMR ( $CDCl_3$ )  $\delta$  8.92 (s, 1 H), 8.15-8.12 (m, 1 H), 8.05 (d,  $J = 8.1$  Hz, 1 H), 7.52 (s, 1 H), 7.24-7.18 (m, 3 H), 7.03-7.00 (m, 1 H), 5.93 (s, 1 H), 4.46-4.40 (m, 1 H), 4.30 (s, 1 H), 2.42-2.39 (m, 1 H), 2.03-1.93 (m, 1 H), 1.80-1.58 (m, 6 H), 1.48-1.37 (m, 4 H), 1.26-1.15 (m, 1 H), 1.10 (s, 9 H) ppm; MS (FAB)  $m/z$  524 [ $(M+H)^+$ ]. Anal. ( $C_{28}H_{33}N_3O_5S$ ) C, H, N.

**4-Cyano-N-[3-[1-[(4a,5,6,7,8,8a-hexahydro-4-hydroxy-2-oxo-2H-1-benzopyran-3-yl)propyl]phenyl]-benzenesulfonamide (12)** 0.236 g (61%) of white solid: mp 127-130 °C;  $^1H$  NMR ( $CDCl_3$ )  $\delta$  7.94-7.84 (m, 2 H), 7.79-7.69 (m, 2 H), 7.25-6.94 (m, 4 H), 4.47-4.40 (m, 1 H), 3.35-3.32 (m, 1 H), 2.52-2.42 (m, 1 H), 2.28-1.20 (m, 10 H), 0.82-0.73 (m, 3 H) ppm; MS (EI)  $m/z$  466 ( $M^+$ ); HRMS (EI) calcd for  $C_{25}H_{26}N_2O_5S$  466.1562, found 466.1583.