Diastereoselective tandem oxidation/Michael/aldol reaction: Unprecedented formation of dispirocyclopentanebisoxindoles and dispiro[acenaphthylene-1,1'-cyclopentane-3',1''-acenaphthylene]-2,2''diones

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General methods

All the reagents were purchased from Sigma-Aldrich and used without further purification. Pre-coated plates (Merck, silica gel 60 GF254, 0.25 mm) were used for TLC analysis. The $^1$H and $^{13}$C NMR spectra were recorded on Bruker Avance 400 MHz Spectrometer. The DEPT-135 experiments were carried out on Bruker Avance 400 MHz Spectrometer. Mass spectra were recorded under EI/HRMS at 60,000 resolution using Thermo Scientific Exactive mass spectrometer. The $^1$H and $^{13}$C chemical shift values ($\delta$) are given in ppm with reference to TMS as internal standard (zero ppm). Coupling constants are given in Hertz.

General Procedure:

In an oven dried 50-mL round-bottom flask 1.0 mmol 3-phenacyloxindole/phenacylacenaphthylenone, 10 mL ethanol and 15 mol % DIPEA were taken and the contents of the flask were heated under reflux $\sim$80 ºC until the starting materials were consumed. The reaction mixture was allowed to cool to room temperature. The crude product was separated by filtration, washed with 10-15 mL of ethanol to obtain pure white solid that did not require further purification.
2'-benzoyl-1,1''-dibenzyl-5'-hydroxy-5'-phenyl-1,1'',2,2''-tetrahydrodispiro[indole-3,1'-cyclopentane-3',3''-indole]-2,2''-dione (2a): The title compound was prepared according to the general procedure as a white solid in 78 % yield; mp = 248-250 °C; IR (KBr): 3307, 3064, 3025, 2922, 1709, 1686, 1609, 1493, 1470, 1389, 1351, 1301, 1231, 1181, 1100, 1012, 931, 862, 804, 758, 696, 604, 554, 457 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.21 (d, J = 7.4 Hz, 1H), 7.97 (d, J = 6.8 Hz, 1H), 7.37 – 7.21 (m, 9H), 7.19 – 7.06 (m, 10H), 7.01 (t, J = 9.5 Hz, 3H), 6.94 (t, J = 7.7 Hz, 1H), 6.53 (d, J = 7.2 Hz, 2H), 6.40 (d, J = 7.5 Hz, 1H), 6.30 (d, J = 7.8 Hz, 1H), 5.38 (s, 1H), 5.24 (d, J = 15.3 Hz, 1H), 5.17 (d, J = 16.1 Hz, 1H), 4.51 (d, J = 14.9 Hz, 1H), 4.47 (d, J = 16.8 Hz, 1H), 4.26 (d, J = 15.3 Hz, 1H), 2.57 (d, J = 13.9 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 196.46, 183.88, 176.83, 143.79, 137.11, 135.71, 134.88, 132.29, 128.98, 128.67, 128.52, 128.33, 128.04, 127.83, 127.75, 127.65, 127.08, 126.81, 126.39, 126.10, 125.73, 124.26, 121.93, 108.78, 108.52, 84.55, 66.62, 65.29, 54.28, 46.70, 44.53, 43.80. HRMS Calcd for [C₄₆H₃₆N₂O₄]+Na: 703.25728; found: 703.25772.

1,1''-dibenzyl-2''-(4-bromobenzoyl)-5'--(4-bromophenyl)-5'-hydroxy-1,1'',2,2''-tetrahydrodispiro[indole-3,3'-cyclopentane-1',3''-indole]-2,2''-dione (2b): The title compound was prepared according to the general procedure as a white solid in 73 % yield; mp = 234-236 °C; IR (KBr): 3318, 3068, 2933, 1689, 1605, 1489, 1431, 1343, 1231, 1085, 996, 915, 815, 750, 696, 577, 465 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.15 (d, J = 8.1 Hz, 1H), 7.92 (d, J = 7.3 Hz, 1H), 7.37 (dt, J = 11.1, 7.3 Hz, 5H), 7.23 (dd, J = 14.7, 7.8 Hz, 5H), 7.14 (t, J = 7.5 Hz, 1H), 7.10 – 7.05 (m, 1H), 7.05 – 6.99 (m, 6H), 6.99 – 6.93 (m, 3H), 6.60 (t, J = 7.0 Hz, 2H), 6.46 (t, J = 6.1 Hz, 2H), 5.25 (s, 1H), 5.22 (d, J = 16.3 Hz, 1H), 5.08 (d, J = 15.1 Hz, 1H), 4.50 (d, J = 15.3 Hz, 1H), 4.46 (d, J = 15.8 Hz, 1H), 4.42 (d, J = 13.5 Hz, 1H), 2.53 (d, J = 13.9 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 195.28, 183.61, 176.59, 143.71, 141.58, 137.34, 135.61, 135.45, 134.67, 131.07, 130.78, 130.03, 129.13, 128.91, 128.64, 128.38, 128.25, 128.07, 128.00, 127.35, 127.11, 126.99, 126.37, 126.11, 125.74, 124.35, 122.14, 122.04, 108.89, 108.62, 84.01, 66.53, 65.01, 54.08, 46.76, 44.79, 43.91. HRMS (EI) Calcd for [C₄₆H₃₄Br₂N₂O₄]+Na: 859.07830; found: 859.07831.
1,1''-dibenzyl-5'-hydroxy-2'-(4-phenylbenzoyl)-5''-(4-phenylphenyl)-1,1'',2,2''-
tetrahydrodispiro[indole-3,3'-cyclopentane-1',3''-indole]-2,2''-dione (2c): The title compound
was prepared according to the general procedure as a white solid in 70
% yield; mp = 240-242 °C; IR (KBr): 3287, 3060, 2902, 1709, 1686,
1609, 1585, 1482, 1428, 1352, 1274, 1239, 1104, 1096, 1070, 1000,
846, 811, 731, 692, 573, 554, 534, 453 cm⁻¹; ¹H NMR (400 MHz, 
CDCl₃) δ 8.25 (d, J = 7.5 Hz, 1H), 8.02 (d, J = 7.3 Hz, 1H), 7.57 (d, J
= 7.6 Hz, 2H), 7.46 (d, J = 4.0 Hz, 4H), 7.41 (d, J = 7.3 Hz, 1H), 7.35 (d, 
J = 7.6 Hz, 1H), 7.33–7.21 (m, 10H), 7.19 (d, J = 8.3 Hz, 2H), 7.15 – 7.10 (m, 2H), 7.05 (s, 2H),
6.98–6.94 (dd, J = 9.9, 7.4 Hz, 4H), 6.59 (d, J = 7.0 Hz, 2H), 6.43 (d, J = 7.2 Hz, 1H), 6.34 (d, J =
7.7 Hz, 1H), 5.42 (s, 1H), 5.23 (dd, J = 23.1, 15.7 Hz, 2H), 4.56 (d, J = 13.9 Hz, 1H), 4.47 (d, J =
16.1 Hz, 1H), 4.36 (d, J = 15.2 Hz, 1H), 2.61 (d, J = 13.9 Hz, 1H);¹³C NMR (100 MHz, CDCl₃) δ
195.97, 183.96, 176.95, 144.92, 143.92, 141.72, 140.54, 140.28, 139.59, 137.55, 135.80, 135.69,
134.89, 130.49, 128.99 (2C), 128.94 (2C), 128.80, 128.74, 128.51 (2C), 128.39, 128.29, 128.09,
127.83 (2C), 127.67, 127.35, 127.31, 127.13 (2C), 127.04 (2C), 126.84, 126.68, 126.39 (2C),
126.24, 125.82, 124.31, 121.97, 108.88, 108.51, 84.44, 66.75, 65.44, 54.42, 47.04, 44.72, 43.97;
HRMS (EI) Calcd for [C₅₈H₄₄N₂O₄+Na]: 855.31988; found: 855.32002.

2'-benzoyl-1,1''-dibenzyl-5,5''-difluoro-5'-hydroxy-5'-phenyl-1,1'',2,2''-
-tetrahydrodispiro[indole-3,1'-cyclopentane-3',3''-indole]-2,2''-dione (2d): The title compound
was prepared according to the general procedure as a white solid in 71
% yield; mp = 244-246 °C; IR (KBr): 3245, 3056, 2914, 1695, 1635, 1534,
1475, 1421, 1563, 1275, 1225, 1100, 821, 715, 689, 570, 545 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.07 (d, J = 6.8 Hz, 1H), 7.73 (d, J = 6.5 Hz, 1H), 7.31 (m,
8H), 7.16 (m 8H), 7.04 (t, J = 7.6 Hz, 2H), 6.96 (s, 1H), 6.83 (t, J = 7.8 Hz, 1H),
6.65 (t, J = 7.7 Hz, 1H), 6.50 (d, J = 7.0 Hz, 2H), 6.30 (dd, J = 8.2, 4.2 Hz, 1H),
6.22 (dd, J = 8.5, 3.9 Hz, 1H), 5.30 (s, 1H), 5.25 (d, J = 15.4 Hz, 1H), 5.18 (d, J = 16.2 Hz, 1H),
4.47 (d, J = 2.0 Hz, 1H), 4.43 (d, J = 5.2 Hz, 1H), 4.23 (d, J = 15.4 Hz, 1H), 2.57 (d, J = 14.0 Hz,
1H). ¹³C NMR (100 MHz, CDCl₃) δ 196.08, 183.51, 176.56, 161.27, 159.92, 158.86, 157.55,
139.75, 137.78, 137.54, 136.84, 135.30, 134.48, 132.59, 131.91, 129.07, 128.59, 128.20, 128.01,
127.95, 127.87, 127.62, 127.09, 126.97, 126.33, 126.02, 115.30, 115.13, 114.98, 114.89, 114.74,
113.94, 113.69, 109.22, 84.50, 66.99, 65.33, 54.51, 46.56, 44.70, 43.94, 29.70. HRMS (EI) Calcd for [C_{46}H_{34}F_{2}N_{2}O_{4}+Na]: 739.23843; found: 739.23888.

2'-benzoyl-1,1''-dibenzyl-5'-hydroxy-5,5''-dimethyl-5'-phenyl-1,1'',2,2''-tetrahydrodispiro[indole-3,1'-cyclopentane-3',3''-indole]-2,2''-dione (2e): The title compound was prepared according to the general procedure as a white solid in 76 % yield; mp = 255-257 °C; IR (KBr): 3349, 3129, 2929, 2852, 1705, 1682, 1609, 1497, 1381, 1347, 1197, 1004, 811, 758, 696, 554 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.02 (s, 1H), 7.78 (s, 1H), 7.34 – 7.22 (m, 7H), 7.18 – 7.05 (m, 10H), 7.01 (t, J = 7.6 Hz, 2H), 6.92 (d, J = 7.8 Hz, 1H), 6.74 (d, J = 7.8 Hz, 1H), 6.46 (d, J = 7.0 Hz, 2H), 6.29 (d, J = 7.9 Hz, 1H), 5.34 (s, 1H), 5.23 (d, J = 15.0 Hz, 1H), 4.50 (d, J = 13.8 Hz, 1H), 4.41 (d, J = 16.2 Hz, 1H), 4.23 (d, J = 15.3 Hz, 1H), 2.56 (d, J = 13.9 Hz, 1H), 2.38 (s, 3H), 2.30 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 196.59, 183.79, 176.77, 141.47, 139.21, 138.48, 137.32, 135.84, 134.97, 134.00, 132.14, 131.25, 130.43, 129.02, 128.95, 128.53, 128.47, 127.98, 127.78, 127.72, 127.63, 127.54, 127.08, 126.73, 126.53, 126.34, 126.13, 108.43, 108.28, 84.51, 66.60, 65.45, 54.34, 46.74, 44.52, 43.88, 21.32, 21.30. HRMS (EI) Calcd for [C_{48}H_{40}N_{2}O_{4}+Na]: 731.28858; found: 731.28961.

2'-benzoyl-1,1''-dibenzyl-5,5''-dichloro-5'-hydroxy-5'-phenyl-1,1'',2,2''-tetrahydrodispiro[indole-3,1'-cyclopentane-3',3''-indole]-2,2''-dione (2f): The title compound was prepared according to the general procedure as a white solid in 68 % yield; mp = 250-252 °C; IR (KBr): 3310, 3068, 2933, 1697, 1609, 1485, 1431, 1343, 1181, 1081, 992, 915, 815, 750, 700, 619, 580, 554 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.24 (s, 1H), 7.95 (s, 1H), 7.32 (t, J = 8.6 Hz, 3H), 7.29 – 7.23 (m, 3H), 7.22 – 7.15 (m, 7H), 7.10 (t, J = 7.7 Hz, 4H), 7.03 (t, J = 7.5 Hz, 2H), 6.93 (d, J = 8.4 Hz, 1H), 6.90 (s, 1H), 6.47 (d, J = 7.3 Hz, 2H), 6.30 (d, J = 8.3 Hz, 1H), 6.22 (d, J = 8.4 Hz, 1H), 5.28 (s, 1H), 5.24 (d, J = 8.4 Hz, 1H), 5.20 (d, J = 9.2 Hz, 1H), 4.46 (d, J = 4.4 Hz, 1H), 4.42 (d, J = 6.8 Hz, 1H), 4.24 (d, J = 15.3 Hz, 1H), 2.55 (d, J = 14.0 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 196.08, 183.38, 176.36, 142.44, 140.15, 137.75, 136.92, 135.16, 134.35, 132.53, 131.96, 129.79, 129.09, 128.70, 128.60, 128.46, 128.29, 128.26, 128.02, 127.98, 127.91, 127.64, 127.27, 127.08, 126.99, 126.29, 126.05, 109.76, 109.54, 84.48, 66.82, 65.49, 54.30, 46.56, 44.68, 43.95. HRMS (EI) Calcd for [C_{48}H_{40}Cl_{2}N_{2}O_{4}+Na]: 771.17933; found: 771.17953.
1,1''-dibenzyl-2'-[4-bromobenzoyl]-5',5''-dichloro-5'-hydroxy-1,1'',2,2''-tetrahydrodispiro[indole-3,1'-cyclopentane-3',3''-indole]-2,2''-dione (2g) : The title compound was prepared according to the general procedure as a white solid in 70 % yield; mp = 256-258 °C; IR (KBr): 3283, 3064, 2898, 1713, 1686, 1609, 1585, 1482, 1431, 1351, 1274, 1235, 1177, 1096, 1070, 1000, 931, 846, 808, 731, 692, 573, 550, 534, 453 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) δ 8.17 (s, 1H), 7.90 (s, 1H), 7.45 – 7.18 (m, 10H), 7.15 – 6.96 (m, 7H), 6.91 (s, 1H), 6.54 (d, $J$ = 6.3 Hz, 2H), 6.38 (dd, $J$ = 15.9, 8.3 Hz, 2H), 5.26 (d, $J$ = 16.0 Hz, 1H), 5.16 (s, 1H), 5.05 (d, $J$ = 15.2 Hz, 1H), 4.52 (d, $J$ = 15.1 Hz, 1H), 4.42 (d, $J$ = 16.1 Hz, 1H), 4.36 (d, $J$ = 14.0 Hz, 1H), 2.52 (d, $J$ = 13.9 Hz, 1H), 1.25 (s, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 195.00, 183.13, 176.17, 142.36, 140.13, 136.81, 134.91, 134.18, 131.63, 131.29, 130.99, 129.92, 129.30, 128.97, 128.75, 128.51, 128.38, 128.14, 127.97, 127.71, 127.41, 127.31, 127.25, 126.28, 126.08, 122.43, 109.93, 109.65, 83.96, 66.73, 65.21, 54.10, 46.63, 45.00, 44.08. HRMS (EI) Calcd for [C$_{46}$H$_{32}$Br$_2$Cl$_2$N$_2$O$_4$+Na]: 927.00036; found: 927.00075.

2'-benzoyl-5'-hydroxy-1,1''-bis[(3-methylphenyl)methyl]-5'-phenyl-1,1'',2,2''-tetrahydrodispiro[indole-3,1'-cyclopentane-3',3''-indole]-2,2''-dione (2h) : The title compound was prepared according to the general procedure as a white solid in 74 % yield; mp = 249-251 °C; IR (KBr): 3320, 2975, 2860, 1711, 1684, 1601, 1575, 1480, 1421, 1347, 1311, 1245, 1150, 1065, 927, 805, 775, 690, 650, 550, 511 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) δ 8.22 (d, $J$ = 6.8 Hz, 1H), 7.96 (d, $J$ = 6.1 Hz, 1H), 7.38 – 6.91 (m, 21H), 6.85 (s, 1H), 6.42 (d, $J$ = 6.9 Hz, 1H), 6.30 (d, $J$ = 6.8 Hz, 1H), 6.11 (s, 1H), 5.36 (s, 1H), 5.23 (d, $J$ = 15.1 Hz, 1H), 5.06 (d, $J$ = 16.0 Hz, 1H), 4.48 (t, $J$ = 14.4 Hz, 2H), 4.16 (d, $J$ = 15.1 Hz, 1H), 2.57 (d, $J$ = 13.8 Hz, 1H), 2.33 (s, 3H), 2.27 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 196.38, 183.88, 176.87, 143.96, 141.70, 138.75, 138.22, 137.92, 137.13, 135.76, 134.81, 132.23, 130.40, 128.84, 128.64, 128.55, 128.35, 128.31, 127.81, 127.78, 127.72, 127.63, 127.38, 127.07, 127.02, 126.72, 126.05, 125.69, 124.72, 124.21, 123.45, 121.86, 108.84, 108.53, 84.54, 66.63, 65.38, 54.29, 46.67, 44.53, 43.95, 21.45, 21.43. HRMS (EI) Calcd for [C$_{48}$H$_{40}$N$_2$O$_4$+Na]: 731.28858; found: 731.28998.
cyclopentane-3',3''-indole]-2,2''-dione (2i): The title compound was prepared according to the general procedure as a white solid in 64% yield; mp = 238-240 °C; IR (KBr): 3312, 3062, 2925, 1687, 1605, 1481, 1431, 1340, 1221, 1086, 986, 910, 812, 750, 696, 575, 463 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.27 (d, \(J = 2.0\) Hz, 1H), 7.99 (d, \(J = 2.0\) Hz, 1H), 7.61 – 7.54 (m, 2H), 7.50 – 7.38 (m, 9H), 7.35 (t, \(J = 7.3\) Hz, 1H), 7.31 – 7.18 (m, 11H), 7.11 (dd, \(J = 8.3, 2.1\) Hz, 1H), 6.95 (dq, \(J = 9.7, 7.2\) Hz, 5H), 6.53 (d, \(J = 7.1\) Hz, 2H), 6.33 (d, \(J = 8.3\) Hz, 1H), 6.26 (d, \(J = 8.4\) Hz, 1H), 5.32 (s, 1H), 5.29 (d, \(J = 16.3\) Hz, 1H), 5.18 (d, \(J = 15.3\) Hz, 1H), 4.49 (d, \(J = 14.0\) Hz, 1H), 4.44 (d, \(J = 16.2\) Hz, 1H), 4.36 (d, \(J = 15.2\) Hz, 1H), 2.59 (d, \(J = 14.0\) Hz, 1H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 196.14, 184.08, 177.09, 145.06, 140.41, 139.70, 137.65, 135.89, 135.79, 135.00, 130.59, 129.13, 129.07, 128.88, 128.64, 128.53, 128.22, 127.95, 127.80, 127.45, 127.26, 127.17, 126.97, 126.79, 126.50, 126.39, 125.95, 124.45, 122.12, 109.02, 108.65, 84.57, 66.86, 65.56, 54.53, 44.83, 44.08, 29.84.

HRMS (EI) Calcd for [C\(_{58}\)H\(_{42}\)Cl\(_2\)N\(_2\)O\(_4\)+Na]: 923.24193; found: 923.24227.

1,1''-dibenzyl-2'-(4-bromobenzoyl)-5'-(4-bromophenyl)-5'-hydroxy-5,5''-dimethyl-1,1'',2,2''-tetrahydrodispiro[indole-3,1'-cyclopentane-3',3''-indole]-2,2''-dione (2j): The title compound was prepared according to the general procedure as a white solid in 67% yield; mp = 258-260 °C; IR (KBr): 3280, 2964, 2910, 2856, 1709, 1686, 1616, 1585, 1493, 1431, 1351, 1324, 1258, 1204, 1162, 1100, 1070, 1008, 931, 808, 777, 692, 657, 561, 534 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.96 (s, 1H), 7.73 (s, 1H), 7.40 – 7.32 (m, 5H), 7.29 – 7.18 (m, 5H), 7.06 – 6.92 (m, 8H), 6.81 (d, \(J = 7.6\) Hz, 1H), 6.55 (d, \(J = 6.9\) Hz, 2H), 6.33 (d, \(J = 7.9\) Hz, 2H), 5.26 (d, \(J = 16.1\) Hz, 1H), 5.21 (s, 1H), 5.07 (d, \(J = 15.1\) Hz, 1H), 4.48 (d, \(J = 15.1\) Hz, 1H), 4.41 (d, \(J = 13.8\) Hz, 1H), 4.39 (d, \(J = 2.4\) Hz, 1H), 2.51 (d, \(J = 13.9\) Hz, 1H), 2.37 (s, 3H), 2.29 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 195.42, 183.55, 176.53, 141.42, 139.21, 137.59, 135.86, 135.61, 134.80, 134.14, 131.41, 131.04, 130.82, 130.12, 129.27, 129.11, 128.86, 128.61, 128.41, 128.20, 128.05, 127.52, 127.19, 127.04, 126.52, 126.33, 126.19, 122.11, 108.57, 108.40, 84.00, 66.56, 65.16, 54.18, 46.83, 44.79, 44.01, 21.29. HRMS (EI) Calcd for [C\(_{48}\)H\(_{38}\)Br\(_2\)N\(_2\)O\(_4\)+Na]: 887.10960; found: 887.10953.

2'acetyl-1,1''-dibenzyl-4'-hydroxy-4'-methyl-1,1'',2,2''-tetrahydrodispiro[indole-3,3'-cyclopentane-1',3''-indole]-2,2''-dione (2k): The title compound was prepared according to the general procedure as a white solid in
48 % yield; mp = 218-220 °C; IR (KBr): 3335, 3056, 1702, 1676, 1611, 1367, 1183, 805, 772, 690, 647, 506, 537.  \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.15 (d, \(J = 7.1\) Hz, 1H), 7.59 (d, \(J = 7.1\) Hz, 1H), 7.48 (d, \(J = 7.4\) Hz, 2H), 7.36 (ddd, \(J = 28.6, 13.6, 7.9\) Hz, 6H), 7.29 – 7.24 (m, 2H), 7.24 – 7.20 (m, 1H), 7.14 (dd, \(J = 16.6, 8.1\) Hz, 2H), 7.02 (t, \(J = 7.3\) Hz, 1H), 6.88 (d, \(J = 7.7\) Hz, 1H), 6.71 (d, \(J = 7.7\) Hz, 1H), 6.11 (s, 1H), 5.17 – 5.05 (m, 2H), 5.03 (t, \(J = 17.2\) Hz, 2H), 4.43 (s, 1H), 3.51 (d, \(J = 14.1\) Hz, 1H), 2.32 (d, \(J = 14.1\) Hz, 1H), 1.22 (s, 3H), 1.05 (s, 3H).  \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 201.60, 183.96, 178.00, 143.78, 141.97, 136.31, 135.29, 131.03, 129.05, 128.74, 128.39, 128.21, 127.61, 127.41, 127.30, 126.95, 125.87, 124.72, 122.07, 109.31, 108.76, 82.48, 69.97, 54.19, 49.81, 44.69, 44.15, 28.32, 20.02. HRMS (EI) Calcd for \([C_{36}H_{32}N_2O_4]^+\): 556.2362; found: 556.2760.

\(1,1''\)-dibenzyl-2'-cyclopropanecarbonyl-4'-cyclopropyl-4'-hydroxy-1,1'',2,2''-tetrahydrodispiro[indole-3,3'-cyclopentane-1',3''-indole]-2,2''-dione (2l): The title compound was prepared according to the general procedure as a white solid in 43 % yield; mp = 235-237 °C; IR (KBr): 3332, 3049, 1701, 1671, 1601, 1410, 1360, 1173, 805, 770, 690, 640, 507, 532 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.12 (d, \(J = 7.7\) Hz, 1H), 7.59–7.57 (d, \(J = 7.6\) Hz, 1H), 7.43–7.17 (m, 12H), 7.14–7.10 (t, \(J = 7.0\) Hz, 1H), 7.04–7.00 (t, \(J = 7.0\) Hz, 1H), 6.90–6.88 (d, \(J = 8.0\) Hz, 1H), 6.75–6.73 (d, \(J = 8.0\) Hz, 1H), 6.08 (s, 1H), 5.32–5.28 (d, \(J = 16.0\) Hz, 1H), 5.18–5.14 (d, \(J = 16.0\) Hz, 1H), 4.95–4.91 (d, \(J = 16.0\) Hz, 1H), 4.87–4.83 (d, \(J = 16.0\) Hz, 1H), 3.47–3.36 (m, 2H), 3.29–3.23 (m, 1H), 2.92–2.88 (m, 1H), 2.67–2.63 (m, 1H), 2.34–2.30 (d, \(J = 16.0\) Hz, 1H), 1.83–1.78 (m, 2H), 1.72–1.66 (m, 1H), 1.48–1.36 (m, 1H), 1.22–1.19 (m, 1H), 0.99–0.97 (m, 1H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 202.90, 183.87, 177.63, 143.68, 142.07, 132.25, 135.17, 130.54, 129.10 (2C), 128.97, 128.81 (2C), 128.57, 128.31, 127.76 (3C), 127.54, 127.49 (2C), 127.22, 127.02, 125.83, 124.70, 122.20, 109.59, 108.81, 84.37, 69.06, 64.99, 54.09, 45.36, 44.79, 44.22, 43.71, 38.86, 31.40, 27.18, 25.62; HRMS (EI) Calcd for \([C_{40}H_{36}N_2O_4]^+\): 608.2675; found: 608.2674.
4'-hydroxy-4'-(naphthalen-1-yl)-2'(naphthalene-1-carbonyl)-2H,2''H-dispiro[acenaphthylene-1,3'-cyclopentane-1',1''-acenaphthylene]-2,2''-dione (4a) : The title compound was prepared according to the general procedure as a white solid in 77 % yield; mp = 254-256 °C; IR (KBr): 3372, 3072, 2918, 1701, 1682, 1605, 1482, 1424, 1366, 1339, 1274, 1231, 1174, 1077, 1000, 927, 815, 765, 746, 700, 554 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.46 (d, \(J=6.0\) Hz, 1H), 8.25 (d, \(J=7.2\) Hz, 1H), 8.07 (d, \(J=6.8\) Hz, 1H), 7.96 (d, \(J = 8.0\) Hz, 1H), 7.88–7.83 (dd, \(J = 5.2, 5.6\) Hz, 2H), 7.83 (d, \(J = 6.8\) Hz, 1H), 7.75–7.72 (dd, \(J = 7.2, 8.0\) Hz, 1H), 7.66–7.54 (m, 3H), 7.39–7.29 (m, 4H), 7.24 (merged dd, 1H), 7.16–7.12 (m, 3H), 7.06 (d, \(J = 8.1\) Hz, 2H), 6.94 (d, \(J = 8.4\) Hz, 2H), 6.81 (d, \(J = 8.8\) Hz, 2H), 6.72 (s, 1H), 5.63 (s, 1H), 5.30 (s, 1H), 4.61 (d, \(J = 14.0\) Hz, 1H), 2.73 (d, \(J = 14.0\) Hz, 1H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 213.12, 204.61, 197.05, 144.42, 142.24, 141.78, 140.56, 139.98, 139.78, 139.56, 137.86, 137.49, 134.84, 134.39, 133.13, 131.47, 130.74, 130.24, 130.74, 129.55, 128.72 (2C), 128.55 (2C), 128.18, 128.00, 127.76, 127.71, 127.18, 127.09, 126.97 (2C), 126.86 (2C), 125.85 (3C), 124.84, 124.53, 122.85, 122.81, 120.79, 84.62, 71.86, 67.90, 59.84, 47.28; HRMS (EI) Calcd for [C\(_{48}\)H\(_{30}\)O\(_4\)+Na]: 693.20418; found: 693.20439.

2'-(4-bromobenzoyl)-4'-(4-bromophenyl)-4'-hydroxy-2H,2''H-dispiro[acenaphthylene-1,3'-cyclopentane-1',1''-acenaphthylene]-2,2''-dione (4b) : The title compound was prepared according to the general procedure as a white solid in 74 % yield; mp = 250-252 °C; IR (KBr): 3334, 3056, 2925, 1713, 1693, 1605, 1562, 1497, 1435, 1393, 1324, 1270, 1235, 1096, 1077, 1012, 838, 777, 700, 538 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.39 (d, \(J = 6.7\) Hz, 1H), 8.17 (d, \(J = 6.8\) Hz, 1H), 8.06 (d, \(J = 7.0\) Hz, 1H), 7.98 (d, \(J = 8.0\) Hz, 2H), 7.83 (dd, \(J = 11.3, 7.8\) Hz, 2H), 7.74–7.65 (m, 3H), 7.61 (dd, \(J = 11.9, 7.2\) Hz, 2H), 7.01 (d, \(J = 8.2\) Hz, 2H), 6.88 (d, \(J = 8.1\) Hz, 2H), 6.71 (q, \(J = 8.2\) Hz, 4H), 6.66 (s, 1H), 5.47 (s, 1H), 4.48 (d, \(J = 14.3\) Hz, 1H), 2.64 (d, \(J = 14.2\) Hz, 1H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 212.77, 204.32, 196.46, 141.63, 139.53, 137.40, 137.23, 134.78, 134.10, 133.47, 131.00, 130.41, 130.32, 130.02, 129.50, 128.17, 127.99, 127.92, 126.62, 125.04, 124.79, 124.46, 123.08, 122.86, 121.64, 120.97, 84.25, 71.67, 67.70, 59.62, 47.03. HRMS (EI) Calcd for [C\(_{48}\)H\(_{23}\)Br\(_2\)O\(_4\)+Na]: 748.99390; found: 748.99408.

4'-hydroxy-2'-(4-phenylbenzoyl)-4'-(4-phenylphenyl)-2H,2''H-dispiro[acenaphthylene-1,3'-cyclopentane-1',1''-acenaphthylene]-2,2''-dione (4c) : The title
compound was prepared according to the general procedure as a white solid in 71 % yield; mp = 244-246 °C; IR (KBr): 3372, 3045, 2925, 1716, 1689, 1601, 1493, 1424, 1343, 1270, 1231, 1096, 1008, 919, 842, 777, 700, 646 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.47 (d, J = 5.6 Hz, 1H), 8.25 (d, J = 6.9 Hz, 1H), 8.07 (d, J = 7.0 Hz, 1H), 7.96 (d, J = 8.1 Hz, 1H), 7.86 (d, J = 5.5 Hz, 1H), 7.84 (d, J = 5.2 Hz, 1H), 7.80 (d, J = 6.9 Hz, 1H), 7.77 – 7.70 (m, 1H), 7.62 (td, J = 14.9, 7.8 Hz, 3H), 7.58 – 7.53 (m, 1H), 7.38 – 7.30 (m, 6H), 7.29 – 7.21 (m, 3H), 7.16 – 7.11 (m, 4H), 7.06 (d, J = 8.5 Hz, 2H), 6.94 (d, J = 8.3 Hz, 2H), 6.81 (d, J = 8.3 Hz, 1H), 6.72 (s, 1H), 5.63 (s, 1H), 4.61 (d, J = 14.2 Hz, 1H), 2.73 (d, J = 14.2 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 213.12, 204.61, 197.05, 144.42, 142.24, 140.56, 139.98, 139.78, 139.56, 137.86, 137.49, 134.84, 134.39, 133.13, 131.47, 130.74, 130.24, 130.01, 129.55, 128.72, 128.55, 128.18, 128.00, 127.76, 127.71, 127.18, 127.09, 126.97, 126.86, 126.58, 125.87, 124.84, 124.53, 122.85, 122.81, 120.79, 84.62, 71.86, 67.90, 59.84, 47.28. HRMS (EI) Calcd for \([C_{52}H_{34}O_4^+Na]\): 745.23548; found: 745.23567.

**2'-(4-chlorobenzoyl)-4'-(4-chlorophenyl)-4'-hydroxy-2H,2''H-dispiro[acenaphthylene-1,3'-cyclopentane-1',1''-acenaphthylene]-2,2''-dione (4d)**: The title compound was prepared according to the general procedure as a white solid in 64 % yield; mp = 240-242 °C; IR (KBr): 3368, 3045, 2929, 1720, 1682, 1601, 1489, 1428, 1343, 1270, 1227, 1093, 1008, 923, 846, 777, 700 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.38 (d, J = 6.7 Hz, 1H), 8.16 (d, J = 6.8 Hz, 1H), 8.05 (d, J = 7.0 Hz, 1H), 7.97 (d, J = 8.0 Hz, 2H), 7.84 (d, J = 8.5 Hz, 1H), 7.81 (d, J = 7.1 Hz, 1H), 7.72 – 7.66 (m, 2H), 7.60 (dd, J = 11.9, 7.2 Hz, 2H), 7.25 (s, 1H), 7.00 (d, J = 8.2 Hz, 2H), 6.87 (d, J = 8.1 Hz, 2H), 6.71 (q, J = 8.2 Hz, 4H), 6.66 (s, 1H), 5.46 (s, 1H), 4.48 (d, J = 14.3 Hz, 1H), 2.63 (d, J = 14.2 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 212.76, 204.31, 196.45, 141.63, 139.53, 137.39, 137.23, 134.78, 134.09, 133.46, 131.00, 130.41, 130.31, 130.01, 129.50, 128.17, 127.98, 127.92, 126.62, 125.03, 124.79, 124.46, 123.07, 122.86, 121.63, 120.97, 84.25, 71.66, 67.70, 59.61, 47.02. HRMS (EI) Calcd for \([C_{46}H_{24}Cl_2O_4^+Na]\): 661.09493; found: 661.09497.

**NMR Spectra of compounds 2a-j and 4a-d**
Figure S1. $^1$H NMR spectrum of compound 2a

Figure S2. $^{13}$C NMR spectrum of compound 2a
Figure S3. HMBC spectrum of compound 2a

Figure S4. DEPT-135 spectrum of compound 2a
Figure S5. $^1$H NMR spectrum of compound 2b

Figure S6. $^{13}$C NMR spectrum of compound 2b
Figure S7. DEPT-135 spectrum of compound 2b

Figure S8. $^1$H NMR spectrum of compound 2c
Figure S9. $^{13}$C NMR spectrum of compound 2c

Figure S10. DEPT-135 spectrum of compound 2c
Figure S11. $^1$H NMR spectrum of compound 2d

Figure S12. $^{13}$C NMR spectrum of compound 2d
Figure S13. DEPT-135 spectrum of compound 2d

Figure S14. $^1$H NMR spectrum of compound 2e
Figure S15. $^{13}$C NMR spectrum of compound 2e

Figure S16. DEPT-135 spectrum of compound 2e
Figure S17. $^1$H NMR spectrum of compound 2f

Figure S18. $^{13}$C NMR spectrum of compound 2f
Figure S19. DEPT-135 spectrum of compound 2f

Figure S20. $^1$H NMR spectrum of compound 2g
Figure S21. $^{13}$C NMR spectrum of compound 2g

Figure S22. DEPT-135 spectrum of compound 2g
Figure S23. $^1$H NMR spectrum of compound 2h

Figure S24. $^{13}$C NMR spectrum of compound 2h
Figure S25. DEPT-135 spectrum of compound 2h

Figure S26. 1H NMR spectrum of compound 2i
Figure S27. $^{13}$C NMR spectrum of compound 2i

Figure S28. DEPT-135 spectrum of compound 2i
Figure S29. $^1$H NMR spectrum of compound 2j

Figure S30. $^{13}$C NMR spectrum of compound 2j
Figure S31. DEPT-135 spectrum of compound 2j

Figure S32. 1H NMR spectrum of compound 2k
Figure S33. $^{13}$C NMR spectrum of compound 2k

Figure S34. DEPT-135 spectrum of compound 2k
Figure S35. $^1$H NMR spectrum of compound 2l

Figure S36. $^{13}$C NMR spectrum of compound 2l
Figure S37. DEPT-135 spectrum of compound 21

Figure S38. $^1$H NMR spectrum of compound 4a
Figure S39. $^{13}$C NMR spectrum of compound 4a

Figure S40. DEPT-135 spectrum of compound 4a
Figure S41. $^1$H NMR spectrum of compound 4b

Figure S42. $^{13}$C NMR spectrum of compound 4b
Figure S43. DEPT-135 spectrum of compound 4b

Figure S44. $^1$H NMR spectrum of compound 4c
Figure S45. $^{13}$C NMR spectrum of compound 4c

Figure S46. DEPT-135 spectrum of compound 4c
Figure S47. $^1$H NMR spectrum of compound 4d

Figure S48. $^{13}$C NMR spectrum of compound 4d
Figure S49. DEPT-135 spectrum of compound 4d

Figure S50. Mass spectrum of 2a
Figure S51. Mass spectrum of 2b

Figure S52. Mass spectrum of 2c
Figure S53. Mass spectrum of 2d

Figure S54. Mass spectrum of 2e
Figure S55. Mass spectrum of 2f

Figure S56. Mass spectrum of 2g
Figure S57. Mass spectrum of 2h

Figure S58. Mass spectrum of 2i
Figure S59. Mass spectrum of 2j

Figure S60. Mass spectrum of 2k
Figure S61. Mass spectrum of 2l

Figure S62. Mass spectrum of 4a
Figure S63. Mass spectrum of 4b

Figure S64. Mass spectrum of 4c
Figure S65. Mass spectrum of 4d

Detecting the formation of intermediate ii (Scheme 2) through ESI MS method
Figure S66. Mass spectrum of substrate 1a
Figure S67. Mass spectrum of substrate 1a and intermediate ii in Scheme 2

Spectrum was recorded using an aliquot withdrawn from the reaction mixture after 30 minutes. Reaction mixture contained 20 mg 1a, 150 μL DIPEA and 20 mL ethanol.
**Figure S68.** Mass spectrum of substrate 1a and product 2a

Spectrum was recorded using an aliquot withdrawn from the reaction after 5 h.
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Channel Name: 2998 Ch1 260nm@1.2nm  
Sample Set Name:  

**Control Experiment**

![Chemical Structure]

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Page: 1 of 1  
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Page: 1 of 1
S49

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Report Method: Untitled
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An aliquot was withdrawn after 11.5 h for HPLC experiment.

Molecular weight = 339

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Sample Set Name

18 HPLC profile of the precipitated isolated from the reaction mixture by centrifugation.

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ESI-MS Profile of the HPLC fraction collected at ~48 minute sample: An aliquot withdrawn after 5.5 h
ESI-MS profile of the fraction collected at ~40 minute
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<td>F000’</td>
<td>716.31</td>
<td>716.31</td>
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<tr>
<td>h,k,lmax</td>
<td>14,15,16</td>
<td>14,15,16</td>
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<tr>
<td>Nref</td>
<td>7356</td>
<td>7171</td>
<td></td>
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<tr>
<td>Tmin, Tmax</td>
<td>0.986, 0.992</td>
<td>0.984, 0.992</td>
<td></td>
</tr>
<tr>
<td>Tmin’</td>
<td>0.984</td>
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</tbody>
</table>

Correction method= # Reported T Limits: Tmin=0.984 Tmax=0.992
AbsCorr = ?

Data completeness= 0.975  Theta(max)= 26.430

R(reflections)= 0.0497( 3717)  wR2(reflections)= 0.1568( 7171)

S = 1.006  Npar = 471