SUPPPORTING INFORMATION

One-Pot Palladium Catalyzed Synthesis of

Benzo[b]carbazolatediones

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General information

All used chemicals are commercially available and used without further purification. All reactions were carried out in dried pressure tubes under an argon atmosphere. Analytical TLC on Merck silica gel 60 F254 plates was visualized by fluorescence quenching. Column chromatography was performed on Merck Geduran Si 60 (0.063-0.200 mm).

$^1$H NMR and $^{13}$C NMR spectra were carried out on a Bruker Advance DRX-500 MHz, 300 MHz or 250 MHz. Chemical shifts in ppm were corrected by residual solvent CDCl$_3$ ($^1$H, 7.26 ppm, $^{13}$C, 77.16 ppm) or DMSO-d6 ($^1$H, 2.50 ppm, $^{13}$C, 39.52 ppm). Multiplicities: s = singlet, d = doublet, dd = doublet of doublets, t = triplet, td = triplet of doublets, tt = triplet of triplets, m = multiplet, q = quartet. Nicolet 550 FT – IR spectrometer was used with ATR sampling technique for solids. Signal characterization: vw = very weak, w = weak, m = medium, s = strong, vs = very strong.

Gas chromatography - mass analysis was performed on an Agilent HP-5890 instrument with an Agilent HP-5973 Mass Selective Detector (EI) and HP-5 capillary column using helium carrier gas. Agilent 1969A TOF mass spectrometer was used for ESI HR-MS measurements. High Resolution MS (HRMS) was performed on a Finnigan MAT 95 XP.

Single crystal X-Ray structure determination was carried out on a Bruker X8Apex diffractometer with CCD camera (Mo Ka radiation and graphite monochromator, a = 0.071073 Å).

Melting points were determined on a Micro-Hot-Stage GalenTM III Cambridge Instruments. The melting points are not corrected.
General procedures

Procedure A: 2,3-dibromo-1,4-naphthoquinone 1 (0.3 mmol), the appropriate amine (0.3 mmol) and H$_2$O (1 mL) were poured into a pressure tube. The reaction was set up at 60 °C for 6 h, then, 2-bromophenylboronic acid (0.3 mmol), Pd(PPh$_3$)$_4$ (5 mol%), Pd$_2$dba$_3$ (5 mol%), RuPhos (10 mol%), K$_3$PO$_4$ (0.9 mmol) and 1,4-dioxane (10 mL) were added under argon. The tube was sealed with a Teflon valve and stirred at 90 °C. After 24 h, the mixture was allowed to reach room temperature, diluted with water and extracted with dichloromethane. The combined organic layers were dried over sodium sulfate and concentrated under vacuum. The crude material was purified by flash column chromatography on silica gel.

Procedure B: An argon purged pressure tube was charged with 2,3-dibromo-1,4-naphthoquinone 1 (0.3 mmol) and the secondary amine (0.3 mmol), Pd(OAc)$_2$ (5 mol%), PPh$_3$ (10 mol%), tBuONa (0.9 mmol) and toluene (10 mL). The reaction was set up at 90 °C for 24 h. Afterwards the mixture was allowed to reach room temperature, was diluted with water and extracted with ethyl acetate. The combined organic layers were dried over sodium sulfate and concentrated under vacuum. The crude material was purified by flash column chromatography on silica gel.
Analytical data

2-Bromo-3- (p-tolylamino)naphthalene-1,4-dione (2b)

Starting from p-toluidine with 2,3-dibromonaphthalene-1,4-dione in water at 60 °C for 6 h, 2b was obtained as a dark red solid, mp = 157 - 158 °C. $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ = 8.19 (dd, $^3J = 7.6$ Hz, $^4J = 1.3$ Hz, 1H), 8.14 – 8.05 (m, 1H), 7.81 – 7.60 (m, 3H), 7.15 (d, $^3J = 8.2$ Hz, 2H), 7.00 (d, $^3J = 8.3$ Hz, 2H), 2.36 (s, 3H, CH$_3$). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ = 180.3, 177.4 (C=O), 144.4, 136.0 (C), 135.1 (CH), 135.0 (C), 133.0 (CH), 132.6, 130.0 (C), 129.2 (2CH), 127.5, 127.2 (CH), 125.0 (2CH), 107.0 (C), 21.2 (CH$_3$). IR (ATR, cm$^{-1}$): = 3302 (m), 3223 (w), 3095 (w), 3024 (w), 2916 (w), 1672 (s), 1645 (m), 1630 (m), 1591 (m), 1581 (m), 1566 (m), 1547 (s), 1516 (m), 1497 (m), 1479 (m), 1454 (m), 1410 (m), 1369 (w), 1329 (m), 1298 (m), 1282 (m), 1263 (s), 1234 (s), 1188 (m), 1174 (m), 1161 (m), 1124 (s), 1111 (s), 1097 (m), 1078 (m), 1043 (m), 1026 (m), 1012 (m), 974 (m), 960 (m), 941 (m), 908 (m), 895 (m), 845 (m), 831 (m), 818 (m), 804 (m), 787 (s), 773 (s), 752 (m), 717 (s), 700 (vs), 679 (s), 660 (m), 636 (m), 625 (s), 604 (m), 542 (s), 530 (m). MS (El, 70 eV): m/z (%) = 341 (M$^+$, 73), 326 (4), 262 (100), 247 (12), 219 (9), 178 (10), 158 (3), 130 (5), 105 (13), 91 (29), 76 (14), 65 (21), 50 (7), 39 (6). HRMS (El): calcd C$_{17}$H$_{13}$BrNO$_2$ (M$^+$) 341.00459, found 341.00382.

2-(2-Bromophenyl)-3- (p-tolylamino)naphthalene-1,4-dione (3b)

Starting from p-toluidine, following general procedure A, 3b was obtained as a red solid (21 mg, 17 %), mp = 175 - 176 °C. $^1$H NMR (250 MHz, CDCl$_3$) $\delta$ = 8.23 – 8.11 (m, 2H), 7.82 – 7.66 (m, 3H), 7.27 – 7.20 (m, 1H), 7.04 – 6.81 (m, 3H), 6.78 – 6.62 (m, 4H), 2.14 (s, 3H, CH$_3$). $^{13}$C NMR (63 MHz, CDCl$_3$) $\delta$ = 182.9, 181.5 (C=O), 142.2, 135.2 (C), 135.1 (CH), 134.6, 134.3, 133.6 (C), 132.9, 132.4, 132.2 (CH), 130.4 (C), 128.7 (CH), 128.5 (2CH), 126.9, 126.5, 126.5 (CH), 125.3 (C), 124.6 (2CH), 115.9 (C), 20.9 (CH$_3$). IR (ATR, cm$^{-1}$): = 3331 (w), 3298 (s), 3064 (w), 3045 (w), 3010 (w), 2920 (w), 1674 (s), 1633 (m), 1612 (w), 1595 (m), 1569 (s), 1516 (s), 1505 (s), 1469 (s), 1425 (m), 1407 (m), 1379 (w), 1328 (s), 1294 (s), 1279 (s), 1253 (s), 1211 (m), 1173 (m), 1156 (m), 1133 (m), 1106 (s), 1052 (m), 1040 (m), 1029 (m), 1018 (s), 1006 (s), 966 (m), 942 (m), 920 (m), 863 (m), 850 (m), 835 (m), 811 (s), 795 (s), 748 (vs), 727 (s), 714 (vs), 670 (s), 655 (s), 647 (s), 632 (s), 599 (s), 578 (s), 530.5 (s). MS (El, 70 eV): m/z (%) = 419 (4), 417 (M$^+$, 5), 339 (27), 338 (100), 324 (7), 323 (26), 295 (3) 294 (5), 281 (2), 266 (2), 239 (2), 221 (1), 204 (2), 190 (6), 176 (7), 169 (3), 165 (6), 161 (9), 147 (2), 139 (1), 104 (3), 91 (4), 76 (5), 65 (4), 51 (1), 39 (2). HR-MS (El): calcd for C$_{21}$H$_{18}$BrNO$_2$ (M$^+$) 417.03589, found 417.03524; calcd for C$_{22}$H$_{16}$$^{81}$BrNO$_2$ (M$^+$) 419.03385, found 419.03478.
5-Phenyl-5H-benzo[b]carbazole-6,11-dione (4a)

Starting from aniline, following general procedure A, 4a was obtained as a light red solid (49 mg, 51 %), and starting from diphenylamine, following general procedure B the same compound was isolated in 38 % yield (37 mg), mp = 253 - 255 °C. 1H NMR (250 MHz, CDCl3) δ = 8.58 – 8.48 (m, 1H), 8.25 (dd, 3J = 7.6 Hz, 4J = 1.3 Hz, 1H), 8.05 (dd, 3J = 7.5 Hz, 4J = 1.4 Hz, 1H), 7.78 – 7.57 (m, 5H), 7.49-7.37 (m, 4H), 7.21 – 7.14 (m, 1H). 13C NMR (63 MHz, CDCl3) δ = 181.8, 177.7 (C=O), 141.2 (C), 136.9, 135.7, 134.2 (C), 133.9 (CH), 133.7 (C), 133.2 (CH), 129.7 (2CH), 129.4, 127.8 (CH), 127.8 (2CH), 126.6 (CH), 126.5, 124.9 124.0 (C), 123.9 (CH), 120.0 (C), 112.3 (CH). IR (ATR, cm⁻¹): 3070 (m), 3047 (w), 2924 (w), 1649 (s), 1612 (m), 15894 (s), 1570 (m), 1516 (s), 1499 (m), 1487 (s), 1470 (m), 1456 (s), 1448 (s), 1435 (m), 1412 (s), 1402 (s), 1354 (m), 1335 (m), 1315 (m), 1281 (m), 1269 (s), 1234 (m), 1215 (vs), 1188 (m), 1173 (m), 1163 (m), 1148 (m), 1134 (s), 1108.92 (m), 1095.42 (m), 1080 (m), 1045 (s), 1026 (m), 1016 (s), 997 (s), 970 (m), 964 (m), 955 (s), 918 (m), 891 (m), 878 (m), 860 (m), 843 (m), 812 (m), 793 (m), 762 (vs), 750 (s), 721 (m), 706 (vs), 696 (vs), 665 (s), 625 (m), 617 (m), 594 (vs), 571 (m), 532 (m). MS (EI, 70 eV): m/z (%) = 323 (100), 294 (11), 265 (14), 239 (4), 218 (1), 190 (6), 161 (7), 147 (3), 132 (9), 119 (2), 88 (1), 77 (4), 63 (1), 51 (3), 39 (1). HR-MS (EI): Calcd for C22H11O2N (M+):323.09406; found 323.09334.

5-(p-Toluidine)-5H-benzo[b]carbazole-6,11-dione (4b)

Starting from p-toluidine, following general procedure A, 4b was obtained as an orange solid (48 mg, 49 %), mp = 266 - 268 °C. 1H NMR (250 MHz, CDCl3) δ = 8.56 – 8.46 (m, 1H), 8.30 – 8.19 (m, 1H), 8.10 – 7.96 (m, 1H), 7.79 – 7.58 (m, 2H), 7.46 – 7.29 (m, 6H), 7.21 – 7.09 (m, 1H), 2.52 (s, 3H, CH3). 13C NMR (63 MHz, CDCl3) δ = 181.7, 177.7 (C=O), 141.3, 139.4, 135.7, 134.3 (C), 134.2 (C), 133.8 (CH), 133.7 (C), 133.1 (CH), 130.3 (2CH), 127.7 (CH), 127.4 (2CH), 126.6 (CH), 126.4 (CH), 124.9 (CH), 124.0 (C), 123.8 (CH),119.9 (C), 112.3 (CH), 21.5 (CH3). IR (ATR, cm⁻¹): 3062 (w), 3050 (w), 2956 (w), 2921 (m), 2851 (m), 1732 (w), 1660 (m), 1641 (m), 1612 (w), 1588 (m), 1516 (s), 1488 (m), 1458 (s), 1418 (m), 1401 (m), 1350 (w), 1318 (m), 1310 (w), 1282 (m), 1158 (m), 1149 (m), 1131 (m), 1111 (m), 1095 (m), 1045 (s), 1017 (s), 1003 (s), 971 (m), 961.3 (m), 947 (m), 937 (m), 894 (m), 877 (m), 847 (m), 832 (s), 799 (s), 790 (s), 744 (vs), 725 (m), 709 (s), 697 (s), 664 (m), 648 (m), 640 (m), 610 (m), 596 (s), 571 (m). MS (EI, 70 eV): m/z (%) = 337 (M⁺, 100), 322 (38), 308 (9), 278 (12), 252 (2), 239 (1), 204 (1), 190 (3), 176 (1), 168 (11), 163 (5), 152 (2), 147 (2), 139 (4), 132 (7), 126 (2), 113 (1), 91 (2), 76 (2), 65 (3), 51 (1), 39 (2). HR-MS (EI): Calcd for C23H13NO2 (M+): 337.10973, found 337.10925.

5-(4-Fluorophenyl)-5H-benzo[b]carbazole-6,11-dione (4c)
Starting from 4-fluoroaniline, following general procedure A, 4c was obtained as a orange solid (62 mg, 60 %), mp = 275 - 277 °C. ¹H NMR (300 MHz, CDCl₃) δ = 8.56 – 8.48 (m, 1H), 8.24 (dd, ³J = 7.6 Hz, ⁴J = 1.2 Hz, 1H), 8.04 (dd, ³J = 7.5 Hz, ⁴J = 1.2 Hz, 1H), 7.77 – 7.63 (m, 2H), 7.47 – 7.40 (m, 4H), 7.34 - 7.27 (m, 2H), 7.18 – 7.11 (m, 1H). ¹⁹F NMR (282 MHz, CDCl₃) δ = -111.41; ¹³C NMR (126 MHz, CDCl₃) δ = 181.7, 177.8 (C=O), 162.9 (d, ³J = 249.7 Hz, CF), 141.3, 135.7, 134.2, 134.0 (C), 133.6 (CH), 133.2 (CH), 132.9 (d, ³J = 3.5 Hz, C), 129.6 (d, ³J = 9.0 Hz, 2CH), 128.0 (CH), 126.6 (CH), 125.1 (CH), 124.0 (CH), 124.0 (CH), 120.2 (C), 116.7 (d, ³J = 23.1 Hz, 2CH), 112.0 (C). IR (ATR, cm⁻¹): = 3062 (w), 3047 (w), 3009 (vw), 2959 (w), 2921 (m), 2850 (m), 1658 (s), 1650 (s), 1613 (m), 1590 (m), 1555 (w), 1512 (s), 1486 (s), 1461 (s), 1454 (s), 1423 (m), 1401 (m), 1319 (m), 1286 (m), 1272 (s), 1218 (s), 1157 (s), 1146 (s), 1130 (m), 1093 (s), 1045 (s), 1017 (s), 999 (s), 960 (m), 941 (s), 894 (m), 877 (m), 851 (s), 831 (m), 813 (s), 795 (s), 765 (m), 744 (vs), 709 (vs), 666 (m), 646 (s), 598 (s), 570 (s), 529 (s). MS (EI, 70 eV): m/z (%) = 341 (M⁺), 100, 324 (3), 312 (16), 296 (7), 283 (15), 257 (4), 208 (2), 190 (4), 170 (9), 163 (5), 156 (3), 141 (6), 119 (2), 95 (3), 75 (5), 63 (1), 50 (2), 39 (1). HR-MS (EI): Calcd for C₂₃H₂₂N₂O₅ (M⁺): 341.08466, found: 341.08426.

5-(4-Nitrophenyl)-5H-benzo[b]carbazole-6,11-dione (4d)

Starting from 4-Nitroaniline, following general procedure A, 4d was obtained as an orange solid (42 mg, 37 %), mp = 286 - 288 °C. ¹H NMR (300 MHz, DMSO) δ = 8.52 – 8.45 (m, 2H), 8.40 – 8.34 (m, 1H), 8.13 (dd, ³J = 7.4, ⁴J = 1.3 Hz, 1H), 8.00 – 7.92 (m, 3H), 7.90 – 7.78 (m, 2H), 7.55 – 7.45 (m, 2H), 7.32 – 7.24 (m, 1H). ¹³C NMR (63 MHz, DMSO) δ = 180.9, 176.7 (C=O), 147.4, 142.1, 139.8, 135.8 (C), 134.3, 133.6 (CH), 133.2, 133.0 (C), 129.4 (2CH), 128.2, 126.2, 125.9, 125.1 (CH), 124.7 (2CH), 123.4 (C), 122.9 (CH), 119.3 (C), 112.0 (CH). IR (ATR, cm⁻¹): = 3116 (w), 3077 (w), 2922 (w), 2851 (w), 1662 (m), 1646 (m), 1607 (w), 1590 (m), 1556 (w), 1518 (m), 1485 (m), 1473 (w), 1456 (w), 1449 (m), 1424 (w), 1399 (m), 1351 (m), 1318 (m), 1283 (m), 1272 (m), 1215 (s), 1186 (m), 1155 (m), 1133 (m), 1108 (w), 1095 (m), 1087 (m), 1045 (m), 1018 (m), 1002 (m), 973 (w), 961 (w), 949 (m), 900 (w), 877 (w), 855 (m), 827 (w), 812 (w), 792 (m), 781 (m), 747 (s), 715 (s), 705 (s), 685 (m), 666 (m), 637 (w), 629 (m), 607 (w), 594 (s), 545 (m). MS (EI, 70 eV): m/z (%) = 368 (M⁺, 100), 339 (4), 322 (16), 310 (2), 293 (9), 265 (23), 253 (2), 239 (3), 190 (5), 161 (4), 133 (7), 120 (2), 75 (2), 63 (1), 50 (2), 30 (2). HR-MS (EI): calcd for C₂₃H₂₁N₂O₄ (M⁺): 368.07916, found 368.07895.

5-(4-Methoxyphenyl)-5H-benzo[b]carbazole-6,11-dione (4e)

Starting from p-anisidine, following general procedure A, 4e was obtained as a light orange solid (50 mg, 47 %), mp = 237 - 239 °C. ¹H NMR (300 MHz, CDCl₃) δ = 8.56 – 8.47 (m, 1H), 8.25 (dd, ³J = 7.6 Hz, ⁴J = 1.2 Hz, 1H), 8.11 – 8.01 (m, 1H), 7.77 – 7.61 (m, 2H), 7.45 – 7.33 (m, 4H), 7.21 – 7.15 (m, 1H), 7.13 – 7.07 (m,
2H), 3.93 (s, 3H, OCH₃). ¹³C NMR (126 MHz, CDCl₃) δ = 181.6, 177.7 (C=O), 160.0, 141.4, 135.6, 134.1 (CH), 133.7 (C), 133.6 (CH), 133.0 (C), 129.4 (CH), 128.7 (2CH), 127.6, 126.5, 126.3, 124.8 (C), 123.8 (CH), 123.7 (C), 119.7 (CH), 114.7 (2CH), 112.2 (C), 55.6 (OCH₃). IR (ATR, cm⁻¹): = 3296 (m), 3228 (w), 3091 (m), 3072 (m), 3006 (m), 2957 (m), 2923 (m), 2849 (m), 2835 (m), 1651 (s), 1610 (m), 1593 (s), 1514 (s), 1486 (s), 1471 (m), 1456 (s), 1441 (s), 1397 (s), 1353 (m), 1322 (m), 1300 (m), 1280 (m), 1269 (s), 1246 (s), 1214 (s), 1186 (s), 1168 (s), 1146 (s), 1130 (s), 1094 (s), 1044 (s), 1028 (s), 1018 (vs), 1011 (vs), 999 (vs), 969 (s), 958 (s), 951 (s), 928 (s), 897 (m), 877 (m), 869 (m), 837 (s), 810 (s), 798 (s), 768 (vs), 758 (vs), 726 (m), 706 (vs), 695 (s), 663 (s), 651 (s), 636 (m), 595 (s), 580 (s), 531 (s). MS (EI, 70 eV): m/z (%) = 353 (M⁺, 100), 338 (9), 322 (11), 310 (8), 293 (4), 281 (5), 265 (6), 254 (9), 226 (4), 201 (2), 190 (4), 176 (8), 163 (4), 140 (2), 133 (3), 126 (6) 113 (3), 100 (2), 88 (2), 76 (4), 63 (4), 50 (2), 39 (1). HR-MS (EI): calcd for C₃₂H₂₇NO₃ (M⁺): 353.0464, found 353.0441.

5-(3,5-Dimethylphenyl)-5H-benzo[b]carbazole-6,11-dione (4f)

Starting from 3,5-Dimethylaniline, following general procedure A, 4f was obtained as a yellow solid (49 mg, 47 %), mp = 248 - 250 °C. ¹H NMR (300 MHz, CDCl₃) δ = 8.57 – 8.47 (m, 1H), 8.25 (dd, ¹J = 7.6 Hz, ¹J = 1.2 Hz, 1H), 8.06 (dd, ³J = 7.6, ⁴J = 1.3 Hz, 1H), 7.77 – 7.61 (m, 2H), 7.47 – 7.35 (m, 2H), 7.22 – 7.14 (m, 2H), 7.04 (s, 2H), 2.43 (s, 6H, 2CH₃). ¹³C NMR (75 MHz, CDCl₃) δ = 181.8, 177.6 (C=O), 141.4 (C), 139.4 (2C), 136.7, 135.7, 134.2 (C), 133.8 (CH), 133.7 (C), 133.1, 131.1, 127.7, 126.6, 126.5 (CH), 125.3 (2CH), 124.8 (CH), 124.0 (C), 123.8 (CH), 119.8 (C), 112.5 (CH), 21.5 (2CH₃). IR (ATR, cm⁻¹): = 3055 (w), 3010 (w), 2918 (w), 2861 (w), 1660 (s), 1650 (s), 1614 (m), 1594 (m), 1586 (m), 1571 (w), 1518 (s), 1491 (m), 1473 (m), 1456 (m), 1409 (m), 1395 (m), 1354 (w), 1319 (m), 1291 (m), 1263 (w), 1249 (m), 1220 (s), 1175 (m), 1157 (m), 1148 (m), 1132 (m), 1107 (w), 1090 (w), 1056 (m), 1027 (m), 1014 (m), 995 (m), 970 (m), 947 (m), 911 (w), 902 (w), 894 (w), 869 (m), 854 (m), 808 (w), 790 (m), 745 (s), 736 (m), 723 (w), 708 (vs), 692 (s), 666 (m), 633 (w), 613 (w), 604 (m), 574 (w), 549 (w). MS (EI, 70 eV): m/z (%) = 351 (M⁺, 100), 336 (88), 322 (6), 306 (4), 291 (7), 278 (11), 265 (2), 252 (3), 240 (1), 226 (1), 218 (2), 202 (1), 190 (4), 176 (8), 168 (8), 163 (5), 154 (4), 145 (2), 139 (8), 132 (3), 126 (2), 113 (1), 105 (2), 89 (2), 77 (6), 63 (2), 50 (1), 39 (2). HR-MS (EI): calcd for C₃₂H₂₇NO₃ (M⁺): 351.0238, found 351.02488.

5-(4-(Tert-butyl)phenyl)-5H-benzo[b]carbazole-6,11-dione (4g)

Starting from 4-(tert-butyl)aniline, following general procedure A, 4g was obtained as a yellow solid (55 mg, 48 %), mp = 294 - 296 °C. ¹H NMR (300 MHz, CDCl₃) δ = 8.56 – 8.48 (m, 1H), 8.25 (dd, ³J = 7.6 Hz, ⁴J = 1.3 Hz, 1H), 8.07 (dd, ³J = 7.6, ⁴J = 1.2 Hz, 1H), 7.79 – 7.57 (m, 4H), 7.49 – 7.32 (m, 4H), 7.24 – 7.13
Starting from C2198791171, 132.9, 127.3, 126.6, 126.1), 773), 278 m, 3069 (w), 3041 (w), 2965 (m), 2868 (m), 1663 (s), 1650 (s), 1613 (m), 1591 (m), 1584 (m), 1556 (m), 1519 (s), 1488 (s), 1465 (s), 1400 (s), 1370 (m), 1352 (m), 1320 (m), 1283 (m), 1270 (s), 1182 (m), 1159 (m), 1150 (m), 1130 (m), 1109 (m), 1095 (m), 1044 (s), 1014 (s), 1000 (s), 977 (m), 956 (s), 941 (m), 903 (m), 877 (m), 850 (m), 839 (s), 825 (m), 814 (m), 797 (s), 778 (s), 750 (vs), 713 (vs), 698 (s), 664 (m), 647 (m), 635 (m), 597 (vs), 573 (m), 553 (s). MS (EI, 70 eV): m/z (%) = 379 (M+, 60), 364 (100), 348 (3), 336 (5), 322 (10), 306 (2), 291 (3), 278 (3), 265 (4), 239 (1), 190 (3), 182 (2), 168 (9), 165 (2), 154 (2), 139 (3), 133 (2), 115 (2), 105 (2), 91 (1), 77 (2), 57 (1), 41 (1). HR-MS (EI): Calcd for C26H25NO2 (M+) 379.15668, found 379.15654.

5-Butyl-5H-benzo[b]carbazole-6,11-dione (4k)

Starting from 1-n-butylamine, following general procedure A, 4k was obtained as a yellow solid (38 mg, 42%), mp = 109 - 111 °C. 1H NMR (300 MHz, CDCl3) δ = 8.50 - 8.46 (m, 1H), 8.25 - 8.11 (m, 2H), 7.78 - 7.63 (m, 2H), 7.52 - 7.35 (m, 3H), 4.78 - 4.67 (m, 2H, CH2), 1.96 - 1.79 (m, 2H, CH2), 1.54 - 1.41 (m, 2H, CH2), 0.99 (t, J = 7.3 Hz, 3H, CH3). 13C NMR (63 MHz, CDCl3) δ = 181.4, 179.1 (C=O), 139.6, 134.9, 134.3 (C), 133.9 (CH), 133.8 (C), 132.9, 127.3, 126.6, 126.4, 124.6 (CH), 124.2 (C), 124.1 (CH), 119.2 (C), 111.2 (CH), 45.3 (CH2), 32.5 (CH3), 20.3 (CH3), 14.0 (CH3). IR (ATR, cm⁻¹): = 3063 (w), 3010 (w), 2997 (w), 2960 (m), 2916 (m), 2862 (m), 2848 (m), 1657 (s), 1643 (s), 1614 (m), 1593 (s), 1576 (m), 1516 (s), 1497 (m), 1473 (s), 1460 (s), 1423 (m), 1400 (s), 1379 (m), 1346 (m), 1340 (m), 1315 (m), 1296 (m), 1284 (m), 1259 (s), 1236 (s), 1198 (s), 1171 (s), 1155 (m), 1134 (m), 1107 (m), 1088 (m), 1059 (s), 1030 (m), 1012 (m), 980 (s), 933 (m), 891 (m), 879 (m), 862 (m), 851 (m), 808 (m), 797 (s), 777 (m), 748 (vs), 714 (vs), 694 (s), 669 (s), 617 (m), 602 (s), 579 (m), 565 (m), 544 (m). MS (EI, 70 eV): m/z (%) = 303 (M+, 80), 274 (17), 260 (100), 247 (31), 232 (7), 219 (9), 203 (8), 190 (17), 176 (10), 130 (3), 105 (4), 88 (4), 77 (4), 63 (1), 41 (3). HR-MS (EI): Calcd for C26H25NO2 (M+): 303.12538, found: 303.12599

5-Pentyl-5H-benzo[b]carbazole-6,11-dione (4l)

Starting from 1-n-pentylamine, following general procedure A, 4l was obtained as a yellow solid (46 mg, 48%), mp = 102 - 104 °C. 1H NMR (300 MHz, CDCl3) δ = 8.46 (d, J = 7.9 Hz, 1H), 8.26 - 8.09 (m, 2H), 7.78 - 7.60 (m, 2H), 7.49 - 7.32 (m, 3H), 4.79 - 4.59 (m, 2H), 1.99 - 1.79 (m, 2H, CH2), 1.46 - 1.34 (m, 4H, 2CH2), 0.91 (t, J = 6.9 Hz, 3H, CH3). 13C NMR (75 MHz, CDCl3) δ = 181.3, 179.0 (C=O), 139.5, 134.9, 134.3 (C), 133.8 (CH), 133.8 (C), 132.9, 127.3, 126.6, 126.4, 124.5 (CH), 124.2 (C), 124.1 (CH), 119.1 (C), 111.2 (CH),...
5-Hexyl-5H-benzo[b]carbazole-6,11-dione (4m)

Starting from 1-n-hexylamine, following general procedure A, 4m was obtained as a yellow solid (57 mg, 57 %), mp = 115 - 117 °C. ¹H NMR (250 MHz, CDCl₃) δ = 8.46 (d, J = 7.8 Hz, 1H), 8.26 - 8.09 (m, 2H), 7.77 - 7.60 (m, 2H), 7.51 - 7.33 (m, 3H), 4.74 - 4.63 (m, 2H, CH₂), 1.95 - 1.78 (m, 2H, CH₂), 1.53 - 1.28 (m, 6H, 3CH₂), 0.88 (t, J = 6.7 Hz, 3H, CH₃). ¹³C NMR (63 MHz, CDCl₃) δ = 181.3, 179.0 (C=O), 139.5, 134.9, 134.3, 133.8 (C), 133.8, 132.9, 127.3, 126.6, 126.4, 124.5 (CH), 124.2 (C), 124.1 (CH), 119.1 (C), 111.2 (CH), 45.5 (CH₂), 31.6 (CH₂), 30.3 (CH₂), 26.7 (CH₂), 22.7 (CH₂), 14.1 (CH₃). IR (ATR, cm⁻¹): = 2968 (w), 2953 (m), 2922 (m), 2870 (w), 2854 (m), 1657 (s), 1643 (s), 1612 (m), 1591 (s), 1516 (s), 1495 (m), 1475 (s), 1460 (m), 1421 (m), 1398 (s), 1373 (m), 1362 (m), 1344 (m), 1315 (w), 1294 (w), 1281 (m), 1255 (s), 1225 (s), 1209 (m), 1194 (m), 1167 (m), 1149 (m), 1128 (m), 1120 (m), 1088 (m), 1059 (s), 1026 (m), 1014 (m), 962 (s), 939 (m), 897 (w), 887 (w), 862 (m), 852 (m), 829 (w), 812 (w), 802 (w), 793 (m), 775 (w), 760 (m), 743 (vs), 727 (m), 712 (vs), 694 (m), 667 (m), 621 (m), 598 (m), 577 (m), 538 (w). MS (EI, 70 eV): m/z (%) = 331 (M⁺, 96), 302 (8), 260 (100), 247 (30), 232 (11), 190 (18), 176 (13), 128 (3), 105 (5), 88 (2), 77 (5), 55 (3), 41 (7). HR-MS (EI): calcd for C₂₃H₁₉NO₂ (M⁺): 331.15668, found: 331.15677.

5-Heptyl-5H-benzo[b]carbazole-6,11-dione (4o)

Starting from 1-n-heptylamine, following general procedure A, 4o was obtained as a yellow solid (46 mg, 44 %), mp = 106 - 108 °C. ¹H NMR (300 MHz, CDCl₃) δ = 8.05 - 8.43 (m, 1H), 8.26 - 8.12 (m, 2H), 7.78 - 7.62 (m, 2H), 7.51 - 7.34 (m, 3H), 4.70 (m, 2H, CH₂), 1.98 - 1.77 (m, 2H, CH₂), 1.47 - 1.26 (m, 8H, 4CH₂), 0.87 (t, J = 6.8 Hz, 3H, CH₃). ¹³C NMR (63 MHz, CDCl₃) δ = 181.3, 179.0 (C=O), 139.5, 134.9, 134.3 (C), 133.8 (CH), 133.8 (C), 132.9, 127.3, 126.6,
126.4, 124.6 (CH), 124.2 (C), 124.1 (CH), 119.1 (C), 111.2 (CH), 45.5 (CH₂), 31.8 (CH₃), 30.4 (CH₂), 29.1 (CH₂), 27.0 (CH₂), 22.7 (CH₂), 14.2 (CH). IR (ATR, cm⁻¹): δ = 2953 (m), 2918 (s), 2870 (m), 2854 (m), 1657 (vs), 1640 (s), 1614 (m), 1593 (s), 1514 (s), 1495 (s), 1475 (s), 1454 (s), 1433 (m), 1421 (m), 1400 (m), 1375 (m), 1362 (m), 1346 (m), 1315 (w), 1296 (w), 1286 (w), 1259 (s), 1244 (s), 1221 (s), 1190 (m), 1169 (m), 1155 (m), 1138 (m), 1128 (m), 1099 (w), 1088 (m), 1059 (m), 1043 (m), 1014 (m), 991 (w), 966 (s), 941 (m), 916 (m), 901 (w), 864 (m), 851 (m), 833 (w), 812 (w), 795 (m), 760 (w), 746 (vs), 725 (m), 712 (vs), 698 (m), 665 (m), 621 (m), 600 (m), 579 (m), 567 (w). MS (EI): m/z (%) = 345 (M⁺, 100), 302 (6), 260 (83), 247 (28), 232 (13), 190 (14), 128 (2), 105 (5), 88 (1), 77 (4), 55 (2), 41 (10). HRMS (ESI-TOF): calcd for C₂₅H₂₃NO₂ ([M+H⁺]: m/z= 346.18016, found 346.18013; calcd for C₂₅H₂₃NaNO₂ ([M+ Na⁺]): 368.1621, found 368.16218.

5-Octyl-5H-benzo[b]carbazole-6,11-dione (4p)

Starting from 1-n-octylamine, following general procedure A, 4p was obtained as a yellow solid (43 mg, 33 %), mp = 77 - 78 °C. ¹H NMR (250 MHz, CDCl₃) δ = 8.52 – 8.39 (m, 1H), 8.26 – 8.08 (m, 2H), 7.79 – 7.61 (m, 2H), 7.50 – 7.31 (m, 3H), 4.81 – 4.60 (m, 2H, CH₂), 1.96 – 1.77 (m, 2H, CH₃), 1.47 – 1.24 (m, 10H, 5CH₃), 0.93 – 0.81 (m, 3H, CH₃). ¹³C NMR (63 MHz, CDCl₃) δ = 181.3, 179.0 (C=O), 139.5, 134.9, 134.3 (C), 133.8 (CH), 133.8 (C), 132.9, 127.3, 126.6, 126.4, 124.6 (CH), 124.2 (C), 124.1 (CH), 119.1 (C), 111.2 (CH), 45.5 (CH₂), 31.9 (CH₂), 30.4 (CH₂), 29.4 (CH₂), 29.3 (CH₂), 27.1 (CH₂), 22.7 (CH₂), 14.2 (CH₃). IR (ATR, cm⁻¹): δ = 2955 (w), 2920 (m), 2854 (m), 1657 (s), 1641 (s), 1612 (w), 1595 (s), 1512 (s), 1493 (m), 1477 (s), 1454 (m), 1433 (w), 1421 (m), 1398 (m), 1377 (m), 1362 (m), 1344 (m), 1315 (w), 1294 (w), 1281 (w), 1259 (s), 1238 (s), 1219 (s), 1207 (m), 1186 (m), 1167 (m), 1153 (m), 1138 (m), 1128 (m), 1088 (w), 1061 (m), 1030 (m), 1018 (m), 1009 (m), 989 (w), 970 (m), 939 (m), 862 (w), 851 (w), 812 (w), 791 (m), 744 (vs), 725 (w), 709 (vs), 696 (s), 663 (m), 621 (m), 600 (m), 579 (m), 567 (w). MS (EI): m/z (%) = 359 (M⁺, 100), 330 (3), 274 (13), 260 (80), 247 (21), 232 (8), 190 (12), 128 (2), 105 (4), 77 (2), 41 (9). HR-MS (EI): calcd for C₂₅H₂₃NO₂ (M⁺): 359.18798, found: 359.18793.

5-Benzyl-5H-benzo[b]carbazole-6,11-dione (4q)

Starting from benzylamine, following general procedure A, 4q was obtained as a yellow solid (53mg, 52 %), mp = 175 - 177 °C. ¹H NMR (300 MHz, CDCl₃) δ = 8.55 – 8.46 (m, 1H), 8.28 – 8.19 (m, 1H), 8.17 – 8.12 (m, 1H), 7.78 – 7.62 (m, 2H), 7.49 – 7.38 (m, 3H), 7.32 – 7.26 (m, 2H), 7.25 – 7.17 (m, 3H), 6.01 (s, 2H, CH₂); ¹³C NMR (63 MHz, CDCl₃) δ = 181.5, 179.2 (C=O), 140.0, 136.7, 135.0, 134.3 (C), 134.1 (CH), 133.8 (C), 133.1 (CH), 129.1 (2CH), 128.0, 127.8 (CH), 126.9 (2CH), 126.8, 126.5, 124.9 (CH), 124.4 (C), 124.2 (CH), 119.7 (C), 111.7 (CH), 48.7 (CH₂). IR
(ATR, cm⁻¹): = 3305 (w), 3282 (w), 3086 (w), 3056 (w), 3029 (m), 3007 (w), 2922 (w), 2850 (w), 1658 (s), 1645 (vs), 1594 (m), 1585 (s), 1520 (s), 1493 (s), 1466 (s), 1452 (s), 1423 (m), 1396 (s), 1365 (m), 1341 (s), 1317 (m), 1310 (m), 1286 (m), 1263 (m), 1244 (s), 1220 (m), 1192 (s), 1166 (s), 1155 (m), 1130 (s), 1090 (m), 1059 (s), 1027 (m), 1015 (s), 994 (m), 975 (m), 940 (s), 900 (m), 875 (w), 864 (m), 840 (m), 813 (m), 794 (s), 742 (vs), 713 (vs), 698 (vs), 693 (vs), 666 (s), 658 (s), 625 (s), 598 (s), 584 (s), 564 (m), 550 (m). MS (EI, 70 eV): m/z (%) = 337 (M⁺, 100), 320 (5), 292 (3), 278 (3), 260 (6), 231 (3), 204 (3), 190 (6), 163 (4), 139 (3), 114 (2), 91 (98), 77 (4), 65 (11), 51 (2), 39 (3). HR-MS (EI): calcd for C₁₉H₁₈N₂O₂ (M⁺) 337.10973, found 337.10948.

5-Phenethyl-5H-benzo[b]carbazole-6,11-dione (4r)

Starting from 2-phenylethanamine, following general procedure A. 4r was obtained as a yellow solid (58 mg, 55 %), mp = 171 - 172 °C. ¹H NMR (250 MHz, CDCl₃) δ = 8.52 – 8.41 (m, 1H), 8.26 – 8.12 (m, 2H), 7.80 – 7.59 (m, 2H), 7.48 – 7.32 (m, 3H), 7.29 – 7.16 (m, 5H), 4.97 – 4.84 (m, 2H, CH₂), 3.23 – 3.11 (m, 2H, CH₂). ¹³C NMR (63 MHz, CDCl₃) δ = 181.4, 179.0 (C=O), 139.4, 137.9, 134.8, 134.3 (C), 133.9 (CH), 133.7 (C), 132.9 (CH), 129.1, 128.8 (2CH), 127.4, 127.0, 126.6, 126.4, 124.6 (CH), 124.1 (C), 124.1 (CH), 119.3 (C), 111.0 (CH), 47.0 (CH₂), 36.7 (CH₂). IR (ATR, cm⁻¹): = 3074 (m), 3055 (m), 3022 (m), 2958 (m), 2920 (m), 2848 (m), 1655 (s), 1643 (s), 1612 (m), 1587 (s), 1514 (s), 1491 (s), 1468 (s), 1450 (s), 1419 (s), 1398 (s), 1369 (m), 1358 (m), 1344 (s), 1321 (m), 1288 (m), 1275 (m), 1259 (s), 1232 (s), 1223 (s), 1207 (s), 1182 (s), 1167 (s), 1149 (m), 1132 (s), 1084 (m), 1057 (s), 1036 (m), 1028 (s), 1007 (m), 959 (s), 932 (m), 910 (m), 893 (m), 866 (m), 843 (m), 833 (m), 810 (m), 789 (s), 764 (m), 748 (s), 733 (vs), 719 (m), 708 (vs), 700 (vs), 665 (s), 623 (s), 604 (m), 581 (s), 563 (m), 536 (m). MS (EI, 70 eV): m/z (%) = 351 (M⁺, 33), 260 (100), 247 (14), 232 (4), 203 (6), 176 (9), 151 (3), 128 (2), 91 (4), 77 (4), 65 (2.36), 51 (2), 39 (1). HR-MS (EI): calcd for C₂₀H₁₇N₂O₂ (M⁺): 351.12538, found 351.12567

5-(3-Phenylpropyl)-5H-benzo[b]carbazole-6,11-dione (4s)

Starting from 3-phenylprop-1-amine, following general procedure A. 4s was obtained as a yellow solid (53 mg, 48 %), mp = 155 - 156 °C. ¹H NMR (250 MHz, CDCl₃) δ = 8.49 – 8.42 (m, 1H), 8.25 – 8.13 (m, 2H), 7.77 – 7.64 (m, 2H), 7.41 – 7.27 (m, 5H), 7.24 – 7.16 (m, 3H), 4.91 – 4.59 (m, 2H, CH₂), 2.92 – 2.64 (m, 2H, CH₂), 2.35 – 2.06 (m, 2H, CH₂). ¹³C NMR (63 MHz, CDCl₃) δ = 181.3, 179.0 (C=O), 140.9, 139.4, 134.9, 134.3 (C), 133.9 (CH), 133.7 (C), 132.9 (CH), 128.6, 128.4 (2CH), 127.4, 126.6, 126.4, 126.3, 124.6 (CH), 124.2 (C), 124.1 (CH), 119.2 (C), 111.1 (CH), 44.9, 33.2, 31.5 (CH₃). IR (ATR, cm⁻¹): = 3024 (w), 2960 (w), 2947 (w), 2931 (w), 2914 (w), 2850 (w), 1657 (vs), 1645 (s), 1614 (w), 1593 (m), 1576 (w), 1516 (s),
Starting from (4-fluorophenyl)methanamine, following general procedure A, 4t was obtained as a yellow solid (56 mg, 52 %), mp = 200 - 201 °C. ¹H NMR (250 MHz, CDCl₃) δ = 8.53 – 8.44 (m, 1H), 8.25 – 8.18 (m, 1H), 8.16 – 8.09 (m, 1H), 7.78 – 7.62 (m, 2H), 7.48 – 7.35 (m, 3H), 7.23 – 7.14 (m, 2H), 7.03 – 6.90 (m, 2H), 5.94 (s, 2H, CH₃). ¹³F NMR (235 MHz, CDCl₃) δ = -114.39. ¹³C NMR (63 MHz, CDCl₃) δ = 181.4, 179.1 (C=O), 162.4 (d, J = 246.5 Hz, CF), 139.7, 134.7, 134.2 (C), 134.0 (CH), 133.6 (C), 133.0 (CH), 132.4 (d, J = 3.2 Hz, C), 128.7 (d, J = 8.2 Hz, 2CH), 127.8, 126.7, 126.5, 124.8 (CH), 124.3 (C), 124.2 (CH), 119.6 (C), 115.9 (d, J = 21.7 Hz, 2CH), 111.4 (CH), 47.7 (CH₂). IR (ATR, cm⁻¹): ν = 3284 (w), 3066 (w), 3055 (w), 3003 (w), 1645 (s), 1595 (s); MS (EI, 70 eV): m/z (%) = 365 (M⁺, 72), 336 (2), 274 (23), 261 (100), 247 (6), 232 (9), 219 (5), 207 (4), 204 (10), 190 (9), 176 (8), 164 (4), 151 (2), 133 (2), 128 (2), 115 (4), 104 (4), 91 (12), 77 (5), 65 (6), 51 (3), 39 (3). HR-MS (EI): calcd for C₂₅H₁₆NFO₂ (M⁺) 365.14103, found 365.14055.

5-(4-Methoxybenzyl)-5H-benzol[b]carbazole-6,11-dione (4u)

Starting from (4-methoxyphenyl)methanamine, following general procedure A, 4u was obtained as a yellow solid (71 mg, 58 %), mp = 182 - 183 °C. ¹H-NMR (300 MHz, CDCl₃): δ = 8.50 – 8.44 (m, 1H), 8.20 (dd, J = 7.5 Hz, J = 1.3 Hz, 1H), 8.14 (dd, J = 7.4 Hz, J = 1.4 Hz, 1H), 7.61 – 7.75 (m, 2H), 7.51 – 7.34 (m, 3H), 7.10 (d, J = 5.8 Hz, 2H) 6.84 – 6.77 (m, 2H), 5.90 (s, 2H, CH₂), 3.73 (s, 3H, OCH₃). ¹³C NMR (75 MHz, CDCl₃) δ = 181.4, 179.1 (C=O), 159.2, 139.8, 134.8, 134.2 (C), 133.9 (CH), 133.7 (C), 132.9 (CH), 128.8 (C), 128.3 (2CH), 127.6, 126.7, 126.4, 124.7 (CH), 124.3 (C), 124.1 (CH), 119.5 (C), 114.3 (2CH), 111.6 (CH), 55.4 (OCH₃), 48.0

1495 (m), 1470 (s), 1454 (m), 1435 (w), 1421 (m), 1398 (m), 1367 (w), 1356 (m), 1344 (m), 1319 (w), 1284 (m), 1275 (w), 1263 (m), 1242 (s), 1221 (m), 1207 (s), 1167 (m), 1157 (w), 1128 (m), 1065 (m), 1057 (m), 1043 (w), 1030 (m), 1011 (m), 986 (m), 968 (w), 947 (m), 916 (w), 889 (w), 854 (w), 812 (w), 795 (w), 748 (s), 741 (vs), 712 (vs), 704 (vs), 667 (w), 642 (m), 598 (m), 586 (m), 573 (w). MS (EI, 70 eV): m/z (%) = 365 (M⁺, 72), 336 (2), 274 (23), 261 (100), 247 (6), 232 (9), 219 (5), 207 (4), 204 (10), 190 (9), 176 (8), 164 (4), 151 (2), 133 (2), 128 (2), 115 (4), 104 (4), 91 (12), 77 (5), 65 (6), 51 (3), 39 (3). HR-MS (EI): calcd for C₂₅H₁₆NFO₂ (M⁺) 356.10813, found 356.10823; calcd for C₂₅H₁₆NFO₂ ([M+ Na⁺]): m/z= 368.1621, found 378.09.
(CH₃). IR (ATR, cm⁻¹): δ = 3289 (w), 3052 (m), 2933 (m), 2840 (m), 1725 (w), 1651 (vs), 1612 (m), 1591 (s), 1584 (s), 1515 (vs), 1493 (s), 1466 (s), 1439 (s), 1420 (m), 1396 (s), 1365 (m), 1341 (s), 1316 (m), 1305 (m), 1286 (m), 1252 (s), 1239 (vs), 1189 (s), 1166 (s), 1132 (m), 1116 (m), 1089 (m), 1057 (s), 1026 (s), 1016 (s), 991 (s), 956 (m), 943 (m), 937 (m), 916 (s), 863 (m), 843 (m), 829 (m), 819 (s), 798 (m), 791 (m), 780 (m), 760 (m), 750 (s), 738 (s), 722 (m), 709 (vs), 695 (s), 660 (s), 636 (m), 619 (s), 598 (s), 581 (s), 563 (m), 540 (s). MS (EI, 70 eV): m/z (%) = 367 (M⁺, 37), 203 (2), 190 (7), 183 (3), 163 (3), 139 (2), 121 (100), 105 (2), 91 (6), 78 (8), 77 (9), 65 (2), 51 (3). HR-MS (EI): calcd for C₁₃H₁₀O₃N [(M+H)⁺] 368.12812, found 368.12771.

5-(3-(Trifluoromethyl)benzyl)-5H-benz[a]carbazole-6,11-dione (4v)

Starting from (3-(trifluoromethyl)phenyl)methanamine, following general procedure A, 4v was obtained as a yellow solid (72 mg, 59 %), mp = 198 - 199 °C. 

¹H NMR (300 MHz, CDCl₃) δ = 8.56 – 8.47 (m, 1H), 8.28 – 8.19 (m, 1H), 8.18 – 8.10 (m, 1H), 7.80 – 7.64 (m, 2H), 7.60 – 7.34 (m, 6H), 7.30 – 7.27 (m, 1H, overlap with solvent signal), 6.04 (s, 2H, CH₂). ¹³C NMR (282 MHz, CDCl₃) δ = -62.62. 

Starting from 2-(3,4-dimethoxyphenethyl)ethanamine, following general procedure A, 4w was obtained as a yellow solid (87 mg, 66 %), mp = 153 - 154 °C. 

¹H NMR (250 MHz, CDCl₃) δ = 8.51 – 8.24 (m, 1H), 8.20 – 7.86 (m, 2H), 7.73 – 7.45 (m, 2H), 7.41 – 7.06 (m, 3H), 6.74 – 6.43 (m, 3H), 5.05 – 4.48 (m, 2H, CH₂), 3.69 (s, 3H, CH₃), 3.67 (s, 3H, CH₃), 3.09 – 2.85 (m, 2H, CH₂). ¹³C NMR (63 MHz, CDCl₃) δ = 181.2, 178.8 (C=O), 149.1, 148.0, 139.4, 134.8, 134.1 (C), 133.8 (CH), 133.6 (C), 132.9 (CH), 130.4 (C), 127.3, 126.5, 126.3, 124.5 (CH), 124.0 (C), 123.9, 121.1 (CH), 119.1 (C), 112.3, 111.5, 111.0 (CH), 56.0, 55.9 (OCH₃),
47.0 (CH₃), 36.2 (CH₂). IR (ATR, cm⁻¹): δ = 3063 (w), 2997 (w), 2953 (m), 2918 (m), 2848 (m), 2827 (w), 1712 (vw), 1655 (s), 1643 (m), 1616 (w), 1605 (w), 1591 (m), 1539 (w), 1512 (s), 1495 (m), 1468 (s), 1462 (s), 1452 (s), 1437 (m), 1419 (m), 1398 (m), 1375 (m), 1363 (m), 1348 (m), 1329 (m), 1317 (w), 1294 (w), 1281 (m), 1257 (s), 1225 (s), 1196 (m), 1182 (s), 1171 (m), 1153 (s), 1138 (s), 1082 (m), 1063 (m), 1047 (m), 1028 (s), 984 (m), 960 (s), 933 (m), 891 (m), 864 (m), 851 (m), 839 (s), 810 (s), 795 (m), 789 (m), 771 (m), 756 (m), 741 (s), 708 (vs), 692 (s), 663 (s), 646 (m), 629 (m), 615 (m), 592 (m), 581 (s), 565 (m), 544 (m). MS (EI, 70 eV): m/z (%) = 411 (M⁺, 54), 260 (49), 247 (13), 203 (8), 176 (11), 164 (29), 151 (100), 107 (10), 91 (6), 77 (8), 65 (5), 51 (3). HR-MS (EI): calcd for C₁₄H₁₂O₂N (M⁺) 411.14651, found 411.14639.

2-Methyl-5-phenyl-5H-benzo[b]carbazole-6,11-dione (5b)

Starting from 4-methyl-N-phenylaniline, following general procedure B, the mixture 5b2 and 4b was obtained as a yellow solid (42 mg, 41%). A small amount of pure compound 5b2 was isolated by recrystallizing from heptane and ethyl acetate (5:1), then washed by cold ethyl acetate, (12 mg), mp = 301 - 302 °C. ¹H NMR (300 MHz, CDCl₃) δ = 8.34 - 8.22 (m, 2H), 8.07 - 8.01 (m, 1H), 7.77 - 7.56 (m, 5H), 7.47 - 7.41 (m, 2H), 7.25 - 7.18 (m, 1H), 7.06 (d, J = 8.6 Hz, 1H), 2.53 (s, 3H, CH₃). ¹³C NMR (75 MHz, CDCl₃) δ = 181.8, 177.7 (C=O), 139.8, 137.1, 135.0, 134.3 (C), 133.8 (CH), 133.8 (C), 133.0, 129.8 (CH), 129.6 (2CH), 129.3 (CH), 127.7 (2CH), 126.6 (CH), 126.5, 124.9 (C), 124.3 (CH), 123.1 (CH), 119.6 (C), 111.9 (CH), 21.7 (CH₃). IR (ATR, cm⁻¹): δ = 3061 (w), 2910 (w), 2858 (w), 1651 (s), 1590 (m), 1518 (s), 1487 (s), 1460 (m), 1417 (m), 1390 (m), 1373 (m), 1332 (m), 1223 (m), 1286 (m), 1265 (s), 1219 (s), 1176 (m), 1157 (m), 1138 (m), 1113 (w), 1093 (m), 1082 (w), 1070 (w), 1047 (m), 1030 (m), 1016 (m), 993 (m), 978 (w), 960 (m), 922 (m), 903 (m), 897 (m), 879 (m), 852 (w), 835 (w), 795 (s), 779 (m), 754 (m), 743 (s), 712 (vs), 692 (s), 673 (m), 662 (m), 619 (s), 600 (m), 586 (m), 557 (m). MS (EI, 70 eV): m/z (%) = 337 (M⁺, 100), 308 (7), 278 (9), 233 (1), 203 (3), 168 (12), 153 (3), 139 (4), 88 (1), 77 (4), 63 (1), 51 (4), 39 (1). HR-MS (EI): calcd for C₁₄H₁₄N₂O₂ (M⁺) 337.10973, found 337.10948.

2-Methyl-5-(p-tolyl)-5H-benzo[b]carbazole-6,11-dione (5c)

Starting from di-p-tolylamine, following general procedure B, 5c was obtained as a yellow solid (36 mg, 34%), mp = 301 - 302 °C. ¹H NMR (300 MHz, CDCl₃) δ = 8.33 - 8.28 (m, 1H), 8.26-8.21 (m, 1H), 8.09 - 8.02 (m, 1H), 7.68 (m, 2H), 7.40 (d, J = 8.1 Hz, 2H), 7.34 - 7.29 (m, 2H), 7.21 (dd, J = 8.6 Hz, J = 1.4 Hz, 1H), 7.06 (d, J = 8.6 Hz, 1H), 2.52, 2.51 (CH₃). ¹³C NMR (63 MHz, CDCl₃) δ = 181.9, 177.8 (C=O), 139.9, 139.4, 135.7, 135.1, 134.5, 134.4 (C), 133.4, 133.1 (CH), 130.4 (2CH), 130.0 (CH), 129.8 (C), 127.5 (2CH), 126.7, 126.5 (CH), 124.4 (C), 123.2 (CH), 119.6 (C), 112.1
(CH), 21.8, 21.6 (CH₃). IR (ATR, cm⁻¹): = 3063 (w), 2997 (w), 2933 (w), 2922 (m), 2854 (w), 2827 (m), 1655 (s), 1643 (s), 1616 (w), 1605 (w), 1591 (s), 1512 (s), 1495 (m), 1470 (s), 1462 (s), 1452 (s), 1437 (m), 1419 (s), 1398 (s), 1373 (m), 1350 (m), 1329 (m), 1317 (m), 1294 (w), 1281 (m), 1257 (s), 1234 (s), 1225 (s), 1196 (m), 1182 (s), 1171 (m), 1153 (s), 1138 (s), 1088 (m), 1063 (s), 1047 (m), 1026 (s), 960 (s), 935 (m), 891 (m), 866 (m), 851 (m), 839 (s), 810 (s), 795 (m), 789 (m), 771 (m), 758 (m), 741 (s), 708 (vs), 692 (s), 663 (s), 629 (s), 615 (m), 592 (s), 581 (s), 565 (m), 544 (s). MS (EI, 70 eV): m/z (%) = 351 (M⁺, 100), 336 (29), 322 (6), 294 (6), 278 (6), 234 (1), 203 (2), 168 (9), 151 (3), 132 (4), 91 (4), 76 (3), 65 (3), 50 (1), 39 (2). HRMS (ESI-TOF): calcd for C₂₉H₂₇NO₂ [(M+H)⁺]: m/z= 352.13321, found 352.13327; calcd for C₂₉H₂₇NaNO₂ [(M+ Na)⁺]: m/z= 374.11515, found 374.11509.

1-Methoxy-5-(2-methoxyphenyl)-5H-benzo[b]carbazole-6,11-dione (5d)

Starting from bis (2-methoxyphenyl)amine, following general procedure B, 5d was obtained as an orange solid (40 mg, 35 %), mp = 239 - 240 °C. ¹H NMR (300 MHz, CDCl₃) δ = 8.22 (d, ¹J= 7.5 Hz, 1H), 8.04 (dd, J = 7.5, 1.2 Hz, 1H), 7.88 (d, J = 1.8 Hz, 1H), 7.75 – 7.59 (m, 2H), 7.39 – 7.32 (m, 2H), 7.15 – 6.99 (m, 4H), 3.95, 3.93 (s, 3H, OCH₃). ¹³C NMR (75 MHz, CDCl₃) δ = 181.8, 177.5 (C=O), 160.0, 158.2, 136.8, 135.5, 134.3, 133.9 (C), 133.8, 133.0 (CH), 129.5 (C), 128.7 (2CH), 126.6, 126.3 (CH), 124.8 (C), 119.7 (CH), 119.3 (C), 114.8 (2CH), 113.4, 102.9 (CH), 56.0, 55.7 (OCH₃). IR (ATR, cm⁻¹): = 3074 (w), 3061 (w), 3014 (w), 2997 (w), 2951 (w), 2831 (w), 2550 (w), 2355 (w), 1649 (s), 1612 (m), 1589 (m), 1514 (s), 1491 (s), 1475 (s), 1464 (s), 1439 (m), 1423 (m), 1398 (m), 1344 (m), 1317 (m), 1302 (m), 1286 (m), 1250 (s), 1217 (s), 1205 (s), 1169 (s), 1157 (s), 1128 (m), 1109 (m), 1092 (m), 1084 (m), 1045 (s), 1026 (s), 1014 (s), 962 (s), 937 (m), 912 (m), 893 (m), 874 (m), 847 (s), 835 (s), 816 (m), 808 (s), 800 (s), 795 (s), 779 (s), 758 (m), 748 (s), 723 (m), 708 (s), 698 (s), 669 (m), 642 (m), 613 (s), 557 (s), 528 (m). MS (EI, 70 eV); m/z (%) = 383 (M⁺, 100), 368 (30), 340 (7), 297 (6), 269 (3), 240 (5), 192 (5), 176 (2), 155 (2), 126 (2), 107 (3), 92 (1), 77 (2), 63 (2), 50 (1). HRMS (EI): calcd for C₂₉H₂₇NO₄ (M⁺): m/z= 383.11521, found 383.11509;

Benzo[b]indolo[3,2,1-ij]carbazole-9,14-dione (5g)

Starting from carbazole, following general procedure B, 5g was obtained as a light red solid (29 mg, 28 %), mp = 251 - 253 °C. ¹H NMR (300 MHz, CDCl₃) δ = 8.64 (d, ³J= 8.1 Hz, 1H), 8.27 – 8.15 (m, 3H), 7.99 – 7.87 (m, 2H), 7.81 – 7.70 (m, 2H), 7.64 – 7.59 (m, 2H), 7.44 – 7.35 (m, 1H). ¹³C NMR (75 MHz, CDCl₃) δ = 181.7, 177.9 (C=O), 142.2, 139.4, 136.5 (C), 134.2 (CH), 133.9 (C), 133.3 (CH), 132.9, 131.6 (C), 128.4, 127.5, 126.9, 126.8 (CH), 126.3 (C), 125.3, 124.0, 123.3, 122.0 (CH), 121.0, 117.3 (C), 117.0 (CH). IR (ATR, cm⁻¹): = 3415 (m), 3304 (w), 3286 (w), 3061 (w), 3049 (w), 3030 (w), 3005 (w), 2920 (m), 2850 (m), 2679 (w), 1655 (s), 1593 (s), 1479 (s), 1471...
Starting from carbazole, following general procedure B, 6g was obtained as a black-violet solid (47 mg, 32 %), mp = 103 - 105 °C. $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ = 8.40 – 8.33 (m, 2H), 7.92 – 7.85 (m, 2H), 7.75 – 7.67 (m, 4H), 7.11 – 6.96 (m, 12H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$= 180.0 (C=C=O), 138.9 (4C), 136.7 (2C), 134.7 (2CH), 131.9 (2C), 127.6 (2CH), 125.7 (4CH), 124.8 (4C), 121.3 (4CH), 120.0 (4CH), 111.7 (4CH). IR (ATR, cm$^{-1}$): $\nu$ = 3045 (w), 3024 (w), 2924 (w), 2849 (w), 1666 (m), 1624 (w), 1591 (m), 1562 (m), 1489 (w), 1477 (m), 1448 (m), 1441 (m), 1371 (m), 1331 (m), 1311 (m), 1263 (s), 1223 (s), 1180 (m), 1159 (m), 1113 (m), 1101 (m), 1053 (w), 1038 (w), 1028 (m), 1018 (w), 993 (m), 935 (w), 930 (w), 901 (w), 847 (w), 824 (w), 808 (w), 797 (m), 791 (m), 770 (w), 744 (s), 719 (vs), 673 (m), 635 (w), 617 (m), 604 (s), 565 (m), 536 (m), 528 (m). MS (EI, 70 eV): m/z (%) = 490 ([M+2H]$^+$, 100), 489 ([M+H]$^+$, 9), 488 (M$^+$, 15), 403 (2), 353 (4), 322 (9), 245 (8), 207 (2), 167 (18), 91 (7), 69 (13), 43 (19).

HRMS (ESI-TOF): calcd for C$_{34}$H$_{32}$N$_2$O$_2$ ([M+H]$^+$): m/z= 489.15975, found 489.16001; calcd for C$_{34}$H$_{30}$NaN$_2$O$_2$ ([M+ Na]$^+$): m/z= 511.1417, found 489.1600.
NMR-Spectra

$^1$H NMR (300 MHz, CDCl$_3$) of 2-bromo-3- (phenylamino)naphthalene-1,4-dione (2b)

$^{13}$C NMR (75 MHz, CDCl$_3$) of 2-bromo-3- (phenylamino)naphthalene-1,4-dione (2b)
$^1$H NMR (250 MHz, CDCl$_3$) 2- (2-bromophenyl)-3- (phenylamino)naphthalene -1,4-dione (3b)

$^{13}$C NMR (63 MHz, CDCl$_3$) 2- (2-bromophenyl)-3- (phenylamino)naphthalene -1,4-dione (3b)
$^1$H NMR (250 MHz, CDCl$_3$) spectra of 5-phenyl-$5H$-benzo[$b$]carbazole-6,11-dione (4a)

$^{13}$C NMR (63 MHz, CDCl$_3$) spectra of 5-phenyl-$5H$-benzo[$b$]carbazole-6,11-dione (4a)
$^1$H NMR (250 MHz, CDCl$_3$) spectra of 5-$(p$-tolyl)-5$H$-benzo[b]carbazole-6,11-dione (4b)

$^{13}$C NMR (63 MHz, CDCl$_3$) spectra of 5-$(p$-tolyl)-5$H$-benzo[b]carbazole-6,11-dione (4b)
$^1$H NMR (300 MHz, CDCl$_3$) of 5-(4-fluorophenyl)-5H-benzo[\textit{b}]carbazole-6,11-dione (4c)

$^{13}$C NMR (126 MHz, CDCl$_3$) of 5-(4-fluorophenyl)-5H-benzo[\textit{b}]carbazole-6,11-dione (4c)
$^1$H NMR (300 MHz, DMSO) spectra of 5-(4-nitrophenyl)-5H-benzo[b]carbazole-6,11-dione (4d)

$^{13}$C NMR (63 MHz, DMSO) spectra of 5-(4-nitrophenyl)-5H-benzo[b]carbazole-6,11-dione (4d)
$^1$H NMR (300 MHz, CDCl$_3$) spectra of 5-(4-methoxyphenyl)-5H-benzo[b]carbazole-6,11-dione (4e)

$^{13}$C NMR (126 MHz, CDCl$_3$) spectra of 5-(4-methoxyphenyl)-5H-benzo[b]carbazole-6,11-dione (4e)
$^1$H NMR (300 MHz, CDCl$_3$) spectra of 5-(3,5-dimethylphenyl)-5H-benzo[b]carbazole-6,11-dione (4f)

$^{13}$C NMR (75 MHz, CDCl$_3$) spectra of 5-(3,5-dimethylphenyl)-5H-benzo[b]carbazole-6,11-dione (4f)
$^1$H NMR (300 MHz, CDCl$_3$)  5-(4-$(\text{tert}-\text{butyl})$phenyl)-$5H$-benzo[b]-carbazole-6,11-dione (4g)

$^{13}$C NMR (75 MHz, CDCl$_3$) of 5-(4-$(\text{tert}-\text{butyl})$phenyl)-$5H$-benzo[b]-carbazole-6,11-dione (4g)
$^1$H NMR (300 MHz, CDCl$_3$) of 5-butyl-5$H$-benzo[b]carbazole-6,11-dione (4k)

$^{13}$C NMR (63 MHz, CDCl$_3$) of 5-butyl-5$H$-benzo[b]carbazole-6,11-dione (4k)
$^1$H NMR (300 MHz, CDCl$_3$) 5-pentyl-5H-benzo[b]carbazole-6,11-dione (4l)

13C NMR (75 MHz, CDCl$_3$) 5-pentyl-5H-benzo[b]carbazole-6,11-dione (4l)
$^1$H NMR (250 MHz, CDCl$_3$) spectra of 5-hexyl-$5H$-benzo[b]carbazole-6,11-dione ($4m$)

$^{13}$C NMR (63 MHz, CDCl$_3$) spectra of 5-hexyl-$5H$-benzo[b]carbazole-6,11-dione ($4m$)
$^1$H NMR (300 MHz, CDCl$_3$) of 5-heptyl-$5H$-benzo[b]carbazole-6,11-dione (4o)

$^{13}$C NMR (63 MHz, CDCl$_3$) of 5-heptyl-$5H$-benzo[b]carbazole-6,11-dione (4o)
$^1$H NMR (250 MHz, CDCl$_3$) of 5-octyl-5$H$-benzo[b]carbazole-6,11-dione (4p)

$^{13}$C NMR (63 MHz, CDCl$_3$) of 5-octyl-5$H$-benzo[b]carbazole-6,11-dione (4p)
$^1$H NMR (300 MHz, CDCl$_3$) of 5-benzyl-5H-benzo[b]carbazole-6,11-dione (4q)

$^{13}$C NMR (63 MHz, CDCl$_3$) of 5-benzyl-5H-benzo[b]carbazole-6,11-dione (4q)
$^1$H NMR (250 MHz, CDCl$_3$) of 5-phenethyl-5H-benzo[b]carbazole-6,11-dione (4r)

$^{13}$C NMR (63 MHz, CDCl$_3$) of 5-phenethyl-5H-benzo[b]carbazole-6,11-dione (4r)
$^1$H NMR (250 MHz, CDCl$_3$) of 5-(3-phenylpropyl)-5$H$-benzo[b]carbazole-6,11-dione (4s)

$^{13}$C NMR (63 MHz, CDCl$_3$) of 5-(3-phenylpropyl)-5$H$-benzo[b]carbazole-6,11-dione (4s)
$^1$H NMR (250 MHz, CDCl$_3$) of 5-(4-fluorobenzyl)-$5H$-benzo[\textit{b}]carbazole-6,11-dione (41)

$^{13}$C NMR (63 MHz, CDCl$_3$) of 5-(4-fluorobenzyl)-$5H$-benzo[\textit{b}]carbazole-6,11-dione (41)
$^1$H-NMR (300 MHz, CDCl$_3$) of 5-(4-methoxybenzyl)-5H-benzo[b]carbazole-6,11-dione (4u)

$^{13}$C NMR (75 MHz, CDCl$_3$) of 5-(4-methoxybenzyl)-5H-benzo[b]carbazole-6,11-dione (4u)
$^1$H NMR (300 MHz, CDCl$_3$) of 5-((3-(trifluoromethyl)benzyl)-5$H$-benzo[b] carbazole-6,11-dione (4v)

$^{13}$C NMR (75 MHz, CDCl$_3$) of 5-((3-(trifluoromethyl)benzyl)-5$H$-benzo[b] carbazole-6,11-dione (4v)
$^1$H NMR (250 MHz, CDCl$_3$) of 5-(3,4-dimethoxypheneylethyl)-5$H$-benzo$[b]$ carbazole-6,11-dione (4w)

$^{13}$C NMR (63 MHz, CDCl$_3$) of 5-(3,4-dimethoxypheneylethyl)-5$H$-benzo$[b]$ carbazole-6,11-dione (4w)
$^1$H NMR (300 MHz, CDCl$_3$) of 2-methyl-5-phenyl-$5H$-benzo[$b$]carbazole-6,11-dione (5b)

$^{13}$C NMR (75 MHz, CDCl$_3$) of 2-methyl-5-phenyl-$5H$-benzo[$b$]carbazole-6,11-dione (5b)
$^1$H NMR (300 MHz, CDCl$_3$) of 2-methyl-5-($p$-tolyl)-5$H$-benzo[$b$]carbazole-6,11-dione (5c)

$^{13}$C NMR (63 MHz, CDCl$_3$) of 2-methyl-5-($p$-tolyl)-5$H$-benzo[$b$]carbazole-6,11-dione (5c)
$^1$H NMR (300 MHz, CDCl$_3$) of 1-methoxy-5-(2-methoxyphenyl)-5$H$-benzo[b] carbazole-6,11-dione (5d)

$^{13}$C NMR (75 MHz, CDCl$_3$) of 1-methoxy-5-(2-methoxyphenyl)-5$H$-benzo[b] carbazole-6,11-dione (5d)
$^1$H NMR (300 MHz, CDCl$_3$) of Benzo[$b$]indolo[3,2,1-$jk$]carbazole-9,14-dione (5g)

$^{13}$C NMR (75 MHz, CDCl$_3$) of Benzo[$b$]indolo[3,2,1-$jk$]carbazole-9,14-dione (5g)
$^1$H NMR (300 MHz, CDCl$_3$) of 2,3-di (9H-carbazol-9-yl)naphthalene-1,4-dione (6g)

$^{13}$C NMR (75 MHz, CDCl$_3$) of 2,3-di (9H-carbazol-9-yl)naphthalene-1,4-dione (6g)