Supporting Information
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Remarkably mild and efficient CTAB-catalyzed Friedel-Crafts amidoalkylation of sesamols with in situ generated N-Boc-imines in aqueous medium: synthesis of novel 6-amidoalkyl sesamols

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1. General Methods.

N-Boc α-amido sulfones 1 were prepared according to literature procedures.\(^1\) Other reagents were obtained from commercial sources and were used without further purification. All reactions were conducted in a closed system with an atmosphere of air and were monitored by TLC. \(^1^H\) and \(^1^3^C\) NMR spectra were performed on a Brucker-300 MHz spectrometer for products dissolved by DMSO-\(d_6\) with tetramethylsilane (TMS) as an internal standard. Melting points were recorded on a Buchi Melting Point B-545.

2. General experimental procedure for the Friedel-Crafts amidoalkylation

N-Boc α-amido sulfone (0.2 mmol, 1.0 equiv), Na\(_2\)CO\(_3\) (0.3 mmol, 1.5 equiv), CTAB (0.02 mmol, 10%) and 2 mL of water was put in a 10 mL glass vial equipped with a small magnetic stirring bar. To the solution was added sesamol (0.24 mmol, 1.2eq). After stirring for the stipulated time at 30°C, the mixture was diluted with water (3 mL) and extracted with ethyl ether (3 ×25 mL). The organic layers were combined, dried over anhydrous sodium sulfate and concentrated under reduced pressure. The residue was subjected to silica gel flash chromatography (Ethyl acetate / Hexanes = 1 / 10) to give the pure product.

Tert-butyl (6-hydroxybenzo[d][1,3]dioxol-5-yl)(phenyl)methylcarbamate (3a)

Yield: 97%; White solid; m.p.: 168.5-168.9 °C; \(^1^H\) NMR (300 MHz, d\(_6\)-DMSO): \(\delta\) 9.31 (s, 1H), 7.61 (d, 1H, \(J = 9.6\) Hz), 7.29-7.22 (m, 4H), 7.19-7.14 (m, 1H), 6.92 (s, 1H), 6.42 (s, 1H), 6.11 (d, 1H, \(J = 9.6\) Hz), 5.88 (s, 1H, \(J = 0.6\) Hz), 5.84 (s, 1H, \(J = 0.6\) Hz), 1.39 (s, 9H); \(^1^3^C\) NMR (75 MHz, d\(_6\)-DMSO): \(\delta\) 154.99, 148.49, 146.09, 143.67, 139.83, 128.03, 126.63, 126.36, 121.09, 107.39, 100.60, 97.39, 77.99, 51.21, 28.28; HRMS (ESI) Calcd for C\(_{19}\)H\(_{21}\)NNaO\(_5\) (M+Na): 366.1317; Found: 366.1312.

Tert-butyl (6-hydroxybenzo[d][1,3]dioxol-5-yl)(p-tolyl)methylcarbamate (3b)

Yield: 95%; White solid; m.p.: 180.8-181.6 °C; \(^1^H\) NMR (300 MHz, d\(_6\)-DMSO): \(\delta\) 9.25 (s, 1H), 7.56 (d, 1H, \(J = 9.6\) Hz), 7.12-7.04 (m, 4H), 6.89 (s, 1H), 6.39 (s, 1H), 6.04 (d, 1H, \(J = 9.6\) Hz), 5.88 (s, 1H, \(J = 0.6\) Hz), 5.84 (s, 1H, \(J = 0.6\) Hz), 2.23 (s, 3H), 1.38 (s, 9H); \(^1^3^C\) NMR (75 MHz, d\(_6\)-DMSO): \(\delta\) 154.94, 148.41, 145.97, 140.69, 139.76, 135.32, 128.54, 126.55, 121.29, 107.35, 100.55, 97.33, 77.90, 50.89, 28.28, 20.58; HRMS (ESI) Calcd for C\(_{20}\)H\(_{23}\)NNaO\(_5\) (M+Na): 380.1474; Found: 380.1468.
Tert-butyl (6-hydroxybenzo[d][1,3]dioxol-5-yl)(m-tolyl)methylcarbamate (3c)
Yield: 90%; White solid; m.p.: 78.1-79.7 °C; \(^1\)H NMR (300 MHz, \(d_6\)-DMSO): \(\delta\) 9.29 (s, 1H), 7.58 (d, 1H, \(J = 9.6\) Hz), 7.15 (t, 1H, \(J = 7.5\) Hz), 7.05-6.97 (m, 3H), 6.91 (s, 1H), 6.41 (s, 1H), 6.06 (d, 1H, \(J = 9.6\) Hz), 5.88 (s, 1H, \(J = 0.6\) Hz), 5.85 (s, 1H, \(J = 0.6\) Hz), 2.25 (s, 3H), 1.39 (s, 9H); \(^{13}\)C NMR (75 MHz, \(d_6\)-DMSO): \(\delta\) 154.97, 148.45, 146.04, 143.65, 139.79, 136.98, 127.96, 127.24, 127.00, 123.79, 121.21, 107.40, 100.59, 97.37, 77.95, 51.16, 28.29, 21.16; HRMS (ESI) Calcd for C\(_{20}\)H\(_{23}\)NNaO\(_5\) (M+Na): 380.1474; Found: 380.1468.

Tert-butyl (6-hydroxybenzo[d][1,3]dioxol-5-yl)(4-methoxyphenyl)methylcarbamate (3d)
Yield: 91%; White solid; m.p.: 82.5-85.8 °C; \(^1\)H NMR (300 MHz, \(d_6\)-DMSO): \(\delta\) 9.24 (s, 1H), 7.54 (d, 1H, \(J = 9.6\) Hz), 7.13 (d, 2H, \(J = 8.7\) Hz), 6.91 (s, 1H), 6.82 (d, 2H, \(J = 8.7\) Hz), 6.40 (s, 1H), 6.03 (d, 1H, \(J = 9.6\) Hz), 5.88 (s, 1H, \(J = 0.6\) Hz), 5.85 (s, 1H, \(J = 0.6\) Hz), 3.70 (s, 3H), 1.38 (s, 9H); \(^{13}\)C NMR (75 MHz, \(d_6\)-DMSO): \(\delta\) 157.83, 154.92, 148.38, 145.95, 139.77, 135.67, 127.96, 127.24, 127.00, 123.79, 121.21, 107.40, 100.54, 97.35, 77.89, 55.02, 50.69, 28.27; HRMS (ESI) Calcd for C\(_{20}\)H\(_{23}\)NNaO\(_6\) (M+Na): 396.1423; Found: 396.1418.

Tert-butyl (6-hydroxybenzo[d][1,3]dioxol-5-yl)(2-methoxyphenyl)methylcarbamate (3e)
Yield: 85%; White solid; m.p.: 185.3-186.2 °C; \(^1\)H NMR (300 MHz, \(d_6\)-DMSO): \(\delta\) 9.16 (s, 1H), 7.22-7.15 (m, 3H), 6.93-6.85 (m, 2H), 6.62 (s, 1H), 6.39 (s, 1H), 6.24 (d, 1H, \(J = 9.6\) Hz), 5.86 (s, 1H, \(J = 0.6\) Hz), 5.84 (d, 1H, \(J = 0.6\) Hz), 3.72 (s, 3H), 1.38 (s, 9H); \(^{13}\)C NMR (75 MHz, \(d_6\)-DMSO): \(\delta\) 156.53, 154.64, 149.14, 146.02, 139.29, 130.99, 127.90, 127.67, 120.39, 119.88, 110.90, 107.97, 100.52, 97.41, 77.65, 55.43, 47.14, 28.29; HRMS (ESI) Calcd for C\(_{20}\)H\(_{23}\)NNaO\(_6\) (M+Na): 396.1423; Found: 396.1418.
Tert-butyl (4-fluorophenyl)(6-hydroxybenzo[d][1,3]dioxol-5-yl)methylcarbamate (3f) Yield: 95%; White solid; m.p.: 111.6-112.4 °C; $^1$H NMR (300 MHz, $d_6$-DMSO): $\delta$ 9.33 (s, 1H), 7.63 (d, 1H, $J = 9.3$ Hz), 7.26 (q, 2H, $J_1 = 8.4$ Hz, $J_2 = 5.7$ Hz), 7.09 (t, 2H, $J = 8.8$ Hz), 6.93 (s, 1H), 6.43 (s, 1H), 6.09 (d, 1H, $J = 9.3$ Hz), 5.89 (d, 1H, $J = 0.6$ Hz), 5.85 (d, 1H, $J = 0.6$ Hz), 1.39 (s, 9H); $^{13}$C NMR (75 MHz, $d_6$-DMSO): $\delta$ 162.49, 159.28, 154.96, 148.78, 146.20, 139.90, 139.83, 139.79, 128.58, 128.48, 120.90, 114.85, 114.57, 107.18, 100.63, 97.46, 78.09, 50.75, 28.25; HRMS (ESI) Calcd for C$_{19}$H$_{20}$FNNaO$_5$ (M+Na)$^+$: 384.1223; Found: 384.1218.

Tert-butyl (3-fluorophenyl)(6-hydroxybenzo[d][1,3]dioxol-5-yl)methylcarbamate (3g) Yield: 91%; White solid; m.p.: 145.6-146.8 °C; $^1$H NMR (300 MHz, $d_6$-DMSO): $\delta$ 9.40 (s, 1H), 7.70 (d, 1H, $J = 9.6$ Hz), 7.31 (q, 1H, $J_1 = 14.4$ Hz, $J_2 = 7.5$ Hz), 7.08 (d, 1H, $J = 7.8$ Hz), 7.03-6.97 (m, 2H), 6.92 (s, 1H), 6.43 (s, 1H), 6.12 (d, 1H, $J = 9.6$ Hz), 5.89 (d, 1H, $J = 0.7$ Hz), 5.85 (d, 1H, $J = 0.7$ Hz), 1.39 (s, 9H); $^{13}$C NMR (75 MHz, $d_6$-DMSO): $\delta$ 163.72, 160.50, 154.98, 148.49, 146.81, 146.72, 146.32, 139.94, 130.09, 129.98, 122.75, 122.71, 120.46, 113.27, 113.02, 112.98, 107.15, 100.69, 97.46, 78.20, 50.85, 28.25; HRMS (ESI) Calcd for C$_{19}$H$_{20}$FNNaO$_5$ (M+Na)$^+$: 384.1223; Found: 384.1218.

Tert-butyl (4-chlorophenyl)(6-hydroxybenzo[d][1,3]dioxol-5-yl)methylcarbamate (3h) Yield: 92%; White solid; m.p.: 81.2-82.8 °C; $^1$H NMR (300 MHz, $d_6$-DMSO): $\delta$ 9.33 (s, 1H), 7.65 (d, 1H, $J = 9.6$ Hz), 7.33 (d, 2H, $J = 8.4$ Hz), 7.22 (d, 2H, $J = 8.4$ Hz), 6.90 (s, 1H), 6.40 (s, 1H), 6.06 (d, 1H, $J = 9.6$ Hz), 5.88 (d, 1H, $J = 0.6$ Hz), 5.85 (d, 1H, $J = 0.6$ Hz), 1.38 (s, 9H); $^{13}$C NMR (75 MHz, $d_6$-DMSO): $\delta$ 154.58, 148.42, 146.19, 142.57, 139.82, 130.89, 128.41, 127.91, 120.44, 107.11, 100.57, 97.38, 78.06, 50.75, 28.17; HRMS (ESI) Calcd. for C$_{19}$H$_{20}$ClNNaO$_5$ (M+Na)$^+$: 400.0928; Found: 400.0922.
Tert-butyl (4-bromophenyl)(6-hydroxybenzo[d][1,3]dioxol-5-yl)methylcarbamate (3i) Yield: 96%; White solid; m.p.: 92.3-94.5 °C; $^1$H NMR (300 MHz, d$_6$-DMSO): δ 9.36 (s, 1H), 7.64 (d, 1H, $J = 9.0$ Hz), 7.46 (d, 2H, $J = 8.4$ Hz), 7.18 (d, 2H, $J = 8.4$ Hz), 6.90 (s, 1H), 6.42 (s, 1H), 6.06 (d, 1H, $J = 9.0$ Hz), 5.89 (d, 1H, $J = 0.6$ Hz), 5.85 (d, 1H, $J = 0.6$ Hz), 1.38 (s, 9H); $^{13}$C NMR (75 MHz, d$_6$-DMSO): δ 154.95, 148.51, 146.28, 143.10, 139.90, 130.93, 128.89, 120.45, 119.47, 107.19, 100.66, 97.45, 78.15, 50.87, 28.24; HRMS (ESI) Calcd for C$_{19}$H$_{20}$BrNNaO$_5$ (M+Na)$^+$: 444.0423; Found: 444.0417.

Tert-butyl (3-bromophenyl)(6-hydroxybenzo[d][1,3]dioxol-5-yl)methylcarbamate (3j) Yield: 97%; White solid; m.p.: 85.5-87.7 °C; $^1$H NMR (300 MHz, d$_6$-DMSO): 9.41 (s, 1H), 7.69 (d, 1H, $J = 9.6$ Hz), 7.41-7.36 (m, 2H), 7.25-7.23 (m, 2H), 6.92 (s, 1H), 6.43 (s, 1H), 6.09 (d, 1H, $J = 9.6$ Hz), 5.89 (d, 1H, $J = 0.6$ Hz), 5.86 (d, 1H, $J = 0.6$ Hz), 1.38 (s, 9H); $^{13}$C NMR (75 MHz, d$_6$-DMSO): δ 154.97, 148.49, 146.50, 146.38, 139.97, 130.34, 129.29, 129.20, 125.80, 121.47, 120.30, 107.10, 100.71, 97.50, 78.26, 50.94, 28.23; HRMS (ESI) Calcd for C$_{19}$H$_{20}$BrNNaO$_5$ (M+Na)$^+$: 444.0423; Found: 444.0417.

Tert-butyl (2-bromophenyl)(6-hydroxybenzo[d][1,3]dioxol-5-yl)methylcarbamate (3k) Yield: 90%; White solid; m.p.: 100.8-103.0 °C; $^1$H NMR (300 MHz, d$_6$-DMSO): δ 9.28 (s, 1H), 7.56 (d, 1H, $J = 7.8$ Hz), 7.46 (d, 1H, $J = 8.4$ Hz), 7.33 (t, 1H, $J = 7.2$ Hz), 7.30 (d, 1H, $J = 7.2$ Hz), 7.20-7.14 (m, 1H), 6.43 (d, 2H, $J = 8.4$ Hz), 6.20 (d, 1H, $J = 8.4$ Hz), 5.88 (s, 1H, $J = 0.6$ Hz), 5.86 (s, 1H, $J = 0.6$ Hz), 1.37 (s, 9H); $^{13}$C NMR (75 MHz, d$_6$-DMSO) δ 154.57, 149.55, 146.41, 141.99, 139.34, 132.59, 128.94, 128.69, 127.36, 123.41, 119.04, 107.74, 100.65, 97.41, 77.81, 51.82, 28.28; HRMS (ESI) Calcd for C$_{19}$H$_{20}$BrNNaO$_5$ (M+Na)$^+$: 444.0423; Found: 444.0417.
Tert-butyl(6-hydroxybenzo[d][1,3]dioxol-5-yl)(3-(trifluoromethyl)phenyl)methyl carbamate (3l)
Yield: 88%; White solid; m.p.: 75.5-76.1 °C; 1H NMR (300 MHz, d$_6$-DMSO): δ 9.45 (s, 1H), 7.79 (d, 1H, $J = 9.6$ Hz), 7.59-7.51 (m, 4H), 6.94 (s, 1H), 6.43 (s, 1H), 6.17 (d, 1H, $J = 9.6$ Hz), 5.88 (s, 1H, $J = 0.6$ Hz), 5.86 (s, 1H, $J = 0.6$ Hz), 1.39 (s, 9H); 13C NMR (75 MHz, d$_6$-DMSO): δ 155.02, 148.56, 146.45, 145.08, 139.99, 130.81, 130.48, 129.71, 129.50, 129.20, 129.09, 128.67, 128.26, 126.10, 123.24, 123.19, 122.90, 122.85, 122.80, 122.75, 122.49, 107.06, 100.71, 97.52, 78.32, 51.19, 28.18; HRMS (ESI) Calcd for C$_{20}$H$_{20}$F$_3$NNaO$_5$ (M+Na)$^+$: 434.1191; Found: 434.1186.

Tert-butyl (6-hydroxybenzo[d][1,3]dioxol-5-yl)(naphthen-1-yl)methyl-carbamate (3m)
Yield: 93%; White solid; m.p.: 111.8-113.3 °C; 1H NMR (300 MHz, d$_6$-DMSO): δ 9.43 (s, 1H), 8.12 (d, 1H, $J = 5.4$ Hz), 7.91 (dd, 1H, $J_1 = 5.4$ Hz, $J_2 = 1.5$ Hz), 7.80 (d, 1H, $J = 5.4$ Hz), 7.68 (d, 1H, $J = 9.0$ Hz), 7.51-7.37 (m, 4H), 6.81 (d, 1H, $J = 9.0$ Hz), 6.70 (s, 1H), 6.48 (d, 2H, $J = 4.8$ Hz), 5.89 (s, 1H, $J = 0.6$ Hz), 5.84 (s, 1H, $J = 0.6$ Hz), 1.39 (s, 9H); 13C NMR (75 MHz, d$_6$-DMSO): δ 154.81, 148.60, 146.54, 139.59, 139.40, 133.40, 130.86, 128.53, 127.24, 126.00, 125.55, 125.46, 123.75, 123.52, 120.13, 108.11, 100.64, 97.44, 77.91, 47.67, 28.28; HRMS (ESI) Calcd for C$_{23}$H$_{23}$NNaO$_5$ (M+Na)$^+$: 416.1474; Found: 416.1468.

Tert-butyl furan-2-yl(6-hydroxybenzo[d][1,3]dioxol-5-yl)methylcarbamate (3n)
Yield: 91%; White solid; m.p.: 168.5-168.9 °C; 1H NMR (300 MHz, d$_6$-DMSO): δ 9.33 (s, 1H), 7.62 (d, 1H, $J = 9.0$ Hz), 7.52 (s, 1H), 6.88 (s, 1H), 6.42 (s, 1H), 6.31 (s, 1H), 6.10 (d, 1H, $J = 9.0$ Hz), 5.90 (s, 1H), 5.89 (d, 1H, $J = 0.6$ Hz), 5.87 (d, 1H, $J = 0.6$ Hz), 1.38 (s, 9H); 13C NMR (75 MHz, d$_6$-DMSO): δ 155.69, 154.84, 148.77, 146.49, 141.90, 139.73, 118.50, 110.23, 107.56, 106.16, 100.67, 97.33, 78.11, 45.73, 28.26; HRMS (ESI) Calcd for C$_{17}$H$_{19}$NNaO$_6$ (M+Na)$^+$: 356.1100; Found: 356.1105.
Tert-butyl (6-hydroxybenzo[d][1,3]dioxol-5-yl)(thiophen-2-yl)methylcarbamate (3o) Yield: 85%; White solid; m.p.: 171.2-172.6 °C; \(^1\)H NMR (300 MHz, d\(_6\)-DMSO): \(\delta\) 9.38 (s, 1H), 7.78 (d, 1H, \(J = 9.6\) Hz), 7.31 (d, 1H, \(J = 4.5\) Hz), 6.98 (s, 1H), 6.88 (dd, 1H, \(J_1 = 4.8\) Hz, \(J_2 = 3.6\) Hz), 6.67 (d, 1H, \(J = 3.0\) Hz), 6.43 (s, 1H), 6.27 (d, 1H, \(J = 9.6\) Hz), 5.90 (d, 1H, \(J = 0.6\) Hz), 5.88 (d, 1H, \(J = 0.6\) Hz), 1.39 (s, 9H); \(^{13}\)C NMR (75 MHz, d\(_6\)-DMSO): \(\delta\) 154.80, 148.44, 148.08, 146.40, 139.78, 126.47, 124.33, 123.96, 120.52, 107.23, 100.64, 97.31, 78.16, 47.32, 28.22; HRMS (ESI) Calcd for C\(_{17}\)H\(_{19}\)NNaO\(_5\)S (M+Na): 372.0882; Found: 372.0876.

Tert-butyl 1-(6-hydroxybenzo[d][1,3]dioxol-5-yl)-2-methylpropylcarbamate (3p) Yield: 93%; White solid; m.p.: 88.5-89.8 °C; \(^1\)H NMR (300 MHz, d\(_6\)-DMSO): \(\delta\) 7.60 (d, 1H, \(J = 9.9\) Hz), 6.77 (d, 1H, \(J = 8.4\) Hz), 6.63 (d, 1H, \(J = 2.4\) Hz), 6.41 (dd, 1H, \(J_1 = 8.4\) Hz, \(J_2 = 2.4\) Hz), 5.93 (d, 1H, \(J = 0.6\) Hz), 5.92 (d, 1H, \(J = 0.6\) Hz), 5.07 (dd, 1H, \(J_1 = 8.7\) Hz, \(J_2 = 7.8\) Hz), 1.95 (q, 1H, \(J = 6.9\) Hz), 1.35 (s, 9H), 0.94 (d, 3H, \(J = 6.9\) Hz), 0.88 (d, 3H, \(J = 6.9\) Hz); \(^{13}\)C NMR (75 MHz, d\(_6\)-DMSO): \(\delta\) 155.17, 152.28, 147.69, 141.47, 108.53, 107.96, 100.93, 99.44, 85.18, 78.35, 41.79, 28.29, 28.07, 27.58, 25.85, 25.24, 25.15; HRMS (ESI) Calcd for C\(_{16}\)H\(_{23}\)NO\(_3\) M+: 309.1576; Found: 309.1571.

Tert-butyl cyclohexyl(6-hydroxybenzo[d][1,3]dioxol-5-yl)methylcarbamate (3q) Yield: 95%; White solid; m.p.: 107.0-108.9 °C; \(^1\)H NMR (300 MHz, d\(_6\)-DMSO): \(\delta\) 7.58 (d, 1H, \(J = 9.9\)Hz), 6.76 (d, 1H, \(J = 8.4\) Hz), 6.63 (d, 1H, \(J = 2.4\) Hz), 6.41 (dd, 1H, \(J_1 = 8.4\) Hz, \(J_2 = 2.4\) Hz), 5.93-5.88 (m, 2H), 5.08 (dd, 1H, \(J_1 = 8.7\) Hz, \(J_2 = 7.8\) Hz), 1.88 (d, 1H, \(J = 12.0\) Hz), 1.68-1.37 (m, 6H), 1.35 (s, 9H), 1.25-0.94 (m, 4H); \(^{13}\)C NMR (75 MHz, d\(_6\)-DMSO): \(\delta\) 155.17, 152.28, 147.69, 141.47, 108.53, 107.96, 100.93, 99.44, 85.18, 78.35, 41.79, 28.29, 28.07, 27.58, 25.85, 25.24, 25.15; HRMS (ESI) Calcd for C\(_{19}\)H\(_{27}\)NNaO\(_5\) (M+Na): 372.1787; Found: 372.1781.
Tert-butyl (6-hydroxy-7-methylbenzo[d][1,3]dioxol-5-yl)(phenyl)methylcarbamate (3r) Yield: 83%; White solid; m.p.: 88.2-90.9 °C; \(^1\)H NMR (300 MHz, \(d_6\)-DMSO): \(\delta\) 8.28 (s, 1H), 7.63 (d, 1H, \(J = 9.6\) Hz), 7.29-7.14 (m, 5H), 6.77 (s, 1H), 6.20 (s, 1H), 5.87 (d, 1H, \(J = 0.6\) Hz), 5.85 (d, 1H, \(J = 0.6\) Hz), 2.04 (s, 3H), 1.39 (s, 9H); \(^{13}\)C NMR (75 MHz, \(d_6\)-DMSO) \(\delta\) 155.05, 146.49, 144.74, 143.69, 139.78, 128.02, 126.74, 126.34, 123.01, 108.23, 104.47, 100.40, 77.99, 51.69, 28.28, 9.63; HRMS (ESI) Calcd for C\(_{20}\)H\(_{23}\)NNaO\(_5\) (M+Na)\(^+\): 380.1474; Found: 380.1468.

Tert-butyl (6-hydroxy-7-iodobenzo[d][1,3]dioxol-5-yl)(phenyl)methylcarbamate (3s) Yield: 95%; White solid; m.p.: 127.3-128.9 °C; \(^1\)H NMR (300 MHz, \(d_6\)-DMSO): \(\delta\) 8.70 (s, 1H), 7.70 (d, 1H, \(J = 9.3\) Hz), 7.31-7.16 (m, 5H), 6.88 (s, 1H), 6.23 (d, 1H, \(J = 9.3\) Hz), 6.00 (d, 1H, \(J = 0.6\) Hz), 5.96 (d, 1H, \(J = 0.6\) Hz), 1.39 (s, 9H); \(^{13}\)C NMR (75 MHz, \(d_6\)-DMSO) \(\delta\) 155.01, 148.42, 147.45, 143.00, 139.83, 128.11, 126.75, 126.56, 124.27, 107.07, 100.53, 78.16, 69.12, 52.18, 28.25; HRMS (ESI) Calcd for C\(_{19}\)H\(_{20}\)INaO\(_5\) (M+Na)\(^+\): 492.0284; Found: 492.0296.

Tert-butyl (2-hydroxynaphthalen-1-yl)(phenyl)methylcarbamate (3t) Yield: 91%; White solid; m.p.: 218.0-219.1 °C; \(^1\)H NMR (300 MHz, \(d_6\)-DMSO): \(\delta\) 10.13 (s, 1H), 7.94 (d, 1H, \(J = 8.7\) Hz), 7.83-7.76 (m, 2H), 7.42 (t, 1H), 7.31-7.16 (m, 8H), 6.81 (d, 1H, \(J = 8.7\) Hz), 1.40 (s, 9H); \(^{13}\)C NMR (75 MHz, \(d_6\)-DMSO) \(\delta\) 155.22, 152.75, 142.72, 132.04, 129.21, 128.56, 128.31, 128.06, 126.67, 126.28, 125.90, 122.72, 122.57, 119.07, 118.53, 78.41, 49.81, 28.15; HRMS (ESI) Calcd for C\(_{22}\)H\(_{23}\)NNaO\(_5\) (M+Na)\(^+\): 372.1576; Found: 372.1570.
Tert-butyl (6-bromo-2-hydroxynaphthalen-1-yl)(phenyl)methylcarbamate (3u)
Yield: 87%; White solid; m.p.: 211.3-212.4 °C; $^1$H NMR (300 MHz, $d_6$-DMSO): δ 10.29 (s, 1H), 8.08 (d, 1H, $J = 1.8$ Hz), 7.87 (d, 1H, $J = 9.0$ Hz), 7.76 (d, 1H, $J = 9.0$ Hz), 7.49 (d, 1H, $J = 8.4$ Hz), 7.28-7.14 (m, 7H), 6.77 (d, 1H, $J = 9.0$ Hz), 1.39 (s, 9H); $^{13}$C NMR (75 MHz, $d_6$-DMSO): δ 155.27, 153.29, 142.37, 130.71, 130.17, 129.65, 129.24, 128.52, 128.10, 126.35, 125.88, 125.50, 119.69, 119.46, 115.37, 78.42, 49.71, 28.15; C$_{22}$H$_{22}$BrNaO$_3$ (M+Na)$^+$: 450.0681; Found: 450.0675.

Reference:

Tert-butyl (1-hydroxynaphthalen-2-yl)(phenyl)methylcarbamate (3v)
Yield: 52%; White solid; m.p.: 94.6-96.8 °C; $^1$H NMR (300 MHz, $d_6$-DMSO): δ 10.11 (s, 1H), 8.17 (dd, 1H, $J_1 = 9.3$ Hz, $J_2 = 1.5$ Hz), 7.96 (d, 1H, $J = 8.1$ Hz), 7.85 (d, 1H, $J = 8.1$ Hz), 7.49-7.44 (m, 2H), 7.31-7.23 (m, 5H), 7.05 (d, 1H, $J = 7.8$ Hz), 6.78 (d, 1H, $J = 7.8$ Hz), 6.44 (s, 1H), 5.89 (s, 1H), 5.87 (s, 1H), 1.33 (s, 9H); $^{13}$C NMR (75 MHz, $d_6$-DMSO): δ 152.63, 142.83, 131.90, 128.64, 128.20, 127.53, 126.78, 126.40, 125.81, 124.79, 124.30, 123.30, 122.63, 107.10, 78.00, 53.73, 28.27; HRMS (ESI) Calcd for C$_{22}$H$_{23}$NNaO$_3$ (M+Na)$^+$: 372.1576; Found: 372.1570.
$^{1}H$ and $^{13}C$ NMR of 3a
$^1$H and $^{13}$C NMR of 3b
$^{1}$H and $^{13}$C NMR of 3c
$^1$H and $^{13}$C NMR of 3d
\[ ^{1}H \text{ and } ^{13}C \text{ NMR of 3e} \]
$^{1}H$ and $^{13}C$ NMR of 3f
$^1$H and $^{13}$C NMR of 3g
$^1$H and $^{13}$C NMR of 3h
$^1$H and $^{13}$C NMR of 3i
$^{1}$H and $^{13}$C NMR of 3j
$^{1}H$ and $^{13}C$ NMR of 3k
$^1$H and $^{13}$C NMR of 3l
$^1$H and $^{13}$C NMR of 3m
$^1$H and $^{13}$C NMR of 3n
$^1$H and $^{13}$C NMR of 3o
$^1$H and $^{13}$C NMR of 3q
$^1$H and $^{13}$C NMR of 3r
$^1$H and $^{13}$C NMR of 3t
\textbf{H and $^{13}$C NMR of 3u}
$^1$H and $^{13}$C NMR of 3v