

Supporting Information

Phthalide Synthesis through Dehydrogenated Lactonization of C(sp³)-H Bond by Photoredox Catalysis

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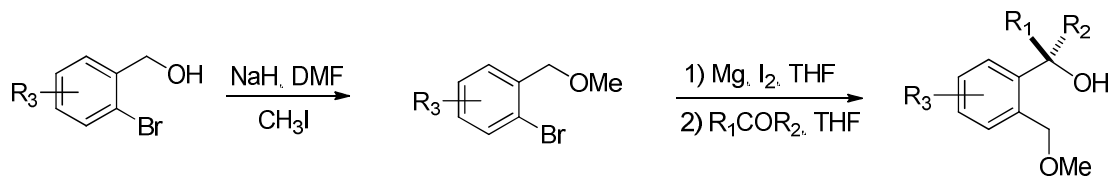
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Materials and methods

All the chemicals were purchased commercially, and used without further purification. Thin-layer chromatography (TLC) was conducted with 0.25 mm Tsingdao silica gel plates (60F-254) and visualized by exposure to UV light (254 nm) or stained with potassium permanganate. Flash column chromatography was performed using Tsingdao silica gel (60, particle size 0.040–0.063 mm). Reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated. ^1H NMR spectra were recorded on JEOL spectrometers (at 400 MHz) and were reported relative to deuterated solvent signals. Data for ^1H NMR spectra were reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration. ^{13}C NMR spectra were recorded on JEOL Spectrometers (at 100 MHz). Data for ^{13}C NMR spectra were reported in terms of chemical shift. Mass spectrometric data were obtained using Bruker Apex IV RTMS. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad.

Typical procedures for the preparations of tertiary alcohols

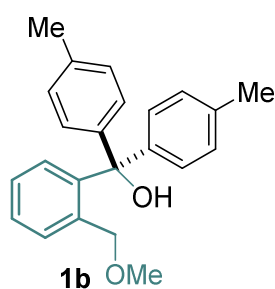


2-Bromobenzyl alcohol (5.0 mmol) was added to a flame-dried round bottomed flask equipped with stirrer bar, and purged with N₂ *via* an inlet needle. Anhydrous DMF (7.5 mL) was added via syringe and stirring commenced. The temperature was adjusted to 0 °C with an external ice bath, then NaH (60% w/w dispersion in mineral oil, 240 mg, 6.0 mmol) added portion-wise over approximately 15 minutes. After that, the reaction mixture was stirred for a further 30 minutes at 0 °C, then methyl iodide (0.63 mL, 10.0 mmol) added in one portion via syringe. The reaction mixture was allowed to stir at room temperature a further 2 hours. The reaction was quenched by addition of a saturated NH₄Cl solution (10 mL), then extracted into Et₂O (3 × 30 mL). The combined organic extracts were washed with water (30 mL), brine (30 mL), dried over MgSO₄, and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel (Hex/EtOAc = 9:1) to afford the desired pure product as a colourless oil.

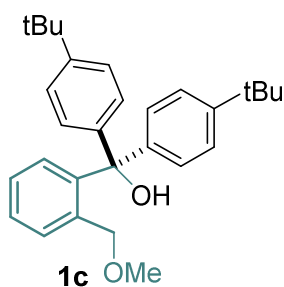
A Schlenk tube was dried under vacuum. After cooling, the tube was placed under N₂ atmosphere. Under a N₂ atmosphere, Mg turnings (5.5 mmol, 1.4 equiv.), iodine (a few grains) and THF (2 mL) were added. In a separate flask under Ar atmosphere, a solution of aryl bromide (4.8 mmol, 1.2 equiv.) in THF (2 mL) was prepared. A few drops of the aryl bromide solution in THF were added to the tube containing Mg, and the mixture was gently heated with a heat gun until the solution color changed from brown to colorless. The remaining aryl bromide solution was then slowly added. Upon complete addition, the mixture was stirred at 60 °C for a further 2 hours. After

that, the solution of ketones (4.0 mmol, 1.0 equiv.) in THF (2 mL) was subsequently added dropwise over 20 mins at 60 °C. The reaction mixture was then allowed to continually stir at the same temperature for 6 hours, after which time TLC indicated complete consumption of starting material ketone. The reaction mixture was quenched with saturated NH₄Cl solution and extracted with EtOAc (3 × 50 mL). The combined organic layers were washed with water (50 mL) and brine (50 mL), dried over Na₂SO₄ and concentrated. The residue was purified by flash chromatography on silica gel (Hex/EtOAc = 6:1) to give the desired product tertiary alcohol.

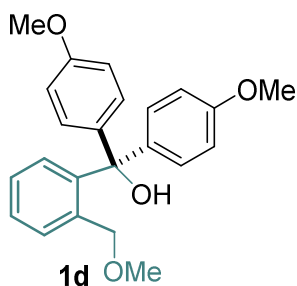
¹H and ¹³C spectra data of compounds 1b-1y, 3a-3q



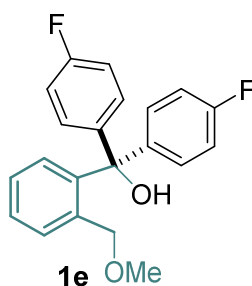
(2-(methoxymethyl)phenyl)di-p-tolylmethanol (1b): ¹H NMR (400 MHz, CDCl₃) δ 7.32- 7.09 (m, 11H), 6.78-6.72 (d, *J* = 8.0 Hz, 1H), 6.08 (s, 1H), 4.10 (s, 2H), 3.22 (s, 3H), 2.34 (s, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 148.2, 144.3, 136.5, 135.2, 132.8, 130.5, 128.6, 128.2, 127.7, 127.6, 82.0, 75.1, 57.7, 21.1; HRMS calculated for C₂₃H₂₃O₂ (M - H⁺): 331.1697, found:331.1704.



bis(4-(tert-butyl)phenyl)(2-(methoxymethyl)phenyl)methanol (1c): ^1H NMR (400 MHz, CDCl_3) δ 7.32-7.20 (m, 11H), 7.16-7.13 (m, 1H), 6.76-6.73 (d, $J = 7.6$ Hz, 1H), 5.99 (s, 1H), 4.07 (s, 2H), 3.18 (s, 3H), 1.30 (s, 18H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.7, 148.3, 144.1, 135.3, 132.7, 130.5, 128.1, 127.6, 127.5, 124.7, 82.0, 75.0, 57.6, 34.5, 31.5; HRMS calculated for $\text{C}_{29}\text{H}_{36}\text{O}_2\text{Na}$ ($\text{M} + \text{Na}^+$):439.2613, found:439.2608.

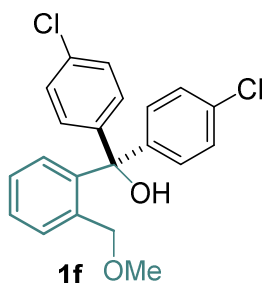


(2-(methoxymethyl)phenyl)bis(4-methoxyphenyl)methanol (1d): ^1H NMR (400 MHz, CDCl_3) δ 7.31-7.22 (m, 6H), 7.18-7.12 (m, 1H), 6.86-6.82 (d, $J = 8.8$ Hz, 4H), 6.75-6.71 (d, $J = 7.6$ Hz, 1H), 6.10 (s, 1H), 4.10 (s, 2H), 3.78 (s, 6H), 3.22 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 158.5, 148.9, 139.6, 135.2, 132.8, 132.2, 130.4, 129.0, 128.2, 127.7, 113.1, 81.7, 75.0, 57.7, 55.3; $\text{C}_{23}\text{H}_{23}\text{O}_4$ ($\text{M} - \text{H}^+$): 363.1596, found: 363.1602.

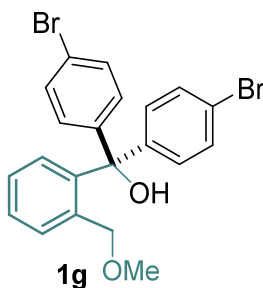


bis(4-fluorophenyl)(2-(methoxymethyl)phenyl)methanol (1e): ^1H NMR (400 MHz, CDCl_3) δ 7.32-7.27 (m, 6H), 7.22-7.16 (m, 1H), 7.04-6.98 (m, 4H), 6.69-6.65 (d, $J = 8.0$ Hz, 1H), 6.40 (s, 1H), 4.08 (s, 2H), 3.25 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 161.8 (d, $J = 243.7$ Hz), 147.6, 142.9, 134.9, 133.2, 130.3, 129.5, 129.4, 128.5, 128.0,

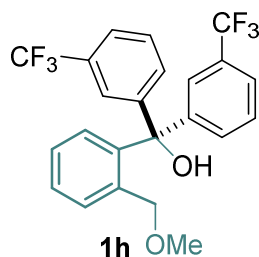
114.8 (d, $J = 21.1$ Hz), 81.5, 75.0, 57.7; ^{19}F NMR (377 MHz, CDCl_3) δ -115.7; HRMS calculated for $\text{C}_{21}\text{H}_{17}\text{F}_2\text{O}_2$ ($\text{M} - \text{H}^+$): 339.1196, found: 339.1202.



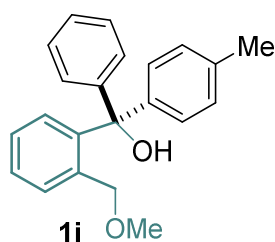
bis(4-chlorophenyl)(2-(methoxymethyl)phenyl)methanol (1f): ^1H NMR (400 MHz, CDCl_3) δ 7.31-7.27 (m, 10H), 7.22-7.17 (m, 1H), 6.70-6.66 (d, $J = 8.0$ Hz, 1H), 6.40 (s, 1H), 4.10 (s, 2H), 3.24 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 147.0, 145.3, 134.9, 133.3, 133.2, 130.4, 129.2, 128.6, 128.1, 81.5, 75.1, 57.8; HRMS calculated for $\text{C}_{21}\text{H}_{17}\text{Cl}_2\text{O}_2$ ($\text{M} - \text{H}^+$): 371.0605, found: 371.0611.



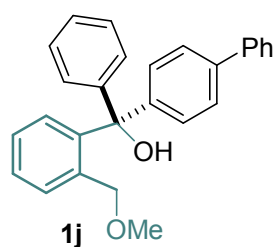
bis(4-bromophenyl)(2-(methoxymethyl)phenyl)methanol (1g): ^1H NMR (400 MHz, CDCl_3) δ 7.46-7.41 (d, $J = 8.8$ Hz, 4H), 7.31-7.27 (m, 2H), 7.24-7.16 (m, 5H), 6.70-6.66 (d, $J = 8.0$ Hz, 1H), 6.40 (s, 1H), 4.07 (s, 2H), 3.23 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 146.9, 145.8, 134.9, 133.3, 131.2, 130.4, 129.6, 128.6, 128.2, 121.5, 81.6, 75.1, 57.1; HRMS calculated for $\text{C}_{21}\text{H}_{18}\text{Br}_2\text{O}_2\text{Na}$ ($\text{M} + \text{Na}^+$): 482.9572, found: 482.9566.



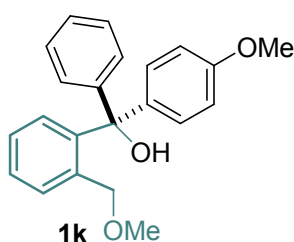
(2-(methoxymethyl)phenyl)bis(3-(trifluoromethyl)phenyl)methanol (1h): ^1H NMR (400 MHz, CDCl_3) δ 7.85 (s, 2H), 7.58-7.54 (d, $J = 6.8$ Hz, 2H), 7.48-7.39 (m, 4H), 7.33-7.29 (m, 2H), 7.23-7.18 (m, 1H), 6.71-6.63 (m, 2H), 4.01 (s, 2H), 3.22 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 147.6, 146.5, 134.8, 133.5, 131.3, 131.2, 131.1 (q, $J = 32.6$ Hz), 130.6, 130.4, 128.8, 128.5, 125.6, 124.3 (q, $J = 271.2$ Hz), 81.7, 75.1, 57.7; ^{19}F NMR (377 MHz, CDCl_3) δ -62.2; HRMS calculated for $\text{C}_{23}\text{H}_{17}\text{F}_6\text{O}_2$ ($\text{M} - \text{H}^+$): 439.1132, found:439.1138.



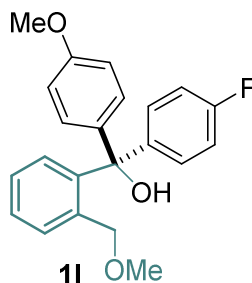
(methoxymethyl)phenyl(phenyl)(p-tolyl)methanol (1i): ^1H NMR (400 MHz, CDCl_3) δ 7.38-7.34 (m, 2H), 7.31-7.22 (m, 7H), 7.17-7.08 (m, 3H), 6.75-6.71 (d, $J = 8.0$ Hz, 1H), 6.14 (s, 1H), 4.08-4.06 (d, $J = 2.0$ Hz, 2H), 3.20 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 148.2, 147.3, 144.2, 136.7, 135.3, 132.9, 130.5, 128.7, 128.3, 128.0, 127.9, 127.8, 127.7, 127.0, 82.1, 75.1, 57.8, 21.2; HRMS calculated for $\text{C}_{22}\text{H}_{22}\text{O}_2\text{Na}$ ($\text{M} + \text{Na}^+$): 341.1518, found: 341.1512.



[1,1'-biphenyl]-4-yl(2-(methoxymethyl)phenyl)(phenyl)methanol (1j): ^1H NMR (400 MHz, CDCl_3) δ 7.60-7.59 (m, 4H), 7.45-7.33 (m, 6H), 7.31-7.20 (m, 6H), 7.15-7.11 (m, 1H), 6.81-6.77 (d, $J = 7.6$ Hz, 1H), 6.14 (s, 1H), 6.28 (s, 1H), 4.11-4.05 (m, 2H), 3.17 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 148.0, 147.2, 146.3, 140.8, 139.8, 137.6, 135.4, 133.1, 130.6, 129.0, 128.5, 128.1, 127.9, 127.5, 127.3, 127.2, 126.7, 82.3, 75.2, 57.8; HRMS calculated for $\text{C}_{27}\text{H}_{23}\text{O}_2$ ($\text{M} - \text{H}^+$): 379.1697, found: 379.1704.

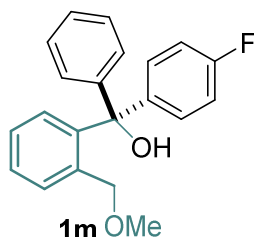


(2-(methoxymethyl)phenyl)(4-methoxyphenyl)(phenyl)methanol (1k): ^1H NMR (400 MHz, CDCl_3) δ 7.37-7.33 (d, $J = 7.6$ Hz, 2H), 7.31-7.20 (m, 7H), 7.16-7.11 (m, 1H), 6.84-6.80 (d, $J = 8.8$ Hz, 2H), 6.74-6.71 (d, $J = 7.6$ Hz, 1H), 6.15 (s, 1H), 4.09-4.06 (m, 2H), 3.75 (s, 3H), 3.20 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 158.6, 148.2, 147.4, 139.3, 135.3, 132.9, 130.5, 129.1, 128.3, 128.0, 127.9, 127.8, 127.0, 113.2, 82.0, 75.0, 57.7, 55.3; HRMS calculated for $\text{C}_{22}\text{H}_{21}\text{O}_3$ ($\text{M} - \text{H}^+$): 331.1490, found: 331.1496.

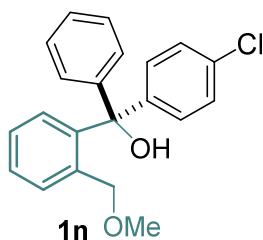


(4-fluorophenyl)(2-(methoxymethyl)phenyl)(4-methoxyphenyl)methanol (1l): ^1H NMR (400 MHz, CDCl_3) δ 7.34-7.22 (m, 6H), 7.18-7.13 (m, 1H), 7.00-6.94 (m, 2H),

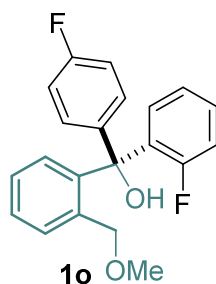
6.86-6.82 (m, 2H), 6.73-6.69 (m, 1H), 6.25 (s, 1H), 4.13-4.07 (dd, $J_1 = 16.0$ Hz, $J_2 = 10.8$ Hz, 2H), 3.77 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 161.9 (d, $J = 244.3$ Hz), 148.0, 143.3, 139.2, 158.7, 135.1, 133.1, 130.4, 129.6, 129.5, 128.9, 128.3, 127.9, 114.6 (d, $J = 21.4$ Hz), 113.3, 81.6, 75.0, 57.7, 55.2; ^{19}F NMR (377 MHz, CDCl_3) δ -115.9; HRMS calculated for $\text{C}_{22}\text{H}_{20}\text{FO}_3$ ($\text{M} - \text{H}^+$): 351.1396, found: 351.1402.



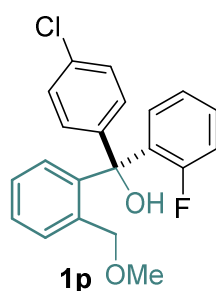
(4-fluorophenyl)(2-(methoxymethyl)phenyl)(phenyl)methanol (1m): ^1H NMR (400 MHz, CDCl_3) δ 7.34-7.21 (m, 9H), 7.16-7.11 (m, 1H), 6.98-6.92 (t, $J = 8.8$ Hz, 2H), 6.72-6.68 (d, $J = 7.2$ Hz, 1H), 6.30 (s, 1H), 4.05 (s, 2H), 3.18 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 161.9 (d, $J = 244.3$ Hz), 147.8, 147.1, 143.1, 135.2, 133.2, 130.5, 129.8, 128.4, 128.1, 128.0, 127.8, 127.3, 114.7 (d, $J = 21.1$ Hz), 82.0, 75.1, 57.7; ^{19}F NMR (377 MHz, CDCl_3) δ -115.7; HRMS calculated for $\text{C}_{21}\text{H}_{18}\text{FO}_2$ ($\text{M} - \text{H}^+$): 321.1290, found: 321.1296.



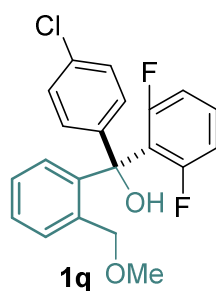
(4-chlorophenyl)(2-(methoxymethyl)phenyl)(phenyl)methanol (1n): ^1H NMR (400 MHz, CDCl_3) δ 7.35-7.23 (m, 11H), 7.18-7.13 (m, 1H), 6.72-6.68 (d, $J = 7.6$ Hz, 1H), 6.31 (s, 1H), 4.06 (s, 2H), 3.20 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 147.6, 146.8, 145.7, 135.1, 133.2, 133.0, 130.5, 129.4, 128.5, 128.1, 128.0, 127.7, 127.3, 81.9, 75.1, 57.7; HRMS calculated for $\text{C}_{21}\text{H}_{18}\text{ClO}_2$ ($\text{M} - \text{H}^+$): 337.0995, found: 337.1008.



(2-fluorophenyl)(4-fluorophenyl)(2-(methoxymethyl)phenyl)methanol (1o): ^1H NMR (400 MHz, CDCl_3) δ 7.39-7.35 (d, $J = 7.2$ Hz, 2H), 7.31-7.22 (m, 4H), 7.18-7.12 (m, 1H), 7.09-7.03 (m, 1H), 7.02-6.94 (m, 3H), 6.86-6.82 (d, $J = 8.0$ Hz, 1H), 5.74-5.71 (d, $J = 4.0$ Hz, 1H), 4.25-4.14 (m, 2H), 3.16 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 162.1 (d, $J = 245.3$ Hz), 160.3 (d, $J = 246.2$ Hz), 145.2, 141.5, 135.7, 132.0, 129.8, 129.7, 129.6, 129.3, 128.2, 128.1, 124.2, 124.1, 116.4 (d, $J = 22.1$ Hz), 114.8 (d, $J = 21.1$ Hz), 80.7, 74.3, 57.8; ^{19}F NMR (377 MHz, CDCl_3) δ -107.1, -115.2; HRMS calculated for $\text{C}_{21}\text{H}_{17}\text{F}_2\text{O}_2$ ($\text{M} - \text{H}^+$): 339.1196, found: 339.1202.

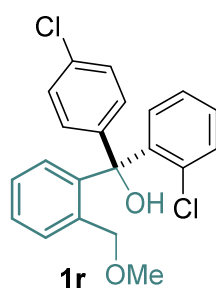


(4-chlorophenyl)(2-fluorophenyl)(2-(methoxymethyl)phenyl)methanol (1p): ^1H NMR (400 MHz, CDCl_3) δ 7.39-7.35 (m, 2H), 7.33-7.24 (m, 6H), 7.19-7.14 (m, 1H), 7.10-7.05 (m, 1H), 7.02-6.96 (m, 1H), 6.85-6.82 (d, $J = 8.4$ Hz, 1H), 5.78-5.75 (d, $J = 3.2$ Hz, 1H), 4.25-4.13 (d, $J = 14.8$ Hz, 2H), 3.17 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 161.5 (d, $J = 246.3$ Hz), 144.9, 144.2, 135.5, 133.3, 133.2, 132.1, 129.9, 129.8, 129.5, 129.4, 129.3, 128.1, 124.2, 124.1, 116.6 (d, $J = 23.0$ Hz), 80.6, 74.4, 57.8; ^{19}F NMR (377 MHz, CDCl_3) δ -106.9; HRMS calculated for $\text{C}_{21}\text{H}_{17}\text{O}_2\text{ClF}$ ($\text{M} - \text{H}^+$): 355.0900, found: 355.0907.



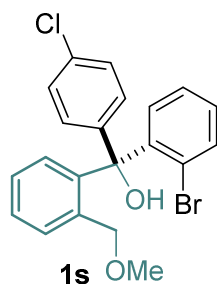
(4-chlorophenyl)(2,6-difluorophenyl)(2-(methoxymethyl)phenyl)methanol (1q):

^1H NMR (400 MHz, CDCl_3) δ 7.43-7.40 (m, 1H), 7.36-7.33 (d, $J = 8.8$ Hz, 2H), 7.30-7.20 (m, 5H), 7.17-7.12 (m, 1H), 6.85-6.78 (m, 1H), 6.75-6.72 (d, $J = 7.2$ Hz, 1H), 5.66-5.63 (t, $J = 3.2$ Hz, 1H), 4.40-4.32 (d, $J = 5.6$ Hz, 2H), 3.20 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 160.8 (d, $J = 249.1$ Hz), 144.9, 143.9, 135.7, 133.5, 131.7, 129.9, 129.8, 129.1, 128.5, 128.2, 128.1, 123.3, 113.0 (d, $J = 21.1$ Hz), 80.4, 74.1, 57.9; ^{19}F NMR (377 MHz, CDCl_3) δ -105.2; $\text{C}_{21}\text{H}_{17}\text{O}_2\text{ClF}_2\text{Na}$ ($\text{M} + \text{Na}^+$): 397.0783, found: 397.0777.

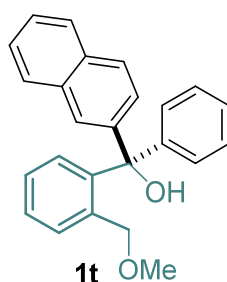


(2-chlorophenyl)(4-chlorophenyl)(2-(methoxymethyl)phenyl)methanol (1r):

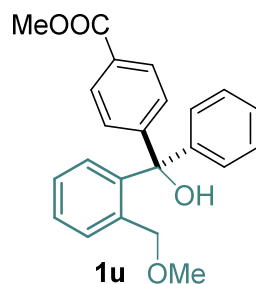
^1H NMR (400 MHz, CDCl_3) δ 7.45-7.41 (m, 1H), 7.40-7.36 (dd, $J_1 = 8.0$ Hz, $J_2 = 12.0$ Hz, 1H), 7.31-7.26 (m, 3H), 7.25-7.19 (m, 2H), 7.17-7.09 (m, 3H), 6.98-6.94 (m, 1H), 6.75-6.72 (m, 1H), 5.82 (s, 1H), 4.37-4.32 (d, $J = 12.0$ Hz, 1H), 4.24-4.19 (d, $J = 12.0$ Hz, 1H), 3.20 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 144.4, 143.9, 143.8, 136.0, 133.7, 133.4, 131.9, 131.8, 130.6, 129.8, 129.6, 129.2, 128.1, 127.8, 126.5, 82.9, 74.0, 57.9; HRMS calculated for $\text{C}_{21}\text{H}_{17}\text{Cl}_2\text{O}_2$ ($\text{M} - \text{H}^+$): 371.0605, found: 371.0611.



(2-bromophenyl)(4-chlorophenyl)(2-(methoxymethyl)phenyl)methanol (1s): ^1H NMR (400 MHz, CDCl_3) δ 7.65-7.62 (m, 1H), 7.45-7.41 (d, $J = 7.6$ Hz, 1H), 7.35-7.28 (m, 3H), 7.20-7.13 (m, 4H), 6.89-6.84 (m, 1H), 6.75-6.71 (d, $J = 8.4$ Hz, 1H), 5.94 (s, 1H), 4.38-4.33 (d, $J = 11.6$ Hz, 1H), 4.24-4.19 (d, $J = 12.0$ Hz, 1H), 3.24 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 145.1, 144.4, 144.0, 135.9, 135.4, 133.4, 131.9, 131.0, 129.9, 129.8, 129.3, 128.0, 127.7, 126.9, 123.5, 83.5, 74.0, 57.8; HRMS calculated for $\text{C}_{21}\text{H}_{18}\text{BrClO}_2\text{Na}(\text{M} + \text{Na}^+)$:439.0074 found:439.0071.

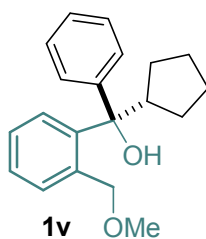


(2-(methoxymethyl)phenyl)(naphthalen-2-yl)(phenyl)methanol (1t): ^1H NMR (400 MHz, CDCl_3) δ 7.85-7.73 (m, 4H), 7.56-7.52 (m, 1H), 7.48-7.43 (m, 2H), 7.43-7.39 (m, 2H), 7.35-7.25 (m, 5H), 7.20-7.15 (m, 1H), 6.78-6.75 (d, $J = 8.0$ Hz, 1H), 6.37 (s, 1H), 4.08 (s, 2H), 3.22 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 154.4, 147.7, 146.9, 144.5, 135.2, 133.1, 133.0, 132.5, 130.7, 128.5, 128.4, 128.0, 127.9, 127.8, 127.6, 127.5, 127.1, 126.5, 126.3, 126.0, 82.3, 75.1, 57.7; HRMS calculated for $\text{C}_{25}\text{H}_{21}\text{O}_2$ ($\text{M} - \text{H}^+$): 353.1541, found:352.1470.

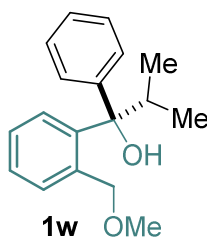


methyl (R)-4-(hydroxy(2-(methoxymethyl)phenyl)(phenyl)methyl)benzoate (1u):

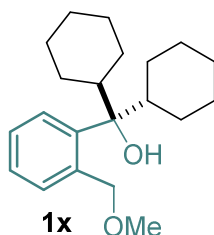
^1H NMR (400 MHz, CDCl_3) δ 8.02-7.96 (d, $J = 8.4$ Hz, 2H), 7.51-7.45 (d, $J = 8.4$ Hz, 2H), 7.38-7.25 (m, 7H), 7.21-7.14 (m, 1H), 6.72-6.67 (d, $J = 7.6$ Hz, 1H), 6.37 (s, 1H), 4.07-4.04 (d, $J = 2.8$ Hz, 2H), 3.90 (s, 3H), 3.22 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.1, 152.3, 147.3, 146.3, 135.0, 133.1, 130.4, 129.2, 128.9, 128.5, 128.1, 128.0, 127.9, 127.7, 127.3, 82.1, 75.1, 57.7, 52.1; HRMS calculated for $\text{C}_{23}\text{H}_{21}\text{O}_4$ ($\text{M} - \text{H}^+$): 361.1439, found: 361.1443.



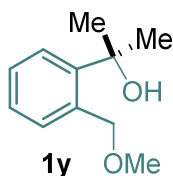
(R)-cyclopentyl(2-(methoxymethyl)phenyl)(phenyl)methanol (1v): ^1H NMR (400 MHz, CDCl_3) δ 7.76-7.73 (d, $J = 8.0$ Hz, 1H), 7.40-7.30 (m, 3H), 7.22-7.17 (m, 4H), 7.16-7.10 (m, 1H), 5.38 (s, 1H), 4.15-4.11 (d, $J = 11.2$ Hz, 1H), 3.87-3.83 (d, $J = 11.2$ Hz, 1H), 3.22 (s, 3H), 2.92-2.85 (m, 1H), 1.93-1.84 (m, 1H), 1.80-1.75 (m, 1H), 1.66-1.50 (m, 4H), 1.38-1.25 (m, 1H), 1.16-1.07 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 147.9, 147.4, 135.4, 133.1, 128.3, 128.2, 127.6, 127.3, 126.2, 126.1, 80.5, 74.6, 57.7, 49.4, 28.6, 28.4, 26.8, 26.5; HRMS calculated for $\text{C}_{20}\text{H}_{24}\text{O}_2\text{Na}$ ($\text{M} + \text{Na}^+$): 319.1674, found: 319.1669.



1-(2-(methoxymethyl)phenyl)-2-methyl-1-phenylpropan-1-ol (1w): ^1H NMR (400 MHz, CDCl_3) δ 7.68-7.66 (d, $J = 8.0$ Hz, 1H), 7.36-7.32 (m, 3H), 7.23-7.16 (m, 4H), 7.14-7.10 (m, 1H), 5.20 (s, 1H), 4.27-4.22 (d, $J = 10.8$ Hz, 1H), 3.94-3.89 (d, $J = 11.2$ Hz, 1H), 3.20 (s, 3H), 1.11-1.07 (d, $J = 6.8$ Hz, 3H), 0.78-0.74 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 146.7, 146.4, 135.7, 133.2, 128.2, 128.0, 127.6, 127.2, 126.5, 126.2, 80.9, 74.9, 57.7, 36.3, 18.2; HRMS calculated for $\text{C}_{18}\text{H}_{22}\text{O}_2\text{Na}$ ($\text{M} + \text{Na}^+$): 293.1518, found: 293.1512.

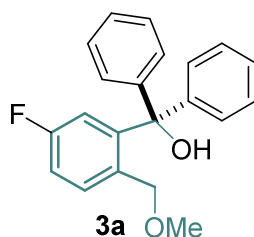


dicyclohexyl(2-(methoxymethyl)phenyl)methanol (1x): ^1H NMR (400 MHz, CDCl_3) δ 7.30-7.25 (m, 1H), 7.24-7.20 (d, $J = 7.6$ Hz, 1H), 7.18-7.15 (d, $J = 5.6$ Hz, 2H), 4.68 (s, 2H), 3.35 (s, 3H), 1.96-1.88 (m, 4H), 1.75-1.55 (m, 7H), 1.49-1.40 (m, 2H), 1.30-1.15 (m, 3H), 1.08-0.99 (m, 3H), 0.88-0.80 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 144.5, 135.7, 133.2, 128.0, 127.9, 125.9, 83.7, 57.5, 45.8, 27.6, 27.1, 26.8, 26.7; HRMS calculated for $\text{C}_{21}\text{H}_{32}\text{O}_2\text{Na}$ ($\text{M} + \text{Na}^+$): 339.2300, found: 339.2295.

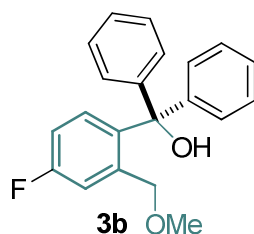


2-(2-(methoxymethyl)phenyl)propan-2-ol (1y): ^1H NMR (400 MHz, CDCl_3) δ 7.37-7.33 (m 1H), 7.31-7.26 (m, 2H), 7.23-7.19 (m, 1H), 4.75 (s, 2H), 3.40 (s, 3H),

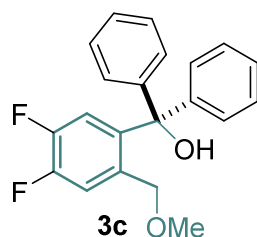
1.63 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 147.7, 134.0, 132.1, 128.4, 126.9, 126.8, 75.3, 74.1, 57.8, 32.4; HRMS calculated for $\text{C}_{11}\text{H}_{16}\text{O}_2\text{Na}$ ($\text{M} + \text{Na}^+$): 203.1048, found:203.1042.



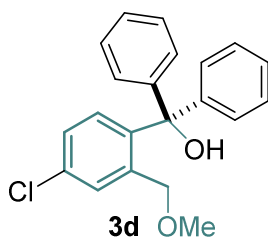
(5-fluoro-2-(methoxymethyl)phenyl)diphenylmethanol (3a): ^1H NMR (400 MHz, CDCl_3) δ 7.42-7.34 (d, $J = 6.8$ Hz, 4H), 7.33-7.20 (m, 8H), 6.95-6.89 (m, 1H), 6.50-6.44 (d, $J = 10.8$ Hz, 1H), 6.23 (s, 1H), 4.00 (s, 2H), 3.16 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 162.2 (d, $J = 246.3$ Hz), 150.9, 146.4, 134.6, 131.3, 128.1, 127.8, 127.4, 118.2 (d, $J = 23.9$ Hz), 114.0 (d, $J = 21.1$ Hz), 82.0, 74.2, 57.7; ^{19}F NMR (377 MHz, CDCl_3) δ -111.9; HRMS calculated for $\text{C}_{21}\text{H}_{18}\text{FO}_2$ ($\text{M} - \text{H}$): 321.1290, found:321.1296.



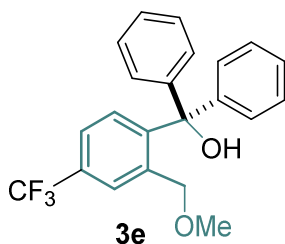
(4-fluoro-2-(methoxymethyl)phenyl)diphenylmethanol (3b): ^1H NMR (400 MHz, CDCl_3) δ 7.36-7.25 (m, 10H), 7.08-7.04 (dd, $J_1 = 9.2$ Hz, $J_2 = 2.8$ Hz, 1H), 6.84-6.78 (m, 1H), 6.71-6.66 (m, 1H), 5.81 (s, 1H), 4.05 (s, 2H), 3.18 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 161.7 (d, $J = 246.3$ Hz), 146.8, 143.6, 138.3, 132.4, 128.0, 127.8, 127.3, 119.3 (d, $J = 22.0$ Hz), 114.0 (d, $J = 19.1$ Hz), 81.8, 74.2, 57.9; ^{19}F NMR (377 MHz, CDCl_3) δ -115.1; HRMS calculated for $\text{C}_{21}\text{H}_{18}\text{FO}_2$ ($\text{M} - \text{H}$): 321.1290, found:321.1295.



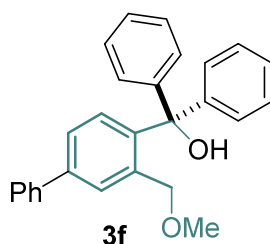
(4,5-difluoro-2-(methoxymethyl)phenyl)diphenylmethanol (3c): ^1H NMR (400 MHz, CDCl_3) δ 7.35-7.27 (m, 10H), 7.18-7.13 (m, 2H), 6.60-6.53(m, 1H), 5.82 (s, 1H), 3.99 (s, 2H), 3.18 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 148.8 (d, $J = 248.2$ Hz), 148.7 (d, $J = 246.3$ Hz), 146.1, 144.6 (d, $J = 72.8$ Hz) 132.9, 130.2, 127.6, 127.3, 121.2 (d, $J = 17.2$ Hz), 120.2 (d, $J = 19.2$ Hz), 81.6, 73.6, 57.9; ^{19}F NMR (377 MHz, CDCl_3) δ -137.4, -139.7; HRMS calculated for $\text{C}_{21}\text{H}_{18}\text{F}_2\text{O}_2$ ($\text{M} - \text{H}^+$): 339.1196, found:339.1202.



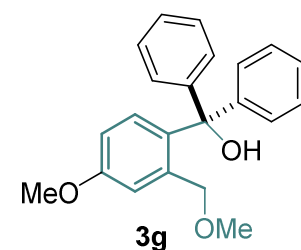
(4-chloro-2-(methoxymethyl)phenyl)diphenylmethanol (3d): ^1H NMR (400 MHz, CDCl_3) δ 7.38-7.22 (m, 11H), 7.13-7.08 (d, $J = 8.4$ Hz, 1H), 6.68-6.63(d, $J = 8.0$ Hz, 1H), 5.90 (s, 1H), 4.03 (s, 2H), 3.18 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 146.5, 146.2, 137.6, 133.4, 132.3, 131.9, 128.1, 127.9, 127.8, 127.3, 81.9, 74.2, 57.9; HRMS calculated for $\text{C}_{21}\text{H}_{18}\text{ClO}_2$ ($\text{M} - \text{H}^+$): 337.0995, found:337.1008.



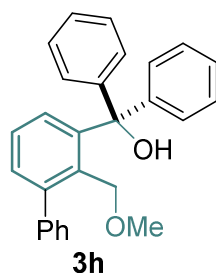
(2-(methoxymethyl)-4-(trifluoromethyl)phenyl)diphenylmethanol (3e): ^1H NMR (400 MHz, CDCl_3) δ 7.61-7.59 (m, 2H), 7.43-7.39 (m, 1H), 7.36-7.25 (m, 10H), 6.89-6.85 (d, $J = 8.0$ Hz, 1H), 5.96 (s, 1H), 4.12 (s, 2H), 3.18 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 151.5, 146.2, 136.7, 130.9, 130.2 (q, $J = 32.6$ Hz), 128.2, 127.7, 127.5, 125.0, 124.8, 123.9 (q, $J = 271.2$ Hz), 82.1, 74.3, 58.0; ^{19}F NMR (377 MHz, CDCl_3) δ -62.4; HRMS calculated for $\text{C}_{22}\text{H}_{18}\text{F}_3\text{O}_2$ ($\text{M} - \text{H}^+$): 371.1258, found: 371.1264.



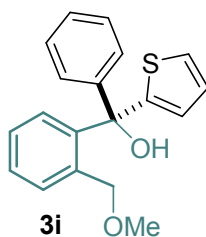
(3-(methoxymethyl)-[1,1'-biphenyl]-4-yl)diphenylmethanol (3f): ^1H NMR (400 MHz, CDCl_3) δ 7.58-7.50 (m, 3H), 7.44-7.22 (m, 14H), 6.82-6.76 (d, $J = 8.0$ Hz, 1H), 6.20 (s, 1H), 4.13 (s, 2H), 3.22 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 147.0, 146.9, 140.5, 135.8, 131.7, 131.2, 128.9, 128.0, 127.7, 127.2, 127.1, 126.6, 82.1, 75.2, 57.8; HRMS calculated for $\text{C}_{27}\text{H}_{23}\text{O}_2$ ($\text{M} - \text{H}^+$): 379.1697, found: 379.1704.



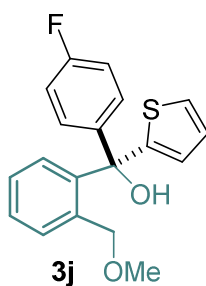
(4-methoxy-2-(methoxymethyl)phenyl)diphenylmethanol (3g): ^1H NMR (400 MHz, CDCl_3) δ 7.38-7.22 (m, 10H), 6.89-6.86 (d, $J = 2.4$ Hz, 1H), 6.68-6.60 (m, 2H), 6.00 (s, 1H), 4.04 (s, 2H), 3.78 (s, 3H), 3.20 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 158.6, 147.3, 140.0, 136.7, 131.9, 127.9, 127.8, 127.0, 119.1, 117.7, 81.8, 75.0, 57.7, 55.3; HRMS calculated for $\text{C}_{22}\text{H}_{21}\text{O}_3$ ($\text{M} - \text{H}^+$): 331.1490, found: 331.1496.



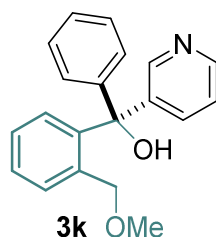
(2-(methoxymethyl)-[1,1'-biphenyl]-3-yl)diphenylmethanol (3h): ^1H NMR (400 MHz, CDCl_3) δ 7.41-7.37 (m, 8H), 7.34-7.29 (m, 5H), 7.27-7.24 (m, 2H), 7.20-7.17 (m, 1H), 6.72-6.69 (m, 1H), 6.60 (s, 1H), 3.94 (s, 2H), 3.20 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 148.9, 147.7, 145.9, 141.5, 130.1, 129.8, 129.5, 128.1, 128.0, 127.9, 127.5, 127.3, 127.0, 82.9, 69.9, 57.6; HRMS calculated for $\text{C}_{27}\text{H}_{23}\text{O}_2$ ($\text{M} - \text{H}^+$): 379.1697, found: 379.1704.



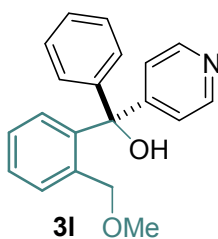
(2-(methoxymethyl)phenyl)(phenyl)(thiophen-2-yl)methanol (3i): ^1H NMR (400 MHz, CDCl_3) δ 7.44-7.40 (m, 2H), 7.35-7.26 (m, 6H), 6.95-6.91 (m, 1H), 6.88-6.85 (d, $J = 7.6$ Hz, 1H), 6.60-6.58 (m, 1H), 6.56 (s, 1H), 4.08 (s, 2H), 3.26 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 152.6, 147.6, 146.9, 134.7, 132.8, 129.9, 128.5, 128.1, 127.4, 126.7, 126.5, 126.4, 125.3, 80.9, 74.9, 57.8; HRMS calculated for $\text{C}_{19}\text{H}_{18}\text{O}_2\text{NaS}$ ($\text{M} + \text{Na}^+$): 333.0926, found: 333.0919.



(4-fluorophenyl)(2-(methoxymethyl)phenyl)(thiophen-2-yl)methanol (3j): ^1H NMR (400 MHz, CDCl_3) δ 7.42-7.36 (m, 2H), 7.25-7.15 (m, 3H), 7.00-6.94 (m, 2H), 6.91-6.87 (t, $J = 3.6$ Hz, 1H), 6.85-6.81 (d, $J = 8.0$ Hz, 1H), 6.66 (s, 1H), 6.56-6.53 (m, 1H), 4.12-4.04 (m, 2H), 3.23 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 162.1 (d, $J = 244.4$ Hz), 152.7, 147.3, 143.0, 134.8, 133.1, 129.9, 128.8, 128.7, 128.3, 126.6, 126.5, 125.6, 114.9 (d, $J = 21.1$ Hz), 80.7, 74.9, 57.8; ^{19}F NMR (377 MHz, CDCl_3) δ -115.1; HRMS calculated for $\text{C}_{19}\text{H}_{16}\text{FO}_2\text{S}$ ($\text{M} - \text{H}^+$): 327.0854, found: 327.0861.

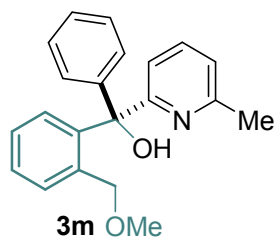


(2-(methoxymethyl)phenyl)(phenyl)(pyridin-3-yl)methanol (3k): ^1H NMR (400 MHz, CDCl_3) δ 8.55-8.51 (m, 2H), 7.80-7.75 (m, 1H), 7.38-7.25 (m, 8H), 7.23-7.18 (m, 1H), 6.70-6.68 (d, $J = 8.0$ Hz, 1H), 4.12-4.02 (m, 2H), 3.24 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.2, 147.8, 146.8, 146.2, 142.7, 135.9, 135.0, 133.2, 130.3, 128.5, 128.2, 127.5, 123.0, 80.9, 75.0, 57.7; HRMS calculated for $\text{C}_{20}\text{H}_{20}\text{O}_2\text{N}$ ($\text{M} + \text{H}^+$): 306.1495, found: 306.1489.

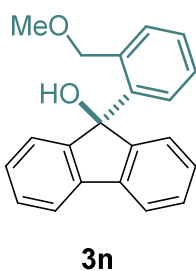


(2-(methoxymethyl)phenyl)(phenyl)(pyridin-4-yl)methanol (3l): ^1H NMR (400 MHz, CDCl_3) δ 8.56-8.52 (d, $J = 6.4$ Hz, 2H), 7.38-7.28 (m, 9H), 7.23-7.18 (m, 1H), 6.71-6.88 (d, $J = 7.6$ Hz, 1H), 4.13-4.01 (m, 2H), 3.20 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 156.5, 148.9, 146.2, 145.7, 135.0, 133.2, 130.2, 128.6, 128.3, 128.2, 127.6, 127.4, 123.1, 81.5, 75.0, 57.7; HRMS calculated for $\text{C}_{20}\text{H}_{20}\text{O}_2\text{N}$ ($\text{M} + \text{H}^+$): 306.1495,

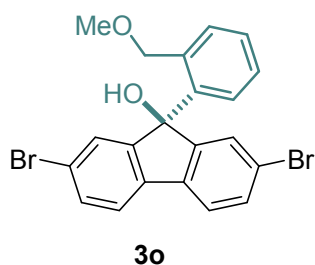
found: 306.1489.



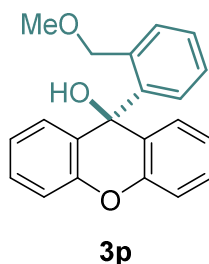
(2-(methoxymethyl)phenyl)(6-methylpyridin-2-yl)(phenyl)methanol (3m): ^1H NMR (400 MHz, CDCl_3) δ 7.64-7.61 (d, $J = 7.6$ Hz, 1H), 7.54-7.48 (m, 1H), 7.36-7.24 (m, 7H), 7.10-7.09 (m, 2H), 6.88-6.84 (d, $J = 7.6$ Hz, 1H), 6.63 (s, 1H), 6.55-6.51 (m, 1H), 4.49-4.43 (d, $J = 13.2$ Hz, 1H), 4.33-4.29 (d, $J = 13.2$ Hz, 1H), 3.10 (s, 3H), 2.57 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 162.1, 156.9, 146.2, 143.6, 139.2, 136.7, 128.8, 128.6, 128.0, 127.8, 127.6, 127.3, 126.0, 121.9, 120.4, 81.3, 72.2, 58.1, 24.3; HRMS calculated for $\text{C}_{21}\text{H}_{22}\text{NO}_2$ ($\text{M} + \text{H}^+$): 320.1651, found: 320.1645.



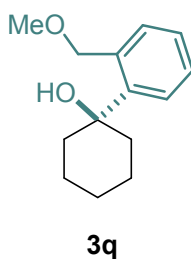
9-(2-(methoxymethyl)phenyl)-9H-fluoren-9-ol (3n): ^1H NMR (400 MHz, CDCl_3) δ 7.71-7.66 (d, $J = 7.2$ Hz, 3H), 7.39-7.31 (m, 5H), 7.23-7.19 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 139.7, 135.1, 129.1, 128.6, 127.6, 127.5, 124.6, 120.3, 57.8; HRMS calculated for $\text{C}_{21}\text{H}_{18}\text{O}_2\text{Na}$ ($\text{M} + \text{Na}^+$): 325.1205, found: 325.1199.



2,7-dibromo-9-(2-(methoxymethyl)phenyl)-9H-fluoren-9-ol (3o): ^1H NMR (400 MHz, CDCl_3) δ 7.54-7.47 (m, 6H), 7.35-7.30 (m 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 137.6, 134.7, 132.3, 128.1, 127.7, 122.6, 121.7, 58.0; HRMS calculated for $\text{C}_{21}\text{H}_{16}\text{Br}_2\text{O}_2\text{Na}(\text{M} + \text{Na}^+)$:480.9415, found:480.9409.



9-(2-(methoxymethyl)phenyl)-9H-xanthen-9-ol (3p): ^1H NMR (400 MHz, CDCl_3) δ 8.16-8.12 (d, $J = 8.0$ Hz, 1H), 7.44-7.28 (m, 5H), 7.22-7.18 (m, 2H), 7.08-7.04 (m, 2H), 7.02-6.96 (m, 2H), 3.74 (s, 2H), 2.79 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.7, 143.5, 135.3, 129.4, 129.1, 128.9, 127.9, 127.1, 127.0, 126.3, 123.6, 116.5, 71.7, 70.2, 57.6; HRMS calculated for $\text{C}_{21}\text{H}_{18}\text{O}_3\text{Na}(\text{M} + \text{Na}^+)$: 341.1154, found: 341.1148.



1-(2-(methoxymethyl)phenyl)cyclohexan-1-ol (3q): ^1H NMR (400 MHz, CDCl_3) δ 7.37-7.33 (d, $J = 7.6$ Hz, 1H), 7.28-7.23 (m, 2H), 7.19-7.14 (m, 1H), 4.74 (s, 2H), 3.35 (s, 3H), 1.90-1.79 (m, 5H), 1.78-1.70 (m, 2H), 1.61-1.54 (m, 2H), 1.26-1.22 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 148.3, 134.6, 132.1, 128.2, 126.8, 126.6, 75.4, 74.9, 57.4, 39.4, 25.8, 22.1; HRMS calculated for $\text{C}_{14}\text{H}_{20}\text{O}_2\text{Na}(\text{M} + \text{Na}^+)$:243.1361,

found:243.1356.

Copies of ^1H NMR and ^{13}C NMR spectra

