

ELECTRONIC SUPPLEMENTARY INFORMATION

Palladium-Catalyzed Tsuji–Trost-Type Reaction of benzofuran-2-ylmethyl acetates with nucleophiles

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1. GENERAL INFORMATION

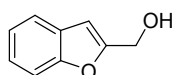
1.1. Reagents and methods

All of the commercially available reagents, catalysts, bases and solvents were used as purchased, without further purification. Starting materials and reaction products were purified by flash chromatography using SiO₂ as stationary phase, eluting with *n*-hexane/ethyl acetate. ¹³C NMR (100.6 MHz), and ¹⁹F spectra (376.5 MHz) were recorded with a Bruker Avance 400 spectrometer, equipped with Nanobay console and Cryoprobe Prodigy probe. Splitting patterns are designed as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), bs (broad singlet), quint (quintuplet), or sext (sextuplet). IR spectra were recorded with a PerkinElmer SpectrumOne FT-ATR spectrophotometer. HRMS were recorded with an Orbitrap Exactive Mass spectrometer with ESI source. Melting points were determined with a Büchi B-545 apparatus and are uncorrected.

2. SYNTHETIC PROCEDURES

2.1. Typical procedure for the preparation of benzofuran-2-ylmethanol **4a**:

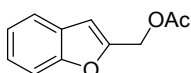
In a flame dried two-necked round bottom flask charged with a stir bar, LiAlH₄ (2 M, 3.4 mL, 6.787 mmol, 1.1 equiv.) was added drop to drop to a solution of benzofuran-2-carboxylic acid **3a** (1.0 g, 6.170 mmol, 1 equiv.) at 0°C in anhydrous THF (20 mL) under Ar. The mixture was allowed to warm to room temperature and stirred for 2 hours. After the complete consumption of the starting material (TLC, hexane/EtOAc 90/10 v/v), the reaction was cooled down to 0°C and quenched by slow addition of an 80 percent aqueous MeOH solution. The mixture was extracted with AcOEt, washed with brine and the combined organic phase was dried over MgSO₄, filtered, and concentrated in vacuo. The residue was purified by flash chromatography (silica gel, *n*-hexane/EtOAc 85/15 v/v, *R_f* = 0.24) to afford 0.713 g of benzofuran-2-ylmethanol **4a** as a white solid (80% yield).



4a: pale yellow oil; 80% yield (6.170 mmol scale, 0.713 g); IR (neat): 3347, 2921, 1605, 1454, 1254, 1010; cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 7.58 (dd, *J*₁ = 7.6 Hz, *J*₂ = 0.6 Hz, 1 H), 7.49 (dd, *J*₁ = 8.2 Hz, *J*₂ = 0.6 Hz, 1H), 7.31 (td, *J*₁ = 7.4 Hz, *J*₂ = 1.3 Hz, 1H), 7.24 (td, *J*₁ = 7.4 Hz, *J*₂ = 1.1 Hz, 1H), 6.69 (d, *J* = 0.6 Hz, 1H), 4.80 (s, 2H), 1.96 (bs, 1 H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 156.5 (q), 155.1 (q), 128.1 (q), 124.4 (CH), 122.8 (CH), 121.1 (CH), 111.3 (CH), 104.1 (CH), 58.1; HRMS: *m/z* [M + Na]⁺ calcd for C₉H₈O₂Na: 171.0417; found: 171.0418.

2.2. Typical procedure for the preparation of benzofuran-2-ylmethyl acetate **2a**:

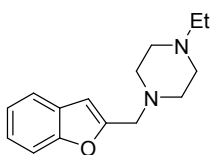
To a stirred solution of benzofuran-2-ylmethanol **4a** (0.700 g, 4.7 mmol) in THF (10 mL) was successively added acetic anhydride (280 μL, 5.170 mmol, 1.1 equiv.) and triethylamine (350 μL, 5.640 mmol, 1.2 equiv.) at 0 °C. The mixture was allowed to warm to room temperature and stirred for 24 h. After fully consumption of substrate **4a**, the reaction was quenched with a solution of KHSO₄ (10% w/w), diluted with AcOEt and washed with a saturated NaHCO₃ solution and with brine. The combined organic layer was dried over Na₂SO₄, filtered and concentrated under vacuum. The crude product **2a** was pure enough to be used directly in the next step (quantitative yield).



2a: colorless oil; quantitative yield (4.7 mmol scale, 0.892 g); IR (neat): 2914, 1698, 1420, 1223, 1045 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.59 (dd, J_1 = 7.7 Hz, J_2 = 0.5 Hz, 1 H), 7.51 (dd, J_1 = 8.2 Hz, J_2 = 0.7 Hz, 1 H), 7.33 (td, J_1 = 7.3 Hz, J_2 = 1.3 Hz, 1 H), 7.26 (td, J_1 = 7.5 Hz, J_2 = 1.0 Hz, 1 H), 6.80 (s, 1 H), 5.23 (s, 2H), 2.14 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 170.6 (q), 155.2 (q), 151.9 (q), 127.9 (q), 124.9 (CH), 123.0 (CH), 121.3 (CH), 111.4 (CH), 107.0 (CH), 58.6 (CH_2), 20.9 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{11}\text{H}_{11}\text{O}_3\text{Na}$: 213.0522; found: 213.0523.

2.3. Typical procedure for the preparation of 1-(benzofuran-2-ylmethyl)-4-ethylpiperazine 6aa:

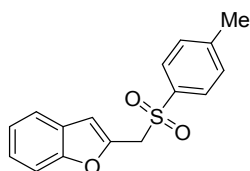
In a 50 mL Carousel Tube Reactor (Radely Discovery Technology) containing a magnetic stirring bar Pd_2dba_3 (9.2 mg, 0.010 mmol, 0.025 equiv.) and dppf (11.1 mg, 0.020 mmol, 0.05 equiv.) were dissolved at room temperature with 1.0 mL of anhydrous MeCN. Then, benzofuran-2-ylmethyl acetate **2a** (76.0 mg, 0.4 mmol, 1.0 equiv.), *N*-ethylpiperazine **5a** (152 μl , 0.80 mmol, 2.0 equiv.), K_2CO_3 (165.6 mg, 1.20 mmol, 2.0 equiv.), and 1.0 mL of solvent were added. The mixture was stirred for 24 h at 100 $^\circ\text{C}$ under Ar. After this time, the reaction mixture was cooled to room temperature, diluted with Et_2O , washed with a saturated NaHCO_3 solution and with brine. The organic extract was dried over Na_2SO_4 , filtered and concentrated under reduced pressure. The residue was purified by chromatography on SiO_2 (25-40 μm), eluting with a 80/20 (v/v) *n*-hexane/AcOEt mixture (R_f = 0.22) to obtain 84.9 mg (87% yield) of 1-(benzofuran-2-ylmethyl)-4-ethylpiperazine **6aa**.



6aa: pale yellow oil; 87% yield (84.9 mg); IR (neat): 2935, 2810, 1454, 1254, 1163, 941 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.54 (d, J = 7.4 Hz, 1H), 7.49 (d, J = 7.9 Hz, 1H), 7.31-7.18 (m, 2H), 6.62 (s, 1H), 3.72 (s, 2H), 2.87-2.27 (m, 10 H), 1.10 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 155.1 (q), 154.4 (q), 128.3 (q), 123.9 (CH), 122.6 (CH), 120.7 (CH), 111.3 (CH), 105.7 (CH), 55.5 (CH_2), 53.1 (CH_2), 52.6 (CH_2), 52.2 (CH_2), 11.9 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{15}\text{H}_{20}\text{N}_2\text{ONa}$: 267.1468; found: 267.1471.

2.4. Typical procedure for the preparation of 2-(tosylmethyl)benzofuran 8aa:

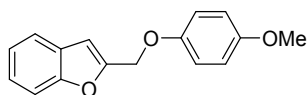
In a 50 mL Carousel Tube Reactor (Radely Discovery Technology) containing a magnetic stirring bar $[\text{Pd}(\eta^3\text{-C}_3\text{H}_5\text{Cl})_2]$ (3.7 mg, 0.010 mmol, 0.025 equiv.) and XPhos (9.5 mg, 0.020 mmol, 0.05 equiv.) were dissolved at room temperature with 0.5 mL of anhydrous THF under Ar. Then, benzofuran-2-ylmethyl acetate **2a** (76.0 mg, 0.4 mmol, 1.0 equiv.), sodium 4-tolylsulphinate **7a** (142.5 mg, 0.80 mmol, 2.0 equiv.), K_2CO_3 (110.4 mg, 0.80 mmol, 2.0 equiv.), and 1.0 mL of anhydrous MeCN were added. The mixture was stirred for 1 h at 120 $^\circ\text{C}$ under Ar. After this time, the reaction mixture was cooled to room temperature, diluted with Et_2O , washed with brine. The organic extract was dried over Na_2SO_4 , filtered and concentrated under reduced pressure. The residue was purified by chromatography on SiO_2 (25-40 μm), eluting with a 70/30 (v/v) *n*-hexane/AcOEt mixture (R_f = 0.24) to obtain 112.2 mg (98% yield) of 12-(tosylmethyl)benzofuran **8aa**.



8aa: pale yellow solid; 98% yield (112.2 mg); mp: 194-195°C; IR (neat): 1451, 1310, 1144, 1084 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.57 (d, J = 8.1 Hz, 2H), 7.45(d, J =7.7 Hz, 1H), 7.28 (d, J = 8.1 Hz, 1H), 7.23-7.11 (m, 4H), 6.59 (s, 1H), 4.45 (s, 2H), 2.35 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 155.2 (q), 145.3 (q), 145.1 (q), 135.3 (q), 129.8 (CH), 128.5 (CH), 127.9 (q), 124.9 (CH), 123.1 (CH), 121.2 (CH), 111.3 (CH), 108.9 (CH), 56.5 (CH₂), 21.7 (CH₃); HRMS: m/z [$\text{M} + \text{Na}$]⁺ calcd for C₁₆H₁₄O₃Na: 309.0556; found: 309.0551.

2.5. Typical procedure for the preparation of 2-((4-methoxyphenoxy)methyl)benzofuran 10aa:

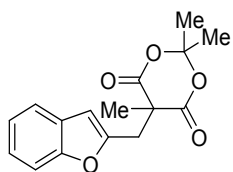
In a 50 mL Carousel Tube Reactor (Radely Discovery Technology) containing a magnetic stirring bar [$\text{Pd}(\eta^3\text{-C}_3\text{H}_5\text{Cl})_2$ (3.7 mg, 0.010 mmol, 0.025 equiv.) and XPhos (9.5 mg, 0.020 mmol, 0.05 equiv.) were dissolved at room temperature with 0.5 mL of anhydrous THF under Ar. Then, benzofuran-2-ylmethyl acetate **2a** (76.0 mg, 0.4 mmol, 1.0 equiv.), 4-methoxyphenol **9a** (99.0 mg, 0.80 mmol, 2.0 equiv.), K₂CO₃ (110.4 mg, 0.80 mmol, 2.0 equiv.), and 1.0 mL of anhydrous MeCN were added. The mixture was stirred for 1 h at 120 °C under Ar. After this time, the reaction mixture was cooled to room temperature, diluted with Et₂O, washed with a saturated NaHCO₃ solution and with brine. The organic extract was dried over Na₂SO₄, filtered and concentrated under reduced pressure. The residue was purified by chromatography on SiO₂ (25-40 μm), eluting with a 90/10 (v/v) *n*-hexane/AcOEt mixture (R_f = 0.19) to obtain 91.5 mg (90% yield) of 2-((4-methoxyphenoxy)methyl)benzofuran **10aa**.



10aa: white solid; 90% yield (91.5 mg); mp: 92-93 °C; IR (Neat): 2926, 1505, 1453, 1376, 1225, 1030 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.59 (d, J = 7.6 Hz, 1H), 7.53 (d, J = 8.2 Hz, 1H), 7.35-7.30 (m, 1H), 7.28-7.23 (m, 1H), 6.98 (d, J = 9.1 Hz, 2H), 6.88 (d, J = 9.1 Hz, 2H), 6.79 (s, 1H), 5.14 (s, 2H), 3.80 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 155.2 (q), 154.4 (q), 153.2 (q), 152.4 (q), 128.0 (q), 124.6 (CH), 122.9 (CH), 121.2 (CH), 116.2 (CH), 114.7 (CH), 111.4 (CH), 106.0 (CH), 64.0 (CH₂), 55.7 (CH₃); HRMS: m/z [$\text{M} + \text{Na}$]⁺ calcd for C₁₆H₁₄O₃Na: 277.0835; found: 277.0833.

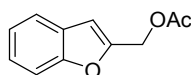
2.5. Typical procedure for the preparation of 5-(benzofuran-2-ylmethyl)-2,2,5-trimethyl-1,3-dioxane-4,6-dione 13ad:

In a 50 mL Carousel Tube Reactor (Radely Discovery Technology) containing a magnetic stirring bar [$\text{Pd}(\eta^3\text{-C}_3\text{H}_5\text{Cl})_2$ (3.7 mg, 0.010 mmol, 0.025 equiv.) and XPhos (9.5 mg, 0.020 mmol, 0.05 equiv.) were dissolved at room temperature with 0.5 mL of anhydrous THF under Ar. Then, benzofuran-2-ylmethyl acetate **2a** (76.0 mg, 0.4 mmol, 1.0 equiv.), 2,2,5-trimethyl-1,3-dioxane-4,6-dione **12d** (126.4 mg, 0.80 mmol, 2.0 equiv.), K₂CO₃ (110.4 mg, 0.80 mmol, 2.0 equiv.), and 1.0 mL of anhydrous MeCN were added. The mixture was stirred for 1 h at 120 °C under Ar. After this time, the reaction mixture was cooled to room temperature, diluted with Et₂O, washed with a saturated NaHCO₃ solution and with brine. The organic extract was dried over Na₂SO₄, filtered and concentrated under reduced pressure. The residue was purified by chromatography on SiO₂ (25-40 μm), eluting with a 95/5 (v/v) *n*-hexane/AcOEt mixture (R_f = 0.19) to obtain 111.9 mg (97% yield) of 5-(benzofuran-2-ylmethyl)-2,2,5-trimethyl-1,3-dioxane-4,6-dione **13ad**.

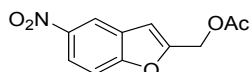


13ad: pale yellow oil; 97% yield (111.9 mg); IR (neat): 2923, 1776, 1737, 1453, 1380, 1251, 1055 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.52-7.47 (m, 1H), 7.44-7.40 (m, 1H), 7.25 (td, $J_1 = 7.2$, $J_2 = 1.4$, 1H), 7.25 (td, $J_1 = 7.2$ Hz, $J_2 = 1.4$ Hz, 1H), 6.55 (s, 1H), 3.56 (s, 2H), 1.80 (s, 3H), 1.71 (s, 3H), 1.28 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 169.3 (q), 154.7 (q), 152.2 (q), 128.1 (q), 124.3 (CH), 122.9 (CH), 120.9 (CH), 111.1 (CH), 106.0 (CH), 105.5 (q), 49.6 (q), 37.8 (CH₂), 29.3 (CH₃), 28.4 (CH₃), 25.7 (CH₃); HRMS: m/z [$\text{M} + \text{Na}$]⁺ calcd for C₁₆H₁₆O₅Na: 311.0890; found: 311.0890.

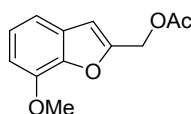
3. CHARACTERIZATION DATA OF 2a – c, 4a – c, 6aa – bk, 8aa – cb, 10aa – ci, 11, 12, 13aa –bd



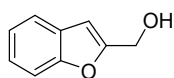
2a: colorless oil; quantitative yield (4.7 mmol scale, 0.892 g); IR (neat): 2914, 1698, 1420, 1223, 1045 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.59 (dd, J_1 = 7.7 Hz, J_2 = 0.5 Hz, 1 H), 7.51 (dd, J_1 = 8.2 Hz, J_2 = 0.7 Hz, 1 H), 7.33 (td, J_1 = 7.3 Hz, J_2 = 1.3 Hz, 1 H), 7.26 (td, J_1 = 7.5 Hz, J_2 = 1.0 Hz, 1 H), 6.80 (s, 1 H), 5.23 (s, 2H), 2.14 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 170.6 (q), 155.2 (q), 151.9 (q), 127.9 (q), 124.9 (CH), 123.0 (CH), 121.3 (CH), 111.4 (CH), 107.0 (CH), 58.6 (CH_2), 20.9 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{11}\text{H}_{10}\text{O}_3\text{Na}$: 213.0522; found: 213.0523.



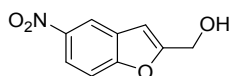
2b: yellow solid; 95% yield; (4.0 mmol scale, 0.893 g); mp: 90-91°C; IR (neat): 1732, 1549, 1244, 1019 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 8.50 (d, J = 2.3 Hz, 1H), 8.24 (dd, J_1 = 9.1 Hz, J_2 = 2.3 Hz, 1H), 7.56 (d, J = 9.1 Hz, 1H), 6.91 (d, J = 0.5 Hz, 1H), 5.23 (s, 2H), 2.14 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 170.3 (q), 157.9 (q), 155.5 (q), 144.3 (q), 128.3 (q), 120.7 (CH), 117.9 (CH), 111.8 (CH), 107.4 (CH), 58.1 (CH_2), 20.7 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{11}\text{H}_9\text{NO}_5\text{Na}$: 258.0373; found: 258.0371.



2c: dark yellow oil; 97% yield (4.0 mmol scale, 0.853 g); IR (neat): 2940, 1739, 1493, 1377, 1363, 1220, 1203, 1094, 1024 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.19-7.16 (m, 2H), 6.86-6.81 (m, 1H), 6.79 (s, 1H), 5.22 (s, 2H), 4.03 (s, 3H), 2.12 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 170.5 (q), 152.0 (q), 145.3 (q), 144.6 (q), 129.5 (q), 123.7 (CH), 113.6 (CH), 107.4 (CH), 106.7 (CH), 58.5 (CH_2), 56.0 (CH_3), 20.8 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{12}\text{H}_{12}\text{O}_4\text{Na}$: 243.0628; found: 243.0628.

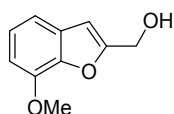


4a: pale yellow oil; 80% yield (6.170 mmol scale, 0.713 g); IR (neat): 3347, 2921, 1605, 1454, 1254, 1010 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.58 (dd, J_1 = 7.6 Hz, J_2 = 0.6 Hz, 1 H), 7.49 (dd, J_1 = 8.2 Hz, J_2 = 0.6 Hz, 1H), 7.31 (td, J_1 = 7.4 Hz, J_2 = 1.3 Hz, 1H), 7.24 (td, J_1 = 7.4 Hz, J_2 = 1.1 Hz, 1H), 6.69 (d, J = 0.6 Hz, 1H), 4.80 (s, 2H), 1.96 (bs, 1 H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 156.5 (q), 155.1 (q), 128.1 (q), 124.4 (CH), 122.8 (CH), 121.1 (CH), 111.3 (CH), 104.1 (CH), 58.1; HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_9\text{H}_8\text{O}_2\text{Na}$: 171.0417; found: 171.0418.

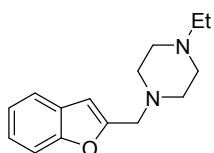


4b: pale yellow solid; quantitative yield (4.0 mmol scale, 0.772 g); mp: 90-91°C; IR (neat): 3407, 1517, 1342, 1016 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 8.48 (d, J = 2.3 Hz, 1H), 8.21 (dd, J_1 = 9.1 Hz, J_2 = 2.3 Hz, 1H), 7.53 (d, J = 9.1 Hz, 1H), 6.81 (d, J = 0.74 Hz, 1H), 4.83 (s, 2H), 2.09 (s, 1H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ =

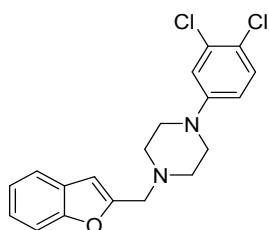
160.0 (q), 157.9 (q), 144.2 (q), 128.5(q), 120.3 (CH), 117.7 (CH), 111.6 (CH), 104.7 (CH), 57.9 (CH₂); HRMS: m/z [M + Na]⁺ calcd for C₉H₇NO₄Na: 216.0267; found: 216.0265.



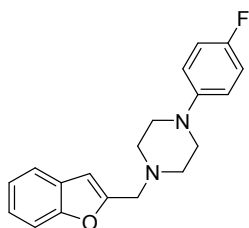
4c: pale yellow solid; quantitative yield (4.0 mmol scale, 0.712 g); mp 45-46 °C, lit. ¹46-47 °C. IR (Neat): 3392, 2941, 2840, 1735, 1606, 1622, 1588, 1436, 1284, 1096 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 7.18-7.11 (m, 2H), 6.83-6.76 (m, 1H), 6.66 (s, 1H), 4.79 (d, J = 5.4 Hz, 2H), 4.00 (s, 3H), 1.65 (t, J = 5.4 Hz, 1H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 156.8 (q), 145.3 (q), 144.3 (q), 129.8 (q), 123.5 (CH), 113.5 (CH), 106.4 (CH), 104.4 (CH), 58.0 (CH₂), 56.0 (CH₃); HRMS: m/z [M + Na]⁺ calcd for C₁₀H₁₀O₃Na: 201.0522; found: 201.0519.



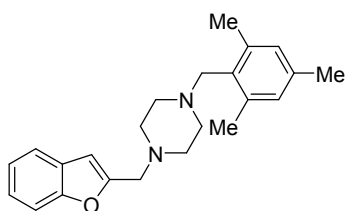
6aa: pale yellow oil; 87% yield (84.9 mg); IR (neat): 2935, 2810, 1454, 1254, 1163, 941 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 7.54 (d, J = 7.4 Hz, 1H), 7.49 (d, J = 7.9 Hz, 1H), 7.31-7.18 (m, 2H), 6.62 (s, 1 H), 3.72 (s, 2H), 2.87-2.27 (m, 10 H), 1.10 (t, J = 7.6 Hz, 3H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 155.1 (q), 154.4 (q), 128.3 (q), 123.9 (CH), 122.6 (CH), 120.7 (CH), 111.3 (CH), 105.7 (CH), 55.5 (CH₂), 53.1 (CH₂), 52.6 (CH₂), 52.2 (CH₂), 11.9 (CH₃); HRMS: m/z [M + Na]⁺ calcd for C₁₅H₂₀N₂O₂Na: 267.1468; found: 267.1471.



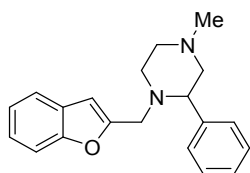
6ab: pale yellow solid; 78% yield (112.7 mg); mp: 113-114°C; IR (neat): 2843, 1592, 1453, 1385, 1240, 1138 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 7.57-7.54 (m, 1H), 7.53-7.49 (m, 1H), 7.32- 7.21 (m, 3H), 6.96 (d, J =2.9 Hz, 1H), 6.74 (dd, J_1 = 8.6 J_2 = 2.9, 1H), 6.66 (s, 1H), 3.77 (s, 2H), 3.28-3.18 (m, 4H), 2.78-2.66 (m, 4H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 155.1 (q), 154.0 (q), 150.6 (q), 132.8 (q), 130.4 (CH), 128.2 (q), 124.1 (CH), 122.8 (CH), 122.2 (q), 120.8 (CH), 117.3 (CH), 115.3 (CH), 111.3 (CH), 105.9 (CH), 55.4 (CH₂), 52.7 (CH₂), 48.6 (CH₂); HRMS: m/z [M + H]⁺ calcd for C₁₉H₁₉Cl₂N₂O: 361.0869; found: 361.0865.



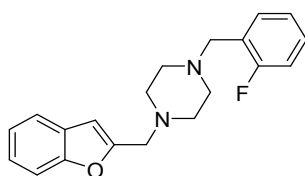
6ac: pale yellow solid; 76% yield (94.3 mg); mp: 91-92°C; IR (neat): 2811, 1510, 1453, 1384, 1229, 1142 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 7.59-7.49 (m, 2H), 7.34-7.20 (m, 2H), 7.02-6.93 (m, 2H), 6.93-6.85 (m, 2H), 6.66 (s, 1H), 3.78 (s, 2H), 3.19 (bt, *J* = 4.9 Hz, 4H), 2.75 (bt, *J* = 4.9 Hz, 4H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 157.2 (q) (d, *J* = 239 Hz), 155.1 (q), 154.2 (q), 147.9 (q) (d, *J* = 2 Hz), 128.2 (q), 124.0 (CH), 122.7 (CH), 120.7 (CH), 115.2 (CH) (d, *J* = 8 Hz), 115.5 (CH) (d, *J* = 22 Hz), 111.3 (CH), 105.9 (CH), 55.5 (CH₂), 53.1 (CH₂), 50.1 (CH₂); ¹⁹F NMR (376.5 MHz) (CDCl₃): δ = -124.5; HRMS: *m/z* [M + H]⁺ calcd for C₁₉H₂₀FN₂O: 311.1554; found: 311.1555.



6ad: pale yellow solid; 88% yield (122.7mg); mp: 117-118°C; IR (neat): 2919, 2807, 1454, 1253, 1137, 1008 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 7.56-7.50 (m, 2H), 7.31-7.21 (m, 2H), 6.87 (s, 2H), 6.61 (s, 1H), 3.70 (s, 2H), 3.50 (s, 2H), 2.63-2.57 (bs, 8H), 2.38 (s, 6H), 2.30 (s, 3H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 155.1 (q), 154.7 (q), 138.1 (q), 136.3 (q), 131.9 (q), 128.9 (CH), 128.4 (q), 123.9 (CH), 122.6 (CH), 120.7 (CH), 111.3 (CH), 105.5 (CH), 55.8 (CH₂), 55.5 (CH₂), 53.4 (CH₂), 52.5 (CH₂), 20.9 (CH₃), 20.1 (CH₃); HRMS: *m/z* [M + H]⁺ calcd for C₂₃H₂₉N₂O: 349.2274; found: 349.2273.

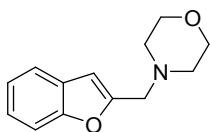


6ae: pale yellow solid; 94% yield (115.2 mg); mp: 80-81°C; IR (neat): 2935, 2794, 1454, 1149, 1026, 940 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 7.55-7.43 (m, 4H), 7.40 (t, *J* = 7.2 Hz, 2H), 7.34-7.29 (m, 1H), 7.27-7.16 (m, 2H), 6.49 (s, 1H), 3.84 (d, *J* = 14.7, 1H), 3.50 (dd, *J*₁ = 10.3 Hz, *J*₂ = 2.8 Hz, 1H), 3.36 (d, *J* = 14.7, 1H), 3.08 (dt, *J*₁ = 11.5 Hz, *J*₂ = 2.4 Hz, 1H), 2.86-2.78 (m, 2H), 2.59 (td, *J*₁ = 11.5 Hz, *J*₂ = 2.8 Hz, 1H), 2.34-2.26 (m, 4H), 2.16 (t, *J* = 10.7 Hz, 1H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 155.2 (q), 155.0 (q), 141.3 (q), 128.6 (CH), 128.4 (q), 128.1 (CH), 127.7 (CH), 123.6 (CH), 122.5 (CH), 120.5 (CH), 111.2 (CH), 105.3 (CH), 66.4 (CH), 63.9 (CH₂), 55.2 (CH₂), 52.2 (CH₂), 51.4 (CH₂), 45.8 (CH₃); HRMS: *m/z* [M + H]⁺ calcd for C₂₀H₂₃N₂O: 307.1805; found: 307.1804.

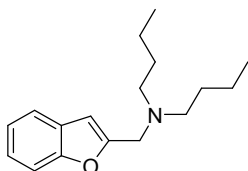


6af: yellow wax; 91% yield (118.1 mg); IR (neat): 2936, 2813, 1586, 1490, 1455, 1010 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 7.56-7.46 (m, 2H), 7.36 (dt, *J*₁ = 7.4 Hz, *J*₂ = 1.7 Hz, 1H), 7.29-7.18 (m, 3H), 7.10 (dt, *J*₁ = 7.4 Hz, *J*₂ = 1.1 Hz, 1H), 7.07-7.00 (m, 1H), 6.60 (s, 1H), 3.71 (s, 2H), 3.63 (s, 2H), 2.60 (bs, 8H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 161.4 (q) (d, *J* = 246 Hz), 155.1 (q), 154.5 (q), 131.7 (CH) (d, *J* = 4 Hz), 128.7 (CH) (d, *J* = 8 Hz), 128.3 (q), 124.5 (q) (d, *J* = 15 Hz), 123.9 (CH), 123.8 (CH) (d, *J* = 3.6 Hz), 122.6 (CH), 120.6 (CH), 115.3 (CH) (d,

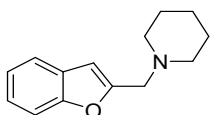
$J = 22$ Hz), 111.3 (CH), 105.7 (CH), 55.4 (CH₂), 55.2 (CH₂) (d, $J = 2$ Hz), 53.0 (CH₂), 52.5 (CH₂); ¹⁹F NMR (376.5 MHz) (CDCl₃): $\delta = -124.5$; HRMS: m/z [M + Na]⁺ calcd for C₂₀H₂₁FN₂O₂Na: 347.1530; found: 347.1529.



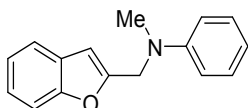
6ag: pale yellow solid; 73% yield (63.4 mg); mp: 85-86°C; IR (neat): 2815, 1450, 1291, 1119, 1006, 939 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): $\delta = 7.55$ (d, $J = 8.0$ Hz, 1H), 7.51 (d, $J = 7.0$ Hz, 1H), 7.32-7.19 (m, 2H), 6.63 (s, 1H), 4.63 (bt, $J = 4.6$ Hz, 4H) 3.70 (s, 2H), 2.57 (bt, $J = 4$ Hz, 4H); ¹³C NMR (100.6 MHz) (CDCl₃): $\delta = 155.1$ (q), 154.0 (q), 128.2 (q), 124.0 (CH), 122.7 (CH), 120.7 (CH), 111.3 (CH), 105.9 (CH), 66.8 (CH₂), 55.9 (CH₂), 53.5 (CH₂); HRMS: m/z [M + H]⁺ calcd for C₁₃H₁₆NO₂: 218.1176; found: 218.1177.



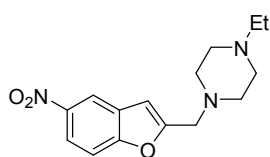
6ah: pale yellow oil; 75% yield (77.8 mg); IR (neat): 2956, 2931, 1454, 1384, 1254, 741 cm⁻¹; ¹H NMR (400.13 MHz) (DMSO-*d*₆): $\delta = 7.60$ -7.56 (m, 1H), 7.55-7.50 (m, 1H), 7.28- 7.16 (m, 2H), 6.72 (s, 1H), 3.74 (s, 2H), 2.43 (bt, $J = 7.0$ Hz, 4H), 1.48-1.38 (m, 4H), 1.28 (sext, $J = 7.5$ Hz, 4H), 0.85 (t, $J = 7.3$, 6H); ¹³C NMR (100.6 MHz) (CDCl₃): $\delta = 156.9$ (q), 154.7 (q), 128.6 (q), 124.1 (CH), 123.1 (CH), 121.1 (CH), 111.3 (CH), 105.2 (CH), 53.5 (CH₂), 50.5 (CH₂), 29.4 (CH₂), 20.4 (CH₂), 14.3 (CH₃); HRMS: m/z [M + H]⁺ calcd for C₁₇H₂₆NO: 260.2009; found: 260.2008.



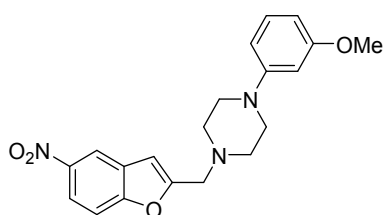
6ai: brown wax; 92% yield (79.2 mg); IR (neat): 2934, 1454, 1384, 1256, 1102, 741 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): $\delta = 7.56$ -7.47 (m, 2H), 7.29- 7.19 (m, 2H), 6.59 (s, 1H), 3.67 (s, 2H), 2.22-2.45 (m, 4H), 1.64 (quint, $J = 5.4$ Hz, 4H), 1.49-1.41 (m, 2H); ¹³C NMR (100.6 MHz) (CDCl₃): $\delta = 155.1$ (q) (overlapping), 128.4 (q), 123.7 (CH), 122.6 (CH), 120.6 (CH), 111.3 (CH), 105.4 (CH), 56.2 (CH₂), 54.4 (CH₂), 25.8 (CH₂), 24.1 (CH₂); HRMS: m/z [M + H]⁺ calcd for C₁₄H₁₈NO: 216.1383; found: 216.1387.



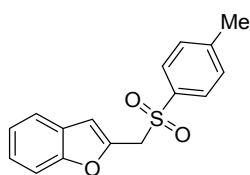
6aj: yellow wax; 48% yield (45.6 mg); mp: IR (neat): 2917, 1600, 1505, 1454, 1383, 1253 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): $\delta = 7.52$ -7.54 (m, 2H), 7.31-7.24 (m, 3H), 7.21 (td, $J_1 = 7.5$ Hz, $J_2 = 1.1$ Hz, 1H), 6.88 (d, $J = 7.9$ Hz, 2H), 6.79 (t, $J = 7.9$ Hz, 1H), 6.53 (d, $J = 0.8$ Hz, 1H), 4.65 (s, 2H), 3.12 (s, 3H); ¹³C NMR (100.6 MHz) (CDCl₃): $\delta = 155.2$ (q), 154.9 (q), 149.1 (q), 129.2 (CH), 128.4 (q), 123.8 (CH), 122.7 (CH), 120.7 (CH), 117.3 (CH), 112.9 (CH), 111.1 (CH), 103.9 (CH), 50.5 (CH₂), 38.7 (CH₃); HRMS: m/z [M + H]⁺ calcd for C₁₆H₁₆NO: 238.1226; found: 238.1232.



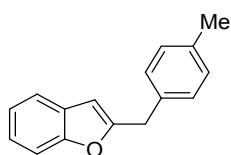
6ba: pale yellow solid; 84% yield (97.2 mg); mp: 89-90°C; IR (neat): 2941, 1520, 1453, 1343, 1265, 1158, 1008 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 8.46 (d, J = 2.3 Hz, 1H), 8.19 (dd, J_1 = 9.0 Hz, J_2 = 2.4 Hz, 1H), 7.54 (d, J = 9.0 Hz, 1H), 6.76 (d, J = 0.3 Hz, 1H), 3.76 (s, 2H), 2.76-2.48 (m, 8H), 2.44 (q, J = 7.2 Hz, 2H), 1.09 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 158.3 (q), 157.9(q), 144.1(q), 128.7 (q), 119.9 (CH), 117.2 (CH), 111.6 (CH), 106.3 (CH), 55.2 (CH_2), 53.1 (CH_2), 52.5 (CH_2), 52.2 (CH_2), 11.9 (CH_3); HRMS: m/z [$\text{M} + \text{H}$] $^+$ calcd for $\text{C}_{15}\text{H}_{20}\text{N}_3\text{O}_3$: 290.1499; found: 290.1504.



6bk: pale yellow solid; 84% yield (123.4 mg); mp: 106-107°C; IR (neat): 1732, 1518, 1446, 1341, 815 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 8.50 (d, J = 2.3 Hz, 1H), 8.23 (dd, J_1 = 9.1 Hz, J_2 = 2.3 Hz, 1H), 7.58 (d, J = 9.0 Hz, 1H), 7.19 (t, J = 8.2 Hz, 1H), 6.82 (s, 1H), 6.56 (dd, J_1 = 8.3 Hz, J_2 = 1.8 Hz, 1H), 6.48 (t, J = 2.3 Hz, 1H), 6.45 (dd, J_1 = 8.0 Hz, J_2 = 2.2 Hz, 1H), 3.82 (s, 2H), 3.81 (s, 3H), 3.31-3.24 (m, 4H), 2.78-2.72 (m, 4H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 160.6 (q), 158.1 (q), 157.9 (q), 152.5 (q), 144.2 (q), 129.8 (CH), 128.7 (q), 120.0 (CH), 117.3 (CH), 111.6 (CH), 109.0 (CH), 106.4 (CH), 104.6 (CH), 102.7 (CH), 55.3 (CH_2), 55.2 (CH_3), 53.1 (CH_2), 49.0 (CH_2); HRMS: m/z [$\text{M} + \text{H}$] $^+$ calcd for $\text{C}_{20}\text{H}_{22}\text{N}_3\text{O}_4$: 368.1605; found: 368.1612.

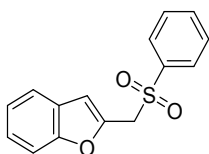


8aa: pale yellow solid; 98% yield (112.2 mg); mp: 194-195°C; IR (neat): 1451, 1310, 1144, 1084 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.57 (d, J = 8.1 Hz, 2H), 7.45(d, J = 7.7 Hz, 1H), 7.28 (d, J = 8.1 Hz, 1H), 7.23-7.11 (m, 4H), 6.59 (s, 1H), 4.45 (s, 2H), 2.35 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 155.2 (q), 145.3 (q), 145.1 (q), 135.3 (q), 129.8 (CH), 128.5 (CH), 127.9 (q), 124.9 (CH), 123.1 (CH), 121.2 (CH), 111.3 (CH), 108.9 (CH), 56.5 (CH_2), 21.7 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{16}\text{H}_{14}\text{O}_3\text{SNa}$: 309.0556; found: 309.0551.

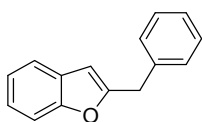


8'aa: pale yellow oil; IR (neat): 2917, 1457, 954, 750 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3) (selected signals): δ = 7.50-7.39 (m, 2H), 7.26-7.13 (m, 6H), 6.38 (d, J = 0.7 Hz, 1H), 4.09 (s, 2H), 2.37 (s, 3H); ^{13}C NMR (100.6 MHz)

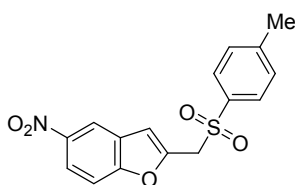
(CDCl₃): δ = 158.1 (q), 155.0 (q), 136.3 (q), 134.1 (q), 129.3 (CH), 128.8 (CH) (overlapping), 123.3 (CH), 122.5 (CH), 120.4 (CH), 110.9 (q), 103.2 (CH), 34.6 (CH₂), 21.1 (CH₃); HRMS: m/z [M + Na]⁺ calcd for C₁₆H₁₄ONa: 245.0937; found: 245.0938.



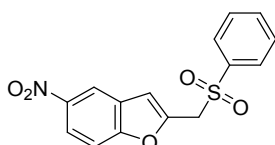
8ab: white solid; 89% yield (96.9 mg); mp: 165-166°C; IR (neat): 1445, 1306, 1147, 1081 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 7.79 (d, J = 7.5 Hz, 2H), 7.66 (t, J = 7.5 Hz, 1H), 7.57-7.49 (m, 3H), 7.37 (d, J = 8.1 Hz, 1H), 7.33-7.21 (m, 2H), 6.70 (s, 1H), 4.58 (s, 2H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 155.2 (q), 145.0 (q), 138.2 (q), 134.1 (CH), 129.1 (CH), 128.5 (CH), 127.9 (q), 125.0 (CH), 123.1 (CH), 121.2 (CH), 111.3 (CH), 109.0 (CH), 56.5 (CH₂); HRMS: m/z [M + Na]⁺ calcd for C₁₅H₁₂O₃SNa: 295.0399; found: 295.0399.



8'ab: yellow oil; 5% yield (4.1 mg); IR (neat): 2922, 2854, 1454, 1252 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 7.42-7.37 (m, 1H), 7.33 (d, J =8.0 Hz, 1H), 7.28-7.21 (m, 4H), 7.28-7.22 (m, 4H), 7.21-7.06 (m, 3H), 6.29 (d, J = 0.6 Hz, 1H), 4.03 (s, 2H), 2.35 (s, 3H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 157.8 (q), 155.0 (q), 137.2 (q), 128.9 (CH), 128.8 (q), 128.6 (CH), 126.8 (CH), 123.4 (CH), 122.5 (CH), 120.4, 110.9. (CH), 103.4 (CH), 35.0 (CH₂); HRMS: m/z [M + Na]⁺ calcd for C₁₅H₁₂ONa: 231.0780; found: 231.0783.

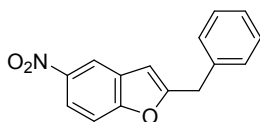


8ba: pale orange solid; 84% yield (111.3 mg); mp: 174-175°C; IR (neat): 1515, 1343, 1323, 1152, 1082 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 8.47 (d, J = 2.3 Hz, 1H), 8.22 (dd, J_1 = 9.1 Hz, J_2 = 2.3 Hz, 1H), 7.67 (d, J = 8.2 Hz, 2H), 7.48 (d, J = 9.1 Hz, 1H), 7.30 (d, J = 8.2 Hz, 2H), 6.81 (d, J = 0.3 Hz, 1H), 4.57 (s, 2H), 2.45 (s, 3H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 157.8 (q), 149.0 (q), 145.6 (q), 144.4 (q), 135.0 (q), 130.0 (CH), 128.5 (CH), 128.2 (q), 120.8 (CH), 117.8 (CH), 111.8 (CH), 109.5 (CH), 56.2 (CH₂), 21.7 (CH₃); HRMS: m/z [M + Na]⁺ calcd for C₁₆H₁₃NO₅SNa: 354.0407; found: 354.040.

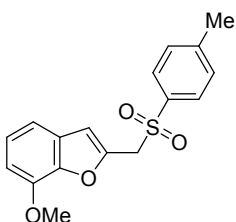


8bb: pale yellow solid; 84% yield (106.6 mg); mp: 214-215°C; IR (neat): 1516, 1346, 1298, 1266, 1148, 1066, 821 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 8.49 (d, J = 2.3 Hz, 1H), 8.24 (dd, J_1 = 9.1 Hz, J_2 = 2.3 Hz, 1H), 7.84-7.79 (m, 2H), 7.72-7.66 (m, 1H), 7.54 (d, J = 7.8 Hz, 2H), 7.48 (d, J = 9.1 Hz, 1H), 6.84 (s, 1H), 4.61 (s, 2H); ¹³C

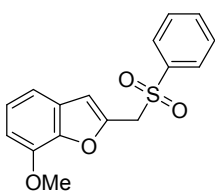
NMR (100.6 MHz) (CDCl₃): δ = 157.8 (q), 148.8 (q), 144.5 (q), 137.9 (q), 134.4 (CH), 129.4 (CH), 128.4 (CH), 128.2 (q), 120.9 (CH), 117.9 (CH), 111.8 (CH), 109.6 (CH), 56.2 (CH₂); HRMS: m/z [M + Na]⁺ calcd for C₁₅H₁₁NO₅Na: 340.0250; found: 340.0246.



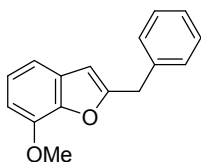
8'bb: pale yellow gum; 9% yield (9.1 mg); IR (neat): 2923, 2853, 1517, 1341, 1262 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 8.42 (d, J = 2.4 Hz, 1H), 8.18 (dd, J_1 = 9.1, J_2 = 2.4 Hz, 1H), 7.50 (d, J = 9.1 Hz, 1H), 7.41–7.30 (m, 5H), 6.53 (d, J = 0.9 Hz, 1H), 4.17 (s, 2H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 161.5 (q), 157.8 (q), 144.1 (q), 136.1 (q), 129.2 (q), 128.9 (CH), 128.8 (CH), 127.2 (CH), 119.5 (CH), 116.9 (CH), 111.2 (CH), 104.1 (CH), 35.0 (CH₂), 29.7 (CH₃); HRMS: m/z [M + Na]⁺ calcd for C₁₅H₁₁NO₃Na: 276.0631; found: 276.0628.



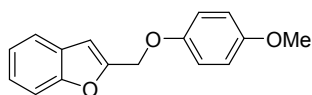
8ca: pale yellow oil; 84% yield (106.3 mg); IR (neat): 2929, 2840, 1492, 1286, 1148, 1084 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 7.68 (d, J = 8.2 Hz, 2H), 7.31–7.26 (m, 2H), 7.20–7.13 (m, 2H), 6.80 (dd, J_1 = 6.5 Hz, J_2 = 2.4 Hz, 1H), 6.75 (s, 1H), 4.57 (s, 2H), 3.95 (s, 3H), 2.45 (s, 3H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 145.4 (q), 145.2 (q), 145.1 (q), 144.6 (q), 135.4 (q), 129.8 (CH), 129.7 (q), 128.6 (CH), 123.8 (CH), 113.5 (CH), 109.0 (CH), 107.2 (CH), 56.3 (CH₂), 56.1 (CH₃), 21.6 (CH₃); HRMS: m/z [M + Na]⁺ calcd for C₁₇H₁₆O₄Na: 339.0662; found: 339.0655.



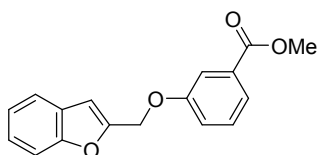
8cb: pale yellow oil; 91% yield (110.0 mg); IR (neat): 2931, 2841, 1483, 1285, 1149, 1083 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 7.84–7.78 (m, 2H), 7.65 (t, J = 7.5 Hz, 1H), 7.51 (t, J = 7.9 Hz, 2H), 7.20–7.12 (m, 2H), 6.80 (dd, J_1 = 6.7 Hz, J_2 = 2.1 Hz, 1H), 6.75 (s, 1H), 4.60 (s, 2H), 3.94 (s, 3H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 145.4 (q), 145.3 (q), 144.7 (q), 138.4 (q), 134.1 (CH), 129.8 (q), 129.3 (CH), 128.7 (CH), 124.0 (CH), 113.6 (CH), 109.3 (CH), 107.4 (CH), 56.4 (CH₂), 56.2 (CH₃); HRMS: m/z [M + Na]⁺ calcd for C₁₆H₁₄O₄Na: 325.0505; found: 325.0501.



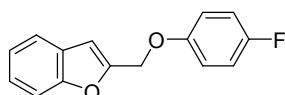
8'cb: pale yellow oil; 6% yield (5.7 mg); IR (neat): 2928, 1629, 1492, 1149, 1105 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.38-7.30 (m, 4H), 7.30-7.25 (m, 1H), 7.16-7.01 (m, 2H), 6.76 (dd, J_1 = 7.6 Hz, J_2 = 1.2 Hz, 1H), 6.34 (s, 1H), 4.16 (s, 2H), 4.01 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 158.1 (q), 145.0 (q), 144.0 (q), 137.1 (q), 130.5 (q), 129.0 (CH), 128.6 (CH), 126.7 (CH), 123.2 (CH), 112.9 (CH), 105.7 (CH), 103.7 (CH), 56.0 (CH_2), 35.9 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{16}\text{H}_{14}\text{O}_2\text{Na}$: 261.0886; found: 261.0886.



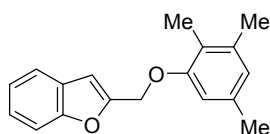
10aa: white solid; 90% yield (91.5 mg); mp: 92-93 $^\circ\text{C}$; IR (neat): 2926, 1505, 1453, 1376, 1225, 1030 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.59 (d, J = 7.6 Hz, 1H), 7.53 (d, J = 8.2 Hz, 1H), 7.35-7.30 (m, 1H), 7.28-7.23 (m, 1H), 6.98 (d, J = 9.1 Hz, 2H), 6.88 (d, J = 9.1 Hz, 2H), 6.79 (s, 1H), 5.14 (s, 2H), 3.80 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 155.2 (q), 154.4 (q), 153.2 (q), 152.4 (q), 128.0 (q), 124.6 (CH), 122.9 (CH), 121.2 (CH), 116.2 (CH), 114.7 (CH), 111.4 (CH), 106.0 (CH), 64.0 (CH_2), 55.7 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{16}\text{H}_{14}\text{O}_3\text{Na}$: 277.0835; found: 277.0833.



10ab: beige solid; 92% yield (103.9 mg); mp: 70-71 $^\circ\text{C}$; IR (neat): 1717, 1591, 1220, 1101, 1073, 1015 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.76-7.68 (m, 2H), 7.59 (d, J = 7.8 Hz, 1H), 7.53 (d, J = 8.1 Hz, 1H), 7.39 (t, J = 8.1 Hz, 1H), 7.36-7.30 (m, 1H), 7.17-7.11 (m, 2H), 6.72 (s, 1H), 5.24 (s, 2H), 3.94 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 166.8 (q), 158.2 (q), 155.3 (q), 152.3 (q), 131.6 (q), 129.6 (CH), 127.9 (q), 124.7 (CH), 123.0 (CH), 122.8 (CH), 121.3 (CH), 120.4 (CH), 115.1 (CH), 111.4 (CH), 106.5 (CH), 63.2 (CH_2), 52.2 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{17}\text{H}_{14}\text{O}_4\text{Na}$: 305.0784; found: 305.0782.

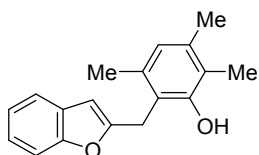


10ac: yellow solid; 98% yield (95.0 mg); mp: 90-91 $^\circ\text{C}$; IR (neat): 2930, 1602, 1506, 1453, 1226, 1173, 1093 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.57 (d, J = 7.2 Hz, 1H), 7.50 (d, J = 8.2 Hz, 1H), 7.33-7.21 (m, 2H), 7.03-6.93 (m, 4H), 6.78 (s, 1H), 5.13 (s, 2H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 157.7 (d, J = 239.2 Hz) (q), 155.2 (q), 154.4 (d, J = 2.10 Hz) (q), 152.7 (q), 127.9 (q), 124.7 (CH), 123.0 (CH), 121.2 (CH), 116.2 (CH, J = 8.0 Hz), 115.9 (CH, J = 23.0 Hz), 111.4 (CH), 106.3 (CH), 63.9 (CH_2); ^{19}F NMR (376.5 MHz) (CDCl_3): δ = (-122.879) - (-122.946) HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{15}\text{H}_{11}\text{FO}_2\text{Na}$: 265.0635; found: 265.0638.

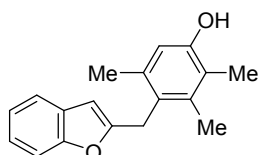


10ad: white solid; 75% yield (79.9 mg); mp: 85-86 $^\circ\text{C}$; IR (neat): 2923, 1754, 1453, 1384, 1255, 1208, 1098 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.60 (d, J = 7.5 Hz, 1H), 7.53 (d, J = 8.0 Hz, 1H), 7.36-7.23 (m, 2H), 6.79

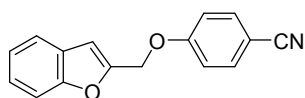
(s, 1H), 6.70 (s, 2H), 5.16 (s, 2H), 2.33 (s, 3H), 2.27 (s, 3H), 2.17 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 156.3 (q), 155.1 (q), 153.7 (q), 137.9 (q), 135.5 (q), 128.2 (q), 124.4 (CH), 123.9 (CH), 122.8 (CH), 122.7 (q), 121.1 (CH), 111.4 (CH), 110.9 (CH), 105.5 (CH), 63.8 (CH_2), 21.3 (CH_3), 20.0 (CH_3), 11.4 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{18}\text{H}_{18}\text{O}_2\text{Na}$: 289.1199; found: 289.1198.



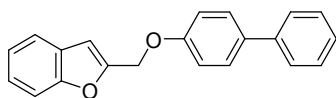
11a: white solid; 6% yield (6.4 mg); mp: 120-121°C; IR (neat): 3390, 2923, 1454, 1160 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.48-7.40 (m, 2H), 7.25-7.14 (m, 2H), 6.69 (s, 1H), 6.31 (d, J = 0.8 Hz, 1H), 5.08 (s, 1H), 4.14 (s, 2H), 2.35 (s, 3H), 2.27 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 157.1 (q), 154.8 (q), 152.2 (q), 136.1 (q), 134.6 (q), 128.8 (q), 124.3 (CH), 123.4 (CH), 122.6 (CH), 120.32 (CH), 120.26 (q), 119.5 (q), 110.9 (CH), 102.5 (CH), 26.1 (CH_2), 20.0 (CH_3), 19.4 (CH_3), 11.7 (CH_3); HRMS: m/z [$\text{M} + \text{H}$] $^+$ calcd for $\text{C}_{18}\text{H}_{19}\text{O}_2$: 267.1380; found: 267.1377.



11b: pale yellow oil; 8% yield (8.5 mg); IR (neat): 3388, 2935, 1440, 1161 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3) (selected signals) : δ = 7.42 (d, 1H, J = 8.1 Hz), 7.25-7.12 (m, 3H), 6.57 (s, 1H), 6.07 (d, 1H, J = 0.8 Hz), 4.65 (s, 1H), 4.10 (s, 2H), 2.31 (s, 3H), 2.27 (s, 3H), 2.11 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3) (selected signals) : δ = 158.0 (q), 154.9 (q), 152.1 (q), 137.3(q), 135.2(q), 128.9(q), 126.0(q), 123.1 (CH), 122.4 (CH), 120.3 (q), 120.2 (CH), 114.6 (CH), 110.8 (CH), 102.5 (CH), 29.0 (CH_2), 20.1 (CH_3), 16.1 (CH_3), 12.0 (CH_3); HRMS: m/z [$\text{M} + \text{H}$] $^+$ calcd for $\text{C}_{18}\text{H}_{19}\text{O}_2$: 267.1380; found: 267.1378.

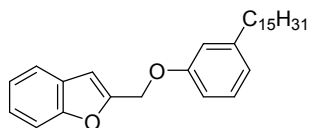


10ae: pale yellow solid; 84% yield (83.7 mg); mp: 103-104°C; IR (neat): 2923, 2219, 1602, 1451, 1243, 1172, 1000 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.67-7.58 (m, 3H), 7.52 (d, J = 8.1 Hz, 1 H), 7.38-7.32 (m, 1H), 7.10 (d, J = 8.9 Hz, 1H), 6.84 (s, 1H), 5.24 (s, 2H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 161.4 (q), 155.3 (q), 151.4 (q), 134.1 (CH), 127.7 (q), 125.0 (CH), 123.1 (CH), 122.7 (q), 121.4 (CH), 119.0 (q), 115.6 (CH), 111.5 (CH), 106.9 (CH), 104.8 (q), 63.1 (CH_2); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{16}\text{H}_{11}\text{NO}_2\text{Na}$: 272.0682; found: 272.0683.

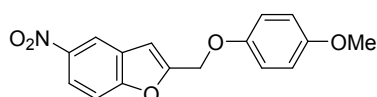


10af: white solid; 87% yield (104.5 mg); mp: 147-148°C; IR (neat): 1722, 1592, 1488, 1223, 1102, 1074 cm^{-1} ; ^1H NMR (400.13 MHz) ($\text{DMSO}-d_6$): δ = 7.67 (d, 1H, J = 7.5 Hz), 7.65-7.58 (m, 5H), 7.44 (t, J = 7.8 Hz, 2H), 7.37-7.22 (m, 3H), 7.18 (d, J = 8.6 Hz, 2H), 7.09 (bs, 1H), 5.33 (s, 2H); ^{13}C NMR (100.6 MHz) ($\text{DMSO}-d_6$): δ = 157.9

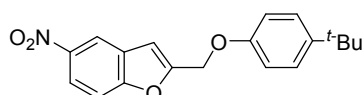
(q), 155.0 (q), 153.5 (q), 140.2 (q), 133.6 (q), 129.3 (CH), 128.3 (CH), 128.1 (q), 127.3 (CH), 126.7 (CH), 125.2 (CH), 123.5 (CH), 121.9 (CH), 115.8 (CH), 111.7 (CH), 107.2 (CH), 62.7 (CH₂); HRMS: m/z [M + Na]⁺ calcd for C₂₁H₁₆O₂Na: 323.104; found: 323.1045.



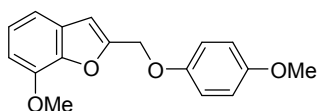
10ag: white solid; 82% yield (142.6 mg); mp: 59-60°C; IR (neat): 1915, 2847, 1593, 1464, 1257, 1023 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 7.61-7.56 (m, 1H), 7.54-7.50 (m, 1H), 7.34-7.29 (m, 1H), 7.27-7.19 (m, 2H), 6.89-6.82 (m, 3H), 6.80 (s, 1H), 5.18 (s, 2H), 2.60 (t, J = 7.6 Hz, 2H), 1.68-1.58 (m, 2H), 1.38-1.24 (m, 26H), 0.91 (t, J = 6.8 Hz, 3H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 158.3 (q), 155.2 (q), 153.1 (q), 144.8 (q), 129.2 (CH), 128.1 (q), 124.5 (CH), 122.9 (CH), 121.7 (CH), 121.2 (CH), 115.3 (CH), 111.7 (CH), 111.4 (CH), 106.0 (CH), 63.0 (CH₂), 36.0 (CH₂), 31.9 (CH₂), 31.3 (CH₂), 29.71 (CH₂), 29.69 (CH₂), 29.67 (CH₂), 29.60 (CH₂), 29.5 (CH₂), 29.4 (CH₂), 29.3 (CH₂), 22.7 (CH₂), 14.1 (CH₃); HRMS: m/z [M + Na]⁺ calcd for C₃₀H₄₂O₂Na: 457.3077; found: 457.3080.



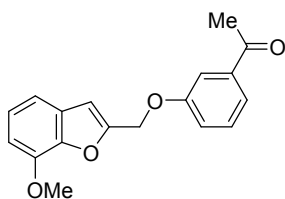
10ba: pale yellow solid; 87% yield (104.2 mg); mp: 110-111°C; IR (neat): 1506, 1340, 1225, 1034 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 8.52 (d, J = 2.3 Hz, 1H), 8.26 (dd, J_1 = 9.0 Hz, J_2 = 2.3 Hz, 1H), 7.60 (d, J = 9.0 Hz, 1H), 6.98 (d, J = 9.1 Hz, 2H), 6.94-6.91 (m, 1H), 6.88 (d, J = 9.1 Hz, 2H), 6.80 (d, J = 2.7 Hz, 1H), 5.07 (s, 2H), 3.71 (s, 3H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 157.9 (q), 156.8 (q), 154.7 (q), 152.1 (q), 144.3 (q), 128.4 (q), 120.5 (CH), 117.8 (CH), 116.2 (CH), 114.8 (CH), 111.8 (CH), 106.5 (CH), 63.7 (CH₂), 55.7 (CH₃); HRMS: m/z [M + Na]⁺ calcd for C₁₆H₁₃NO₅Na: 322.0686; found: 322.0685.



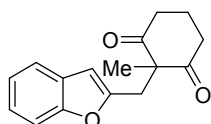
10bh: pale yellow solid; 93% yield (121.0 mg); mp: 98-99°C; IR (neat): 2924, 1509, 1342, 1230, 1019 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 8.52 (d, J = 2.3 Hz, 1H), 8.25 (dd, J_1 = 9.0 Hz, J_2 = 2.4 Hz, 1H), 7.59 (d, J = 9.1 Hz, 1H), 7.40-7.34 (m, 2H), 7.01-6.93 (m, 3H), 5.21 (s, 2H), 1.34 (s, 9H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 157.9 (q), 156.8 (q), 155.7 (q), 144.6 (q), 144.3 (q), 128.5 (q), 126.5 (CH), 120.4 (CH), 117.7 (CH), 114.3 (CH), 111.8 (CH), 106.5 (CH), 62.8 (CH₂), 34.2 (q), 31.5 (CH₃); HRMS: m/z [M + Na]⁺ calcd for C₁₉H₁₉NO₄Na: 348.1206; found: 348.1208.



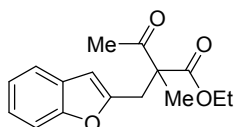
10ca: white solid; 90% yield (102.3 mg); mp: 101-102°C; IR (neat): 2930, 1506, 1283, 1226, 1173, 1093 cm⁻¹; ¹H NMR (400.13 MHz) (CDCl₃): δ = 7.20-7.16 (m, 2H), 6.97-6.95 (m, 2H), 6.89-6.80 (m, 3H), 6.77 (s, 1H), 5.15 (s, 2H), 4.04 (s, 3H), 3.79 (s, 3H); ¹³C NMR (100.6 MHz) (CDCl₃): δ = 154.3 (q), 153.4 (q), 152.4 (q), 145.4 (q), 144.4 (q), 129.7 (q), 123.6 (CH), 116.1 (CH), 114.6 (CH), 113.5 (CH), 106.6 (CH), 106.2 (CH), 63.8 (CH₂), 56.0 (CH₃), 55.7 (CH₃); HRMS: m/z [M + Na]⁺ calcd for C₁₇H₁₆O₄Na: 307.0941; found: 307.0941.



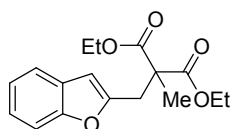
10ci: yellow wax; 85% yield (100.7 mg); IR (neat): 2941, 1682, 1583, 1493, 1264, 1199, 1016 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.65-7.62 (m, 1H), 7.60 (d, 1H, J = 7.7 Hz), 7.40 (t, 1H, J = 8.0 Hz), 7.24-7.16 (m, 3H), 6.85-6.83 (m, 2H), 5.25 (s, 2H), 4.04 (s, 3H), 2.62 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 197.8 (q), 158.4 (q), 152.5 (q), 145.4 (q), 144.5 (q), 138.5 (q), 129.7 (CH), 129.6 (q), 123.7 (CH), 121.7 (CH), 120.4 (CH), 113.61 (CH), 113.57 (CH), 106.83 (CH), 106.76 (CH), 63.0 (CH_2), 56.0 (CH_3), 26.7 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{18}\text{H}_{16}\text{O}_4\text{Na}$: 319.0941; found: 319.0937.



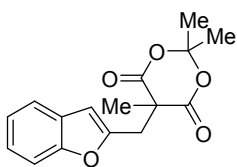
13aa: pale yellow oil; 87% yield (89.3 mg); IR (neat): 2961, 1726, 1694, 1453, 1250, 1172 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.49-7.44 (m, 1H), 7.38-7.34 (m, 1H), 7.24-7.15 (m, 2H), 6.40 (d, J = 0.8 Hz, 1H), 3.36 (s, 2H), 2.77-2.56 (m, 4H), 2.02-1.90 (m, 1H), 1.89-1.77 (m, 1H), 1.40 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 210.0 (q), 154.4 (q), 154.3 (q), 128.5 (q), 123.6 (CH), 122.7 (CH), 120.6 (CH), 110.8 (CH), 104.6 (CH), 63.3 (q), 38.3 (CH_2), 34.9 (CH_2), 23.1 (CH_3), 17.2 (CH_2); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{16}\text{H}_{16}\text{O}_3\text{Na}$: 279.0992; found: 279.0992.



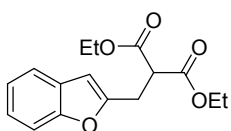
13ab: pale yellow oil; 86% yield (94.4 mg); IR (neat): 2983, 2928, 1712, 1454, 1356, 1250, 1018 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.52-7.47 (m, 1H), 7.42-7.36 (m, 1H), 7.27-7.16 (m, 2H), 6.47 (d, J = 0.7 Hz, 1H), 4.26 (dq, J_1 = 7.2 Hz, J_2 = 1.3 Hz, 2H), 3.43 (d, J = 15.2 Hz, 1H), 3.31 (d, J = 15.2 Hz, 1H), 2.25 (s, 3H), 1.42 (s, 3H), 1.29 (t, J = 7.12 Hz, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 204.4 (q), 172.0 (q), 154.8 (q), 154.2 (q), 128.5 (q), 123.7 (CH), 122.6 (CH), 120.5 (CH), 110.9 (CH), 105.4 (CH), 61.8 (CH_2), 59.6 (q), 33.7 (CH_2), 26.1 (CH_3), 19.2 (CH_3), 14.0 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{16}\text{H}_{18}\text{O}_4\text{Na}$: 297.1097; found: 297.1094.



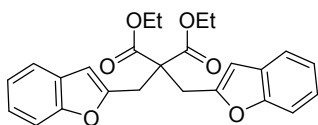
13ac: pale yellow oil; 70% yield (85.2 mg); IR (neat): 2982, 2939, 1730, 1454, 1238, 1174, 1106, 1019 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.52-7.48 (m, 1H), 7.42-7.37 (m, 1H), 7.27-7.16 (m, 2H), 6.49 (s, J = 0.5 Hz, 1H), 4.26 (dq, J_1 = 7.1 Hz, J_2 = 1.8 Hz, 4H), 3.43 (s, 2H), 1.48 (s, 3H), 1.29 (t, J = 7.1 Hz, 6H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 171.4 (q), 154.8 (q), 154.1 (q), 128.5 (q), 123.7 (CH), 122.6 (CH), 120.5 (CH), 110.9 (CH), 105.3 (CH), 61.6 (CH_2), 53.7 (q), 34.5 (CH_2), 19.9 (CH_3), 14.0 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{17}\text{H}_{20}\text{O}_5\text{Na}$: 327.1203; found: 327.1202.



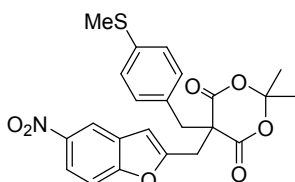
13ad: pale yellow oil; 97% yield (111.9 mg); IR (neat): 2923, 1776, 1737, 1453, 1380, 1251, 1055 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.52-7.47 (m, 1H), 7.44-7.40 (m, 1H), 7.25 (td, $J_1 = 7.2$, $J_2 = 1.4$, 1H), 7.25 (td, $J_1 = 7.2$ Hz, $J_2 = 1.4$ Hz, 1H), 6.55 (s, 1H), 3.56 (s, 2H), 1.80 (s, 3H), 1.71 (s, 3H), 1.28 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 169.3 (q), 154.7 (q), 152.2 (q), 128.1 (q), 124.3 (CH), 122.9 (CH), 120.9 (CH), 111.1 (CH), 106.0 (CH), 105.5 (q), 49.6 (q), 37.8 (CH_2), 29.3 (CH_3), 28.4 (CH_3), 25.7 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{16}\text{H}_{16}\text{O}_5\text{Na}$: 311.0890; found: 311.0890.



13ae: pale yellow oil; 57% yield (70 mg, on 0.42 mmol scale); IR (neat): 2982, 1730, 1454, 1251, 1147, 1031 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.50 (d, $J = 7.2$ Hz, 1H), 7.42 (d, $J = 7.5$ Hz, 1H), 7.27-7.17 (m, 2H), 6.50 (s, 1H), 4.30-4.16 (m, 4H), 3.87 (t, $J = 7.3$ Hz, 1H), 3.4 (d, $J = 7.6$ Hz, 2H), 1.27 (t, $J = 7.1$ Hz, 6H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 168.4 (q), 161.4 (q), 154.8 (q), 128.6 (q), 123.7 (CH), 122.6 (CH), 120.5 (CH), 110.9 (CH), 103.8 (CH), 61.8 (CH_2), 50.7 (CH), 27.8 (CH_2), 14.0 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{16}\text{H}_{18}\text{O}_5\text{Na}$: 313.1046; found: 313.1046.



13'ae: pale yellow oil; 15% yield (15.0 mg, on 0.42 mmol scale); IR (neat): 2981, 1732, 1454, 1252, 1170, 1041 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 7.54 (d, $J = 7.3$ Hz, 2H), 7.43 (d, $J = 7.9$ Hz, 2H), 7.28-7.20 (m, 4H), 6.60 (s, 2H), 4.30 (q, $J = 7.3$ Hz, 4H), 3.49 (s, 4H), 1.31 (t, $J = 7.3$ Hz, 8H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 169.8 (q), 153.7 (q), 128.4 (q), 123.8 (CH), 122.7 (CH), 120.6 (CH), 120.5 (q), 110.9 (CH), 106.0 (CH), 62.0 (CH_2), 57.1 (q), 31.5 (CH_2), 14.0 (CH_3); HRMS: m/z [$\text{M} + \text{Na}$] $^+$ calcd for $\text{C}_{25}\text{H}_{24}\text{O}_6\text{Na}$: 443.1465; found: 443.1462.



13bf: pale yellow solid; 98% yield (178.5 mg); mp: 150-151 $^{\circ}\text{C}$; IR (neat): 1962, 1740, 1521, 1440, 1341, 1259, 1085, 1014 cm^{-1} ; ^1H NMR (400.13 MHz) (CDCl_3): δ = 8.46 (d, $J = 2.3$ Hz, 1H), 8.21 (dd, $J_1 = 9.1$, Hz, $J_2 = 2.3$ Hz, 1H), 7.53 (d, $J = 9.1$ Hz, 1H), 7.21 (d, $J = 8.4$ Hz, 2H), 7.15 (d, $J = 8.4$ Hz, 2H), 6.77 (s, 1H), 3.69 (s, 2H), 3.45 (s, 2H), 2.45 (s, 3H), 1.00 (s, 3H), 0.76 (s, 3H); ^{13}C NMR (100.6 MHz) (CDCl_3): δ = 167.5 (q), 157.4 (q), 155.6 (q), 144.4 (q), 139.0 (q), 130.64 (CH), 130.61 (q), 128.4 (q), 126.8 (CH), 120.5 (CH), 117.6 (CH), 11.53 (CH), 107.3

(CH), 106.3 (q), 56.9 (q), 44.5 (CH₂), 37.8 (CH₂), 29.0 (CH₃), 28.6 (CH₃), 15.7 (CH₃); HRMS: m/z [M + Na]⁺ calcd for C₂₃H₂₁NO₇SNa: 478.0931; found: 478.0933.

REFERENCES

¹ Mukhanova, T. I.; Kukushkin, S. Y.; Ivanov, P. Y.; Alekseeva, L. M.; Granik, V. G. Russ. Chem. Bull. 2007, 56, 325-329.