Supporting Information

Radical (Phenylsulfonyl)difluoromethylation with Iododifluoromethyl Phenyl Sulfone

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Table of contents

General	S2
Preparation and physical data of compounds 3 and 4	S3-6
Preparation and physical data of compounds 5	S7-9
Preparation and physical data of compounds 6	S10
Preparation and physical data of compounds 7	S11-13
NMR spectra for all products.....	S14-81
Proposed mechanism for the formation of 4	S-82

General:

Unless otherwise mentioned, solvents and reagent were purchased from commercial sources and used as received. ^1H NMR spectra were recorded with Me_4Si as internal standard. ^{19}F NMR spectra were recorded with CFCl_3 as external standard. ^{13}C NMR spectra were recorded on the 300-MHz, 500-MHz or 400-MHz NMR spectrometers. Low-resolution Mass spectra were obtained on a low-resolution mass spectrometer. High-resolution mass data were recorded on a high-resolution mass spectrometer in the EI, ESI or MALDI mode.

Typical procedure for the radical addition of iododifluoromethyl phenyl sulfone with alkenes:

In the presence of air, into a 30-mL Schlenk flask containing iododifluoromethyl phenyl sulfone (**1**) (64 mg, 0.2 mmol) and 1-hexene (**2a**) (34 mg, 0.4 mmol) in 1.2 mL of CH₂Cl₂ at –30 °C, was added Et₃B (0.2 mL, 1.0 mol in hexane). The reaction mixture was stirred at this temperature for 0.5 h. After removing of volatile solvents under vacuum, the crude product was further purified by silica gel column chromatography with ethyl acetate/petroleum ether (1: 30) to give product **3a** as an oil, yield 75% (60 mg).

oil; IR (film): 2931, 1585, 1449, 1341, 1164, 1071, 718, 685cm⁻¹; ¹H NMR: δ 7.98 (d, J = 8.1 Hz, 2H), 7.76-7.81 (m, 1H), 7.61-7.66 (m, 2H), 4.30-4.39 (m, 1H), 2.97-3.27 (m, 2H), 1.75-1.85 (m, 2H), 1.25-1.52 (m, 4H), 0.91 (t, J=7.2Hz, 3H); ¹⁹F NMR: δ –101.75 (ddd, J = 227.8, 25.8, 11.0Hz, 1F), –103.26 (ddd, J = 228.4, 23.7, 11.3Hz, 1F); ¹³C NMR: δ 135.5, 131.8, 130.8, 129.3, 123.4 (t, J = 287.5 Hz), 40.0, 39.7 (t, J = 19.1 Hz), 31.4, 21.6, 21.1, 13.8; EI (m/z %): 275 (M⁺-127, 6.8), 143(100.0); HRMS (EI) calcd. For C₁₃H₁₇F₂IO₂S (M⁺-I): 275.0917, Found 275.0919.

oil; IR (film): 2925, 2360, 1584, 1448, 1347, 1163, 717, 614 cm⁻¹; ¹H NMR: δ 7.98 (d, J = 8.1 Hz, 2H), 7.76-7.81 (m, 1H), 7.60-7.66 (m, 2H), 4.30-4.39 (m, 1H), 3.02-3.21 (m, 2H), 1.70-1.87 (m, 2H), 1.25-1.55 (m, 16H), 0.87 (t, J=6.6Hz, 3H); ¹⁹F NMR: δ –101.69 (ddd, J = 226.4, 26.2, 10.7 Hz, 1F), –103.28 (ddd, J = 226.1, 25.2, 11.3 Hz, 1F); ¹³C NMR: δ 135.5, 131.8, 130.8, 129.3, 123.4 (t, J = 287.3 Hz), 40.3, 39.7 (t, J = 19.0 Hz), 31.8, 29.5, 29.4, 29.3, 29.2, 28.5, 22.6, 21.2, 14.0; ESI 504(M+NH₄⁺); HRMS (ESI) calcd. For C₁₉H₂₉F₂IO₂SNa (M+Na⁺): 509.07932, Found

509.07928.

solid; IR (KBr): 2953, 1582, 1450, 1338, 1254, 1157, 847, 723 cm^{-1} ; ^1H NMR: δ 8.01 (d, J = 7.2 Hz, 2H), 7.68-7.82 (m, 1H), 7.62-7.68 (m, 2H), 3.25 (dd, J = 10.8, 2.7 Hz, 1H), 3.04-3.22 (m, 1H), 2.70-2.90 (m, 1H), 0.22 (s, 9H); ^{19}F NMR: δ -102.02 (ddd, J = 224.7, 26.8, 7.0 Hz, 1F), -106.01 (ddd, J = 224.4, 26.2, 8.8 Hz, 1F); ^{13}C NMR: δ 135.4, 132.0, 130.8, 129.3, 123.6 (t, J = 286.0 Hz), 33.3 (t, J = 19.6 Hz), 1.7, -2.3; ESI: 436 ($\text{M}+\text{NH}_4^+$), 441 ($\text{M}+\text{Na}^+$); HRMS (ESI) calcd. For $\text{C}_{12}\text{H}_{17}\text{F}_2\text{IO}_2\text{SSiNa}$ ($\text{M}+\text{Na}^+$): 440.96235, Found 440.9617.

oil; IR (film): 2360, 1715, 1583, 1448, 1339, 1163, 717, 685 cm^{-1} ; ^1H NMR δ 7.98 (d, J =7.2Hz, 2H), 7.65-7.81 (m, 1H), 7.61-7.66 (m, 2H), 4.34-4.43 (m, 1H), 2.96-3.30 (m, 2H), 2.60-2.75 (m, 2H), 2.17 (s, 3H), 1.92-2.15 (m, 2H); ^{19}F NMR δ -101.47 (ddd, J =228.6, 26.8, 8.5Hz, 1F), -103.25 (ddd, J =228.3, 23.4, 9.3Hz, 1F); ^{13}C NMR δ 206.4, 135.6, 131.6, 130.8, 129.4, 123.2 (t, J =287.3Hz), 43.4, 39.9 (t, J =19.1Hz), 33.9, 30.0, 20.1; ESI 417 ($\text{M}+\text{H}^+$), 434 ($\text{M}+\text{NH}_4^+$); HRMS (ESI) calcd. For $\text{C}_{13}\text{H}_{16}\text{F}_2\text{IO}_3\text{S}$ (M^+): 416.98274, Found 416.9847.

oil; IR (film): 2939, 1736, 1584, 1448, 1344, 1242, 1163, 717 cm^{-1} ; ^1H NMR: δ 7.98 (d, J = 8.7 Hz, 2H), 7.76-7.81 (m, 1H), 7.61-7.66 (m, 2H), 4.34-4.40 (m, 1H), 4.05-4.09 (m, 2H), 3.00-3.23 (m, 2H), 2.05 (s, 3H), 1.79-1.88 (m, 2H), 1.43-1.72 (m, 4H); ^{19}F NMR: δ -101.47 (ddd, J = 227.6, 25.3, 10.2Hz, 1F), -103.25 (ddd, J = 227.0, 23.4, 10.7Hz, 1F); ^{13}C NMR: δ 171.1, 135.6, 131.6, 130.8, 129.4, 123.3 (t, J = 294.6Hz), 64.0, 39.8 (t, J = 18.4 Hz), 39.7, 27.5, 26.0, 20.9, 20.6; ESI

478 ($M+NH_4^+$), 483 ($M+Na^+$); HRMS (ESI) calcd. For $C_{15}H_{19}F_2IO_4SNa$ ($M+Na^+$): 482.99090, Found 482.9925.

solid; IR (KBr) 1702, 1449, 1338, 1164, 717, 605 cm^{-1} ; 1H NMR: δ 7.98 (d, J = 8.1 Hz, 2H), 7.69-7.81 (m, 1H), 7.61-7.66 (m, 2H), 4.36-4.45 (m, 1H), 2.97-3.32 (m, 2H), 2.50-2.71 (m, 2H), 2.01-2.23 (m, 2H); ^{19}F NMR: δ -101.24 (ddd, J = 228.3, 27.9, 8.7Hz, 1F), -103.25 (ddd, J = 228.3, 24.8, 8.8Hz, 1F); ^{13}C NMR: δ 177.7, 135.6, 131.6, 130.9, 129.4, 123.2 (t, J = 287.2 Hz), 39.9 (t, J = 19.7Hz), 34.8, 34.1, 19.2; ESI 419 ($M+H^+$), 436 ($M+NH_4^+$); HRMS (ESI) calcd. For $C_{12}H_{13}F_2IO_4SNa$ ($M+Na^+$): 440.94395, Found 440.9450.

oil; IR (film): 3542, 2359, 1583, 1448, 1337, 1162, 1054, 684 cm^{-1} ; 1H NMR: δ 7.98 (d, J = 7.5 Hz, 2H), 7.77-7.82 (m, 1H), 7.61-7.66 (m, 2H), 4.45-4.52 (m, 1H), 3.78-3.82 (m, 2H), 2.94-3.38 (m, 2H), 2.20-2.24 (m, 1H); ^{19}F NMR: δ -102.02 (ddd, J = 227.8, 23.1, 11.6 Hz, 1F), -102.32 (ddd, J = 228.3, 22.6, 12.7 Hz, 1F); ^{13}C NMR: δ 135.6, 131.6, 130.8, 129.4, 123.2 (t, J = 286.2 Hz), 67.8, 36.0 (t, J = 19.5 Hz), 22.4; ESI: 394 ($M+NH_4^+$), 399 ($M+Na^+$); HRMS (ESI) calcd. For $C_{10}H_{11}F_2IO_3SNa$ ($M+Na^+$): 398.93339, Found 398.9351.

solid; IR (KBr): 2921, 1590, 1508, 1464, 1338, 1232, 1162, 1066, 830, 717 cm^{-1} ; 1H NMR: δ 7.98 (d, J = 8.4 Hz, 2H), 7.75-7.80 (m, 1H), 7.60-7.65 (m, 2H), 6.80-6.87 (m, 4H), 4.49-4.57 (m, 1H), 4.09-4.25 (m, 2H), 3.76 (s, 3H), 3.36-3.54 (m, 1H), 2.95-3.15 (m, 1H); ^{19}F NMR: δ -102.66 (td, J = 18.2, 9.6 Hz, 2F); ^{13}C NMR: δ 154.6, 151.8, 135.6, 131.6, 130.9, 129.4, 123.2 (t, J = 286.2 Hz),

116.2, 114.7, 73.6, 55.7, 36.1 (t, J = 19.2 Hz), 14.0; ESI: 500 (M+NH₄⁺), 505 (M+Na⁺); HRMS (MALDI) calcd. For C₁₇H₁₇F₂IO₄SNa (M+Na⁺): 504.97525, Found 504.9771

oil; IR (film): 2981, 1646, 1585, 1450, 1344, 1166, 1072, 717, 687 cm⁻¹; ¹H NMR: δ 7.98 (d, J = 7.8 Hz, 2H), 7.74-7.79 (m, 1H), 7.59-7.64 (m, 2H), 4.97 (t, J = 5.4 Hz, 1H), 3.50-3.70 (m, 4H), 2.70 (td, J = 18.0, 5.7 Hz, 2H), 1.20 (t, J = 7.2 Hz, 6H); ¹⁹F NMR: δ -102.69 (t, J = 18.0 Hz, 2F); ¹³C NMR: δ 135.3, 132.1, 130.8, 129.3, 123.0 (t, J = 285.3 Hz), 97.1, 61.8, 33.8 (t, J = 18.5 Hz), 15.0; EI 279 (M⁺-29, 0.6), 121 (100.0); HRMS (FTMS) calcd. for C₁₃H₁₈F₂O₄SNa (M+Na⁺): 331.07860, found: 331.07900.

One-pot Preparation of PhSO₂CF₂-substituted alkanes **5**:

In the presence of air, into a 30-mL Schlenk flask containing iododifluoromethyl phenyl sulfone (**1**) (95 mg, 0.3 mmol) and 1-hexene (**2a**) (50 mg, 0.6 mmol) in 2 mL of CH₂Cl₂ at –30 °C, was added Et₃B (1.0 mL, 1.0 mol in hexane). The reaction mixture was stirred at this temperature for 0.5h. After removing of volatile solvents under vacuum, the flask was further added Bu₃SnH (114 mg, 0.39 mmol) and toluene (2.0 mL). Under N₂ atmosphere, the reaction mixture was heated for 0.5h under 90°C, followed by adding a saturated KF-H₂O solution (5 mL) at rt. The solution mixture was extracted with Et₂O (25 mL x 3), and the combined organic phase was dried over MgSO₄. After the removal of volatile solvents under vacuum, the crude product was further purified by silica gel column chromatography with ethyl acetate/petroleum ether (1:50) to give product **5a** as an oil, yield 75% (62 mg).

oil; IR (film): 2958, 1585, 1449, 1345, 1164, 1040, 686, 600 cm⁻¹; ¹H NMR: δ 7.98 (d, J = 7.8 Hz, 2H), 7.76 (t, J = 7.5 Hz, 1H), 7.59-7.64 (m, 2H), 2.24-2.42 (m, 2H), 1.58-1.68 (m, 2H), 1.25-1.44 (m, 6H), 0.89 (t, J = 6.6 Hz, 3H); ¹⁹F NMR: δ –103.59 (t, J = 18.1 Hz, 2F); ¹³C NMR: δ 135.2, 132.6, 130.7, 129.2, 124.7 (t, J = 287.8 Hz), 31.3, 29.1 (t, J = 20.7 Hz), 28.8, 22.4, 20.7 (t, J = 2.9 Hz), 13.9; MS (EI, %): 277 (M⁺+1, 2.4), 143 (73.3), 43 (100.0); HRMS (EI) calcd. For C₁₃H₁₈F₂O₂S (M⁺): 276.0996, Found 276.1008.

oil; IR (film): 2918, 1585, 1473, 1450, 1345, 1262, 1167, 1023, 802 cm⁻¹; ¹H NMR: δ 7.98 (d, J = 6.9 Hz, 2H), 7.72-7.78 (m, 1H), 7.59-7.64 (m, 2H), 2.23-2.41 (m, 2H), 1.58-1.66 (m, 2H), 1.21-1.40 (m, 18H), 0.88 (t, J = 6.9 Hz, 3H); ¹⁹F NMR: δ –103.59 (t, J = 17.8 Hz, 2F); ¹³C NMR: δ 135.1, 132.6, 130.7, 129.2, 124.7 (t, J = 283.2 Hz), 31.9, 29.6, 29.5, 29.35, 29.32, 29.1, 28.9,

22.6, 20.7, 17.3, 14.0; MS (ESI): 361 ($M+H^+$), 378 ($M+NH_4^+$), 383 ($M+Na^+$); HRMS (ESI) calcd. For $C_{19}H_{30}F_2O_2SNa$ ($M+Na^+$): 383.18267, Found 383.18292.

solid; IR (KBr): 2957, 1584, 1452, 1348, 1195, 1158, 863, 721, 584 cm^{-1} ; 1H NMR: δ 7.98 (d, J = 8.1 Hz, 2H), 7.73-7.79 (m, 1H), 7.60-7.65 (m, 2H), 2.22-2.40 (m, 2H), 0.74-0.80 (m, 2H), 0.02 (s, 9H); ^{19}F NMR: δ -105.38 (t, J = 18.1 Hz, 2F); ^{13}C NMR: δ 135.2, 132.7, 130.6, 129.2, 124.8 (t, J = 285.5 Hz), 24.2 (t, J = 20.8 Hz). 1.0, -2.0; MS (ESI): 293 ($M+H^+$), 310 ($M+NH_4^+$), 315 ($M+Na^+$); HRMS (MALDI) calcd. For $C_{12}H_{18}F_2O_2SSiNa$ ($M+Na^+$): 315.06571, Found 315.0676.

oil; 1H NMR: δ 7.97 (d, J = 7.8 Hz, 2H), 7.74-7.79 (m, 1H), 7.59-7.65 (m, 2H), 2.46-2.50 (m, 2H), 2.46-2.50 (m, 2H), 2.26-2.40 (m, 2H), 2.14 (s, 3H), 1.61-1.72 (m, 4H); ^{19}F NMR: δ -103.48 (t, J = 18.3 Hz, 2F); ^{13}C NMR: δ 207.9, 135.2, 132.4, 130.6, 129.2, 124.4 (t, J = 284.1 Hz), 42.9, 29.8, 29.1 (t, J = 20.3 Hz), 23.0, 20.4 (t, J = 3.2 Hz); EI (m/z %): 290 (M^+ , 0.7), 43 (100.0); HRMS (EI) calcd. for $C_{13}H_{16}F_2O_3S$ (M^+): 290.0788, found: 290.0784

oil; IR (film): 2943, 1737, 1448, 1343, 1240, 1163, 1037, 600 cm^{-1} ; 1H NMR: δ 7.97 (d, J = 8.1 Hz, 2H), 7.74-7.79 (m, 1H), 7.59-7.64 (m, 2H), 2.25-2.43 (m, 2H), 2.04 (s, 3H), 1.61-1.70 (m, 4H), 1.38-1.42 (m, 4H); ^{19}F NMR: δ -103.53 (t, J = 18.2 Hz, 2F); ^{13}C NMR: δ 171.1, 135.2, 132.5, 130.6, 129.2, 124.5 (t, J = 284.0 Hz), 64.2, 29.1 (t, J = 20.1 Hz), 28.7, 28.3, 25.5, 20.9, 20.7 (t, J = 3.1 Hz); EI (m/z %): 335 (M^++1 , 1.1), 193 (1.1), 143 (8.6), 43 (100.0); HRMS (EI) calcd. for $C_9H_{15}F_2O_2$ (M^+-PhSO_2): 193.1040, found: 193.1042.

solid; IR (KBr): 2952, 1510, 1448, 1236, 1166, 825, 720 cm^{-1} ; ^1H NMR: δ 7.98 (d, J = 8.1 Hz, 2H), 7.73-7.78 (m, 1H), 7.59-7.64 (m, 2H), 6.82 (s, 4H), 3.98 (t, J = 6.0 Hz, 2H), 3.76 (s, 3H), 2.48-2.66 (m, 2H), 2.06-2.16 (m, 4H); ^{19}F NMR: δ -103.34 (t, J = 18.3 Hz, 2F); ^{13}C NMR: δ 154.0, 152.7, 135.2, 132.4, 130.8, 129.3, 124.5 (t, J = 285.5 Hz), 115.5, 114.7, 66.9, 55.7, 26.5 (t, J = 27.1 Hz), 21.3 (t, J = 4.5 Hz); EI (m/z %) 356 (M^+ , 39.2), 125 (100.0); HRMS (EI) calcd. for $\text{C}_{17}\text{H}_{18}\text{F}_2\text{O}_4\text{S}$ (M^+): 356.0894, found: 356.0897.

Preparation of PhSO₂CF₂-substituted alkanes **6**:

In the presence of air, into a 30-mL Schlenk flask containing iododifluoromethyl phenyl sulfone (**1**) (159 mg, 0.5 mmol), vinyl ether (180 mg, 2.5 mmol) and Bu₃SnH (189 mg, 0.65 mmol) in 2.5 mL of toluene at $-78\text{ }^{\circ}\text{C}$, was added Et₃B (0.65 mL, 1.0 mol in hexane). The reaction mixture was stirred at this temperature for 0.5 h, followed by adding a saturated KF-H₂O solution (10 mL) at rt. The solution mixture was extracted with Et₂O (25 mL x 3), and the combined organic phase was dried over MgSO₄. After the removal of volatile solvents under vacuum, the crude product was further purified by silica gel column chromatography with ethyl acetate/petroleum ether (1:50) to give product **6** as an oil, yield 64% (85 mg).

oil; IR (film): 2876, 1585, 1449, 1344, 1166, 1122, 687, 605 cm^{-1} ; ¹H NMR: δ 7.98 (d, $J = 8.1\text{ Hz}$, 2H), 7.73-7.79 (m, 1H), 7.62 (t, $J = 7.8\text{ Hz}$, 2H), 3.76 (t, $J = 6.6\text{ Hz}$, 2H), 3.47-3.54 (m, 2H), 2.56-2.73 (m, 2H), 1.19 (t, $J = 7.2\text{ Hz}$, 3H); ¹⁹F NMR: δ -102.5 (t, $J = 17.8\text{ Hz}$, 2F); ¹³C NMR: δ 135.4, 132.2, 130.8, 129.3, 123.7 (t, $J = 292.1\text{ Hz}$), 65.5, 62.7 (t, $J = 4.2\text{ Hz}$), 30.0 (t, $J = 19.3\text{ Hz}$), 15.0; EI (m/z %): 264 (M^+ , 0.1), 235 (0.8), 59 (100.0); HRMS (EI) calcd. for C₉H₉F₂O₃S (M^+ -29): 235.0240, found: 235.0248.

Preparation of PhSO₂CF₂-substituted alkenes 7:

Under air atmosphere, into a 30-mL Schlenk flask containing iododifluoromethyl phenyl sulfone (**1**) (318 mg, 1.0 mmol) and 1-hexene (**2a**) (168 mg, 2.0 mmol) in 6 mL CH₂Cl₂ at –30 °C, was added Et₃B (1.0 mL, 1.0 mol in Hexane). The reaction mixture was stirred at this temperature for 0.5h. After removing of volatile solvents under vacuum, the flask was further added DBU (182 mg, 1.2 mmol) and toluene (8 mL) at 0°C. The reaction mixture was stirred under this temperature for 3.0h, followed by removal of volatile solvents under vacuum. The crude product was further purified by silica gel column chromatography with ethyl acetate/petroleum ether (1:50) to give product **7a** as an oil, yield 71% (195 mg).

oil; IR(film) 2960, 1666, 1585, 1450, 1345, 1167, 1081, 973, 686 cm⁻¹; ¹H NMR: δ 7.98 (d, J = 8.1 Hz, 2H), 7.72-7.77 (m, 1H), 7.58-7.72 (m, 2H), 6.42-6.52 (m, 1H), 5.71-5.85 (m, 1H), 2.21-2.25 (m, 2H), 1.25-1.63 (m, 4H), 0.91 (t, J = 7.2 Hz, 3H); ¹⁹F NMR: δ –101.33 (dd, J = 13.0, 1.9 Hz, 2F); ¹³C NMR: δ 146.5 (t, J = 8.4 Hz), 135.1, 133.0, 130.7, 129.1, 120.8 (t, J = 281.1 Hz), 114.9 (t, J = 2.2 Hz), 32.0, 29.9, 22.0, 13.7; EI (m/z %): 275 (M⁺+1, 20.8), 143 (100.0); HRMS (EI) calcd. for C₁₃H₁₆F₂O₂S (M⁺): 274.0839, found: 274.0843.

oil; IR (film): 2926, 1665, 1466, 1346, 1166, 998, 686, 592 cm⁻¹; ¹H NMR: δ 7.98 (d, J = 7.8 Hz, 2H), 7.75 (d, J = 7.5 Hz, 1H), 7.58-7.63 (m, 2H), 6.42-6.52 (m, 1H), 5.72-5.85 (m, 1H), 2.22-2.24 (m, 2H), 1.26-1.48 (m, 16H), 0.88 (t, J = 6.6 Hz, 3H); ¹⁹F NMR: δ –101.99 (d, J = 11.6 Hz, 2F); ¹³C NMR: δ 146.6 (t, J = 8.4 Hz), 135.1, 133.0, 130.7, 129.2, 124.6 (t, J = 282.7 Hz), 114.8 (t, J = 21.3 Hz), 32.4, 31.9, 29.6, 29.5, 29.37, 29.34, 29.0, 27.9, 22.7, 14.1; ESI: 376 (M+NH₄⁺), 381

(M+Na⁺); HRMS (FTMS) calcd. for C₁₉H₂₈F₂O₂SNa (M+Na⁺): 381.16702, found: 381.1668.

solid; IR (KBr): 2959, 1585, 1450, 1348, 1168, 984, 846, 726 cm⁻¹; ¹H NMR: δ 7.92 (d, J = 7.2 Hz, 2H), 7.68-7.91 (m, 1H), 7.54-7.59 (m, 2H), 6.65-6.73 (m, 1H), 6.09-6.23 (m, 1H), 0.09 (s, 9H); ¹⁹F NMR: δ -103.55 (dd, J = 12.7, 2.2 Hz, 2F); ¹³C NMR: δ 147.4 (t, J = 5.2 Hz), 135.2, 133.0, 130.8, 129.2, 128.3 (t, J = 22.5 Hz), 120.0 (t, J = 282.1 Hz), -2.0; ESI: 291 (M+H⁺), 308 (M+NH₄⁺); HRMS (FTMS) calcd. for C₁₂H₁₆F₂O₂SSi (M⁺): 290.0608, found: 290.0611.

oil; IR (film): 2360, 1716, 1665, 1448, 1337, 1165, 971, 617 cm⁻¹; ¹H NMR: δ 7.98 (d, J = 7.5 Hz, 2H), 7.73-7.96 (m, 1H), 7.59-7.65 (m, 2H), 6.42-6.51 (m, 1H), 5.74-5.87 (m, 1H), 2.60-2.66 (m, 2H), 2.47-2.54 (m, 2H), 2.17 (s, 3H); ¹⁹F NMR: δ -101.77 (dd, J = 13.5, 1.7 Hz, 2F); ¹³C NMR: δ 206.4, 144.5 (t, J = 8.5 Hz), 135.2, 132.8, 130.7, 129.2, 120.6 (t, J = 281.1 Hz), 115.9 (t, J = 22.2 Hz), 41.3, 29.9, 29.1; ESI: 306 (M+NH₄⁺), 311 (M+Na⁺); HRMS (MALDI) calcd. for C₁₃H₁₄F₂O₃SNa (M+Na⁺): 311.05239, found: 311.05245.

oil; IR (film): 2947, 2359, 1738, 1665, 1448, 1339, 1240, 1166, 972 cm⁻¹; ¹H NMR: δ 7.98 (d, J = 8.1 Hz, 2H), 7.73-7.78 (m, 1H), 7.59-7.64 (m, 2H), 6.42-6.50 (m, 1H), 5.75-5.88 (m, 1H), 4.07 (t, J = 6.6 Hz, 2H), 2.25-2.32 (m, 2H), 2.06 (s, 3H), 1.52-1.72 (m, 4H); ¹⁹F NMR: δ -101.53 (dd, J = 11.3, 2.5 Hz, 2F); ¹³C NMR: δ 171.0, 145.7 (t, J = 8.3 Hz), 135.1, 132.9, 130.7, 129.2, 120.7 (t, J = 281.4 Hz), 115.4 (t, J = 21.7 Hz), 63.9, 31.8, 27.9, 24.3, 20.8; ESI 333 (M+H⁺), 350 (M+NH₄⁺); HRMS (MALDI) calcd. for C₁₅H₁₈F₂O₄SNa (M+Na⁺): 355.07861, found: 355.0781.

oil; IR (film) 3542, 1671, 1450, 1330, 1161, 1081, 996, 582 cm^{-1} ; ^1H NMR: δ 8.00 (d, J = 7.8 Hz, 2H), 7.77-7.79 (m, 1H), 7.59-7.76 (m, 2H), 6.57-6.65 (m, 1H), 6.07-6.21 (m, 1H), 4.34-4.39 (m, 2H), 2.33-2.39 (m, 1H); ^{19}F NMR: δ -101.40 (dd, J = 11.9, 1.7 Hz, 2F); ESI: 249 ($\text{M}+\text{H}^+$), 266 ($\text{M}+\text{NH}_4^+$), 271 ($\text{M}+\text{Na}^+$); HRMS (FTMS) calcd. for $\text{C}_{10}\text{H}_{10}\text{F}_2\text{O}_3\text{SNa}$ ($\text{M}+\text{Na}^+$): 271.02109, found: 271.02144.

solid; IR (KBr): 1677, 1584, 1512, 1336, 1237, 1164, 1079, 822, 587 cm^{-1} ; ^1H NMR: δ 7.98 (d, J = 8.1 Hz, 2H), 7.73-7.78 (m, 1H), 7.58-7.63 (m, 2H), 6.59-6.66 (m, 1H), 6.20-6.25 (m, 1H), 4.63-4.67 (m, 2H), 3.77 (s, 3H); ^{19}F NMR: δ -102.02 (dd, J = 13.2, 2.2 Hz, 2F); ^{13}C NMR: δ 154.4, 152.0, 140.4 (t, J = 8.2 Hz), 135.3, 132.7, 130.8, 129.3, 120.9 (t, J = 282.6 Hz), 116.0 (t, J = 23.1 Hz), 115.8, 114.7, 67.0, 55.7; EI: 354 (M^+ , 13.6), 123 (100.0); HRMS (EI) calcd. for $\text{C}_{17}\text{H}_{16}\text{F}_2\text{O}_4\text{S}$ (M^+): 354.0737, found: 354.0740.

NMR spectra for all products

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^{19}F NMR (CDCl_3)

7a

^{13}C NMR (CDCl_3)

7c