Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Solvent-controlled Nucleophilic Trifloromethylthiolation of Morita-Baylis-Hillman Carbonates: Dual roles of DABCO in activating the Zard's trifluoromethylthiolation reagent and the MBH carbonates

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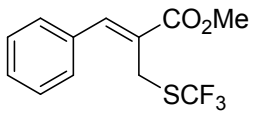
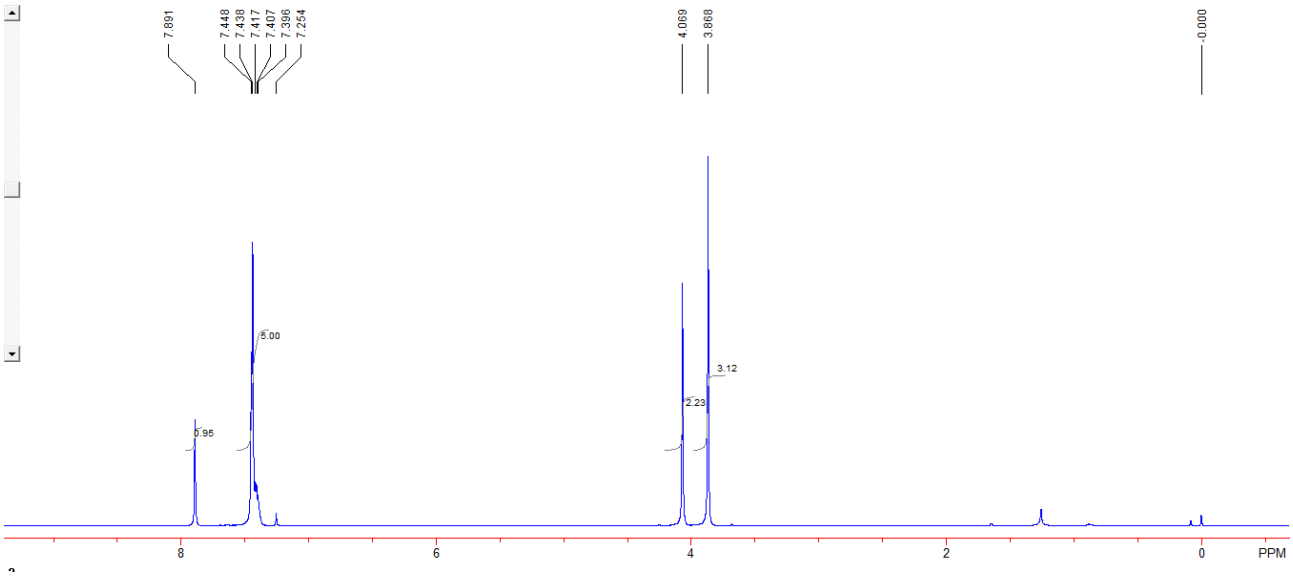
General remarks. ^{1}H and ^{13}C NMR spectra were recorded at 400 (or 300) MHz, respectively. HRMS spectra were recorded by EI, MALDI or ESI method. The employed solvents and commercially obtained reagents were used without further purification. All reactions were monitored by TLC with silica gel coated plates. Flash column chromatography was carried out using 300-400 mesh silica gel at increased pressure.

Procedure for DABCO-catalyzed primary allylic trifluoromethylthiolation reaction of MBH adducts:

To a solution of MBH adducts **I-3-2** (0.2 mmol) and *O*-octadecyl-*S*-trifluorothiolcarbonate **I-3-1a** (0.4 mmol) in THF (2 mL) was added DABCO (0.04 mmol) and the reaction mixture was stirred for 2 h at room temperature under air. The reaction mixture was concentrated and the crude product was purified by flash chromatograph (silica gel, petroleum ether:dichloromethane = 10:1-5:1) to give the corresponding products **3**.



Compound 3a: Yield: 50 mg, 90%. A colourless oil. IR (neat) ν 3004, 2984, 2947, 1716, 1449, 1437, 1275, 1267, 1151, 1114, 1083, 764, 750 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.87 (s, 3H), 4.07 (s, 2H), 7.40-7.45 (m, 5H), 7.89 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 27.0 (q, J = 2.2 Hz), 52.5, 125.8, 128.8, 129.2, 129.5, 130.6 (q, J = 304.6 Hz), 134.1, 143.6, 166.8; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.9; HRMS (EI) Calcd. for $C_{12}H_{11}F_3O_2S$ requires (M^+): 276.0432, Found: 276.0426.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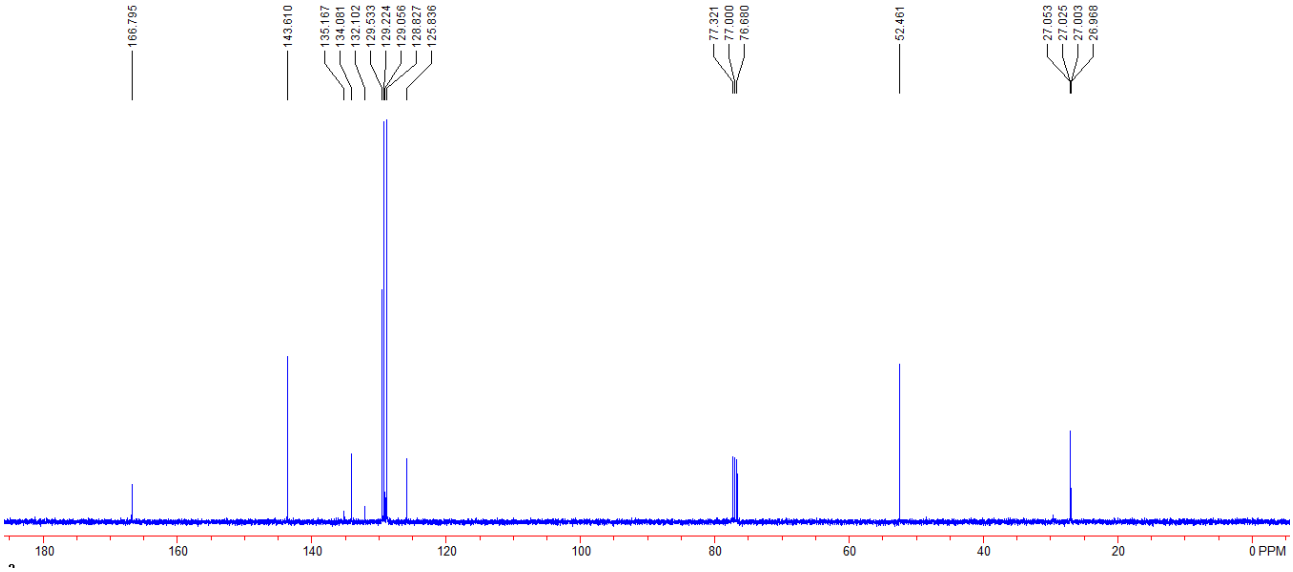
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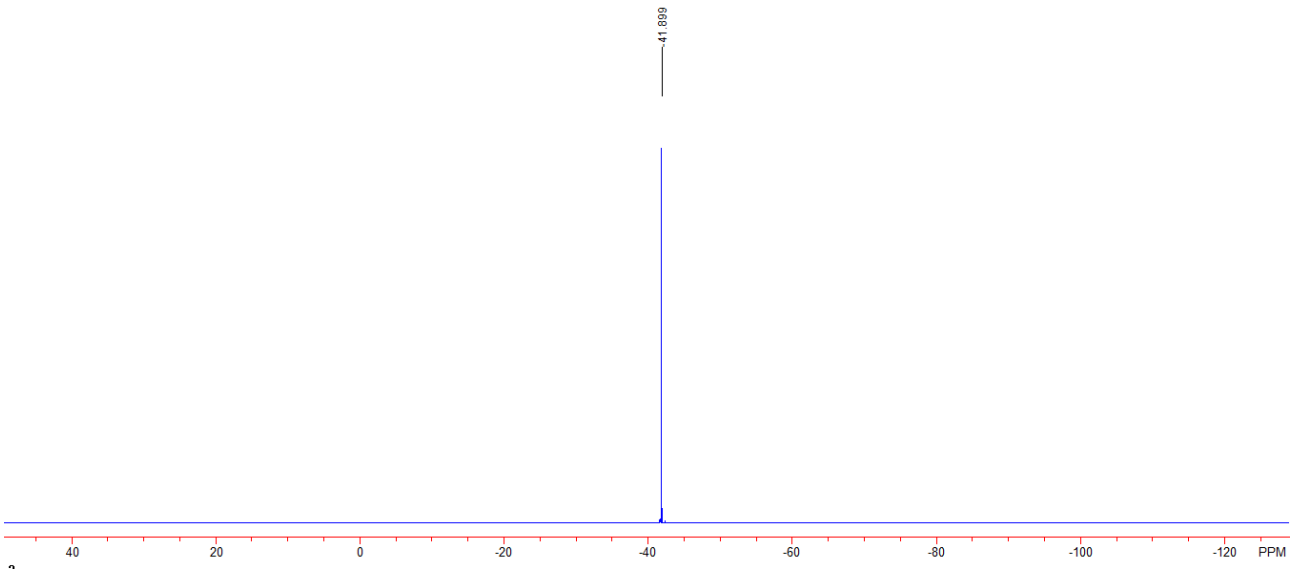
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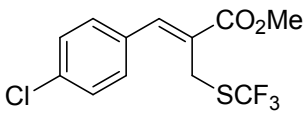
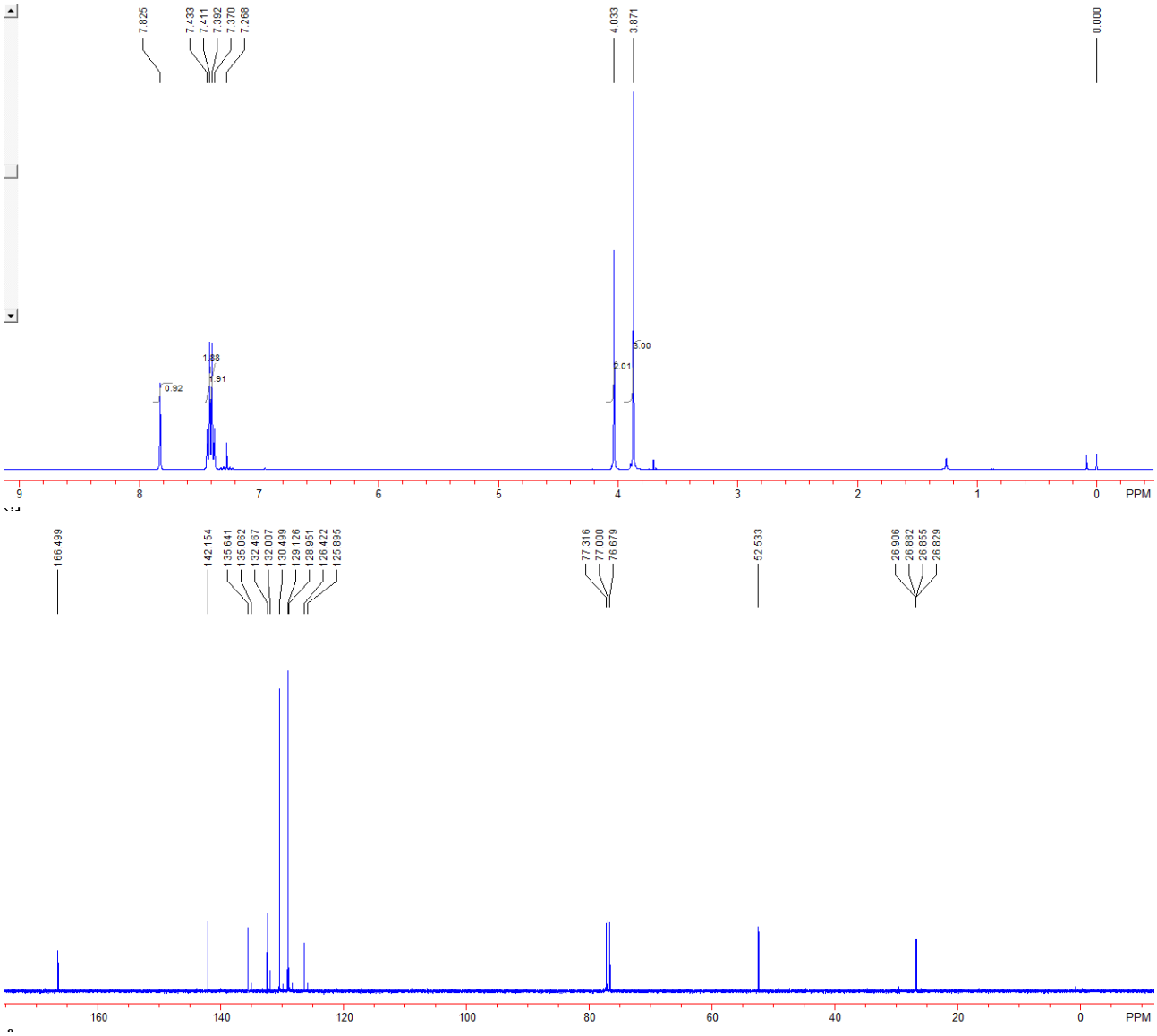
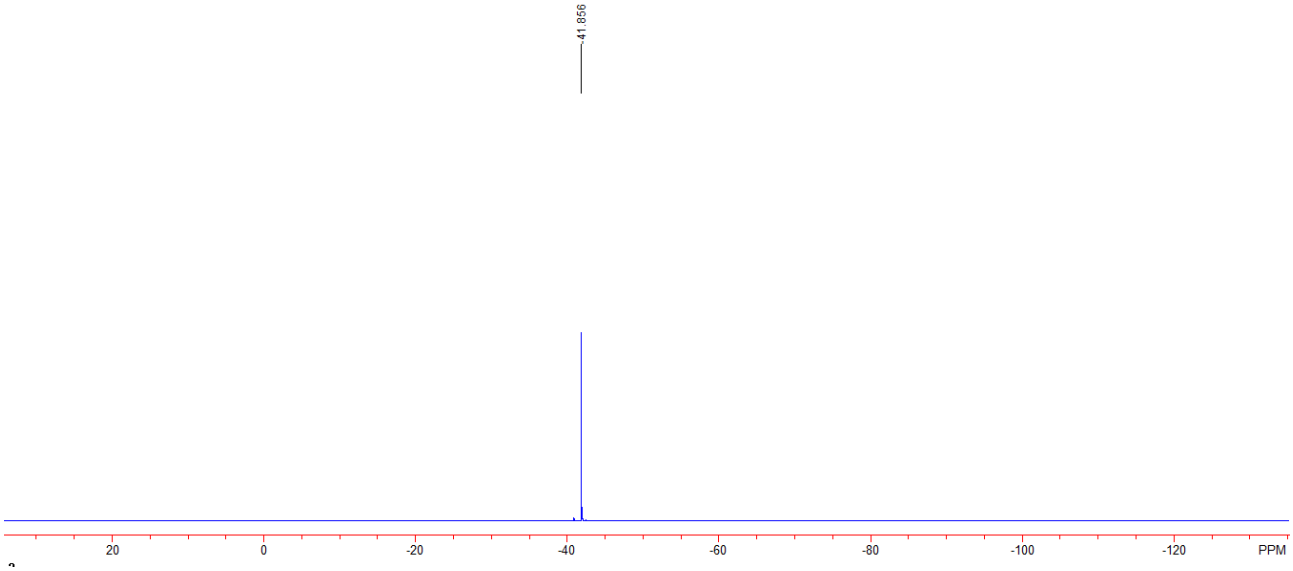
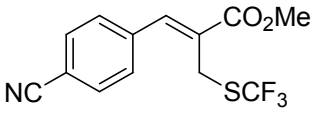


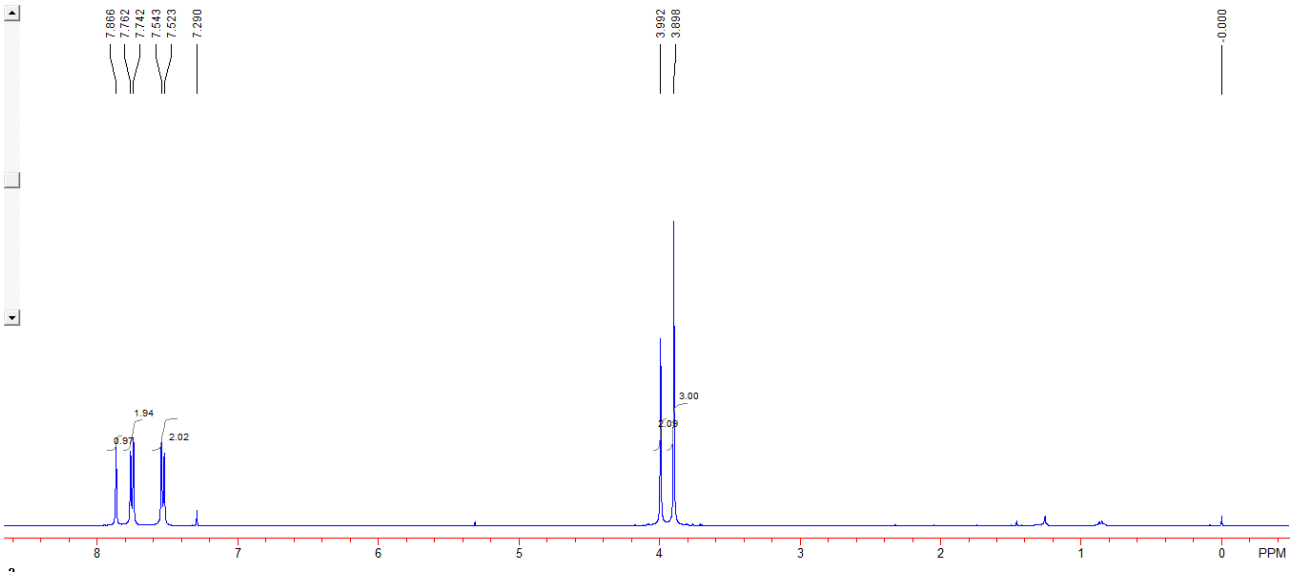
Compound 3b: Yield: 58 mg, 93%. A white solid. m. p.: 65-67 ^{o}C. IR (neat) ν 3004, 2964, 2850, 1717, 1491, 1437, 1276, 1262, 1153, 1113, 1081, 764, 750 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.87 (s, 3H), 4.03 (s, 2H), 7.20-7.35 (m, 4H), 7.38 (d, *J* = 8.8 Hz, 2H), 7.42 (d, *J* = 8.8 Hz, 2H), 7.83 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 26.9 (q, *J* = 2.7 Hz), 52.5, 126.4, 129.1, 130.48 (q, *J* = 305.6 Hz), 130.50, 132.5, 135.6, 142.2, 166.5; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.9; HRMS (EI) Calcd. for $C_{12}H_{10}ClF_3O_2S$ requires (M^+): 310.0042, Found: 310.0049

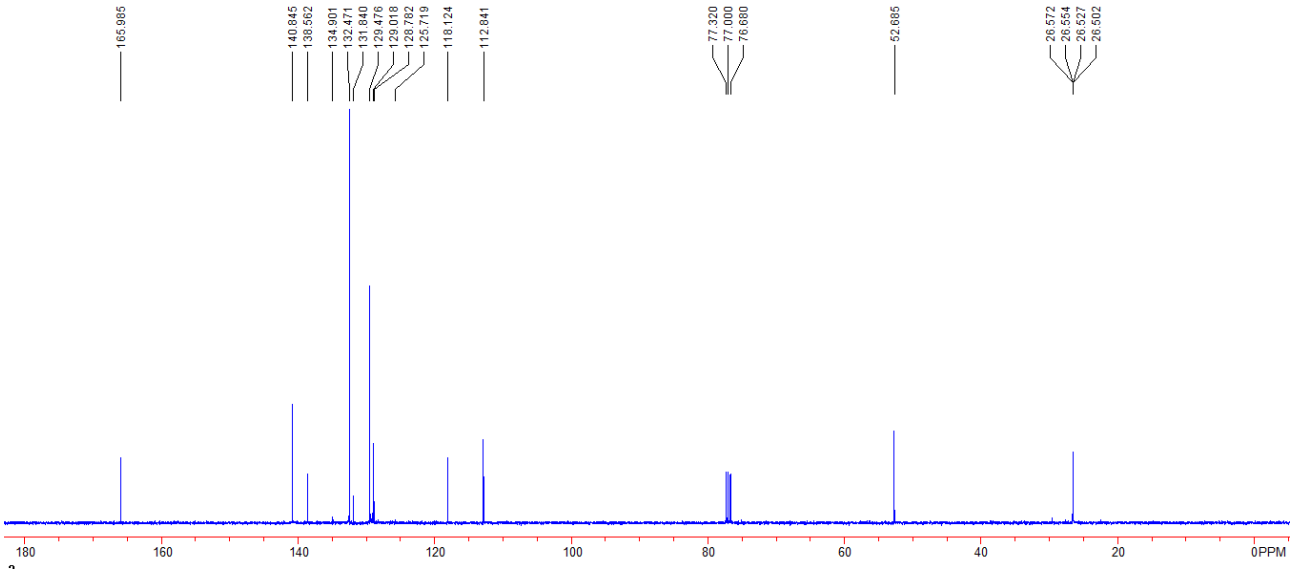


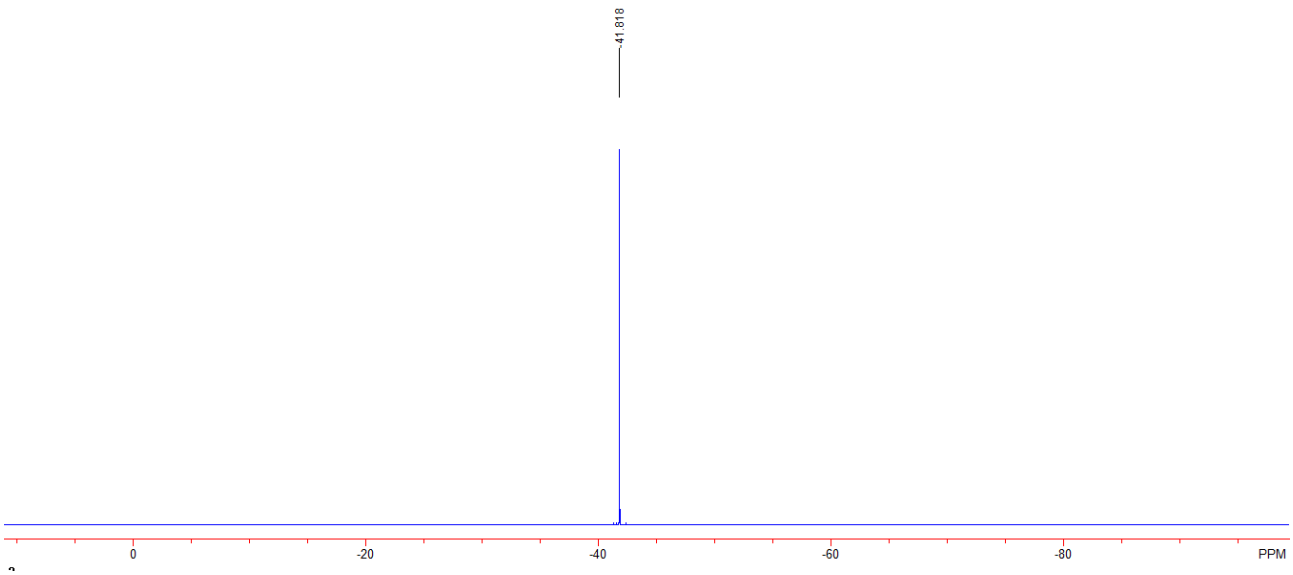


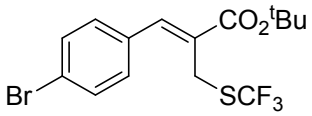


Compound 3c: Yield: 55 mg, 91%. A white solid. m. p.: 61-62 ^{o}C. IR (neat) ν 3008, 2960, 2915, 2850, 2230, 1717, 1438, 1275, 1267, 1152, 1112, 1082, 764, 750 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.90 (s, 3H), 3.99 (s, 2H), 7.53 (d, J = 8.0 Hz, 2H), 7.75 (d, J = 8.0 Hz, 2H), 7.87 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 26.5 (q, J = 2.7 Hz), 52.7, 112.8, 118.1, 129.0, 129.5, 130.3 (q, J = 305.8 Hz), 132.5, 138.6, 140.8, 166.0; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.8; HRMS (EI) Calcd. for $C_{13}H_{10}F_3NO_2S$ requires (M^+): 301.0384, Found: 301.0383.

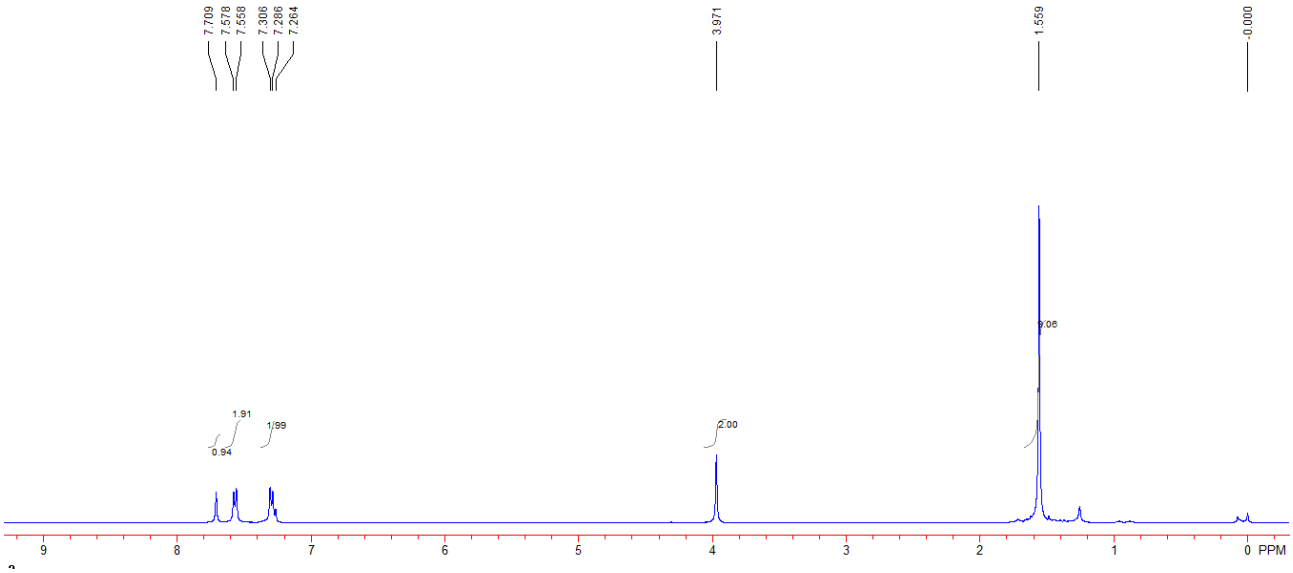








Compound 3d: Yield: 40 mg, 50%. A white solid. m. p.: 48-50 ^{o}C. IR (neat) ν 2979, 2918, 2849, 1711, 1488, ,1369, 1287, 1257, 1153, 1114, 1074, 1011, 849, 811, 756 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 1.56 (s, 9H), 3.97 (s, 2H), 7.30 (d, J = 8.0 Hz, 2H), 7.57 (d, J = 8.0 Hz, 2H), 7.71 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 27.0 (q, J = 2.3 Hz), 28.0, 82.2, 123.6, 128.2, 130.59 (q, J = 305.6 Hz), 130.61, 132.0, 133.3, 141.1, 165.1; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -42.2; HRMS (EI) Calcd. for $C_{15}H_{16}BrF_3O_2S$ requires (M^+): 396.0006, Found: 395.9998.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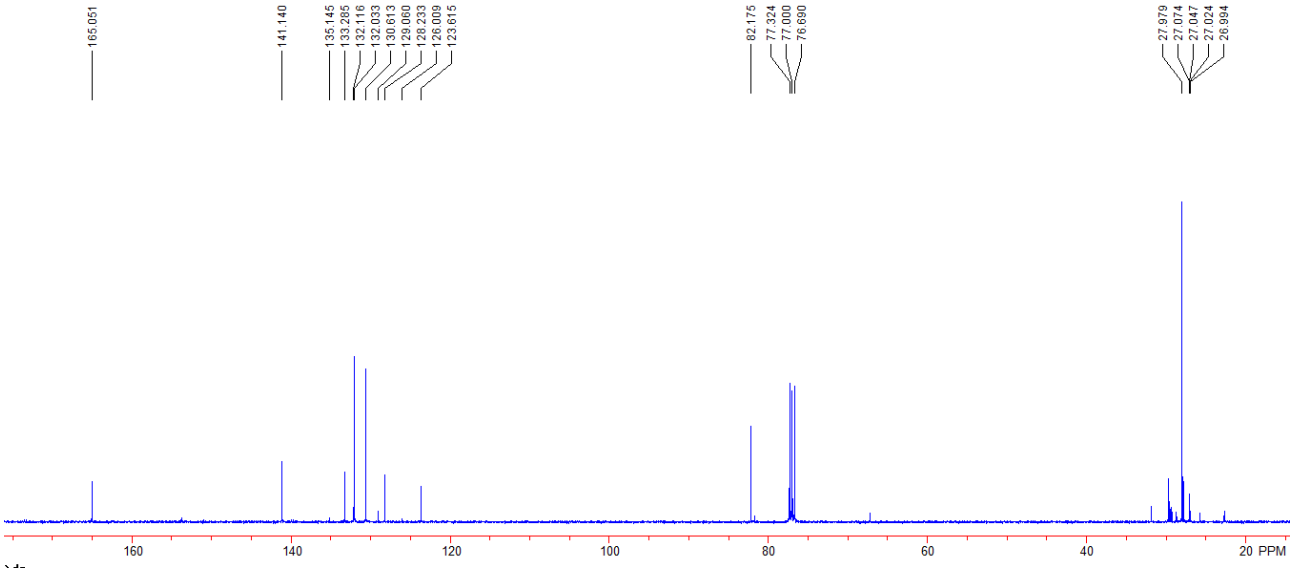
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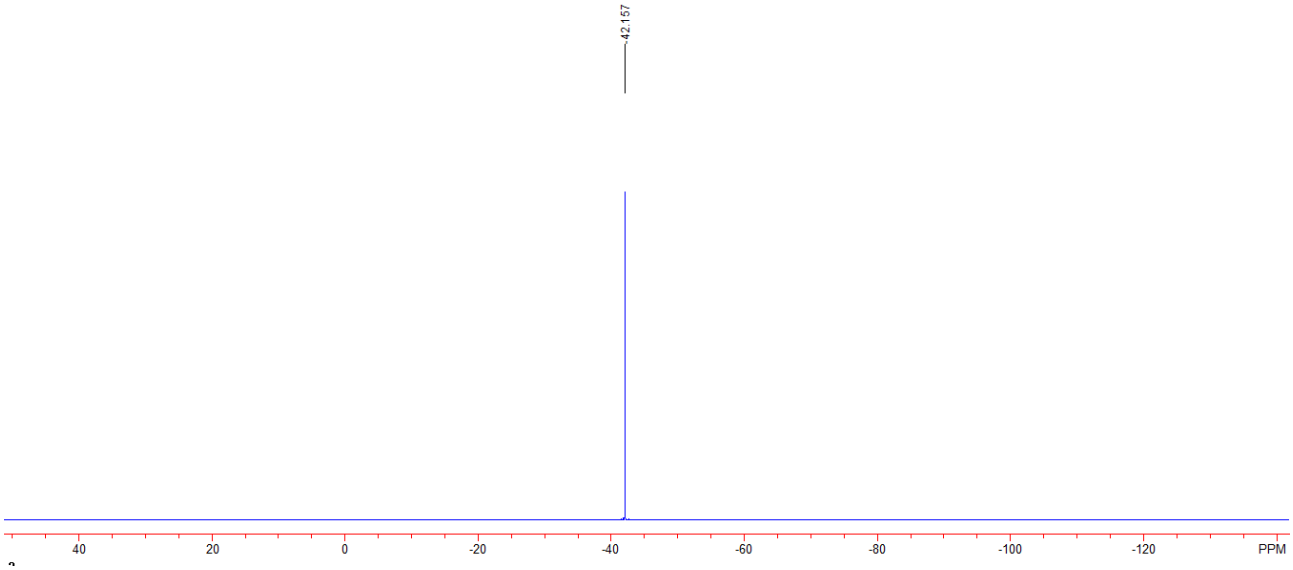
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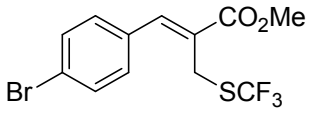
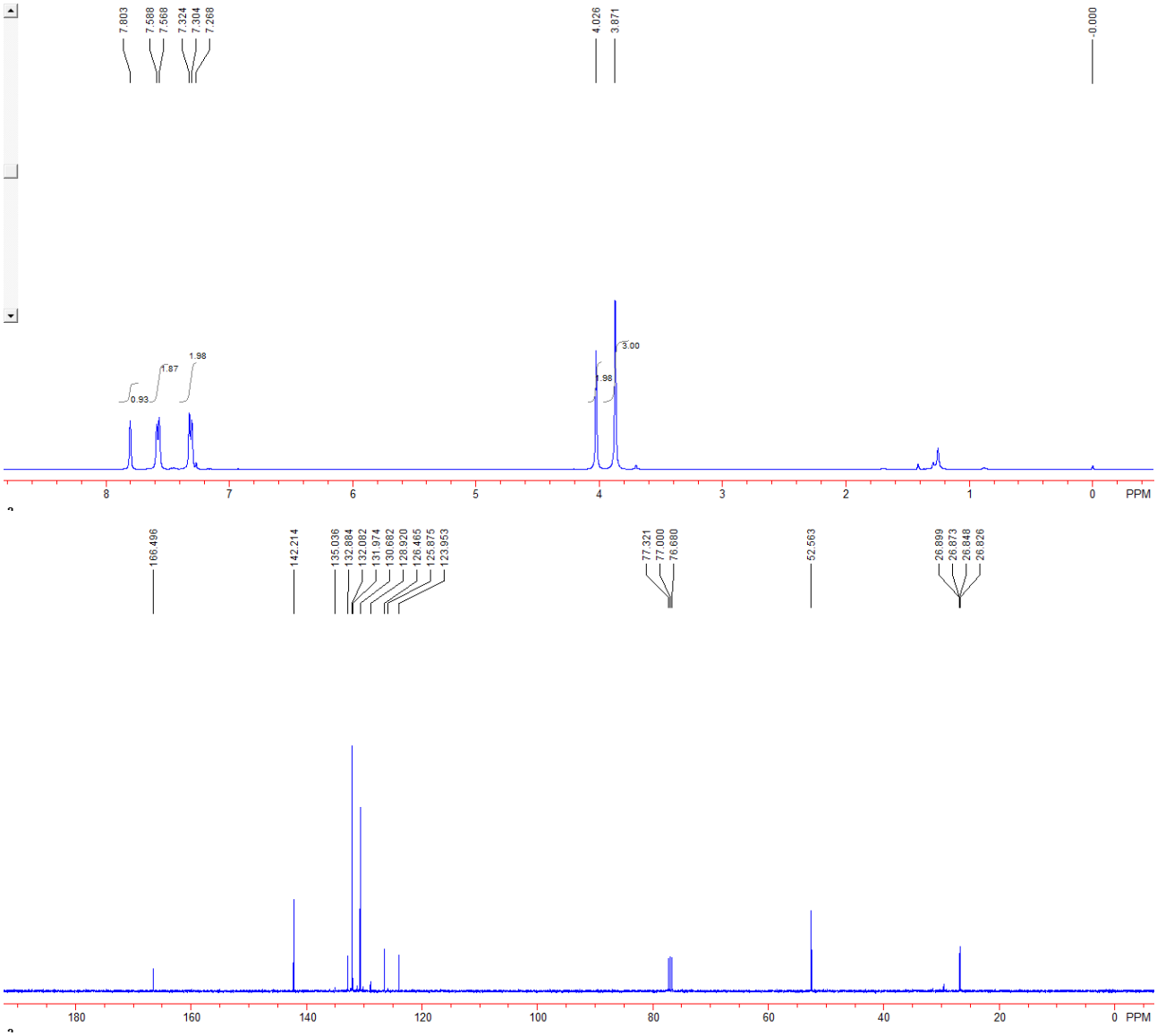
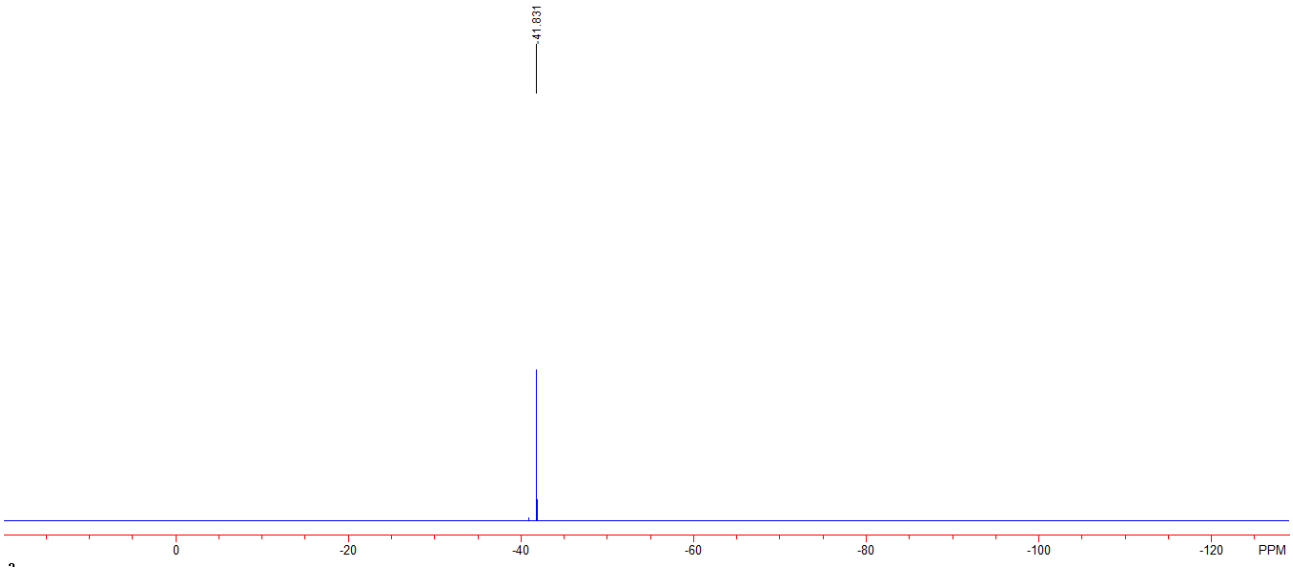
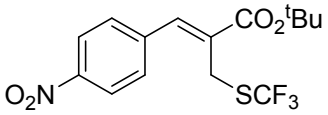


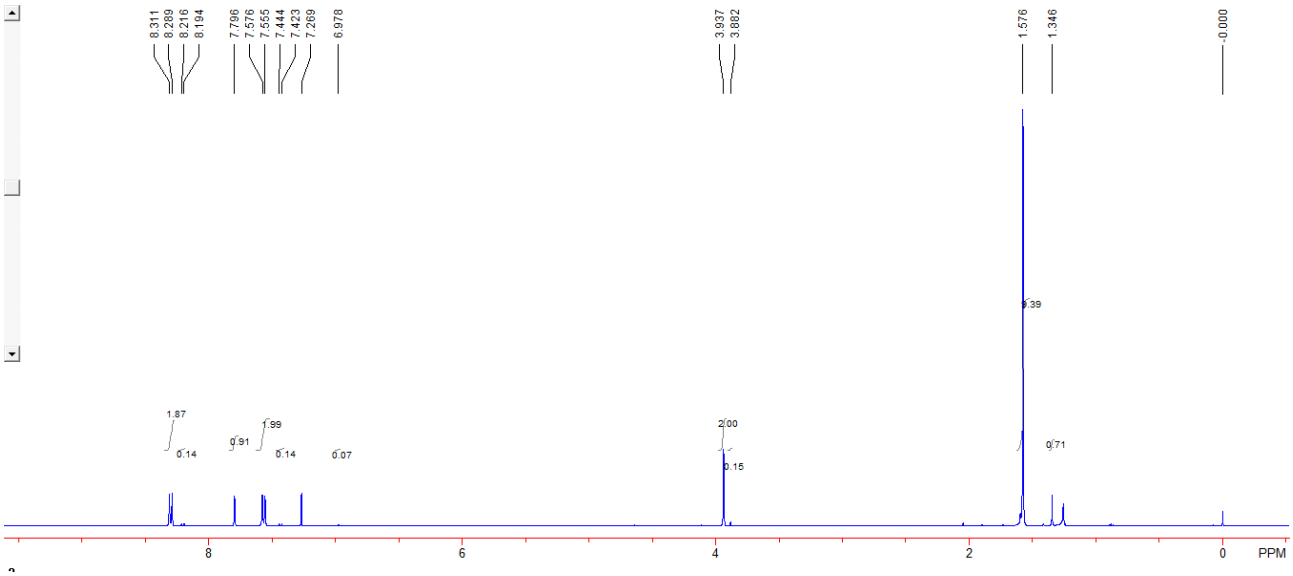
Compound 3e: Yield: 67 mg, 94%. A white solid. m. p.: 40-42 ^{o}C. IR (neat) ν 2954, 2919, 2850, 1719, 1634, 1587, 1488, 1437, 1281, 1154, 1115, 1075, 833, 806 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.87 (s, 3H), 4.03 (s, 2H), 7.31 (d, J = 8.0 Hz, 2H), 7.58 (d, J = 8.0 Hz, 2H), 7.80 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 26.9 (q, J = 2.5 Hz), 52.6, 124.0, 126.5, 130.4 (q, J = 305.4 Hz), 130.7, 132.1, 132.9, 142.2, 166.5; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.8; HRMS (EI) Calcd. for $C_{12}H_{10}BrF_3O_2S$ requires (M^+): 353.9537, Found: 353.9542.







Compound 3f: Yield: 44 mg, 61%. Z:E = 16.3:1. A white solid. m. p.: 78-80 ^{o}C. IR (neat) ν 2984, 2927, 2854, 1712, 1600, 1523, 1370, 1346, 1277, 1260, 1153, 1113, 852, 764, 750 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 1.35 (s, 9H, minor isomer), 1.58 (s, 9H), 3.88 (s, 2H, minor isomer), 3.94 (s, 2H), 6.98 (s, 1H, minor isomer), 7.43 (d, *J* = 8.4 Hz, 2H, minor isomer), 7.57 (d, *J* = 8.4 Hz, 2H), 7.80 (s, 1H), 8.21 (d, *J* = 8.8 Hz, 2H, minor isomer), 8.30 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 26.7 (q, *J* = 2.1 Hz), 27.7 (minor isomer), 27.9, 82.7, 82.9 (minor isomer), 123.2 (minor isomer), 124.0, 129.2 (minor isomer), 129.7, 130.4 (q, *J* = 305.7 Hz), 131.2, 133.4 (minor isomer), 135.5 (minor isomer), 139.4, 140.9, 142.4 (minor isomer), 147.8, 164.4, 164.7 (minor isomer); ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -40.9(minor), -41.8 (major); HRMS (EI) Calcd. for $C_{15}H_{16}F_3NO_4S$ requires (M^+): 363.0752, Found: 363.0748.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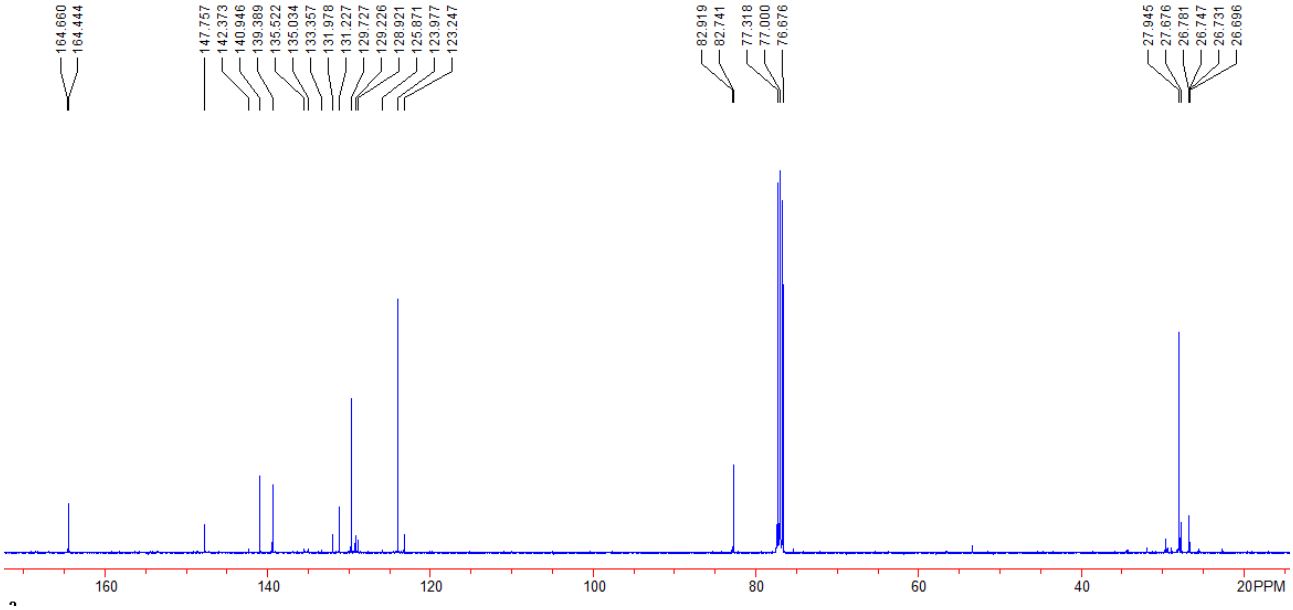
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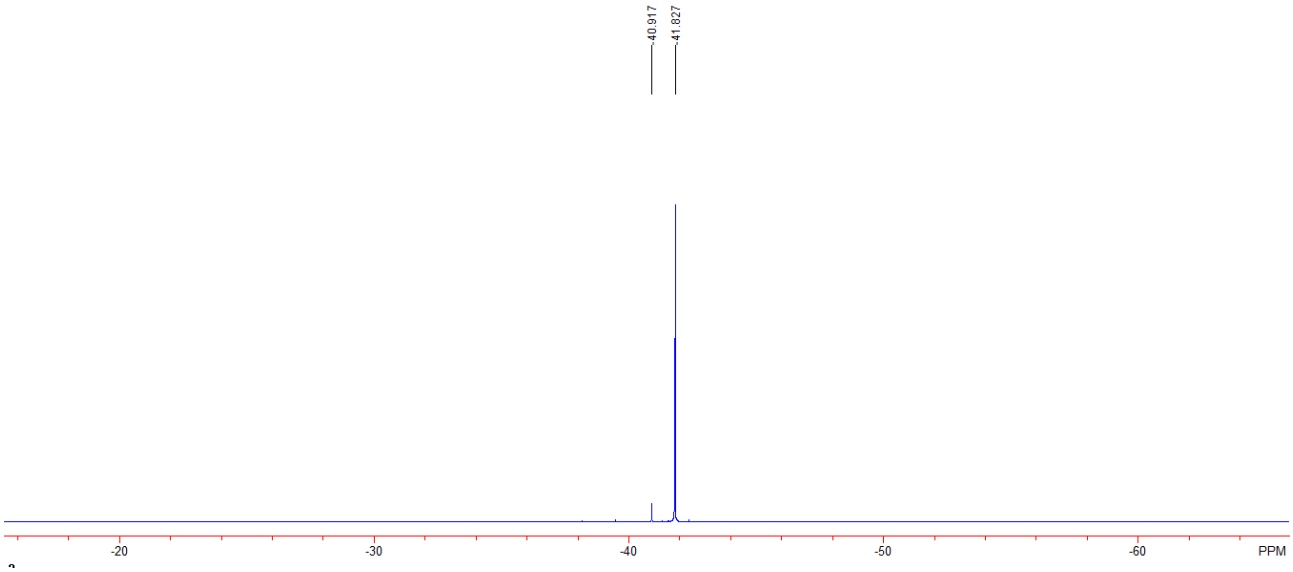
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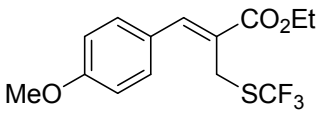
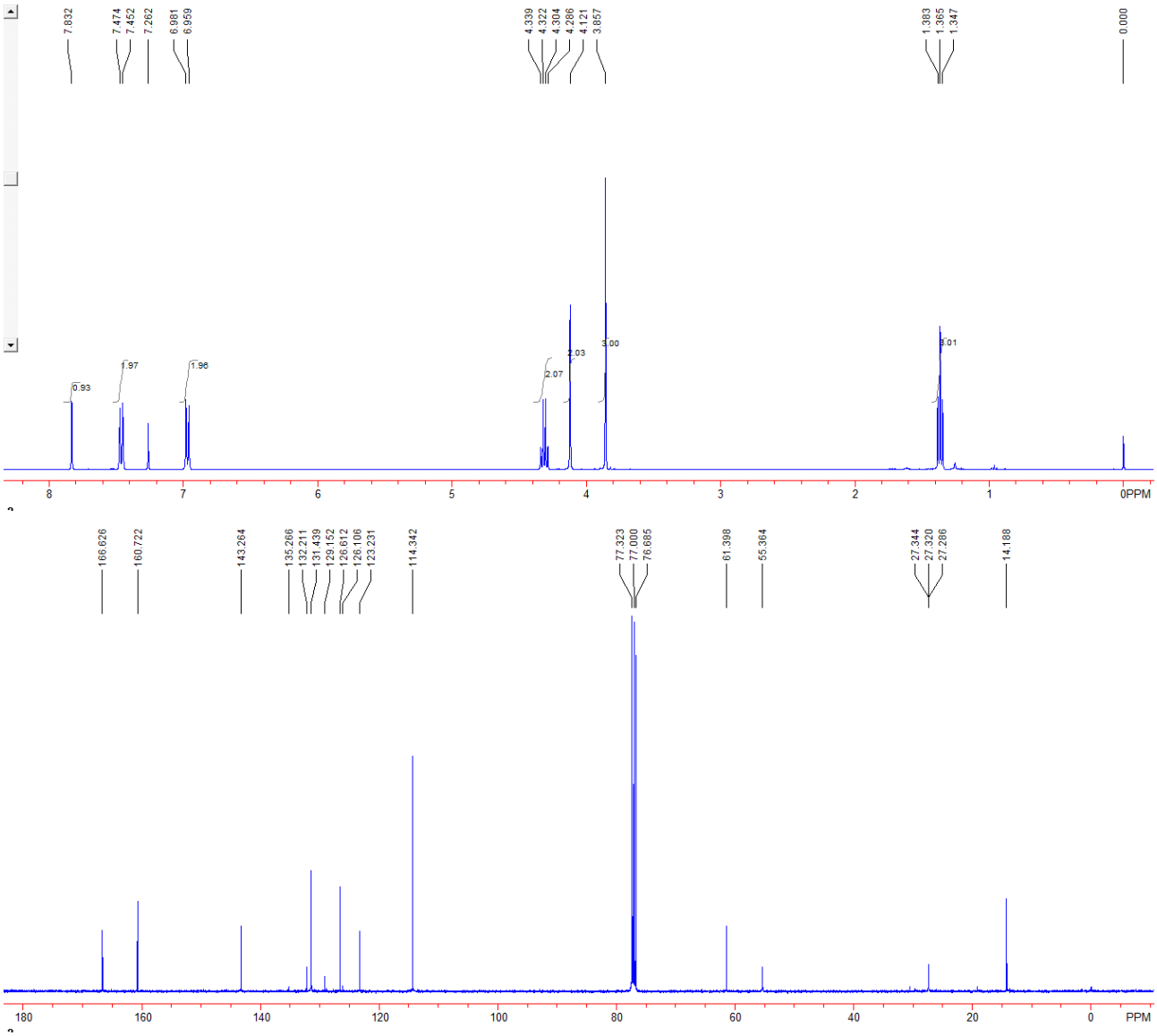
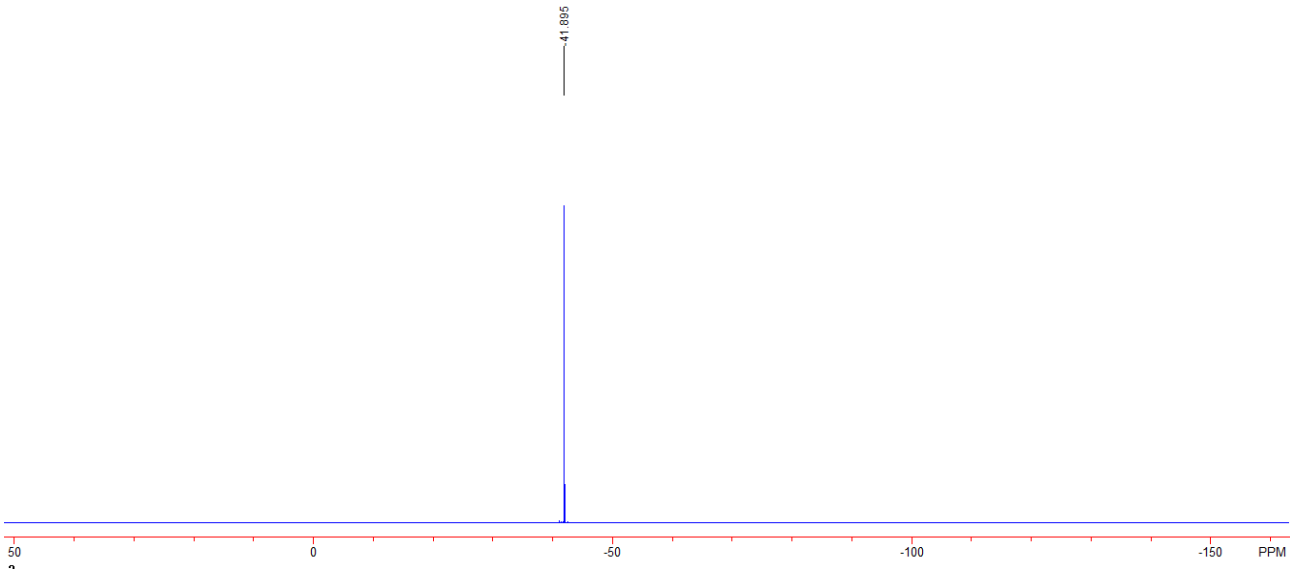
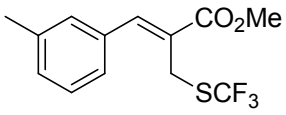


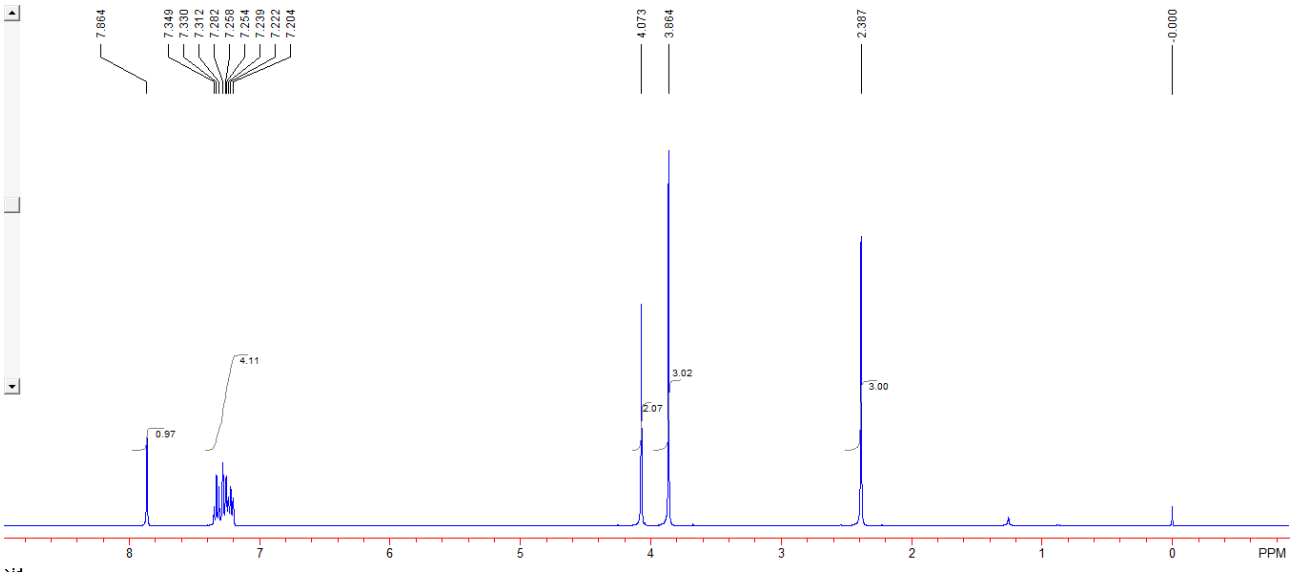
Compound 3g: Yield: 30 mg, 47%. A white solid. m. p.: 40-42 ^{o}C. IR (neat) ν 2964, 2931, 2838, 1706, 1605, 1512, 1306, 1259, 1175, 1152, 1111, 1081, 1030, 828 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 1.37 (t, *J* = 7.2 Hz, 3H), 3.86 (s, 3H), 4.12 (s, 2H), 4.31 (q, *J* = 7.2 Hz, 2H), 6.97 (d, *J* = 8.8 Hz, 2H), 7.46 (d, *J* = 8.8 Hz, 2H), 7.83 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 14.2, 27.3 (q, *J* = 2.4 Hz), 55.4, 61.4, 114.3, 123.2, 126.6, 130.7 (q, *J* = 305.9 Hz), 131.4, 143.3, 160.7, 166.6; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.9; HRMS (EI) Calcd. for $C_{14}H_{15}F_3O_3S$ requires (M^+): 320.0694, Found: 320.0696.

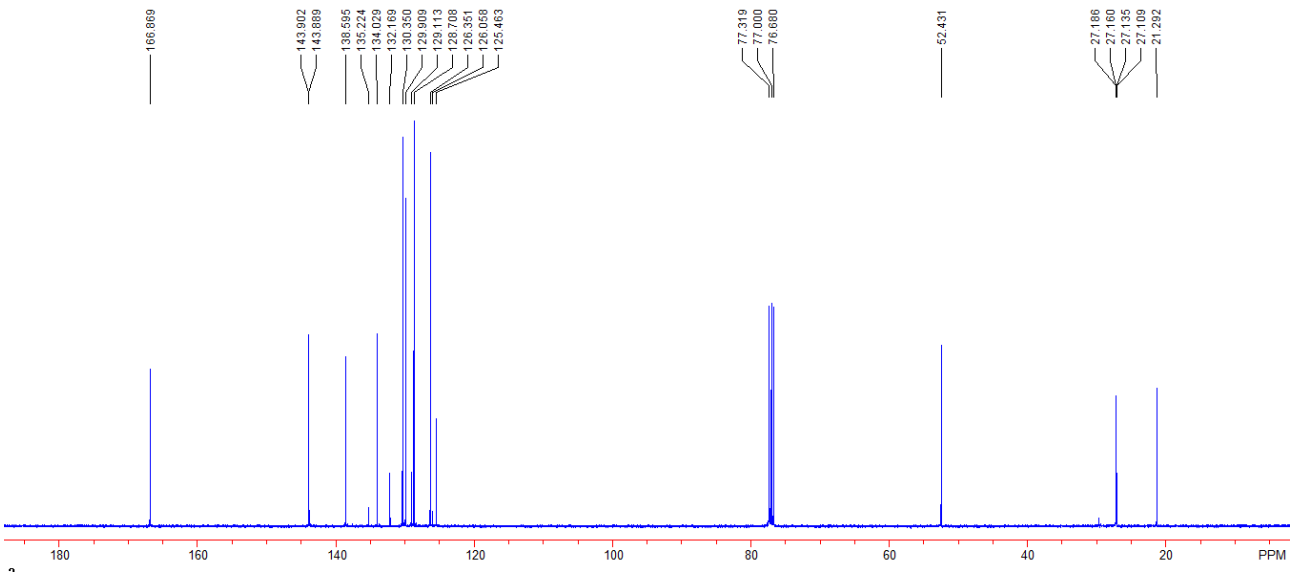


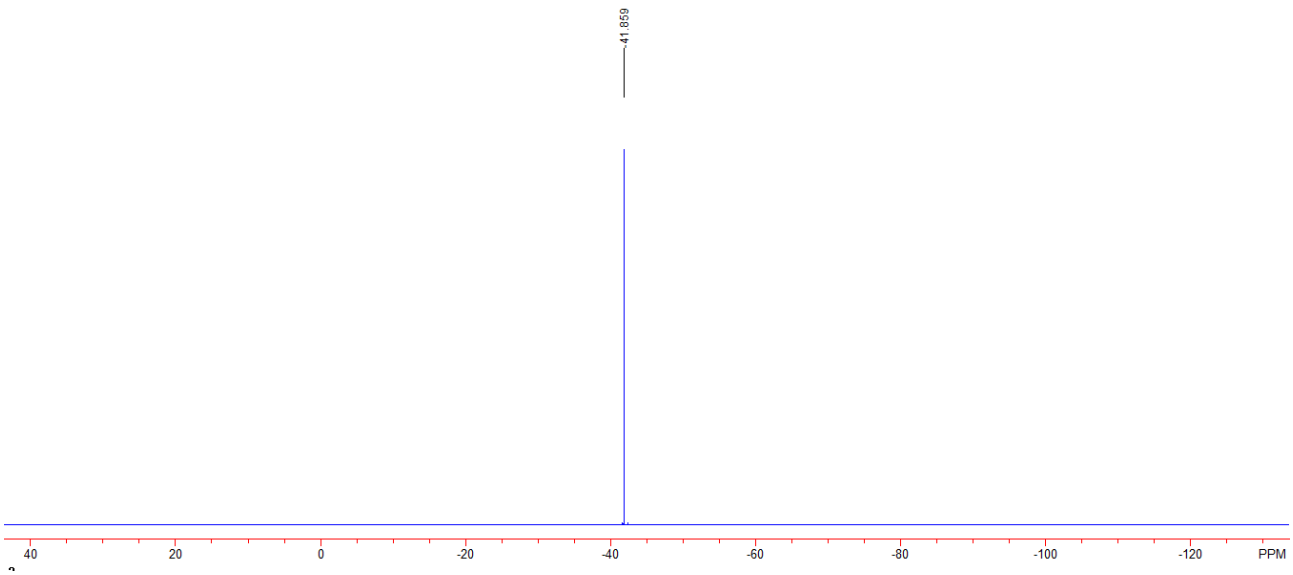


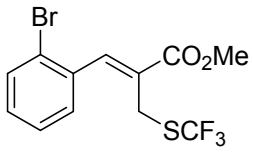


Compound 3h: Yield: 46 mg, 80%. A colourless oil. IR (neat) ν 3008, 2960, 2927, 2846, 1715, 1438, 1276, 1261, 1243, 1151, 1112, 1082, 764, 750 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 2.39 (s, 3H), 3.86 (s, 3H), 4.07 (s, 2H), 7.20-7.35 (m, 4H), 7.86 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 21.3, 27.1 (q, J = 2.5 Hz), 52.4, 125.5, 126.4, 128.7, 129.9, 130.4, 130.6 (q, J = 305.6 Hz), 134.0, 138.6, 143.89, 143.90, 166.9; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.9; HRMS (EI) Calcd. for $C_{13}H_{13}F_3O_2S$ requires (M^+): 290.0588, Found: 290.0584.



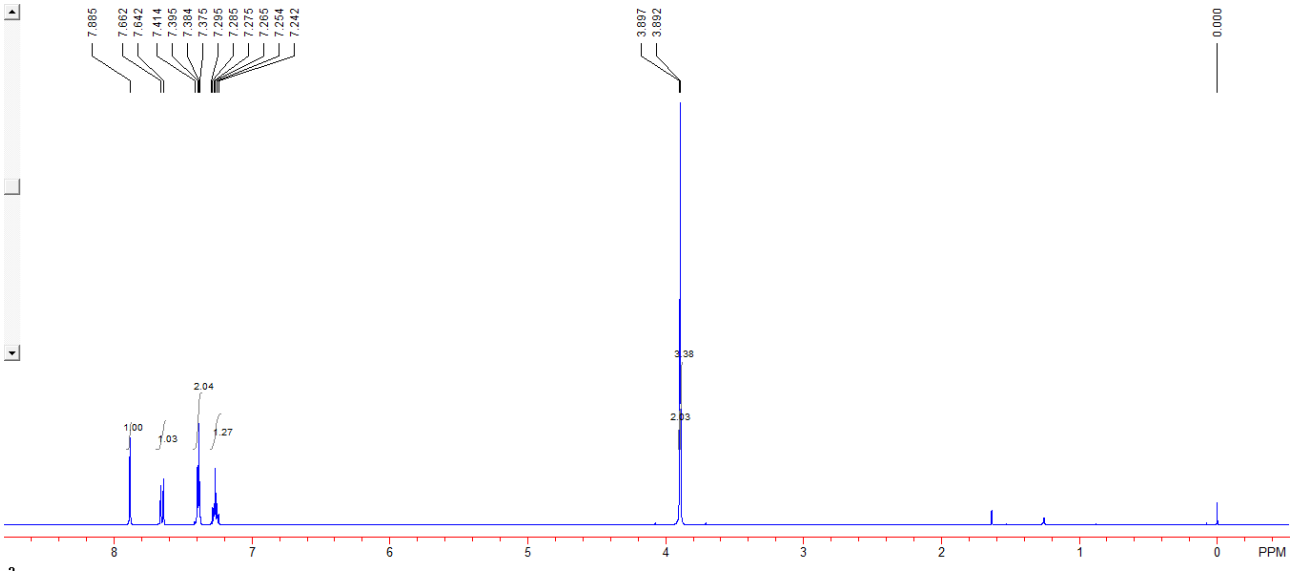


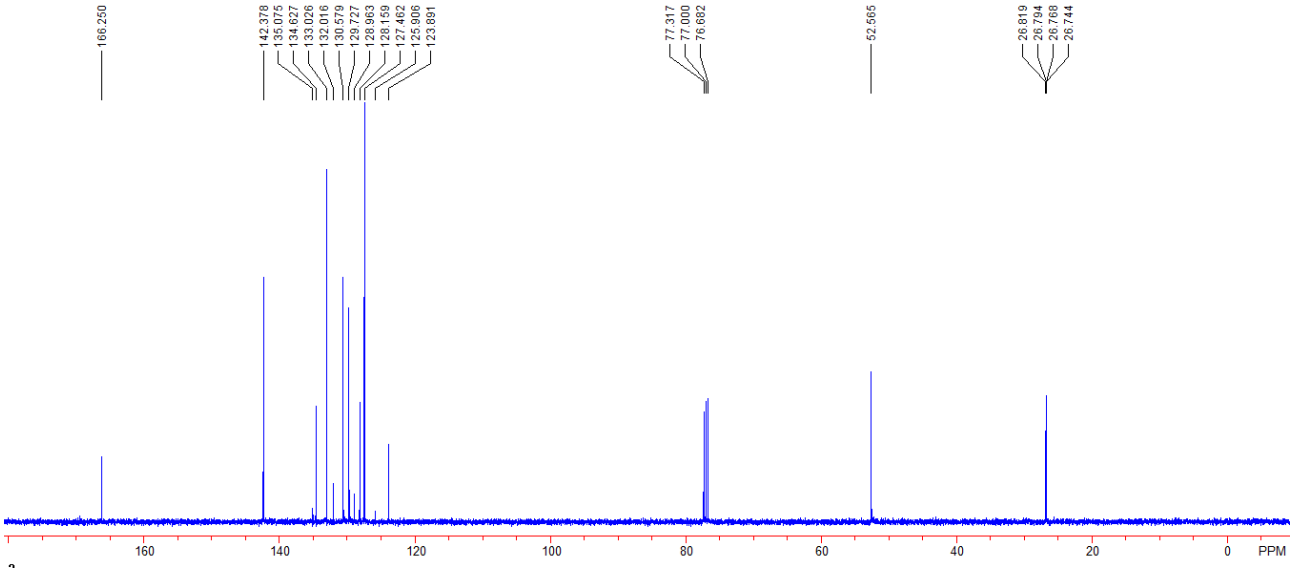


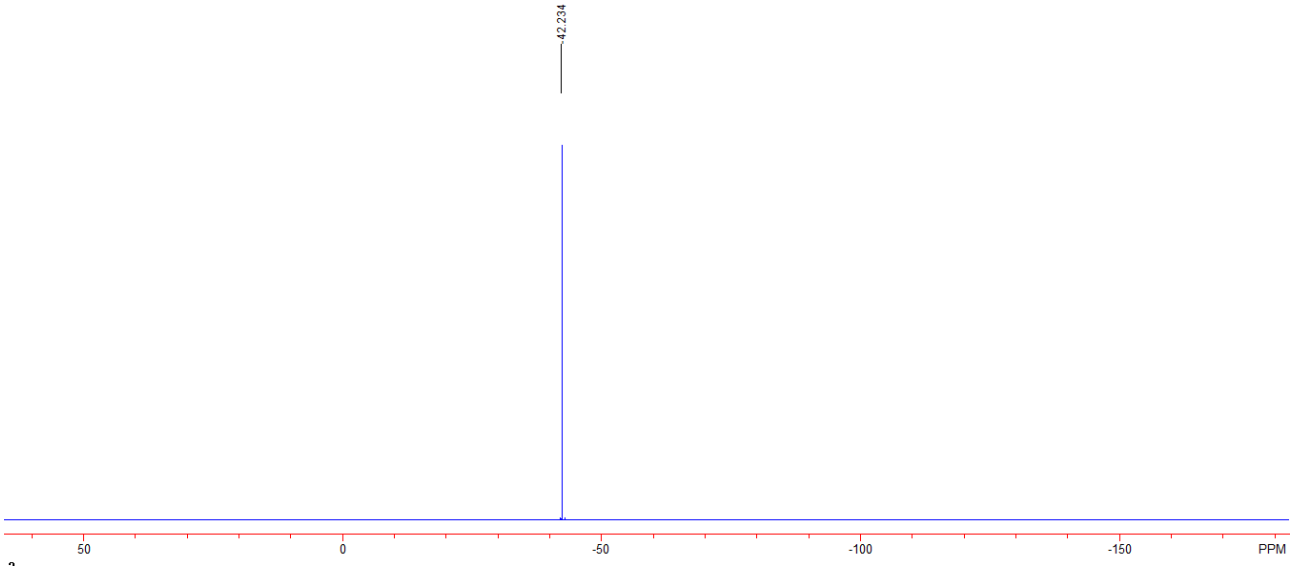


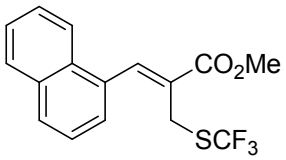
Compound 3i: Yield: 64 mg, 90%. A white solid. m. p.: 30-32 ^{o}C. IR (neat) ν 2955, 2919, 2846, 1717, 1466, 1436, 1288, 1264, 1206, 1151, 1111, 1083, 1046, 1027, 783, 755 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.892 (s, 3H), 3.897 (s, 2H), 7.24-7.30 (m, 1H), 7.38-7.41 (m, 2H), 7.65 (d, *J* = 8.0 Hz, 1H), 7.89 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 26.8 (q, *J* = 2.6 Hz), 52.6, 123.9, 127.5, 128.2, 129.7, 130.5 (q, *J* = 305.3 Hz), 130.6, 133.0, 134.6, 142.4, 166.3; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -42.2; HRMS (EI) Calcd. for $C_{12}H_{10}BrF_3O_2S$ requires (M^+-OMe): 322.9353,

Found: 322.9348.

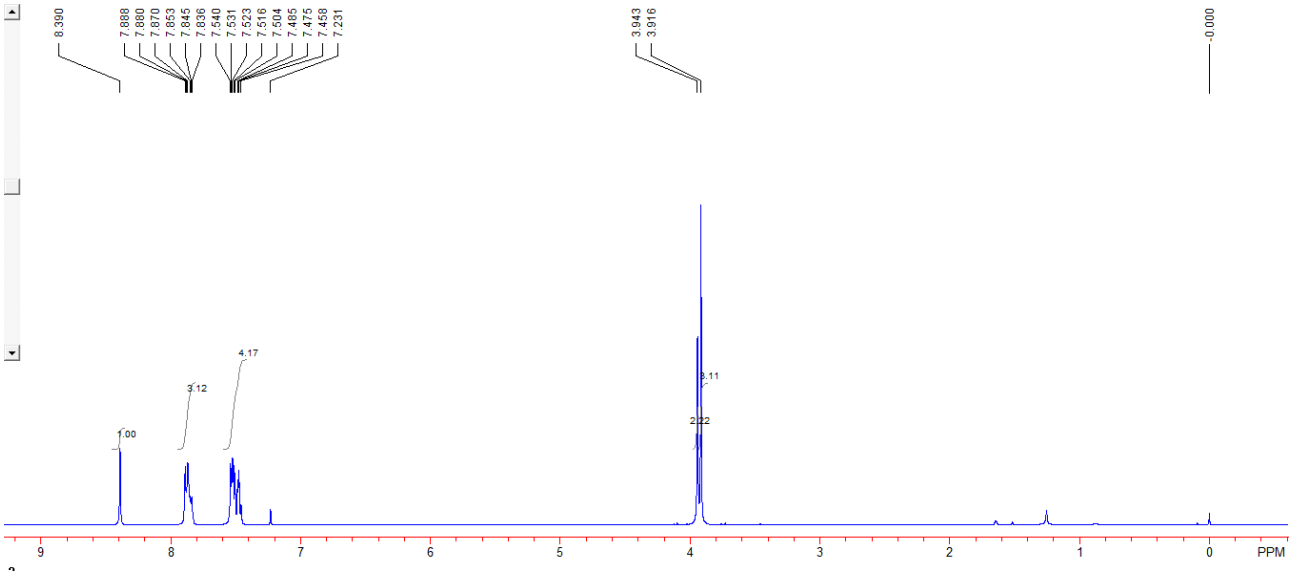


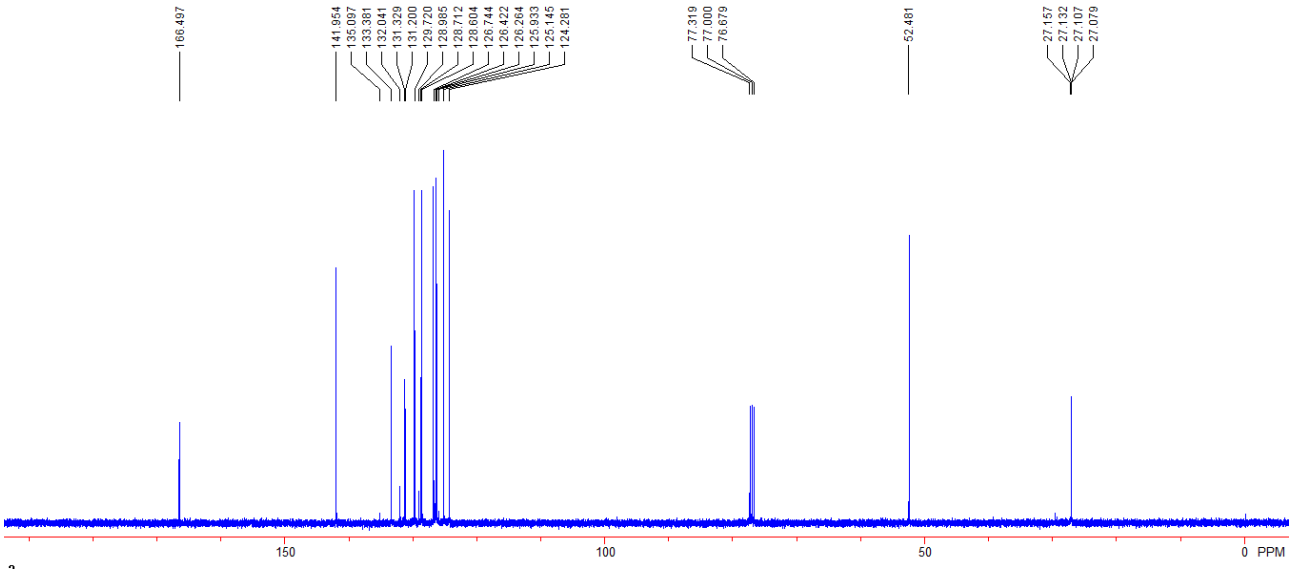


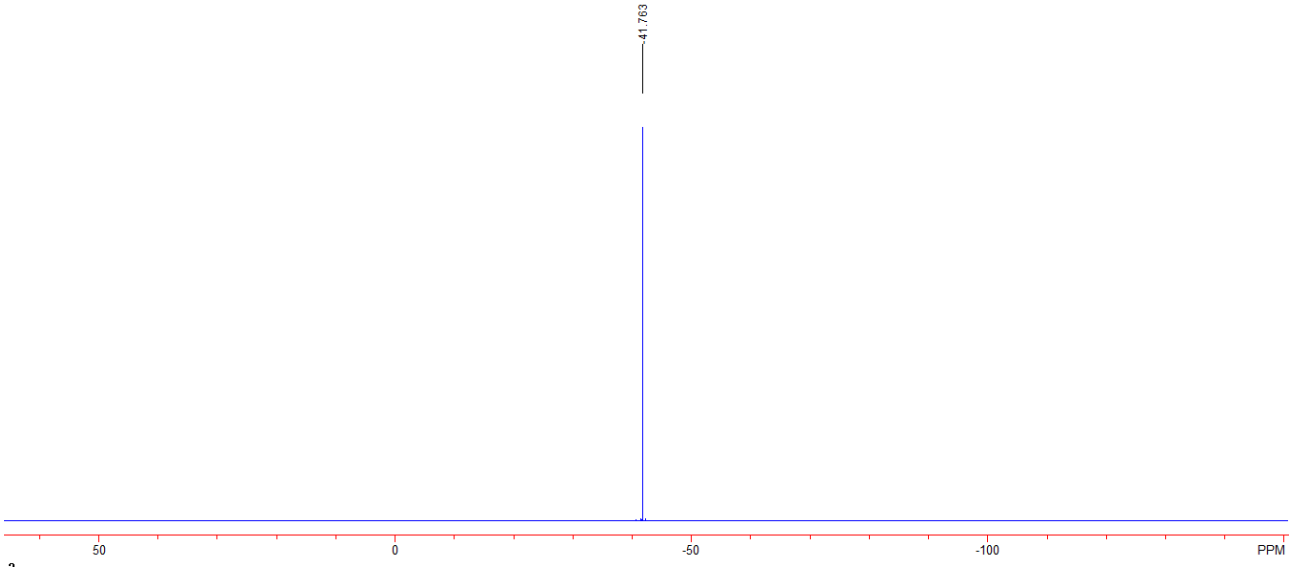


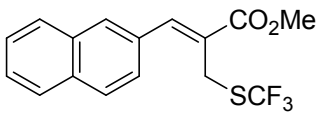


Compound 3j: Yield: 59 mg, 91%. A white solid. m. p.: 36-38 ^{o}C. IR (neat) ν 3065, 3004, 2955, 2850, 1716, 1508, 1437, 1342, 1279, 1261, 1197, 1149, 1111, 1074, 803, 782, 755 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.92 (s, 3H), 3.94 (s, 2H), 7.46-7.54 (m, 4H), 7.84-7.89 (m, 3H), 8.39 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 27.1 (q, J = 2.5 Hz), 52.5, 124.3, 125.1, 126.3, 126.4, 126.7, 128.6, 128.7, 129.7, 130.5 (q, J = 305.6 Hz), 131.2, 131.3, 133.4, 142.0, 166.5; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.8; HRMS (EI) Calcd. for $C_{16}H_{13}F_3O_2S$ requires (M^+): 326.0588, Found: 326.0587.

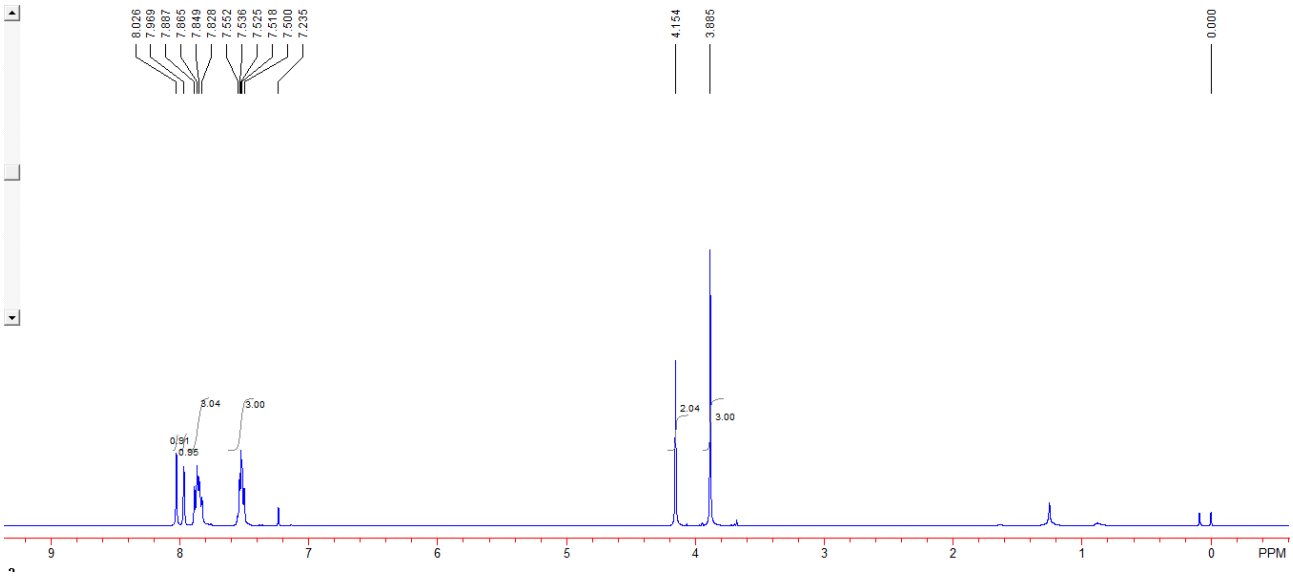


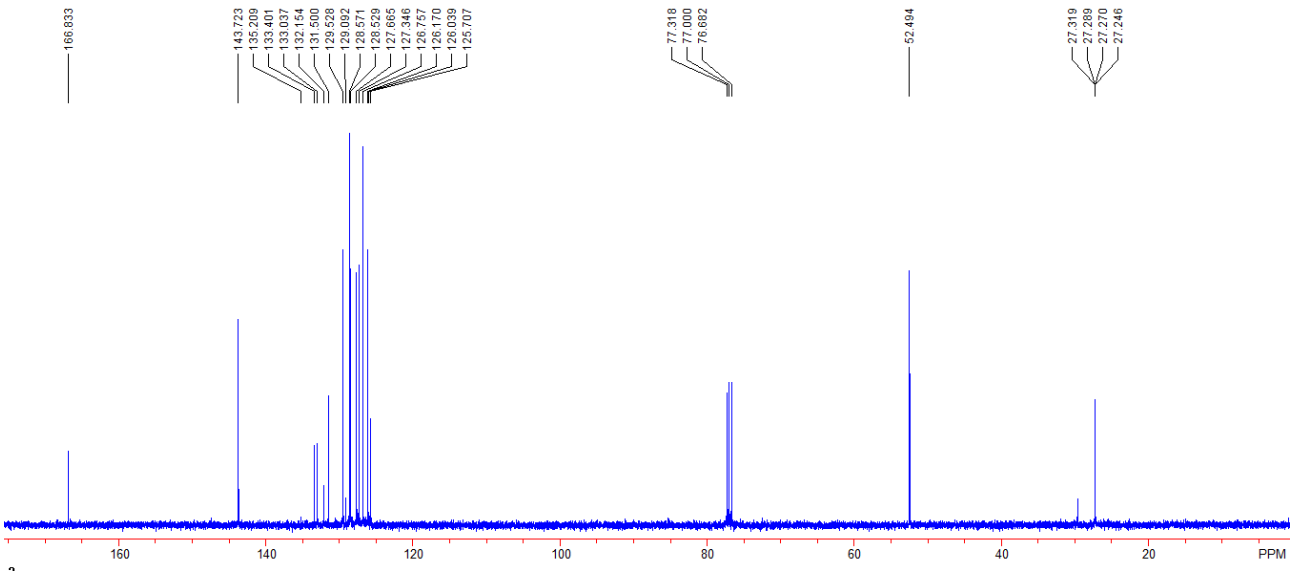


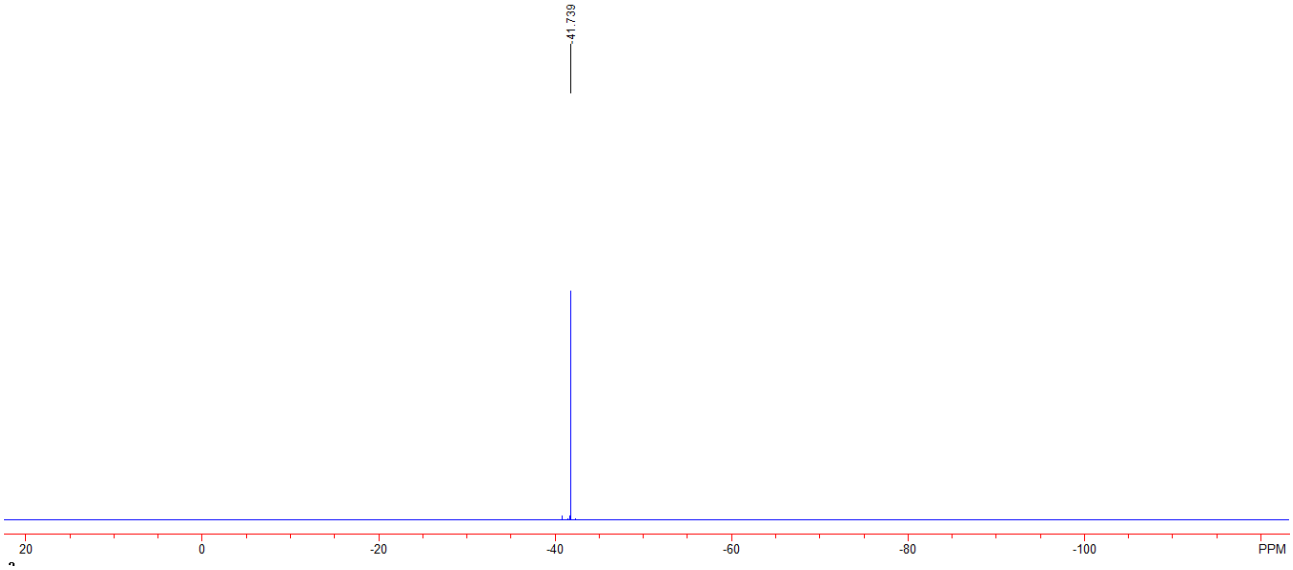


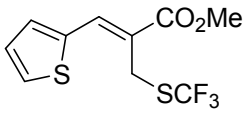


Compound 3k: Yield: 58 mg, 89%. A white solid. m. p.: 44-45 ^{o}C. IR (neat) ν 3061, 3004, 2951, 2923, 2846, 1712, 1623, 1437, 1276, 1261, 1152, 1112, 1083, 764, 750 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.89 (s, 3H), 4.15 (s, 2H), 7.50-7.55 (m, 3H), 7.83-7.89 (m, 3H), 7.97 (s, 1H), 8.03 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 27.3 (q, J = 2.0 Hz), 52.5, 125.7, 126.2, 126.8, 127.3, 127.7, 128.5, 128.6, 129.5, 130.6 (q, J = 306.2 Hz), 131.5, 133.0, 133.4, 143.7, 166.8; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.7; HRMS (EI) Calcd. for $C_{16}H_{13}F_3O_2S$ requires (M^+): 326.0588, Found: 326.0590.

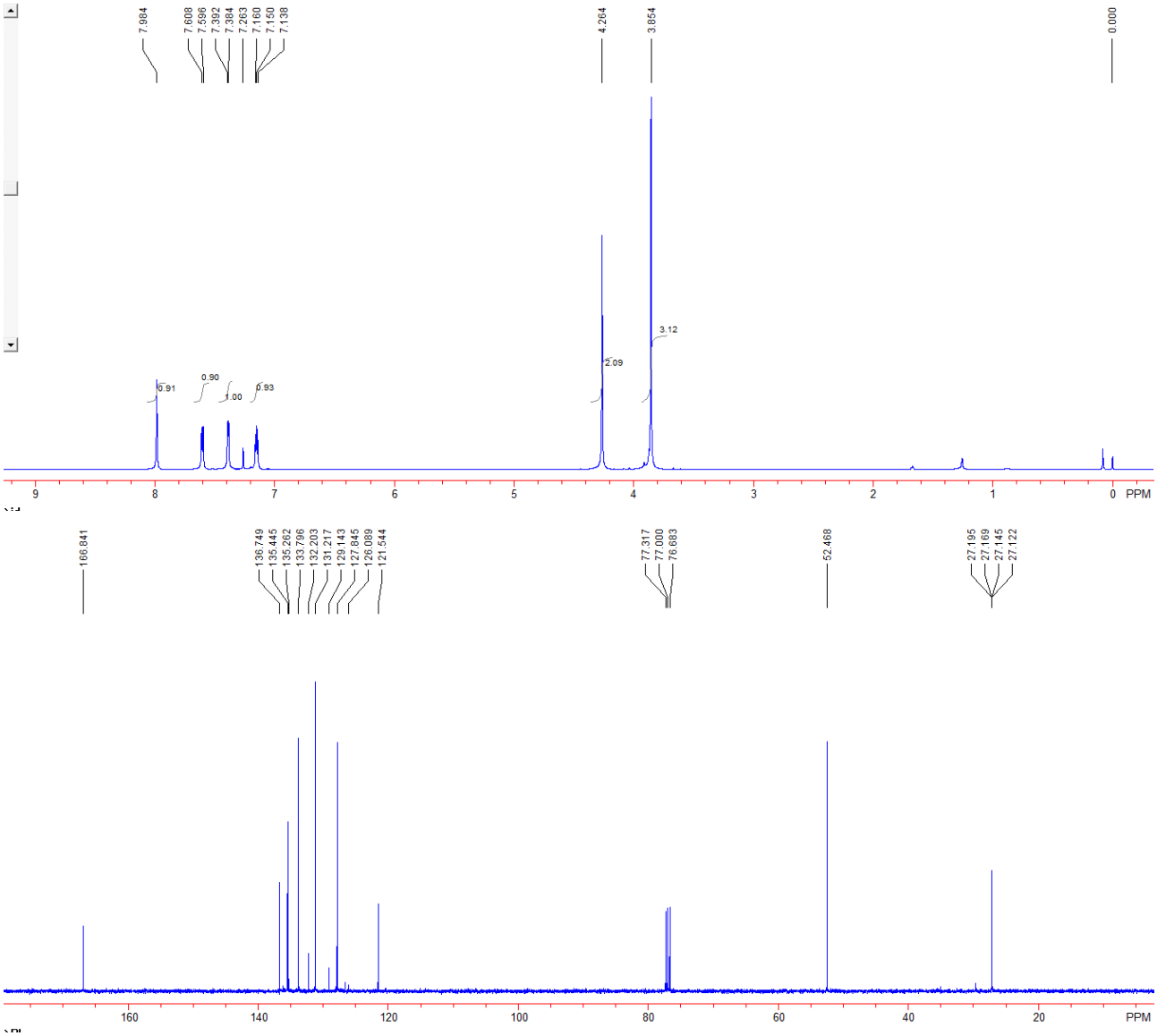

8.026
7.969
7.887
7.865
7.849
7.828
7.552
7.536
7.525
7.518
7.500
7.235
4.154
3.885
0.000
0.91
0.95
4.04
3.00
2.04
3.00
9
8
7
6
5
4
3
2
1
0
PPM

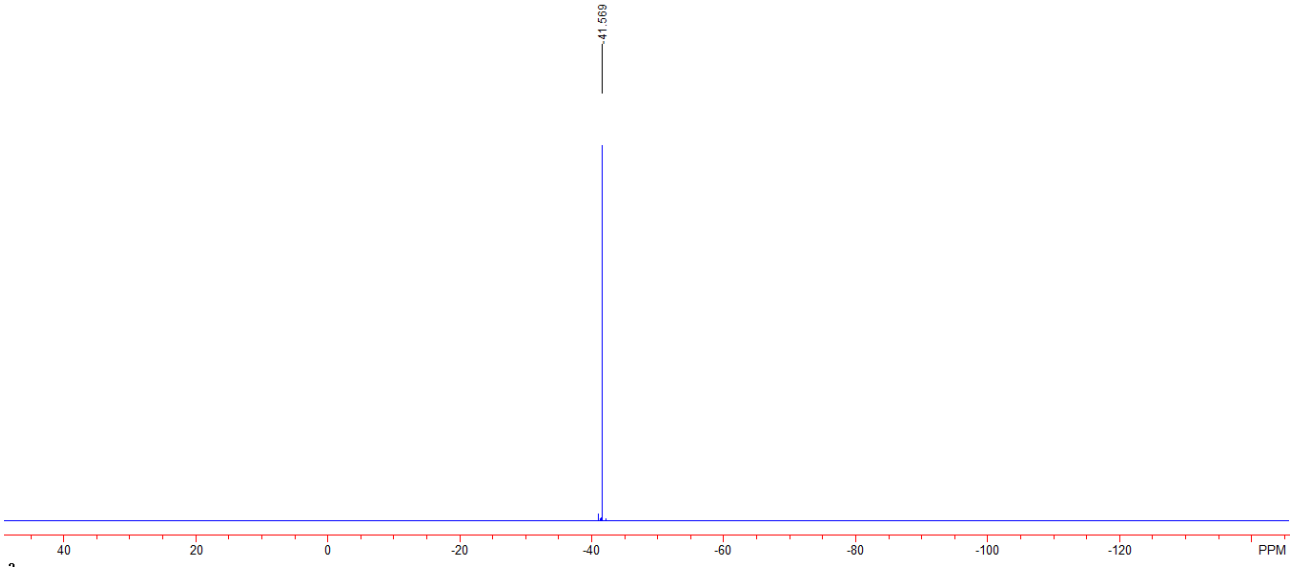

166.833
143.723
135.209
133.401
133.037
132.154
131.500
129.528
129.092
128.571
128.529
127.665
127.346
126.757
126.170
126.039
125.707
77.318
77.000
76.682
52.494
27.319
27.289
27.270
27.246
160
140
120
100
80
60
40
20
PPM

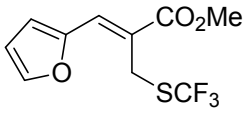

41.739
20
0
-20
-40
-60
-80
-100
PPM



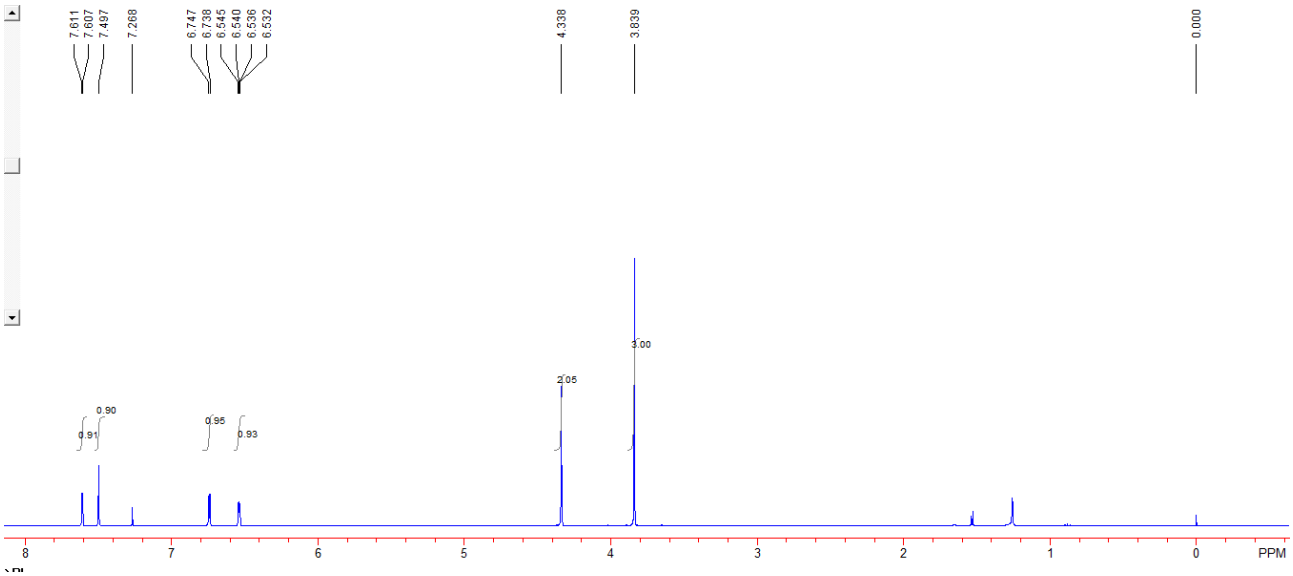
Compound 3l: Yield: 53 mg, 94%. A white solid. m. p.: 69-70 ^{o}C. IR (neat) ν 3008, 2984, 2951, 1710, 1618, 1435, 1417, 1344, 1276, 1257, 1210, 1113, 764, 750 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.85 (s, 3H), 4.26 (s, 2H), 7.15 (t, *J* = 4.4 Hz, 1H), 7.39 (d, *J* = 3.2 Hz, 1H), 7.60 (d, *J* = 4.4 Hz, 1H), 7.98 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 27.2 (q, *J* = 2.4 Hz), 52.5, 121.5, 127.8, 130.7 (q, *J* = 306.0 Hz), 131.2, 133.8, 135.4, 136.7, 166.8; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.6; HRMS (EI) Calcd. for $C_{10}H_9F_3O_2S_2$ requires (M^+): 281.9996, Found: 281.9997.







Compound 3m: Yield: 40 mg, 75%. A colourless oil. IR (neat) ν 3004, 2955, 2923, 2850, 1709, 1633, 1437, 1275, 1267, 1213, 1195, 1111, 1084, 1020, 884, 764, 750 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.84 (s, 3H), 4.34 (s, 2H), 6.54 (dd, J = 3.6 Hz, 2.0 Hz, 1H), 6.74 (d, J = 3.6 Hz, 1H), 7.50 (s, 1H), 7.61 (d, J = 1.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 26.8 (q, J = 2.3 Hz), 52.4, 112.4, 118.2, 122.3, 127.7, 131.4 (q, J = 305.5 Hz), 145.7, 150.3, 167.1; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.6; HRMS (EI) Calcd. for $C_{10}H_9F_3O_3S$ requires (M^+): 266.0225, Found: 266.0218.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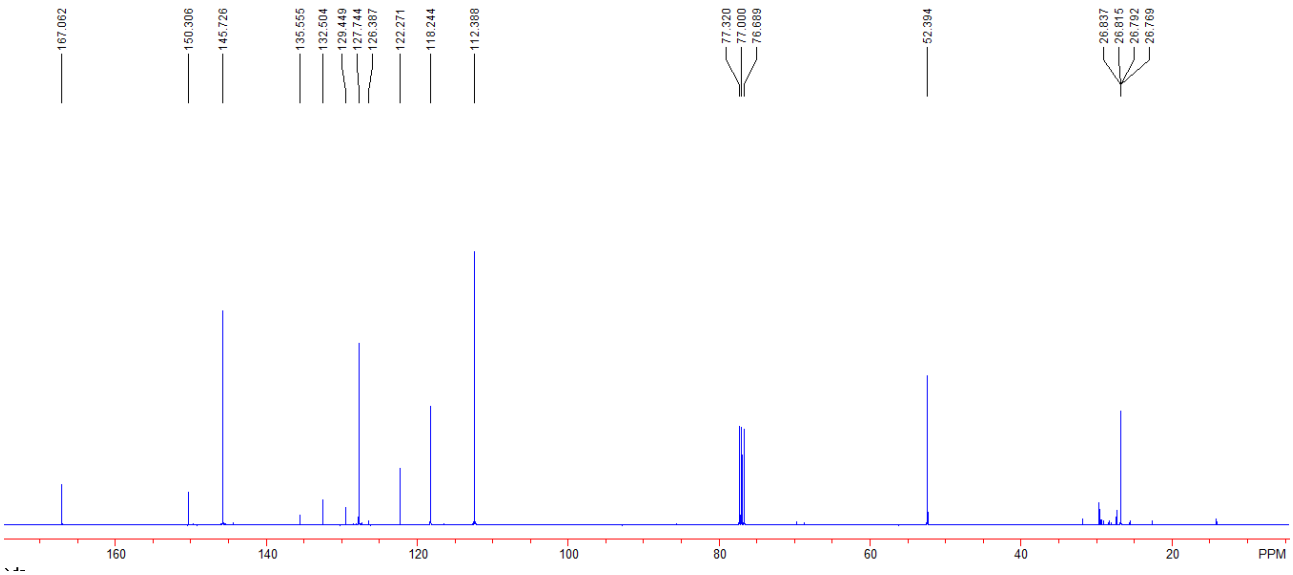
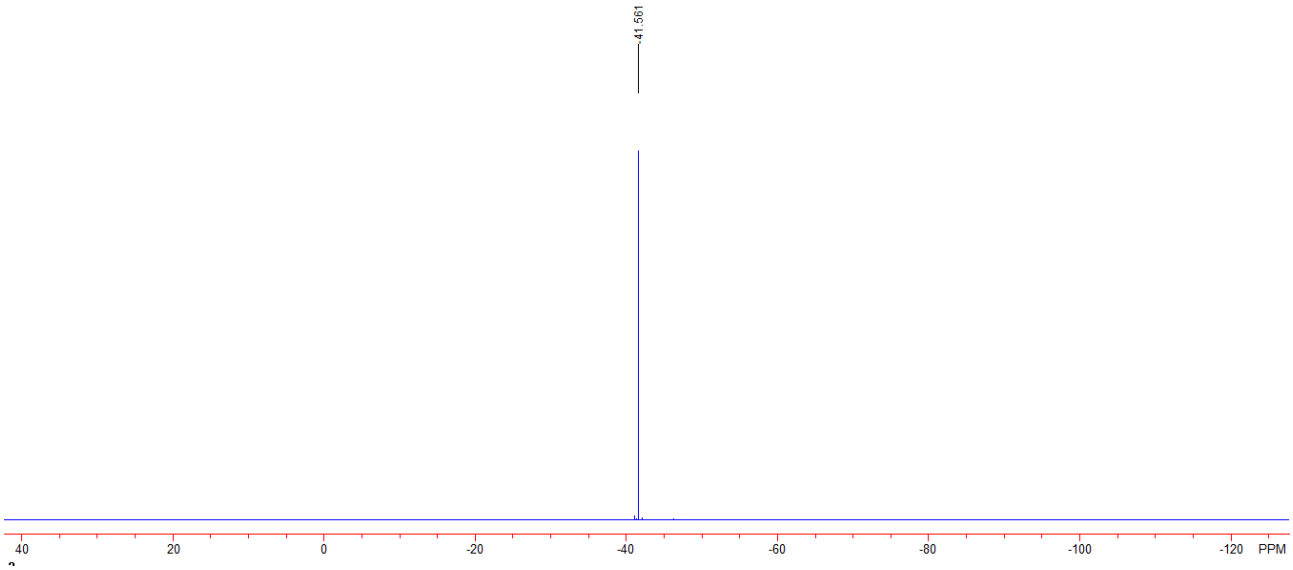
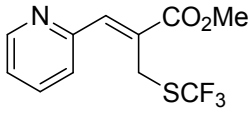
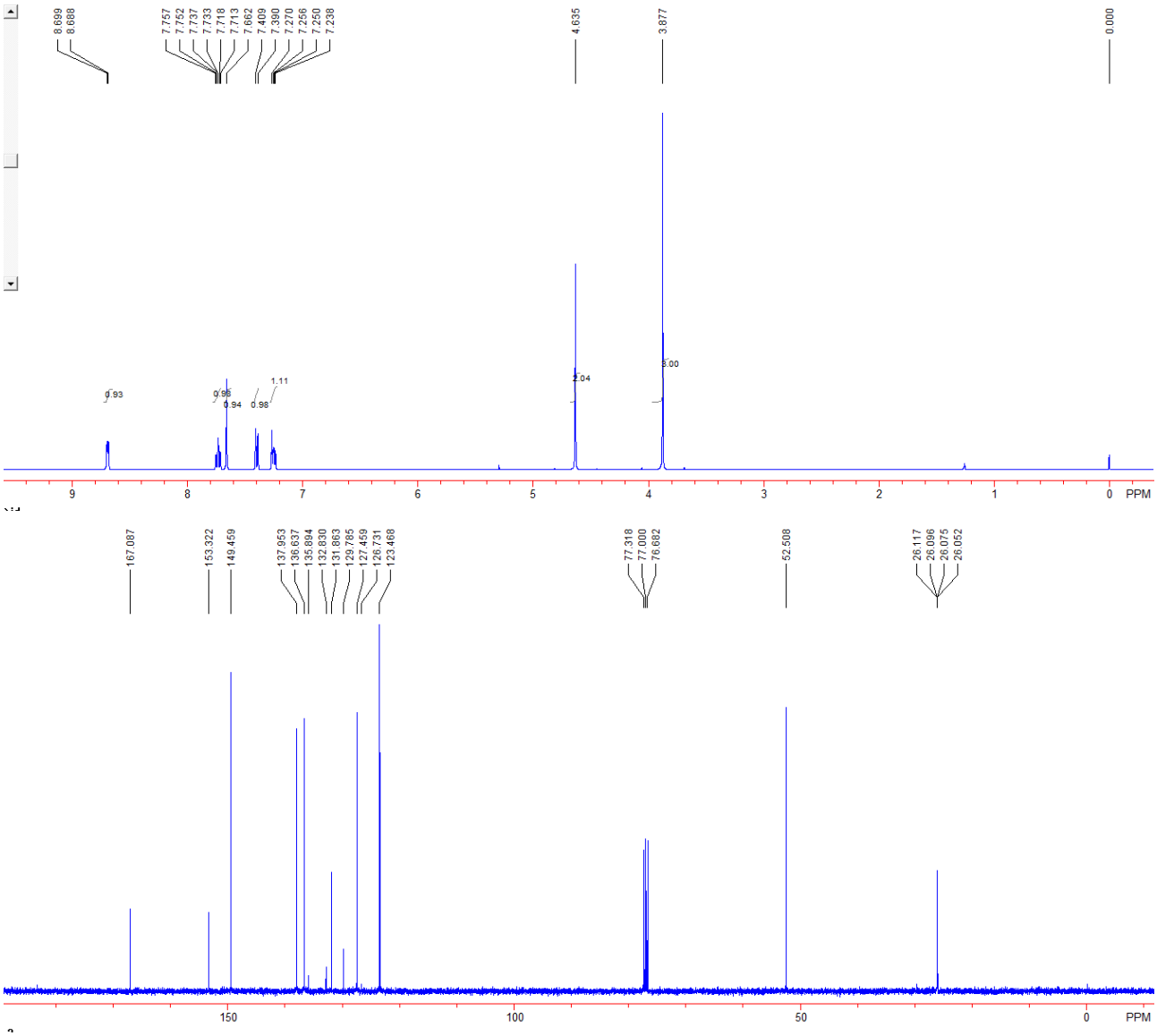
7.607
7.497
7.268
6.747
6.738
6.545
6.540
6.536
6.532
4.338
3.839
0.000
0.90
0.91
0.95
0.93
2.05
3.00
8
7
6
5
4
3
2
1
0
PPM

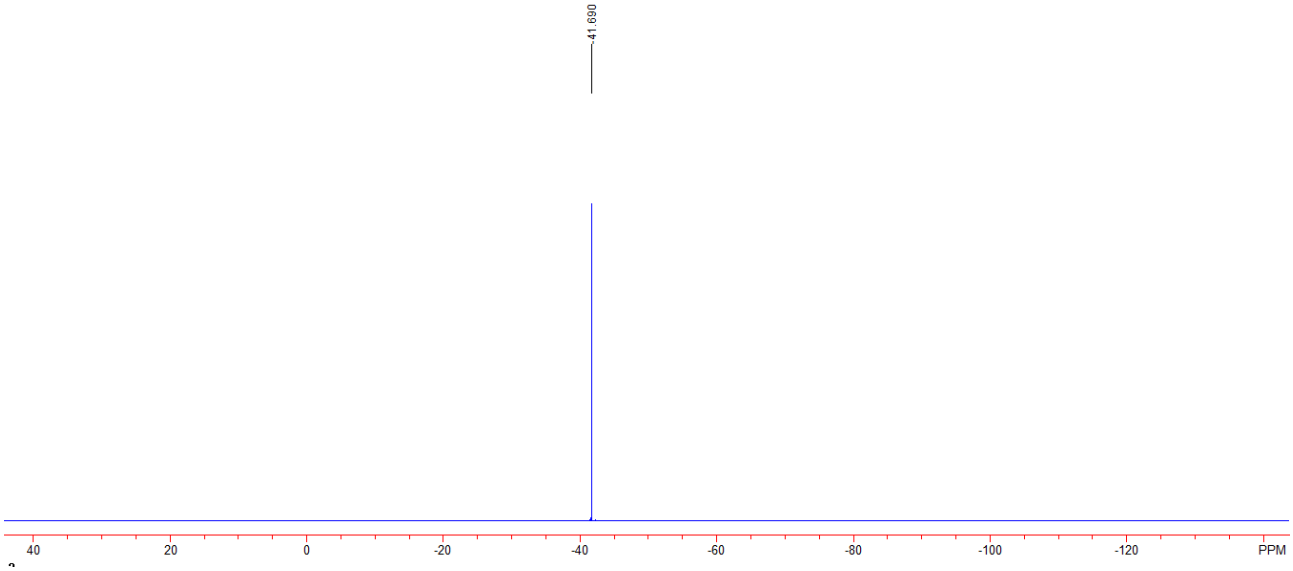

167.062
150.306
145.726
135.555
132.504
129.449
127.744
126.387
122.271
118.244
112.388
77.320
77.000
76.689
52.394
26.837
26.815
26.792
26.769
160
140
120
100
80
60
40
20
PPM

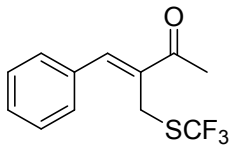

41.561
40
20
0
-20
-40
-60
-80
-100
-120
PPM



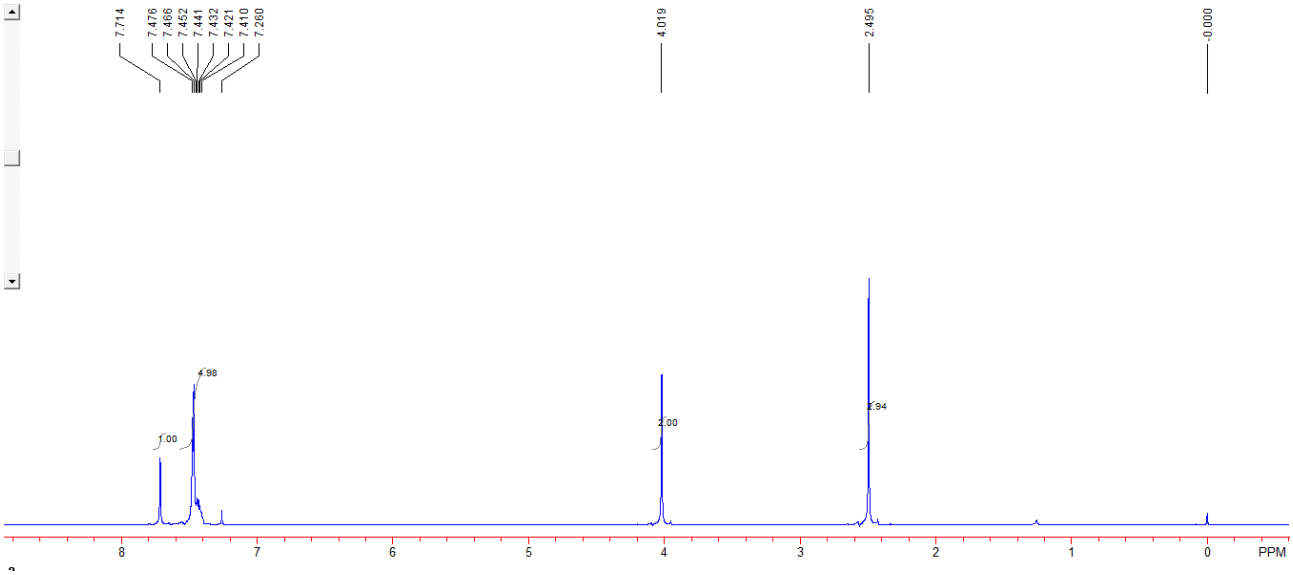
Compound 3n: Yield: 48 mg, 87%. A white solid. m. p.: 48-50 ^{o}C. IR (neat) ν 3008, 2984, 2951, 1716, 1583, 1437, 1276, 1261, 1225, 1115, 1079, 789, 764, 750 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.88 (s, 3H), 4.64 (s, 2H), 7.25 (dd, *J* = 7.2 Hz, 4.8 Hz, 1H), 7.40 (d, *J* = 7.6 Hz, 1H), 7.66 (s, 1H), 7.74 (td, *J* = 8.0 Hz, 2.0 Hz, 1H), 8.69 (d, *J* = 4.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 26.1 (q, *J* = 2.1 Hz), 52.5, 123.5, 127.5, 131.3 (q, *J* = 304.5 Hz), 131.9, 136.6, 138.0, 149.5, 153.3, 167.1; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.7; HRMS (EI) Calcd. for $C_{11}H_{10}F_3NO_2S$ requires (M^+): 277.0384, Found: 277.0380.

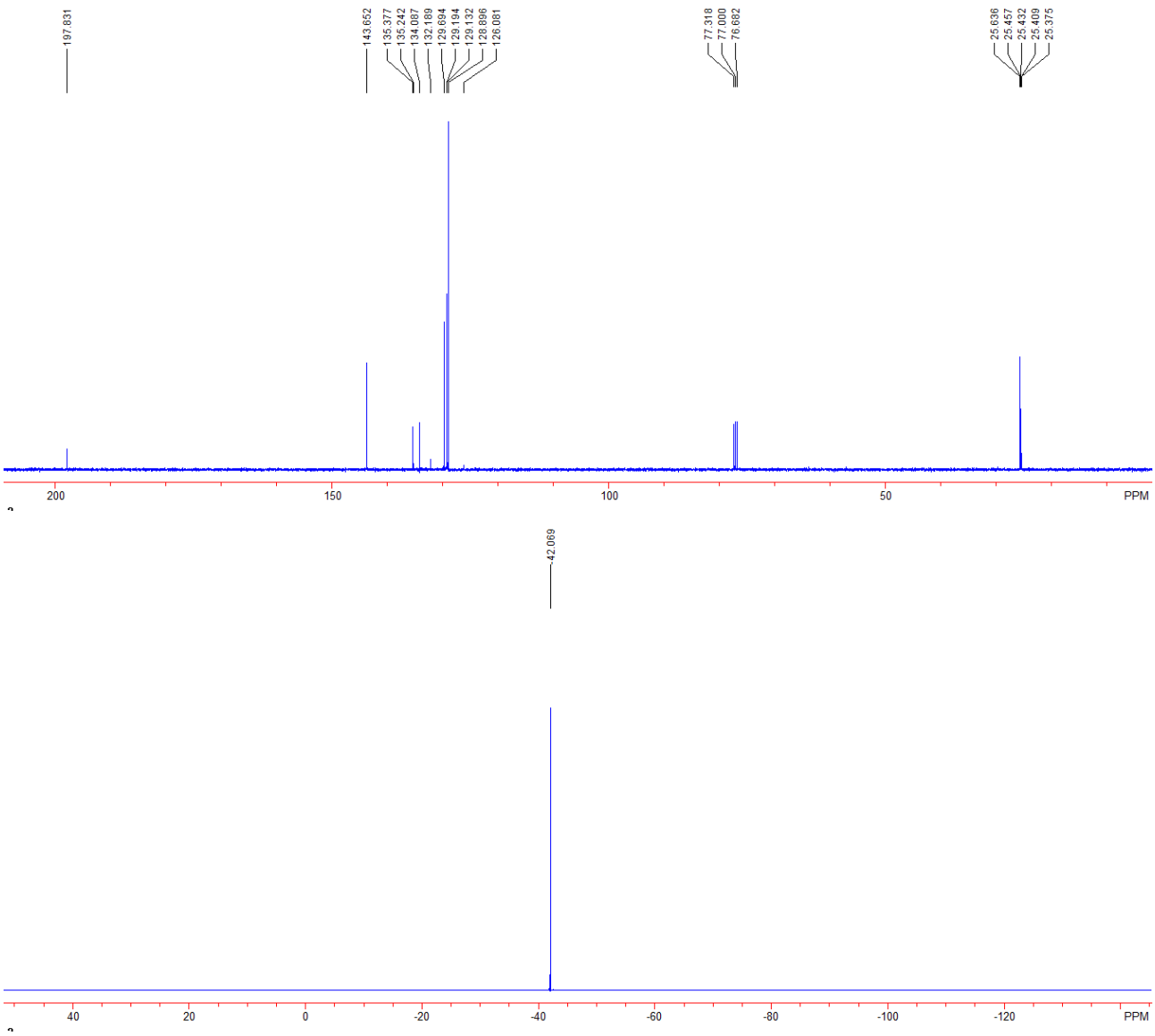






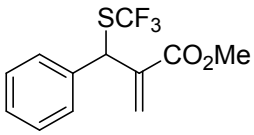
Compound 3o: Yield: 50 mg, 96%. A white solid. m. p.: 42-43 ^{o}C. IR (neat) ν 3004, 2923, 2846, 1670, 1624, 1447, 1378, 1275, 1260, 1216, 1147, 1109, 965, 765, 750, 696 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 2.50 (s, 3H), 4.02 (s, 2H), 7.41-7.48 (m, 5H), 7.71 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 25.4 (q, J = 2.3 Hz), 25.6, 128.9, 129.2, 129.7, 130.7 (q, J = 305.7 Hz), 134.1, 135.4, 143.7, 197.8; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -42.1; HRMS (EI) Calcd. for $C_{12}H_{11}F_3OS$ requires (M^+): 260.0483, Found: 260.0480.



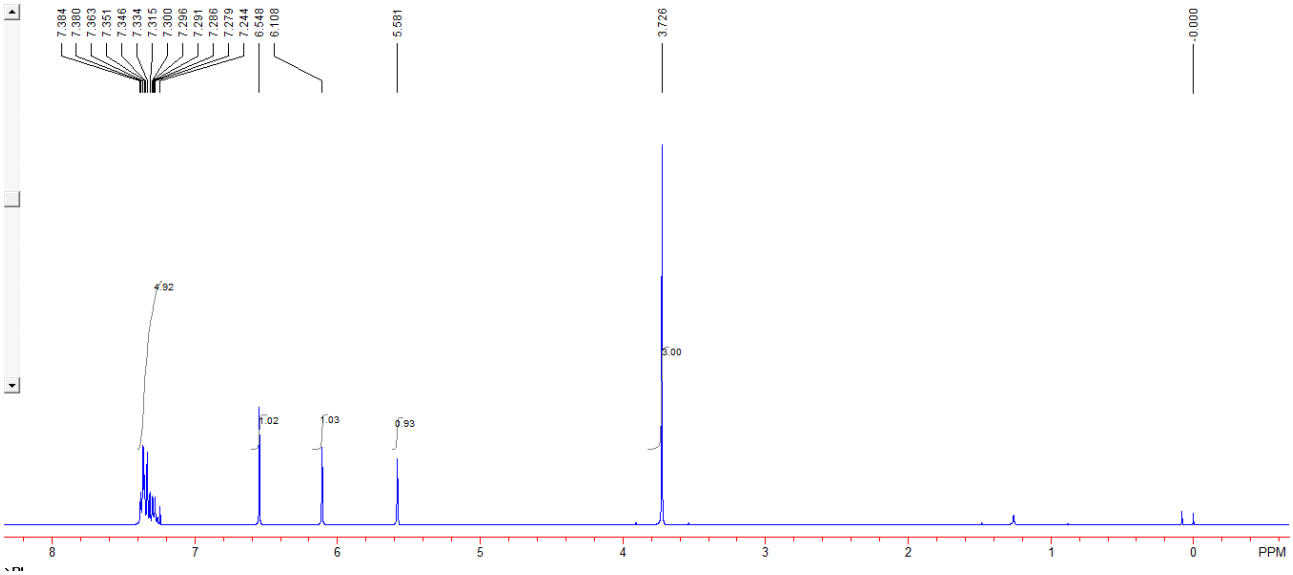


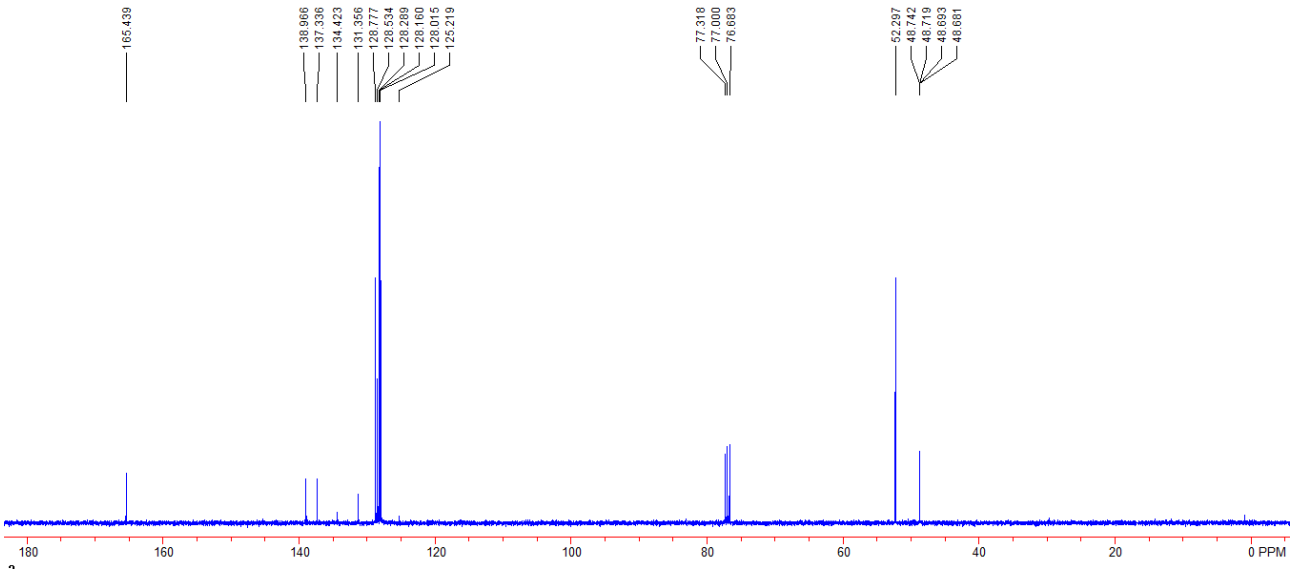
Procedure for DABCO-catalyzed secondary allylic trifluoromethylthiolation reaction of MBH adducts:

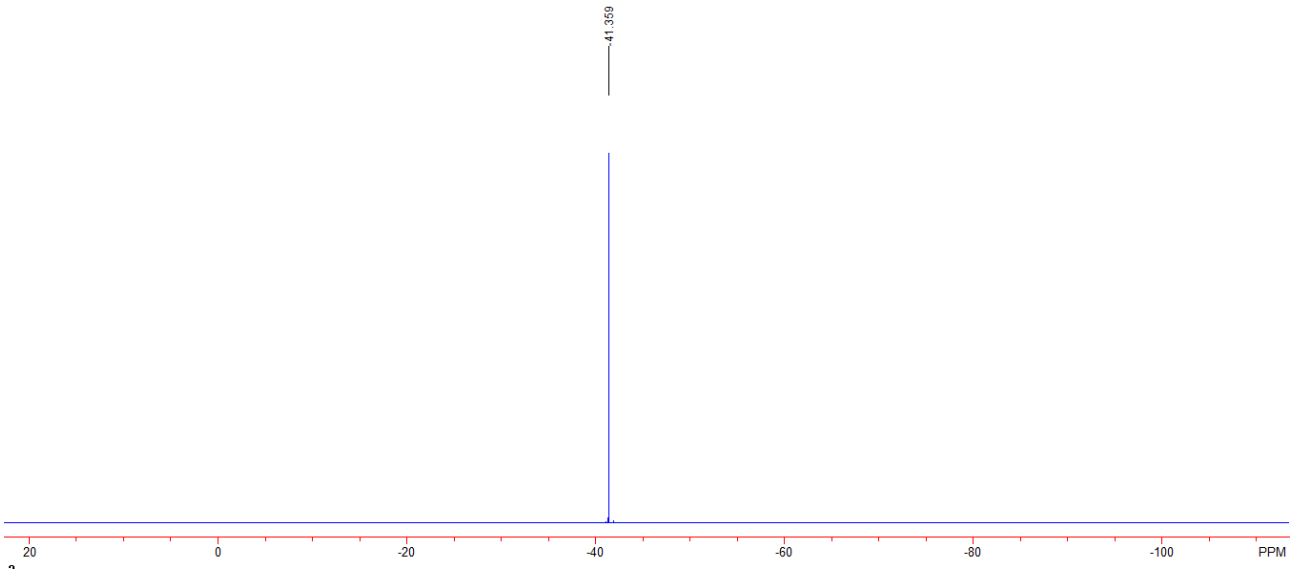
To a solution of MBH adducts **I-3-2** (0.2 mmol) and *O*-octadecyl-*S*-trifluorothiolcarbonate **I-3-1a** (0.4 mmol) in $CHCl_3$ (2 mL) was added DABCO (0.04 mmol) and the reaction mixture was stirred for 10 h at 0 ^oC under air. The reaction mixture was concentrated and the crude product was purified by flash chromatograph (silica gel, petroleum ether : dichloromethane = 10:1-5:1) to give the corresponding products **4**.

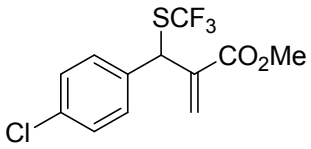


Compound 4a: Yield: 39 mg, 71%. A colourless oil. IR (neat) ν 2961, 2928, 2856, 1725, 1631, 1454, 1440, 1316, 1259, 1111, 1017, 953, 802, 756, 700 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.73 (s, 3H), 5.58 (s, 1H), 6.11 (s, 1H), 6.55 (s, 1H), 7.24-7.38 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 48.7 (q, J = 2.6 Hz), 52.3, 128.0, 128.2, 128.5, 128.8, 129.8 (q, J = 306.7 Hz), 137.3, 139.0, 165.4; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.4; HRMS (EI) Calcd. for $C_{12}H_{11}F_3O_2S$ requires (M^+): 276.0432, Found: 276.0435.

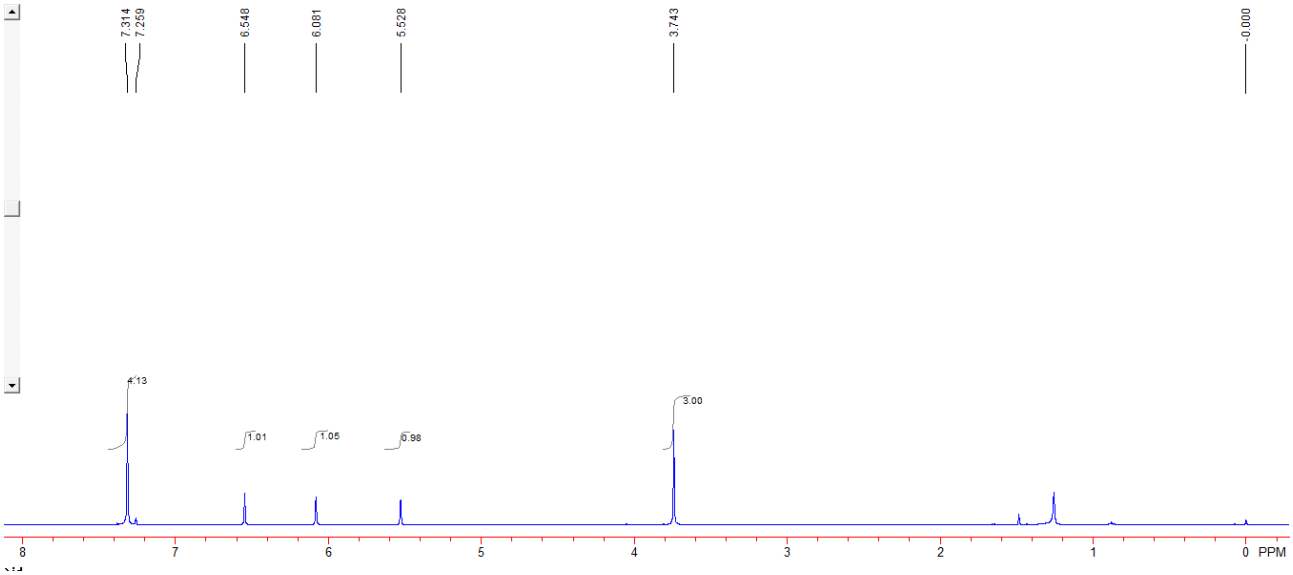


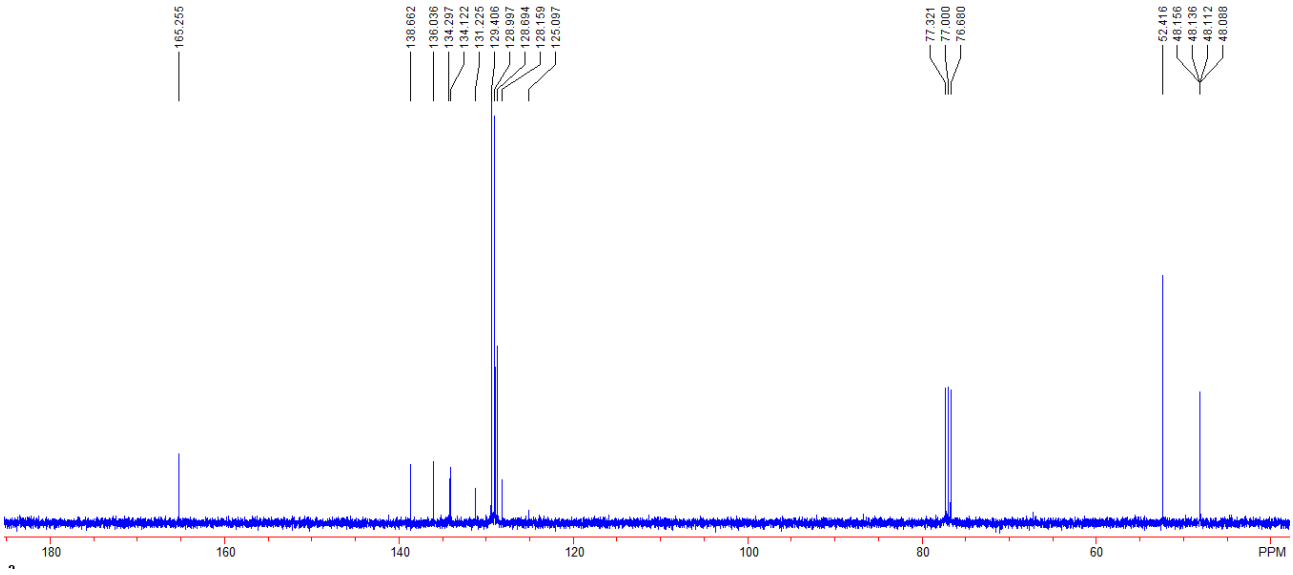


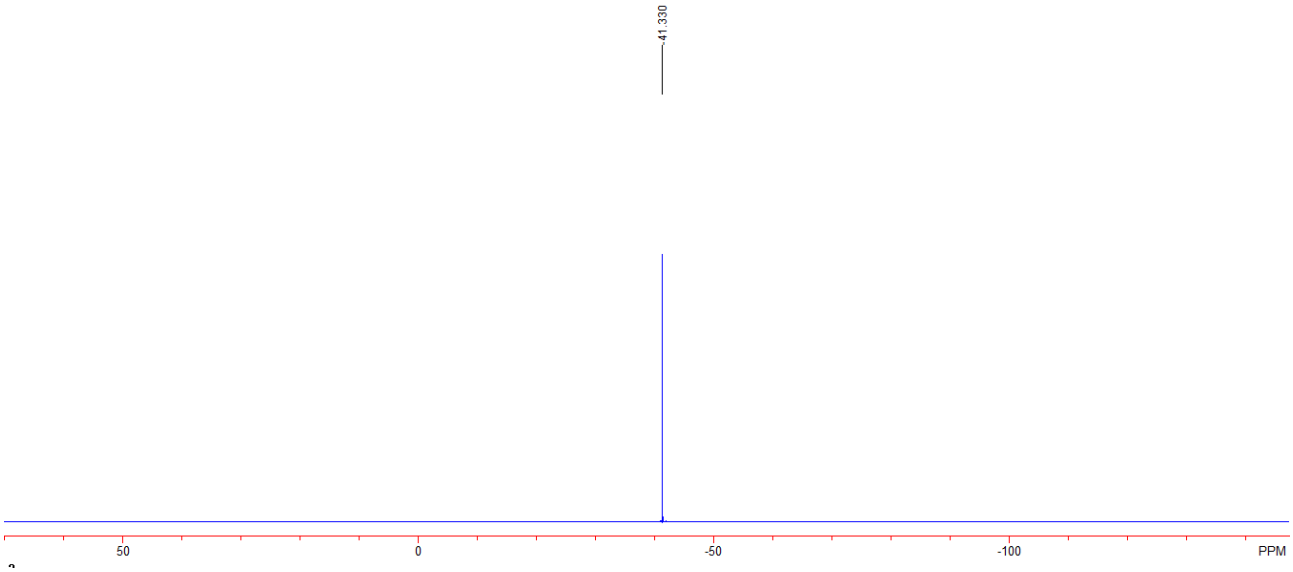


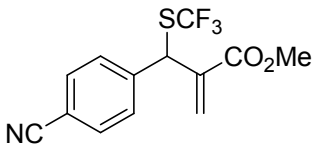


Compound 4b: Yield: 52 mg, 84%. A colourless oil. IR (neat) ν 2960, 2919, 2850, 1723, 1491, 1439, 1250, 1110, 1015, 954, 817, 755 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.74 (s, 3H), 5.53 (s, 1H), 6.08 (s, 1H), 6.55 (s, 1H), 7.31 (brs, 4H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 48.1 (q, J = 2.4 Hz), 52.4, 128.7, 129.0, 129.4, 129.7 (q, J = 306.6 Hz), 134.1, 136.0, 138.7, 165.3; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.3; HRMS (EI) Calcd. for $C_{12}H_{10}ClF_3O_2S$ requires (M^+): 310.0042, Found: 310.0037.

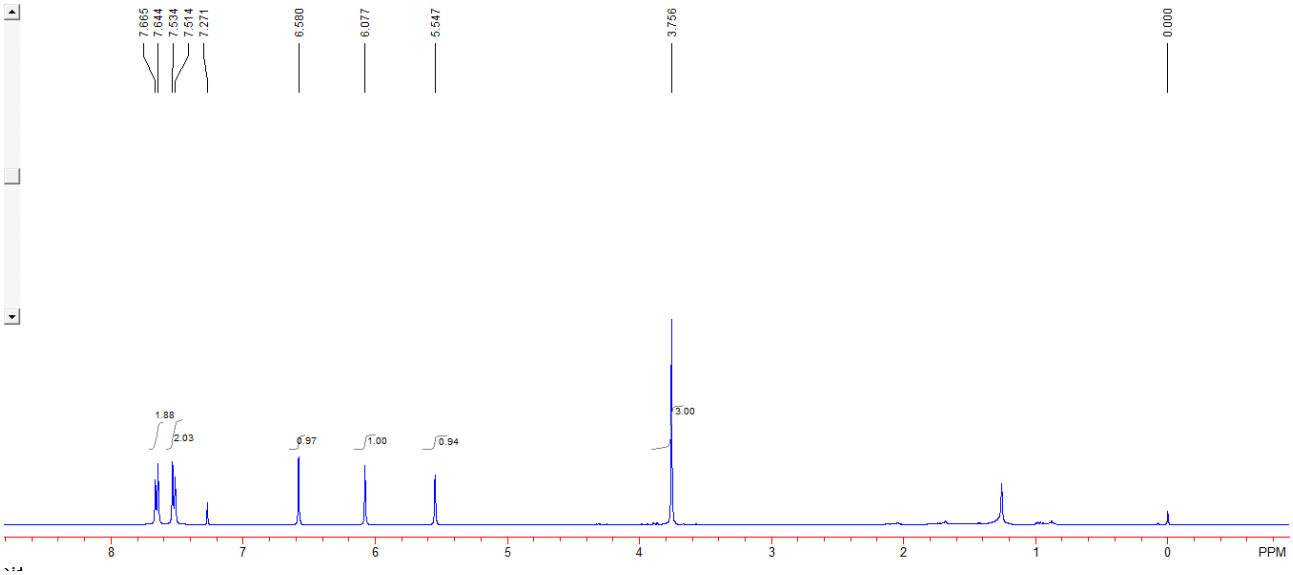


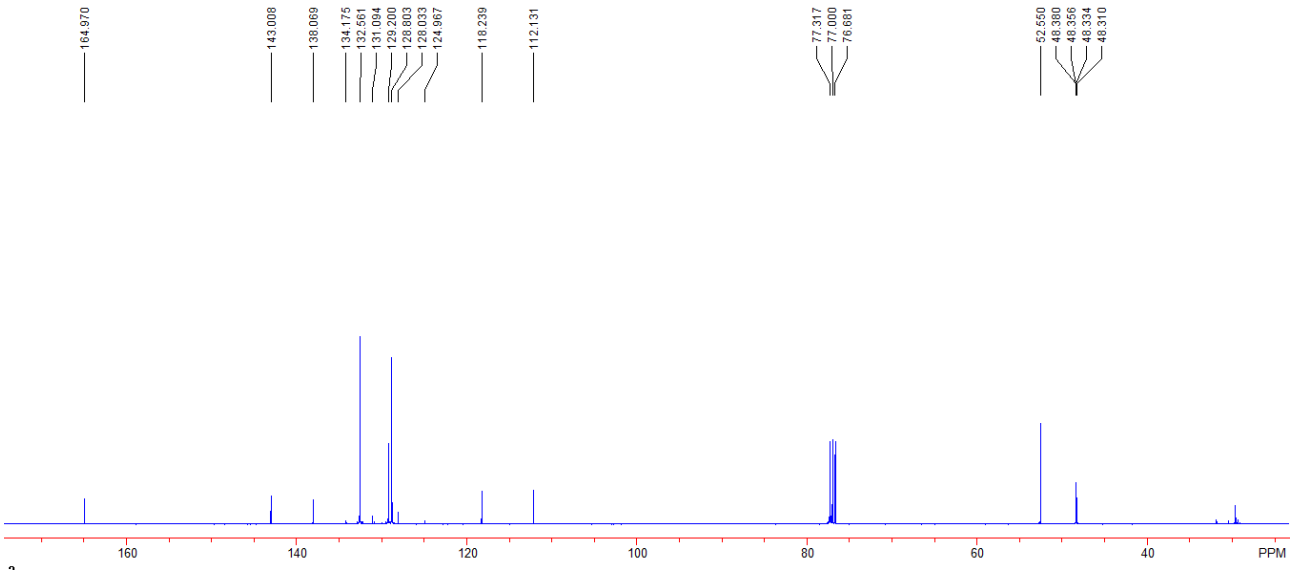


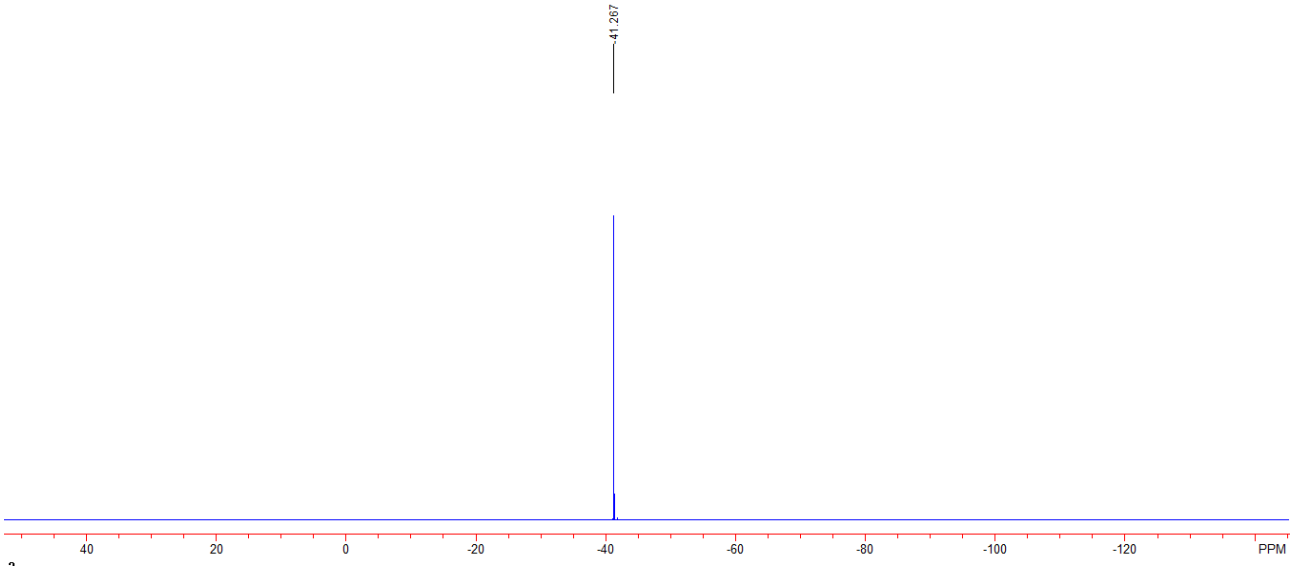


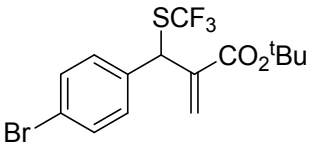


Compound 4c: Yield: 28 mg, 46%. A colourless oil. IR (neat) ν 3004, 2955, 2927, 2850, 2231, 1720, 1439, 1334, 1316, 1276, 1258, 1104, 961, 828, 751 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.76 (s, H), 5.55 (s, 1H), 6.08 (s, 1H), 6.58 (s, 1H), 7.52 (d, J = 8.0 Hz, 2H), 7.65 (d, J = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 48.3 (q, J = 2.2 Hz), 52.6, 112.1, 118.2, 128.8, 129.2, 129.6 (q, J = 306.1 Hz), 132.6, 138.1, 143.0, 165.0; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.3; HRMS (EI) Calcd. for $C_{13}H_{10}F_3NO_2S$ requires (M^+): 301.0384, Found: 301.0381.

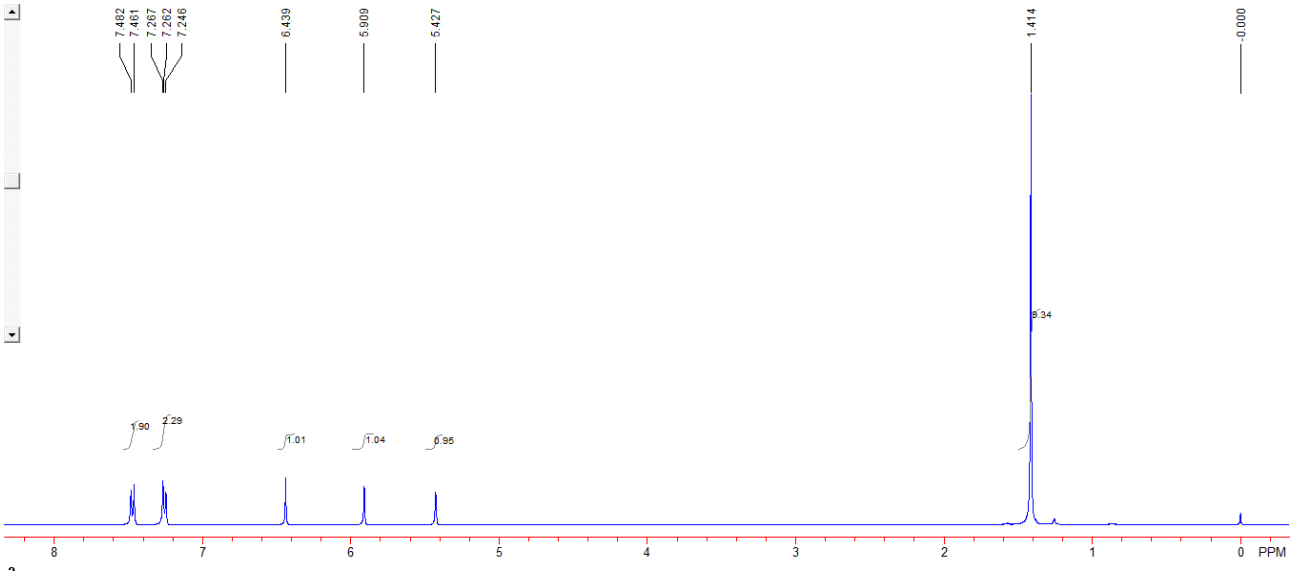

7.665
7.644
7.534
7.514
7.271
6.580
6.077
5.547
3.756
0.000
1.88
2.03
0.97
1.00
0.94
3.00
8
7
6
5
4
3
2
1
0
PPM

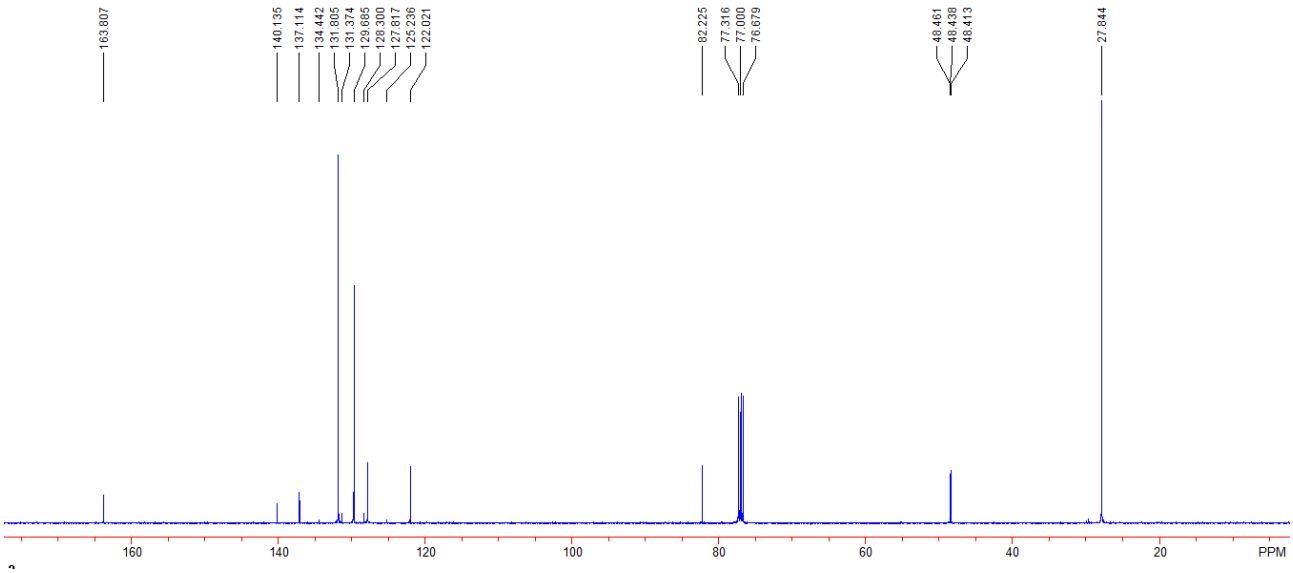

164.970
143.008
138.069
134.175
132.561
131.094
129.200
128.803
128.033
124.967
118.239
112.131
77.317
77.000
76.681
52.550
48.380
48.356
48.334
48.310
160
140
120
100
80
60
40
PPM

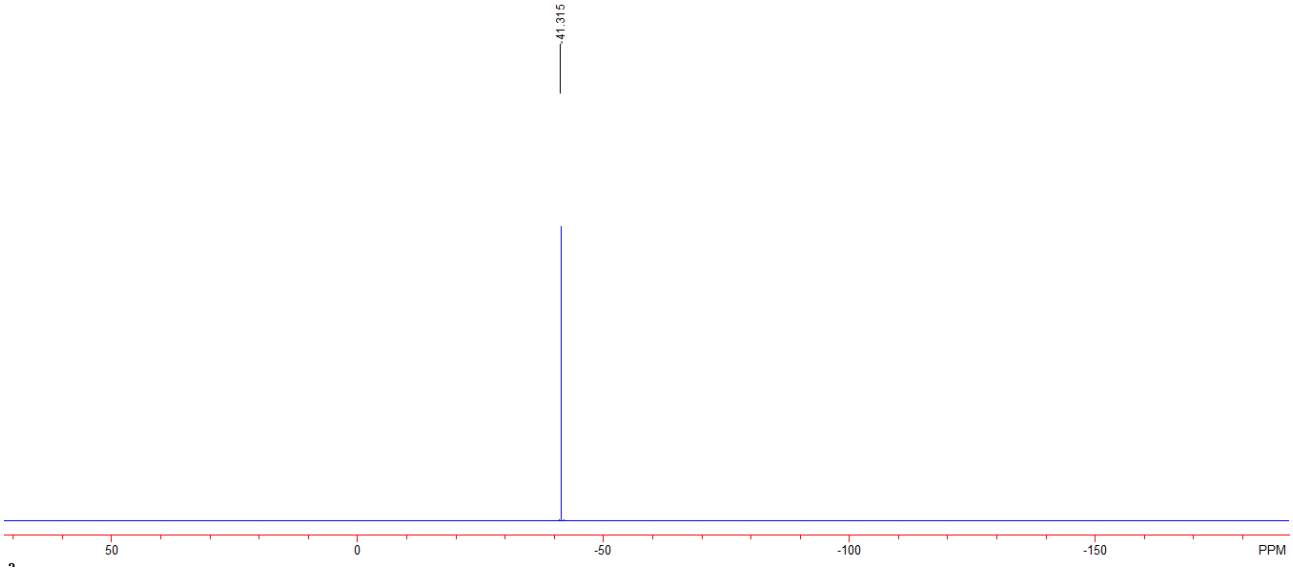

41.267
40
20
0
-20
-40
-60
-80
-100
-120
PPM

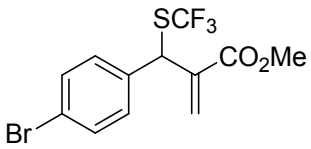


Compound 4d: Yield: 60 mg, 76%. A colourless oil. IR (neat) ν 3004, 2980, 2923, 1722, 1706, 1275, 1260, 1149, 1112, 1012, 764, 750 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 1.41 (s, 9H), 5.43 (s, 1H), 5.91 (s, 1H), 6.44 (s, 1H), 7.26 (d, J = 8.4 Hz, 2H), 7.47 (d, J = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 27.8, 48.4 (q, J = 2.5 Hz), 82.2, 122.0, 127.8, 129.7, 129.8 (q, J = 307.4 Hz), 131.8, 137.1, 140.1, 163.8; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.3; HRMS (EI) Calcd. for $C_{15}H_{16}BrF_3O_2S$ requires (M^+): 396.0006, Found: 396.0009.

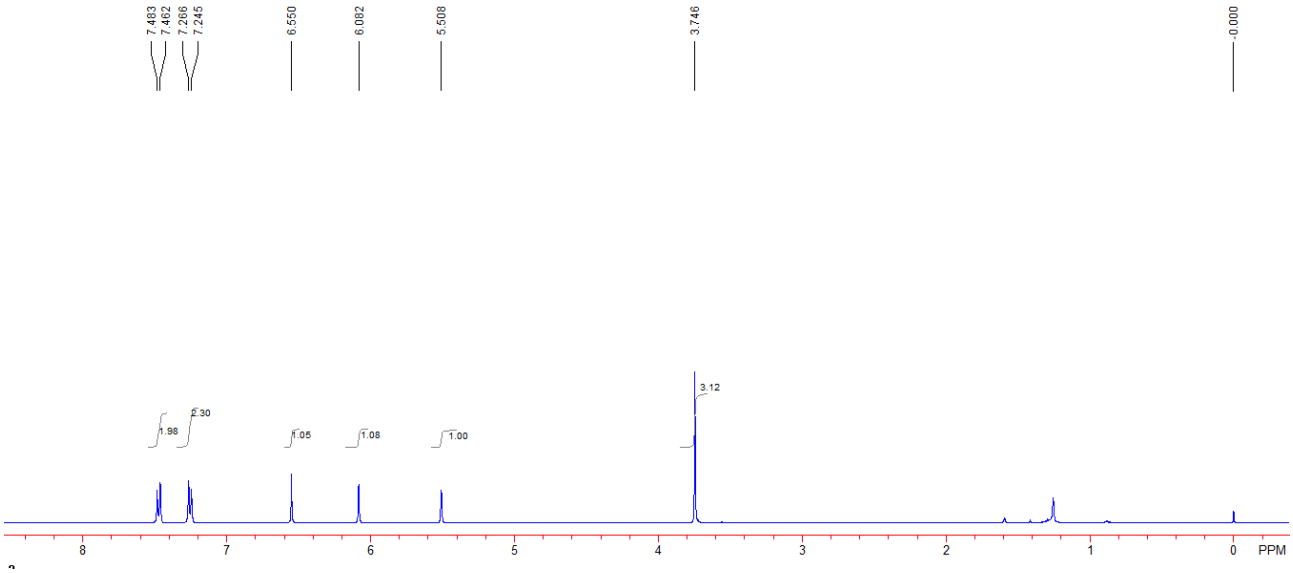


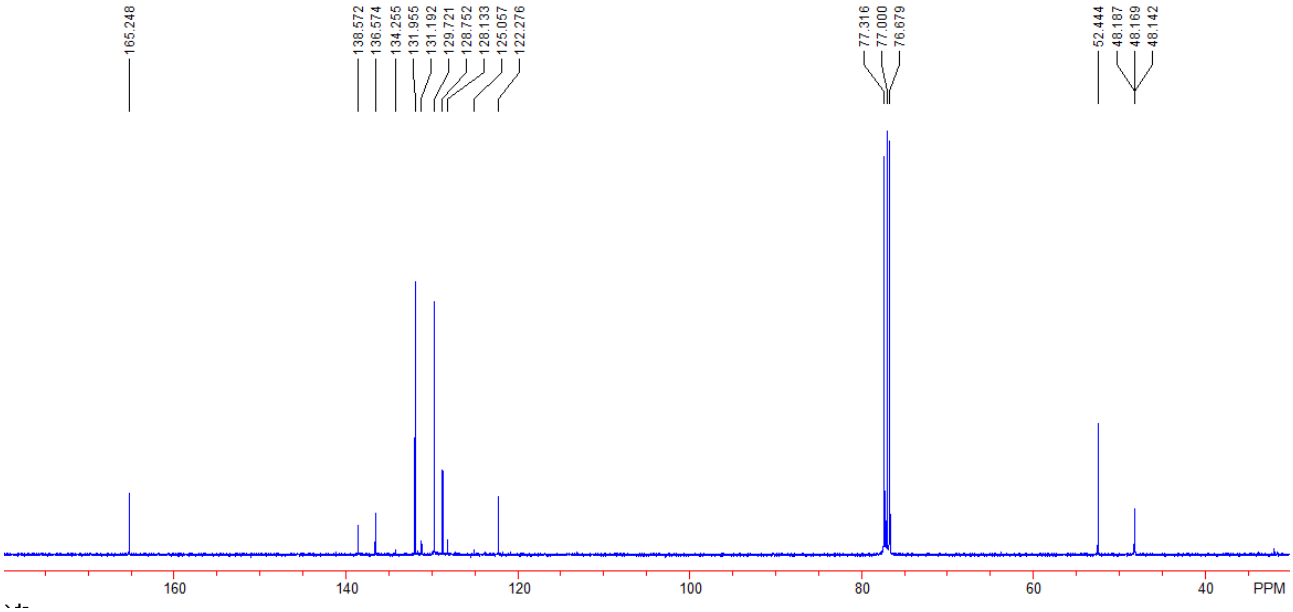


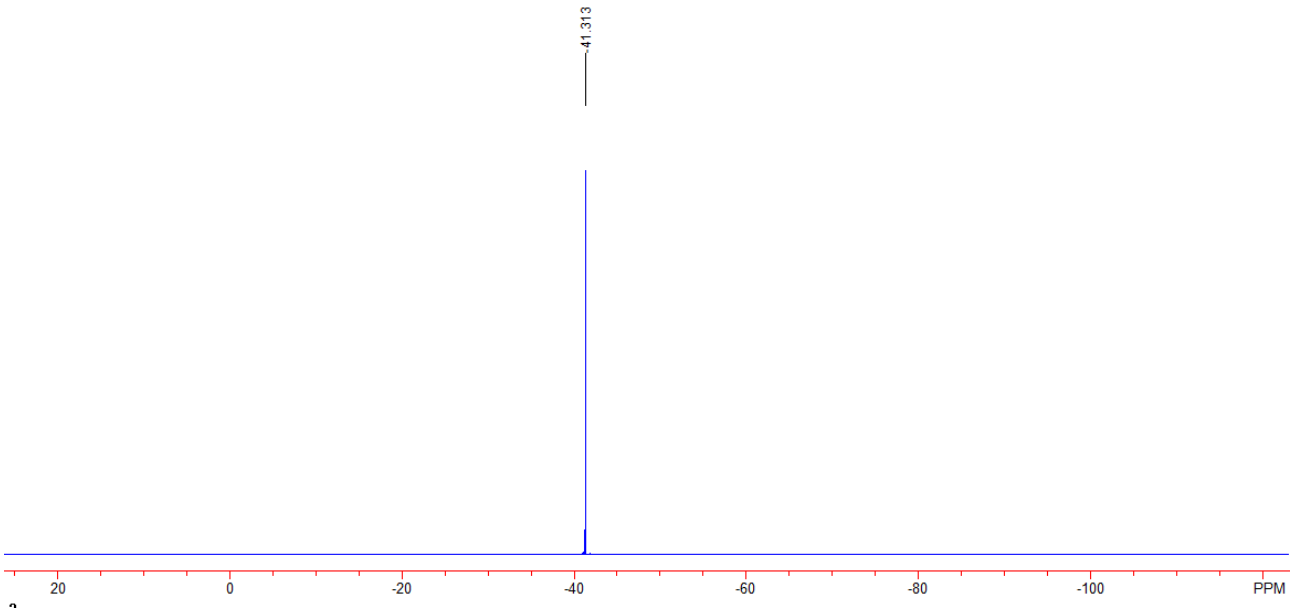


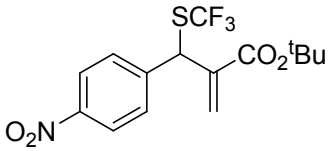


Compound 4e: Yield: 36 mg, 50%. A colourless oil. IR (neat) ν 2960, 2926, 2854, 1723, 1629, 1488, 1439, 1404, 1330, 1249, 1149, 1108, 1074, 1012, 849, 815 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.75 (s, 3H), 5.51 (s, 1H), 6.08 (s, 1H), 6.55 (s, 1H), 7.26 (d, J = 8.4 Hz, 2H), 7.47 (d, J = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 48.2 (q, J = 2.7 Hz), 52.4, 122.3, 128.8, 129.66 (q, J = 305.9 Hz), 129.7, 132.0, 136.6, 138.6, 165.2; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.3; HRMS (EI) Calcd. for $C_{12}H_{10}BrF_3O_2S$ requires (M^+): 353.9537, Found: 353.9535.



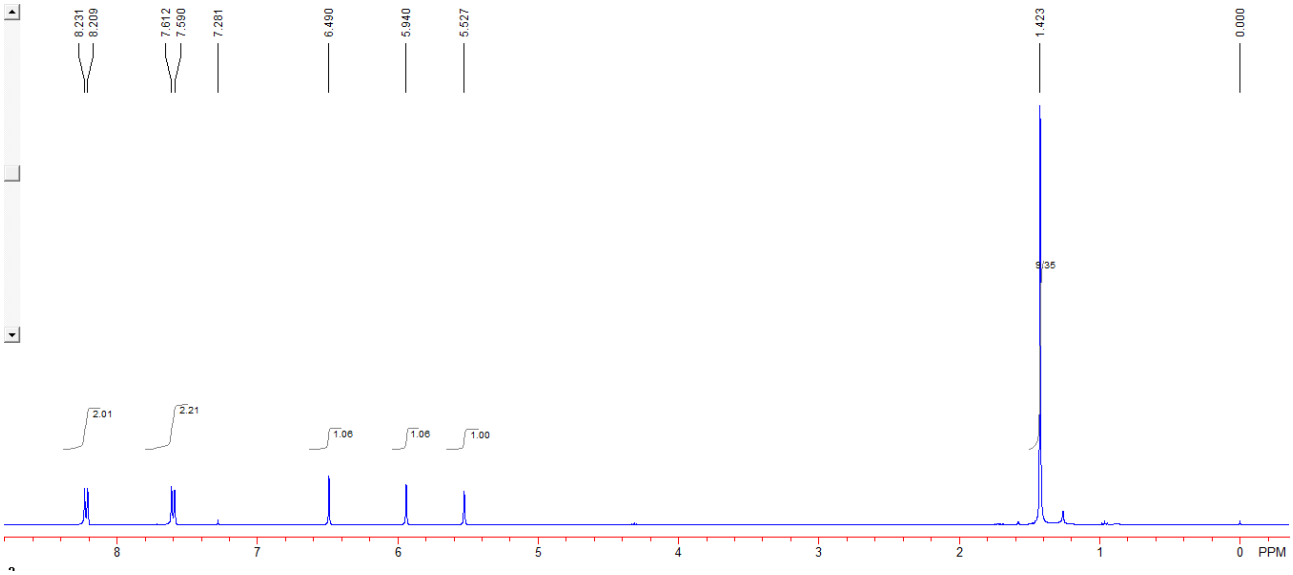


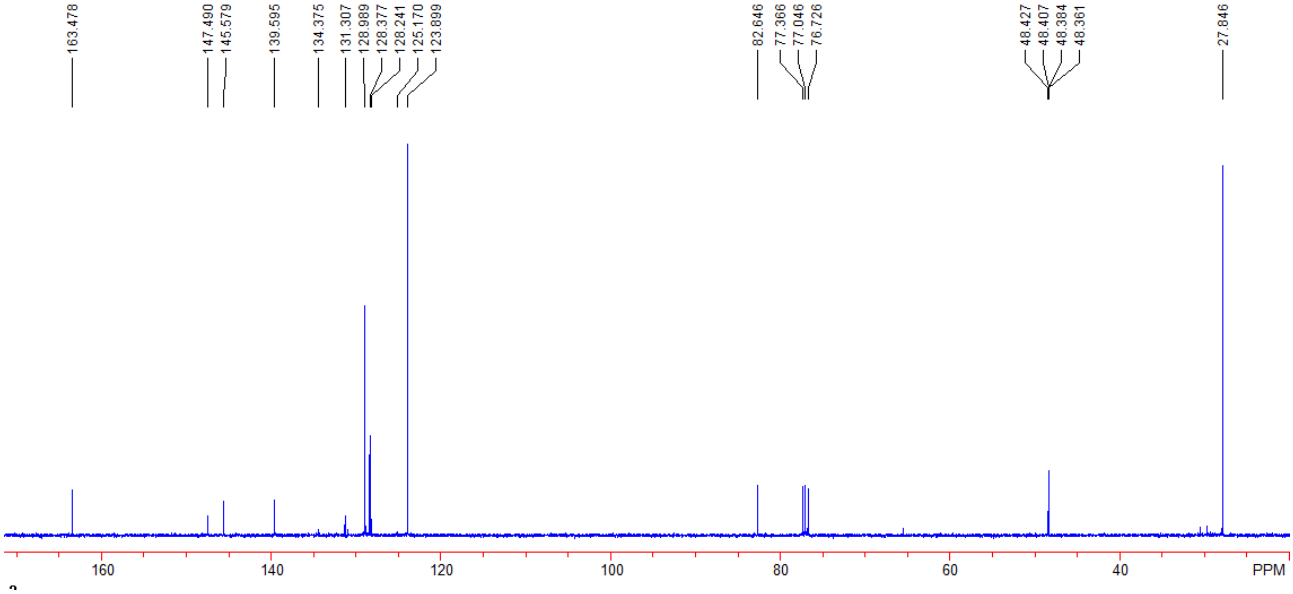


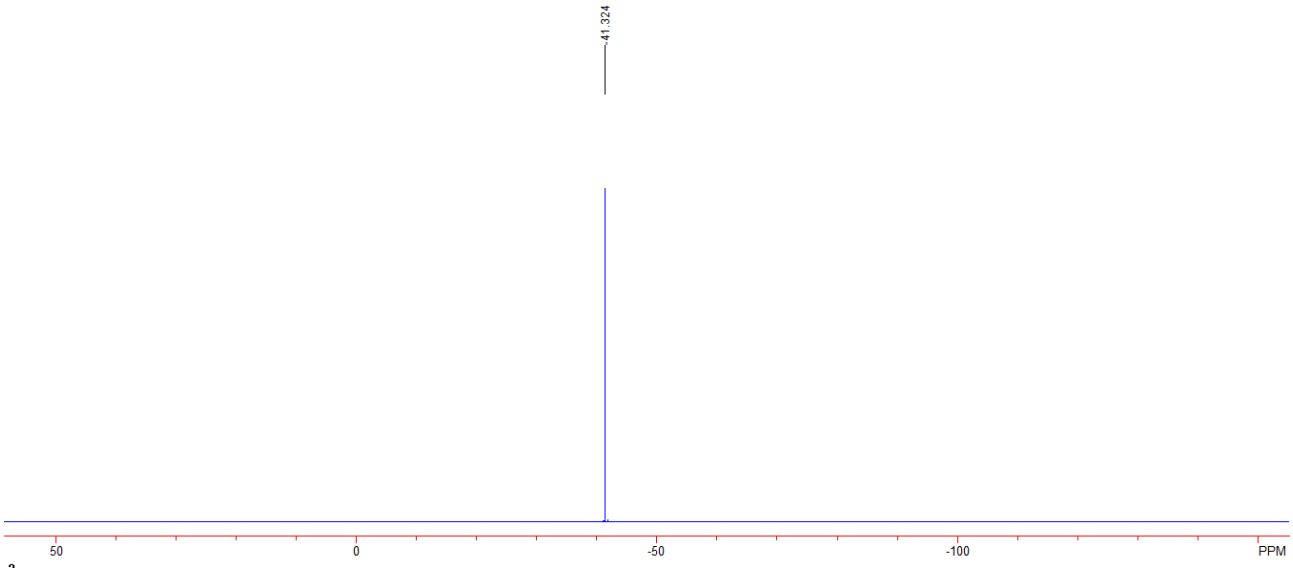


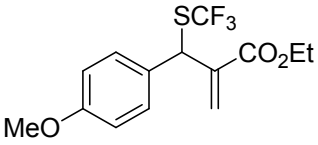
Compound 4f: Yield: 60 mg, 83%. A colourless oil. IR (neat) ν 2980, 2931, 2854, 1725, 1708, 1607, 1597, 1526, 1370, 1348, 1253, 1147, 1109, 963, 851, 833, 756 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 1.42 (s, 9H), 5.53 (s, 1H), 5.94 (s, 1H), 6.49 (s, 1H), 7.60 (d, J = 8.8 Hz, 2H), 8.22 (d, J = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 27.8, 48.4 (q, J = 2.3 Hz), 82.6, 123.9, 128.4, 129.0, 129.8 (q, J = 306.6 Hz), 139.6, 145.6, 147.5, 163.5; ^{19}F NMR (376 MHz, $CDCl_3$,

$CFCl_3$) δ -41.3; HRMS (EI) Calcd. for $C_{15}H_{16}F_3NO_4S$ requires (M^+): 363.0752, Found: 363.0755.

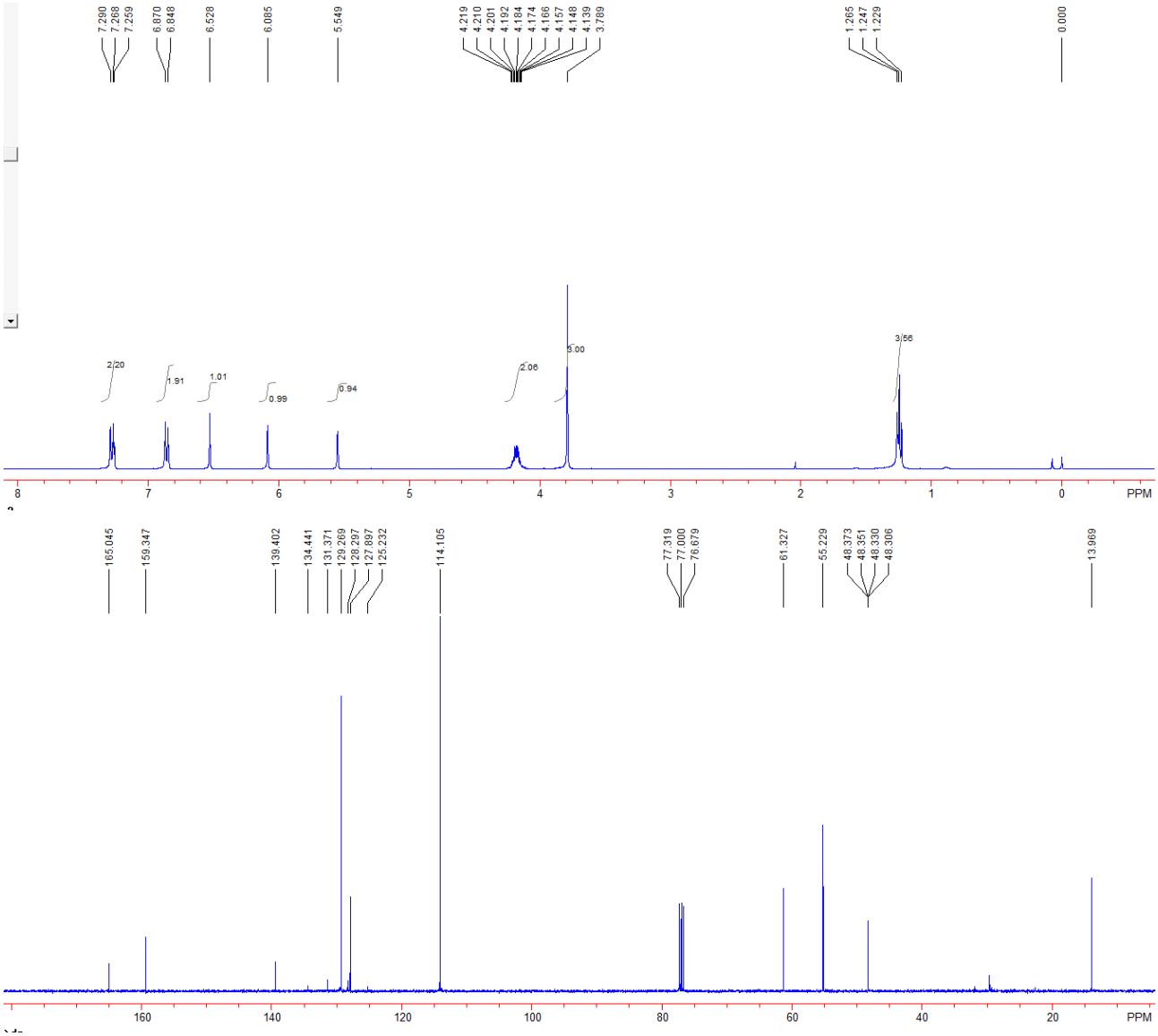


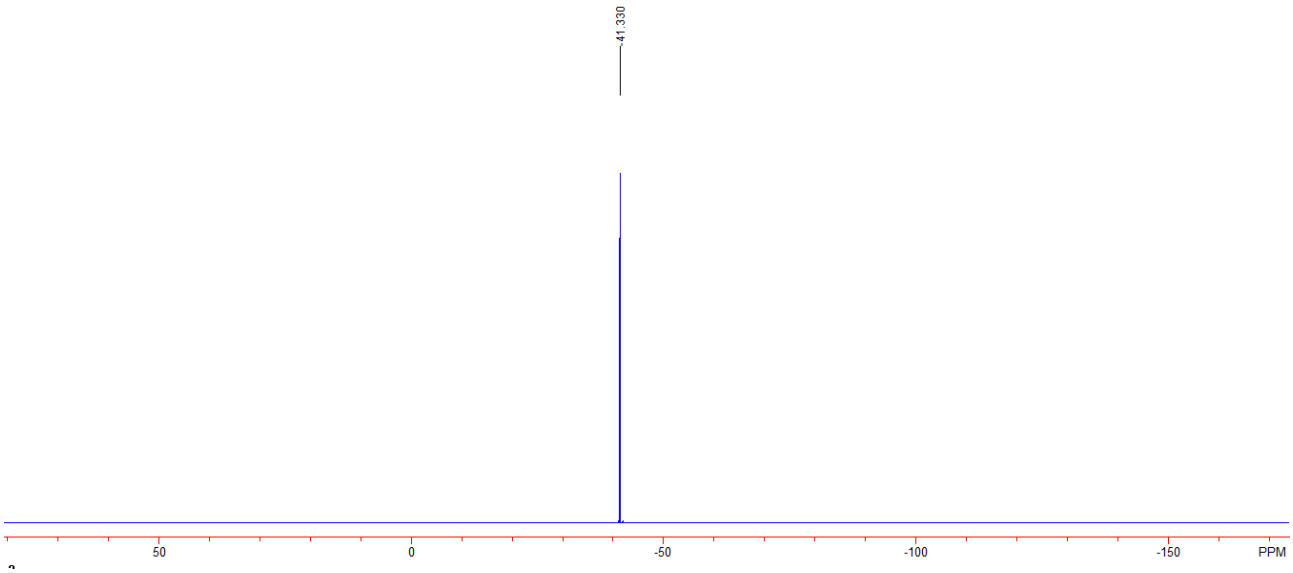


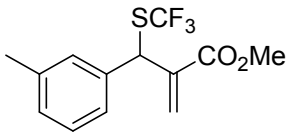




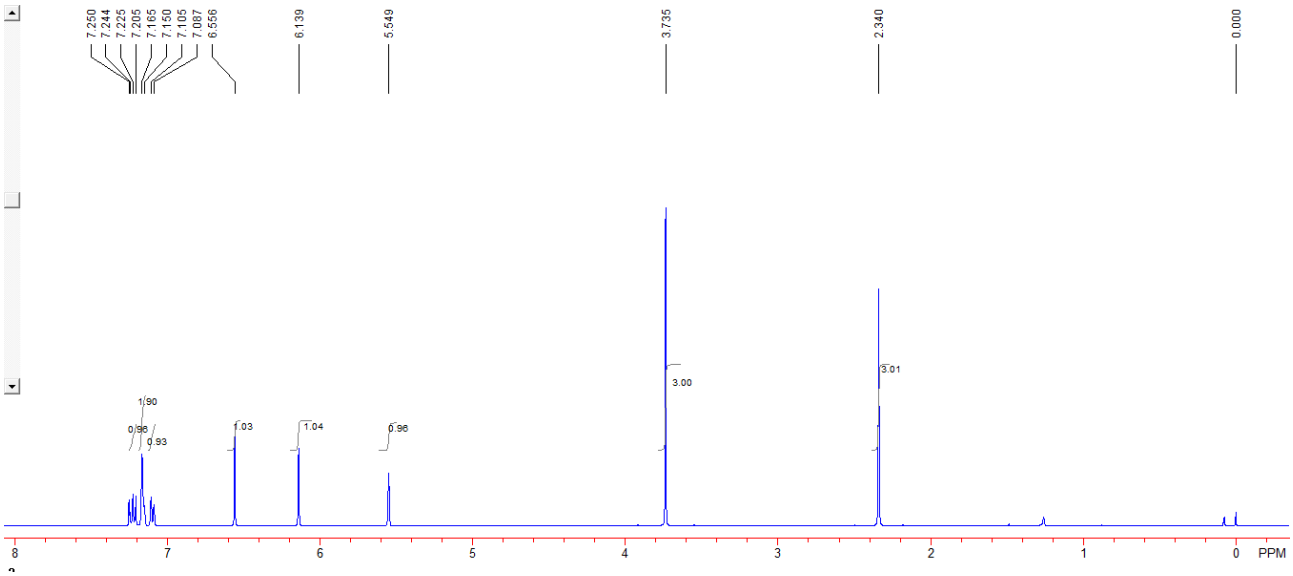
Compound 4g: Yield: 39 mg, 63%. A colourless oil. IR (neat) ν 2964, 2928, 2850, 1717, 1610, 1511, 1463, 1304, 1255, 1175, 1108, 1034 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 1.25 (t, *J* = 7.2 Hz, 3H), 3.79 (s, 3H), 4.14-4.22 (m, 2H), 5.55 (s, 1H), 6.09 (s, 1H), 6.53 (s, 1H), 6.86 (d, *J* = 8.8 Hz, 2H), 7.28 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 14.0, 48.3 (q, *J* = 2.2 Hz), 55.2, 61.3, 114.1, 127.9, 129.3, 129.8 (q, *J* = 307.4 Hz), 139.4, 159.3, 165.0; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.3; HRMS (EI) Calcd. for $C_{14}H_{15}F_3O_3S$ requires (M^+): 320.0694, Found: 320.0688.







Compound 4h: Yield: 49 mg, 84%. A colourless oil. IR (neat) ν 3008, 2955, 2919, 2854, 1722, 1439, 1331, 1276, 1259, 1235, 1108, 958, 752, 704 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 2.34 (s, 3H), 3.74 (s, 3H), 5.55 (s, 1H), 6.14 (s, 1H), 6.56 (s, 1H), 7.10 (d, J = 7.2 Hz, 1H), 7.15-7.17 (m, 2H), 7.23 (t, J = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 21.4, 48.7 (q, J = 2.0 Hz), 52.3, 125.1, 128.5, 128.67, 128.68, 129.0, 129.8 (q, J = 306.5 Hz), 137.1, 138.6, 139.0, 165.5; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.4; HRMS (EI) Calcd. for $C_{13}H_{13}F_3O_2S$ requires (M^+): 290.0588, Found: 290.0586.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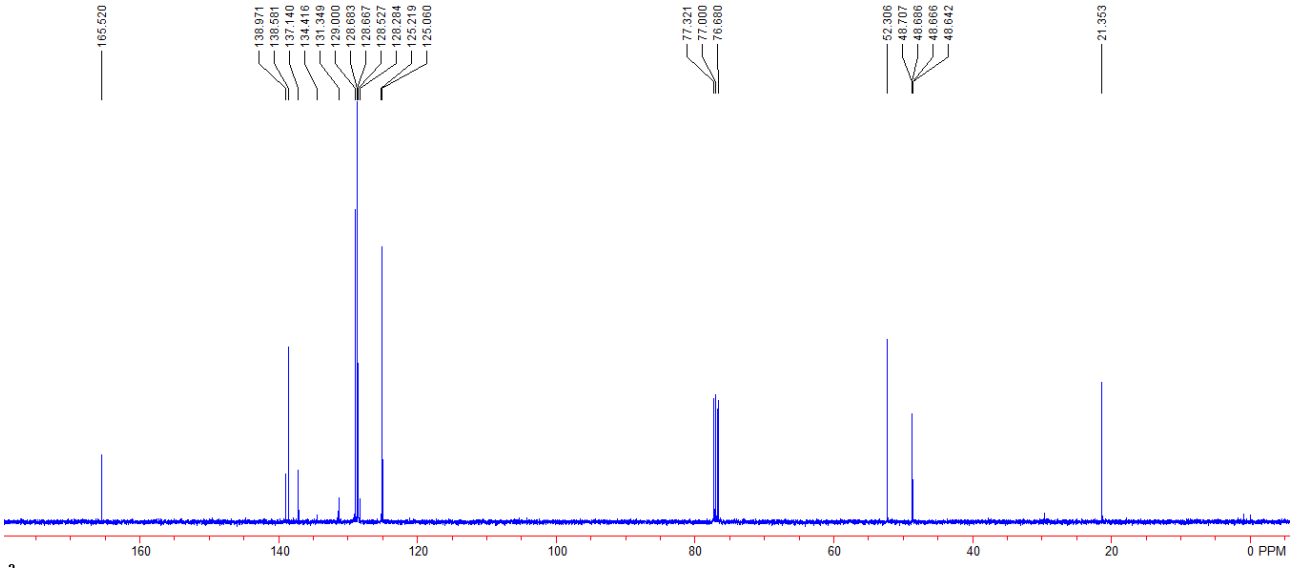
7.225
7.205
7.165
7.150
7.105
7.087
6.556
6.139
5.549
3.735
2.340
0.000
1.90
0.98
0.93
1.03
1.04
0.96
3.00
3.01
8
7
6
5
4
3
2
1
0
PPM


165.520
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138.581
137.140
134.416
131.349
129.000
128.683
128.667
128.527
128.284
125.219
125.000
77.321
77.000
76.680
52.306
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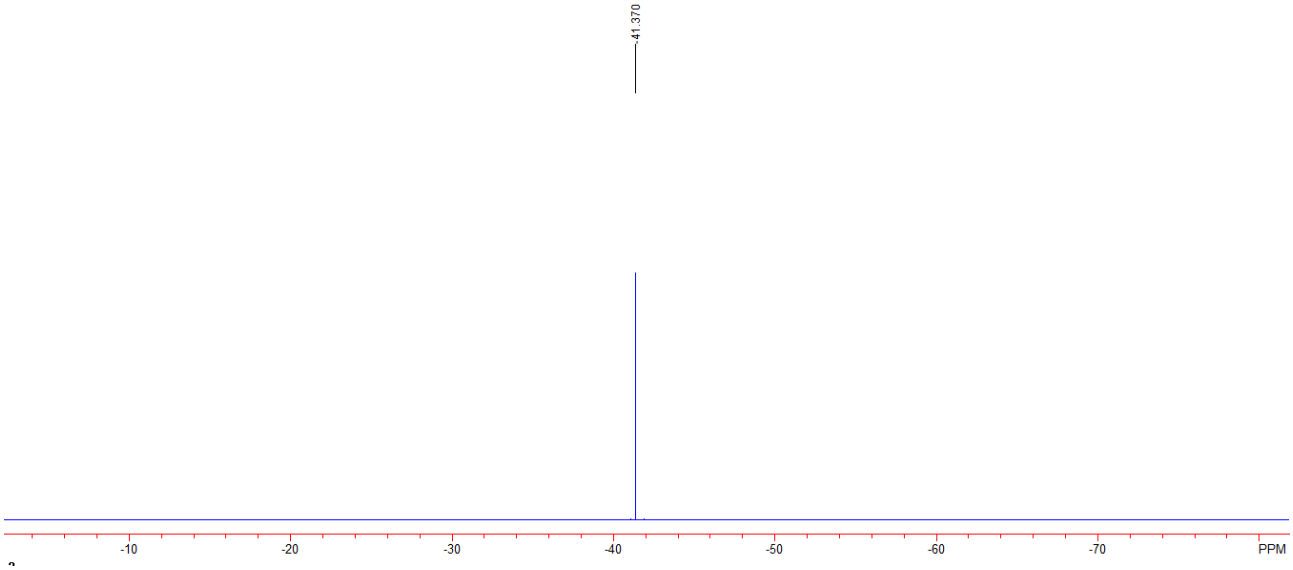
48.686
48.666
48.642
21.353
160
140
120
100
80
60
40
20
0 PPM


41.370
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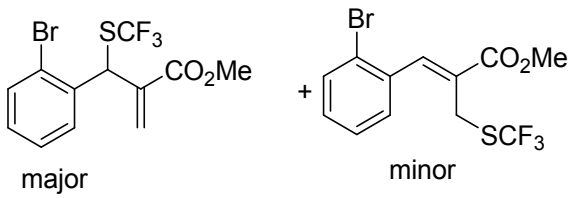
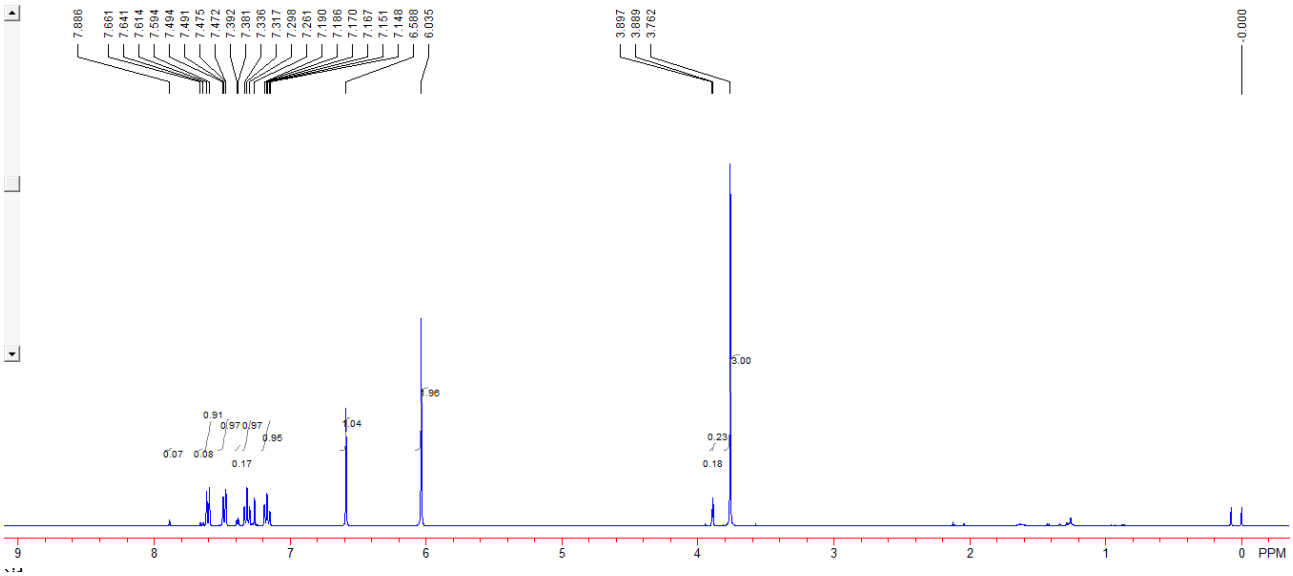
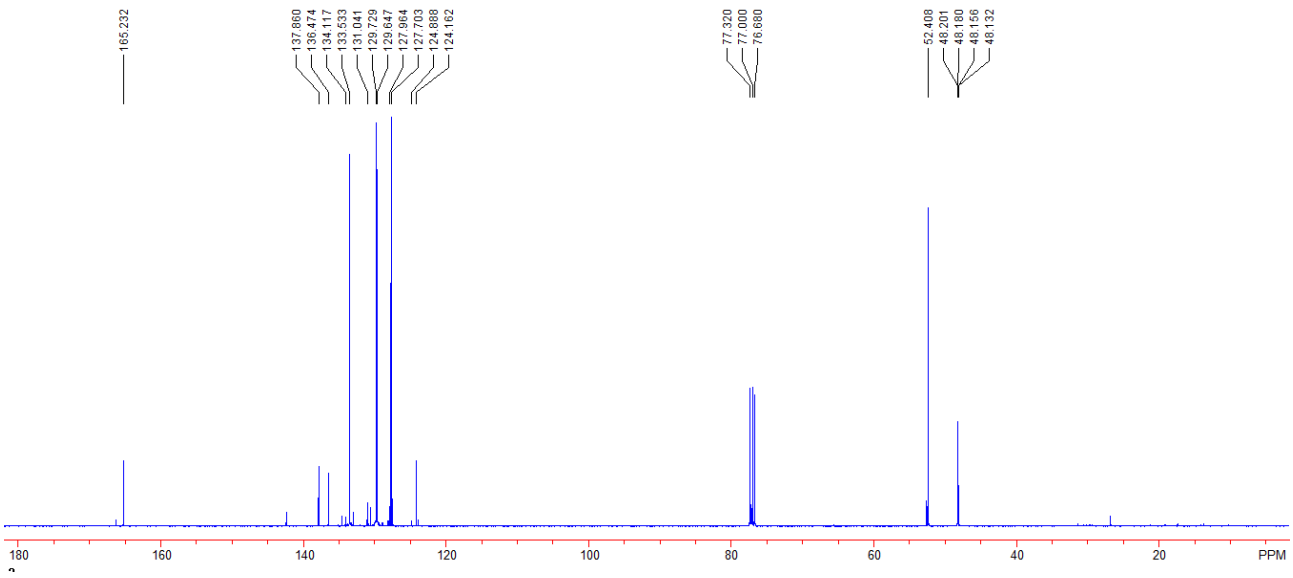
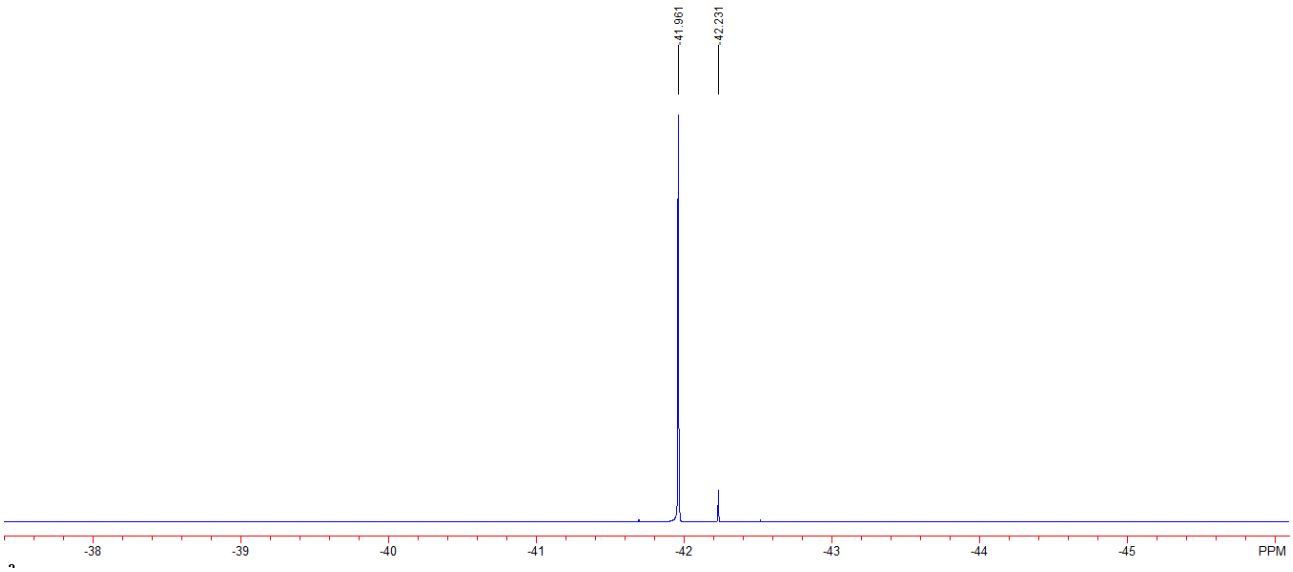
-30
-40
-50
-60
-70
PPM

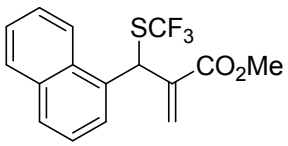


Compound 4i: Yield: 56 mg, 79%. A colourless oil. **4i**:**3i** = 13.0:1. IR (neat) ν 3008, 2988, 2955, 1725, 1467, 1440, 1276, 1260, 1108, 764, 750 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.76 (s, 3H), 3.88 (s, 3H, minor isomer), 3.90 (s, 2H, minor isomer), 6.04 (brs, 2H), 6.59 (s, 1H), 7.17 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.32 (t, *J* = 7.6 Hz, 1H), 7.38-7.39 (m, 2H, minor isomer), 7.48 (dd, *J* = 7.6 Hz, 1.2 Hz, 2H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.65 (d, *J* = 8.0 Hz, 1H, minor isomer), 7.89 (s, 1H, minor isomer); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 48.2 (q, *J* = 2.4 Hz), 52.4, 124.2, 127.7, 129.5 (q, *J* = 307.7 Hz), 129.6, 129.7, 133.5, 136.5, 137.9, 165.2; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -42.0 (major isomer), -42.2 (minor isomer); HRMS (EI) Calcd. for $C_{12}H_{10}BrF_3O_2S$ requires (M^+): 353.9537, Found: 353.9531.



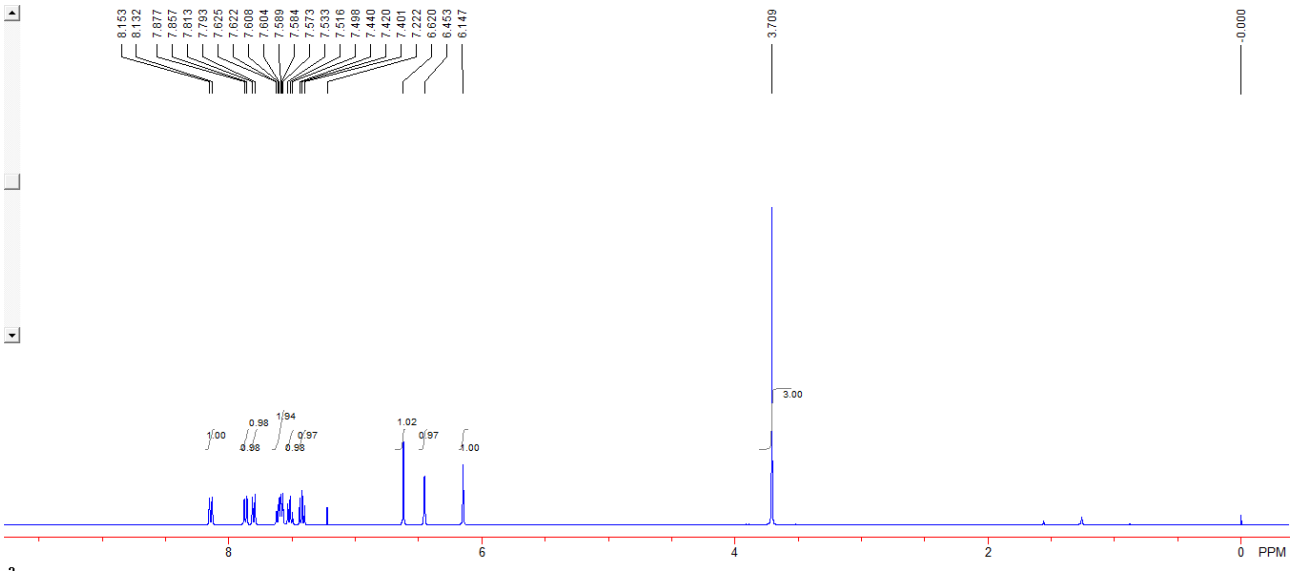


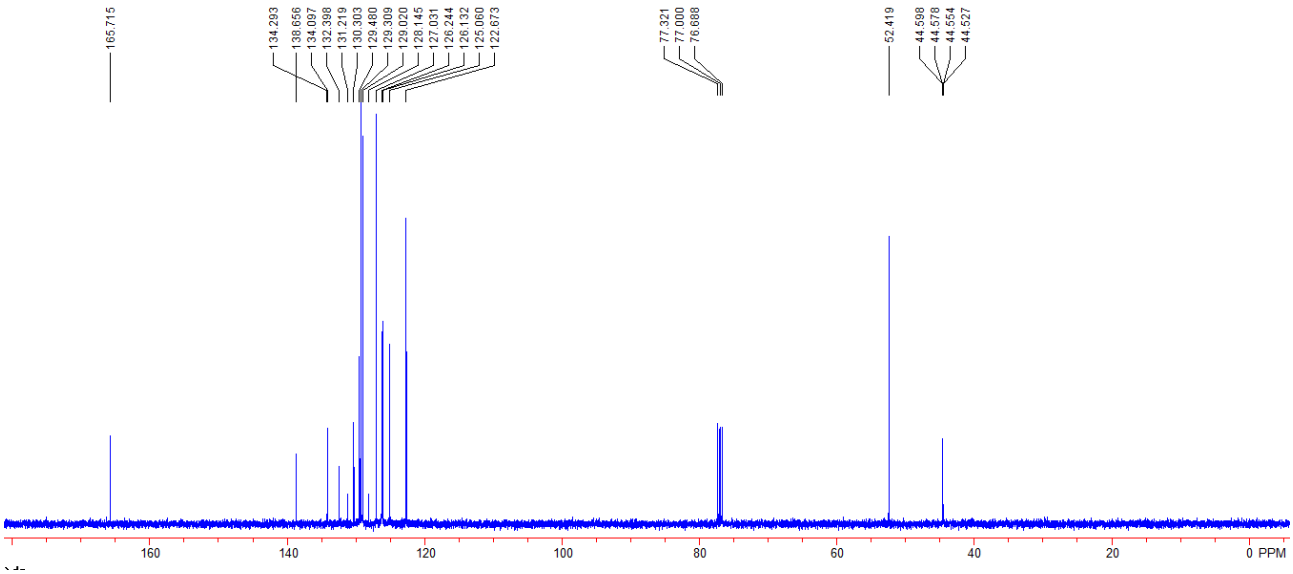


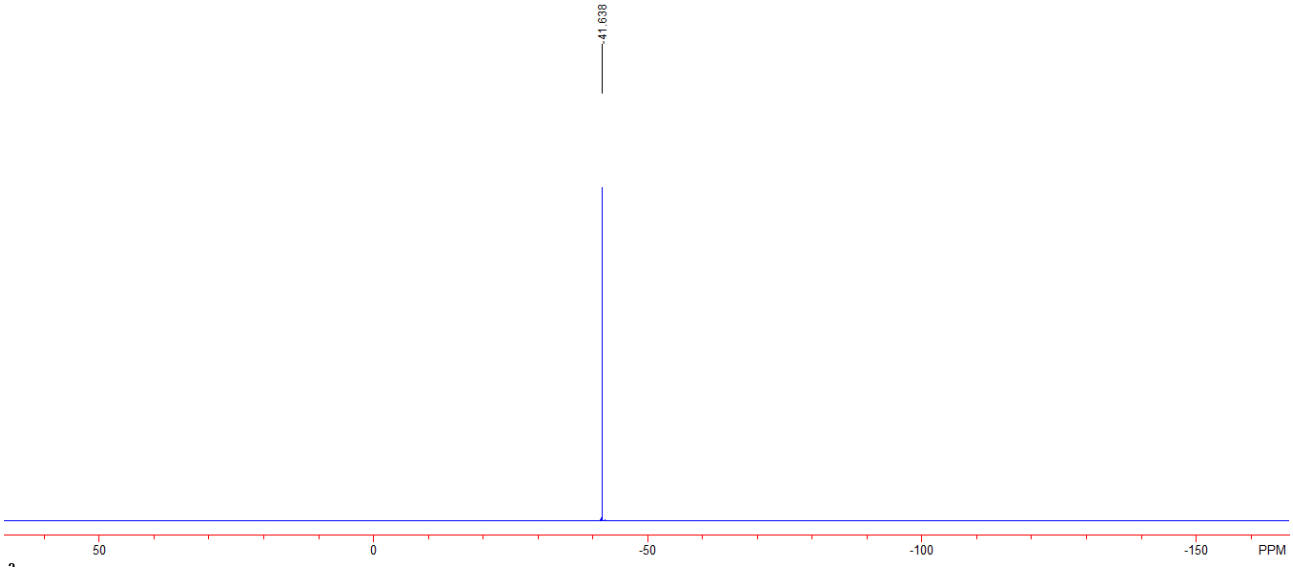


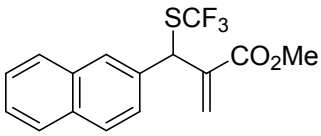
Compound 4j: Yield: 53 mg, 81%. A colourless oil. IR (neat) ν 3061, 3004, 2955, 2854, 1721, 1634, 1511, 1439, 1397, 1327, 1281, 1246, 1106, 962, 802, 778, 755 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.71 (s, 3H), 6.15 (s, 1H), 6.45 (s, 1H), 6.62 (s, 1H), 7.42 (t, *J* = 7.6 Hz, 1H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.57-7.63 (m, 2H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.87 (d, *J* = 8.0 Hz, 1H), 8.14 (d, *J* = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 44.6 (q, *J* = 2.4 Hz), 52.4, 122.7, 125.1, 126.1, 126.2, 127.0, 129.0, 129.3, 129.5, 129.7 (q, *J* = 307.4 Hz), 130.3, 132.4, 134.1, 138.7, 165.7; ^{19}F

NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.6; HRMS (EI) Calcd. for $C_{16}H_{13}F_3O_2S$ requires (M^+): 326.0588, Found: 326.0583.

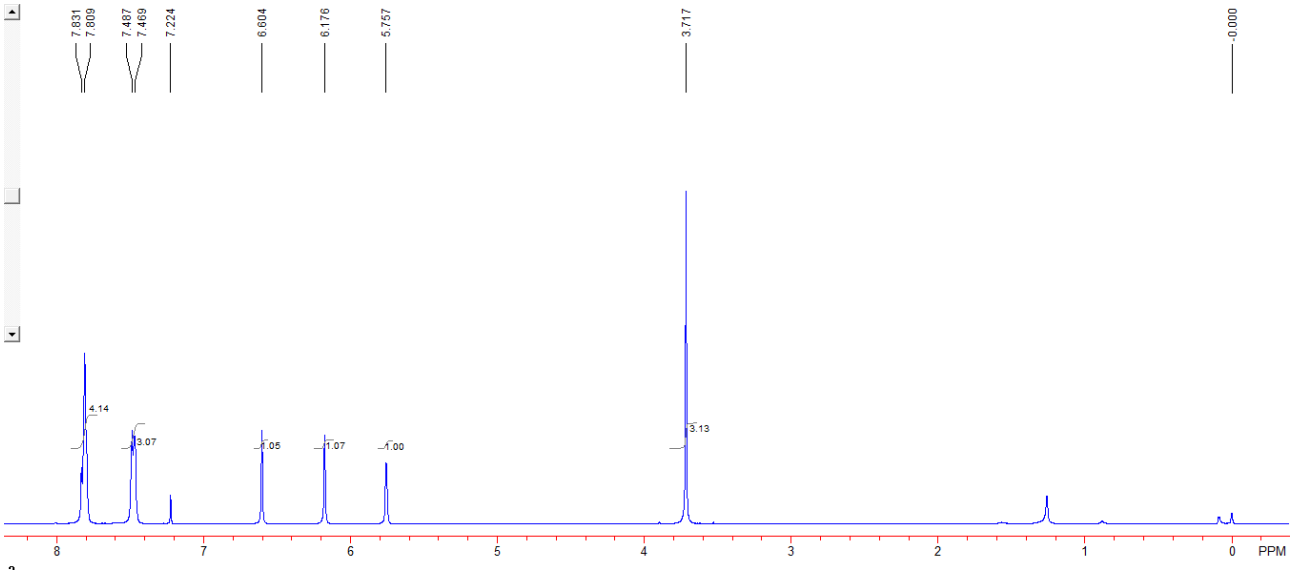


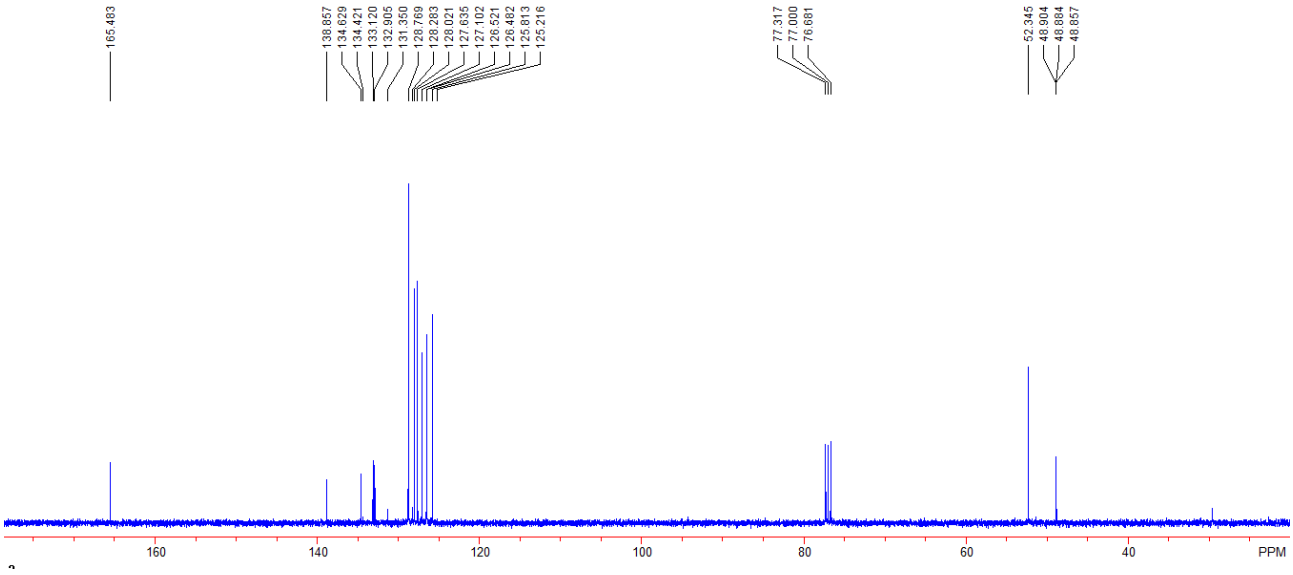


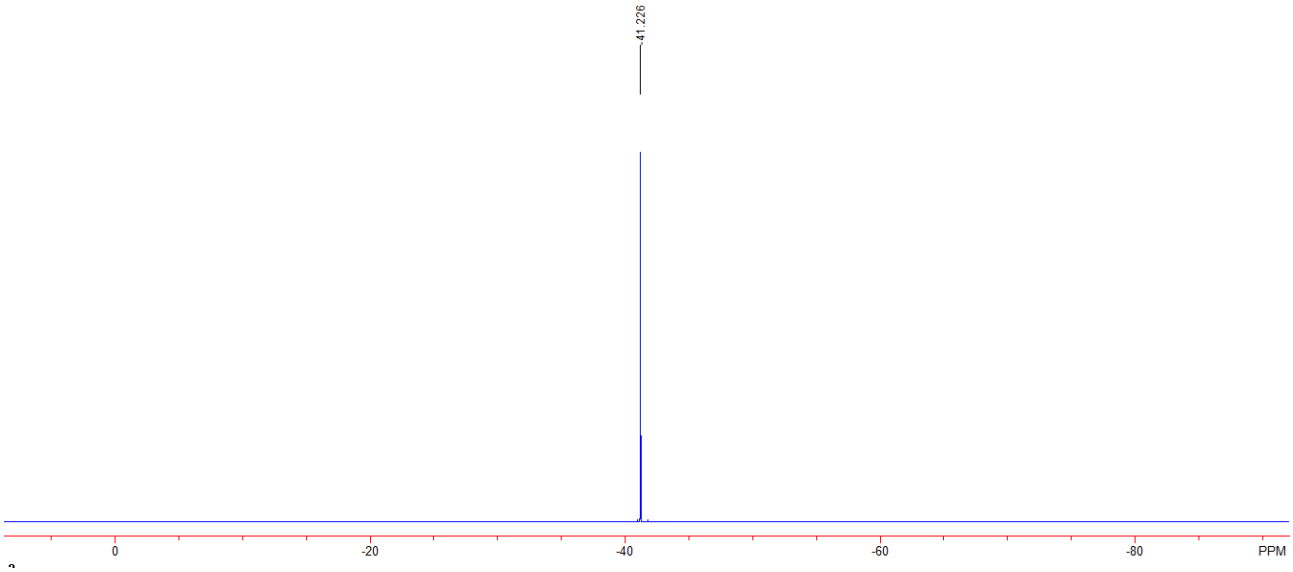


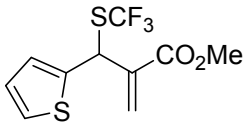


Compound 4k: Yield: 50 mg, 77%. A colourless oil. IR (neat) ν 3061, 3004, 2951, 2923, 1720, 1631, 1508, 1439, 1328, 1275, 1259, 1241, 1102, 962, 861, 815, 752 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.72 (s, 3H), 5.76 (s, 1H), 6.18 (s, 1H), 6.60 (s, 1H), 7.47-7.49 (m, 3H), 7.81-7.83 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 48.9 (q, J = 2.0 Hz), 52.3, 125.8, 126.48, 126.5, 127.1, 127.6, 128.0, 128.8, 129.8 (q, J = 306.7 Hz), 132.9, 133.1, 134.6, 138.9, 165.5; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.2; HRMS (EI) Calcd. for $C_{16}H_{13}F_3O_2S$ requires (M^+): 326.0588, Found: 326.0591.

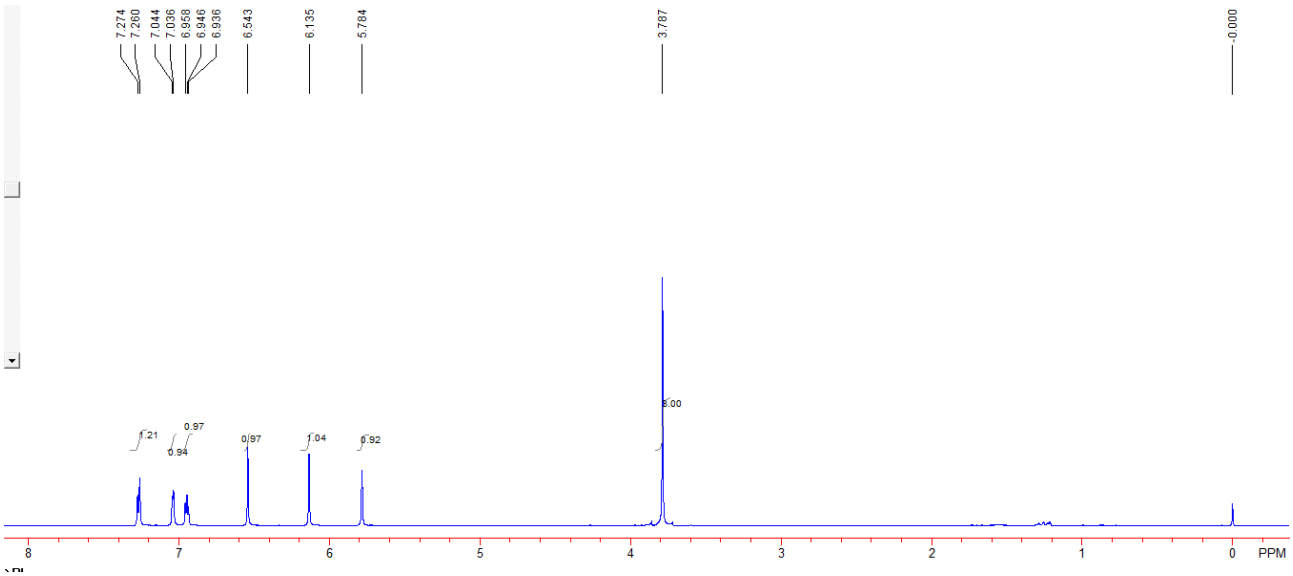








Compound 4l: Yield: 34 mg, 60%. A colourless oil. IR (neat) ν 2955, 2927, 2850, 1724, 1629, 1439, 1332, 1265, 1220, 1110, 702 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.79 (s, 3H), 5.78 (s, 1H), 6.14 (s, 1H), 6.54 (s, 1H), 6.95 (t, J = 4.4 Hz, 1H), 7.04 (d, J = 0.8 Hz, 1H), 7.27 (d, J = 5.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 44.1 (q, J = 2.6 Hz), 52.5, 126.3, 127.06, 127.07, 128.8, 129.8 (q, J = 307.5 Hz), 139.2, 141.0, 165.2; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.6; HRMS (EI) Calcd. for $C_{10}H_9F_3O_2S_2$ requires (M^+): 281.9996, Found: 281.9992.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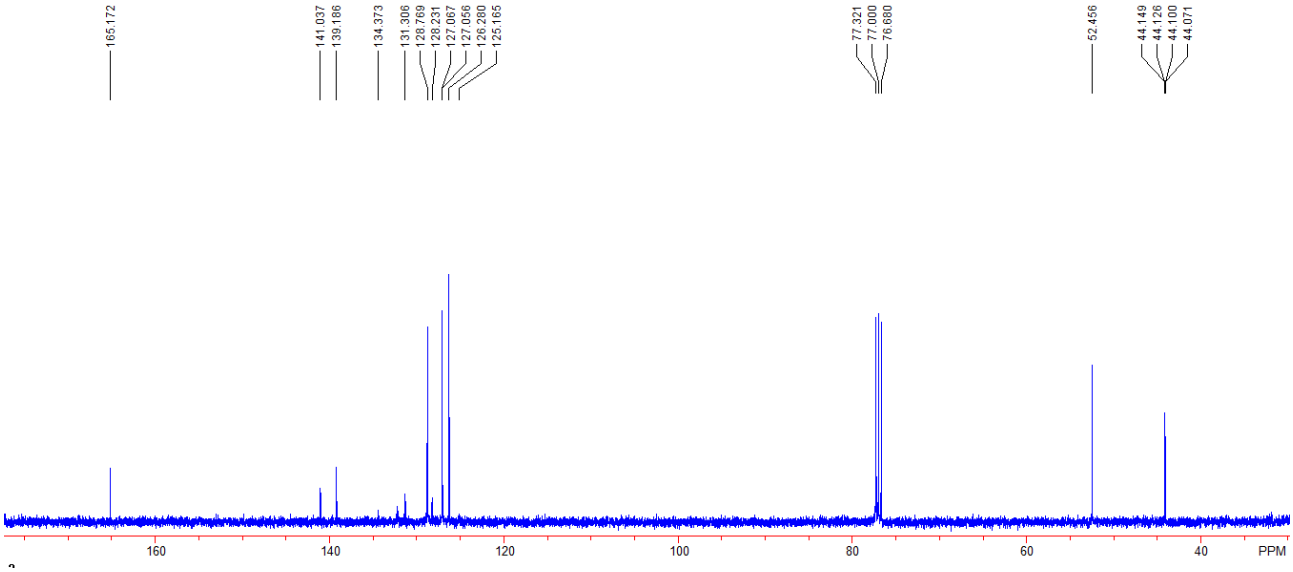
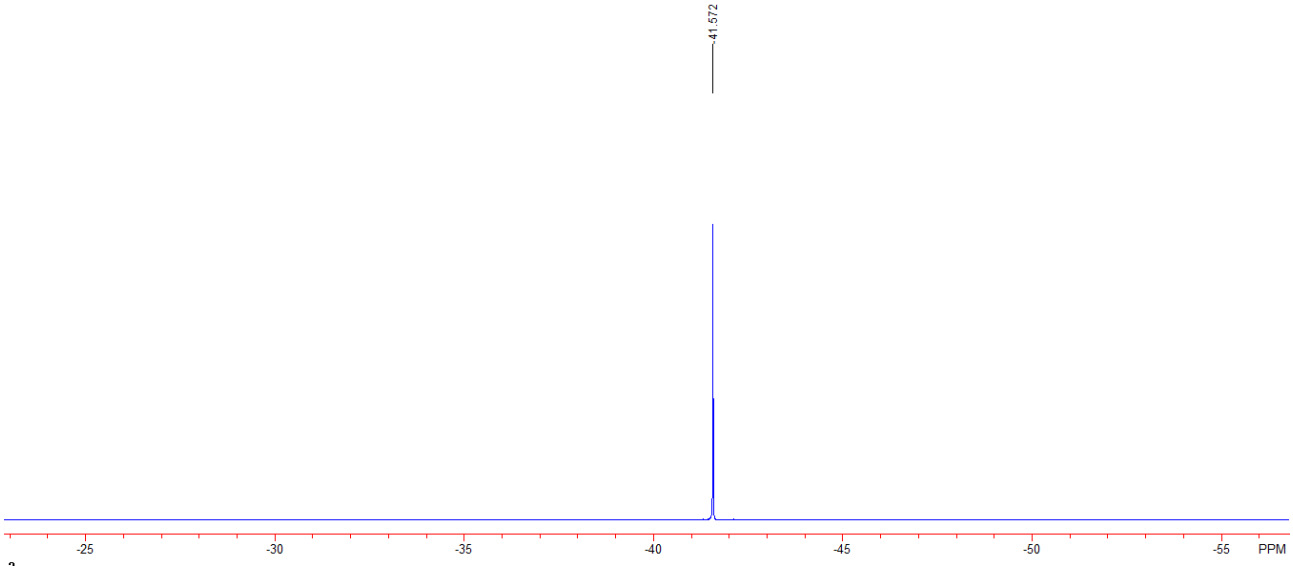
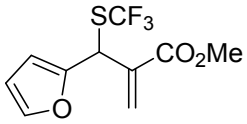
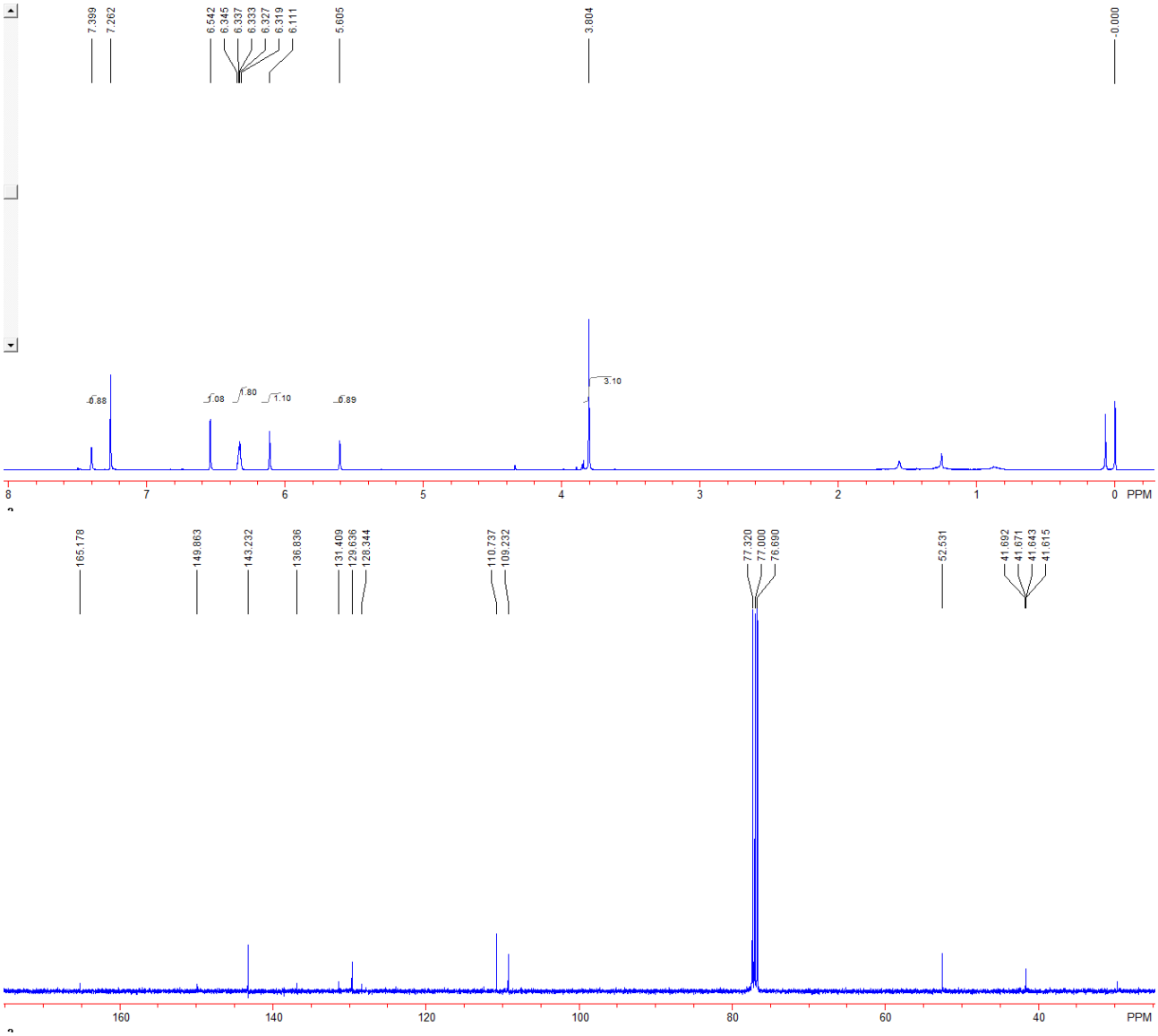
7.260
7.044
7.036
6.958
6.946
6.936
6.543
6.135
5.784
3.787
-0.000
1.21
0.94
0.97
0.97
1.04
0.92
5.00
8
7
6
5
4
3
2
1
0
PPM

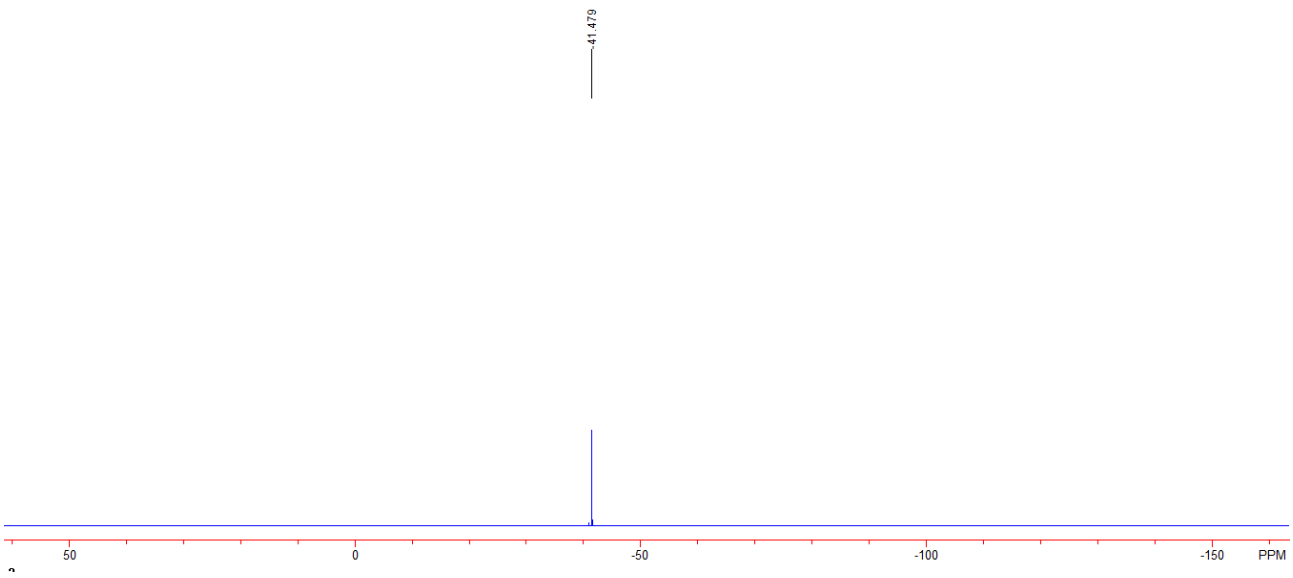

165.172
141.037
139.186
134.373
131.306
128.769
128.231
127.067
127.056
126.280
125.165
77.321
77.000
76.680
52.456
44.149
44.126
44.100
44.071
160
140
120
100
80
60
40
PPM

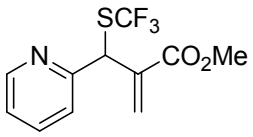

41.572
-25
-30
-35
-40
-45
-50
-55
PPM



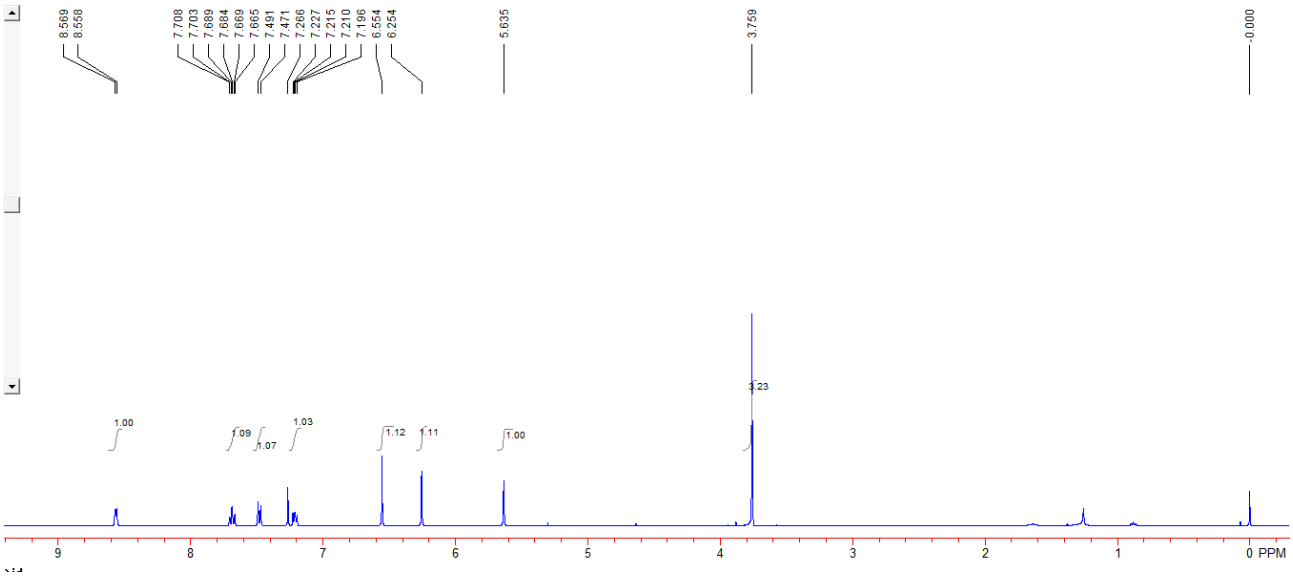
Compound 4m: Yield: 14 mg, 26%. A colourless oil. IR (neat) ν 2955, 2925, 2854, 1727, 1439, 1261, 1214, 1112, 1014, 955, 817, 737 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.80 (s, 3H), 5.61 (s, 1H), 6.11 (s, 1H), 6.32-6.35 (m, 2H), 6.54 (s, 1H), 7.40 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 41.7 (q, J = 2.8 Hz), 52.5, 109.2, 110.7, 129.6, 129.9 (q, J = 306.5 Hz), 136.8, 143.2, 149.9, 165.2; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.5; HRMS (EI) Calcd. for $C_{10}H_9F_3O_3S$ requires (M^+): 266.0225, Found: 266.0230.







Compound 4n: Yield: 28 mg, 51%. A colourless oil. IR (neat) ν 2960, 2926, 2850, 1720, 1588, 1470, 1436, 1312, 1240, 1112, 996, 749 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 3.76 (s, 3H), 5.64 (s, 1H), 6.25 (s, 1H), 6.55 (s, 1H), 7.21 (dd, *J* = 6.8 Hz, 4.8 Hz, 1H), 7.48 (d, *J* = 8.0 Hz, 1H), 7.69 (td, *J* = 7.6 Hz, 2.0 Hz, 1H), 8.56 (d, *J* = 4.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 49.9 (q, *J* = 2.6 Hz), 52.3, 122.6, 122.8, 129.6, 130.3 (q, *J* = 305.5 Hz), 137.0, 138.4, 149.6, 157.1, 165.6; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.4; HRMS (EI) Calcd. for $C_{11}H_{10}F_3NO_2S$ requires (M^+): 277.0384, Found: 277.0380.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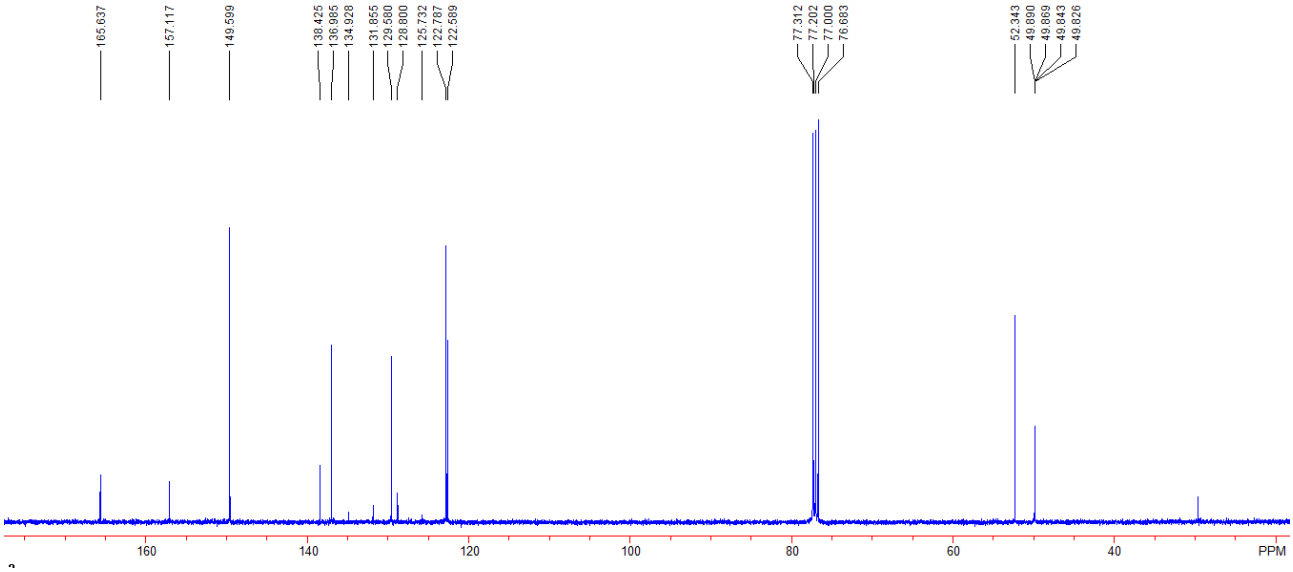
7.708
7.703
7.689
7.684
7.669
7.665
7.491
7.471
7.266
7.227
7.215
7.210
7.196
6.554
6.254
5.635
3.759
-0.000
1.00
1.09
1.07
1.03
1.12
1.11
1.00
3.23
9
8
7
6
5
4
3
2
1
0 PPM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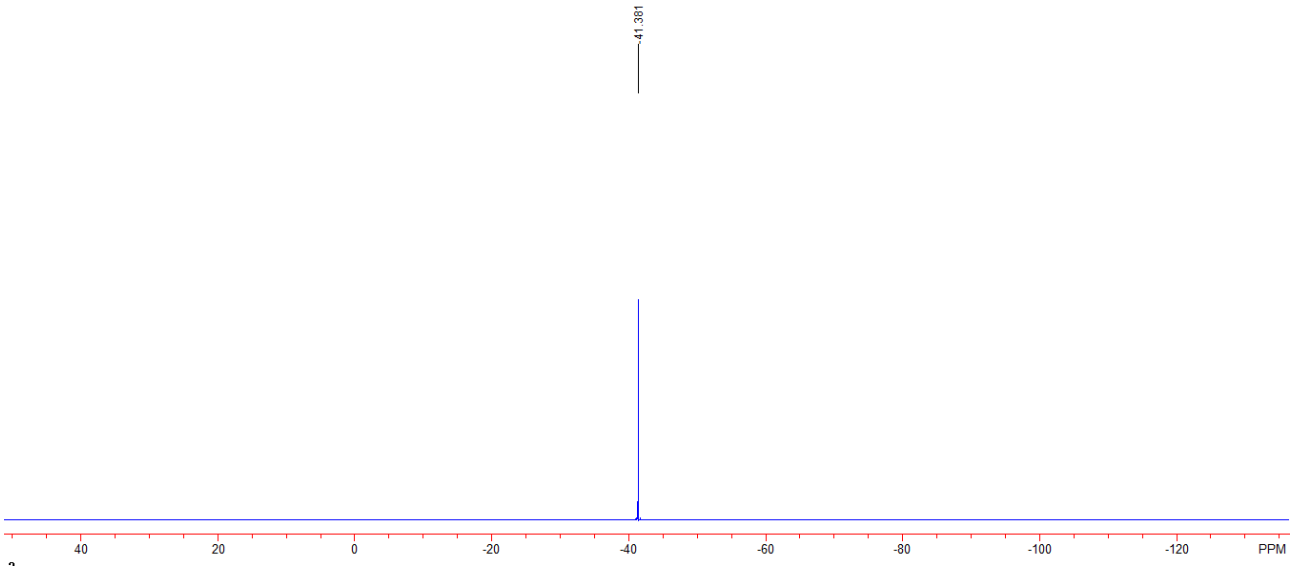
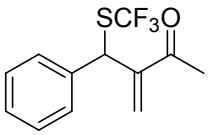
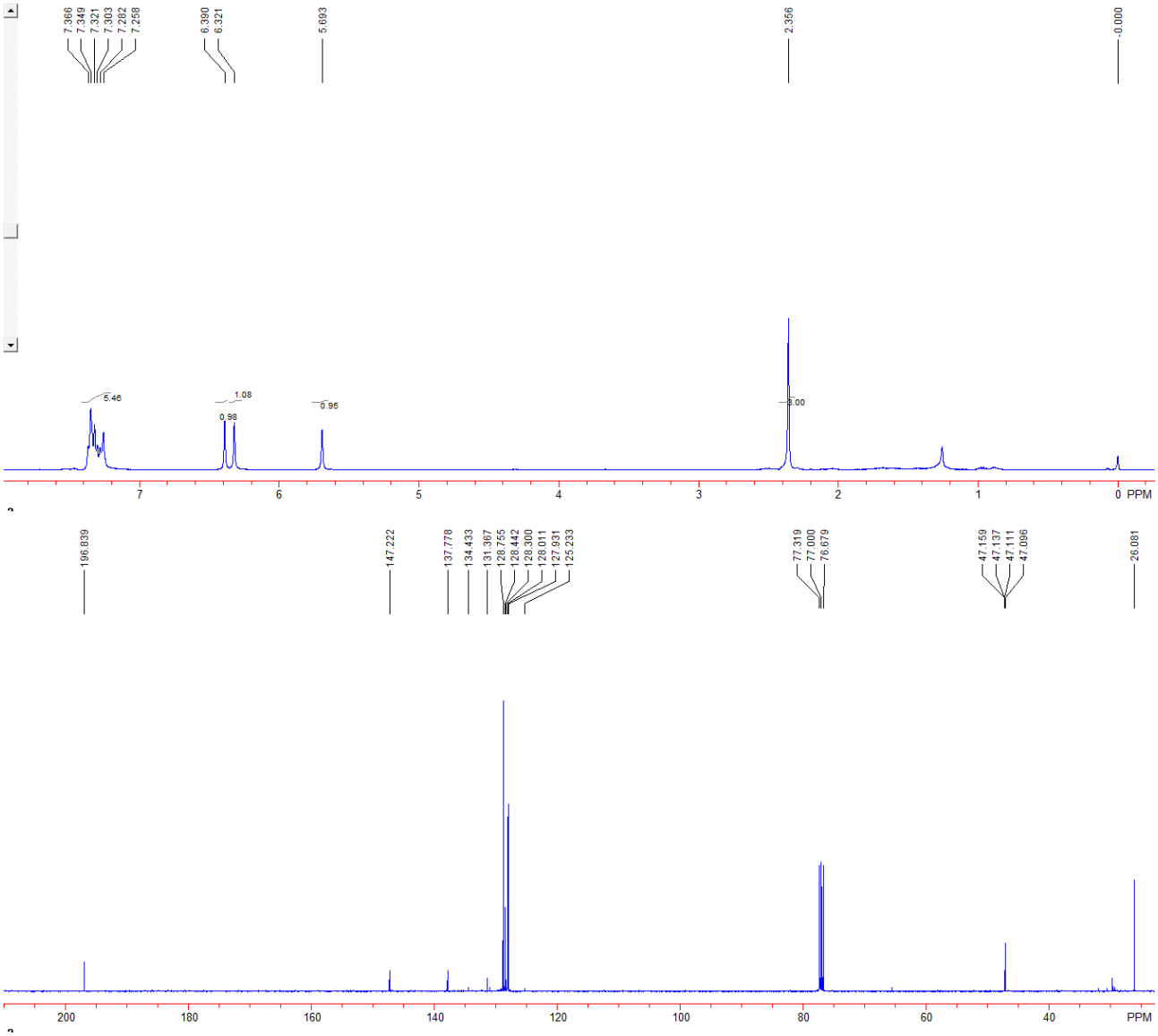
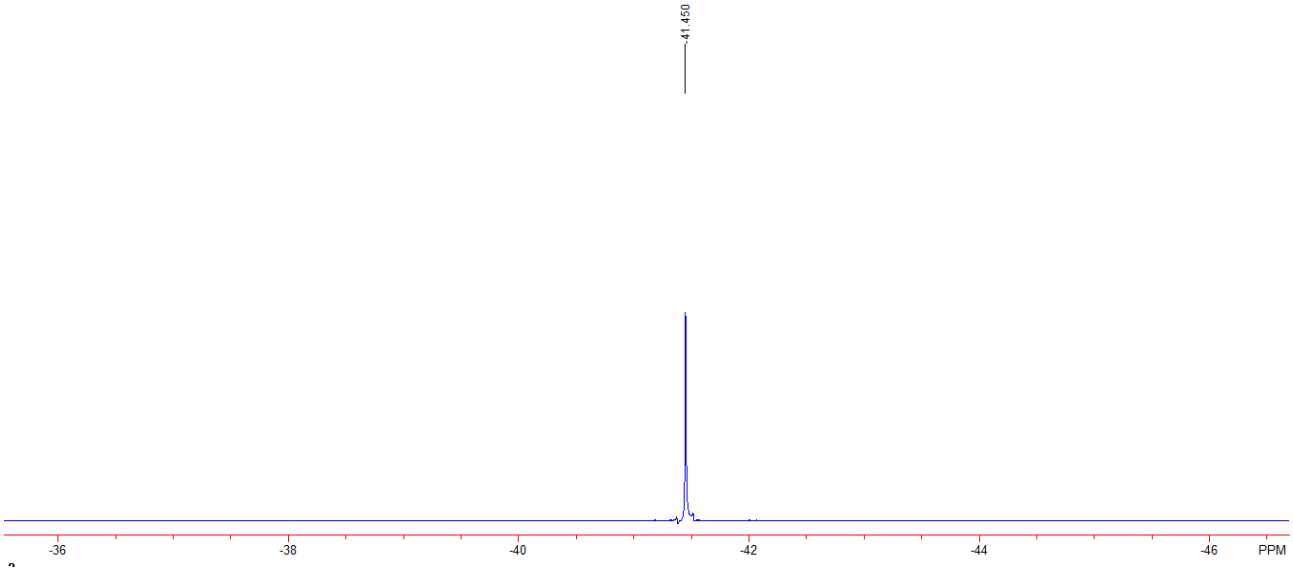
157.117
149.599
138.425
136.985
134.928
131.855
129.580
128.800
125.732
122.787
122.589
77.312
77.202
77.000
76.683
52.343
49.690
49.869
49.843
49.826
160
140
120
100
80
60
40
PPM

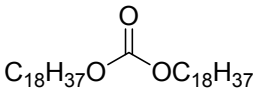

41.381
40
20
0
-20
-40
-60
-80
-100
-120
PPM



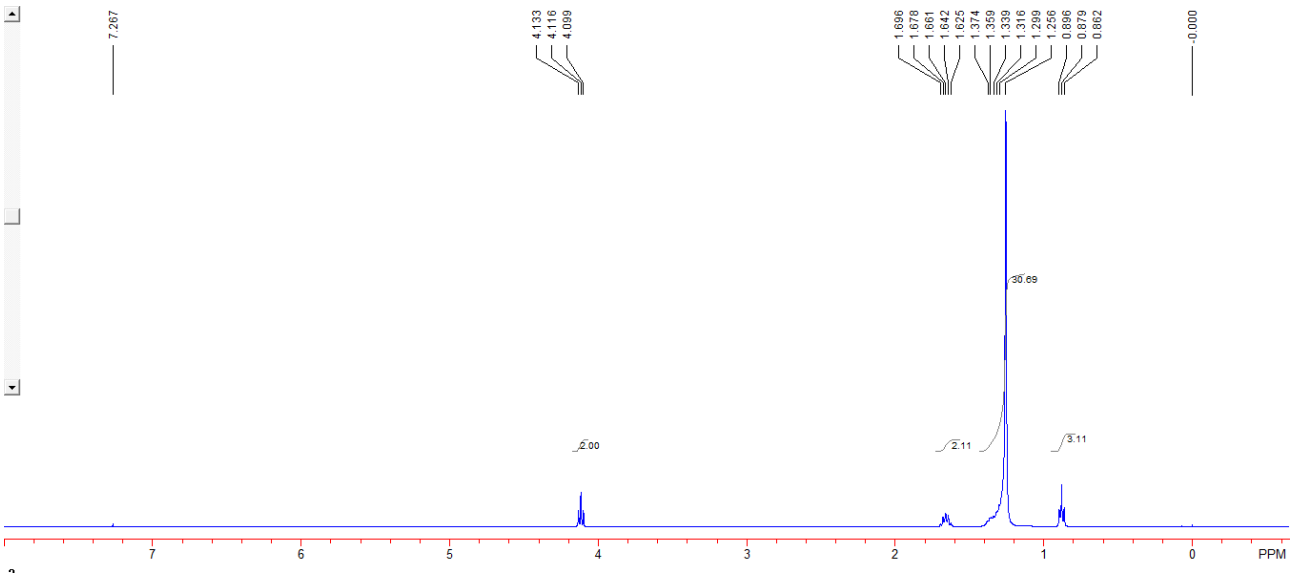
Compound 4o: Yield: 19 mg, 37%. A colourless oil. IR (neat) ν 2964, 2927, 2850, 1682, 1366, 1275, 1260, 1114, 954, 764, 750, 697 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 2.36 (s, 3H), 5.69 (s, 1H), 6.32 (s, 1H), 6.39 (s, 1H), 7.26-7.37 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 26.1, 47.1 (q, J = 2.6 Hz), 127.9, 128.0, 128.4, 128.8, 129.8 (q, J = 306.7 Hz), 137.8, 147.2, 196.8; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -41.5; HRMS (EI) Calcd. for $C_{12}H_{11}F_3OS$ requires (M^+): 260.0483, Found: 260.0479.

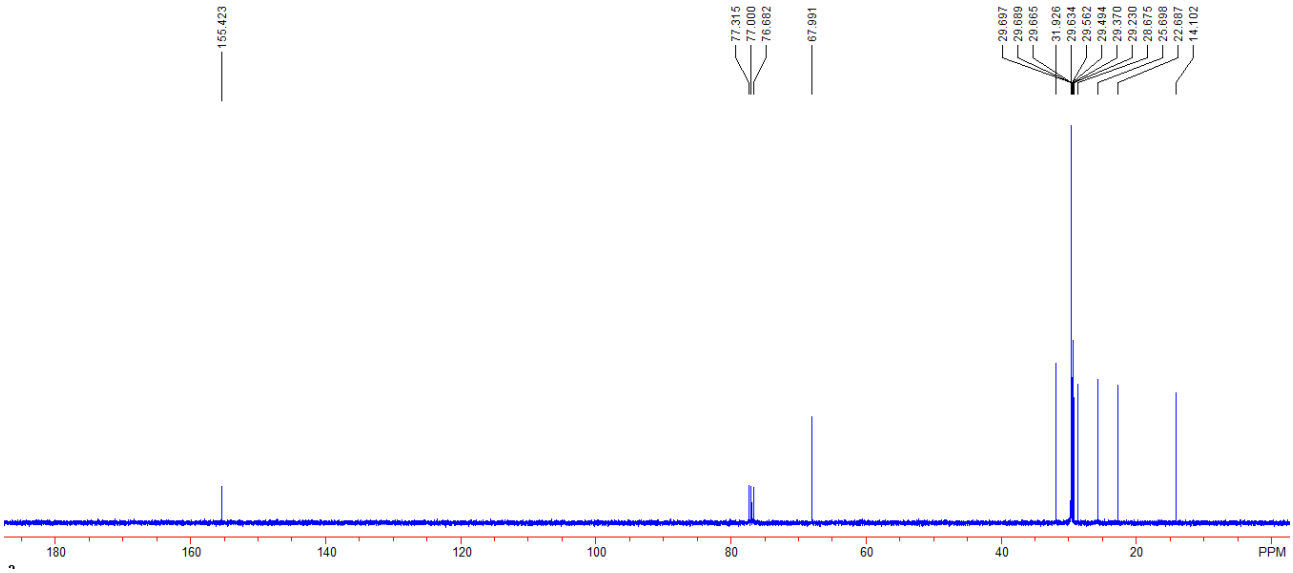


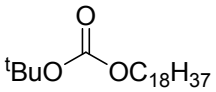




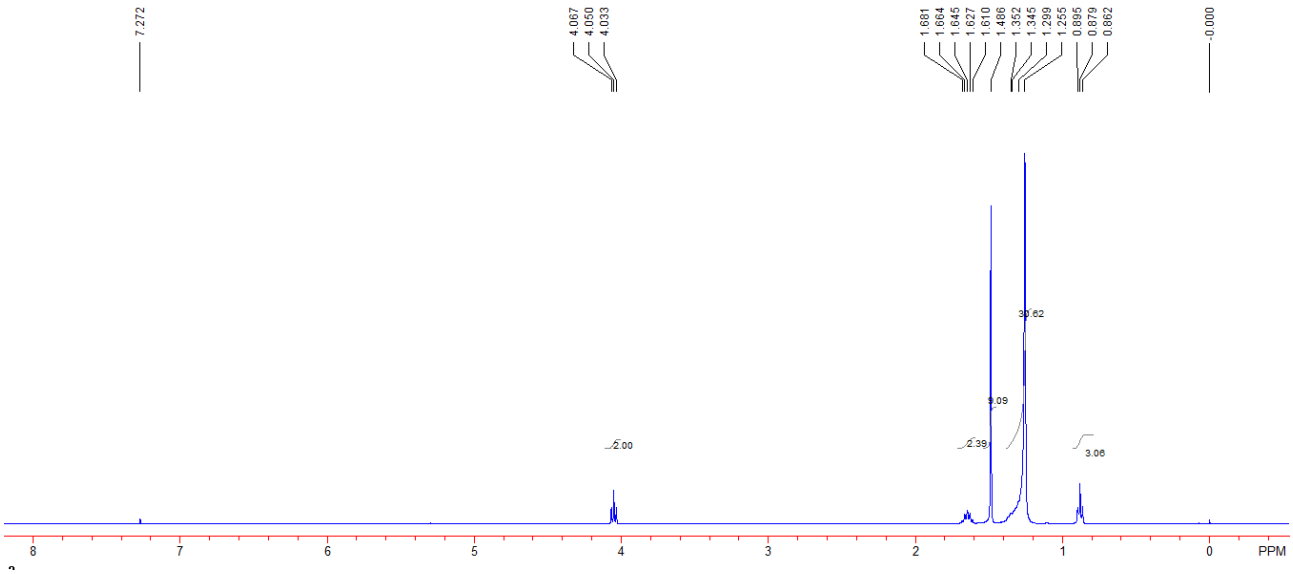
Compound 5a: A white solid. ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 0.88 (t, J = 6.8 Hz, 3H), 1.26-1.37 (m, 30H), 1.66 (quint, J = 6.8 Hz, 2H), 4.12 (t, J = 6.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 14.1, 22.7, 25.7, 28.7, 29.2, 29.4, 29.5, 29.56, 29.63, 29.67, 29.69, 29.70, 31.9, 68.0, 155.4; HRMS (MALDI) Calcd. for $C_{37}H_{74}O_3$ requires (M^++H): 567.5711, Found: 567.5705.

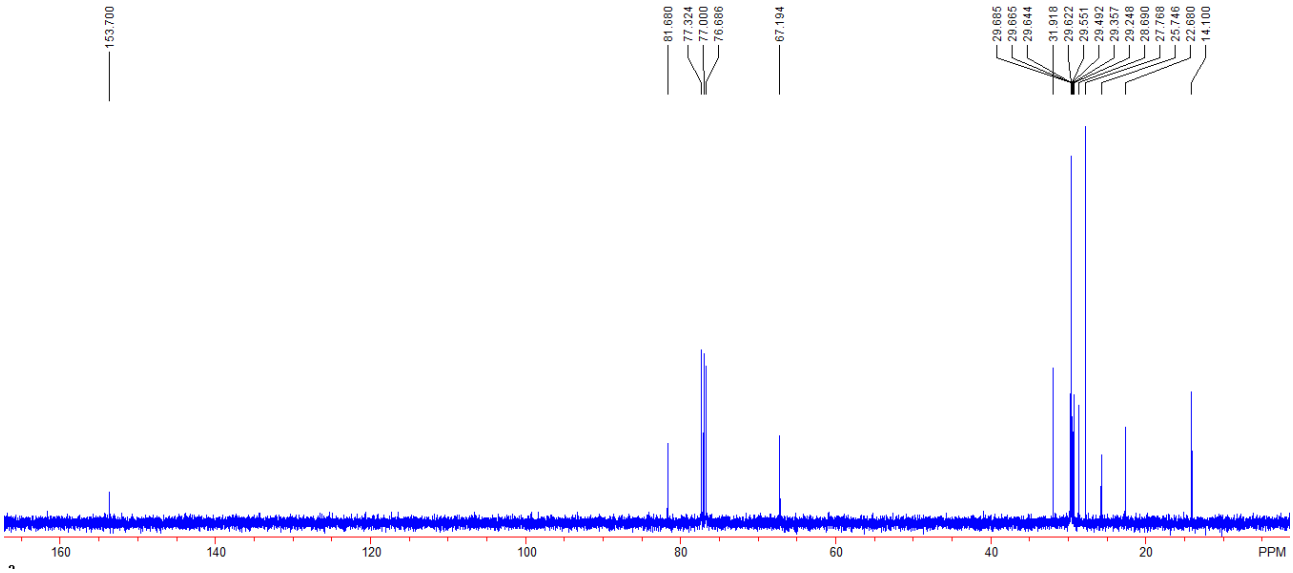




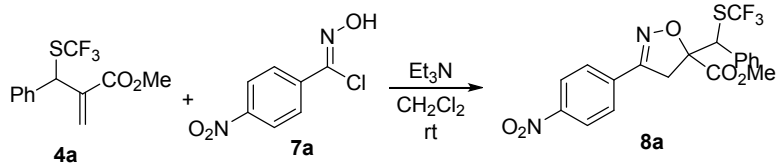


Compound 6a: white solid. ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 0.88 (t, *J* = 6.8 Hz, 3H), 1.26-1.35 (m, 30H), 1.49 (s, 9H), 1.65 (quint, *J* = 6.8 Hz, 2H), 4.05 (t, *J* = 6.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 14.1, 22.7, 25.7, 27.8, 28.7, 29.2, 29.4, 29.5, 29.55, 29.62, 29.64, 29.67, 29.69, 31.9, 67.2, 81.7, 153.7; HRMS (ESI) Calcd. for $C_{23}H_{46}O_3$ requires (M^++NH_4): 388.3785, Found: 388.3787.

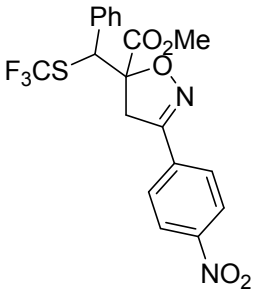




Procedure for the conversion of product 4a.

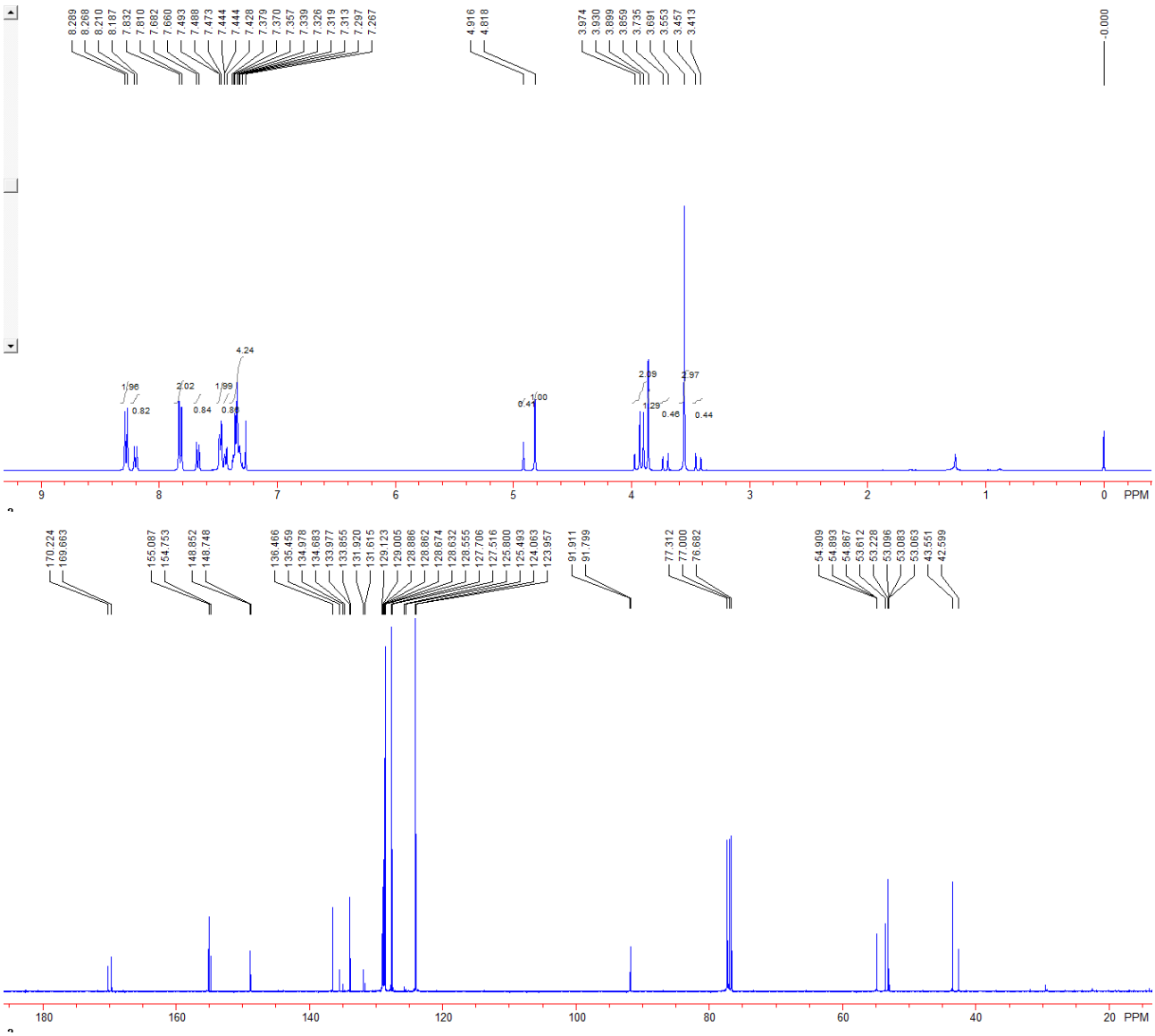


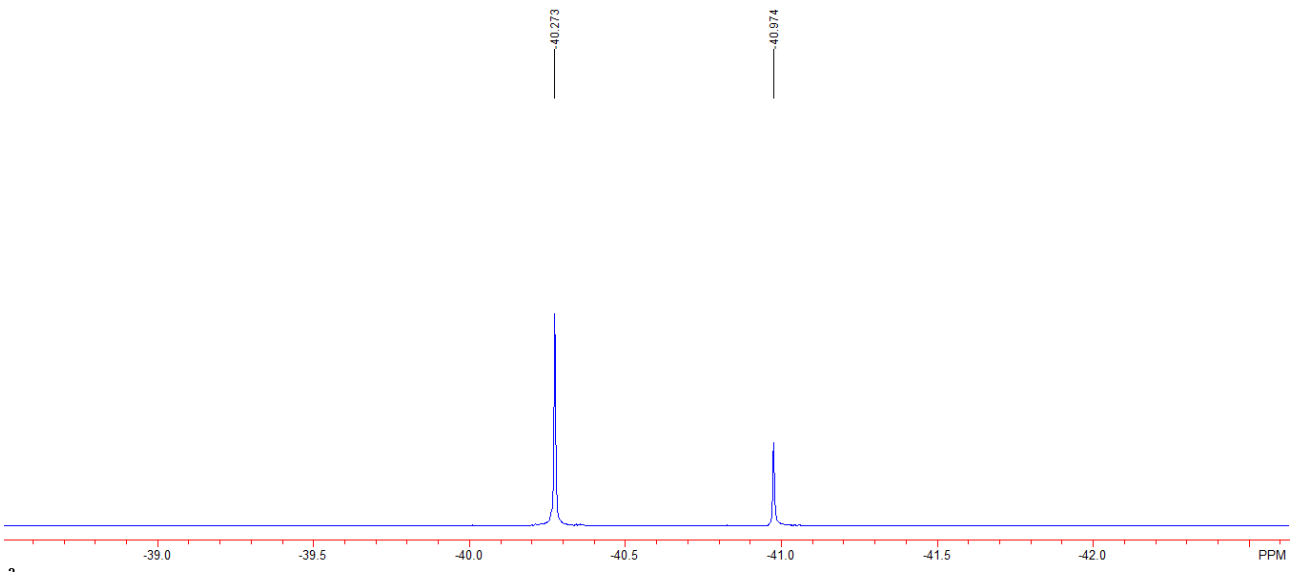
Compound **4a** (55 mg, 0.2 mmol), *N*-hydroxy-4-nitrobenzimidoyl chloride **7a** (60 mg, 0.3 mmol), and Et_3N (42 μL, 0.3 mmol) were stirred in 2 mL of CH_2Cl_2 at rt. The solvent was removed under reduced pressure and the residue was purified by column chromatography (silica gel, ethyl acetate: petroleum ether = 1: 5) to give compound **8a** (46 mg, 52%).



Compound 8a: Yield: 46 mg, 52%. A white solid. m. p.: 148-152 °C. IR (neat) ν 2955, 2923, 2850, 1762, 1739, 1599, 1584, 1521, 1436, 1344, 1275, 1260, 1153, 1107, 1081, 914, 850, 825, 765, 751, 699 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, TMS) δ 3.44 (d, J = 17.6 Hz, 1H, minor isomer), 3.55 (s, 3H), 3.71 (d, J = 17.6 Hz, 1H, minor isomer),

3.86 (s, 3H, minor isomer), 3.88 (d, J = 17.6 Hz, 1H), 3.95 (d, J = 17.6 Hz, 1H), 4.82 (s, 1H), 4.92 (s, 1H, minor isomer), 7.30-7.38 (m, 3H), 7.43-7.44 (m, 2H, minor isomer), 7.47-7.49 (m, 2H), 7.67 (d, J = 8.8 Hz, 2H, minor isomer), 7.82 (d, J = 8.8 Hz, 2H), 8.20 (d, J = 9.2 Hz, 2H, minor isomer), 8.28 (d, J = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 42.6, 43.6, 53.1 (q, J = 2.0 Hz), 53.2, 53.6, 54.9 (q, J = 2.0 Hz), 91.8, 91.9, 124.0, 124.1, 127.5, 127.7, 128.6, 128.7, 128.86, 128.89, 129.0, 129.1, 130.1 (q, J = 306.0 Hz), 130.4 (q, J = 305.8 Hz), 133.9, 134.0, 135.5, 136.5, 148.7, 148.9, 154.8, 155.1, 169.7, 170.2; ^{19}F NMR (376 MHz, $CDCl_3$, $CFCl_3$) δ -40.3 (major isomer), -41.0 (minor isomer); HRMS (MALDI) Calcd. for $C_{19}H_{16}F_3N_2O_5S$ requires (M^++H): 441.0732, Found: 441.0718.

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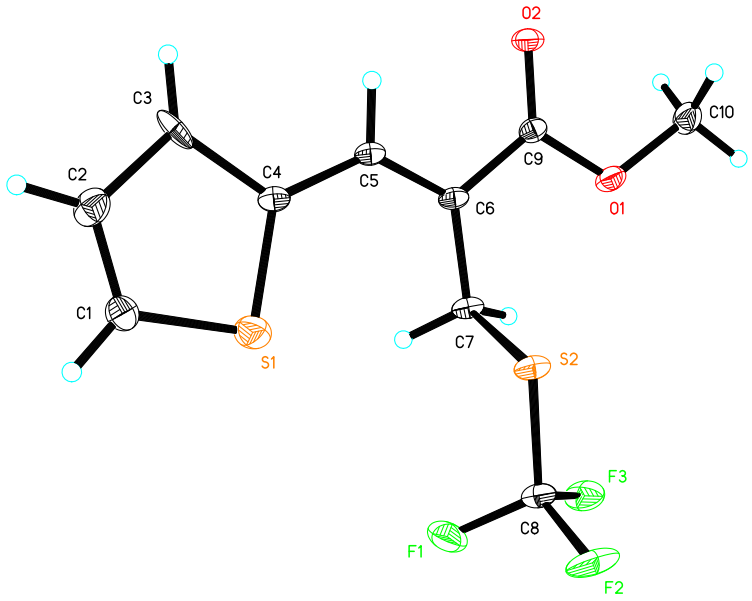
-41.0
-41.5
-42.0
PPM



The crystal data of **3l** have been deposited in CCDC with number 1048652. Empirical Formula: $C_{10}H_9F_3O_2S_2$; Formula Weight: 282.29; Crystal Dimensions: 0.156 x 0.123 x 0.086 mm; Crystal System: Monoclinic; Lattice Parameters: a = 9.4647(7)Å, b = 5.7312(5)Å, c = 22.1678(18)Å, α = 90°, β = 100.2830(10)°, γ = 90°, V = 1183.16(17)Å^3; Space group: P 1 21/n 1; Z = 4; D_{calc} = 1.585 g/cm^3; F_{000} = 576; Final R indices [I>2sigma(I)] R1 = 0.0645, wR2 = 0.1972.