Supporting Information

Transition Metal-Free Synthesis of Polyfluoroaryl Sulfides via *S*-Transfer Reaction

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Table of Contents

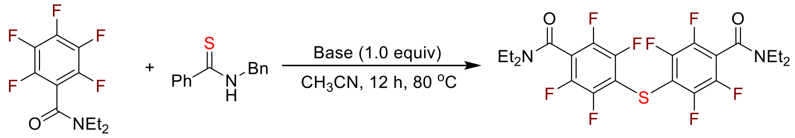
1. General Information

All reactions were monitored by thin layer chromatography (TLC) using Macherey-Nagel 0.20 mm silica gel 60 plates. Flash column chromatography was performed on silica gel 60 (particle size 300-400 mesh ASTM, purchased from Taizhou, China). ^{1}H, ^{13}C spectra were recorded with, Bruker 400 MHz (Avance-400) instrument. All ^{1}H NMR data are reported in δ units, parts per million (ppm), and were measured relative to the residual proton signal in the deuterated solvent at 7.26 ppm ($CDCl_3$). All ^{13}C NMR spectra are decoupled and reported in ppm relative to the solvent signal at 77.16 ppm ($CDCl_3$). High-resolution mass spectra HRMS (ESI-TOF) were recorded on Brucker microtof. Compounds were visualized by irradiation with UV light, or stained with iodine/silica gel, or potassium permanganate. Preparatory thin-layer chromatography (Prep-TLC) was performed on silica gel GF with UV 254 (20 × 20 cm, 1000 microns, from Yantai Jiang you Silica Gel Development Co., Ltd.) and visualized with UV light.

Materials. Reaction solvents THF and toluene were distilled over sodium and stored under nitrogen atmosphere. While DCM, DCE and CH_3CN was distilled over CaH_2 and stored under nitrogen atmosphere. Compounds **1a-1i** were known and prepared according to the previously reported procedures[1]. All other commercial reagents and solvents were purchased from Energy-Chemical Ltd, and used as received unless otherwise noted.

2. Detailed Optimization Studies

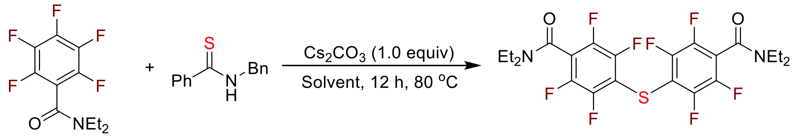
Table S1. Screening of base.[a]



Entry	Base	Yield 4a (%)[b]
1	Cs_2CO_3	36
2	K_2CO_3	22
3	$KHCO_3$	trace
4	Na_2CO_3	trace
5	^{t}BuOK	28
6	NaOH	25
7	$^{i}Pr_2EtN$	n.d.
8	DMAP	trace
9	Et_3N	n.d.

[a]Reaction conditions: **1a** (0.2 mmol), **2** (0.10 mmol), base (1.0 equiv), solvent (0.2 M), 12 h. [b] ^{19}F NMR yields using fluorobenzene as internal standard.

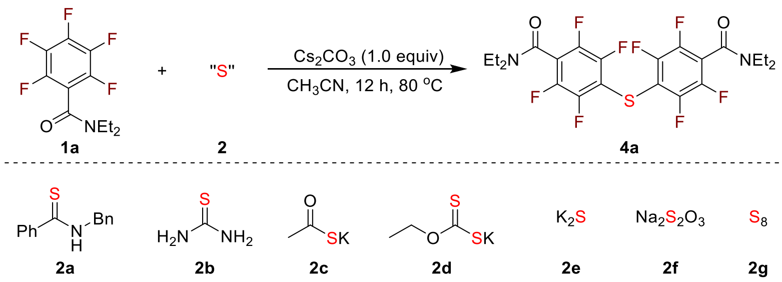
Table S2. Screening of solvent.[a]



Entry	Solvent	Yield 4a (%)[b]
1	Toluene	n.d.
2	DCE	n.d.
3	CCl_4	n.d.
4	DMF	34
5	DMSO	30
6	CH_3CN	36

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.10 mmol), Cs_2CO_3 (1.0 equiv), solvent (0.2 M), 12 h. [b] ^{19}F NMR yields using fluorobenzene as internal standard.

Table S3. Screening of sulfur source.[a]

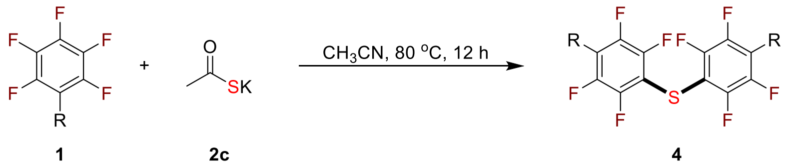


Entry	2	Yield 4a (%)[b]
1	**2a**	36
2	**2b**	43
3[c]	**2c**	65
4[c,d]	**2d**	32
5[c]	**2e**	38
6[c]	**2f**	trace
7[c]	**2g**	n.d.
8[c,e]	**2c**	91

[a] Reaction conditions: **1a** (0.2 mmol), **2** (0.10 mmol), Cs_2CO_3 (1.0 equiv), CH_3CN (0.2 M), 12 h. [b] ^{19}F NMR yields using fluorobenzene as internal standard. [c] Without any base. [d] Since potassium ethyl xanthogenate **2d** is sensitive to light the experiments were carried out under dark conditions. [e] **2c** (0.15 mmol).

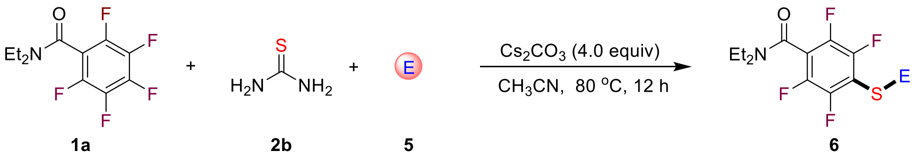
3. Experimental Procedures

a) General Procedure for 4



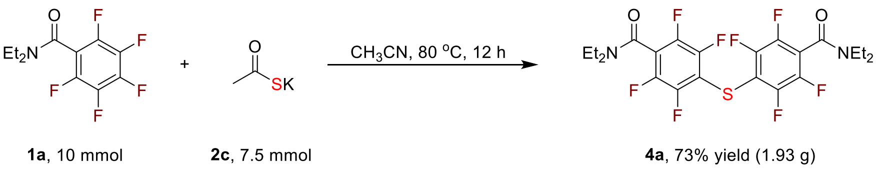
Added polyfluoroarene **1** (0.2 mmol), potassium thioacetate **2c** (17.1 mg, 0.15 mmol, 0.75 equiv) in 1.0 mL of acetonitrile and the mixture was allowed to stir for 12 h at 80 ℃. Until the reaction was complete as indicated by TLC. The reaction mixture was then quenched with H_2O, extracted with CH_2Cl_2 (3×10 mL) and the combined organic layers were concentrated in vacuo. The resulting crude product was purified by flash column chromatography on silica gel to obtain product **4**.

b) General Procedure for 6

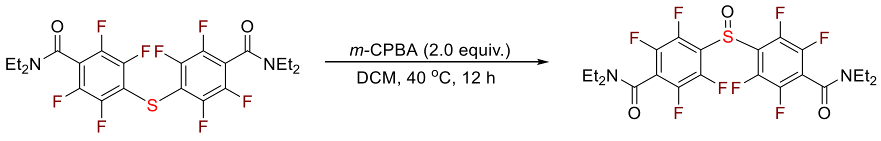


Added *N*,*N*-diethyl-2,3,4,5,6-pentafluorobenzamide **1a** (53.4 mg, 0.2 mmol, 1.0 equiv), thiourea **2b** (22.8 mg, 0.30 mmol, 1.5 equiv), electroophiles **5** (0.8 mmol, 4.0 equiv) and Cs_2CO_3 (260 mg, 0.8 mmol, 4.0 equiv) in 2.0 mL of acetonitrile and the mixture was allowed to stir for 12 h at 80 ℃. Until the reaction was complete as indicated by TLC. The reaction mixture was then quenched with H_2O, extracted with CH_2Cl_2 (3×10 mL) and the combined organic layers were concentrated in vacuo. The resulting crude product was purified by flash column chromatography on silica gel to obtain product **6**.

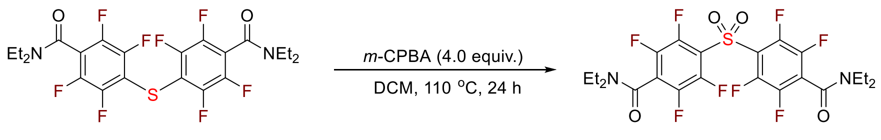
c) A Gram scale synthesis and synthetic applications



Added *N*,*N*-diethyl-2,3,4,5,6-pentafluorobenzamide **1a** (2.67g, 10 mmol, 1.0 equiv), and potassium thioacetate **2c** (0.86 g, 7.5 mmol, 0.75 equiv) in 30 mL of acetonitrile, The solution was warmed up to 80 ℃ and it was stirred for 12 hours, until the reaction was complete as indicated by TLC. The reaction mixture was then quenched with H_2O, extracted with CH_2Cl_2 (3×30 mL) and the combined organic layers were concentrated in vacuo. The resulting crude product was purified by flash column chromatography on silica gel to obtain product **4a** (1.93 g, 73%).



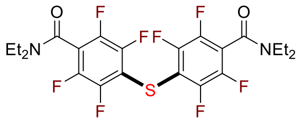
Added **4a** (105.6 mg, 0.2 mmol) and *m*-CPBA (68.8 mg, 0.4 mmol, 2.0 equiv) in 2 mL of DCM and the mixture was allowed to stir for 12 h at 40 ℃, until the reaction was complete as indicated by TLC. The reaction mixture was quenched with saturated aq. Na_2SO_3, extracted with CH_2Cl_2 (3×10 mL) and the combined organic layers were concentrated in vacuo. The resulting crude product was purified by flash column chromatography on silica gel to obtain product **7** (94.7 mg, 87%).



A heavy wall pressure vessel was charged with pentafluorophenyl sulfide (105.6 mg, 0.2 mmol, 1.00 equiv.) and *m*-CPBA (137.6 mg, 0.8 mmol, 4.0 equiv). The mixture was suspended in 2 mL of dichloromethane and the tube was sealed with a Teflon cap and the mixture was allowed to stir for 24 h at 110 ℃, until the reaction was complete as indicated by TLC. The reaction mixture was quenched with saturated aq. Na_2SO_3, extracted with CH_2Cl_2 (3×10 mL) and the combined organic layers were concentrated in vacuo. The resulting crude product was purified by flash column chromatography on silica gel to obtain product **8** (72.8 mg, 65%).

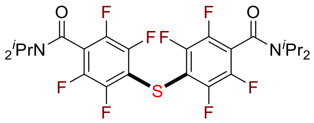
4. Analytical data of New Compounds

4,4'-Thiobis(*N,N*-diethyl-2,3,5,6-tetrafluorobenzamide) (4a)



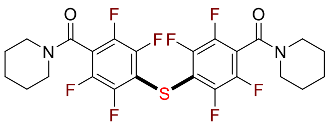
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 10:1 to afford **4a** as colorless oil (96.1 mg, 91% yield), TLC: R_f = 0.25 (Petroleum ether : Ethyl acetate = 10:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.60 (q, *J* = 7.2 Hz, 4H), 3.24 (q, *J* = 7.2 Hz, 4H), 1.27 (t, *J* = 7.2 Hz, 6H), 1.16 (t, *J* = 7.2 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.0, 147.0 (dm, *J* = 251.0 Hz), 142.5 (dm, *J* = 238.0 Hz), 118.9 (t, *J* = 22.0 Hz), 111.7 (t, *J* = 22.0 Hz), 43.4, 40.0, 14.3, 12.8. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -130.66 – -130.92 (m, 4F), -140.17 – -140.40 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{22}H_{21}F_8N_2O_2S$ ($[M + H]^+$), 529.1191, found, 529.1180.

4,4'-Thiobis(2,3,5,6-tetrafluoro-*N,N*-diisopropylbenzamide) (4b)



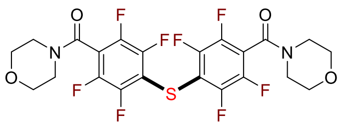
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 10:1 to afford **4b** as colorless oil (85.3 mg, 73% yield), TLC: R_f = 0.26 (Petroleum ether : Ethyl acetate = 10:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.73 – 3.64 (m, 2H), 3.61 – 3.52 (m, 2H), 1.54 (d, *J* = 6.8 Hz, 12H), 1.21 (d, *J* = 6.4 Hz, 12H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.4, 147.1 (dm, *J* = 253.0 Hz), 142.4 (dm, *J* = 242.0 Hz), 120.1 (t, *J* = 46.0 Hz), 111.7 (t, *J* = 46.0 Hz), 52.1, 47.0, 21.0, 20.3. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -130.68 – -130.83 (m, 4F), -141.32 – -141.50 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{26}H_{29}F_8N_2O_2S$ ($[M + H]^+$), 585.1817, found, 585.1813.

(Thiobis(2,3,5,6-tetrafluoro-4,1-phenylene))bis(piperidin-1-ylmethanone) (4c)



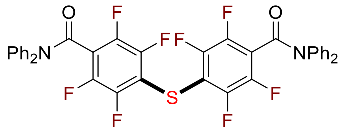
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 15:1 to afford **4c** as colorless oil (88.3 mg, 80% yield), TLC: R_f = 0.17 (Petroleum ether : Ethyl acetate = 15:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.75 (t, *J* = 5.2 Hz, 4H), 3.34 – 3.24 (m, 4H), 1.74 – 1.55 (m, 12H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 156.9, 147.0 (dm, *J* = 254.0 Hz), 142.5 (dm, *J* = 250.0 Hz), 118.4 (t, *J* = 22.0 Hz), 111.7 (t, *J* = 22.0 Hz), 48.3, 43.3, 26.6, 25.5, 24.4. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -130.79 – -131.01 (m, 4F), -139.79 – -139.96 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{24}H_{21}F_8N_2O_2S$ ($[M + H]^+$), 553.1191, found, 553.1190.

(Thiobis(2,3,5,6-tetrafluoro-4,1-phenylene))bis(morpholinomethanone) (4d)



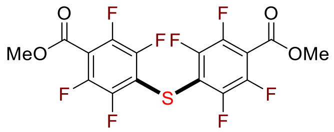
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 10:1 to afford **4d** as colorless oil (85.6 mg, 77% yield), TLC: R_f = 0.14 (Petroleum ether : Ethyl acetate = 10:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.86 – 3.77 (m, 8H), 3.72 – 3.67 (m, 4H), 3.35 (t, *J* = 5.2 Hz, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.2, 147.0 (dm, *J* = 253.0 Hz), 142.6 (dm, *J* = 240.0 Hz), 117.3 (t, *J* = 22.0 Hz), 112.4 (t, *J* = 20.0 Hz), 66.9, 66.7, 47.4, 42.8. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -130.36 – -130.48 (m, 4F), -139.22 – -19.47 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{22}H_{17}F_8N_2O_4S$ ($[M + H]^+$), 557.0776, found, 557.0769.

4,4'-Thiobis(2,3,5,6-tetrafluoro-*N*,*N*-diphenylbenzamide) (4e)



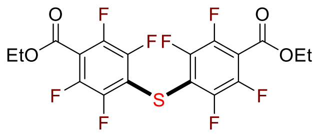
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 15:1 to afford **4e** as colorless oil (129.6 mg, 90% yield), TLC: R_f = 0.13 (Petroleum ether : Ethyl acetate = 15:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43 – 7.37 (m, 8H), 7.31 – 7.20 (m, 12H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.5, 146.0 (dm, *J* = 253.0 Hz), 142.0 (dm, *J* = 244.0 Hz), 119.4 (t, *J* = 21.0 Hz), 112.2 (t, *J* = 20.0 Hz), 126.0, 119.6, 119.4, 112.4, 112.2, 112.0. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -132.27 – -132.40 (m, 4F), -139.10 – -139.29 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{38}H_{21}F_8N_2O_2S$ ($[M + H]^+$), 721.1191, found, 721.1187.

Dimethyl 4,4'-thiobis(2,3,5,6-tetrafluorobenzoate) (4f)



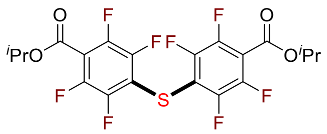
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 15:1 to afford **4f** as colorless oil (82.9 mg, 93% yield), TLC: R_f = 0.16 (Petroleum ether : Ethyl acetate = 15:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.98 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.6, 146.6 (dm, *J* = 253.0 Hz), 144.7 (dm, *J* = 259.0 Hz), 114.2 (t, *J* = 20.0 Hz), 114.1 (t, *J* = 16.0 Hz), 53.6. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -131.90 – -132.08 (m, 4F), -137.63 – -137.79 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{16}H_6F_8O_6S$ ($[M]^+$), 445.9859, found, 445.9866.

Diethyl 4,4'-thiobis(2,3,5,6-tetrafluorobenzoate) (4g)



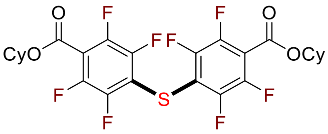
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 15:1 to afford **4g** as colorless oil (76.8 mg, 81% yield), TLC: R_f = 0.14 (Petroleum ether : Ethyl acetate = 15:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.45 (q, *J* = 7.2 Hz, 4H), 1.40 (t, *J* = 7.2 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.1, 146.6 (dm, *J* = 250.5 Hz), 144.6 (dm, *J* = 258.0 Hz), 114.6 (t, *J* = 16.5 Hz), 114.0 (t, *J* = 18.0 Hz), 63.3, 14.2. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -131.87 – -132.08 (m, 4F), -137.85 – -138.13 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{18}H_{10}F_8O_4S$ ($[M]^+$), 474.0172, found, 474.0178.

Diisopropyl 4,4'-thiobis(2,3,5,6-tetrafluorobenzoate) (4h)



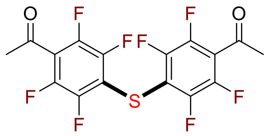
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 15:1 to afford **4h** as colorless oil (89.4 mg, 89% yield), TLC: R_f = 0.15 (Petroleum ether : Ethyl acetate = 15:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.32 – 5.28 (m, 2H), 1.37 (d, *J* = 6.4 Hz, 12H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.6, 146.8 (dm, *J* = 248.0 Hz), 144.4 (dm, *J* = 257.0 Hz), 115.1 (t, *J* = 16.5 Hz), 113.7 (t, *J* = 20.0 Hz), 71.6, 21.8. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -132.02 – -132.19 (m, 4F), -138.42 – -138.58 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{20}H_{14}F_8O_4S$ ($[M]^+$), 502.0485, found, 502.0492.

Dicyclohexyl 4,4'-thiobis(2,3,5,6-tetrafluorobenzoate) (4i)



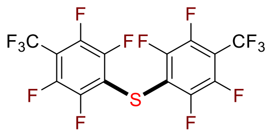
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 15:1 to afford **4i** as colorless oil (84.9 mg, 73% yield), TLC: R_f = 0.17 (Petroleum ether : Ethyl acetate = 15:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.15 – 5.07 (m, 2H), 2.01 – 1.90 (m, 4H), 1.82 – 1.73 (m, 4H), 1.67 – 1.53 (m, 6H), 1.48 – 1.30 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.5, 146.6 (dm, *J* = 250.0 Hz), 144.5 (dm, *J* = 257.5 Hz), 115.1 (t, *J* = 17.0 Hz), 113.7 (t, *J* = 19.5 Hz), 76.1, 31.4, 25.3, 23.4. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -132.04 – -132.26 (m, 4F), -138.29 – -138.47 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{26}H_{22}F_8O_4S$ ($[M]^+$), 582.1111, found, 582.1105.

1,1'-(Thiobis(2,3,5,6-tetrafluoro-4,1-phenylene))bis(ethan-1-one) (4j)



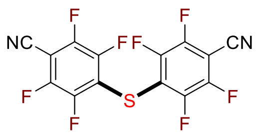
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 15:1 to afford **4j** as colorless oil (51.5 mg, 62% yield), TLC: R_f = 0.15 (Petroleum ether : Ethyl acetate = 15:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.64 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.5, 146.7 (dm, *J* = 250.0 Hz), 143.9 (dm, *J* = 241.0 Hz), 121.0 (t, *J* = 17.5 Hz), 113.8 (t, *J* = 20.0 Hz), 32.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -131.48 – -131.84 (m, 4F), -140.00 – -140.16 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{16}H_5F_8O_2S$ ($[M - H]^-$), 412.9888, found, 412.9886.

Bis(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenyl)sulfane (4k)



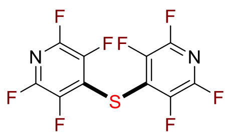
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 50:1 to afford **4k** as colorless oil (55.0 mg, 59% yield), TLC: R_f = 0.14 (Petroleum ether : Ethyl acetate = 50:1) [UV]. **^{13}C NMR** (100 MHz, $CDCl_3$) δ 146.9 (dm, *J* = 250.0 Hz), 144.0 (dm, *J* = 211.0 Hz), 120.6 (d, *J* = 275.0 Hz), 115.4 (t, *J* = 17.5 Hz), 111.3 (t, *J* = 20.0 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -56.48 (t, *J* = 21.8 Hz, 6F), -130.63 – -130.72 (m, 4F), -138.10 – -138.28 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{14}F_{14}S$ ($[M]^+$), 465.9497, found, 465.9496.

4,4'-Thiobis(2,3,5,6-tetrafluorobenzonitrile) (4l)



The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 40:1 to afford **4l** as colorless oil (46.3 mg, 61% yield), TLC: R_f = 0.15 (Petroleum ether : Ethyl acetate = 40:1) [UV]. **^{13}C NMR** (100 MHz, $CDCl_3$) δ 147.2 (dm, *J* = 263.3 Hz), 146.4 (dm, *J* = 250.0 Hz), 117.5 (t, *J* = 19.4 Hz), 106.8, 96.2 (t, *J* = 17.1 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -129.30 – -129.45 (m, 4F), -129.86 – -130.02 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{14}F_8N_2S$ ($[M]^+$), 379.9654, found, 379.9658.

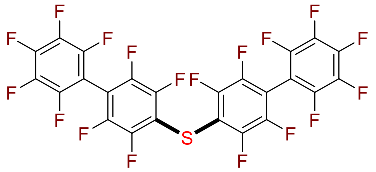
Bis(perfluoropyridin-4-yl)sulfane (4m)



The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 30:1 to afford **4m** as colorless oil (29.9 mg, 45% yield), TLC: R_f = 0.14 (Petroleum ether : Ethyl acetate = 30:1) [UV]. **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.6 (dm, *J* = 246.0 Hz), 141.3 (dm, *J* = 261.0 Hz), 123.9 (t, *J* = 17.5 Hz). **^{19}F**

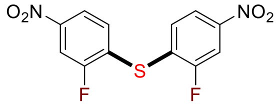
NMR (376 MHz, $CDCl_3$) δ -87.54 – -87.71 (m, 4F), -134.75 – -135.00 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{10}F_8N_2S$ ($[M]^+$), 331.9654, found, 331.9659.

Bis(perfluoro-[1,1'-biphenyl]-4-yl)sulfane (4n)



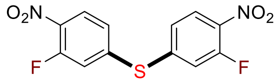
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 50:1 to afford **4n** as colorless oil (70.0 mg, 56% yield), TLC: R_f = 0.21 (Petroleum ether : Ethyl acetate = 50:1) [UV]. ^{13}**C NMR** (100 MHz, $CDCl_3$) δ 147.0 (dm, *J* = 250.0 Hz), 144.5 (dm, *J* = 253.0 Hz), 144.1 (m), 141.6 (m), 138.1 (dm, *J* = 249.0 Hz), 113.3 (t, *J* = 20.5 Hz), 108.3 (t, *J* = 18.0 Hz), 102.0 (t, *J* = 19.0 Hz). ^{19}**F NMR** (376 MHz, $CDCl_3$) δ -131.54 – -131.68 (m, 4F), -136.10 – -136.29 (m, 4F), -136.77 – -136.98 (m, 4F), -149.14 – -149.28 (m, 2F), -159.95 – -160.13 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{24}F_{18}S$ ($[M + H]^+$), 661.9433, found, 661.9430.

Bis(2-fluoro-4-nitrophenyl)sulfane (4o)



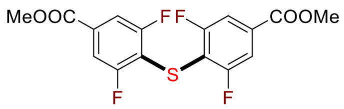
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 70:1 to afford **4o** as colorless oil (28.1 mg, 45% yield), TLC: R_f = 0.15 (Petroleum ether : Ethyl acetate = 70:1) [UV]. ^{1}**H NMR** (400 MHz, $CDCl_3$) δ 8.10 – 7.98 (m, 4H), 7.49 – 7.42 (m, 2H). ^{13}**C NMR** (100 MHz, $CDCl_3$) δ 160.4 (d, *J* = 252.0 Hz), 148.7 (d, *J* = 8.0 Hz), 133.0, 128.5 (d, *J* = 18.0 Hz), 120.1 (d, *J* = 3.0 Hz), 112.2 (d, *J* = 27.0 Hz). ^{19}**F NMR** (376 MHz, $CDCl_3$) δ -103.16 (s, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{12}H_6F_2N_2O_4S$ ($[M + H]^+$), 312.0016, found, 312.0011.

Bis(3-fluoro-4-nitrophenyl)sulfane (4p)



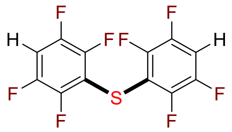
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 70:1 to afford **4p** as colorless oil (25.6 mg, 41% yield), TLC: R_f = 0.16 (Petroleum ether : Ethyl acetate = 70:1) [UV]. ^{1}**H NMR** (400 MHz, $CDCl_3$) δ 8.24 (dd, *J* = 9.2, 5.2 Hz, 2H), 7.21 (ddd, *J* = 9.2, 7.2, 2.8 Hz, 2H), 6.99 (dd, *J* = 8.4, 2.8 Hz, 2H). ^{13}**C NMR** (100 MHz, $CDCl_3$) δ 164.8 (d, *J* = 259.0 Hz), 145.5 (d, *J* = 2.0 Hz), 134.5 (d, *J* = 9.0 Hz), 128.6 (d, *J* = 10.0 Hz), 120.5 (d, *J* = 25.0 Hz), 116.5 (d, *J* = 23.0 Hz). ^{19}**F NMR** (376 MHz, $CDCl_3$) δ -101.49 (s, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{12}H_6F_2N_2O_4S$ ($[M + H]^+$), 312.0016, found, 312.0010.

Dimethyl 4,4'-thiobis(3,5-difluorobenzoate) (4q)



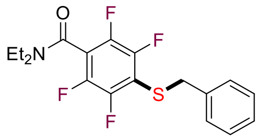
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 60:1 to afford **4q** as colorless oil (33.0 mg, 37% yield), TLC: R_f = 0.16 (Petroleum ether : Ethyl acetate = 60:1) [UV]. ^{1}**H NMR** (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 7.2 Hz, 4H), 3.93 (s, 6H). ^{13}**C NMR** (100 MHz, $CDCl_3$) δ 164.6, 162.0 (dm, *J* = 247.0 Hz), 132.6 (t, *J* = 10.0 Hz), 113.6, 112.8, 53.0. ^{19}**F NMR** (376 MHz, $CDCl_3$) δ -103.73 (s, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{16}H_6F_8O_4S$ ($[M]^+$), 445.9859, found, 445.9850.

Bis(2,3,5,6-tetrafluorophenyl)sulfane (4r)



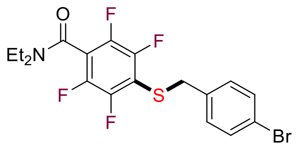
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 100:1 to afford **4r** as colorless oil (25.0 mg, 38% yield), TLC: R_f = 0.31 (Petroleum ether : Ethyl acetate = 100:1) [UV]. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.21 – 7.05 (m, 2H). **^{13}C NMR** (100 MHz, Chloroform-*d*) δ 146.8 (dm, *J* = 244.7 Hz), 146.1 (dm, *J* = 245.5 Hz), 112.1 (t, *J* = 21.9 Hz), 107.7 (t, *J* = 22.8 Hz). **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -133.16 – -133.26 (m, 4F), -137.13 – -137.24 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{12}H_2F_8S$ ($[M]^+$), 329.9749, found, 329.9738.

4-(Benzylthio)-*N*,*N*-diethyl-2,3,5,6-tetrafluorobenzamide (6a)



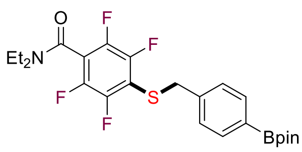
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 50:1 to afford **6a** as colorless oil (60.8 mg, 82% yield), TLC: R_f = 0.15 (Petroleum ether : Ethyl acetate = 50:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32 – 7.14 (m, 5H), 4.14 (s, 2H), 3.57 (q, *J* = 8.0 Hz, 2H), 3.15 (q, *J* = 8.0 Hz, 2H), 1.25 (t, *J* = 8.0 Hz, 3H), 1.09 (t, *J* = 8.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.4, 147.0 (dm, *J* = 246.5 Hz), 142.2 (dm, *J* = 248.0 Hz), 136.2, 128.9, 128.7, 127.9, 116.9 (t, *J* = 22.5 Hz), 114.9 (t, *J* = 20.5 Hz), 43.3, 39.8, 38.8, 14.1, 12.8. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -131.65 – -131.88 (m, 2F), -141.73 – -142.04 (m, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{18}H_{18}F_4NOS$ ($[M+H]^+$), 372.1040, found, 372.1049.

4-((4-Bromobenzyl)thio)-*N*,*N*-diethyl-2,3,5,6-tetrafluorobenzamide (6b)



The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 50:1 to afford **6b** as colorless oil (70.9 mg, 79% yield), TLC: R_f = 0.14 (Petroleum ether : Ethyl acetate = 50:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.4 Hz, 2H), 7.06 (d, *J* = 8.4 Hz, 2H), 4.02 (s, 2H), 3.50 (q, *J* = 7.2 Hz, 2H), 3.07 (q, *J* = 7.2 Hz, 2H), 1.18 (t, *J* = 7.2 Hz, 3H), 1.03 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.3, 147.0 (dm, *J* = 260.0 Hz), 142.3 (dm, *J* = 237.0 Hz), 135.4, 131.8, 130.6, 121.9, 117.1 (t, *J* = 23.0 Hz), 114.5 (t, *J* = 21.0 Hz), 43.3, 39.9, 38.1, 14.1, 12.8. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -131.55 – -131.87 (m, 2F), -141.33 – -141.64 (m, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{18}H_{17}BrF_4NOS$ ($[M+H]^+$), 450.0145, found, 450.0146.

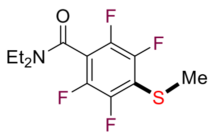
***N*,*N*-diethyl-2,3,5,6-tetrafluoro-4-((4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)thio)benzamide (6c)**



The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 15:1 to afford **6c** as colorless oil (70.6 mg, 71% yield), TLC: R_f = 0.15 (Petroleum ether : Ethyl acetate = 30:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 7.6 Hz, 2H), 7.18 (d, *J* = 7.6 Hz, 2H), 4.07 (s, 2H), 3.50 (q, *J* = 6.8 Hz, 2H), 3.05 (q, *J* = 7.2 Hz, 2H), 1.26 (s, 12H), 1.17 (t, *J* = 6.8 Hz, 4H), 1.02 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.5, 147.0 (dm, *J* = 261.0 Hz), 142.3 (dm, *J* = 261.0 Hz), 139.4, 135.2, 128.3, 116.9 (t, *J* = 22.5 Hz), 114.8 (t,

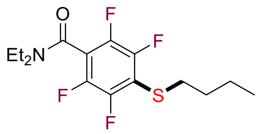
J = 20.0 Hz), 84.0, 43.3, 39.9, 38.8, 25.0, 14.1, 12.9. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -131.53 – -131.84 (m, 2F), -141.63 – -142.00 (m, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{24}H_{29}BF_4NO_3S$ ($[M+H]^+$), 498.1892, found, 498.1890.

***N,N*-diethyl-2,3,5,6-tetrafluoro-4-(methylthio)benzamide (6d)**



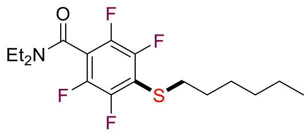
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 50:1 to afford **6d** as colorless oil (53.7 mg, 91% yield), TLC: R_f = 0.16 (Petroleum ether : Ethyl acetate = 50:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.58 (q, J = 6.8 Hz, 2H), 3.23 (q, J = 7.2 Hz, 2H), 2.53 (s, 3H), 1.26 (t, J = 7.2 Hz, 3H), 1.13 (t, J = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.6, 146.5 (dm, J = 247.0 Hz), 142.5 (dm, J = 237.0 Hz), 117.5 (t, J = 20.0 Hz), 116.2 (t, J = 22.0 Hz), 43.4, 39.8, 17.7, 14.2, 12.9. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -133.25 – -133.53 (m, 2F), -141.83 – -142.08 (m, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{12}H_{14}F_4NOS$ ($[M+H]^+$), 296.0727, found, 296.0720.

4-(Butylthio)-*N,N*-diethyl-2,3,5,6-tetrafluorobenzamide (6e)



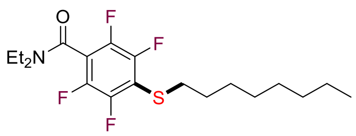
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 50:1 to afford **6e** as colorless oil (46.5 mg, 69% yield), TLC: R_f = 0.15 (Petroleum ether : Ethyl acetate = 50:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.60 (q, J = 7.2 Hz, 2H), 3.52 (t, J = 7.4 Hz, 2H), 3.24 (q, J = 7.2 Hz, 2H), 1.80 – 1.69 (m, 2H), 1.52 – 1.39 (m, 2H), 1.27 (t, J = 7.2 Hz, 3H), 1.16 (t, J = 7.2 Hz, 3H), 0.97 (t, J = 7.4 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.8, 142.9 (dm, J = 258.0 Hz), 135.7 (dm, J = 250.0 Hz), 118.9 (t, J = 22.0 Hz), 116.4 (t, J = 21.0 Hz), 54.8, 43.5, 40.0, 30.0, 19.4, 14.2, 13.7, 12.9. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -140.20 – -140.48 (m, 2F), -144.56 – -144.80 (m, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{15}H_{20}F_4NOS$ ($[M+H]^+$), 338.1196, found, 338.1190.

***N,N*-diethyl-2,3,5,6-tetrafluoro-4-(hexylthio)benzamide (6f)**



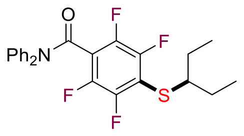
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 50:1 to afford **6f** as colorless oil (60.6 mg, 83% yield), TLC: R_f = 0.15 (Petroleum ether : Ethyl acetate = 50:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.58 (q, J = 7.2 Hz, 2H), 3.23 (q, J = 7.2 Hz, 2H), 2.93 (t, J = 7.4 Hz, 2H), 1.63 – 1.50 (m, 2H), 1.45 – 1.33 (m, 2H), 1.30 – 1.19 (m, 7H), 1.12 (t, J = 7.2 Hz, 3H), 0.87 (t, J = 6.8 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.6, 147.0 (dm, J = 246.0 Hz), 142.3 (dm, J = 263.0 Hz), 116.4 (t, J = 22.0 Hz), 116.1 (t, J = 20.5 Hz), 43.4, 39.8, 34.8, 31.3, 29.9, 28.2, 22.6, 14.1, 14.1, 12.9. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -132.33 – -132.71 (m, 2F), -141.76 – -142.09 (m, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{17}H_{24}F_4NOS$ ($[M+H]^+$), 366.1509, found, 366.1510.

***N,N*-diethyl-2,3,5,6-tetrafluoro-4-(octylthio)benzamide (6g)**



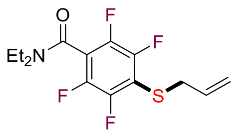
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 60:1 to afford **6g** as colorless oil (70.7 mg, 90% yield), TLC: R_f = 0.15 (Petroleum ether : Ethyl acetate = 60:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.57 (q, J = 7.2 Hz, 2H), 3.22 (q, J = 7.2 Hz, 2H), 2.92 (t, J = 7.4 Hz, 2H), 1.61 – 1.49 (m, 2H), 1.44 – 1.32 (m, 2H), 1.28 – 1.21 (m, 11H), 1.12 (t, J = 7.2 Hz, 3H), 0.85 (t, J = 6.8 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.5, 146.9 (dm, J = 245.5 Hz), 142.2 (dm, J = 249.0 Hz), 116.3 (t, J = 22.0 Hz), 116.0 (t, J = 20.5 Hz), 43.2, 39.7, 34.7, 31.7, 29.8, 29.1, 29.0, 28.4, 22.6, 14.1, 14.0, 12.7. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -132.38 – -132.68 (m, 2F), -141.80 – -142.08 (m, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{19}H_{28}F_4NOS$ ($[M+H]^+$), 394.1822, found, 394.1824.

2,3,5,6-Tetrafluoro-4-(pentan-3-ylthio)-*N,N*-diphenylbenzamide (6h)



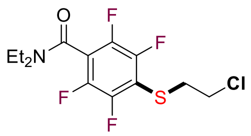
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 60:1 to afford **6h** as colorless oil (51.9 mg, 74% yield), TLC: R_f = 0.17 (Petroleum ether : Ethyl acetate = 60:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.44 – 7.22 (m, 10H), 3.14 – 3.03 (m, 1H), 1.59 – 1.38(m, 4H), 0.94 (t, J = 7.4 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.3, 147.0 (dm, J = 245.0 Hz), 141.9 (dm, J = 248.0 Hz), 141.3, 141.3, 129.6, 129.4, 128.7, 127.9, 127.3, 126.1, 117.7 (t, J = 21.0 Hz), 116.2 (t, J = 21.0 Hz), 53.3, 27.1, 10.9. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -131.69 – -132.04 (m, 2F), -140.40 – -140.76 (m, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{24}H_{22}F_4NOS$ ($[M+H]^+$), 448.1353, found, 448.1350.

4-(Allylthio)-*N,N*-diethyl-2,3,5,6-tetrafluorobenzamide (6i)



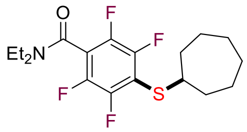
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 60:1 to afford **6i** as colorless oil (59.7 mg, 93% yield), TLC: R_f = 0.17 (Petroleum ether : Ethyl acetate = 60:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.84 – 5.69 (m, 1H), 5.10 – 4.93 (m, 2H), 3.60 – 3.49 (m, 4H), 3.19 (q, J = 7.2 Hz, 2H), 1.24 (t, J = 7.2 Hz, 3H), 1.10 (t, J = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.5, 147.1 (dm, J = 246.0 Hz), 142.3 (dm, J = 249.0 Hz), 132.7, 118.9, 116.9 (t, J = 22.0 Hz), 114.7 (t, J = 20.5 Hz), 43.3, 39.9, 37.5, 14.1, 12.8. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -131.63 – -131.95 (m, 2F), -141.76 – -142.08 (m, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{14}H_{16}F_4NOS$ ($[M+H]^+$), 322.0883, found, 322.0886.

4-((2-Chloroethyl)thio)-*N,N*-diethyl-2,3,5,6-tetrafluorobenzamide (6j)



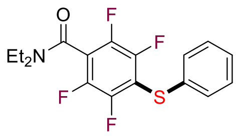
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 60:1 to afford **6j** as colorless oil (42.5 mg, 62% yield), TLC: R_f = 0.19 (Petroleum ether : Ethyl acetate = 60:1) [UV]. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 3.68 – 3.55 (m, 4H), 3.30 – 3.18 (m, 4H), 1.27 (t, J = 7.2 Hz, 3H), 1.15 (t, J = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.3, 147.1 (dm, J = 257.5 Hz), 142.4 (dm, J = 252.7 Hz), 117.6 (t, J = 27.5 Hz), 114.2 (t, J = 21.2 Hz), 43.4, 42.7, 39.9, 36.5, 14.2, 12.9. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -131.51 – -131.61(m, 2F), -140.87 – -140.97 (m, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{13}H_{15}ClF_4NOS$ ($[M+H]^+$), 344.0494, found, 344.0485.

4-(Cycloheptylthio)-*N*,*N*-diethyl-2,3,5,6-tetrafluorobenzamide (6k)



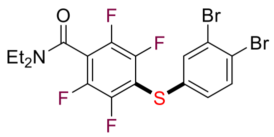
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 60:1 to afford **6j** as colorless oil (63.3 mg, 84% yield), TLC: R_f = 0.18 (Petroleum ether : Ethyl acetate = 60:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.59 (q, *J* = 7.2 Hz, 2H), 3.53 – 3.40 (m, 1H), 3.23 (q, *J* = 7.2 Hz, 2H), 1.98 – 1.87 (m, 2H), 1.80 – 1.68 (m, 2H), 1.62 – 1.50 (m, 6H), 1.48 – 1.38 (m, 2H), 1.26 (t, *J* = 7.0 Hz, 3H), 1.13 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.7, 147.3 (dm, *J* = 245.5 Hz), 142.4 (dm, *J* = 266.0 Hz), 116.7 (t, *J* = 22.0 Hz), 115.7 (t, *J* = 20.5 Hz), 49.0, 43.4, 39.8, 35.1, 28.2, 25.6, 14.1, 12.9. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -131.45 – -131.88 (m, 2F), -141.58 – -141.94 (m, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{18}H_{24}F_4NOS$ ($[M+H]^+$), 378.1509, found, 378.1510.

***N*,*N*-diethyl-2,3,5,6-tetrafluoro-4-(phenylthio)benzamide (6l)**



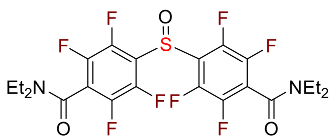
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 30:1 to afford **6l** as colorless oil (51.4 mg, 72% yield), TLC: R_f = 0.16 (Petroleum ether : Ethyl acetate = 30:1) [UV]. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.41 – 7.37 (m, 2H), 7.34 – 7.28 (m, 3H), 3.59 (q, *J* = 7.2 Hz, 2H), 3.24 (q, *J* = 7.2 Hz, 2H), 1.26 (t, *J* = 7.2 Hz, 3H), 1.14 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, Chloroform-*d*) δ 158.3, 147.0 (dm, *J* = 247.0 Hz), 142.4 (dm, *J* = 251.0 Hz), 132.4, 131.0, 129.4, 128.1, 117.6 (t, *J* = 22.0 Hz), 115.2 (t, *J* = 20.0 Hz), 43.3, 39.7, 14.0, 12.7. **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -131.05 – -131.21 (m, 2F), -140.91 – -141.15 (m, 2F). HRMS (ESI-TOF) (m/z): Calcd for $C_{17}H_{16}F_4NOS$ ($[M + H]^+$), 358.0883, found, 358.0880.

4-((3,4-Dibromophenyl)thio)-*N*,*N*-diethyl-2,3,5,6-tetrafluorobenzamide (6m)



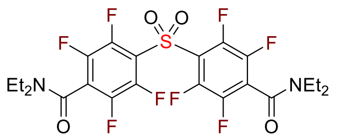
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 15:1 to afford **6m** as colorless oil (44.0 mg, 43% yield), TLC: R_f = 0.19 (Petroleum ether : Ethyl acetate = 15:1) [UV]. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 8.8 Hz, 1H), 7.28 (d, *J* = 2.8 Hz, 1H), 6.83 (dd, *J* = 8.8, 3.2 Hz, 1H), 3.61 (q, *J* = 7.2 Hz, 2H), 3.27 (q, *J* = 7.2 Hz, 2H), 1.28 (t, *J* = 7.2 Hz, 3H), 1.17 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, Chloroform-*d*) δ 158.1, 156.2, 143.1 (dm, *J* = 253.6 Hz), 141.5 (dm, *J* = 253.3 Hz), 134.4, 125.7, 121.2, 119.6, 116.3, 43.5, 40.0, 14.2, 12.9. **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -141.03 – -141.13 (m, 2F), -152.00 – -152.09 (m, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{17}H_{14}Br_2F_4NOS$ ($[M + H]^+$), 513.9093, found, 513.9088.

4,4'-Sulfinylbis(*N*,*N*-diethyl-2,3,5,6-tetrafluorobenzamide) (7)



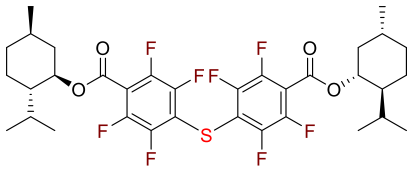
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 10:1 to afford **7** as colorless oil (94.6 mg, 87% yield), TLC: R_f = 0.11 (Petroleum ether : Ethyl acetate = 10:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.59 (q, *J* = 7.2 Hz, 4H), 3.24 (q, *J* = 7.0 Hz, 4H), 1.27 (t, J = 7.0 Hz, 6H), 1.17 (t, J = 7.2 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.4, 144.2 (dm, J = 262.0 Hz), 142.5 (dm, J = 253.0 Hz), 121.4 (t, J = 22.0 Hz), 112.5 (t, J = 26.0 Hz), 43.5, 40.0, 14.2, 12.8. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -137.77 – -137.98 (m, 2F), -138.00 – -138.15 (m, 2F), -138.25 (dd, J = 22.9, 12.0 Hz, 2F), -138.62 (dd, J = 22.6, 11.3 Hz, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{22}H_{20}F_8N_2O_3S$ ([M + H]$^+$), 544.1067, found, 544.1060.

4,4'-Sulfonylbis(*N*,*N*-diethyl-2,3,5,6-tetrafluorobenzamide) (8)



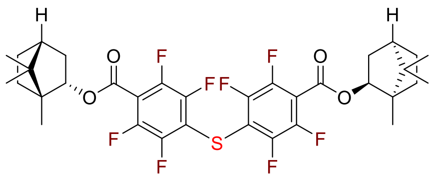
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 10:1 to afford **8** as colorless oil (72.8 mg, 65% yield), TLC: R_f = 0.12 (Petroleum ether : Ethyl acetate = 10:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.59 (q, J = 7.2 Hz, 4H), 3.23 (q, J = 7.2 Hz, 4H), 1.26 (t, J = 7.2 Hz, 6H), 1.16 (t, J = 7.2 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 156.8, 144.5 (dm, J = 266.5 Hz), 142.6 (dm, J = 254.5 Hz), 123.6 (t, J = 22.5 Hz), 120.6 (t, J = 22.0 Hz), 43.4, 40.0, 14.2, 12.8. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -133.95 – -134.27 (m, 4F), -137.51 – -137.79 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{22}H_{20}F_8N_2O_4S$ ([M + H]$^+$), 560.1016, found, 560.1011.

Bis((1R,2S,5R)-2-isopropyl-5-methylcyclohexyl) 4,4'-thiobis(2,3,5,6-tetrafluorobenzoate) (9)



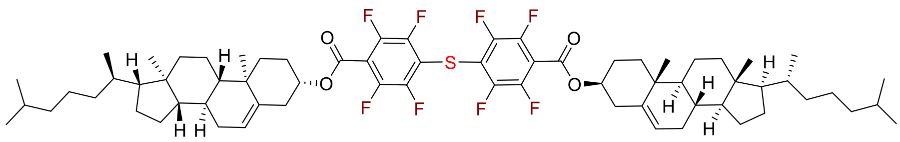
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 50:1 to afford **9** as colorless oil (95.8 mg, 69% yield), TLC: R_f = 0.15 (Petroleum ether : Ethyl acetate = 50:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.05 – 4.94 (m, 2H), 2.19 – 2.13 (m, 2H), 2.03 – 1.94 (m, 2H), 1.78 – 1.70 (m, 4H), 1.59 – 1.45 (m, 4H), 1.19 – 1.07 (m, 4H), 0.93 (m, 14H), 0.81 (d, J = 7.2 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.8, 146.6 (dm, J = 249.0 Hz), 144.3 (dm, J = 254.5 Hz), 115.3 (t, J = 17.5 Hz), 113.6 (t, J = 20.0 Hz), 78.1, 47.0, 40.7, 34.2, 31.7, 26.1, 23.2, 22.1, 20.9, 16.0. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -131.85 – -132.05 (m, 4F), -138.25 – -138.51 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{34}H_{38}F_8O_4S$ ([M]$^+$), 694.2363, found, 694.2366.

Bis((1R,2S,4R)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl) 4,4'-thiobis (2,3,5,6-tetrAfluoroben zoate) (10)



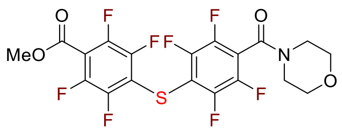
The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 50:1 to afford **10** as colorless oil (85.6 mg, 62% yield), TLC: R_f = 0.17 (Petroleum ether : Ethyl acetate = 50:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.22 – 5.15 (m, 2H), 2.55 – 2.43 (m, 2H), 2.00 – 1.92 (m, 2H), 1.80 – 1.72 (m, 4H), 1.40 – 1.23 (m, 6H), 1.16 (d, J = 3.6 Hz, 1H), 1.13 (d, J = 3.6 Hz, 1H), 0.95 (s, 6H), 0.91 (s, 10H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.5, 146.7 (dm, J = 251.0 Hz), 144.6 (dm, J = 258.0 Hz), 131.0, 129.0, 114.9 (t, J = 16.0 Hz), 113.9 (t, J = 20.0 Hz), 83.8, 49.2, 48.1, 45.0, 36.8, 28.1, 27.2, 19.8, 19.0, 13.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -131.85 – -132.16 (m, 4F), -137.79 – -138.08 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{34}H_{34}F_8O_4S$ ([M]$^+$), 690.2050, found, 690.2052.

Bis((3S,8S,9S,10R,13R,14S,17R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl) 4,4'-thiobis(2,3,5,6-tetrafluorobenzoate) (11)



The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 50:1 to afford **11** as colorless oil (131.6 mg, 57% yield), TLC: R_f = 0.17 (Petroleum ether : Ethyl acetate = 50:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.45 – 5.42 (m, 2H), 4.97 – 4.87 (m, 2H), 2.50 – 2.43 (m, 4H), 2.02 – 1.04 (m, 58H), 0.92 (d, *J* = 6.8 Hz, 6H), 0.87 (dd, *J* = 6.4, 2.0 Hz, 12H), 0.68 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.5, 146.6 (dm, *J* = 250.0 Hz), 144.4 (dm, *J* = 257.5 Hz), 139.1, 123.5, 115.0 (t, *J* = 16.5 Hz), 113.8 (t, *J* = 20.0 Hz), 56.8, 56.1, 50.1, 42.5, 39.9, 39.7, 38.0, 37.0, 36.7, 36.3, 35.9, 32.1, 32.0, 28.4, 28.2, 27.8, 24.4, 24.0, 23.0, 22.7, 21.2, 19.4, 18.9, 12.0. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -131.86 – -132.13 (m, 4F), -138.08 – -138.30 (m, 4F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{68}H_{90}F_8O_4S$ ($[M]^+$), 1154.6432, found, 1154.6449.

Methyl 2,3,5,6-tetrafluoro-4-((2,3,5,6-tetrafluoro-4-(morpholine-4-carbonyl)phenyl)thio) benzoate (12)

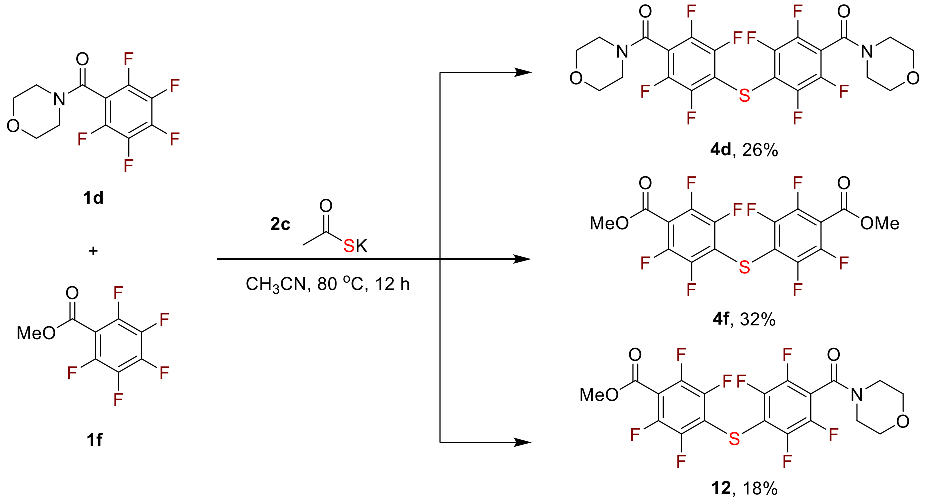


The crude was purified by flash chromatography using Petroleum ether/ Ethyl acetate 20:1 to afford **12** as colorless oil (18.0 mg, 18% yield), TLC: R_f = 0.16 (Petroleum ether : Ethyl acetate = 20:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.99 (s, 3H), 3.84 – 3.78 (m, 4H), 3.70 – 3.67 (m, 2H), 3.35 – 3.32 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.2, 146.7 (dm, *J* = 252.0 Hz), 144.6 (dm, *J* = 258.0 Hz), 117.2 (t, *J* = 21.0 Hz), 114.1 (t, *J* = 17.0 Hz), 66.9, 66.7, 53.7, 47.4, 42.8. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -130.59 – -130.76 (m, 2F), -131.66 – -131.85 (m, 4F), -137.47 – -137.65 (m, 2F), -139.22 – -139.43 (m, 2F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{19}H_{11}F_8NO_4S$ ($[M + H]^+$), 502.0354, found, 502.0353.

4. Mechanistic investigation

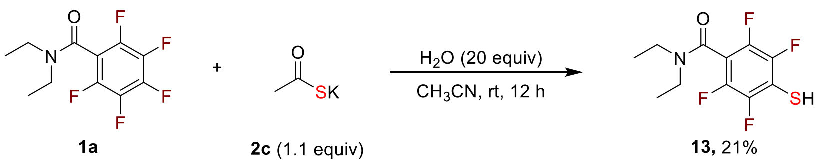
4.1 Cross-over experiment

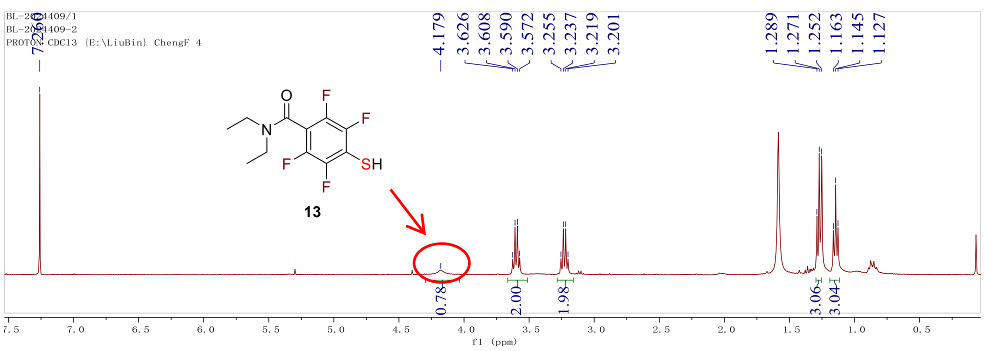
To gain deeper insights into the aforementioned transformation, a cross-over experiment was performed, where **1d** (28.1 mg, 0.1 mmol, 1.0 equiv) and **1f** (22.6 mg, 0.1 mmol, 1.0 equiv) were subjected to the same reaction conditions in one pot manner in the presence of potassium thioacetate **2c** (17.1 mg, 0.15 mmol, 0.75 equiv). The cross-over product **12** was detected in 18% yield, most of the symmetric sulfide **4d** and **4f** were generated, and the yields were 26% and 32%, respectively.



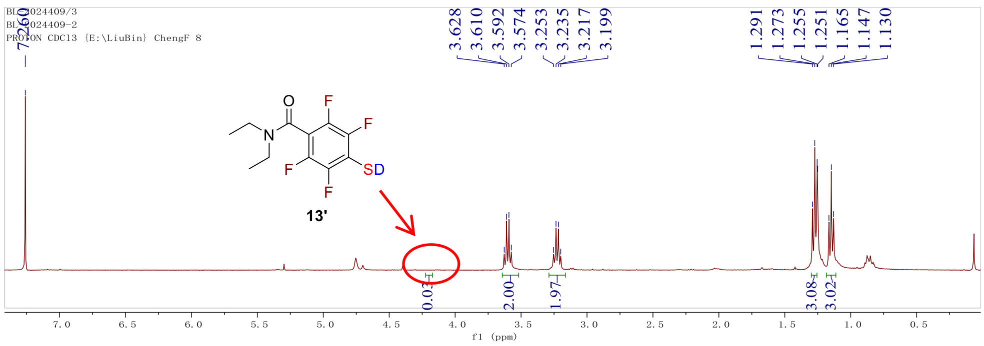
4.2 Intermediate capture experiment

To confirm the formation of thiolate intermediate during the reaction, an intermediate capture experiment was performed, where **1a** (53.4 mg, 0.2 mmol, 1.0 equiv.) and H_2O (36.0 mg, 4.0 mmol, 20 equiv.) were subjected to the same reaction conditions in one pot manner in the presence of potassium thioacetate **2c** (25.1 mg, 0.22 mmol, 1.1 equiv.). The arylthiophenol **13** was detected in 21% yield, indicating that a polyfluoroarene-substituted sulfur anion may serve as a key intermediate in this reaction.





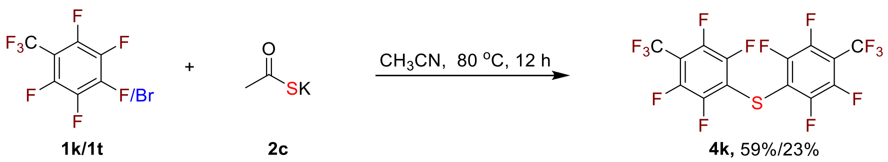
^{1}H NMR of **13**



^{1}H NMR of **13 (Deuterium exchange experiment)**

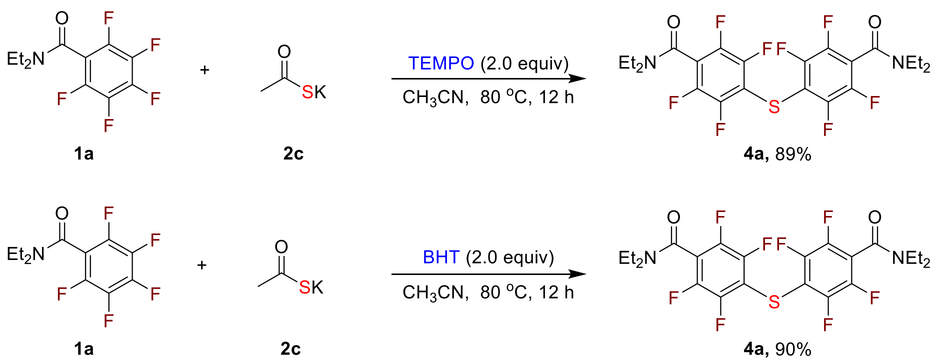
4.3 Controlled experiment

To confirm the special use of fluorine atom in the reaction, a controlled experiment was performed, where brominated polyfluoroarenes **1t** (59.2 mg, 0.2 mmol, 1.0 equiv) was subjected to the same reaction conditions. The product **4k** was detected in 23% yield. Compared with the use of polyfluoroarene **1k**, the yield of product **4k** was significantly reduced. This indicates that compared with fluorine ion, bromine ion has poor leaving ability or nucleophilic property in the reaction process.



4.4 Radical inhibition experiments

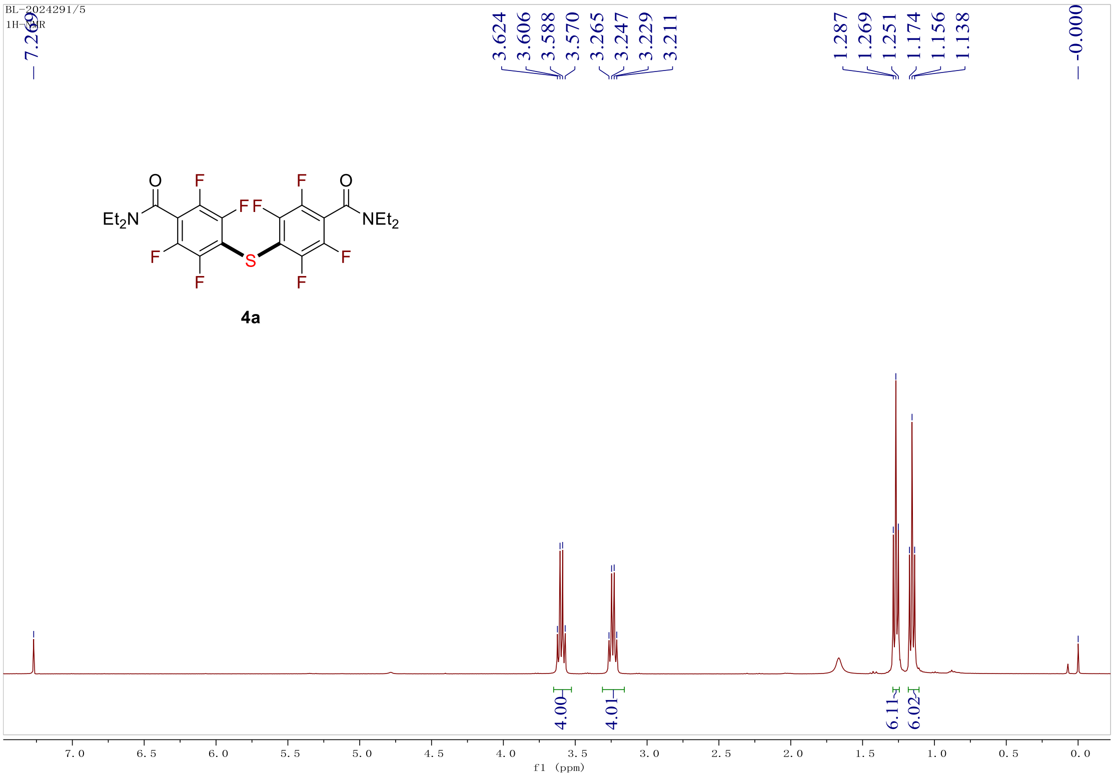
To investigate if the sulfane formation proceeds through the radical pathway, we conducted a couple of reactions in the presence of trapping reagent TEMPO and BHT. Under the standard conditions, two equivalent of radical scavengers (TEMPO, BHT) were added to the reaction. To our delight, standard product **4a** was afforded in 89% and 90% yield. No significant decrease of yield was observed. Hence, it can be concluded that the reaction is not passing through radical pathway.

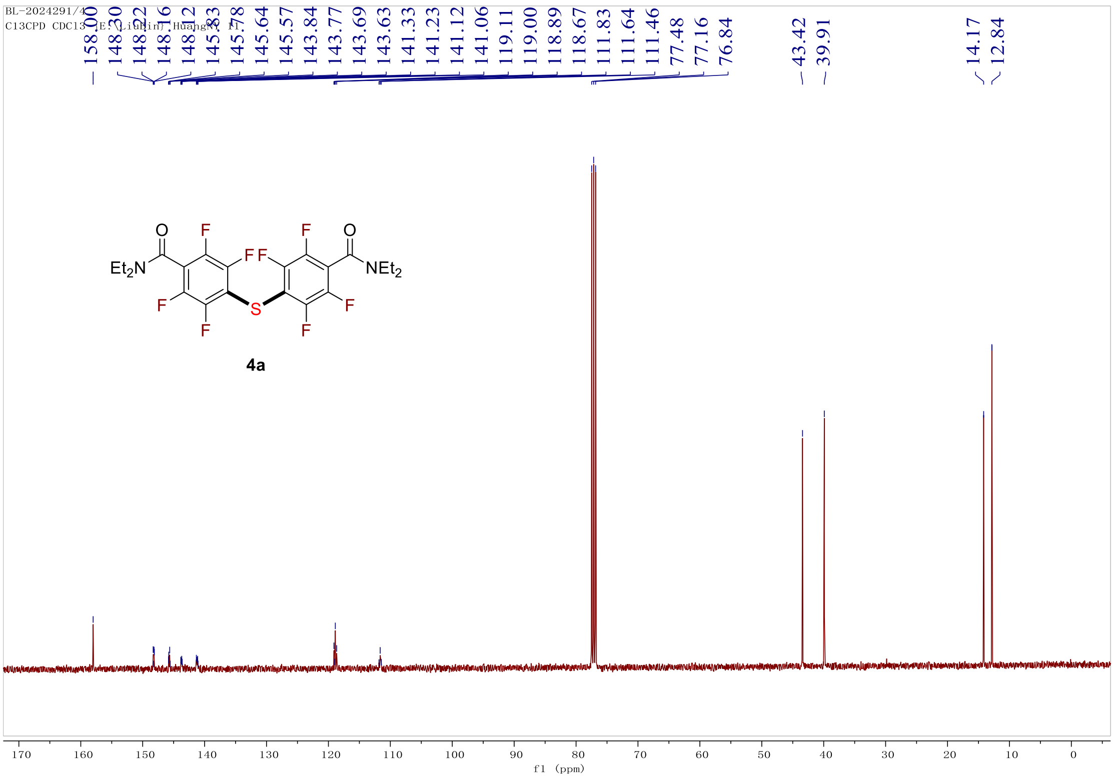


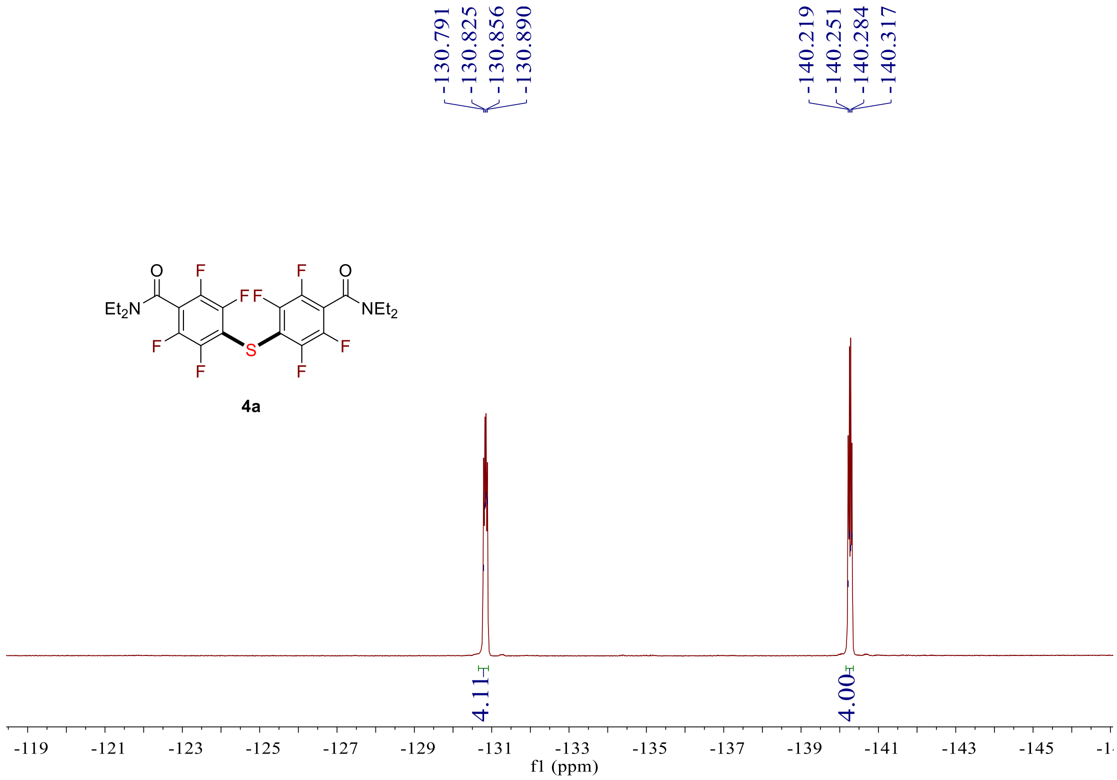
5. References

[1] Zhao, Y.; Fu, B.; Wang, S.; Li, Y.; Yuan, X.; Yin, J.; Xiong, T.; Zhang, Q. *Org. Lett.* **2023**, *25*, 2492.

6. ^{1}H, ^{13}C, ^{19}F Spectra of New Compounds







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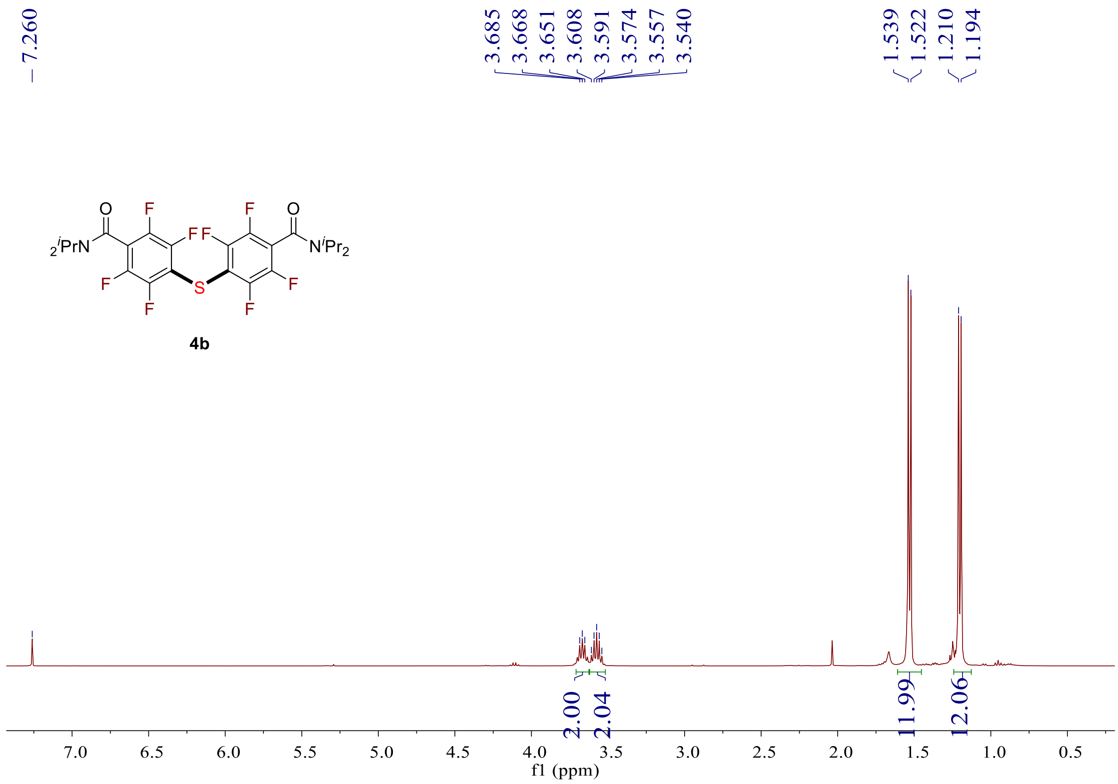
-130.825
-130.856
-130.890
-140.219
-140.251
-140.284
-140.317
Et2N
NEt2
F
S
4a
4.11
4.00
-119 -121 -123 -125 -127 -129 -131 -133 -135 -137 -139 -141 -143 -145
f1 (ppm)



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3.668
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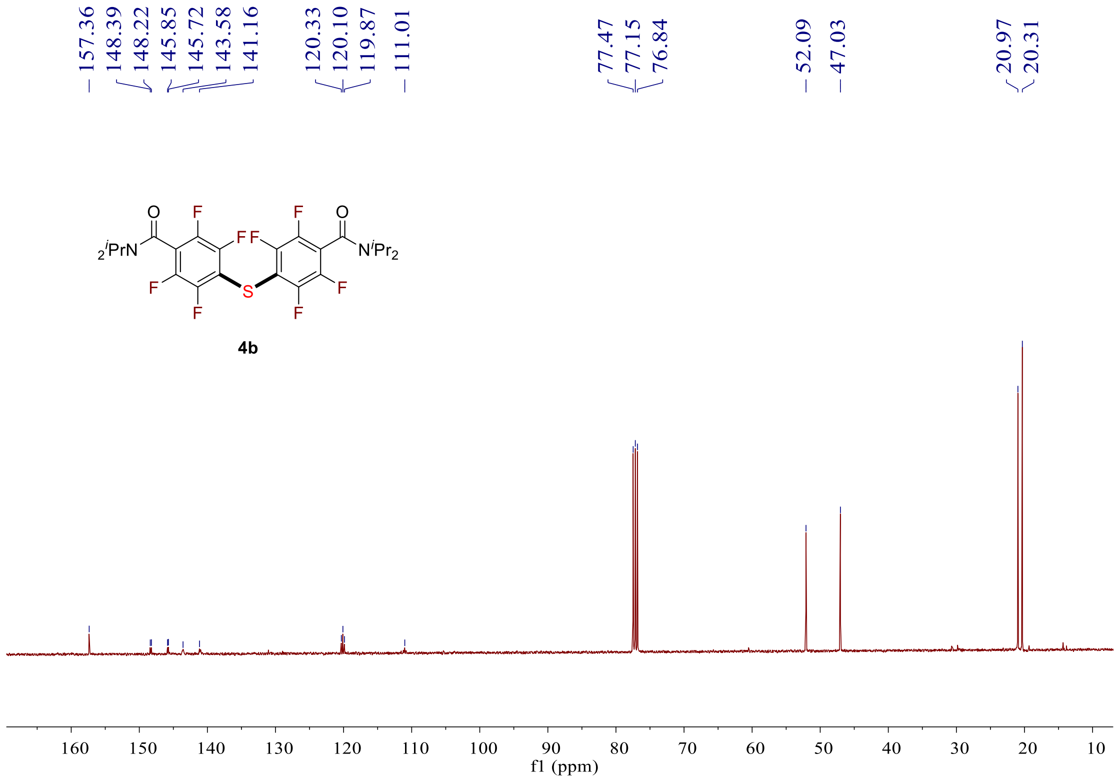
3.608
3.591
3.574
3.557
3.540
1.539
1.522
1.210
1.194
2iPrN
NiPr2
F
S
4b
2.00
2.04
11.99
12.06
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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148.39
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145.72
143.58
141.16
120.33
120.10
119.87
111.01
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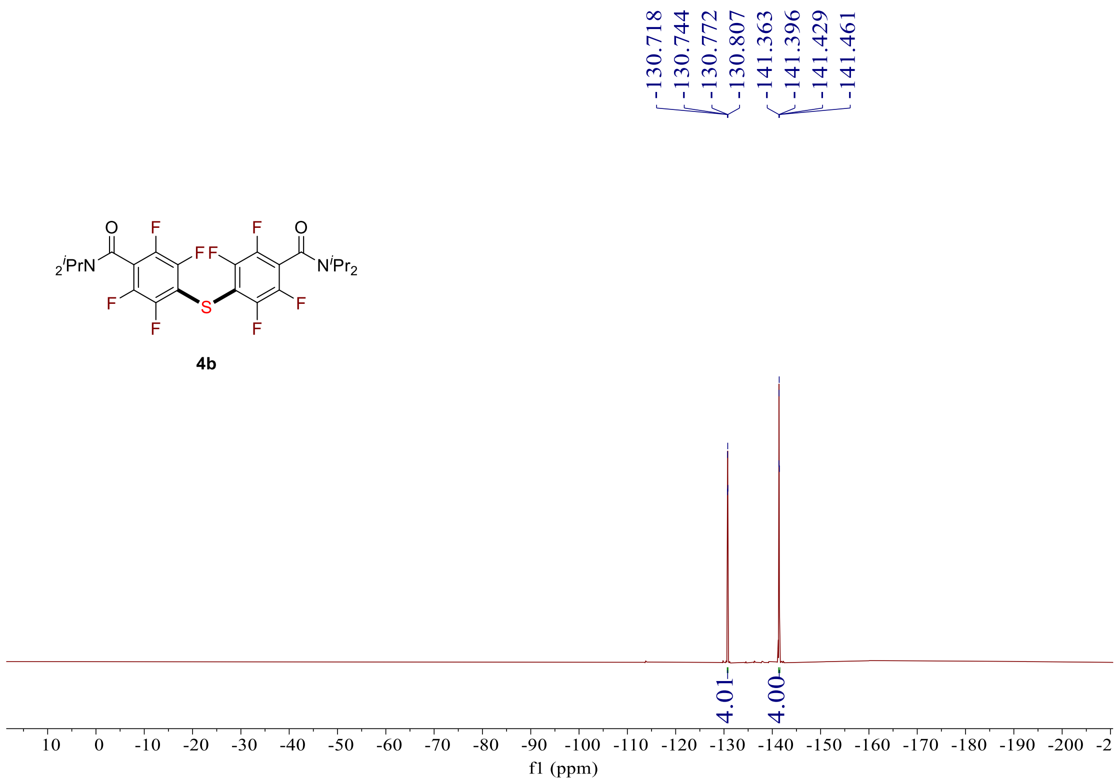
20.31
4b
f1 (ppm)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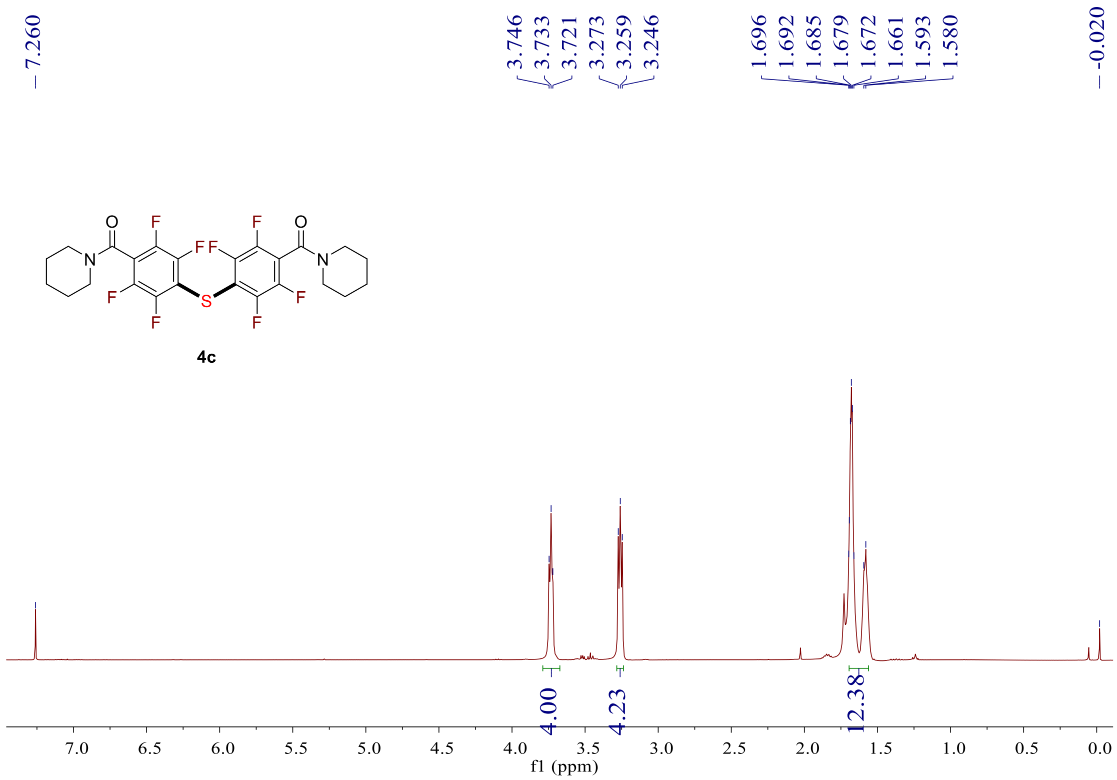
4b
4.01
4.00
f1 (ppm)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1.692
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4c
4.00
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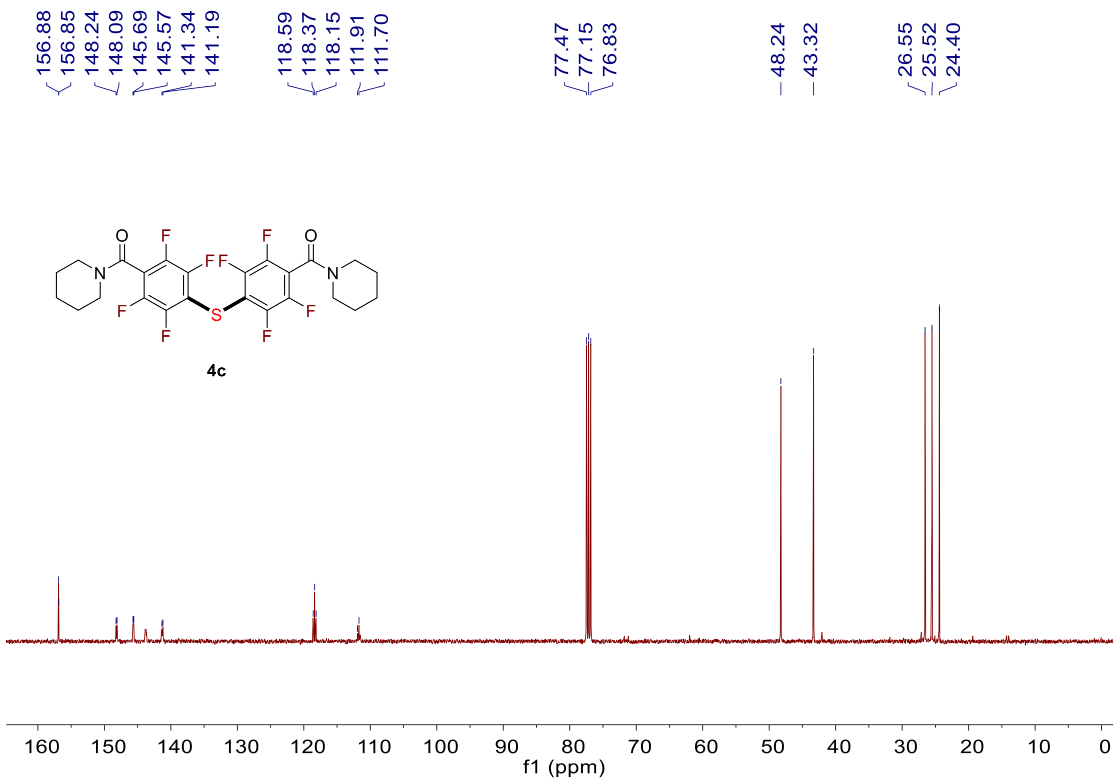
12.38
f1 (ppm)


156.88
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141.19
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118.37
118.15
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111.70
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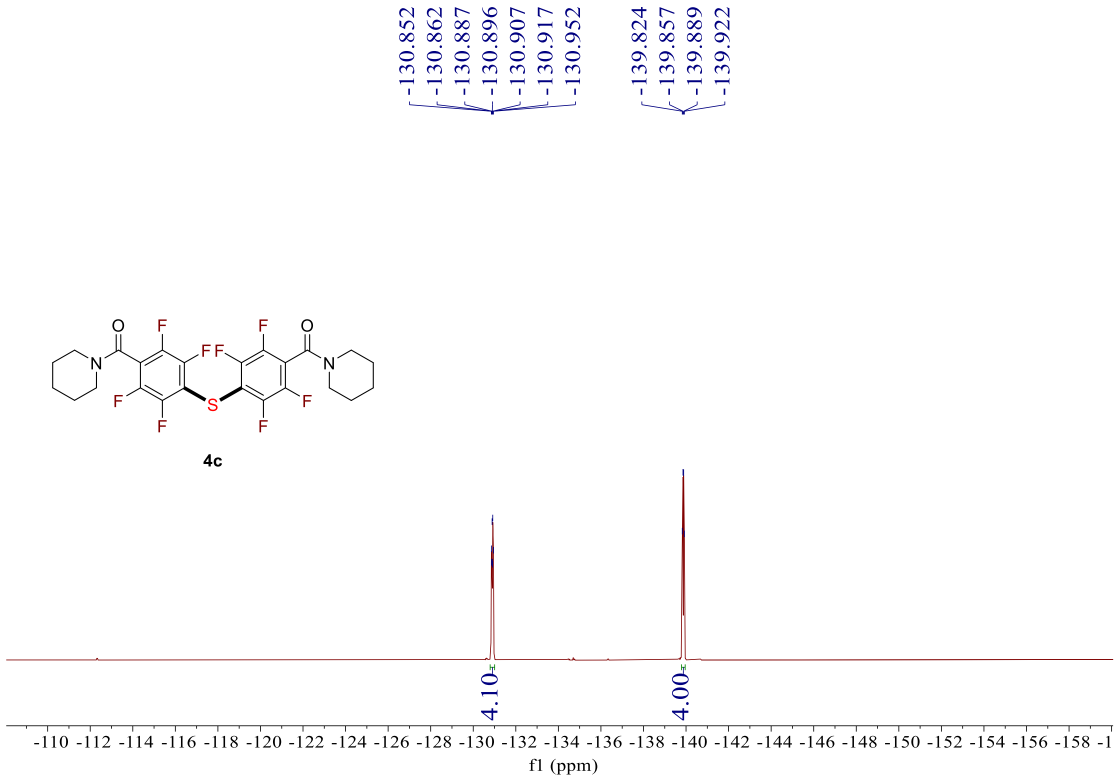
77.15
76.83
48.24
43.32
26.55
25.52
24.40
4c
f1 (ppm)



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-130.862
-130.887
-130.896
-130.907
-130.917
-130.952
-139.824
-139.857
-139.889
-139.922
4c
4.10
4.00
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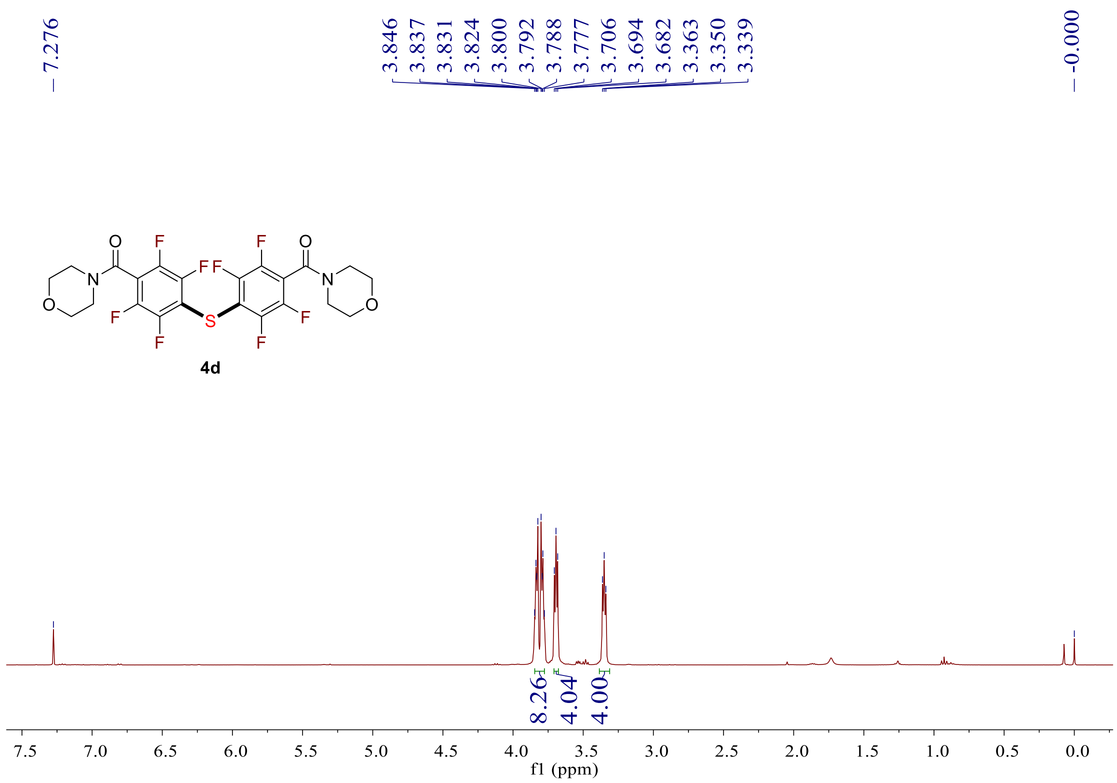
f1 (ppm)



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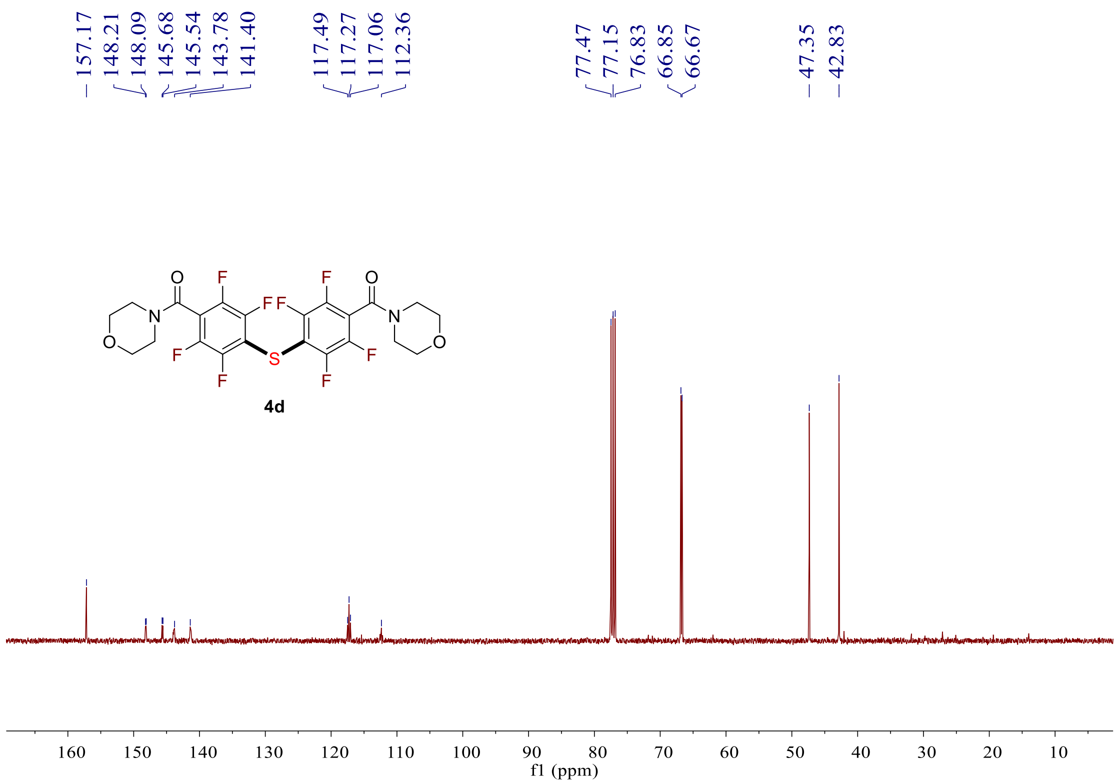
3.846
3.837
3.831
3.824
3.800
3.792
3.788
3.777
3.706
3.694
3.682
3.363
3.350
3.339
-0.000
4d
8.26
4.04
4.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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148.09
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143.78
141.40
117.49
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117.06
112.36
77.47
77.15
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66.85
66.67
47.35
42.83
4d
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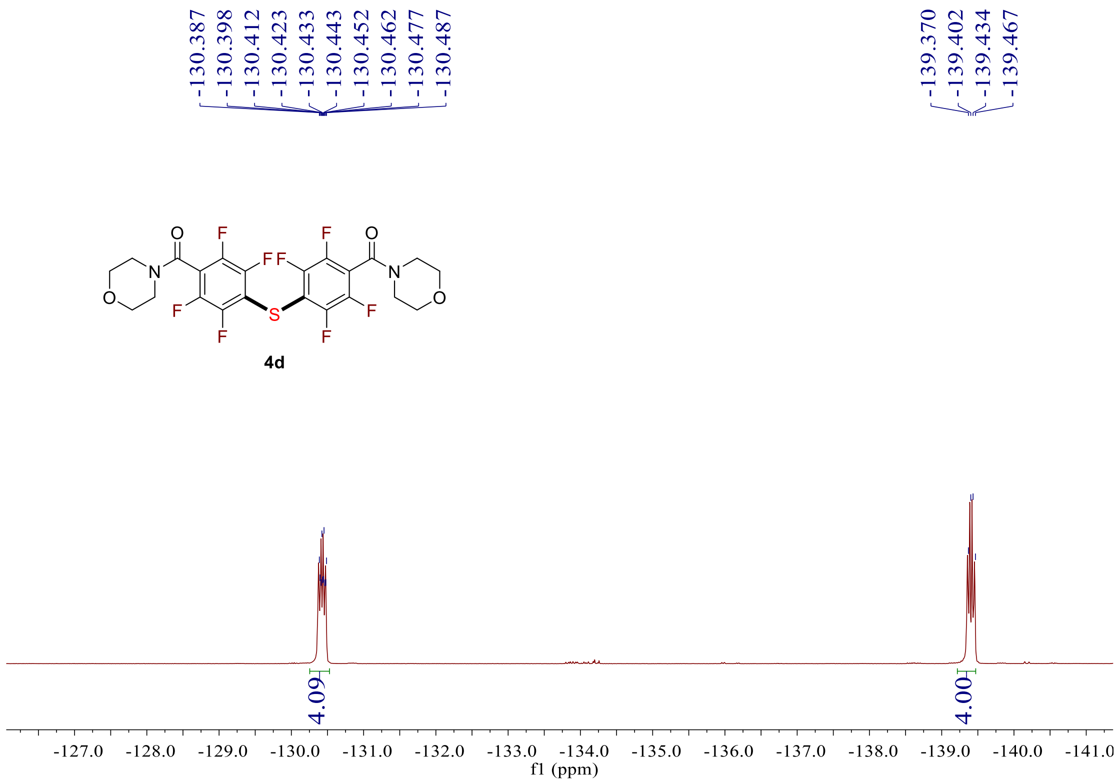
f1 (ppm)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-130.452
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-130.477
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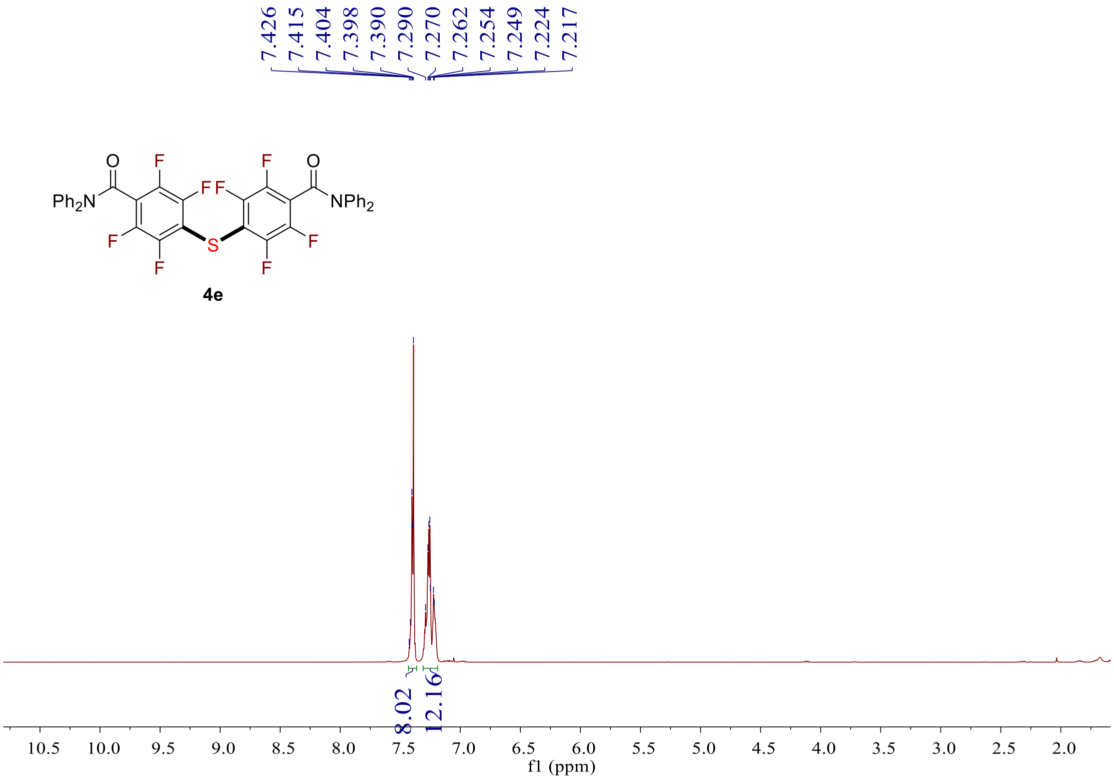
-139.402
-139.434
-139.467
4d
4.09
4.00
-127.0 -128.0 -129.0 -130.0 -131.0 -132.0 -133.0 -134.0 -135.0 -136.0 -137.0 -138.0 -139.0 -140.0 -141.0
f1 (ppm)



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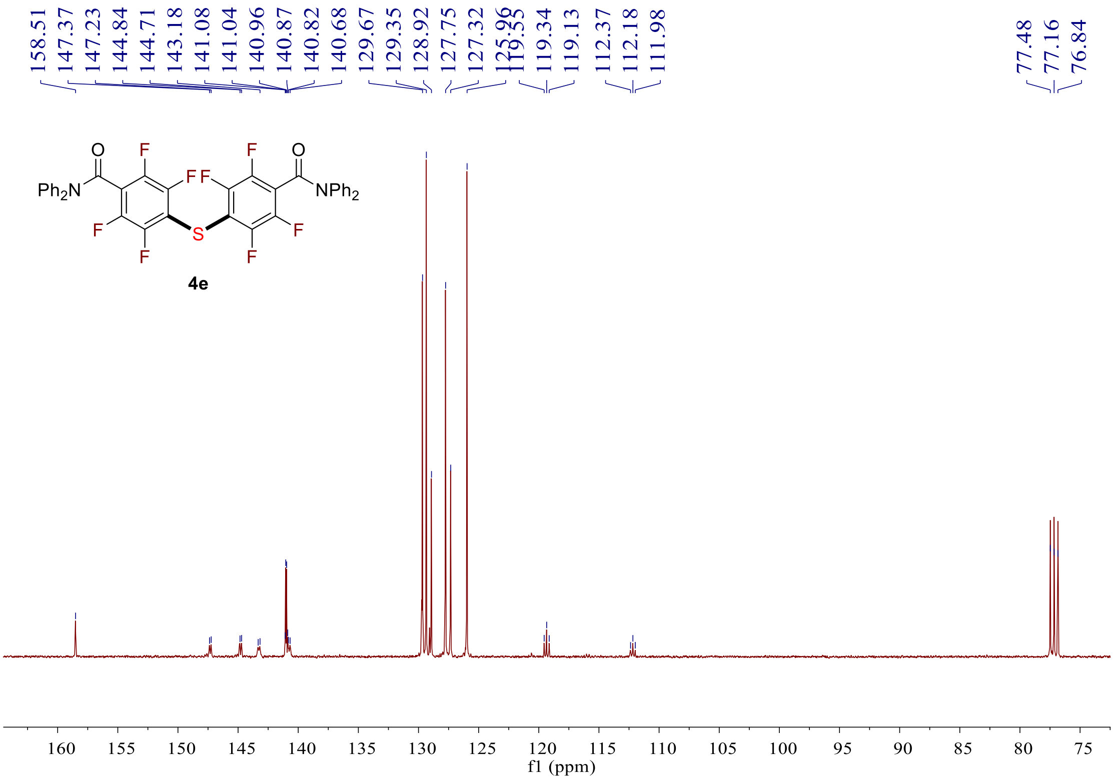
7.415
7.404
7.398
7.390
7.290
7.270
7.262
7.254
7.249
7.224
7.217
4e
8.02
12.16
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0
f1 (ppm)



158.51
147.37
147.23
144.84
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143.18
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141.04
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140.87
140.82
140.68
129.67
129.35
128.92
127.75
127.32
125.96
119.55
119.34
119.13
112.37
112.18
111.98
77.48
77.16
76.84
4e
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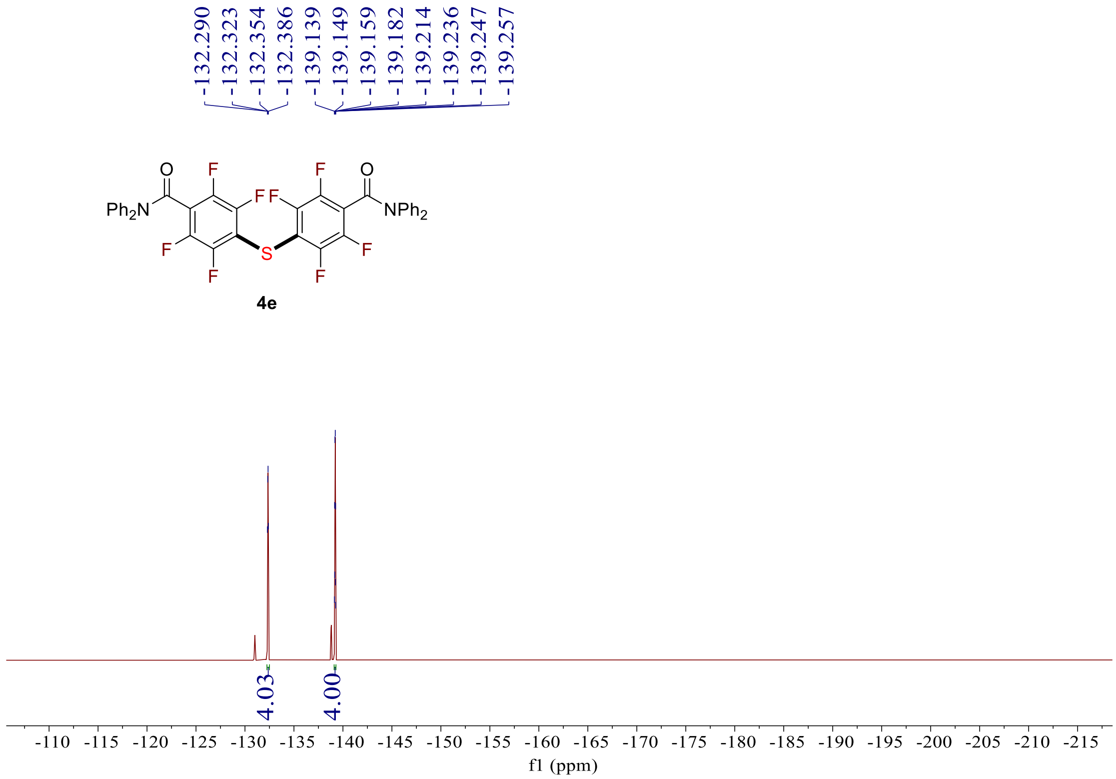
f1 (ppm)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-139.139
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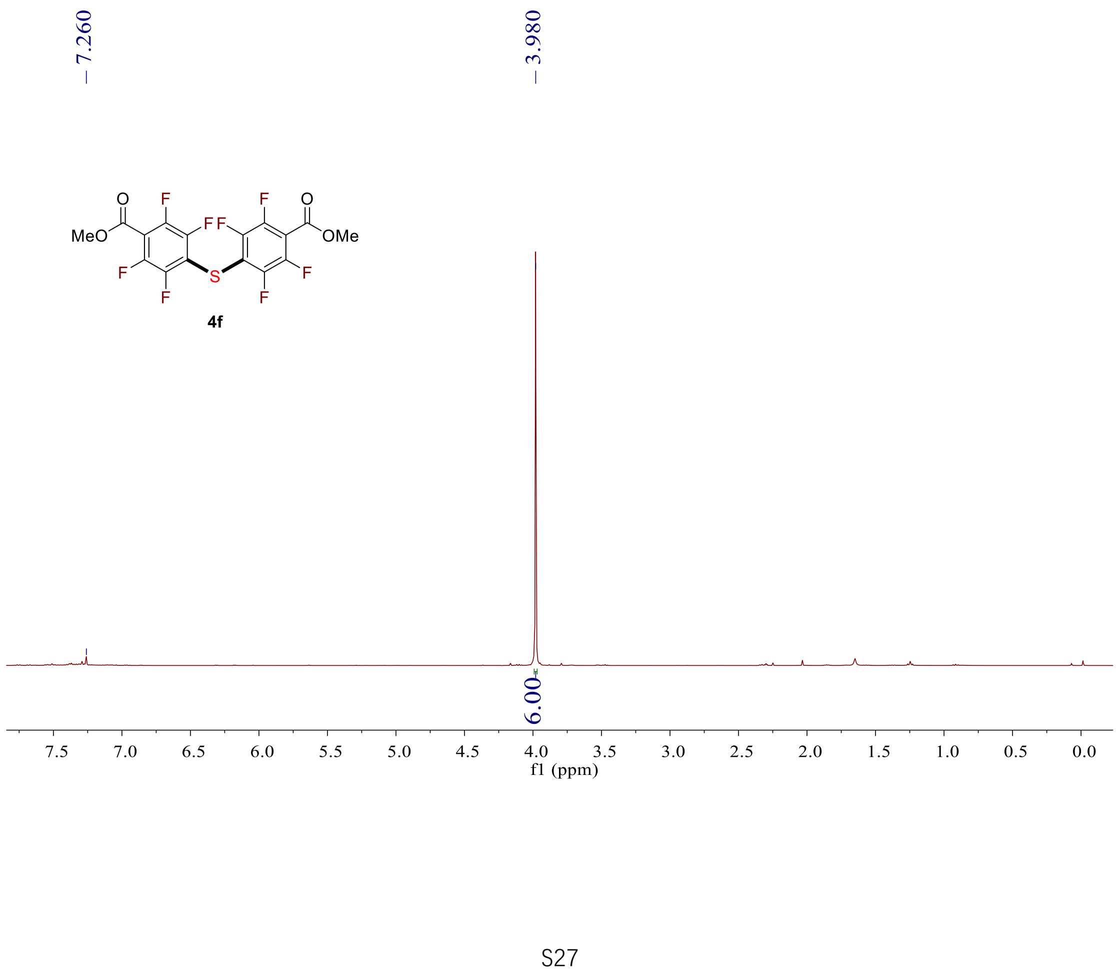
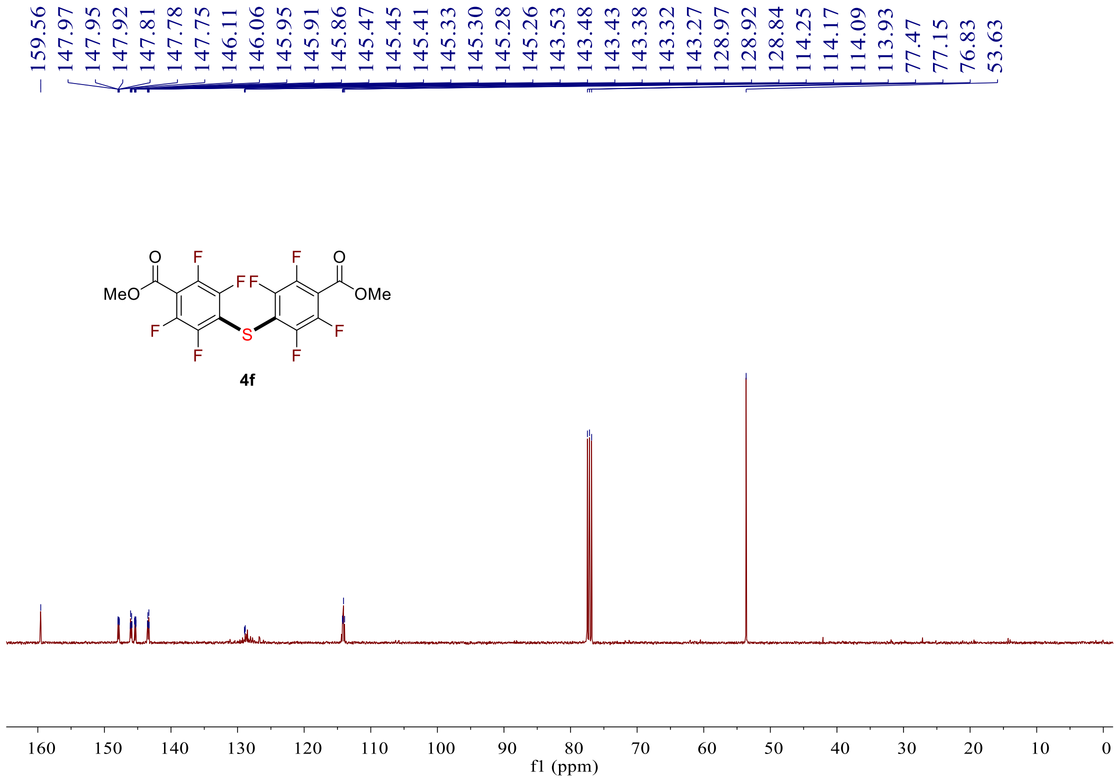
-139.159
-139.182
-139.214
-139.236
-139.247
-139.257
Ph2N
NPh2
O
F
S
4.03
4.00
-110 -115 -120 -125 -130 -135 -140 -145 -150 -155 -160 -165 -170 -175 -180 -185 -190 -195 -200 -205 -210 -215
f1 (ppm)

4e


7.260
3.980
MeO
OMe
O
F
S
6.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

4f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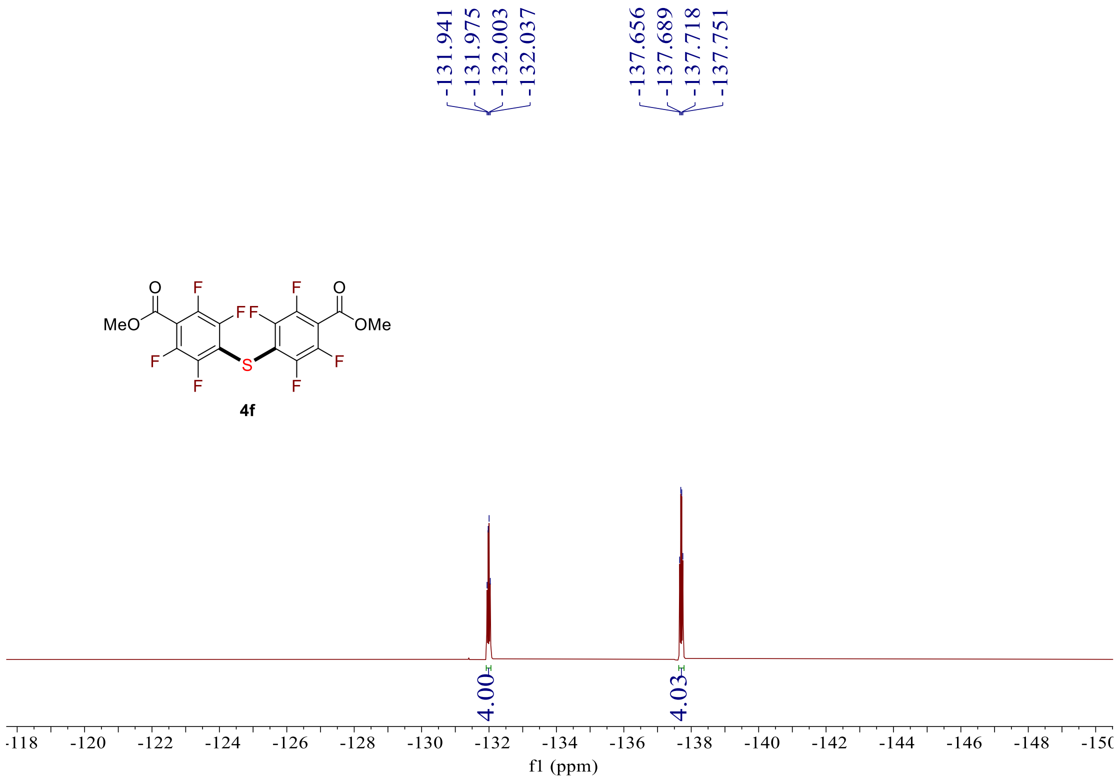
147.97
147.95
147.92
147.81
147.78
147.75
146.11
146.06
145.95
145.91
145.86
145.47
145.45
145.41
145.33
145.30
145.28
145.26
143.53
143.48
143.43
143.38
143.32
143.27
128.97
128.92
128.84
114.25
114.17
114.09
113.93
77.47
77.15
76.83
53.63
MeO
O
F
F
F
F
S
F
F
F
F
O
OMe
4f
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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-132.037
-137.656
-137.689
-137.718
-137.751
MeO
O
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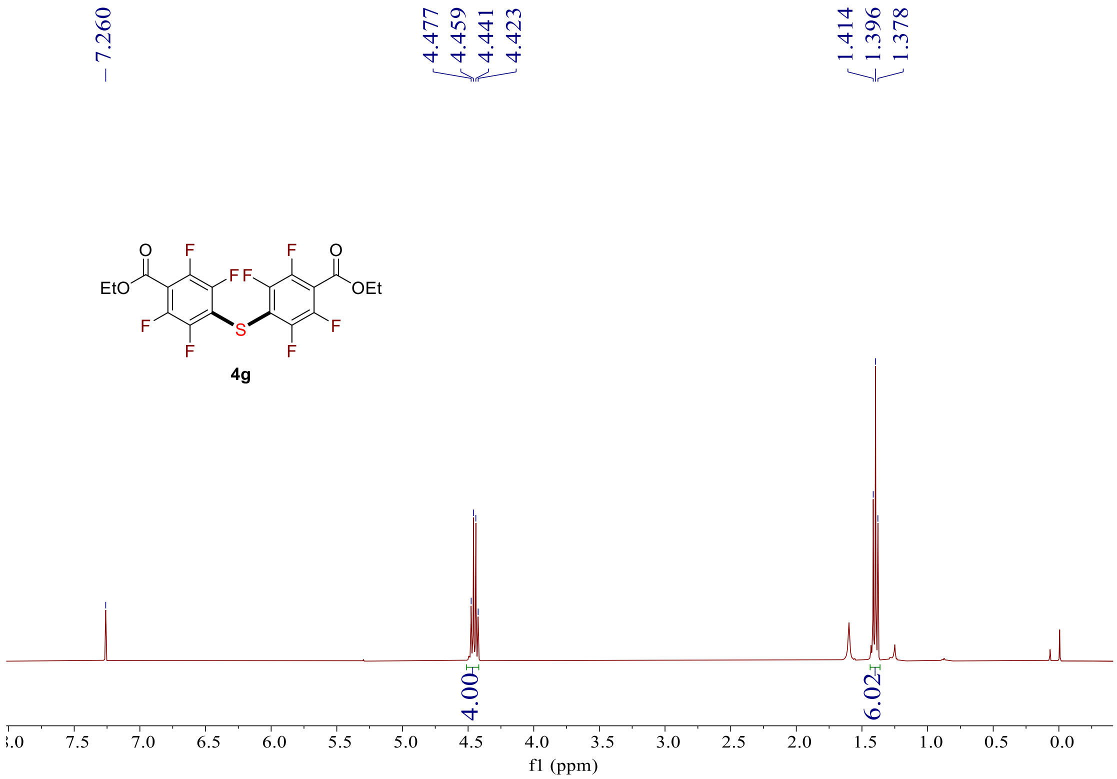
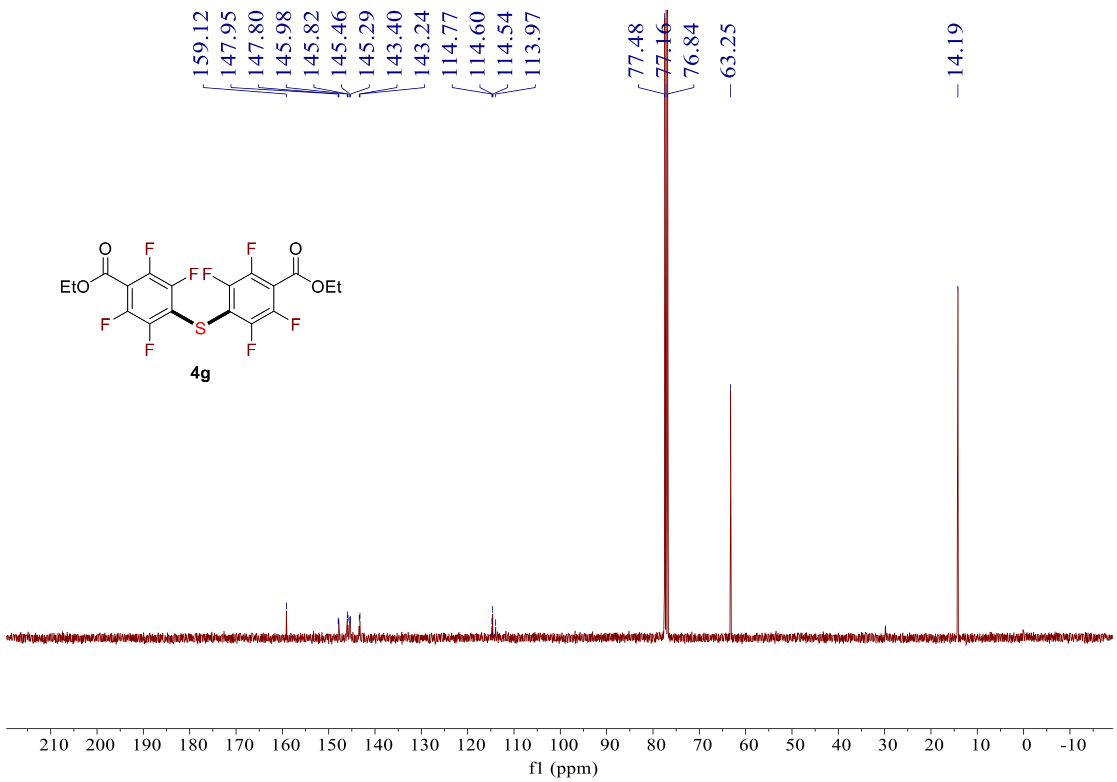
F
F
S
F
F
F
F
O
OMe
4f
4.00
4.03
-118 -120 -122 -124 -126 -128 -130 -132 -134 -136 -138 -140 -142 -144 -146 -148 -150
f1 (ppm)


7.260
4.477
4.459
4.441
4.423
1.414
1.396
1.378
EtO
OEt
O
F
S
4g
4.00
6.02
f1 (ppm)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147.95
147.80
145.98
145.82
145.46
145.29
143.40
143.24
114.77
114.60
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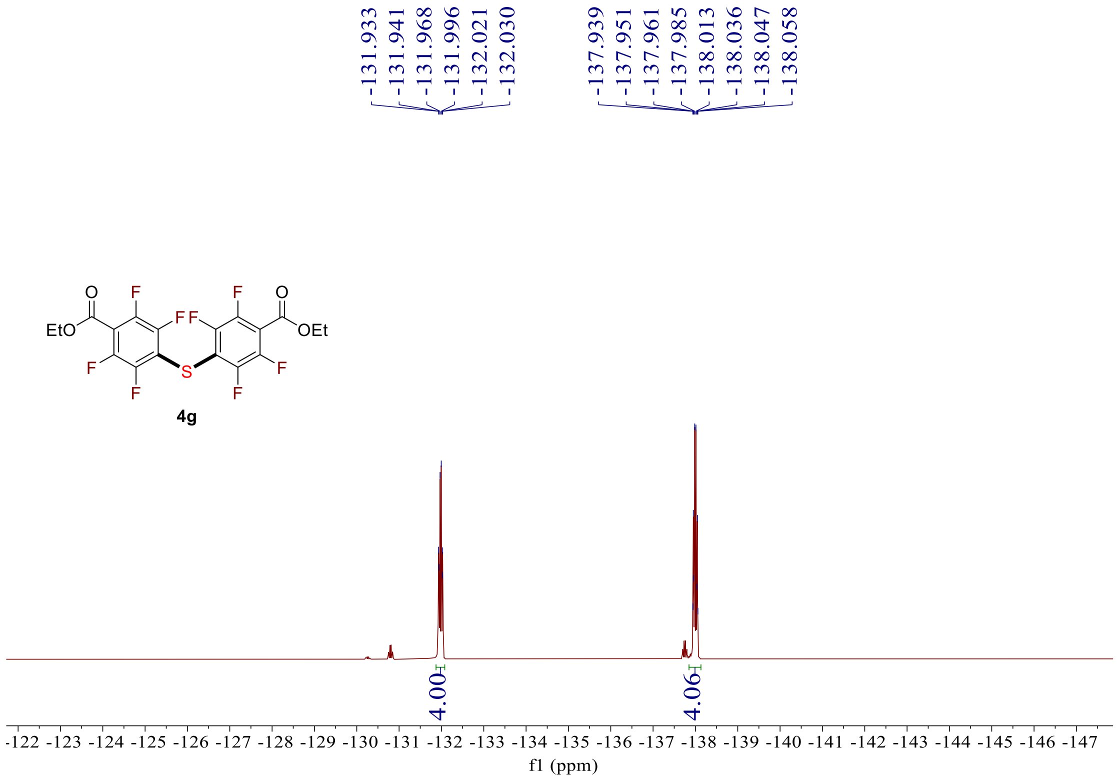
113.97
77.48
77.16
76.84
63.25
14.19
EtO
OEt
O
F
S
4g
f1 (ppm)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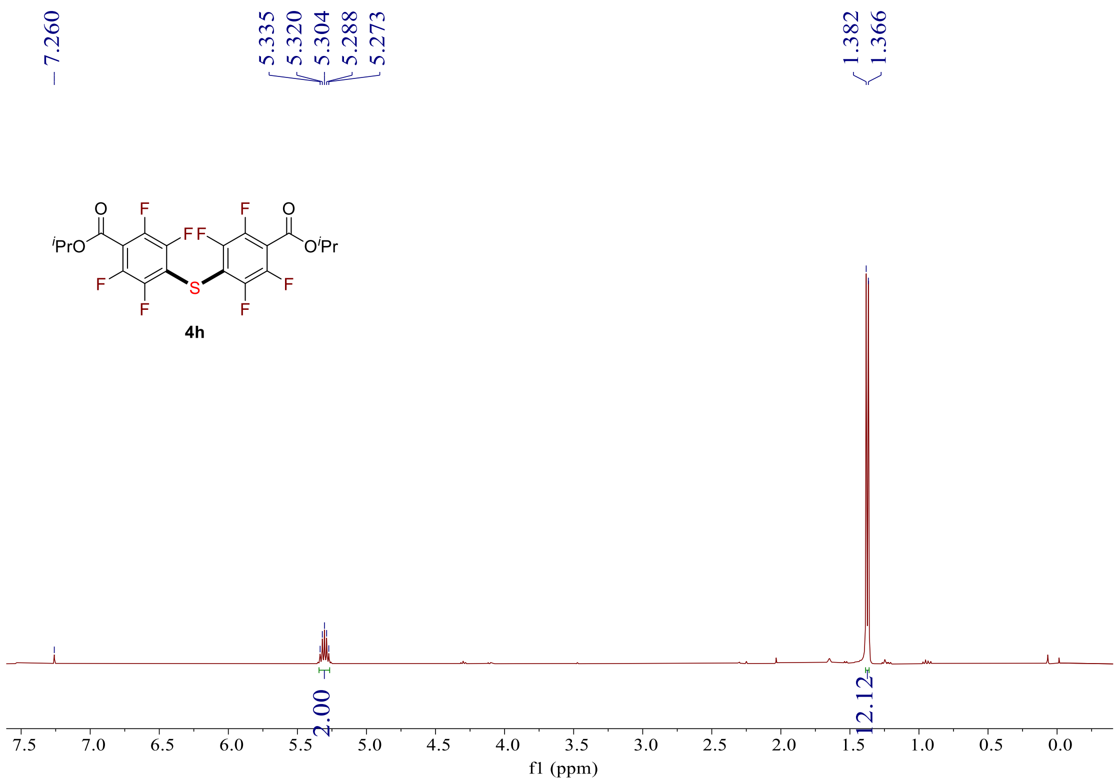
-131.968
-131.996
-132.021
-132.030
-137.939
-137.951
-137.961
-137.985
-138.013
-138.036
-138.047
-138.058
EtO
O
F
F
F
F
S
F
F
F
F
O
OEt
4g
4.00
4.06
-122 -123 -124 -125 -126 -127 -128 -129 -130 -131 -132 -133 -134 -135 -136 -137 -138 -139 -140 -141 -142 -143 -144 -145 -146 -147
f1 (ppm)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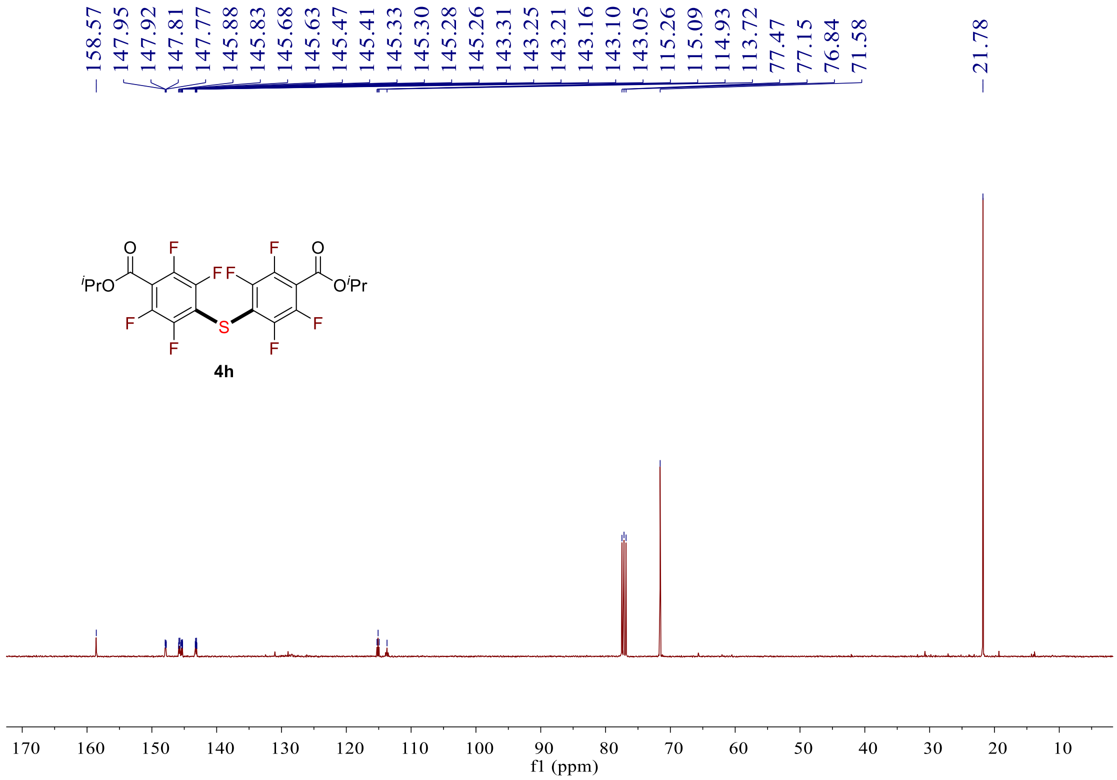
5.320
5.304
5.288
5.273
1.382
1.366
iPrO
O
F
F
F
F
S
F
F
F
F
O
OiPr
4h
2.00
12.12
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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145.68
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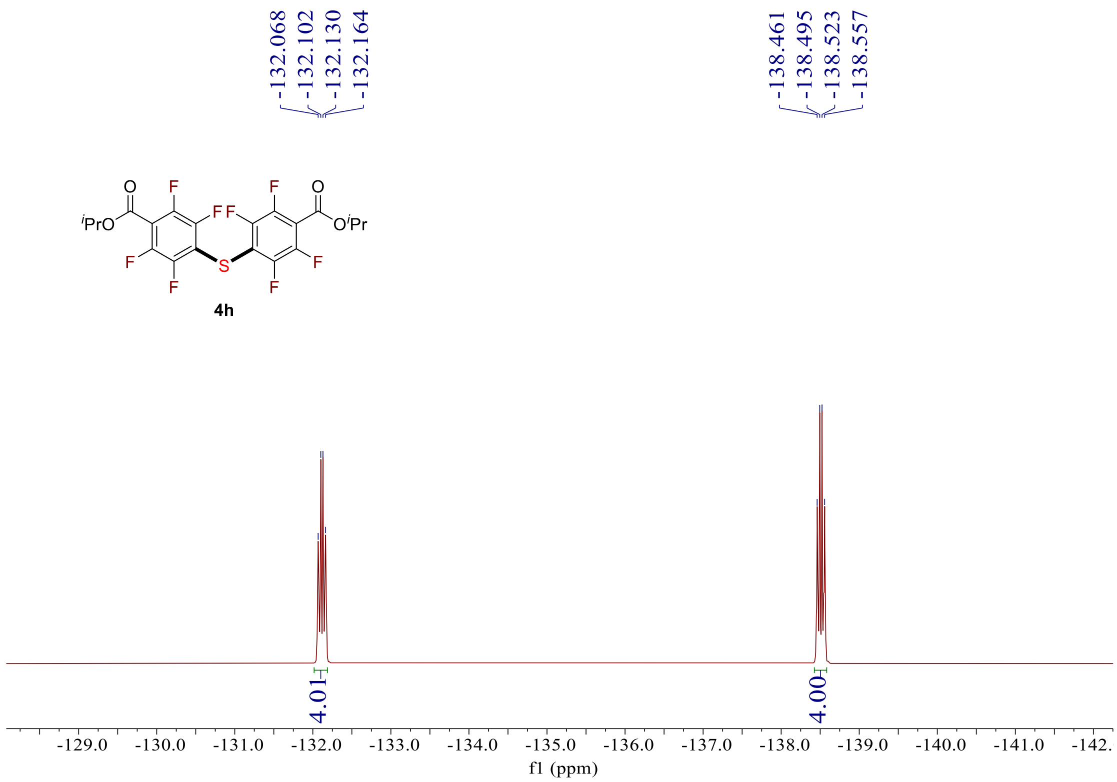
145.41
145.33
145.30
145.28
145.26
143.31
143.25
143.21
143.16
143.10
143.05
115.26
115.09
114.93
113.72
77.47
77.15
76.84
71.58
21.78
4h
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)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-132.130
-132.164
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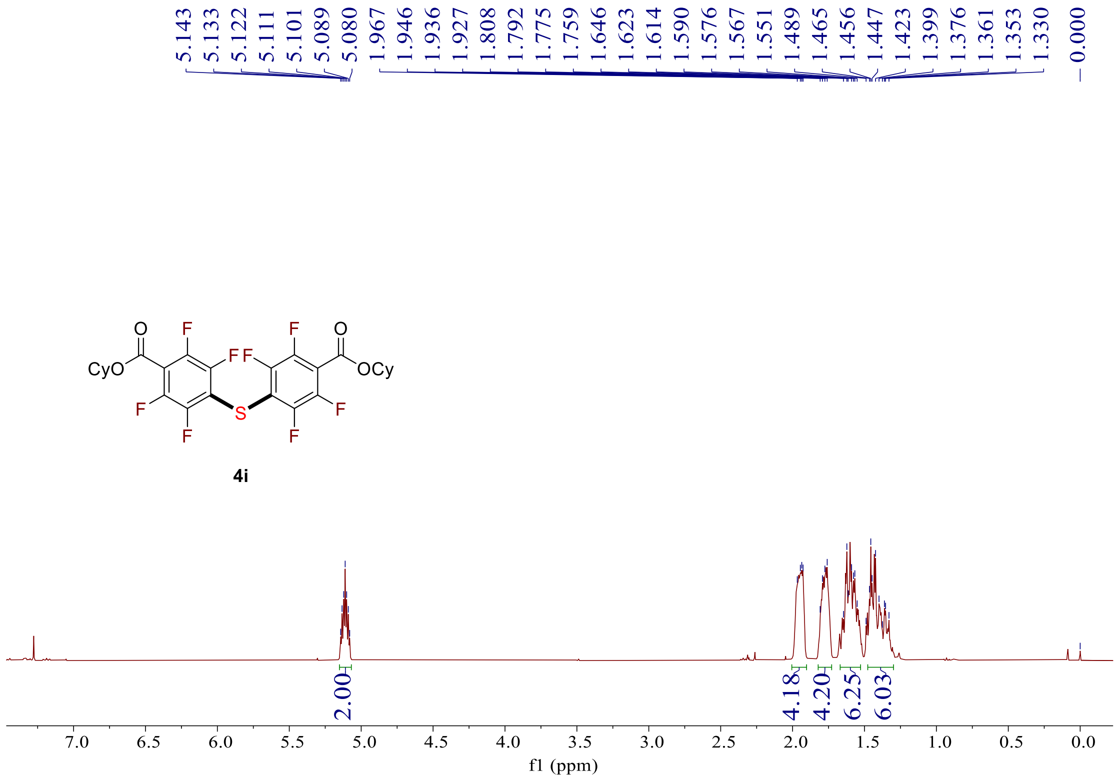
-138.495
-138.523
-138.557
4h
4.01
4.00
-129.0 -130.0 -131.0 -132.0 -133.0 -134.0 -135.0 -136.0 -137.0 -138.0 -139.0 -140.0 -141.0 -142.
f1 (ppm)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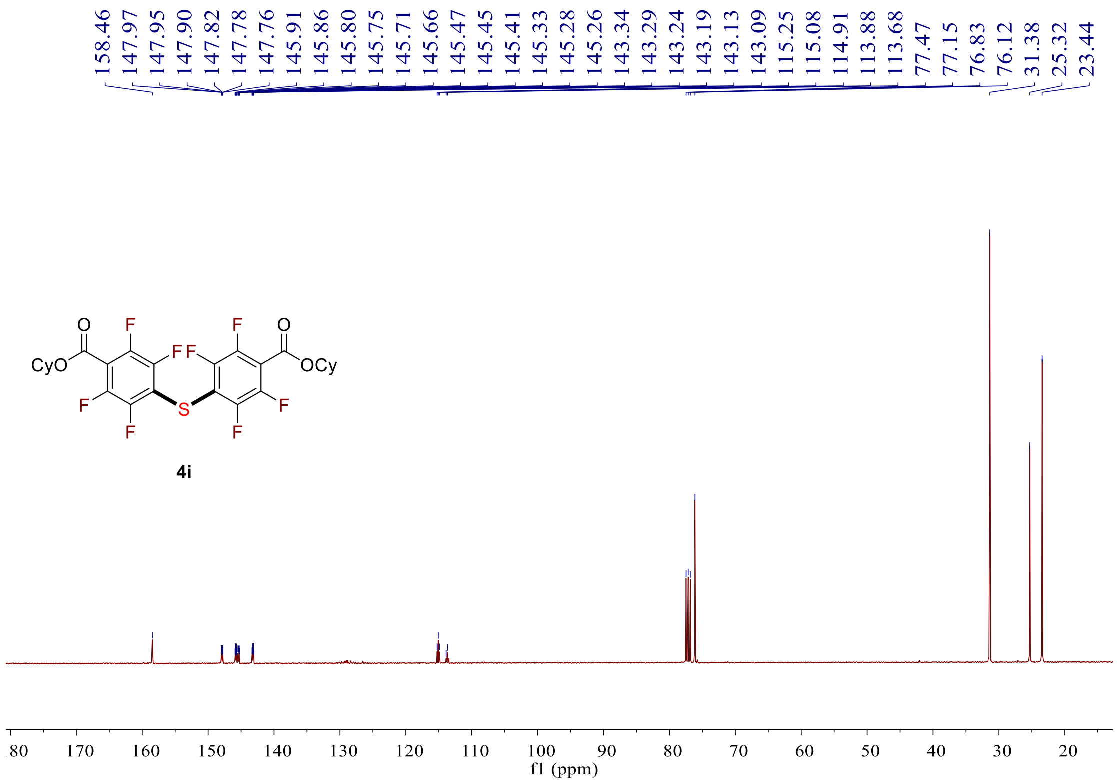
5.133
5.122
5.111
5.101
5.089
5.080
1.967
1.946
1.936
1.927
1.808
1.792
1.775
1.759
1.646
1.623
1.614
1.590
1.576
1.567
1.551
1.489
1.465
1.456
1.447
1.423
1.399
1.376
1.361
1.353
1.330
0.000
CyO
OCy
F
S
2.00
4.18
4.20
6.25
6.03
f1 (ppm)

4i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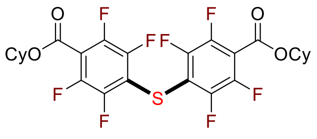
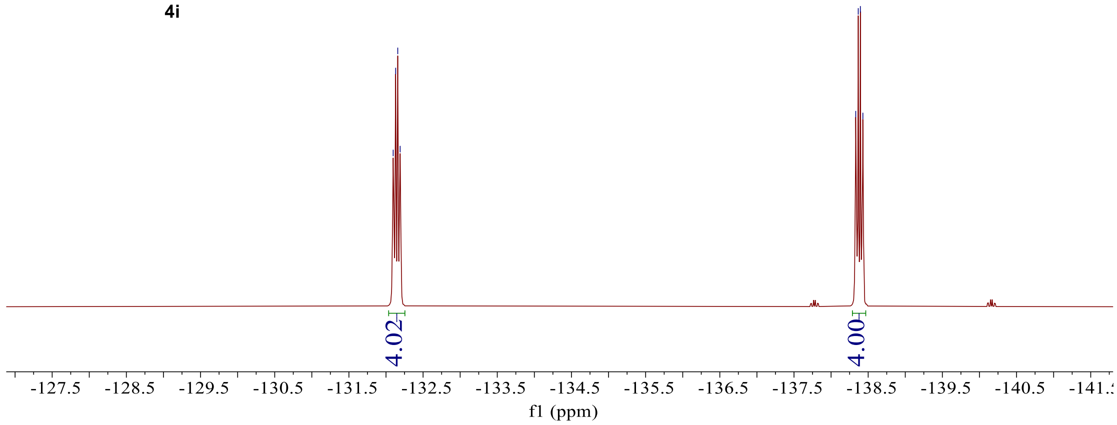
145.71
145.66
145.47
145.45
145.41
145.33
145.28
145.26
143.34
143.29
143.24
143.19
143.13
143.09
115.25
115.08
114.91
113.88
113.68
77.47
77.15
76.83
76.12
31.38
25.32
23.44
CyO
OCy
F
S
f1 (ppm)

4i

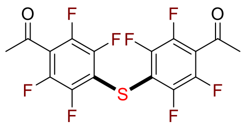


O
CyO
F
F
F
S
F
F
F
F
F
O
OCy

4i

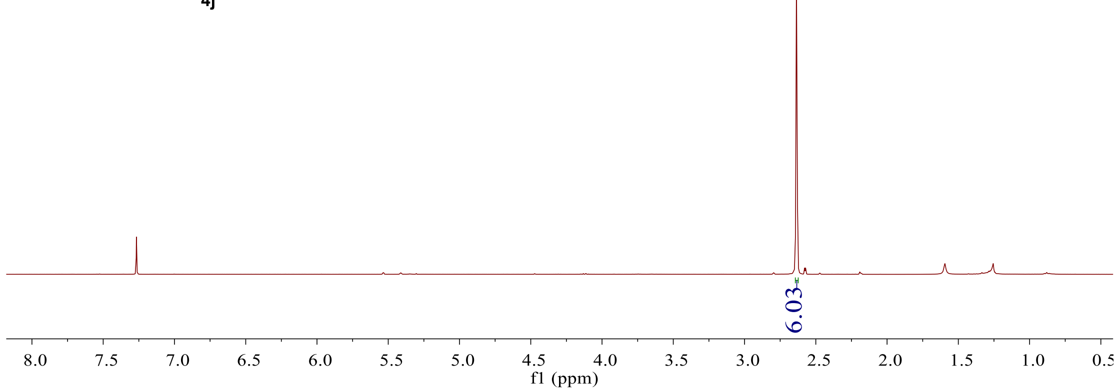


-132.097
-132.130
-132.158
-132.191
-138.332
-138.366
-138.395
-138.429
4.02
4.00
-127.5 -128.5 -129.5 -130.5 -131.5 -132.5 -133.5 -134.5 -135.5 -136.5 -137.5 -138.5 -139.5 -140.5
f1 (ppm)

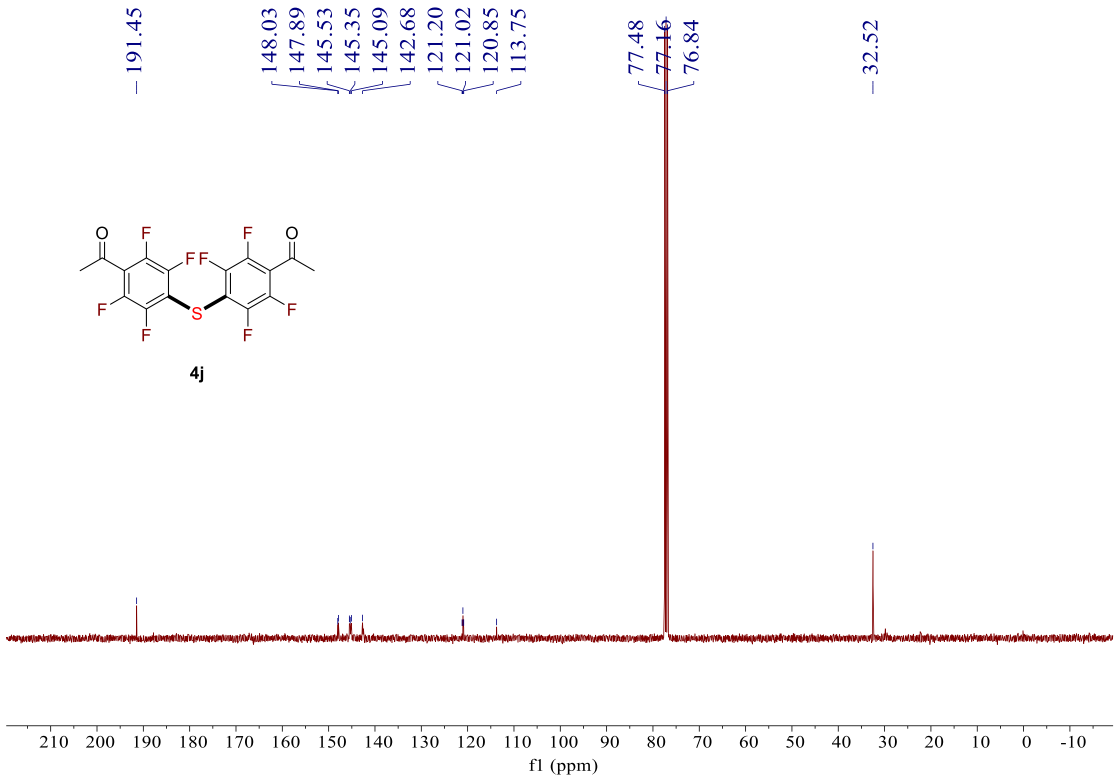


O
F
F
F
F
S
F
F
F
F
O

4j



2.636
6.03
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)


191.45
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147.89
145.53
145.35
145.09
142.68
121.20
121.02
120.85
113.75
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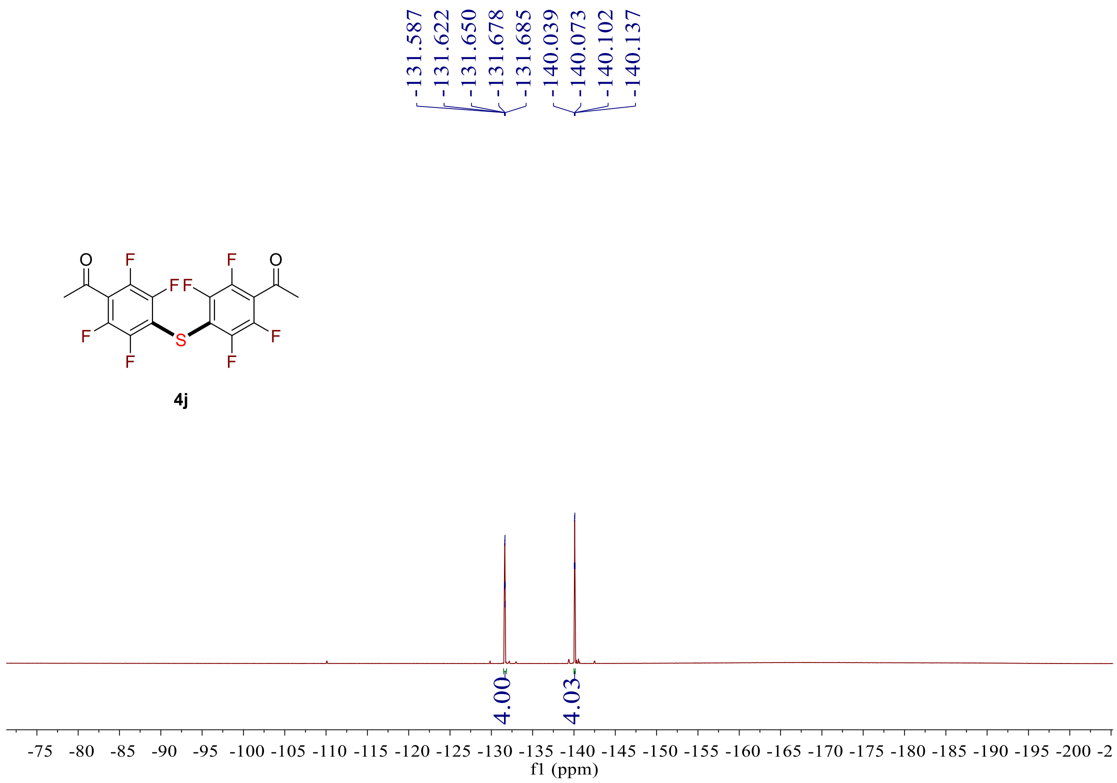
77.16
76.84
32.52
4j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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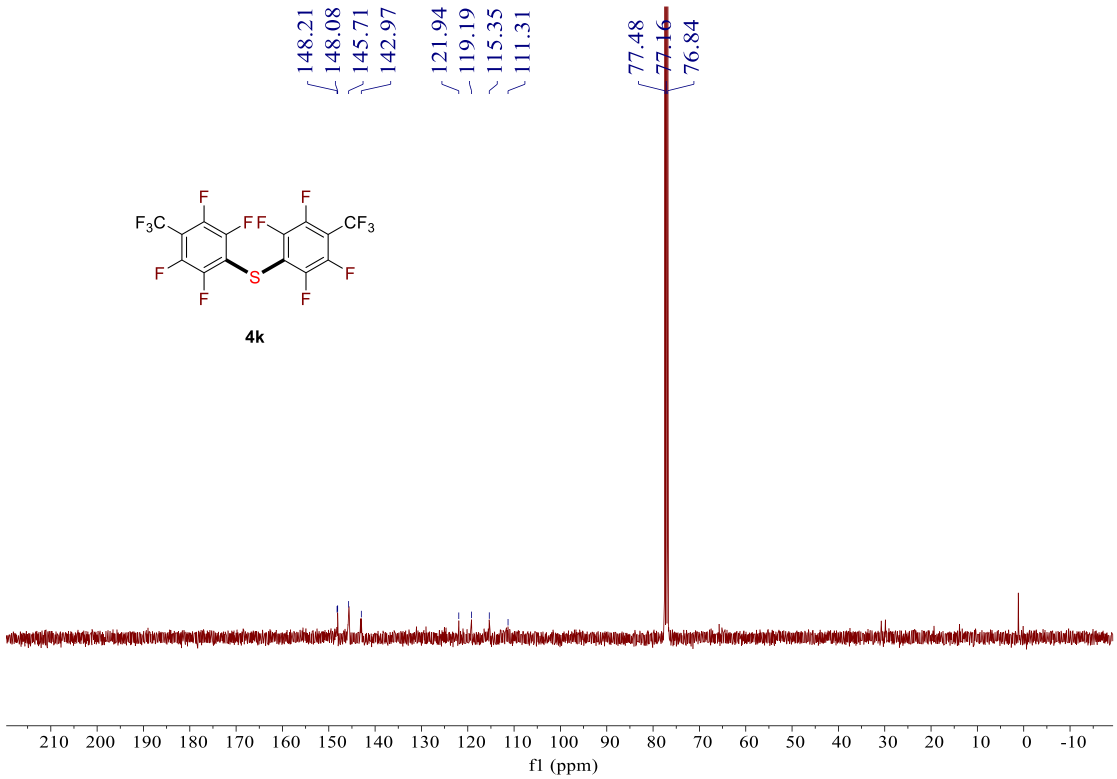
-131.650
-131.678
-131.685
-140.039
-140.073
-140.102
-140.137
4j
4.00
4.03
-75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -150 -155 -160 -165 -170 -175 -180 -185 -190 -195 -200
f1 (ppm)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148.08
145.71
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121.94
119.19
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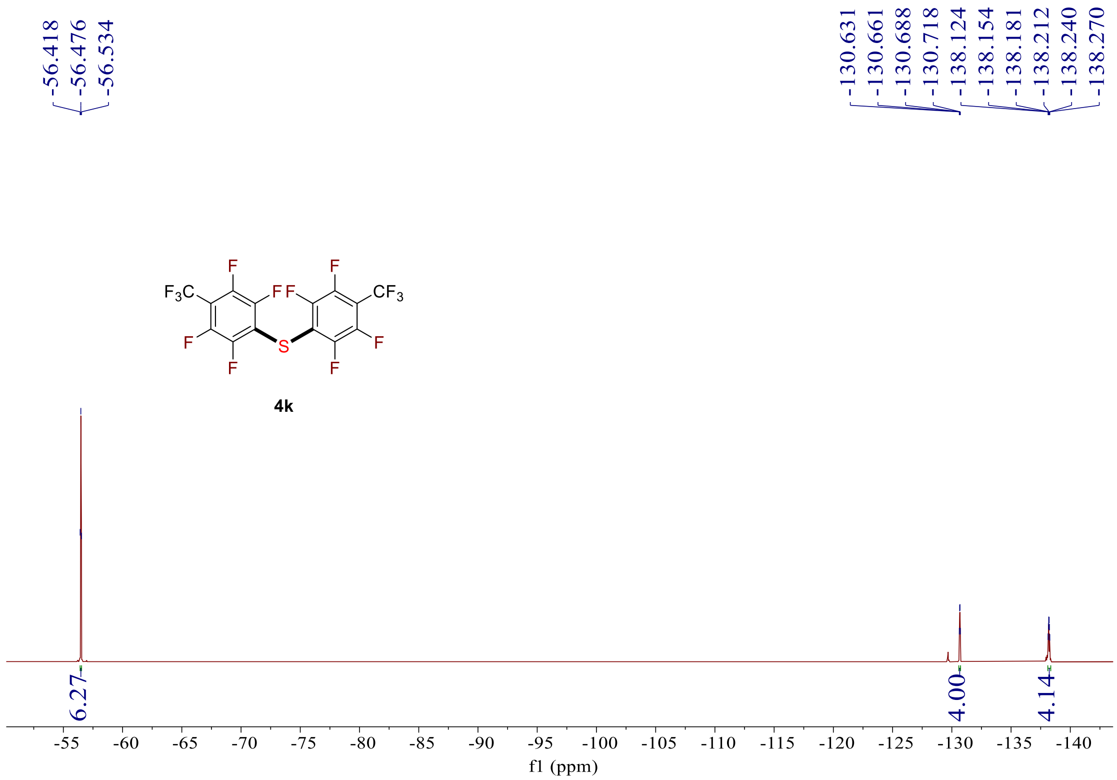
111.31
77.48
77.16
76.84
4k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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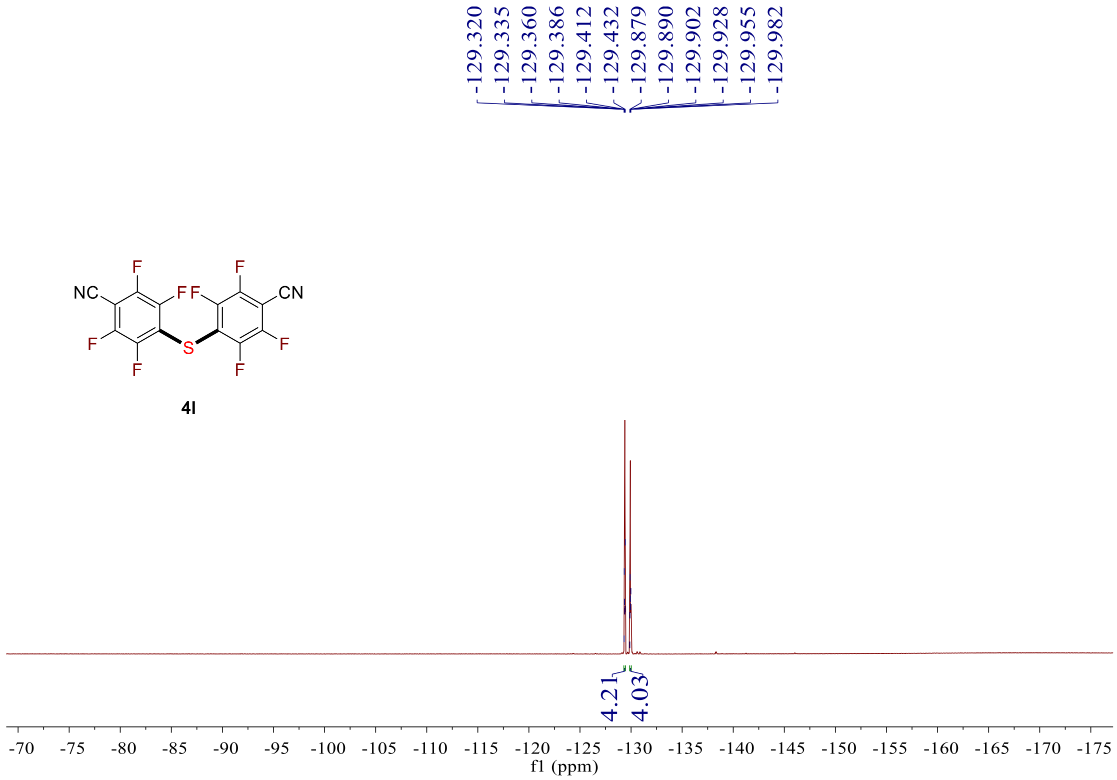
-56.534
-130.631
-130.661
-130.688
-130.718
-138.124
-138.154
-138.181
-138.212
-138.240
-138.270
4k
6.27
4.00
4.14
-55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140
f1 (ppm)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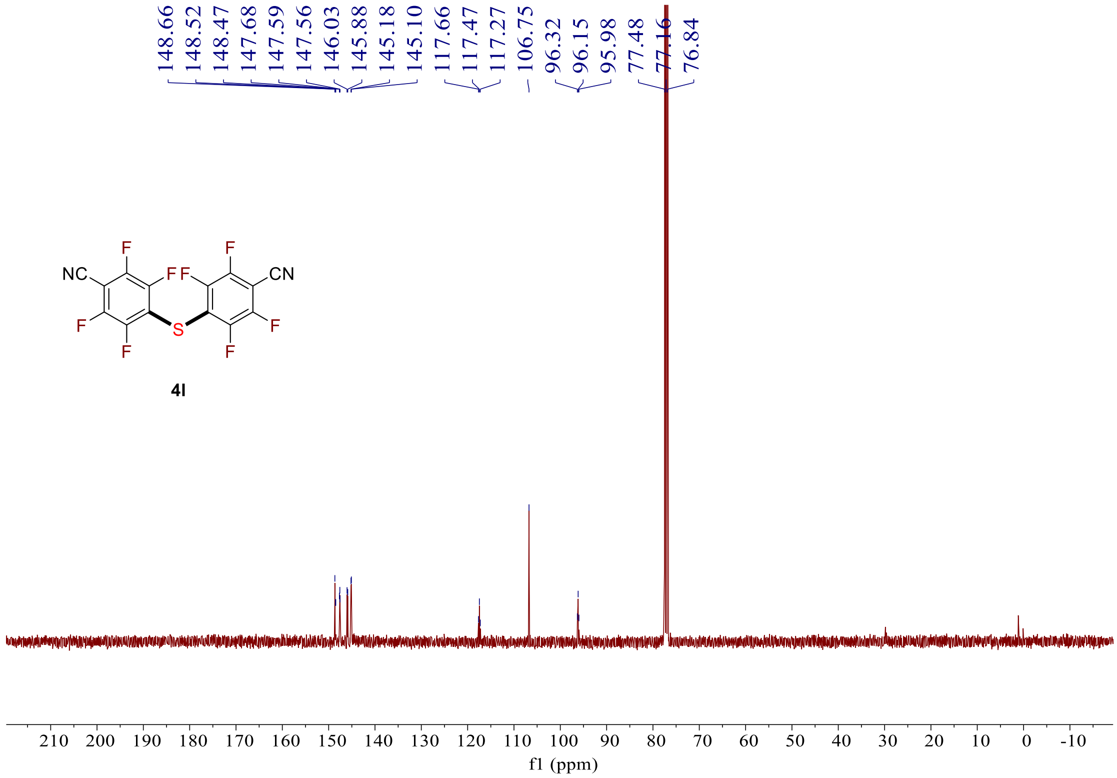
-129.432
-129.879
-129.890
-129.902
-129.928
-129.955
-129.982
NC
F
F
F
F
S
F
F
CN
F
F
4l
4.21
4.03
-70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -150 -155 -160 -165 -170 -175
f1 (ppm)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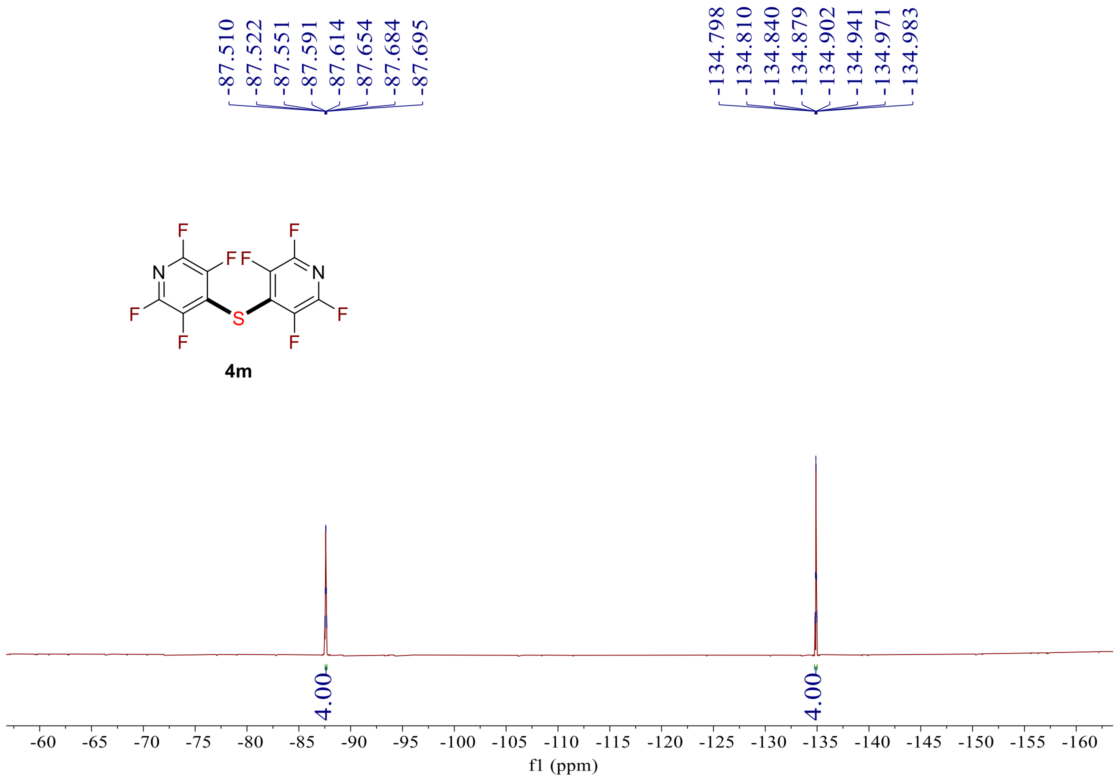
145.18
145.10
117.66
117.47
117.27
106.75
96.32
96.15
95.98
77.48
77.16
76.84
NC
F
F
F
F
S
F
F
CN
F
F
4l
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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-87.684
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-134.798
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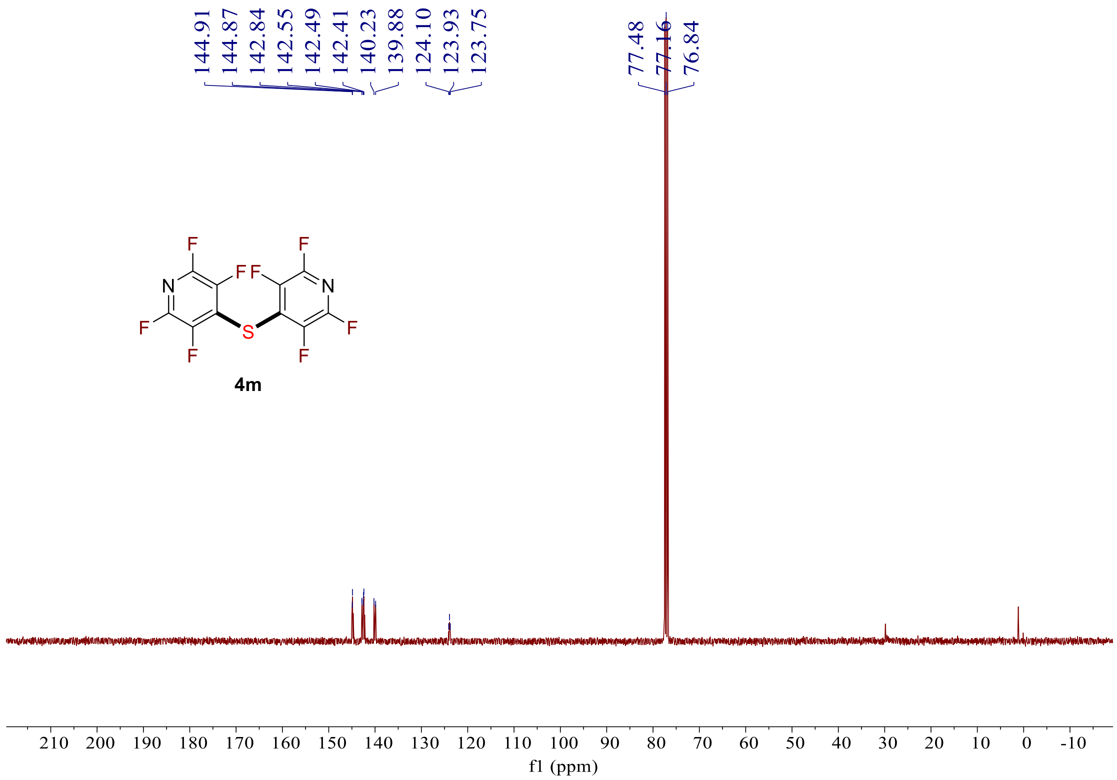
-134.840
-134.879
-134.902
-134.941
-134.971
-134.983
F
F
F
N
F
S
F
F
F
F
N
F
4m
4.00
4.00
-60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -150 -155 -160
f1 (ppm)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123.93
123.75
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76.84
F
F
F
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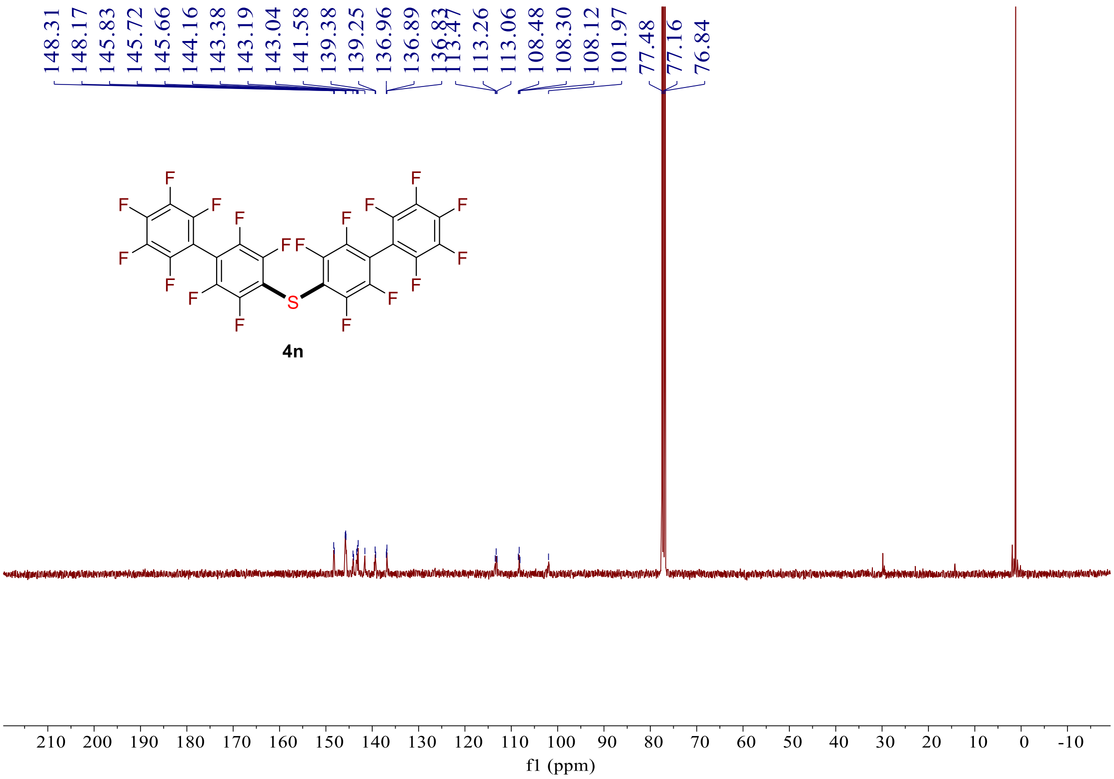
f1 (ppm)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143.38
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143.04
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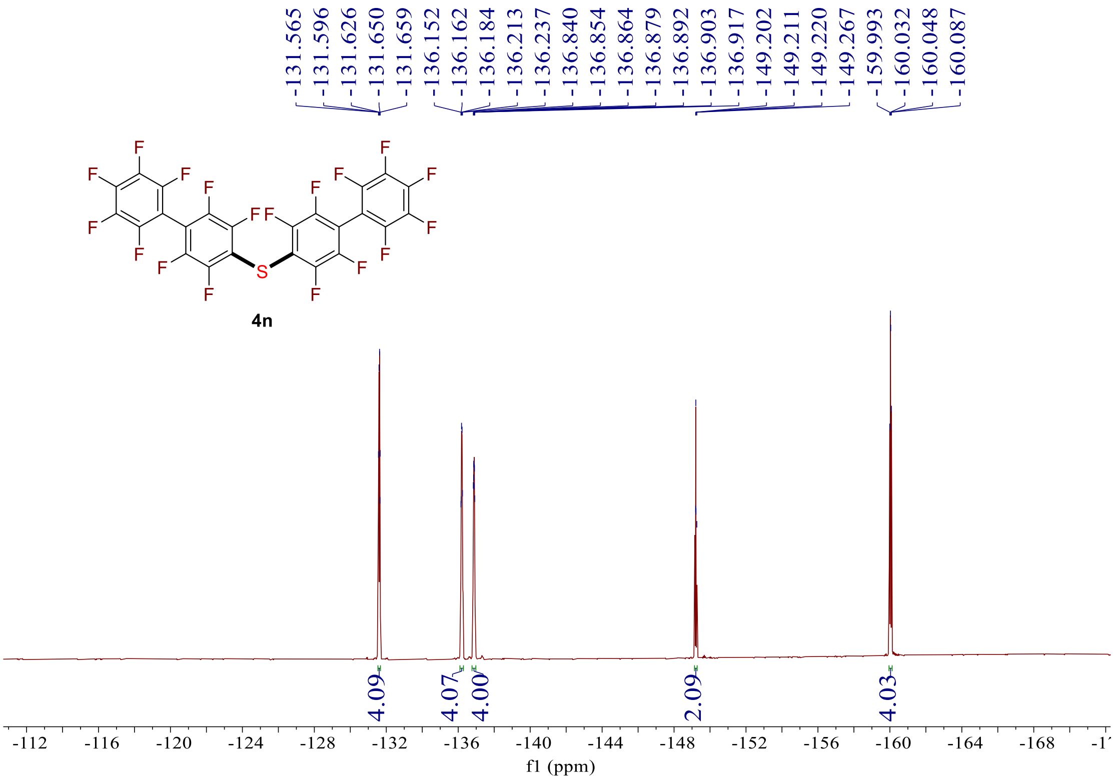
139.38
139.25
136.96
136.89
136.83
113.47
113.26
113.06
108.48
108.30
108.12
101.97
77.48
77.16
76.84
4n
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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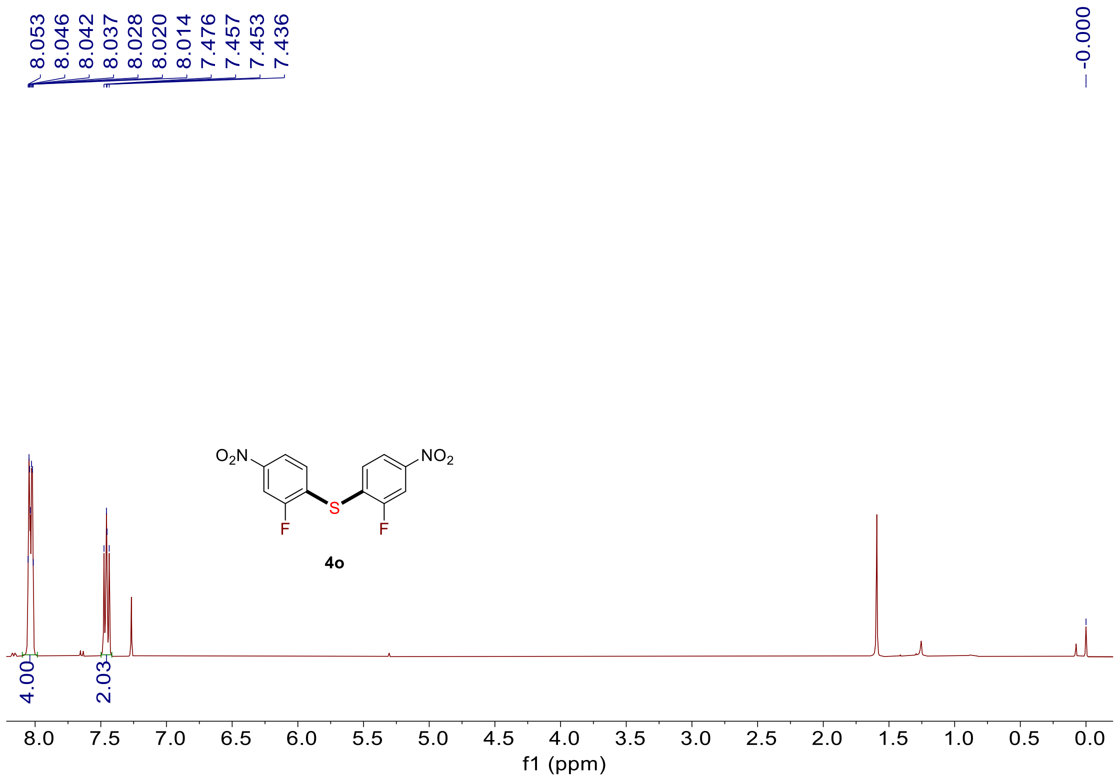
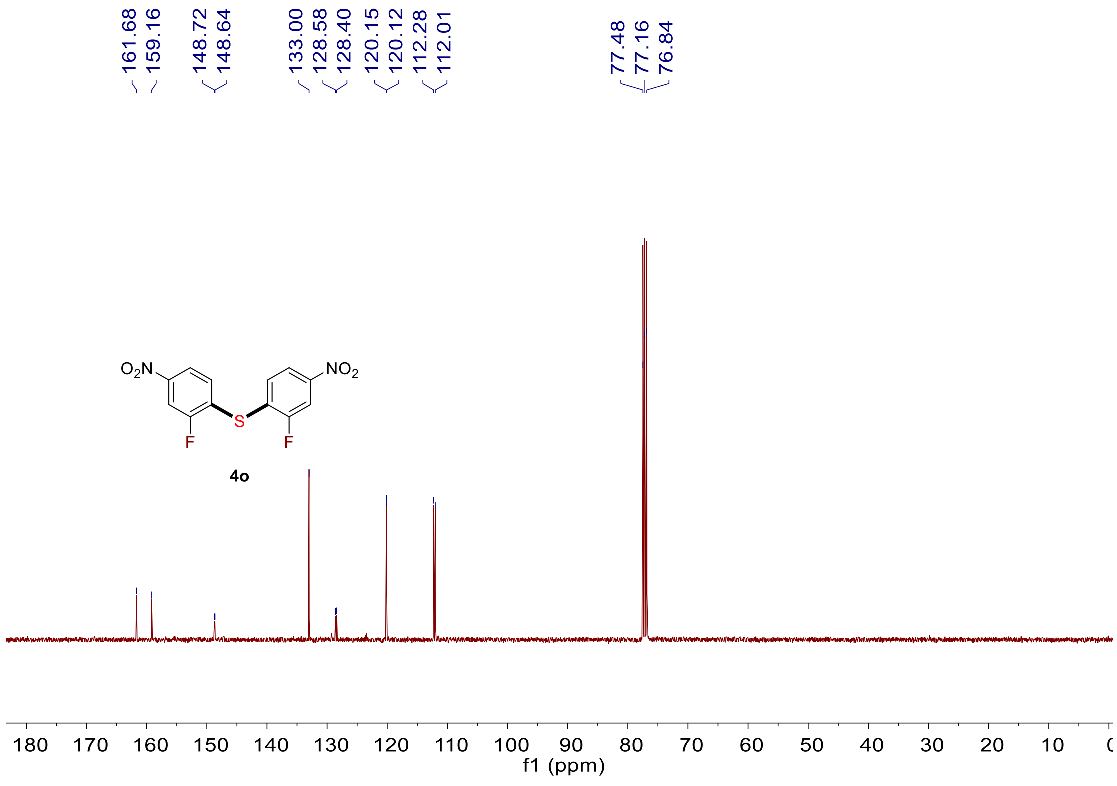
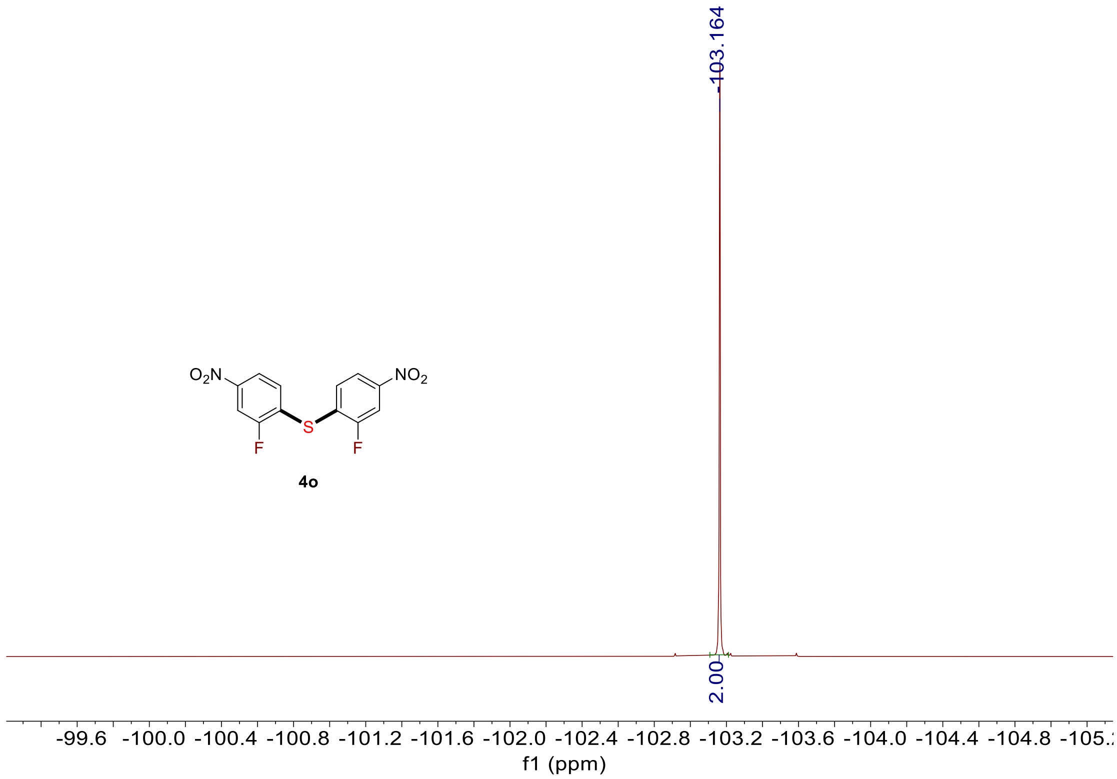
-160.048
-160.087
4n
4.09
4.07
4.00
2.09
4.03
-112 -116 -120 -124 -128 -132 -136 -140 -144 -148 -152 -156 -160 -164 -168
f1 (ppm)



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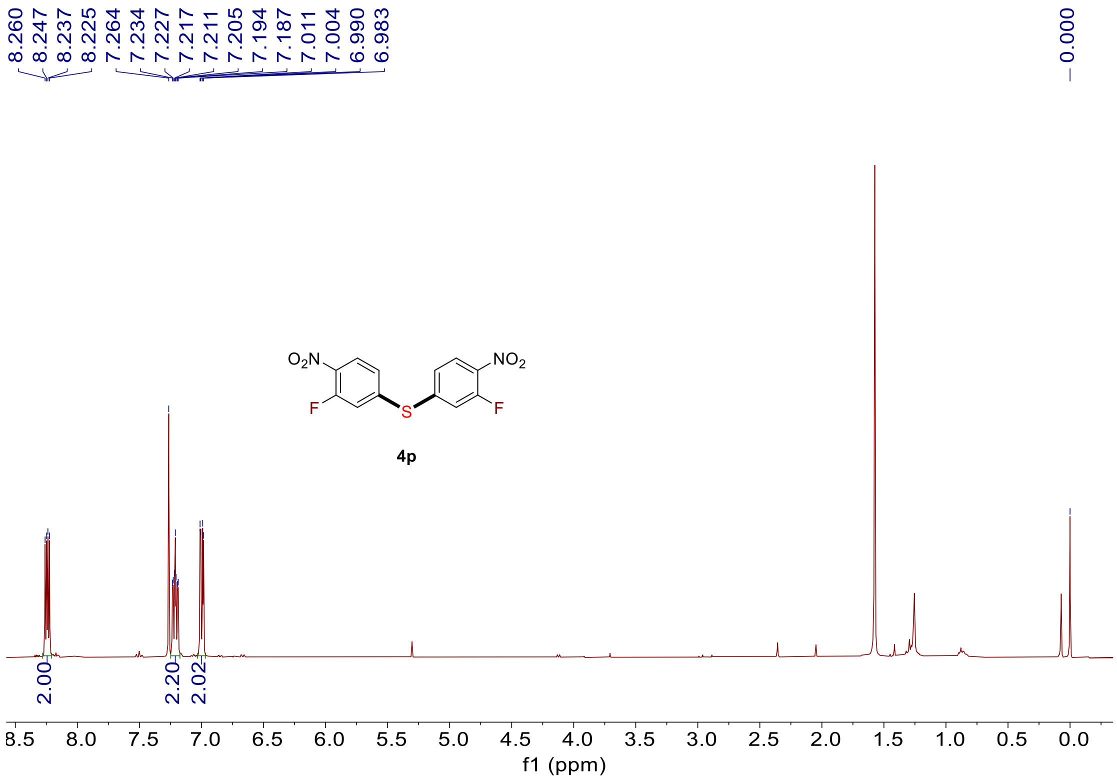
NO2
S
F
F
4o
2.00
-99.6 -100.0 -100.4 -100.8 -101.2 -101.6 -102.0 -102.4 -102.8 -103.2 -103.6 -104.0 -104.4 -104.8
f1 (ppm)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8.237
8.225
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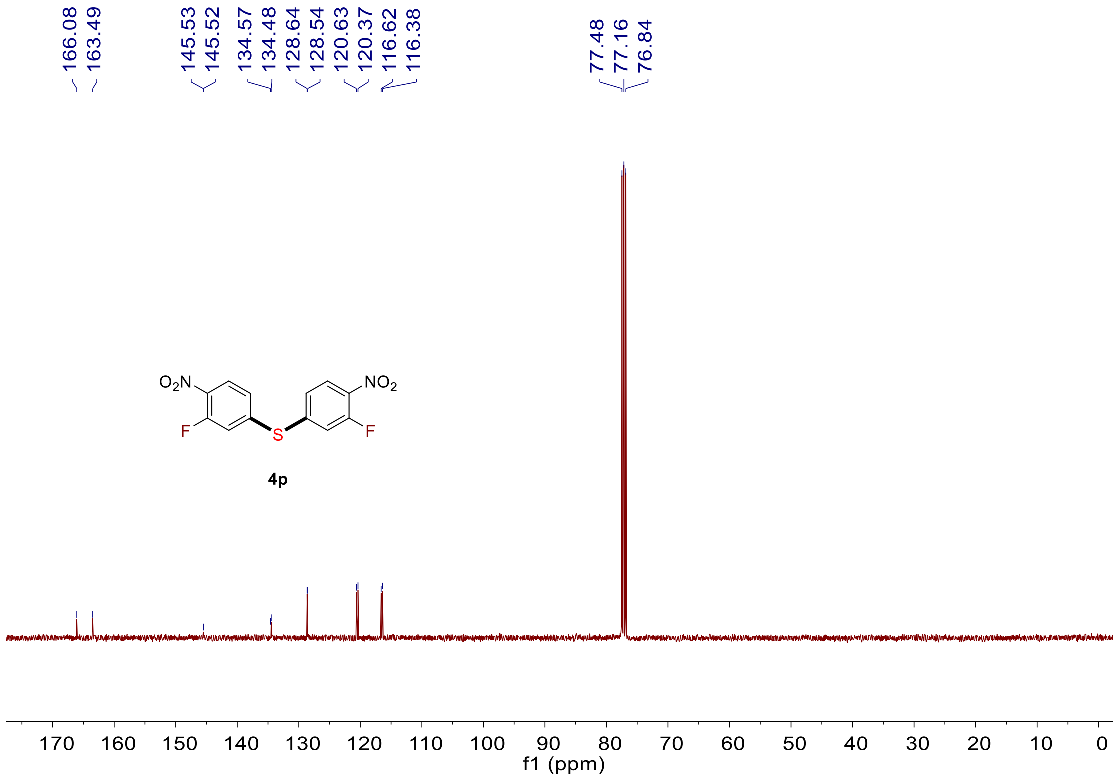
7.234
7.227
7.217
7.211
7.205
7.194
7.187
7.011
7.004
6.990
6.983
0.000
O2N
NO2
F
S
F
4p
2.00
2.20
2.02
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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163.49
145.53
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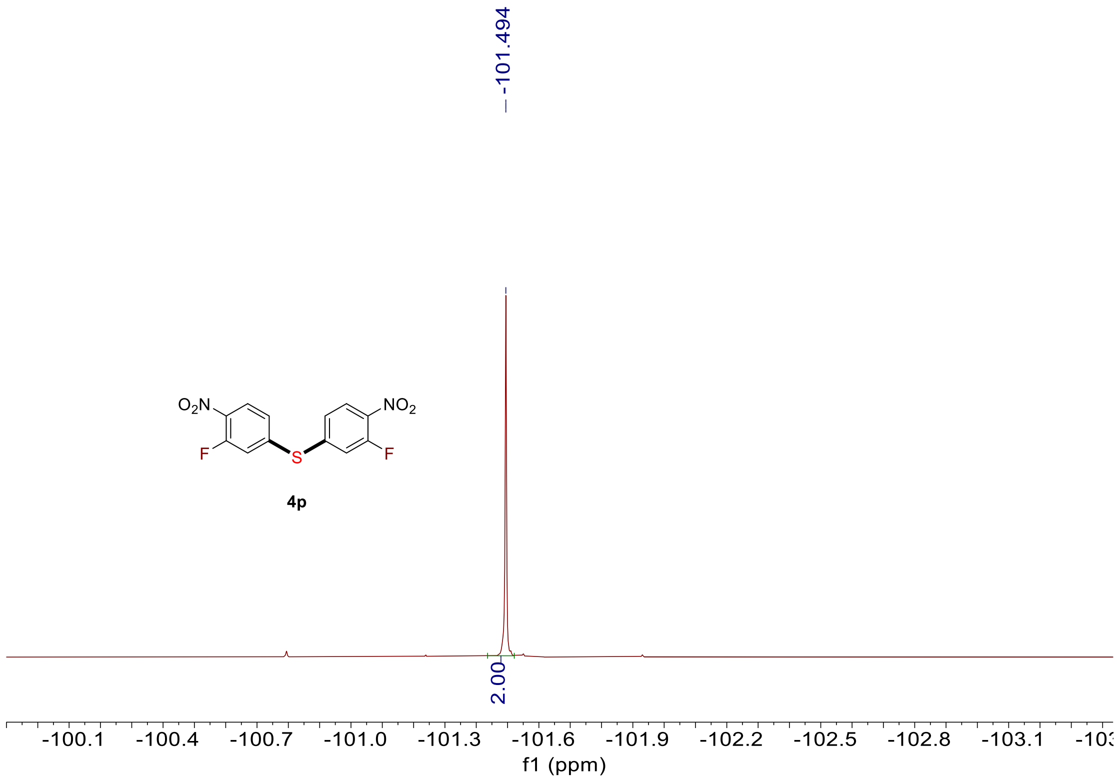
134.57
134.48
128.64
128.54
120.63
120.37
116.62
116.38
77.48
77.16
76.84
O2N
NO2
F
S
F
4p
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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NO2
F
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F
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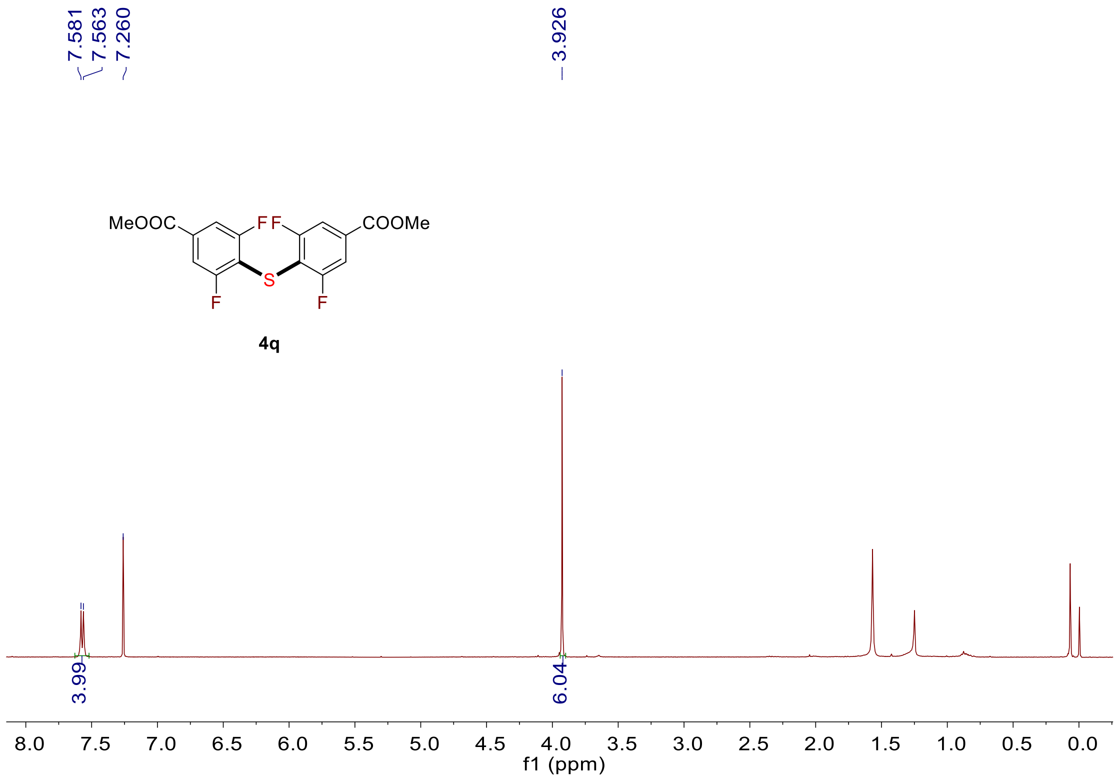
f1 (ppm)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7.563
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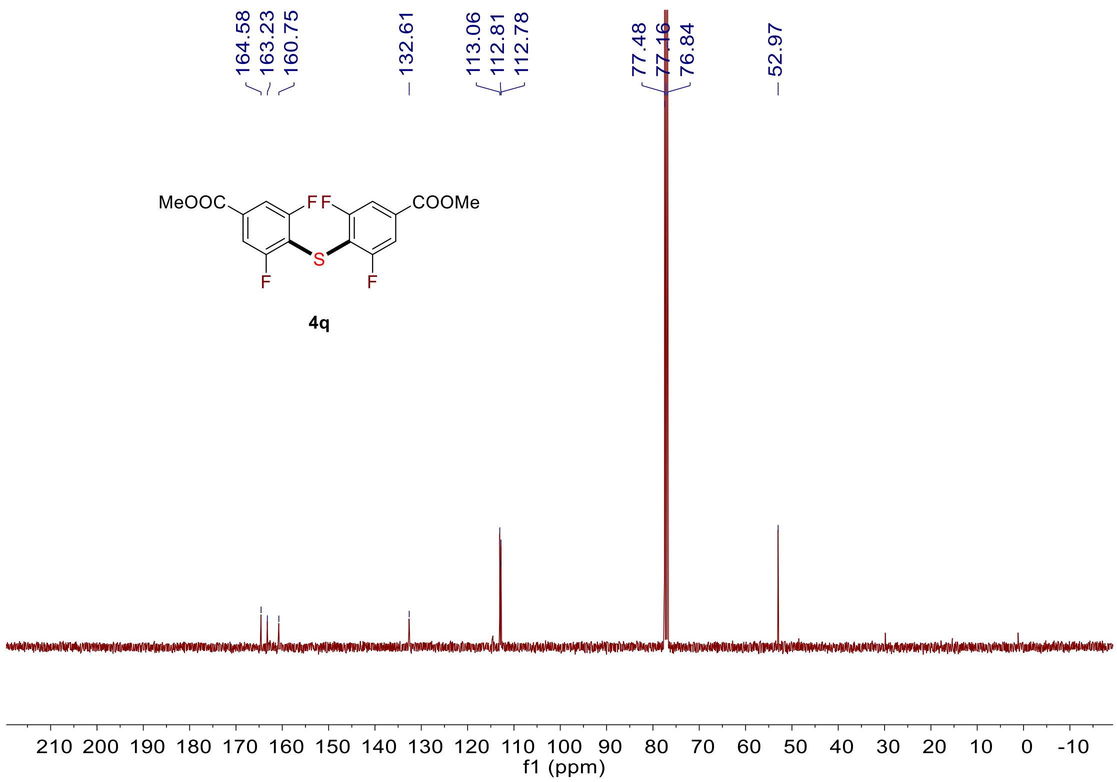
3.926
MeOOC
COOMe
F
S
4q
3.99
6.04
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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163.23
160.75
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113.06
112.81
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77.48
77.16
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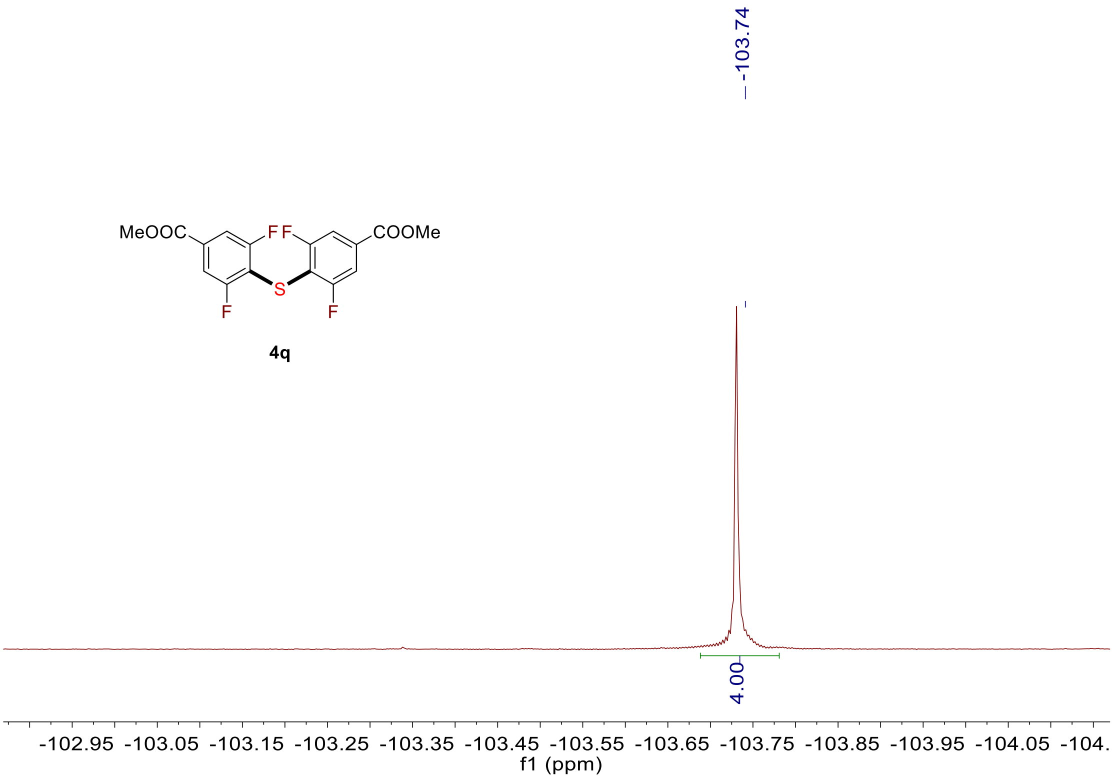
52.97
MeOOC
COOMe
F
S
4q
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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4.00
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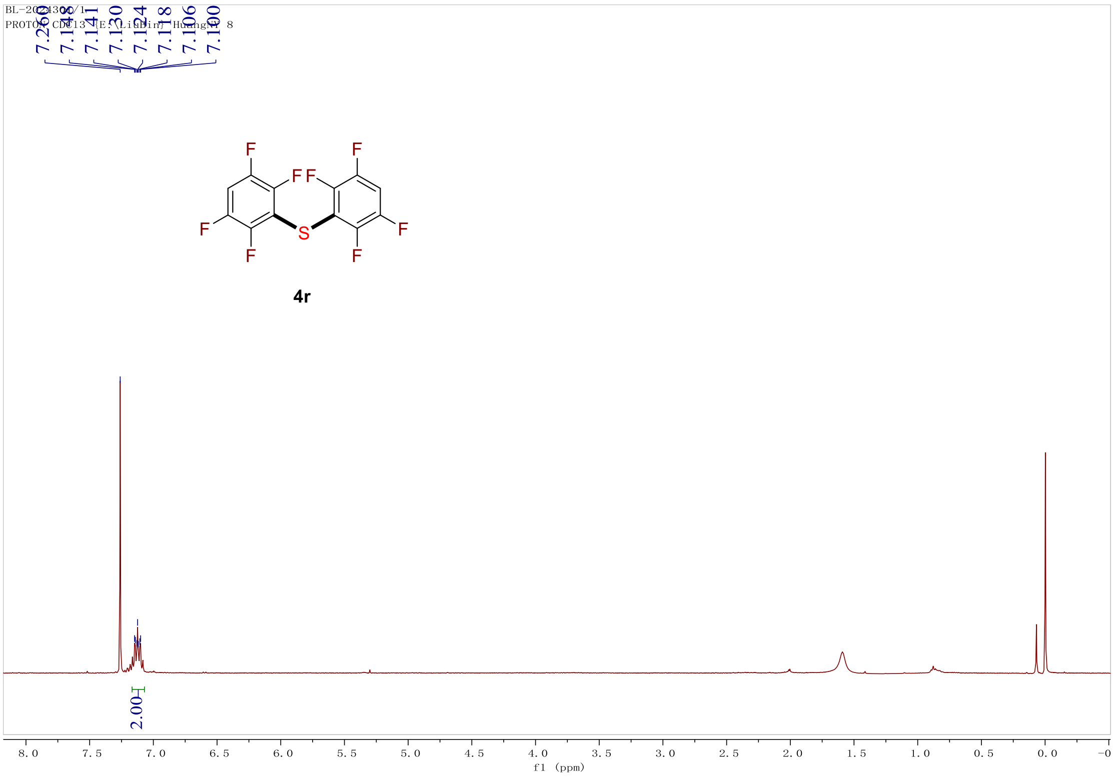
f1 (ppm)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7.148
7.141
7.130
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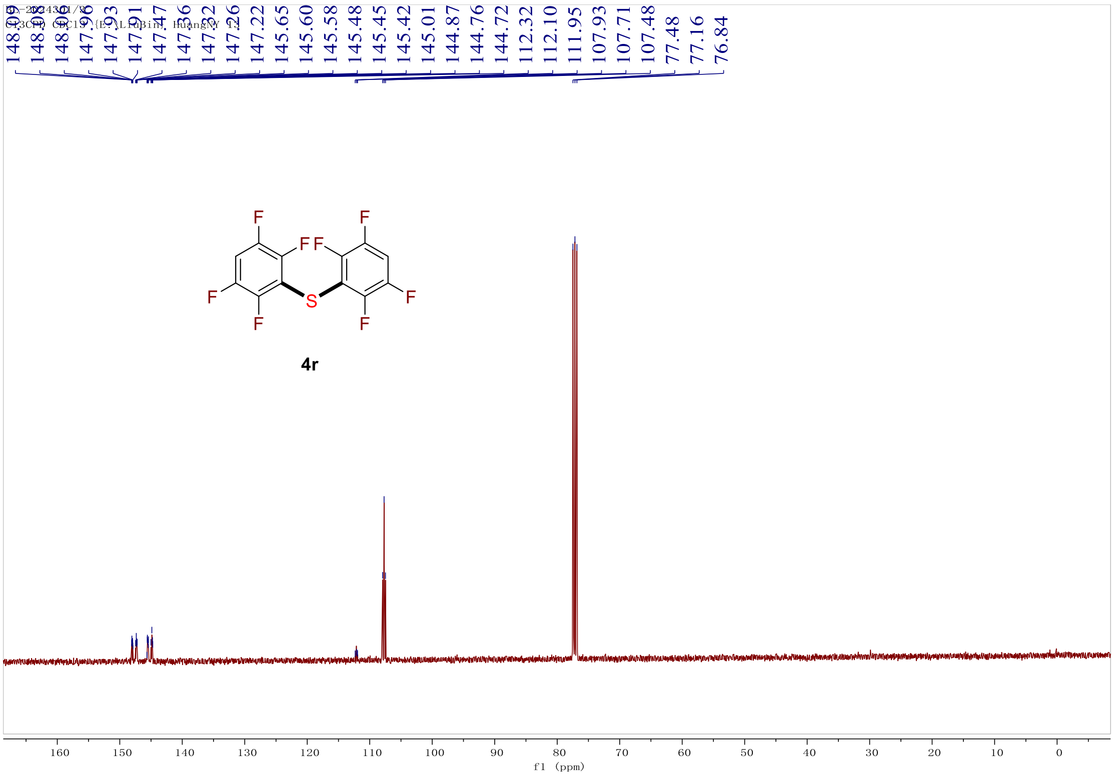
7.118
7.106
7.100
F
S
4r
2.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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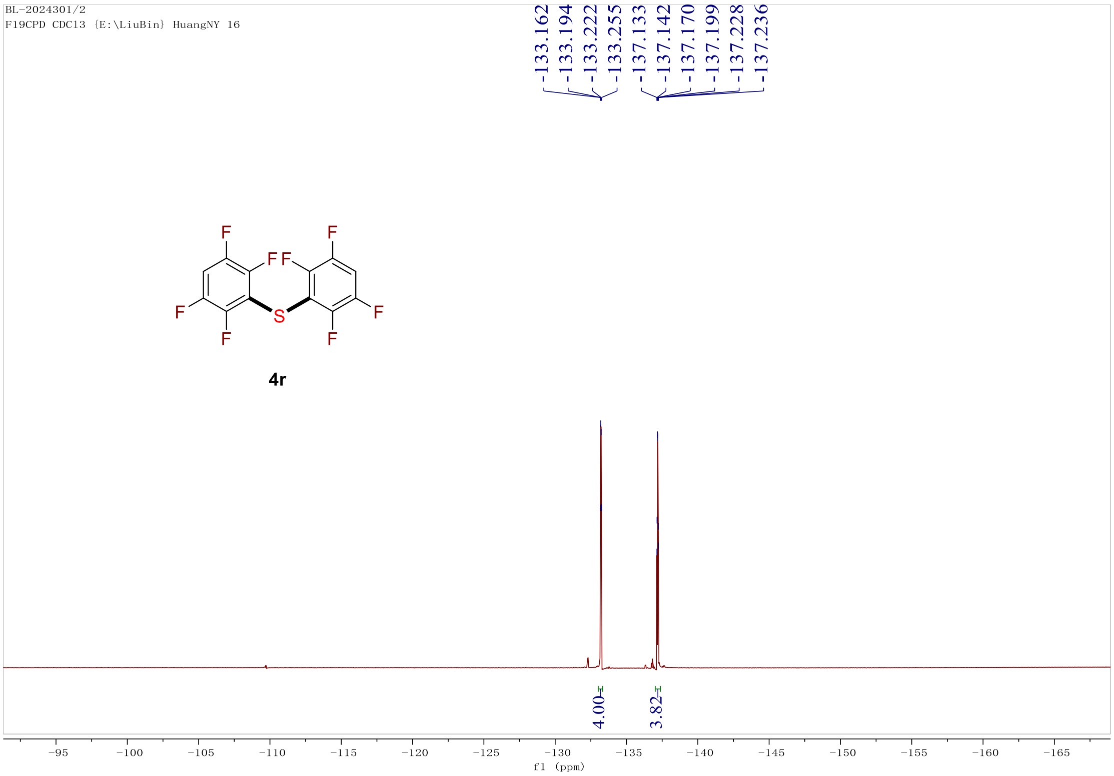
147.22
145.65
145.60
145.58
145.48
145.45
145.42
145.01
144.87
144.76
144.72
112.32
112.10
111.95
107.93
107.71
107.48
77.48
77.16
76.84
4r
f1 (ppm)


BL-2024301/2
F19CPD CDCl3 {E:\LiuBin} HuangNY 16
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-133.194
-133.222
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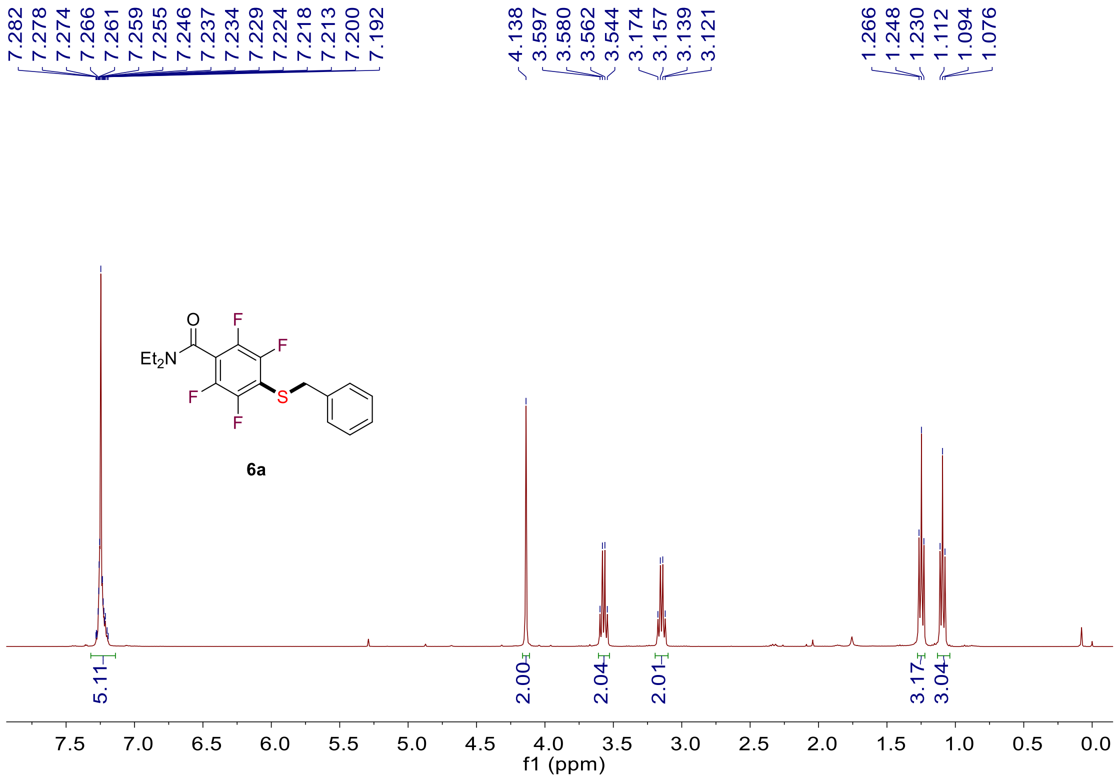
-137.142
-137.170
-137.199
-137.228
-137.236
4r
4.00
3.82
f1 (ppm)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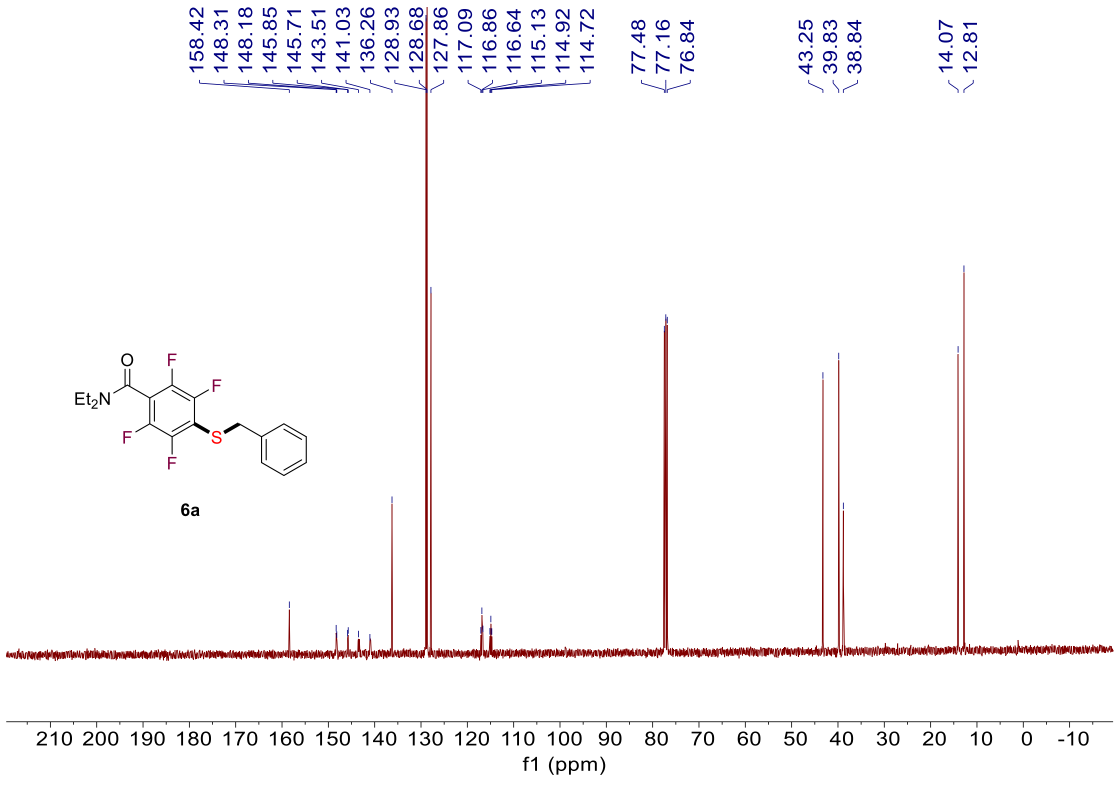
7.278
7.274
7.266
7.261
7.259
7.255
7.246
7.237
7.234
7.229
7.224
7.218
7.213
7.200
7.192
4.138
3.597
3.580
3.562
3.544
3.174
3.157
3.139
3.121
1.266
1.248
1.230
1.112
1.094
1.076
O
F
F
Et2N
S
F
F
6a
5.11
2.00
2.04
2.01
3.17
3.04
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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128.68
127.86
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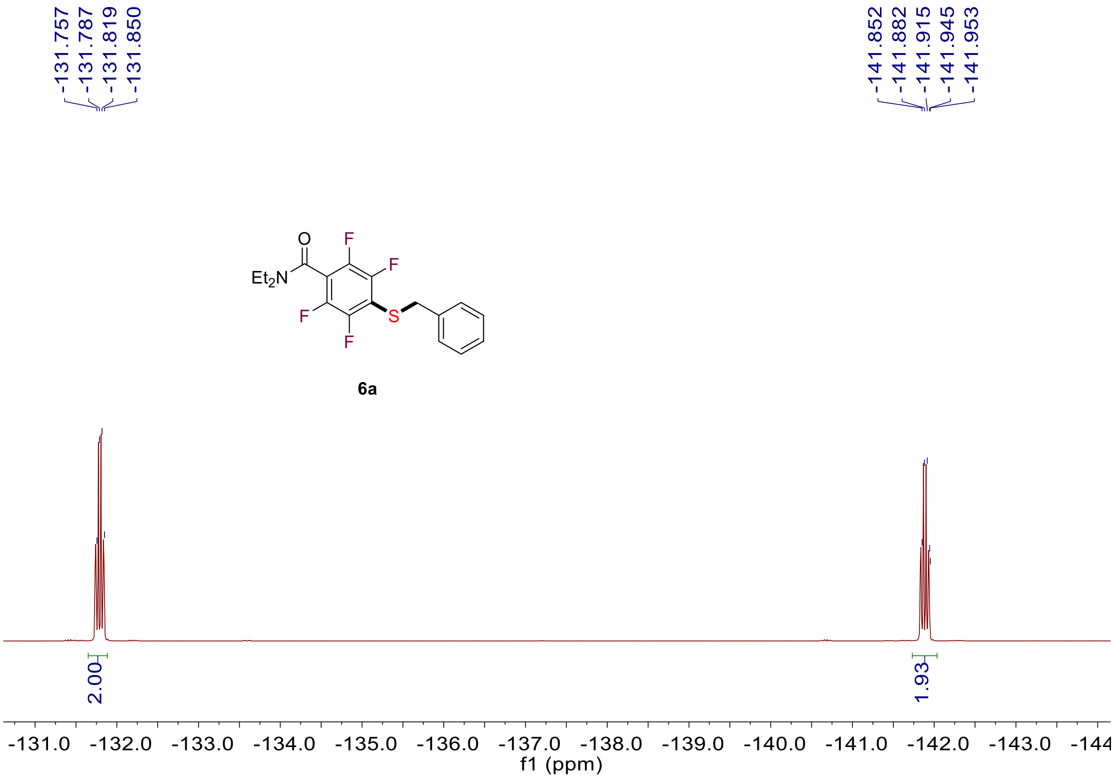
116.86
116.64
115.13
114.92
114.72
77.48
77.16
76.84
43.25
39.83
38.84
14.07
12.81
O
F
F
Et2N
S
F
F
6a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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-131.850
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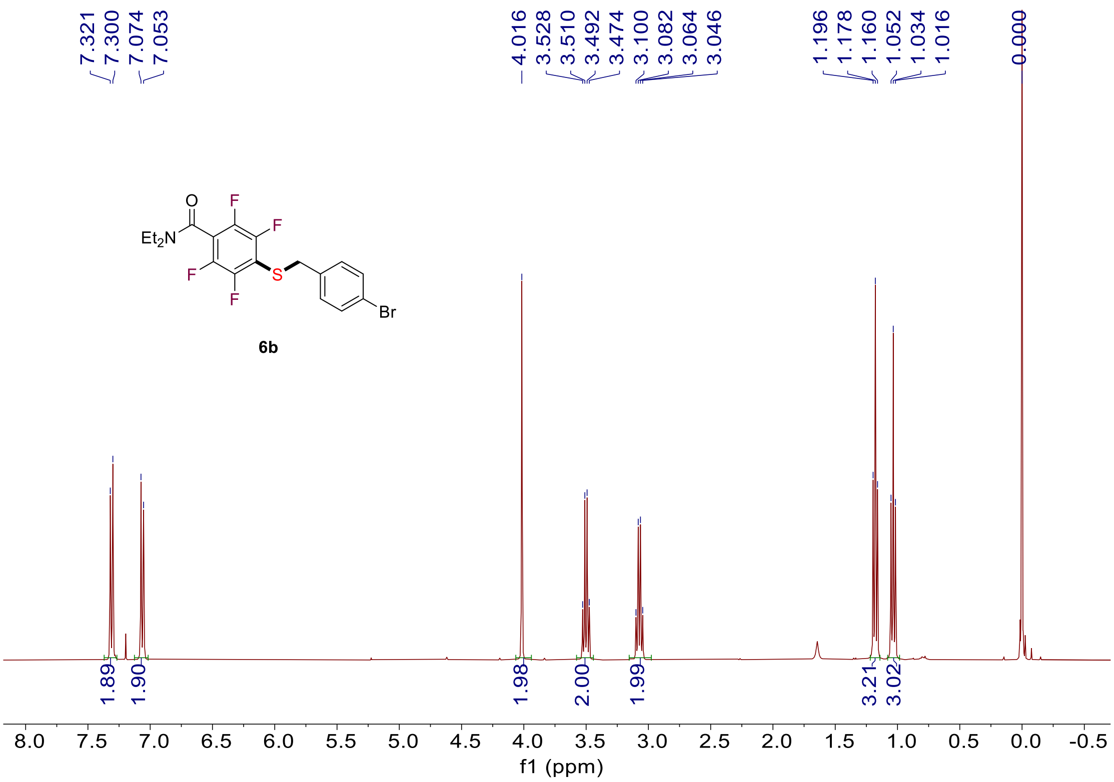
-141.882
-141.915
-141.945
-141.953
6a
2.00
1.93
-131.0 -132.0 -133.0 -134.0 -135.0 -136.0 -137.0 -138.0 -139.0 -140.0 -141.0 -142.0 -143.0 -144
f1 (ppm)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3.492
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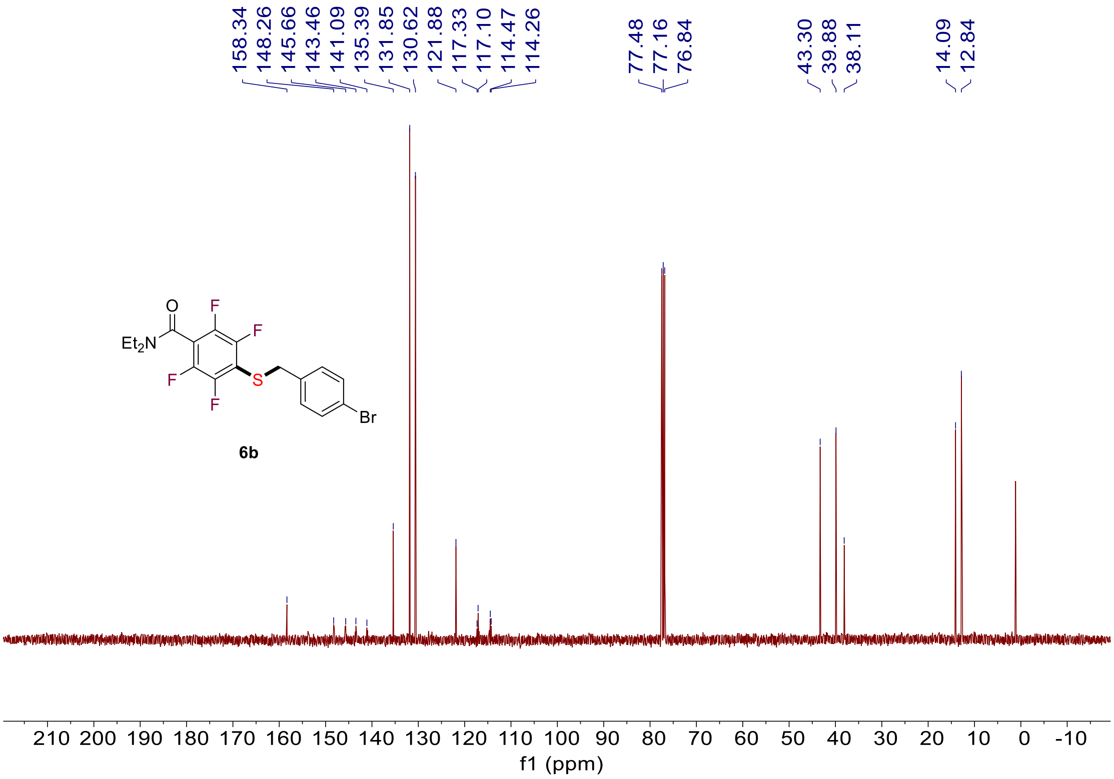
3.100
3.082
3.064
3.046
1.196
1.178
1.160
1.052
1.034
1.016
0.000
6b
1.89
1.90
1.98
2.00
1.99
3.21
3.02
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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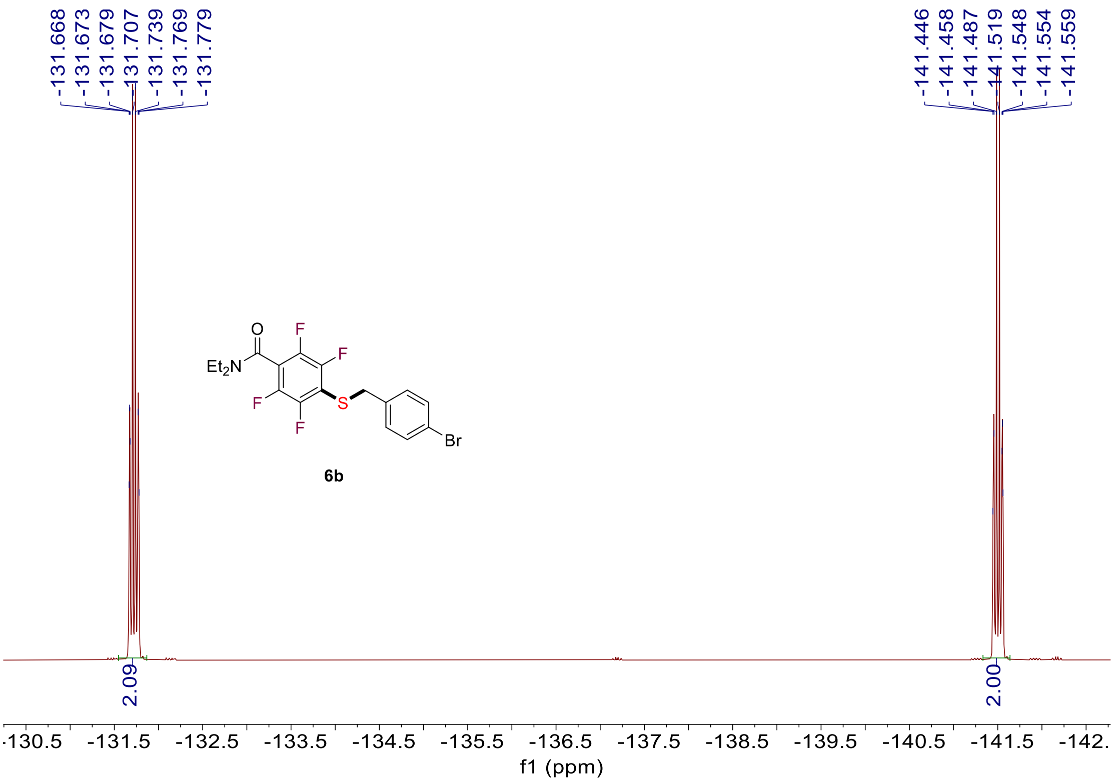
117.33
117.10
114.47
114.26
77.48
77.16
76.84
43.30
39.88
38.11
14.09
12.84
O
F
F
Et2N
F
F
S
Br
6b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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-131.769
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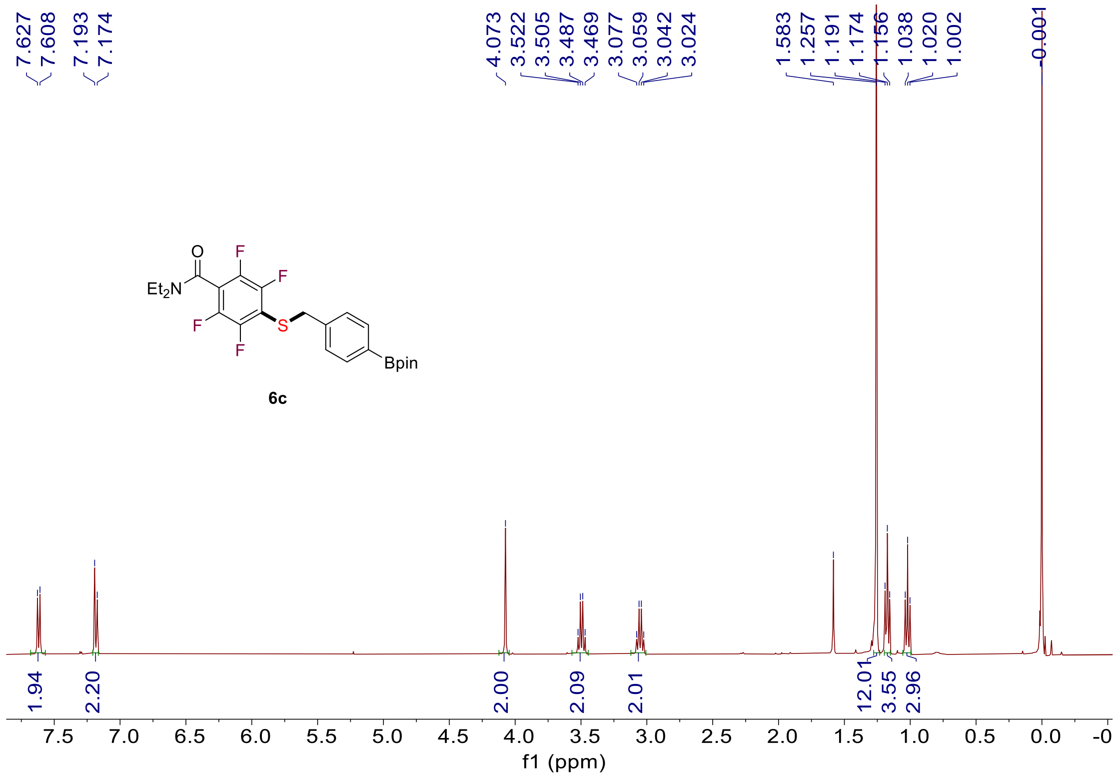
-141.446
-141.458
-141.487
-141.519
-141.548
-141.554
-141.559
O
F
F
Et2N
F
F
S
Br
6b
2.09
2.00
-130.5 -131.5 -132.5 -133.5 -134.5 -135.5 -136.5 -137.5 -138.5 -139.5 -140.5 -141.5 -142.
f1 (ppm)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7.193
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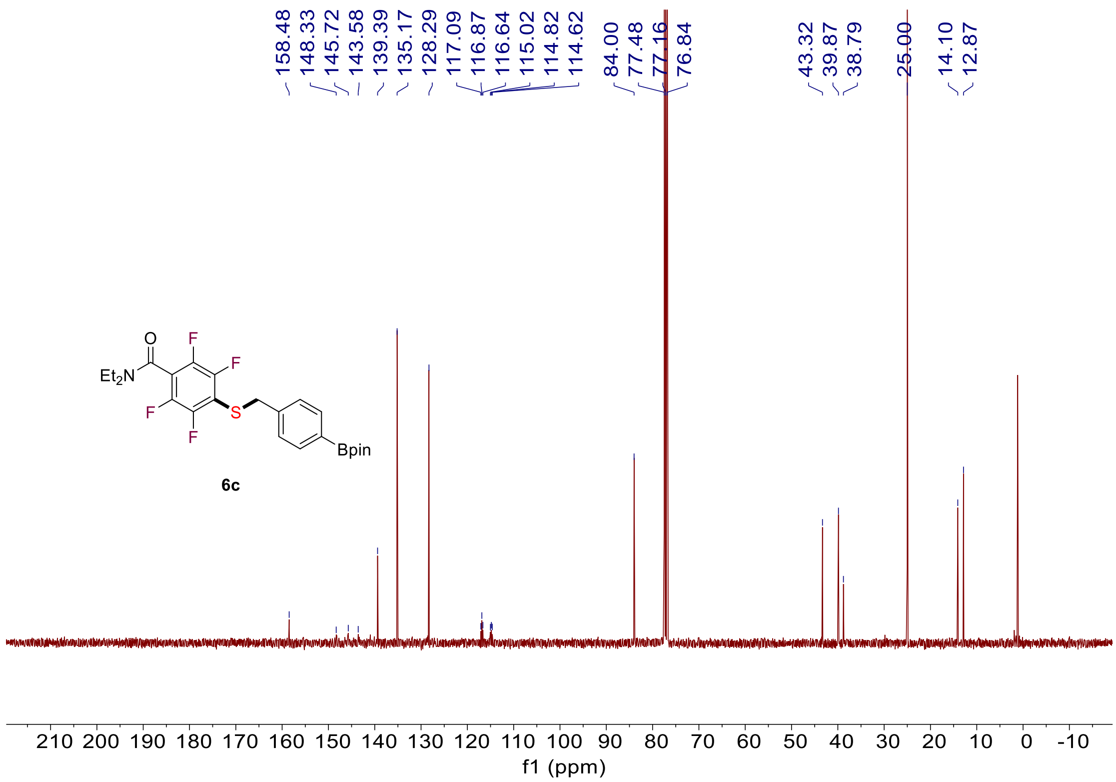
4.073
3.522
3.505
3.487
3.469
3.077
3.059
3.042
3.024
1.583
1.257
1.191
1.174
1.156
1.038
1.020
1.002
-0.001
Et2N
O
F
F
F
F
S
Bpin
6c
1.94
2.20
2.00
2.09
2.01
12.01
3.55
2.96
f1 (ppm)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115.02
114.82
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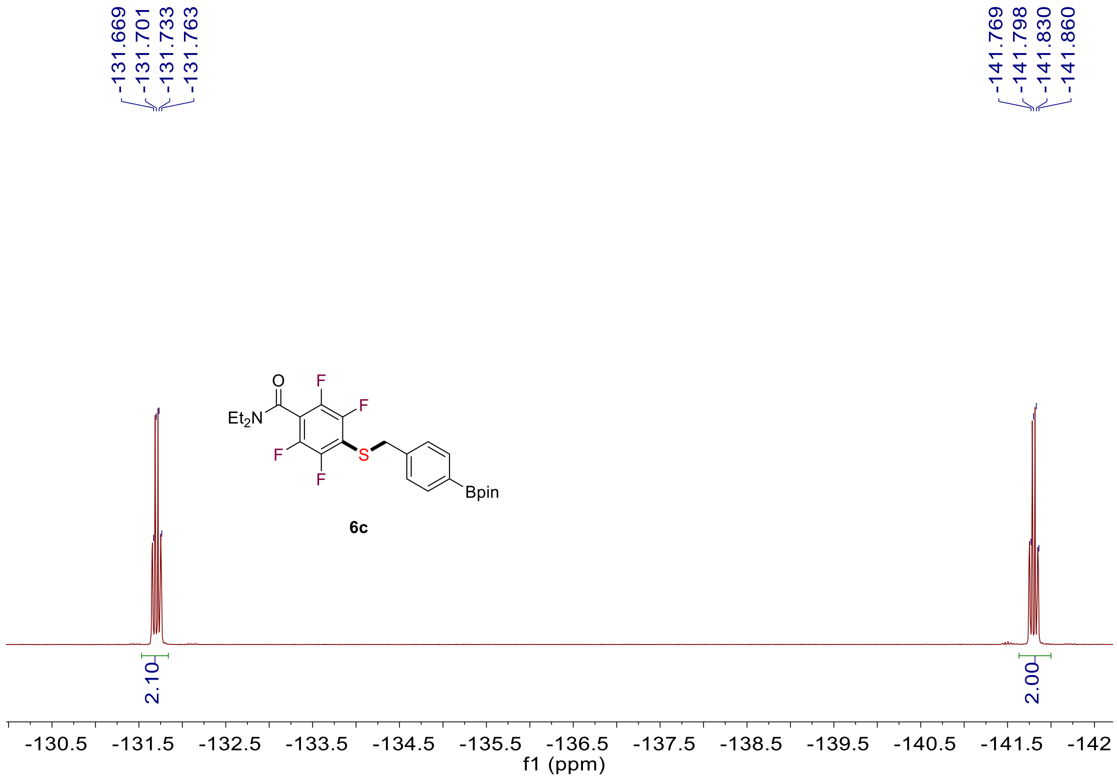
84.00
77.48
77.16
76.84
43.32
39.87
38.79
25.00
14.10
12.87
Et2N
O
F
F
F
F
S
Bpin
6c
f1 (ppm)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-141.860
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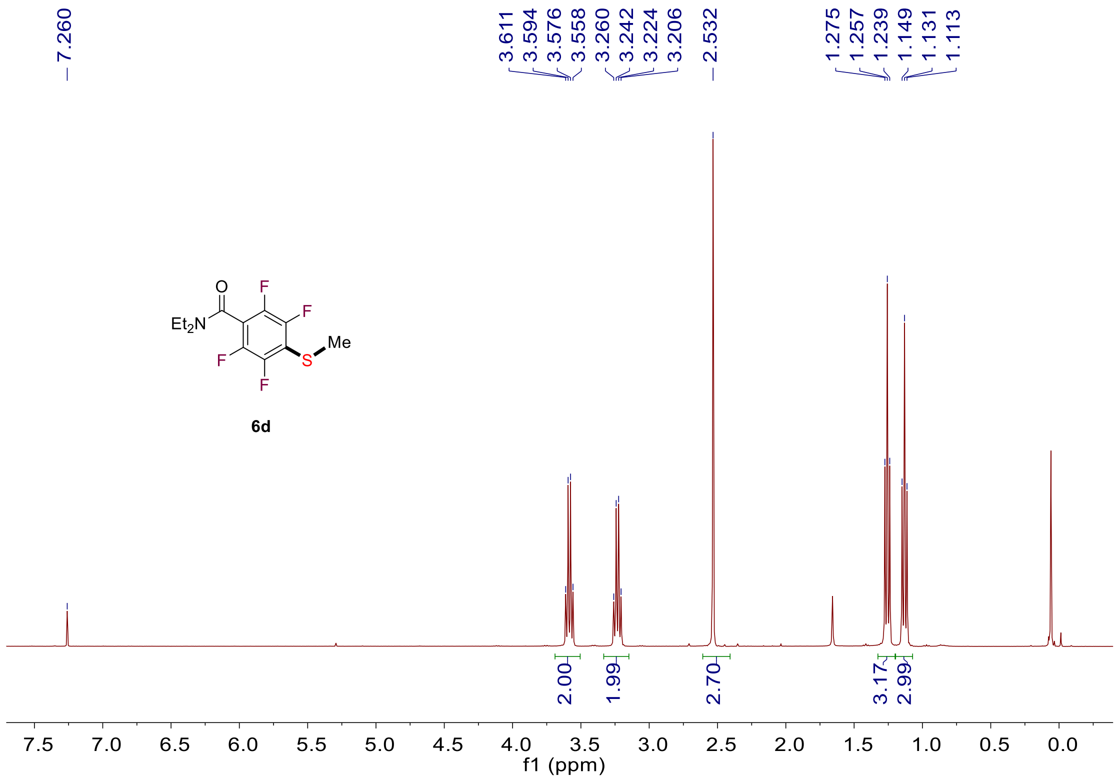
O
F
F
F
F
S
Bpin
6c
2.10
2.00
-130.5 -131.5 -132.5 -133.5 -134.5 -135.5 -136.5 -137.5 -138.5 -139.5 -140.5 -141.5 -142
f1 (ppm)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1.239
1.149
1.131
1.113
Et2N
O
F
F
F
F
S
Me
6d
2.00
1.99
2.70
3.17
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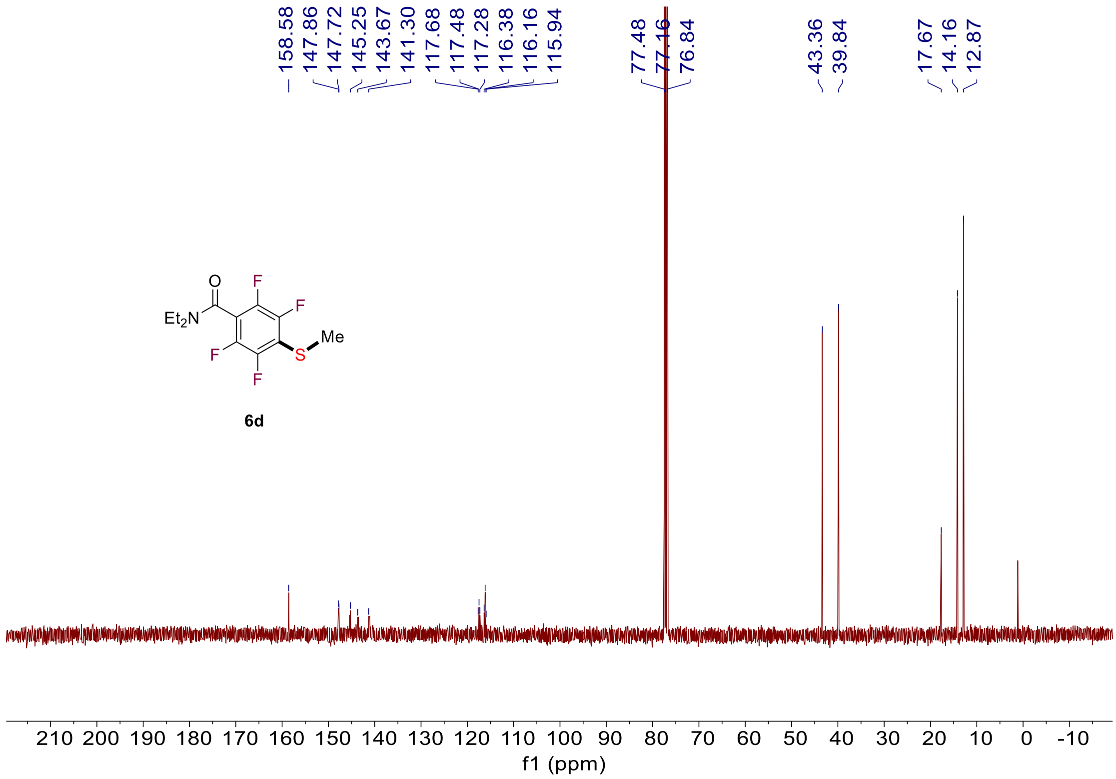
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77.48
77.16
76.84
43.36
39.84
17.67
14.16
12.87
Et2N
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F
F
S
Me
6d
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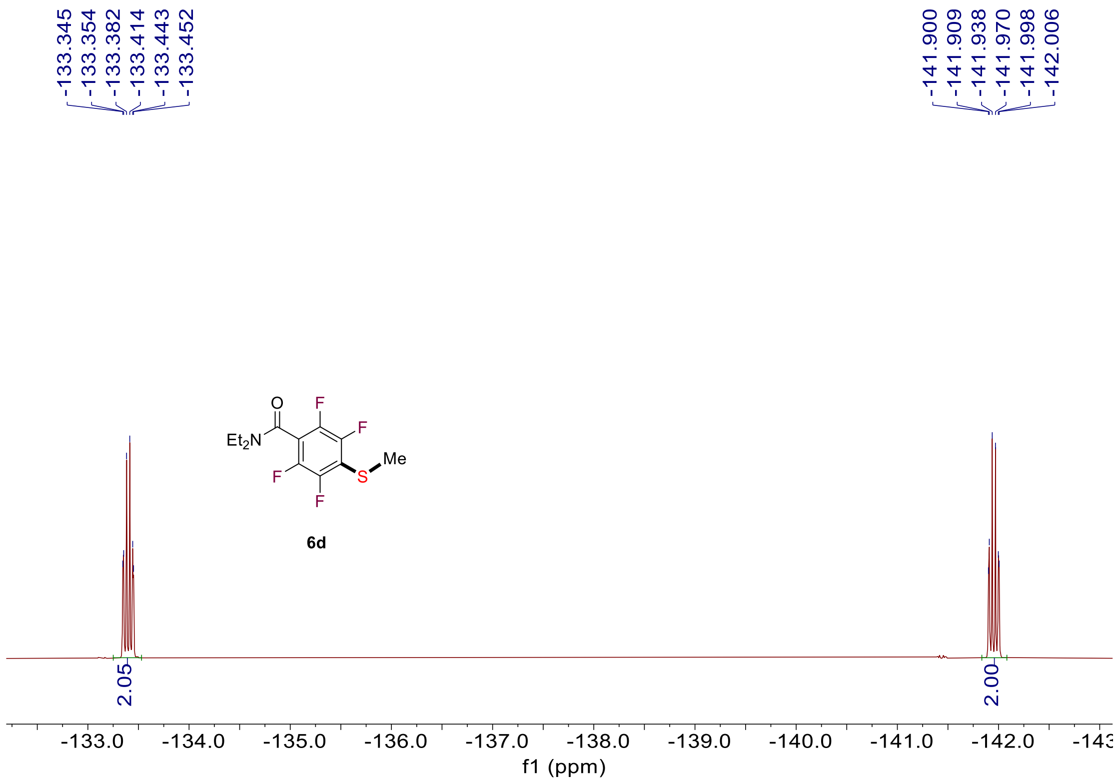
f1 (ppm)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Et2N
O
F
F
F
F
S
Me
6d
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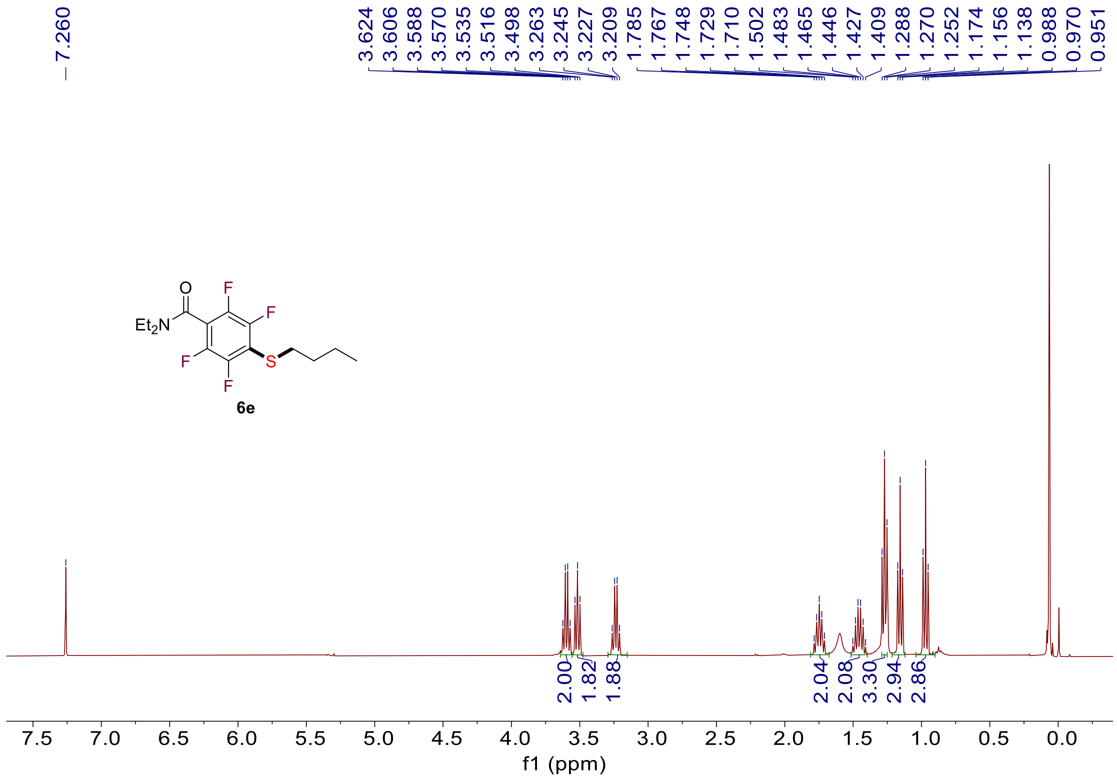
2.00
-133.0 -134.0 -135.0 -136.0 -137.0 -138.0 -139.0 -140.0 -141.0 -142.0
f1 (ppm)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1.710
1.502
1.483
1.465
1.446
1.427
1.409
1.288
1.270
1.252
1.174
1.156
1.138
0.988
0.970
0.951
Et2N
O
F
F
F
F
S
6e
2.00
1.82
1.88
2.04
2.08
3.30
2.94
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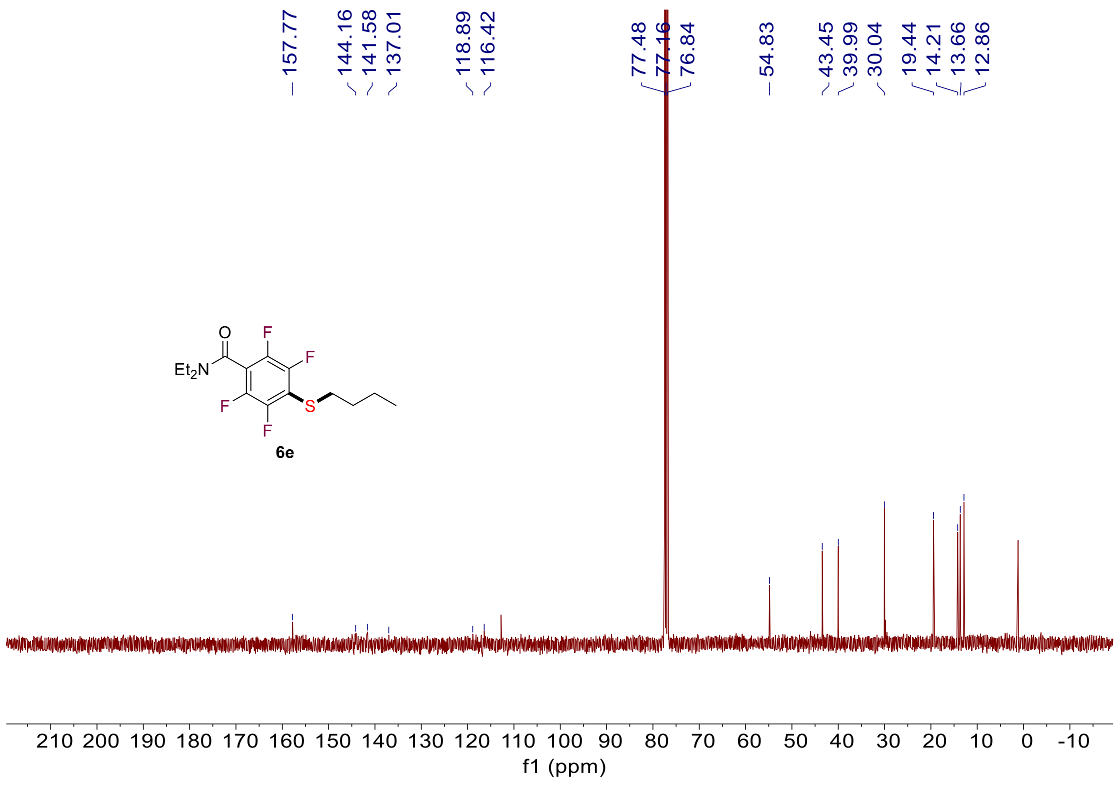
f1 (ppm)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30.04
19.44
14.21
13.66
12.86
Et2N
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F
F
F
S
6e
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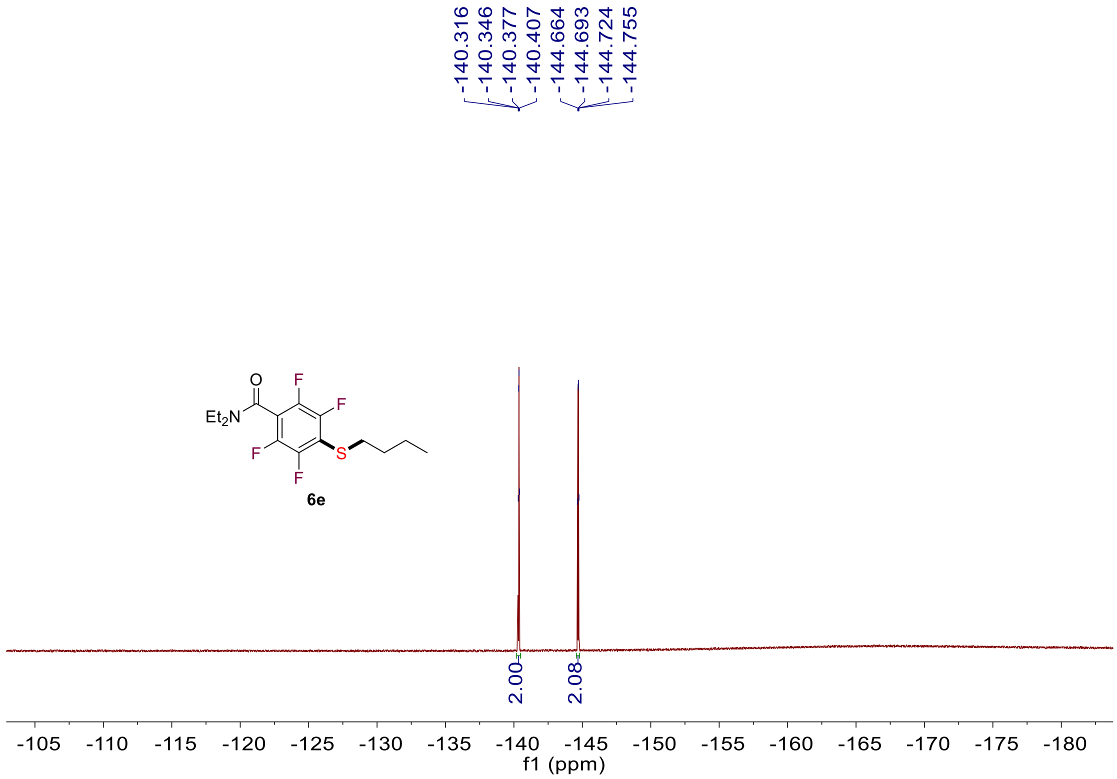
f1 (ppm)



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-144.755
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O
F
F
F
F
S
6e
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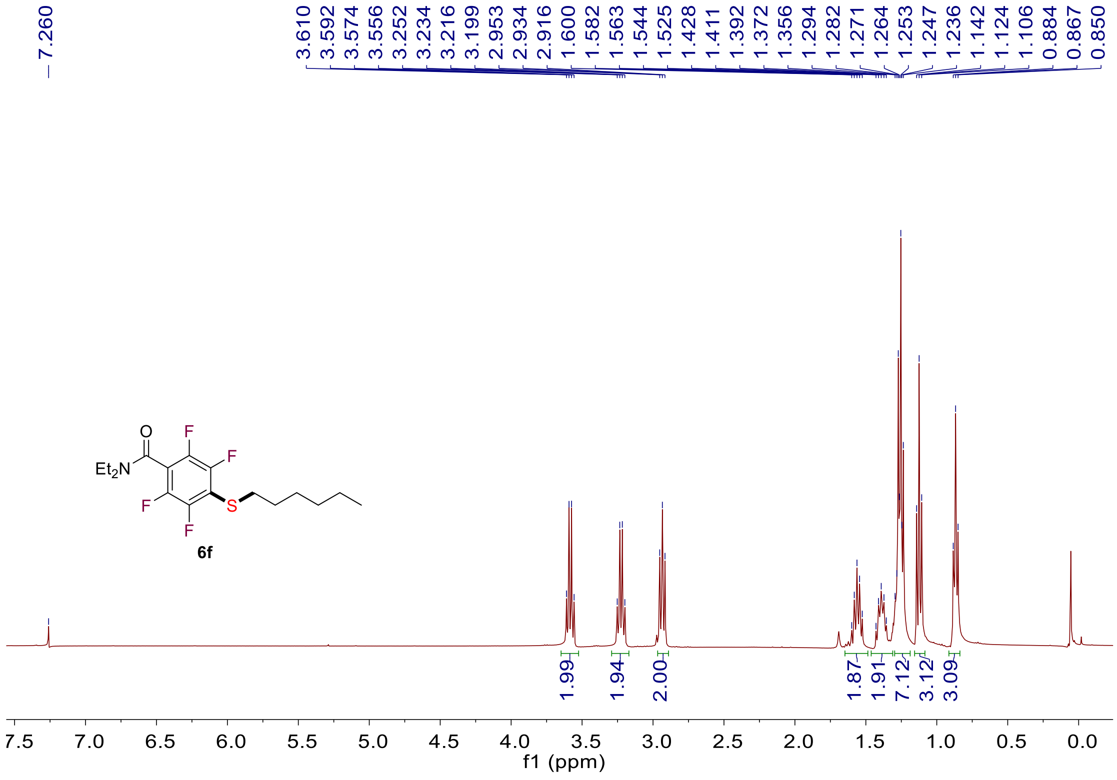
2.08
-105 -110 -115 -120 -125 -130 -135 -140 -145 -150 -155 -160 -165 -170 -175 -180
f1 (ppm)



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O
F
F
F
F
S
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2.00
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1.91
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3.12
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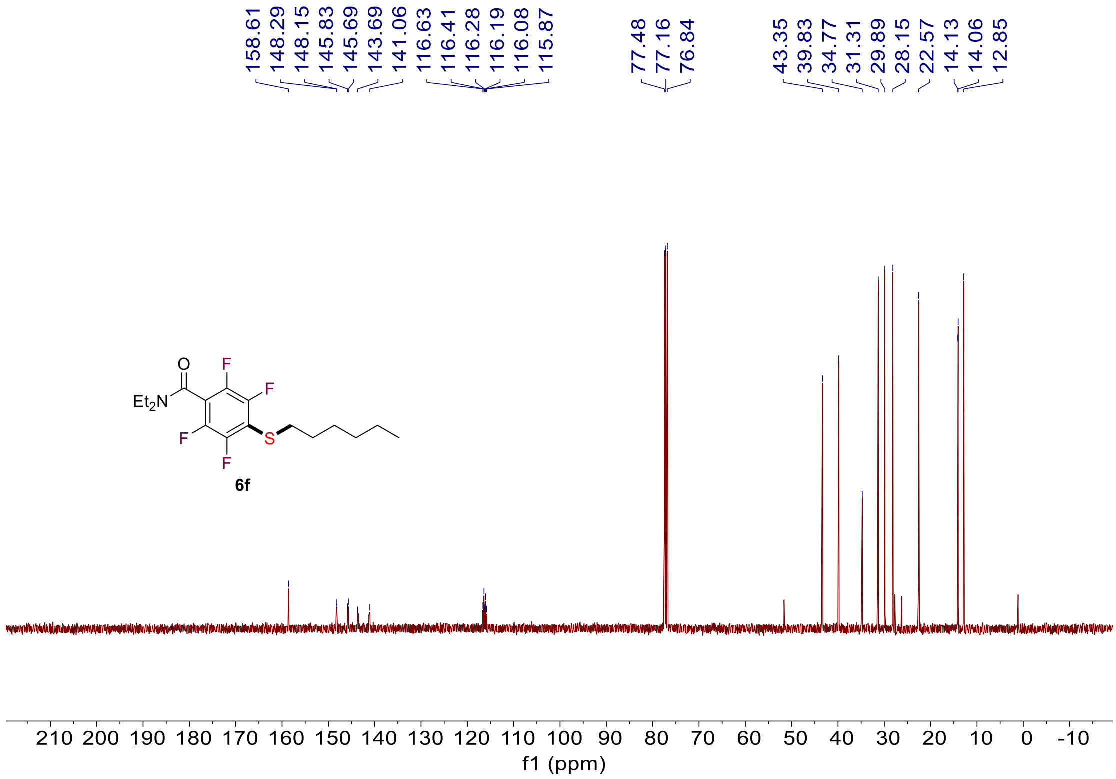
f1 (ppm)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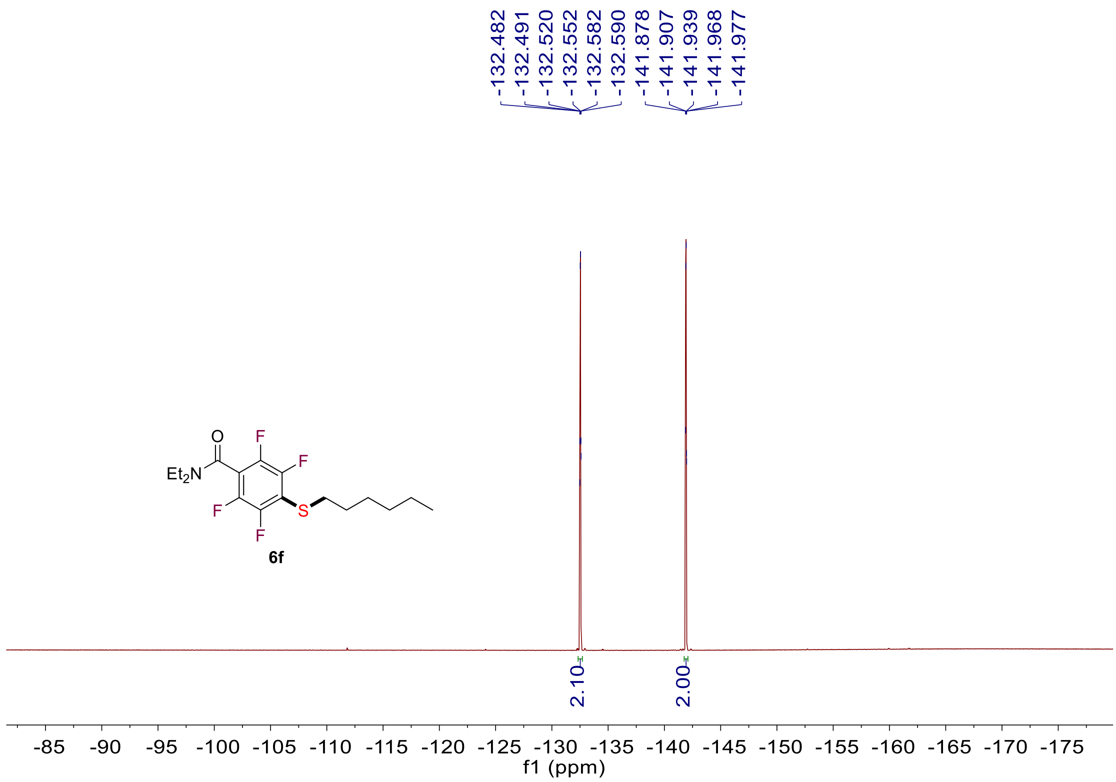
77.16
76.84
43.35
39.83
34.77
31.31
29.89
28.15
22.57
14.13
14.06
12.85
O
Et2N
F
F
F
F
S
6f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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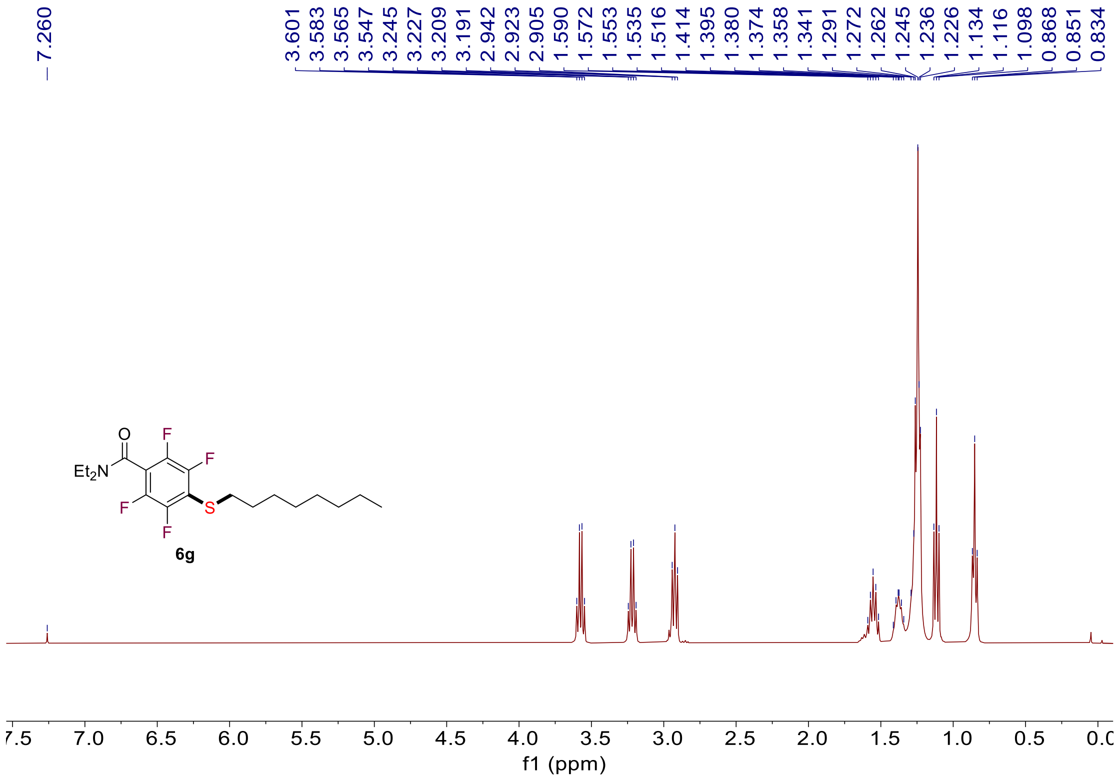
-141.977
O
Et2N
F
F
F
F
S
6f
2.10
2.00
-85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -150 -155 -160 -165 -170 -175
f1 (ppm)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1.572
1.553
1.535
1.516
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1.395
1.380
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1.245
1.236
1.226
1.134
1.116
1.098
0.868
0.851
0.834
Et2N
O
F
F
F
F
S
6g
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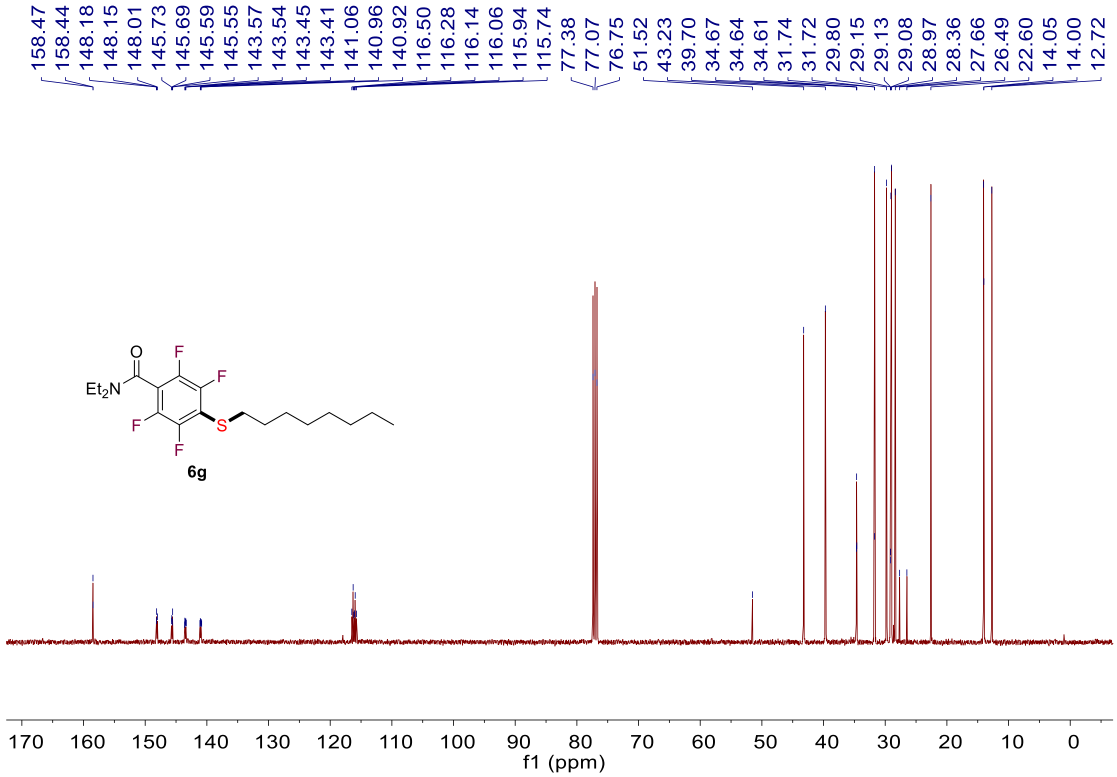
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148.15
148.01
145.73
145.69
145.59
145.55
143.57
143.54
143.45
143.41
141.06
140.96
140.92
116.50
116.28
116.14
116.06
115.94
115.74
77.38
77.07
76.75
51.52
43.23
39.70
34.67
34.64
34.61
31.74
31.72
29.80
29.15
29.13
29.08
28.97
28.36
27.66
26.49
22.60
14.05
14.00
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Et2N
O
F
F
F
F
S
6g
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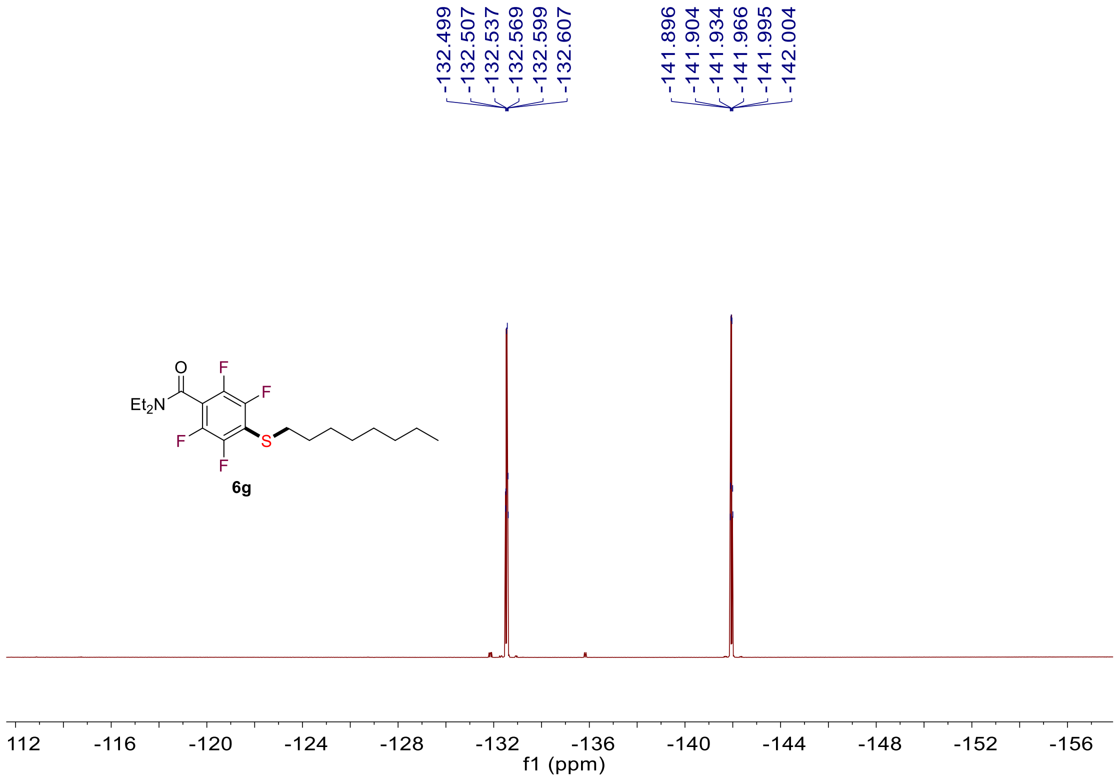
f1 (ppm)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-142.004
O
F
F
Et2N
F
S
F
6g
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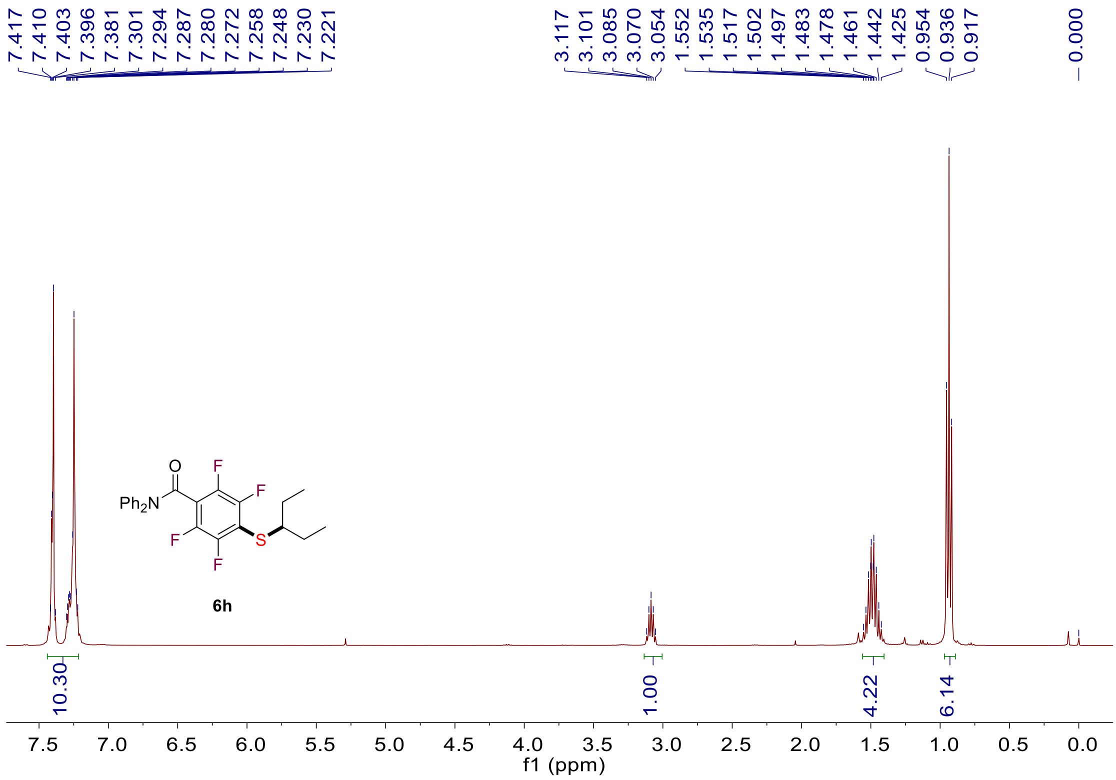
-116
-120
-124
-128
-132
-136
-140
-144
-148
-152
-156
f1 (ppm)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7.248
7.230
7.221
3.117
3.101
3.085
3.070
3.054
1.552
1.535
1.517
1.502
1.497
1.483
1.478
1.461
1.442
1.425
0.954
0.936
0.917
0.000
O
F
F
Ph2N
F
S
F
6h
10.30
1.00
4.22
6.14
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
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1.5
1.0
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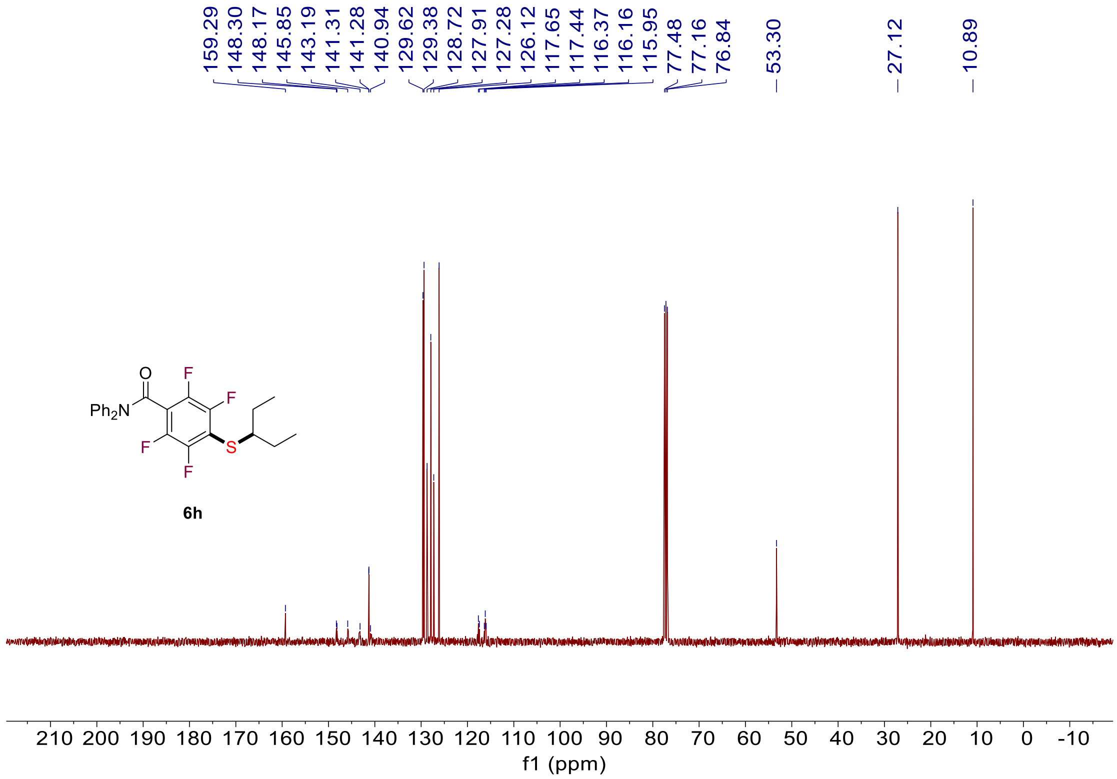
0.0
f1 (ppm)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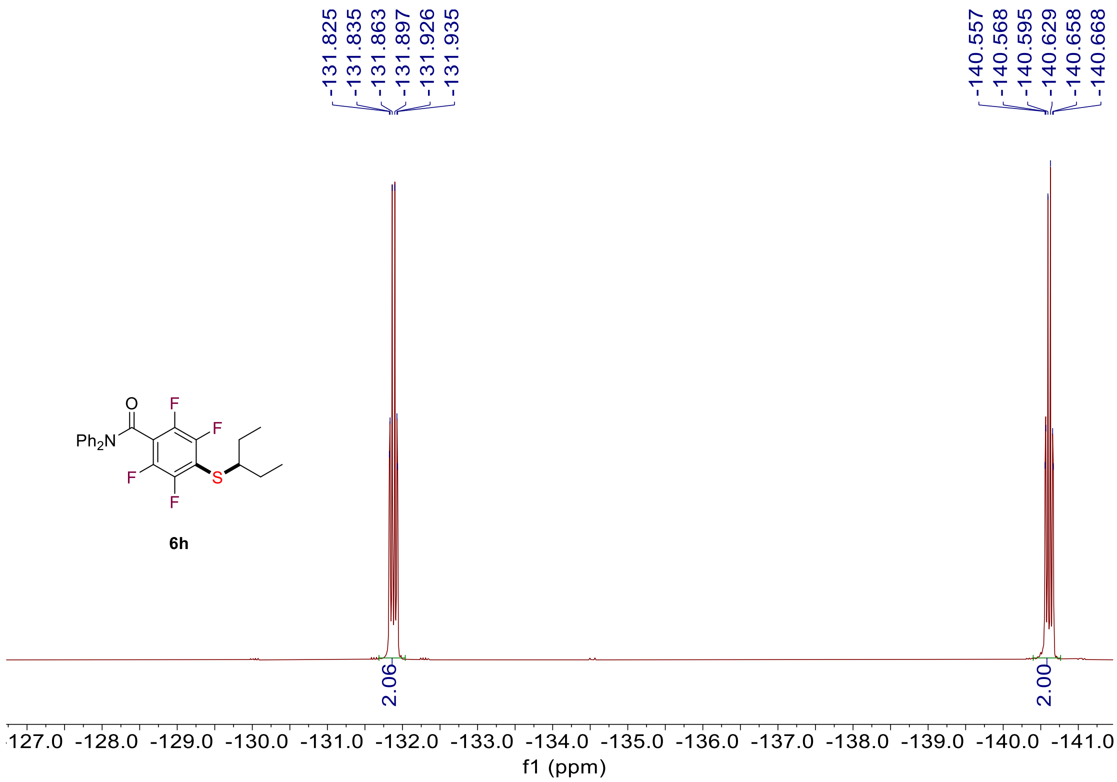
127.28
126.12
117.65
117.44
116.37
116.16
115.95
77.48
77.16
76.84
53.30
27.12
10.89
O
F
F
Ph_2N
S
F
F
6h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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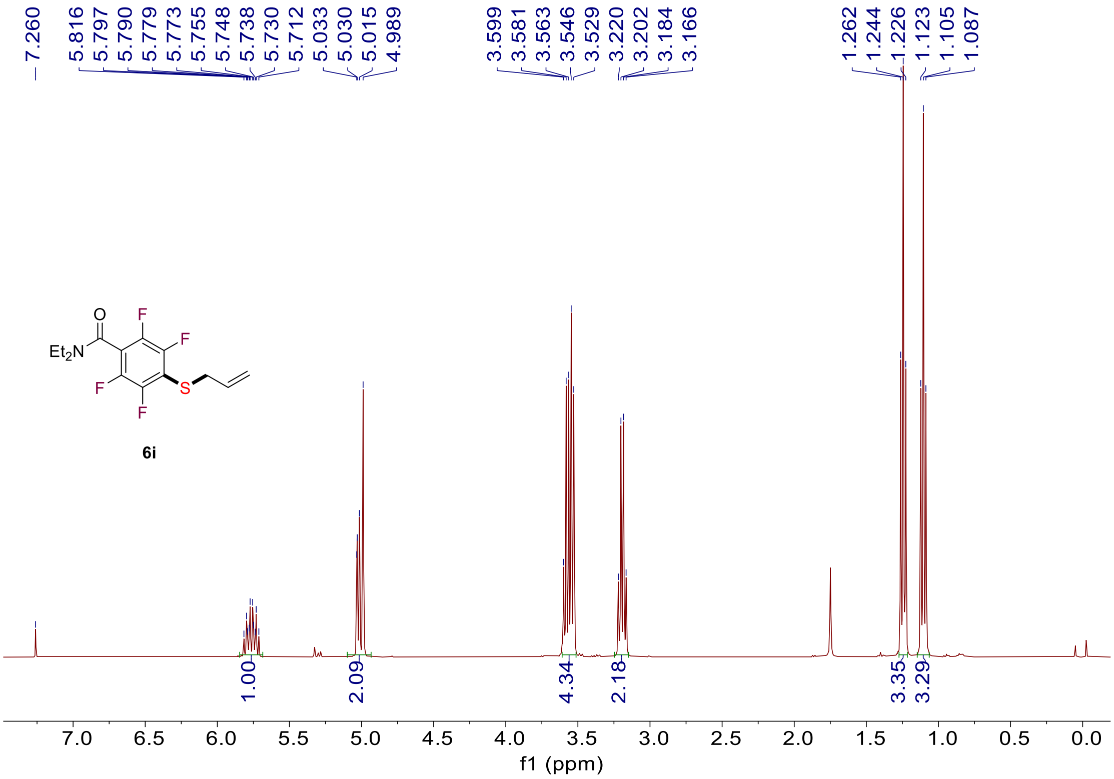
-140.658
-140.668
O
F
F
Ph_2N
S
F
F
6h
2.06
2.00
-127.0 -128.0 -129.0 -130.0 -131.0 -132.0 -133.0 -134.0 -135.0 -136.0 -137.0 -138.0 -139.0 -140.0 -141.0
f1 (ppm)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5.030
5.015
4.989
3.599
3.581
3.563
3.546
3.529
3.220
3.202
3.184
3.166
1.262
1.244
1.226
1.123
1.105
1.087
O
F
F
Et2N
F
S
F
6i
1.00
2.09
4.34
2.18
3.35
3.29
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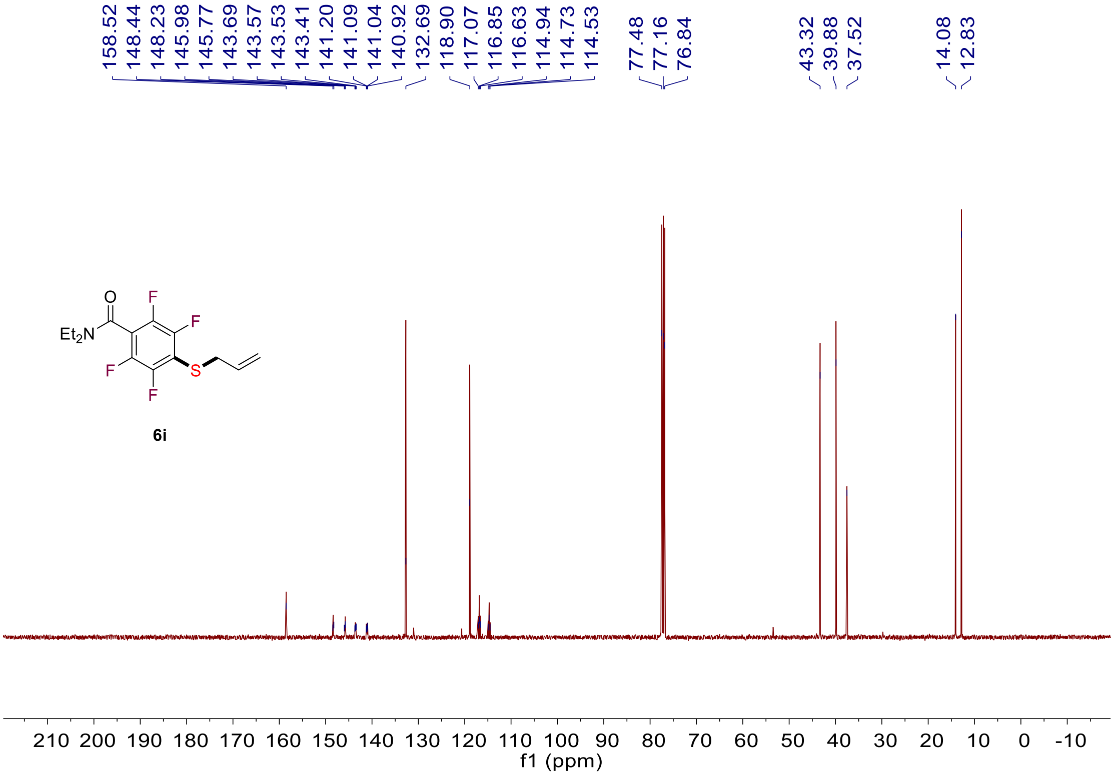
f1 (ppm)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143.53
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141.20
141.09
141.04
140.92
132.69
118.90
117.07
116.85
116.63
114.94
114.73
114.53
77.48
77.16
76.84
43.32
39.88
37.52
14.08
12.83
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F
F
Et2N
F
S
F
6i
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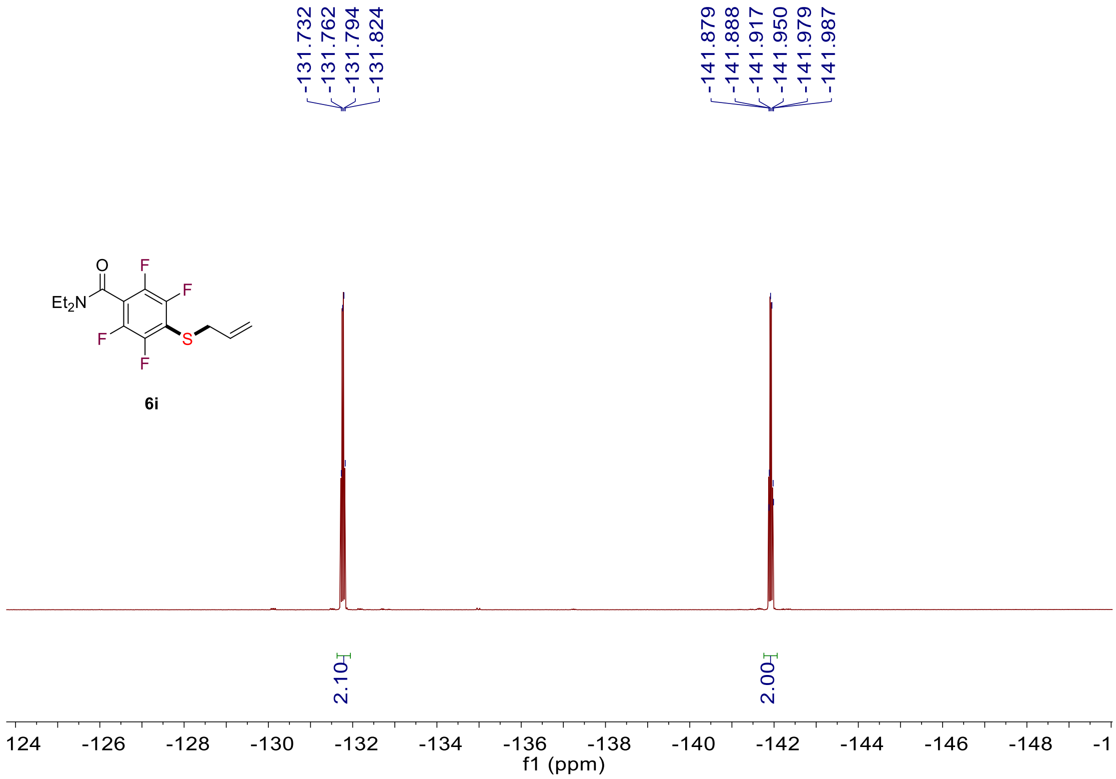
f1 (ppm)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-141.917
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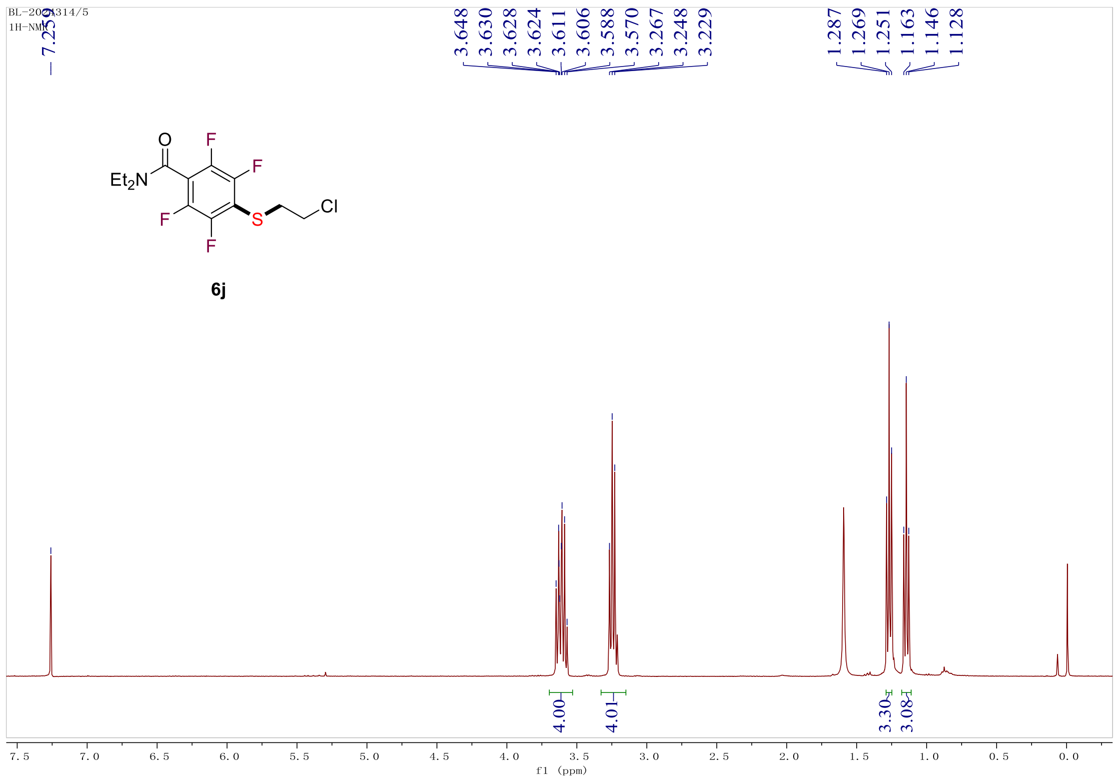
-141.987
Et2N
O
F
F
F
F
S
6i
2.10
2.00
f1 (ppm)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3.648
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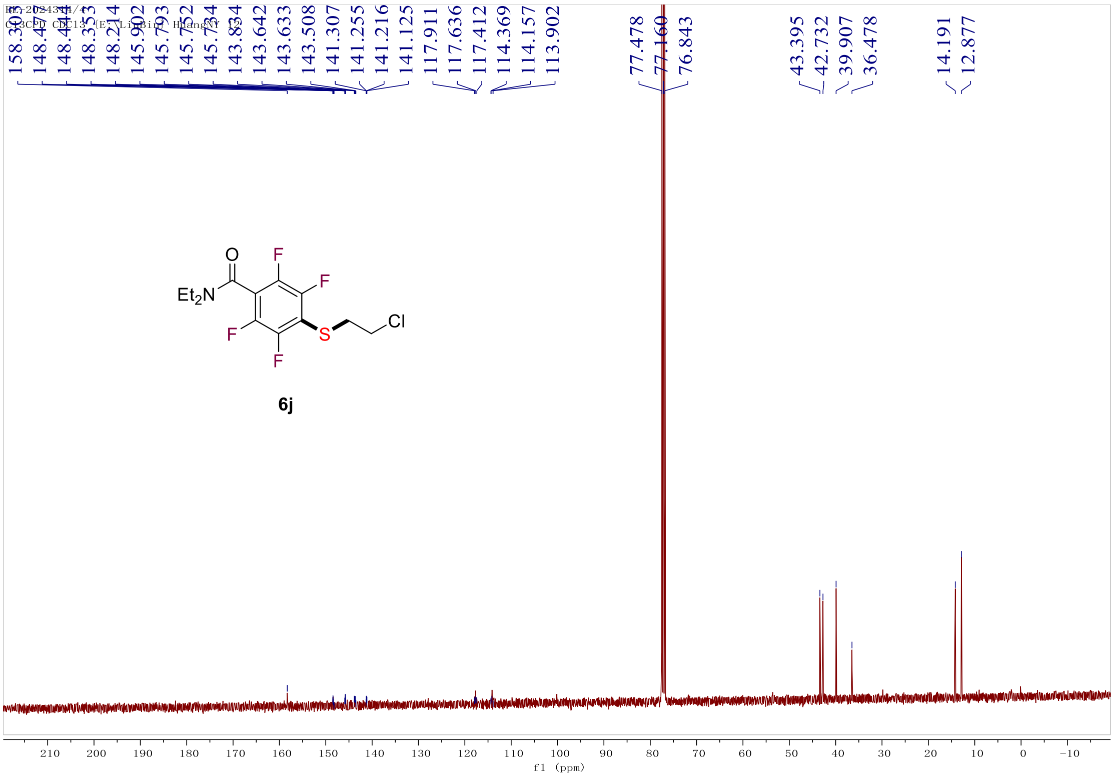
3.624
3.611
3.606
3.588
3.570
3.267
3.248
3.229
1.287
1.269
1.251
1.163
1.146
1.128
Et2N
O
F
F
F
F
S
Cl
6j
4.00
4.01
3.30
3.08
f1 (ppm)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145.752
145.734
143.834
143.642
143.633
143.508
141.307
141.255
141.216
141.125
117.911
117.636
117.412
114.369
114.157
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76.843
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42.732
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36.478
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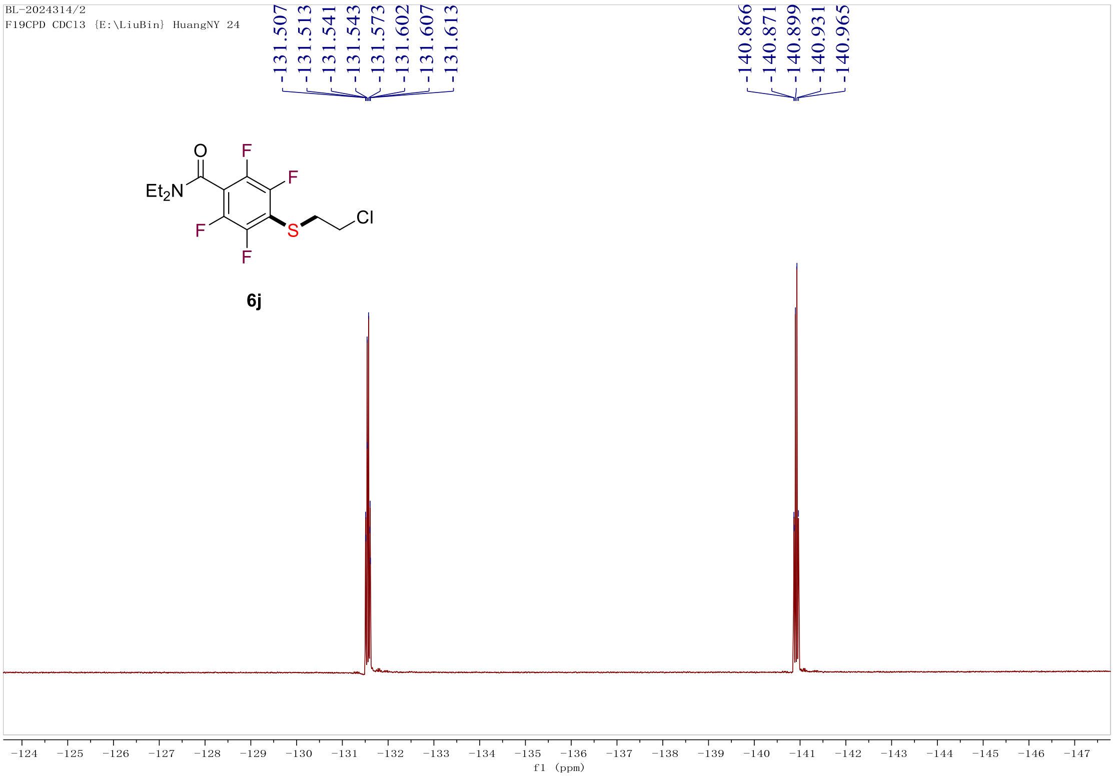
6j
f1 (ppm)


BL-2024314/2
F19CPD CDCl3 {E:\LiuBin} HuangNY 24
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-131.541
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-131.573
-131.602
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-131.613
-140.866
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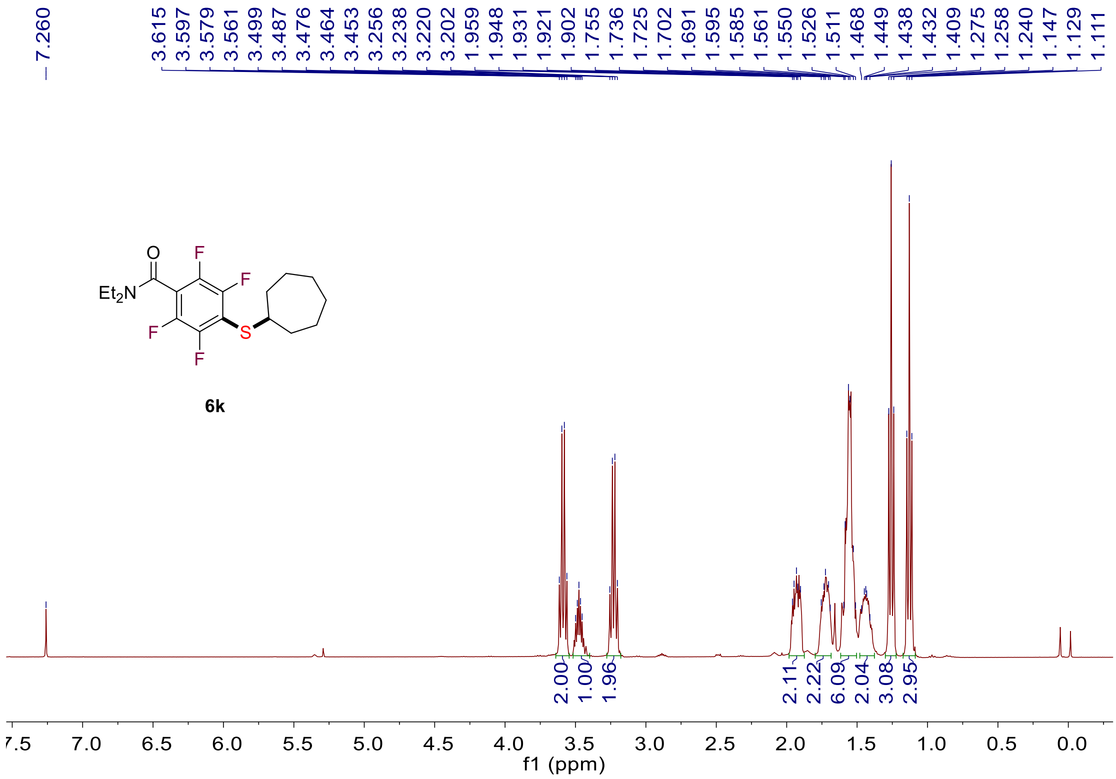
-140.899
-140.931
-140.965
6j
f1 (ppm)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3.476
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3.453
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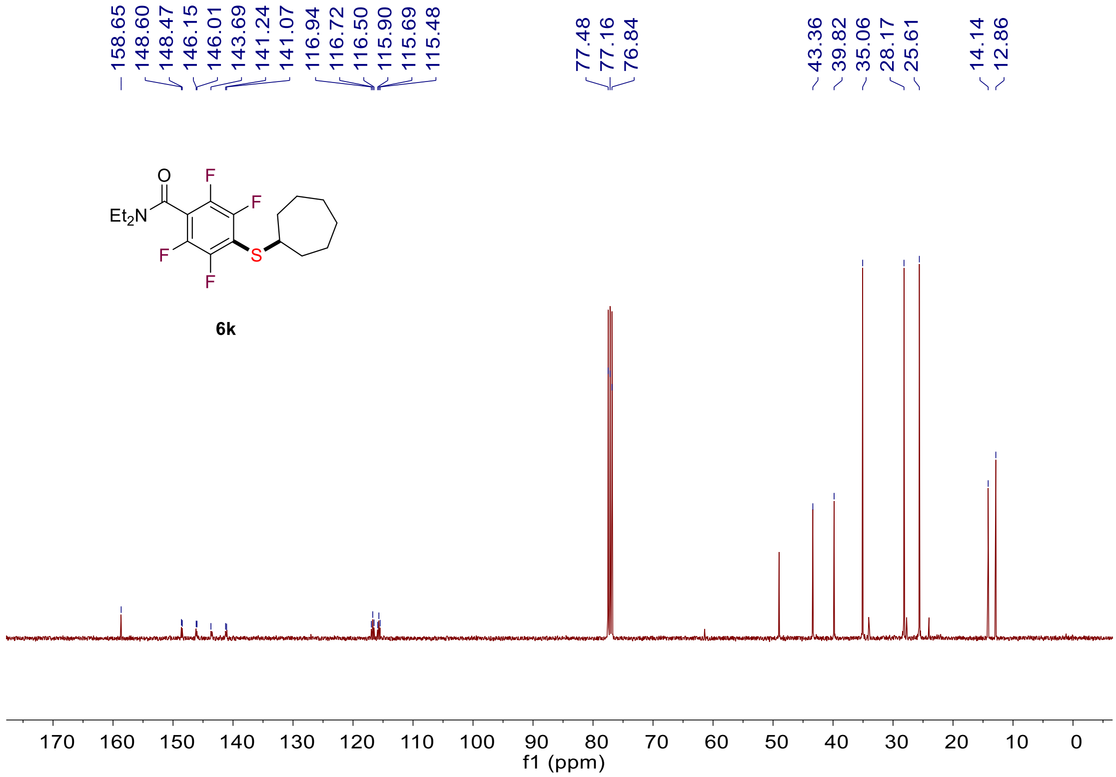
3.238
3.220
3.202
1.959
1.948
1.931
1.921
1.902
1.755
1.736
1.725
1.702
1.691
1.595
1.585
1.561
1.550
1.526
1.511
1.468
1.449
1.438
1.432
1.409
1.275
1.258
1.240
1.147
1.129
1.111
O
F
F
Et2N
S
F
F
2.00
1.00
1.96
2.11
2.22
6.09
2.04
3.08
2.95
f1 (ppm)

6k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141.07
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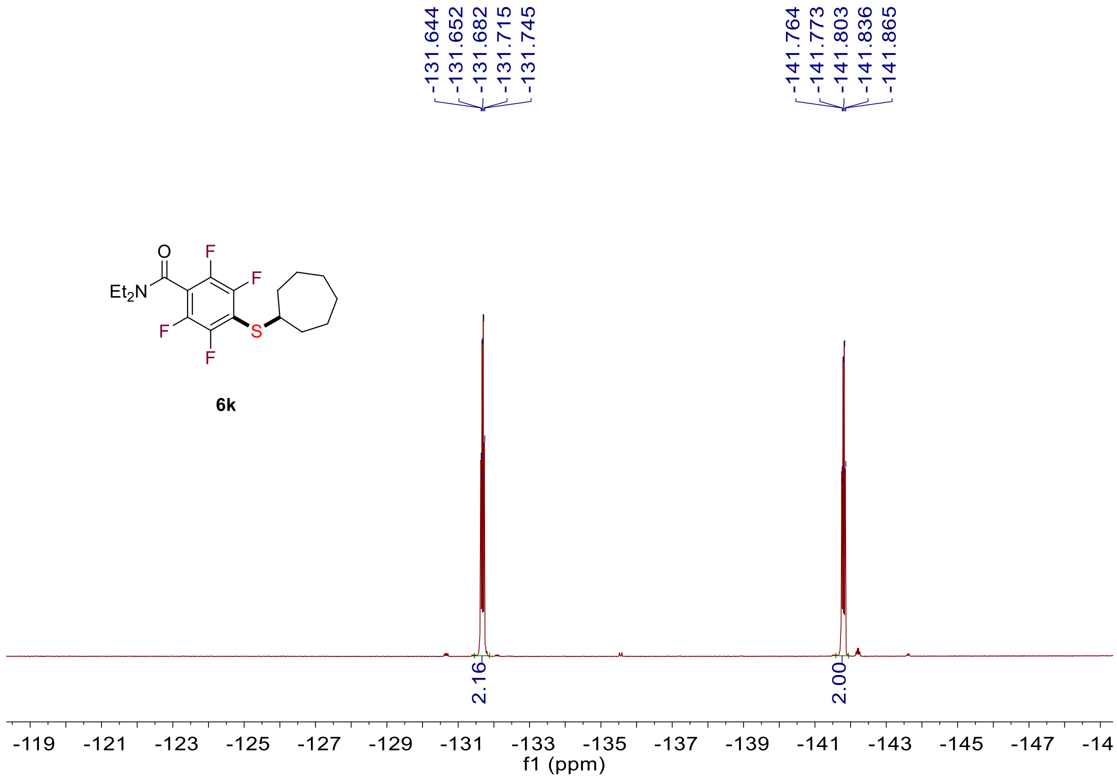
116.72
116.50
115.90
115.69
115.48
77.48
77.16
76.84
43.36
39.82
35.06
28.17
25.61
14.14
12.86
O
F
F
Et2N
S
F
F
f1 (ppm)

6k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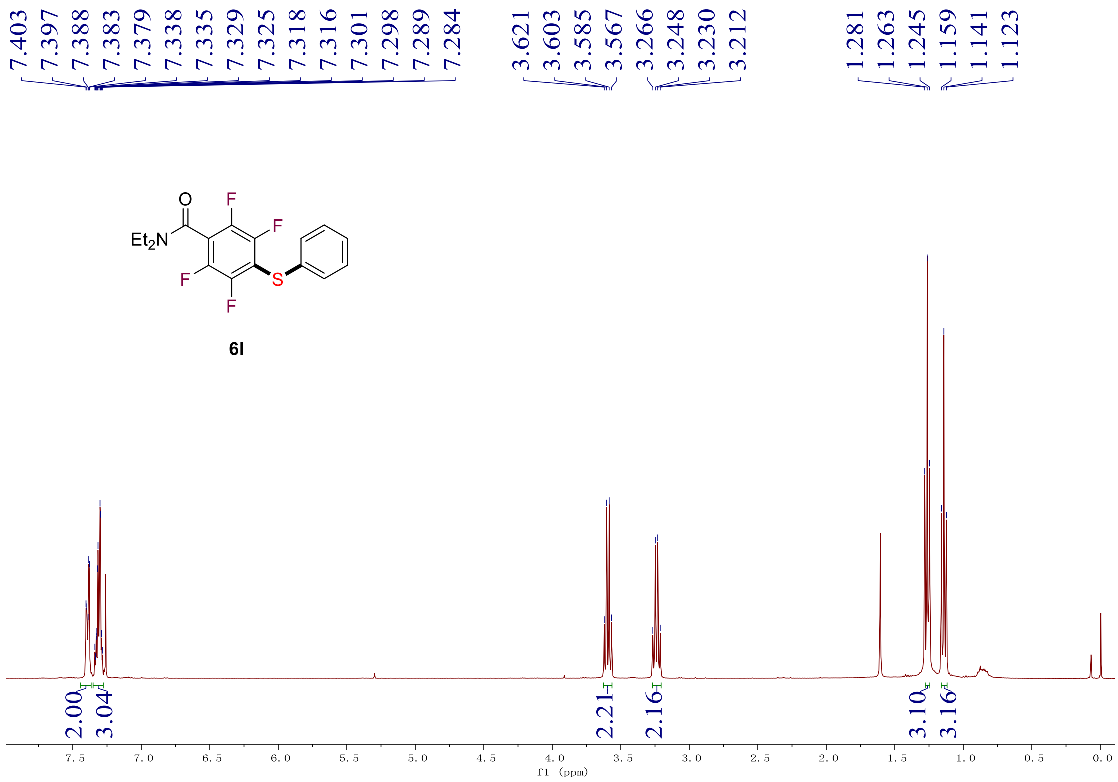
-141.803
-141.836
-141.865
Et2N
O
F
F
F
F
S
6k
2.16
2.00
-119 -121 -123 -125 -127 -129 -131 -133 -135 -137 -139 -141 -143 -145 -147 -14
f1 (ppm)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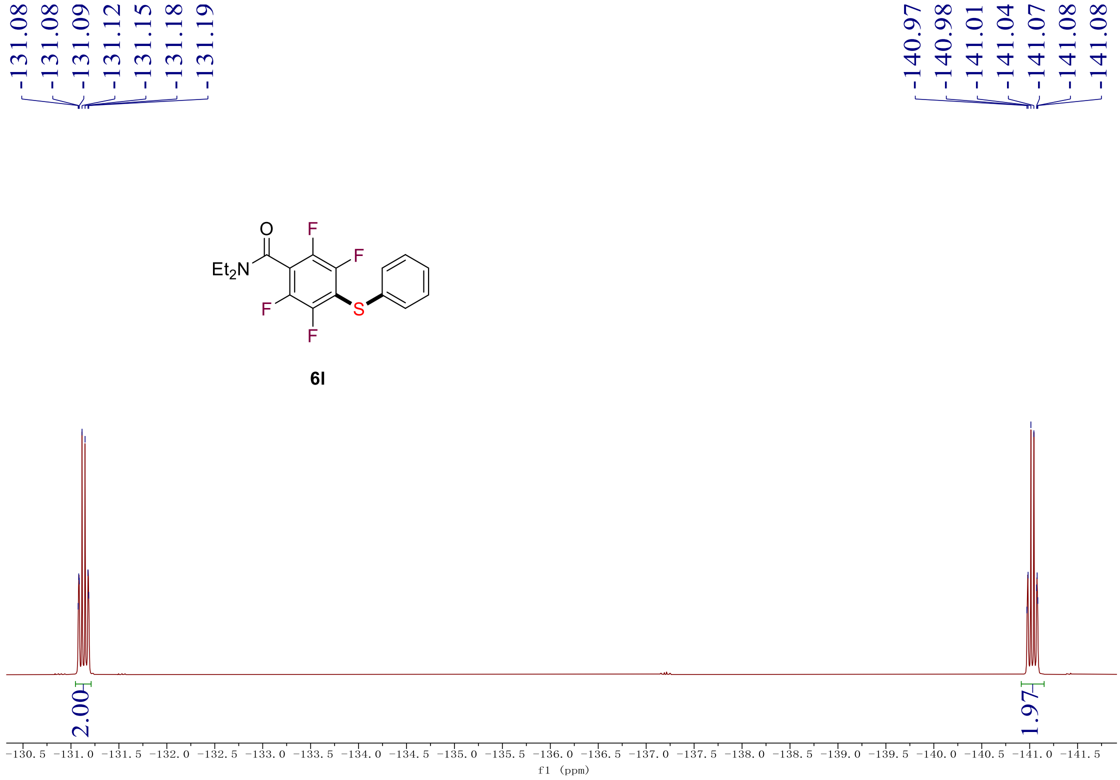
7.316
7.301
7.298
7.289
7.284
3.621
3.603
3.585
3.567
3.266
3.248
3.230
3.212
1.281
1.263
1.245
1.159
1.141
1.123
Et2N
O
F
F
F
F
S
6l
2.00
3.04
2.21
2.16
3.10
3.16
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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-131.09
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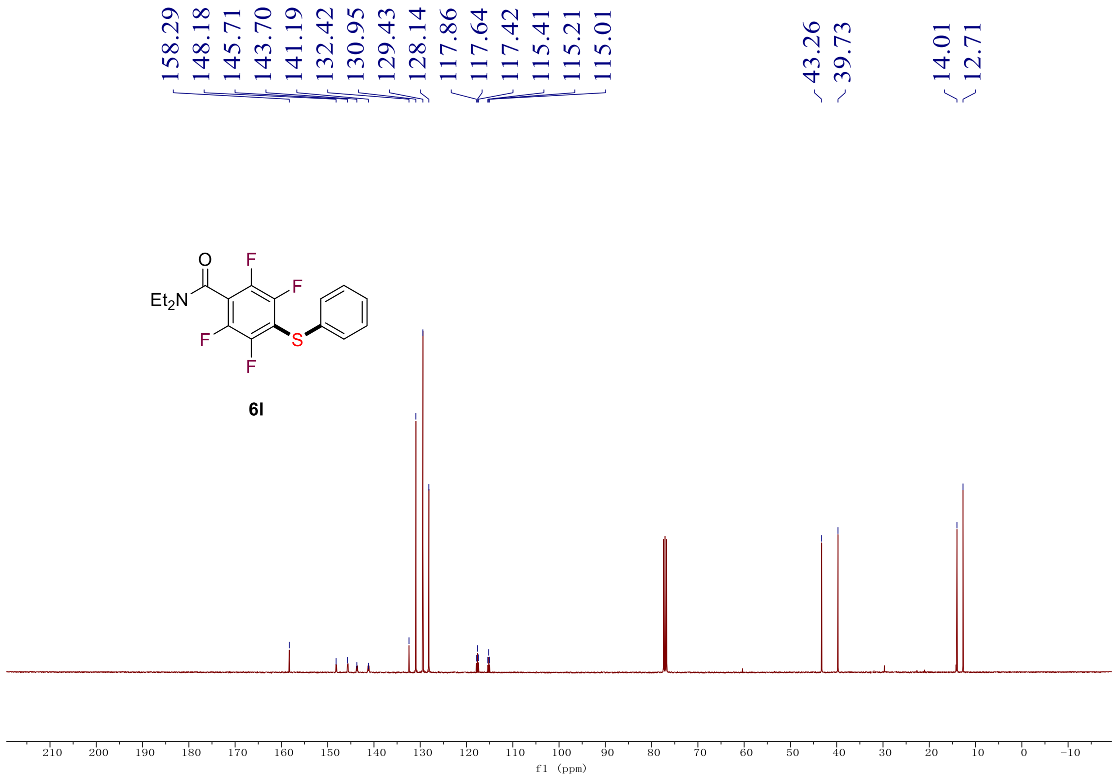
-140.98
-141.01
-141.04
-141.07
-141.08
-141.08
O
F
F
Et2N
S
F
F
6l
2.00
1.97
f1 (ppm)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117.64
117.42
115.41
115.21
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39.73
14.01
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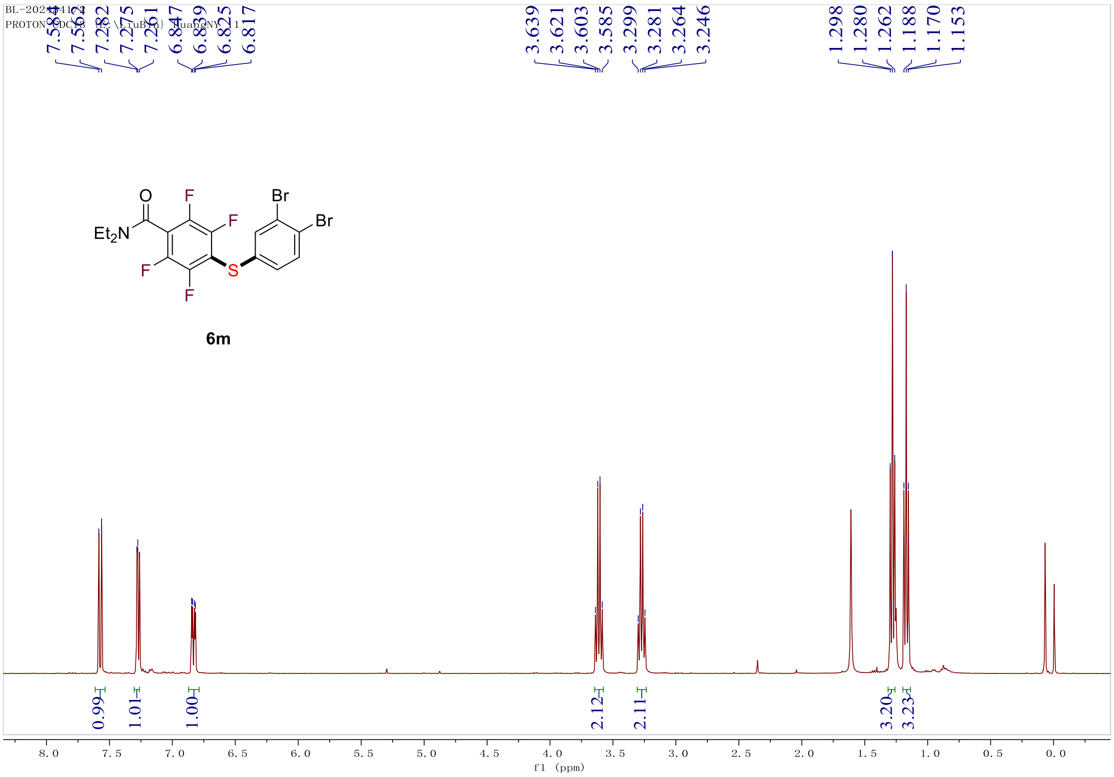
O
F
F
Et2N
S
F
F
6l
f1 (ppm)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3.585
3.299
3.281
3.264
3.246
1.298
1.280
1.262
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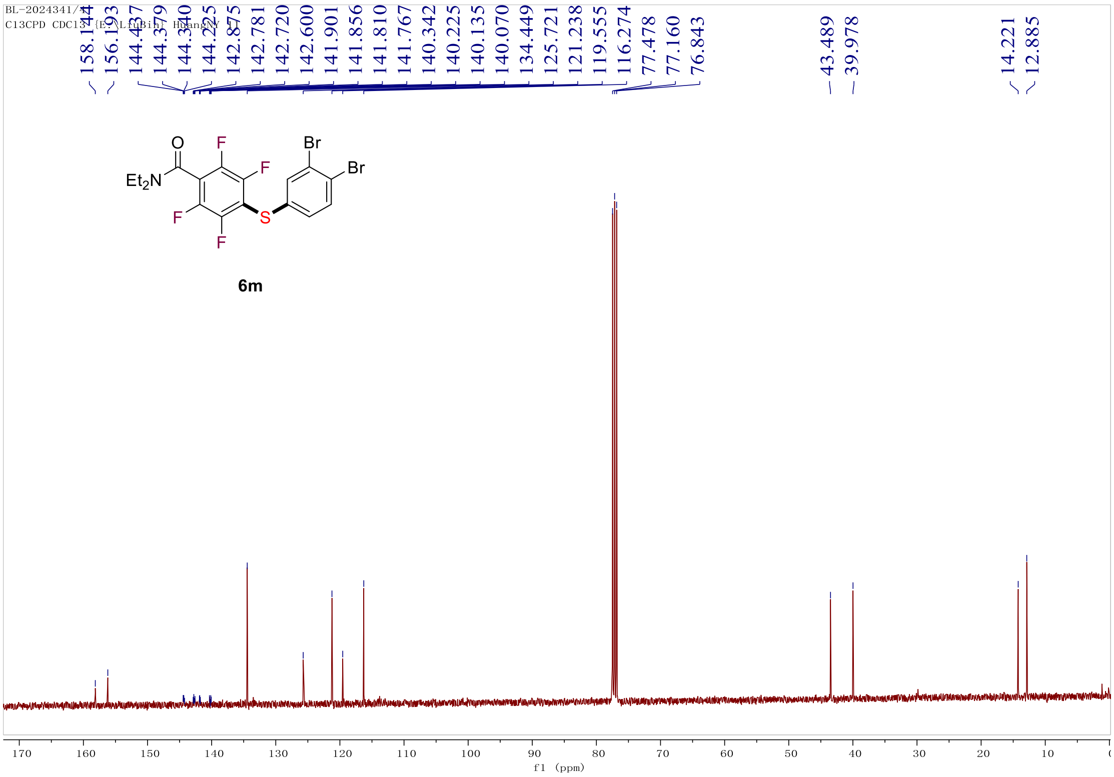
3.20
3.23
f1 (ppm)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141.767
140.342
140.225
140.135
140.070
134.449
125.721
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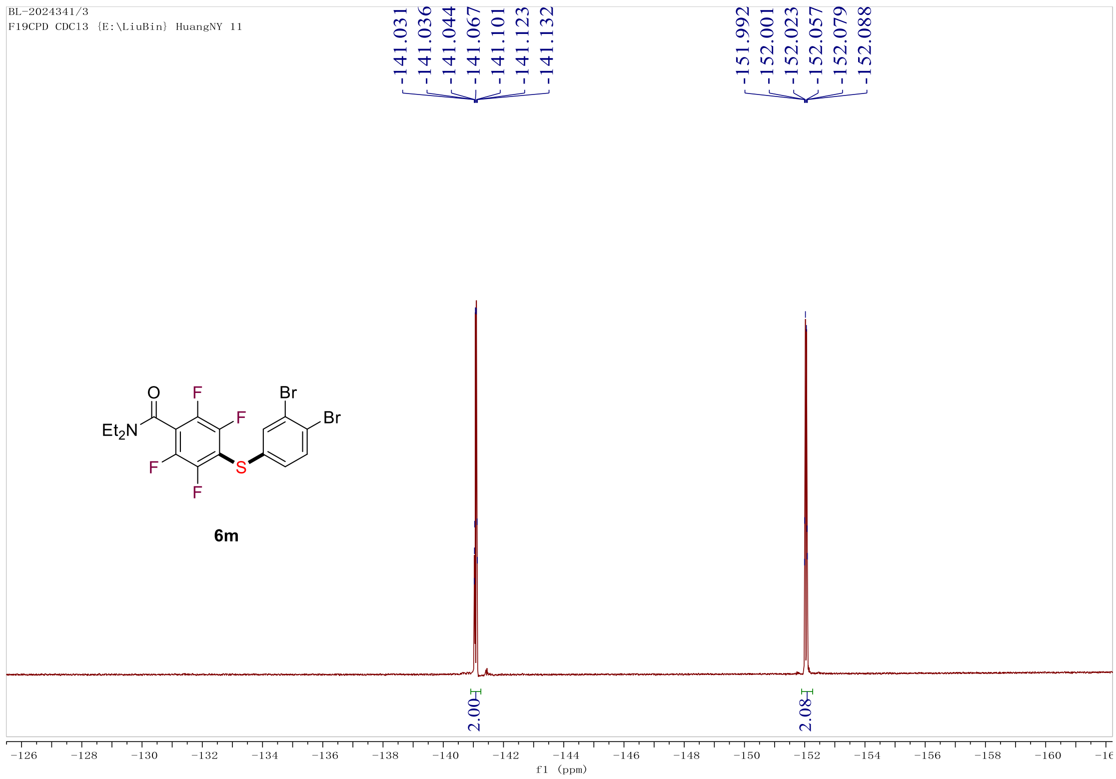
BL-2024341/3
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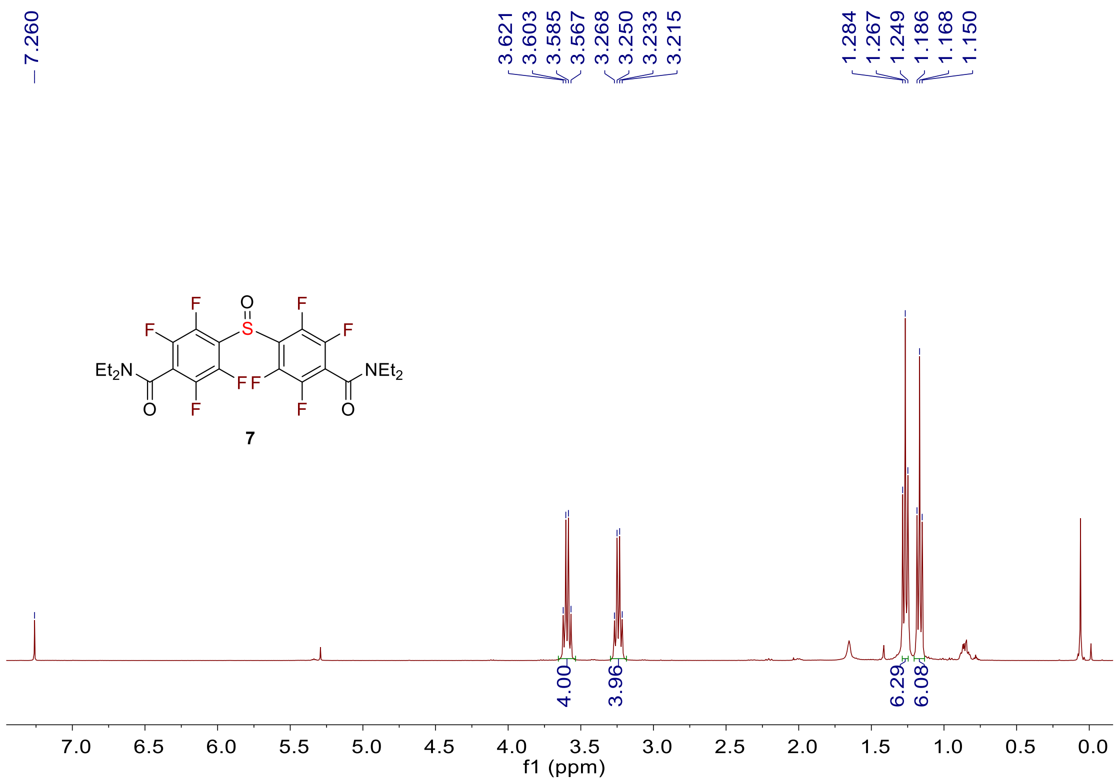
-152.001
-152.023
-152.057
-152.079
-152.088
O
F
Br
F
Br
Et_2N
S
F
F
6m
2.00
2.08
f1 (ppm)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3.233
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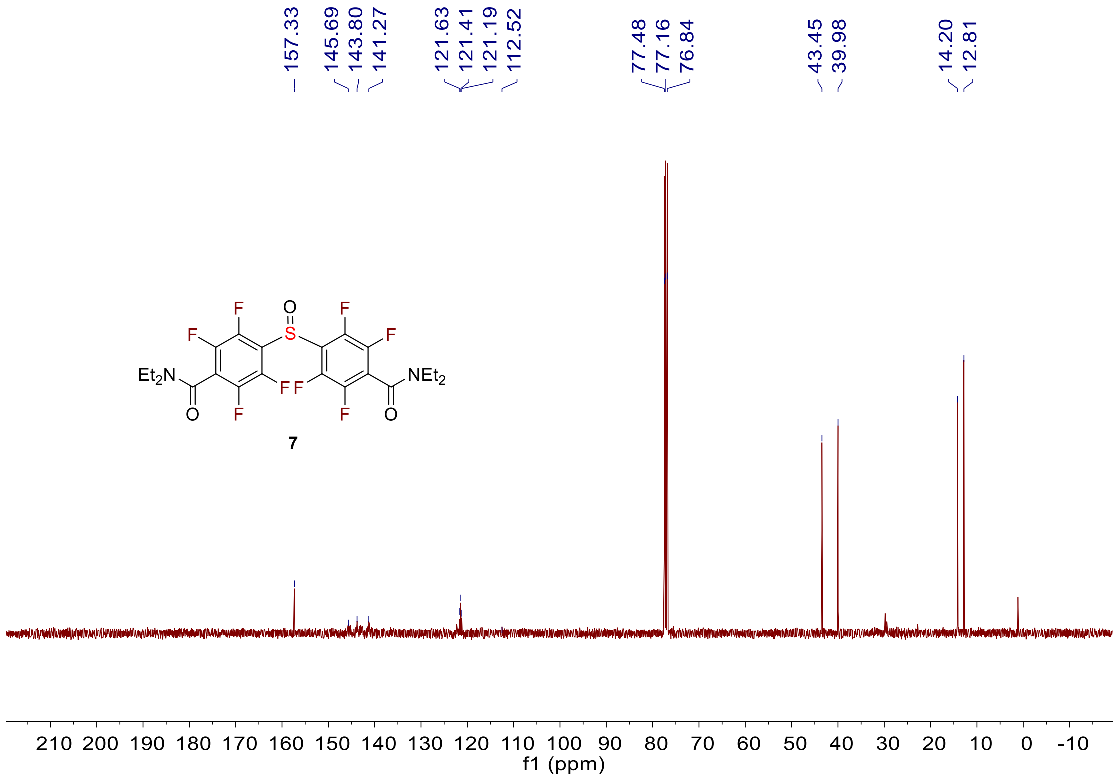
1.267
1.249
1.186
1.168
1.150
F
O
F
F
S
F
Et_2N
F F
NEt_2
O
F
F
O
7
4.00
3.96
6.29
6.08
f1 (ppm)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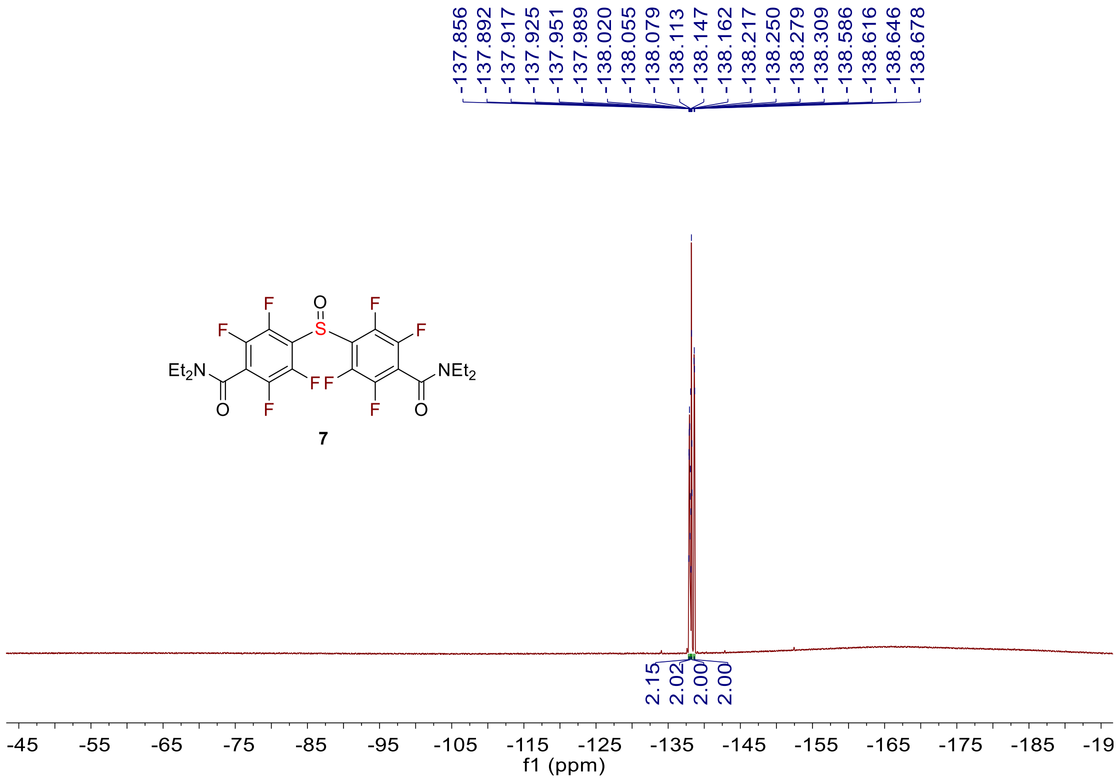
39.98
14.20
12.81
F
O
S
F
F
F
Et2N
NEt2
F F
F
F
O
O
7
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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F
O
S
F
F
F
Et2N
NEt2
F F
F
F
O
O
7
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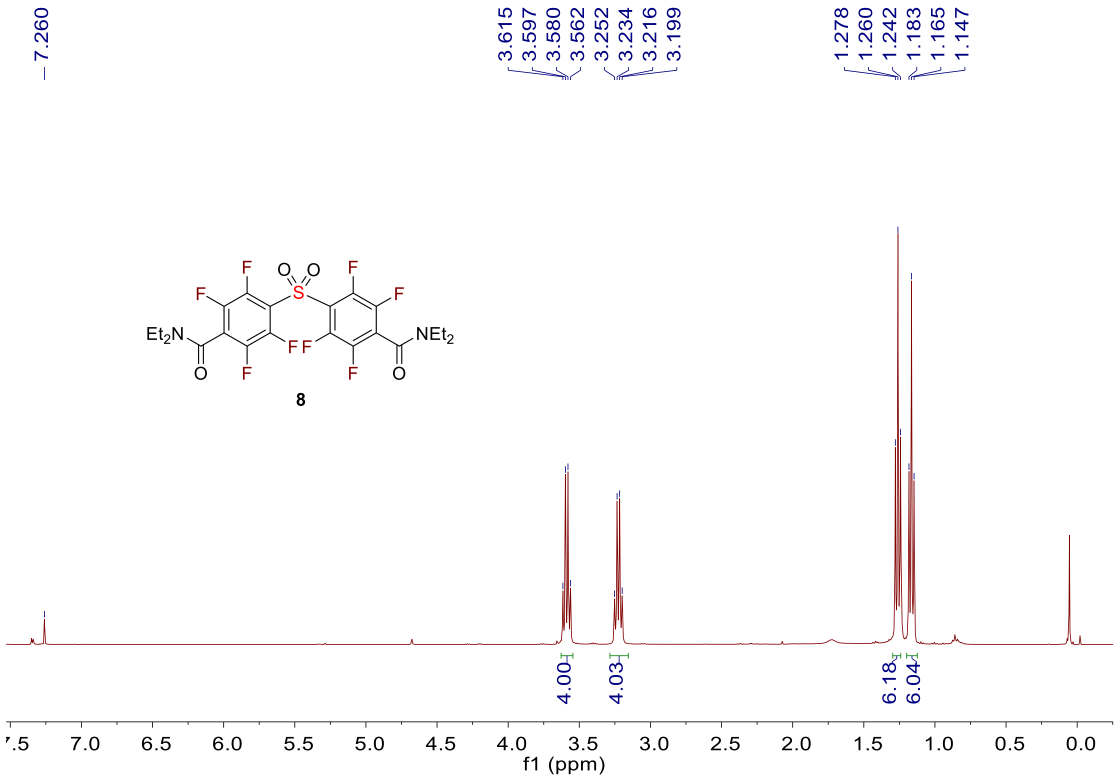
2.02
2.00
2.00
-45 -55 -65 -75 -85 -95 -105 -115 -125 -135 -145 -155 -165 -175 -185 -19
f1 (ppm)



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3.199
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1.260
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1.183
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O
O
S
Et2N
NEt2
8
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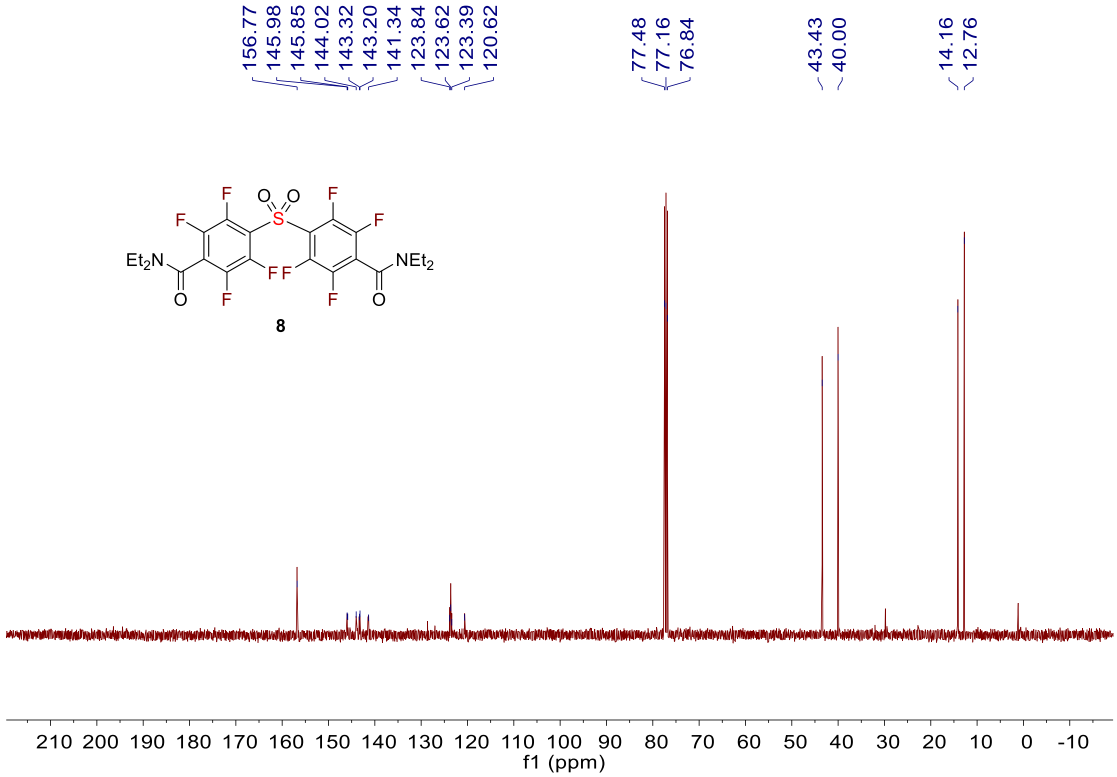
6.04
f1 (ppm)



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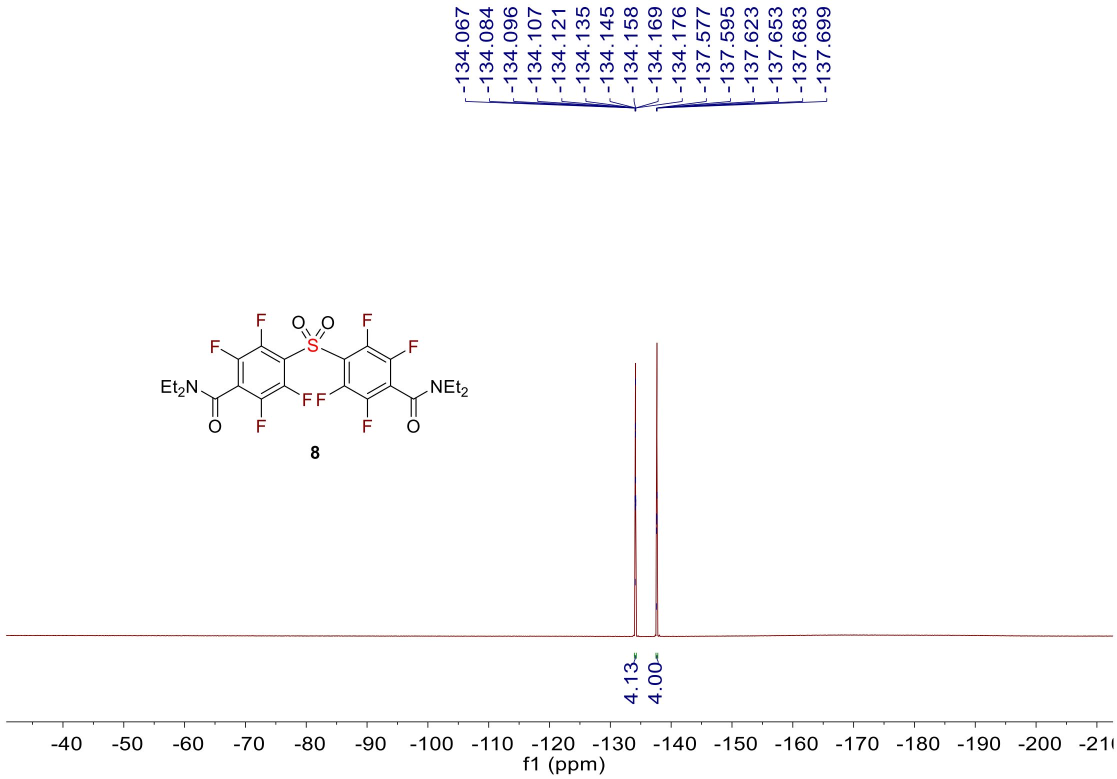
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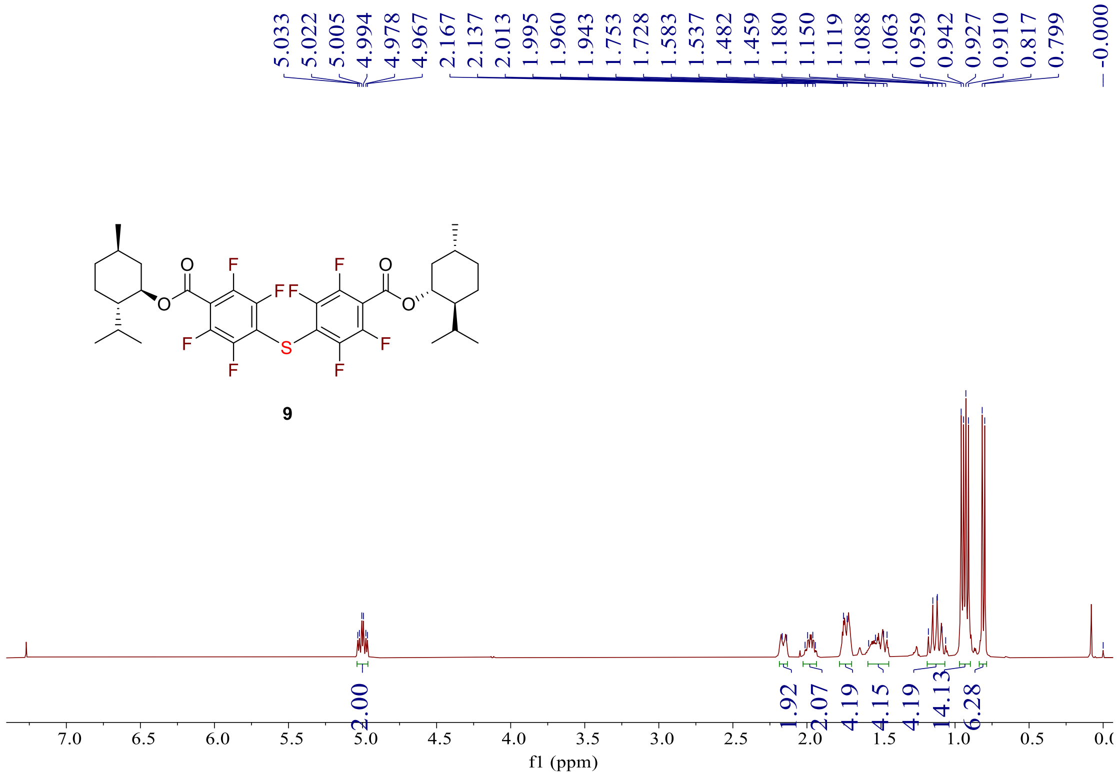
4.00
f1 (ppm)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1.728
1.583
1.537
1.482
1.459
1.180
1.150
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1.088
1.063
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0.942
0.927
0.910
0.817
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2.07
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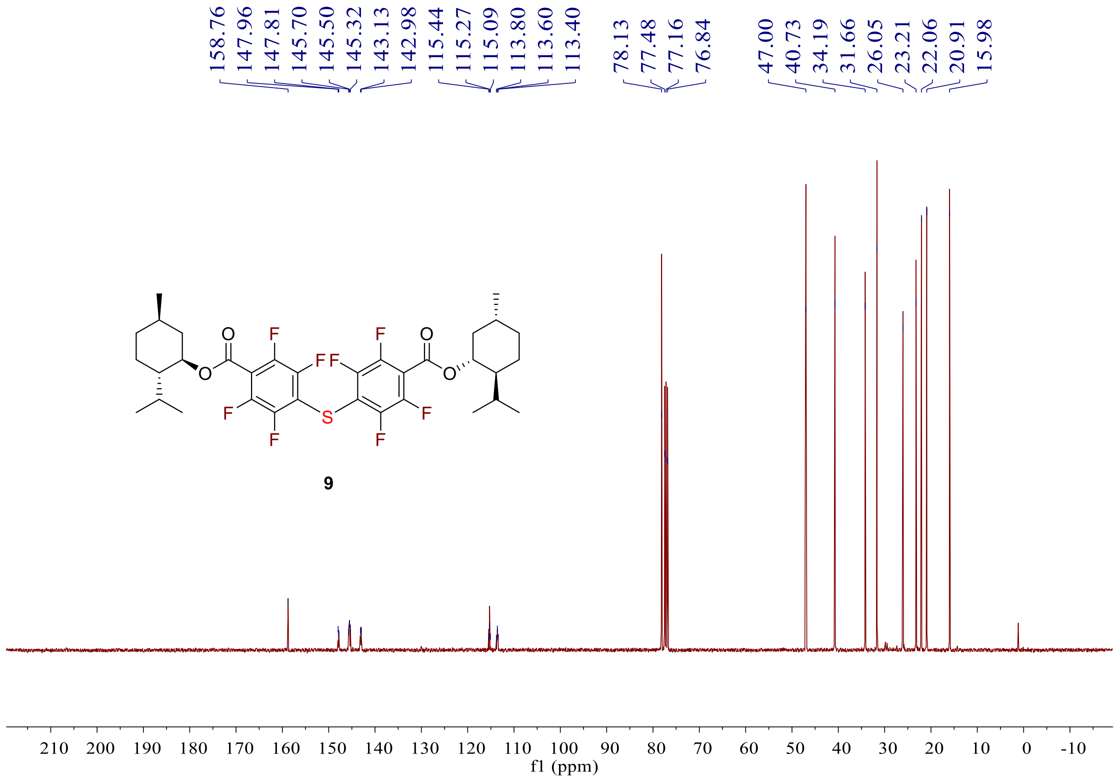
6.28
f1 (ppm)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113.60
113.40
78.13
77.48
77.16
76.84
47.00
40.73
34.19
31.66
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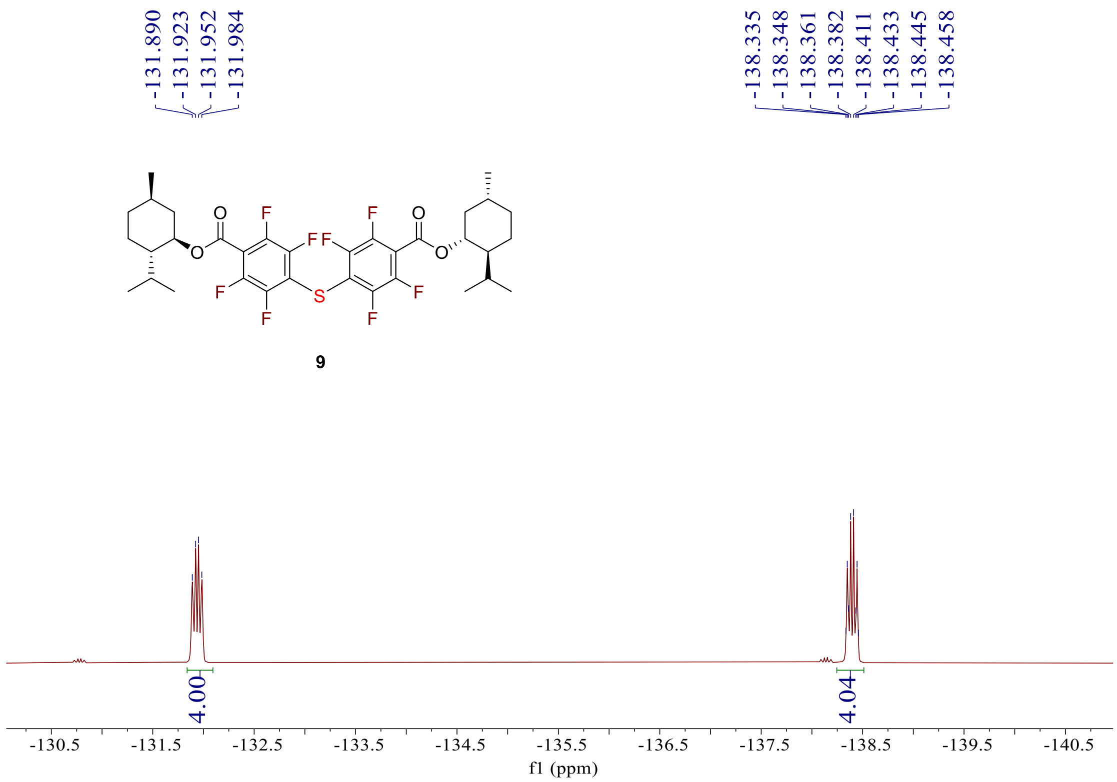
f1 (ppm)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9
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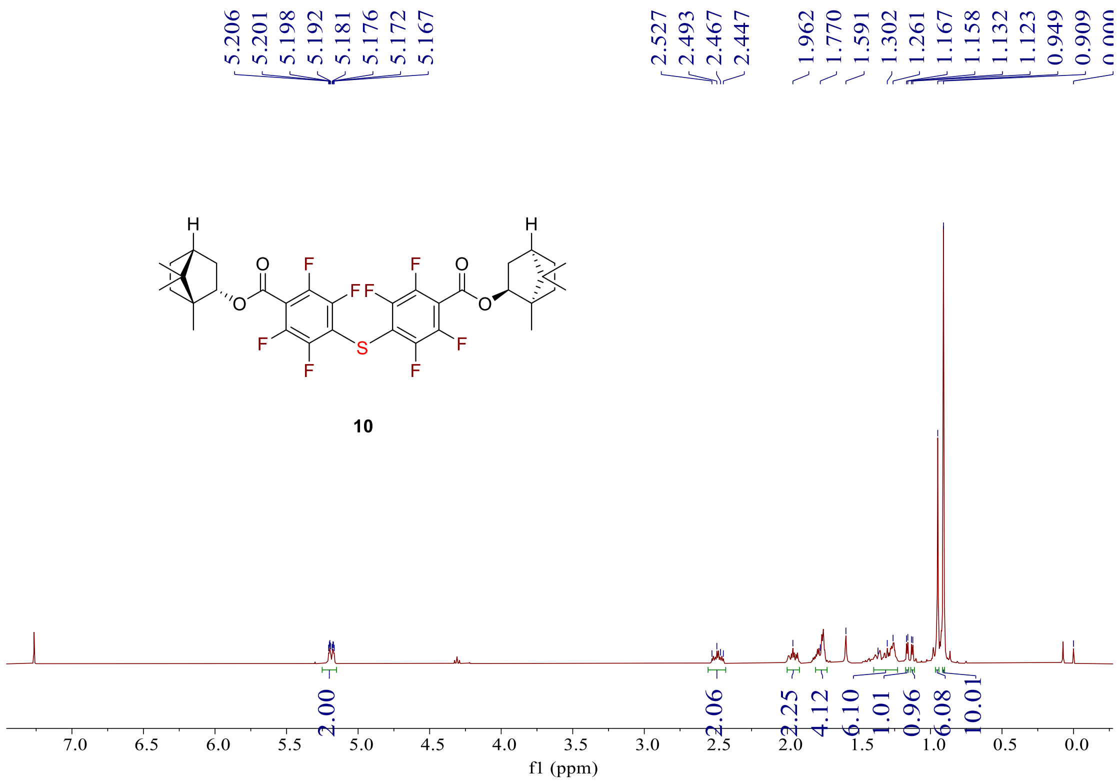
f1 (ppm)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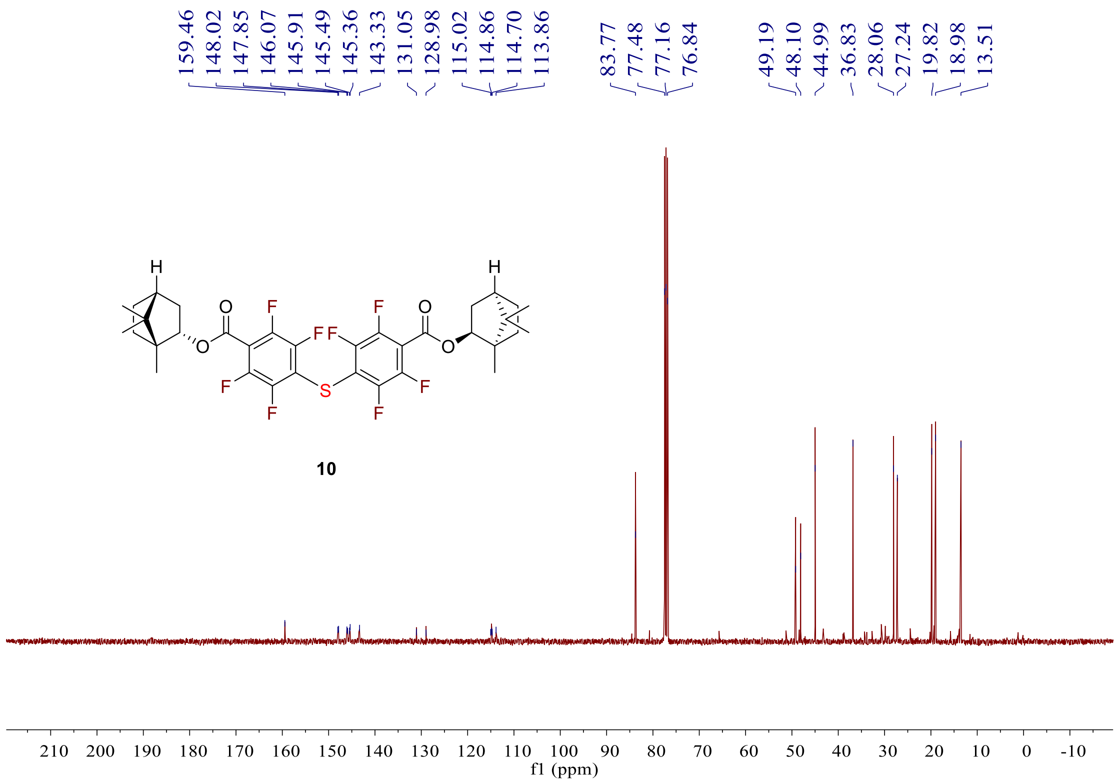
2.467
2.447
1.962
1.770
1.591
1.302
1.261
1.167
1.158
1.132
1.123
0.949
0.909
0.000
2.00
2.06
2.25
4.12
6.10
1.01
0.96
6.08
10.01
f1 (ppm)

10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148.02
147.85
146.07
145.91
145.49
145.36
143.33
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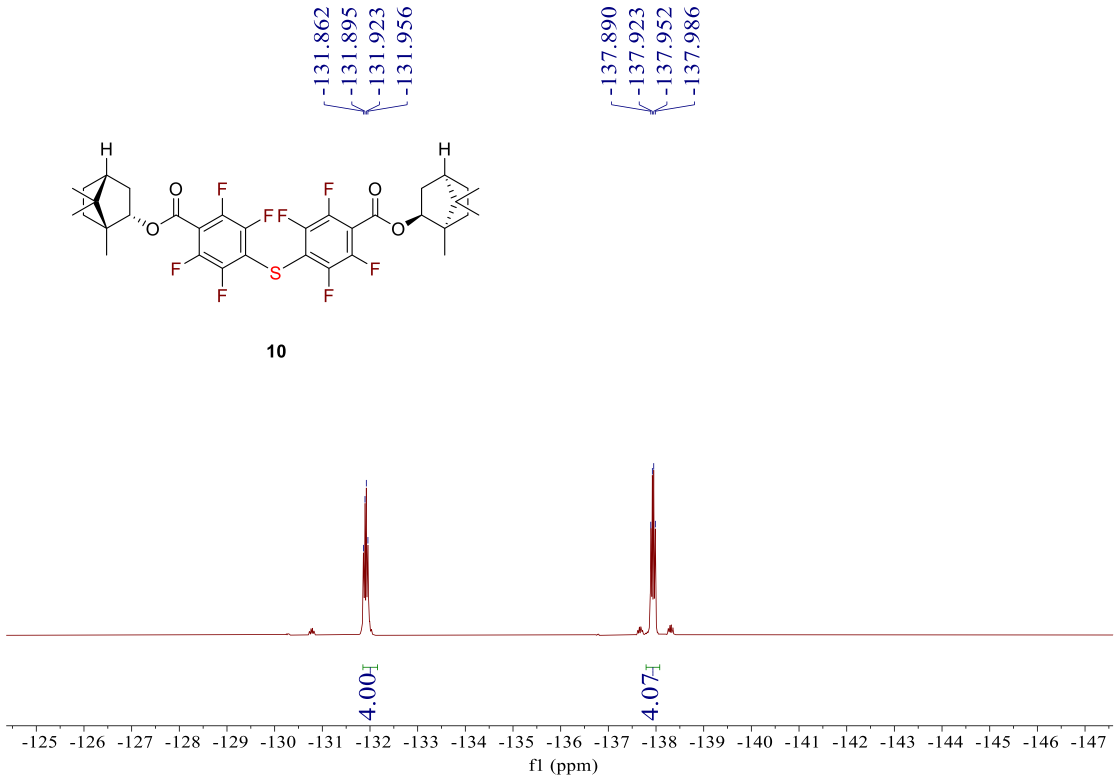
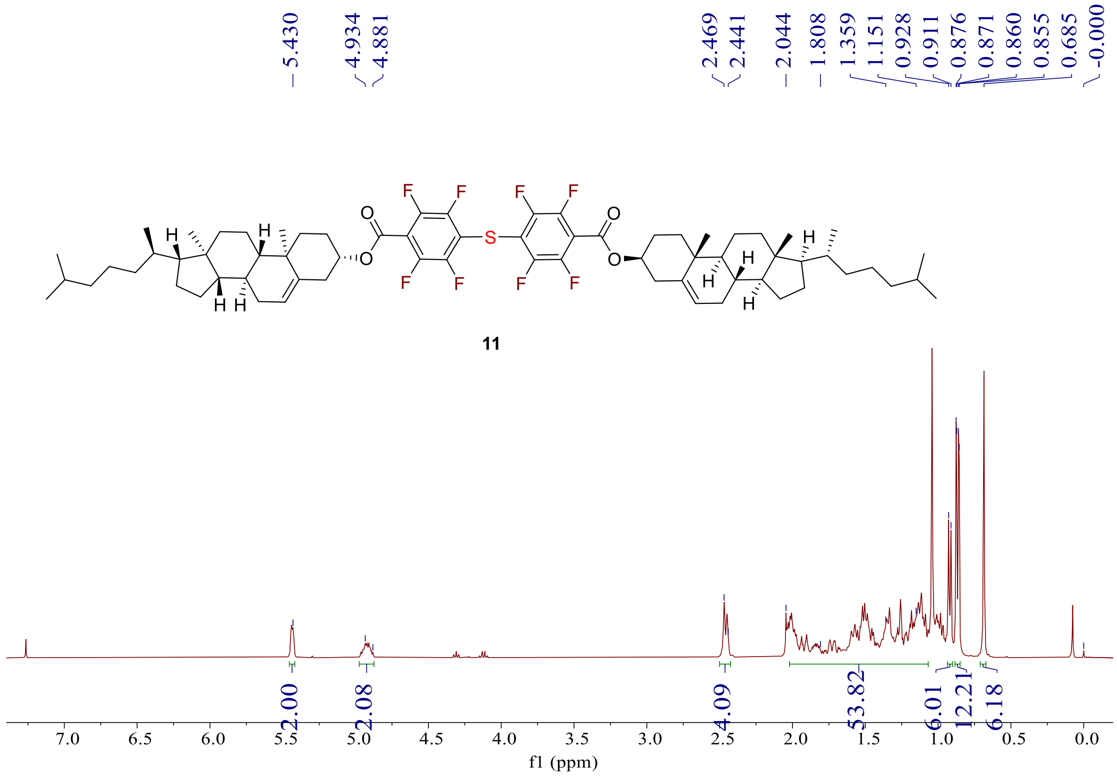
128.98
115.02
114.86
114.70
113.86
83.77
77.48
77.16
76.84
49.19
48.10
44.99
36.83
28.06
27.24
19.82
18.98
13.51
f1 (ppm)

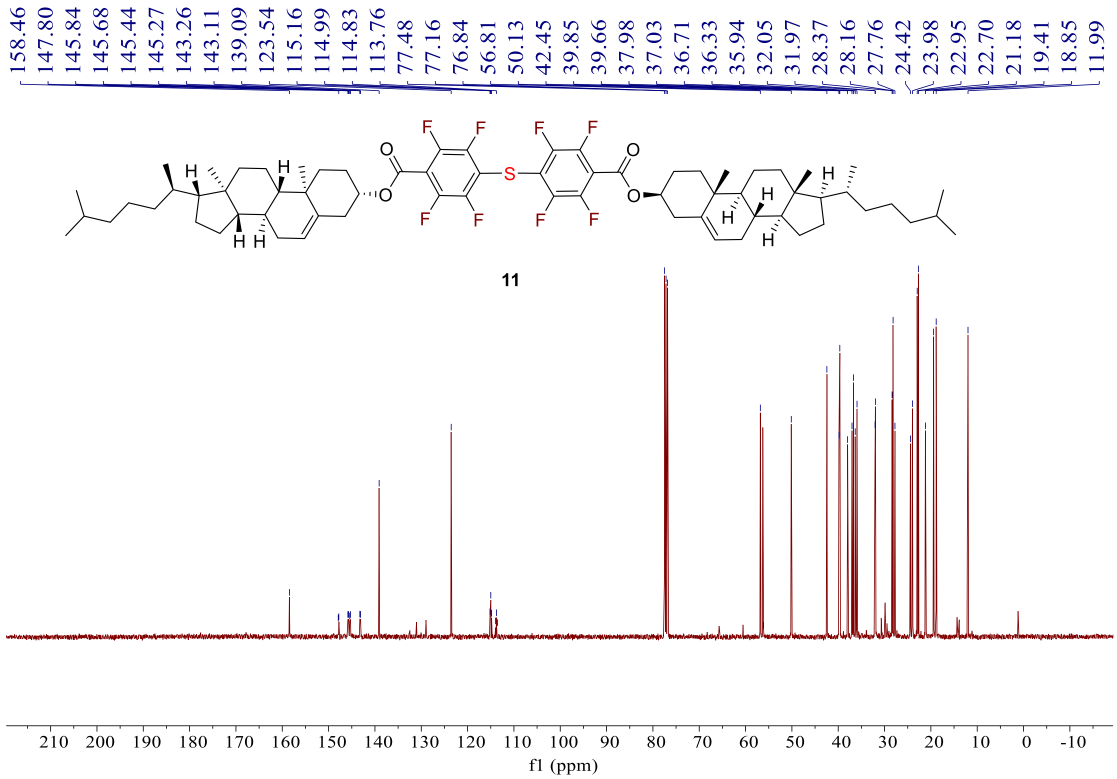
10

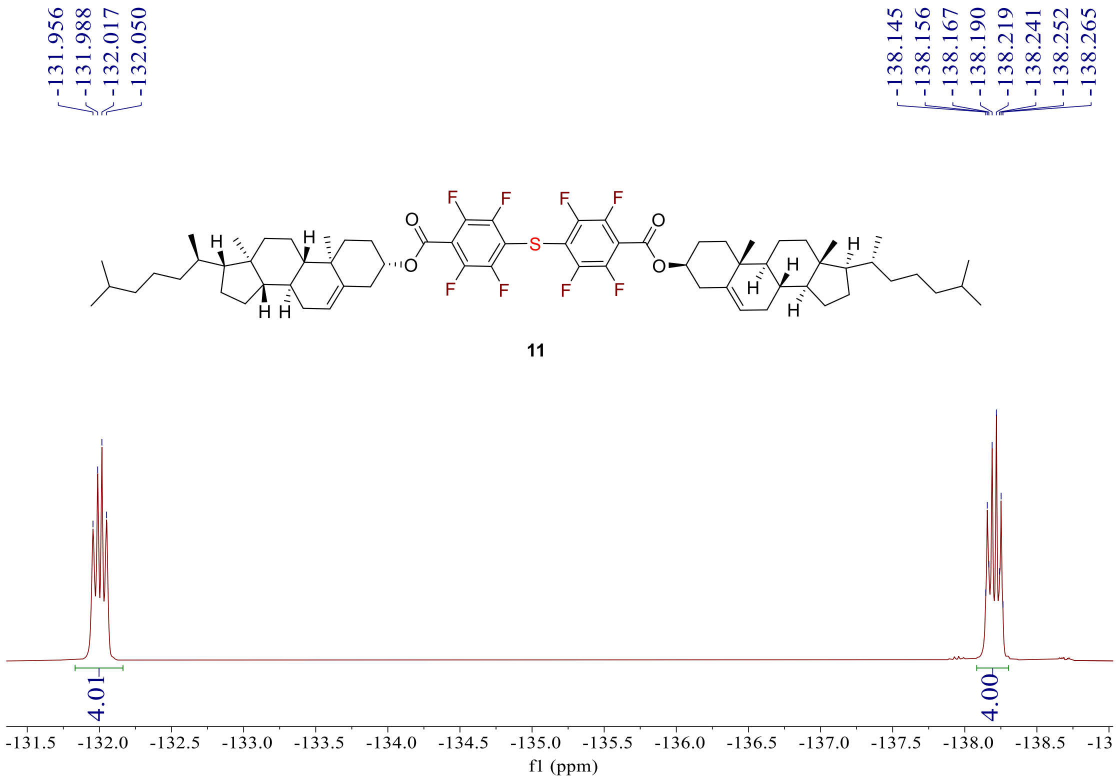

-131.862
-131.895
-131.923
-131.956
-137.890
-137.923
-137.952
-137.986
4.00
4.07
-125 -126 -127 -128 -129 -130 -131 -132 -133 -134 -135 -136 -137 -138 -139 -140 -141 -142 -143 -144 -145 -146 -147
f1 (ppm)

10


5.430
4.934
4.881
2.469
2.441
2.044
1.808
1.359
1.151
0.928
0.911
0.876
0.871
0.860
0.855
0.685
-0.000
2.00
2.08
4.09
53.82
6.01
12.21
6.18
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

11


158.46
147.80
145.84
145.68
145.44
145.27
143.26
143.11
139.09
123.54
115.16
114.99
114.83
113.76
77.48
77.16
76.84
56.81
50.13
42.45
39.85
39.66
37.98
37.03
36.71
36.33
35.94
32.05
31.97
28.37
28.16
27.76
24.42
23.98
22.95
22.70
21.18
19.41
18.85
11.99
11
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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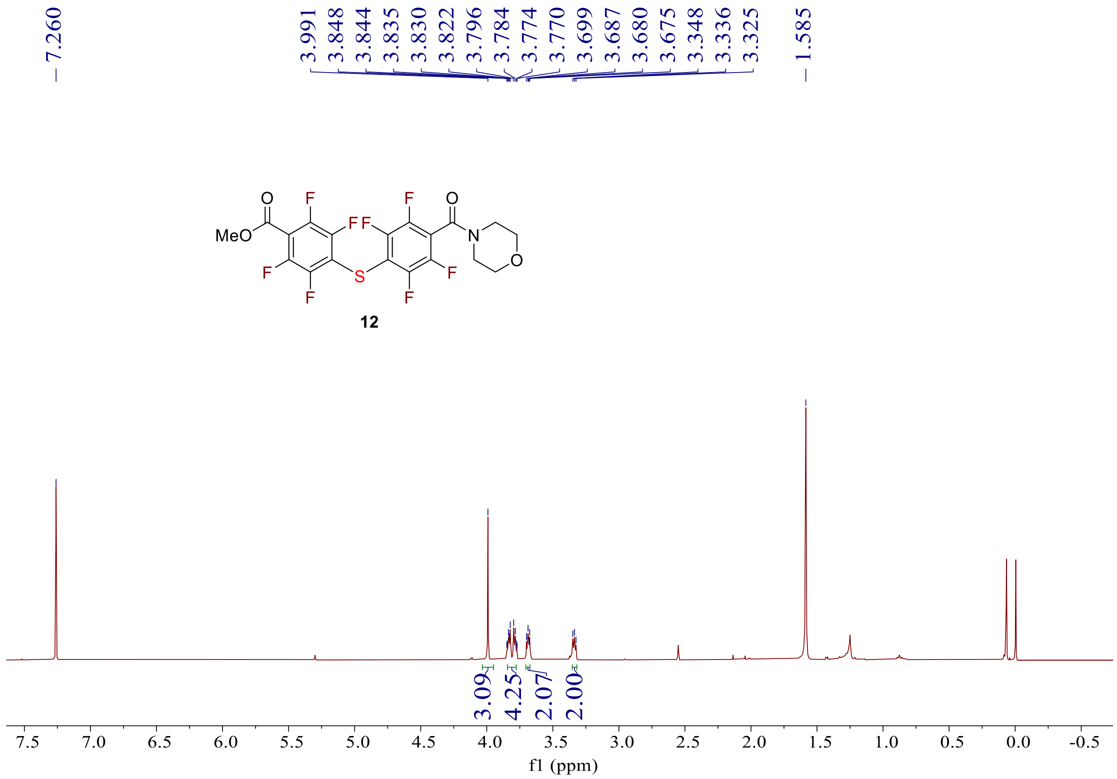
-132.017
-132.050
-138.145
-138.156
-138.167
-138.190
-138.219
-138.241
-138.252
-138.265
11
4.01
4.00
-131.5 -132.0 -132.5 -133.0 -133.5 -134.0 -134.5 -135.0 -135.5 -136.0 -136.5 -137.0 -137.5 -138.0 -138.5 -13
f1 (ppm)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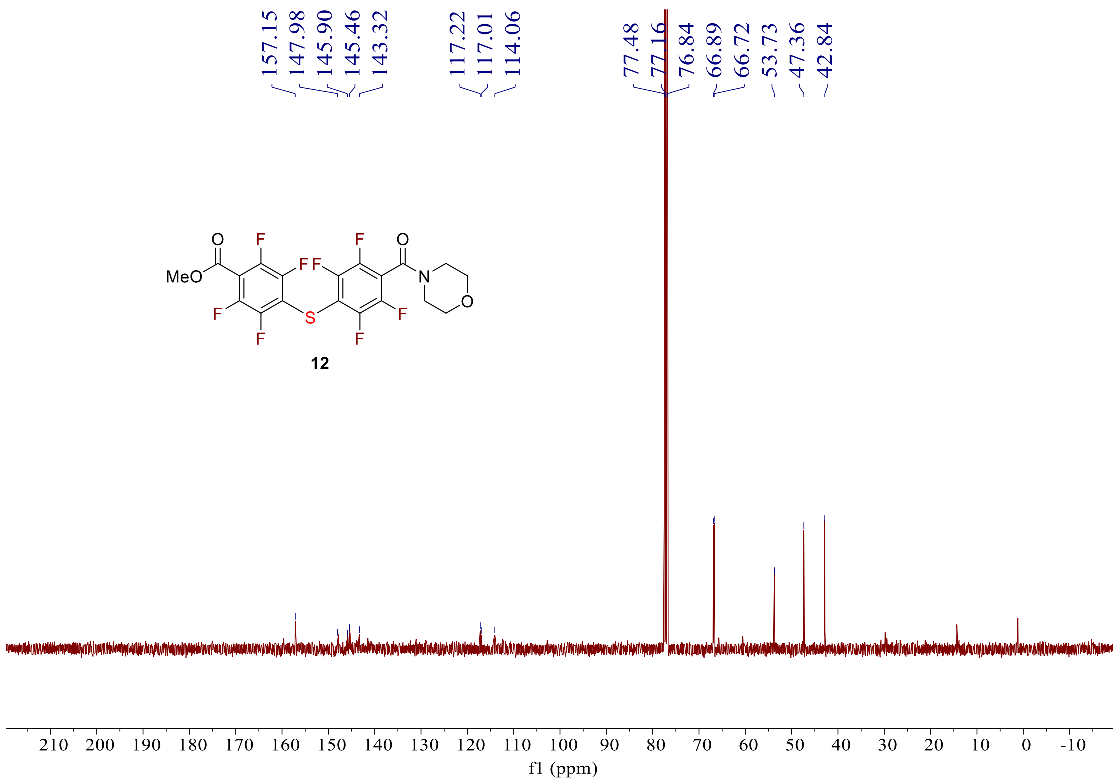
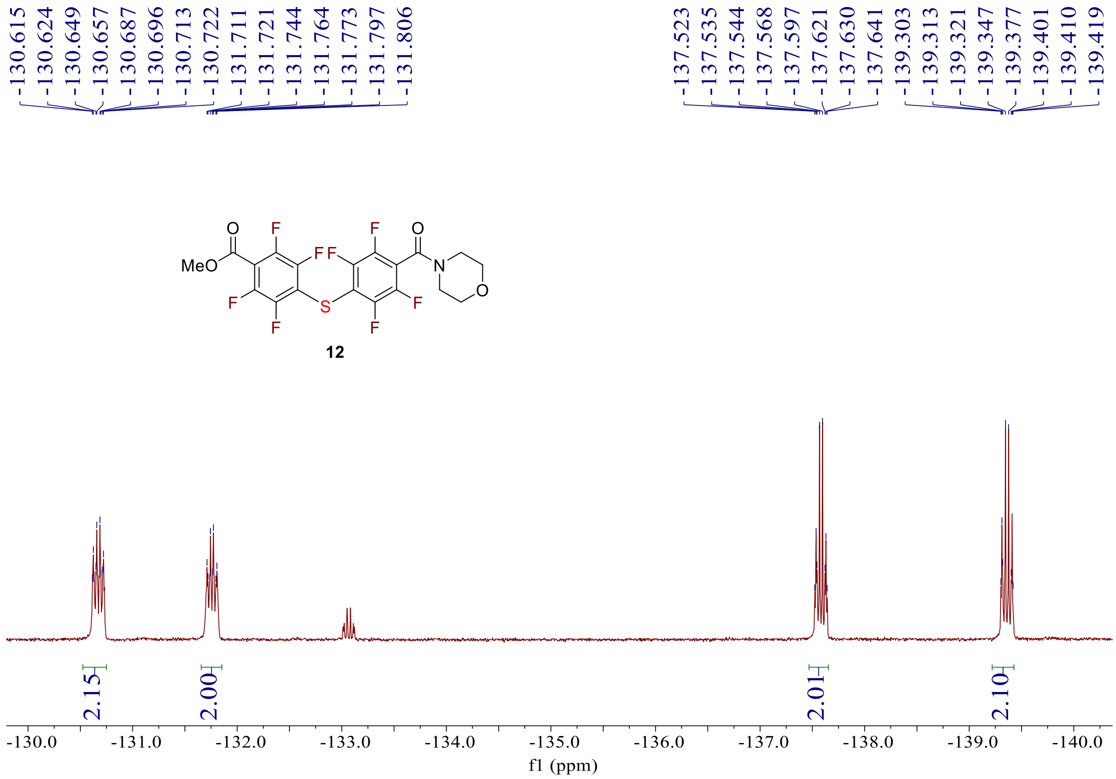
3.991
3.848
3.844
3.835
3.830
3.822
3.796
3.784
3.774
3.770
3.699
3.687
3.680
3.675
3.348
3.336
3.325
1.585
MeO
O
F
S
N
O
12
3.09
4.25
2.07
2.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


157.15
147.98
145.90
145.46
143.32
117.22
117.01
114.06
77.48
77.16
76.84
66.89
66.72
53.73
47.36
42.84
MeO
O
F
S
N
O
12
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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-130.624
-130.649
-130.657
-130.687
-130.696
-130.713
-130.722
-131.711
-131.721
-131.744
-131.764
-131.773
-131.797
-131.806
-137.523
-137.535
-137.544
-137.568
-137.597
-137.621
-137.630
-137.641
-139.303
-139.313
-139.321
-139.347
-139.377
-139.401
-139.410
-139.419
MeO
O
F
F
F
F
S
F
F
F
F
O
N
O
12
2.15
2.00
2.01
2.10
-130.0
-131.0
-132.0
-133.0
-134.0
-135.0
-136.0
-137.0
-138.0
-139.0
-140.0
f1 (ppm)