Supporting information

Photocatalytic method for the generation of the 1,1,1,3,3,3-hexafluoroisopropyl radical

Sergey S. Lunkov,[a] Vladislav S. Kostromitin,[a,b] Artem A. Zemtsov,[a] Vitalij V. Levin[a] and Alexander D. Dilman[a]*

[a] N. D. Zelinsky Institute of Organic Chemistry, 119991 Moscow, Leninsky prosp. 47, Russian Federation

[b] Lomonosov Moscow State University, Department of Chemistry, 119991 Moscow, Leninskie Gory 1-3, Russian Federation

E-mail: adil25@mail.ru

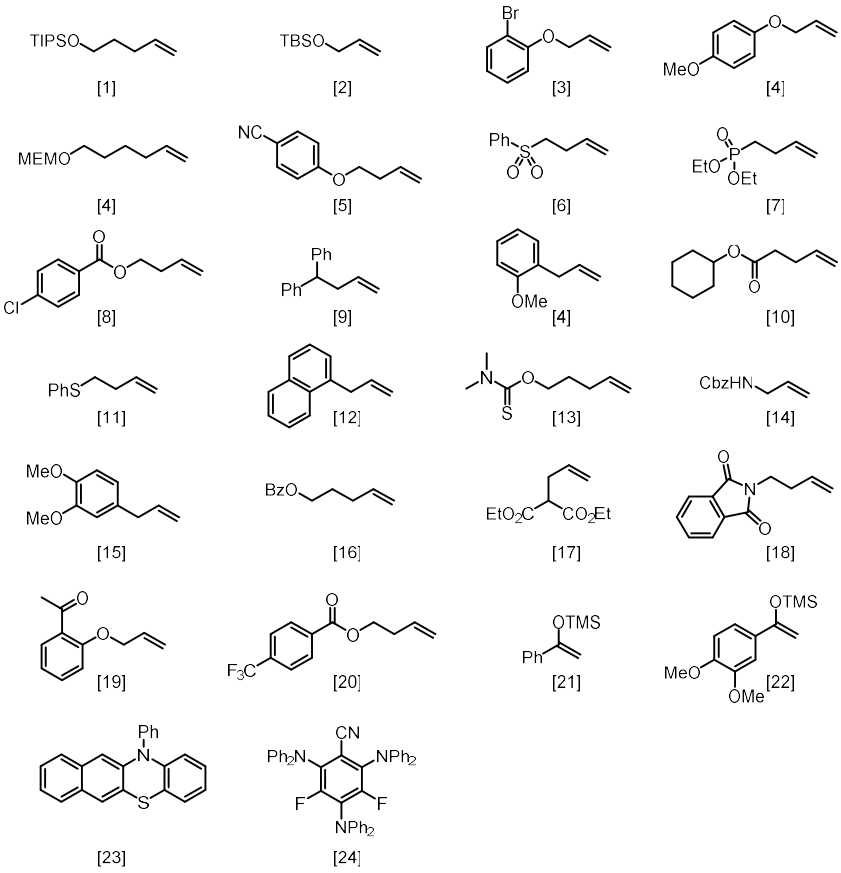
Content

General methods

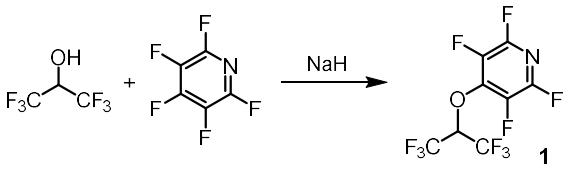
All reactions were performed in an atmosphere of dry argon. Column chromatography was carried out employing silica gel (230−400 mesh). Precoated silica gel plates F-254 were used for thin-layer analytical chromatography visualizing with UV and/or acidic aq. $KMnO_4$ solution. High resolution mass-spectra (HRMS) were measured using electrospray ionization (ESI) and a time-of-flight (TOF) mass analyzer (Bruker MicrOTOF II). The measurements were done in a positive-ion mode (interface capillary voltage −4500 V) or in a negative-ion mode (3200 V); the mass ranged from m/z 50 to m/z 3000. For NMR measurements, a Bruker AM300 spectrometer was used. Infrared spectra were recorded on Brucker Alpha-T spectrometer. Photo-induced reactions were performed in Duran culture tubes (Roth cat. no K248.1, outside diameter = 12 mm). The reaction tube was placed in a glass jacket covered with aluminium foil and cooled with water (18 °C). For irradiation with 400 nm light, COB LED matrix Hontiey (29-32 V, 3000 mA, 100W; the LED matrix was operated at 80W or 60W). For irradiation with 450 nm light, a strip of light emitting diodes (SMD 2835−120 LED 1 M Blue, 12 V, 24 W/m; 50 cm strip length; operated at 10W) was used. The distance between the reaction vessel and diodes was about 1 cm.

Starting materials

DMF, DMSO, and MeCN were distilled from CaH_2 and stored over MS 4Å. Diethyl ether was distilled from lithium aluminum hydride. Following compounds were obtained according to literature procedures:

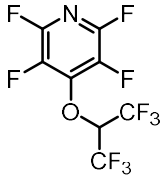


Reagent 1



In a round bottom flask in a stream of argon, sodium hydride (4.0 g, 60% in mineral oil, 100 mmol, 1.2 eq.) was washed with hexane (3×3 mL), and 50 mL of freshly distilled diethyl ether was added. The mixture was immersed into room temperature water bath. Hexafluoroisopropanol (HFIP) (16.8 g, 100 mmol, 1.2 eq.) was added dropwise via a syringe, and the mixture was stirred 10 minutes. The bath was replaced by ice/water, and the mixture was cooled to 0 °C, and then pentafluoropyridine (14.1 g, 83.3 mmol, 1 eq.) was rapidly added. The mixture was allowed to warm up to room temperature over 2 hours, while keeping the flask in the bath. The reaction was quenched with a solution of saturated ammonium chloride (10 mL). The organic layer was separated, the aqueous phase extracted with hexane (3×5 mL). The combined organic layers were dried over Na_2SO_4 and carefully fractionally distilled under stream of argon at ambient pressure. The fraction boiling at 142-143 °C was collected affording 25.3 g (80%). This material contains 5% impurity of the pentafluoropyridine disubstitution product. The resulting impurity does not interfere with the subsequent fluoroalkylation reaction. Its content can be reduced to 2% after additional fractional distillation through a column packed with Raschig rings.

2,3,5,6-Tetrafluoro-4-((1,1,1,3,3,3-hexafluoropropan-2-yl)oxy)pyridine (1)



Yield 25.3 g (80%), colorless liquid. Bp 142-143 °C.

^{1}H NMR (300 MHz, Chloroform-d) δ 5.24 (hept, *J* = 5.2 Hz, 1H).

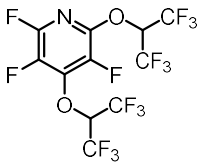
^{13}C NMR (75 MHz, Chloroform-d) δ 144.53 (dtd, *J* = 245.2, 15.1, 2.9 Hz)., 144.4 (tt, *J* = 10.0, 5.2 Hz), 138.0 – 133.7 (m), 120.5 (qq, *J* = 284.3, 2.4 Hz)), 77.0 (hept. t, *J* = 35.0, 3.8 Hz).

^{19}F NMR (282 MHz, Chloroform-d) δ -73.75 (dt, *J* = 5.1, 2.5 Hz, 6F), -86.65 – -87.02 (m, 2F), -155.85 – -156.18 (m, 2F).

MS (EI): 317 [M], 298 [M-F], 248 [M-CF_3], 69 [CF_3].

HRMS (ESI-TOF): calcd for $C_8F_{10}NO$, [M^+] 316.9893; found 316.9882.

Impurity



Selected signals:

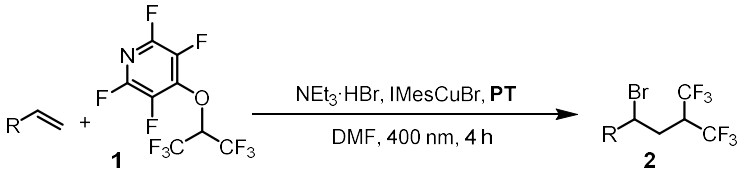
1H NMR (300 MHz, Chloroform-d) δ 6.12 – 6.01 (m, 1H).

^{13}C NMR (75 MHz, Chloroform-d) δ 69.56 (hept, J = 35.4 Hz).

^{19}F NMR (282 MHz, Chloroform-d) δ -73.36, -73.38, -88.28 (dd, J = 26.7, 20.2 Hz), -154.76 (d, J = 26.7 Hz), -159.07 (d, J = 20.1 Hz).

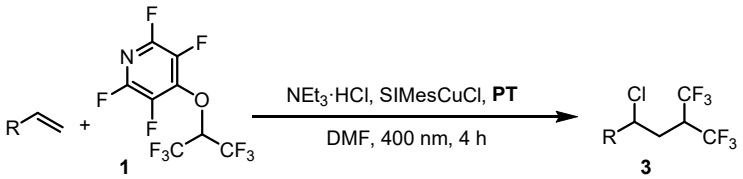
MS (EI): 465 [M], 446 [M-F], 396 [M-CF_3], 69 [CF_3].

Synthesis of bromides 2 (General Procedure A)



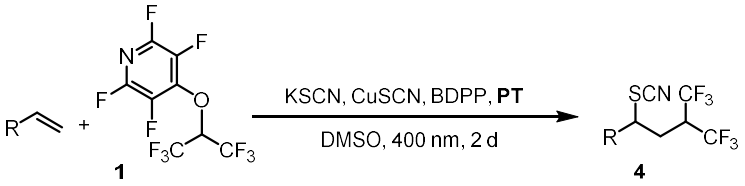
A screw test tube charged with NEt_3·HBr (110 mg, 0.6 mmol 1.2 eq.), IMesCuBr (22 mg, 0.05 mmol 10 mol%), 12-phenyl-12H-benzo[b]phenothiazine (**PT**) (4 mg, 3 mol%) and a PTFE coated stirring bar was evacuated-back filled with argon three times, then dry DMF (1.0 mL) was added. Alkene (0.5 mmol, 1 eq.) and reagent **1** (238 mg, 0.75 mmol, 1.5 eq.) were added with a syringe. The tube was sealed and irradiated using 80W 400 nm LED for 4 hours. For the work-up, the reaction was quenched with water (4 mL) and the resulting mixture was extracted with MTBE (3×3 mL). The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel.

Synthesis of chlorides 3 (General Procedure B)



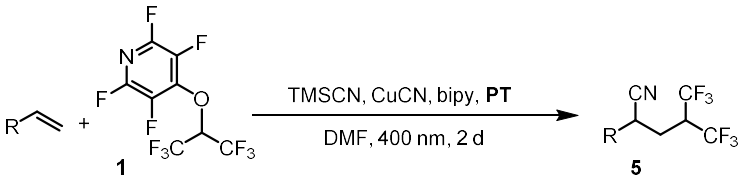
A screw test tube charged with NEt_3·HCl (82 mg, 0.6 mmol 1.2 eq.), SIMesCuCl (20 mg, 0.05 mmol 10 mol%), 12-phenyl-12H-benzo[b]phenothiazine (**PT**) (4 mg, 3 mol%) and a PTFE coated stirring bar was evacuated-back filled with argon three times, then dry DMF (1.0 mL) was added. Alkene (0.5 mmol, 1 eq.) and reagent **1** (238 mg, 0.75 mmol, 1.5 eq.) were added with a syringe. The tube was sealed and irradiated using 80W 400 nm LED for 4 hours. For the work-up, the reaction was quenched with water (4 mL) and the resulting mixture was extracted with MTBE (3×3 mL). The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel.

Synthesis of thiocyanates 4 (General Procedure C)



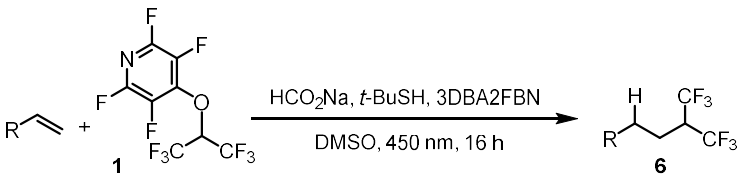
A screw test tube charged with CuSCN (6 mg, 0.05 mmol 10 mol%), KSCN (58 mg, 0.6 mol, 1.2 eq.), 2,6-bis(3,5-dimethyl-1H-pyrazol-1-yl)pyridine (BDPPENT, **L1**) (15 mg, 0.05 mmol 10 mol%), 12-phenyl-12H-benzo[b]phenothiazine (**PT**) (4 mg, 3 mol%) and a PTFE coated stirring bar was evacuated-back filled with argon three times, then dry DMSO (1.0 mL) was added. Alkene (0.5 mmol, 1 eq.) and reagent **1** (238 mg, 0.75 mmol, 1.5 eq.) were added with a syringe. The tube was sealed and irradiated using 60W 400 nm LED for 2 days. For the work-up, the reaction was quenched with water (4 mL) and the resulting mixture was extracted with MTBE (3×3 mL). The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel.

Synthesis of nitriles 5 (General Procedure D)



A screw test tube charged with CuCN (5 mg, 0.05 mmol 10 mol%), 2,2'-bipyridine (bipy) (9 mg, 0.05 mmol 10 mol%), 12-phenyl-12H-benzo[b]phenothiazine (**PT**) (4 mg, 3 mol%) and a PTFE coated stirring bar was evacuated-back filled with argon three times, then dry DMF (1.0 mL) was added. Alkene (0.5 mmol, 1 eq.), trimethylsilyl cyanide (TMSCN) (59 mg, 75 µl, 0.6 mmol, 1.2 eq.) and reagent **1** (238 mg, 0.75 mmol, 1.5 eq.) were added with a syringe. The tube was sealed and irradiated using 400 nm 60W LED for 2 days. For the work-up, the reaction was quenched with water (4 mL) and the resulting mixture was extracted with MTBE (3×3 mL). The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel.

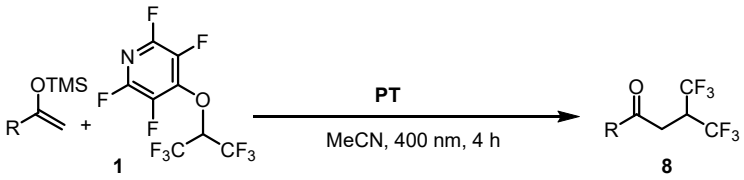
Synthesis of compounds 6 (General procedure E)



A test tube was evacuated and filled with argon. Then, DMSO (1 mL), reagent **1** (317 mg, 1 mmol, 2 eq.), alkene (0.5 mmol, 1 eq.), HCO_2Na (68 mg, 1 mmol, 2 eq.), 2-methylpropane-2-thiol (6 µl,

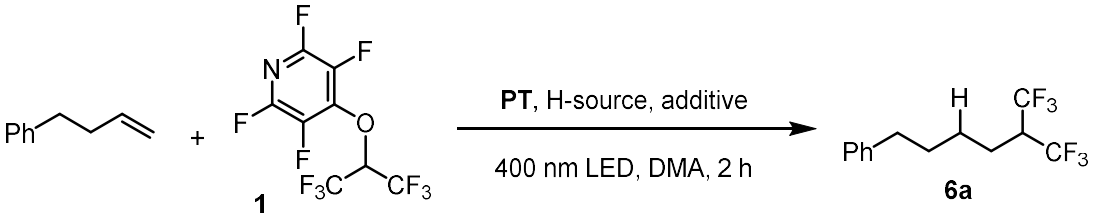
0.05 mmol, 10 mol%), 3DPA2FBN (1.6 mg, 2.5 μmol, 0.5 mol%) were added. The tube was screw-capped and irradiated using 450 nm 10W LED strip for 16 hours. For the work-up, the reaction was quenched with water (5 mL) and extracted with hexane (3×1.5 mL). The combined organic phases were filtered through a short pad of Na_2SO_4 and concentrated on a rotary evaporator. The residue was purified by column chromatography on silica gel.

Synthesis of ketones 8 (General procedure F)



A screw test tube charged with, 12-phenyl-12H-benzo[b]phenothiazine (**PT**) (4 mg, 3 mol%) and a PTFE coated stirring bar was evacuated-back filled with argon three times, then dry MeCN (1.0 mL) was added. Silyl enol ether (0.5 mmol, 1 eq.) and reagent **1** (238 mg, 0.75 mmol, 1.5 eq.) were added with a syringe. The tube was sealed and irradiated using and 400 nm 80W LED for 4 hours. For the work-up, reaction was quenched with water (4 mL) and the resulting mixture was extracted with MTBE (3×3 mL). The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel.

Variation of the source of hydrogen

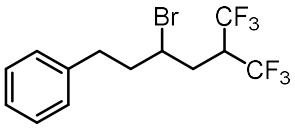


#	H-source	Additive	Conv. of alkene, %	Yield of **6a**, %[a]
1	γ-terpinene	-	41	38
2	DIPEA	CySH	39	21
3	Ascorbic acid	NEt_3	20	13
4	Hantzsch ester	-	trace	trace
5	*t*-Bu-triazinane[b]	-	26	14

[a] Determined by ^{19}F NMR with $PhCF_3$ as an internal standard.
[b] 1,3,5-Tri-*tert*-butyl-1,3,5-triazinane.

Characterization of compounds

(3-Bromo-6,6,6-trifluoro-5-(trifluoromethyl)hexyl)benzene (2a)



General Procedure A.

Yield 150.2 mg (83%). Colorless liquid.

Chromatography: hexanes/EtOAc, 20/1.

Final purification was performed by preparative HPLC (reversed-phase column C18, 21 × 250 mm, 5 μm), flow rate 8 mL min^{-1}; mobile phase isocratic, acetonitrile/water, 15% water; tR = 22.5 min

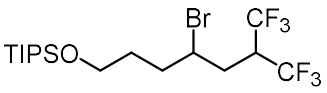
^{1}H NMR (300 MHz, Chloroform-d) δ 7.38 – 7.19 (m, 5H), 4.15 – 4.01 (m, 1H), 3.54 – 3.34 (m, 1H), 3.04 – 2.90 (m, 1H), 2.88 – 2.75 (m, 1H), 2.36 – 2.14 (m, 4H).

^{13}C NMR (76 MHz, Chloroform-d) δ 140.2, 128.8, 128.6, 126.6, 123.9 (qq, *J* = 280.6, 2.4 Hz), 123.6 (qq, *J* = 280.0, 3.3 Hz), 52.5 (q, *J* = 2.1 Hz), 47.1 (hept, *J* = 28.1 Hz), 41.2, 33.7, 33.6 (t, *J* = 1.8 Hz).

^{19}F NMR (282 MHz, Chloroform-d) δ -66.51 (qd, *J* = 9.7, 7.7 Hz, 3F), -67.80 (qd, *J* = 9.8, 8.2 Hz, 3F).

Calcd for $C_{13}H_{13}BrF_6$ (362.01): C 43.00%, H 3.63%; found: C 43.31%, H 3.78%.

((4-Bromo-7,7,7-trifluoro-6-(trifluoromethyl)heptyl)oxy)triisopropylsilane (2b)



General Procedure A.

Yield 192 mg (81%). Colorless oil.

Chromatography: hexane/EtOAc, 50/1.

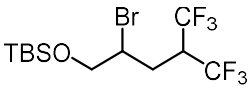
^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.24 – 4.08 (m, 1H), 3.74 (t, *J* = 5.9 Hz, 2H), 3.53 – 3.32 (m, 1H), 2.39 – 2.14 (m, 2H), 2.14 – 1.60 (m, 4H), 1.15 – 0.93 (m, 21H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 124.0 (qq, *J* = 280.7, 2.6 Hz), 123.7 (qq, *J* = 280.0, 3.3 Hz), 62.3, 53.3 (d, *J* = 2.1 Hz), 47.2 (hept, *J* = 28.1 Hz), 36.1, 33.8, 30.7, 18.1, 12.1.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.29 (pent, *J* = 9.6 Hz, 3F), -68.59 (pent, *J* = 9.5 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{17}H_{31}[^{79}Br]F_6OSiNa$ [M+Na]: 495.1124; found 495.1117.

((2-Bromo-5,5,5-trifluoro-4-(trifluoromethyl)pentyl)oxy)(*tert*-butyl)dimethylsilane (2c)



General Procedure A.

Yield 156 mg (73%). Orange oil.

Chromatography: pentane/DCM, 5/1.

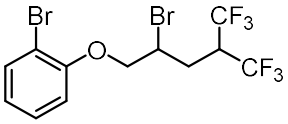
^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.08 (ddt, *J* = 10.6, 7.6, 3.9 Hz, 1H), 3.97 (dd, *J* = 10.7, 3.9 Hz, 1H), 3.76 (dd, *J* = 10.7, 7.7 Hz, 1H), 3.52 – 3.30 (m, 2H), 2.59 (ddd, *J* = 15.7, 9.2, 3.9 Hz, 1H), 2.15 (ddd, *J* = 15.7, 10.6, 1.3 Hz, 1H), 0.91 (s, 9H), 0.10 (s, 3H), 0.09 (s, 3H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 124.0 (qq, *J* = 280.3, 2.3 Hz), 123.8 (qq, *J* = 279.9, 3.2 Hz), 67.5, 50.8 (d, *J* = 2.2 Hz), 46.8 (hept, *J* = 28.1 Hz), 30.2, 25.8, 18.3, -5.3, -5.4.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.49 (pent, *J* = 9.5 Hz, 3F), -68.64 (pent, *J* = 9.5 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{12}H_{21}[^{79}Br]F_6OSiNa$ [M+Na]: 425.0341; found 425.0328.

1-Bromo-2-((2-bromo-5,5,5-trifluoro-4-(trifluoromethyl)pentyl)oxy)benzene (2d)



General Procedure A.

Yield 171 mg (77%). Pale-yellow oil.

Chromatography: hexane/EtOAc, 50/1.

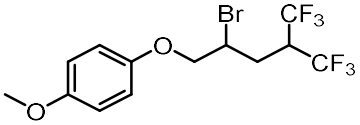
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.57 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.28 (td, *J* = 7.8, 1.5 Hz, 1H), 6.97 – 6.85 (m, 2H), 4.54 – 4.34 (m, 2H), 4.28 – 4.11 (m, 1H), 3.66 – 3.42 (m, 1H), 2.82 (ddd, *J* = 15.8, 9.1, 3.3 Hz, 1H), 2.48 – 2.29 (m, 1H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 154.3, 133.8, 128.7, 123.9 (qq, *J* = 280.3, 2.2 Hz), 123.7 (qq, *J* = 279.9, 3.2 Hz), 123.3, 113.9, 112.6, 72.8, 46.8 (hept, *J* = 28.2 Hz), 46.5, 30.7.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.46 (q, *J* = 9.6 Hz, 3F), -68.55 (q, *J* = 9.8 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{12}H_{10}[^{81}Br]_2F_6ONa$ [M+Na]: 468.8854; found 468.8857.

1-((2-Bromo-5,5,5-trifluoro-4-(trifluoromethyl)pentyl)oxy)-4-methoxybenzene (2e)



General Procedure A.

Yield 146 mg (74%). Pale-yellow oil.

Chromatography: hexane/EtOAc, 35/1.

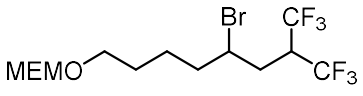
^{1}H NMR (300 MHz, Chloroform-*d*) δ 6.86 (s, 4H), 4.42 – 4.25 (m, 2H), 4.12 (dd, *J* = 9.7, 7.5 Hz, 1H), 3.78 (s, 3H), 3.57 – 3.36 (m, 1H), 2.68 (ddd, *J* = 15.9, 9.5, 3.1 Hz, 1H), 2.29 (t, *J* = 13.5 Hz, 1H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 154.9, 152.1, 123.9 (qq, *J* = 280.5, 2.2 Hz), 123.6 (qq, *J* = 279.4, 2.7, 2.2 Hz), 116.2, 115.0, 72.9, 55.8, 47.4, 46.8 (hept, *J* = 28.2 Hz), 30.4.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.29 (pent, *J* = 9.5 Hz, 3F), -68.52 (pent, *J* = 9.6 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{13}H_{13}[^{79}Br]F_6O_2Na$ [M+Na]: 416.9895; found 416.9905.

4-Bromo-1,1,1-trifluoro-8-((2-methoxyethoxy)methoxy)-2-(trifluoromethyl)octane (2f)



General Procedure A.

Yield 132 mg (63%). Yellow oil.

Chromatography: hexane/EtOAc, 4/1.

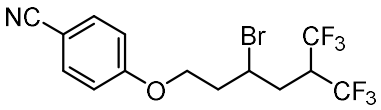
^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.70 (s, 2H), 4.17 – 4.01 (m, 1H), 3.69 (dd, *J* = 6.1, 3.3 Hz, 2H), 3.62 – 3.49 (m, 4H), 3.49 – 3.34 (m, 1H), 3.39 (s, 3H), 2.37 – 2.12 (m, 2H), 1.99 – 1.80 (m, 2H), 1.76 – 1.45 (m, 4H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 123.9 (qq, *J* = 280.6, 2.4 Hz), 123.6 (qq, *J* = 280.0, 3.2 Hz), 95.6, 71.9, 67.4, 66.9, 59.1, 53.0 (q, *J* = 2.0 Hz), 47.1 (hept, *J* = 28.2 Hz), 39.2, 33.5, 29.0, 24.4.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.33 (pent, *J* = 9.6 Hz, 3F), -68.67 (pent, *J* = 9.4 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{13}H_{21}[^{79}Br]F_6O_3Na$ [M+Na]: 441.0470; found 441.0475.

4-((3-Bromo-6,6,6-trifluoro-5-(trifluoromethyl)hexyl)oxy)benzonitrile (2g)



General Procedure A.

Yield 182 mg (90%). White solid. Mp 68-71 °C.

Chromatography: hexane/EtOAc, 8/1.

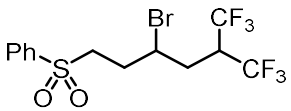
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.59 (d, *J* = 8.6 Hz, 2H), 7.03 – 6.85 (m, 2H), 4.48 – 4.29 (m, 1H), 4.28 – 4.11 (m, 2H), 3.56 – 3.29 (m, 1H), 2.53 – 2.14 (m, 4H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 161.8, 134.2, 123.9 (qq, *J* = 280.7, 2.3 Hz), 123.5 (qq, *J* = 280.3, 3.2 Hz), 119.1, 115.4, 104.6, 65.6, 49.0, 47.0 (hept, *J* = 28.2 Hz), 38.6, 33.7.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.25 (pent, *J* = 9.5 Hz, 3F), -68.53 (pent, *J* = 9.4 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{14}H_{12}[^{81}Br]F_6NONa$ [M+Na]: 427.9879; found 427.9871.

((3-Bromo-6,6,6-trifluoro-5-(trifluoromethyl)hexyl)sulfonyl)benzene (2h)



General Procedure A.

Yield 156 mg (73%). White solid. Mp 80-84 °C.

Chromatography: hexane/EtOAc, 5/1.

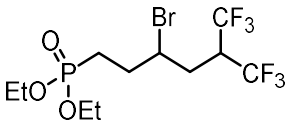
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 7.5 Hz, 2H), 7.72 (t, *J* = 7.5 Hz, 1H), 7.62 (t, *J* = 7.5 Hz, 2H), 4.15 (tt, *J* = 10.3, 3.5 Hz, 1H), 3.54 – 3.22 (m, 3H), 2.52 – 2.34 (m, 1H), 2.34 – 2.12 (m, 3H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 138.8, 134.23, 129.7, 128.1, 123.7 (qq, *J* = 280.7, 2.2 Hz), 123.4 (qq, *J* = 280.2, 3.1 Hz), 54.4, 50.1 (d, *J* = 2.3 Hz), 46.9 (hept, *J* = 28.3 Hz), 33.5, 32.3.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.26 (pent, *J* = 9.5 Hz, 3F), -68.58 (pent, *J* = 9.8 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{13}H_{13}[^{79}Br]F_6O_2SNa$ [M+Na]: 448.9616; found 448.9612.

Diethyl (3-bromo-6,6,6-trifluoro-5-(trifluoromethyl)hexyl)phosphonate (2i)



General Procedure A.

Yield 169 mg (80%). Orange oil.

Chromatography: hexane/EtOAc, 1/1.

^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.19 – 3.95 (m, 5H), 3.49 – 3.23 (m, 1H), 2.34 – 1.74 (m, 6H), 1.29 (t, *J* = 7.1 Hz, 6H).

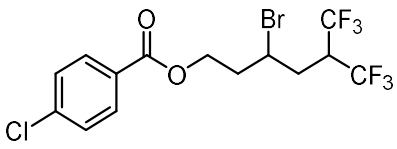
^{13}C NMR (75 MHz, Chloroform-*d*) δ 123.8 (qq, *J* = 280.5, 2.4 Hz), 123.5 (qq, *J* = 279.6, 2.9 Hz), 62.0 (dd, *J* = 6.5, 4.6 Hz), 52.8 (dt, *J* = 17.8, 2.1 Hz), 47.00 (hept, *J* = 28.3 Hz), 33.4, 32.6 (d, *J* = 3.9 Hz), 24.1 (d, *J* = 143.2 Hz), 16.5 (d, *J* = 6.0 Hz).

^{19}F NMR (282 MHz, Chloroform-*d*) δ -66.56 (qd, *J* = 9.7, 8.0 Hz, 3F), -67.89 (qd, *J* = 9.9, 8.1 Hz, 3F).

^{31}P NMR (121 MHz, CDCl3) δ 30.67.

HRMS (ESI-TOF): calcd for $C_{11}H_{18}[^{79}Br]F_6O_3PNa$ [M+Na]: 444.9973; found 444.9978.

3-Bromo-6,6,6-trifluoro-5-(trifluoromethyl)hexyl 4-chlorobenzoate (2j)



General Procedure A.

Yield 168 mg (76%). Colorless oil.

Chromatography: hexane/EtOAc, 20/1.

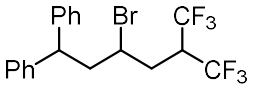
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 8.4 Hz, 2H), 7.42 (d, *J* = 8.7 Hz, 2H), 4.68 – 4.43 (m, 2H), 4.27 (tt, *J* = 9.0, 4.1 Hz, 1H), 3.55 – 3.32 (m, 1H), 2.50 – 2.20 (m, 4H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 165.5, 139.9, 131.1, 129.0, 128.3, 123.9 (qq, *J* = 280.7, 2.4 Hz), 123.5 (qq, *J* = 280.4, 3.1 Hz), 62.6, 48.7, 47.0 (hept, *J* = 28.6 Hz), 38.3, 33.7.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.24 (pent, *J* = 9.6 Hz, 3F), -68.55 (pent, *J* = 9.8 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{14}H_{12}[^{79}Br]F_6ClO_2Na$ [M+Na]: 462.9506; found 462.9504.

(3-Bromo-6,6,6-trifluoro-5-(trifluoromethyl)hexane-1,1-diyl)dibenzene (2k)



General Procedure A.

Yield 213 mg (97%). Pale-red oil.

Chromatography: hexane/EtOAc, 20/1.

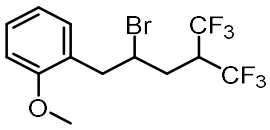
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.40 – 7.14 (m, 10H), 4.35 (t, *J* = 7.8 Hz, 1H), 3.94 – 3.75 (m, 1H), 3.44 – 3.22 (m, 1H), 2.58 (dd, *J* = 8.6, 5.9 Hz, 2H), 2.41 – 2.18 (m, 2H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 143.6, 142.3, 129.0, 128.9, 128.0, 127.7, 127.1, 126.8, 123.8 (qq, *J* = 280.5, 2.1 Hz), 123.6 (qq, *J* = 280.0, 3.2 Hz), 51.5, 49.1, 47.0 (hept, *J* = 28.1 Hz), 45.4, 33.8.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.53 (pent, *J* = 9.6 Hz), -68.64 (pent, *J* = 9.7 Hz).

HRMS (ESI-TOF): calcd for $C_{19}H_{17}[^{79}Br]F_6Na$ [M+Na]: 461.0310; found 461.0322.

1-(2-Bromo-5,5,5-trifluoro-4-(trifluoromethyl)pentyl)-2-methoxybenzene (2l)



General Procedure A.

Yield 135 mg (71%). Pale-yellow oil.

Chromatography: hexane/EtOAc, 50/1.

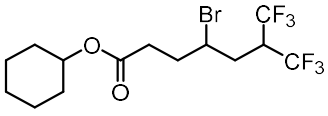
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.23 (t, *J* = 8.0 Hz, 1H), 7.11 (d, *J* = 7.4 Hz, 1H), 6.93 – 6.78 (m, 2H), 4.44 (dtd, *J* = 10.6, 6.8, 3.4 Hz, 1H), 3.77 (s, 3H), 3.48 – 3.26 (m, 1H), 3.19 (d, *J* = 7.0 Hz, 2H), 2.34 – 2.07 (m, 2H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 157.6, 131.4, 128.9, 125.7, 123.9 (qq, *J* = 280.5, 2.3 Hz), 123.7 (qq, *J* = 280.0, 3.3 Hz), 120.7, 110.6, 55.3, 51.8 (d, *J* = 2.2 Hz), 47.2 (hept, *J* = 28.0 Hz), 41.3, 33.0.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.42 (pent, *J* = 9.5 Hz 3F), -68.61 (pent, *J* = 9.4 Hz 3F).

HRMS (ESI-TOF): calcd for $C_{13}H_{13}[^{79}Br]F_6ONa$ [M+Na]: 400.9946; found 400.9945.

Cyclohexyl 4-bromo-7,7,7-trifluoro-6-(trifluoromethyl)heptanoate (2m)



General Procedure A.

Yield 153 mg (76%). Pale-red oil.

Chromatography: hexane/EtOAc, 25/1.

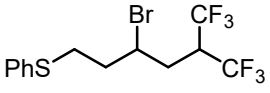
^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.87 – 4.67 (m, 1H), 4.26 – 4.05 (m, 1H), 3.52 – 3.27 (m, 1H), 2.69 – 2.44 (m, 2H), 2.40 – 2.17 (m, 3H), 2.16 – 1.99 (m, 1H), 1.93 – 1.09 (m, 10H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 171.7, 123.9 (qq, *J* = 280.7, 2.2 Hz), 123.6 (qq, *J* = 280.0, 3.3 Hz), 73.3, 52.1, 47.1 (hept, *J* = 28.0 Hz), 34.5, 33.7, 32.6, 31.74, 31.72, 25.5, 23.9.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.36 (pent, *J* = 9.4 Hz, 3F), -68.65 (pent, *J* = 9.6 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{14}H_{19}[^{79}Br]F_6O_2Na$ [M+Na]: 435.0365; found 435.0357.

(3-Bromo-6,6,6-trifluoro-5-(trifluoromethyl)hexyl)(phenyl)sulfane (2n)



General Procedure A.

Yield 174 mg (88%). Yellow oil.

Chromatography: hexane/EtOAc, 50/1.

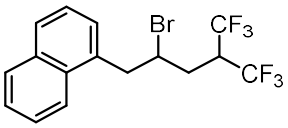
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.49 – 7.22 (m, 5H), 4.44 – 4.25 (m, 1H), 3.54 – 3.35 (m, 1H), 3.28 (ddd, *J* = 13.2, 7.6, 4.9 Hz, 1H), 3.09 (dt, *J* = 13.2, 7.7 Hz, 1H), 2.34 – 2.03 (m, 4H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 135.1, 130.4, 129.3, 126.9, 123.9 (qq, *J* = 280.6, 2.2 Hz), 123.6 (qq, *J* = 280.1, 3.3 Hz), 51.4 (d, *J* = 2.0 Hz), 47.0 (hept, *J* = 28.3 Hz), 38.6, 33.5, 31.9.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.27 (pent, *J* = 9.4 Hz, 3F), -68.59 (pent, *J* = 9.4 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{13}H_{13}[^{81}Br]F_6S[^{107}Ag]$ [M+Ag]: 502.8853; found 502.8855.

1-(2-Bromo-5,5,5-trifluoro-4-(trifluoromethyl)pentyl)naphthalene (2o)



General Procedure A.

Yield 124 mg (62%). Colorless oil.

Chromatography: hexane/EtOAc, 100/1.

Final purification was performed by preparative HPLC (reversed-phase column C18, 21×250 mm, 5 μm), flow rate 6 mL·min−1 ; mobile phase: isocratic, acetonitrile/water, 10% water; tR = 20.9 min).

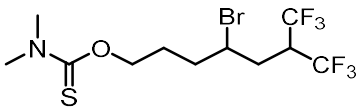
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.99 – 7.87 (m, 2H), 7.84 (d, *J* = 8.2 Hz, 1H), 7.65 – 7.31 (m, 4H), 4.67 – 4.41 (m, 1H), 3.78 (dd, *J* = 14.6, 7.2 Hz, 1H), 3.63 (dd, *J* = 14.6, 7.0 Hz, 1H), 3.55 – 3.33 (m, 1H), 2.52 – 2.21 (m, 2H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 134.2, 133.1, 131.7, 129.3, 128.5, 127.9, 126.7, 126.0, 125.5, 123.8 (qq, *J* = 280.5, 2.2 Hz), 123.6 (qq, *J* = 280.0, 3.1 Hz), 122.9, 51.9, 47.3 (hept, *J* = 28.0 Hz), 43.3, 33.3.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.31 (pent, *J* = 9.4 Hz, 3F), -68.59 (pent, *J* = 9.5 Hz, 3F).

Anal. Calcd for $C_{16}H_{13}BrF_6$: C, 48.14; H, 3.28. Found: C, 48.27, H, 3.36.

O-(4-bromo-7,7,7-trifluoro-6-(trifluoromethyl)heptyl) dimethylcarbamothioate (2p)



General Procedure A.

Yield 156 mg (77%). Orange oil.

Chromatography: hexane/EtOAc, 12/1.

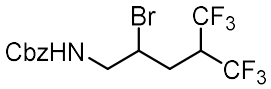
^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.59 – 4.39 (m, 2H), 4.20 – 4.01 (m, 1H), 3.49 – 3.27 (m, 1H), 3.35 (s, 3H), 3.10 (s, 3H), 2.39 – 2.13 (m, 2H), 2.12 – 1.78 (m, 4H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 188.2, 123.9 (qq, *J* = 280.6, 2.3 Hz), 123.5 (qq, *J* = 280.1, 3.3 Hz), 70.4, 52.6 (d, *J* = 2.3 Hz), 47.1 (hept, *J* = 28.2 Hz), 42.8, 37.8, 36.1, 33.6, 27.1.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.24 (pent, *J* = 9.5 Hz, 3F), -68.60 (pent, *J* = 9.6 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{11}H_{16}[^{79}Br]F_6NOSNa$ [M+Na]: 425.9932; found 425.9922.

Benzyl (2-bromo-5,5,5-trifluoro-4-(trifluoromethyl)pentyl)carbamate (2q)



General Procedure A.

Yield 188 mg (89%). Orange oil.

Chromatography: hexane/EtOAc, 5/1.

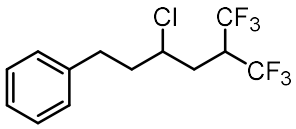
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.44 – 7.30 (m, 5H), 5.25 (s, 1H), 5.14 (s, 2H), 4.33 – 4.15 (m, 1H), 3.76 – 3.49 (m, 2H), 3.49 – 3.29 (m, 1H), 2.36 (ddd, *J* = 13.4, 9.0, 3.9 Hz, 1H), 2.19 (t, *J* = 13.5 Hz, 1H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 156.5, 136.2, 128.8, 128.5, 128.3, 123.7 (qq, *J* = 280.6, 2.2 Hz), 123.5 (qq, *J* = 280.2, 2.9 Hz), 67.5, 51.7, 47.6, 46.8 (hept, *J* = 28.4 Hz), 30.4.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.32 (pent, *J* = 9.6 Hz, 3F), -68.34 (pent, *J* = 9.5 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{14}H_{14}[^{79}Br]F_6NO_2Na$ [M+Na]: 444.0004; found 443.9992.

(3-Chloro-6,6,6-trifluoro-5-(trifluoromethyl)hexyl)benzene (3a)



General Procedure B.

Yield 127.2 mg (80%). Colorless liquid.

Chromatography: hexanes/EtOAc, 10/1.

Final purification was performed by preparative HPLC (reversed-phase column C18, 21 × 250 mm, 5 μm), flow rate 6 mL min−1; mobile phase isocratic, acetonitrile/water, 10% water; tR = 20.0 min.

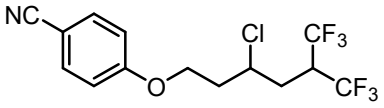
^{1}H NMR (300 MHz, Chloroform-d) δ 7.42 – 7.17 (m, 5H), 4.13 – 3.96 (m, 1H), 3.66 – 3.21 (m, 1H), 2.96 (dt, *J* = 14.3, 7.2 Hz, 1H), 2.81 (dt, *J* = 13.8, 8.1 Hz, 1H), 2.36 – 2.04 (m, 4H).

^{13}C NMR (76 MHz, Chloroform-d) δ 140.3, 128.8, 128.5, 126.53, 124.0 (qq, *J* = 280.4, 2.3 Hz), 123.7 (qq, *J* = 280.1, 3.5 Hz), 59.2 (q, *J* = 1.9 Hz), 45.9 (hept., *J* = 28.2 Hz), 40.6, 33.0, 32.7.

^{19}F NMR (282 MHz, Chloroform-d) δ -67.40 (pent, *J* = 9.6 Hz, 3F), -68.65 (pent, *J* = 9.5 Hz, 3F).

Calcd for $C_{13}H_{13}ClF_6$ (318.06): C 49.00, H 4.11; found: C 48.92, H 4.31.

4-((3-Chloro-6,6,6-trifluoro-5-(trifluoromethyl)hexyl)oxy)benzonitrile (3b)



General Procedure B.

Yield 158.0 mg (88%), colorless liquid.

Chromatography: hexanes/EtOAc, 10/1.

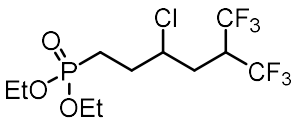
^{1}H NMR (300 MHz, Chloroform-d) δ 7.57 (d, *J* = 8.8 Hz, 2H), 6.95 (d, *J* = 8.8 Hz, 2H), 4.39 – 4.13 (m, 3H), 3.53 – 3.30 (m, 1H), 2.44 – 2.27 (m, 2H), 2.27 – 2.07 (m, 2H).

^{13}C NMR (76 MHz, Chloroform-d) δ 161.8, 134.10, 123.9 (qq, *J* = 280.4, 2.4 Hz), 123.5 (qq, *J* = 280.0, 3.2 Hz), 119.1, 115.3, 104.5, 64.6, 56.2 (d, *J* = 2.0 Hz), 45.7 (hept., *J* = 28.3 Hz), 37.9, 33.0.

^{19}F NMR (282 MHz, Chloroform-d) δ -66.54 (pent, *J* = 9.5 Hz, 3F), -67.80 (qd, *J* = 9.8, 8.0 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{14}H_{12}[^{35}Cl]F_6NONa$, [M+Na] 382.0404; found 382.0390

Diethyl (3-chloro-6,6,6-trifluoro-5-(trifluoromethyl)hexyl)phosphonate (3c)



General Procedure B.

Yield 141.8 mg (75%). yellowish liquid.

Chromatography: hexanes/EtOAc, 1/1.

^{1}H NMR (300 MHz, Chloroform-d) δ 4.19 – 4.02 (m, 5H), 3.48 – 3.26 (m, 1H), 2.34 – 1.77 (m, 6H), 1.33 (t, *J* = 7.1 Hz, 6H).

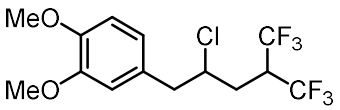
^{13}C NMR (76 MHz, Chloroform-d) δ 123.8 (qq, *J* = 280.5, 2.4 Hz), 123.5 (qq, *J* = 279.9, 3.2 Hz), 61.9 (dd, *J* = 6.5, 4.0 Hz), 59.6 (dd, *J* = 17.4, 2.1 Hz), 45.7 (hept., *J* = 28.3 Hz), 32.7, 32.0 (d, *J* = 4.0 Hz), 22.84 (d, *J* = 143.4 Hz), 16.4 (d, *J* = 5.9 Hz).

^{19}F NMR (282 MHz, Chloroform-d) δ -66.56 (pent, *J* = 9.4 Hz, 3F), -67.87 (qd, *J* = 9.8, 8.1 Hz, 3F).

^{31}P NMR (122 MHz, Chloroform-d) δ 29.98.

HRMS (ESI-TOF): calcd for $C_{11}H_{18}[^{35}Cl]F_6O_3PNa$, [M+Na] 401.0478; found 401.0472.

4-(2-Chloro-5,5,5-trifluoro-4-(trifluoromethyl)pentyl)-1,2-dimethoxybenzene (3d)



General Procedure B.

Yield 147.4 mg (81%), colorless liquid.

Chromatography: hexanes/EtOAc, 10/1.

Final purification was performed by preparative HPLC (reversed-phase column C18, 21 × 250 mm, 5 μm), flow rate 8 mL min−1; mobile phase isocratic, acetonitrile/water, 20% water; tR = 15.1 min.

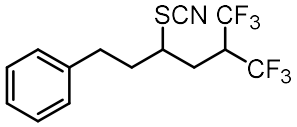
^{1}H NMR (300 MHz, Chloroform-d) δ 6.83 (d, *J* = 8.1 Hz, 1H), 6.79 – 6.67 (m, 2H), 4.32 – 4.14 (m, 1H), 3.88 (s, 6H), 3.53 – 3.30 (m, 1H), 3.12 (dd, *J* = 14.3, 7.1 Hz, 1H), 2.98 (dd, *J* = 14.3, 6.7 Hz, 1H), 2.37 – 2.18 (m, 1H), 2.04 (t, *J* = 13.5 Hz, 1H).

^{13}C NMR (76 MHz, Chloroform-d) δ 149.17, 148.43, 128.90, 123.95 (qq, *J* = 280.6, 2.4 Hz), 123.60 (qq, *J* = 279.9, 3.3 Hz), 121.48, 112.35, 111.40, 59.94 (q, *J* = 1.9 Hz), 56.03, 55.99, 45.88 (hept, *J* = 28.2 Hz), 44.86, 32.16.

^{19}F NMR (282 MHz, Chloroform-d) δ -66.46 (pent, *J* = 9.4 Hz, 3F), -67.84 (pent, *J* = 9.5 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{14}H_{15}[^{35}Cl]F_6O_2Na$, [M+Na] 387.0557; found 387.0567.

(6,6,6-Trifluoro-3-thiocyanato-5-(trifluoromethyl)hexyl)benzene (4a)



General Procedure C.

Yield 102.3 mg (60%), yellowish liquid.

Chromatography: hexanes/EtOAc, 20/1.

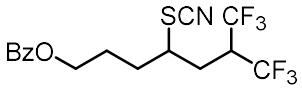
^{1}H NMR (300 MHz, Chloroform-d) δ 7.40 – 7.18 (m, 5H), 3.43 – 3.17 (m, 1H), 3.12 – 2.93 (m, 2H), 2.89 – 2.75 (m, 1H), 2.42 – 2.14 (m, 4H).

^{13}C NMR (76 MHz, Chloroform-d) δ 139.3, 128.9, 128.4, 126.9, 123.7 (qq, *J* = 280.6, 2.4 Hz),123.3 (qq, *J* = 280.6, 3.0 Hz), 108.4, 47.3 (d, *J* = 1.7 Hz), 46.1 (hept., *J* = 28.3 Hz), 37.3, 33.0, 30.3 – 29.3 (m).

^{19}F NMR (282 MHz, Chloroform-d) δ -66.41 (qd, *J* = 9.7, 7.6 Hz, 3F), -67.56 (qd, *J* = 9.8, 7.9 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{14}H_{13}F_6NSNa$, [M+Na] 364.0565; found 364.0578.

7,7,7-Trifluoro-4-thiocyanato-6-(trifluoromethyl)heptyl benzoate (4b)



General Procedure C.

Yield 85.8 mg (43%), yellowish liquid.

Chromatography: hexanes/EtOAc, 20/1.

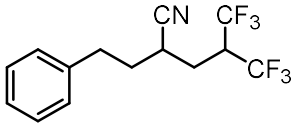
^{1}H NMR (300 MHz, Chloroform-d) δ 8.04 (d, *J* = 7.3 Hz, 2H), 7.58 (t, *J* = 7.3 Hz, 1H), 7.46 (t, *J* = 7.3 Hz, 2H), 4.49 – 4.28 (m, 2H), 4.05 – 3.88 (m, 1H), 3.27 – 3.08 (m, 1H), 2.14 – 1.79 (m, 6H).

^{13}C NMR (76 MHz, Chloroform-d) δ 166.62, 135.60, 133.30, 130.05, 129.72, 128.59, 123.75 (qq, *J* = 280.7, 2.3 Hz), 123.44 (qq, *J* = 280.2, 3.4 Hz), 63.84, 56.19 – 56.01 (m), 45.59 (hept, *J* = 28.5 Hz), 33.72, 30.40, 25.55.

^{19}F NMR (282 MHz, Chloroform-d) δ -67.15 – -67.41 (m, 3F), -68.39 – -68.65 (m, 3F).

HRMS (ESI-TOF): calcd for $C_{16}H_{15}F_6NO_2SNa$, [M+Na] 422.0620; found 422.0606.

5,5,5-Trifluoro-2-phenethyl-4-(trifluoromethyl)pentanenitrile (5a)



General Procedure D.

Yield 109.7 mg (71%), colorless liquid.

Chromatography: hexanes/EtOAc, 15/1.

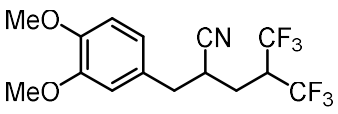
^{1}H NMR (300 MHz, Chloroform-d) δ 7.43 – 7.17 (m, 5H), 3.34 – 3.11 (m, 1H), 3.03 – 2.88 (m, 1H), 2.88 – 2.73 (m, 3H), 2.22 – 1.88 (m, 3H).

^{13}C NMR (76 MHz, Chloroform-d) δ 139.3, 128.9, 128.4, 126.9, 123.6 (qq, *J* = 280.6, 2.5 Hz), 123.3 (qq, *J* = 280.4, 3.2 Hz), 119.8, 46.5 (hept, *J* = 28.5 Hz), 34.3, 33.1, 29.4, 26.9 – 26.6 (m).

^{19}F NMR (282 MHz, Chloroform-d) δ -66.5 (qd, *J* = 9.8, 7.4 Hz, 3F), -67.8 (qd, *J* = 9.9, 7.7 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{14}H_{13}F_6NNa$, [M+Na] 332.0844; found 332.0846.

2-(3,4-Dimethoxybenzyl)-5,5,5-trifluoro-4-(trifluoromethyl)pentanenitrile (5b)



General Procedure D.

Yield 113.6 mg (64%), yellowish liquid.

Chromatography: hexanes/EtOAc, 3/1.

Final purification was performed by preparative HPLC (reversed-phase column C18, 21 × 250 mm, 5 μm), flow rate 8 mL min^{-1}; mobile phase isocratic, acetonitrile/water, 20% water; tR = 10.0 min.

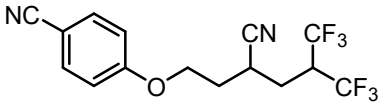
^{1}H NMR (300 MHz, Chloroform-d) δ 6.85 (d, *J* = 7.9 Hz, 1H), 6.81 – 6.73 (m, 2H), 3.88 (s, 6H), 3.31 – 3.12 (m`, 1H), 3.11 – 2.97 (m, 1H) 2.92 (t, *J* = 6.3 Hz, 2H), 2.15 – 2.02 (m, 2H).

^{13}C NMR (76 MHz, Chloroform-d) δ 149.41, 148.81, 127.73, δ 123.61 (qq, *J* = 280.8, 2.4 Hz), 123.25 (qq, *J* = 280.6 Hz, 2.4Hz), 121.34, 119.81, 112.01, 111.64, 56.09, 56.05, 46.55 (hept, *J* = 28.4 Hz), 38.28, 32.13, 26.41.

19F NMR (282 MHz, Chloroform-d) δ -66.3 (qd, *J* = 9.8, 7.4 Hz, 3F), -67.7 (qd, *J* = 9.9, 7.8 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{15}H_{15}F_6NO_2Na$, [M+Na] 378.0899; found 378.0901.

4-((3-Cyano-6,6,6-trifluoro-5-(trifluoromethyl)hexyl)oxy)benzonitrile (5c)



General Procedure D.

Yield 106.8 mg (71%), colorless liquid.

Chromatography: hexanes/EtOAc, 3/1.

Final purification was performed by preparative HPLC (reversed-phase column C18, 21 × 250 mm, 5 μm), flow rate 6 mL min^{-1}; mobile phase isocratic, acetonitrile/water, 15% water; tR = 12.0 min.

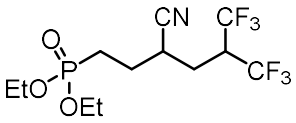
^{1}H NMR (300 MHz, Chloroform-d) δ 7.60 (d, *J* = 8.9 Hz, 2H), 6.96 (d, *J* = 8.9 Hz, 2H), 4.22 (t, *J* = 5.7 Hz, 2H), 3.34 – 3.10 (m, 2H), 2.24 – 2.11 (m, 4H).

^{13}C NMR (76 MHz, Chloroform-d) δ 161.3, 134.3, 123.6 (qq, *J* = 280.8, 2.3 Hz). 123.2 (qq, *J* = 280.4, 3.2 Hz), 119.2, 119.0, 115.3, 105.1, 64.7, 46.5 (hept., *J* = 28.6 Hz), 32.0, 27.2 (d, *J* = 1.9 Hz), 26.8.

^{19}F NMR (282 MHz, Chloroform-d) δ -66.4 (qd, *J* = 9.9, 7.5 Hz, 3F), -67.7 (qd, *J* = 9.9, 7.7 Hz, 3F).

HRMS (ESI-TOF): calcd for $C_{15}H_{12}F_6N_2ONa$, [M+Na] 373.0746; found 373.0748.

Diethyl (3-cyano-6,6,6-trifluoro-5-(trifluoromethyl)hexyl)phosphonate (5d)



General Procedure D.

Yield 147.6 mg (80%), colorless liquid.

Chromatography: EtOAc.

^{1}H NMR (300 MHz, Chloroform-d) δ 4.21 – 4.01 (m, 4H), 3.31 – 3.08 (m, 1H), 3.08 – 2.90 (m, 1H), 2.14 – 1.81 (m, 6H), 1.32 (t, *J* = 7.1 Hz, 6H).

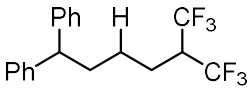
^{13}C NMR (76 MHz, Chloroform-d) δ 123.5 (qq, *J* = 280.6, 2.5 Hz), 123.1 (qq, *J* = 280.3, 3.3 Hz). 119.2, 62.1 (t, *J* = 6.0 Hz), 46.4 (hept,, *J* = 28.5 Hz), 30.4 (d, *J* = 15.4 Hz), 26.6, 26.1 (d, *J* = 4.3 Hz), 23.24 (d, *J* = 143.8 Hz), 16.3 (d, *J* = 6.0 Hz).

^{19}F NMR (282 MHz, Chloroform-d) δ -66.5 (pent, *J* = 9.6 Hz, 3F), -67.8 (pent, *J* = 9.7 Hz, 3F).

^{31}P NMR (122 MHz, Chloroform-d) δ 28.37.

HRMS (ESI-TOF): calcd for $C_{12}H_{19}F_6NO_3P$, [M+H] 370.1001; found 370.1002.

(6,6,6-Trifluoro-5-(trifluoromethyl)hexane-1,1-diyl)dibenzene (6a)



General procedure E.

Yield 159 mg (88%). Colorless oil.

Chromatography: hexane/EtOAc, 30/1.

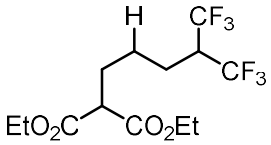
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.48 – 7.21 (m, 10H), 4.01 (t, *J* = 7.8 Hz, 1H), 3.01 – 2.73 (m, 1H), 2.19 (q, *J* = 7.8 Hz, 2H), 1.95 (q, *J* = 7.0 Hz, 2H), 1.77 – 1.49 (m, 2H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 144.6, 128.7, 127.9, 126.5, 124.1 (qq, *J* = 281.0, 3.6 Hz), 51.1, 48.1 (hept, *J* = 28.2, 27.7 Hz), 35.4, 25.9, 23.9 – 23.6 (m).

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.94 (d, *J* = 8.3 Hz).

HRMS (ESI-TOF): calcd for $C_{19}H_{19}F_6$ [M+H]: 361.1385; found 361.1381.

Diethyl 2-(5,5,5-trifluoro-4-(trifluoromethyl)pentyl)malonate (6b)



General procedure E.

Yield 164 mg (93%). Pale-yellow oil.

Chromatography: hexane/EtOAc, 10/1.

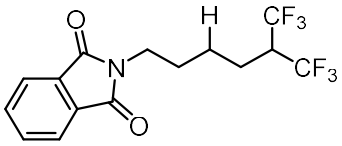
^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.19 (q, *J* = 7.1 Hz, 4H), 3.32 (t, *J* = 7.4 Hz, 1H), 2.97 – 2.71 (m, 1H), 2.01 – 1.73 (m, 4H), 1.68 – 1.45 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 7H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 169.2, 124.0 (qq, *J* = 281.1, 3.9 Hz), 61.6, 51.6, 47.9 (hept, *J* = 28.1 Hz), 28.4, 25.0, 23.6 – 23.4 (m), 14.1.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.96 (d, *J* = 8.3 Hz).

HRMS (ESI-TOF): calcd for $C_{13}H_{18}F_6O_4Na$ [M+Na]: 375.1001; found 375.0997.

2-(6,6,6-Trifluoro-5-(trifluoromethyl)hexyl)isoindoline-1,3-dione (6c)



General procedure E.

Yield 79 mg (45%). White solid (mp 63-66°C).

Chromatography: hexane/EtOAc, 6/1.

Final purification was performed by preparative HPLC (reversed-phase column C18, 21×250 mm, 5 μm), flow rate 6 mL·min^{-1}; mobile phase: isocratic, acetonitrile/water, 20% water; tR = 15.7 min).

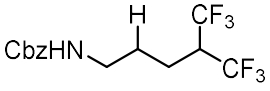
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.84 (t, *J* = 5.4, 3.2 Hz, 2H), 7.71 (dd, *J* = 5.4, 3.1 Hz, 2H), 3.70 (t, *J* = 7.1 Hz, 2H), 2.97 – 2.71 (m, 1H), 1.86 (dt, *J* = 7.3, 7.1 Hz, 2H), 1.73 (pent, *J* = 7.1 Hz, 2H), 1.65 – 1.49 (m, 2H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 168.5, 134.1, 132.2, 124.0 (qq, *J* = 281.2, 3.9 Hz), 123.4, 48.1 (hept, *J* = 27.9 Hz), 37.4, 28.4, 24.6, 23.4 (pent, *J* = 1.9 Hz).

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.90 (d, *J* = 8.3 Hz).

HRMS (ESI-TOF): calcd for $C_{15}H_{13}F_6NO_2Na$ [M+Na]: 376.0743; found 376.0744.

Benzyl (5,5,5-trifluoro-4-(trifluoromethyl)pentyl)carbamate (6d)



General procedure E.

Yield 144 mg (84%). Colorless oil.

Chromatography: hexane/EtOAc, 5/1.

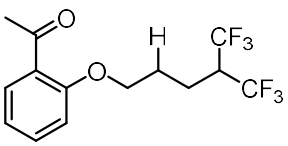
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.44 – 7.27 (m, 5H), 5.10 (s, 2H), 4.94 (s, 1H), 3.23 (q, *J* = 6.6 Hz, 2H), 3.04 – 2.74 (m, 1H), 1.97 – 1.54 (m, 4H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 156.6, 136.6, 128.7, 128.3, 128.2, 124.0 (qq, *J* = 281.1, 3.5 Hz), 66.9, 47.8 (hept, *J* = 27.7, 27.2 Hz), 40.4, 27.7, 21.2 – 20.7 (m).

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.75 (d, *J* = 8.1 Hz).

HRMS (ESI-TOF): calcd for $C_{14}H_{15}F_6NO_2Na$ [M+Na]: 366.0899; found 366.0898.

1-(2-((5,5,5-Trifluoro-4-(trifluoromethyl)pentyl)oxy)phenyl)ethanone (6e)



General procedure E.

Yield 102 mg (62%). White solid (mp 56-58°C).

Chromatography: hexane/EtOAc, 8/1.

Final purification was performed by preparative HPLC (reversed-phase column C18, 21×250 mm, 5 μm), flow rate 6 mL·min−1 ; mobile phase: isocratic, acetonitrile/water, 20% water; tR = 18.3 min)

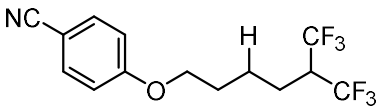
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.73 (dd, *J* = 7.5, 1.8 Hz, 1H), 7.45 (td, *J* = 8.4, 7.5, 1.8 Hz, 1H), 7.02 (t, *J* = 7.5 Hz, 1H), 6.93 (d, *J* = 8.4 Hz, 1H), 4.24 – 4.01 (m, 2H), 3.13 – 2.87 (m, 1H), 2.60 (s, 3H), 2.21 – 2.01 (m, 4H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 199.6, 157.9, 133.7, 130.7, 128.7, 124.0 (qq, *J* = 281.1, 3.6 Hz), 121.1, 112.3, 67.6, 47.9 (hept, *J* = 27.7 Hz), 31.8, 27.1, 21.1 (pent, *J* = 2.0 Hz).

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.77 (d, *J* = 8.0 Hz).

HRMS (ESI-TOF): calcd for $C_{14}H_{15}F_6O_2$ [M+H]: 329.0971; found 329.0981.

4-((6,6,6-Trifluoro-5-(trifluoromethyl)hexyl)oxy)benzonitrile (6f)



General procedure E.

Yield 128 mg (79%). Pale-yellow oil.

Chromatography: hexane/EtOAc, gradient elution from 8/1 to 6/1.

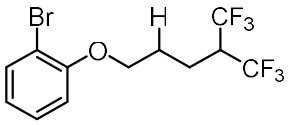
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 8.7 Hz, 2H), 6.92 (d, *J* = 8.7 Hz, 2H), 4.02 (t, *J* = 5.9 Hz, 2H), 3.05 – 2.74 (m, 1H), 2.05 – 1.79 (m, 4H), 1.78 – 1.54 (m, 2H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 162.2, 134.1, 124.0 (qq, *J* = 281.5, 4.0 Hz), 119.3, 115.3, 104.1, 67.7, 48.1 (hept, *J* = 27.9 Hz), 28.8, 24.0, 23.7 – 23.4 (m).

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.86 (d, *J* = 8.1 Hz).

HRMS (ESI-TOF): calcd for $C_{14}H_{13}F_6NONa$ [M+Na]: 348.0794; found 348.0788.

1-Bromo-2-((5,5,5-trifluoro-4-(trifluoromethyl)pentyl)oxy)benzene (6g)



General procedure E.

Yield 84 mg (46%). Colorless oil.

Chromatography: hexane/EtOAc, 50/1.

Final purification was performed by preparative HPLC (reversed-phase column C18, 21×250 mm, 5 μm), flow rate 6 mL·min−1 ; mobile phase: isocratic, acetonitrile/water, 15% water; tR = 22.9 min).

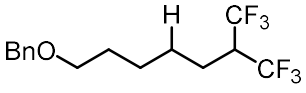
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.55 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.26 (ddd, *J* = 8.3, 7.7, 1.6 Hz, 1H), 6.86 (t, *J* = 7.7 Hz, 2H), 4.07 (t, *J* = 5.3 Hz, 2H), 3.30 – 3.07 (m, 1H), 2.19 – 1.99 (m, 4H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 155.1, 133.6, 128.6, 124.2 (qq, *J* = 281.5, 3.5 Hz), 122.4, 113.3, 112.4, 68.4, 47.8 (hept, *J* = 27.9 Hz), 26.7, 21.2 – 21.0 (m).

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.79 (d, *J* = 8.3 Hz).

HRMS (ESI-TOF): calcd for $C_{12}H_{11}[^{79}Br]F_6ONa$ [M+Na]: 386.9790; found 386.9785.

(((7,7,7-Trifluoro-6-(trifluoromethyl)heptyl)oxy)methyl)benzene (6h)



General procedure E.

Yield 95 mg (58%). Colorless oil.

Chromatography: hexane/EtOAc, 30/1.

Final purification was performed by preparative HPLC (reversed-phase column C18, 21×250 mm, 5 μm), flow rate 6 mL·min−1 ; mobile phase: isocratic, acetonitrile/water, 15% water; tR = 27.1 min)

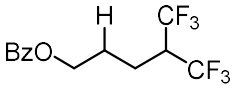
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.40 – 7.27 (m, 5H), 4.51 (s, 2H), 3.48 (t, *J* = 6.3 Hz, 2H), 2.93 – 2.72 (m, 1H), 1.93 – 1.73 (m, 2H), 1.71 – 1.35 (m, 6H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 138.7, 128.5, 127.8, 127.7, 124.2 (qq, *J* = 281.0, 3.7 Hz), 73.1, 70.1, 48.1 (hept, *J* = 27.6 Hz), 29.4, 27.2, 26.2, 23.8 (pent, *J* = 1.9 Hz).

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.89 (d, *J* = 8.3 Hz).

HRMS (ESI-TOF): calcd for $C_{15}H_{18}F_6ONa$ [M+Na]: 351.1154; found 351.1167.

5,5,5-Trifluoro-4-(trifluoromethyl)pentyl benzoate (6i)



General procedure E.

Yield 104 mg (66%). Colorless oil.

Chromatography: hexane/EtOAc, 20/1.

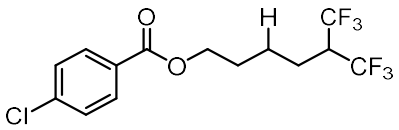
^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.03 (d, *J* = 7.5 Hz, 2H), 7.58 (tt, *J* = 7.5, 1.5 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 2H), 4.46 – 4.26 (m, 2H), 3.10 – 2.88 (m, 1H), 2.12 – 1.95 (m, 4H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 166.5, 133.2, 130.0, 129.6, 128.5, 124.0 (qq, *J* = 281.1, 3.7 Hz), 63.7, 47.8 (hept, *J* = 27.8 Hz), 26.3, 21.1 – 20.1 (m).

^{19}F NMR (282 MHz, Chloroform-d) δ -67.81 (d, *J* = 8.0 Hz).

HRMS (ESI-TOF): calcd for $C_{13}H_{12}F_6O_2Na$ [M+Na]: 337.0634; found 337.0636.

6,6,6-Trifluoro-5-(trifluoromethyl)hexyl 4-chlorobenzoate (6j)



General procedure E.

Yield 129 mg (71%). Colorless oil.

Chromatography: hexane/EtOAc, 15/1.

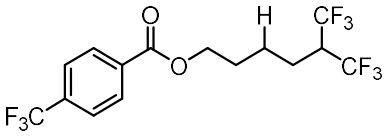
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 8.5 Hz, 2H), 7.39 (d, *J* = 8.5 Hz, 2H), 4.34 (t, *J* = 6.3 Hz, 2H), 3.00 – 2.74 (m, 1H), 1.99 – 1.75 (m, 4H), 1.74 – 1.57 (m, 2H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 165.8, 139.6, 131.0, 128.9, 124.1 (qq, *J* = 281.2, 3.9 Hz), 64.5, 48.1 (hept, *J* = 27.9 Hz), 28.6, 24.0, 23.7 – 23.4 (m).

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.89 (d, *J* = 8.4 Hz).

HRMS (ESI-TOF): calcd for $C_{14}H_{13}[^{35}Cl]F_6O_2Na$ [M+Na]: 385.0400; found 385.0409.

6,6,6-Trifluoro-5-(trifluoromethyl)hexyl 4-(trifluoromethyl)benzoate (6k)



General procedure E.

Yield 149 mg (75%). Colorless oil.

Chromatography: hexane/EtOAc, gradient elution from 25/1 to 15/1.

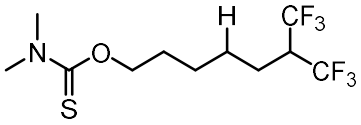
^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.14 (d, *J* = 8.1 Hz, 2H), 7.70 (d, *J* = 8.1 Hz, 2H), 4.38 (t, *J* = 6.3 Hz, 2H), 3.00 – 2.75 (m, 1H), 2.03 – 1.78 (m, 4H), 1.77 – 1.56 (m, 2H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 165.5, 134.7 (q, *J* = 32.7 Hz), 133.6, 130.1, 125.6 (q, *J* = 3.8 Hz), 124.1 (qq, *J* = 281.0, 3.6 Hz), 123.81 (q, *J* = 272.7 Hz), 122.0, 64.8, 48.2 (hept, *J* = 27.8 Hz), 28.6, 24.0, 23.8 – 23.4 (m).

^{19}F NMR (282 MHz, Chloroform-*d*) δ -64.06 (s, 3F), -67.95 (d, *J* = 8.0 Hz, 6F).

HRMS (ESI-TOF): calcd for $C_{15}H_{13}F_9O_2Na$ [M+Na]: 419.0664; found 419.0664.

O-(7,7,7-Trifluoro-6-(trifluoromethyl)heptyl) dimethylcarbamothioate (6l)



General procedure E.

Yield 140 mg (86%). Colorless oil.

Chromatography: hexane/EtOAc, 12/1.

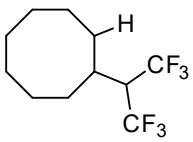
^{1}H NMR (300 MHz, Chloroform-*d*) δ 4.41 (t, *J* = 6.5 Hz, 2H), 3.32 (s, 3H), 3.07 (s, 3H), 2.93 – 2.71 (m, 1H), 1.93 – 1.63 (m, 4H), 1.62 – 1.32 (m, 4H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 188.3, 124.0 (qq, *J* = 281.1, 3.9 Hz), 71.2, 48.0 (hept, *J* = 27.8 Hz), 42.6, 37.6, 28.4, 26.9, 25.8, 23.78 – 23.45 (m).

^{19}F NMR (282 MHz, Chloroform-*d*) δ -67.97 (d, *J* = 8.4 Hz).

HRMS (ESI-TOF): calcd for $C_{11}H_{18}F_6NOS$ [M+H]: 326.1008; found 326.1006.

(1,1,1,3,3,3-Hexafluoropropan-2-yl)cyclooctane (6m)



General Procedure E.

Yield 104 mg (79%). Colorless liquid.

Chromatography: pentane.

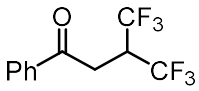
^{1}H NMR (300 MHz, Chloroform-*d*) δ 2.86 (heptd, *J* = 9.0, 1.8 Hz, 1H), 2.24 (t, *J* = 9.8 Hz, 1H), 1.54 (m, 14H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 124.4 (qq, *J* = 282.6, 4.0 Hz), 54.8 (hept, *J* = 25.9 Hz), 34.9, 31.2, 26.7, 26.2, 26.0.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -63.69 (d, *J* = 9.3 Hz).

MS (EI): 262 [M^+], 233, 111, 84, 69 [CF_3].

4,4,4-Trifluoro-1-phenyl-3-(trifluoromethyl)butan-1-one (8a)



General procedure F.

Yield 80 mg (59%). Colorless oil.

Chromatography: pentane/DCM, 5/1.

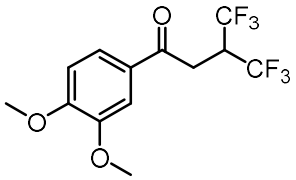
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 7.5 Hz, 2H), 7.65 (t, *J* = 7.5 Hz, 1H), 7.52 (t, *J* = 7.5 Hz, 2H), 4.33 – 4.11 (m, 1H), 3.42 (d, *J* = 5.4 Hz, 2H).

^{13}C NMR (75 MHz, Chloroform-*d*) δ 192.83, 135.51, 134.32, 129.11, 128.38, 123.89 (qq, *J* = 280.4, 3.7 Hz), 43.09 (hept, *J* = 29.3 Hz), 32.54 (pent, *J* = 1.7 Hz), 29.86.

^{19}F NMR (282 MHz, Chloroform-*d*) δ -68.59 (d, *J* = 8.4 Hz).

HRMS (ESI-TOF): calcd for $C_{11}H_8F_6ONa$ [M+Na]: 293.0372; found 293.0363.

1-(3,4-Dimethoxyphenyl)-4,4,4-trifluoro-3-(trifluoromethyl)butan-1-one (8b)



General procedure F.

Yield 142 mg (86%). Yellow oil.

Chromatography: Hexane/EtOAc, 5/1.

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.60 (dd, *J* = 8.4, 2.1 Hz, 1H), 7.53 (d, *J* = 2.1 Hz, 1H), 6.92 (d, *J* = 8.4 Hz, 1H), 4.27 – 4.09 (m, 1H), 3.95 (s, 3H), 3.93 (s, 3H), 3.38 (d, *J* = 5.4 Hz, 2H).

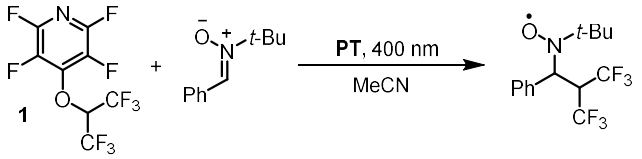
^{13}C NMR (75 MHz, Chloroform-*d*) δ 191.3, 154.4, 149.5, 128.7, 123.9 (qq, *J* = 280.5, 3.7 Hz), 123.1, 110.5, 110.3, 56.3, 56.2, 43.2 (hept, *J* = 29.1 Hz), 32.0 (s).

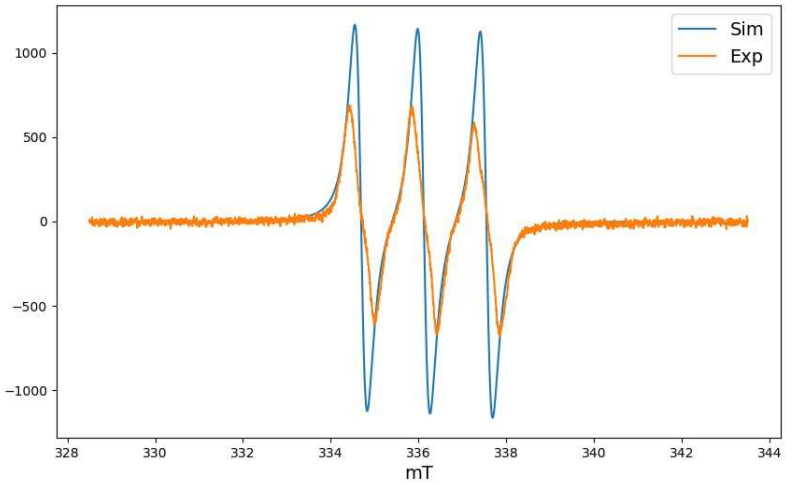
^{19}F NMR (282 MHz, Chloroform-*d*) δ -68.51 (d, *J* = 8.5 Hz).

HRMS (ESI-TOF): calcd for $C_{13}H_{12}F_6O_3Na$ [M+Na]: 353.0583; found 353.0579.

EPR study

A screw test tube was evacuated, refilled with argon, and charged with reagent **1** (32 mg, 0.1 mmol), *N*-*tert*-butyl-1-phenylmethanimine oxide (18 mg, 0.1 mmol) and 12-phenyl-12H-benzo[b]phenothiazine (**PT**) (1 mg, 0.01 mmol), followed by addition of MeCN (0.5 mL). Form the resulting solution, an aliquot (ca. 10 µl) was taken and placed into an EPR tube, which was then irradiated with 400 nm LED matrix (80W) for 180 seconds. The EPR spectrum was immediately recorded at 298 K on EPR spectrometer SPINSCAN X (ADANI).





The X-band EPR spectrum of the nitroxyl radical (orange line). Simulated EPR spectrum (blue line) based on hyperfine coupling constants of a_N= 14.28 G (g-factor = 2.0056).

Experiment parameters:

Center-Field: 336.0 mT

Width: 15 G

Points: 6000

Modulation Amplitude: 100 µT

Modulation Frequency: 9.432985 GHz

Microwave Power: 31.6 mW

Time constant: 0.015 s

Cyclic Voltammetry

Voltammetric studies were carried out using potentiostat P30JM with a scan rates of 0.1 $V \cdot s^{-1}$ in a temperature-controlled (25 °C) glass cell (V = 10 mL) under an argon atmosphere. Software *iR* compensation using ferrocene (R = 1362 Ω) was used. A glassy carbon disk (d = 2.9 mm) was used as the working electrode (carefully polished before each measurement). A saturated calomel electrode (SCE) separated from the solution being studied by a salt bridge filled with the supporting electrolyte (0.1M Et_4NClO_4 in DMSO) was used as the reference electrode. A platinum wire (S = 3 cm^2) was used as the counter electrode. Experiment was performed with the concentration of compound **1** of 1 mM.

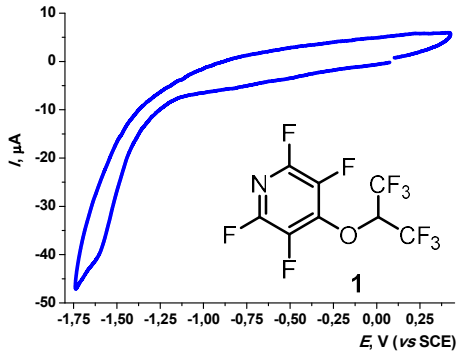


Figure S1. Compound **1** (initial cathodic scan).

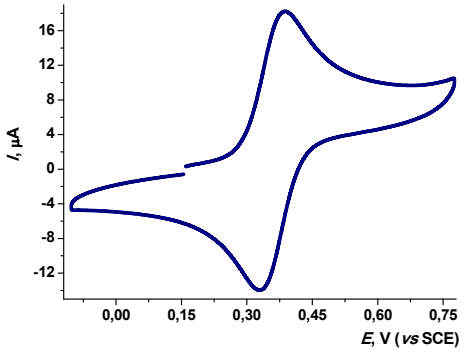


Figure S2. Ferrocene (initial anodic scan).

Compound **1** showed irreversible cathodic peak at – 1.65 V (*vs.* SCE).

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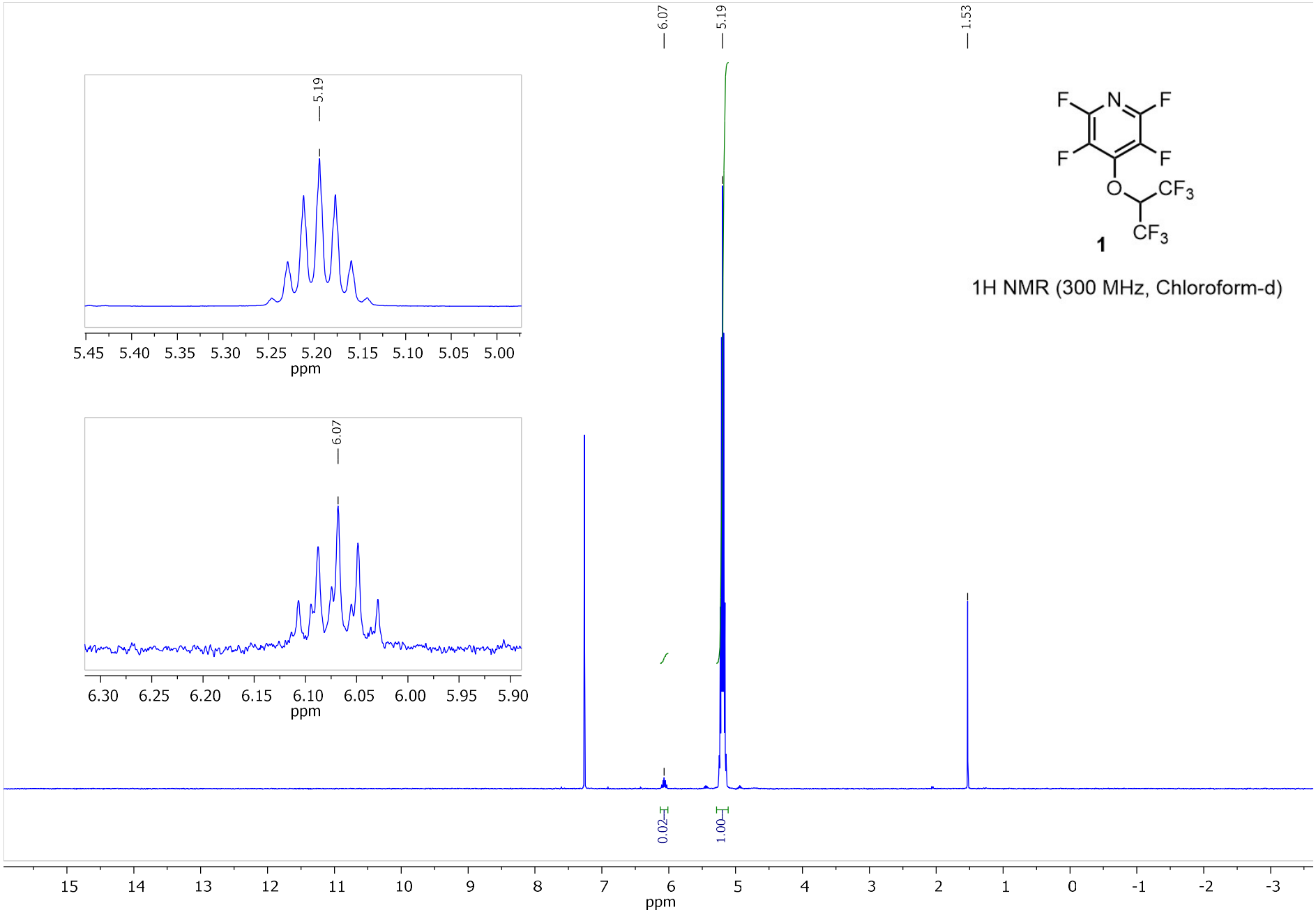
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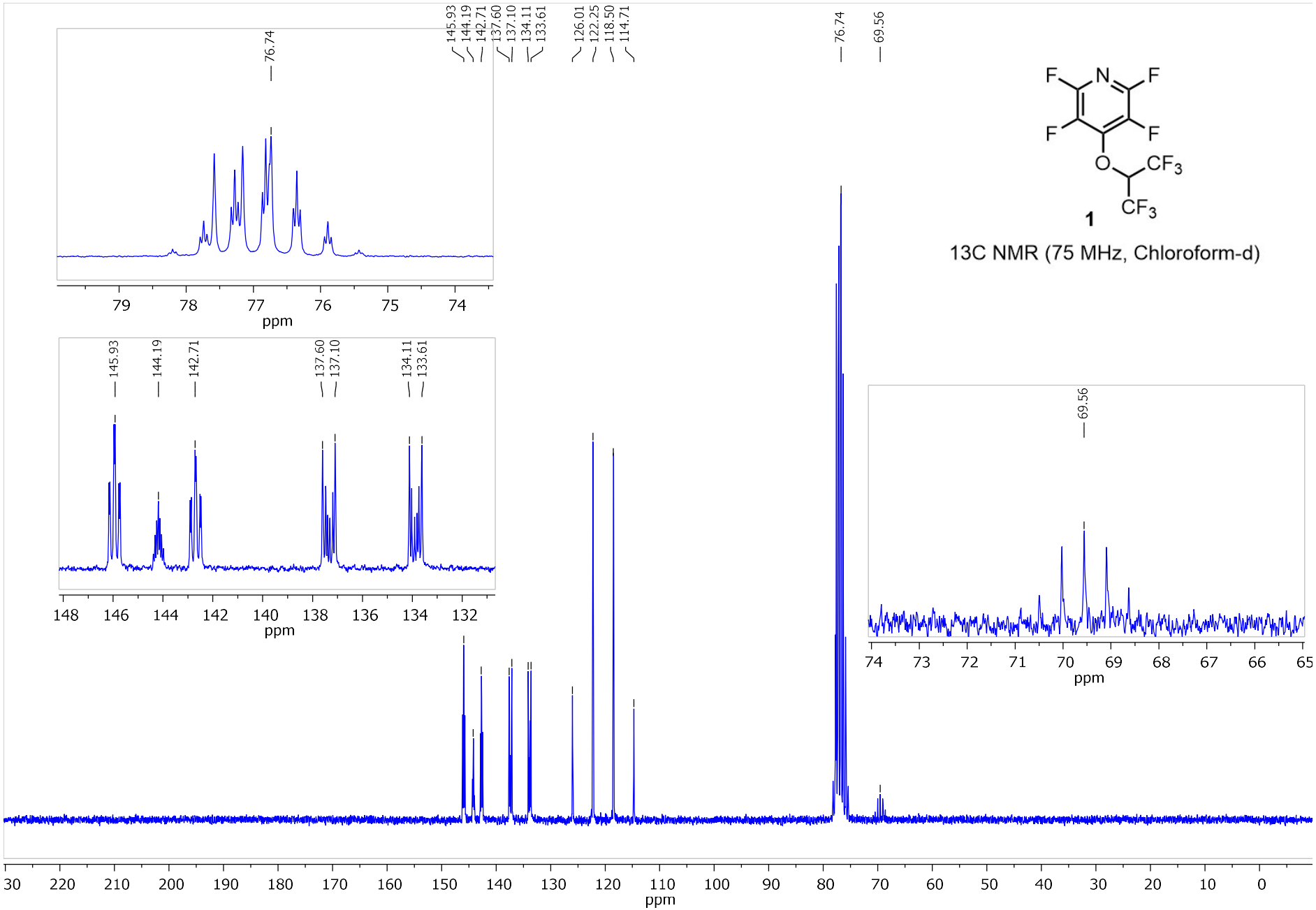
5.19
1.53
5.19
5.45 5.40 5.35 5.30 5.25 5.20 5.15 5.10 5.05 5.00
ppm
6.07
6.30 6.25 6.20 6.15 6.10 6.05 6.00 5.95 5.90
ppm
F
N
F
F
F
O
CF3
CF3
1
1H NMR (300 MHz, Chloroform-d)
0.02
1.00
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
ppm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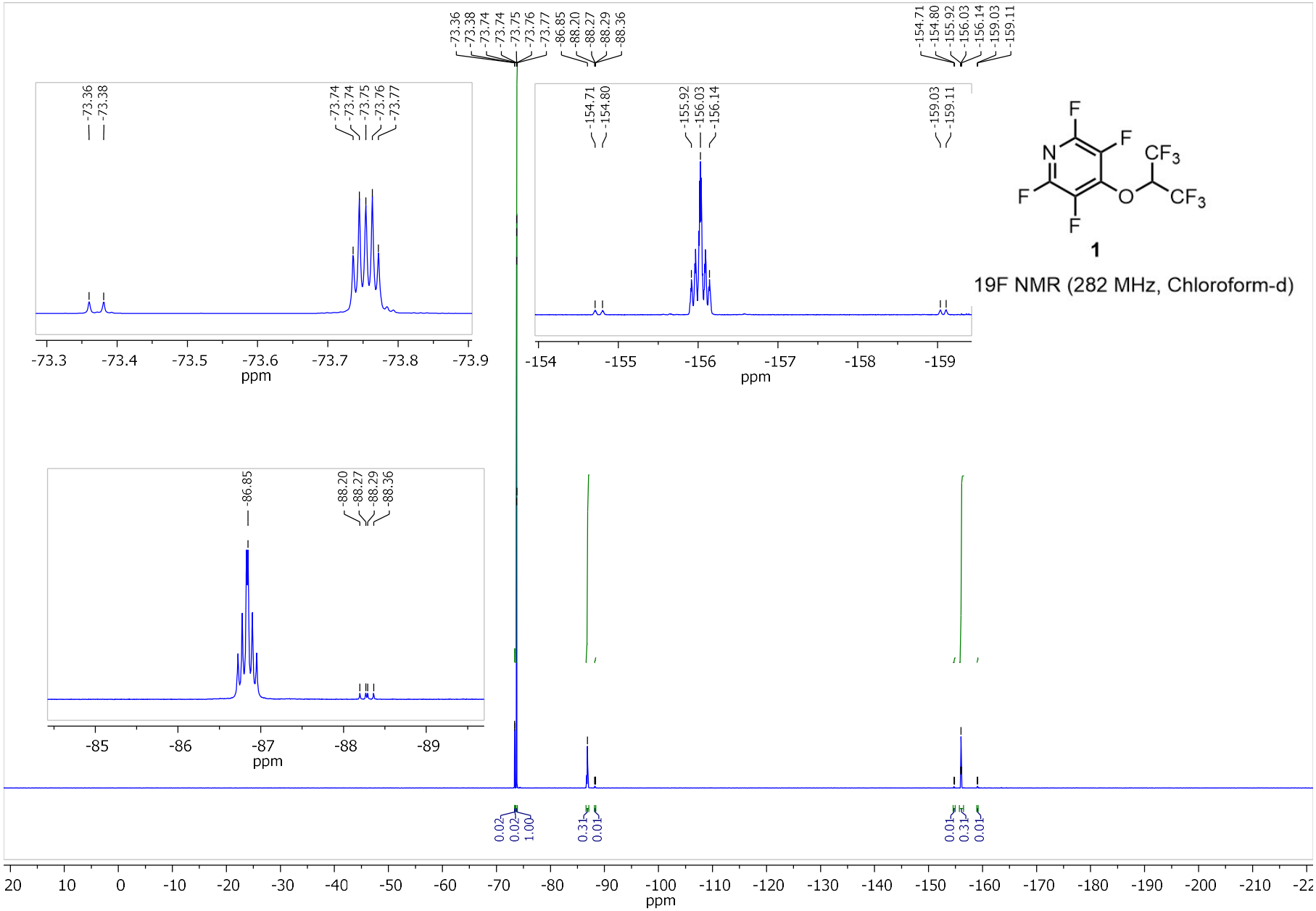
144.19
142.71
137.60
137.10
134.11
133.61
126.01
122.25
118.50
114.71
76.74
69.56
F
N
F
F
F
O
CF3
CF3
1
13C NMR (75 MHz, Chloroform-d)
ppm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-73.74
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-73.77
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-156.03
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ppm
F
F
N
F
CF3
O
CF3
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1
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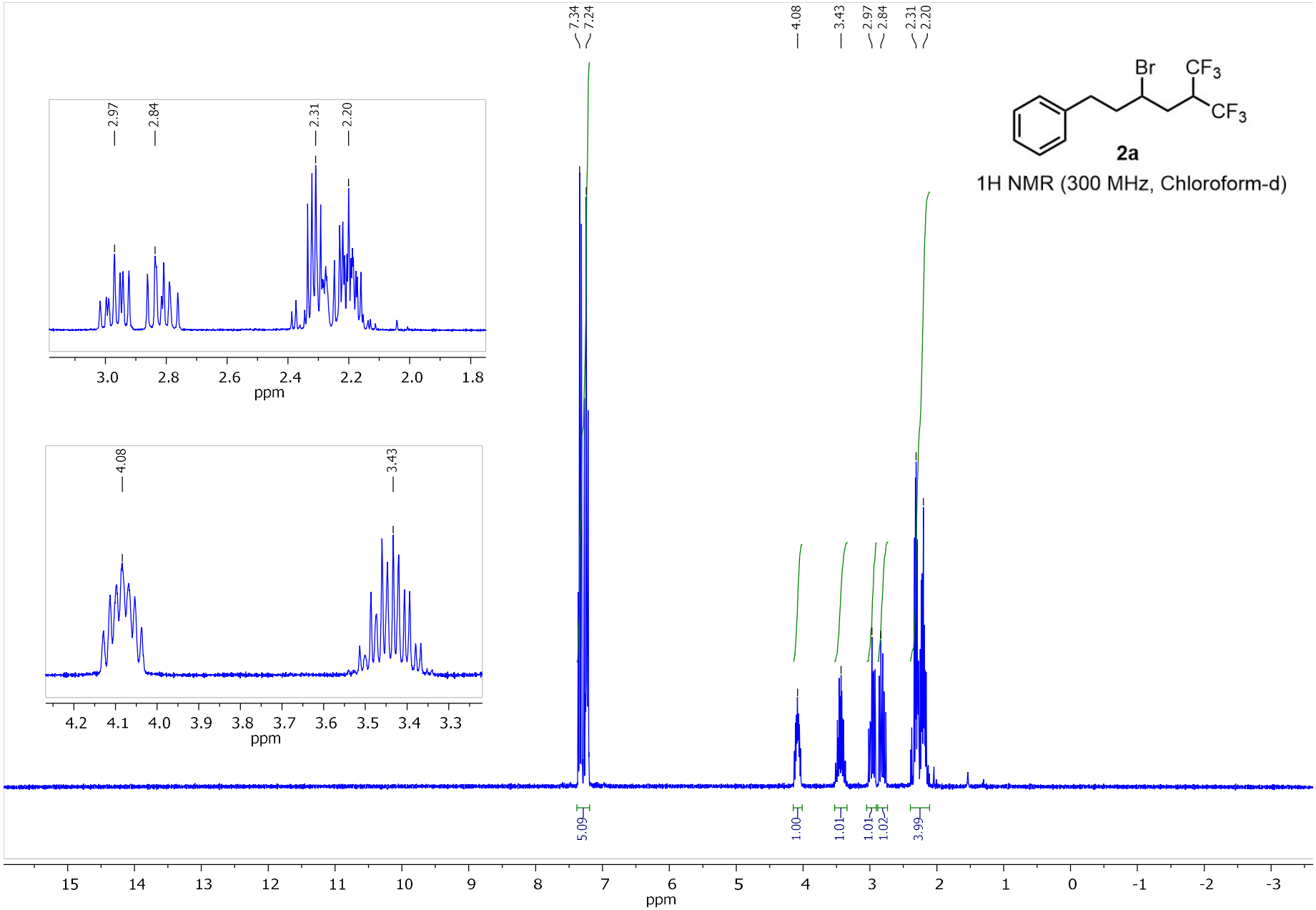
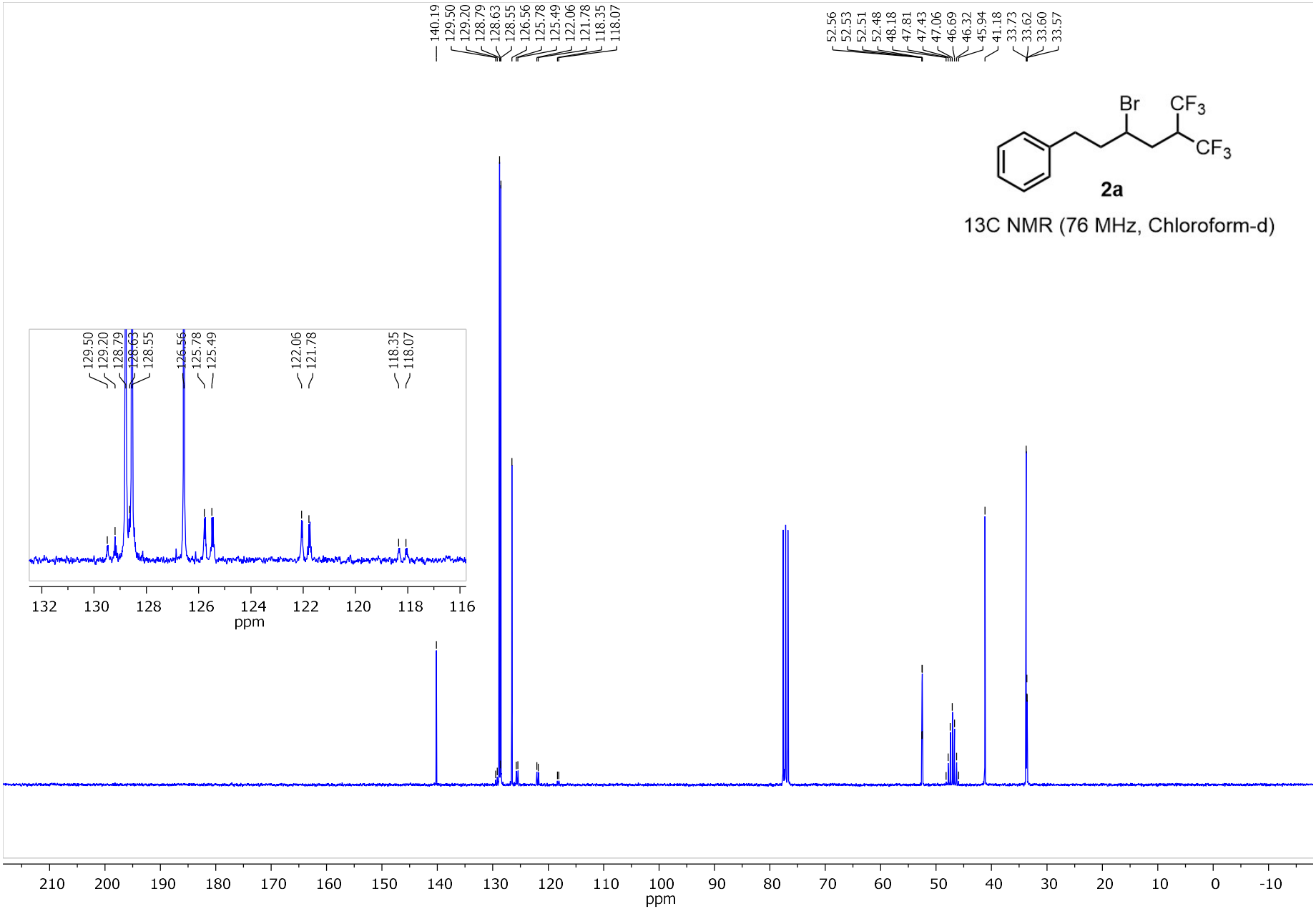
0.02
1.00
0.31
0.01
0.01
0.31
0.01


7.34
7.24
4.08
3.43
2.97
2.84
2.31
2.20
Br
CF3
CF3
2a
1H NMR (300 MHz, Chloroform-d)
ppm
5.09
1.00
1.01
1.01
1.02
3.99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128.63
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52.53
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47.81
47.43
47.06
46.69
46.32
45.94
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33.73
33.62
33.60
33.57
Br
CF3
CF3
2a
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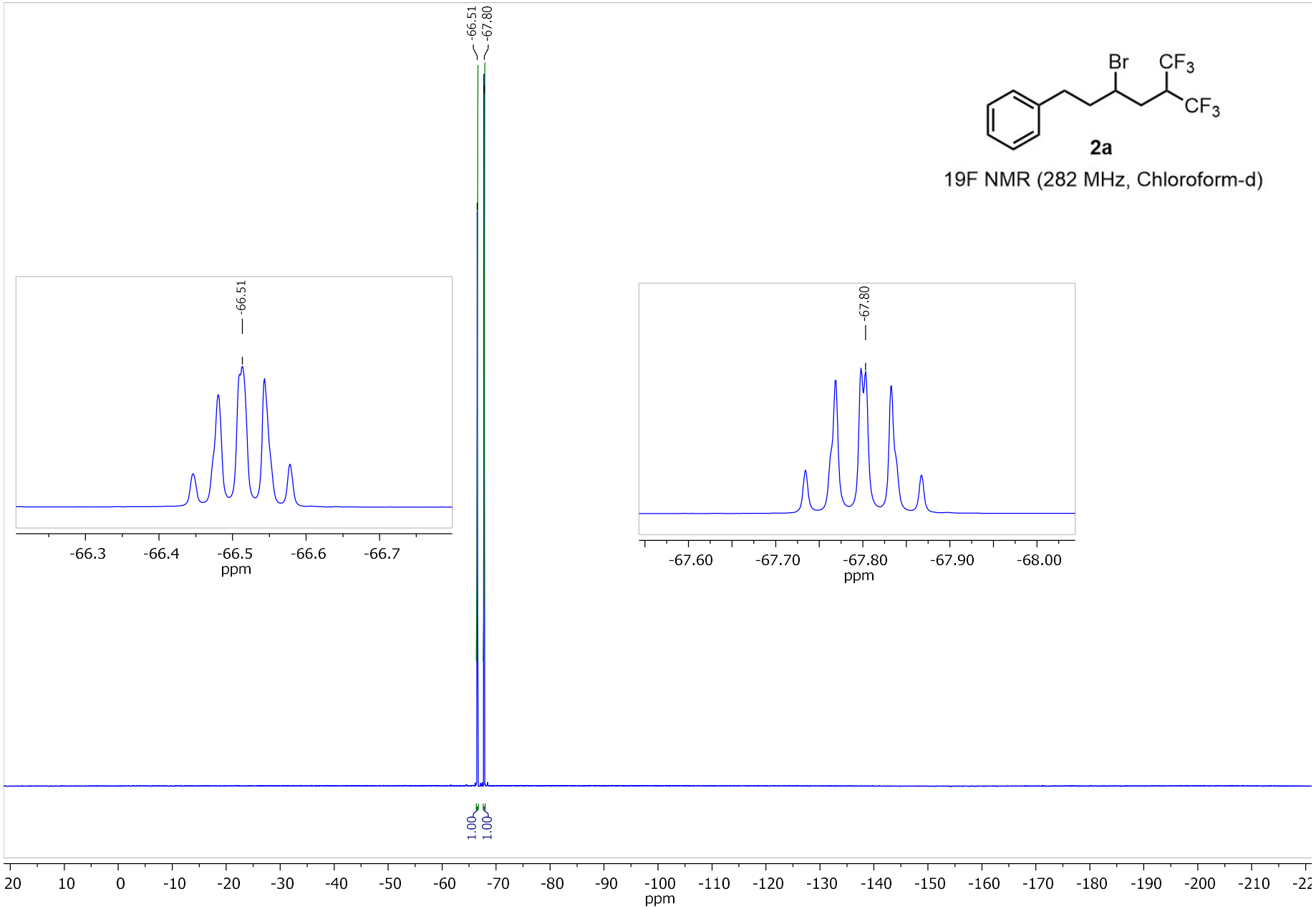
ppm


-66.51
-67.80
Br
CF3
CF3
2a
19F NMR (282 MHz, Chloroform-d)
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-66.3
-66.4
-66.5
-66.6
-66.7
ppm
-67.80
-67.60
-67.70
-67.80
-67.90
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ppm
1.00
1.00
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
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-100
-110
-120
-130
-140
-150
-160
-170
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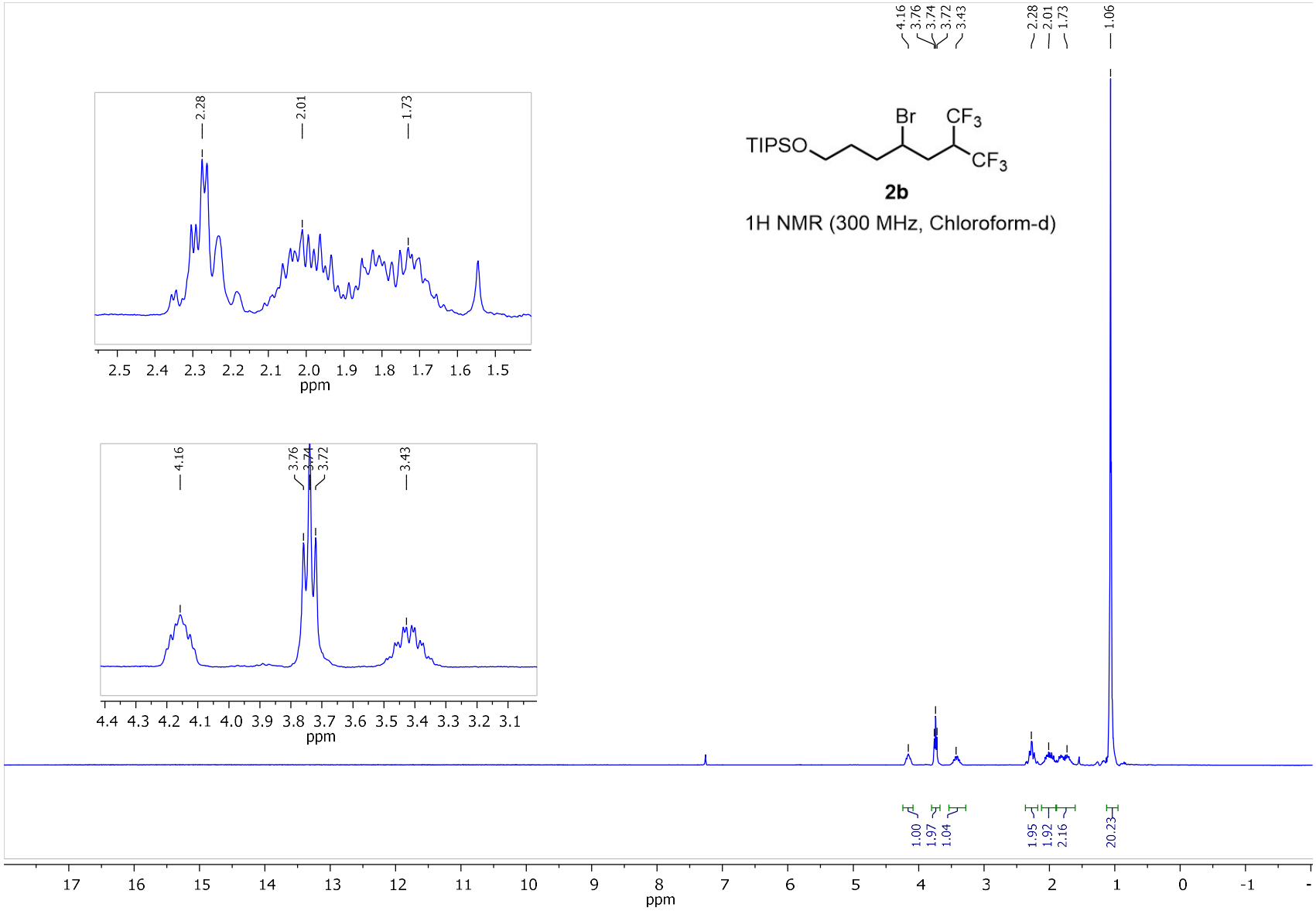
-190
-200
-210
ppm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3.74
3.72
3.43
2.28
2.01
1.73
1.06
Br
CF3
TIPSO
CF3
2b
1H NMR (300 MHz, Chloroform-d)
ppm
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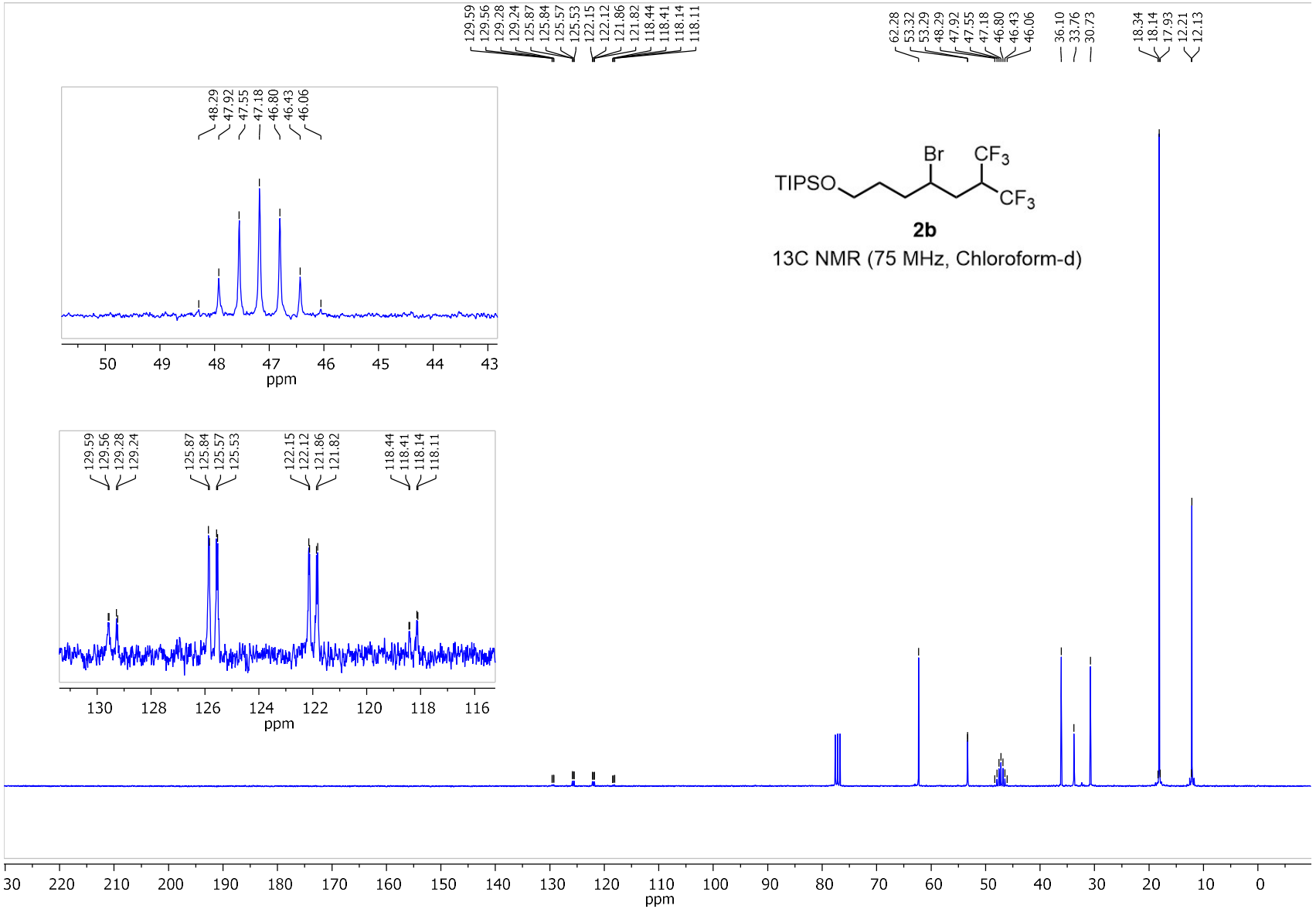
1.97
1.04
1.95
1.92
2.16
20.23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121.82
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118.41
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118.11
62.28
53.32
53.29
48.29
47.92
47.55
47.18
46.80
46.43
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36.10
33.76
30.73
18.34
18.14
17.93
12.21
12.13
48.29
47.92
47.55
47.18
46.80
46.43
46.06
50
49
48
47
46
45
44
43
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116
ppm
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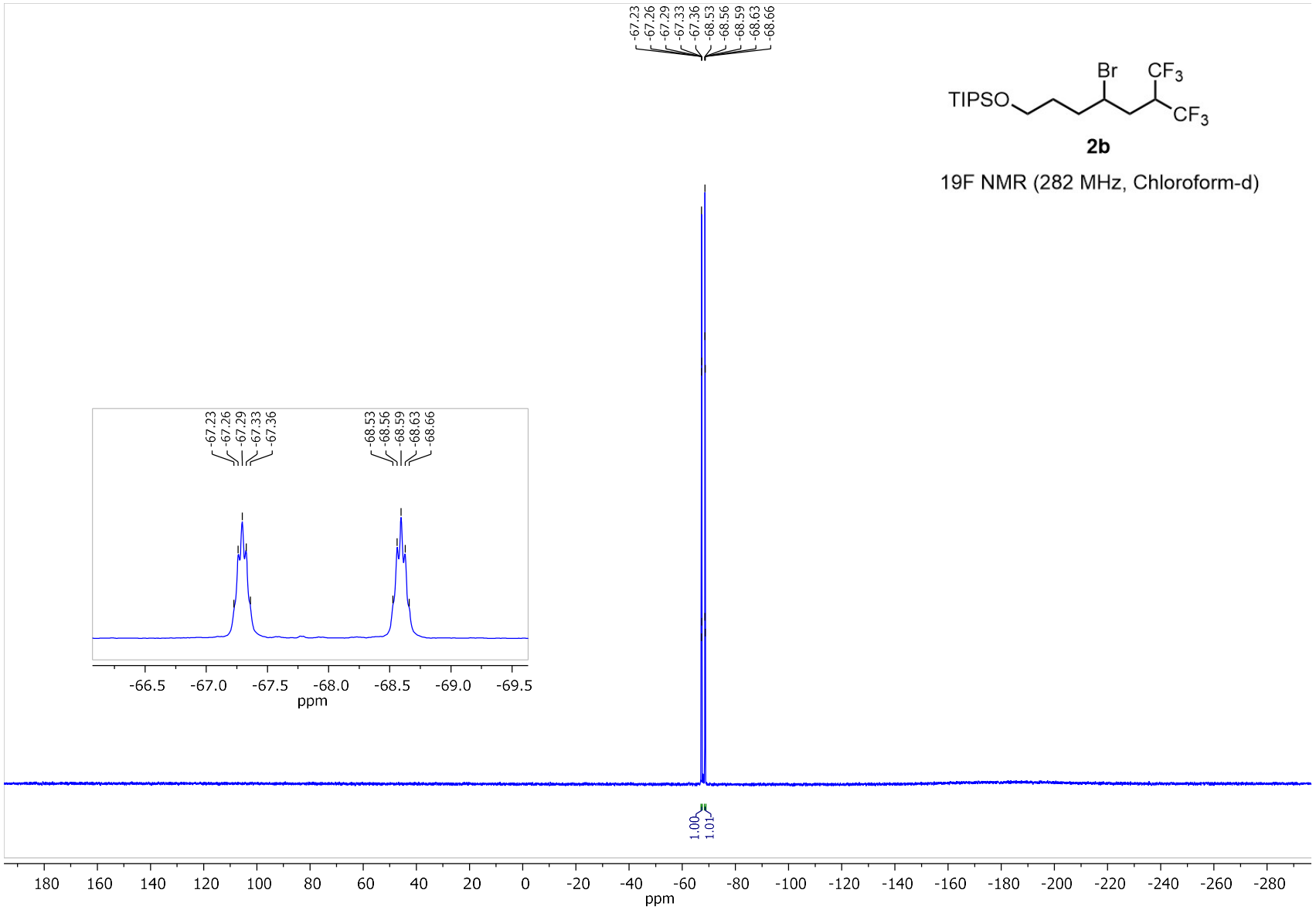
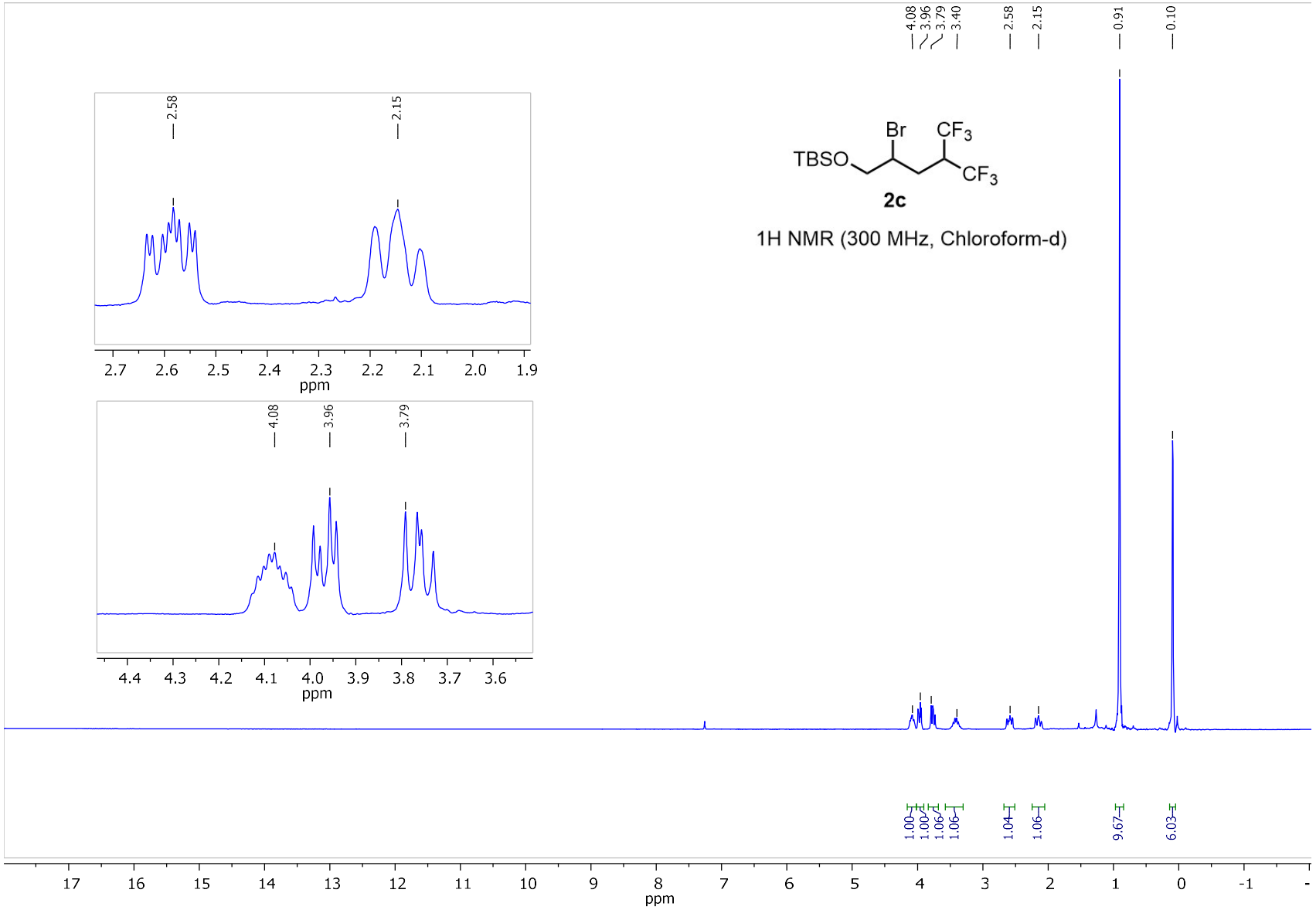
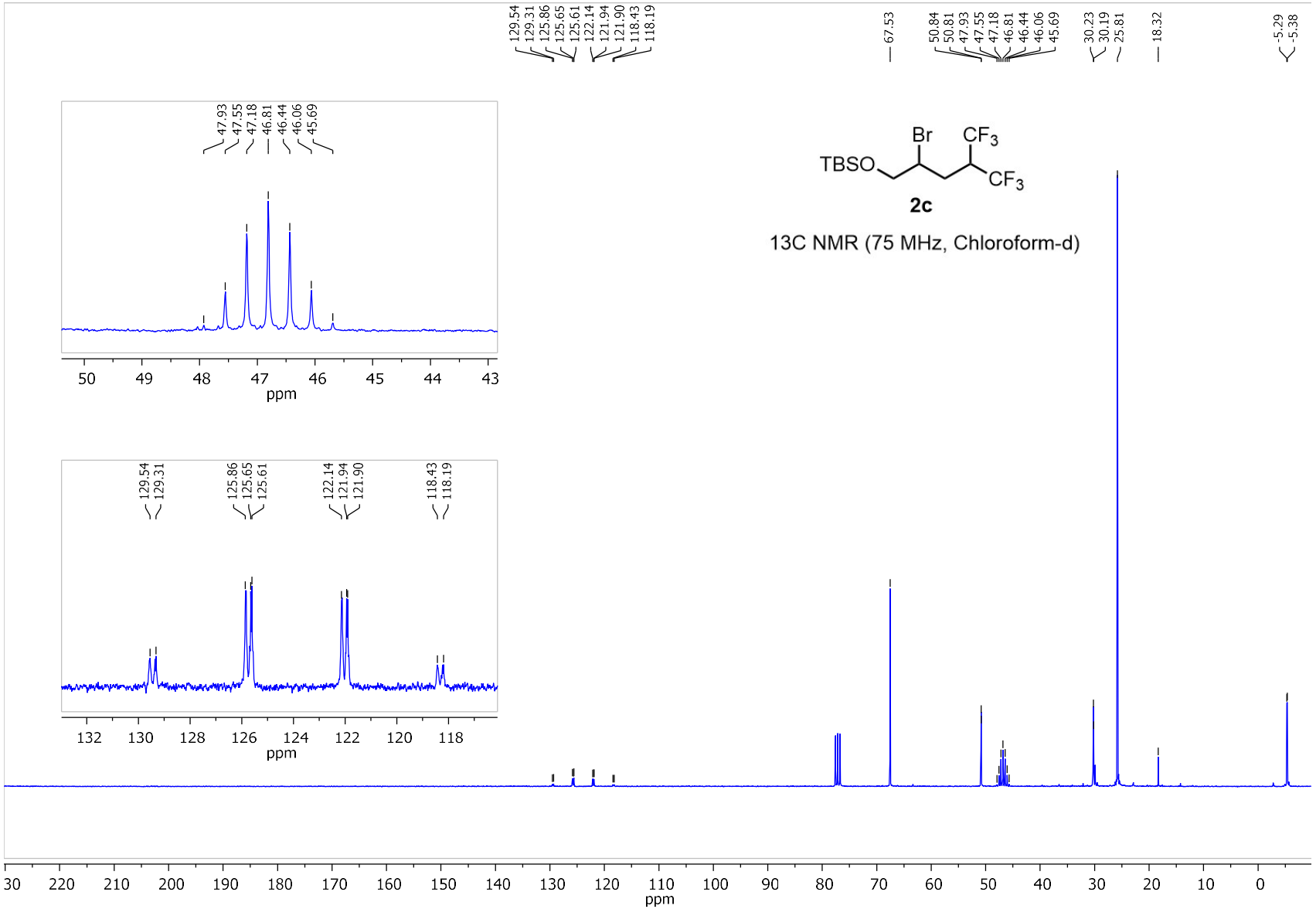
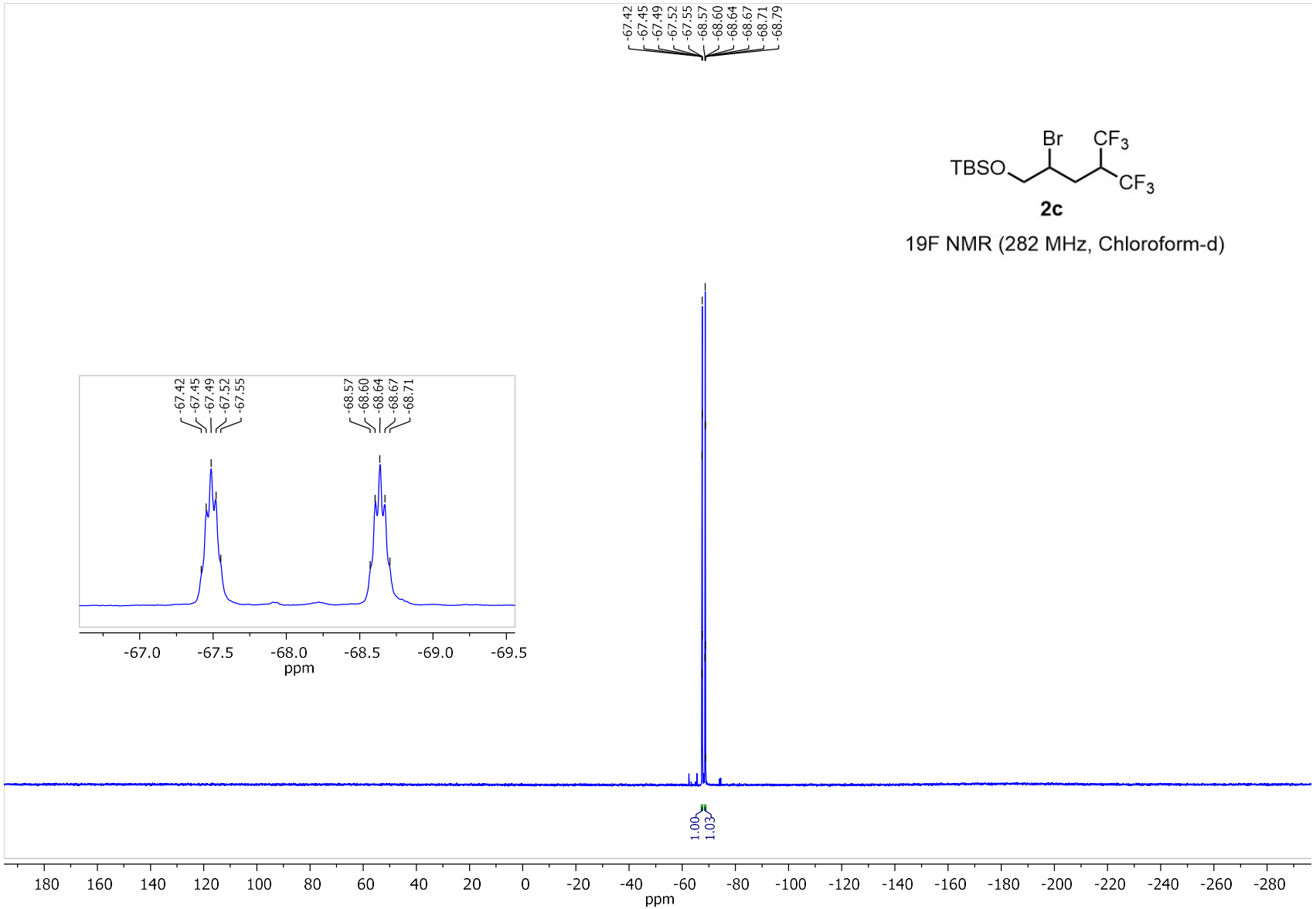
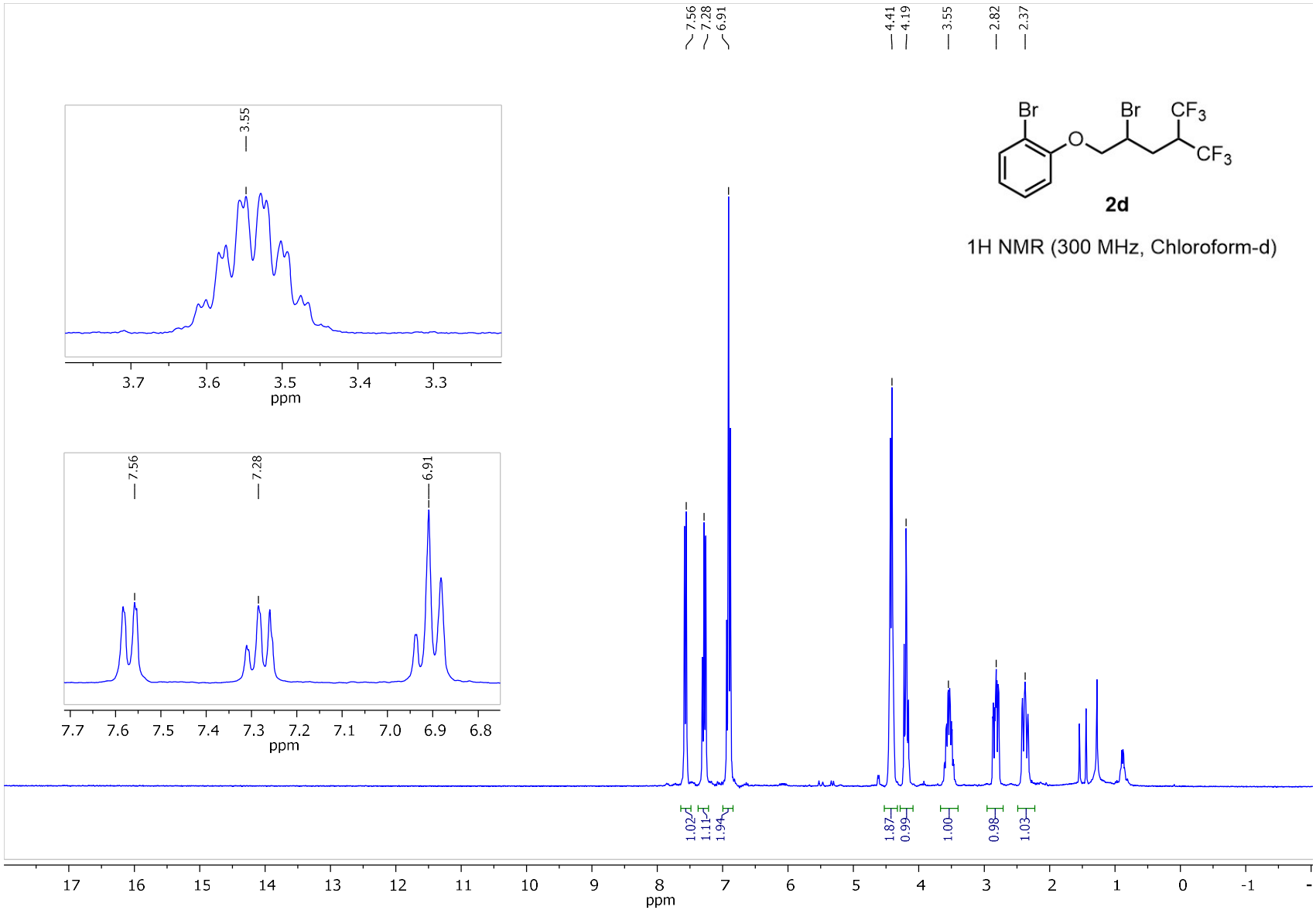
TIPSO
CF3
2b
13C NMR (75 MHz, Chloroform-d)
30
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

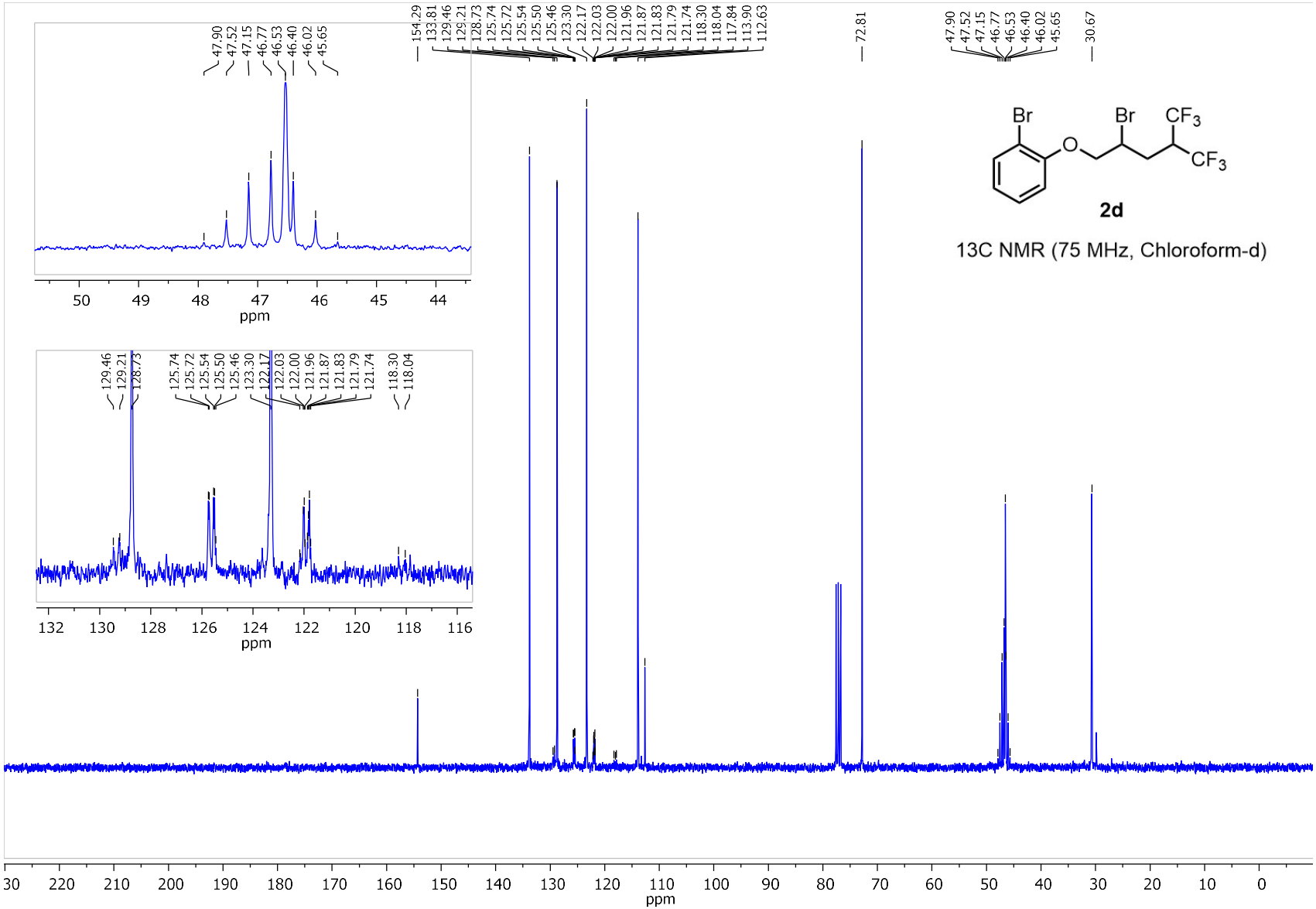

-67.23
-67.26
-67.29
-67.33
-67.36
-68.53
-68.56
-68.59
-68.63
-68.66
Br
CF3
TIPSO
CF3
2b
19F NMR (282 MHz, Chloroform-d)
1.00
1.01
ppm
-66.5
-67.0
-67.5
-68.0
-68.5
-69.0
-69.5
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280

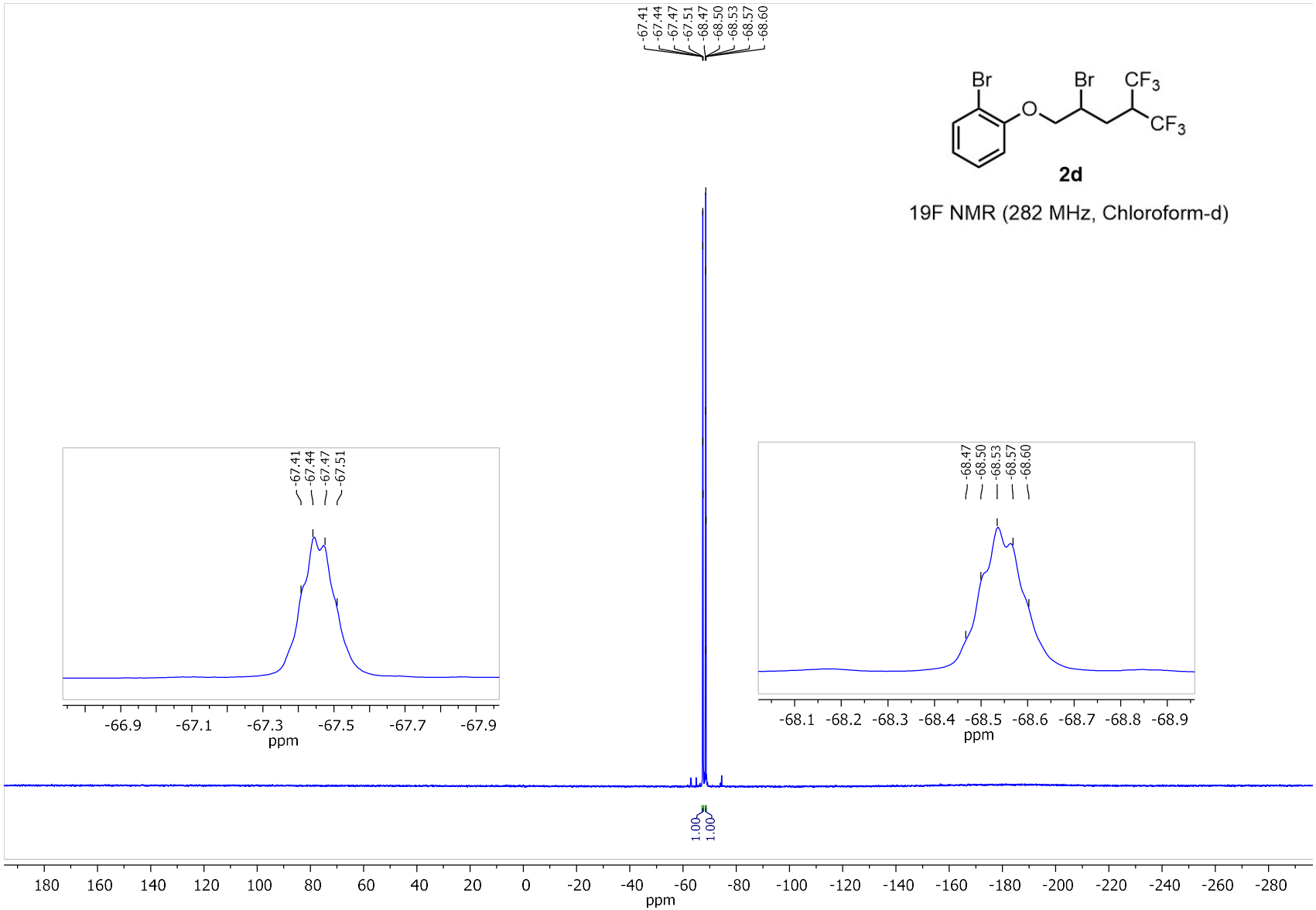

4.08
3.96
3.79
3.40
2.58
2.15
0.91
0.10
Br
CF3
TBSO
CF3
2c
1H NMR (300 MHz, Chloroform-d)
2.58
2.15
ppm
4.08
3.96
3.79
ppm
1.00
1.00
1.06
1.06
1.04
1.06
9.67
6.03
ppm

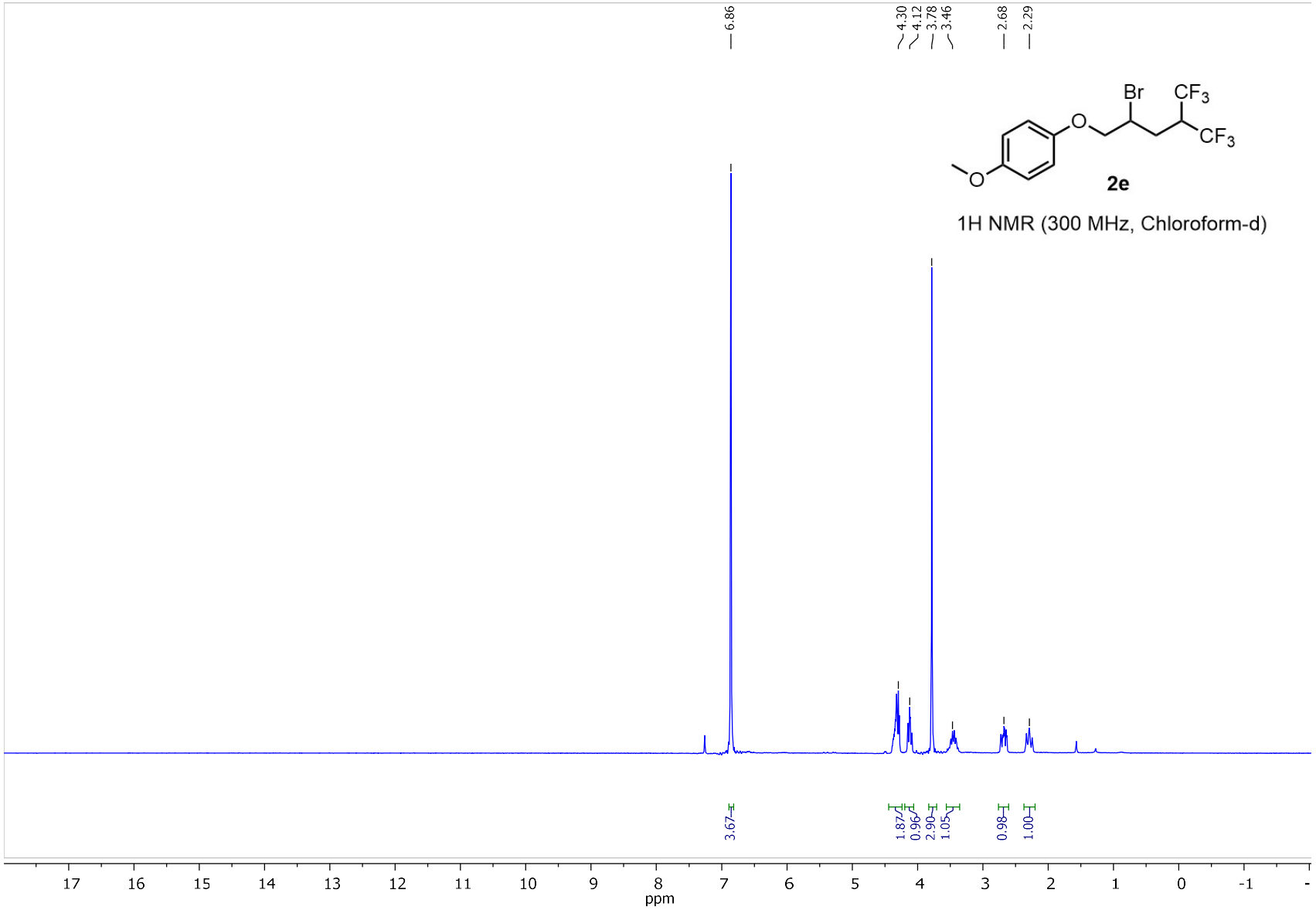

129.54
129.31
125.86
125.65
125.61
122.14
121.94
121.90
118.43
118.19
67.53
50.84
50.81
47.93
47.55
47.18
46.81
46.44
46.06
45.69
30.23
30.19
25.81
18.32
-5.29
-5.38
Br
CF3
TBSO
CF3
2c
13C NMR (75 MHz, Chloroform-d)
ppm

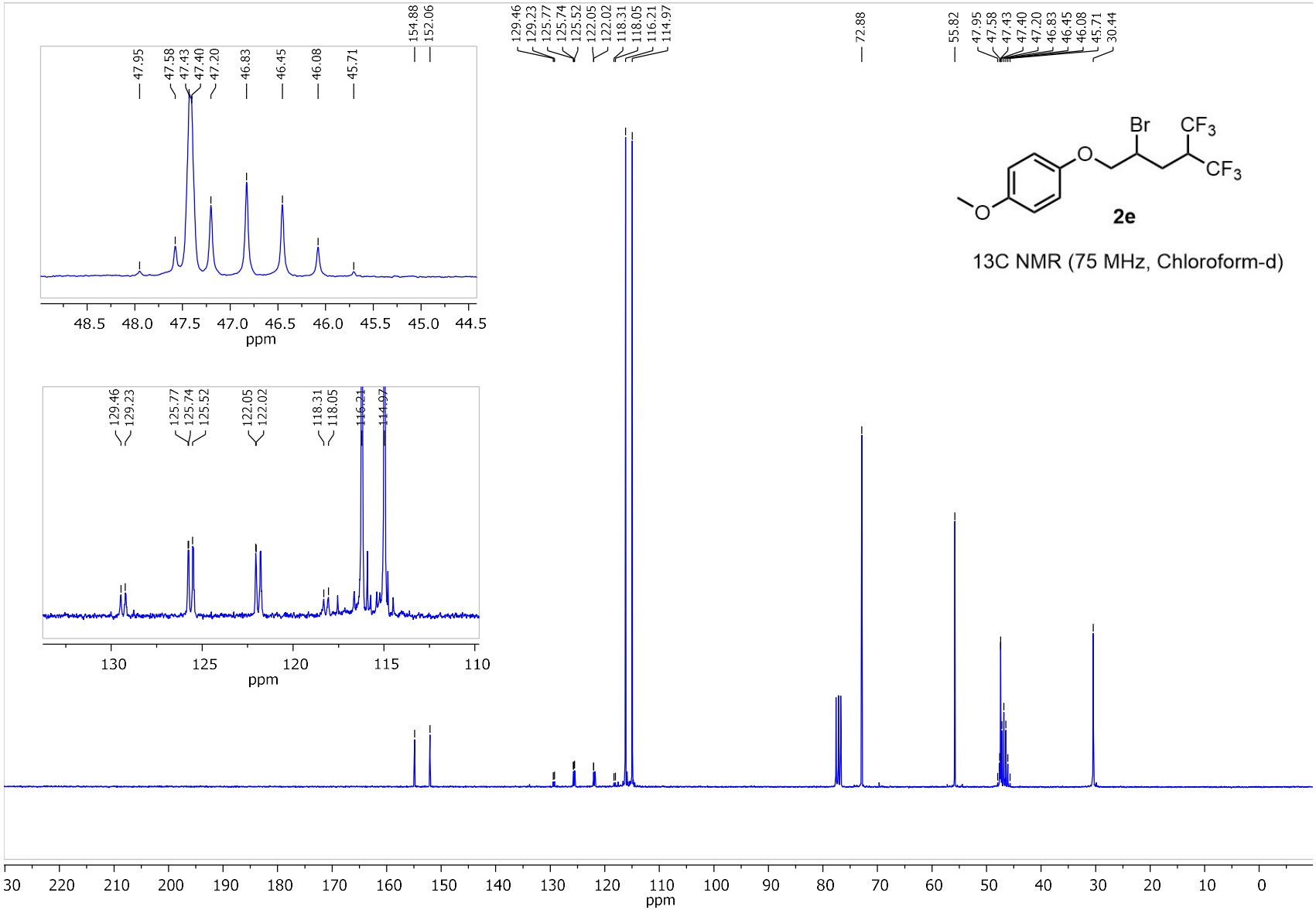

-67.42
-67.45
-67.49
-67.52
-67.55
-68.57
-68.60
-68.64
-68.67
-68.71
-68.79
Br
CF3
TBSO
CF3
2c
19F NMR (282 MHz, Chloroform-d)
-67.42
-67.45
-67.49
-67.52
-67.55
-68.57
-68.60
-68.64
-68.67
-68.71
-67.0
-67.5
-68.0
-68.5
-69.0
-69.5
ppm
1.00
1.03
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm

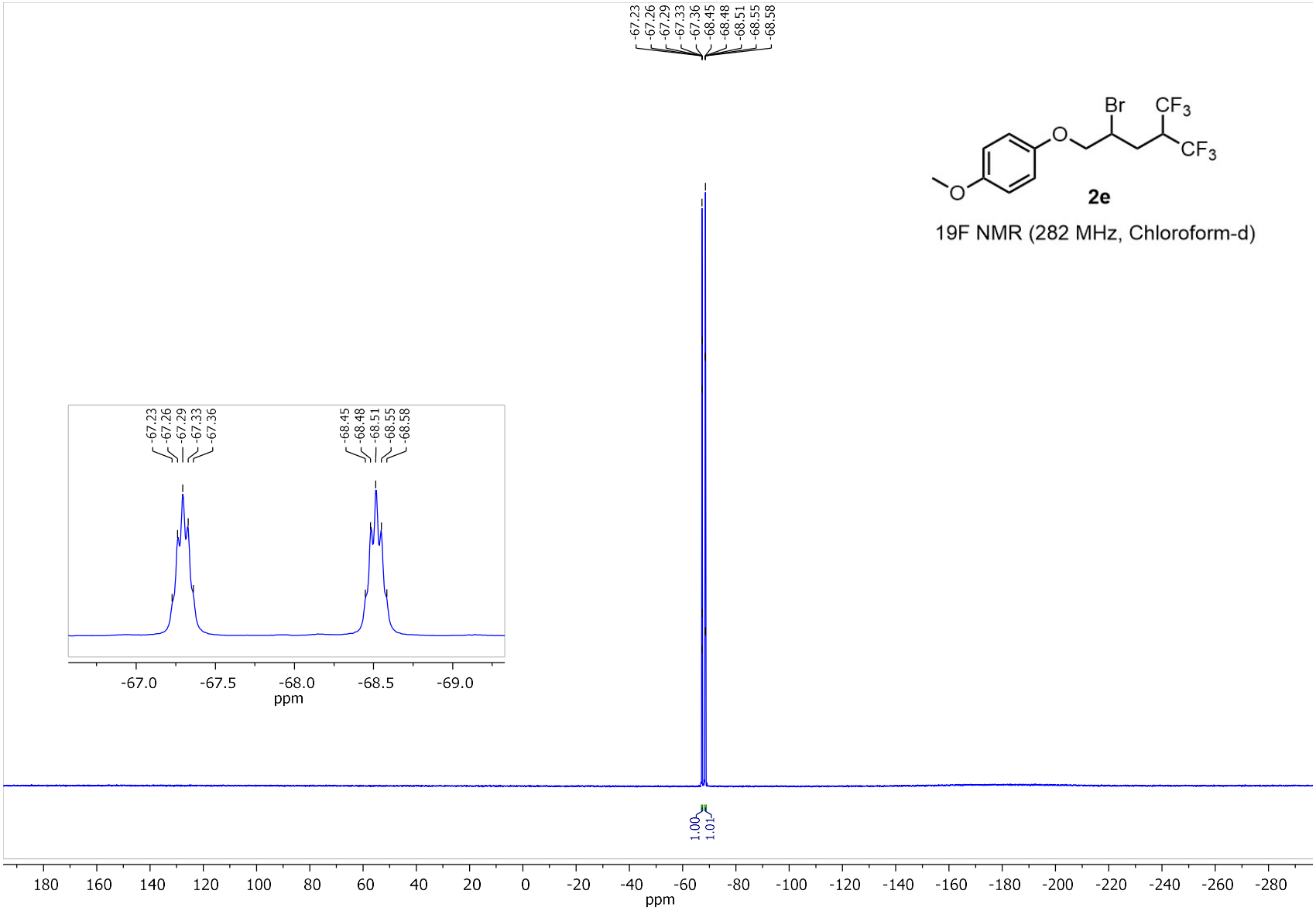

7.56
7.28
6.91
4.41
4.19
3.55
2.82
2.37
Br
Br
CF3
O
CF3
2d
1H NMR (300 MHz, Chloroform-d)
3.55
3.7
3.6
3.5
3.4
3.3
ppm
7.56
7.28
6.91
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
6.8
ppm
1.02
1.11
1.94
1.87
0.99
1.00
0.98
1.03
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

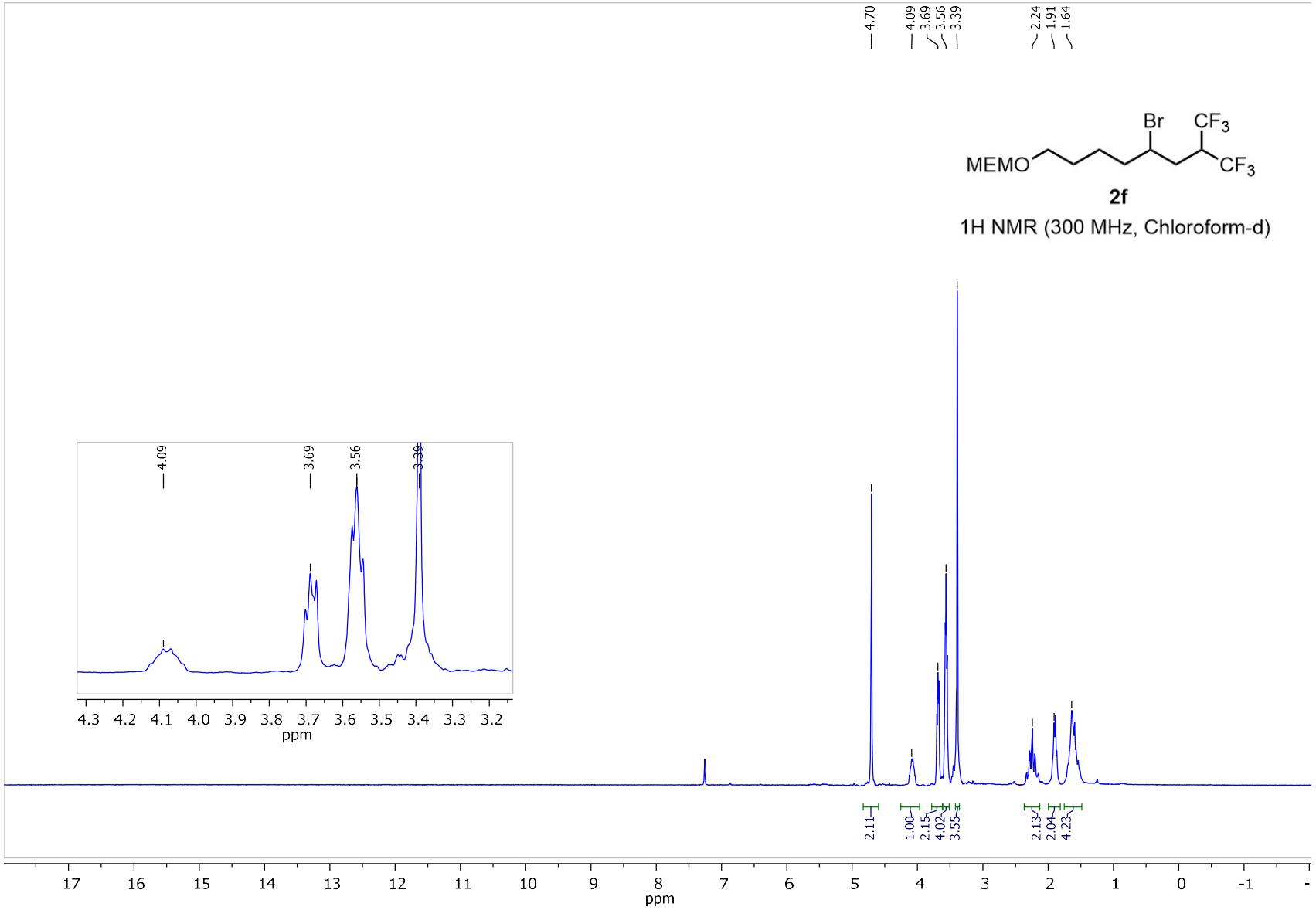

154.29
133.81
129.46
129.21
128.73
125.74
125.72
125.54
125.50
125.46
123.30
122.17
122.03
122.00
121.96
121.87
121.83
121.79
121.74
118.30
118.04
117.84
113.90
112.63
72.81
47.90
47.52
47.15
46.77
46.53
46.40
46.02
45.65
30.67
Br
Br
CF3
CF3
O
2d
13C NMR (75 MHz, Chloroform-d)
ppm

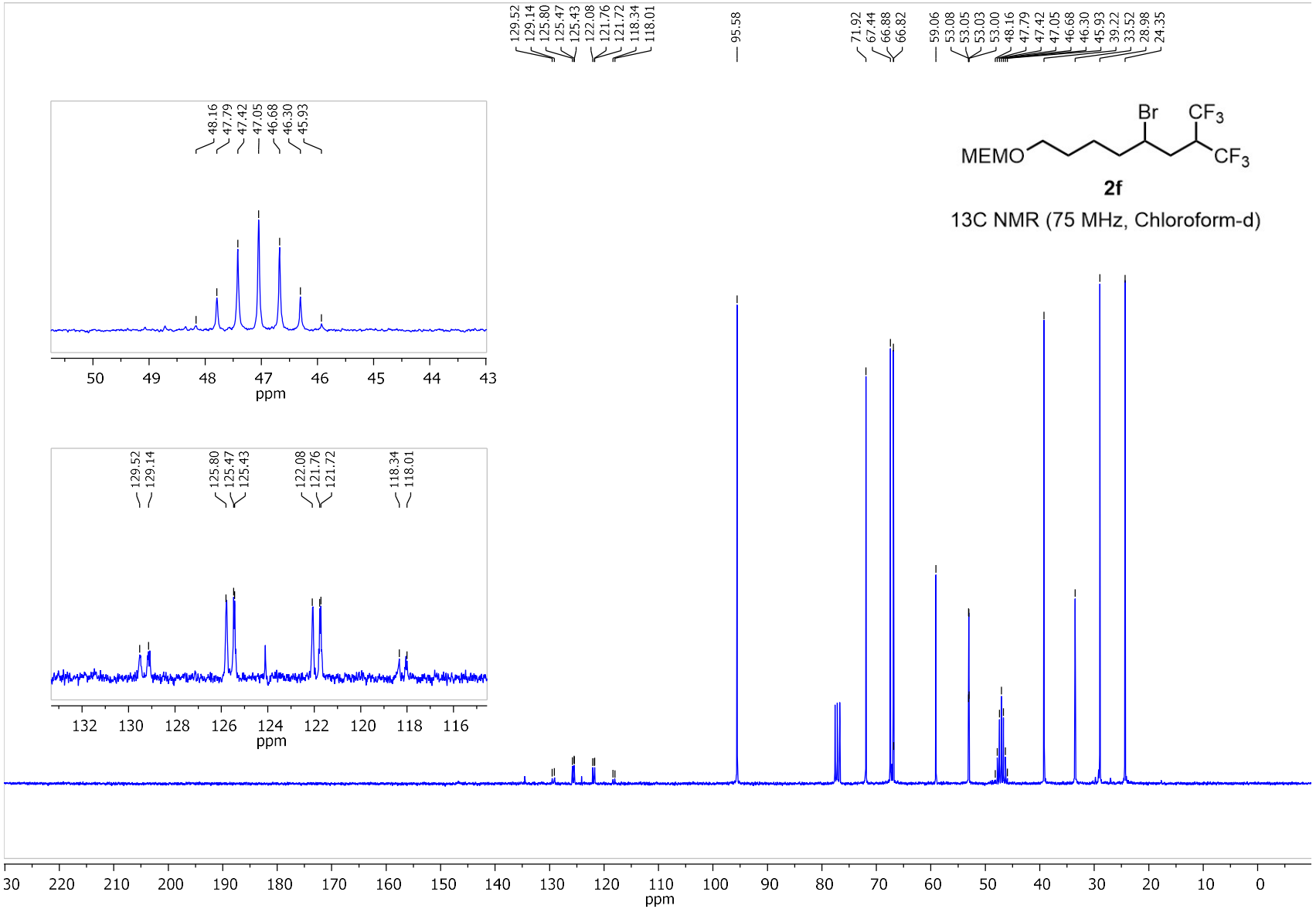

-67.41
-67.44
-67.47
-67.51
-68.47
-68.50
-68.53
-68.57
-68.60
Br
Br
CF3
O
CF3
2d
19F NMR (282 MHz, Chloroform-d)
-66.9
-67.1
-67.3
-67.5
-67.7
-67.9
ppm
-68.1
-68.2
-68.3
-68.4
-68.5
-68.6
-68.7
-68.8
-68.9
ppm
1.00
1.00
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm

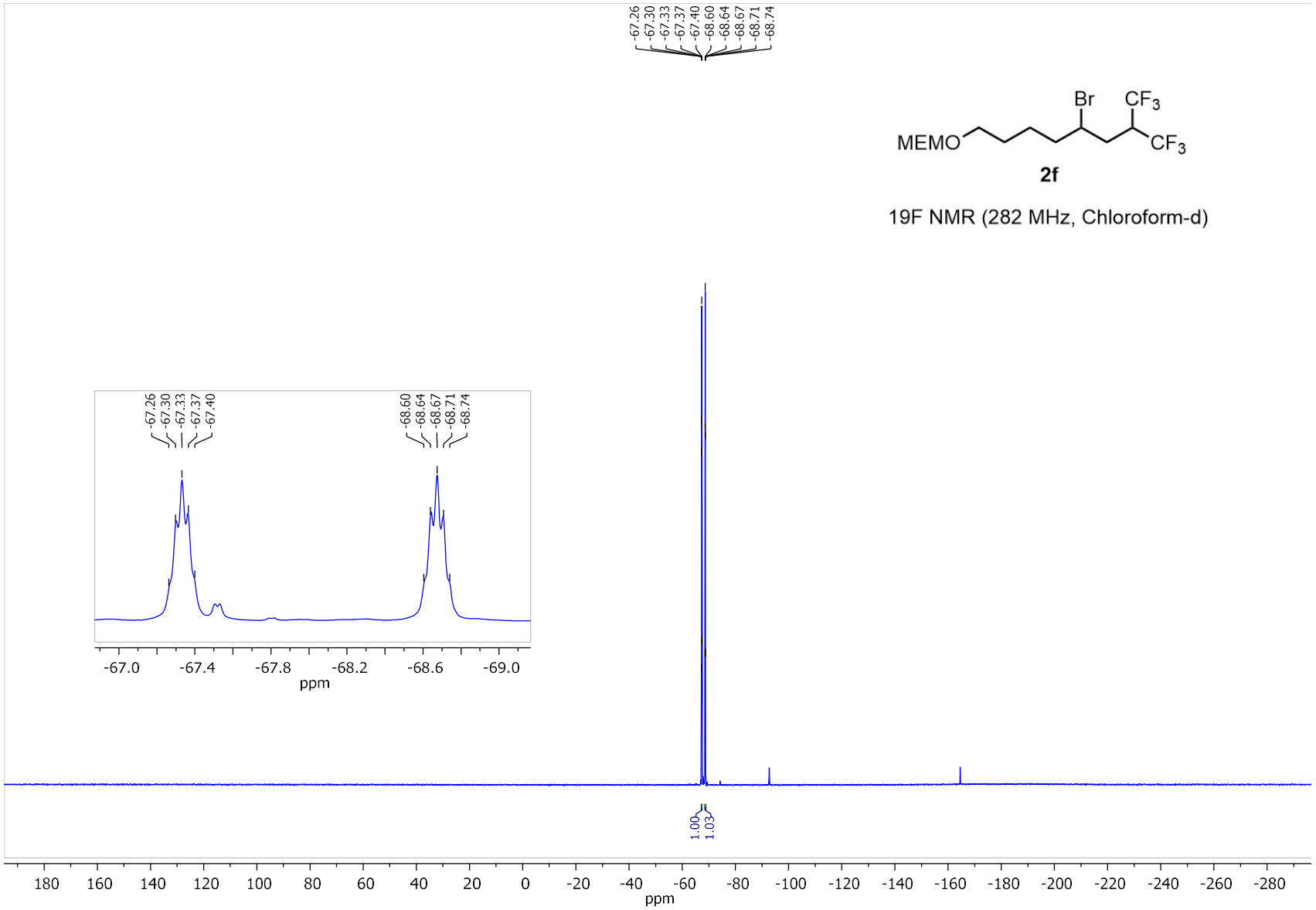

6.86
4.30
4.12
3.78
3.46
2.68
2.29
Br
CF3
CF3
O
O
2e
1H NMR (300 MHz, Chloroform-d)
3.67
1.87
0.96
2.90
1.05
0.98
1.00
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

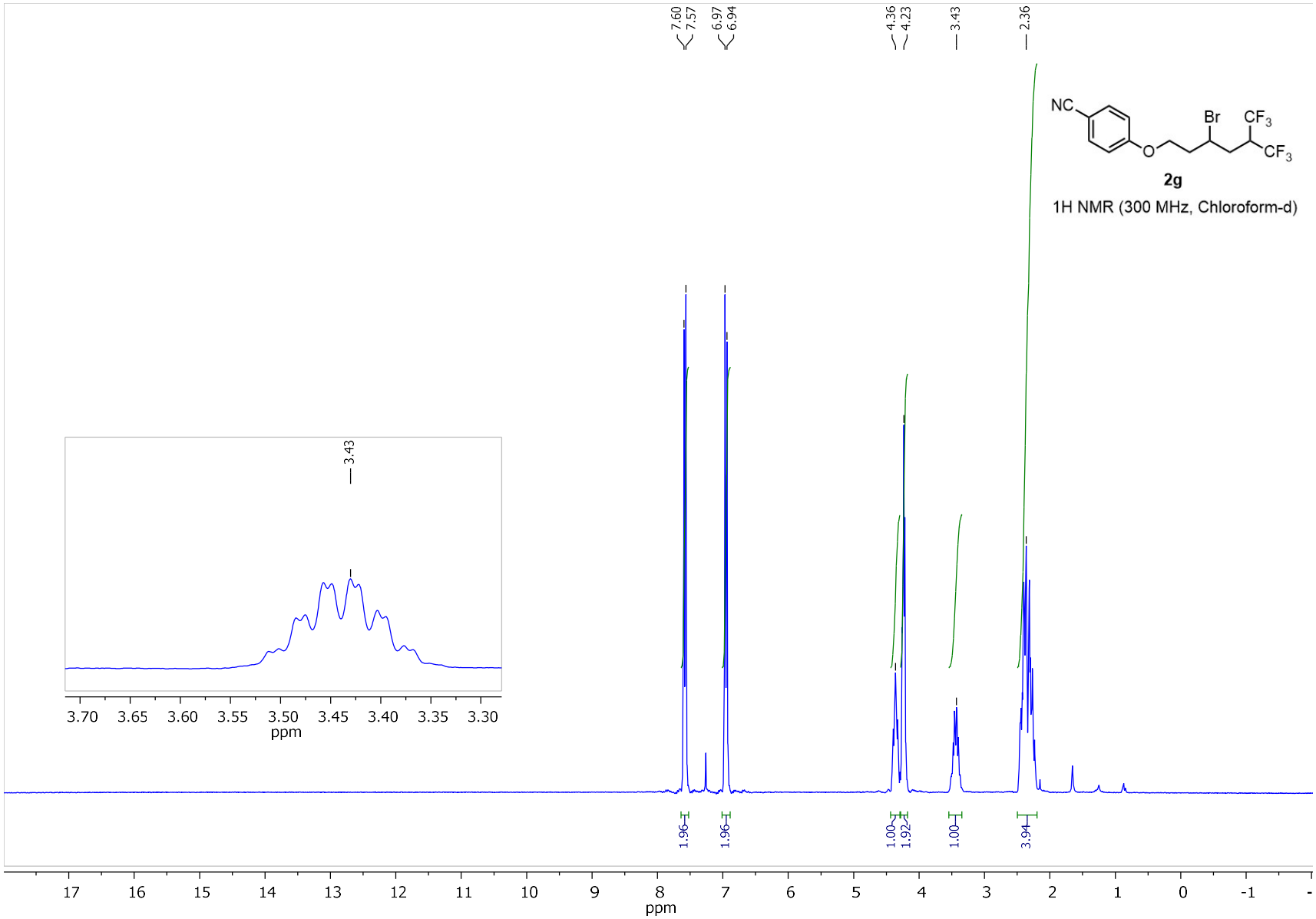

154.88
152.06
129.46
129.23
125.77
125.74
125.52
122.05
122.02
118.31
118.05
116.21
114.97
72.88
55.82
47.95
47.58
47.43
47.40
47.20
46.83
46.45
46.08
45.71
30.44
Br
CF3
CF3
O
O
2e
13C NMR (75 MHz, Chloroform-d)
ppm

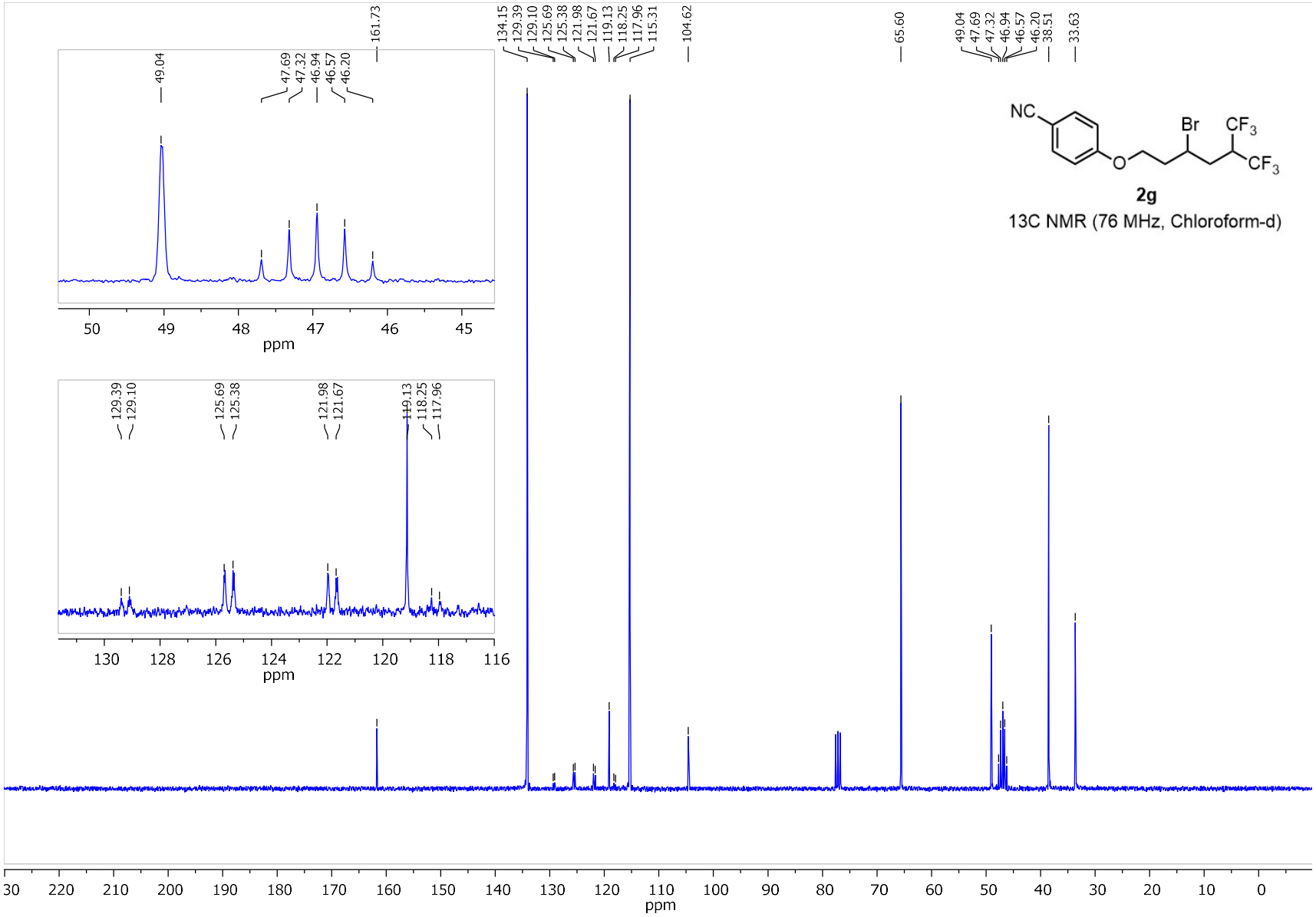

-67.23
-67.26
-67.29
-67.33
-67.36
-68.45
-68.48
-68.51
-68.55
-68.58
Br
CF3
CF3
O
O
2e
19F NMR (282 MHz, Chloroform-d)
1.00
1.01
ppm
-67.0
-67.5
-68.0
-68.5
-69.0
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280

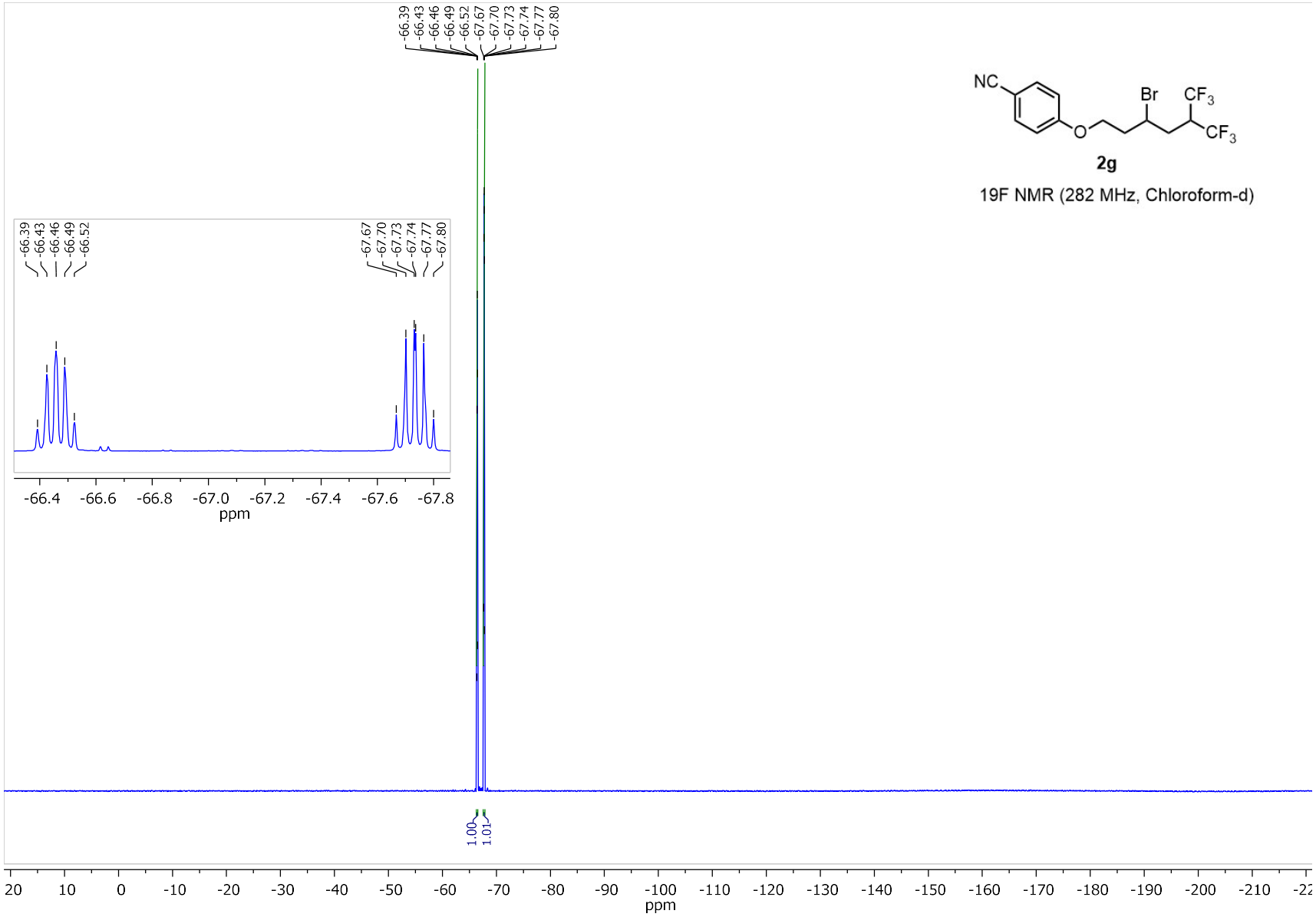

4.70
4.09
3.69
3.56
3.39
2.24
1.91
1.64
Br
CF3
MEMO
CF3
2f
1H NMR (300 MHz, Chloroform-d)
2.11
1.00
2.15
4.02
3.55
2.13
2.04
4.23
ppm

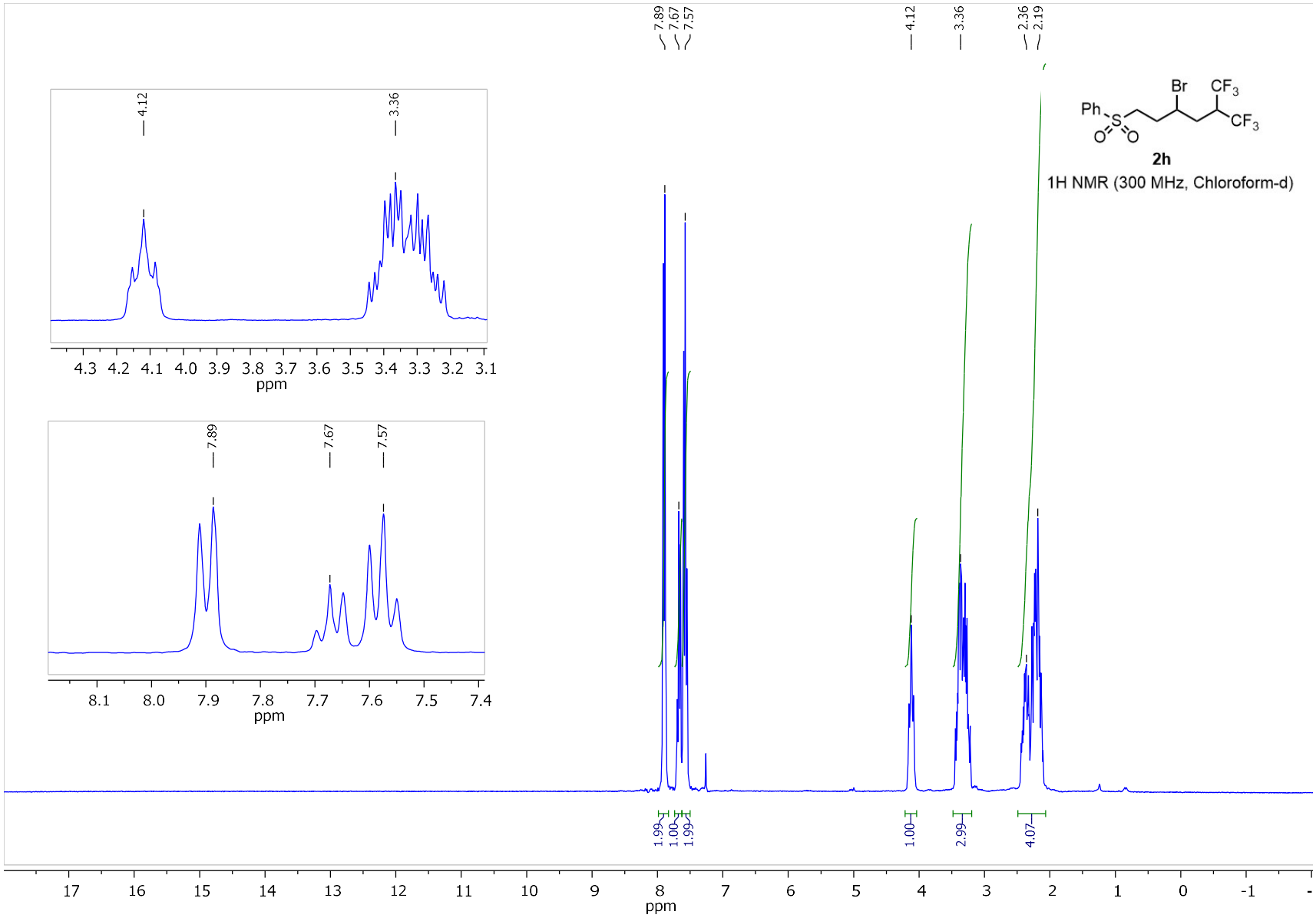

129.52
129.14
125.80
125.47
125.43
122.08
121.76
121.72
118.34
118.01
95.58
71.92
67.44
66.88
66.82
59.06
53.08
53.05
53.03
53.00
48.16
47.79
47.42
47.05
46.68
46.30
45.93
39.22
33.52
28.98
24.35
Br
CF3
MEMO
CF3
2f
13C NMR (75 MHz, Chloroform-d)
ppm

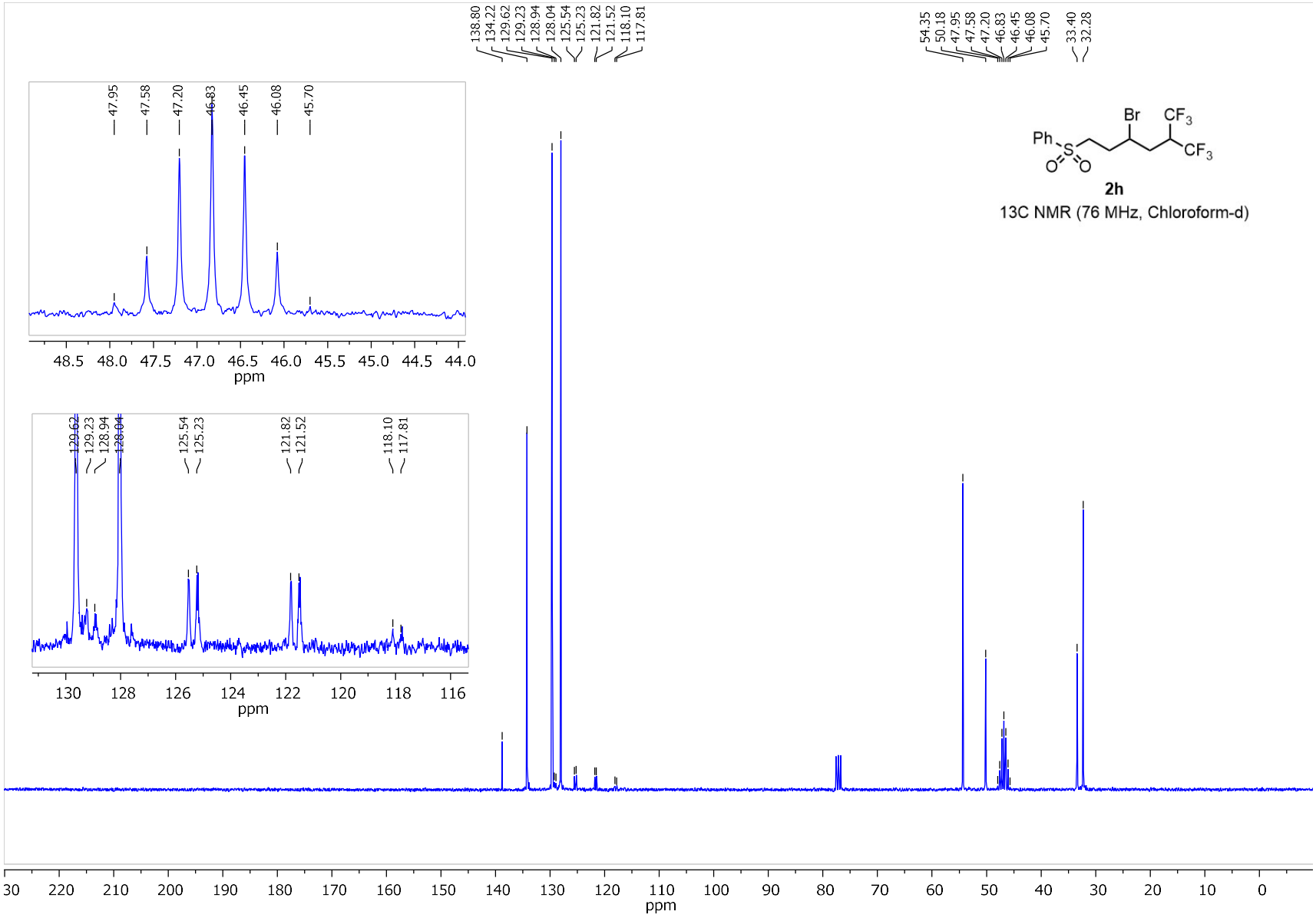

-67.26
-67.30
-67.33
-67.37
-67.40
-68.60
-68.64
-68.67
-68.71
-68.74
Br
CF3
MEMO
CF3
2f
19F NMR (282 MHz, Chloroform-d)
-67.0
-67.4
-67.8
-68.2
-68.6
-69.0
ppm
1.00
1.03
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm

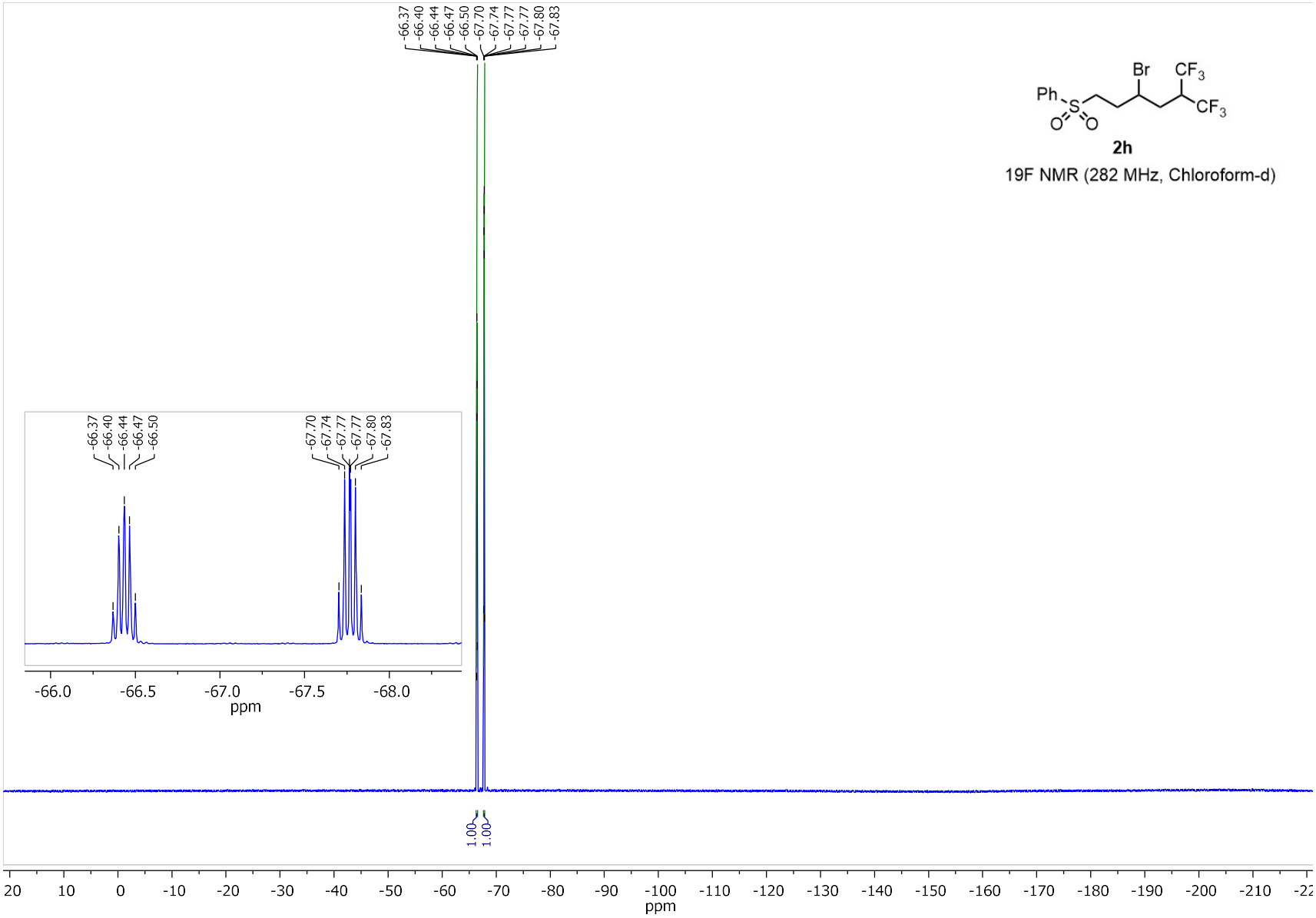

7.60
7.57
6.97
6.94
4.36
4.23
3.43
2.36
NC
Br
CF3
CF3
O
2g
1H NMR (300 MHz, Chloroform-d)
3.43
3.70 3.65 3.60 3.55 3.50 3.45 3.40 3.35 3.30
ppm
1.96
1.96
1.00
1.92
1.00
3.94
17 16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
ppm

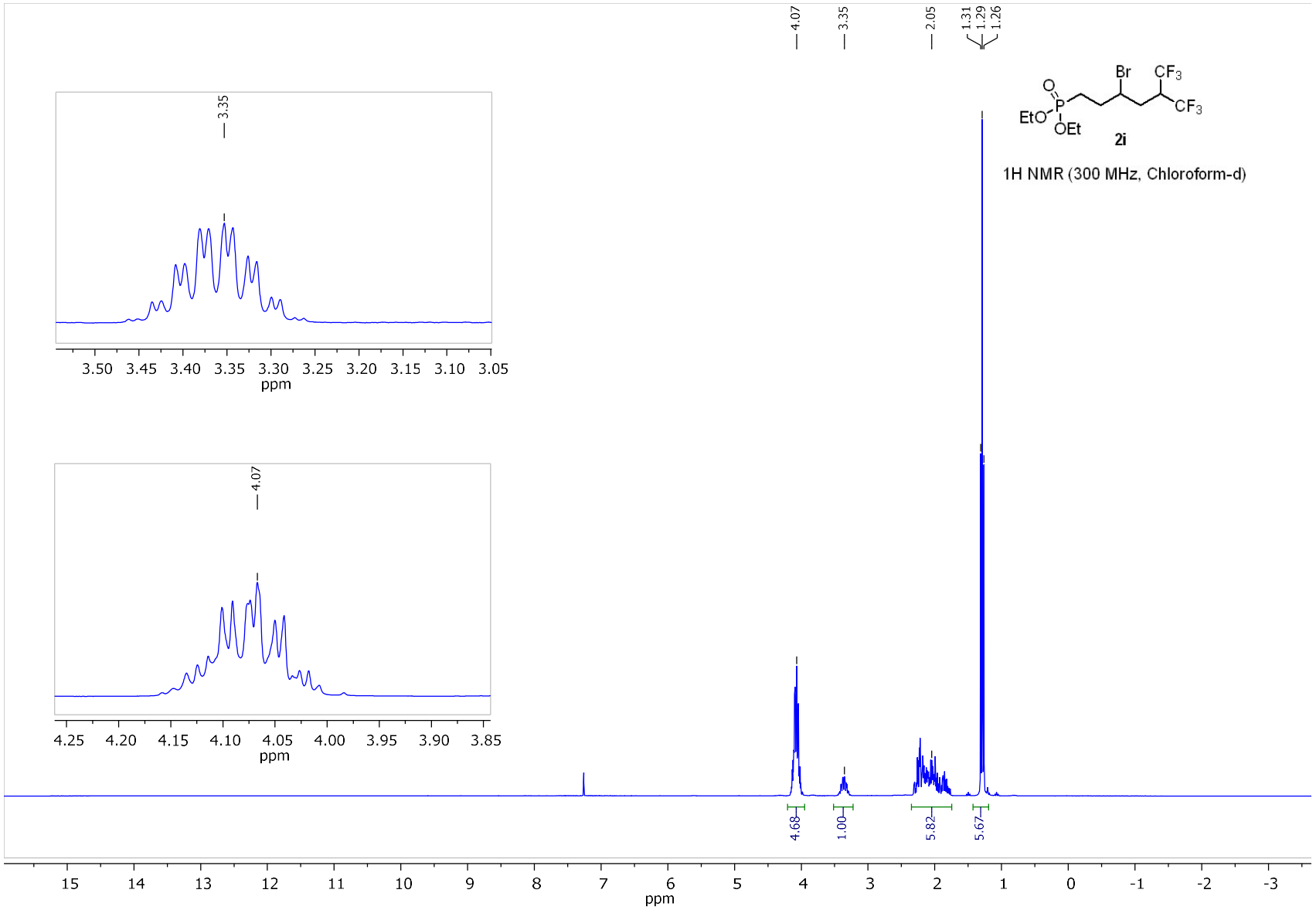

161.73
134.15
129.39
129.10
125.69
125.38
121.98
121.67
119.13
118.25
117.96
115.31
104.62
65.60
49.04
47.69
47.32
46.94
46.57
46.20
38.51
33.63
NC
Br
CF3
CF3
O
2g
13C NMR (76 MHz, Chloroform-d)
ppm

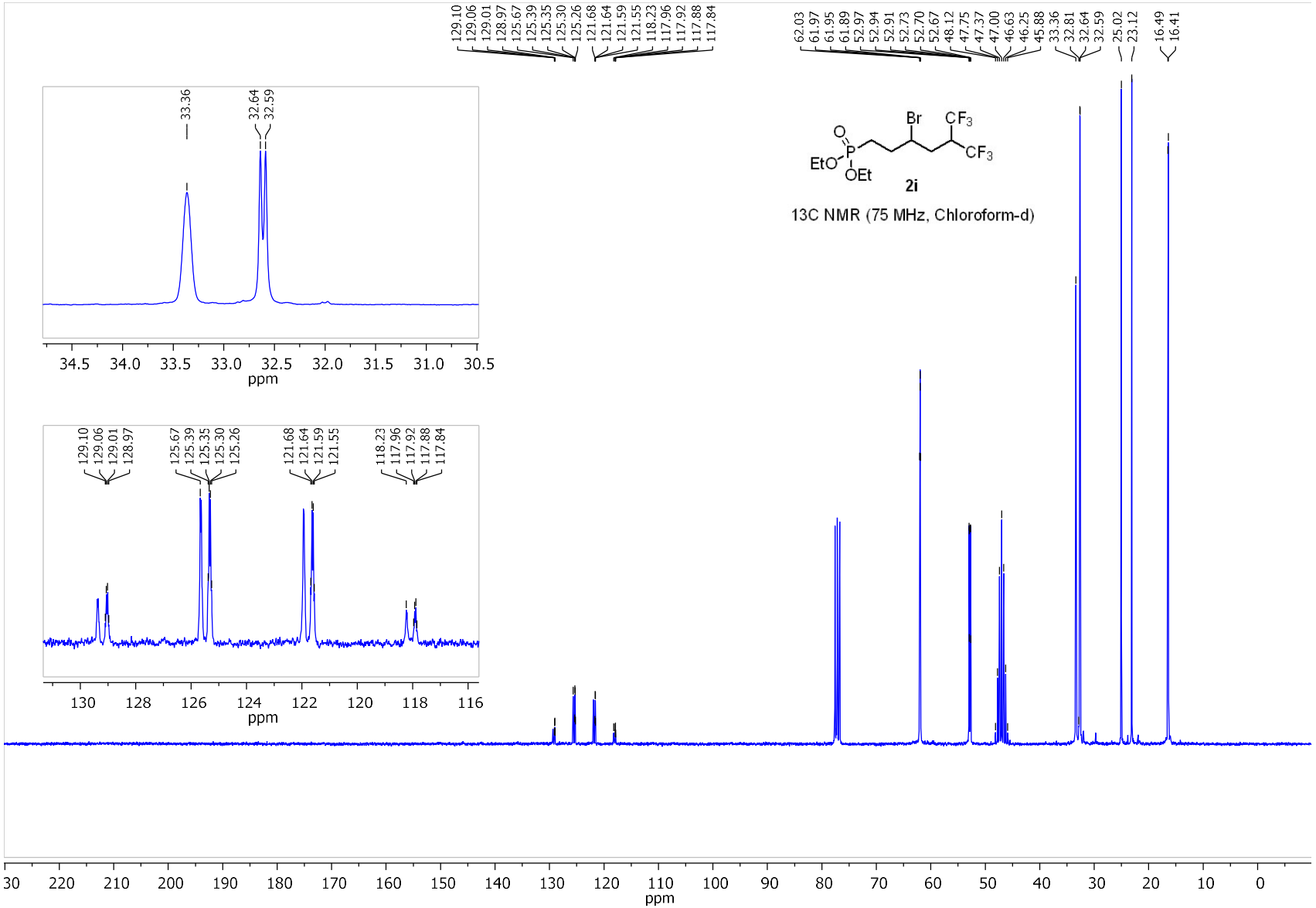

-66.39
-66.43
-66.46
-66.49
-66.52
-67.67
-67.70
-67.73
-67.74
-67.77
-67.80
NC
Br
CF3
CF3
O
2g
19F NMR (282 MHz, Chloroform-d)
-66.4
-66.6
-66.8
-67.0
-67.2
-67.4
-67.6
-67.8
ppm
1.00
1.01
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
ppm

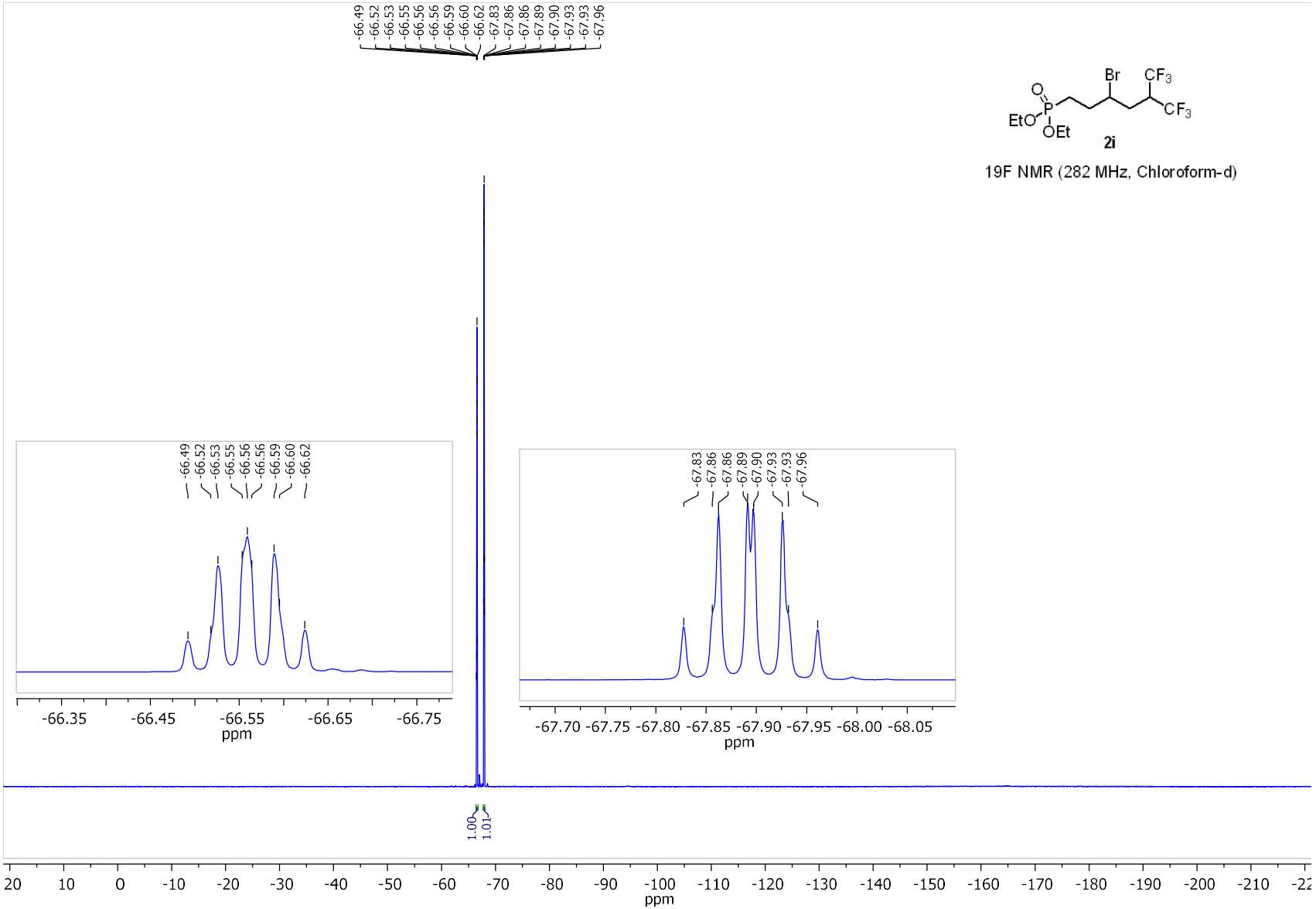

7.89
7.67
7.57
4.12
3.36
2.36
2.19
Br
CF3
Ph
S
O
O
CF3
2h
1H NMR (300 MHz, Chloroform-d)
4.12
3.36
4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6 3.5 3.4 3.3 3.2 3.1
ppm
7.89
7.67
7.57
8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4
ppm
1.99
1.00
1.99
1.00
2.99
4.07
17 16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
ppm

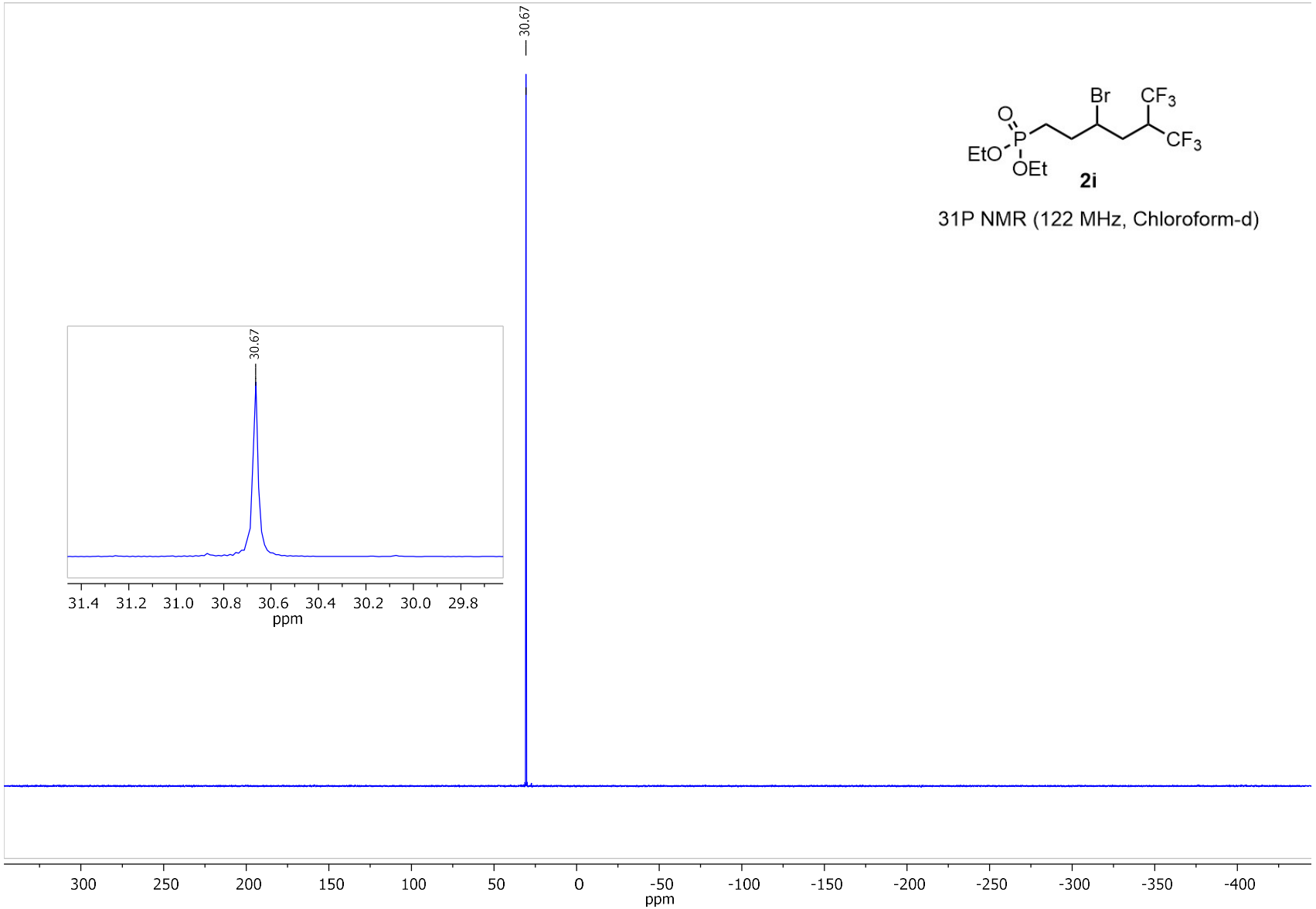

138.80
134.22
129.62
129.23
128.94
128.04
125.54
125.23
121.82
121.52
118.10
117.81
54.35
50.18
47.95
47.58
47.20
46.83
46.45
46.08
45.70
33.40
32.28
Br
CF3
Ph
S
O
O
CF3
2h
13C NMR (76 MHz, Chloroform-d)
ppm
ppm

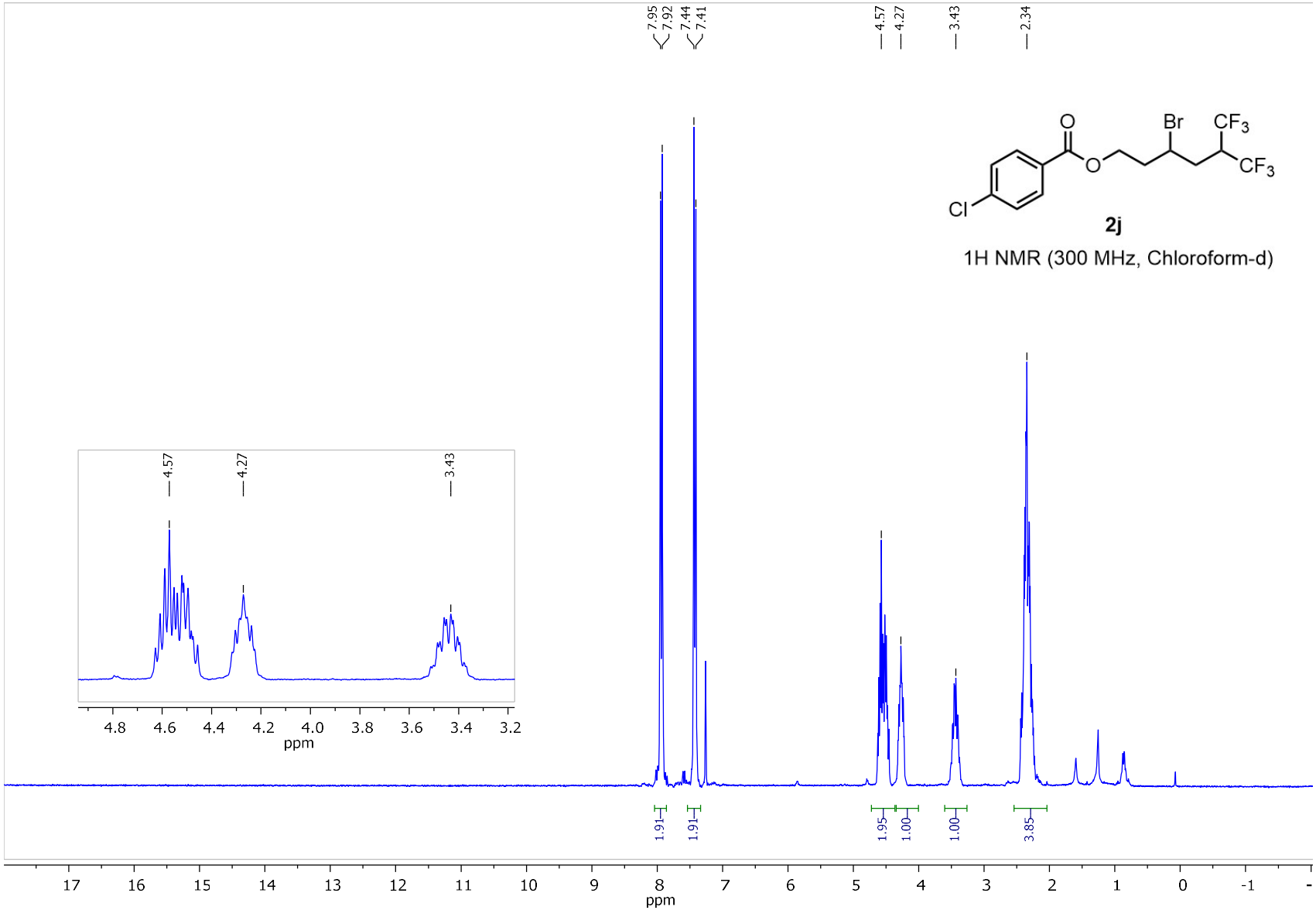

-66.37
-66.40
-66.44
-66.47
-66.50
-67.70
-67.74
-67.77
-67.77
-67.80
-67.83
Br
CF3
Ph
S
O
O
CF3
2h
19F NMR (282 MHz, Chloroform-d)
-66.0
-66.5
-67.0
-67.5
-68.0
ppm
1.00
1.00
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
ppm

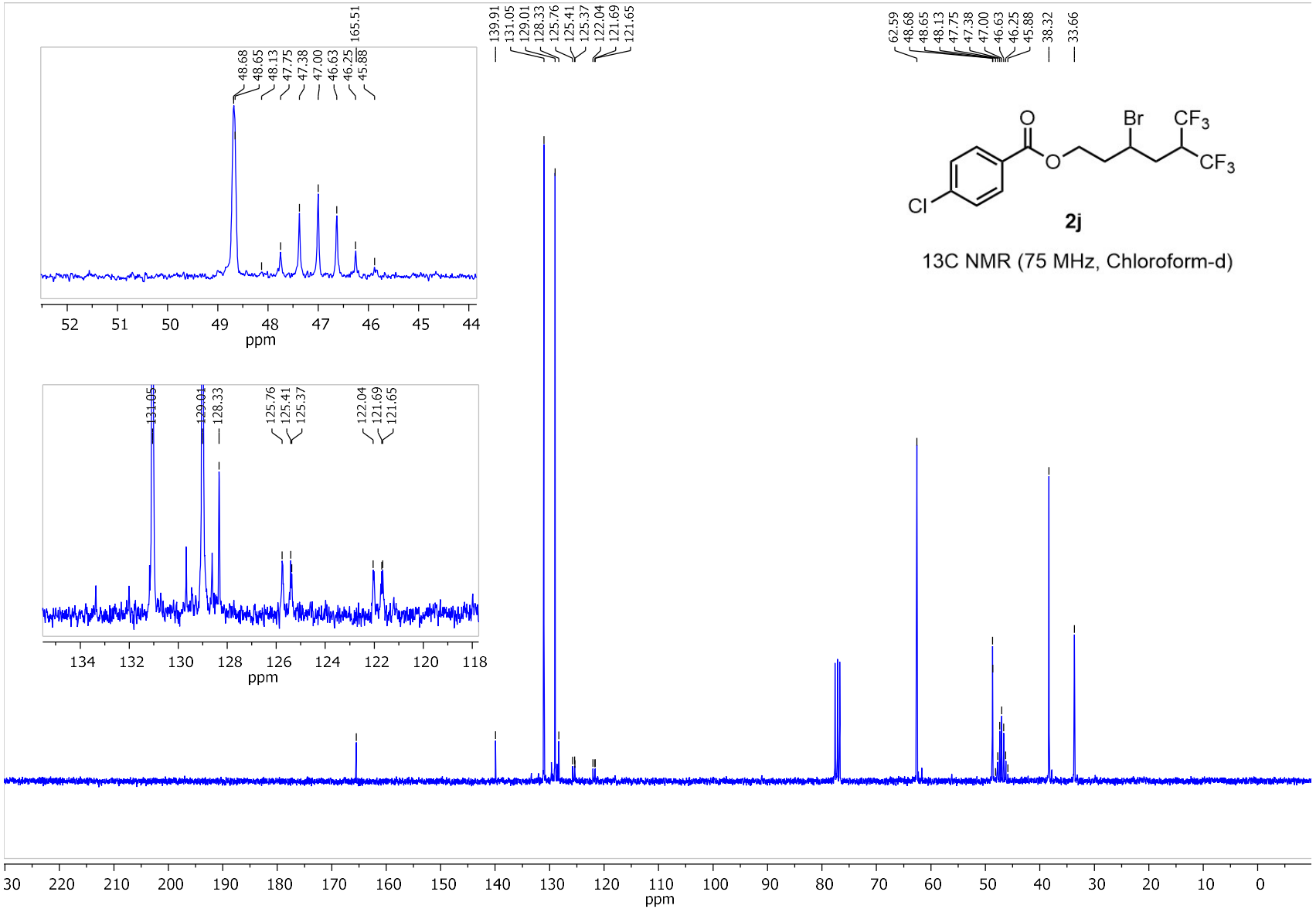

4.07
3.35
2.05
1.31
1.29
1.26
Br
CF3
CF3
EtO
P
O
OEt
2i
1H NMR (300 MHz, Chloroform-d)
3.35
3.50 3.45 3.40 3.35 3.30 3.25 3.20 3.15 3.10 3.05
ppm
4.07
4.25 4.20 4.15 4.10 4.05 4.00 3.95 3.90 3.85
ppm
4.68
1.00
5.82
5.67
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
ppm


129.10
129.06
129.01
128.97
125.67
125.39
125.35
125.30
125.26
121.68
121.64
121.59
121.55
118.23
117.96
117.92
117.88
117.84
62.03
61.97
61.95
61.89
52.97
52.94
52.91
52.73
52.70
52.67
48.12
47.75
47.37
47.00
46.63
46.25
45.88
33.36
32.81
32.64
32.59
25.02
23.12
16.49
16.41
Br
CF3
CF3
O
P
EtO
OEt
2i
13C NMR (75 MHz, Chloroform-d)
ppm


-66.49
-66.52
-66.53
-66.55
-66.56
-66.56
-66.59
-66.60
-66.62
-67.83
-67.86
-67.86
-67.89
-67.90
-67.93
-67.93
-67.96
Br
CF3
O
P
EtO
OEt
CF3
2i
19F NMR (282 MHz, Chloroform-d)
-66.35
-66.45
-66.55
-66.65
-66.75
ppm
-67.70 -67.75 -67.80 -67.85 -67.90 -67.95 -68.00 -68.05
ppm
1.00
1.01
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
ppm


30.67
Br
CF3
O
P
EtO
OEt
CF3
2i
31P NMR (122 MHz, Chloroform-d)
30.67
31.4 31.2 31.0 30.8 30.6 30.4 30.2 30.0 29.8
ppm
300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400
ppm


7.95
7.92
7.44
7.41
4.57
4.27
3.43
2.34
O
Br
CF3
O
CF3
Cl
2j
1H NMR (300 MHz, Chloroform-d)
4.57
4.27
3.43
4.8
4.6
4.4
4.2
4.0
3.8
3.6
3.4
3.2
ppm
1.91
1.91
1.95
1.00
1.00
3.85
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm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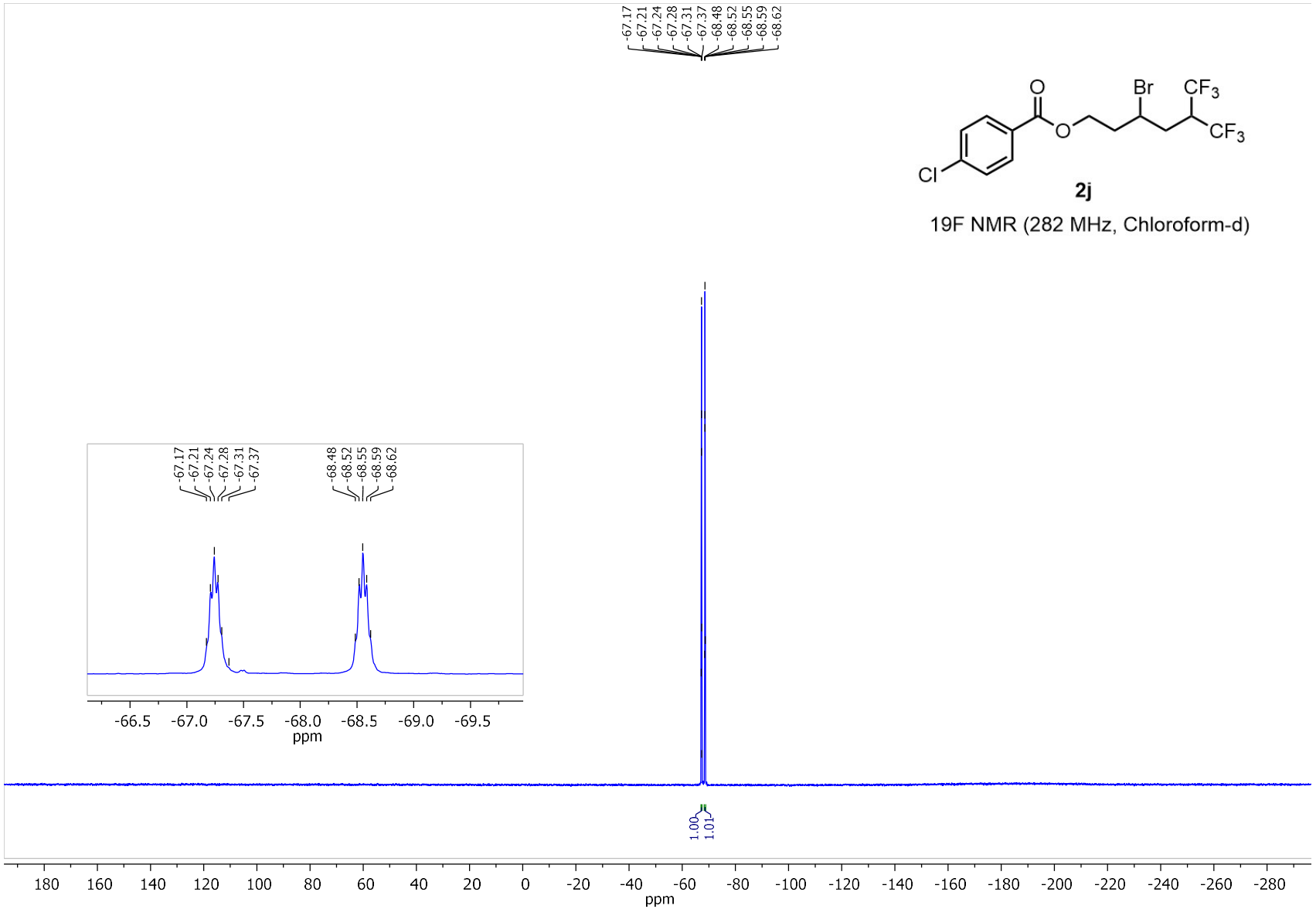
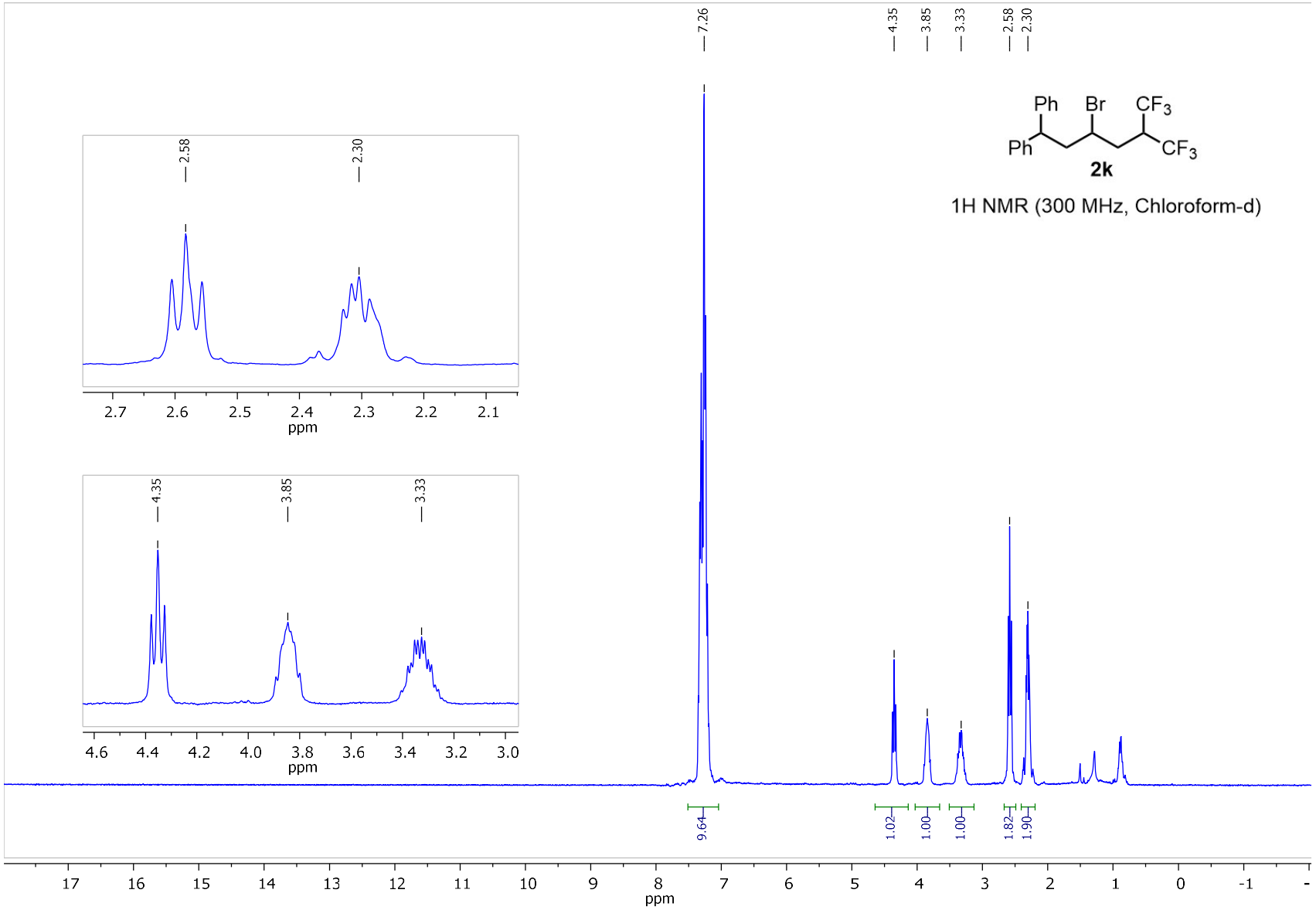
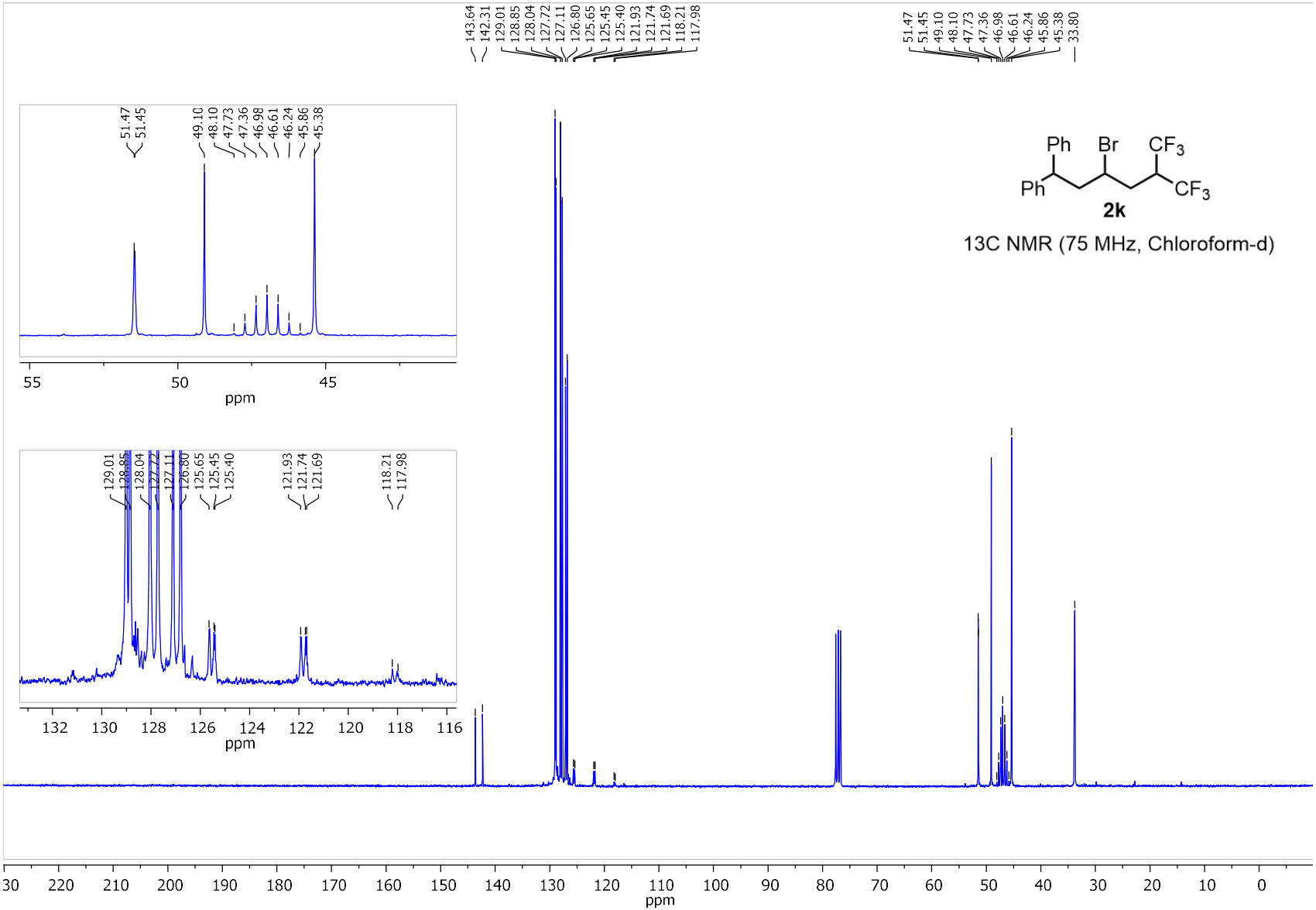
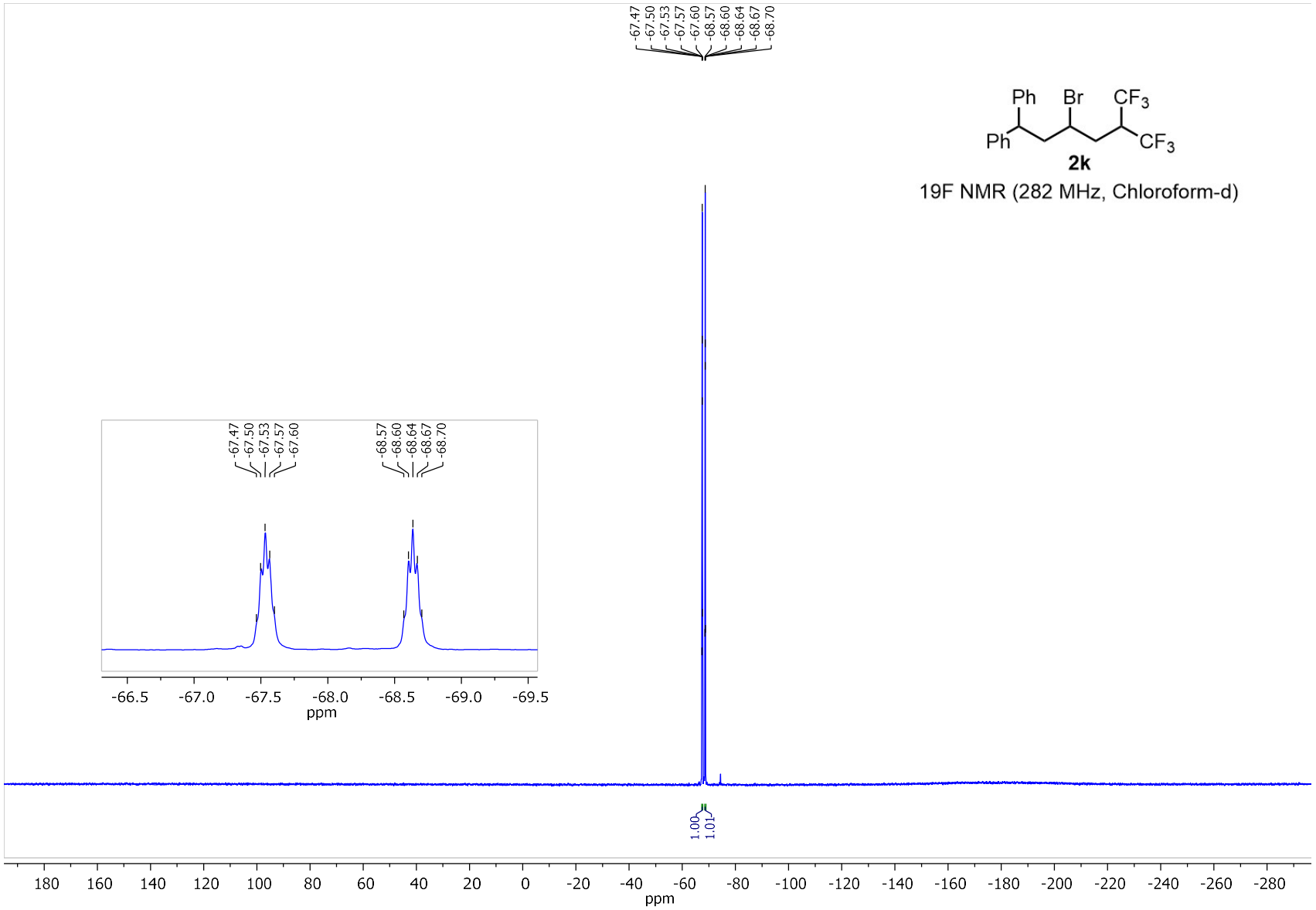
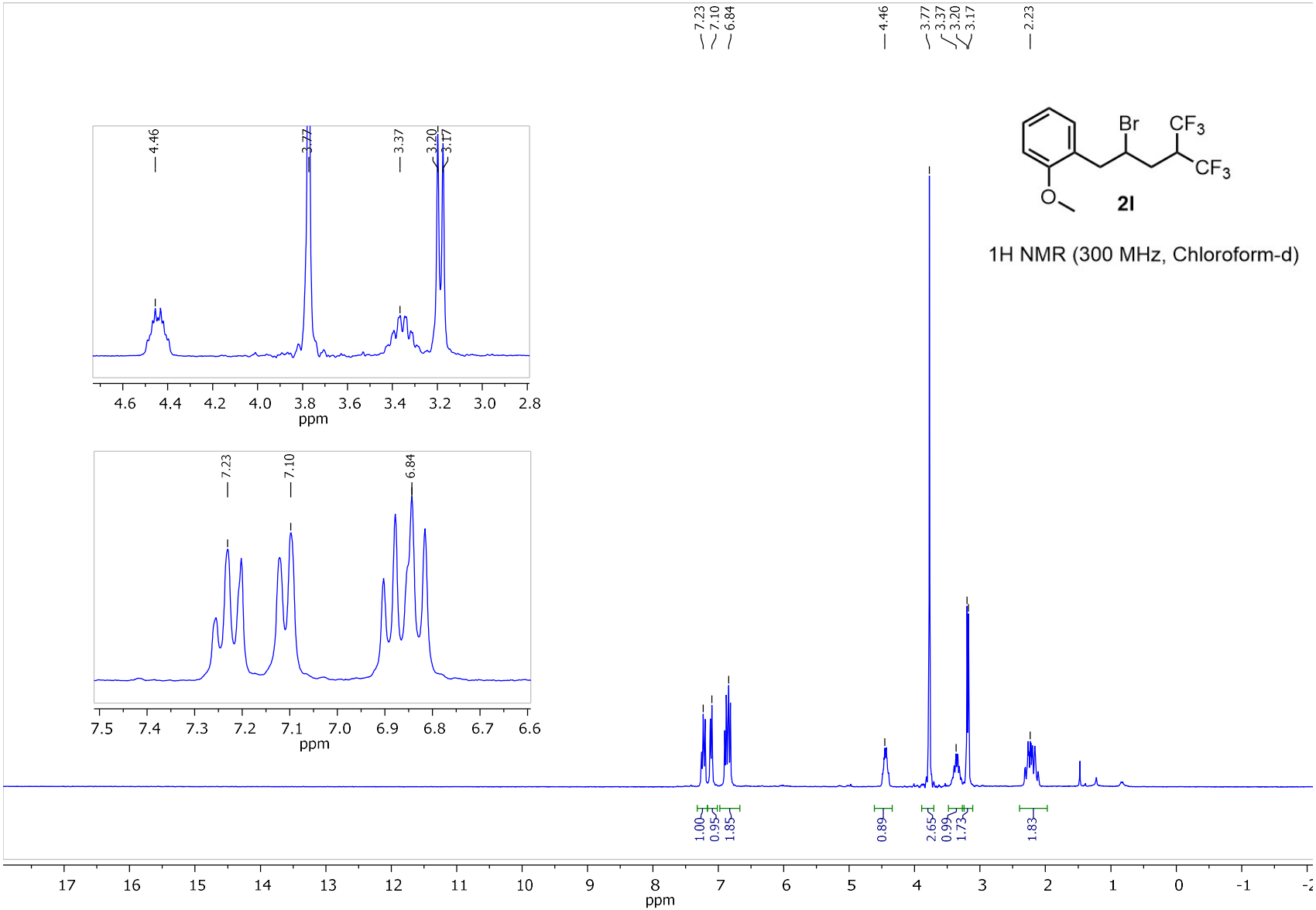
139.91
131.05
129.01
128.33
125.76
125.41
125.37
122.04
121.69
121.65
62.59
48.68
48.65
48.13
47.75
47.38
47.00
46.63
46.25
45.88
38.32
33.66
48.68
48.65
48.13
47.75
47.38
47.00
46.63
46.25
45.88
52 51 50 49 48 47 46 45 44
ppm
131.05
129.01
128.33
125.76
125.41
125.37
122.04
121.69
121.65
134 132 130 128 126 124 122 120 118
ppm
O
Br
CF3
CF3
O
Cl
2j
13C NMR (75 MHz, Chloroform-d)
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

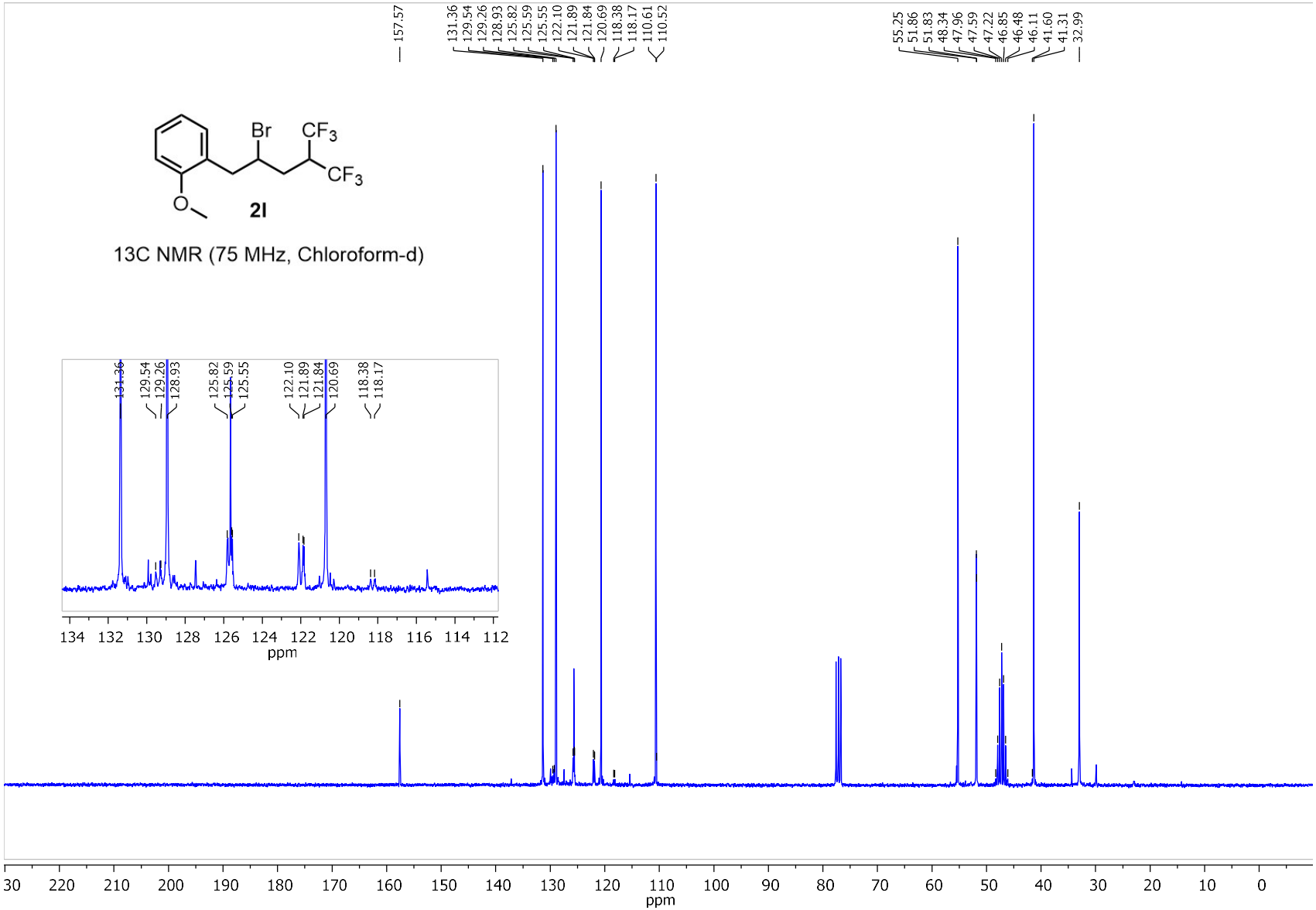

-67.17
-67.21
-67.24
-67.28
-67.31
-67.37
-68.48
-68.52
-68.55
-68.59
-68.62
O
Br
CF3
O
CF3
Cl
2j
19F NMR (282 MHz, Chloroform-d)
1.00
1.01
ppm

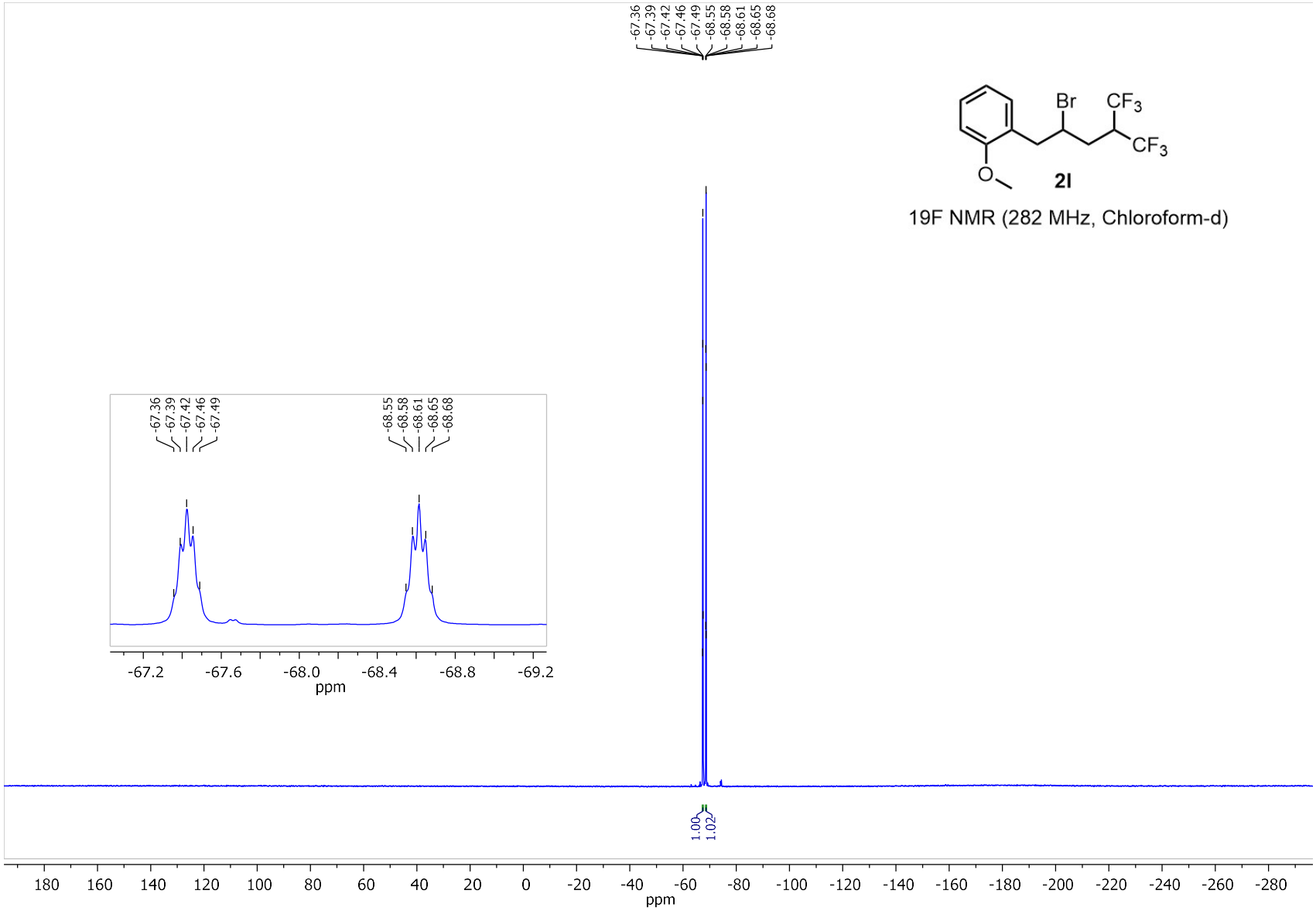

7.26
4.35
3.85
3.33
2.58
2.30
Ph
Br
CF3
Ph
CF3
2k
1H NMR (300 MHz, Chloroform-d)
ppm
9.64
1.02
1.00
1.00
1.82
1.90

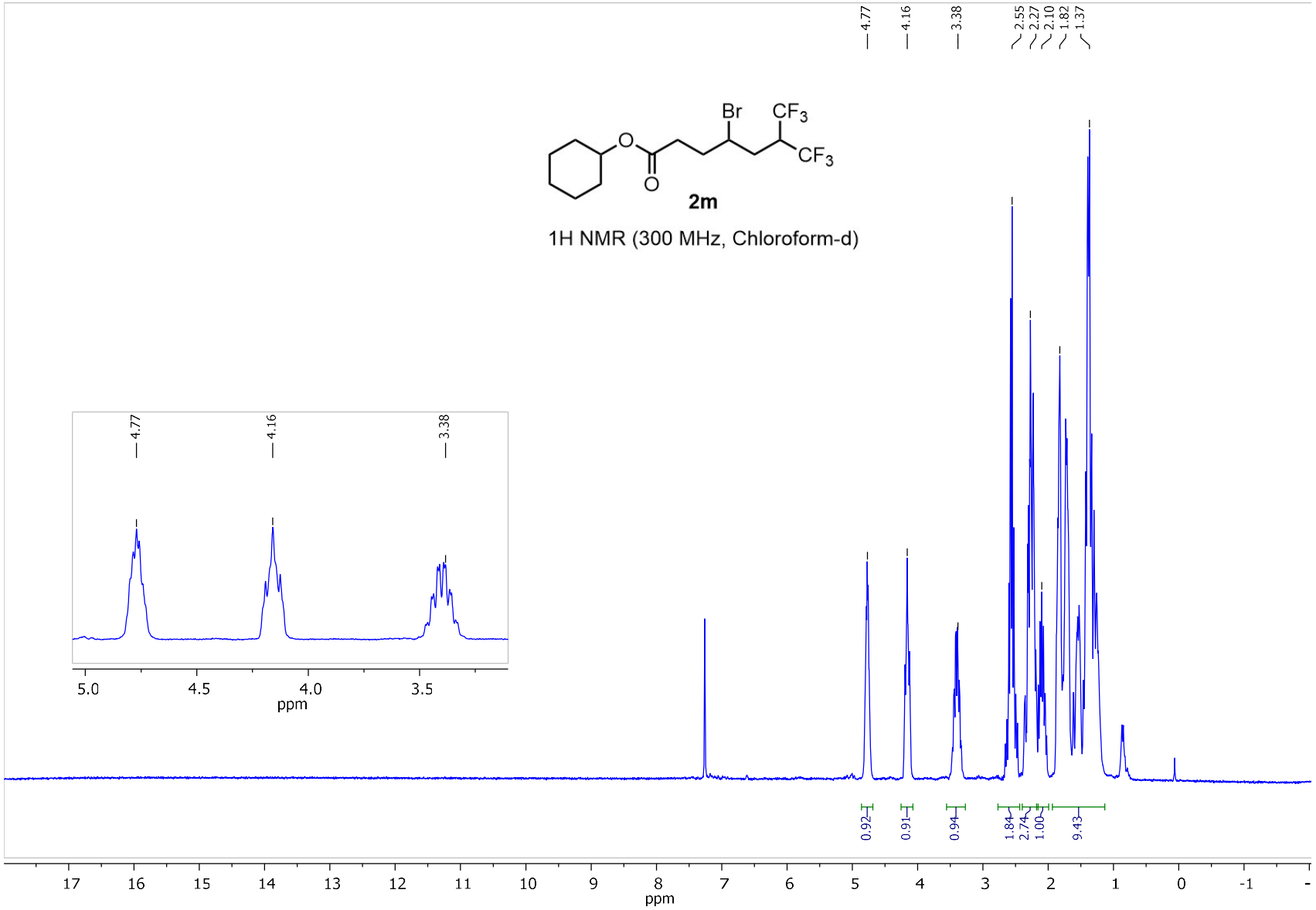

143.64
142.31
129.01
128.85
128.04
127.72
127.11
126.80
125.65
125.45
125.40
121.93
121.74
121.69
118.21
117.98
51.47
51.45
49.10
48.10
47.73
47.36
46.98
46.61
46.24
45.86
45.38
33.80
Ph
Br
CF3
Ph
CF3
2k
13C NMR (75 MHz, Chloroform-d)
ppm

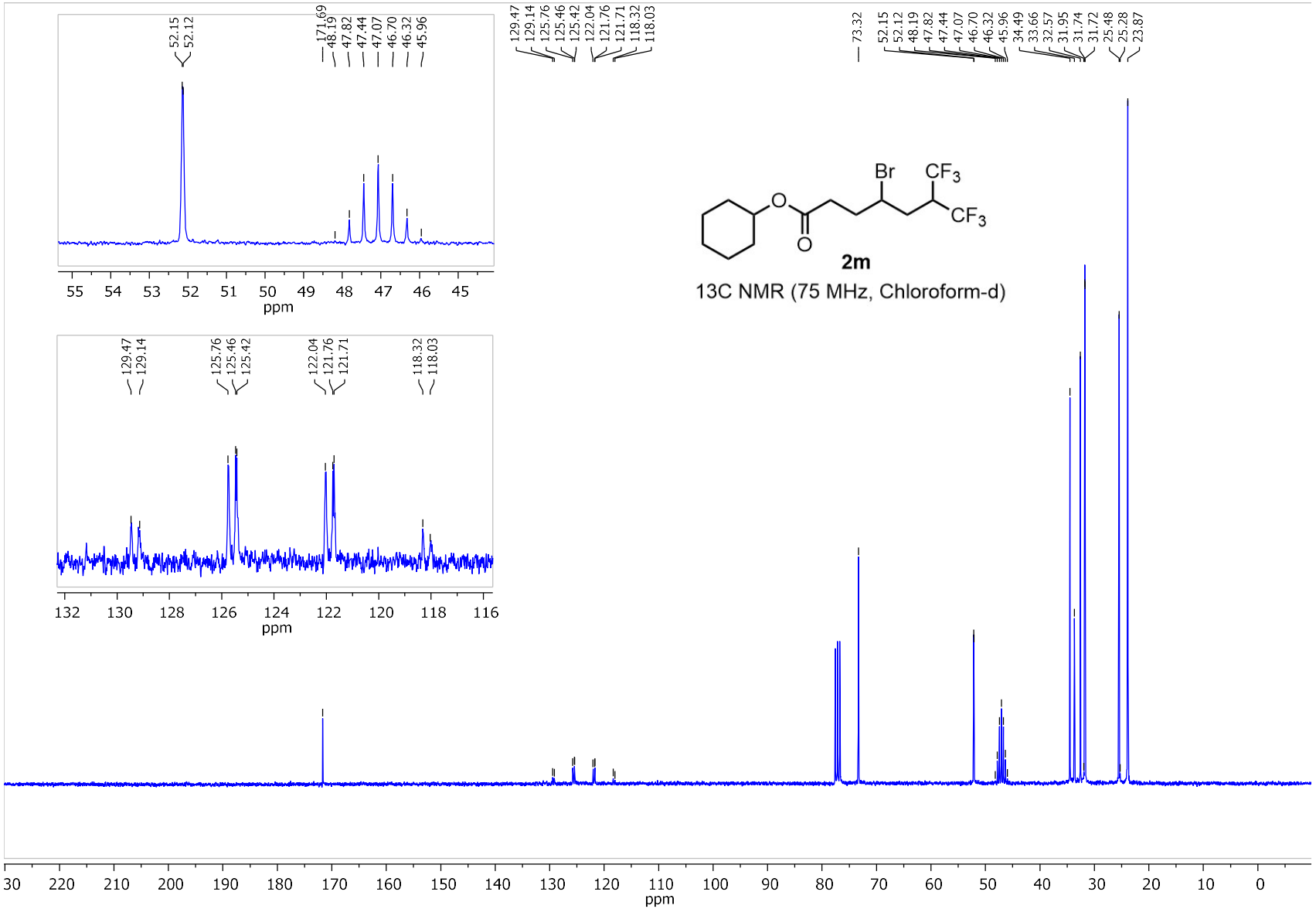

-67.47
-67.50
-67.53
-67.57
-67.60
-68.57
-68.60
-68.64
-68.67
-68.70
Ph
Br
CF3
Ph
CF3
2k
19F NMR (282 MHz, Chloroform-d)
-66.5
-67.0
-67.5
-68.0
-68.5
-69.0
-69.5
ppm
1.00
1.01
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm

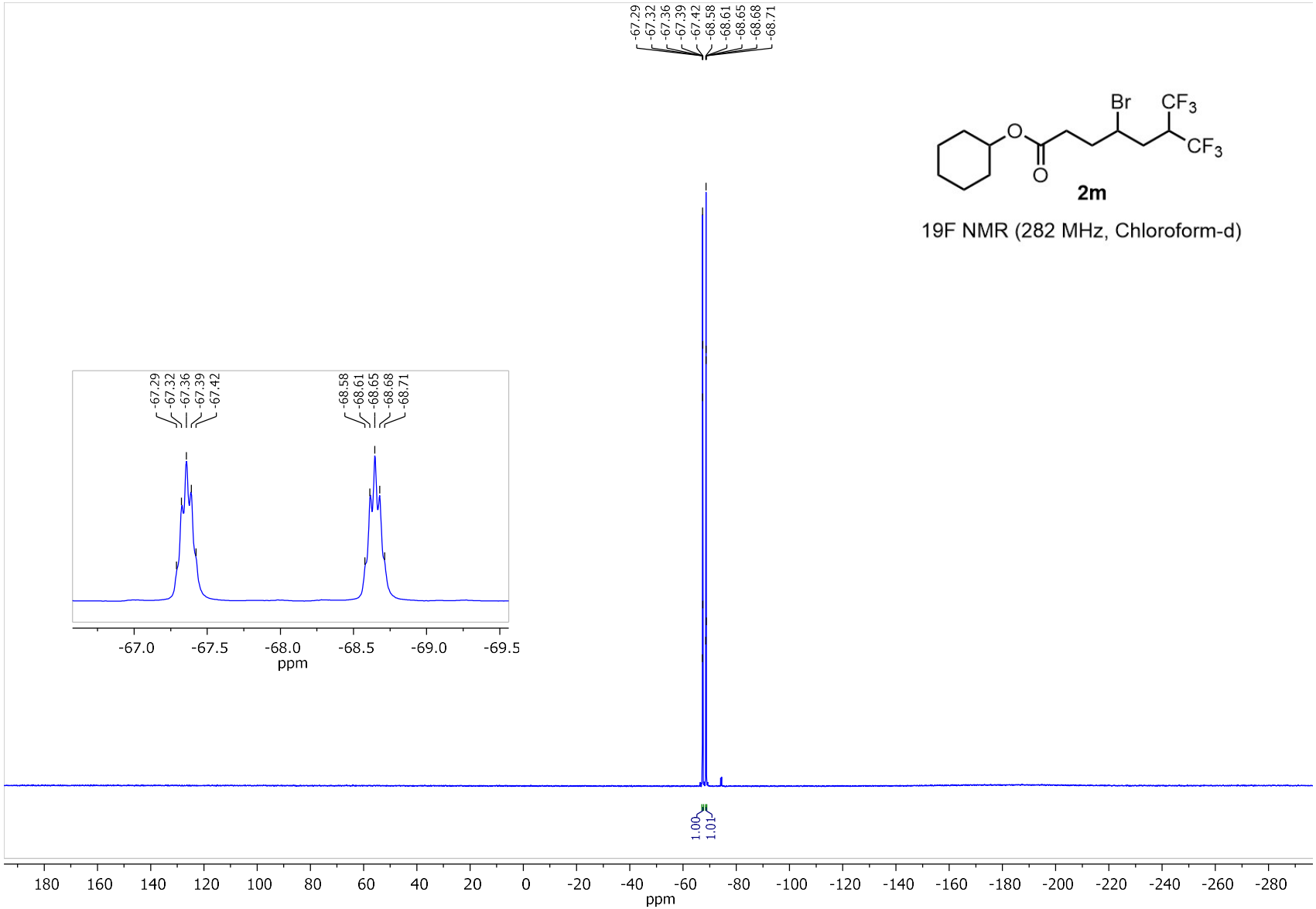

7.23
7.10
6.84
4.46
3.77
3.37
3.20
3.17
2.23
Br
CF3
CF3
O
2l
1H NMR (300 MHz, Chloroform-d)
ppm
1.00
0.95
1.85
0.89
2.65
0.99
1.73
1.83

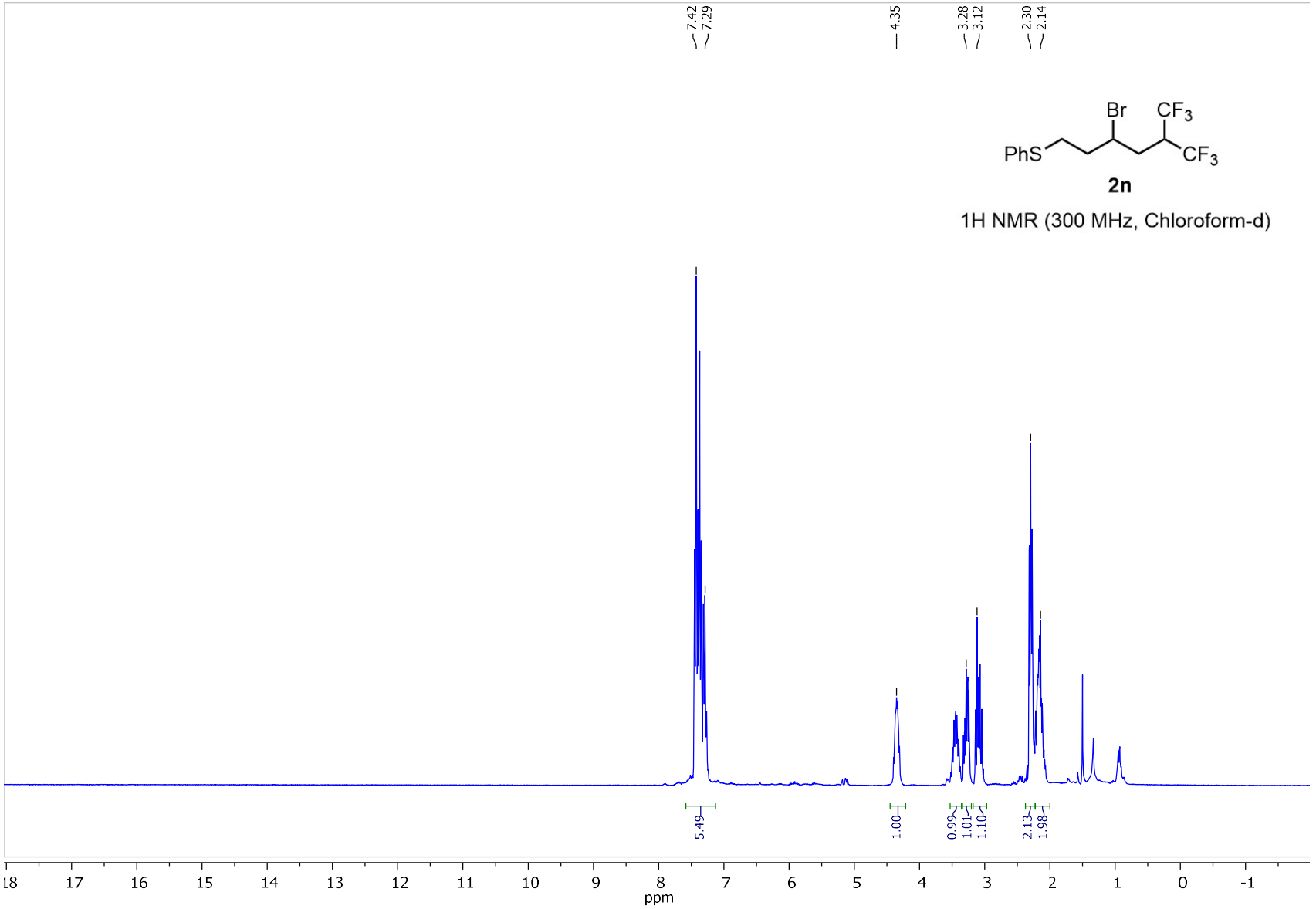

157.57
131.36
129.54
129.26
128.93
125.82
125.59
125.55
122.10
121.89
121.84
120.69
118.38
118.17
110.61
110.52
55.25
51.86
51.83
48.34
47.96
47.59
47.22
46.85
46.48
46.11
41.60
41.31
32.99
Br
CF3
CF3
O
2l
13C NMR (75 MHz, Chloroform-d)
ppm
ppm

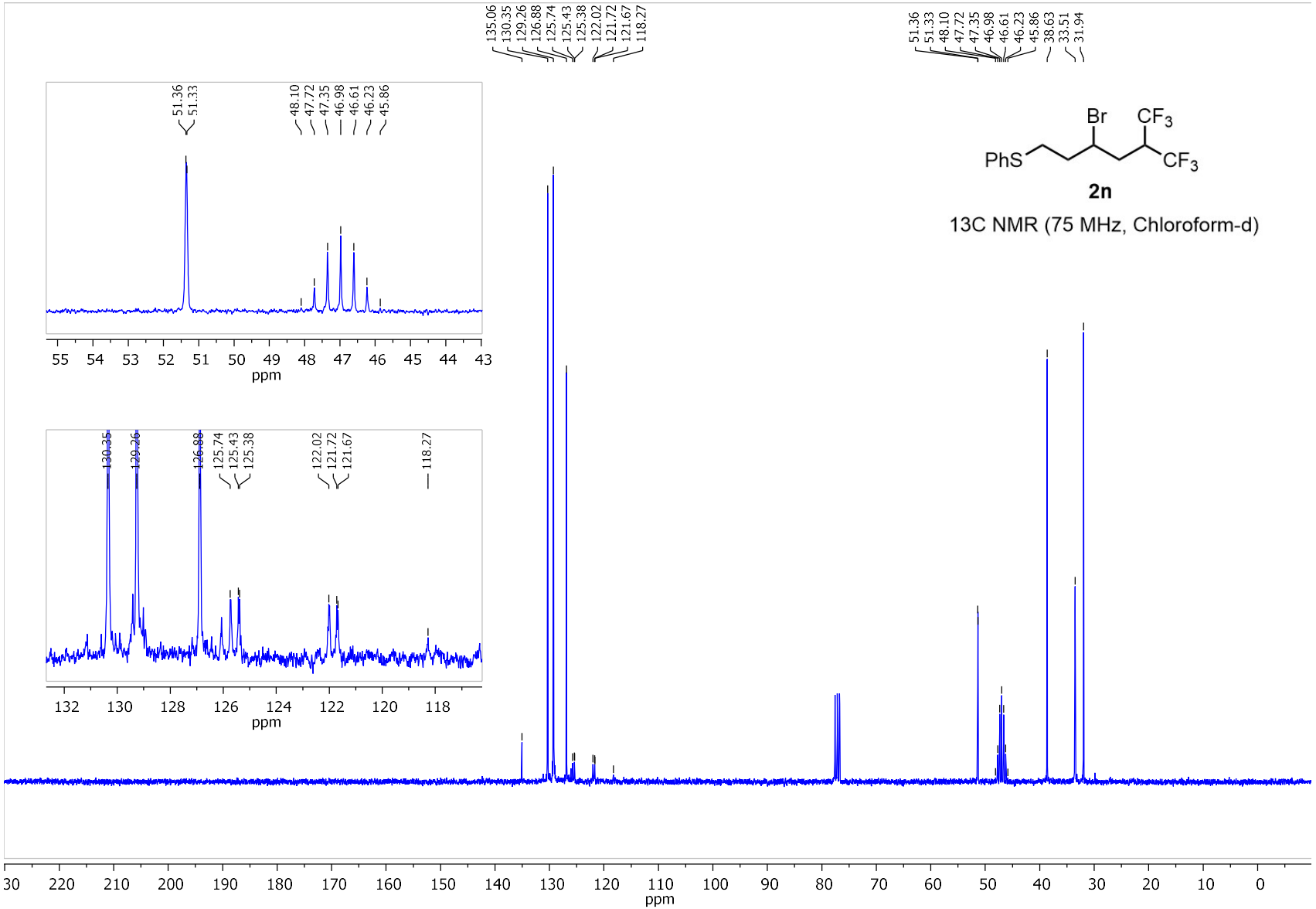

-67.36
-67.39
-67.42
-67.46
-67.49
-68.55
-68.58
-68.61
-68.65
-68.68
Br
CF3
CF3
O
2l
19F NMR (282 MHz, Chloroform-d)
-67.2
-67.6
-68.0
-68.4
-68.8
-69.2
ppm
1.00
1.02
180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280
ppm

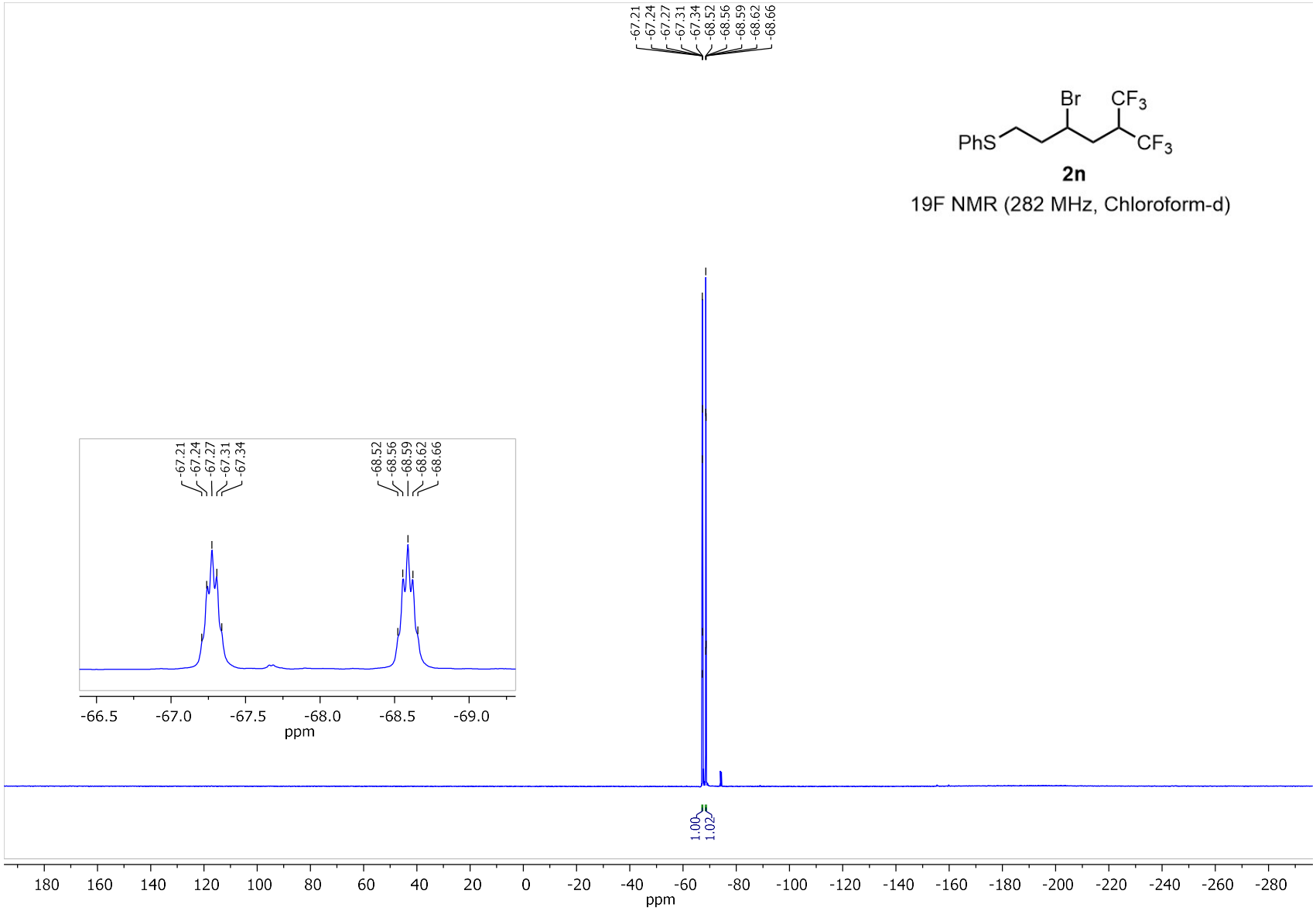

4.77
4.16
3.38
2.55
2.27
2.10
1.82
1.37
Br
CF3
CF3
O
O
2m
1H NMR (300 MHz, Chloroform-d)
0.92
0.91
0.94
1.84
2.74
1.00
9.43
ppm

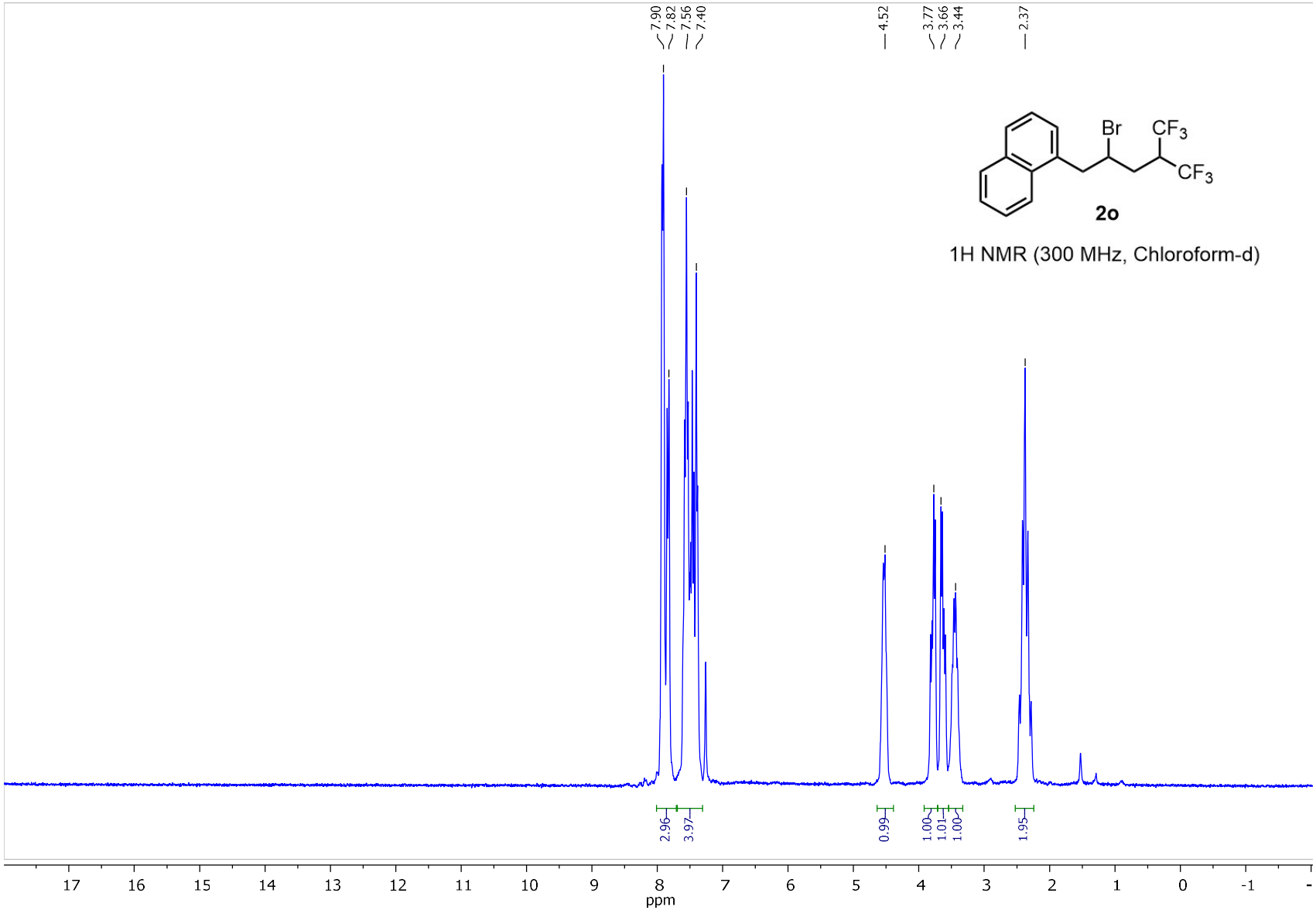

52.15
52.12
171.69
48.19
47.82
47.44
47.07
46.70
46.32
45.96
55 54 53 52 51 50 49 48 47 46 45
ppm
129.47
129.14
125.76
125.46
125.42
122.04
121.76
121.71
118.32
118.03
132 130 128 126 124 122 120 118 116
ppm
73.32
52.15
52.12
48.19
47.82
47.44
47.07
46.70
46.32
45.96
34.49
33.66
32.57
31.95
31.74
31.72
25.48
25.28
23.87
Br
CF3
CF3
O
O
2m
13C NMR (75 MHz, Chloroform-d)
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

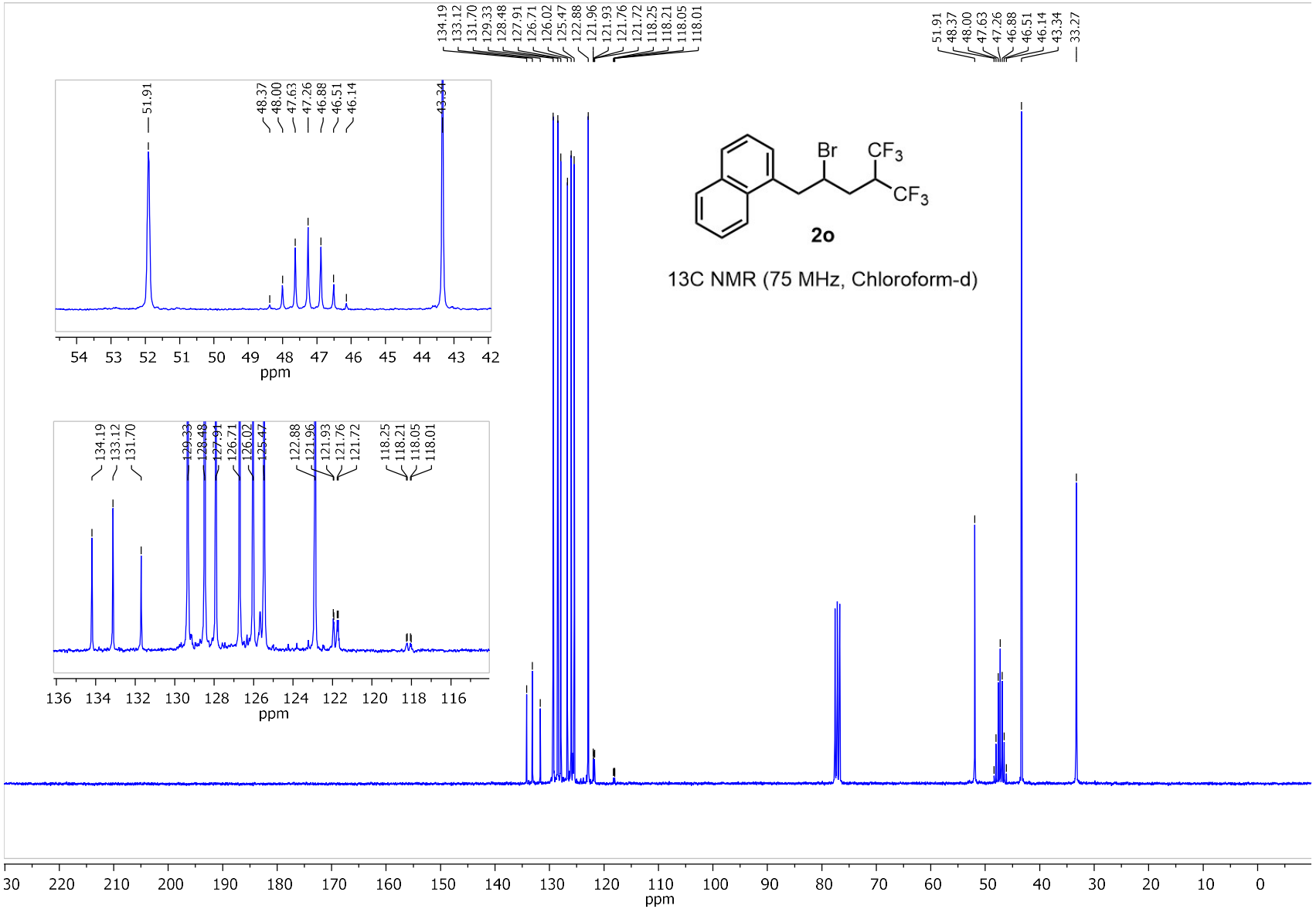

-67.29
-67.32
-67.36
-67.39
-67.42
-68.58
-68.61
-68.65
-68.68
-68.71
Br
CF3
CF3
O
O
2m
19F NMR (282 MHz, Chloroform-d)
-67.0
-67.5
-68.0
-68.5
-69.0
-69.5
ppm
1.00
1.01
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm

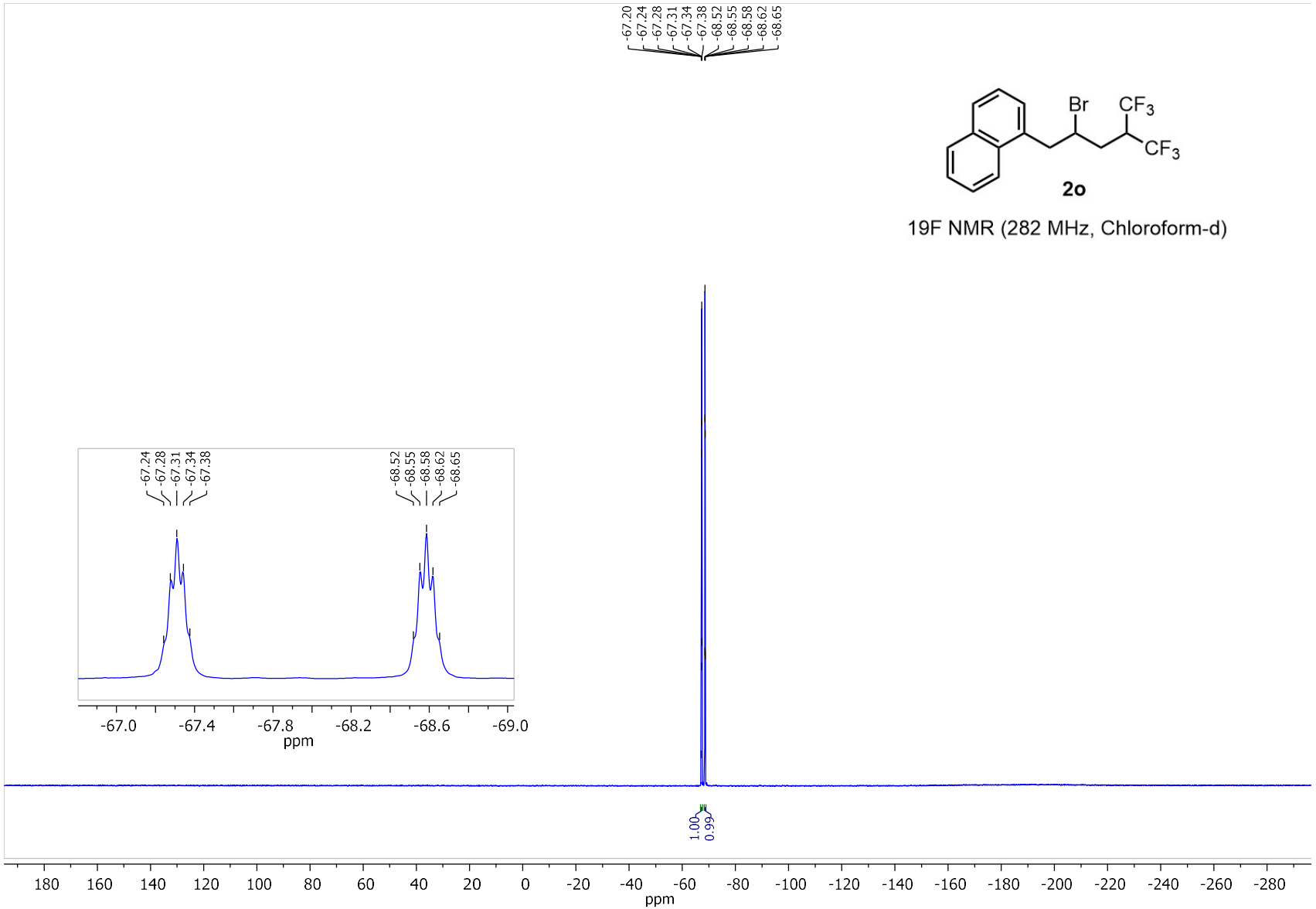

7.42
7.29
4.35
3.28
3.12
2.30
2.14
Br
CF3
PhS
CF3
2n
1H NMR (300 MHz, Chloroform-d)
5.49
1.00
0.99
1.01
1.10
2.13
1.98
18
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

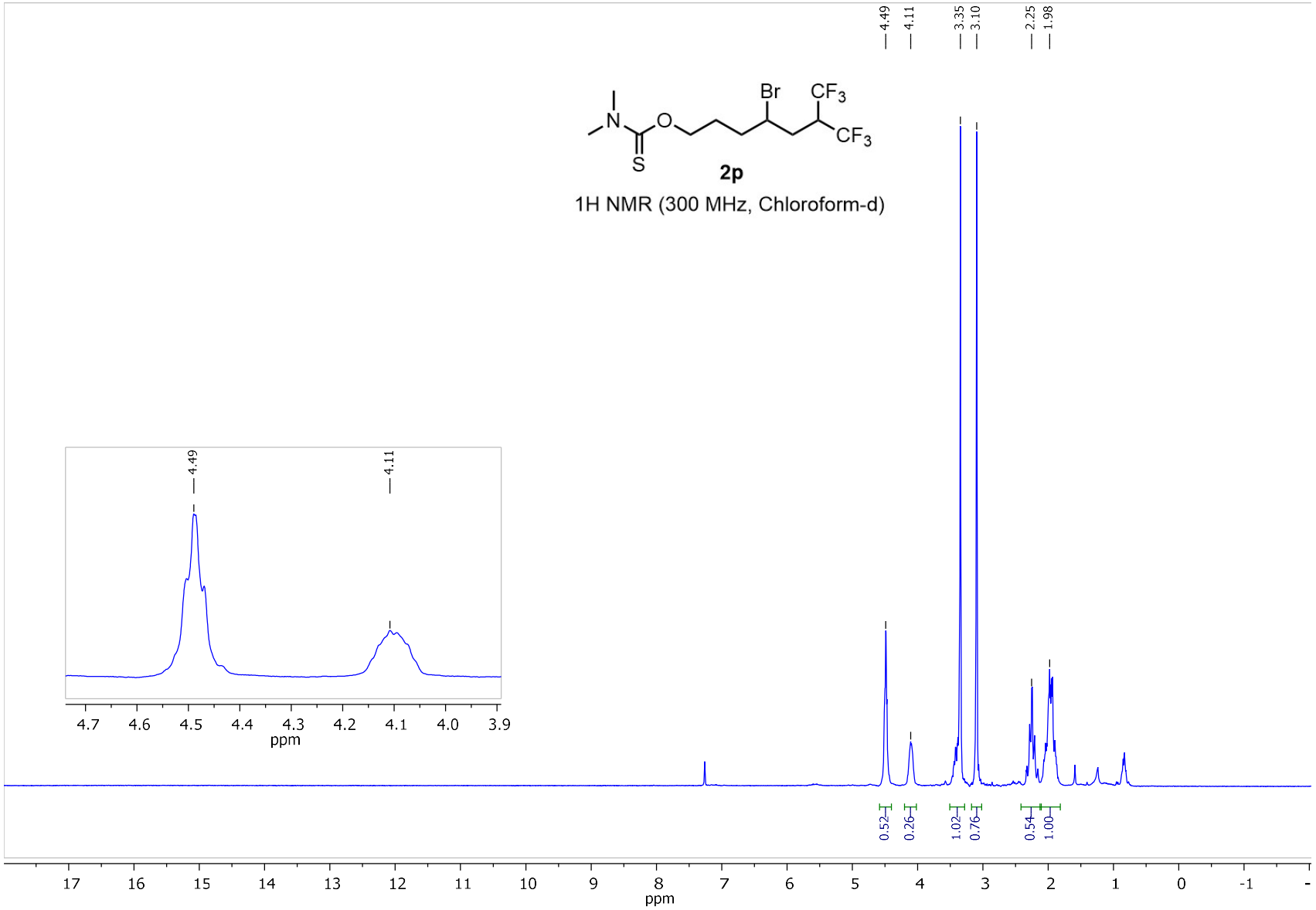

135.06
130.35
129.26
126.88
125.74
125.43
125.38
122.02
121.72
121.67
118.27
51.36
51.33
48.10
47.72
47.35
46.98
46.61
46.23
45.86
38.63
33.51
31.94
Br
CF3
PhS
CF3
2n
13C NMR (75 MHz, Chloroform-d)
ppm
ppm
ppm

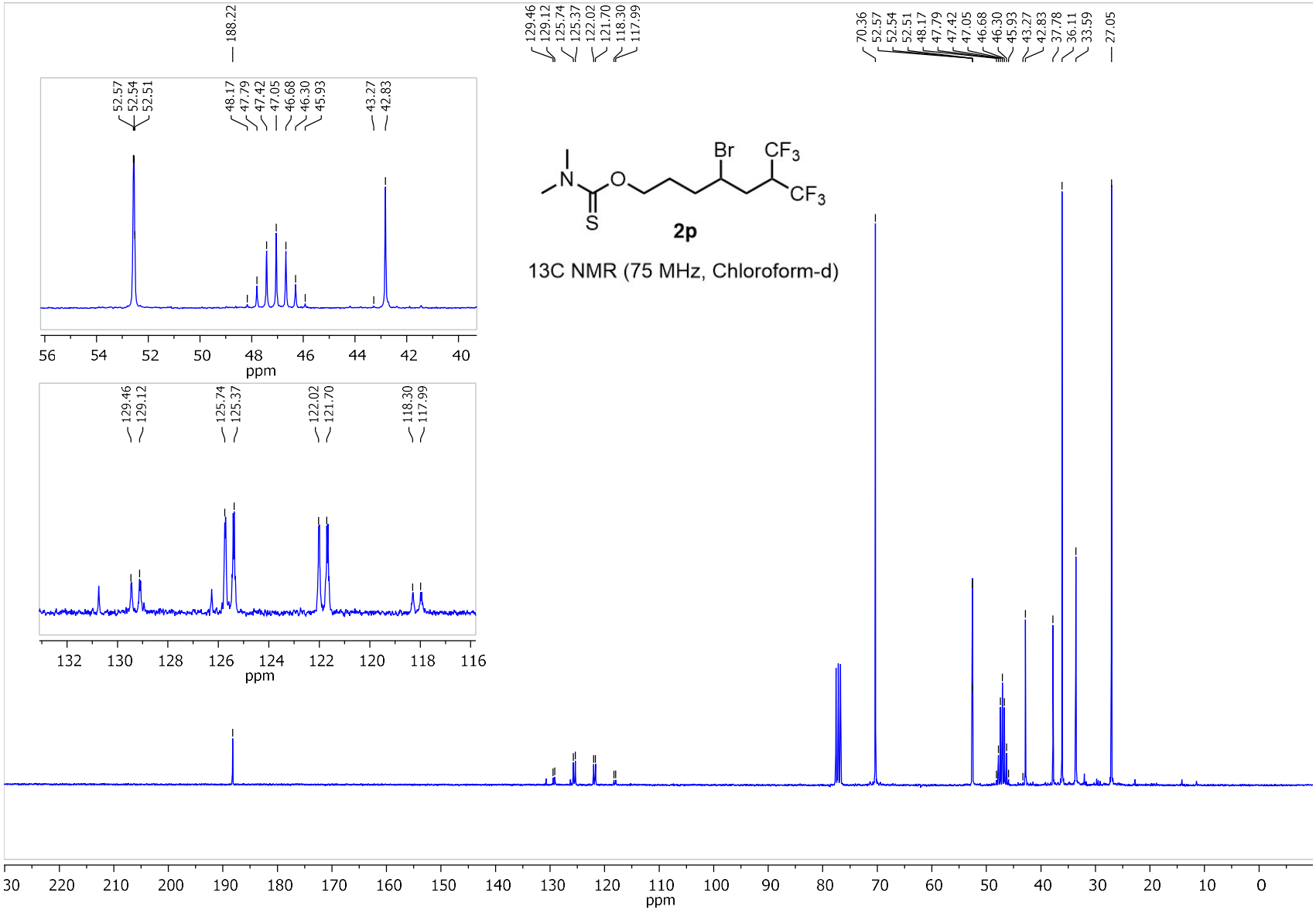

-67.21
-67.24
-67.27
-67.31
-67.34
-68.52
-68.56
-68.59
-68.62
-68.66
Br
CF3
PhS
CF3
2n
19F NMR (282 MHz, Chloroform-d)
-66.5
-67.0
-67.5
-68.0
-68.5
-69.0
ppm
1.00
1.02
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm

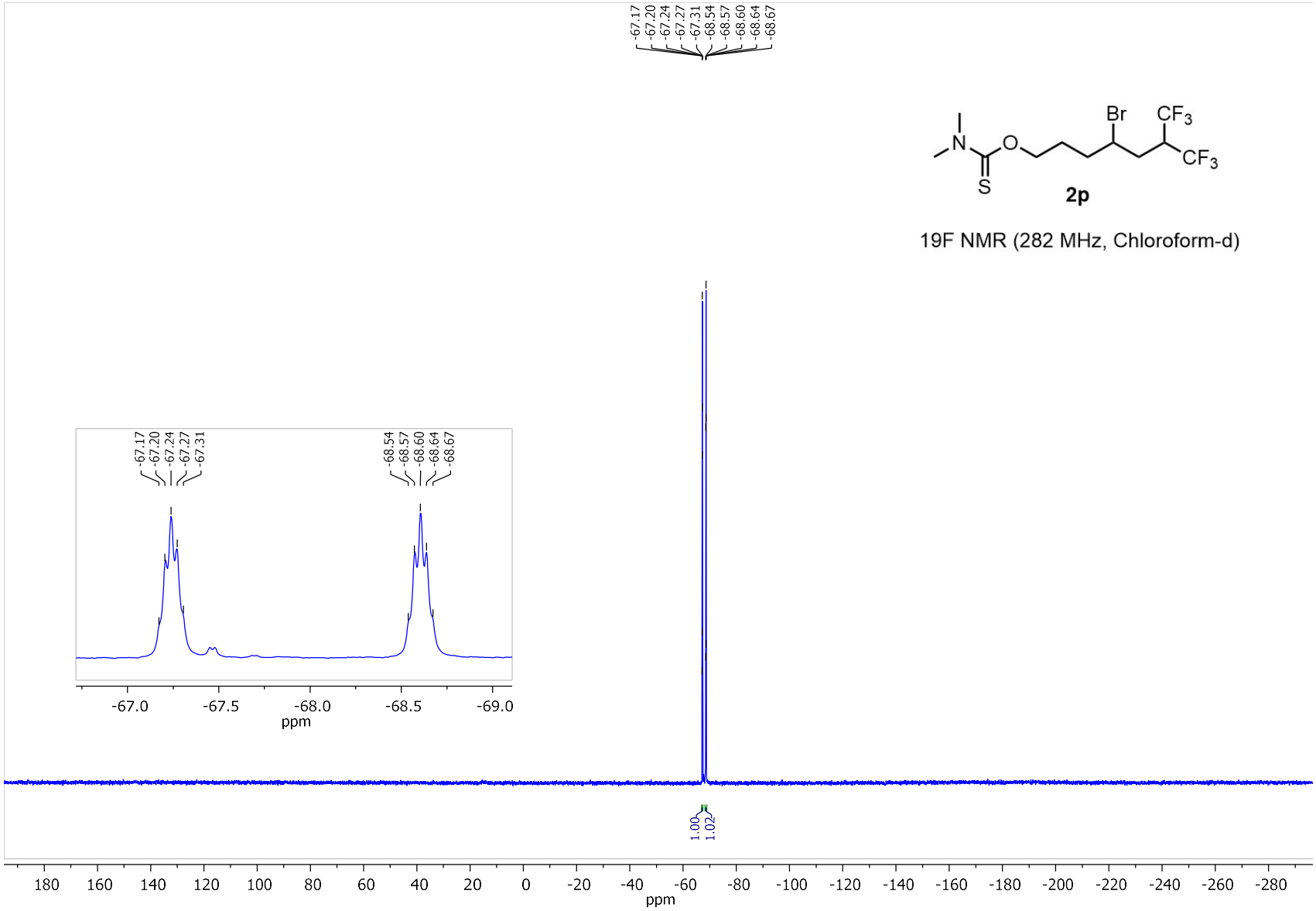

7.90
7.82
7.56
7.40
4.52
3.77
3.66
3.44
2.37
Br
CF3
CF3
2o
1H NMR (300 MHz, Chloroform-d)
2.96
3.97
0.99
1.00
1.01
1.00
1.95
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

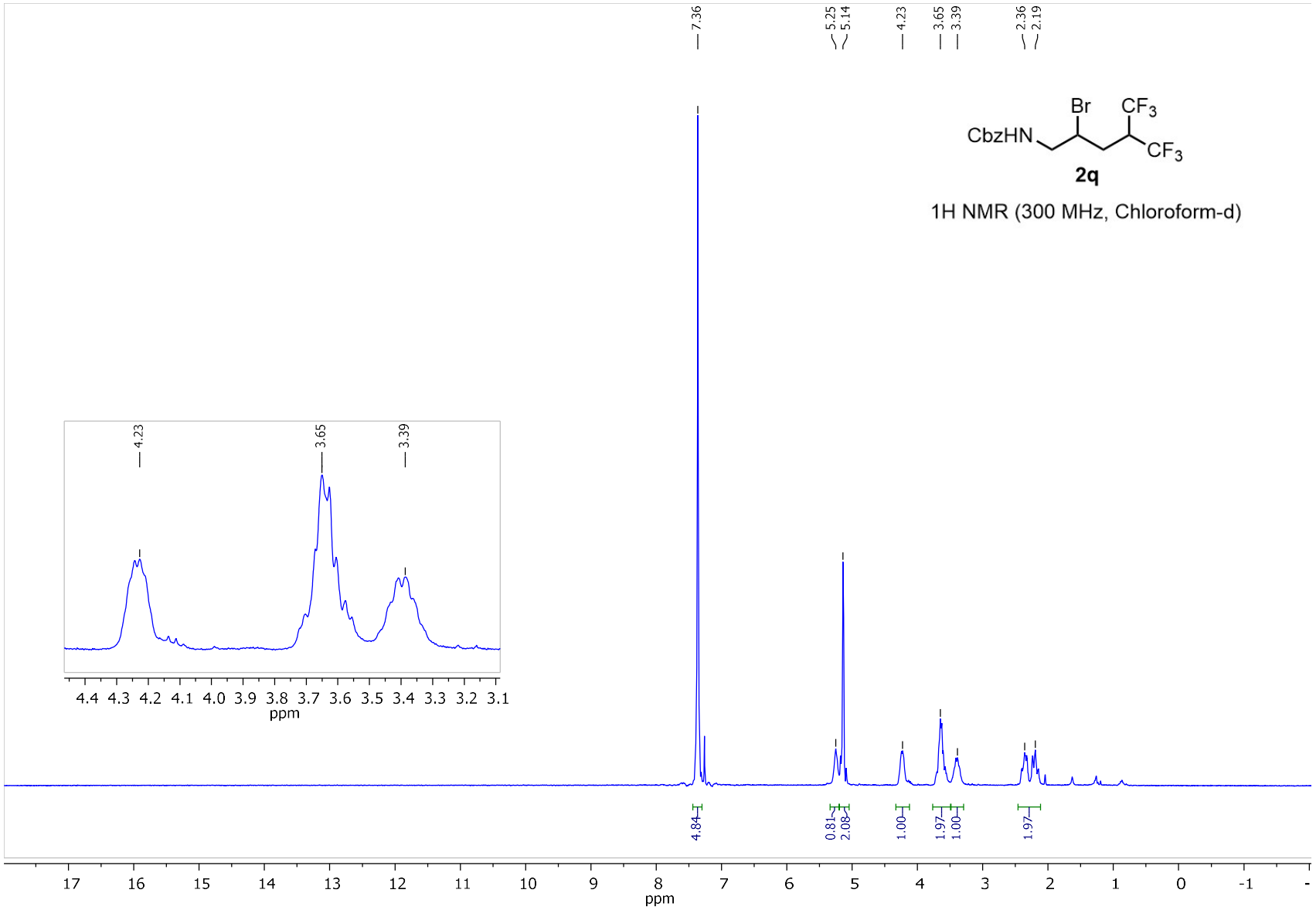

134.19
133.12
131.70
129.33
128.48
127.91
126.71
126.02
125.47
122.88
121.96
121.93
121.76
121.72
118.25
118.21
118.05
118.01
51.91
48.37
48.00
47.63
47.26
46.88
46.51
46.14
43.34
33.27
Br
CF3
CF3
2o
13C NMR (75 MHz, Chloroform-d)
ppm

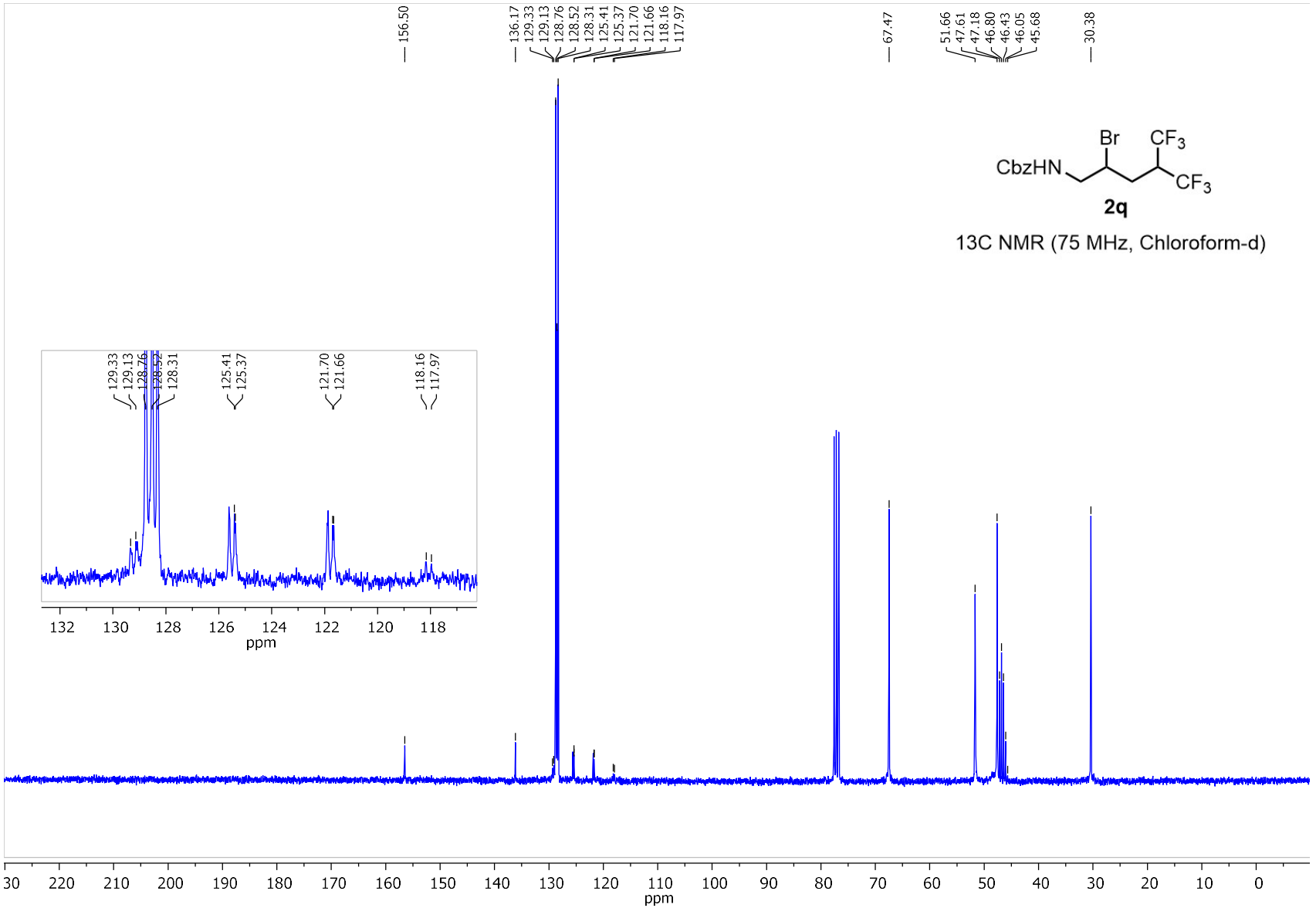

-67.20
-67.24
-67.28
-67.31
-67.34
-67.38
-68.52
-68.55
-68.58
-68.62
-68.65
Br
CF3
CF3
2o
19F NMR (282 MHz, Chloroform-d)
1.00
0.99
ppm

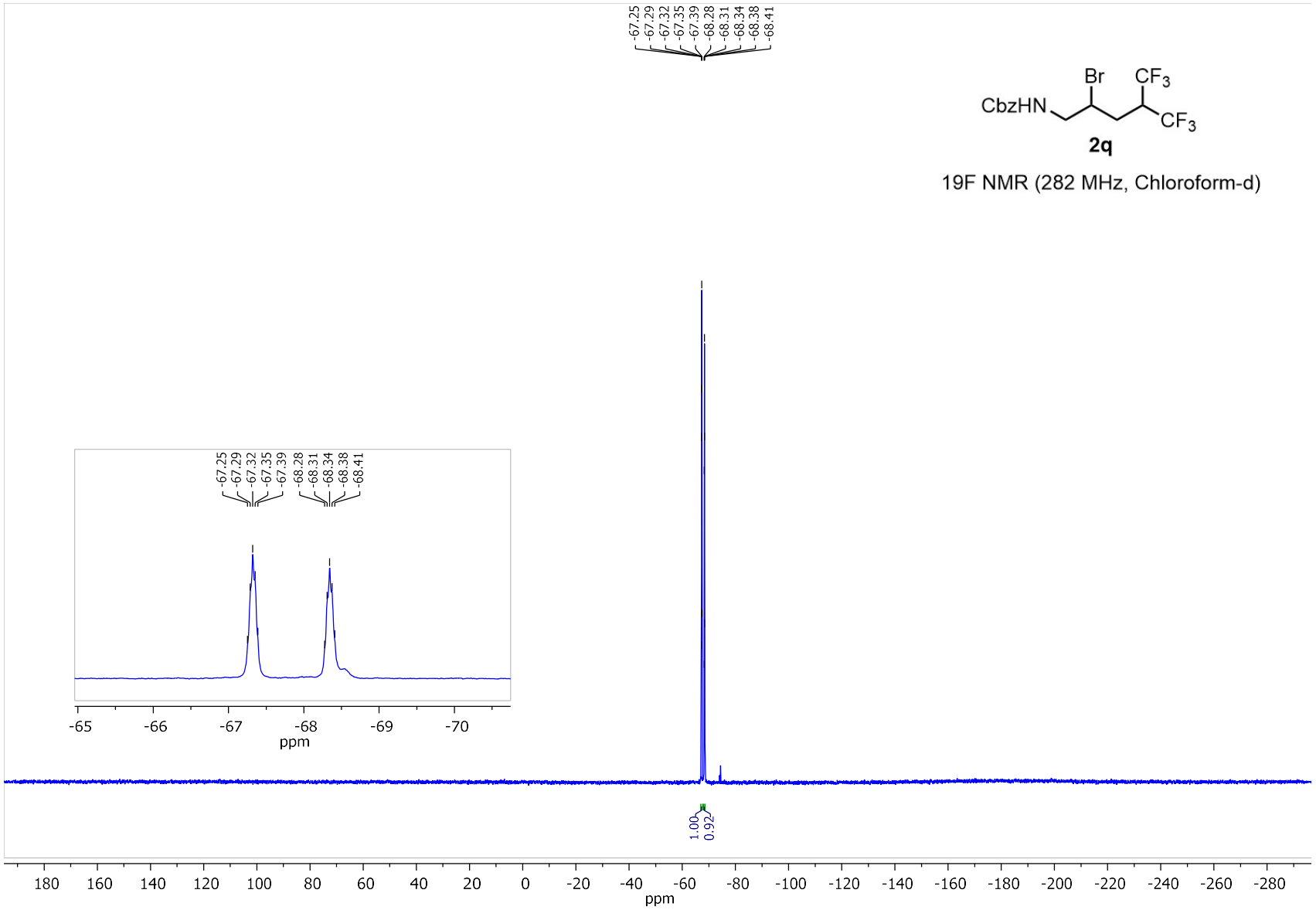

4.49
4.11
3.35
3.10
2.25
1.98
Br
CF3
CF3
N
O
S
2p
1H NMR (300 MHz, Chloroform-d)
0.52
0.26
1.02
0.76
0.54
1.00
ppm

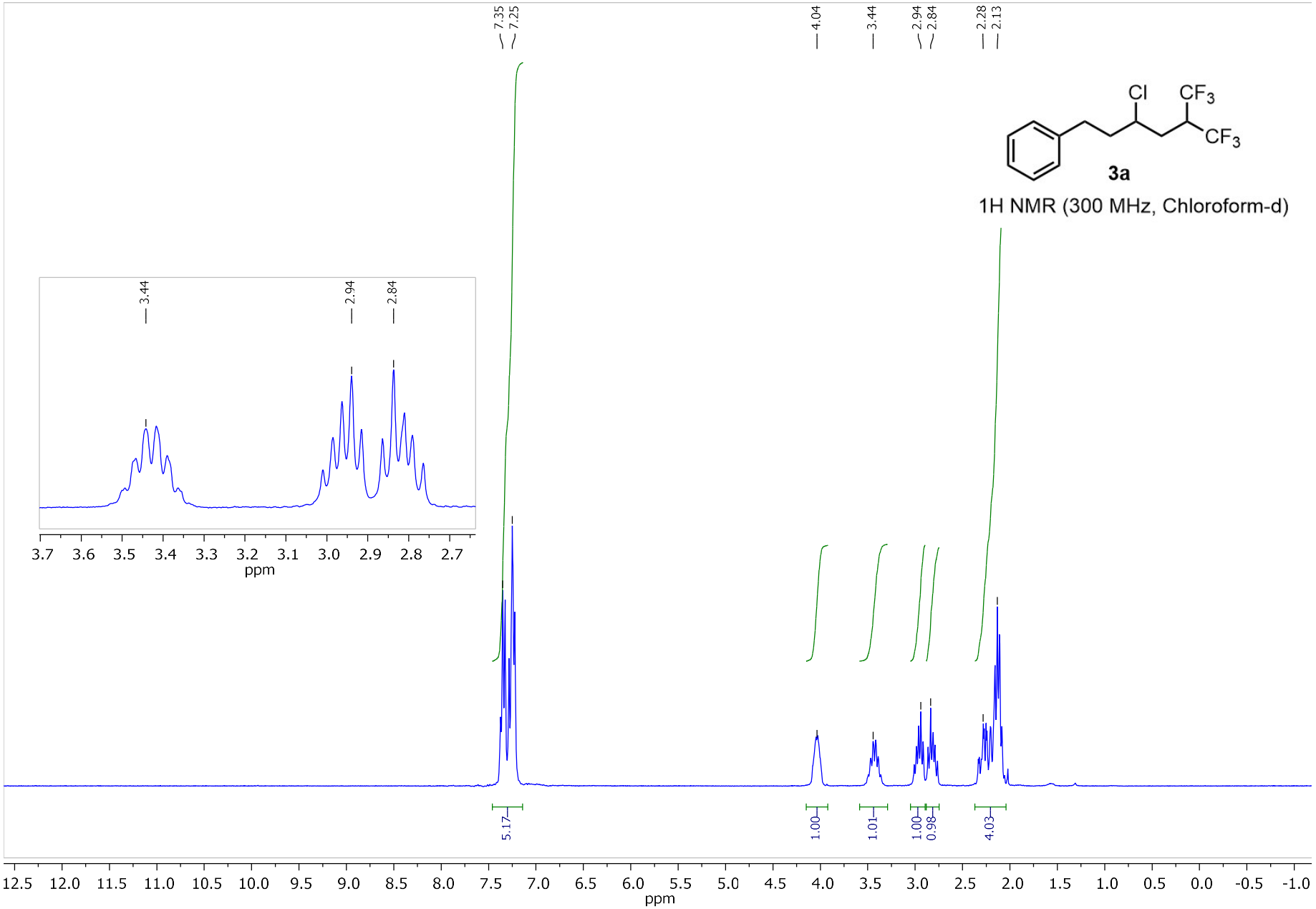

188.22
129.46
129.12
125.74
125.37
122.02
121.70
118.30
117.99
70.36
52.57
52.54
52.51
48.17
47.79
47.42
47.05
46.68
46.30
45.93
43.27
42.83
37.78
36.11
33.59
27.05
Br
CF3
CF3
N
O
S
2p
13C NMR (75 MHz, Chloroform-d)
ppm

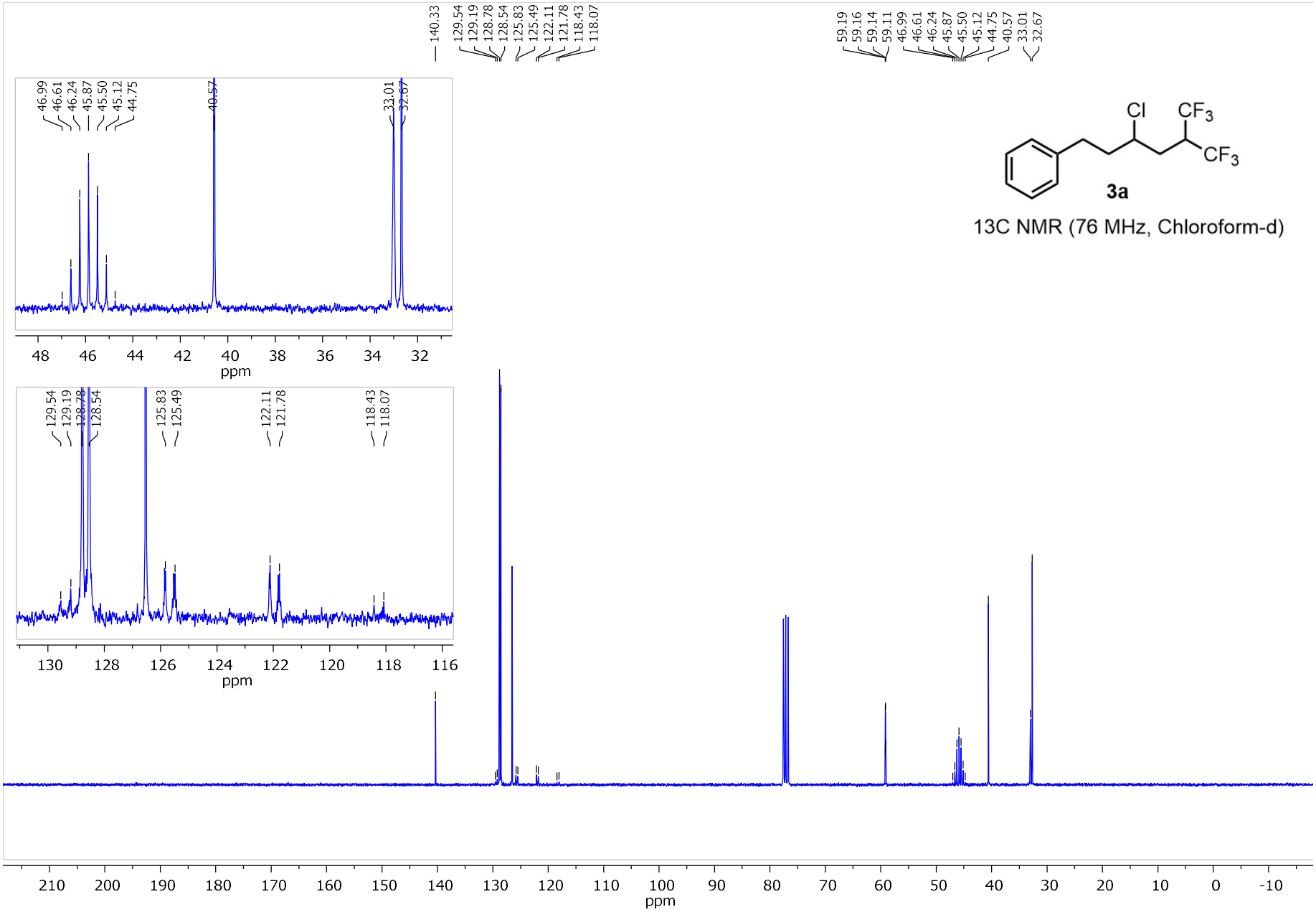

-67.17
-67.20
-67.24
-67.27
-67.31
-68.54
-68.57
-68.60
-68.64
-68.67
Br
CF3
CF3
N
O
S
2p
19F NMR (282 MHz, Chloroform-d)
-67.17
-67.20
-67.24
-67.27
-67.31
-68.54
-68.57
-68.60
-68.64
-68.67
-67.0
-67.5
-68.0
-68.5
-69.0
ppm
1.00
1.02
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm

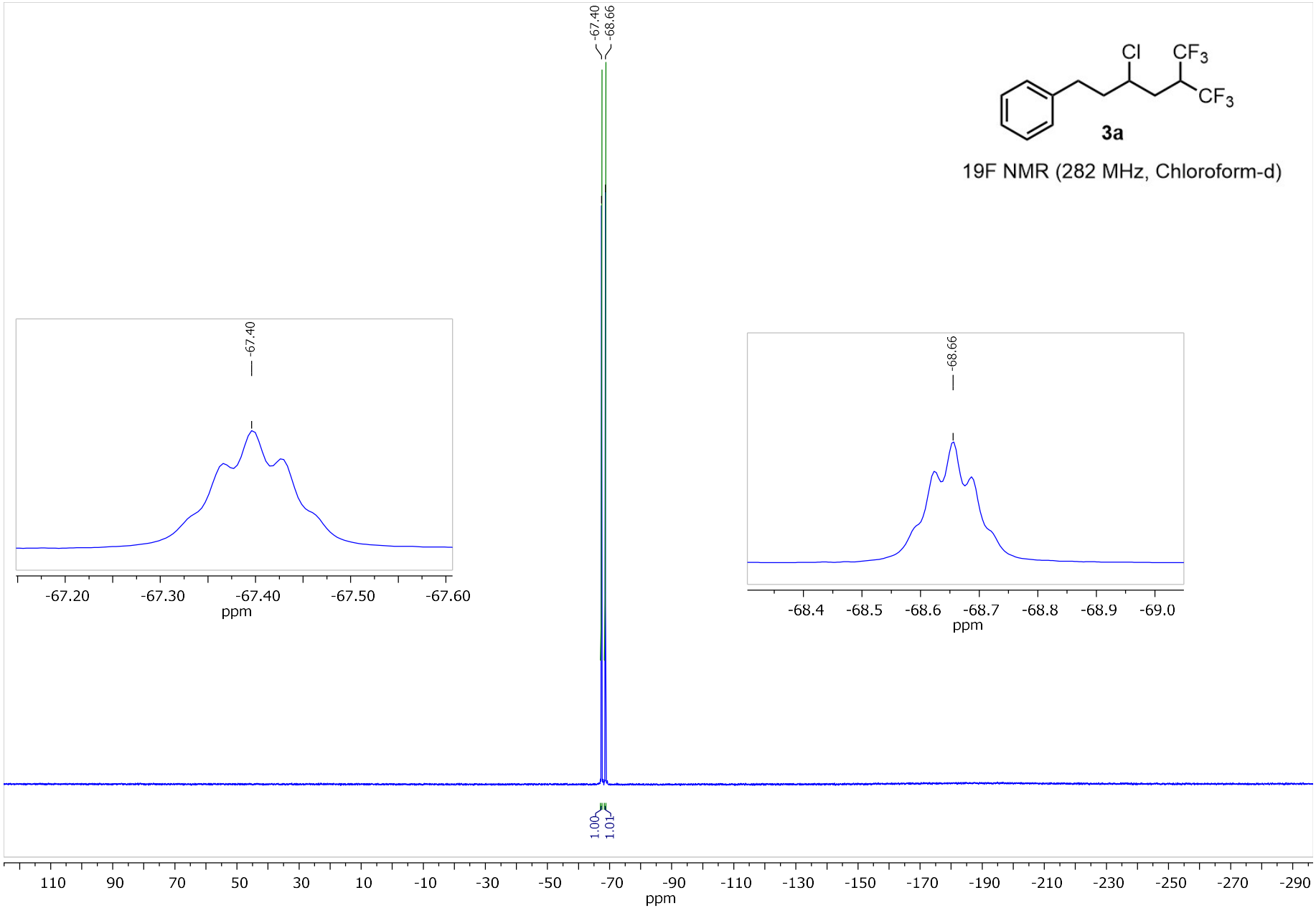

7.36
5.25
5.14
4.23
3.65
3.39
2.36
2.19
Br
CF3
CbzHN
CF3
2q
1H NMR (300 MHz, Chloroform-d)
4.84
0.81
2.08
1.00
1.97
1.00
1.97
ppm

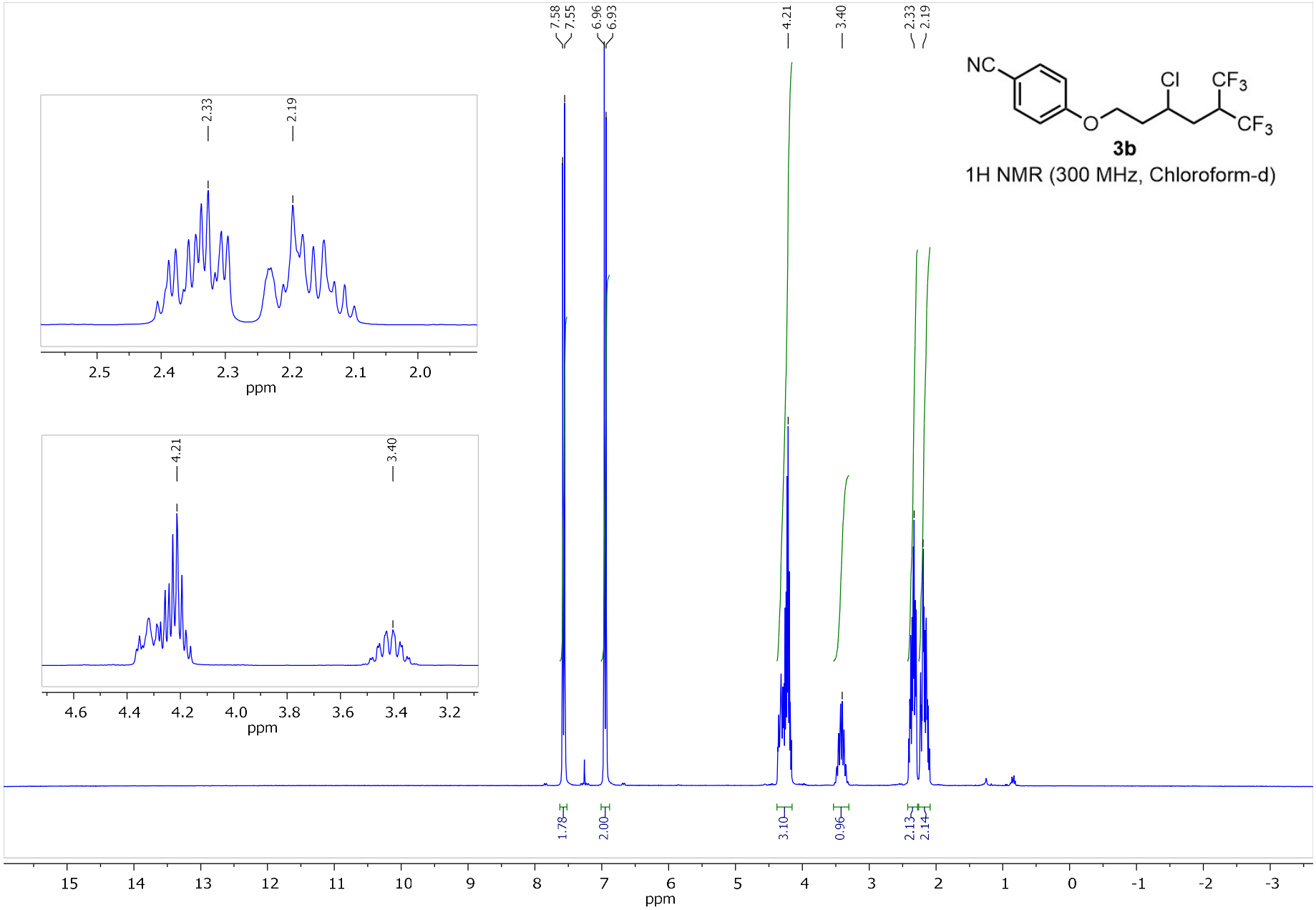

156.50
136.17
129.33
129.13
128.76
128.52
128.31
125.41
125.37
121.70
121.66
118.16
117.97
67.47
51.66
47.61
47.18
46.80
46.43
46.05
45.68
30.38
Br
CF3
CbzHN
CF3
2q
13C NMR (75 MHz, Chloroform-d)
ppm

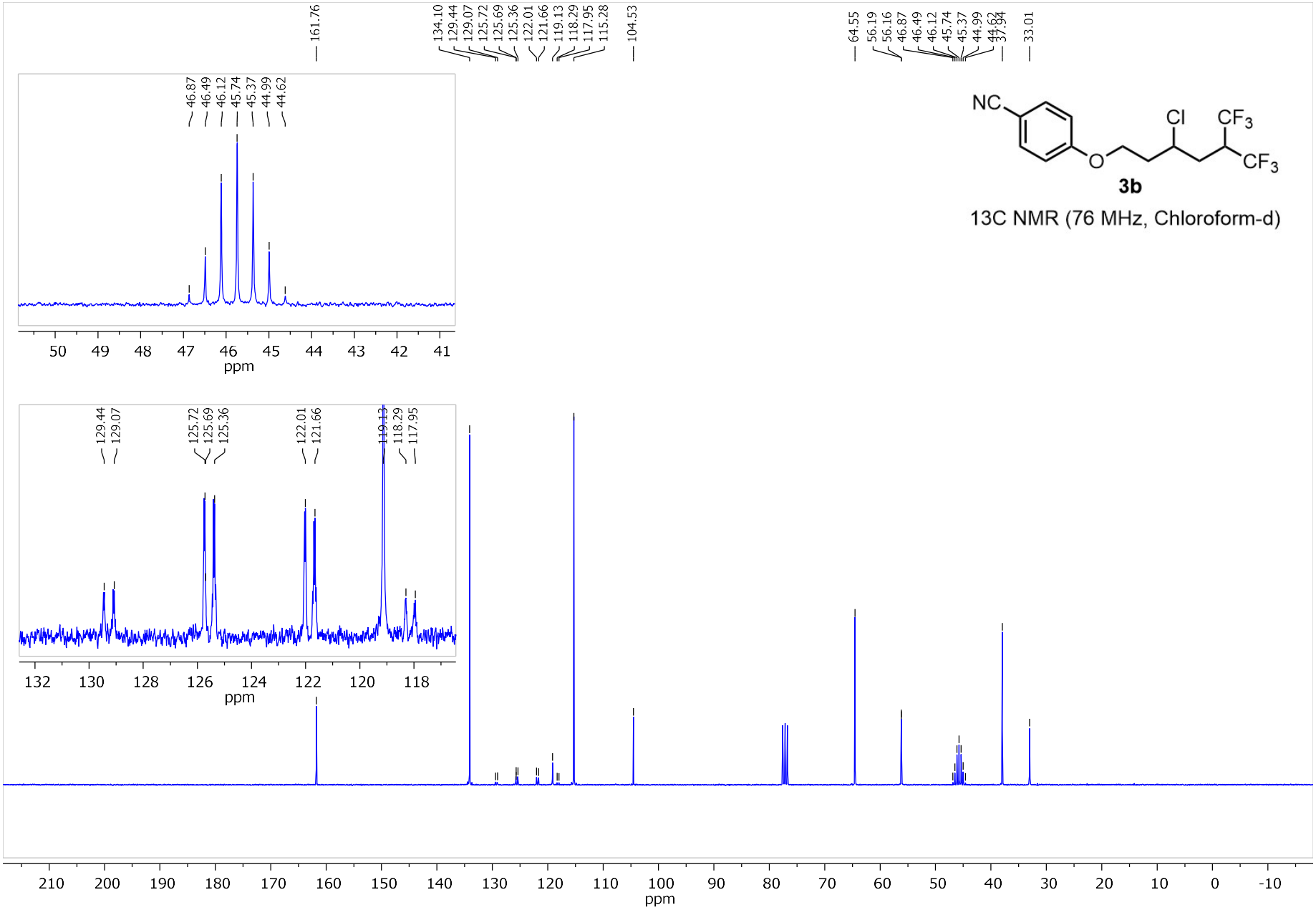

-67.25
-67.29
-67.32
-67.35
-67.39
-68.28
-68.31
-68.34
-68.38
-68.41
Br
CF3
CbzHN
CF3
2q
19F NMR (282 MHz, Chloroform-d)
1.00
0.92
ppm

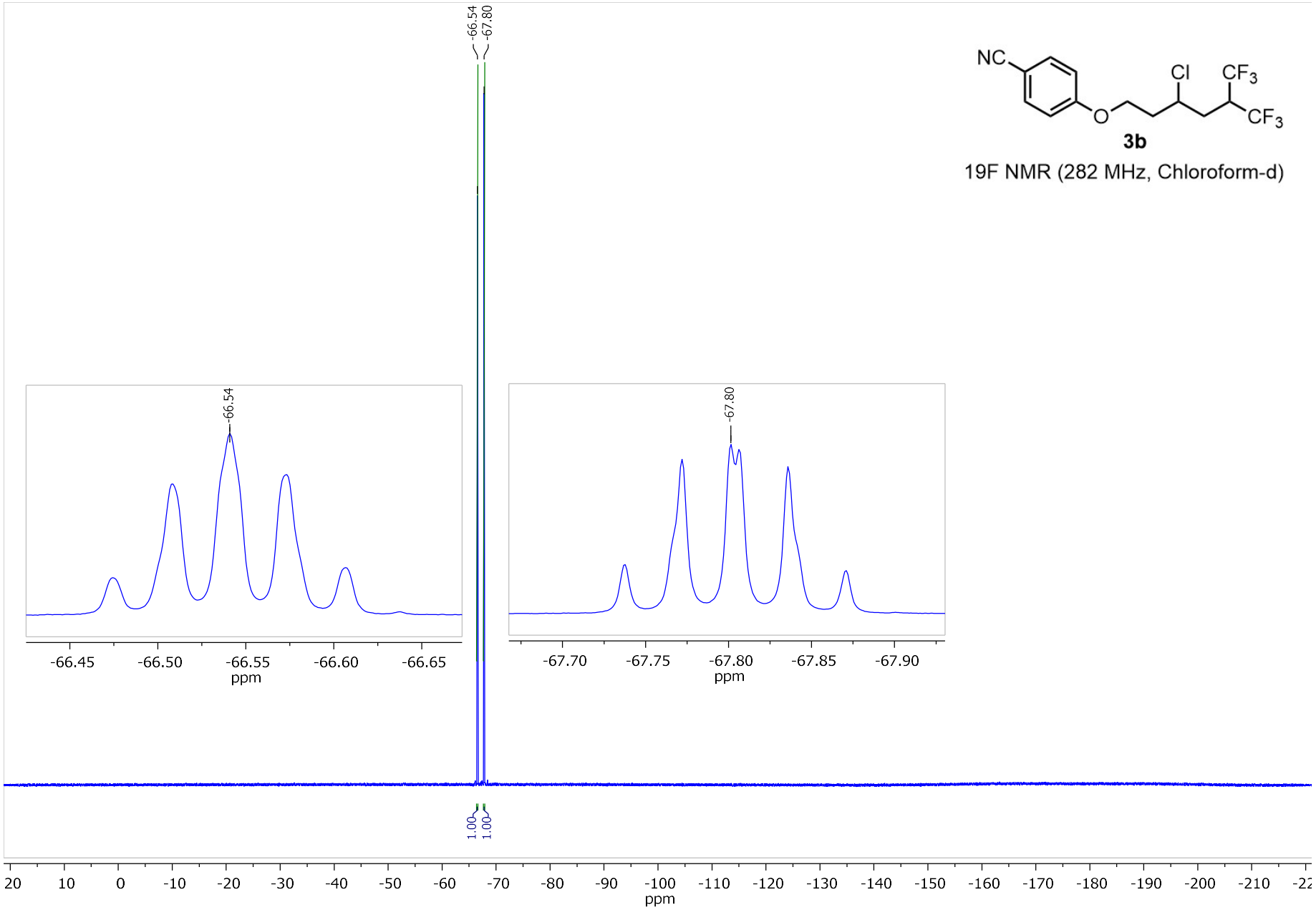

7.35
7.25
4.04
3.44
2.94
2.84
2.28
2.13
Cl
CF3
CF3
3a
1H NMR (300 MHz, Chloroform-d)
3.44
2.94
2.84
3.7 3.6 3.5 3.4 3.3 3.2 3.1 3.0 2.9 2.8 2.7
ppm
5.17
1.00
1.01
1.00
0.98
4.03
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
ppm

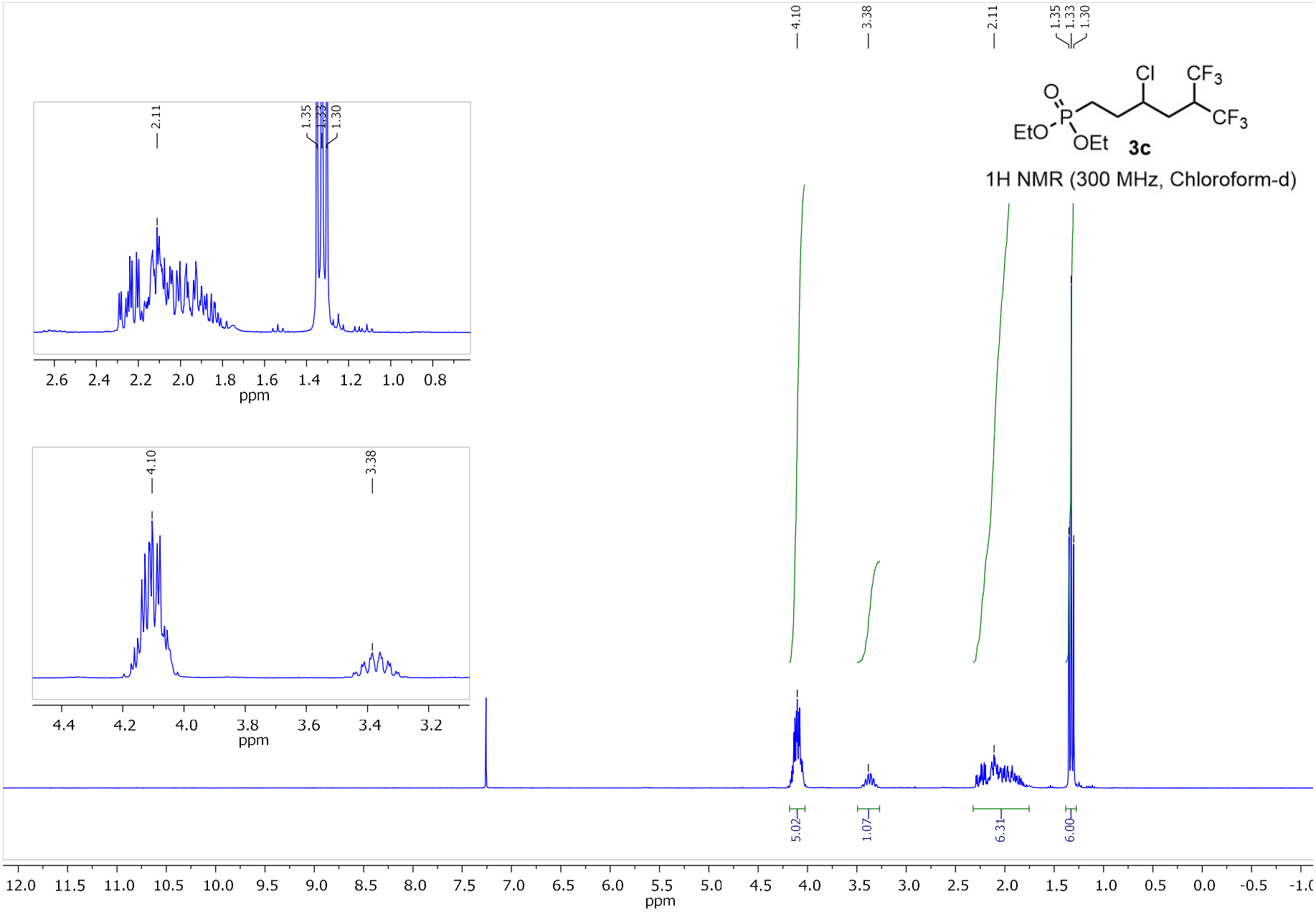

140.33
129.54
129.19
128.78
128.54
125.83
125.49
122.11
121.78
118.43
118.07
59.19
59.16
59.14
59.11
46.99
46.61
46.24
45.87
45.50
45.12
44.75
40.57
33.01
32.67
Cl
CF3
CF3
3a
13C NMR (76 MHz, Chloroform-d)
ppm

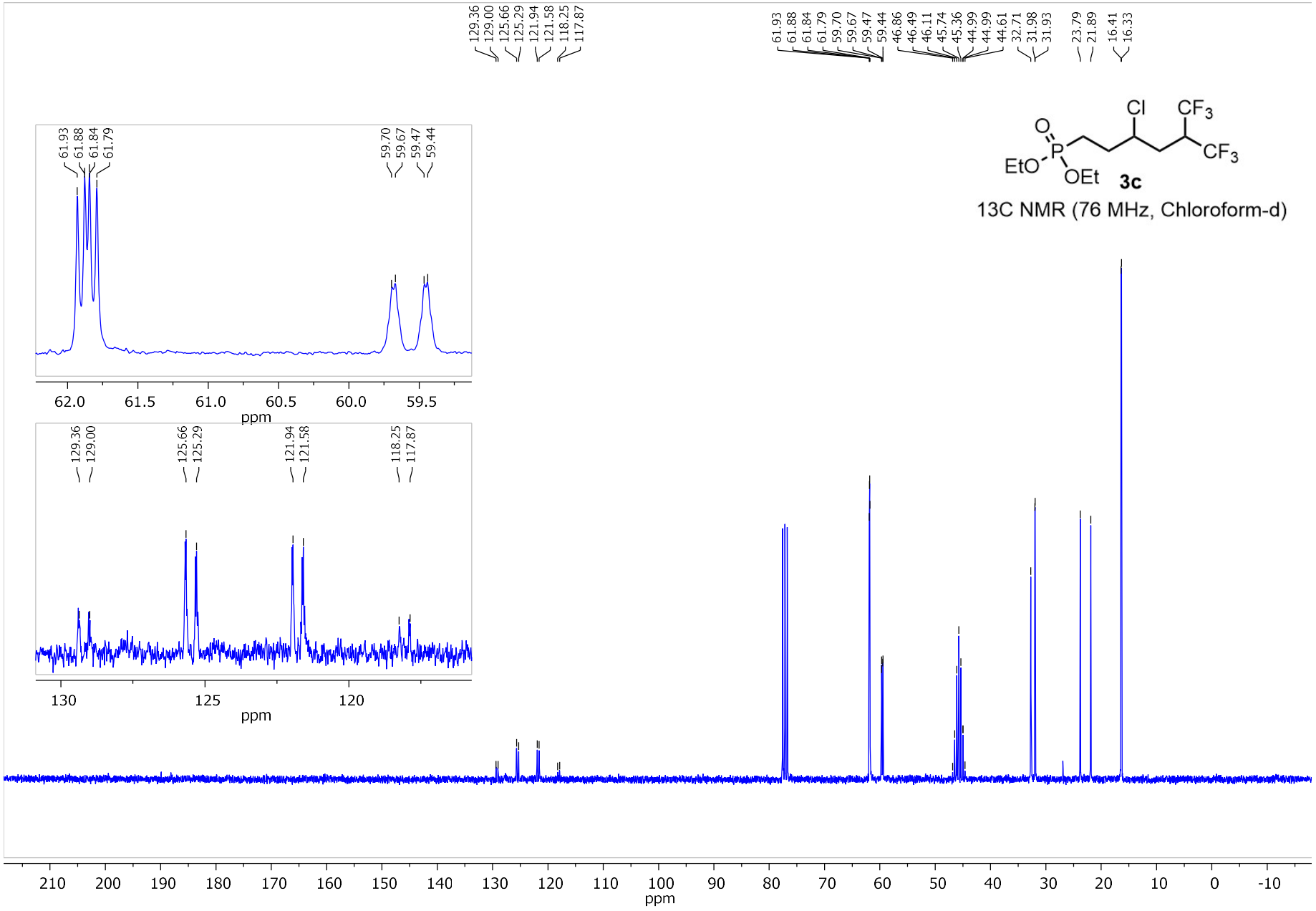

-67.40
-68.66
Cl
CF3
CF3
3a
19F NMR (282 MHz, Chloroform-d)
-67.40
-67.20
-67.30
-67.40
-67.50
-67.60
ppm
-68.66
-68.4
-68.5
-68.6
-68.7
-68.8
-68.9
-69.0
ppm
1.00
1.01
110
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
ppm


7.58
7.55
6.96
6.93
4.21
3.40
2.33
2.19
NC
Cl
CF3
CF3
O
3b
1H NMR (300 MHz, Chloroform-d)
ppm
1.78
2.00
3.10
0.96
2.13
2.14


161.76
134.10
129.44
129.07
125.72
125.69
125.36
122.01
121.66
119.13
118.29
117.95
115.28
104.53
64.55
56.19
56.16
46.87
46.49
46.12
45.74
45.37
44.99
44.62
37.94
33.01
NC
Cl
CF3
CF3
O
3b
13C NMR (76 MHz, Chloroform-d)
ppm


-66.54
-67.80
NC
Cl
CF3
O
CF3
3b
19F NMR (282 MHz, Chloroform-d)
-66.54
-66.45
-66.50
-66.55
-66.60
-66.65
ppm
-67.80
-67.70
-67.75
-67.80
-67.85
-67.90
ppm
1.00
1.00
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
ppm


4.10
3.38
2.11
1.35
1.33
1.30
Cl
CF3
O
P
EtO
OEt
CF3
3c
1H NMR (300 MHz, Chloroform-d)
5.02
1.07
6.31
6.00
ppm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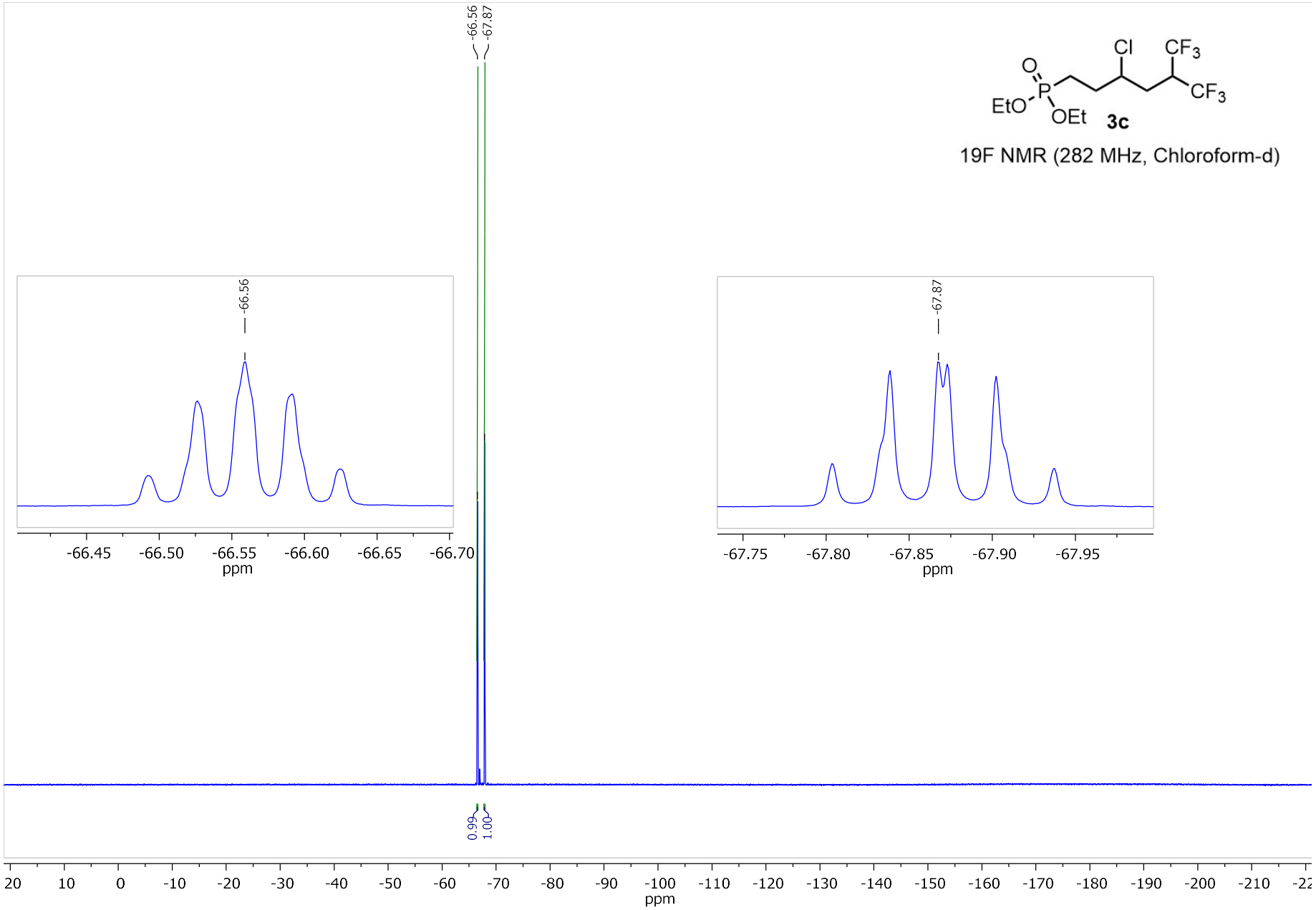
129.00
125.66
125.29
121.94
121.58
118.25
117.87
61.93
61.88
61.84
61.79
59.70
59.67
59.47
59.44
46.86
46.49
46.11
45.74
45.36
44.99
44.99
44.61
32.71
31.98
31.93
23.79
21.89
16.41
16.33
Cl
CF3
O
P
EtO
OEt
CF3
3c
13C NMR (76 MHz, Chloroform-d)
62.0
61.5
61.0
60.5
60.0
59.5
ppm
130
125
120
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm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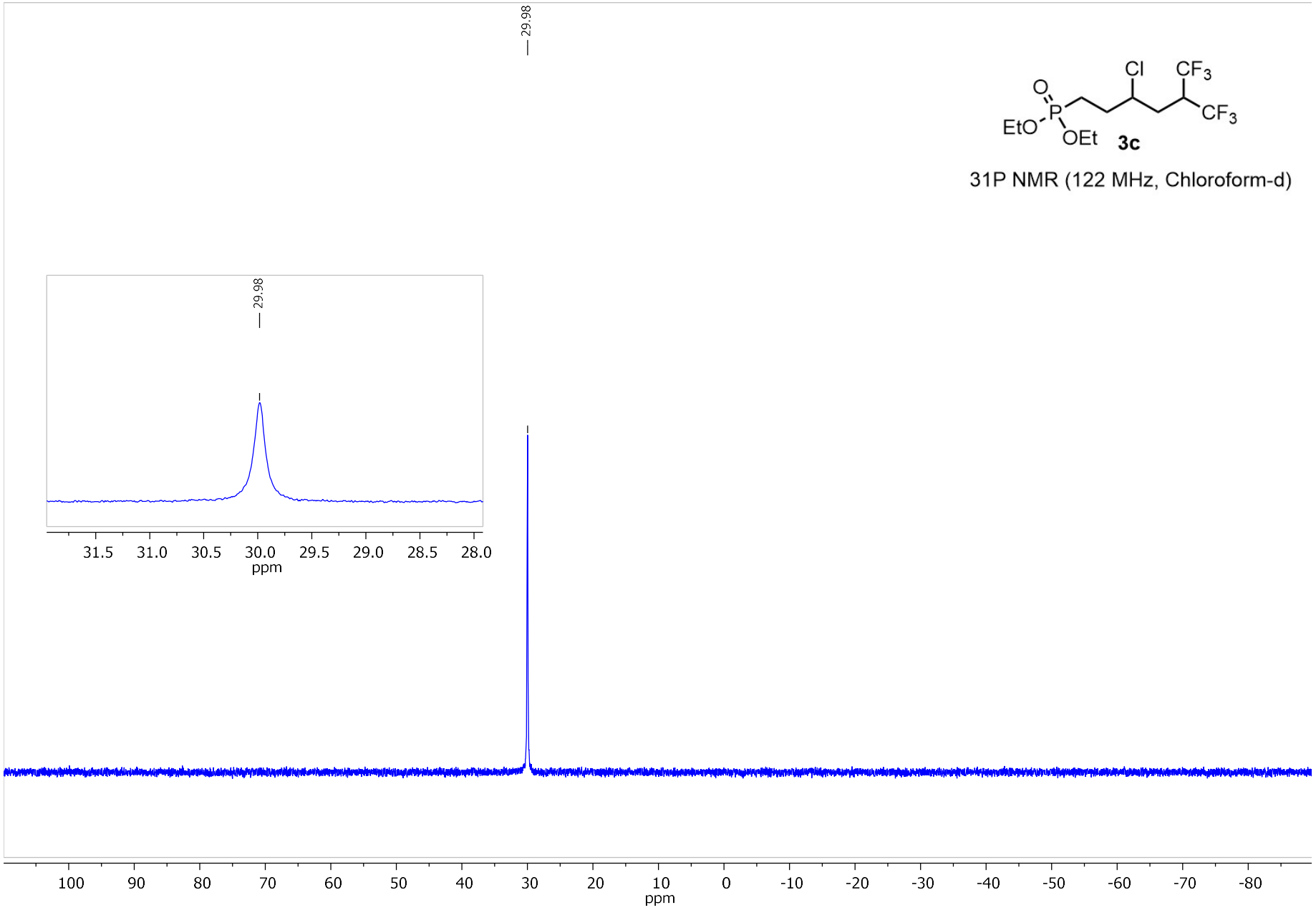
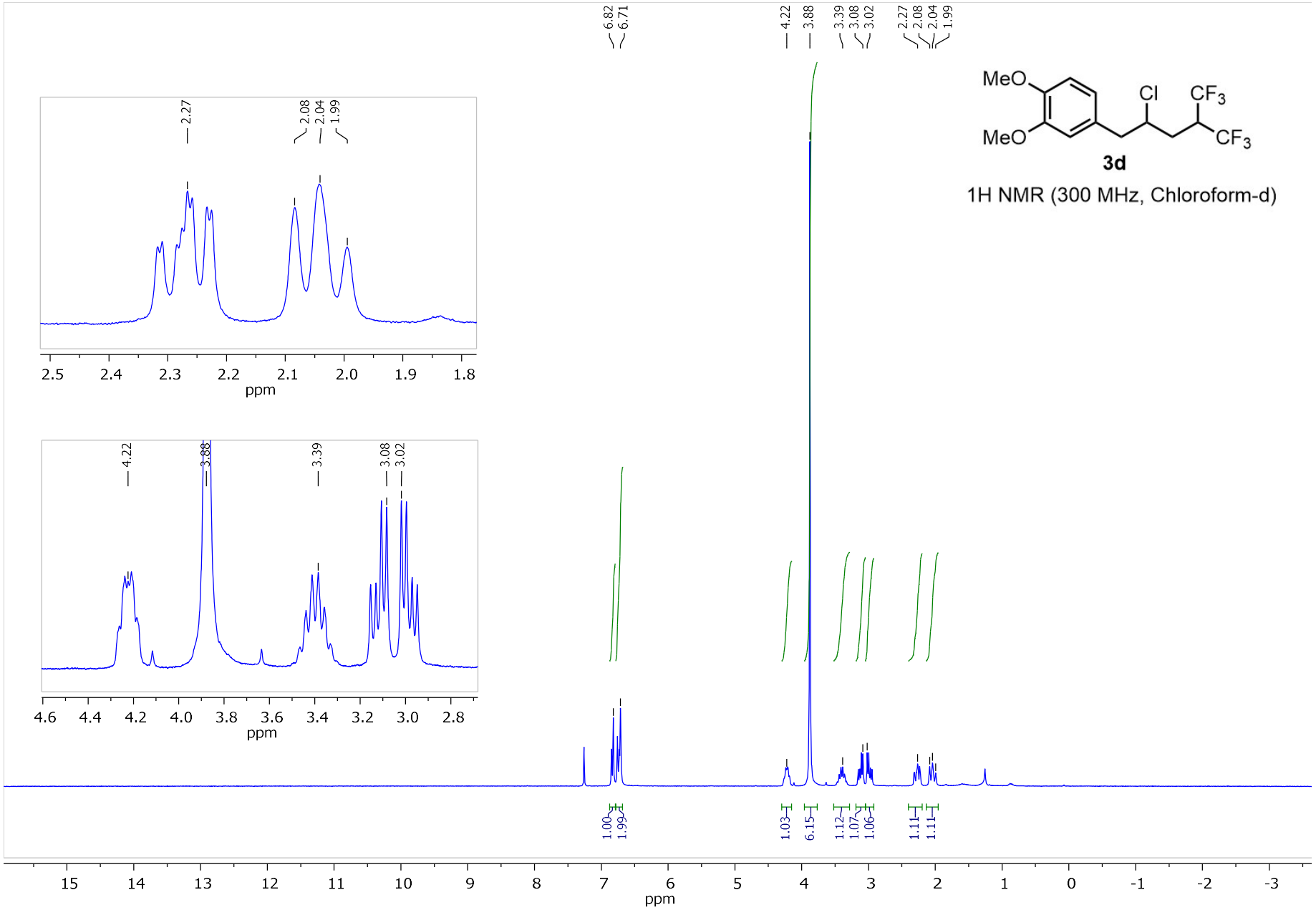
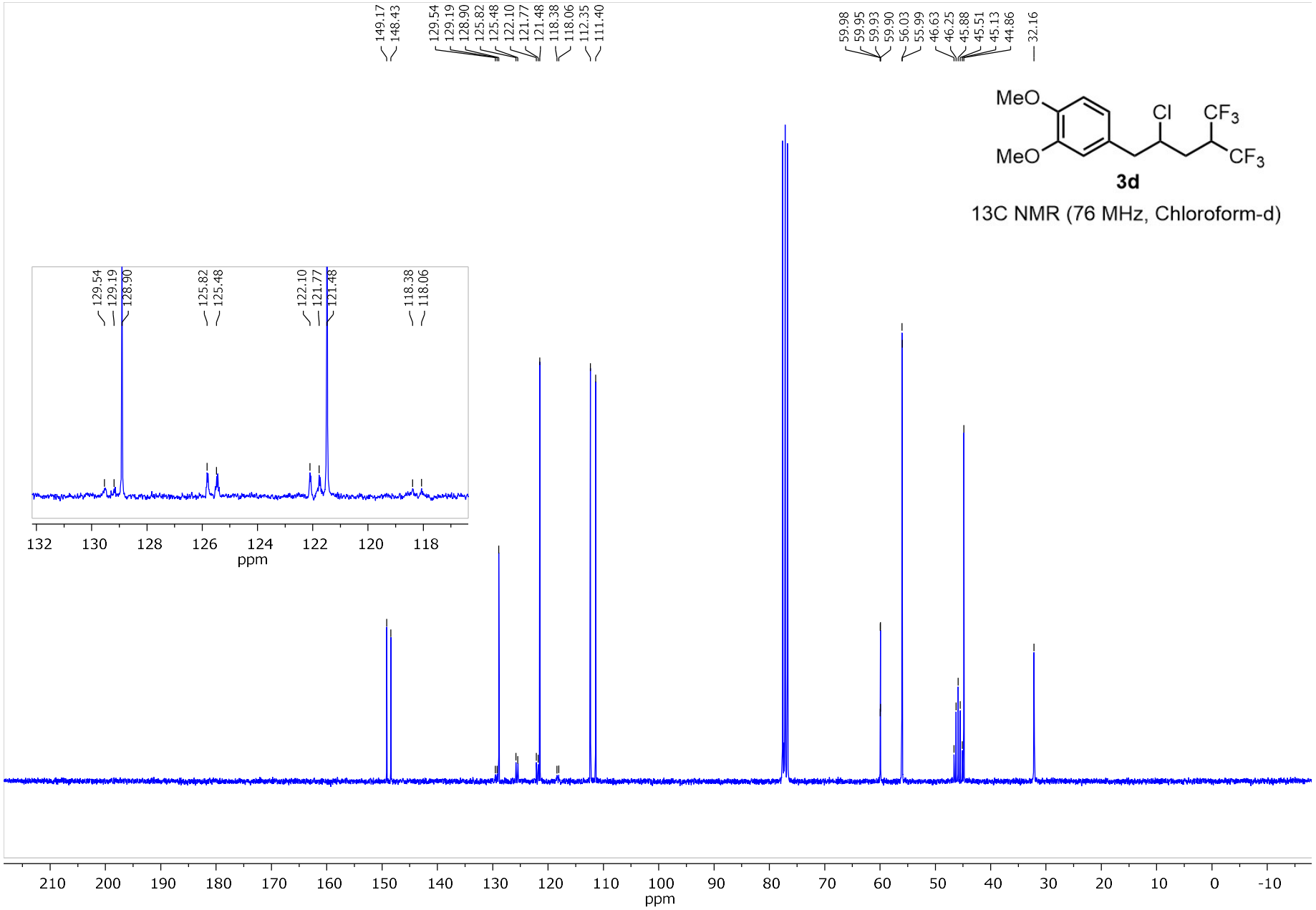
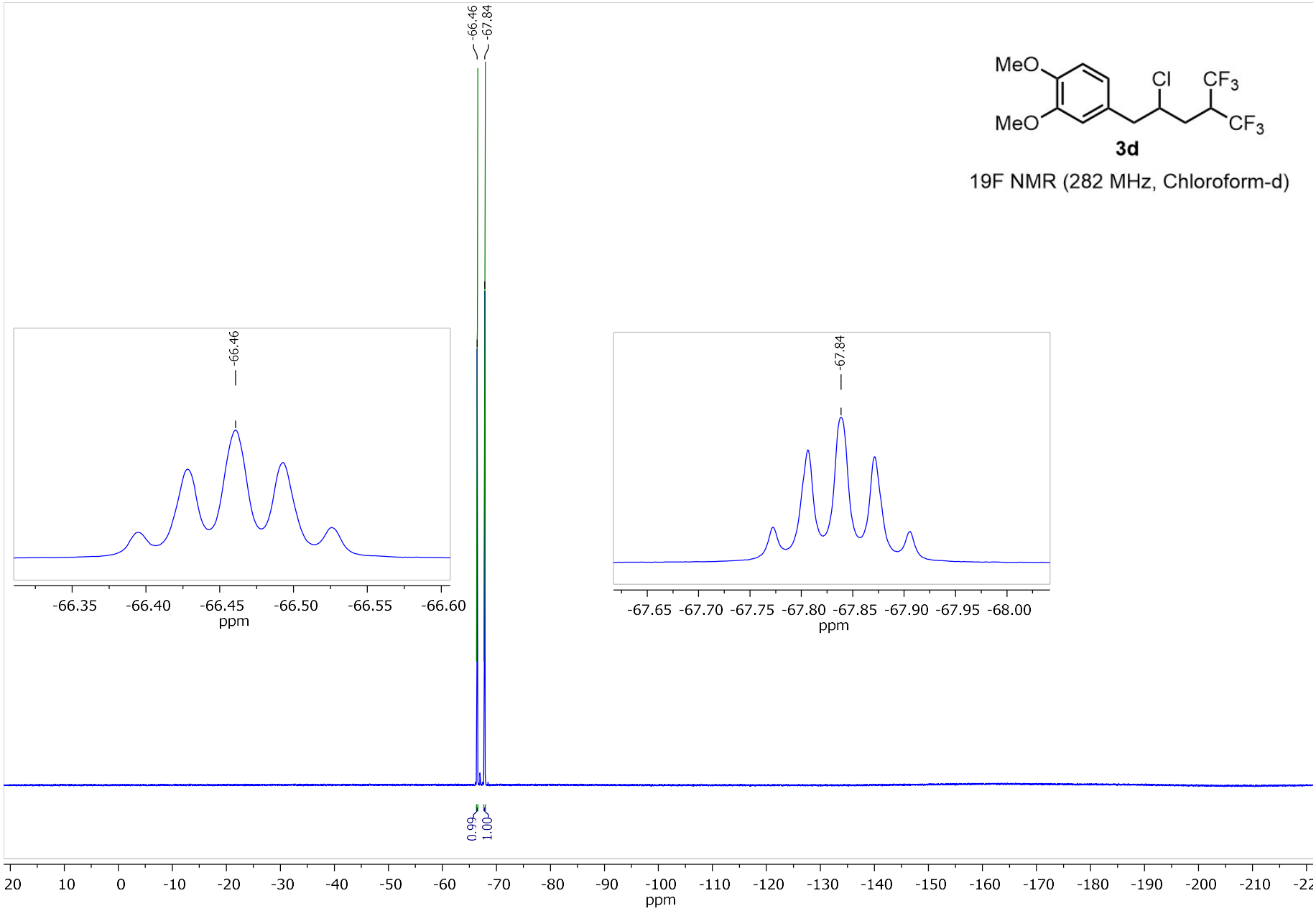
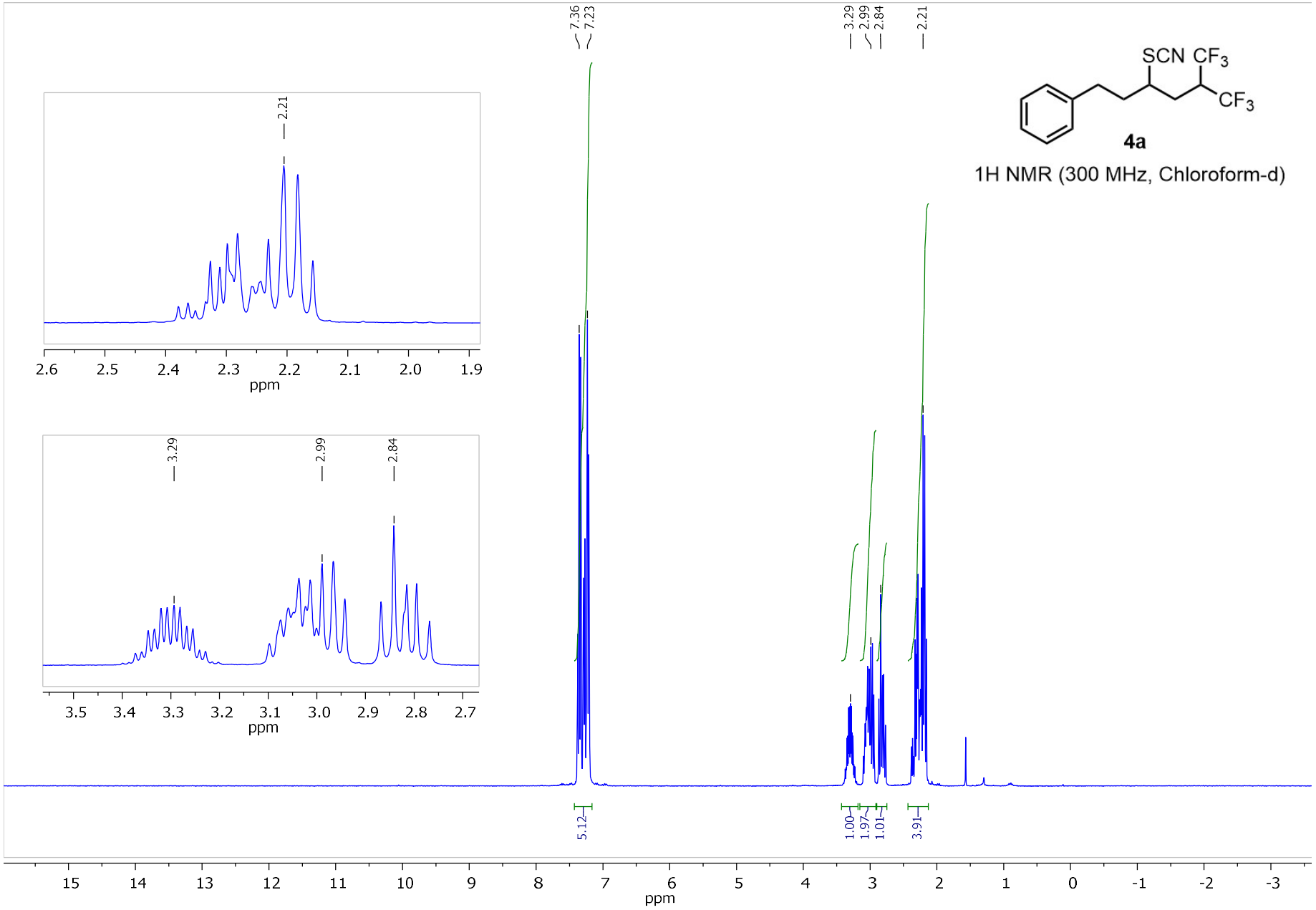
-67.87
Cl
CF3
O
P
EtO
OEt
CF3
3c
19F NMR (282 MHz, Chloroform-d)
-66.56
-66.45
-66.50
-66.55
-66.60
-66.65
-66.70
ppm
-67.87
-67.75
-67.80
-67.85
-67.90
-67.95
ppm
0.99
1.00
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
ppm

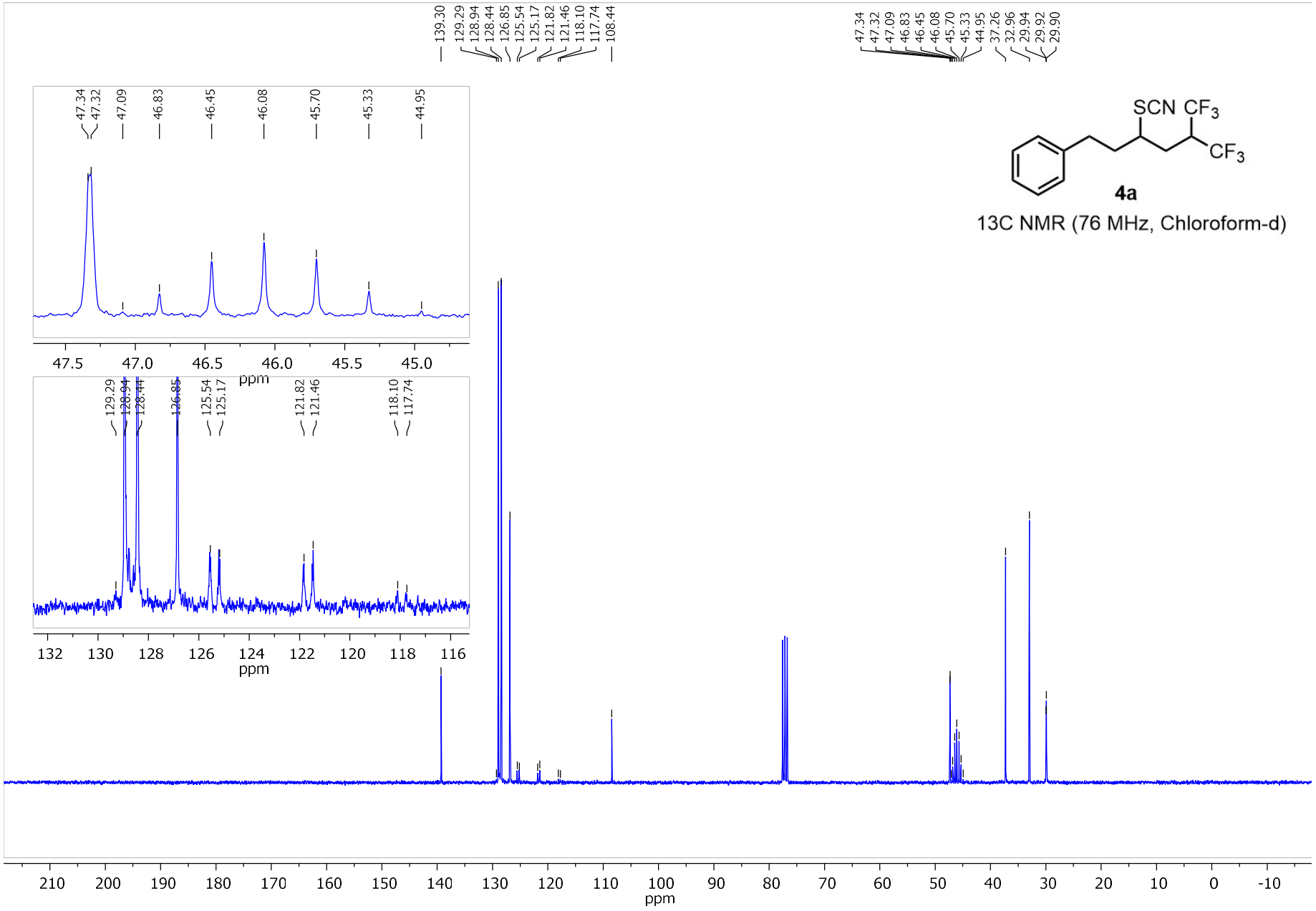

29.98
Cl
CF3
O
P
EtO
OEt
CF3
3c
31P NMR (122 MHz, Chloroform-d)
29.98
31.5
31.0
30.5
30.0
29.5
29.0
28.5
28.0
ppm
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
ppm

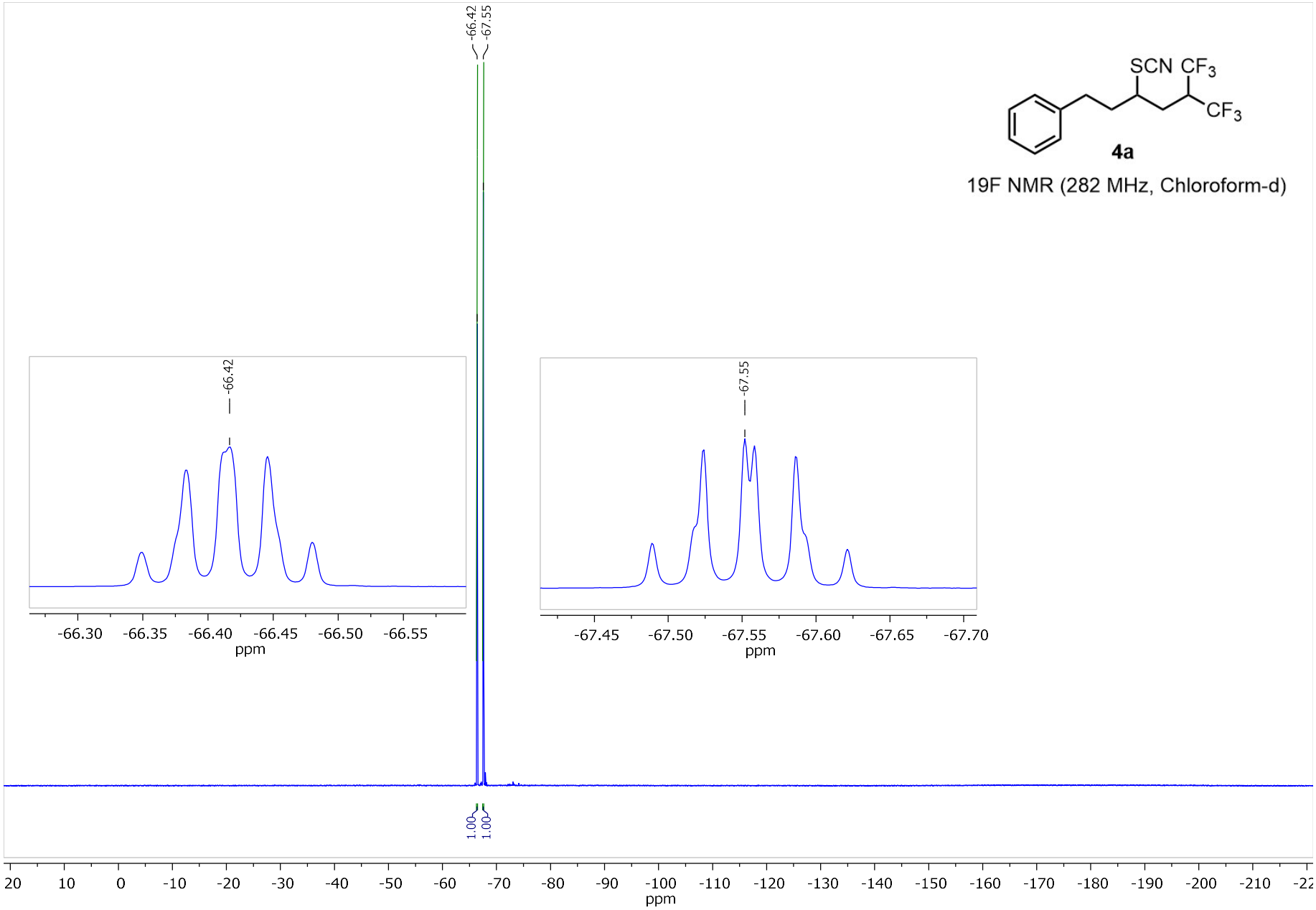

6.82
6.71
4.22
3.88
3.39
3.08
3.02
2.27
2.08
2.04
1.99
MeO
MeO
Cl
CF3
CF3
3d
1H NMR (300 MHz, Chloroform-d)
ppm
1.00
1.99
1.03
6.15
1.12
1.07
1.06
1.11
1.11

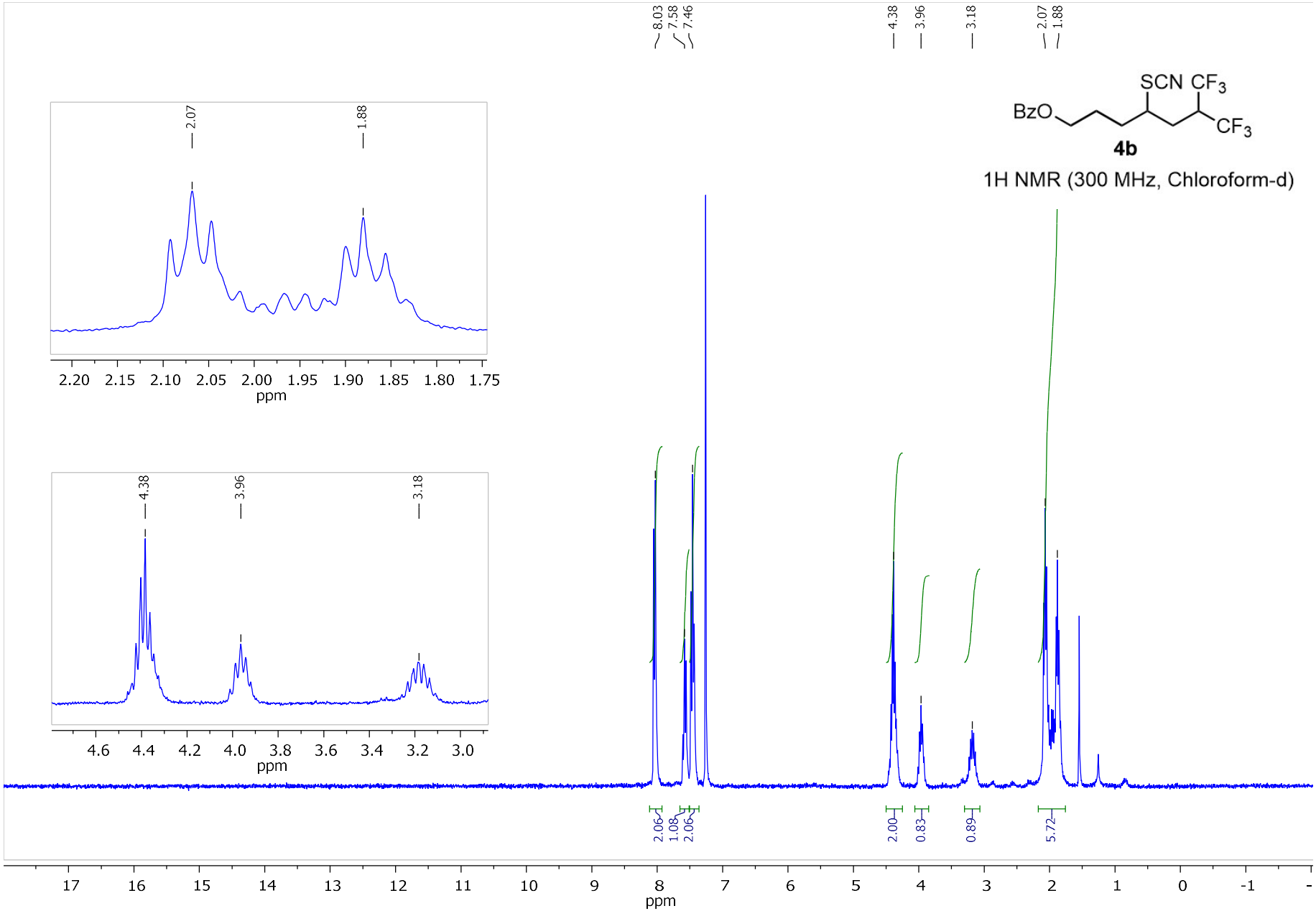

149.17
148.43
129.54
129.19
128.90
125.82
125.48
122.10
121.77
121.48
118.38
118.06
112.35
111.40
59.98
59.95
59.93
59.90
56.03
55.99
46.63
46.25
45.88
45.51
45.13
44.86
32.16
MeO
MeO
Cl
CF3
CF3
3d
13C NMR (76 MHz, Chloroform-d)
ppm
ppm

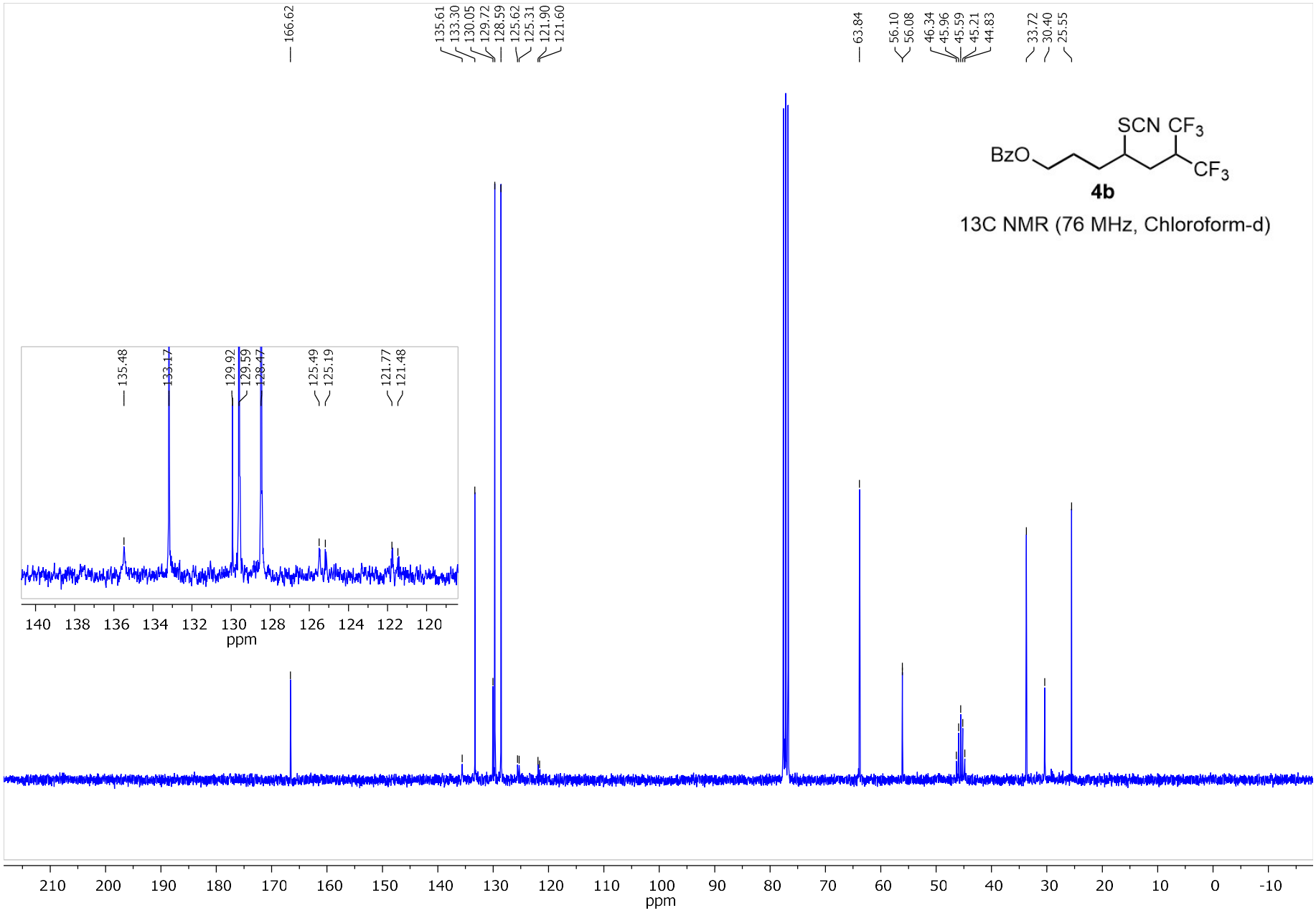

-66.46
-67.84
MeO
MeO
Cl
CF3
CF3
3d
19F NMR (282 MHz, Chloroform-d)
-66.46
-66.35 -66.40 -66.45 -66.50 -66.55 -66.60
ppm
-67.84
-67.65 -67.70 -67.75 -67.80 -67.85 -67.90 -67.95 -68.00
ppm
0.99
1.00
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -22
ppm

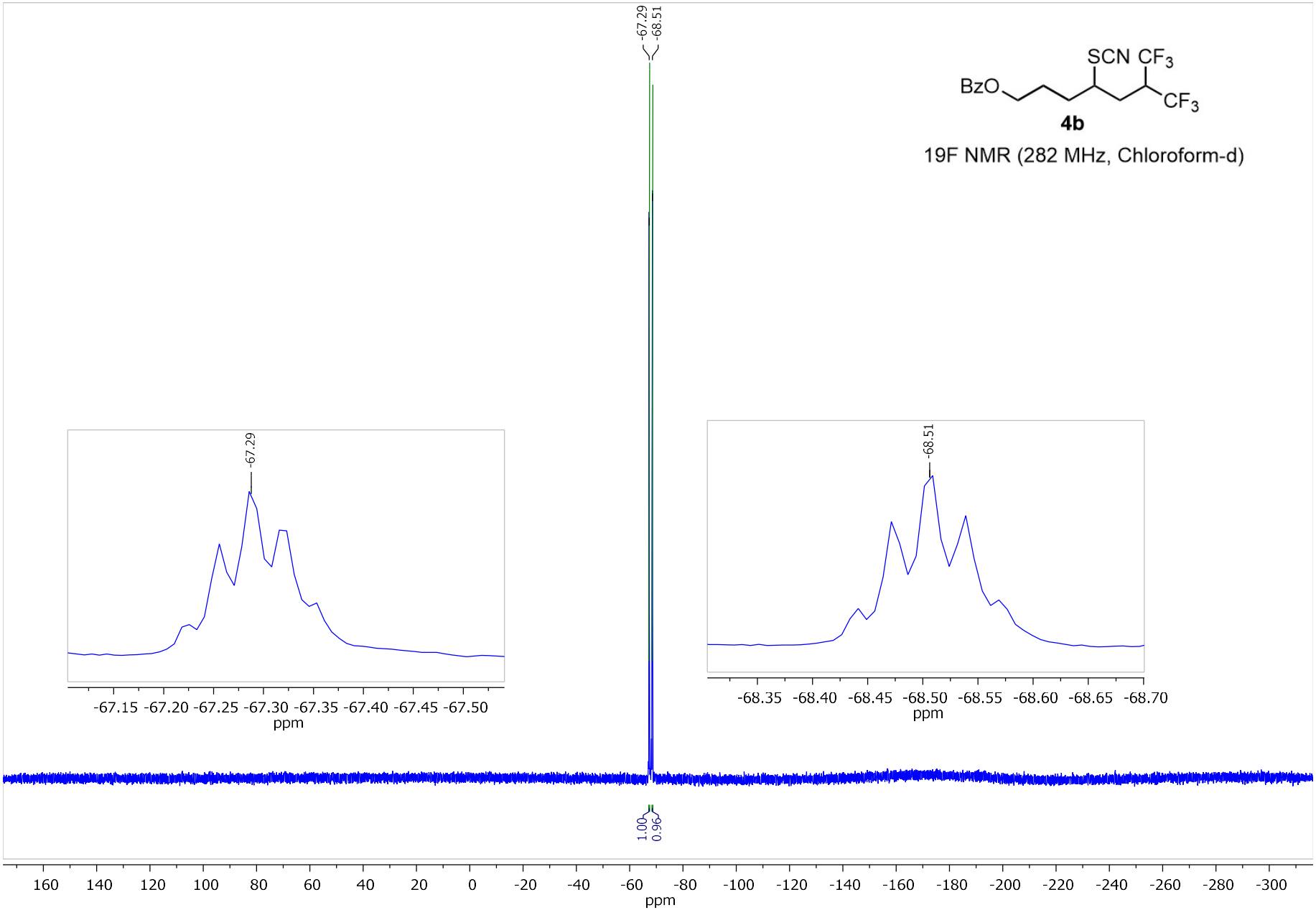

7.36
7.23
3.29
2.99
2.84
2.21
SCN CF3
CF3
4a
1H NMR (300 MHz, Chloroform-d)
ppm
5.12
1.00
1.97
1.01
3.91

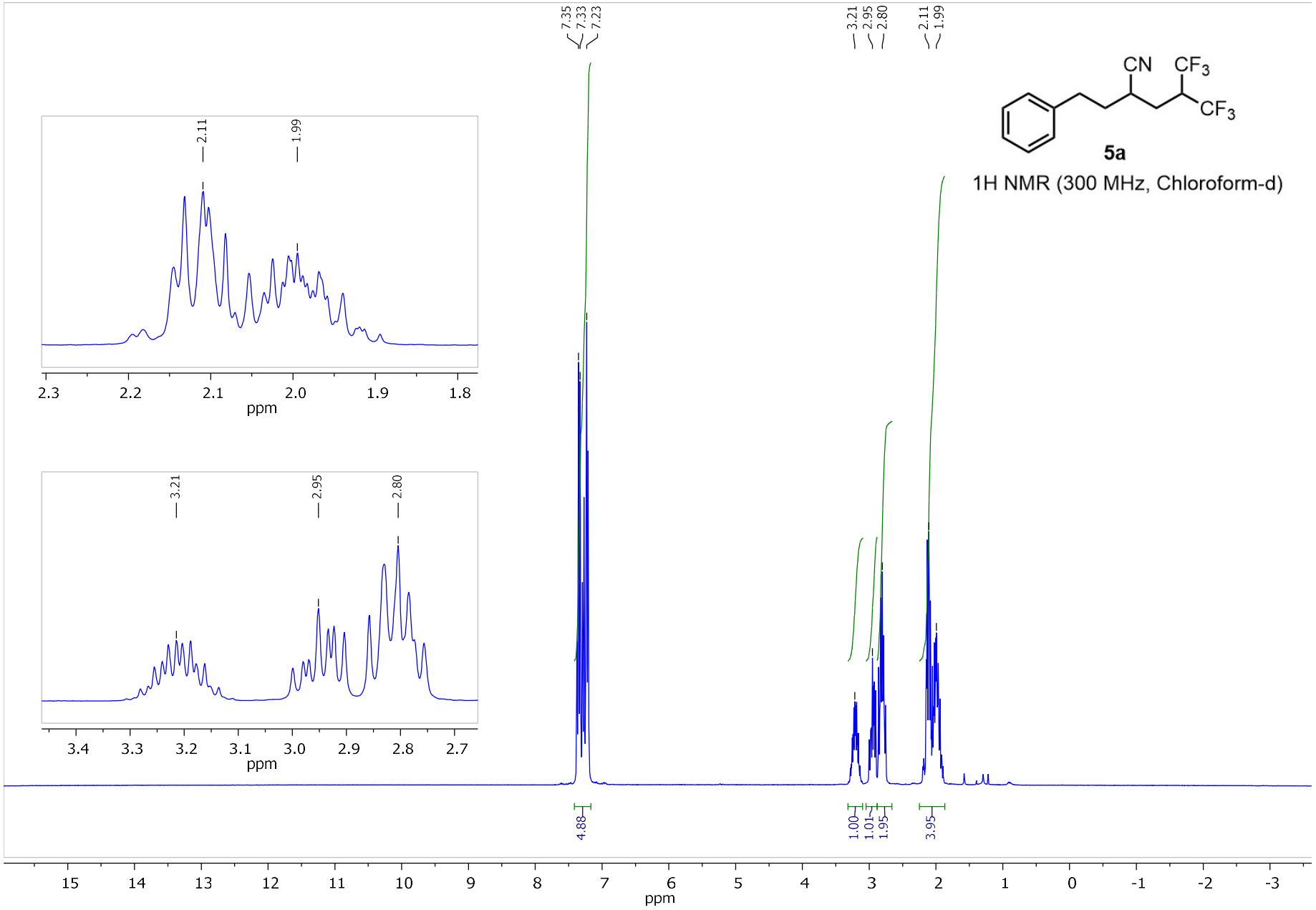

139.30
129.29
128.94
128.44
126.85
125.54
125.17
121.82
121.46
118.10
117.74
108.44
47.34
47.32
47.09
46.83
46.45
46.08
45.70
45.33
44.95
37.26
32.96
29.94
29.92
29.90
SCN CF3
CF3
4a
13C NMR (76 MHz, Chloroform-d)
ppm

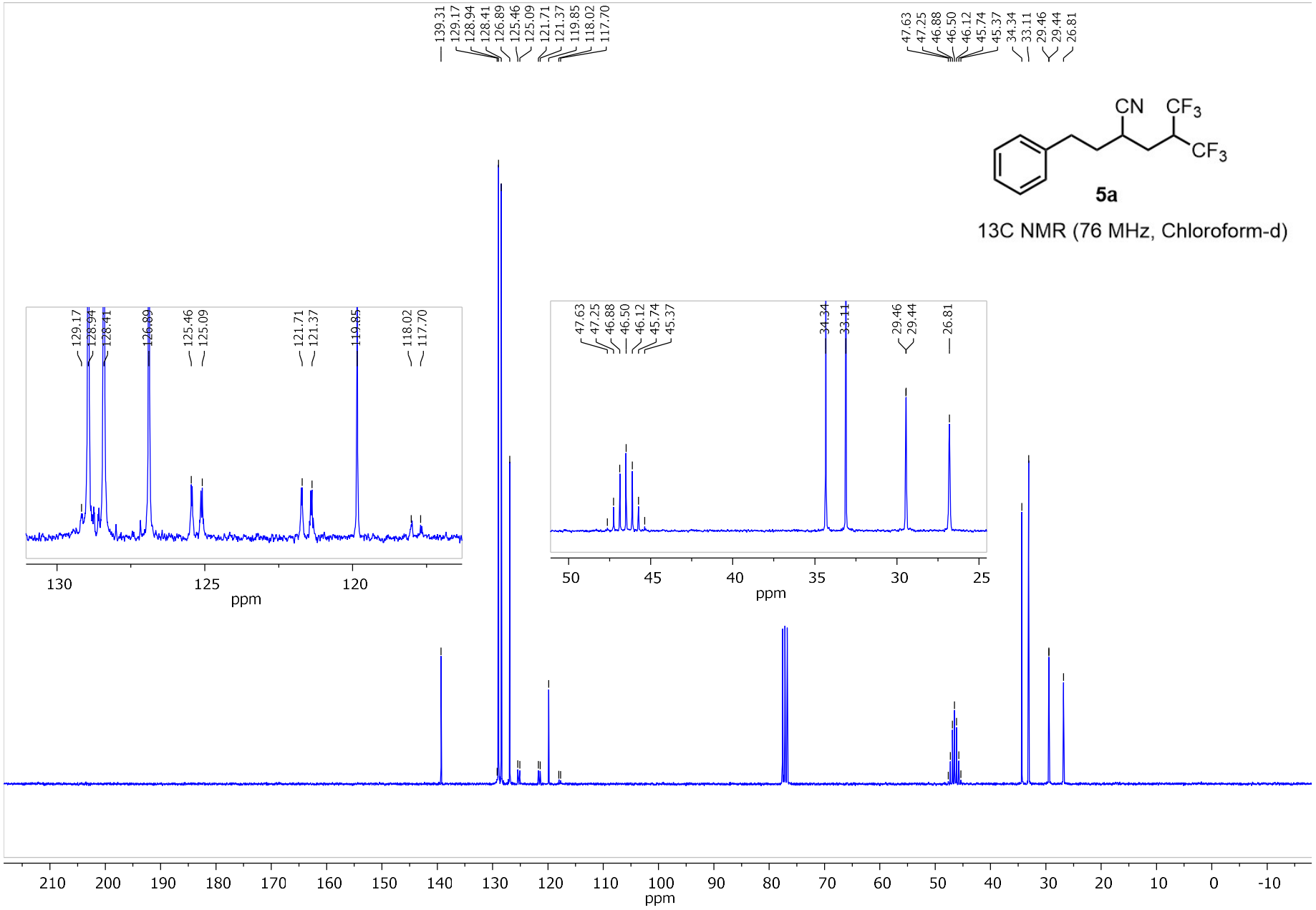

-66.42
-67.55
SCN CF3
CF3
4a
19F NMR (282 MHz, Chloroform-d)
-66.42
-66.30 -66.35 -66.40 -66.45 -66.50 -66.55
ppm
-67.55
-67.45 -67.50 -67.55 -67.60 -67.65 -67.70
ppm
1.00
1.00
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -22
ppm


8.03
7.58
7.46
4.38
3.96
3.18
2.07
1.88
SCN CF3
BzO
CF3
4b
1H NMR (300 MHz, Chloroform-d)
2.06
1.08
2.06
2.00
0.83
0.89
5.72
ppm


166.62
135.61
133.30
130.05
129.72
128.59
125.62
125.31
121.90
121.60
63.84
56.10
56.08
46.34
45.96
45.59
45.21
44.83
33.72
30.40
25.55
SCN CF3
BzO
CF3
4b
13C NMR (76 MHz, Chloroform-d)
135.48
133.17
129.92
129.59
128.47
125.49
125.19
121.77
121.48
140 138 136 134 132 130 128 126 124 122 120
ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm


-67.29
-68.51
SCN CF3
BzO
CF3
4b
19F NMR (282 MHz, Chloroform-d)
-67.29
-67.15 -67.20 -67.25 -67.30 -67.35 -67.40 -67.45 -67.50
ppm
-68.51
-68.35 -68.40 -68.45 -68.50 -68.55 -68.60 -68.65 -68.70
ppm
1.00
0.96
160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280 -300
ppm


7.35
7.33
7.23
3.21
2.95
2.80
2.11
1.99
CN
CF3
CF3
5a
1H NMR (300 MHz, Chloroform-d)
2.11
1.99
3.21
2.95
2.80
ppm
4.88
1.00
1.01
1.95
3.95
ppm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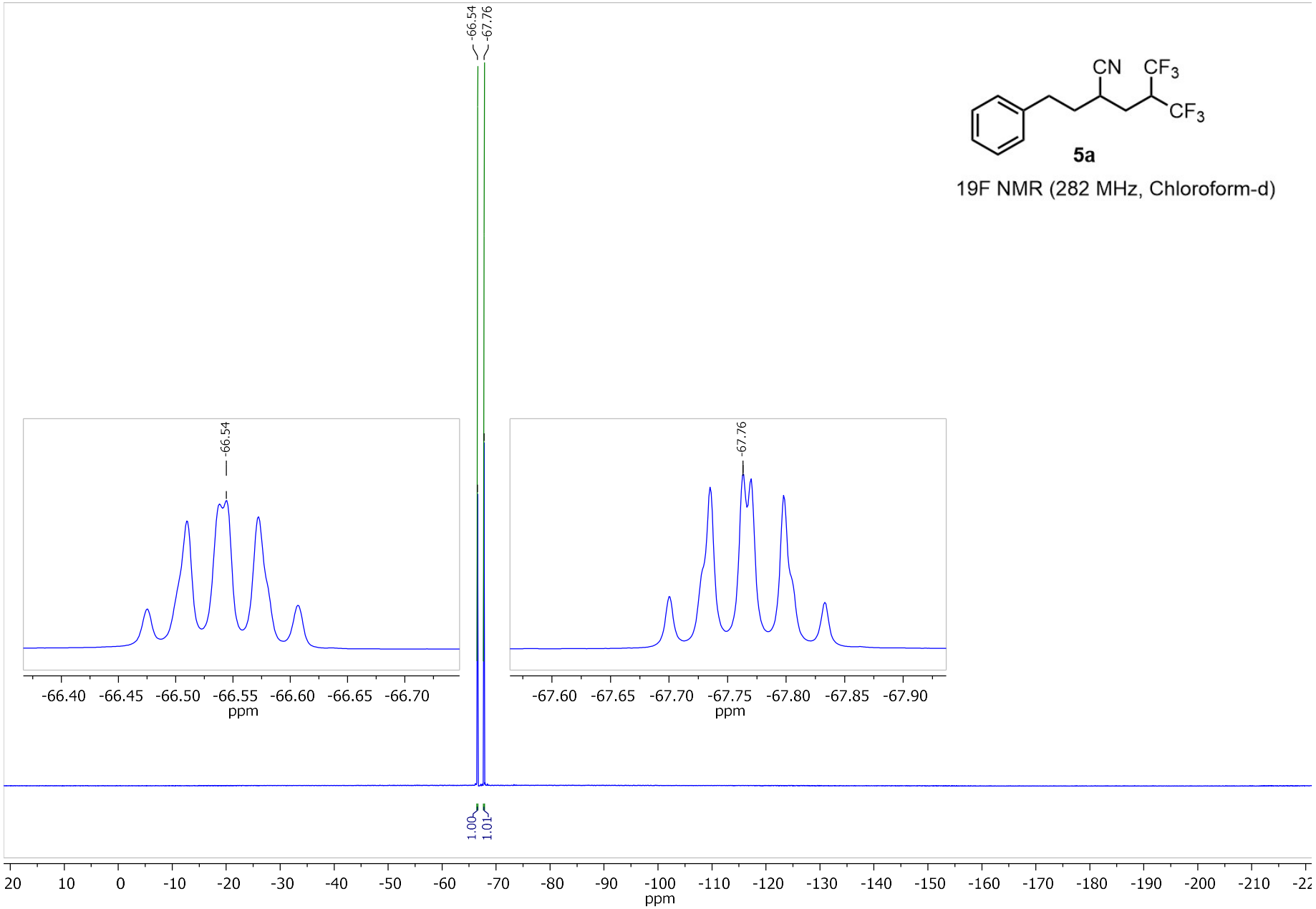
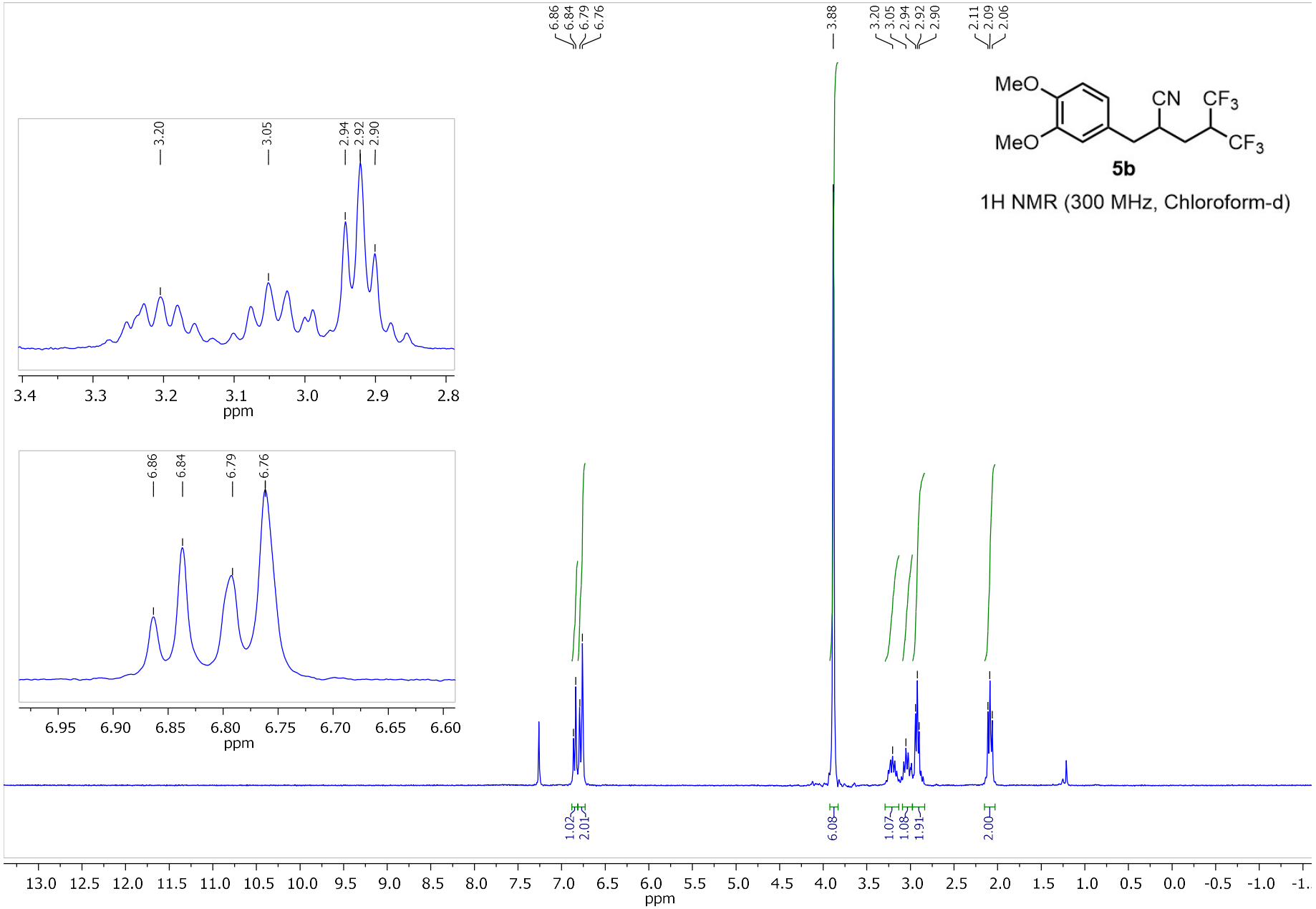
128.94
128.41
126.89
125.46
125.09
121.71
121.37
119.85
118.02
117.70
47.63
47.25
46.88
46.50
46.12
45.74
45.37
34.34
33.11
29.46
29.44
26.81
CN
CF3
CF3
5a
13C NMR (76 MHz, Chloroform-d)
ppm
ppm
ppm


-66.54
-67.76
CN
CF3
CF3
5a
19F NMR (282 MHz, Chloroform-d)
-66.54
-66.40 -66.45 -66.50 -66.55 -66.60 -66.65 -66.70
ppm
-67.76
-67.60 -67.65 -67.70 -67.75 -67.80 -67.85 -67.90
ppm
1.00
1.01
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -22
ppm


6.86
6.84
6.79
6.76
3.88
3.20
3.05
2.94
2.92
2.90
2.11
2.09
2.06
MeO
MeO
CN
CF3
CF3
5b
1H NMR (300 MHz, Chloroform-d)
3.20
3.05
2.94
2.92
2.90
3.4
3.3
3.2
3.1
3.0
2.9
2.8
ppm
6.86
6.84
6.79
6.76
6.95
6.90
6.85
6.80
6.75
6.70
6.65
6.60
ppm
1.02
2.01
6.08
1.07
1.08
1.91
2.00
13.0
12.5
12.0
11.5
11.0
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
1.5
1.0
0.5
0.0
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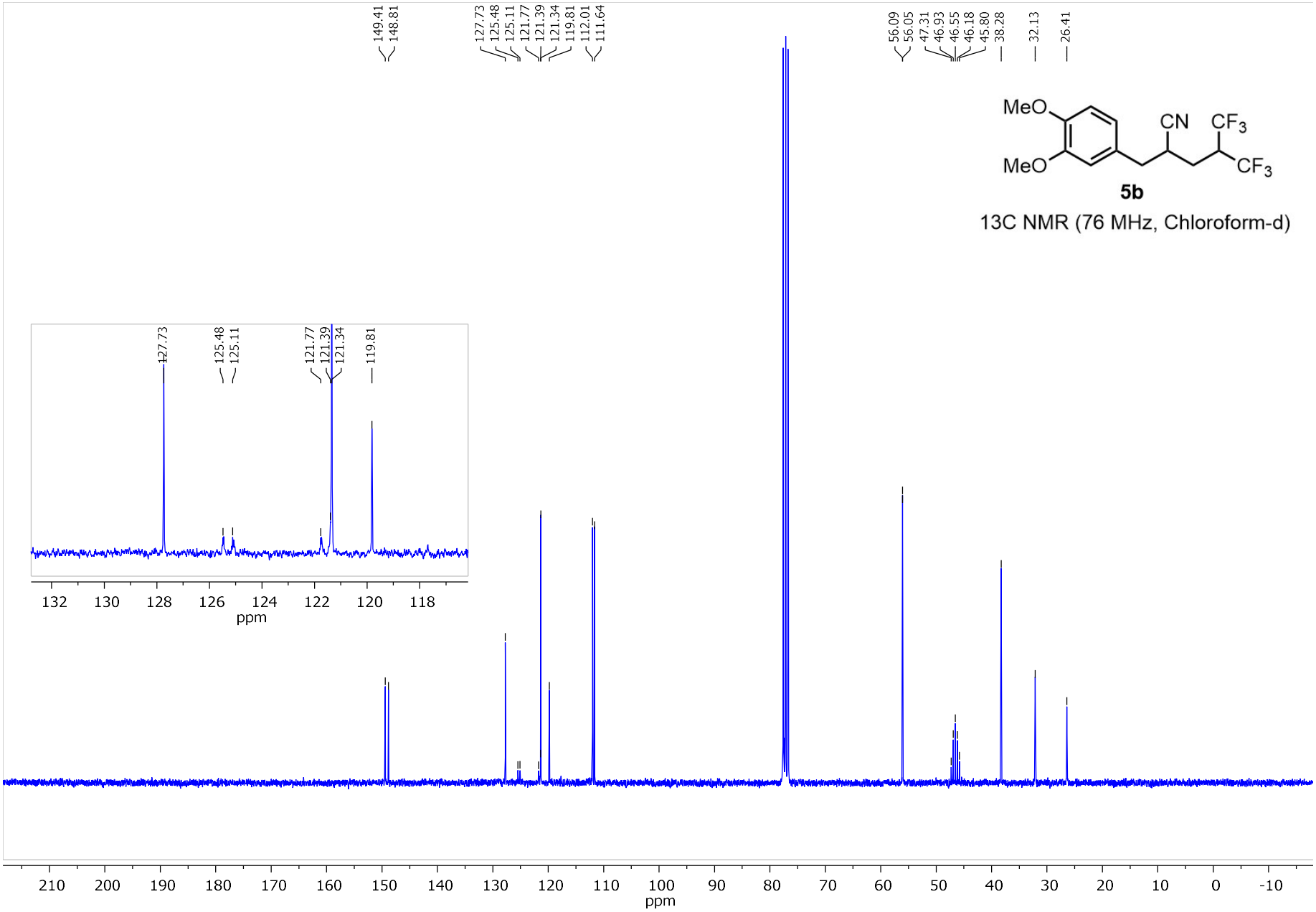
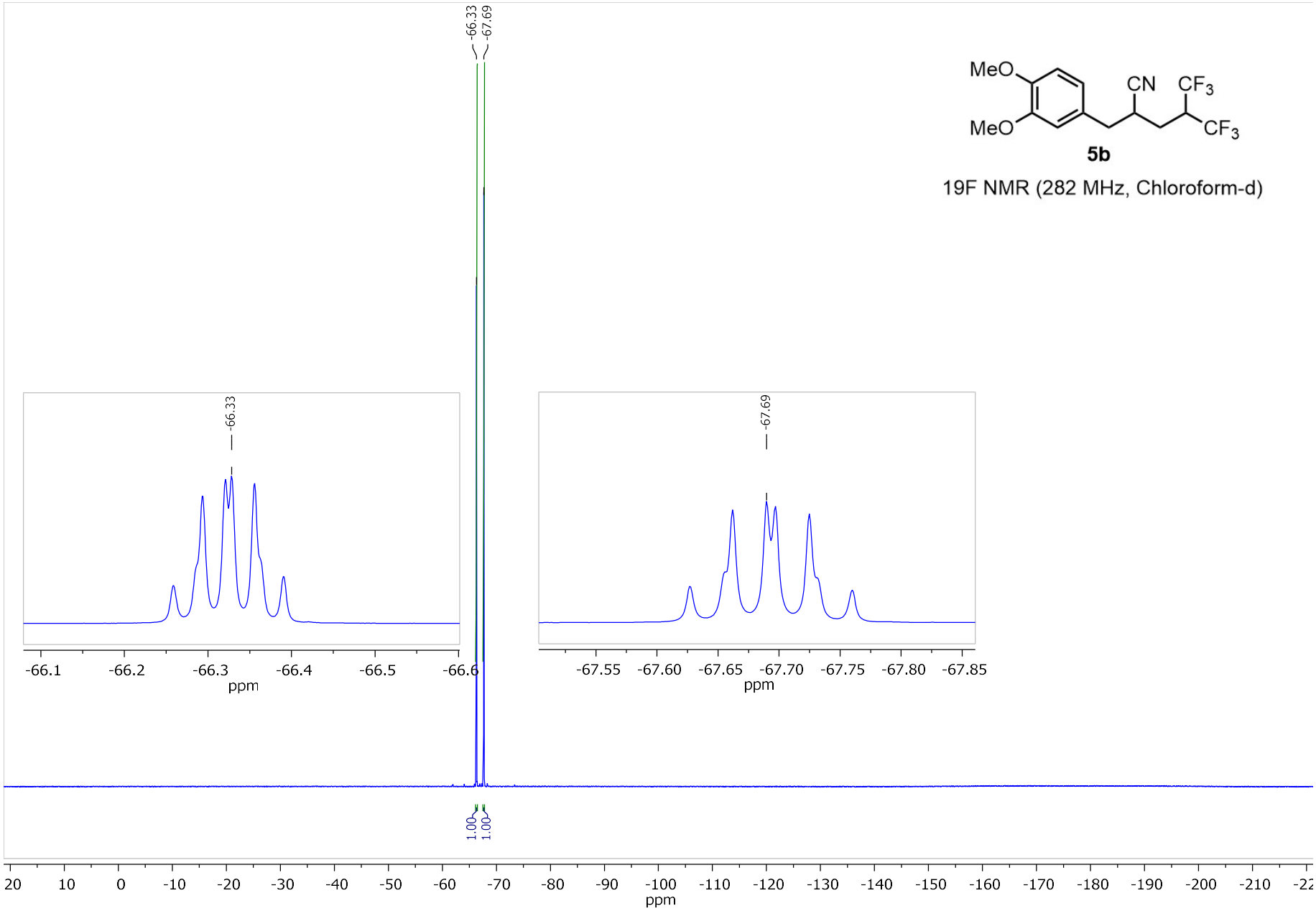
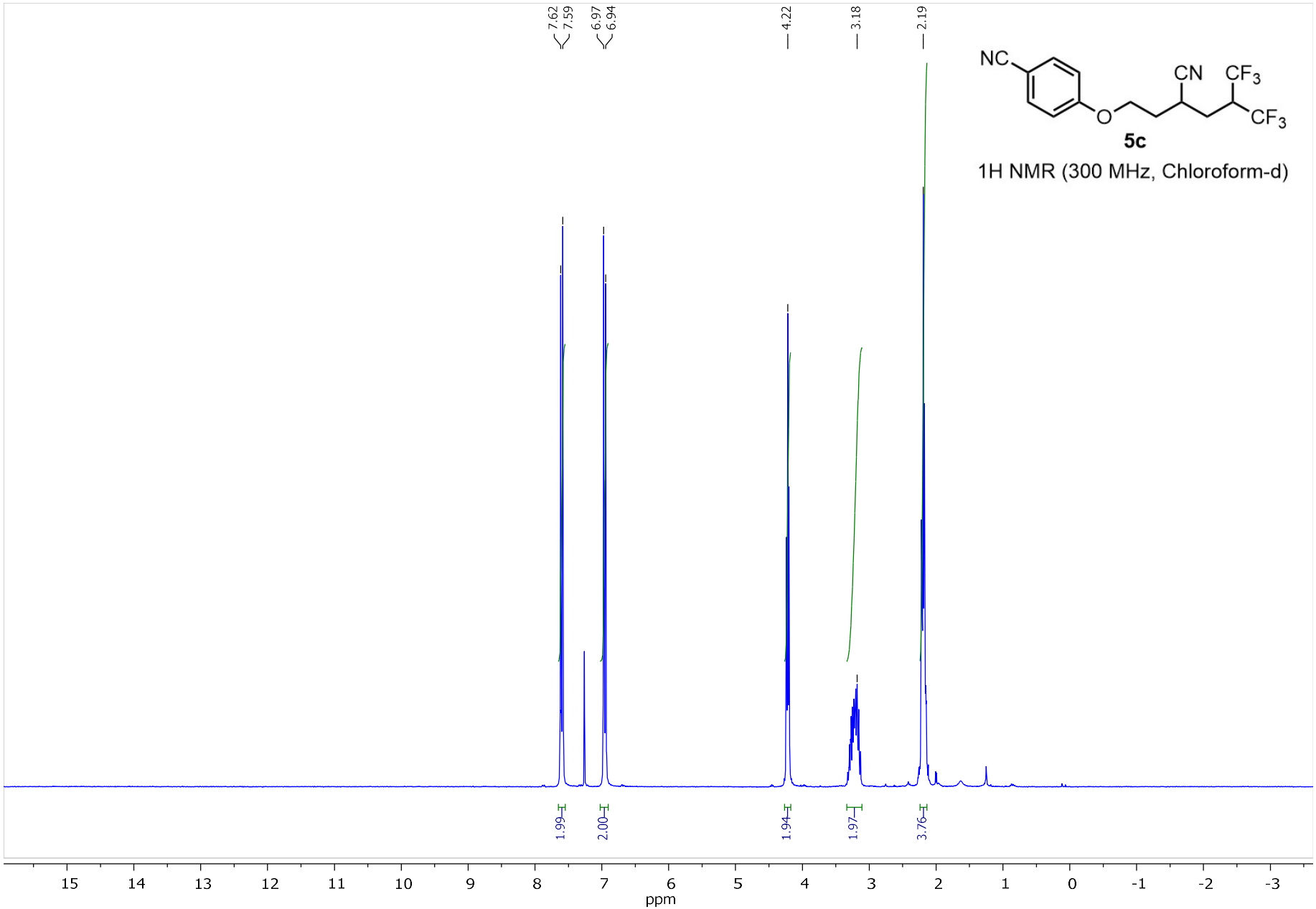
-1.0
ppm


149.41
148.81
127.73
125.48
125.11
121.77
121.39
121.34
119.81
112.01
111.64
56.09
56.05
47.31
46.93
46.55
46.18
45.80
38.28
32.13
26.41
MeO
MeO
CN
CF3
CF3
5b
13C NMR (76 MHz, Chloroform-d)
ppm
ppm


-66.33
-67.69
MeO
MeO
CN
CF3
CF3
5b
19F NMR (282 MHz, Chloroform-d)
-66.33
-67.69
-66.1
-66.2
-66.3
-66.4
-66.5
-66.6
ppm
-67.55
-67.60
-67.65
-67.70
-67.75
-67.80
-67.85
ppm
1.00
1.00
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
ppm


7.62
7.59
6.97
6.94
4.22
3.18
2.19
NC
CN
CF3
CF3
O
5c
1H NMR (300 MHz, Chloroform-d)
1.99
2.00
1.94
1.97
3.76
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
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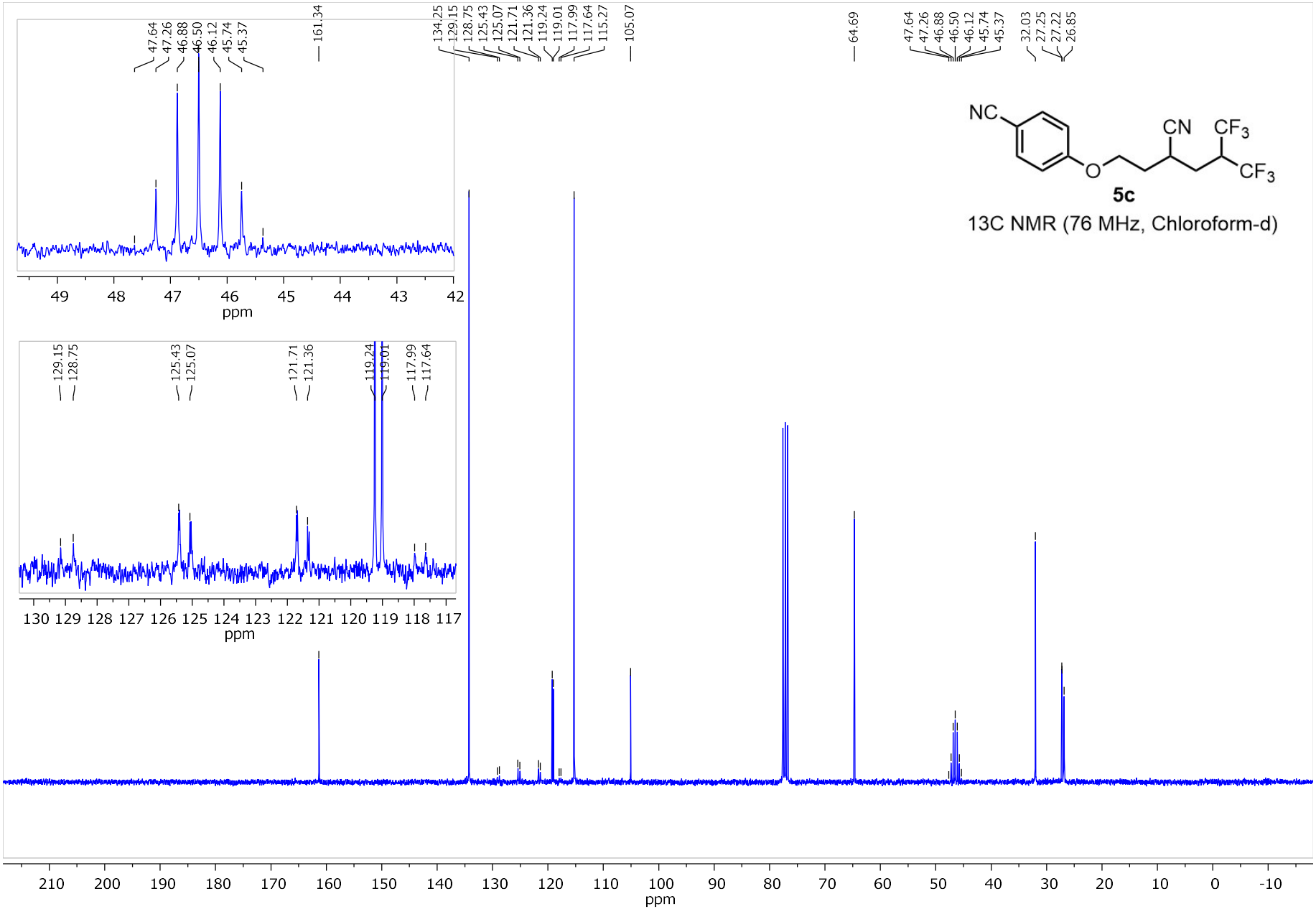
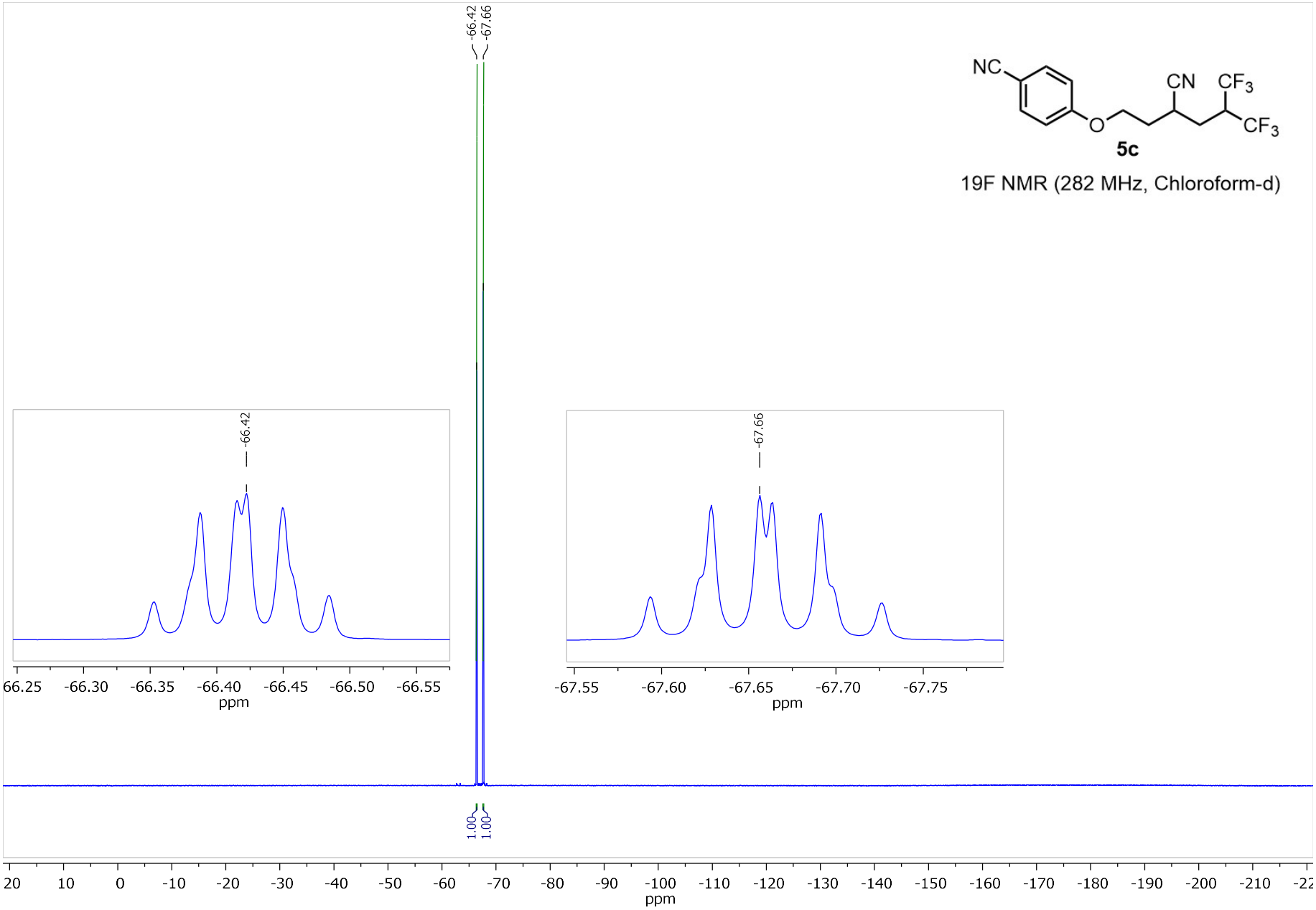
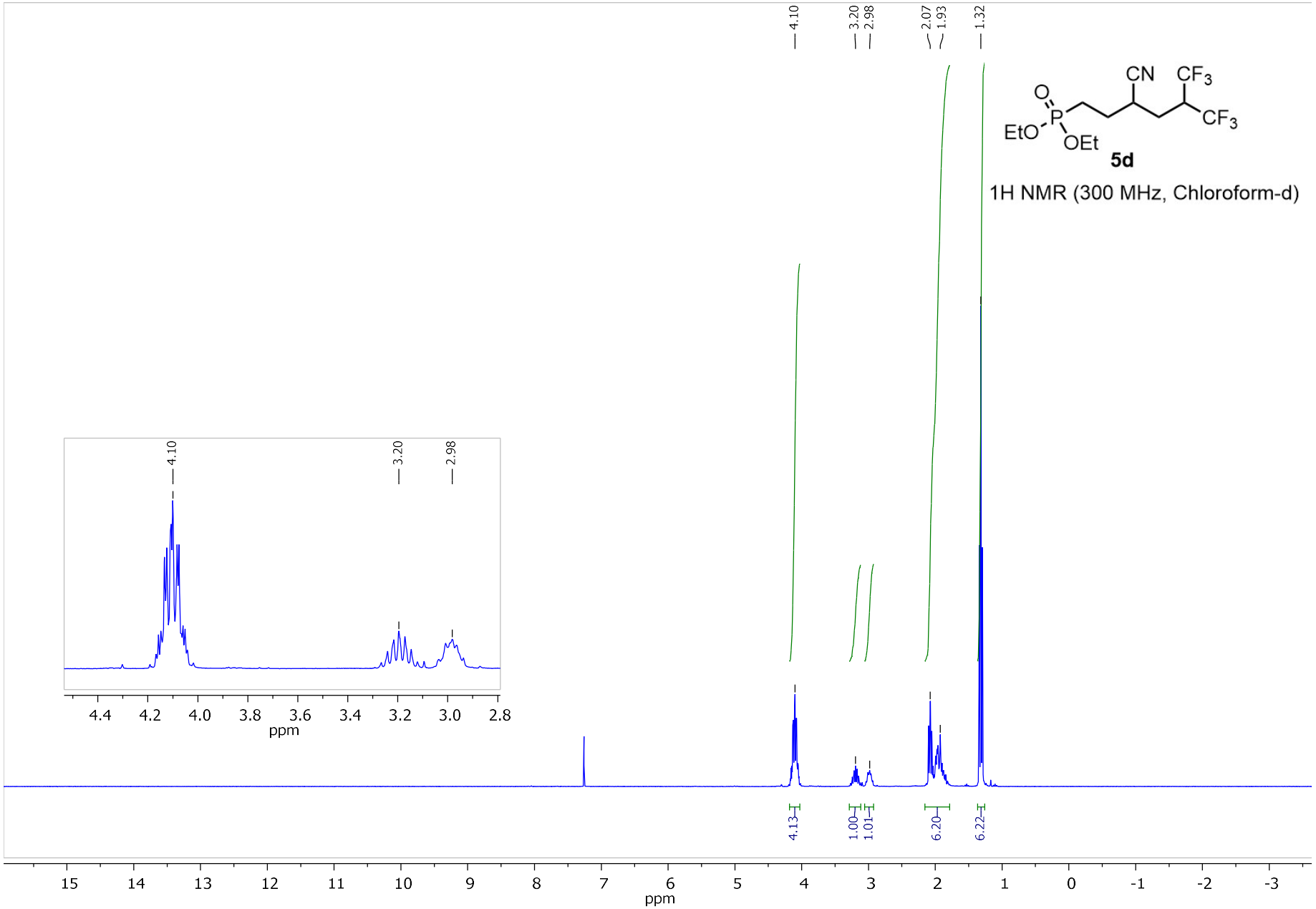
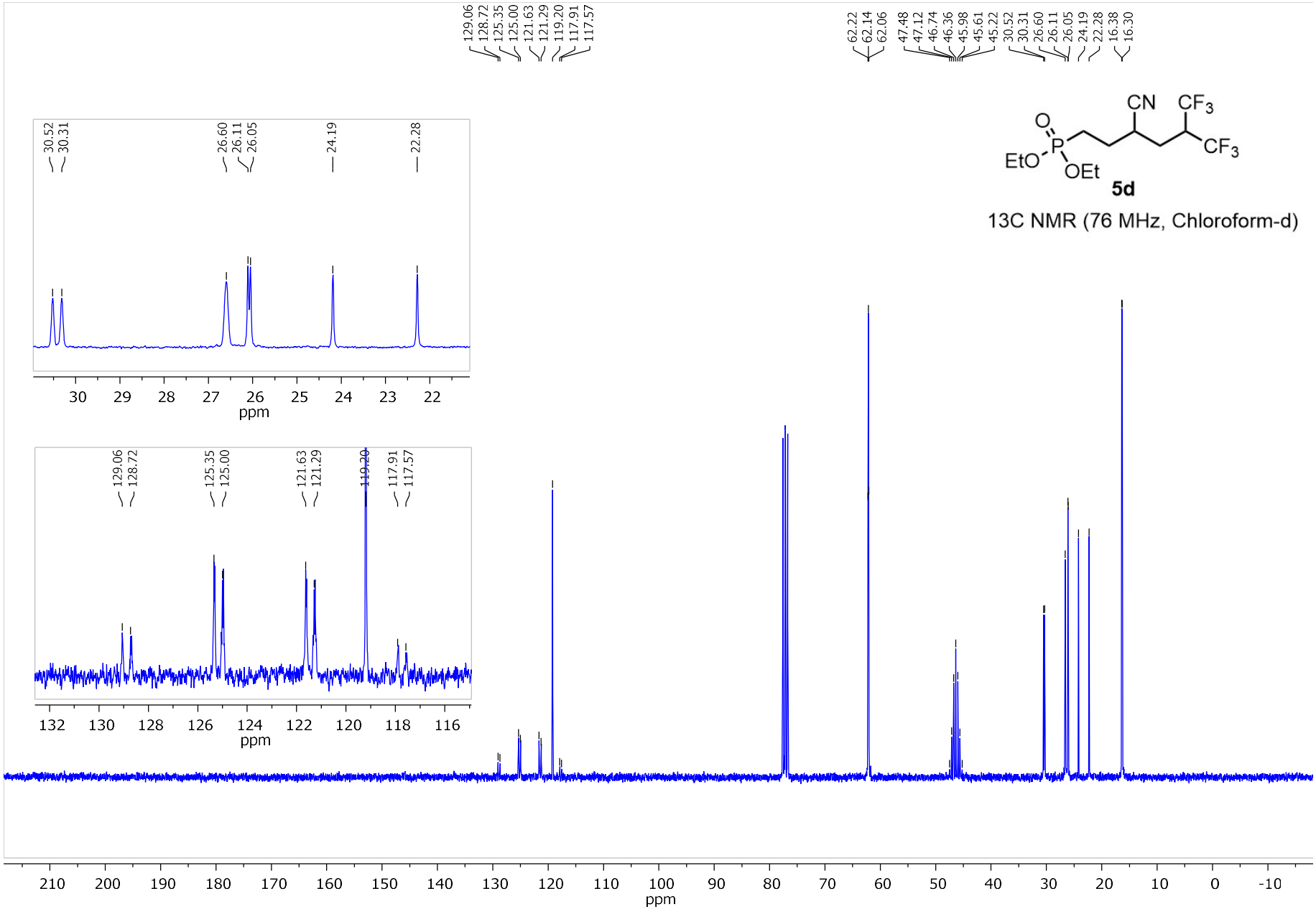
-1
-2
-3
ppm


47.64
47.26
46.88
46.50
46.12
45.74
45.37
161.34
134.25
129.15
128.75
125.43
125.07
121.71
121.36
119.24
119.01
117.99
117.64
115.27
105.07
64.69
32.03
27.25
27.22
26.85
NC
CN
CF3
CF3
O
5c
13C NMR (76 MHz, Chloroform-d)
ppm
ppm


-66.42
-67.66
NC
CN
CF3
CF3
O
5c
19F NMR (282 MHz, Chloroform-d)
-66.42
-67.66
66.25 -66.30 -66.35 -66.40 -66.45 -66.50 -66.55
ppm
-67.55 -67.60 -67.65 -67.70 -67.75
ppm
1.00
1.00
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -22
ppm


4.10
3.20
2.98
2.07
1.93
1.32
CN
CF3
O
P
EtO
OEt
CF3
5d
1H NMR (300 MHz, Chloroform-d)
4.13
1.00
1.01
6.20
6.22
ppm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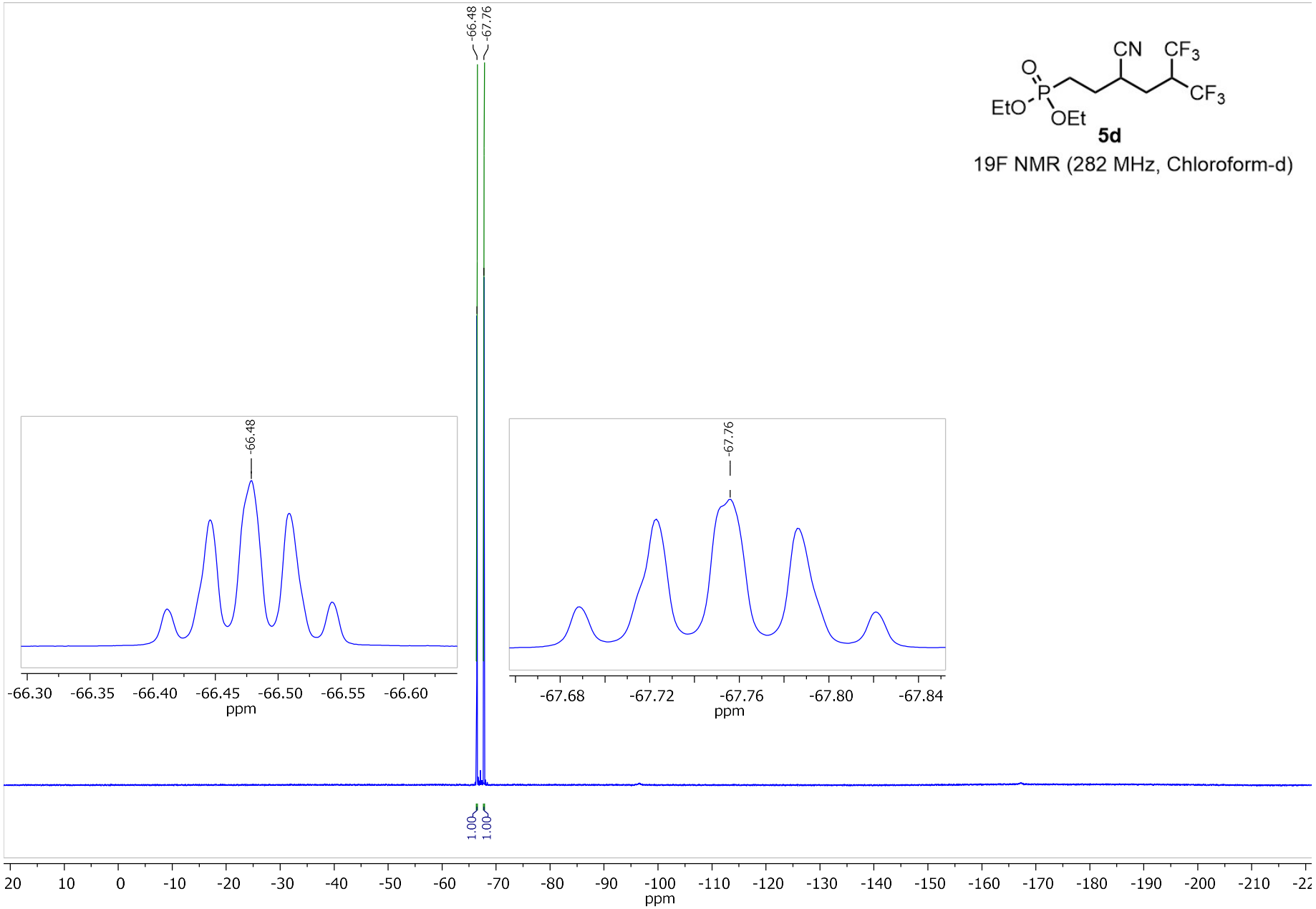
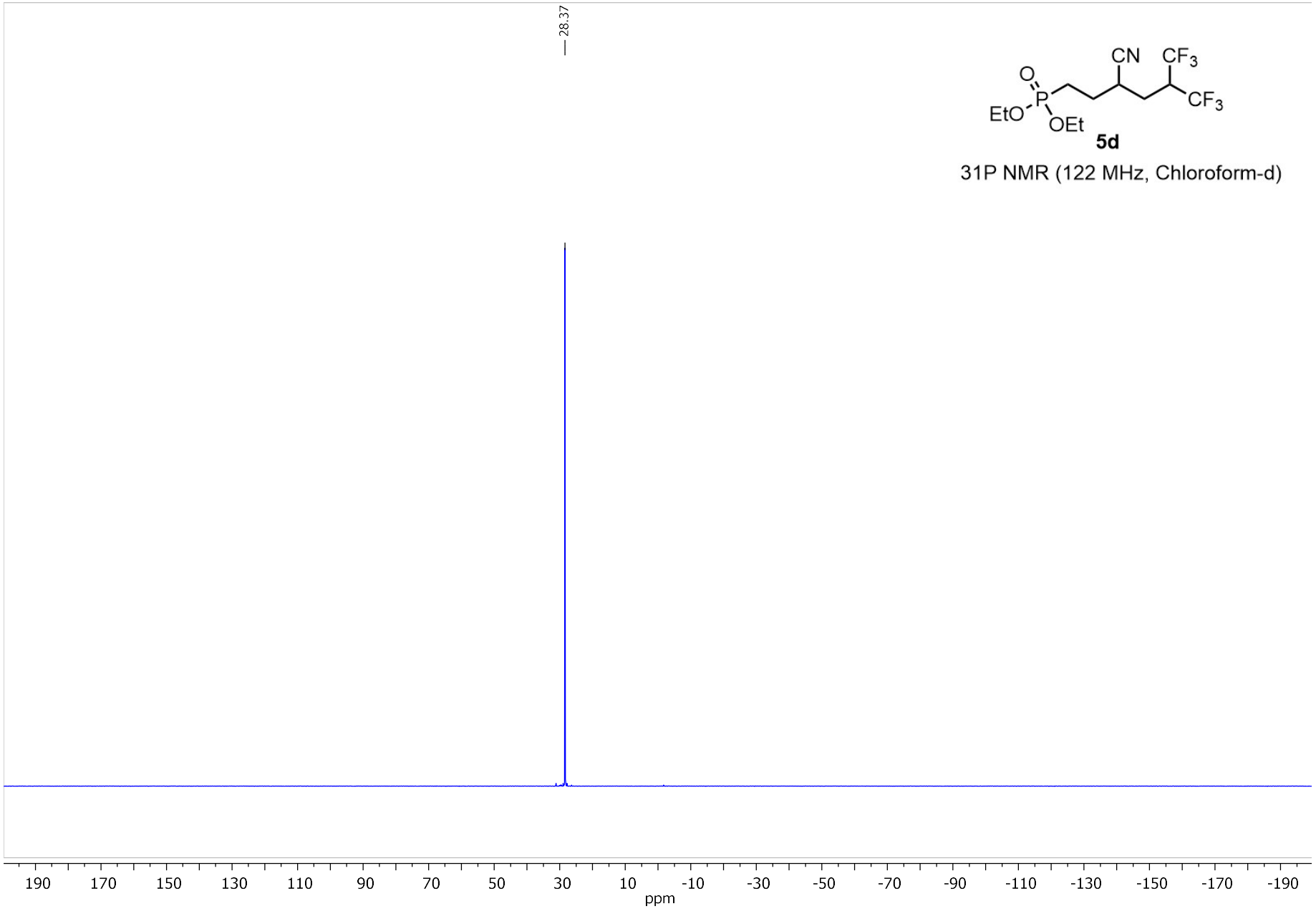
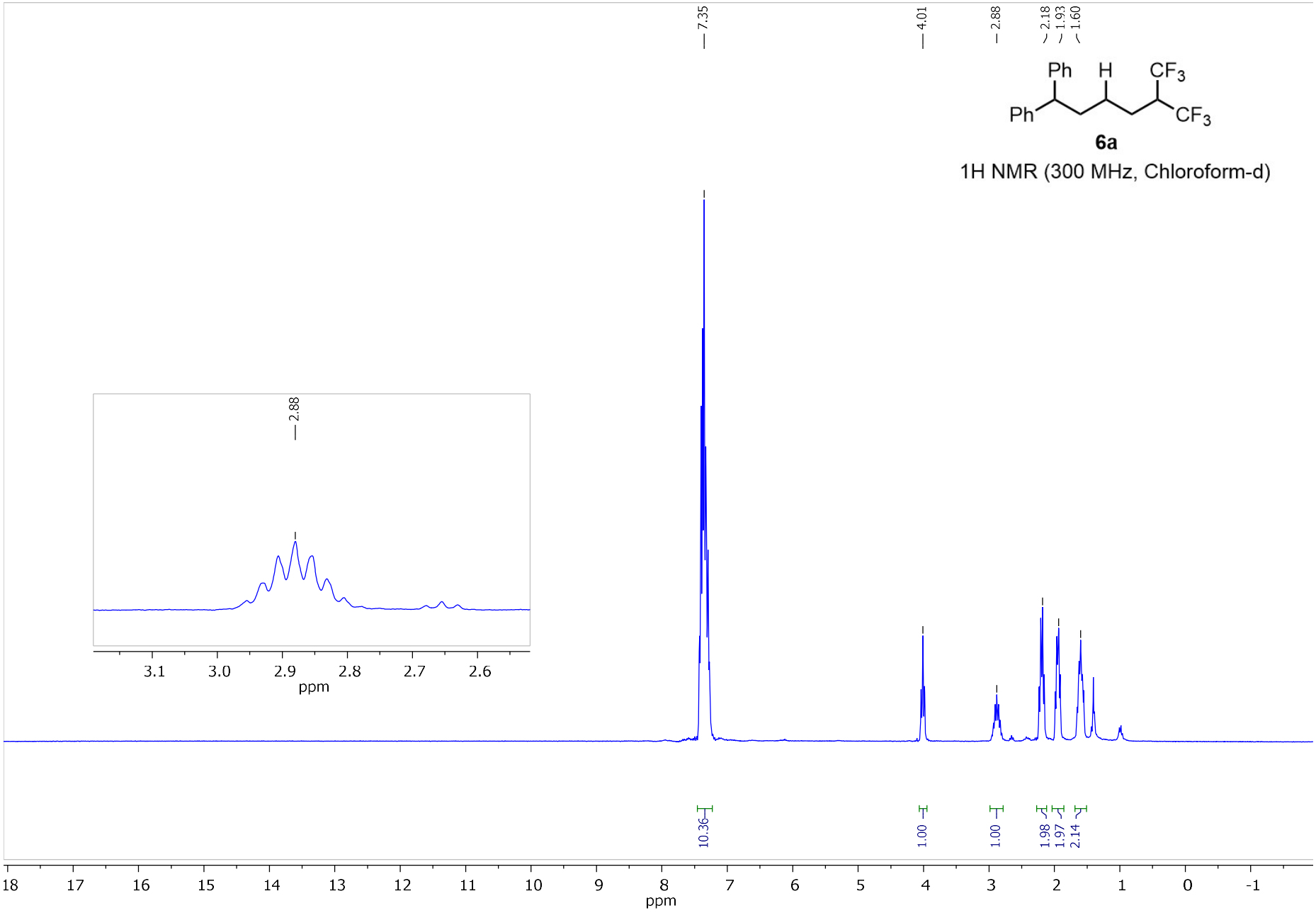
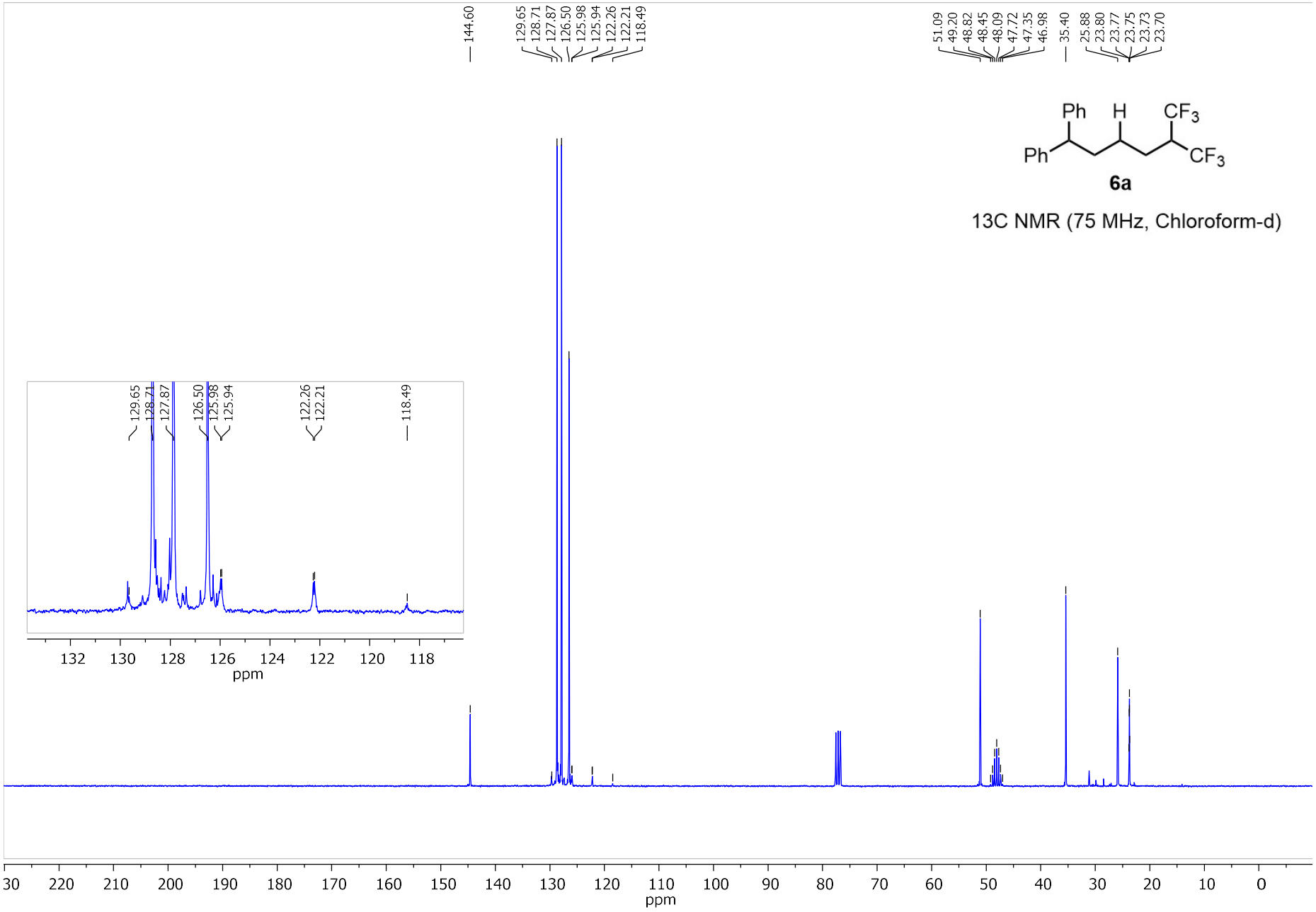
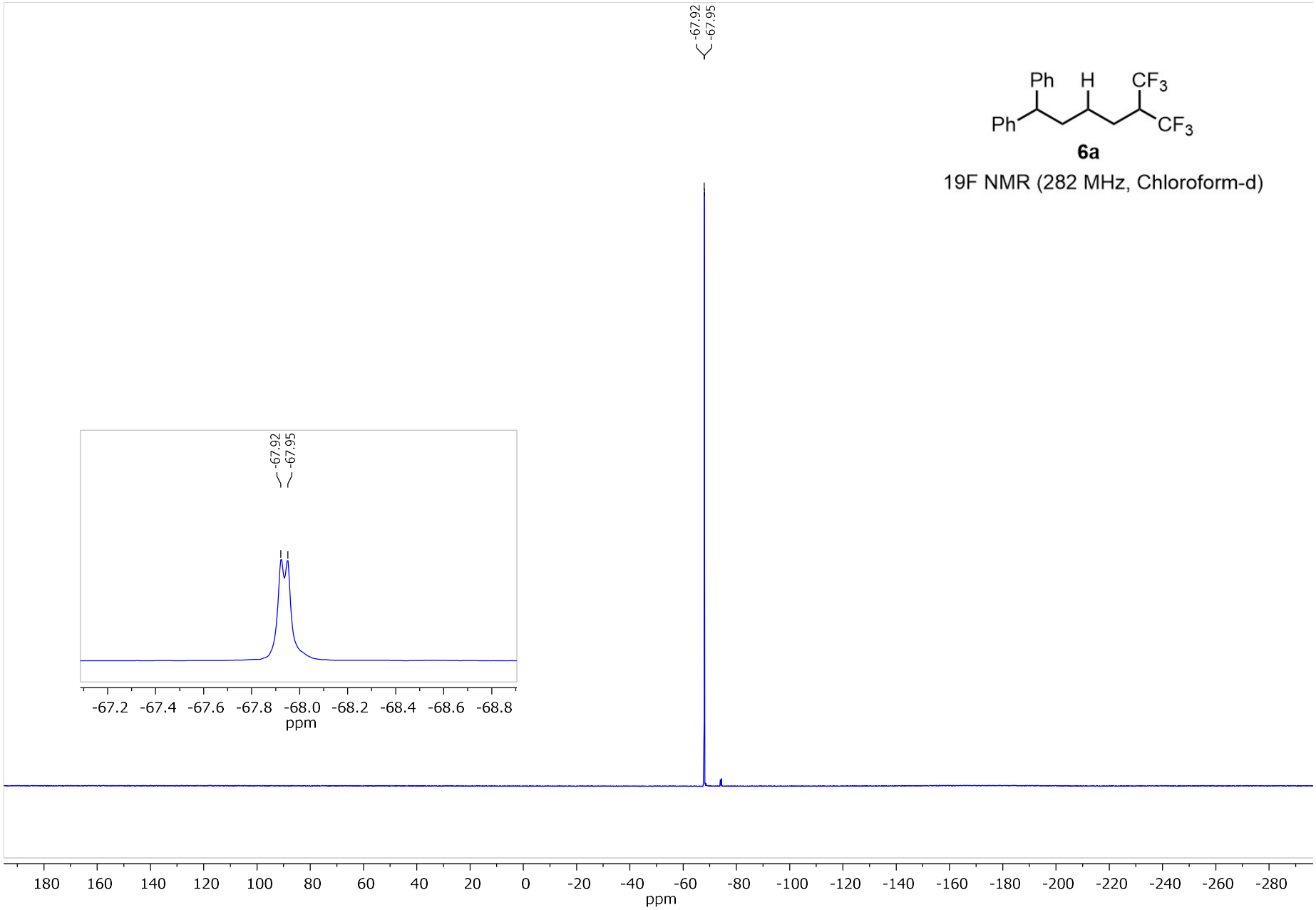
128.72
125.35
125.00
121.63
121.29
119.20
117.91
117.57
62.22
62.14
62.06
47.48
47.12
46.74
46.36
45.98
45.61
45.22
30.52
30.31
26.60
26.11
26.05
24.19
22.28
16.38
16.30
CN
CF3
CF3
O
P
EtO
OEt
5d
13C NMR (76 MHz, Chloroform-d)
ppm
ppm

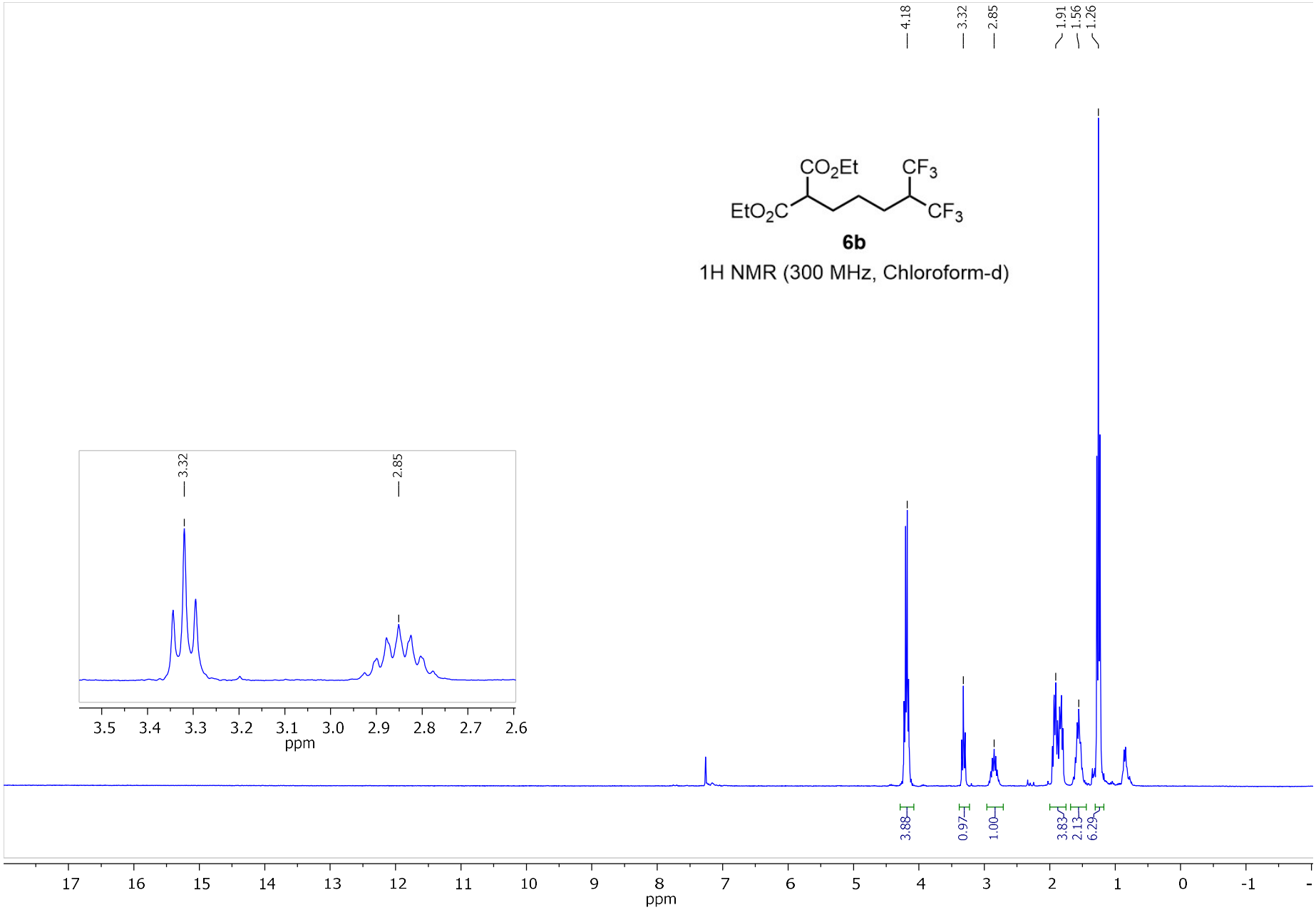

-66.48
-67.76
CN
CF3
O
P
EtO
OEt
CF3
5d
19F NMR (282 MHz, Chloroform-d)
-66.48
-66.30
-66.35
-66.40
-66.45
-66.50
-66.55
-66.60
ppm
-67.76
-67.68
-67.72
-67.76
-67.80
-67.84
ppm
1.00
1.00
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
ppm

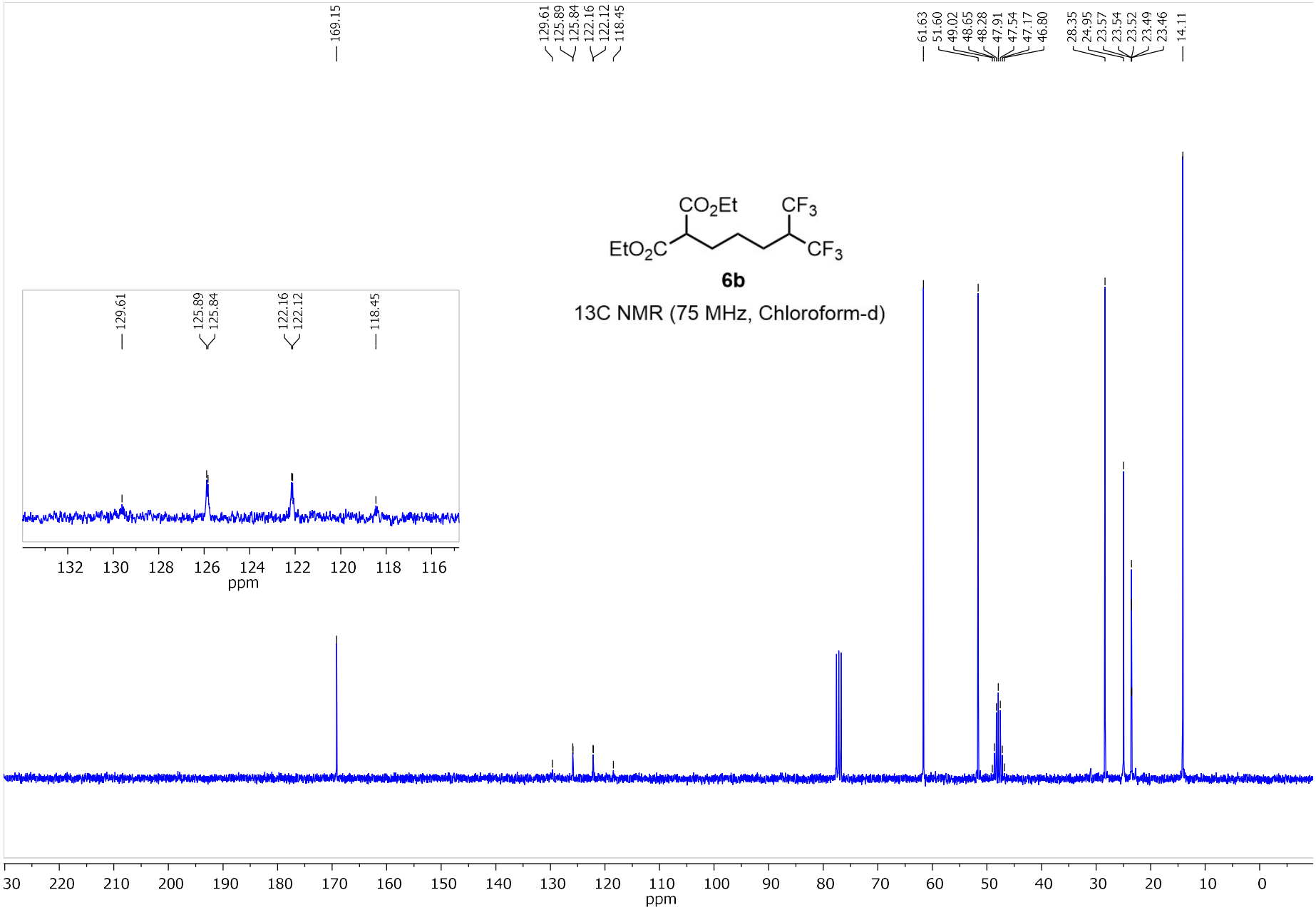

28.37
CN
CF3
O
P
EtO
OEt
CF3
5d
31P NMR (122 MHz, Chloroform-d)
190
170
150
130
110
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
ppm

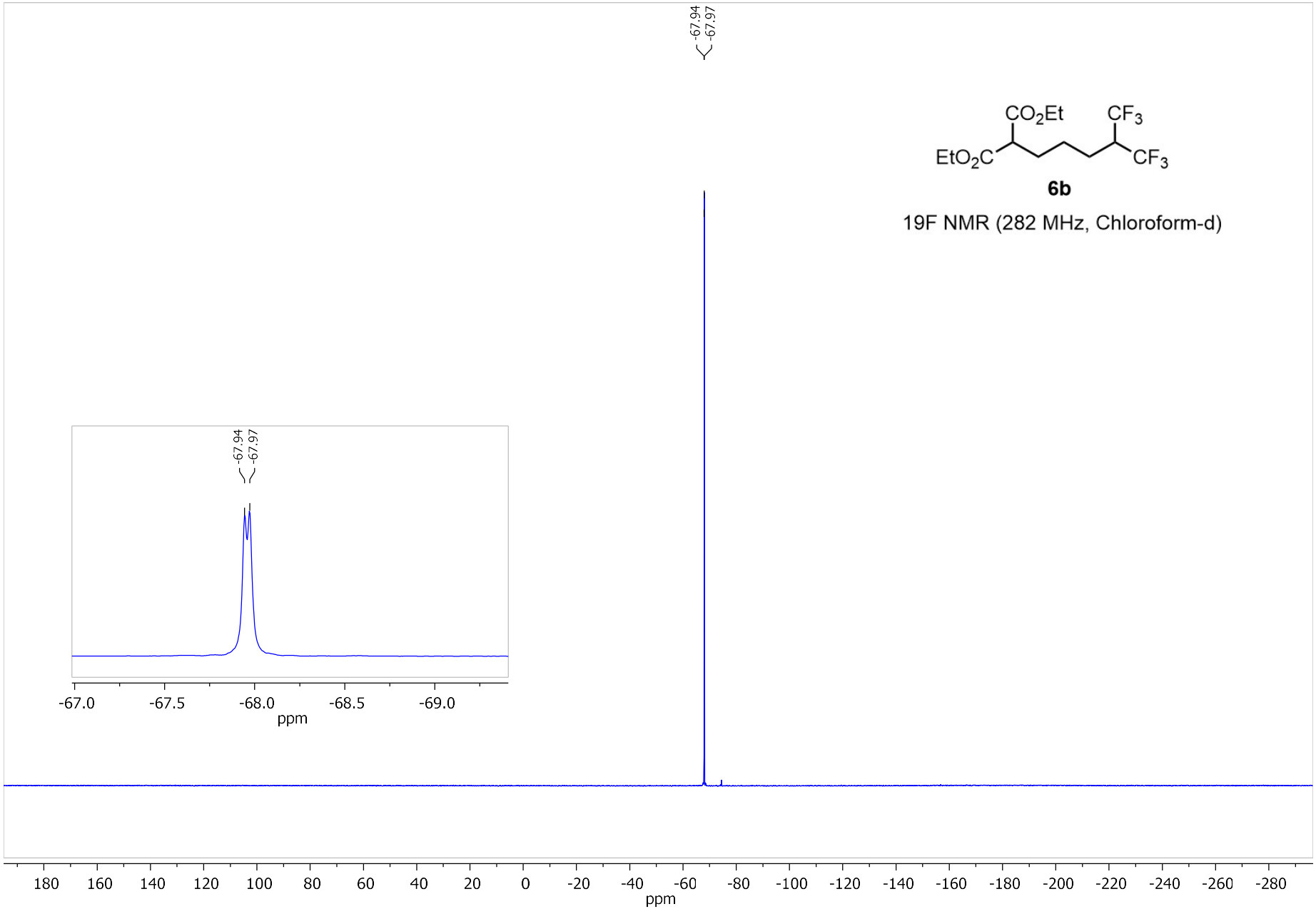

7.35
4.01
2.88
2.18
1.93
1.60
Ph
H
CF3
Ph
CF3
6a
1H NMR (300 MHz, Chloroform-d)
2.88
3.1
3.0
2.9
2.8
2.7
2.6
ppm
10.36
1.00
1.00
1.98
1.97
2.14
18
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

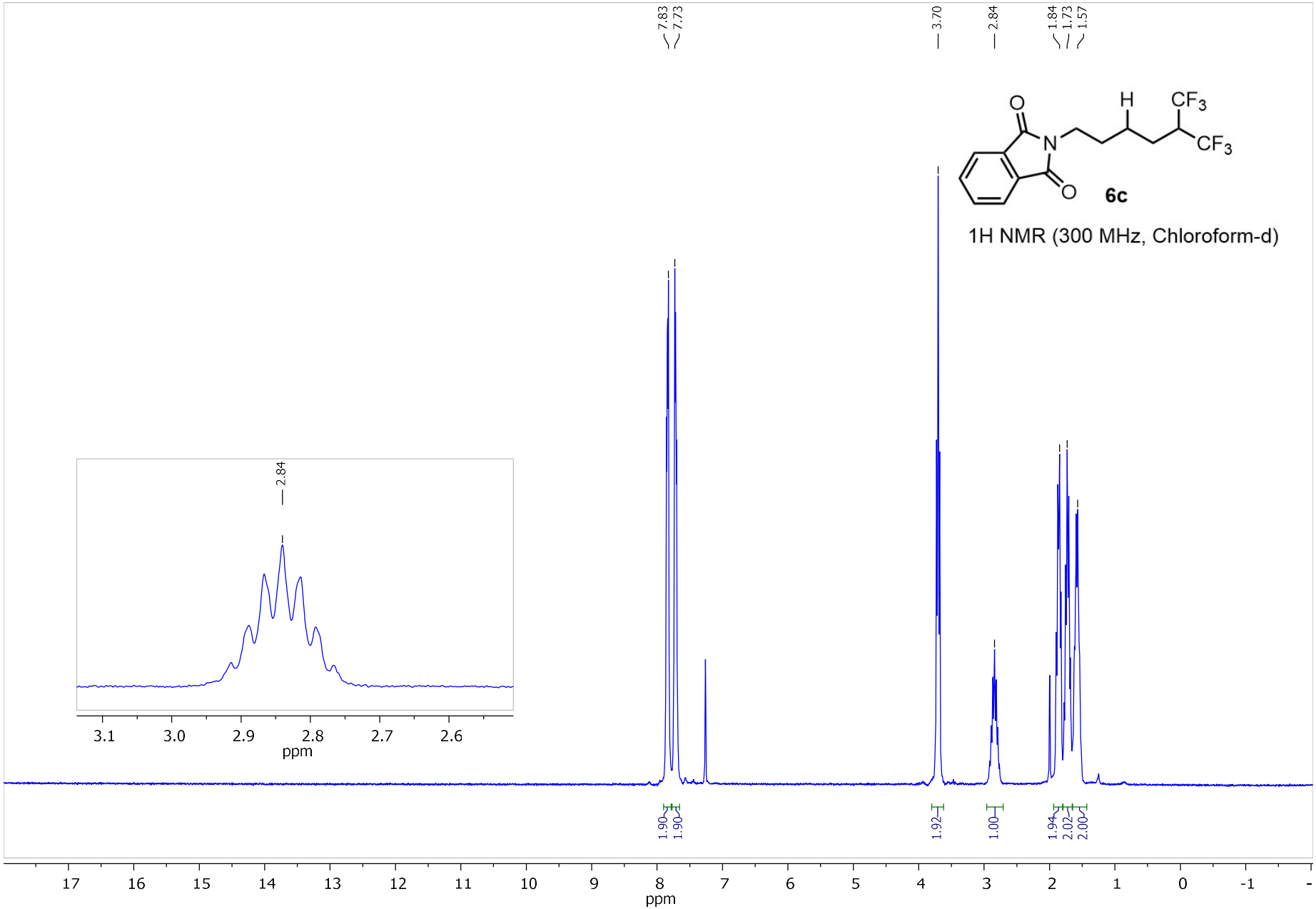

144.60
129.65
128.71
127.87
126.50
125.98
125.94
122.26
122.21
118.49
51.09
49.20
48.82
48.45
48.09
47.72
47.35
46.98
35.40
25.88
23.80
23.77
23.75
23.73
23.70
Ph
H
CF3
Ph
CF3
6a
13C NMR (75 MHz, Chloroform-d)
ppm
ppm

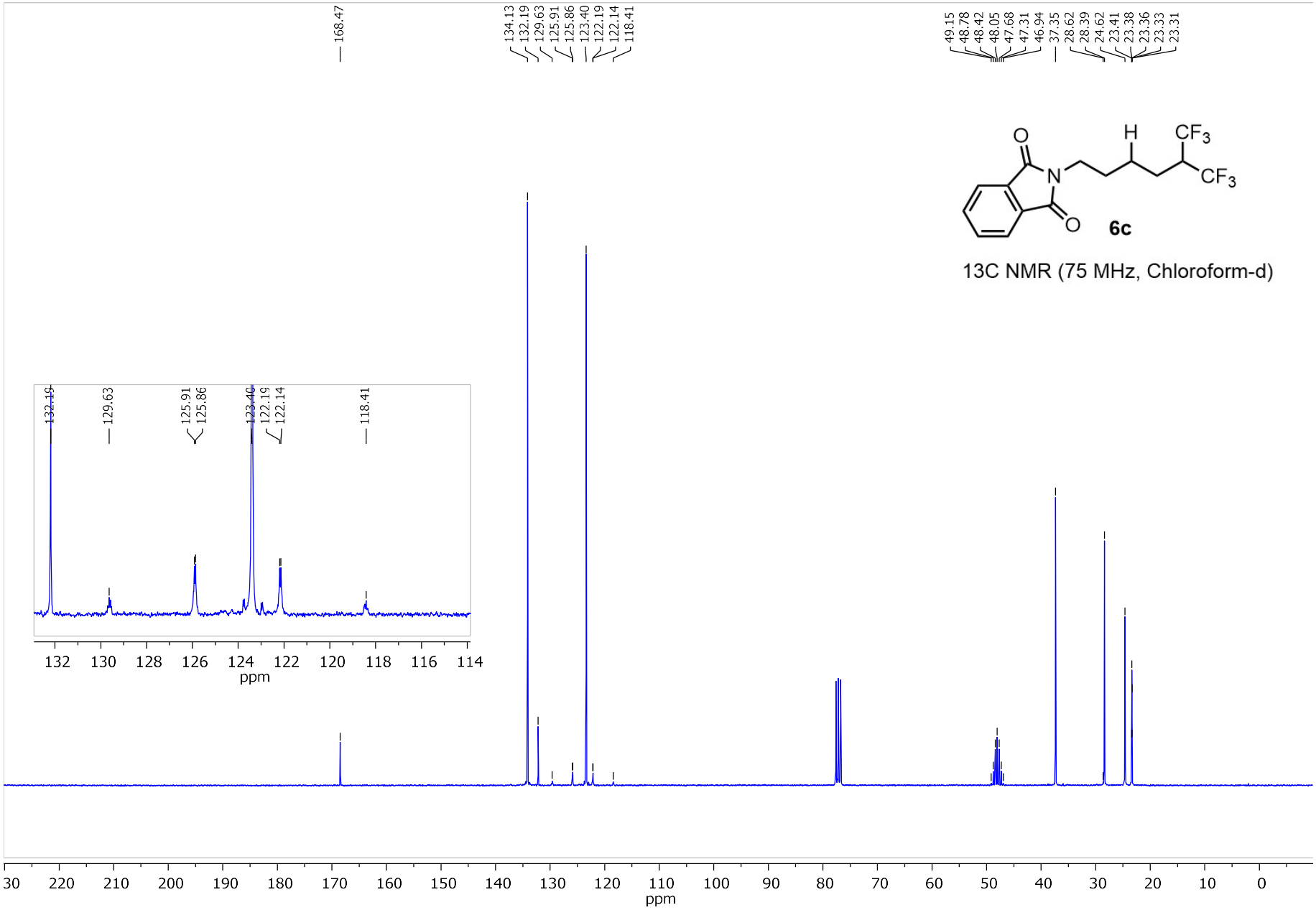

-67.92
-67.95
Ph
H
CF3
Ph
CF3
6a
19F NMR (282 MHz, Chloroform-d)
-67.92
-67.95
-67.2 -67.4 -67.6 -67.8 -68.0 -68.2 -68.4 -68.6 -68.8
ppm
180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280
ppm

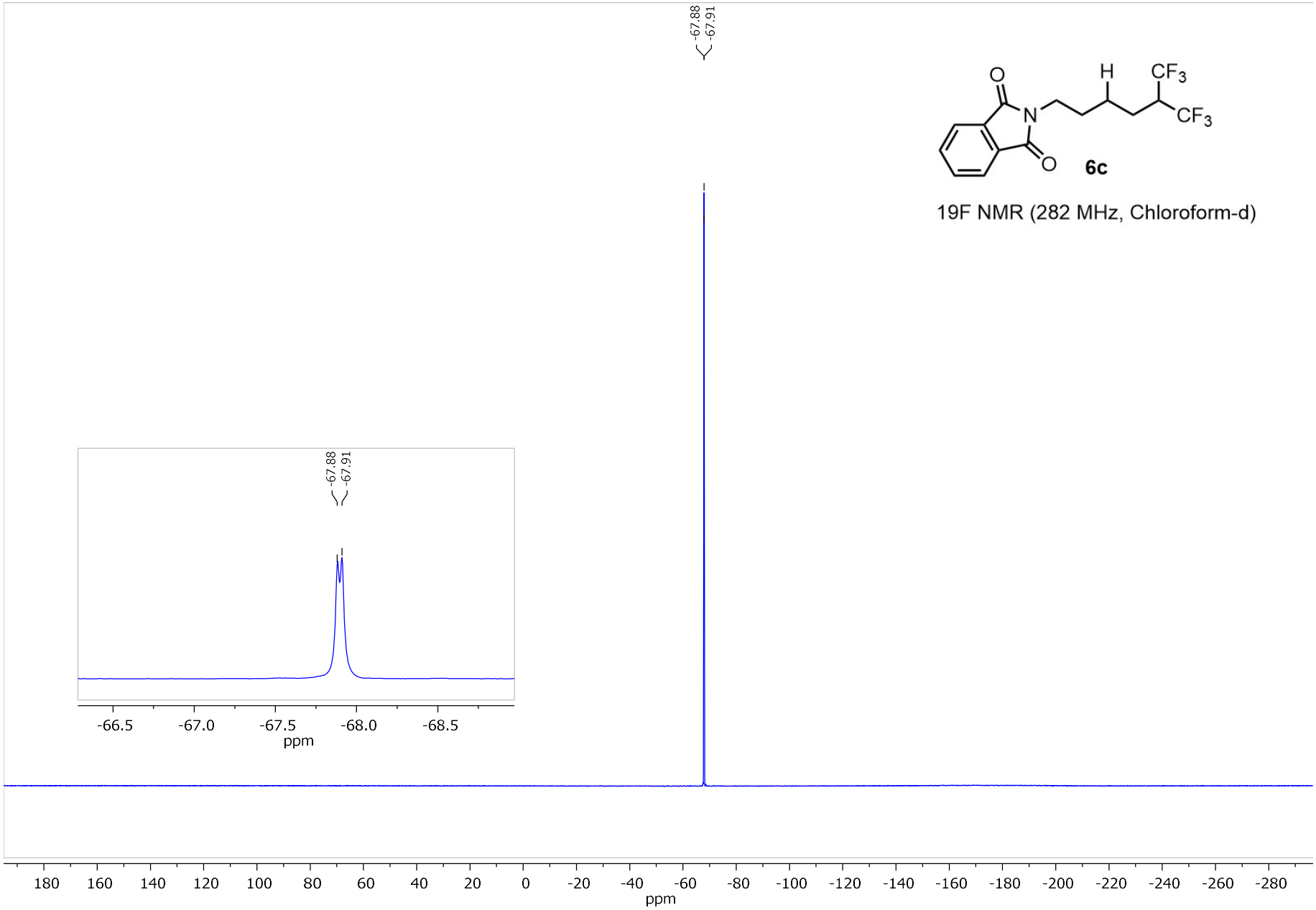

4.18
3.32
2.85
1.91
1.56
1.26
CO2Et
CF3
EtO2C
CF3
6b
1H NMR (300 MHz, Chloroform-d)
3.32
2.85
3.5 3.4 3.3 3.2 3.1 3.0 2.9 2.8 2.7 2.6
ppm
3.88
0.97
1.00
3.83
2.13
6.29
17 16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
ppm

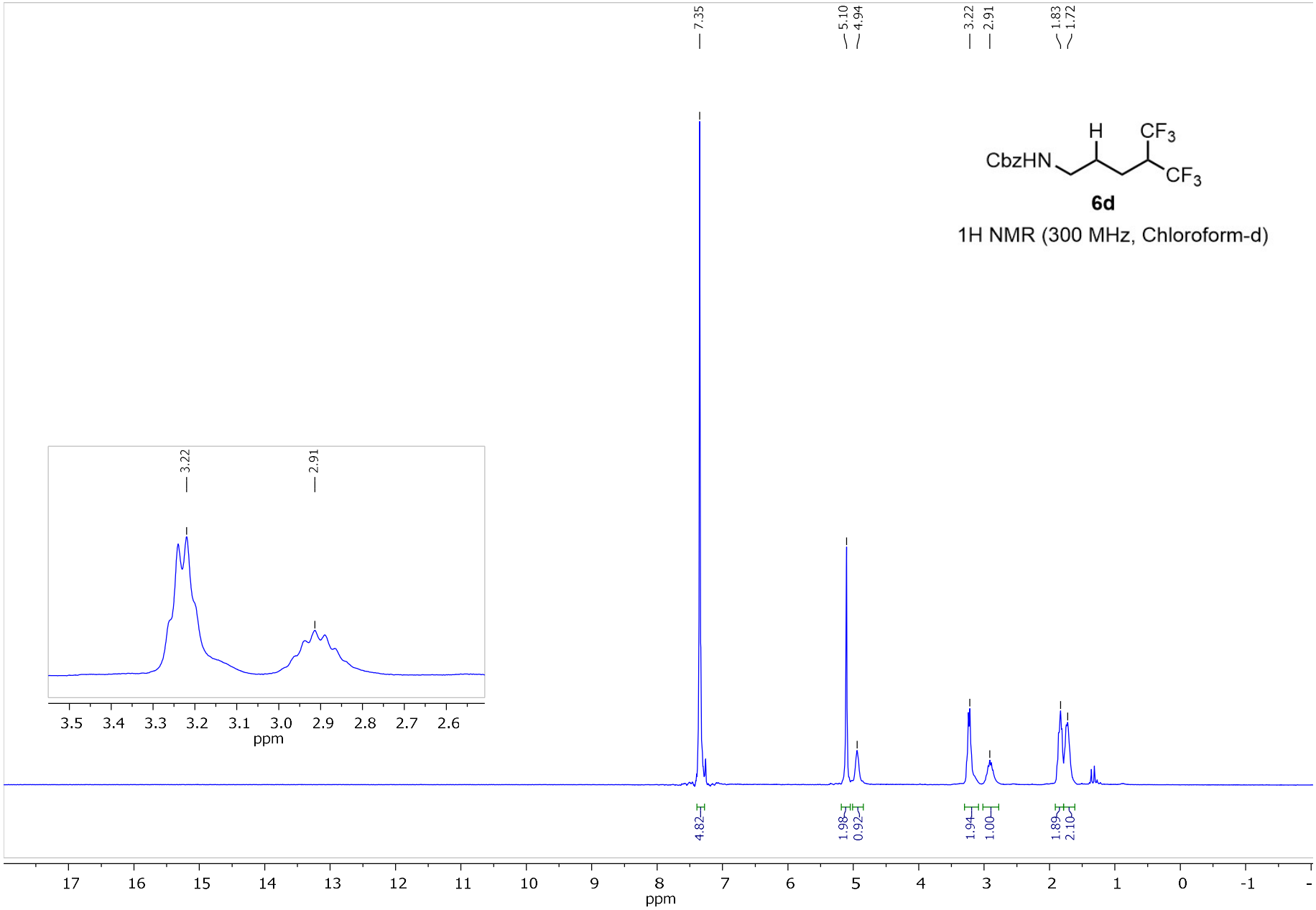

169.15
129.61
125.89
125.84
122.16
122.12
118.45
61.63
51.60
49.02
48.65
48.28
47.91
47.54
47.17
46.80
28.35
24.95
23.57
23.54
23.52
23.49
23.46
14.11
CO2Et
CF3
EtO2C
CF3
6b
13C NMR (75 MHz, Chloroform-d)
ppm
ppm

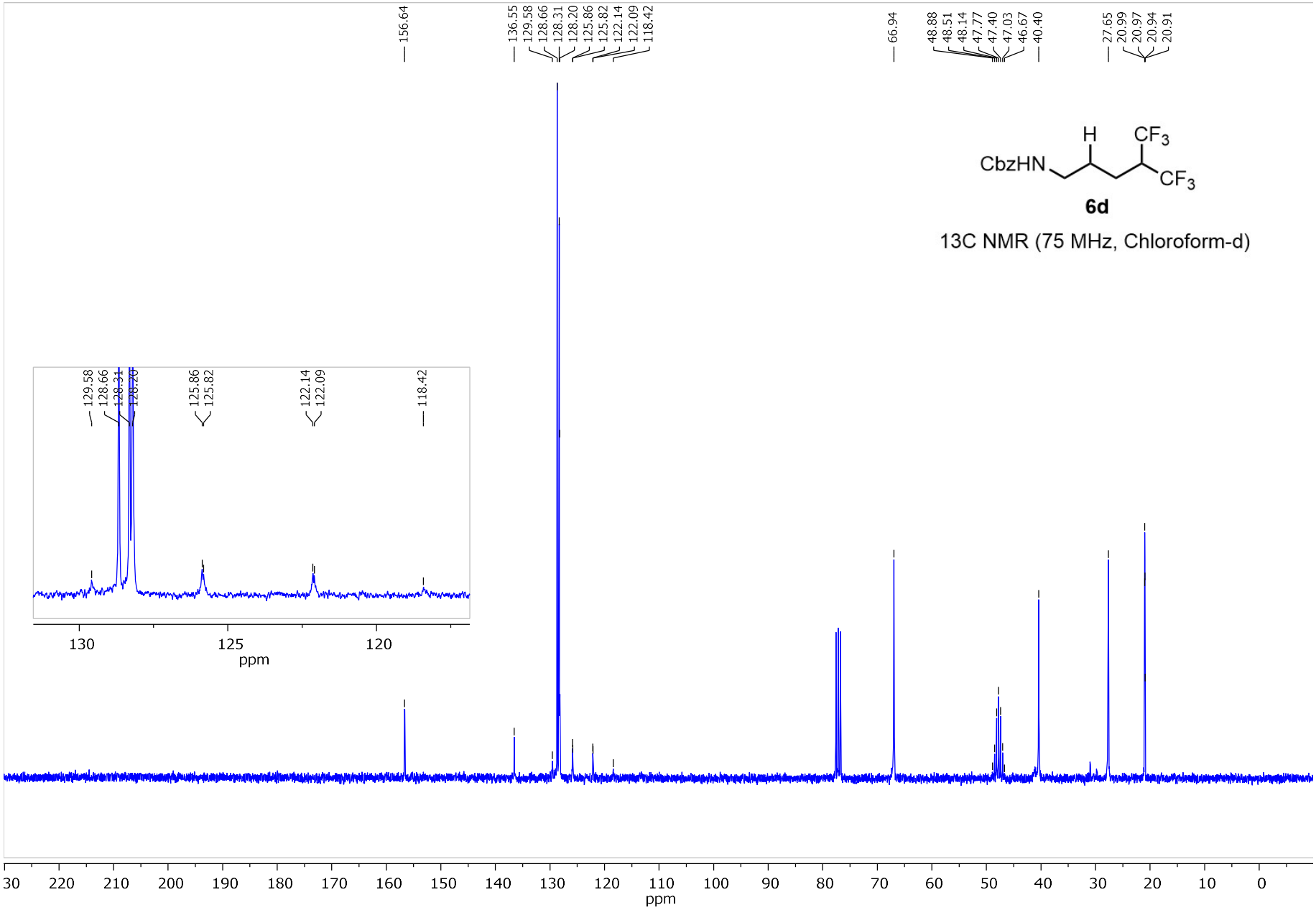

-67.94
-67.97
CO2Et
CF3
EtO2C
CF3
6b
19F NMR (282 MHz, Chloroform-d)
-67.94
-67.97
-67.0
-67.5
-68.0
-68.5
-69.0
ppm
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm

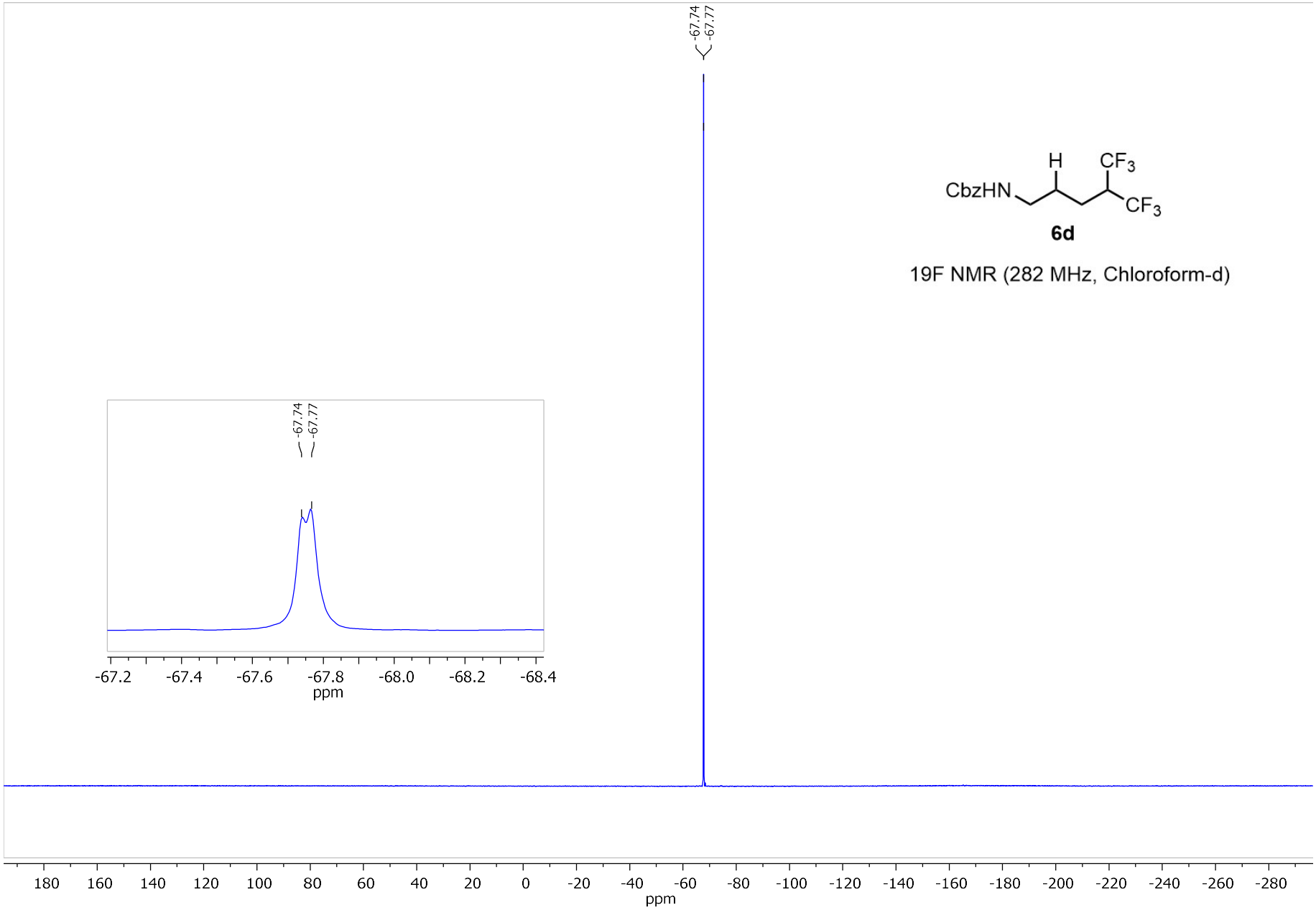

7.83
7.73
3.70
2.84
1.84
1.73
1.57
O
N
O
H
CF3
CF3
6c
1H NMR (300 MHz, Chloroform-d)
2.84
3.1
3.0
2.9
2.8
2.7
2.6
ppm
1.90
1.90
1.92
1.00
1.94
2.02
2.00
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

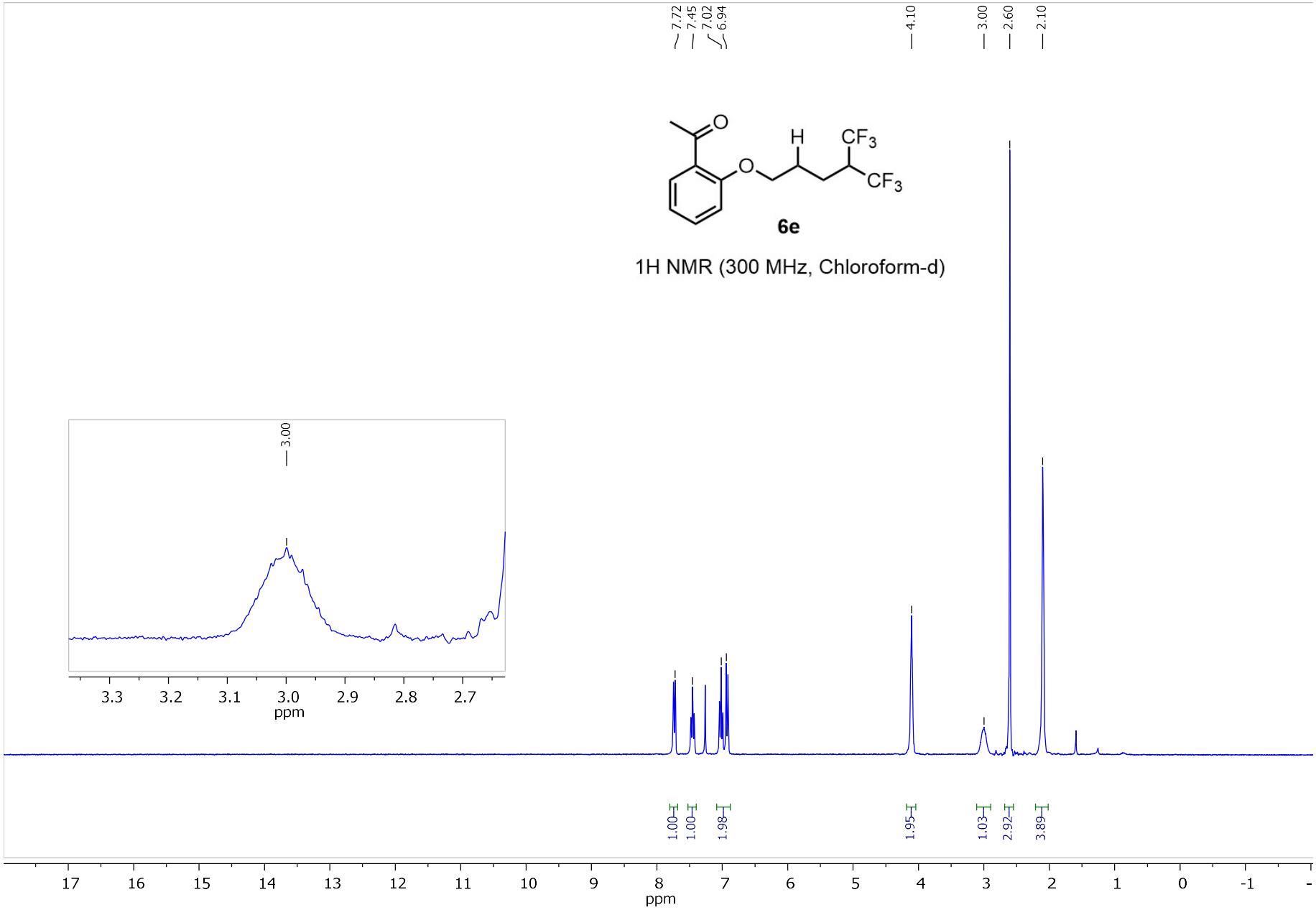

168.47
134.13
132.19
129.63
125.91
125.86
123.40
122.19
122.14
118.41
49.15
48.78
48.42
48.05
47.68
47.31
46.94
37.35
28.62
28.39
24.62
23.41
23.38
23.36
23.33
23.31
O
H
CF3
N
CF3
O
6c
13C NMR (75 MHz, Chloroform-d)
ppm
ppm

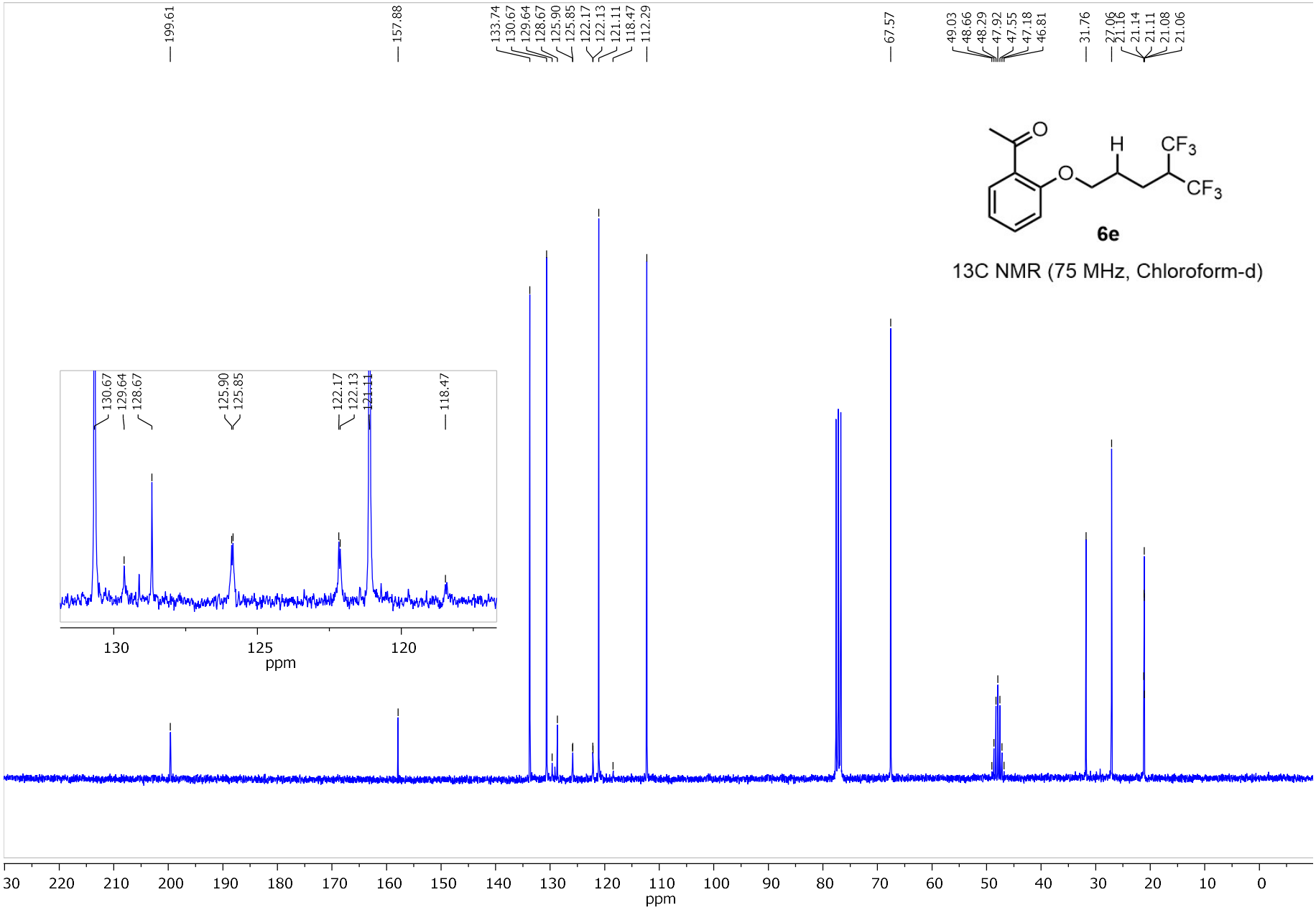

-67.88
-67.91
O
H
CF3
N
CF3
O
6c
19F NMR (282 MHz, Chloroform-d)
-67.88
-67.91
-66.5
-67.0
-67.5
-68.0
-68.5
ppm
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm

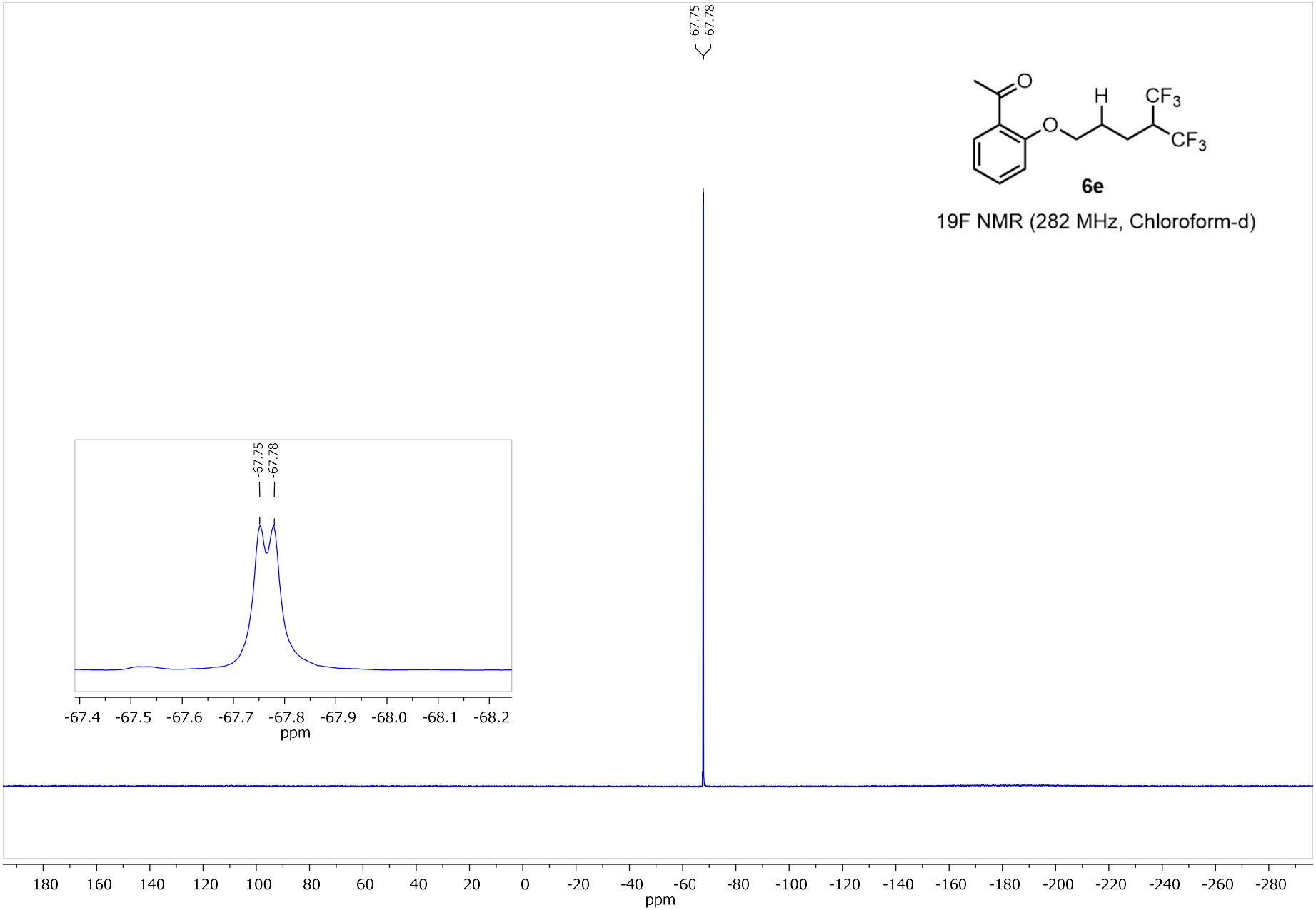

7.35
5.10
4.94
3.22
2.91
1.83
1.72
H
CF3
CbzHN
CF3
6d
1H NMR (300 MHz, Chloroform-d)
3.22
2.91
3.5 3.4 3.3 3.2 3.1 3.0 2.9 2.8 2.7 2.6
ppm
4.82
1.98
0.92
1.94
1.00
1.89
2.10
17 16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
ppm

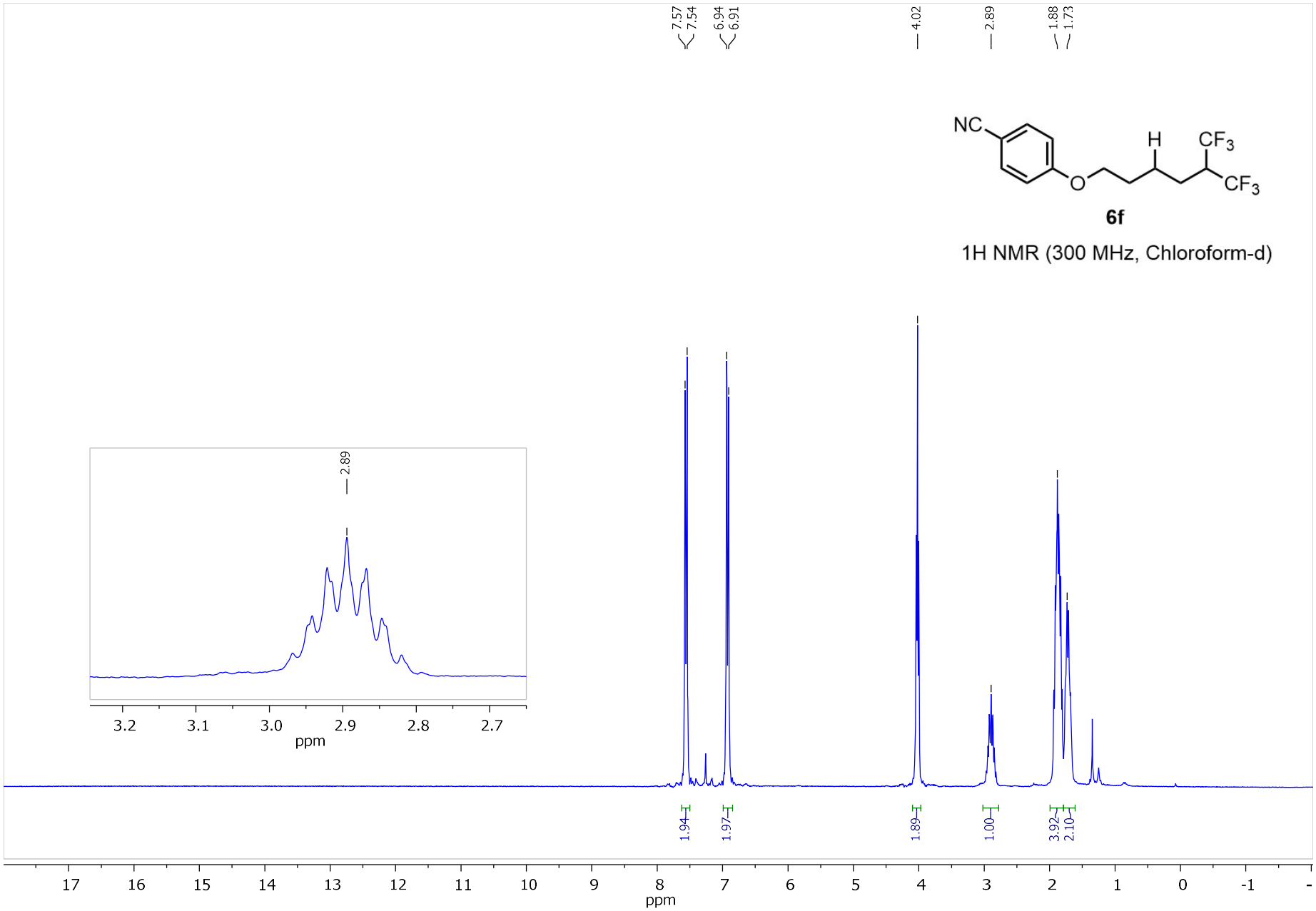

156.64
136.55
129.58
128.66
128.31
128.20
125.86
125.82
122.14
122.09
118.42
66.94
48.88
48.51
48.14
47.77
47.40
47.03
46.67
40.40
27.65
20.99
20.97
20.94
20.91
H
CF3
CbzHN
CF3
6d
13C NMR (75 MHz, Chloroform-d)
ppm
ppm

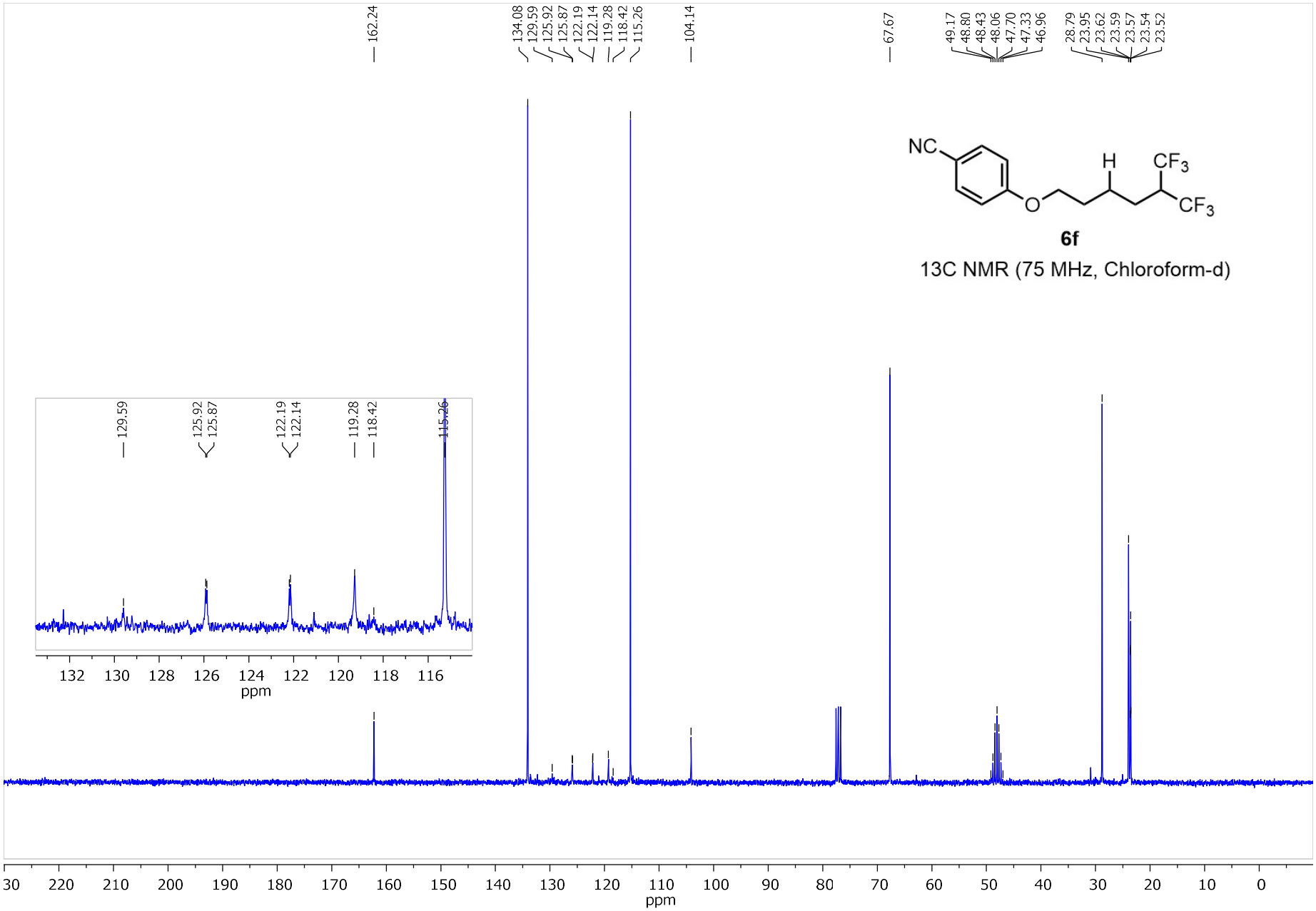

-67.74
-67.77
H
CF3
CbzHN
CF3
6d
19F NMR (282 MHz, Chloroform-d)
-67.74
-67.77
-67.2
-67.4
-67.6
-67.8
-68.0
-68.2
-68.4
ppm
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm

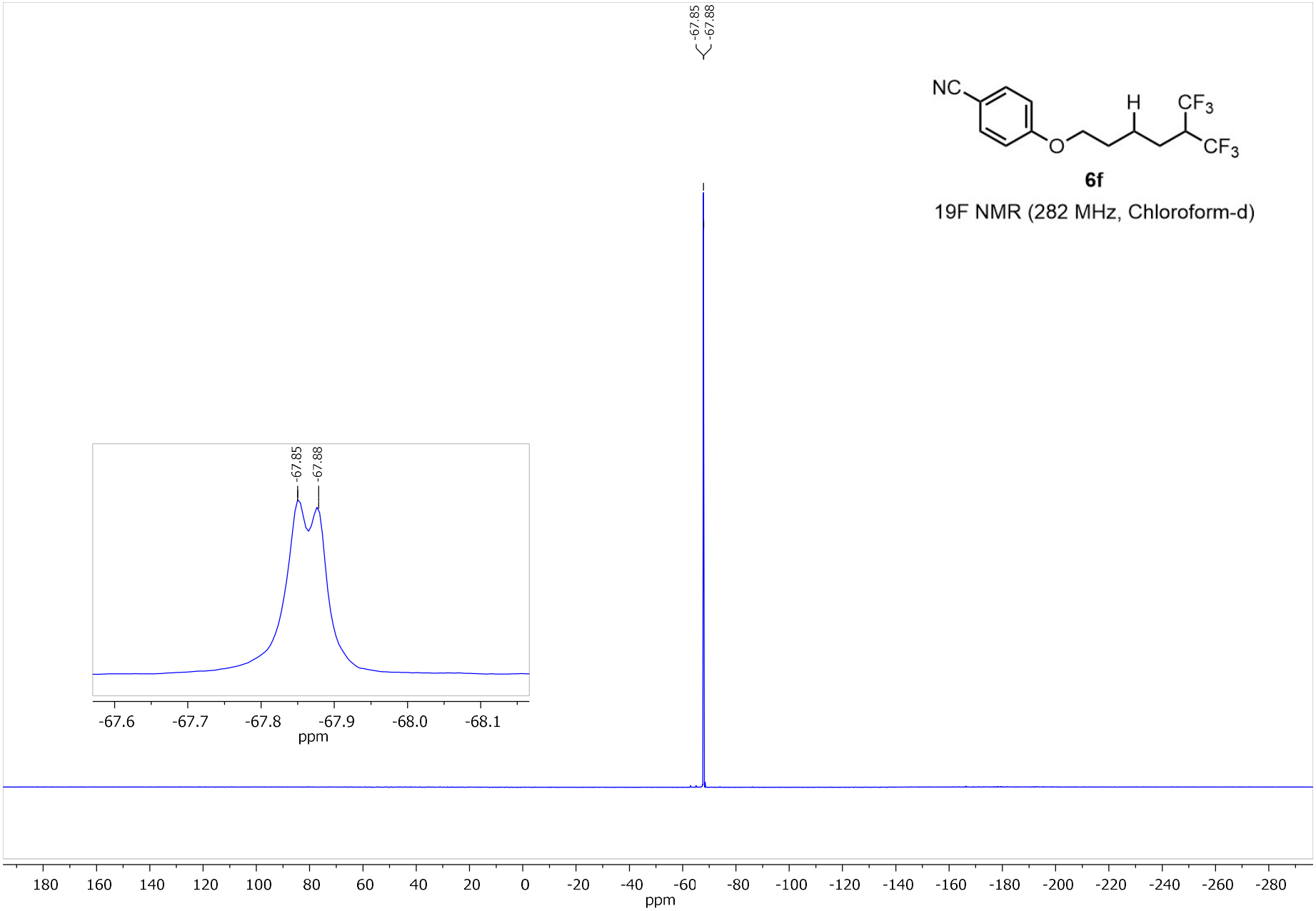

7.72
7.45
7.02
6.94
4.10
3.00
2.60
2.10
O
H
CF3
O
CF3
6e
1H NMR (300 MHz, Chloroform-d)
3.00
3.3
3.2
3.1
3.0
2.9
2.8
2.7
ppm
1.00
1.00
1.98
1.95
1.03
2.92
3.89
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-
ppm

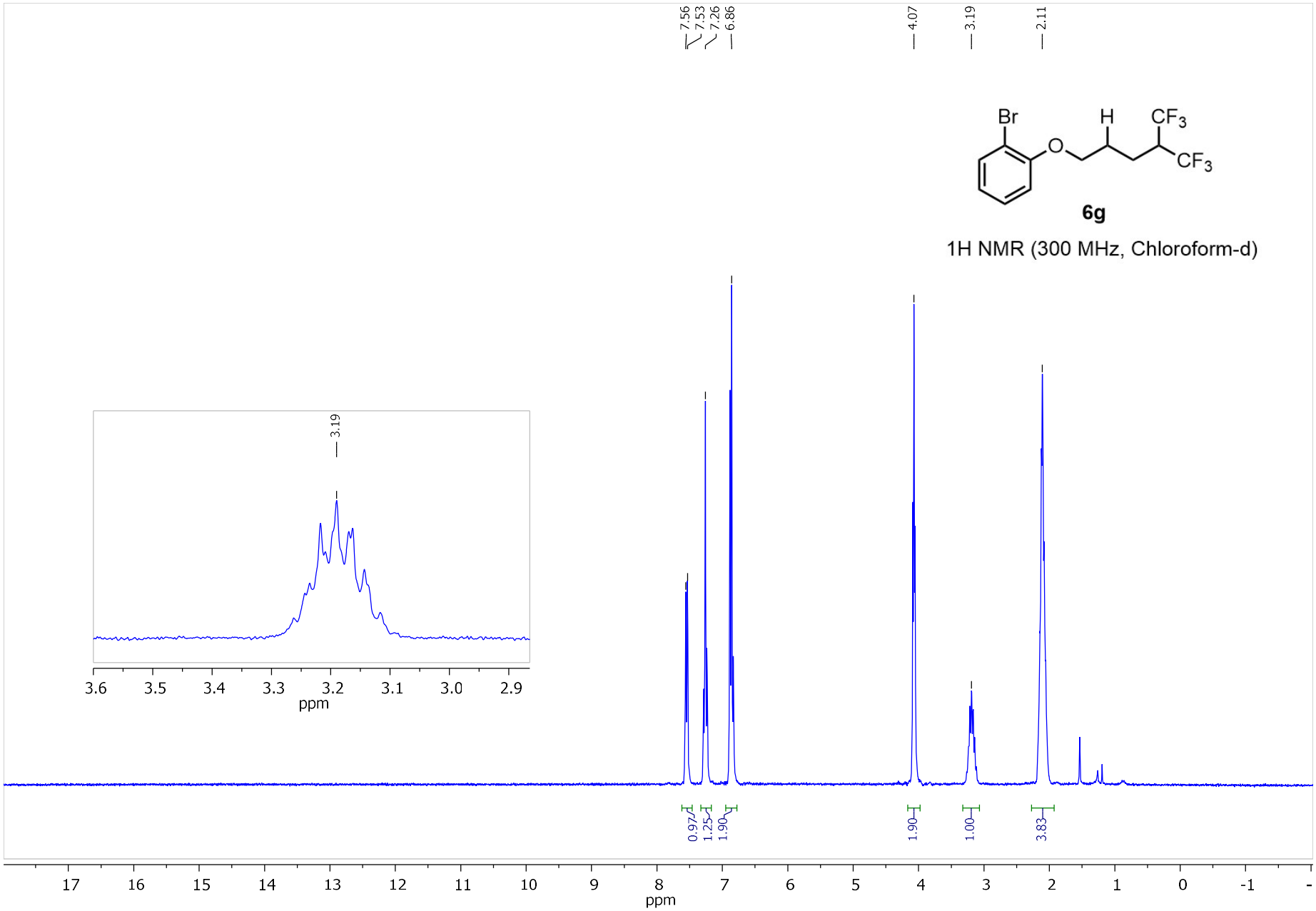

199.61
157.88
133.74
130.67
129.64
128.67
125.90
125.85
122.17
122.13
121.11
118.47
112.29
67.57
49.03
48.66
48.29
47.92
47.55
47.18
46.81
31.76
27.06
21.16
21.14
21.11
21.08
21.06
O
H
CF3
O
CF3
6e
13C NMR (75 MHz, Chloroform-d)
ppm
ppm

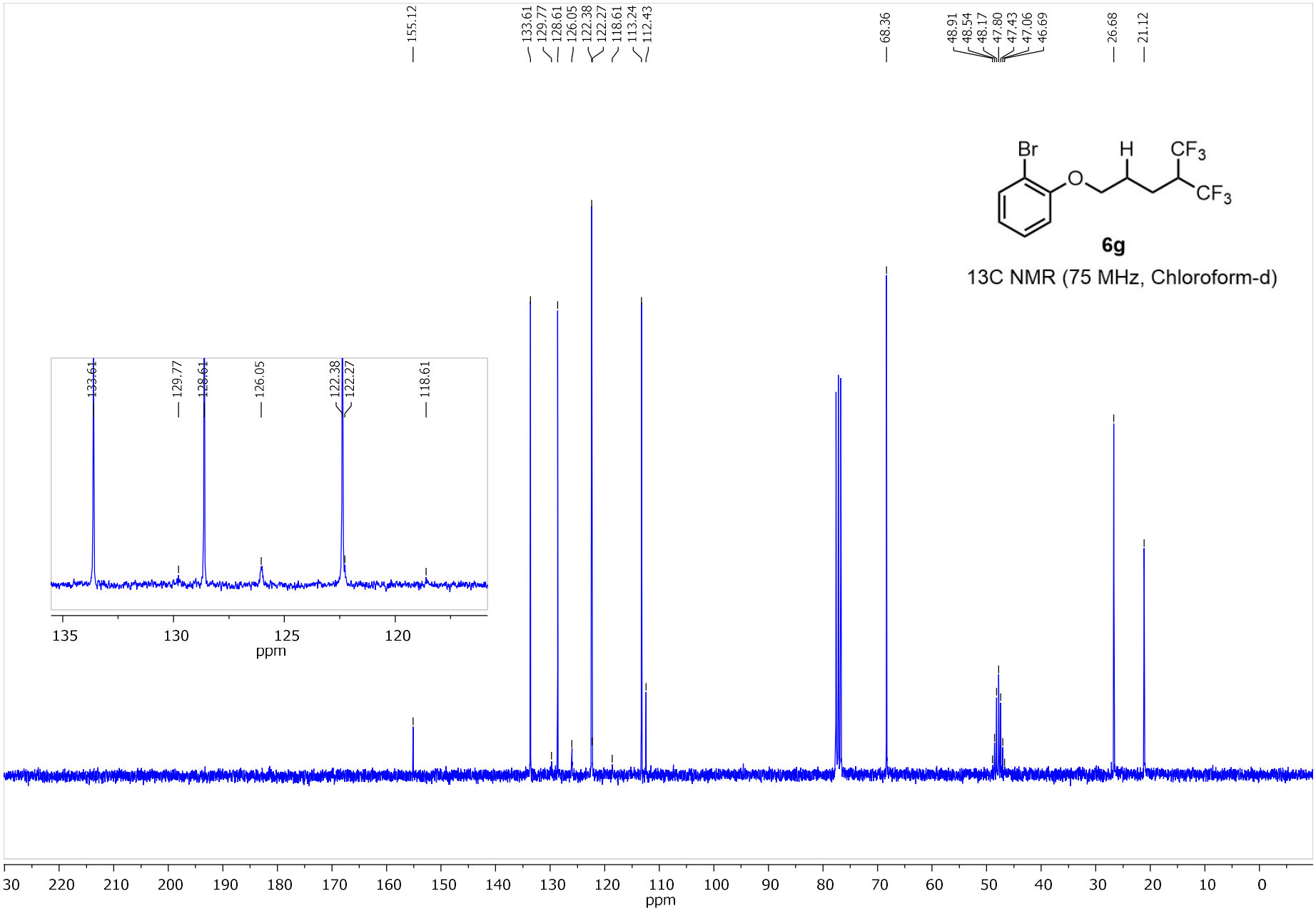

-67.75
-67.78
O
H
CF3
O
CF3
6e
19F NMR (282 MHz, Chloroform-d)
-67.75
-67.78
-67.4 -67.5 -67.6 -67.7 -67.8 -67.9 -68.0 -68.1 -68.2
ppm
180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280
ppm


7.57
7.54
6.94
6.91
4.02
2.89
1.88
1.73
NC
H
CF3
O
CF3
6f
1H NMR (300 MHz, Chloroform-d)
2.89
3.2
3.1
3.0
2.9
2.8
2.7
ppm
1.94
1.97
1.89
1.00
3.92
2.10
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm


162.24
134.08
129.59
125.92
125.87
122.19
122.14
119.28
118.42
115.26
104.14
67.67
49.17
48.80
48.43
48.06
47.70
47.33
46.96
28.79
23.95
23.62
23.59
23.57
23.54
23.52
NC
H
CF3
CF3
O
6f
13C NMR (75 MHz, Chloroform-d)
ppm


-67.85
-67.88
NC
H
CF3
O
CF3
6f
19F NMR (282 MHz, Chloroform-d)
-67.85
-67.88
-67.6
-67.7
-67.8
-67.9
-68.0
-68.1
ppm
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm


7.56
7.53
7.26
6.86
4.07
3.19
2.11
Br
H
CF3
O
CF3
6g
1H NMR (300 MHz, Chloroform-d)
3.19
3.6 3.5 3.4 3.3 3.2 3.1 3.0 2.9
ppm
0.97
1.25
1.90
1.90
1.00
3.83
17 16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
ppm


155.12
133.61
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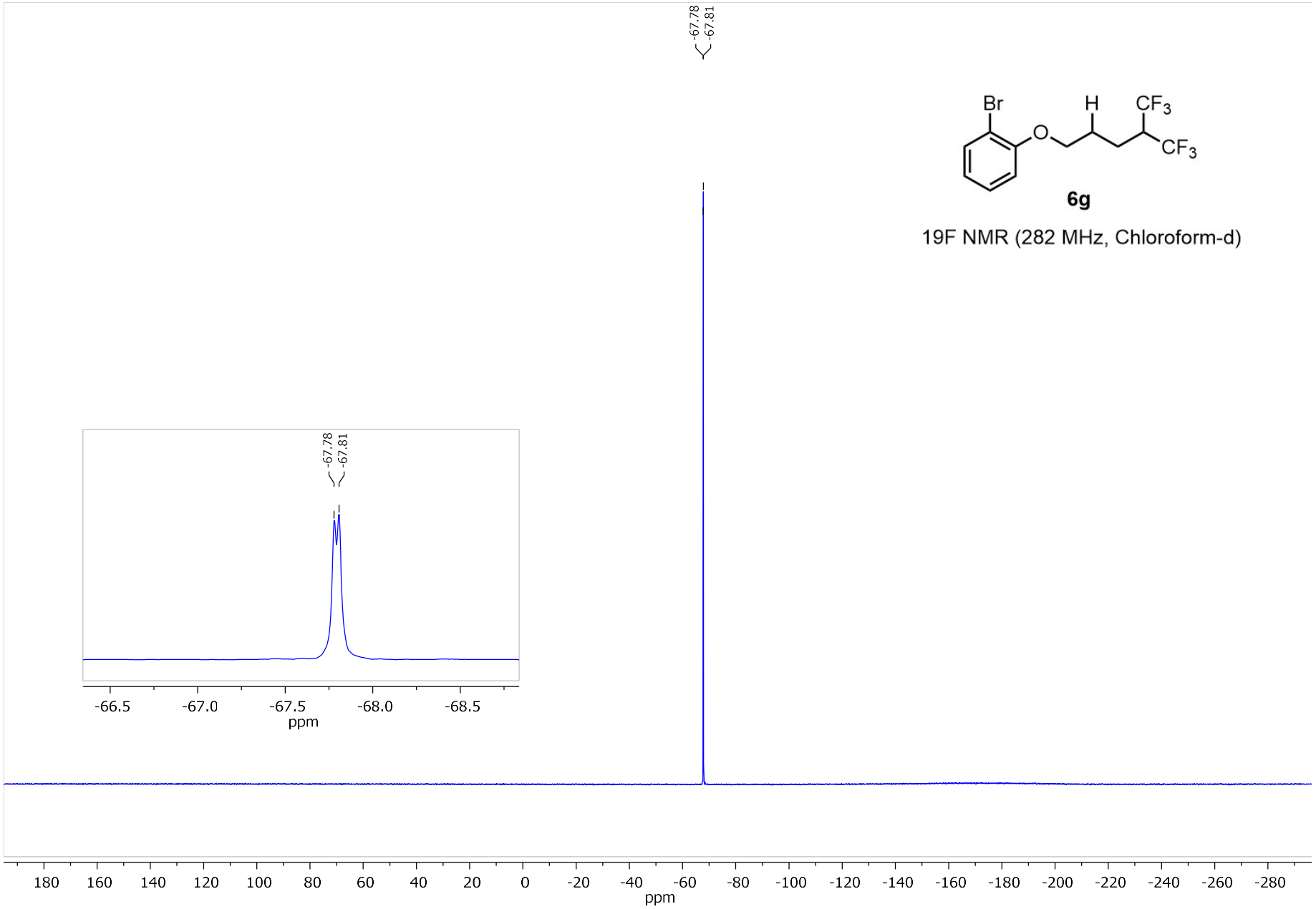
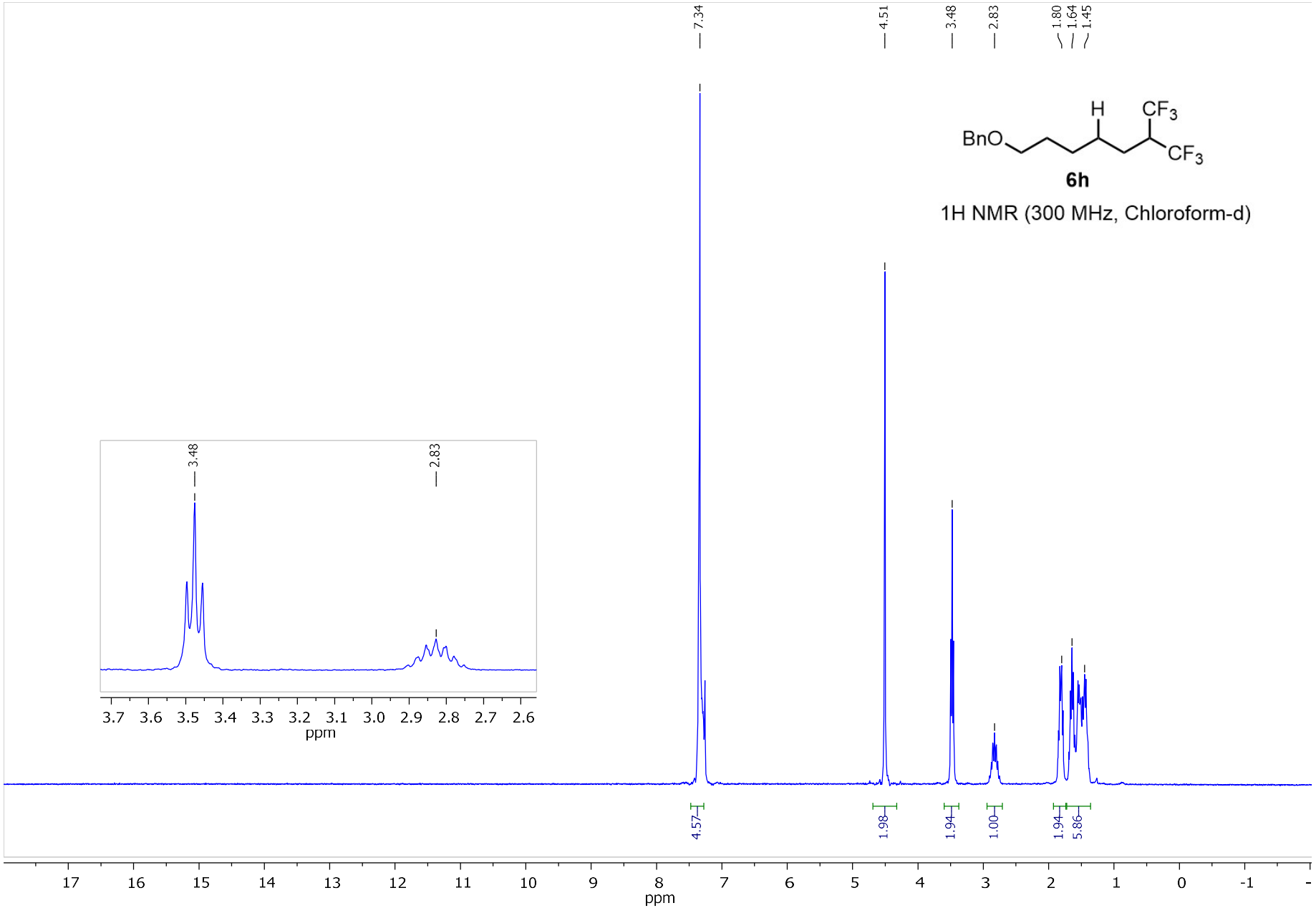
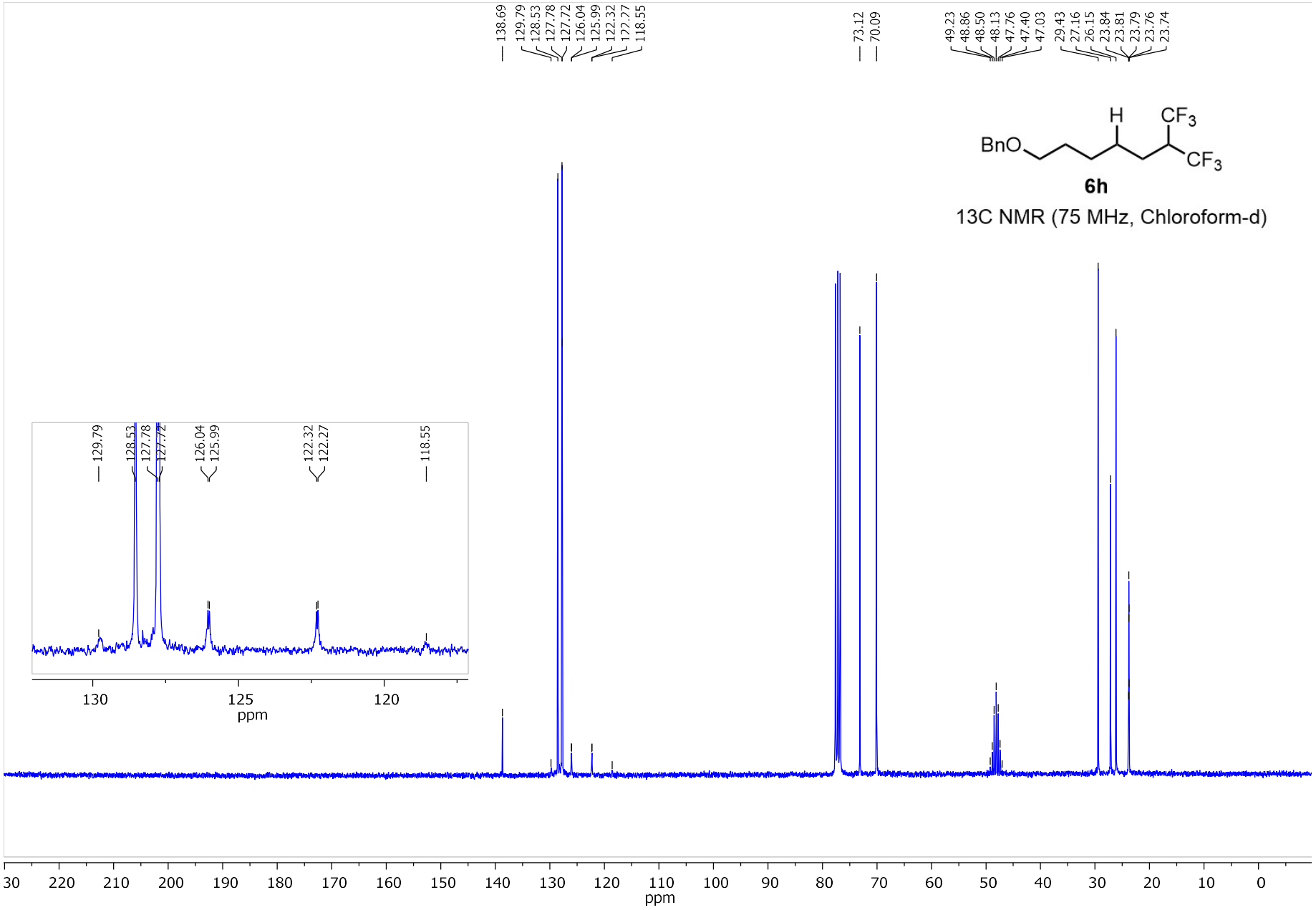
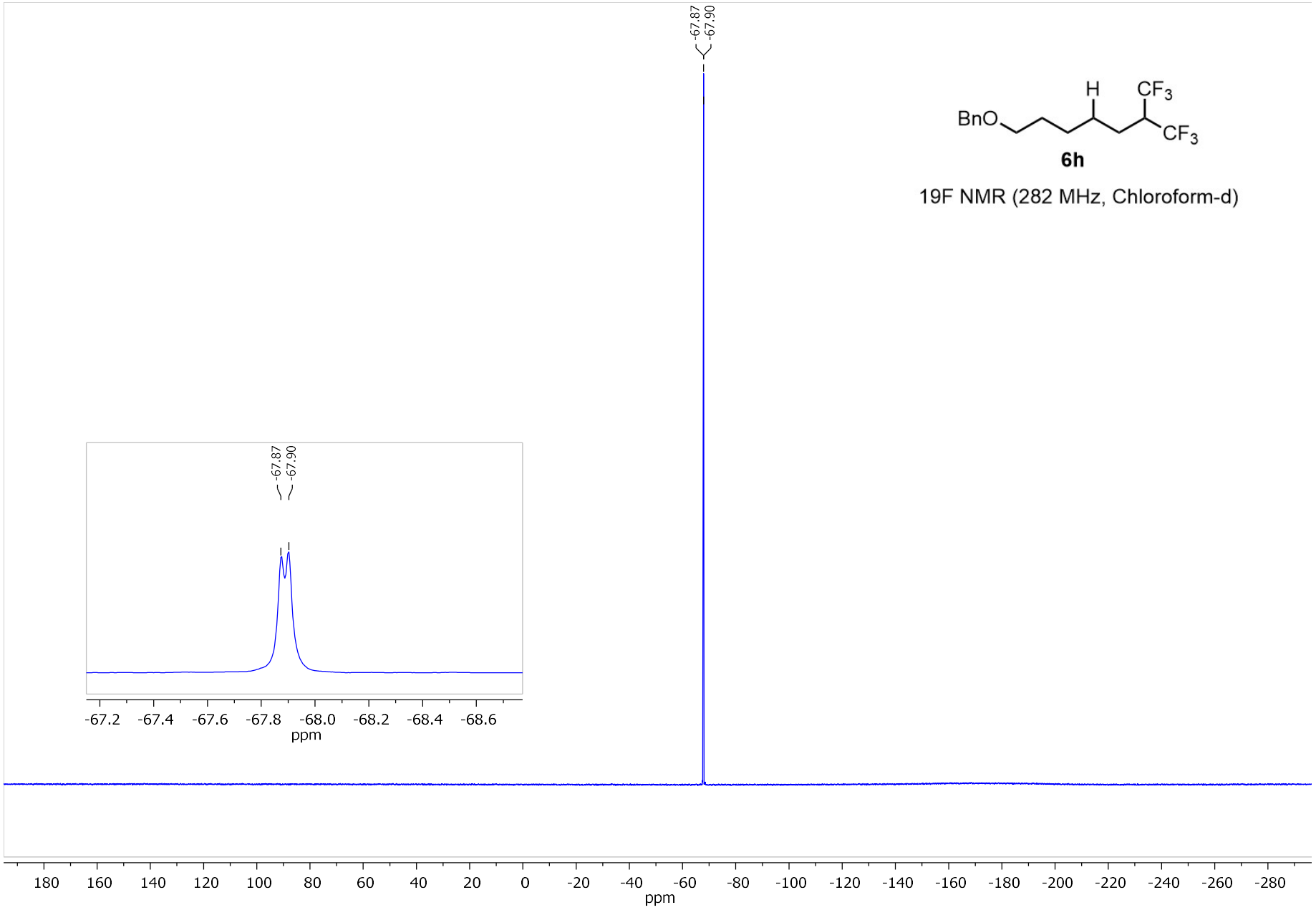
128.61
126.05
122.38
122.27
118.61
113.24
112.43
68.36
48.91
48.54
48.17
47.80
47.43
47.06
46.69
26.68
21.12
Br
H
CF3
O
CF3
6g
13C NMR (75 MHz, Chloroform-d)
ppm
ppm


-67.78
-67.81
Br
H
CF3
O
CF3
6g
19F NMR (282 MHz, Chloroform-d)
-67.78
-67.81
-66.5
-67.0
-67.5
-68.0
-68.5
ppm
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm


7.34
4.51
3.48
2.83
1.80
1.64
1.45
BnO
H
CF3
CF3
6h
1H NMR (300 MHz, Chloroform-d)
4.57
1.98
1.94
1.00
1.94
5.86
ppm
ppm


138.69
129.79
128.53
127.78
127.72
126.04
125.99
122.32
122.27
118.55
73.12
70.09
49.23
48.86
48.50
48.13
47.76
47.40
47.03
29.43
27.16
26.15
23.84
23.81
23.79
23.76
23.74
H
CF3
BnO
CF3
6h
13C NMR (75 MHz, Chloroform-d)
ppm


-67.87
-67.90
BnO
H
CF3
CF3
6h
19F NMR (282 MHz, Chloroform-d)
-67.87
-67.96
-67.2
-67.4
-67.6
-67.8
-68.0
-68.2
-68.4
-68.6
ppm
180
160
140
120
100
80
60
40
20
0
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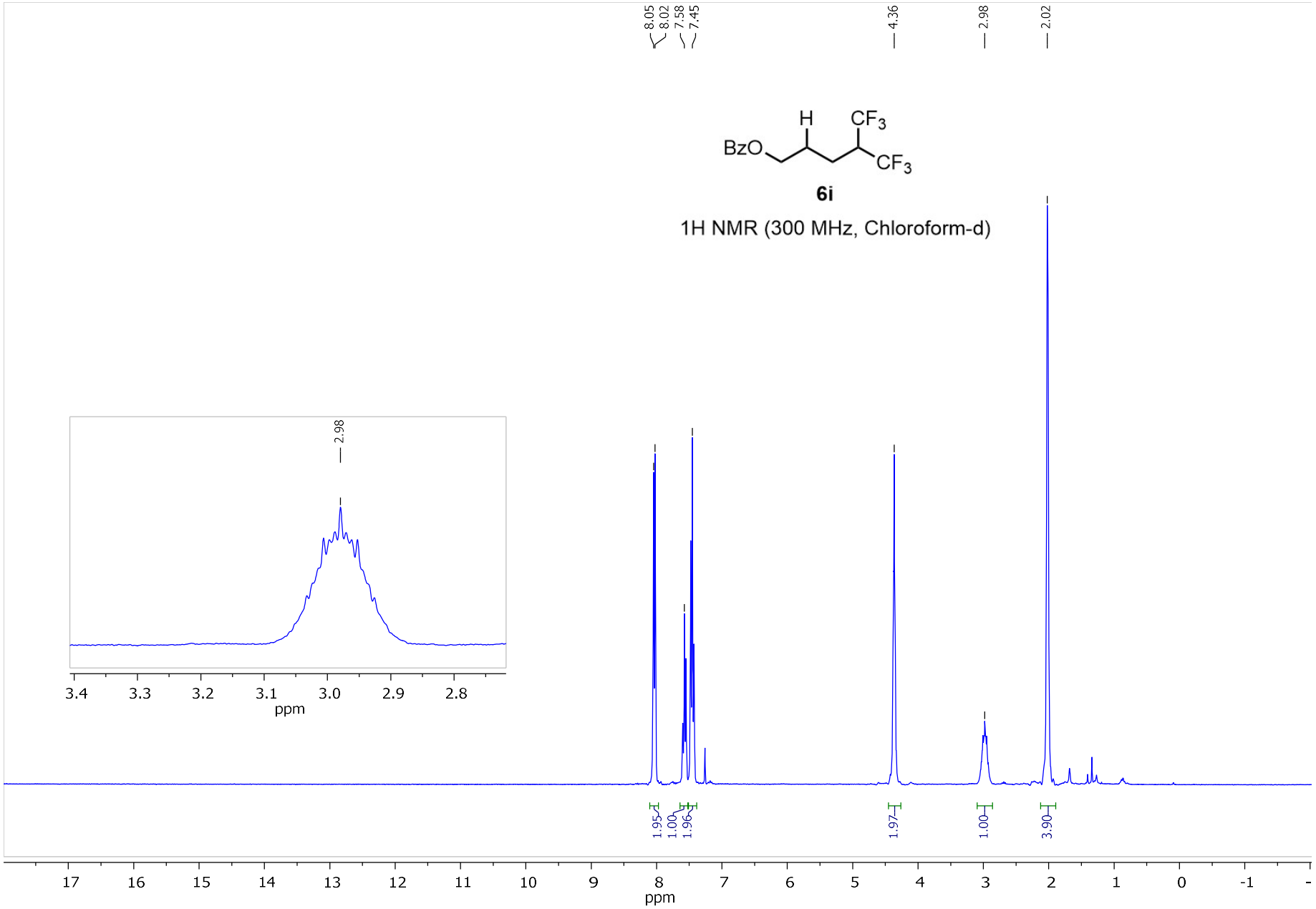
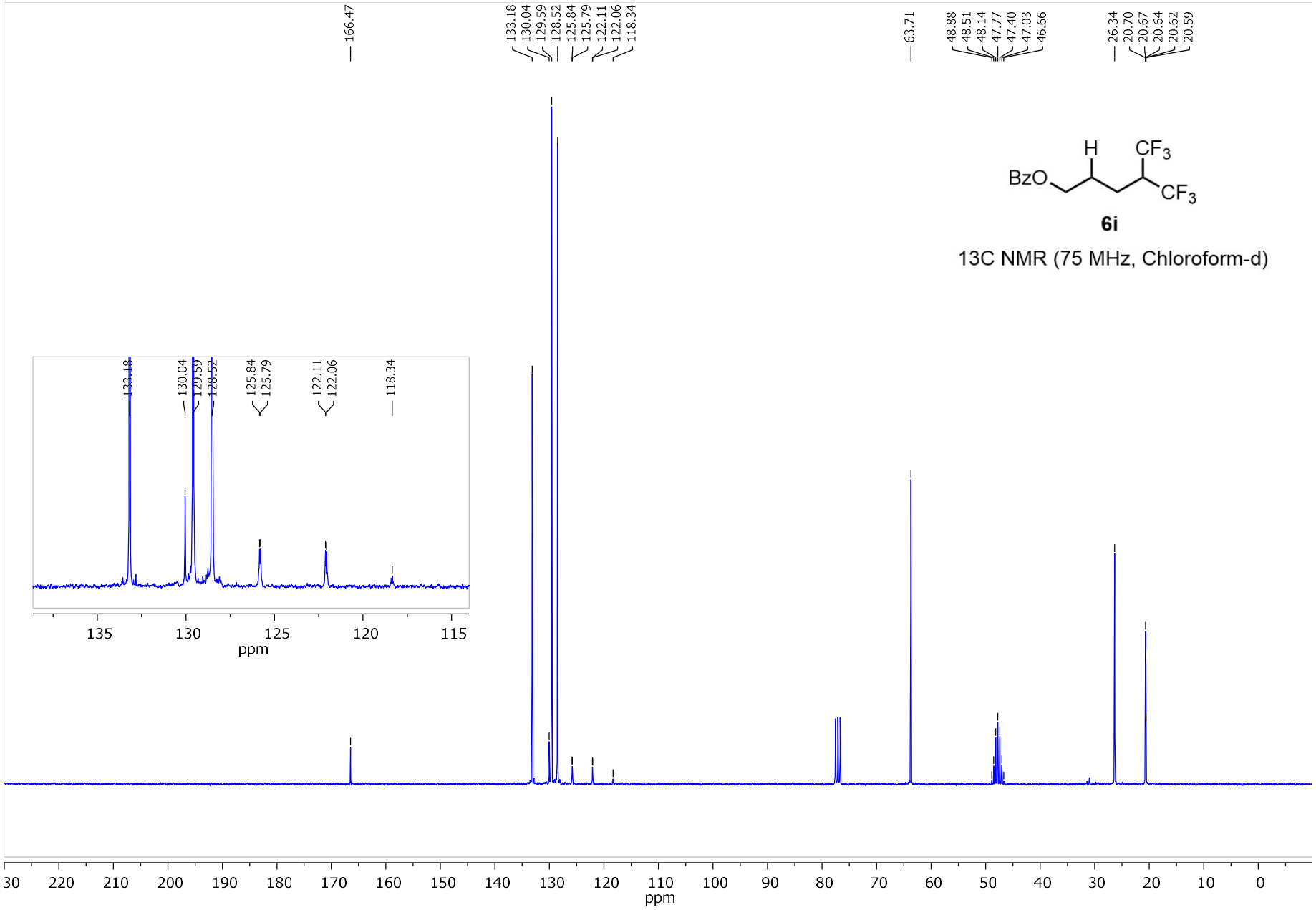
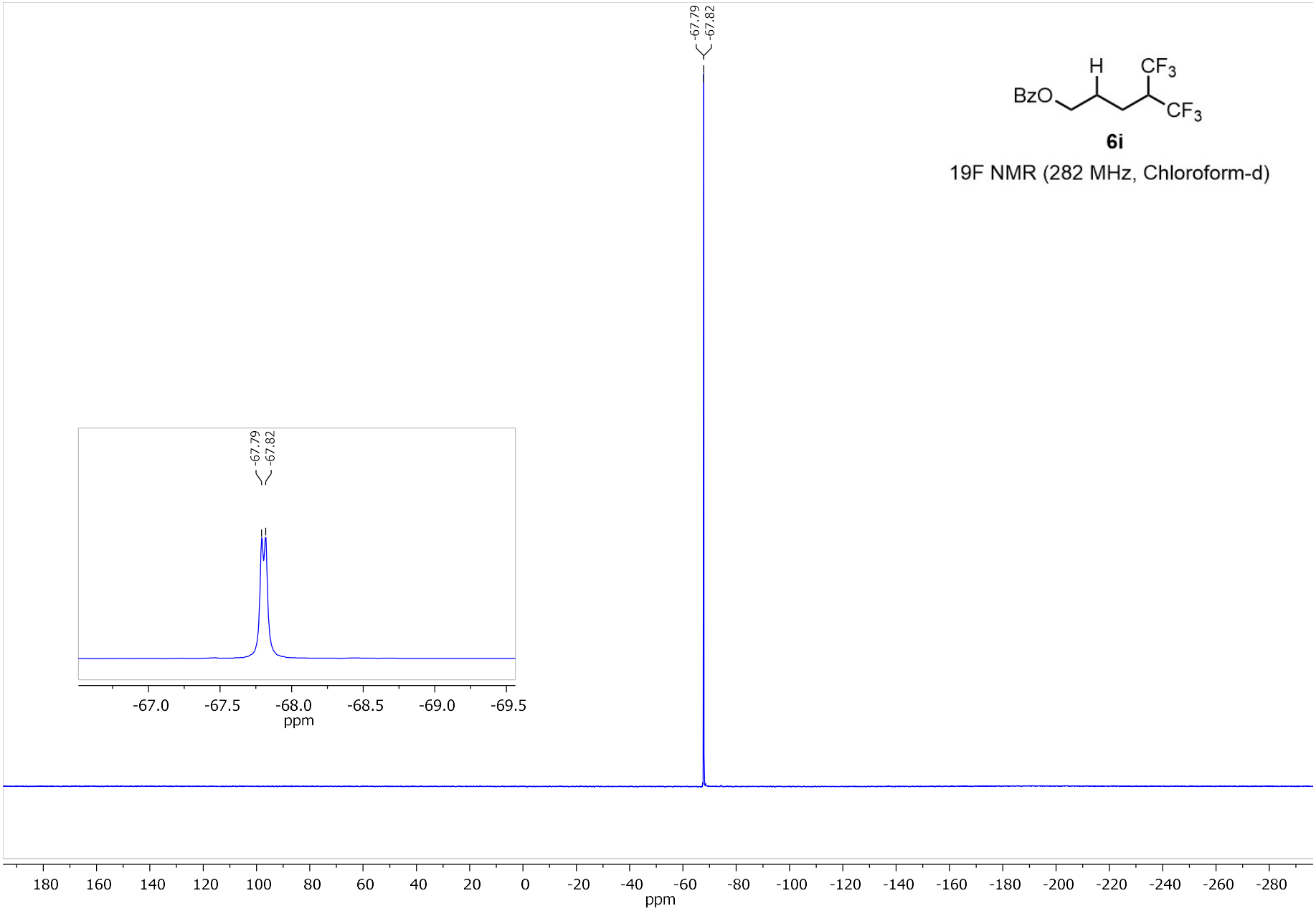
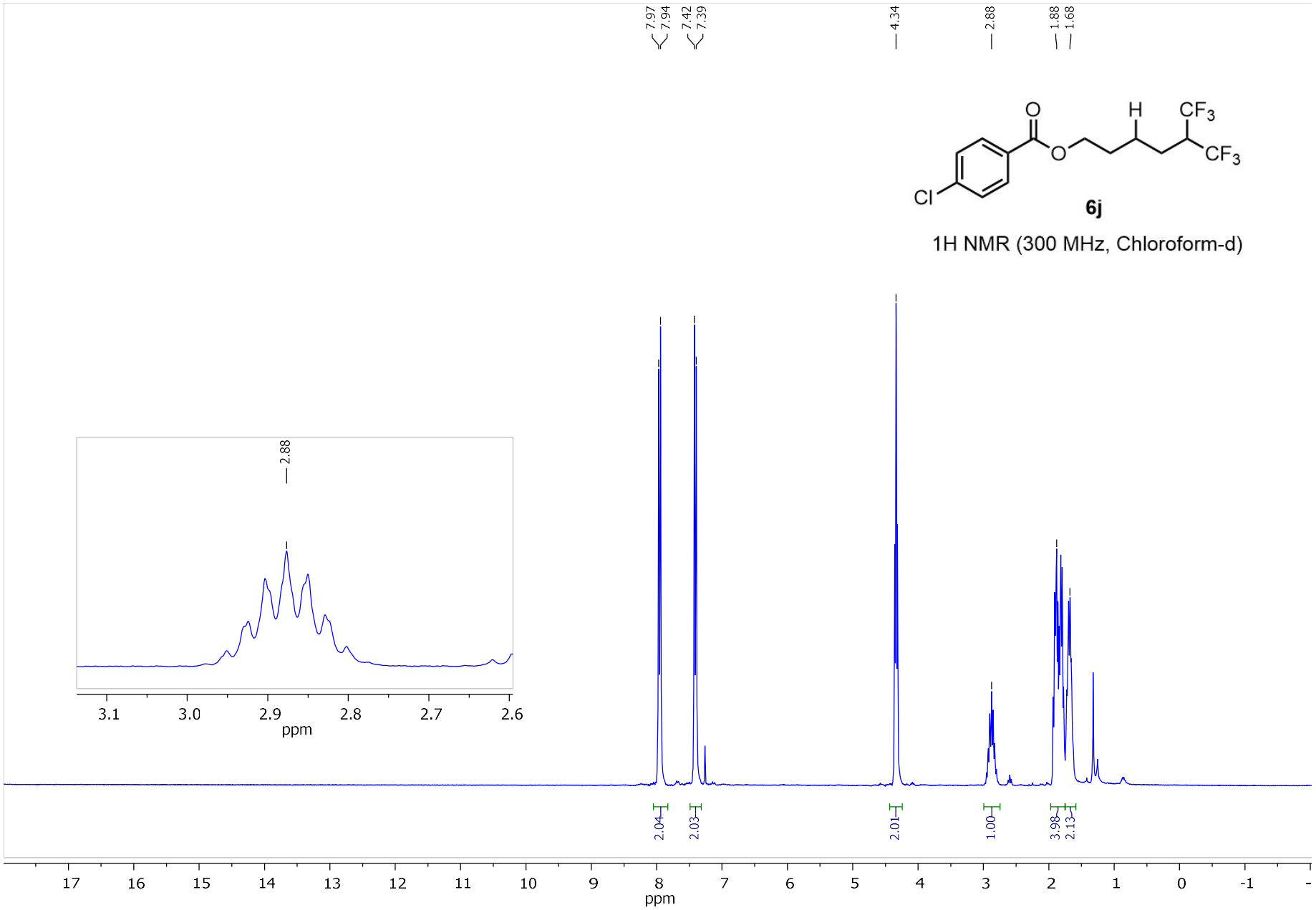
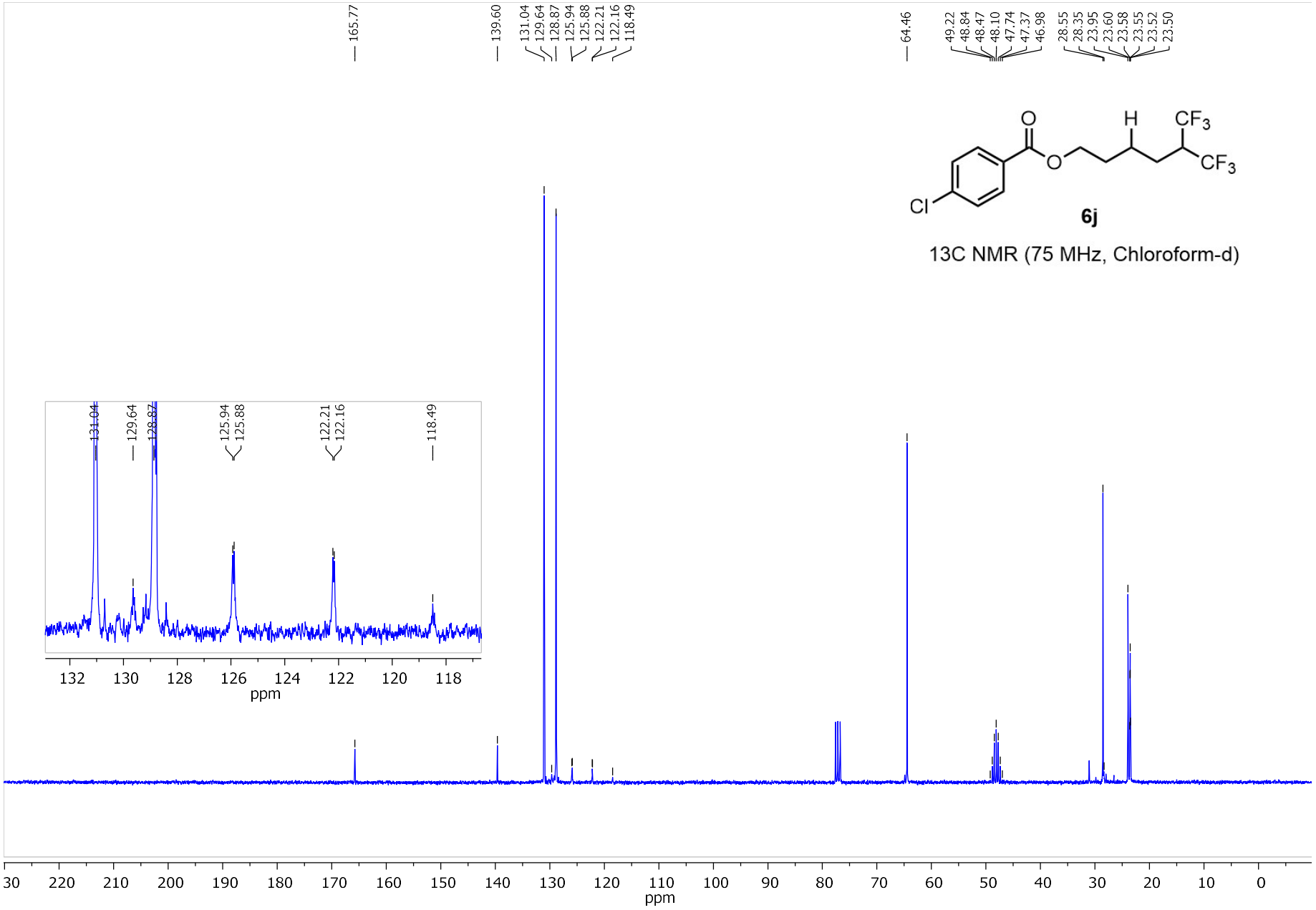
-160
-180
-200
-220
-240
-260
-280
ppm

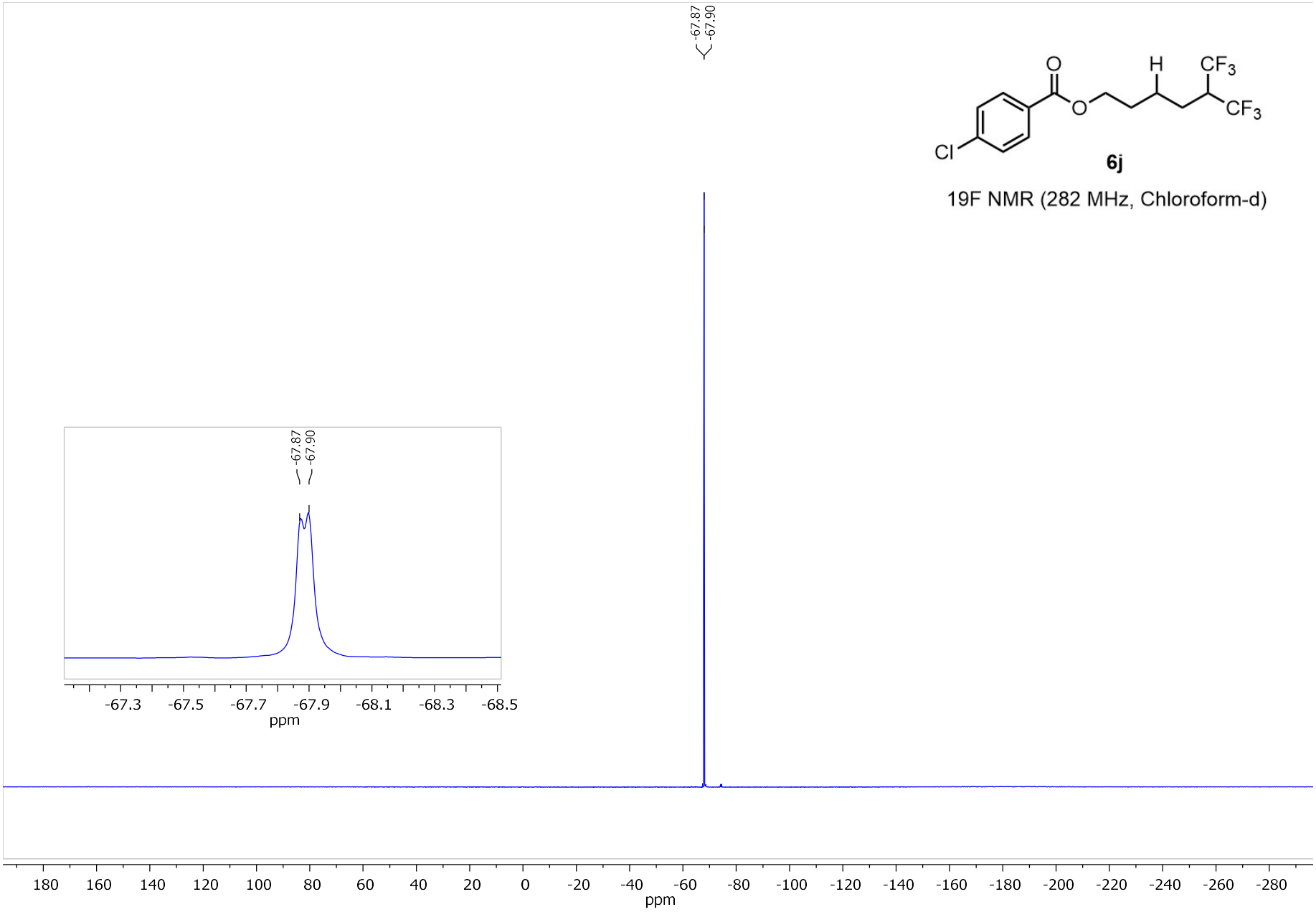

8.05
8.02
7.58
7.45
4.36
2.98
2.02
H
CF3
BzO
CF3
6i
1H NMR (300 MHz, Chloroform-d)
2.98
3.4
3.3
3.2
3.1
3.0
2.9
2.8
ppm
1.95
1.00
1.96
1.97
1.00
3.90
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

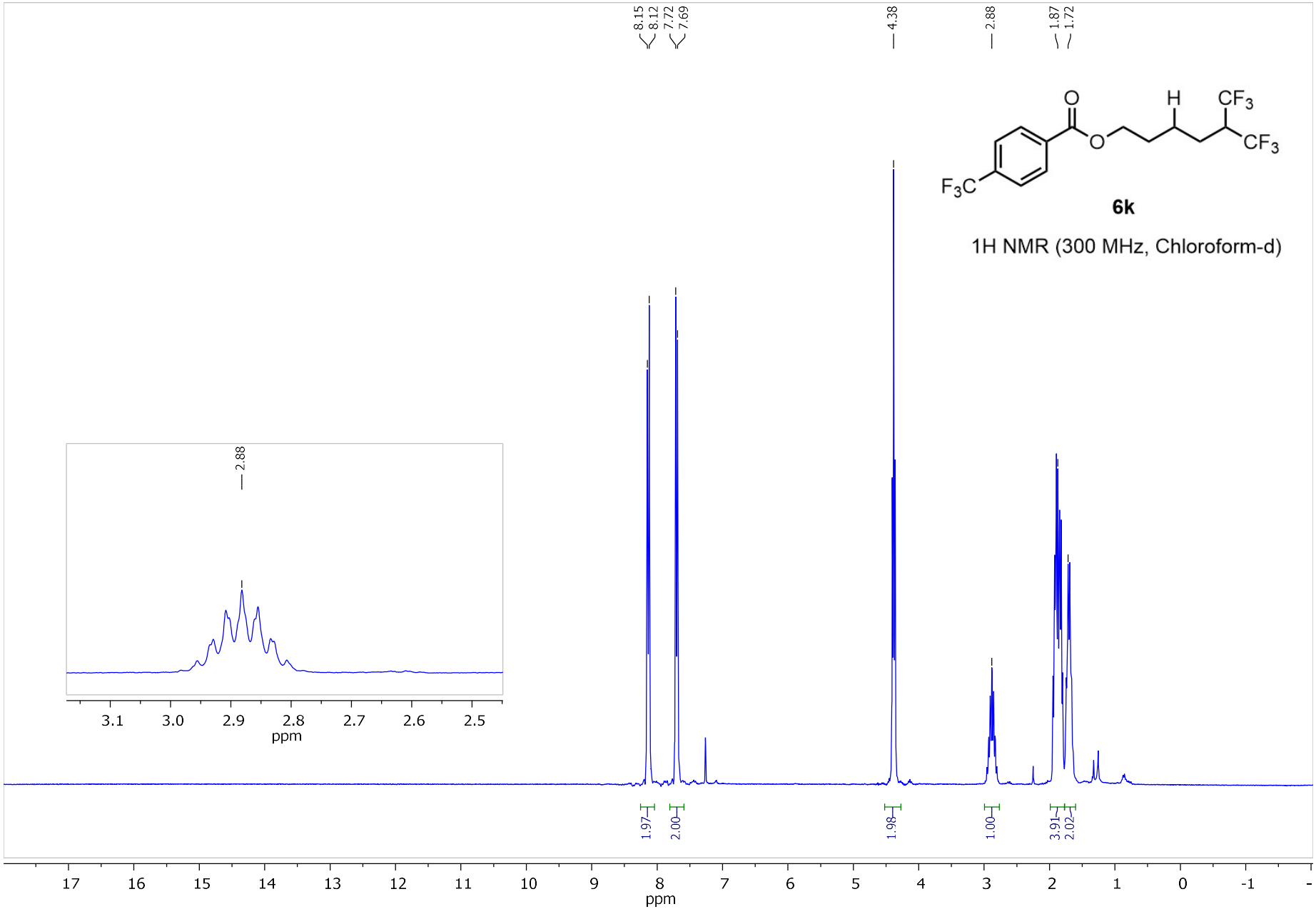

166.47
133.18
130.04
129.59
128.52
125.84
125.79
122.11
122.06
118.34
63.71
48.88
48.51
48.14
47.77
47.40
47.03
46.66
26.34
20.70
20.67
20.64
20.62
20.59
H
CF3
BzO
CF3
6i
13C NMR (75 MHz, Chloroform-d)
ppm
ppm

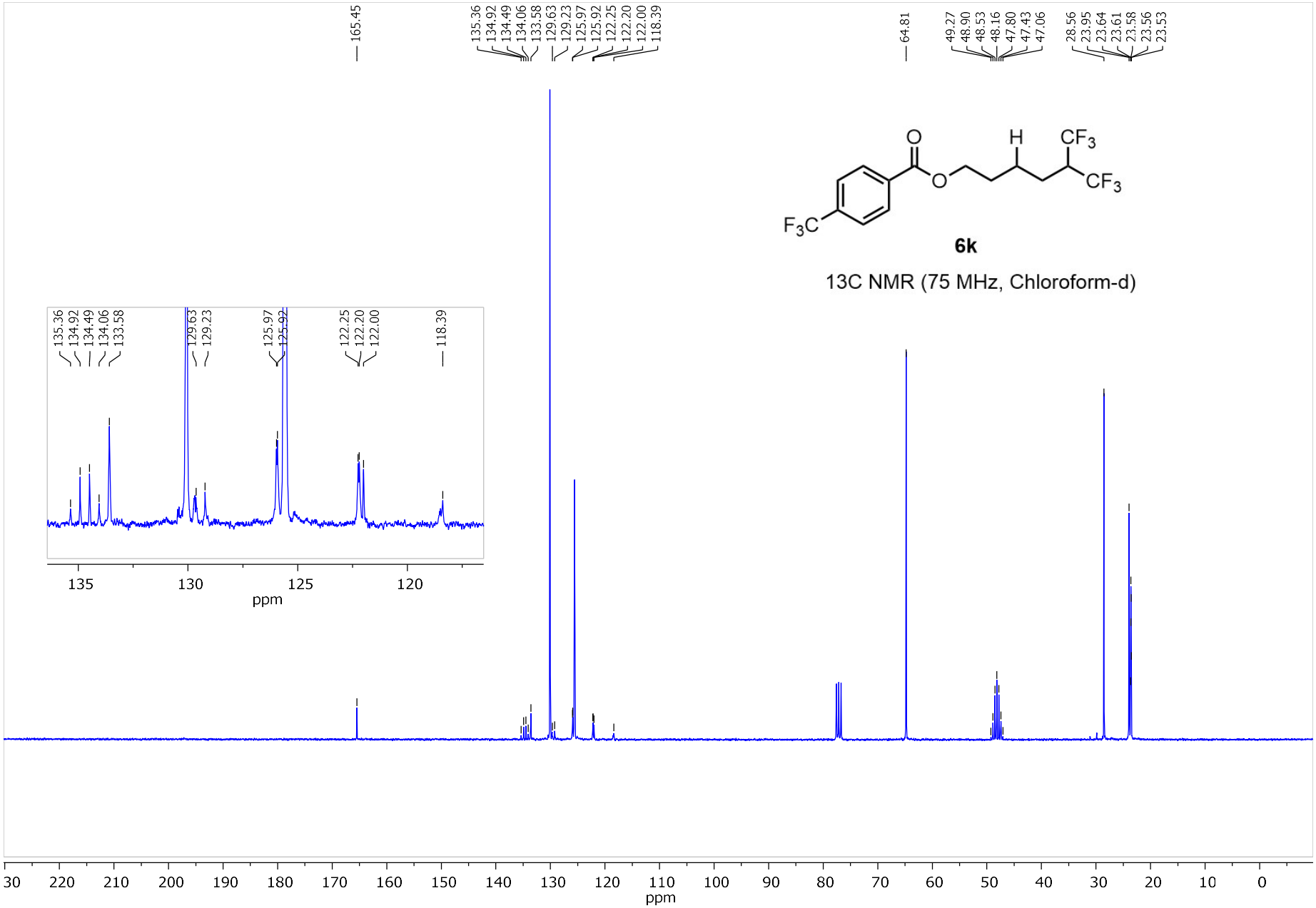

-67.79
-67.82
H
CF3
BzO
CF3
6i
19F NMR (282 MHz, Chloroform-d)
-67.79
-67.82
-67.0
-67.5
-68.0
-68.5
-69.0
-69.5
ppm
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm

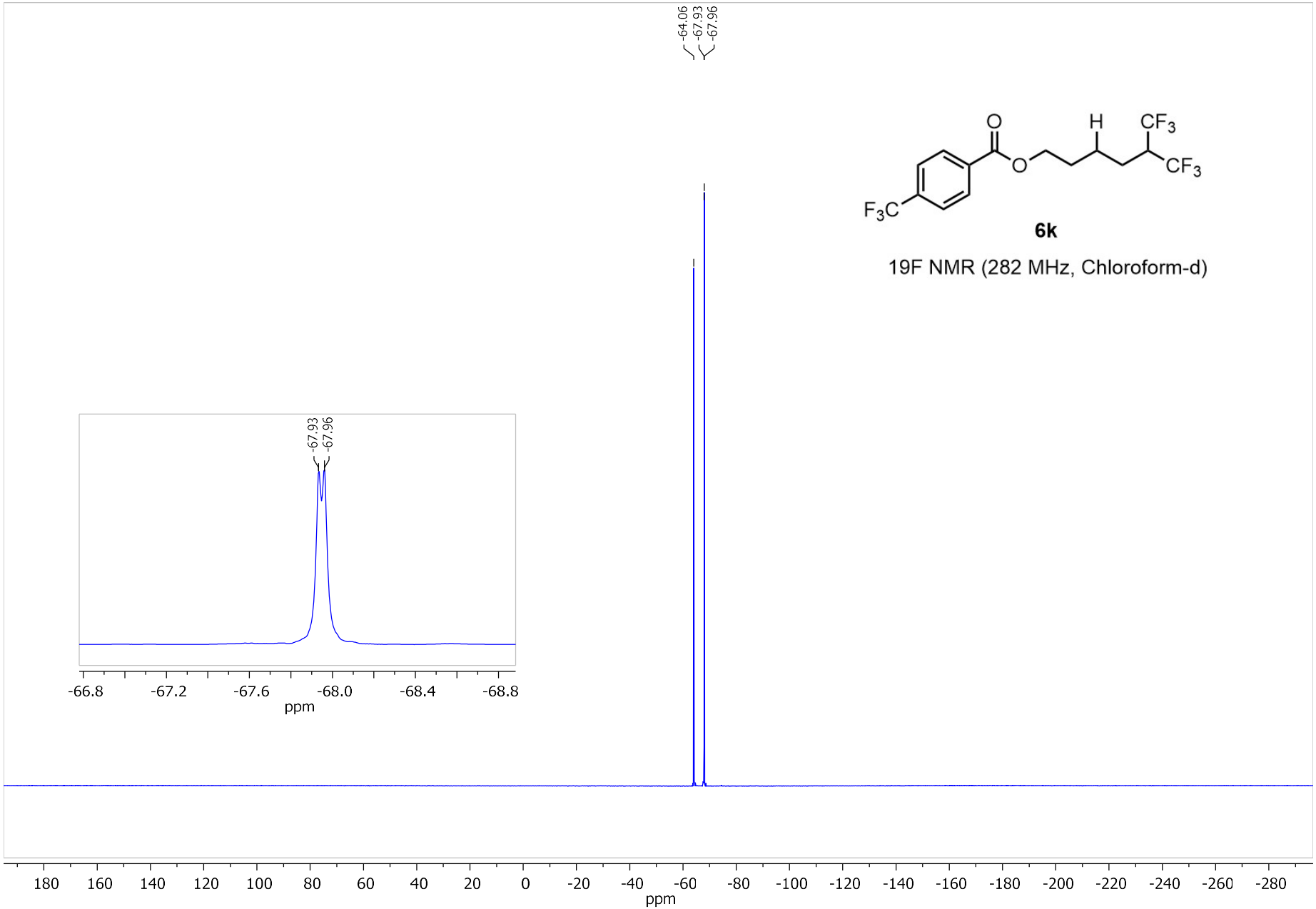

7.97
7.94
7.42
7.39
4.34
2.88
1.88
1.68
O
H
CF3
O
CF3
Cl
6j
1H NMR (300 MHz, Chloroform-d)
2.88
3.1
3.0
2.9
2.8
2.7
2.6
ppm
2.04
2.03
2.01
1.00
3.98
2.13
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

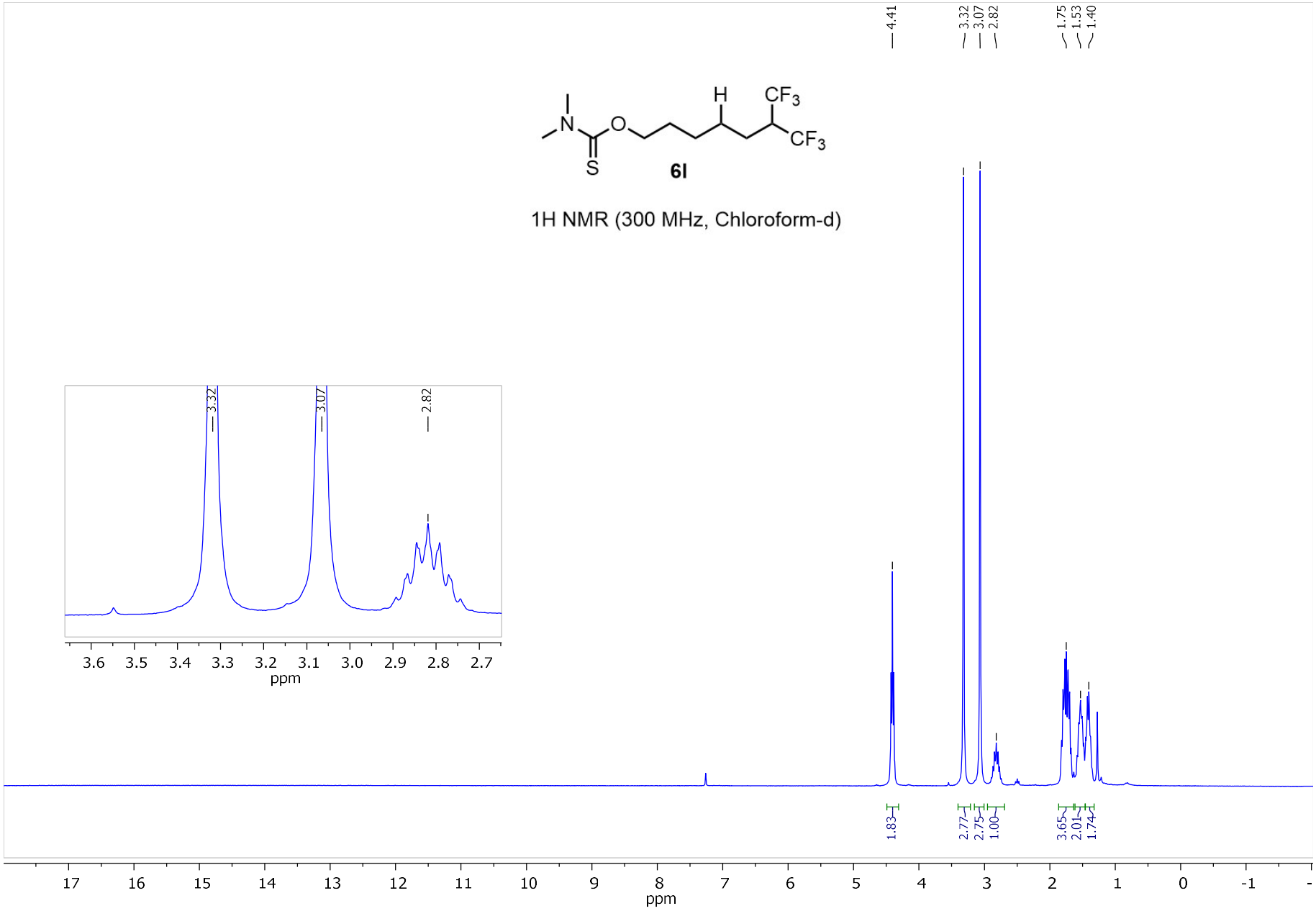

165.77
139.60
131.04
129.64
128.87
125.94
125.88
122.21
122.16
118.49
64.46
49.22
48.84
48.47
48.10
47.74
47.37
46.98
28.55
28.35
23.95
23.60
23.58
23.55
23.52
23.50
O
H
CF3
O
CF3
Cl
6j
13C NMR (75 MHz, Chloroform-d)
ppm
ppm

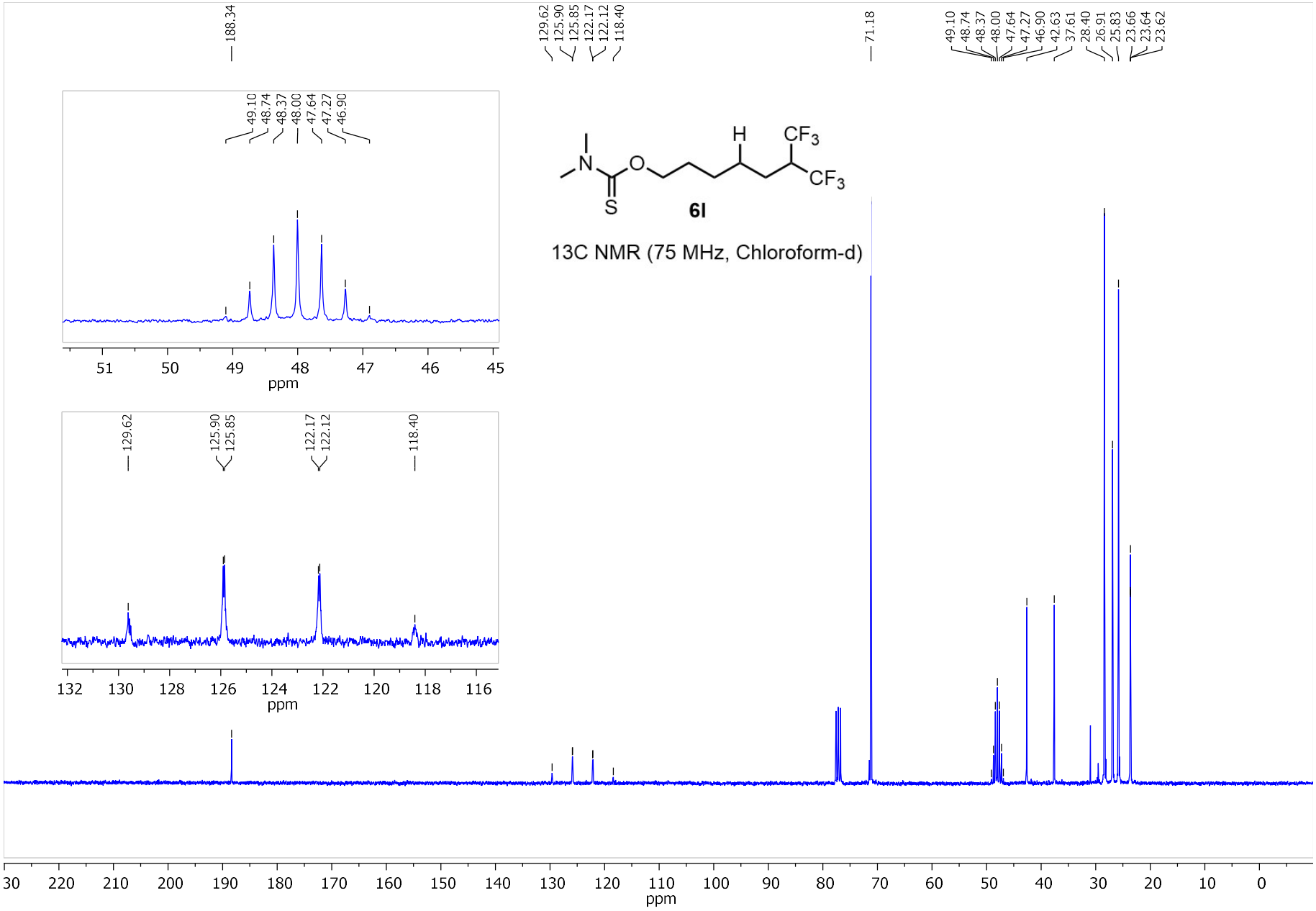

-67.87
-67.90
O
H
CF3
O
CF3
Cl
6j
19F NMR (282 MHz, Chloroform-d)
-67.87
-67.90
-67.3
-67.5
-67.7
-67.9
-68.1
-68.3
-68.5
ppm
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm

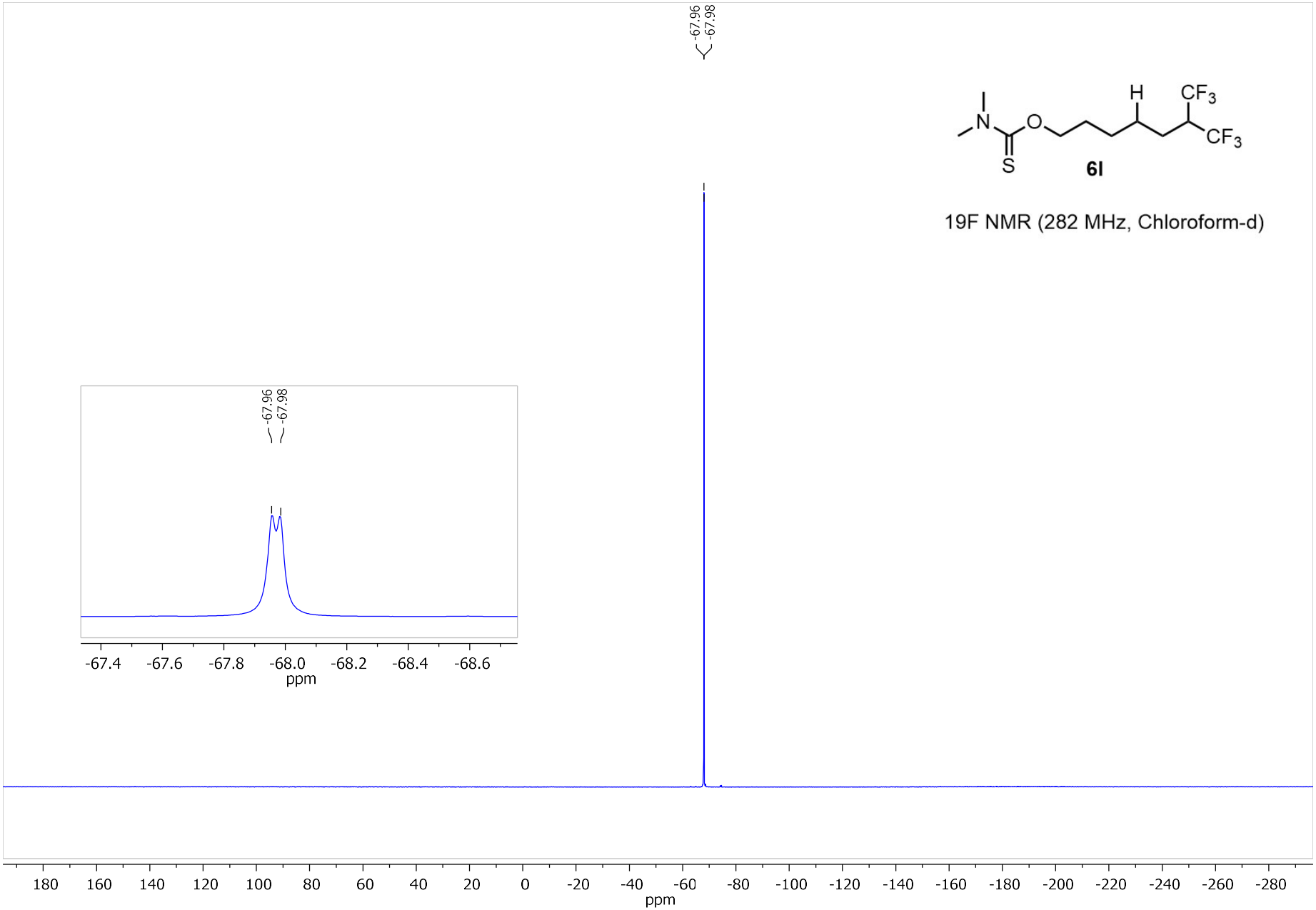

8.15
8.12
7.72
7.69
4.38
2.88
1.87
1.72
O
H
CF3
O
CF3
F3C
6k
1H NMR (300 MHz, Chloroform-d)
2.88
3.1
3.0
2.9
2.8
2.7
2.6
2.5
ppm
1.97
2.00
1.98
1.00
3.91
2.02
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

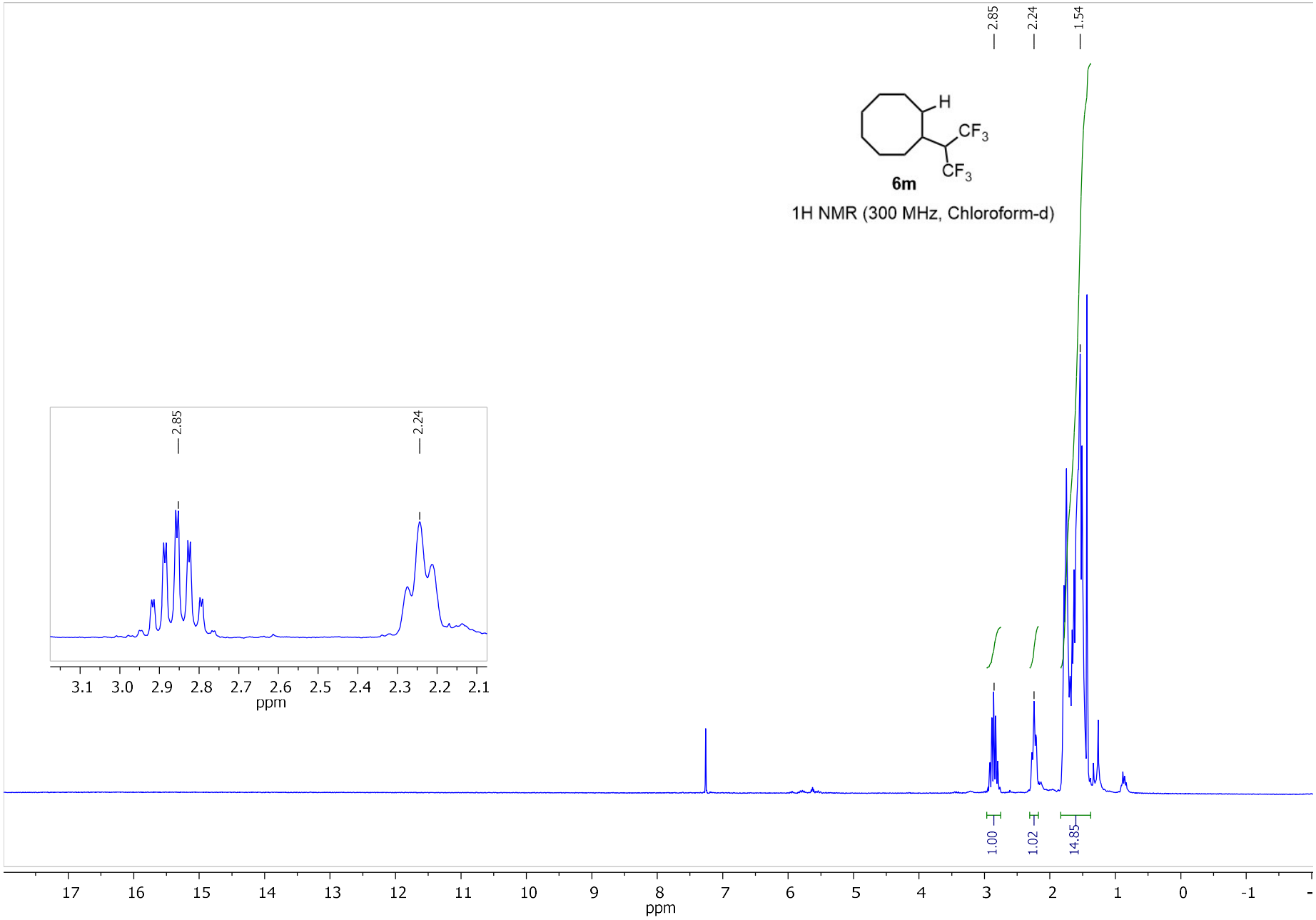

165.45
135.36
134.92
134.49
134.06
133.58
129.63
129.23
125.97
125.92
122.25
122.20
122.00
118.39
64.81
49.27
48.90
48.53
48.16
47.80
47.43
47.06
28.56
23.95
23.64
23.61
23.58
23.56
23.53
O
H
CF3
O
CF3
F3C
6k
13C NMR (75 MHz, Chloroform-d)
ppm
ppm

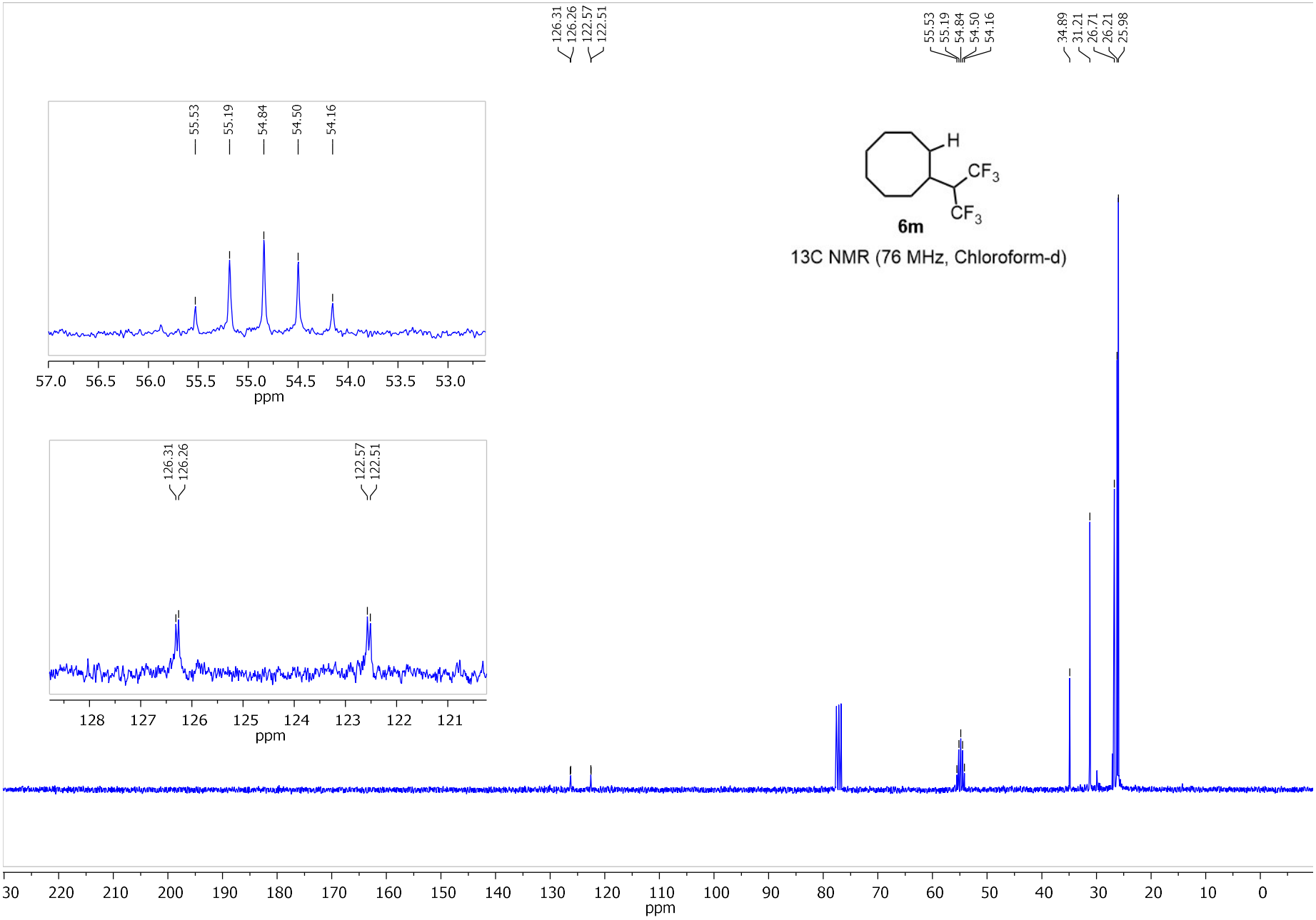

-64.06
-67.93
-67.96
O
H
CF3
O
CF3
F3C
6k
19F NMR (282 MHz, Chloroform-d)
-67.93
-67.96
-66.8
-67.2
-67.6
-68.0
-68.4
-68.8
ppm
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm

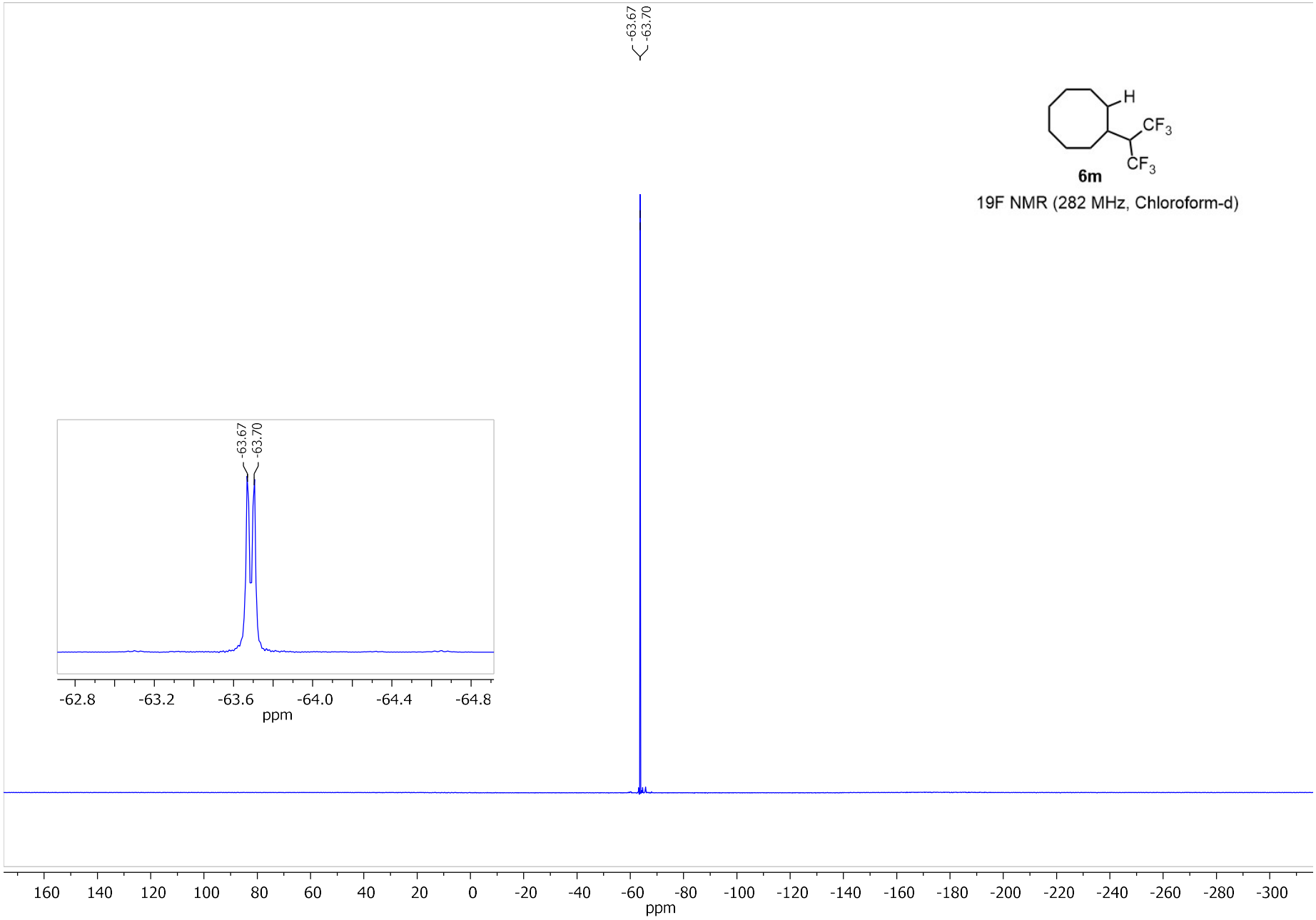

4.41
3.32
3.07
2.82
1.75
1.53
1.40
6l
1H NMR (300 MHz, Chloroform-d)
1.83
2.77
2.75
1.00
3.65
2.01
1.74
ppm

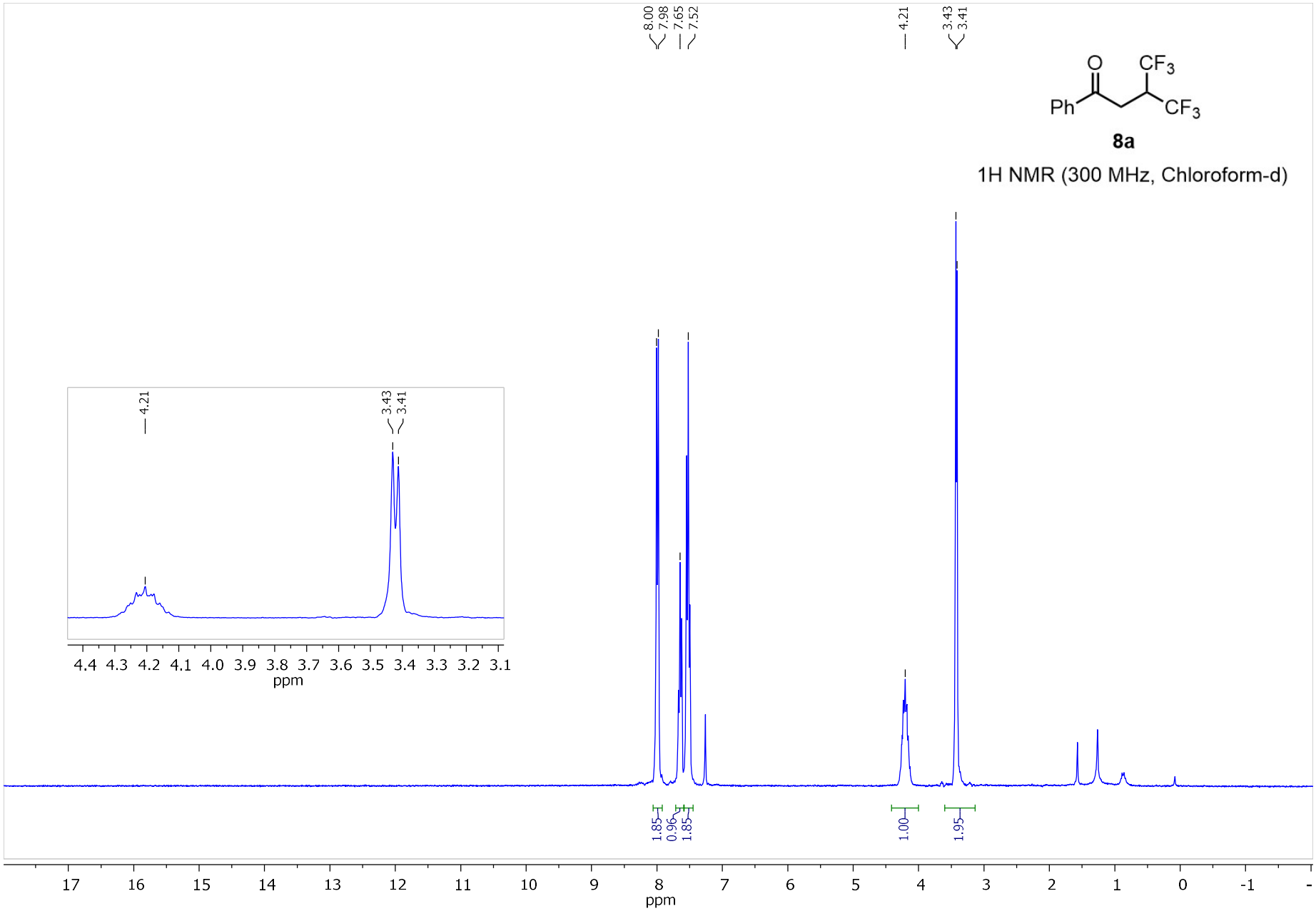

188.34
129.62
125.90
125.85
122.17
122.12
118.40
71.18
49.10
48.74
48.37
48.00
47.64
47.27
46.90
42.63
37.61
28.40
26.91
25.83
23.66
23.64
23.62
CF3
CF3
H
N
O
S
6l
13C NMR (75 MHz, Chloroform-d)
ppm

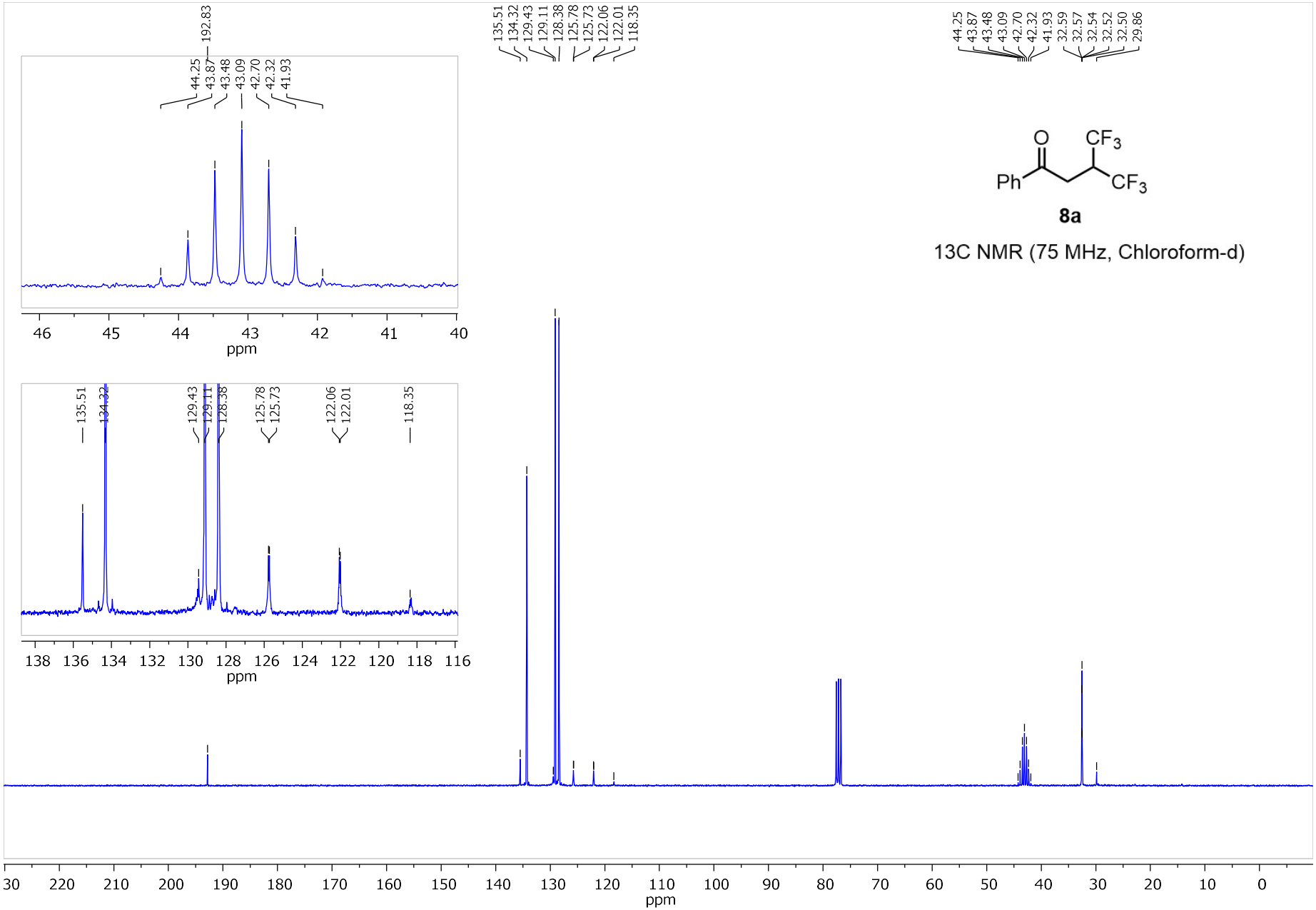

-67.96
-67.98
H
CF3
CF3
N
O
S
6l
19F NMR (282 MHz, Chloroform-d)
-67.96
-67.98
-67.4
-67.6
-67.8
-68.0
-68.2
-68.4
-68.6
ppm
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm

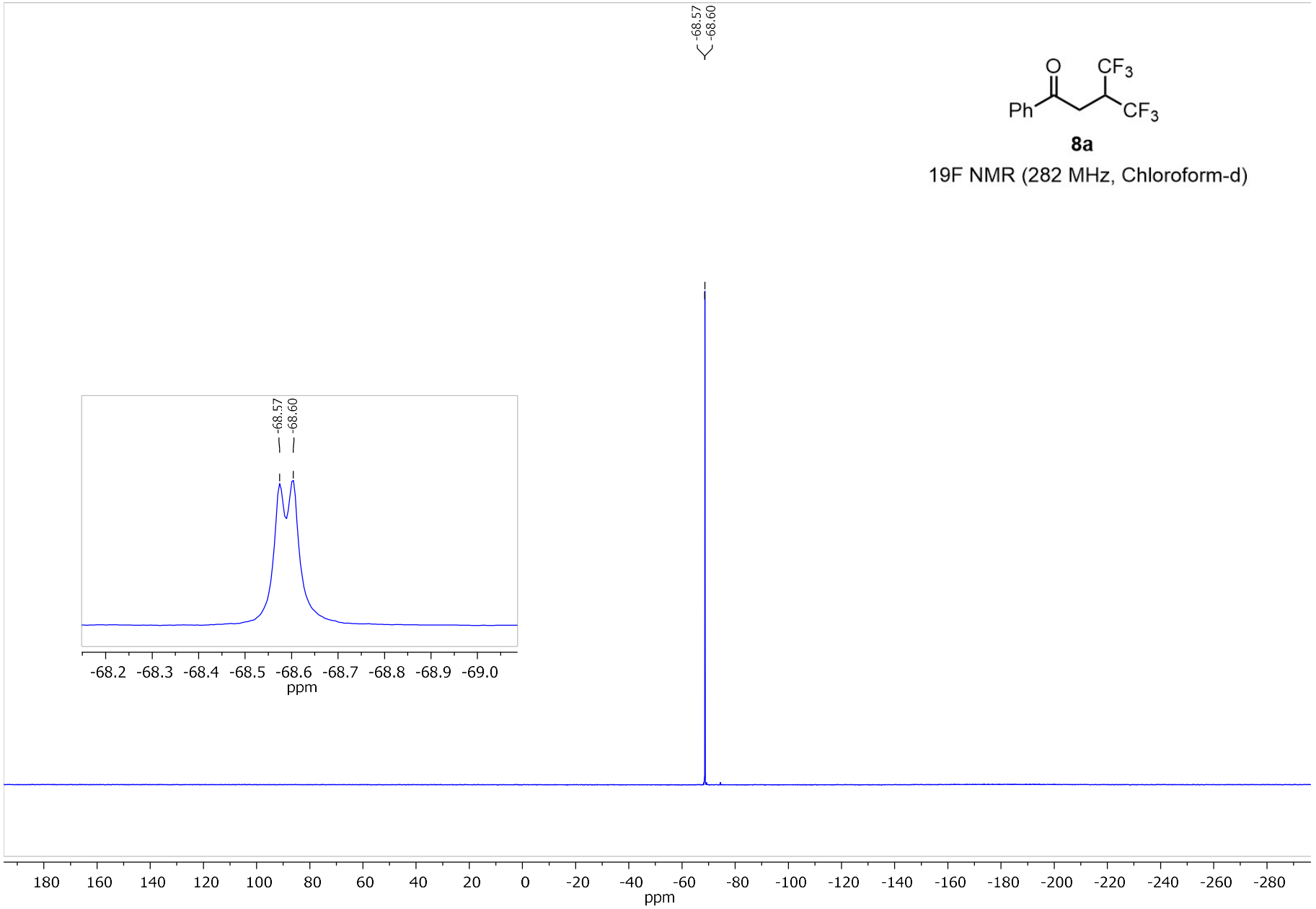

2.85
2.24
1.54
H
CF3
CF3
6m
1H NMR (300 MHz, Chloroform-d)
2.85
2.24
3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3 2.2 2.1
ppm
1.00
1.02
14.85
17 16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
ppm

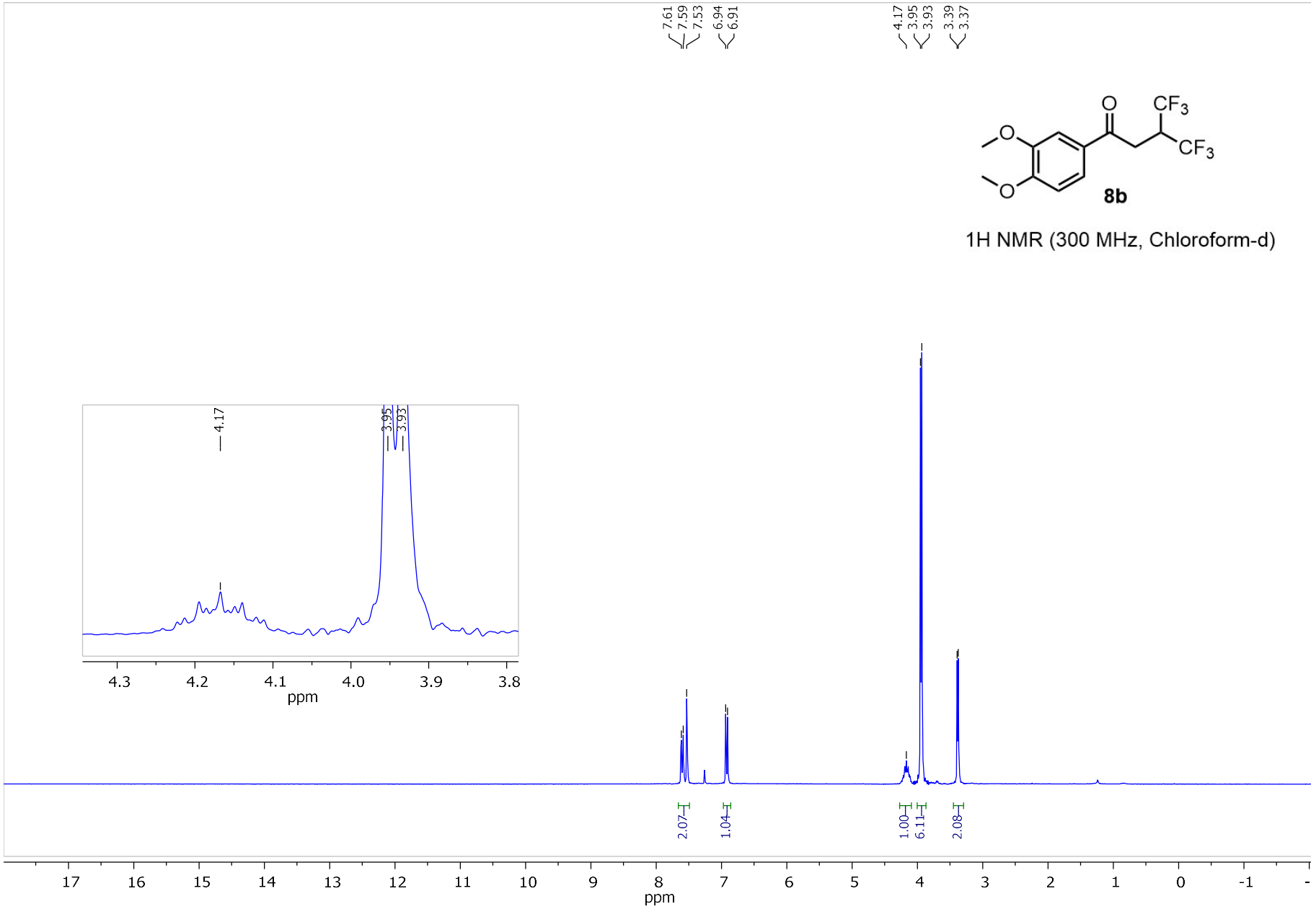

126.31
126.26
122.57
122.51
55.53
55.19
54.84
54.50
54.16
34.89
31.21
26.71
26.21
25.98
H
CF3
CF3
6m
13C NMR (76 MHz, Chloroform-d)
ppm
ppm
ppm

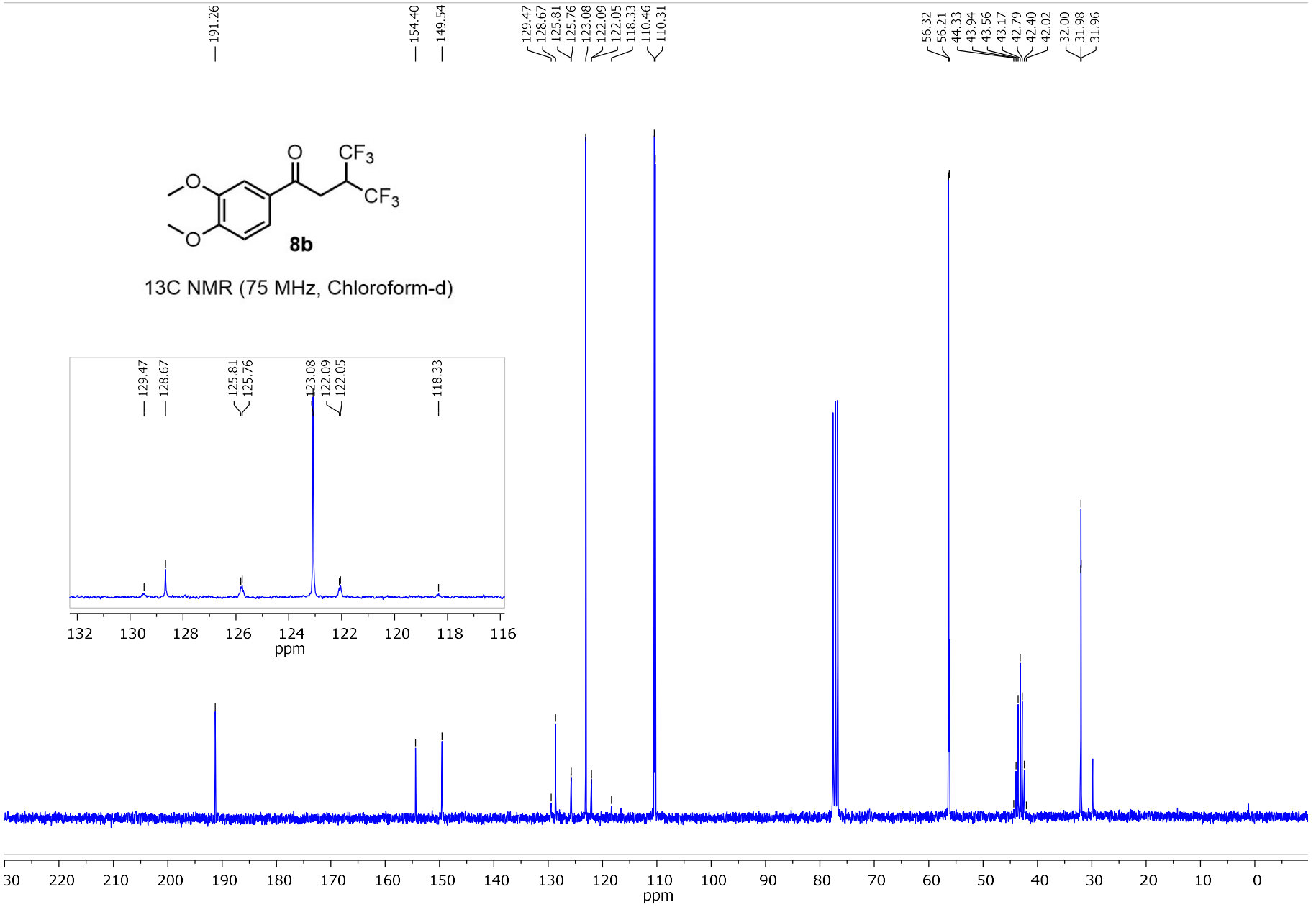

-63.67
-63.70
H
CF3
CF3
6m
19F NMR (282 MHz, Chloroform-d)
-63.67
-63.70
-62.8
-63.2
-63.6
-64.0
-64.4
-64.8
ppm
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
-300
ppm

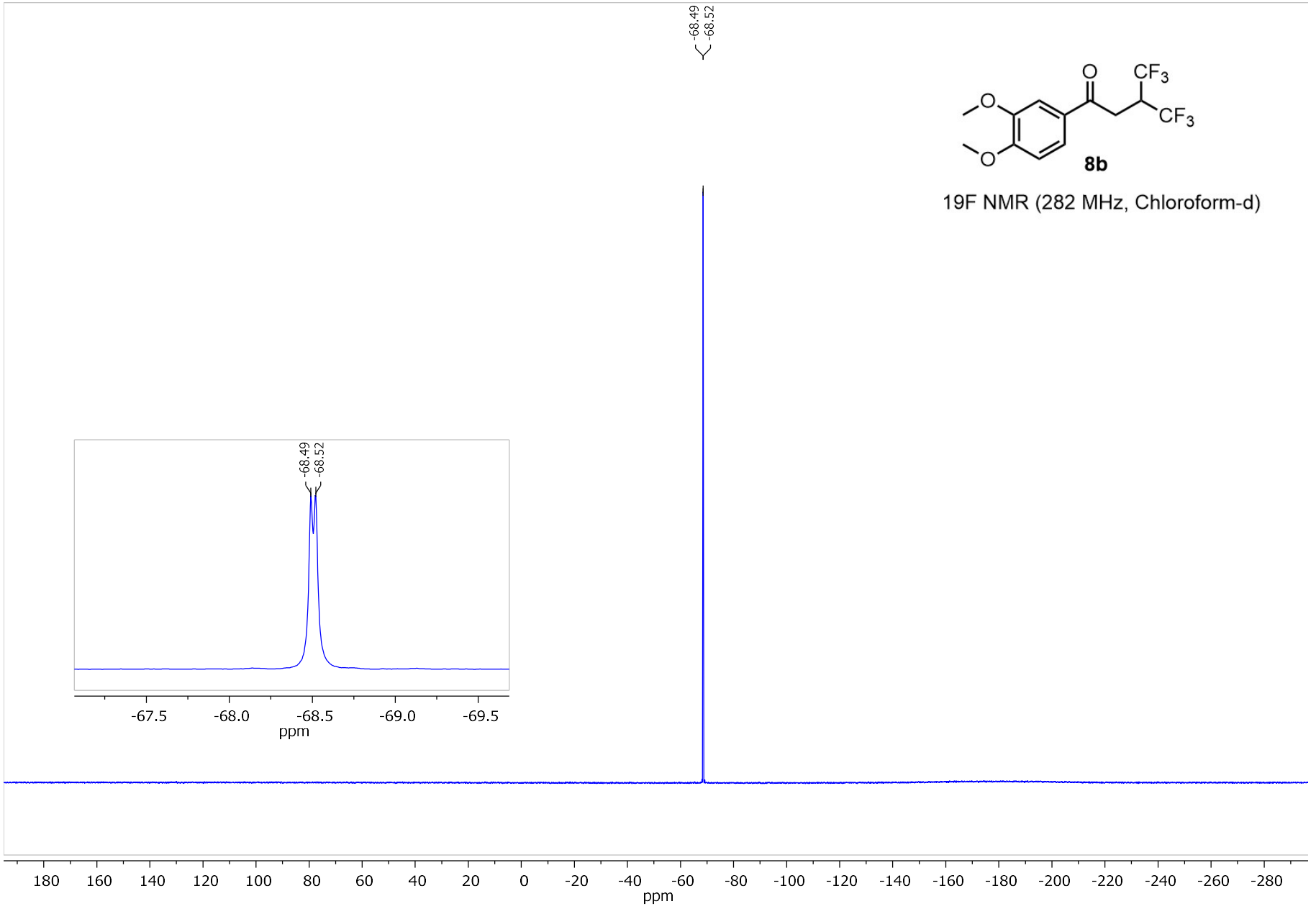

8.00
7.98
7.65
7.52
4.21
3.43
3.41
O
CF3
Ph
CF3
8a
1H NMR (300 MHz, Chloroform-d)
1.85
0.96
1.85
1.00
1.95
ppm


192.83
44.25
43.87
43.48
43.09
42.70
42.32
41.93
135.51
134.32
129.43
129.11
128.38
125.78
125.73
122.06
122.01
118.35
44.25
43.87
43.48
43.09
42.70
42.32
41.93
32.59
32.57
32.54
32.52
32.50
29.86
O
CF3
Ph
CF3
8a
13C NMR (75 MHz, Chloroform-d)
ppm
ppm
ppm


-68.57
-68.60
O
CF3
Ph
CF3
8a
19F NMR (282 MHz, Chloroform-d)
-68.57
-68.60
-68.2 -68.3 -68.4 -68.5 -68.6 -68.7 -68.8 -68.9 -69.0
ppm
180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280
ppm


7.61
7.59
7.53
6.94
6.91
4.17
3.95
3.93
3.39
3.37
O
CF3
CF3
O
O
8b
1H NMR (300 MHz, Chloroform-d)
4.17
3.95
3.93
4.3
4.2
4.1
4.0
3.9
3.8
ppm
2.07
1.04
1.00
6.11
2.08
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm


191.26
154.40
149.54
129.47
128.67
125.81
125.76
123.08
122.09
122.05
118.33
110.46
110.31
56.32
56.21
44.33
43.94
43.56
43.17
42.79
42.40
42.02
32.00
31.98
31.96
O
CF3
CF3
O
O
8b
13C NMR (75 MHz, Chloroform-d)
ppm
ppm


-68.49
-68.52
O
CF3
CF3
O
O
8b
19F NMR (282 MHz, Chloroform-d)
-68.49
-68.52
-67.5
-68.0
-68.5
-69.0
-69.5
ppm
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
ppm