

Supporting Information

Photoinduced Fluoroalkylation-Peroxidation of Alkenes Enabled by Ligand-to-Iron Charge Transfer Mediated Decarboxylation

Qiuwei Huang, Chenhao Lou, Leiyang Lv* and Zhiping Li *

Key Laboratory of Advanced Light Conversion Materials and Biophotonics, School of Chemistry and Life Resources, Renmin University of China, Beijing 100872, People's Republic of China, Beijing 100872 (China)

Email: lvleiyang2020@ruc.edu.cn; zhipingli@ruc.edu.cn

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1. General information

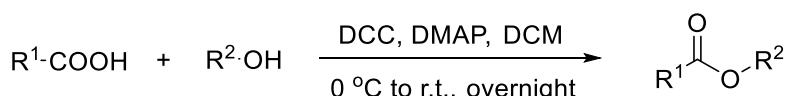
¹H NMR spectra were recorded on Bruker 400 MHz spectrometer and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm) for CDCl₃. The peak patterns are indicated as follows: s, singlet; d, doublet; dd, doublet of doublet; t, triplet; m, multiplet; q, quartet. The coupling constants, J , are reported in Hertz (Hz). ¹³C NMR spectra were obtained at Bruker 100 MHz and referenced to the internal solvent signals (central peak is 77.0 ppm in CDCl₃). ¹⁹F NMR spectra were obtained at Bruker 376 MHz and referenced to CFCl₃ (δ = 0 ppm). CDCl₃ was used as the NMR solvent. High-resolution mass spectra (HRMS) were acquired on Thermo Q-Exactive instrument (quadrupole mass analyzer) using electrospray ionization mode (ESI). Flash column chromatography was performed over silica gel 200-300. All reagents were weighed and handled in air at room temperature. All chemical reagents were purchased from Alfa, Acros, Aldrich, TCI, J&K and used without further purification. Unless otherwise stated, all reactions were carried out under a positive atmosphere of nitrogen in oven-dried or flame-dried glassware. Prior to the reaction set-up, glassware was evacuated and backfilled with nitrogen three times. Experiments upon light irradiation were carried out with a 10 W Kessil® PR160L-390nm (max 40 W) with average intensity of 300 mW/cm² (for light spectrum and other details, see: https://kessil.com/products/science_PR160L.php). The material of the irradiation vessel was Schlenk tube made of borosilicate glass. Irradiation to each tube at a distance of 3.0 cm without any filters (Figure S1). The alkene substrates were synthesized according to the reported literature.



Figure S1 Experimental set-up featuring with a Kessil® PR160L-390 nm lamp

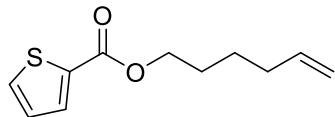
2. Synthesis of starting materials

2.1. Method A¹

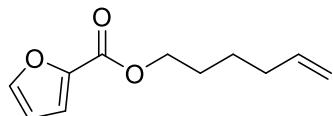


To a Schlenk tube was added alcohol (4.0 mmol), carboxylic acid (2.0 mmol), 4-dimethylamino pyridine (0.2 mmol, 10 mol%), and a stir bar. The Schlenk tube was then evacuated and backfilled with nitrogen gas three times. Dry dichloromethane (0.2 M) was added via syringe to the Schlenk tube. The Schlenk tube was then placed in an ice bath positioned on top of a stirring plate. Dicyclohexyl carbodiimide (4.0 mmol) was added to the mixture via syringe dropwise over a period of 5 min. The ice bath was then removed, allowing the reaction to return to room temperature. The reaction was left to stir overnight. After the reaction was finished, the mixture was concentrated through rotary

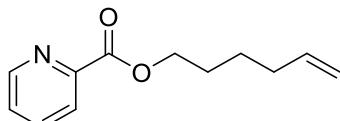
evaporation. Purification by column chromatography afforded the desired alkenes.



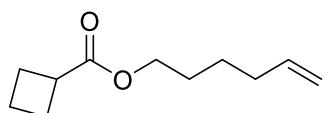
Hex-5-en-1-yl thiophene-2-carboxylate (1i) Colorless oil, 400 mg, 95% yield. ^1H NMR (400 MHz, CDCl_3) δ 7.71 (d, $J = 3.7$ Hz, 1H), 7.46 (d, $J = 4.9$ Hz, 1H), 7.01 (t, $J = 4.4$ Hz, 1H), 5.73 (ddt, $J = 16.9, 10.2, 6.7$ Hz, 1H), 5.00-4.86 (m, 2H), 4.22 (t, $J = 6.6$ Hz, 2H), 2.09-1.99 (m, 2H), 1.74-1.63 (m, 2H), 1.52-1.39 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 162.2, 138.3, 134.0, 133.2, 132.1, 127.6, 114.8, 65.0, 33.2, 28.1, 25.2.



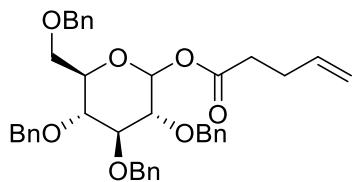
Hex-5-en-1-yl furan-2-carboxylate (1j) Colorless oil, 354 mg, 91% yield. ^1H NMR (400 MHz, CDCl_3) δ 7.58 (s, 1H), 7.17, (d, $J = 3.4$ Hz, 1H), 6.50 (dd, $J = 3.0, 1.3$ Hz, 1H), 5.81 (ddt, $J = 16.9, 10.2, 6.7$ Hz, 1H), 5.07-4.94 (m, 2H), 4.31 (t, $J = 6.7$ Hz, 2H), 2.17-2.07 (m, 2H), 1.82-1.72 (m, 2H), 1.58-1.47 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 158.7, 146.1, 144.8, 138.2, 117.6, 114.8, 111.7, 64.8, 33.2, 28.0, 25.1.



Hex-5-en-1-yl picolinate (1k) Colorless oil, 405 mg, 99% yield. ^1H NMR (400 MHz, CDCl_3) δ 8.78-8.76 (m, 1H), 8.13 (d, $J = 7.7$ Hz, 1H), 7.85 (td, $J = 7.7, 1.6$ Hz, 1H), 7.50-7.45 (m, 1H), 5.81 (ddt, $J = 16.9, 10.2, 6.7$ Hz, 1H), 5.07-4.94 (m, 2H), 4.43 (t, $J = 6.9$ Hz, 2H), 2.16-2.09 (m, 2H), 1.89-1.81 (m, 2H), 1.59-1.50 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 165.2, 149.8, 148.2, 138.2, 136.9, 126.7, 125.0, 114.8, 65.8, 33.2, 28.1, 25.1.

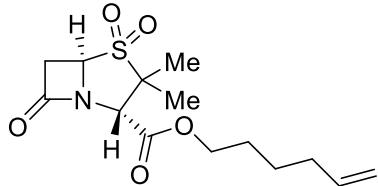


Hex-5-en-1-yl cyclobutanecarboxylate (1l) Colorless oil, 353 mg, 97% yield. ^1H NMR (400 MHz, CDCl_3) δ 7.58 (s, 1H), 7.17, (d, $J = 3.4$ Hz, 1H), 6.50 (dd, $J = 3.0, 1.3$ Hz, 1H), 5.81 (ddt, $J = 16.9, 10.2, 6.7$ Hz, 1H), 5.07-4.94 (m, 2H), 4.31 (t, $J = 6.7$ Hz, 2H), 2.17-2.07 (m, 2H), 1.82-1.72 (m, 2H), 1.58-1.47 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 158.7, 146.1, 144.8, 138.2, 117.6, 114.8, 111.7, 64.8, 33.2, 28.0, 25.1.



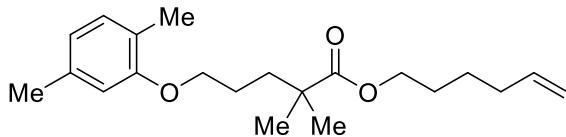
2,3,4,6-Tetra-O-benzyl-D-glucopyranosyl pent-4-enoate (5a) Colorless oil, 1240 mg, 99% yield. **5a** was obtained as a mixture of α and β configuration product. ^1H NMR (400 MHz, CDCl_3) δ 7.39-7.23 (m, 36H), 7.17-7.11 (m, 4H),

6.39 (d, $J = 3.6$ Hz, 1H, α configuration), 5.88-5.74 (m, 2H), 5.63 (d, $J = 8.0$ Hz, 1H, β configuration), 5.10-4.44 (m, 20H), 3.97-3.53 (m, 12H), 2.56-2.30 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3) δ 171.4, 171.3, 138.3, 138.0, 137.9, 137.6, 136.3, 135.2, 128.4, 128.3, 128.1, 128.0, 127.9, 127.8, 127.6, 115.7, 115.6, 94.0, 89.9, 84.8, 81.6, 81.0, 78.9, 76.9, 75.7, 75.6, 75.5, 75.3, 75.0, 73.5, 73.4, 73.1, 72.8, 68.1, 68.0, 33.5, 33.4, 28.7, 28.3.

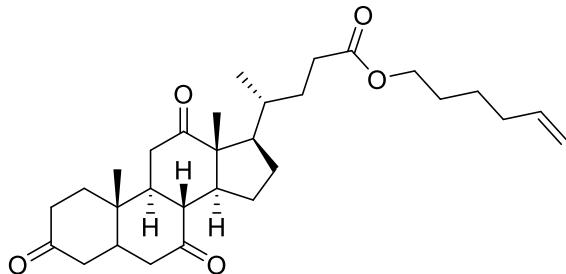


Hex-5-en-1-yl (2S,5R)-3,3-dimethyl-7-oxo-4-thia-1-azabicyclo[3.2.0]heptane-2-carboxylate 4,4-dioxide (5c)

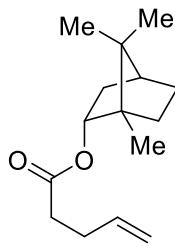
Colorless solid, 491 mg, 78% yield. ^1H NMR (400 MHz, CDCl_3) δ 5.86-5.71 (m, 1H), 5.08-4.95 (m, 2H), 4.61 (s, 1H), 4.38 (s, 1H), 4.21 (t, $J = 6.6$ Hz, 2H), 3.54-3.40 (m, 2H), 2.15-2.06 (m, 2H), 1.76-1.65 (m, 2H), 1.62 (s, 3H), 1.52-1.44 (m, 2H), 1.42 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.7, 167.0, 137.8, 115.2, 66.4, 63.3, 62.6, 61.1, 38.3, 33.0, 27.8, 25.0, 20.3, 18.6.



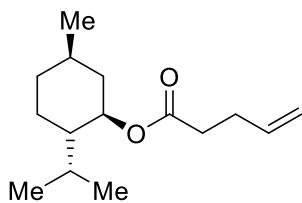
Hex-5-en-1-yl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (5d) Colorless oil, 658 mg, 99% yield. ^1H NMR (400 MHz, CDCl_3) δ 7.00 (d, $J = 7.4$ Hz, 1H), 6.65 (d, $J = 7.5$ Hz, 1H), 6.60 (s, 1H), 5.79 (ddt, $J = 17.0, 10.2, 6.7$ Hz, 1H), 5.06-4.84 (m, 2H), 4.06 (t, $J = 6.6$ Hz, 2H), 3.91 (t, $J = 5.4$ Hz, 2H), 2.30 (s, 3H), 2.17 (s, 3H), 2.12-2.03 (m, 2H), 1.79-1.68 (m, 4H), 1.68-1.59 (m, 2H), 1.52-1.39 (m, 2H), 1.21 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 177.8, 156.9, 138.3, 136.4, 130.3, 123.6, 120.7, 114.8, 111.9, 67.9, 64.3, 42.1, 37.1, 33.2, 28.1, 25.3, 25.2, 21.4, 15.7.



Hex-5-en-1-yl (5 β)-3,7,12-trioxocholan-24-olate (5e) White solid, 902 mg, 93% yield. ^1H NMR (400 MHz, CDCl_3) δ 5.80 (ddt, $J = 16.9, 10.3, 6.7$ Hz, 1H), 5.07-4.90 (m, 2H), 4.07 (t, $J = 6.6$ Hz, 2H), 3.00-2.82 (m, 3H), 2.44-2.21 (m, 8H), 2.18-1.97 (m, 8H), 1.89-1.81 (m, 2H), 1.67-1.60 (m, 3H), 1.50-1.42 (m, 2H), 1.40 (s, 3H), 1.36-1.23 (m, 4H), 1.07 (s, 3H), 0.85 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 211.8, 208.9, 208.6, 174.1, 138.3, 114.8, 64.1, 56.8, 51.7, 49.0, 48.9, 46.8, 45.6, 45.5, 44.9, 42.7, 38.6, 36.4, 36.0, 35.5, 35.2, 33.9, 33.2, 31.5, 30.4, 28.0, 27.6, 25.6, 25.2, 25.1, 24.9, 21.8, 18.6, 11.8.

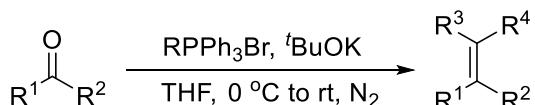


(1*S*,2*R*,4*S*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl pent-4-enoate (5f) Colorless oil, 468 mg, 99% yield. ^1H NMR (400 MHz, CDCl_3) δ 5.83 (ddt, $J = 16.5, 11.5, 6.0$ Hz, 1H), 5.12-4.96 (m, 2H), 4.93-4.85 (m, 1H), 2.47-2.30 (m, 5H), 1.99-1.89 (m, 1H), 1.78-1.70 (m, 1H), 1.69-1.64 (m, 1H), 1.36-1.17 (m, 2H), 0.99-0.92 (m, 1H), 0.90 (s, 3H), 0.87 (s, 3H), 0.83 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 173.3, 136.8, 115.4, 79.8, 48.7, 47.7, 44.9, 36.8, 33.9, 29.0, 28.0, 27.1, 19.7, 18.8, 13.4.

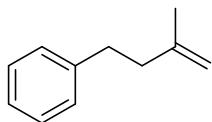


(1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl pent-4-enoate (5g) Colorless oil, 475 mg, 99% yield. ^1H NMR (400 MHz, CDCl_3) δ 5.89-5.75 (m, 1H), 5.10-4.97 (m, 2H), 4.69 (td, $J = 10.9, 4.4$ Hz, 1H), 2.41-2.35 (m, 4H), 2.01-1.94 (m, 1H), 1.92-1.80 (m, 1H), 1.73-1.62 (m, 2H), 1.54-1.42 (m, 1H), 1.37 (tt, $J = 12.0, 3.1$ Hz, 1H), 1.11-1.00 (m, 1H), 1.00-0.92 (m, 1H), 0.89 (dd, $J = 6.5, 3.2$ Hz, 6H), 0.88-0.80 (m, 1H), 0.75 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 172.6, 136.7, 115.4, 74.1, 47.0, 40.9, 34.3, 33.9, 31.4, 29.0, 26.2, 23.4, 22.0, 20.7, 16.3; HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{15}\text{H}_{26}\text{O}_2\text{Na}$ 261.1825; Found: 261.1816.

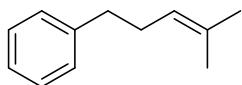
2.2 Method B



Alkyltriphenylphosphonium bromide (13 mmol) and potassium *tert*-butoxide (13 mmol) were stirred in THF (20.0 mL) at 0 °C for 1h. A solution of related ketone (10 mmol) in THF (10.0 mL) was then added drop-wise. The reaction mixture was stirred until the completion of the reaction. Then, saturated aqueous NH_4Cl was added and the aqueous layer was extracted with EtOAc. The combined organic layers were washed with saturated brine, dried over Na_2SO_4 , and concentrated in vacuum. Purification by column chromatography afforded the desired alkenes

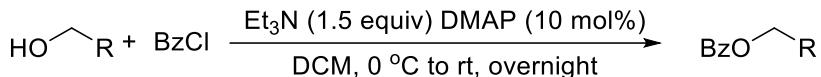


(3-methylbut-3-en-1-yl)benzene (1x) Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.27 (t, $J = 7.5$ Hz, 2H), 7.19 (d, $J = 7.9$ Hz, 3H), 4.72 (d, $J = 11.0$ Hz, 2H), 2.82-2.66 (m, 2H), 2.39-2.24 (m, 2H), 1.77 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 145.3, 142.2, 128.3, 128.3, 125.7, 110.2, 39.6, 34.3, 22.6.

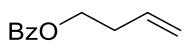


(4-Methylpent-3-en-1-yl)benzene (1ae) Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.30-7.23 (m, 2H), 7.21-7.13 (m, 3H), 5.17 (t, $J = 7.2$ Hz, 1H), 2.62 (t, $J = 7.7$ Hz, 2H), 2.33-2.24 (m, 2H), 1.68 (s, 3H), 1.56 (s, 3H). Spectral data match those previously reported.²

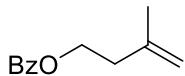
2.3 Method C³



To a solution of alcohol (2.0 mmol, 1.0 equiv), DAMP (0.2 mmol, 10 mol%), and triethylamine (3.0 mmol, 1.5 equiv) in anhydrous CH_2Cl_2 was added benzoyl chloride (3.6 mmol, 1.3 equiv) dropwise at 0 °C. The resulting mixture was stirred at 0 °C for 0.5 hour, and then at room temperature overnight. After completion, the reaction was quenched with water and extracted with CH_2Cl_2 . The combined organic layers were dried with Na_2SO_4 , filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate) to afford the corresponding pure product.

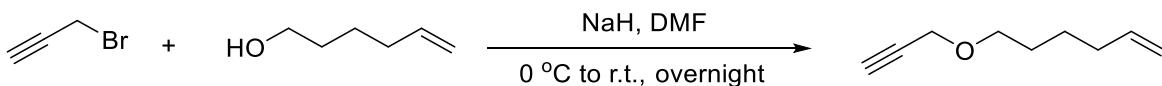


But-3-en-1-yl benzoate (1b) Colorless oil, 352 mg, 100% yield. ^1H NMR (400 MHz, CDCl_3) δ 7.98-7.92 (m, 2H), 7.50-7.41 (m, 1H), 7.39-7.29 (m, 2H), 5.86-5.71 (m, 1H), 5.12-4.97 (m, 2H), 4.29 (t, $J = 6.7$ Hz, 2H), 2.51-2.34 (m, 2H). Spectral data match those previously reported.⁴



3-methylbut-3-en-1-yl benzoate (1t) Colorless oil, 380 mg, 100% yield. ^1H NMR (400 MHz, CDCl_3) δ 8.07 – 8.00 (m, 2H), 7.59 – 7.50 (m, 1H), 7.47 – 7.39 (m, 2H), 4.83 (d, $J = 10.8$ Hz 2H), 4.44 (t, $J = 6.8$ Hz, 2H), 2.48 (t, $J = 6.8$, 2H), 1.81 (t, $J = 1.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.5, 141.7, 132.8, 130.4, 129.5, 128.3, 112.4, 63.1, 36.8, 22.5.

2.4 Method D⁵

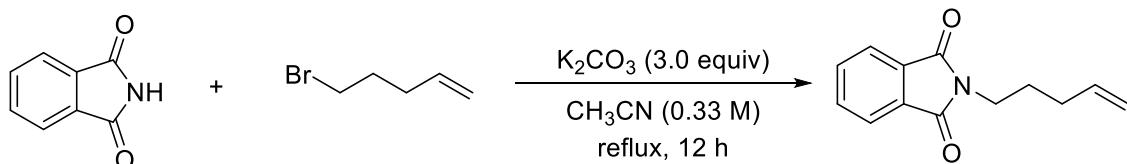


To a suspension of NaH in dry DMF, the alcohol solution of dry DMF was added dropwise at 0 °C. After 2 h, propargyl bromide (80% in toluene) was added at 0 °C and the resulting mixture was stirred at room temperature for 12 h. The reaction was quenched by water and then extracted with diethyl ether. The combined organic layers were washed with water and brine, dried over Na_2SO_4 and filtered. The resulting residue was purified by silica gel flash chromatography.

6-(Prop-2-nyloxy)hex-1-ene (1f) yellow oil, 262 mg, 95% yield. ^1H NMR (400 MHz, CDCl_3) δ 5.87-5.73 (m, 1H), 5.06-4.90 (m, 2H), 4.13 (d, $J = 2.3$ Hz, 2H), 3.52 (t, $J = 6.5$ Hz, 2H), 2.42 (t, $J = 2.2$ Hz, 1H), 2.12-2.03 (m, 2H),

1.66-1.56 (m, 2H), 1.52-1.41 (m, 2H). Spectral data match those previously reported.⁵

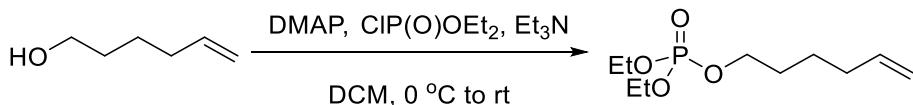
2.5 Method E⁶



To a solution of phthalimide (5 mmol, 1.0 equiv) and K_2CO_3 (15 mmol, 3.0 equiv) in CH_3CN (15 mL) was added 5-bromo-1-pentene (7.5 mmol, 1.5 equiv), and the mixture was refluxed for 12 h. The reaction mixture was concentrated, and the residue was partitioned between CH_2Cl_2 and water. The aqueous layer was extracted with CH_2Cl_2 . The combined organic extracts were washed with water, dried over anhydrous Na_2SO_4 , and concentrated in vacuo. The resulting residue was purified by silica gel flash chromatography.

2-(But-3-en-1-yl)isoindoline-1,3-dione (1g) colorless oil, 1076 mg, 99% yield. ^1H NMR (400 MHz, CDCl_3) δ 7.87-7.81 (m, 2H), 7.74-7.68 (m, 2H), 5.89-5.75 (m, 1H), 5.10-4.94 (m, 2H), 3.70 (t, $J = 7.4$ Hz, 2H), 2.17-2.08 (m, 2H), 1.85-1.74 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 168.4, 137.3, 133.8, 132.1, 115.3, 37.6, 30.9, 27.6. Spectral data match those previously reported.⁶

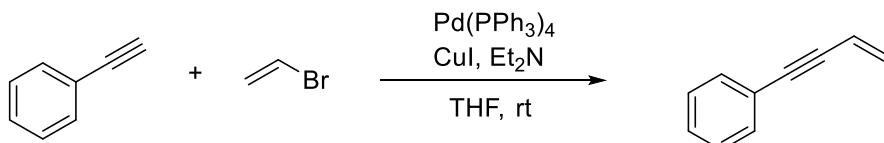
2.6 Method F



Diethyl chlorophosphate (2.0 mmol) was added dropwise to a solution of hex-5-en-1-ol (2.4 mmol), 4-dimethylaminopyridine (0.4 mmol, 20 mol%), and triethylamine (3.0 mmol) in dichloromethane (10 mL) at 0°C . The reaction mixture was warmed up over 2 h to rt and the reaction mixture was stirred overnight at rt. The product mixture was filtered through a pad of silica gel. The filtrates were combined and the combined filtrates were concentrated to dryness. The residue obtained was purified by flash-column chromatography.

Hex-5-en-1-yl phosphate (1k) colorless oil, 1.13 g, 93% yield. ^1H NMR (400 MHz, CDCl_3) δ 5.85-5.70 (m, 1H), 5.07-4.88 (m, 2H), 4.17-4.07 (m, 4H), 4.06-4.00 (m, 2H), 2.13-2.05 (m, 2H), 1.77-1.63 (m, 2H), 1.57-1.41 (m, 2H), 1.38-1.29 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 138.2, 138.1, 114.9, 114.8, 67.7 ($d, J_{\text{C-P}} = 6.0$ Hz), 63.6 ($d, J_{\text{C-P}} = 5.9$ Hz), 33.1, 29.6 ($d, J_{\text{C-P}} = 6.9$ Hz), 24.6, 16.1 ($d, J_{\text{C-P}} = 6.7$ Hz); ^{31}P NMR (162 MHz, CDCl_3) δ -0.9.

2.7 Method G⁷

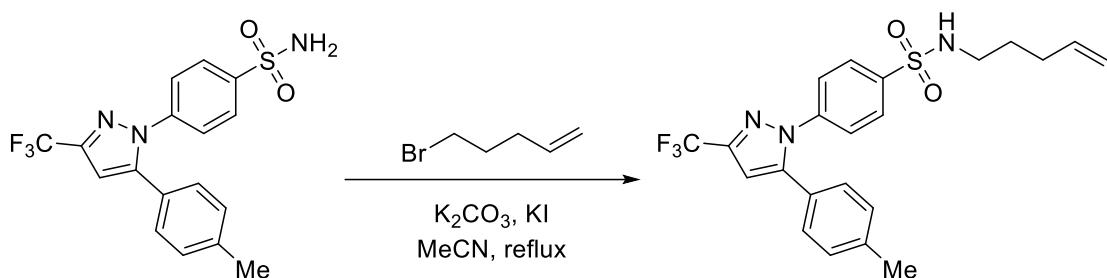


To an oven-dried 50-mL round-bottom flask equipped with a magnetic stirring bar was added $\text{Pd}(\text{PPh}_3)_4$ (2.00 mol%) and CuI (10.0 mol%) in glovebox. The flask was sealed with a rubber septum and removed from the glovebox. Vinyl bromide (1.0 M in THF , 2.50 equiv) was added followed by freshly distilled and degassed Et_2NH (5.0 equiv).

The mixture was allowed to stir at ambient temperature for ca. 5 min and subsequently phenylacetylene (5.0 mmol) was added as a solution in THF (2.0 mL) dropwise. The mixture was then allowed to stir at ambient temperature overnight. The reaction contents were then filtered through a pad of celite, eluting with Et₂O. The solution was then concentrated in vacuo. The residue obtained was purified by flash-column chromatography.

But-3-en-1-yn-1-ylbenzene (1q) colorless oil .640 mg, 99% yield. ¹H NMR (400 MHz, CDCl₃) δ 7.48-7.41 (m, 2H), 7.34-7.26 (m, 3H), 6.01 (dd, *J* = 17.6, 11.1, 1H), 5.73 (dd, *J* = 17.6, 2.0 Hz, 1H), 5.54 (dd, *J* = 11.1, 2.0 Hz, 1H). Spectral data match those previously reported.⁷

2.8 Method H⁸

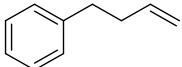
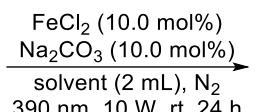
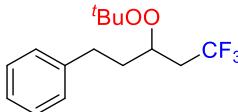


To a suspension of celecoxib (2.0 mmol, 1.0 equiv), K₂CO₃ (4 mmol, 2.0 equiv), KI (0.2 mmol, 10 mol%) in MeCN (6 mL) was added 6-bromopent-1-ene (2.4 mmol, 1.2 equiv). After being stirred at reflux for 12 h, the mixture was cooled to RT. The solvent was removed under reduced pressure. The residue was diluted with H₂O and CH₂Cl₂. The aqueous layer was extracted 3 times with CH₂Cl₂. The combined organic phase was dried over anhydrous MgSO₄, filtered, and concentrated under reduced pressure. The resulting residue was purified by column chromatography.

N-(pent-5-en-1-yl)-4-(5-(p-tolyl)-3-(trifluoromethyl)-1H-pyrazol-1-yl)benzenesulfonamide (5b) white solid, 668 mg, 74% yield. ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, *J* = 7.7 Hz, 2H), 7.47 (d, *J* = 7.7 Hz, 2H), 7.23-7.05 (m, 4H), 6.75 (s, 1H), 5.78-5.61 (m, 1H), 5.08-4.88 (m, 3H), 3.03-2.87 (m, 2H), 2.37 (s, 3H), 2.09-1.97 (m, 2H), 1.64-1.50 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 145.2, 144.0 (q, *J*_{C-F} = 38.9 Hz), 142.4, 139.7, 139.4, 137.0, 129.7, 128.6, 128.0, 125.6, 125.5, 121.0 (q, *J*_{C-F} = 268.6 Hz), 115.7, 106.2, 42.6, 30.5, 28.5, 21.2; ¹⁹F NMR (376 MHz, CDCl₃) δ -62.4 (s, 3F).

3. Additional optimization studies

Table S1 Additional optimization studies

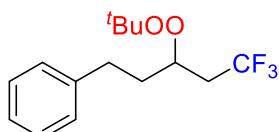
				
entry	solvent			yield of 4a
1	Acetone			51
2	Ph–Cl			0
3	Ph–CF ₃			0
4 ^c	MeCN			33
5 ^d	MeCN			0

^aReaction conditions: **1a** (0.2 mmol), **2a** (0.6 mmol), ^tBuOOH **3** (0.8 mmol, 5–6 M in decane), catalyst (10.0 mol%), base (10.0 mol%), solvent (2.0 mL), 390 nm (10 W), r.t., 24 h in a Schlenk tube under N₂ unless otherwise noted.

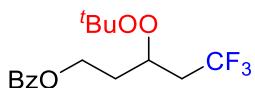
^bNMR yields are based on **1a** and determined by ¹H NMR using mesitylene as an internal standard. ^c1.0 Equiv of Na₂CO₃. ^dAir instead of N₂

4. Experimental procedures and characterization data of products

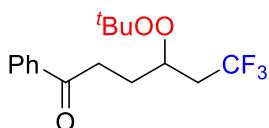
To a mixture of alkene **1** (0.2 mmol), acid **2** (0.6 mmol), Na₂CO₃ (0.02 mmol) and FeCl₂ (2.5 mg, 0.02 mmol), MeCN (2.0 mL) was added under nitrogen at room temperature. Then *tert*-butyl hydroperoxide (TBHP, 0.8 mmol, 5–6 M in decane) was added into the mixture under nitrogen at room temperature. The resulting mixture was stirred under rt, 390 nm, 10 W for 24 h. The resulting reaction solution was directly filtered through a pad of silica by chloroform. The solvent was evaporated in vacuo to give the crude products. NMR yields are determined by ¹H NMR using mesitylene as an internal standard. Solvent was evaporated and the residues were purified by flash column chromatography on silica gel with an eluent to afford the pure products.



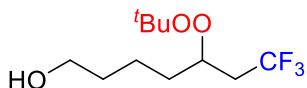
(3-(*tert*-Butylperoxy)-5,5,5-trifluoropentyl)benzene (4a**)** (44.7 mg, 77%). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:5, R_f = 0.5). The title compound was obtained as colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.32–7.26 (m, 2H), 7.23–7.16 (m, 3H), 4.25–4.18 (m, 1H), 2.87–2.76 (m, 1H), 2.75–2.63 (m, 2H), 2.31–2.15 (m, 1H), 2.06–1.88 (m, 2H), 1.25 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 141.4, 128.4, 128.3, 126.1 (q, J_{C-F} = 276.9 Hz), 126.0, 80.2, 76.7 (q, J_{C-F} = 2.7 Hz), 36.9 (q, J_{C-F} = 27.6 Hz), 34.5, 31.5, 26.4; ¹⁹F NMR (376 MHz, CDCl₃) δ -63.1 (s, 3F). HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₁₅H₂₁F₃O₂Na 313.1386; Found: 313.1383.



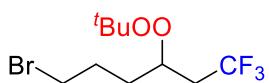
3-(*tert*-Butylperoxy)-5,5,5-trifluoropentyl benzoate (4b**)** (58.8 mg, 88%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.6). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 8.03 (d, J = 7.7 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.45 (t, J = 7.6 Hz, 2H), 4.47 (t, J = 6.1 Hz, 2H), 4.45-4.38 (m, 1H), 2.85-2.68 (m, 1H), 2.40-2.25 (m, 1H), 2.20-2.06 (m, 2H), 1.23 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.4, 133.0, 130.1, 129.5, 128.4, 125.9 (q, $J_{\text{C}-\text{F}}$ = 276.5 Hz) 80.5, 74.5 (q, $J_{\text{C}-\text{F}}$ = 2.9 Hz), 61.2, 37.1 (q, $J_{\text{C}-\text{F}}$ = 27.6 Hz), 32.1, 26.3; ^{19}F NMR (376 MHz, CDCl_3) δ -63.2 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{16}\text{H}_{21}\text{F}_3\text{O}_4\text{Na}$ 357.1284; Found: 357.1276.



4-(*tert*-Butylperoxy)-6,6,6-trifluoro-1-phenylhexan-1-one (4c**)** (38.8 mg, 61%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:20, R_f = 0.3). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 8.01-7.93 (m, 2H), 7.59-7.53 (m, 1H), 7.49-7.41 (m, 2H), 4.36-4.25 (m, 1H), 3.19-3.08 (m, 2H), 2.80-2.56 (m, 1H), 2.36-2.12 (m, 2H), 2.10-1.95 (m, 1H), 1.22 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 199.2, 136.9, 133.1, 128.6, 128.0, 126.1 (q, $J_{\text{C}-\text{F}}$ = 277.2 Hz), 80.3, 76.4 (q, $J_{\text{C}-\text{F}}$ = 3.1 Hz), 37.2 (q, $J_{\text{C}-\text{F}}$ = 28.2 Hz), 34.1, 27.4, 26.4; ^{19}F NMR (376 MHz, CDCl_3) δ -63.2 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{16}\text{H}_{21}\text{F}_3\text{O}_3\text{Na}$ 341.1335; Found: 341.1317.

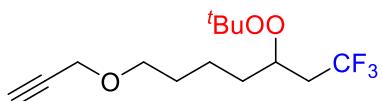


5-(*tert*-Butylperoxy)-7,7,7-trifluoroheptan-1-ol (4d**)** (21.2 mg, 41%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:3, R_f = 0.5). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 4.22-4.14 (m, 1H), 3.66 (t, J = 6.6 Hz, 2H), 2.75-2.58 (m, 1H), 2.29-2.11 (m, 1H), 1.71-1.64 (m, 2H), 1.63-1.56 (m, 2H), 1.54-1.40 (m, 3H), 1.24 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 126.0 (q, $J_{\text{C}-\text{F}}$ = 276.9 Hz), 80.3, 77.3 (q, $J_{\text{C}-\text{F}}$ = 2.7 Hz), 62.6, 36.9 (q, $J_{\text{C}-\text{F}}$ = 27.2 Hz), 32.5, 26.4, 21.4; ^{19}F NMR (376 MHz, CDCl_3) δ -63.1 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{11}\text{H}_{21}\text{F}_3\text{O}_3\text{Na}$ 281.1335; Found: 281.1329.

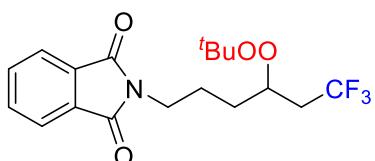


6-Bromo-3-(*tert*-butylperoxy)-1,1,1-trifluorohexane (4e**)** (33.8 mg, 55%). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:5, R_f = 0.5). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 4.24-4.15 (m, 1H), 3.44 (t, J = 6.6 Hz, 2H), 2.78-2.62 (m, 1H), 2.28-2.13 (m, 1H), 2.12-1.90 (m, 2H), 1.89-1.71 (m, 2H), 1.23 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 125.9 (q, $J_{\text{C}-\text{F}}$ = 277.0 Hz), 80.3, 76.6 (q, $J_{\text{C}-\text{F}}$ = 2.6 Hz), 37.0 (q, $J_{\text{C}-\text{F}}$ = 27.5 Hz), 33.3, 31.5, 28.5, 26.4; ^{19}F NMR (376 MHz, CDCl_3) δ -63.2 (s, 3F);

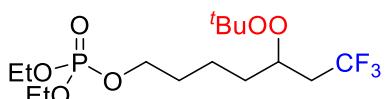
HRMS (ESI) m/z : [M+Na]⁺ Calcd for C₁₀H₁₈BrF₃O₂Na 329.0334; Found: 329.0325.



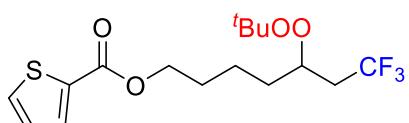
3-(tert-Butylperoxy)-1,1,1-trifluoro-7-(prop-2-yn-1-yloxy)heptane (4f) (36.1 mg, 61%). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:1, R_f = 0.4). The title compound was obtained as colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 4.21-4.15 (m, 1H), 4.13 (d, *J* = 2.4 Hz, 2H), 3.53 (t, *J* = 6.3 Hz, 2H), 2.71-2.59 (m, 1H), 2.41 (t, *J* = 2.3 Hz, 1H), 2.25-2.14 (m, 1H), 1.73-1.59 (m, 4H), 1.56-1.42 (m, 2H), 1.23 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 126.1 (q, *J*_{C-F} = 277.1 Hz), 80.3, 79.9, 77.4 (q, *J*_{C-F} = 2.4 Hz), 74.1, 69.8, 58.0, 36.9 (q, *J*_{C-F} = 27.6 Hz), 32.5, 29.3, 26.4, 21.8; ¹⁹F NMR (376 MHz, CDCl₃) δ -63.1 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for C₁₄H₂₃F₃O₃Na 319.1492; Found: 319.1486.



2-(4-(tert-Butylperoxy)-6,6,6-trifluorohexyl)isoindoline-1,3-dione (4g) (56.0 mg, 75%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.3). The title compound was obtained as colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.86-7.83 (m, 2H), 7.74-7.70 (m, 2H), 4.25-4.16 (m, 1H), 3.72 (t, *J* = 6.9 Hz, 2H), 2.72-2.57 (m, 1H), 2.27-2.12 (m, 1H), 1.93-1.83 (m, 1H), 1.83-1.76 (m, 1H), 1.75-1.67 (m, 2H), 1.21 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 168.3, 133.9, 132.1, 125.9 (q, *J*_{C-F} = 277.0 Hz), 123.2, 80.3, 76.7 (q, *J*_{C-F} = 2.6 Hz), 37.7, 36.8 (q, *J*_{C-F} = 27.5 Hz), 30.1, 26.3, 24.3; ¹⁹F NMR (376 MHz, CDCl₃) δ -63.2 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for C₁₈H₂₂F₃NO₄Na 396.1393; Found: 396.1383.

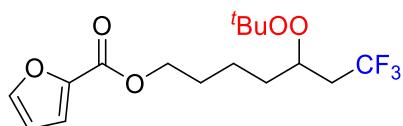


5-(tert-Butylperoxy)-7,7,7-trifluoroheptyl diethyl phosphate (4h) (53.6 mg, 68%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:1, R_f = 0.3). The title compound was obtained as colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 4.20-4.15 (m, 1H), 4.15-4.07 (m, 4H), 4.07-4.00 (m, 2H), 2.76-2.58 (m, 1H), 2.28-2.06 (m, 1H), 1.76-1.63 (m, 4H), 1.61-1.44 (m, 2H), 1.34 (t, *J* = 7.1 Hz, 6H), 1.23 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 126.1 (q, *J*_{C-F} = 276.4 Hz), 80.3, 77.2 (q, *J*_{C-F} = 2.5 Hz), 67.2 (d, *J*_{C-P} = 5.9 Hz), 63.6 (d, *J*_{C-P} = 5.9 Hz), 36.9 (q, *J*_{C-F} = 27.6 Hz), 32.3, 30.1 (d, *J*_{C-P} = 6.7 Hz), 26.3, 21.2, 16.1 (d, *J*_{C-P} = 6.9 Hz); ¹⁹F NMR (376 MHz, CDCl₃) δ -63.1 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for C₁₅H₃₀F₃PO₆Na 417.1624; Found: 417.1612.

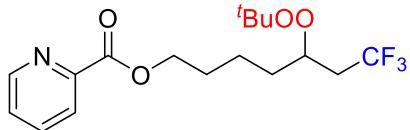


5-(tert-Butylperoxy)-7,7,7-trifluoroheptyl thiophene-2-carboxylate (4i) (67.1 mg, 91%). Isolated by flash column

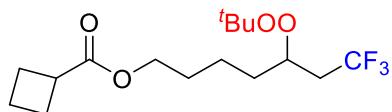
chromatography (ethyl acetate/petroleum ether = 1:20, R_f = 0.4). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.79 (d, J = 3.8 Hz, 1H), 7.55 (d, J = 5.0 Hz, 1H), 7.10 (dd, J = 5.0, 3.7 Hz, 1H), 4.31 (t, J = 6.5 Hz, 2H), 4.23-4.14 (m, 1H), 2.77-2.59 (m, 1H), 2.29-2.13 (m, 1H), 1.86-1.75 (m, 2H), 1.73-1.63 (m, 2H), 1.64-1.58 (m, 1H), 1.58-1.47 (m, 1H), 1.23 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 162.2, 134.0, 133.3, 132.2, 127.7, 126.1 (q, $J_{\text{C}-\text{F}}$ = 276.6 Hz), 80.2, 77.1 (q, $J_{\text{C}-\text{F}}$ = 3.0 Hz), 64.8, 36.9 (q, $J_{\text{C}-\text{F}}$ = 27.3 Hz), 32.4, 28.6, 26.4, 21.7; ^{19}F NMR (376 MHz, CDCl_3) δ -63.1 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{16}\text{H}_{23}\text{F}_3\text{O}_4\text{SNa}$ 391.1161; Found: 391.1142.



5-(tert-Butylperoxy)-7,7,7-trifluoroheptyl furan-2-carboxylate (4j) (53.0 mg, 75%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.4). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.60-7.56 (m, 1H), 7.18-7.16 (m, 1H), 6.52-6.50, 4.32 (t, J = 6.6 Hz), 4.22-4.15 (m, 1H), 2.76-2.60 (m, 1H), 2.27-2.15 (m, 1H), 1.86-1.75 (m, 2H), 1.74-1.66 (m, 2H), 1.63-1.47 (m, 2H), 1.23 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 158.7, 146.2, 144.7, 126.0 (q, $J_{\text{C}-\text{F}}$ = 277.0 Hz), 117.7, 111.8, 80.2, 77.1 (q, $J_{\text{C}-\text{F}}$ = 2.8 Hz), 64.6, 36.9 (q, $J_{\text{C}-\text{F}}$ = 27.7 Hz), 32.4, 28.5, 26.3, 21.7; ^{19}F NMR (376 MHz, CDCl_3) δ -63.1 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{16}\text{H}_{23}\text{F}_3\text{O}_5\text{Na}$ 375.1390; Found: 375.1380.

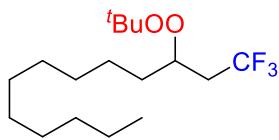


5-(tert-Butylperoxy)-7,7,7-trifluoroheptyl picolinate (4k) (10.9 mg, 15%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, R_f = 0.4). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 8.83-8.72 (m, 1H), 8.13 (d, J = 7.8 Hz, 1H), 7.85 (td, J = 7.7, 1.6 Hz, 1H), 7.53-7.41 (m, 1H), 4.44 (t, J = 6.8 Hz, 2H), 4.22-4.14 (m, 1H), 2.77-2.57 (m, 1H), 2.28-2.11 (m, 1H), 1.93-1.79 (m, 2H), 1.75-1.68 (m, 2H), 1.61-1.46 (m, 2H), 1.22 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 165.2, 149.9, 148.2, 137.0, 126.8, 126.1 (q, $J_{\text{C}-\text{F}}$ = 277.3 Hz), 125.1, 80.3, 77.1 (q, $J_{\text{C}-\text{F}}$ = 3.0 Hz), 65.7, 38.2, 36.9 (q, $J_{\text{C}-\text{F}}$ = 27.5 Hz), 32.5, 28.6, 26.4, 21.8; ^{19}F NMR (376 MHz, CDCl_3) δ -63.1 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{17}\text{H}_{24}\text{F}_3\text{NO}_4\text{Na}$ 386.1550; Found: 386.1534.

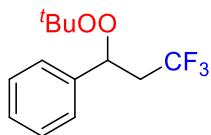


5-(tert-Butylperoxy)-7,7,7-trifluoroheptyl cyclobutanecarboxylate (4l) (59.9 mg, 88%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:20, R_f = 0.3). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 4.21-4.12 (m, 1H), 4.08 (t, J = 6.5 Hz, 2H), 3.18-3.06 (m, 1H), 2.74-2.60 (m, 1H), 2.34-2.21 (m, 3H), 2.20-2.13 (m, 2H), 2.04-1.87 (m, 2H), 1.72-1.61 (m, 4H), 1.59-1.50 (m, 1H), 1.48-1.38 (m, 1H), 1.23 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 175.5, 126.1 (q, $J_{\text{C}-\text{F}}$ = 276.7 Hz), 80.2, 77.2 (q, $J_{\text{C}-\text{F}}$ = 2.8 Hz), 64.0,

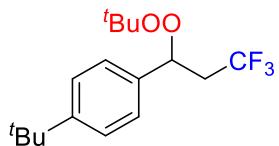
38.2, 36.9 (q, $J_{C-F} = 27.4$ Hz), 32.4, 28.6, 26.4, 25.2, 21.7, 18.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.2 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $C_{16}H_{27}F_3O_4Na$ 363.1754; Found: 363.1737.



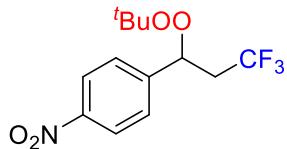
3-(tert-Butylperoxy)-1,1,1-trifluorotridecane (4m) (44.4 mg, 68%). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:10, $R_f = 0.5$). The title compound was obtained as colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ 4.20-4.12 (m, 1H), 2.73-2.57 (m, 1H), 2.27-2.10 (m, 1H), 1.68-1.57 (m, 2H), 1.27 (s, 16H), 1.23 (s, 9H), 0.88 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 126.2 (q, $J_{C-F} = 277.0$ Hz), 80.2, 77.5 (q, $J_{C-F} = 2.9$ Hz), 36.9 (q, $J_{C-F} = 27.6$ Hz), 32.9, 31.9, 29.6, 29.5, 29.4, 29.3, 26.4, 25.2, 22.7, 14.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.1 (s, 3F).



(1-(tert-Butylperoxy)-3,3,3-trifluoropropyl)benzene (4n) (38.8 mg, 74%). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:10, $R_f = 0.5$). The title compound was obtained as colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ 7.40-7.30 (m, 5H), 5.15 (dd, $J = 7.2, 5.8$ Hz, 1H), 2.90-2.75 (m, 1H), 2.55-2.39 (m, 1H), 1.22 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.1, 128.6, 128.5, 126.9, 125.5 (q, $J_{C-F} = 277.1$ Hz), 80.8, 79.8 (q, $J_{C-F} = 2.9$ Hz), 39.2 (q, 28.2 Hz), 26.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.3 (s, 3F).

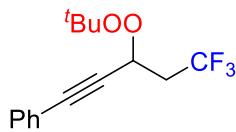


1-(tert-Butyl)-4-(1-(tert-butylperoxy)-3,3,3-trifluoropropyl)benzene (4o) (43.3 mg, 68%). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:10, $R_f = 0.6$). The title compound was obtained as colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, $J = 7.8$ Hz, 2H), 7.26 (d, $J = 7.8$ Hz, 2H), 5.14 (t, $J = 6.1$ Hz, 1H), 2.93-2.75 (m, 1H), 2.56-2.39 (m, 1H), 1.32 (s, 9H), 1.23 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.4, 135.9, 127.0, 125.6 (q, $J_{C-F} = 276.6$ Hz), 125.4, 80.8, 79.6 (q, $J_{C-F} = 3.2$ Hz), 39.1 (q, 27.8 Hz), 34.6, 31.3, 26.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.3 (s, 3F).

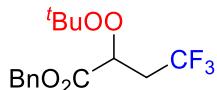


(3-(tert-Butylperoxy)-5,5,5-trifluoropent-1-yn-1-yl)benzene (4p) (28.9 mg, 47%). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:5, $R_f = 0.5$). The title compound was obtained as colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ 8.25 (d, $J = 8.4$ Hz, 2H), 7.54 (d, $J = 8.3$ Hz, 2H), 5.27 (dd, $J = 8.1, 5.1$ Hz, 1H), 2.82-2.66 (m, 1H), 2.51-2.35 (m, 1H), 1.22 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.9, 146.6, 127.5, 125.1 (q, J_{C-F}

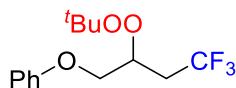
$\text{F} = 277.5$ Hz), 123.8, 81.3, 78.7 (q, $J_{\text{C}-\text{F}} = 3.1$ Hz), 39.2 (q, $J_{\text{C}-\text{F}} = 28.9$ Hz), 26.2; ^{19}F NMR (376 MHz, CDCl_3) δ -63.3 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{13}\text{H}_{16}\text{F}_3\text{NO}_4\text{Na}$ 330.0924; Found: 330.0919.



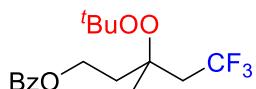
(3-(tert-Butylperoxy)-5,5,5-trifluoropent-1-yn-1-yl)benzene (4q) (14.3 mg, 25%). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:5, $R_f = 0.5$). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.48-7.42 (m, 2H), 7.36-7.28 (m, 3H), 5.07 (t, $J = 6.4$ Hz, 1H), 2.90-2.75 (m, 1H), 2.72-2.57 (m, 1H), 1.29 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 131.8, 128.8, 128.3, 125.1 (q, $J_{\text{C}-\text{F}} = 276.9$ Hz), 122.1, 86.5, 85.0, 68.2 (q, $J_{\text{C}-\text{F}} = 3.9$ Hz), 38.3 (q, $J_{\text{C}-\text{F}} = 28.5$ Hz), 29.7, 26.4; ^{19}F NMR (376 MHz, CDCl_3) δ -63.6 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $\text{C}_{15}\text{H}_{17}\text{F}_3\text{O}_2\text{Na}$ 309.1073; Found: 309.1068. 7.40 – 7.27 (m, 5H), 5.37 – 5.05 (m, 2H), 3.08 – 2.60 (m, 2H), 1.55 (s, 3H), 1.16 (d, $J = 1.4$ Hz, 9H).



Benzyl 2-(tert-butylperoxy)-4,4,4-trifluorobutanoate (4r) (21.1 mg, 33%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, $R_f = 0.5$). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.39-7.33 (m, 5H), 5.30-5.19 (m, 2H), 4.73 (dd, $J = 7.3, 5.5$ Hz, 1H), 2.67-2.54 (m, 2H), 1.22 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.2, 135.2, 128.6, 128.5, 128.3, 125.2 (q, $J_{\text{C}-\text{F}} = 277.2$ Hz), 81.6, 76.4 (q, $J_{\text{C}-\text{F}} = 3.0$ Hz), 67.3, 34.4 (q, $J_{\text{C}-\text{F}} = 30.0$ Hz), 26.1; ^{19}F NMR (376 MHz, CDCl_3) δ -63.9 (s, 3F).

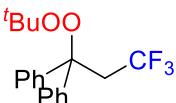


(2-(tert-Butylperoxy)-4,4,4-trifluorobutoxy)benzene (4s) (15.8 mg, 27%). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:4, $R_f = 0.4$). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.32-7.26 (m, 2H), 6.97 (t, $J = 7.5$ Hz, 1H), 6.93 (d, $J = 7.9$ Hz, 2H), 4.57-4.50 (m, 1H), 4.19 (dd, $J = 10.0, 4.1$ Hz, 1H), 4.14 (dd, $J = 10.0, 5.2$ Hz, 1H), 2.65-2.50 (m, 2H), 1.25 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 158.4, 129.5, 126.0 (q, $J_{\text{C}-\text{F}} = 276.8$ Hz), 121.2, 114.6, 81.0, 76.1 (q, $J_{\text{C}-\text{F}} = 3.0$ Hz), 66.7, 34.3 (q, $J_{\text{C}-\text{F}} = 29.5$ Hz), 26.2; ^{19}F NMR (376 MHz, CDCl_3) δ -63.5 (s, 3F).

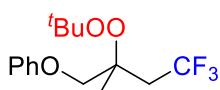


3-(tert-Butylperoxy)-5,5,5-trifluoro-3-methylpentyl benzoate (4t) (53.6 mg, 77%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:20, $R_f = 0.4$). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 8.04 (d, $J = 7.6$ Hz, 2H), 7.56 (t, $J = 7.0$ Hz, 1H), 7.44 (t, $J = 7.4$ Hz, 2H), 4.48 (t, $J = 7.0$ Hz, 2H), 2.67-2.52 (m, 2H), 2.35-2.22 (m, 1H), 2.16-2.06 (m, 1H), 1.41 (s, 3H), 1.23 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.5, 132.9, 130.3, 129.5, 128.3, 126.0 (q, $J_{\text{C}-\text{F}} = 278.2$ Hz), 79.3, 78.2 (q, $J_{\text{C}-\text{F}} = 2.2$ Hz), 60.8, 41.2

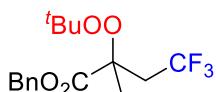
(q, $J_{C-F} = 27.0$ Hz), 36.2, 26.5, 22.2 (q, $J_{C-F} = 1.6$ Hz); ^{19}F NMR (376 MHz, CDCl₃) δ -59.9 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for C₁₇H₂₃F₃O₄Na 371.1441; Found: 371.1435.



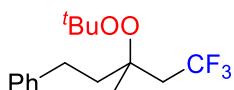
(1-(tert-Butylperoxy)-3,3,3-trifluoropropane-1,1-diyldibenzene (4u)) (26.4 mg, 39%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.3). The title compound was obtained as colorless oil. 1H NMR (400 MHz, CDCl₃) δ 7.31-7.26 (m, 8H), 7.25-7.20 (m, 2H), 3.40 (q, $J = 10.4$ Hz, 2H), 1.14 (s, 9H); ^{13}C NMR (100 MHz, CDCl₃) δ 143.1, 127.7, 127.3, 126.8, 125.7 (q, $J_{C-F} = 278.5$ Hz), 83.0 (q, $J_{C-F} = 2.0$ Hz), 79.9, 39.4 (q, $J_{C-F} = 27.0$ Hz), 26.5; ^{19}F NMR (376 MHz, CDCl₃) δ -58.9 (s, 3F).



(2-(tert-Butylperoxy)-4,4,4-trifluoro-2-methylbutoxybenzene (4v)) (20.8 mg, 34%). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:4, R_f = 0.5). The title compound was obtained as colorless oil. 1H NMR (400 MHz, CDCl₃) δ 7.28 (t, $J = 7.6$ Hz, 2H), 6.96 (t, $J = 7.4$ Hz, 1H), 6.91 (d, $J = 8.1$ Hz, 2H), 4.07 (d, $J = 9.4$ Hz, 1H), 3.97 (d, $J = 9.5$ Hz, 1H), 4.16-4.11 (m, 1H), 2.77-2.56 (m, 2H), 1.46 (s, 3H), 1.21 (s, 9H); ^{13}C NMR (100 MHz, CDCl₃) δ 158.7, 129.4, 126.0 (q, $J_{C-F} = 277.6$ Hz), 121.0, 114.7, 79.6, 79.0 (q, $J_{C-F} = 1.6$ Hz), 70.8, 37.7 (q, $J_{C-F} = 27.6$ Hz), 26.4, 19.7 (q, $J_{C-F} = 1.6$ Hz); ^{19}F NMR (376 MHz, CDCl₃) δ -59.9 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for C₁₅H₂₁F₃O₃Na 329.1335; Found: 329.1327.

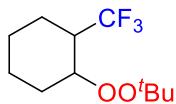


(2-(tert-Butylperoxy)-4,4,4-trifluoro-2-methylbutoxybenzene (4w)) (20.8 mg, 34%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:20, R_f = 0.5). The title compound was obtained as colorless oil. 1H NMR (400 MHz, CDCl₃) δ 7.40-7.27 (m, 5H), 5.37-5.05 (m, 2H), 3.08-2.60 (m, 2H), 1.55 (s, 3H), 1.16 (d, $J = 1.4$ Hz, 9H); ^{13}C NMR (100 MHz, CDCl₃) δ 171.2, 135.5, 128.5, 128.3, 128.2, 125.6 (q, $J_{C-F} = 277.4$ Hz), 80.6 (q, $J_{C-F} = 2.2$ Hz), 80.4, 67.1, 37.6 (q, $J_{C-F} = 28.5$ Hz), 26.3, 19.8 (q, $J_{C-F} = 1.6$ Hz); ^{19}F NMR (376 MHz, CDCl₃) δ -60.0 (s, 3F).

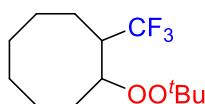


(3-(tert-Butylperoxy)-5,5,5-trifluoro-3-methylpentyl)benzene/(3-((tert-butylperoxy)methyl)-4,4,4-trifluoro-3-methylbutyl)benzene (4x)) (32.3 mg, 53%). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:5, R_f = 0.7). The title compound was obtained as colorless oil. 1H NMR (400 MHz, CDCl₃) δ 7.31-7.24 (m, 2H), 7.22-7.14 (m, 3H), 2.79-2.41 (m, 4H), 2.03 (td, $J = 13.0, 4.9$ Hz, 1H), 1.88 (td, $J = 13.2, 5.4$ Hz, 1H), 1.36 (s, 3H), 1.25 (s, 9H); ^{13}C NMR (100 MHz, CDCl₃) δ 142.4, 128.4, 128.3, 126.2 (q, $J_{C-F} = 278.2$ Hz), 125.8, 79.1, 79.0 (q, $J_{C-F} = 2.1$ Hz), 40.7 (q, $J_{C-F} = 27.0$ Hz), 29.5, 26.6, 22.0 (q, $J_{C-F} = 1.7$ Hz); ^{19}F NMR (376 MHz, CDCl₃) δ -60.0

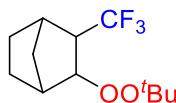
(s, 3F); HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₁₆H₂₃F₃O₂Na 327.1542; Found: 327.1538.



1-(tert-Butylperoxy)-2-(trifluoromethyl)cyclohexane (4y) (38.8 mg, 61%, d.r. = 2.1:1). Isolated by flash column chromatography (dichlorormethane/petroleum ether = 1:20, R_f = 0.4). The title compound was obtained as colorless oil. Major: ¹H NMR (400 MHz, CDCl₃) 4.01 (td, *J* = 9.3, 4.0 Hz, 1H), 2.46-2.32 (m, 1H), 2.28-2.13 (m, 1H), 2.06-1.94 (m, 1H), 1.79-1.66 (m, 2H), 1.42-1.28 (m, 4H), 1.24 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 126.9 (q, *J*_{C-F} = 280.4 Hz), 80.3, 79.5 (q, *J*_{C-F} = 1.8 Hz), 44.1 (q, *J*_{C-F} = 25.5 Hz), 30.2, 26.3, 24.2 (q, *J*_{C-F} = 3.1 Hz), 23.9, 23.0; ¹⁹F NMR (376 MHz, CDCl₃) δ -68.2 (s, 3F); HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₁₁H₁₉F₃O₂Na 263.1229; Found: 263.1226.

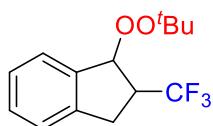


1-(tert-Butylperoxy)-2-(trifluoromethyl)cyclooctane (4z) (35.4 mg, 66%, d.r. = 1.7:1). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:10, R_f = 0.5). The title compound was obtained as colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 4.43-4.36 (m, 0.38H), 4.34-4.26 (m, 0.62H), 2.53-2.17 (m, 1H), 2.15-1.98 (m, 1H), 1.94-1.82 (m, 2H), 1.80-1.35 (m, 9H), 1.23 (s, 5.7H), 1.22 (s, 3.3H); ¹³C NMR (100 MHz, CDCl₃) δ 128.0 (q, *J*_{C-F} = 280.2 Hz), 127.9 (q, *J*_{C-F} = 280.2 Hz), 80.7 (q, *J*_{C-F} = 1.8 Hz), 80.1, 80.0, 78.0 (q, *J*_{C-F} = 2.6 Hz), 44.5 (q, 24.6 Hz), 44.3 (q, *J*_{C-F} = 24.6 Hz), 28.6, 27.8, 27.4, 27.2, 26.4, 26.3, 26.0, 25.5, 25.2, 24.3, 22.4 (q, *J*_{C-F} = 2.9 Hz), 21.0, 20.9 (q, *J*_{C-F} = 2.0 Hz); ¹⁹F NMR (376 MHz, CDCl₃) δ -68.2 (s, 1.89F), -69.2 (s, 1.11F); HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₁₃H₂₄F₃O₂ 269.1723; Found: 269.1723.

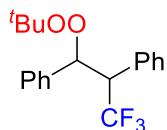


2-(tert-Butylperoxy)-3-(trifluoromethyl)bicyclo[2.2.1]heptane (4aa) (20.7 mg, 41%, d.r. = 1.5:1). **Major:** Isolated by flash column chromatography (dicholorormethane/petroleum ether = 1:10, R_f = 0.7). The title compound was obtained as colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 4.44-4.34 (m, 1H), 2.58 (s, 1H), 2.40 (d, *J* = 4.3 Hz, 1H), 1.91-1.72 (m, 2H), 1.69-1.55 (m, 2H), 1.46-1.38 (m, 1H), 1.35-1.26 (m, 2H), 1.24 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 127.1 (q, *J*_{C-F} = 277.7 Hz), 84.0 (q, *J*_{C-F} = 1.9 Hz), 80.4, 50.5 (q, *J*_{C-F} = 27.6 Hz), 39.4, 37.6 (q, *J*_{C-F} = 1.7 Hz), 35.2 (q, *J*_{C-F} = 1.1 Hz), 29.9, 26.3, 19.6; ¹⁹F NMR (376 MHz, CDCl₃) δ -69.3 (s, 3F); HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₁₂H₁₉F₃O₂Na 275.1229; Found: 275.1218. **Minor:** Isolated by flash column chromatography (dicholorormethane/petroleum ether = 1:10, R_f = 0.6). The title compound was obtained as colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 4.18 (d, *J* = 7.7 Hz, 1H), 2.66 (d, *J* = 4.9 Hz, 1H), 2.43 (s, 1H), 2.37-2.22 (m, 1H), 1.86 (d, *J* = 10.6 Hz, 1H), 1.67-1.59 (m, 1H), 1.57-1.47 (m, 1H), 1.24 (s, 9H), 1.19-1.03 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 126.0 (q, *J*_{C-F} = 279.0 Hz), 85.3 (q, *J*_{C-F} = 1.4 Hz), 80.5, 50.8 (q, *J*_{C-F} = 26.4 Hz), 39.8, 37.0 (q, *J*_{C-F} = 2.0 Hz), 33.7 (q, *J*_{C-F} = 1.3 Hz), 29.6, 26.3, 23.0; ¹⁹F NMR (376 MHz, CDCl₃) δ -62.1 (s, 3F); HRMS (ESI) *m/z*: [M+Na]⁺ Calcd

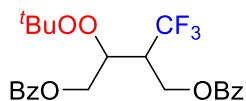
for $C_{12}H_{19}F_3O_2Na$ 275.1229; Found: 275.1218.



1-(tert-Butylperoxy)-2-(trifluoromethyl)-2,3-dihydro-1H-indene (4ab) (10.4 mg, 19%, d.r. = 12.9:1). Isolated by flash column chromatography (dichloromethane/petroleum ether, R_f = 0.4). The title compound was obtained as colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, J = 7.5 Hz, 1H), 7.35-7.29 (m, 1H), 7.28-7.22 (m, 2H), 5.62 (d, J = 2.4 Hz, 0.93H), 5.46 (d, J = 6.0 Hz, 0.07H), 3.40-3.25 (m, 2H), 3.11-3.00 (m, 1H), 1.27 (s, 8.35H), 1.20 (s, 0.65H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.7, 138.5, 129.6, 127.4 (q, J_{C-F} = 278.4 Hz), 127.1, 126.3, 124.7, 86.9 (q, J_{C-F} = 2.8 Hz), 80.7, 47.0 (q, J_{C-F} = 26.9 Hz), 31.0 (q, J_{C-F} = 2.8 Hz), 26.4, 26.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ -65.4 (s, 0.22F), -70.6 (s, 2.78F).

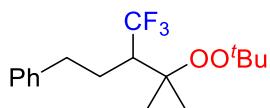


(1-(tert-Butylperoxy)-3,3,3-trifluoropropane-1,2-diyl)dibenzene (4ac) (30.5 mg, 45%, d.r. = 1.4:1). **Major:** Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:5, R_f = 0.4). The title compound was obtained as colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ 7.30-7.18 (m, 8H), 7.13-7.08 (m, 2H), 5.52 (d, J = 4.6 Hz, 1H), 3.51 (qd, J = 9.8, 4.7 Hz, 1H), 1.16 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.7, 130.7 (q, J_{C-F} = 1.3 Hz), 130.6, 128.2, 128.0, 127.9, 127.8, 126.8, 125.7 (q, J_{C-F} = 280.0 Hz), 82.4, (q, J_{C-F} = 2.3 Hz), 80.7, 50.4 (q, J_{C-F} = 23.4 Hz), 26.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ -65.2 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $C_{19}H_{21}F_3O_2Na$ 361.1386; Found: 361.1375. **Minor:** Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:5, R_f = 0.4). The title compound was obtained as colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ 7.20-7.09 (m, 6H), 7.04-6.97 (m, 4H), 5.36 (d, J = 8.8 Hz, 1H), 3.82-3.70 (m, 1H), 1.23 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.8, 132.0 (q, J_{C-F} = 1.9 Hz), 139.6, 128.2, 128.0, 127.8, 127.7, 127.6, 126.1 (q, J_{C-F} = 280.0 Hz), 84.5, (q, J_{C-F} = 1.9 Hz), 80.8, 54.3 (q, J_{C-F} = 26.3 Hz), 26.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.4 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $C_{19}H_{21}F_3O_2Na$ 361.1386; Found: 361.1375.

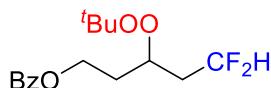


2-(tert-Butylperoxy)-3-(trifluoromethyl)butane-1,4-diyl dibenzoate (4ad) (41.8 mg, 46%, d.r. = 1:1). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.4). The title compound was obtained as colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, J = 9.1 Hz, 4H), 7.60-7.54 (m, 2H), 7.47-7.41 (m, 4H), 4.78-4.68 (m, 4H), 4.67-4.63 (m, 0.50H), 4.55-4.48 (m, 0.50H), 3.40-3.29 (m, 0.50H), 3.21-3.08 (m, 0.50H), 1.25 (s, 4.50H), 1.22 (s, 4.50H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.1, 166.0, 165.9, 165.8, 133.3-133.2, 129.7-129.6, 129.5, 129.4, 128.5, 129.4, 126.0 (q, J_{C-F} = 280.1 Hz), 125.8 (q, J_{C-F} = 281.0 Hz), 81.3, 81.0, 77.9 (q, J_{C-F} = 2.2 Hz), 76.5 (q, J_{C-F} = 2.2 Hz), 62.6 (q, J_{C-F} = 1.3 Hz), 62.3 (q, J_{C-F} = 1.5 Hz), 58.9 (q, J_{C-F} = 2.9 Hz), 58.8 (q, J_{C-F} = 2.8 Hz), 43.9

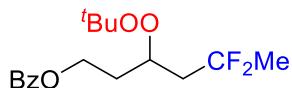
(q, $J_{C-F} = 26.3$ Hz), 43.5 (q, $J_{C-F} = 25.5$ Hz), 26.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ -65.1 (s, 1.5F), -65.8 (s, 1.5F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $C_{23}H_{25}F_3O_6Na$ 477.1495; Found: 477.1478.



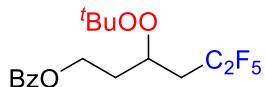
(3-(tert-Butylperoxy)-5,5,5-trifluoro-4,4-dimethylpentyl)benzene (4ae) (15.9 mg, 25%). Isolated by flash column chromatography (petroleum ether, $R_f = 0.5$). The title compound was obtained as colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ 7.31-7.26 (m, 2H), 7.23-7.18 (m, 3H), 2.90-2.81 (m, 1H), 2.78-2.68 (m, 2H), 2.15-2.05 (m, 1H), 1.82-1.70 (m, 1H), 1.42-1.38 (m, 3H), 1.22 (s, 9H), 1.30-1.11 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.1, 128.5, 128.4, 128.2 (q, $J_{C-F} = 281.6$ Hz), 125.9, 80.0 (q, $J_{C-F} = 1.6$ Hz), 78.8, 48.5 (q, $J_{C-F} = 23.3$ Hz), 35.7 (q, $J_{C-F} = 1.5$ Hz), 27.0 (q, $J_{C-F} = 2.3$ Hz), 26.6, 25.4 (q, $J_{C-F} = 2.9$ Hz), 21.1 (q, $J_{C-F} = 1.7$ Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.2 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $C_{17}H_{25}F_3O_2Na$ 341.1699; Found: 341.1693.



3-(tert-Butylperoxy)-5,5-difluorohexyl benzoate (4af) (34.8 mg, 55%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, $R_f = 0.5$). The title compound was obtained as colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ 8.06-8.01 (m, 2H), 7.59-7.53 (m, 1H), 7.48-7.41 (m, 2H), 6.10 (tdd, $J = 56.9, 6.4, 3.3$ Hz, 1H), 4.48-4.42 (m, 2H), 4.39-4.31 (m, 1H), 2.37-2.19 (m, 1H), 2.18-1.99 (m, 3H), 1.23 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 133.0, 130.1, 129.5, 128.4, 115.8 (t, $J_{C-F} = 238.1$ Hz), 80.4, 75.2 (dd, $J_{C-F} = 8.6, 4.4$ Hz), 61.4, 38.5 (t, $J_{C-F} = 21.2$ Hz), 32.3, 26.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -114.1 (d, $J = 285.9, 1F$), -117.4 (d, $J = 285.0, 1F$); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $C_{16}H_{22}F_2O_4Na$ 339.1378; Found: 339.1370.

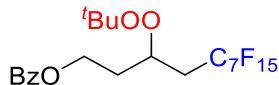


3-(tert-Butylperoxy)-5,5-difluorohexyl benzoate (4ag) (40.3 mg, 61%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, $R_f = 0.5$). The title compound was obtained as colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ 8.07-8.00 (m, 2H), 7.59-7.53 (m, 1H), 7.48-7.41 (m, 2H), 4.47 (t, $J = 6.5$ Hz, 2H), 4.42-4.35 (m, 1H), 2.50-2.34 (m, 1H), 2.22-2.03 (m, 3H), 1.68 (t, $J = 18.9$ Hz, 3H), 1.23 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 132.9, 130.2, 129.5, 128.4, 123.3 (t, $J_{C-F} = 240.1$ Hz), 80.1, 75.6 (dd, $J_{C-F} = 5.5, 3.8$ Hz), 61.7, 40.8 (t, $J_{C-F} = 24.7$ Hz), 32.7, 26.5, 24.1 (t, $J_{C-F} = 27.6$ Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -85.6 (d, $J = 242.8, 1F$), -89.9 (d, $J = 242.7, 1F$); HRMS (ESI) m/z : [M+Na]⁺ Calcd for $C_{17}H_{24}F_2O_4Na$ 353.1535; Found: 353.1527.

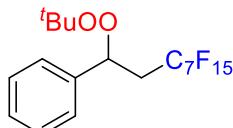


3-(tert-Butylperoxy)-5,5,6,6,6-pentafluorohexyl benzoate (4ah) (53.0 mg, 72%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:20, $R_f = 0.4$). The title compound was obtained as colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ 8.05-8.00 (m, 2H), 7.59-7.54 (m, 1H), 7.47-7.41 (m, 2H), 4.57-4.50 (m, 1H), 4.50-

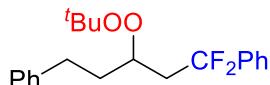
4.44 (m, 2H), 2.82-2.65 (m, 1H), 2.32-2.21 (m, 1H), 2.21-2.11 (m, 2H), 1.23 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 166.4, 133.0, 130.1, 129.5, 128.4, 120.1-111.3 (m, C₂F₅), 80.5, 73.7 (t, J_{C-F} = 2.3 Hz), 61.2, 33.7 (t, J_{C-F} = 21.3 Hz), 32.6, 26.3; ¹⁹F NMR (376 MHz, CDCl₃) δ -85.7 (s, 3F), -115.4 to -117.1 (m, 2F); HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₁₇H₂₁F₅O₄Na 407.1252; Found: 407.1241.



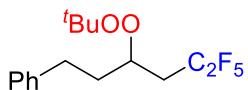
3-(*tert*-Butylperoxy)-5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-pentadecafluoroundecyl benzoate (4ai) (91.4 mg, 72%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:20, R_f = 0.5). The title compound was obtained as colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 8.05-8.01 (m, 2H), 7.59-7.54 (m, 1H), 7.47-7.41 (m, 2H), 4.60-4.52 (m, 1H), 4.52-4.45 (m, 2H), 2.87-2.68 (m, 1H), 2.36-2.09 (m, 3H), 1.23 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 166.4, 133.0, 130.1, 129.5, 128.4, 121.6-104.3 (m, C₇F₁₅), 80.5, 73.8 (t, J_{C-F} = 2.6 Hz), 61.2, 34.0 (t, J_{C-F} = 21.1 Hz), 32.7, 26.3; ¹⁹F NMR (376 MHz, CDCl₃) δ -80.8 (t, J = 9.8 Hz, 3F), -111.4 to -113.5 (m, 2F), -121.4 to -123.7 (m, 8F), -126.0 to -126.2 (m, 2F); HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₂H₂₁F₁₅O₄Na 657.1093; Found: 657.1079.



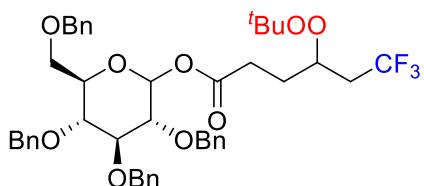
(1-(*tert*-Butylperoxy)-3,3,4,4,5,5,6,6,7,7,8,8,9,9,9-pentadecafluorononyl)benzene (4aj) (48.4 mg, 43%). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:10, R_f = 0.5). The title compound was obtained as colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.41-7.31 (m, 5H), 5.27 (dd, J = 6.9, 5.5 Hz, 1H), 2.89-2.71 (m, 1H), 2.54-2.36 (m, 1H), 1.22 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 139.4, 128.6, 128.5, 126.9, 121.6-104.3 (m, C₇F₁₅), 80.8, 78.9, 36.0 (t, J_{C-F} = 21.1 Hz), 26.3; ¹⁹F NMR (376 MHz, CDCl₃) δ -80.8 (t, J = 10.1 Hz, 3F), -111.2 to -113.1 (m, 2F), -121.4 to -123.8 (m, 8F), -126.1 to -126.3 (m, 2F); HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₁₉H₁₇F₁₅O₂Na 585.0881; Found: 585.0878.



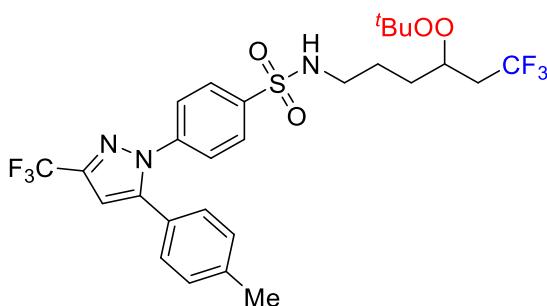
(3-(*tert*-Butylperoxy)-1,1-difluoropentane-1,5-diyl)dibenzene (4ak) (20.2 mg, 29%). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:5, R_f = 0.5). The title compound was obtained as colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.48-7.41 (m, 2H), 7.42-7.38 (m, 3H), 7.30-7.24 (m, 2H), 7.20-7.14 (m, 3H), 4.20-4.12 (m, 1H), 2.85-2.60 (m, 3H), 2.35-2.19 (m, 1H), 2.00-1.90 (m, 2H), 1.17 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 141.9, 137.3 (t, J_{C-F} = 26.3 Hz), 129.7 (t, J_{C-F} = 1.8 Hz), 128.4, 128.3 (t, J_{C-F} = 2.6 Hz), 125.8, 125.0 (t, J_{C-F} = 6.2 Hz), 122.1 (t, J_{C-F} = 243.1 Hz), 80.4, 75.2 (dd, J_{C-F} = 8.6, 4.4 Hz), 61.4, 38.5 (t, J_{C-F} = 21.2 Hz), 32.3, 26.4; ¹⁹F NMR (376 MHz, CDCl₃) δ -89.9 (d, J = 247.4, 1F), -96.0 (d, J = 247.4, 1F); HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₁H₂₆F₂O₂Na 371.1793; Found: 371.1791.



(3-(*tert*-Butylperoxy)-5,5,6,6,6-pentafluorohexyl)benzene (4al) (53.8 mg, 79%). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:10, R_f = 0.5). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.32-7.26 (m, 2H), 7.22-7.16 (m, 3H), 4.36-4.28 (m, 1H), 2.87-2.77 (m, 2H), 2.75-2.57 (m, 2H), 2.21-1.91 (m, 3H), 1.25 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 141.4, 128.4, 128.3, 126.0, 120.8-112.1 (m, C_2F_5), 80.2, 76.0 (t, $J_{\text{C}-\text{F}} = 2.2$ Hz), 35.2, 33.6 (t, $J_{\text{C}-\text{F}} = 21.0$ Hz), 31.5, 26.4; ^{19}F NMR (376 MHz, CDCl_3) δ -85.9 (s, 3F), -115.3 to -117.6 (m, 2F); HRMS (ESI) m/z : [M+Na] $^+$ Calcd for $\text{C}_{16}\text{H}_{21}\text{F}_5\text{O}_2\text{Na}$ 363.1354; Found: 363.1345.

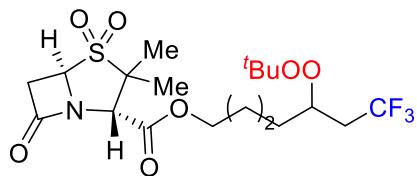


2,3,4,6-Tetra-*O*-benzyl-*D*-glucopyranosyl 4-(*tert*-butylperoxy)-6,6,6-trifluorohexanoate (6a) (110.9 mg, 71%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.5). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.38-7.22 (m, 18H), 7.18-7.07 (m, 2H), 6.40 (s, 0.5H), 5.67-5.60 (m, 0.5H), 5.00-4.43 (m, 8H), 4.25-4.19 (m, 1H), 3.78-3.50 (m, 6H), 2.72-1.89 (m, 6H), 1.26-1.20 (m, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 171.6-171.2 (m), 138.7-137.7 (m), 128.6-127.5 (m), 125.9 (q, $J_{\text{C}-\text{F}} = 277.4$ Hz), 94.1, 94.0, 90.1, 90.0, 84.8, 84.6, 81.6, 81.1, 81.0, 80.3, 75.9, 75.6, 75.2, 74.9, 73.5-73.0 (m), 68.1, 61.4, 36.9 (q, $J_{\text{C}-\text{F}} = 28.3$ Hz), 36.8 (q, $J_{\text{C}-\text{F}} = 27.8$ Hz), 33.8-33.1 (m), 30.0 29.9, 29.8, 27.9, 27.6, 26.4, 26.3, 2.3-21.2 (m); ^{19}F NMR (376 MHz, CDCl_3) δ -62.9 to -63.2 (m), -66.1 to -66.4 (m); HRMS (ESI) m/z : [M+Na] $^+$ Calcd for $\text{C}_{44}\text{H}_{51}\text{F}_3\text{O}_9\text{Na}$ 803.3377; Found: 803.3369.

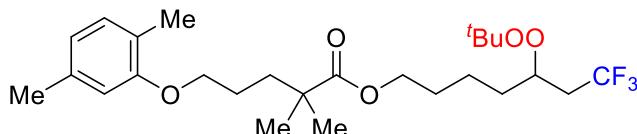


N-(4-(*tert*-Butylperoxy)-6,6,6-trifluorohexyl)-4-(5-(*p*-tolyl)-3-(trifluoromethyl)-1*H*-pyrazol-1-yl)benzenesulfonamide (6b) (96.0 mg, 79%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:3, R_f = 0.5). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.84 (d, J = 8.2 Hz, 2H), 7.47 (d, J = 8.2 Hz, 2H), 7.17 (d, J = 7.7 Hz, 2H), 7.11 (d, J = 7.6 Hz, 2H), 6.74 (s, 1H), 4.89 (t, J = 6.2 Hz, 1H), 4.21-4.07 (m, 1H), 3.06-2.91 (m, 2H), 2.71-2.51 (m, 1H), 2.38 (s, 3H), 2.25-2.06 (m, 1H), 1.75-1.65 (m, 2H), 1.63-1.53 (m, 2H), 1.21 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 145.2, 144.1 (q, $J_{\text{C}-\text{F}} = 38.8$ Hz), 142.5, 139.8, 139.6, 129.7, 128.7, 128.0, 125.9 (q, $J_{\text{C}-\text{F}} = 276.9$ Hz), 125.7, 125.5, 121.0 (q, $J_{\text{C}-\text{F}} = 269.0$ Hz), 106.2, 80.6, 76.8 (q,

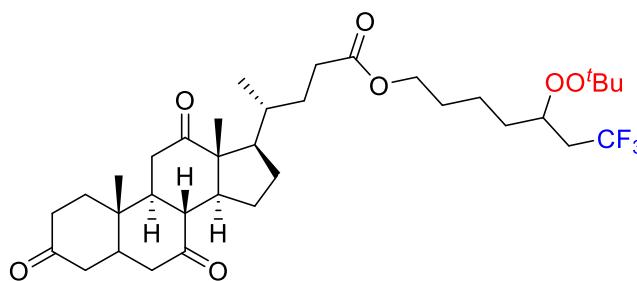
$J_{C-F} = 2.9$ Hz), 43.0, 36.8 (q, $J_{C-F} = 27.7$ Hz), 29.5, 26.3, 25.2, 21.2; ^{19}F NMR (376 MHz, CDCl₃) δ -62.5 (s, 3F), -63.2 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for C₂₇H₃₁F₆N₃O₄SnA 630.1832; Found: 630.1812.



5-(tert-Butylperoxy)-7,7,7-trifluoroheptyl (2S,5R)-3,3-dimethyl-7-oxo-4-thia-1-azabicyclo[3.2.0]heptane-2-carboxylate 4,4-dioxide (6c) (67.2 mg, 71%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:3, R_f = 0.3). The title compound was obtained as colorless oil. 1H NMR (400 MHz, CDCl₃) δ 4.61 (dd, J = 4.1, 2.3 Hz, 1H), 4.38 (s, 1H), 4.22 (t, J = 6.7 Hz, 2H), 4.20-4.13 (m, 1H), 3.55-3.38 (m, 2H), 2.78-2.60 (m, 1H), 2.28-2.11 (m, 1H), 1.78-1.65 (m, 4H), 1.61 (s, 3H), 1.59-1.44 (m, 2H), 1.42 (s, 3H), 1.23 (s, 9H); ^{13}C NMR (100 MHz, CDCl₃) δ 170.7, 167.0, 126.0 (q, J_{C-F} = 277.0 Hz), 80.3, 77.0 (q, J_{C-F} = 1.6 Hz), 66.2, 63.3, 62.6, 61.1, 38.3, 36.9 (q, J_{C-F} = 27.4 Hz), 32.3, 28.3, 26.4, 21.6, 21.6, 20.3, 18.6; ^{19}F NMR (376 MHz, CDCl₃) δ -63.1 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for C₁₉H₃₀F₃NO₇SnA 496.1587; Found: 496.1582.

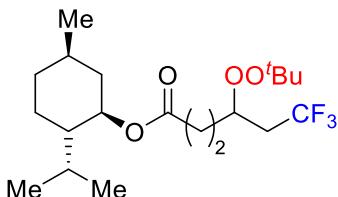


5-(tert-Butylperoxy)-7,7,7-trifluoroheptyl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (6d) (63.8 mg, 65%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:20, R_f = 0.3). The title compound was obtained as colorless oil. 1H NMR (400 MHz, CDCl₃) δ 6.99 (d, J = 7.4 Hz, 1H), 6.65 (d, J = 7.5 Hz, 1H), 6.60 (s, 1H), 4.20-4.11 (m, 1H), 4.10-4.03 (m, 2H), 3.94-3.88 (m, 2H), 2.73-2.57 (m, 1H), 2.30 (s, 3H), 2.17 (s, 3H), 1.74-1.70 (m, 4H), 1.70-1.61 (m, 4H), 1.46-1.36 (m, 2H), 1.22 (s, 9H), 1.21 (s, 6H); ^{13}C NMR (100 MHz, CDCl₃) δ 177.8, 157.0, 136.4, 130.3, 126.1 (q, J_{C-F} = 276.5 Hz), 123.6, 120.7, 112.0, 80.2, 77.2 (q, J_{C-F} = 2.7 Hz), 68.0, 64.1, 42.1, 37.1, 37.0 (q, J_{C-F} = 27.6 Hz), 32.5, 28.6, 26.4, 25.2, 25.1, 21.8, 21.4, 15.7; ^{19}F NMR (376 MHz, CDCl₃) δ -63.1 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for C₂₆H₄₁F₃O₅Na 513.2798; Found: 513.2775.

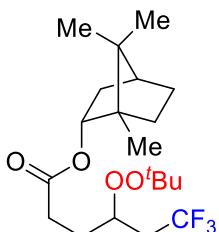


5-(tert-Butylperoxy)-7,7,7-trifluoroheptyl (5β)-3,7,12-trioxocholan-24-oate (6e) (65.6 mg, 51%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:1, R_f = 0.6). The title compound was obtained as white solid. 1H NMR (400 MHz, CDCl₃) δ 4.21-4.12 (m, 1H), 4.07 (t, J = 6.7 Hz, 2H), 2.97-2.80 (m, 3H), 2.75-2.58 (m, 1H), 2.46-2.05 (m, 12H), 2.06-1.91 (m, 4H), 1.90-1.78 (m, 2H), 1.72-1.59 (m, 5H), 1.57-1.44 (m, 2H), 1.40 (s, 3H), 1.35-1.25 (m, 3H), 1.23 (s, 9H), 1.07 (s, 3H), 0.85 (d, J = 6.6 Hz, 3H); ^{13}C NMR (100 MHz, CDCl₃) δ 211.8, 208.9,

208.5, 174.0, 126.1 (q, $J_{C-F} = 276.8$ Hz), 80.2, 77.1 (q, $J_{C-F} = 3.0$ Hz), 64.0, 56.9, 51.8, 49.0, 46.8, 45.7, 45.5, 44.9, 42.8, 38.6, 36.9 (q, $J_{C-F} = 27.5$ Hz), 36.4, 36.0, 35.5, 35.3, 32.4, 31.5, 30.5, 28.5, 27.6, 26.4, 25.1, 21.9, 21.7, 18.6, 11.8; ^{19}F NMR (376 MHz, CDCl₃) δ -63.1 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for C₃₅H₅₃F₃O₇Na 665.3636; Found: 665.3610.



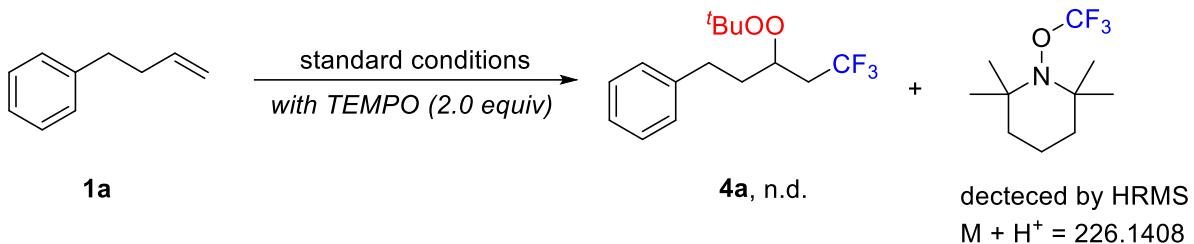
(1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl 4-(tert-butyperoxy)-6,6,6-trifluorohexanoate (6f) (60.3 mg, 76%, d.r. = 1:1). Isolated by flash column chromatography (dichloromethane/petroleum ether = 1:2, R_f = 0.5). The title compound was obtained as colorless oil. 1H NMR (400 MHz, CDCl₃) δ 4.70 (tdd, $J = 10.8, 4.4, 1.5$ Hz, 1H), 4.27-4.18 (m, 1H), 2.78-2.60 (m, 1H), 2.46 (t, $J = 7.6$ Hz, 2H), 2.28-2.13 (m, 1H), 2.08-1.95 (m, 2H), 1.94-1.79 (m, 2H), 1.72-1.63 (m, 2H), 1.55-1.43 (m, 1H), 1.37 (tt, $J = 11.9, 3.0$ Hz), 1.23 (s, 9H), 1.12-1.00 (m, 1H), 0.96 (q, $J = 11.5$ Hz, 1H), 0.91 (d, $J = 3.0$ Hz, 3H), 0.89 (d, $J = 3.5$ Hz, 3H), 0.87-0.80 (m, 1H), 0.76 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl₃) δ 172.5, 125.9 (q, $J_{C-F} = 276.2$ Hz), 76.3 (q, $J_{C-F} = 3.0$ Hz), 74.4, 74.3, 47.0, 40.9, 40.8, 37.0 (q, $J_{C-F} = 27.6$ Hz), 36.9 (q, $J_{C-F} = 27.6$ Hz), 34.2, 30.3, 30.2, 28.2, 26.4, 26.3, 23.4, 22.0, 20.7, 16.3, 16.2; ^{19}F NMR (376 MHz, CDCl₃) δ -63.2 (s, 1.5F), -63.3 (s, 1.5F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for C₂₀H₃₅F₃O₄Na 419.2380; Found: 419.2362.



(1*S*,2*R*,4*S*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl 4-(tert-butyperoxy)-6,6,6-trifluorohexanoate (6g) (53.6 mg, 68%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:20, R_f = 0.5). The title compound was obtained as colorless oil. 1H NMR (400 MHz, CDCl₃) δ 4.91 (d, $J = 9.8$ Hz, 1H), 4.30-4.18 (m, 1H), 2.77-2.61 (m, 1H), 2.49 (t, $J = 7.5$ Hz, 2H), 2.41-2.31 (m, 1H), 2.28-2.14 (m, 1H), 2.10-1.99 (m, 1H), 1.98-1.87 (m, 2H), 1.77-1.67 (m, 2H), 1.31-1.26 (m, 2H), 1.23 (s, 9H), 1.00-0.94 (m, 1H), 0.91 (s, 3H), 0.88 (s, 3H), 0.83 (s, 3H); ^{13}C NMR (100 MHz, CDCl₃) δ 173.1, 126.0 (q, $J_{C-F} = 276.8$ Hz), 80.3, 80.1, 80.0, 76.3 (q, $J_{C-F} = 3.0$ Hz), 48.8, 47.8, 44.9, 37.0 (q, $J_{C-F} = 28.1$ Hz), 36.8, 36.7, 30.3, 28.2, 28.0, 27.1, 26.4, 19.7, 18.8, 13.4; ^{19}F NMR (376 MHz, CDCl₃) δ -63.3 (s, 3F); HRMS (ESI) m/z : [M+Na]⁺ Calcd for C₂₀H₃₃F₃O₄Na 417.2223; Found: 417.2203.

5. Mechanistic experiments

5.1 Radical inhibition experiment



The operation followed the general procedure for the synthesis of product , when 2,2,6,6- tetramethyl piperidin-1-oxyl (TEMPO) (0.4 mmol) was added. ^1H NMR analysis revealed that **4a** was not detected. Besides, the adduct was also detected by HRMS (Figure S2)

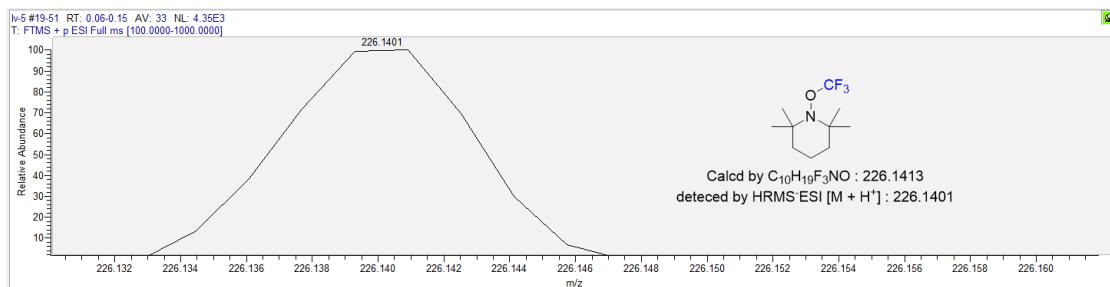
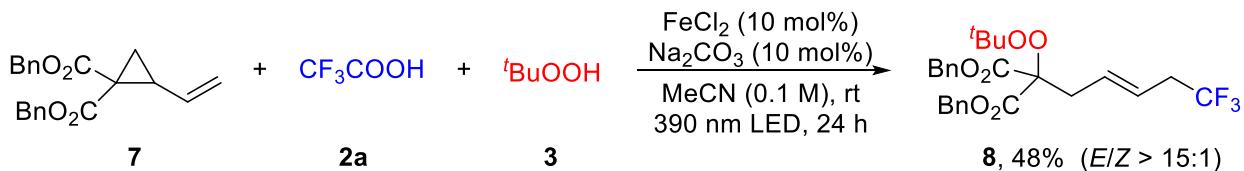
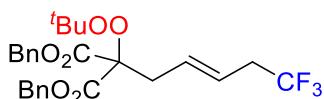


Figure S2 HRMS data for radical inhibition experiment

5.2 Radical clock experiment



To a mixture of alkene **7** (0.2 mmol), acid **2a** (0.6 mmol), Na₂CO₃ (0.02 mmol) and FeCl₂ (2.5 mg, 0.02 mmol), MeCN (2.0 mL) was added under nitrogen at room temperature. Then tert-butyl hydroperoxide (TBHP, 0.8 mmol, 5-6 M in decane) was added into the mixture under nitrogen at room temperature. The resulting mixture was stirred under rt, 390 nm, 10 W for 24 h. The resulting reaction solution was directly filtered through a pad of silica by chloroform. The solvent was evaporated in vacuo to give the crude products. NMR yields are determined by ¹H NMR using mesitylene as an internal standard. Solvent was evaporated and the residues were purified by flash column chromatography on silica gel with an eluent to afford the pure products



Dibenzyl 2-(*tert*-butylperoxy)-2-(5,5,5-trifluoropent-2-en-1-yl)malonate (8) (47.4 mg, 48%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5). The title compound was obtained as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.34-7.26 (m, 10H), 5.65-5.55 (m, 1H), 5.41-5.31 (m, 1H), 5.20-5.11 (m, 4H), 2.96 (d, J = 7.4 Hz, 2H), 2.70-2.57 (m, 2H), 1.18 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.4, 135.1, 130.2, 128.4, 128.3, 125.7 (q, $J_{\text{C-F}}$ = 276.0 Hz), 122.5 (q, $J_{\text{C-F}}$ = 3.4 Hz), 87.0, 81.0, 67.3, 37.3 (q, $J_{\text{C-F}}$ = 29.6 Hz), 35.4, 26.3,

¹⁹F NMR (376 MHz, CDCl₃) δ -66.5 (s, 3F); HRMS (ESI) *m/z*: [M+Na]⁺ Calcd for C₂₆H₂₉F₃O₆Na 517.1808; Found: 517.1790.

5.3 Light on/off experiment

Light on/off experiment was performed with the model reaction with a Kessil® PR160L-390 nm lamp (10 W). The reaction was placed in light and dark in every alternative 2 h. The yield of the product **4a** was determined by ¹H NMR using mesitylene as internal standard. The results show that the reaction proceeds when the light is on, while it is stopped when the light is off.

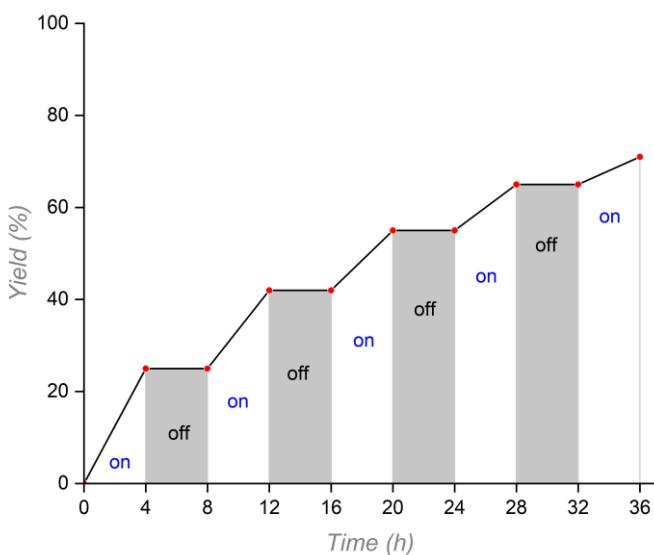


Figure S3 Light on/off experiment

5.4 Quantum yield measurement

5.4.1 Determination of the photon flux

The photon flux of the spectrophotometer was determined by standard ferrioxalate actinometry.⁹ Following a literature procedure. For this purpose, the following two solutions were prepared:

Solution A:

Potassium ferrioxalate hydrate (737 mg, 1.50 mmol) was dissolved in aq. H₂SO₄ (0.05 M, 10 mL) to afford a 0.15 M ferrioxalate solution.

Solution B:

1,10-Phenanthroline monohydrate (20 mg, 0.10 mmol), NaOAc (2.71 g, 41.3 mmol) were dissolved in aq. H₂SO₄ (0.5 M, 20 mL).

Notice: Solution A was prepared in the dark and both solutions were stored in the dark to avoid external irradiation prior to the actinometry. The following procedure was performed in a darkened lab.

First, the photon flux of the 390 nm LED was determined. For this, solution A (1.0 mL) was filled in a 10 mL

Schlenk tube and irradiated for 60 s, at $\lambda_{\max} = 390$ nm. After irradiation, solution B (175 μL) was added to the Schlenk tube and the mixture was stirred in the dark for 1 h to ensure coordination of Fe(II)- ions by phenantroline. The solution was poured into a quartz cuvette and the absorption of the solution was measured at 510 nm. Sample preparation and measurement were repeated two more times. In a similar way a non-irradiated control sample was prepared, measured for absorbance at 510 nm, which was repeated twice. The average of the absorption of the both irradiated and radiated samples were calculated and were used to calculate the conversion factor n applying eq. 1.

$$n(\text{Fe}^{2+}) = \frac{V \cdot \Delta A(510 \text{ nm})}{l \cdot \varepsilon} \quad (1)$$

V refers to the total volume (0.001175 L) of the solution (after addition of solution B), ΔA is the average difference in absorption of irradiated and non-irradiated samples between at 510 nm ($\Delta A = 1.86$), l is the path length (1.0 cm) of the cuvette, and ε is the molar extinction coefficient of the ferrioxalate actinometer at 510 nm (11100 L mol $^{-1}$ cm $^{-1}$).¹⁰ The photon flux (Φ_q) is calculated using eq. 2.

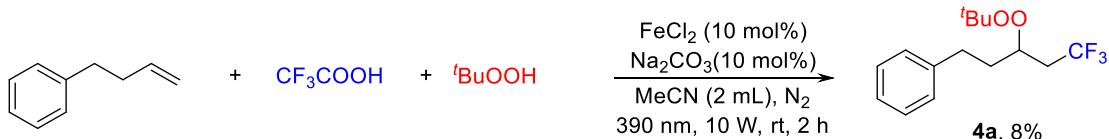
$$\Phi_q = \frac{n(\text{Fe}^{2+})}{\Phi_F \cdot t \cdot f} \quad (2)$$

Φ_F refers to the quantum yield for the ferrioxalate actinometer (1.13 at $\lambda_{\max} = 390$ nm),¹¹ t is the irradiation time for solution A (60 s), and f is the fraction of light absorbed at $\lambda_{\max} = 390$ nm by the ferrioxalate actinometer. This value is calculated using eq. 3, where A (390 nm) is the absorption of the ferrioxalate solution at 390 nm. A measured absorbance value of >8 at 390 nm indicates the fraction of absorbed light (f) to be >0.999 .

$$f = 1 - 10^{-A(390 \text{ nm})} \quad (3)$$

Thus, the average photon flux was calculated to be 2.90×10^{-9} einsteins s $^{-1}$

5.4.2 Determination of the quantum yield of trifluoromethylation-peroxidation



First, a stock solution was prepared: To two oven-dried 25 mL Schlenk tube were added 4-Phenyl-1-butene (30.0 μL , 0.2 mmol, 1.0 equiv.), trifluoroacetic acid (44.8 μL , 0.6 mmol, 3.0 equiv.), *tert*-butyl hydroperoxide (145 μL , 0.8 mmol, 4.0 equiv.), FeCl_2 (2.5 mg, 0.02 mmol, 10mol%), Na_2CO_3 (2.1 mg, 0.02 mmol, 10mol%) and MeCN (2 mL) under nitrogen. Upon homogenization, two 25 mL Schlenk tubes each equipped with Teflon coated stir bars under nitrogen. Both reaction tubes were carefully degassed by two freeze/pump/thaw cycles and irradiated with the single blue LED (10 W, $\lambda_{\max} = 390$ nm) for 2 h. Followed by an addition of 1 mL 0.1 M mesitylene internal standard in CDCl_3 and yield determination by ^1H NMR. Thus, the average yield was calculated to be 8%.

The quantum yield (Φ) of the reaction was determined using eq. 4, where the photon flux (Φ_q) is 2.90×10^{-9} einsteins s $^{-1}$ (see above), t is the reaction time (2 h = 7200 s) and f_R is the fraction of light absorbed by the reaction mixture (indicated in eq. 3; $A_{\text{Rct}}(390 \text{ nm}) = 4.3$, thus $f_R > (0.999)$) determined using eq. 4.

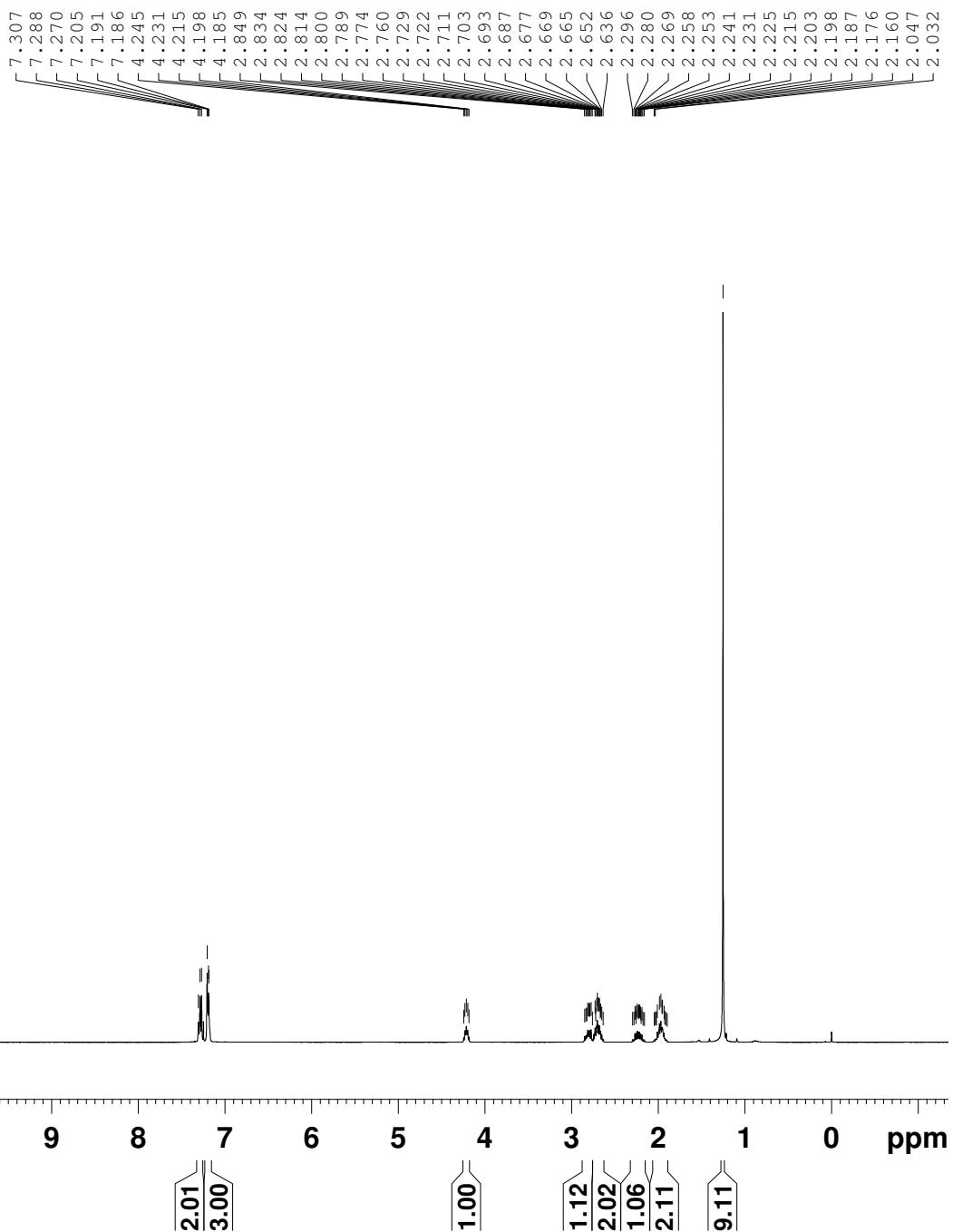
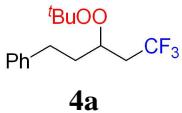
$$\Phi = \frac{n(\text{product})}{\Phi_q \cdot t \cdot f_R} \quad (4)$$

Thus, the quantum yield (Φ) of the trifluoromethylation-peroxidation was determined to be: $\Phi = 0.79$.

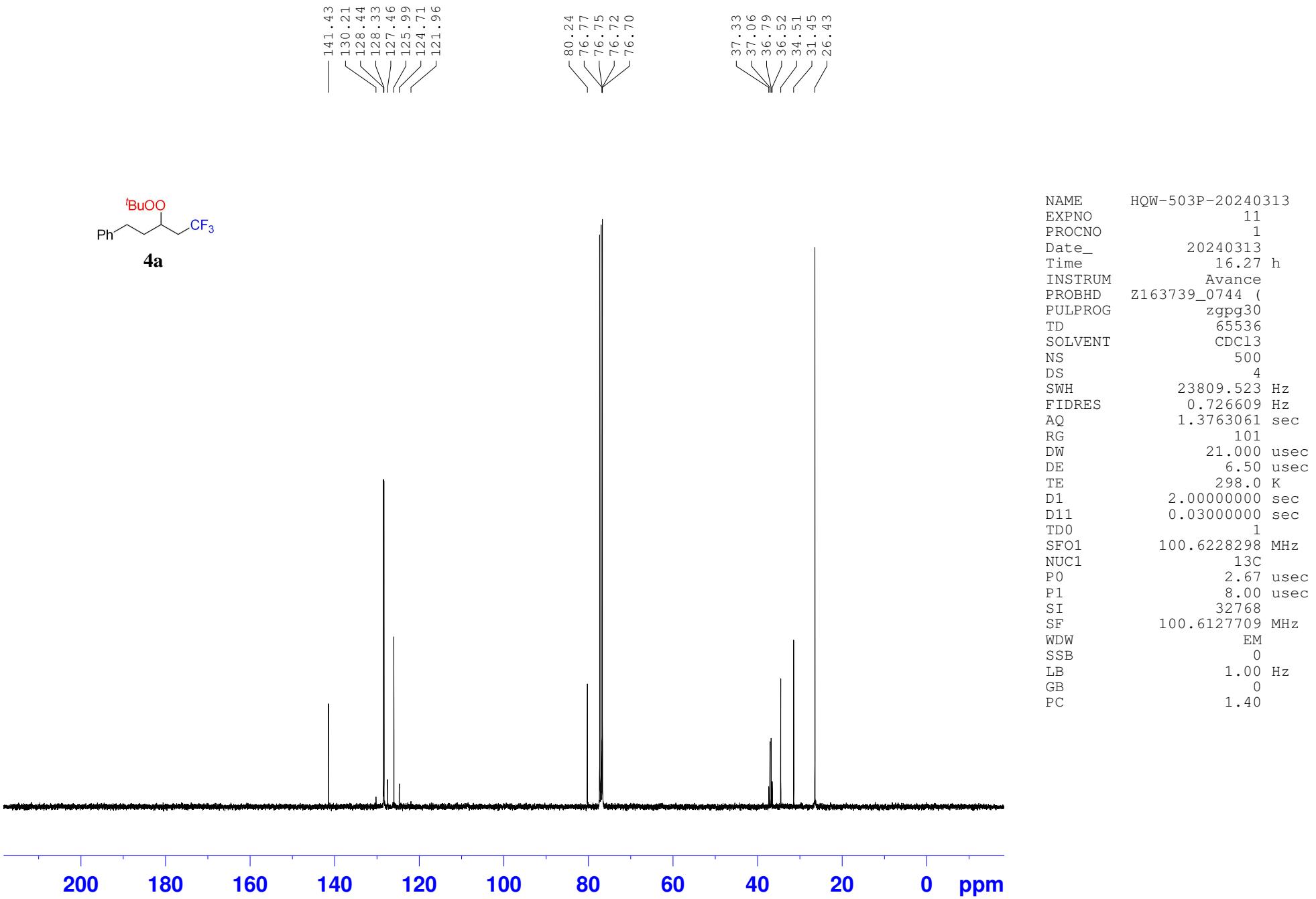
6. References

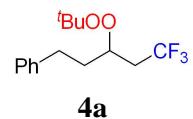
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7. Copies of ^1H NMR, ^{13}C NMR and ^{19}F NMR spectra for products

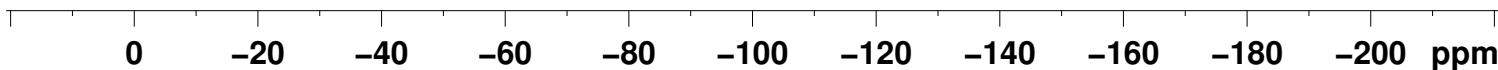


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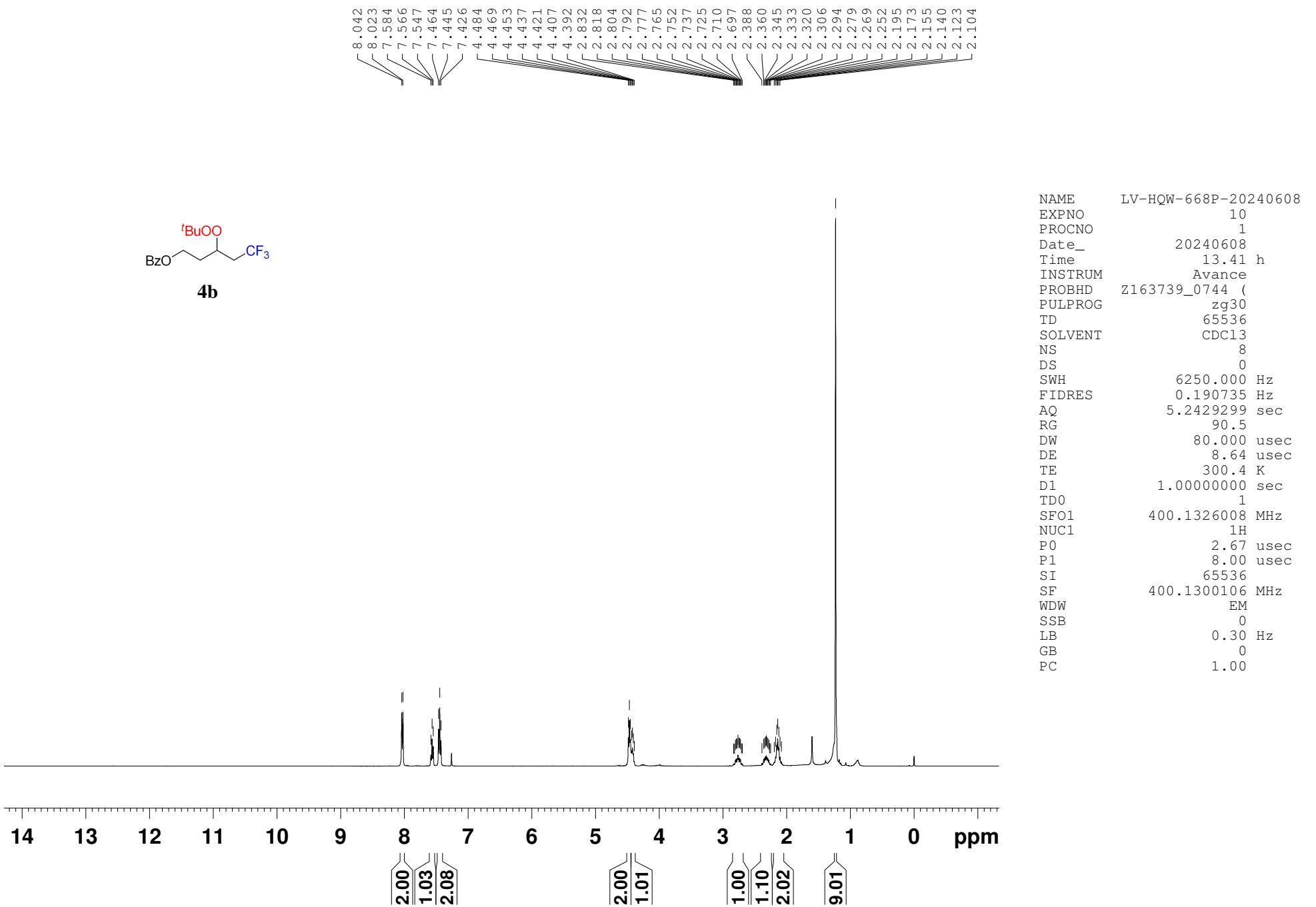


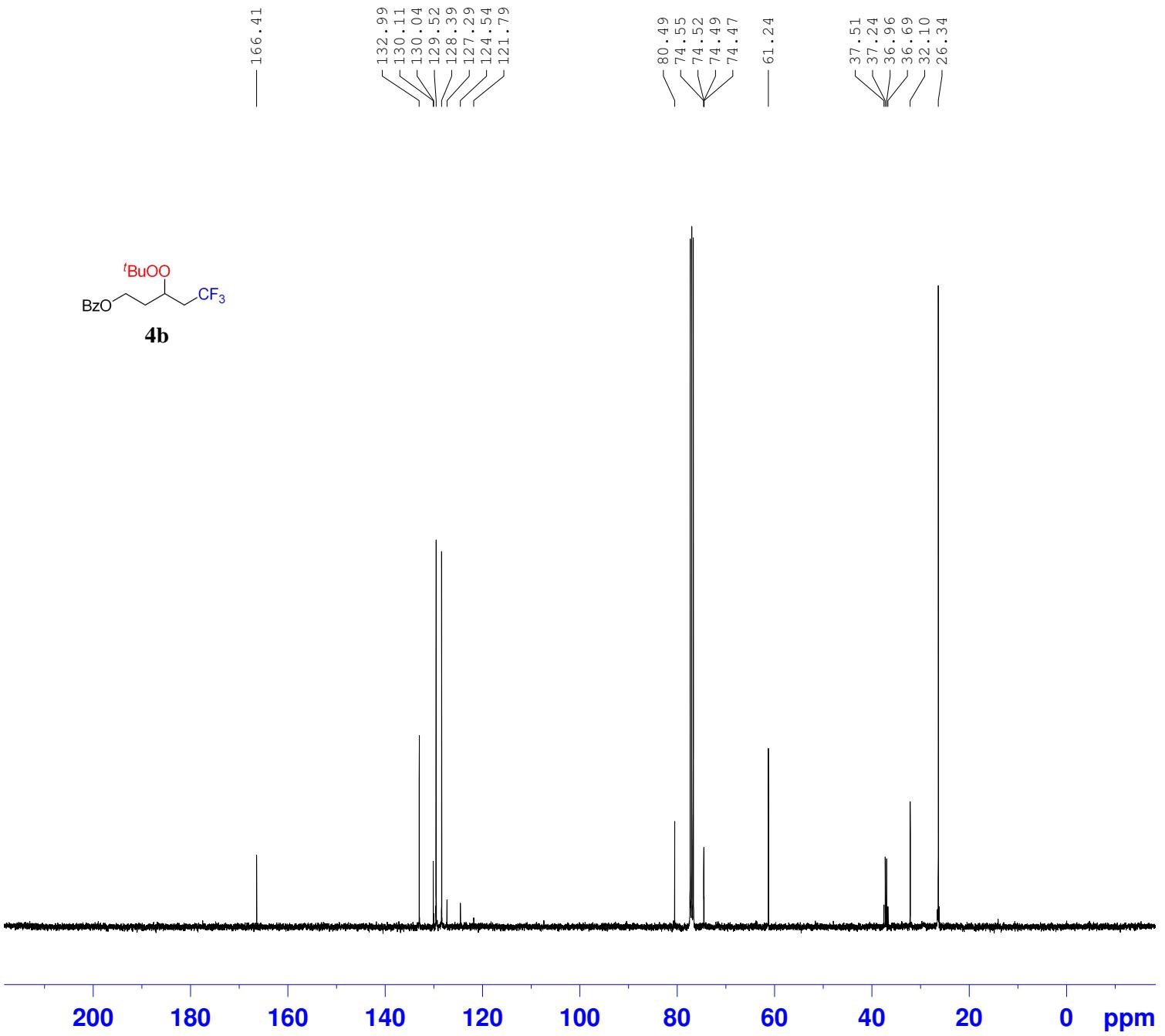


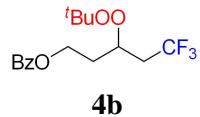
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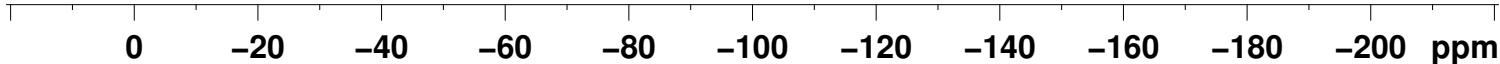
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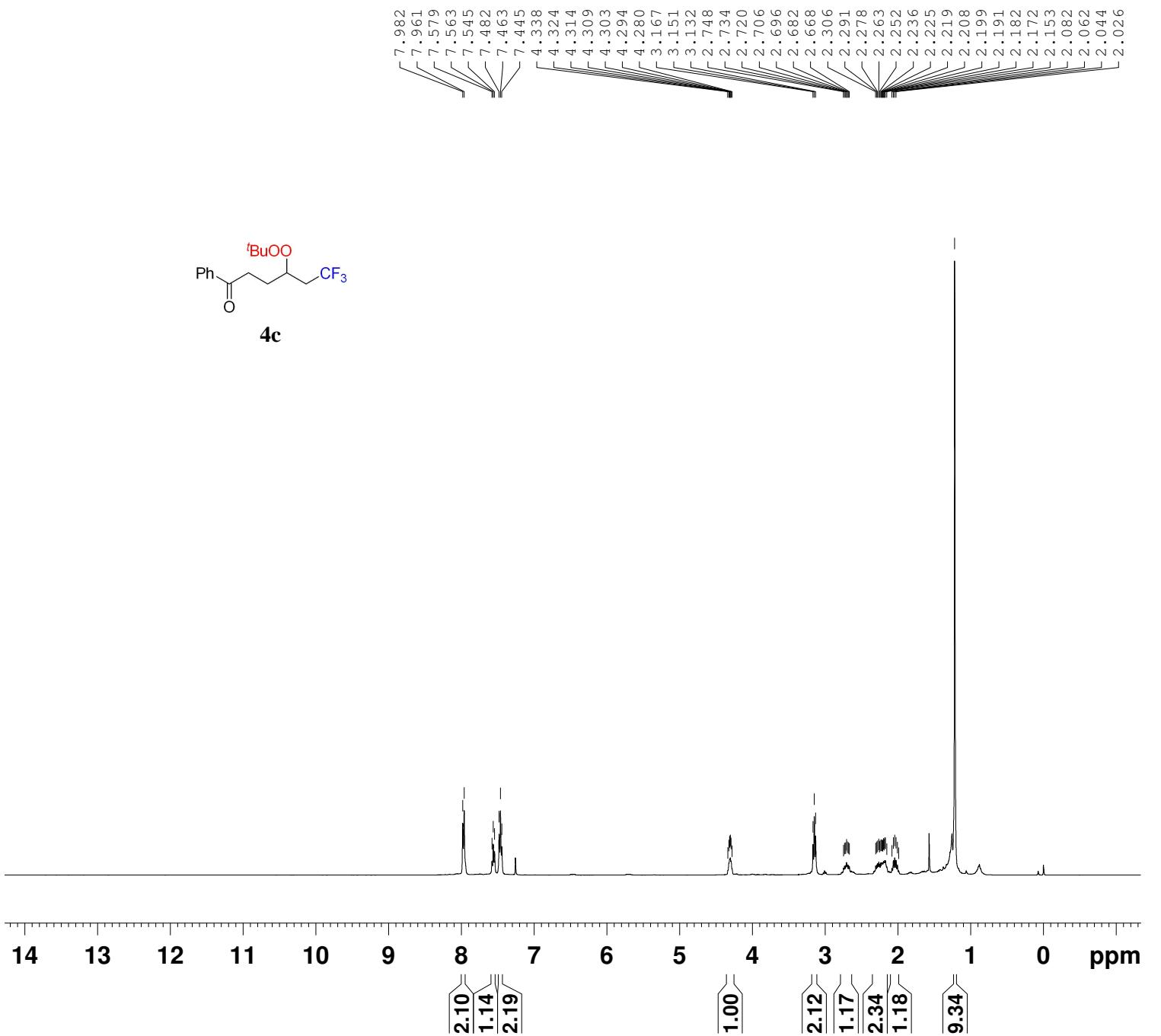


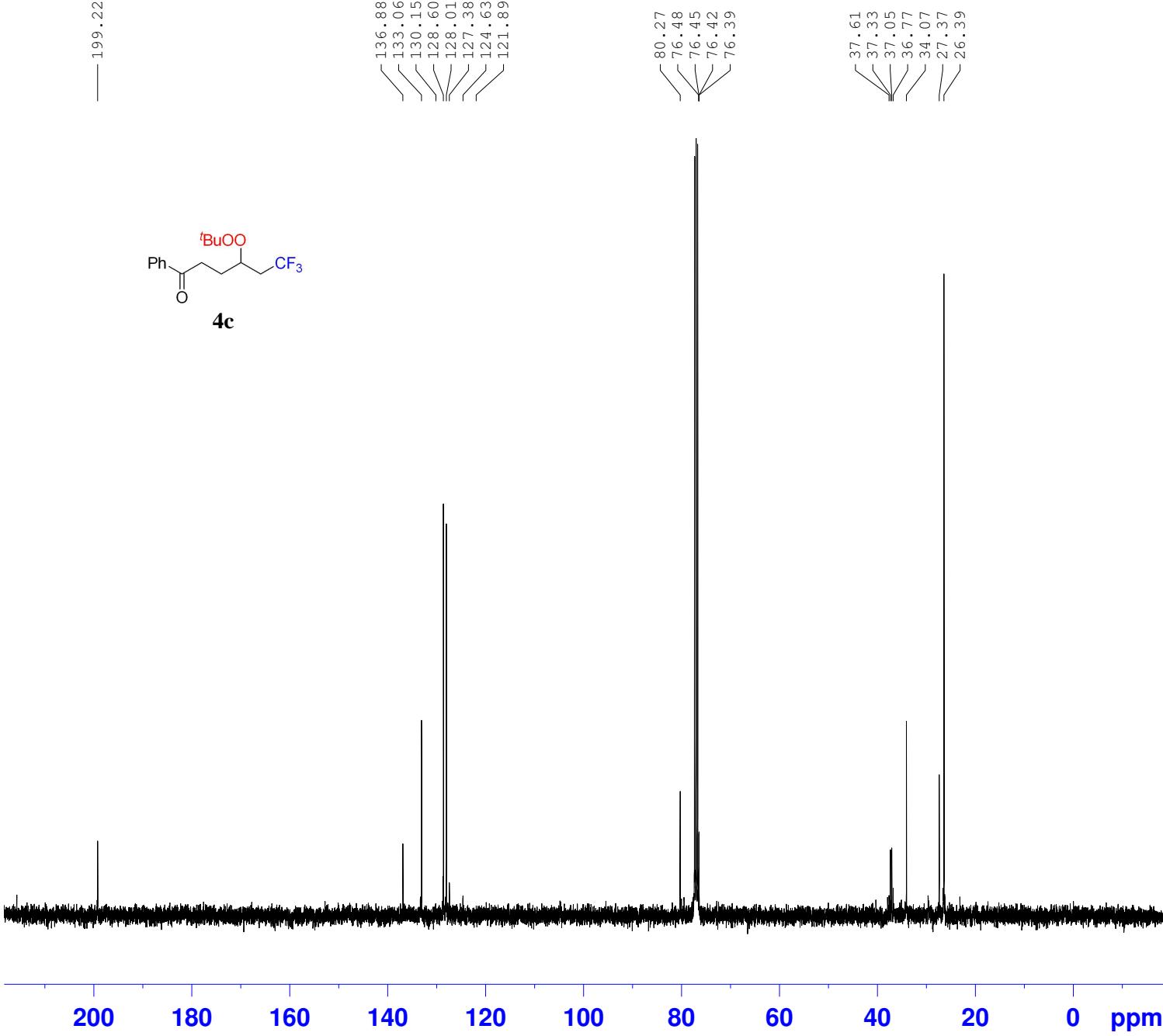


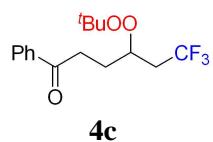
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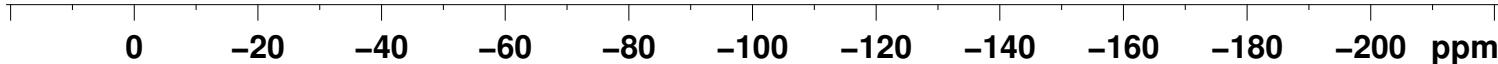
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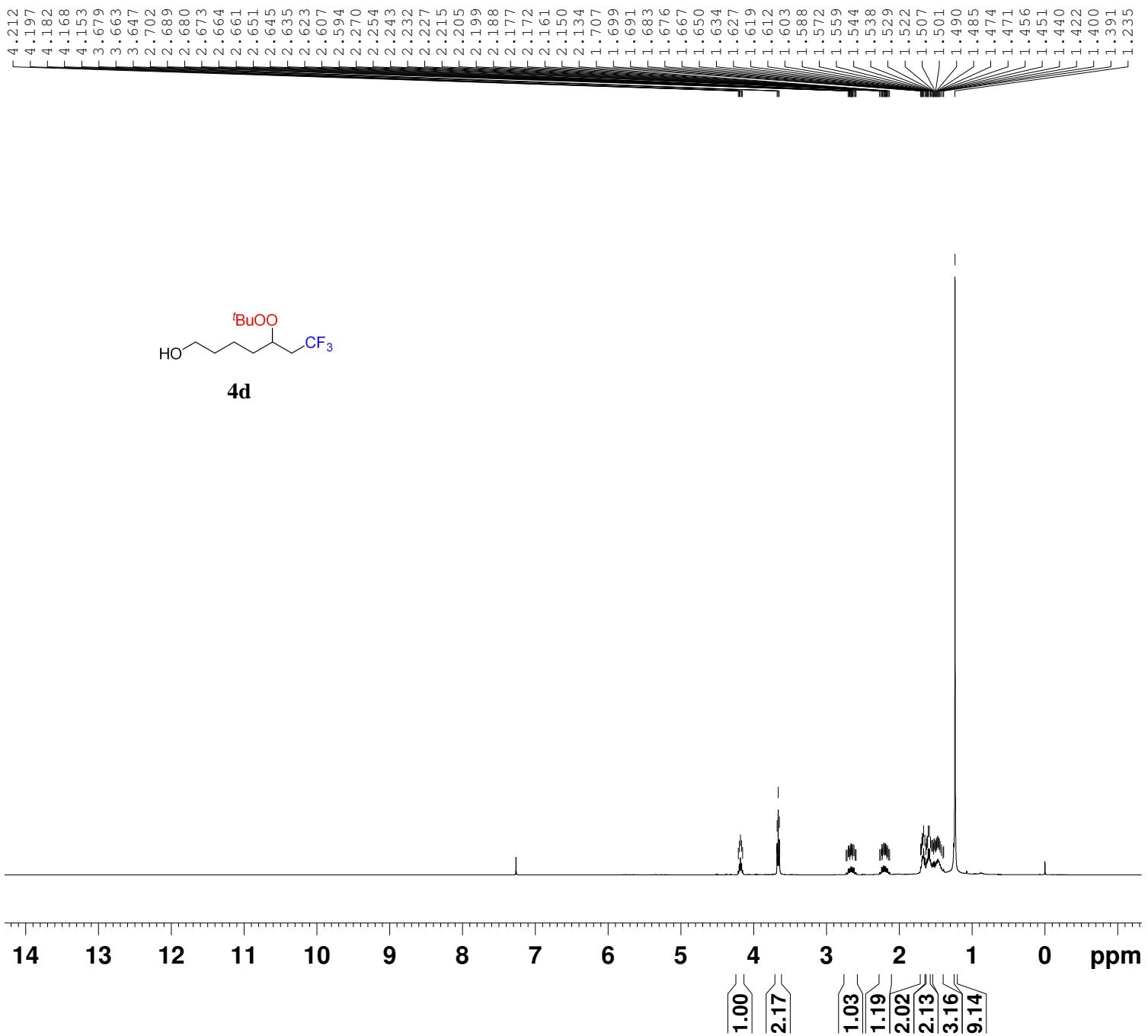




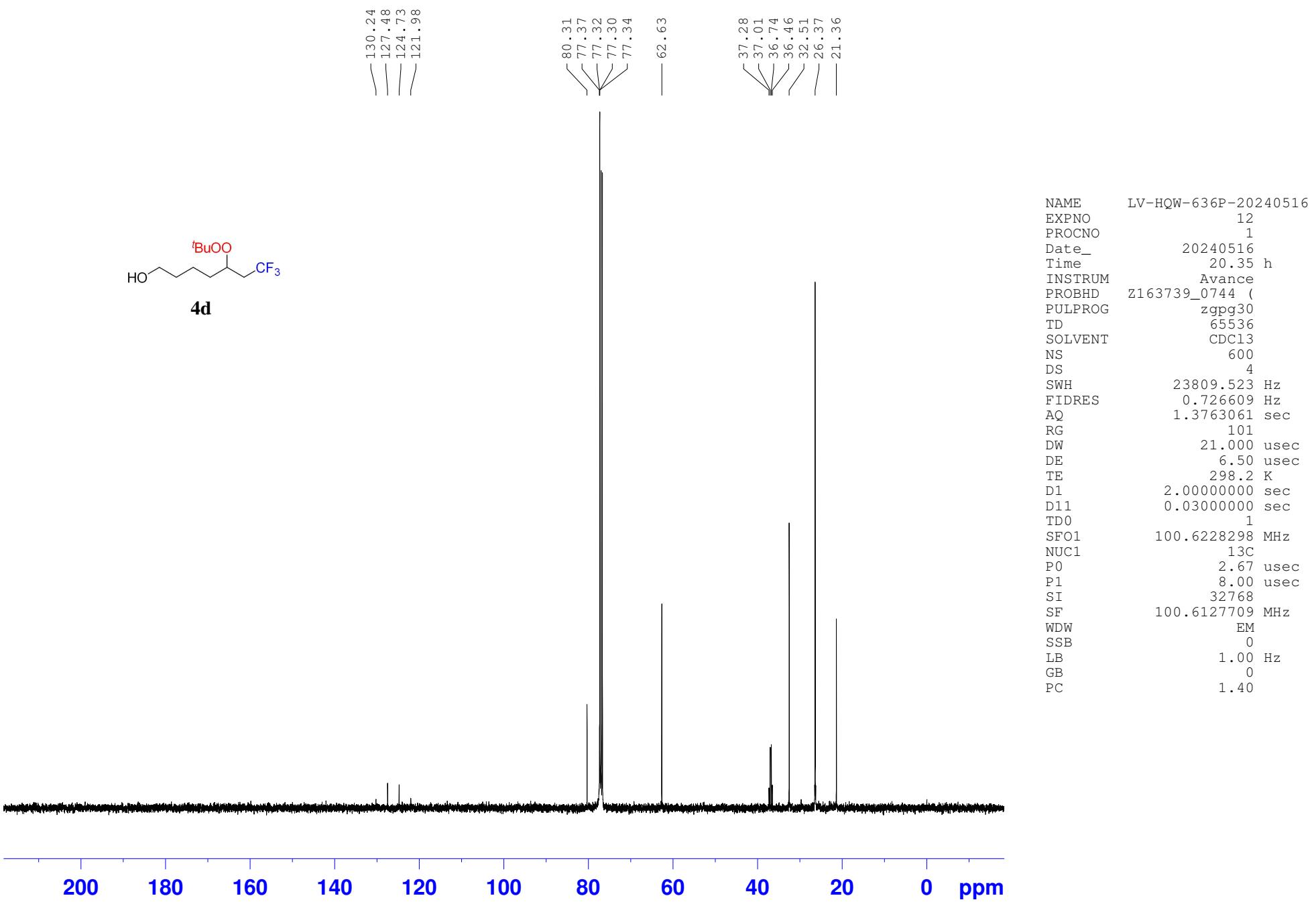
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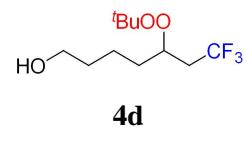


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SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	305.9 K
D1	1.0000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

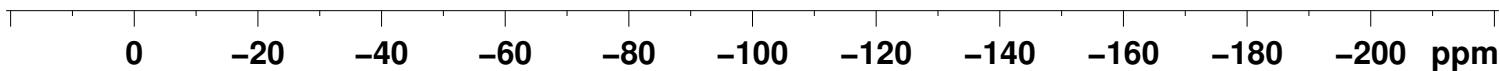


NAME LV-HQW-636P-20240516
 EXPNO 10
 PROCNO 1
 Date_ 20240516
 Time 19.56 h
 INSTRUM Avance
 PROBHD Z163739_0744 (zg30
 PULPROG 65536
 TD 65536
 SOLVENT CDC13
 NS 8
 DS 0
 SWH 6250.000 Hz
 FIDRES 0.190735 Hz
 AQ 5.2429299 sec
 RG 90.5
 DW 80.000 usec
 DE 8.64 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1
 SFO1 400.1326008 MHz
 NUC1 1H
 P0 2.67 usec
 P1 8.00 usec
 SI 65536
 SF 400.1300083 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

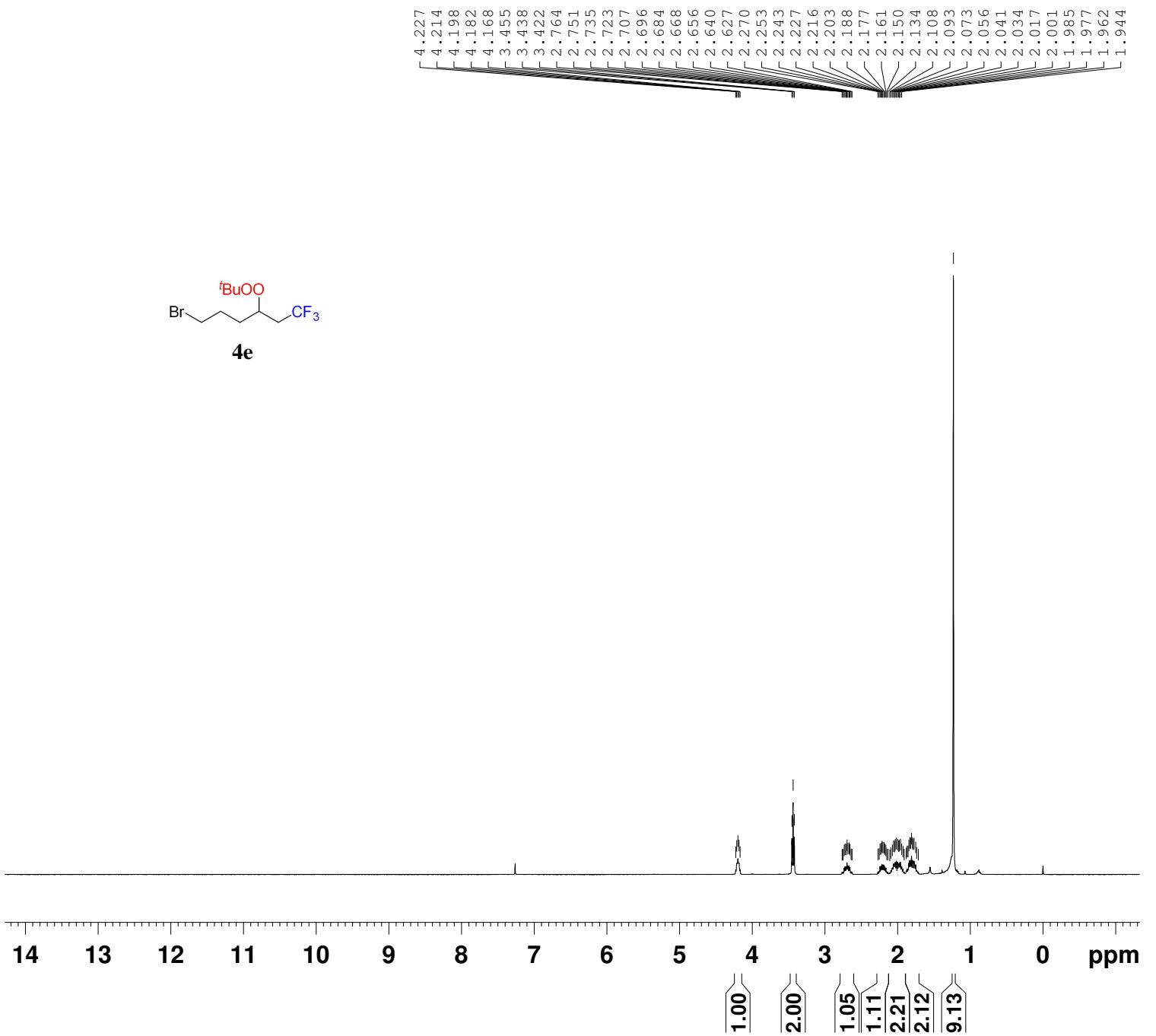
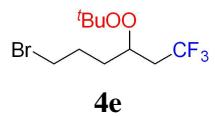




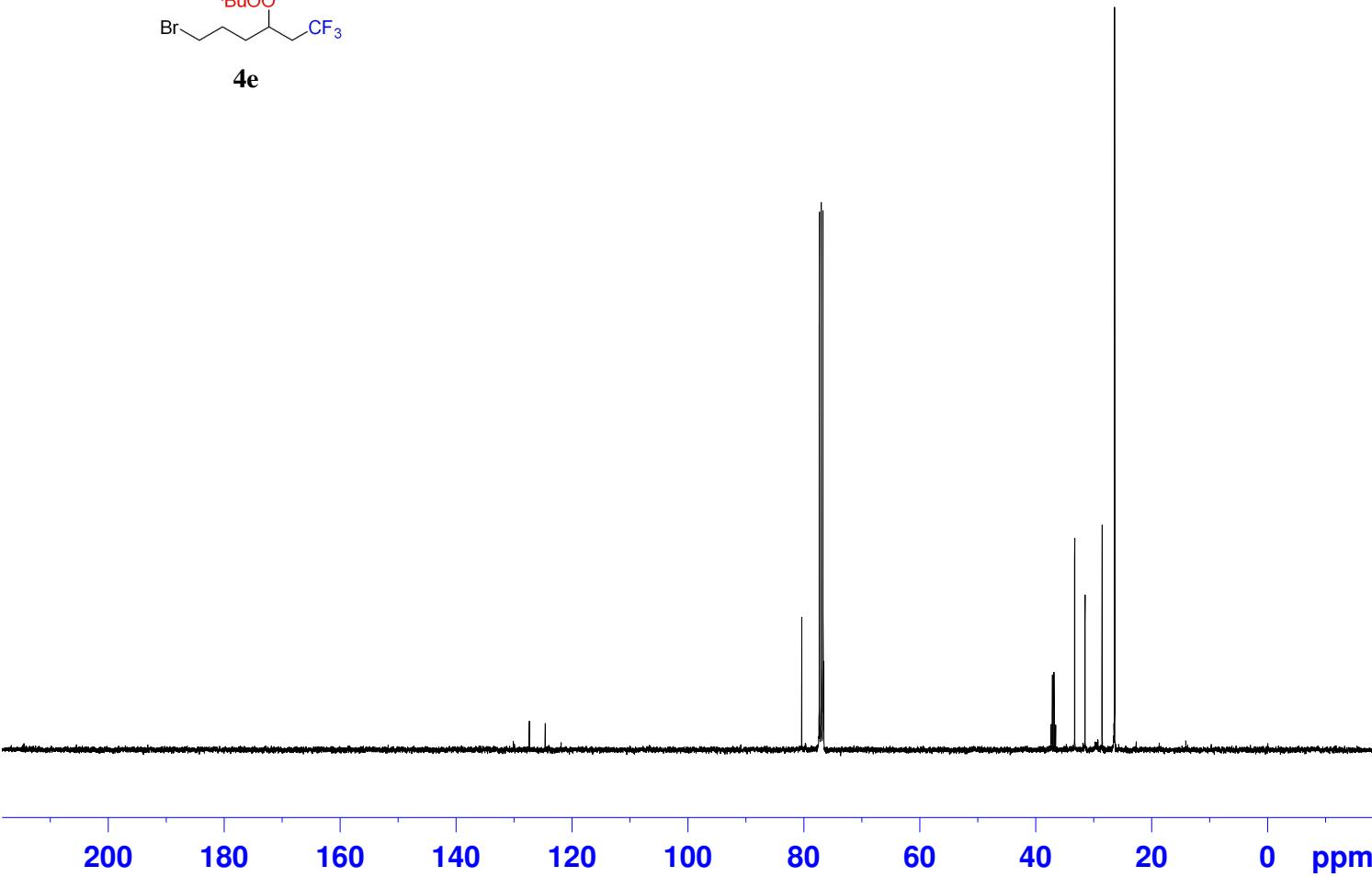
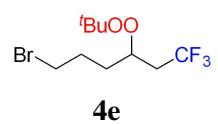
— -63.114



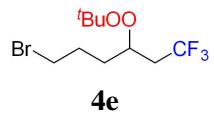
NAME	LV-HQW-636P-20240516
EXPNO	11
PROCNO	1
Date_	20240516
Time	19.58 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.1 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	¹⁹ F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



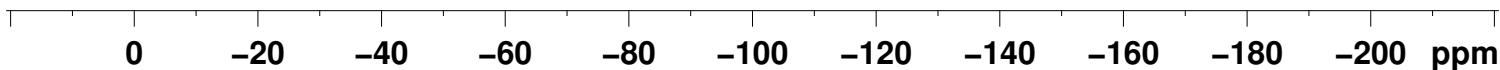
NAME	LV-HQW-635P-20240516
EXPNO	10
PROCNO	1
Date_	20240516
Time	15.38 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zg30
TD	65536
SOLVENT	CDC13
NS	8
DS	0
SWH	6250.000 Hz
FIDRES	0.190735 Hz
AQ	5.2429299 sec
RG	90.5
DW	80.000 usec
DE	8.64 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
SFO1	400.1326008 MHz
NUC1	1H
P0	2.67 usec
P1	8.00 usec
SI	65536
SF	400.1300095 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



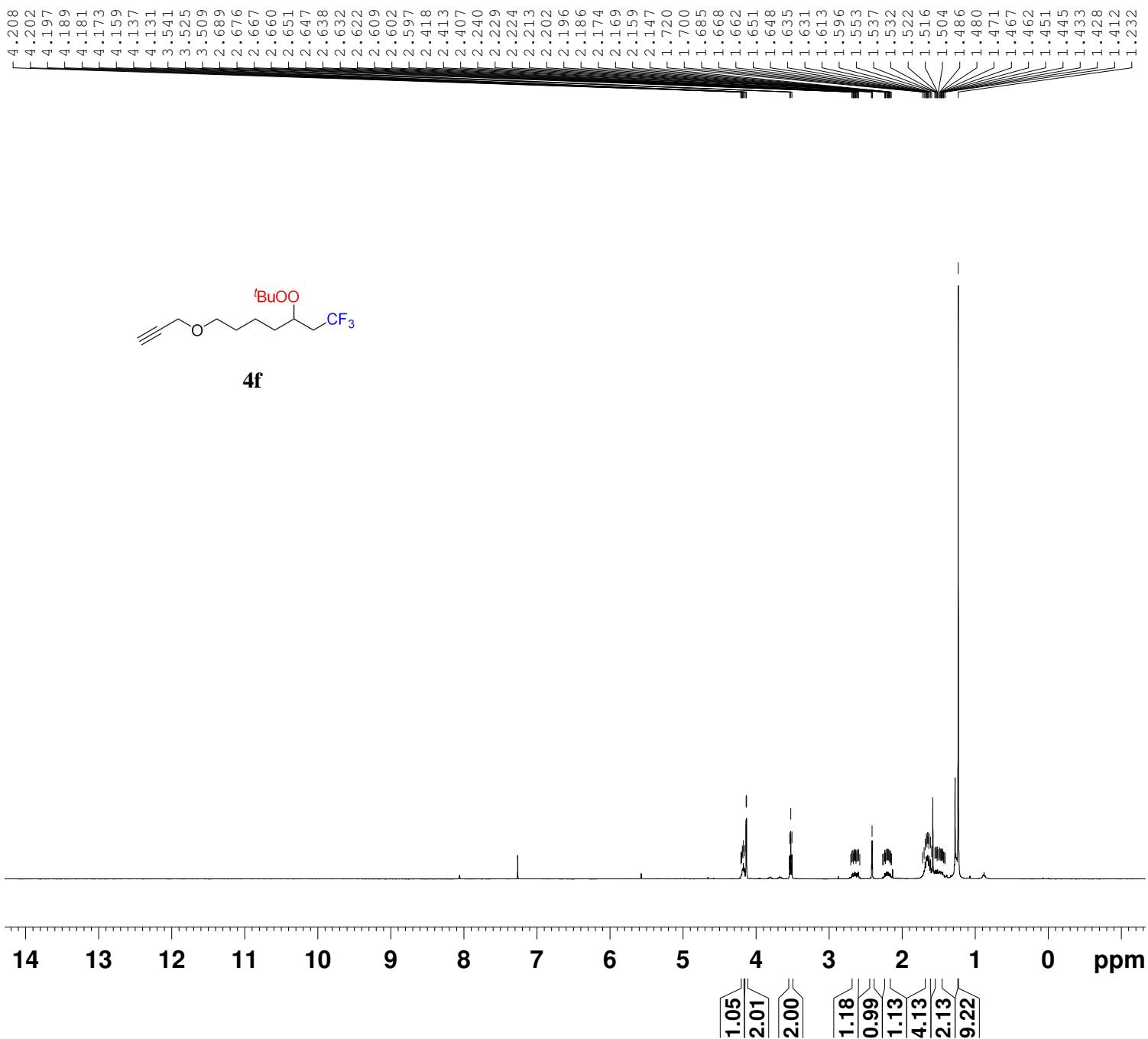
NAME	LV-HQW-635P-20240516
EXPNO	12
PROCNO	1
Date_	20240516
Time	16.15 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl ₃
NS	600
DS	4
SWH	23809.523 Hz
FIDRES	0.726609 Hz
AQ	1.3763061 sec
RG	101
DW	21.000 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TDO	1
SFO1	100.6228298 MHz
NUC1	¹³ C
P0	2.67 usec
P1	8.00 usec
SI	32768
SF	100.6127702 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

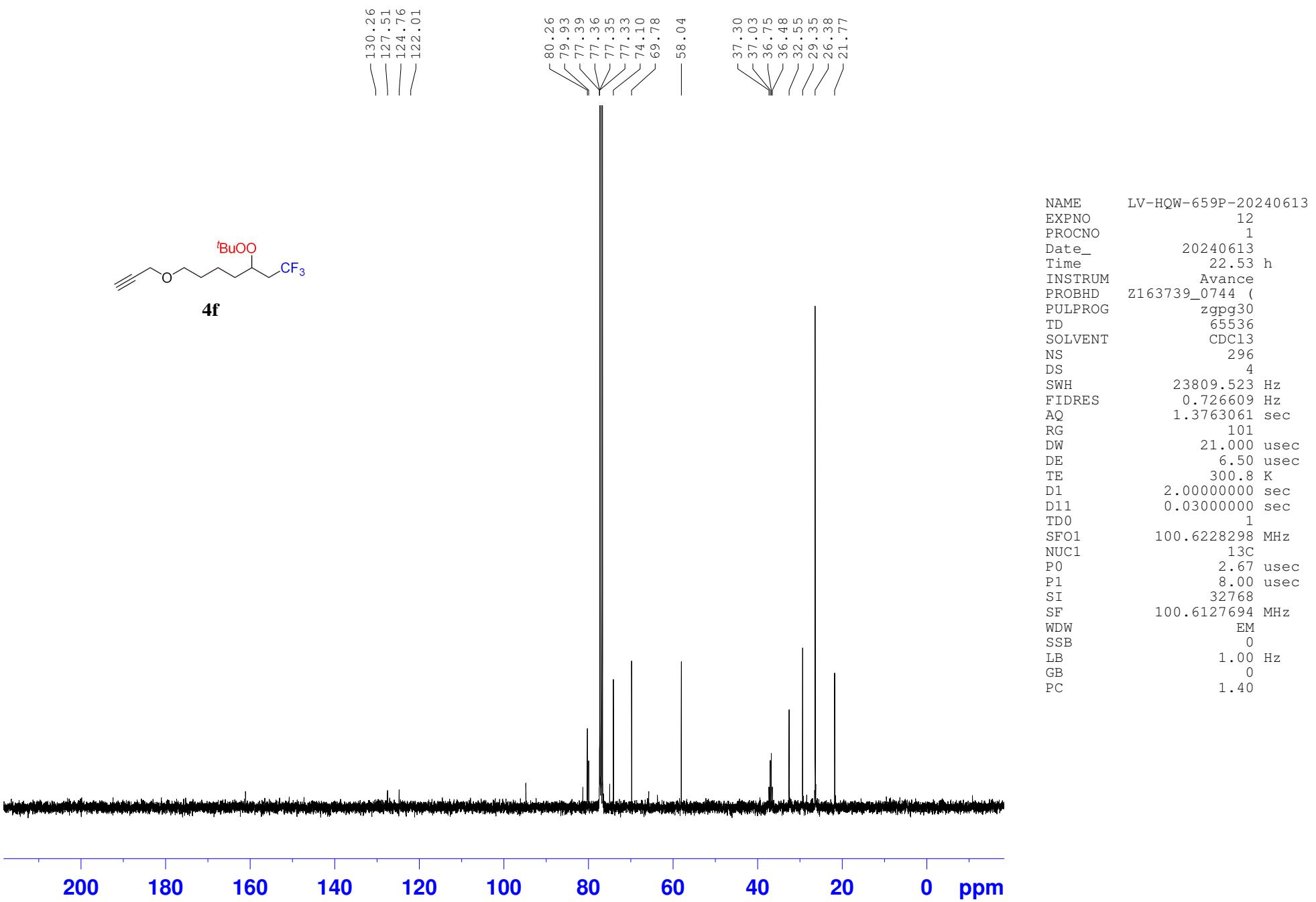


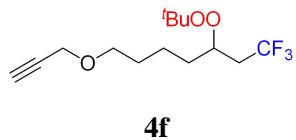
— -63.184 —



NAME	LV-HQW-635P-20240516
EXPNO	11
PROCNO	1
Date_	20240516
Time	15.39 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.1 K
D1	1.0000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



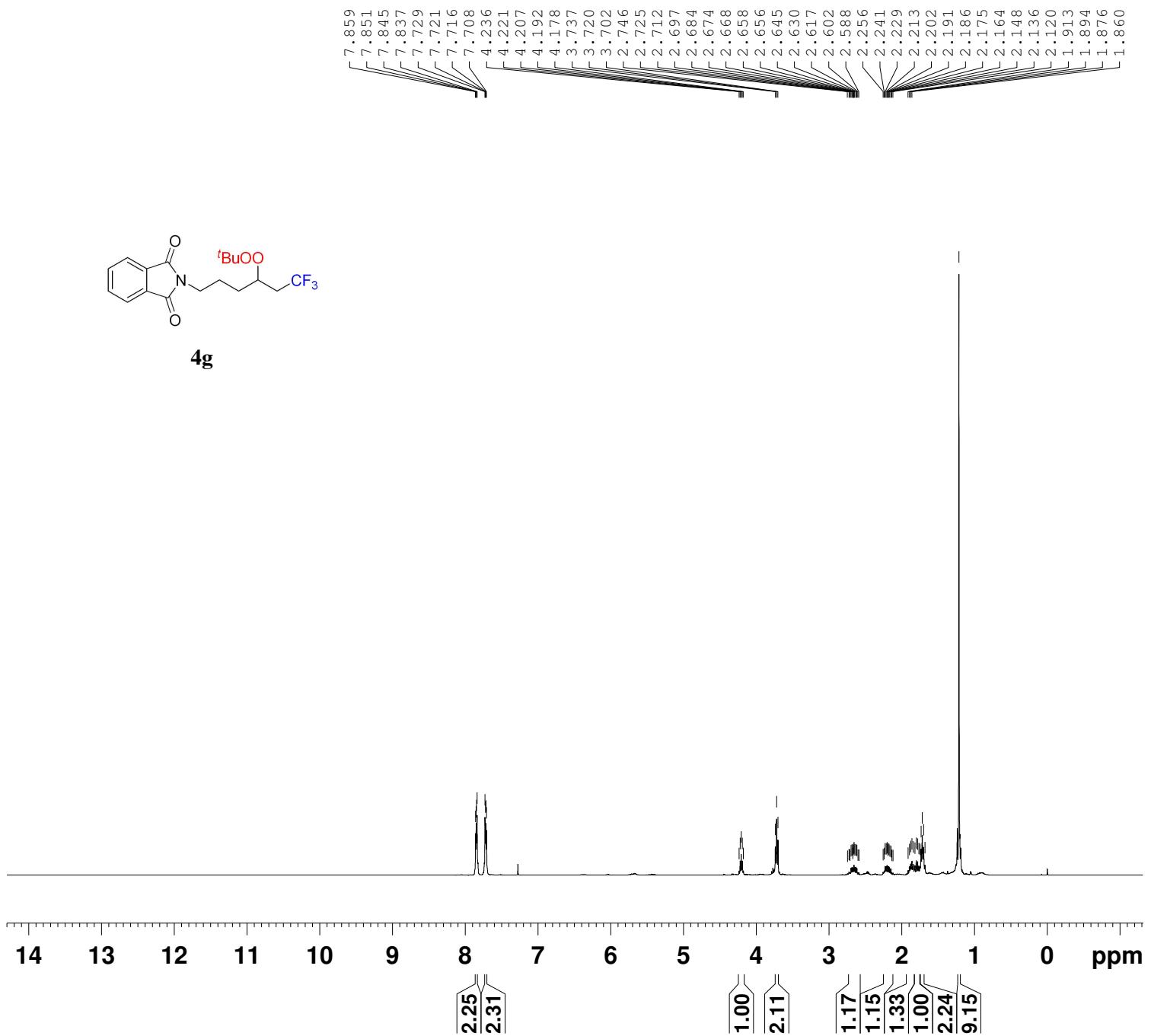


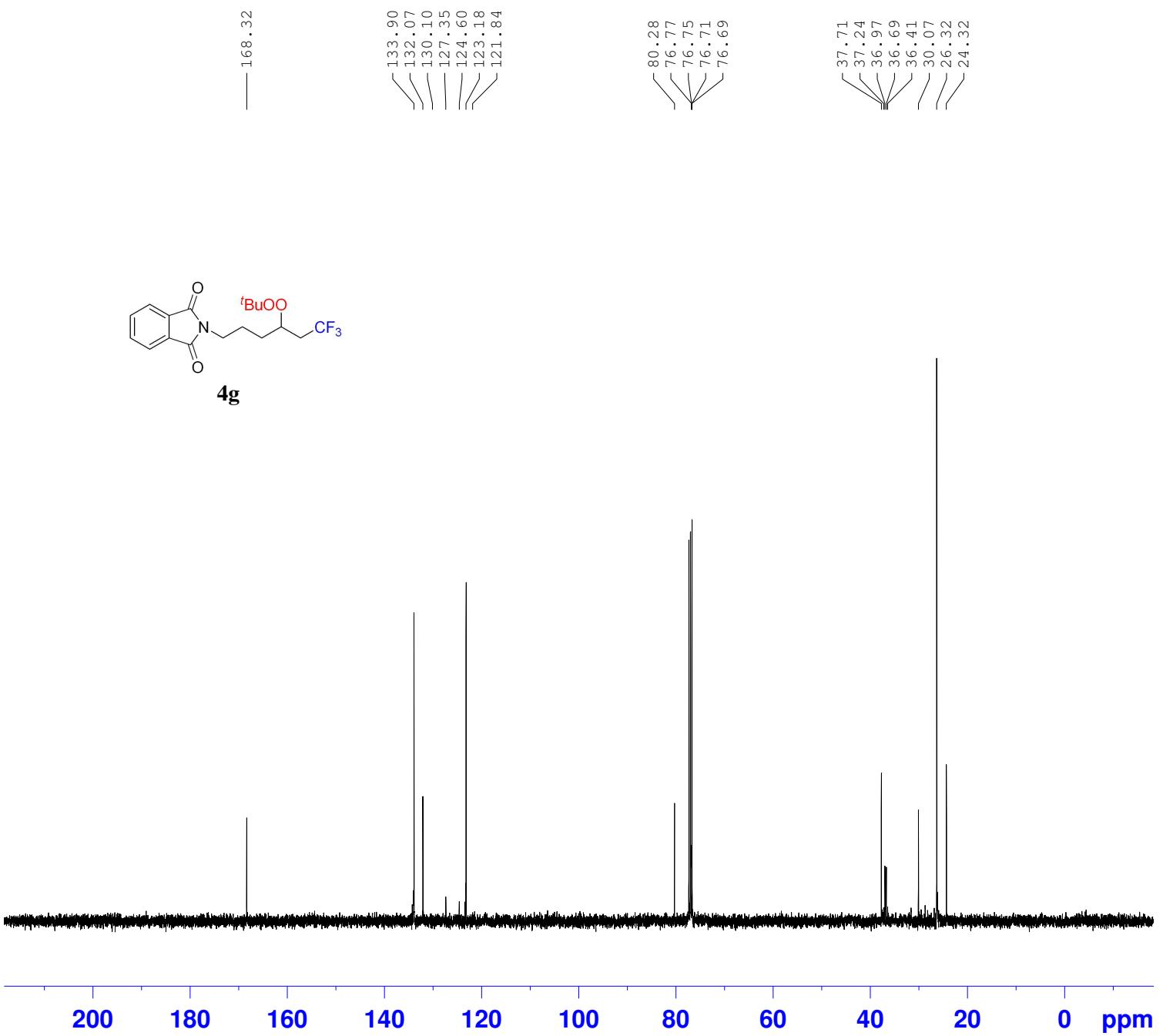


-63.118



NAME	LV-HQW-659P-20240613
EXPNO	11
PROCNO	1
Date_	20240613
Time	22.34 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	300.6 K
D1	1.0000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	¹⁹ F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

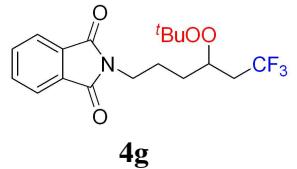




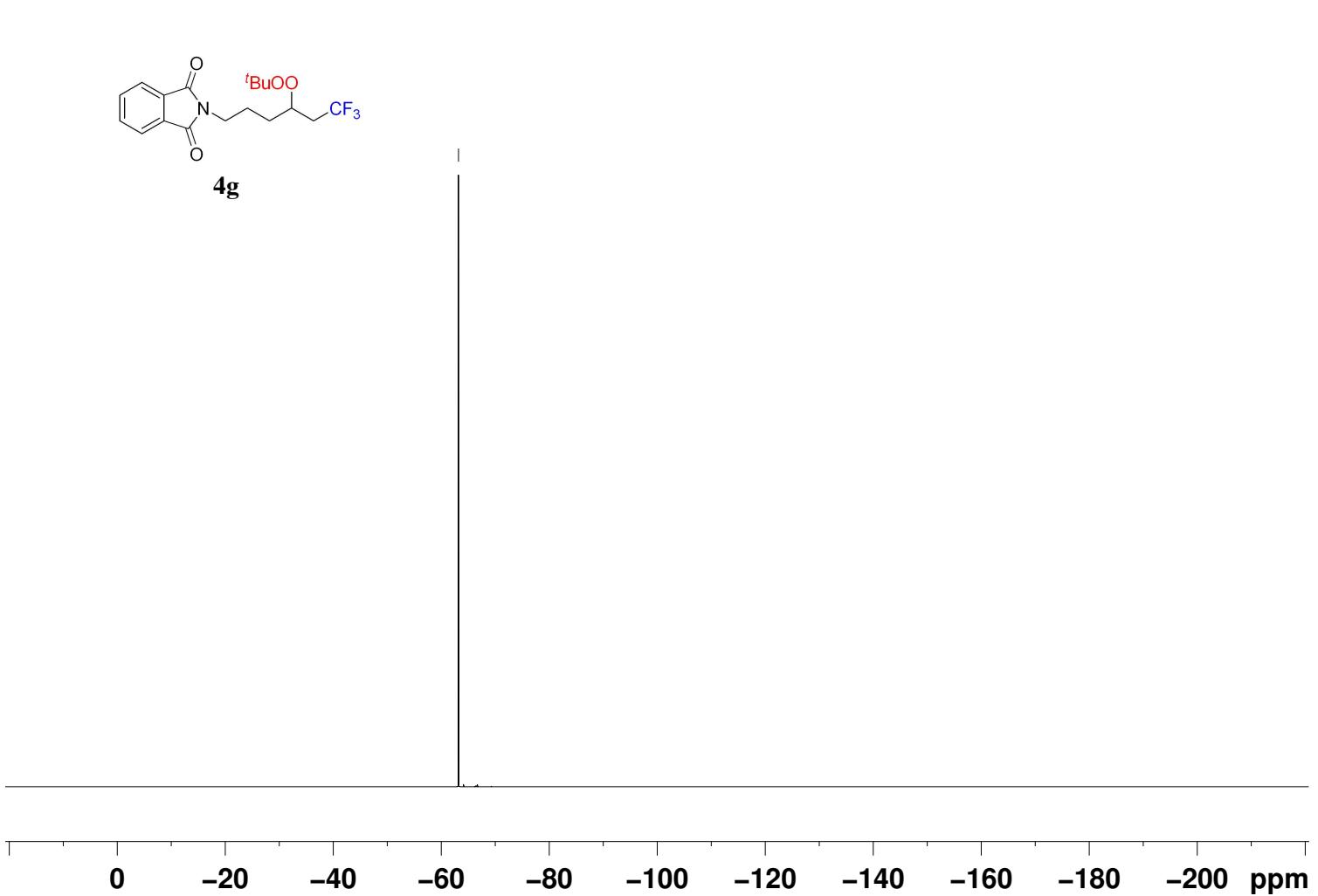
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NAME      LV-HQW-660P-20240530
EXPNO    12
PROCNO   1
Date_   20240530
Time   14.08 h
INSTRUM Avance
PROBHD Z163739_0744 (
PULPROG zgpg30
TD      65536
SOLVENT CDC13
NS      48
DS      4
SWH     23809.523 Hz
FIDRES  0.726609 Hz
AQ      1.3763061 sec
RG      101
DW      21.000 usec
DE      6.50 usec
TE      299.1 K
D1      2.0000000 sec
D11     0.0300000 sec
TD0      1
SFO1    100.6228298 MHz
NUC1    13C
P0      2.67 usec
P1      8.00 usec
SI      32768
SF      100.6127720 MHz
WDW
SSB
LB      1.00 Hz
GB
PC      1.40

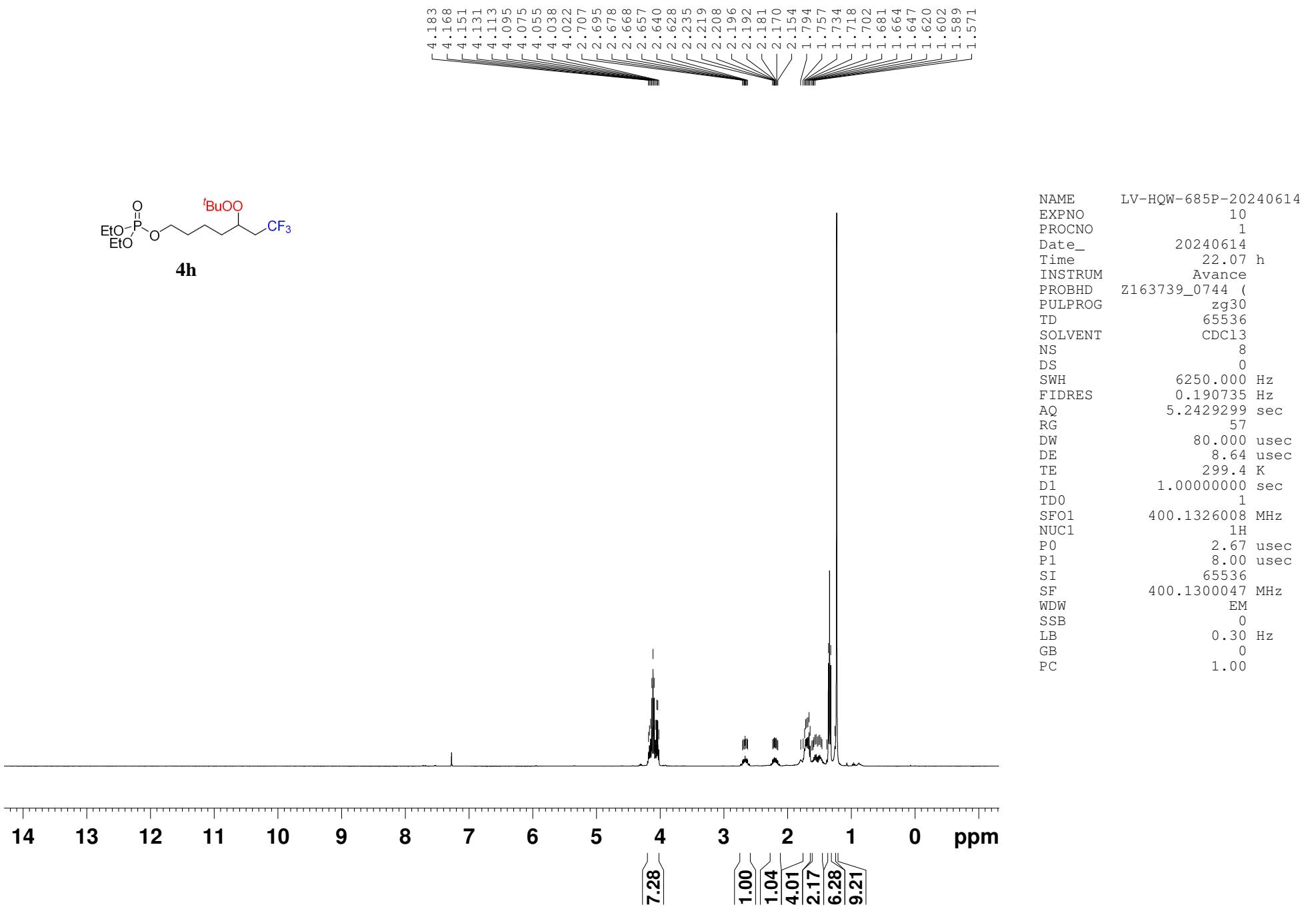
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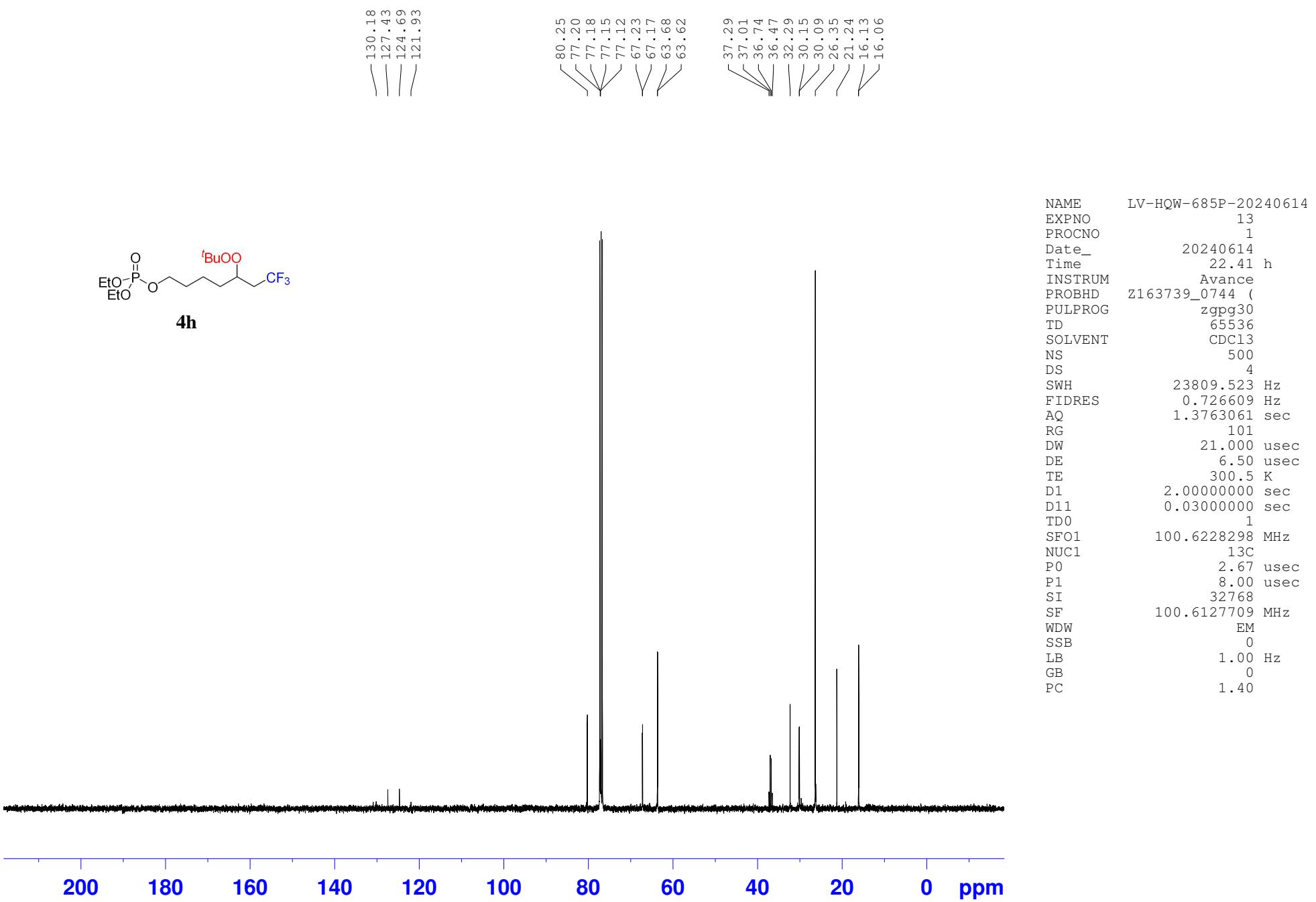


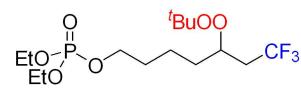
- 63 . 195



NAME LV-HQW-660P-20240530
 EXPNO 11
 PROCNO 1
 Date_ 20240530
 Time 14.04 h
 INSTRUM Avance
 PROBHD Z163739_0744 (
 PULPROG zgig
 TD 131072
 SOLVENT CDC13
 NS 16
 DS 4
 SWH 90909.094 Hz
 FIDRES 1.387163 Hz
 AQ 0.7209460 sec
 RG 101
 DW 5.500 usec
 DE 6.50 usec
 TE 298.9 K
 D1 1.0000000 sec
 D11 0.0300000 sec
 TD0 1
 SFO1 376.4607164 MHz
 NUC1 19F
 P1 12.00 usec
 SI 65536
 SF 376.4983662 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

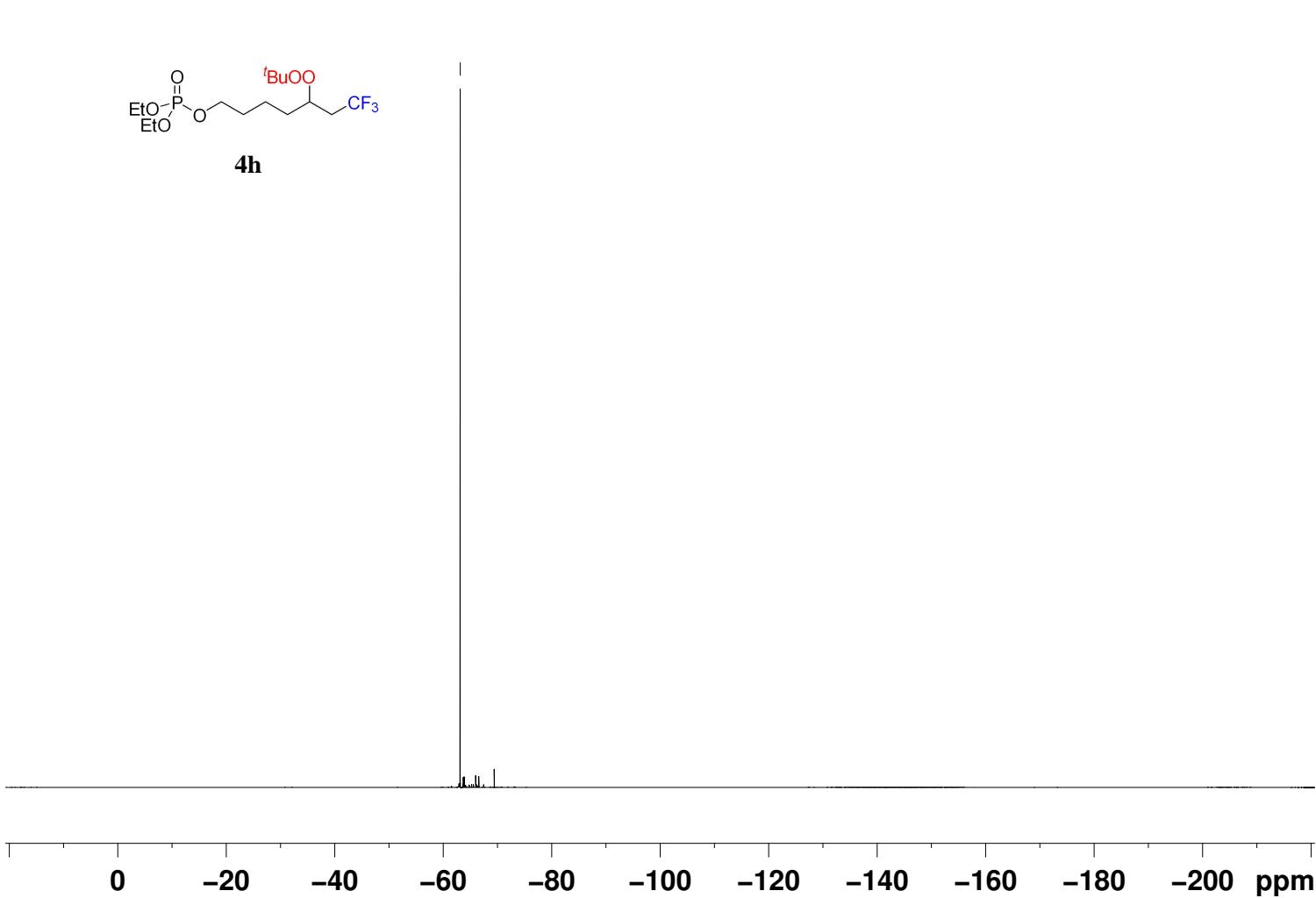




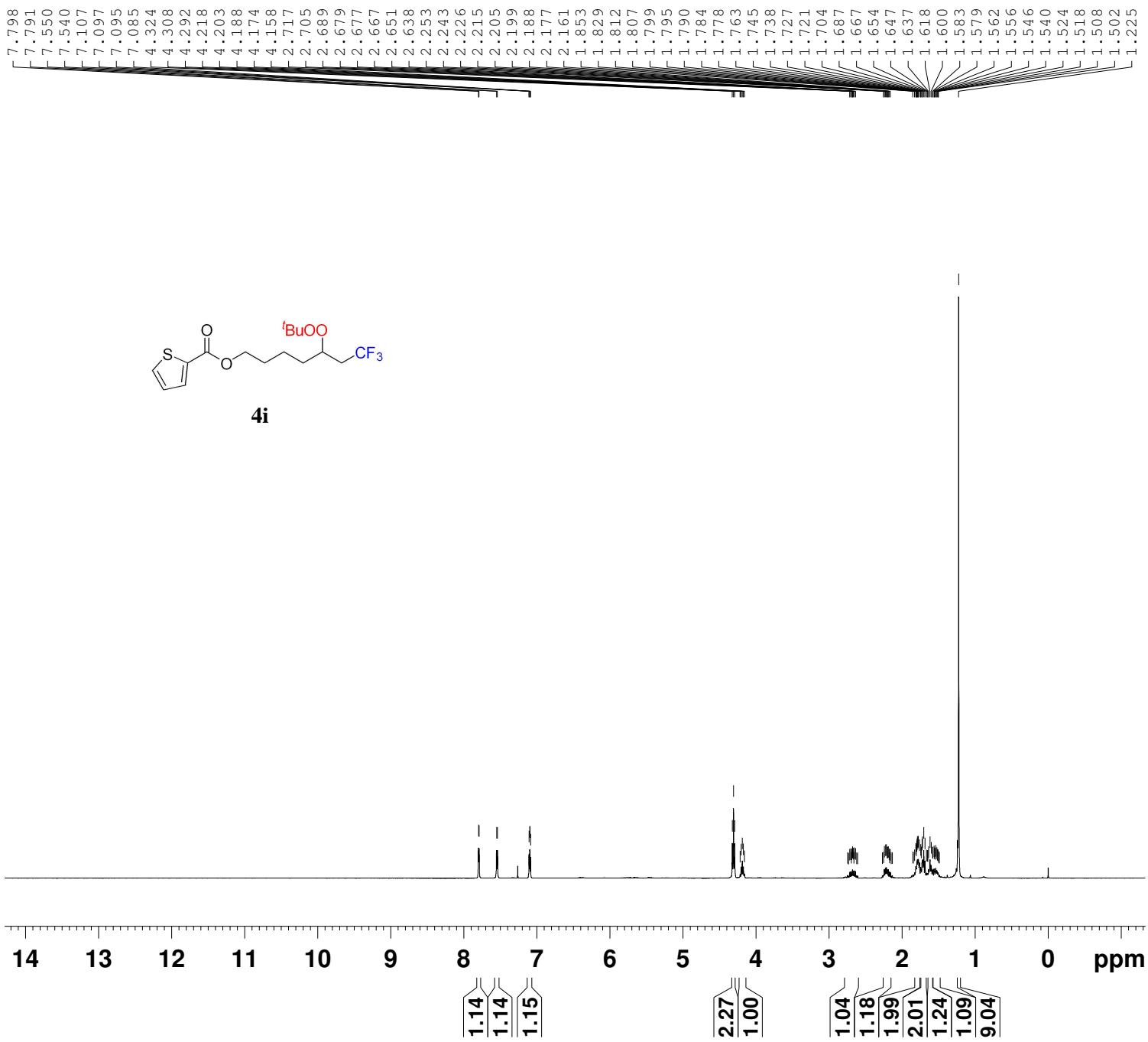


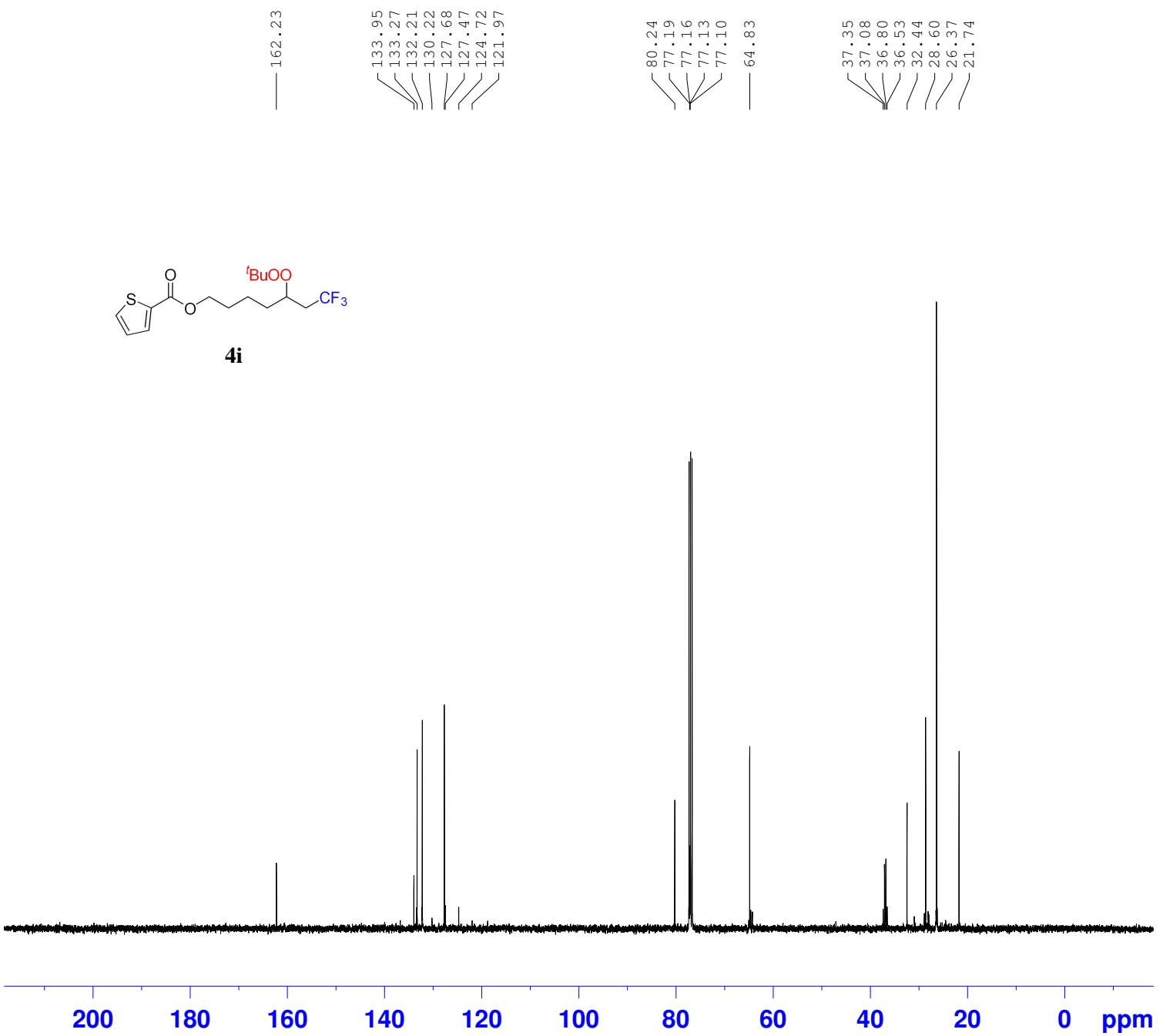
4h

— -63.139

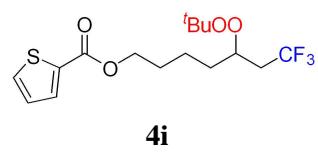


NAME	LV-HQW-685P-20240614
EXPNO	11
PROCNO	1
Date_	20240614
Time	22.08 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	299.6 K
D1	1.0000000 sec
D11	0.0300000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

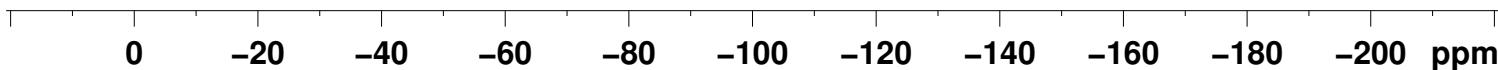




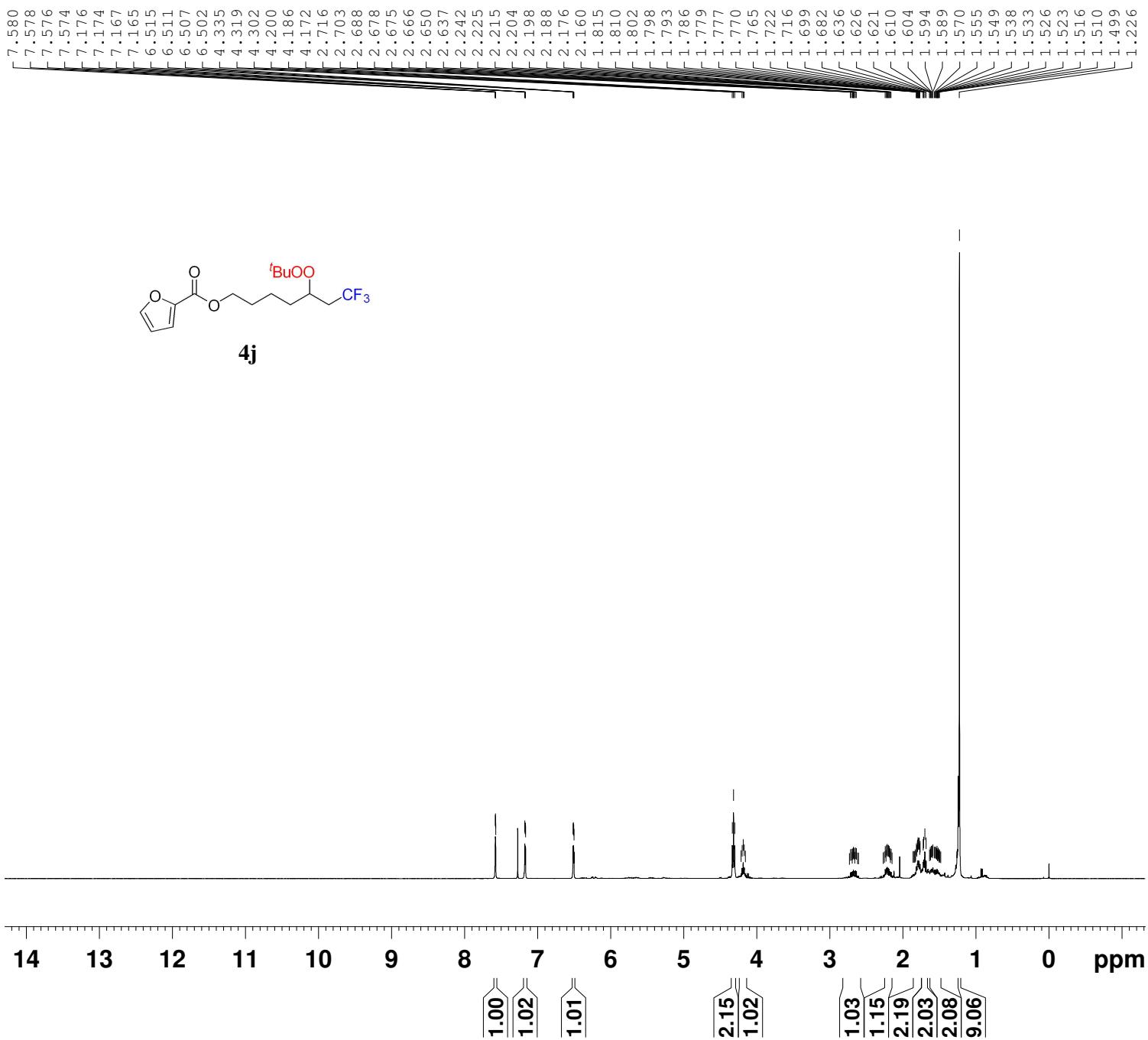
NAME	LV-HQW-697P-20240623
EXPNO	12
PROCNO	1
Date_	20240623
Time	23.18 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl ₃
NS	248
DS	4
SWH	23809.523 Hz
FIDRES	0.726609 Hz
AQ	1.3763061 sec
RG	101
DW	21.000 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TDO	1
SFO1	100.6228298 MHz
NUC1	¹³ C
P0	2.67 usec
P1	8.00 usec
SI	32768
SF	100.6127702 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

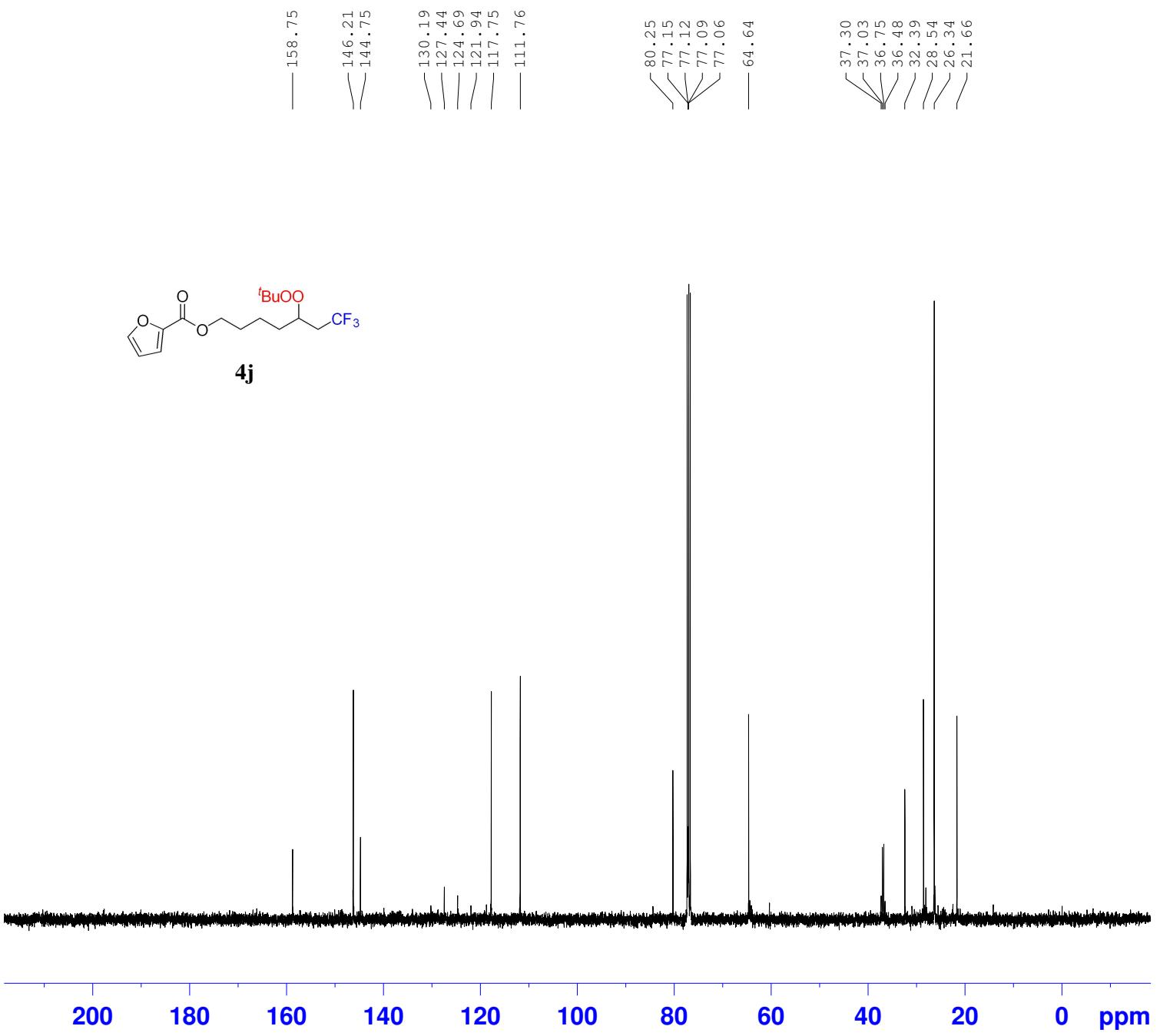


— -63.105



NAME	LV-HQW-697P-20240623
EXPNO	11
PROCNO	1
Date_	20240623
Time	23.02 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl3
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	302.2 K
D1	1.0000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

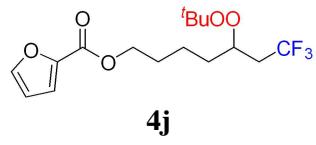




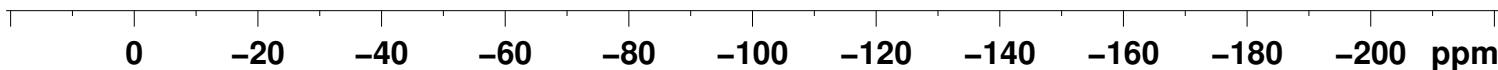
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NAME      LV-HQW-637P-20240517
EXPNO    12
PROCNO   1
Date_   20240518
Time    0.39 h
INSTRUM Avance
PROBHD Z163739_0744 (
PULPROG zgpg30
TD       65536
SOLVENT  CDCl3
NS      104
DS       4
SWH     23809.523 Hz
FIDRES  0.726609 Hz
AQ      1.3763061 sec
RG      101
DW      21.000 usec
DE      6.50 usec
TE      298.0 K
D1      2.00000000 sec
D11     0.03000000 sec
TD0      1
SFO1    100.6228298 MHz
NUC1    13C
P0      2.67 usec
P1      8.00 usec
SI      32768
SF      100.6127716 MHz
WDW
SSB
LB      1.00 Hz
GB
PC      1.40

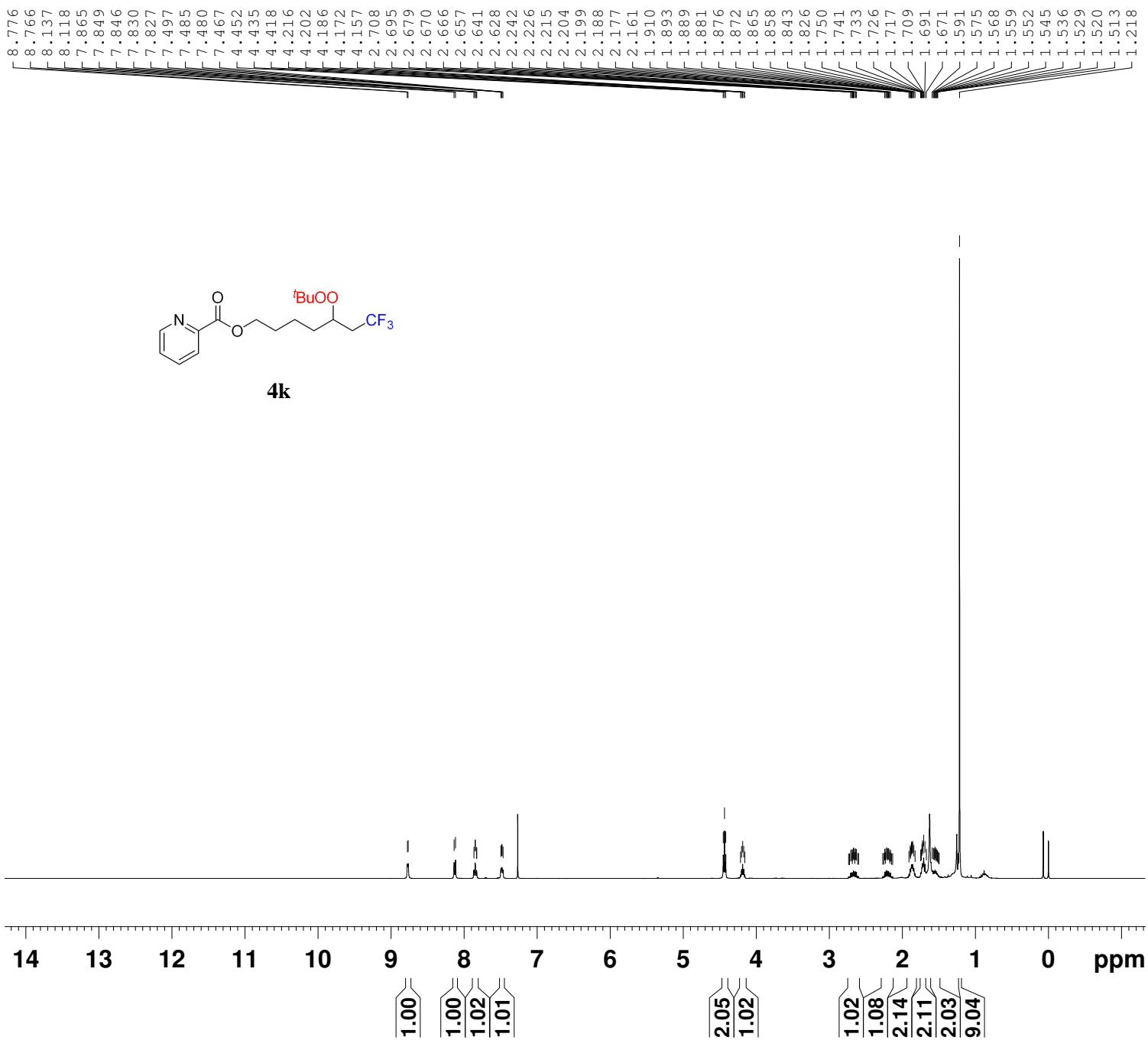
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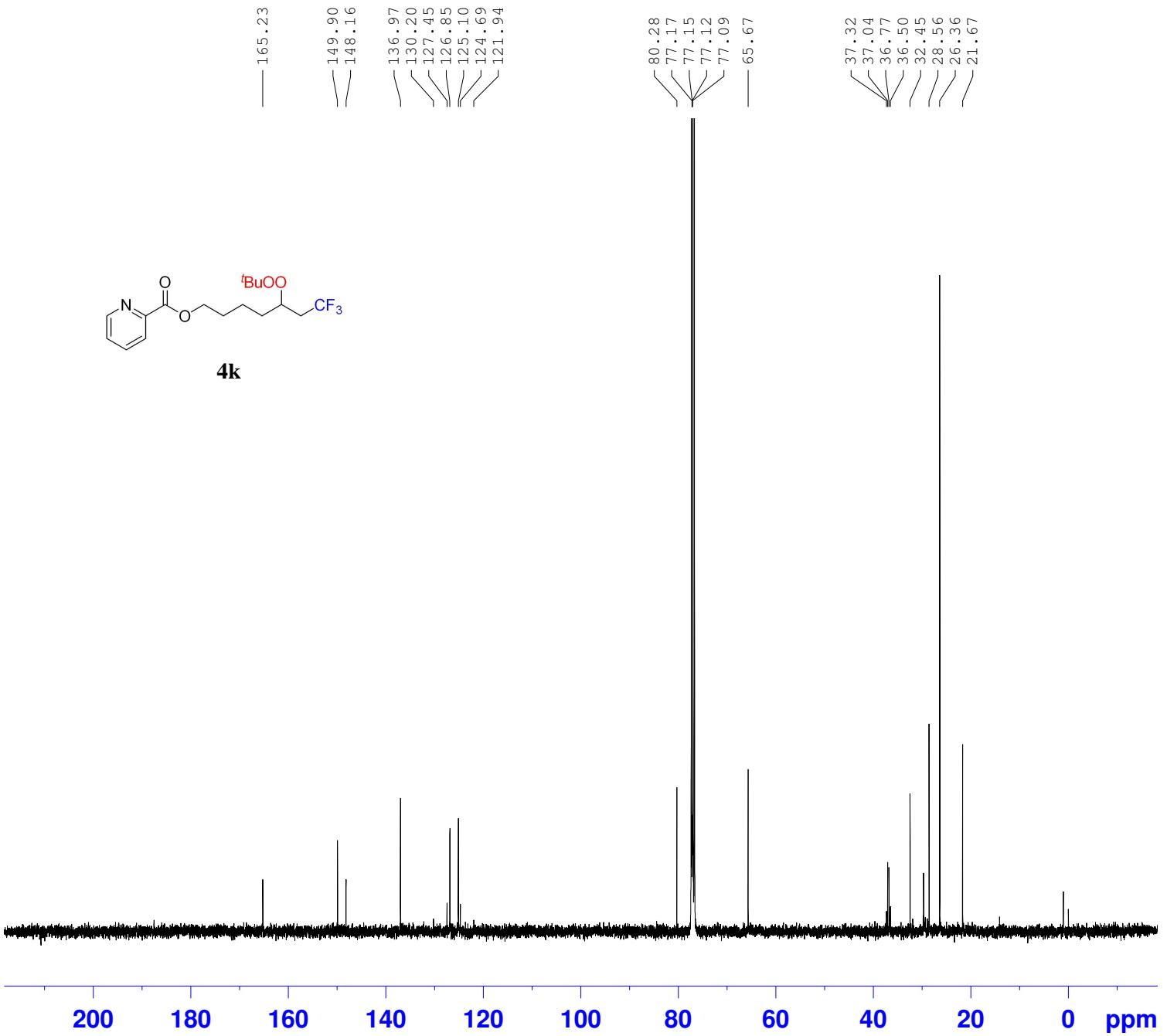


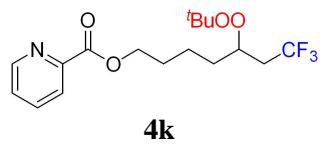
— -63.118



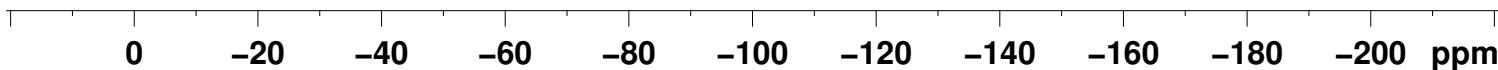
NAME	LV-HQW-637P-20240517
EXPNO	11
PROCNO	1
Date_	20240518
Time	0.31 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.1 K
D1	1.0000000 sec
D11	0.0300000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	¹⁹ F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



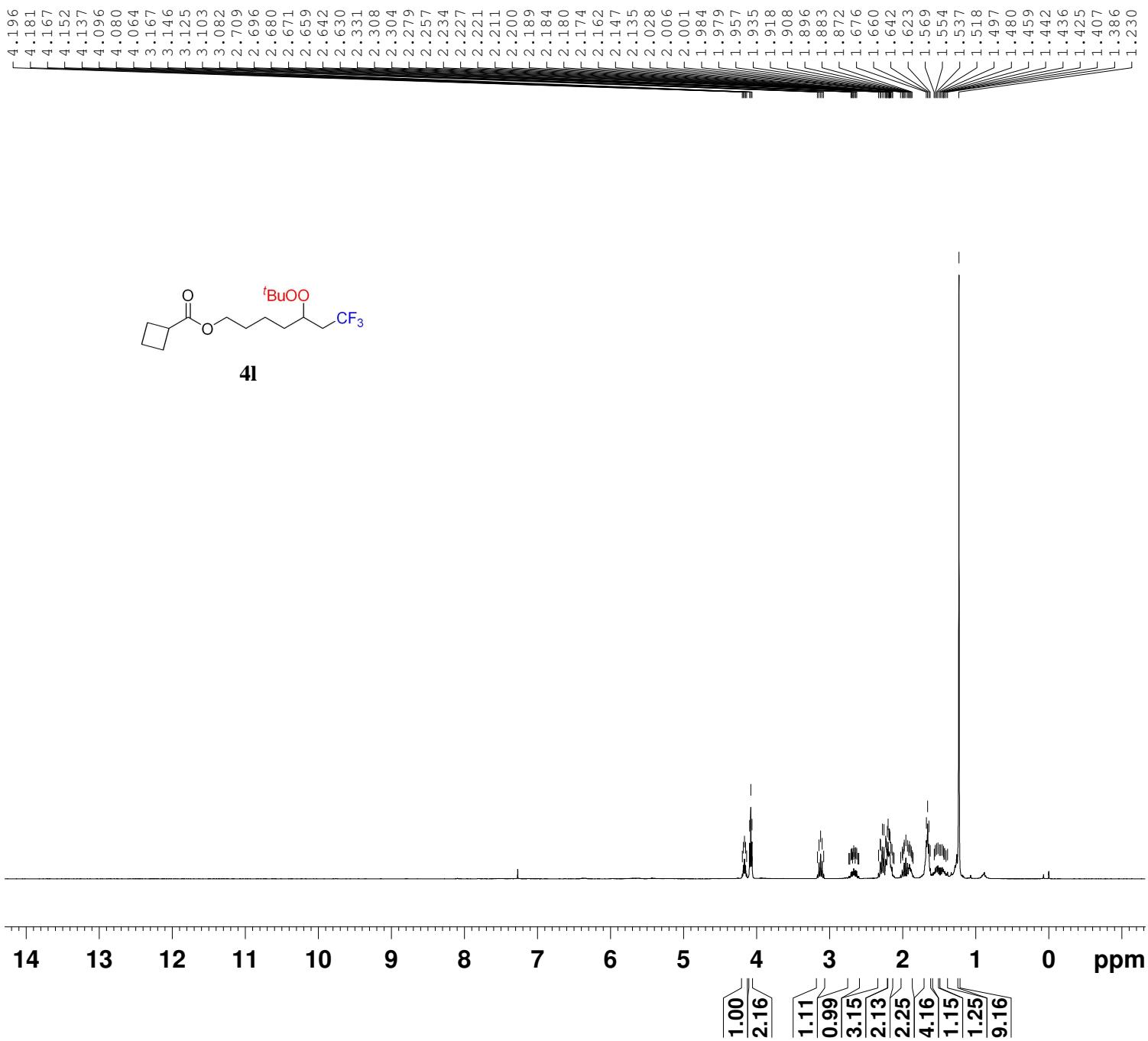


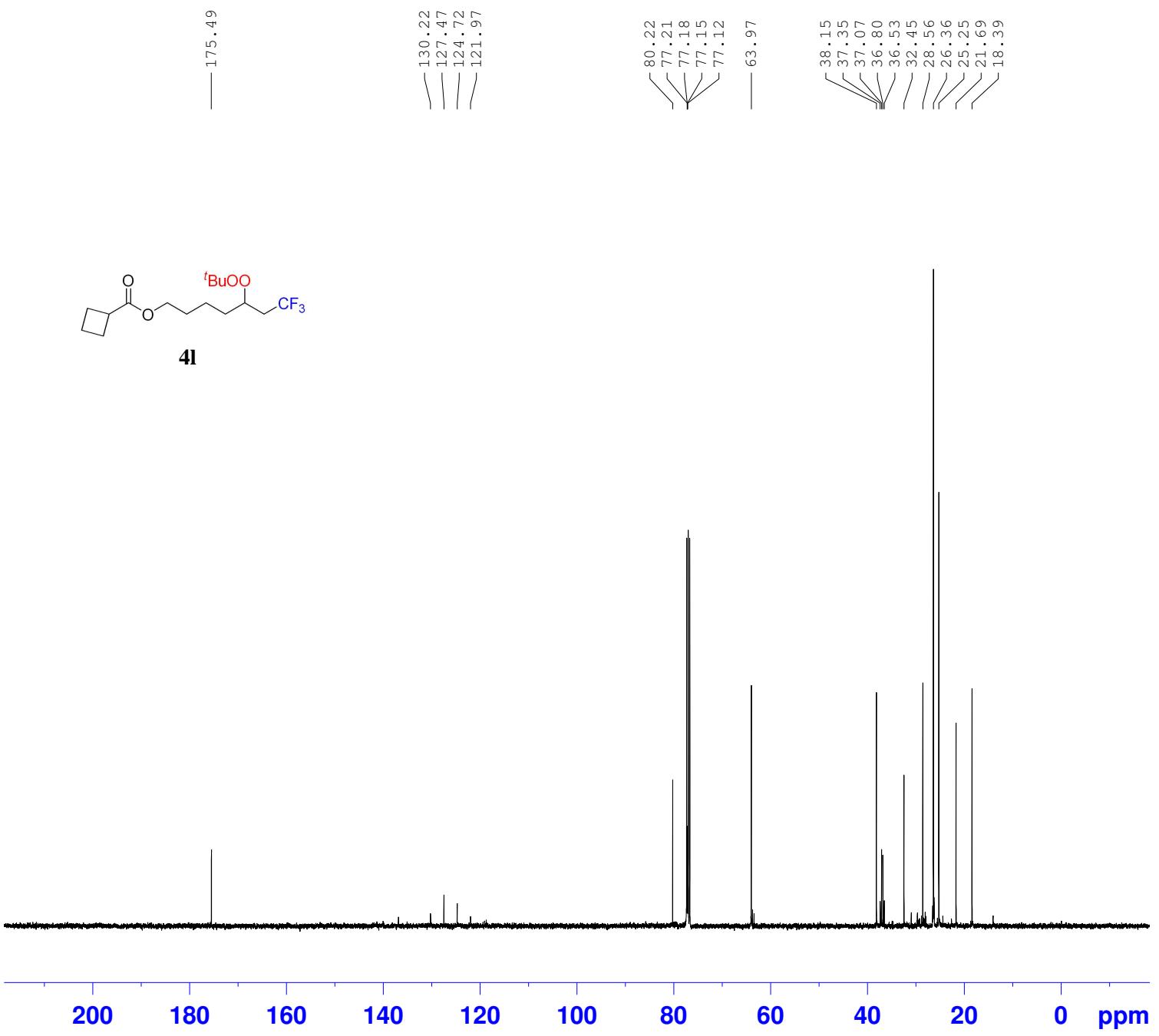


— -63.103

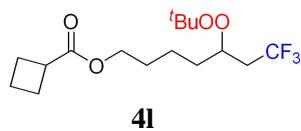


NAME	LV-HQW-762P-20240721
EXPNO	11
PROCNO	1
Date_	20240721
Time	23.19 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.1 K
D1	1.0000000 sec
D11	0.0300000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

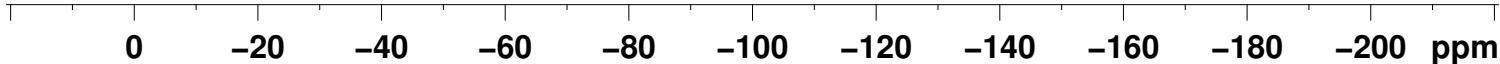




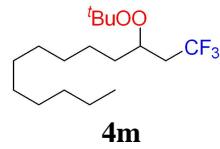
NAME	LV-HQW-699P-20240624
EXPNO	11
PROCNO	1
Date_	20240625
Time	0.25 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl ₃
NS	400
DS	4
SWH	23809.523 Hz
FIDRES	0.726609 Hz
AQ	1.3763061 sec
RG	101
DW	21.000 usec
DE	6.50 usec
TE	303.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TDO	1
SFO1	100.6228298 MHz
NUC1	¹³ C
P0	2.67 usec
P1	8.00 usec
SI	32768
SF	100.6127685 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



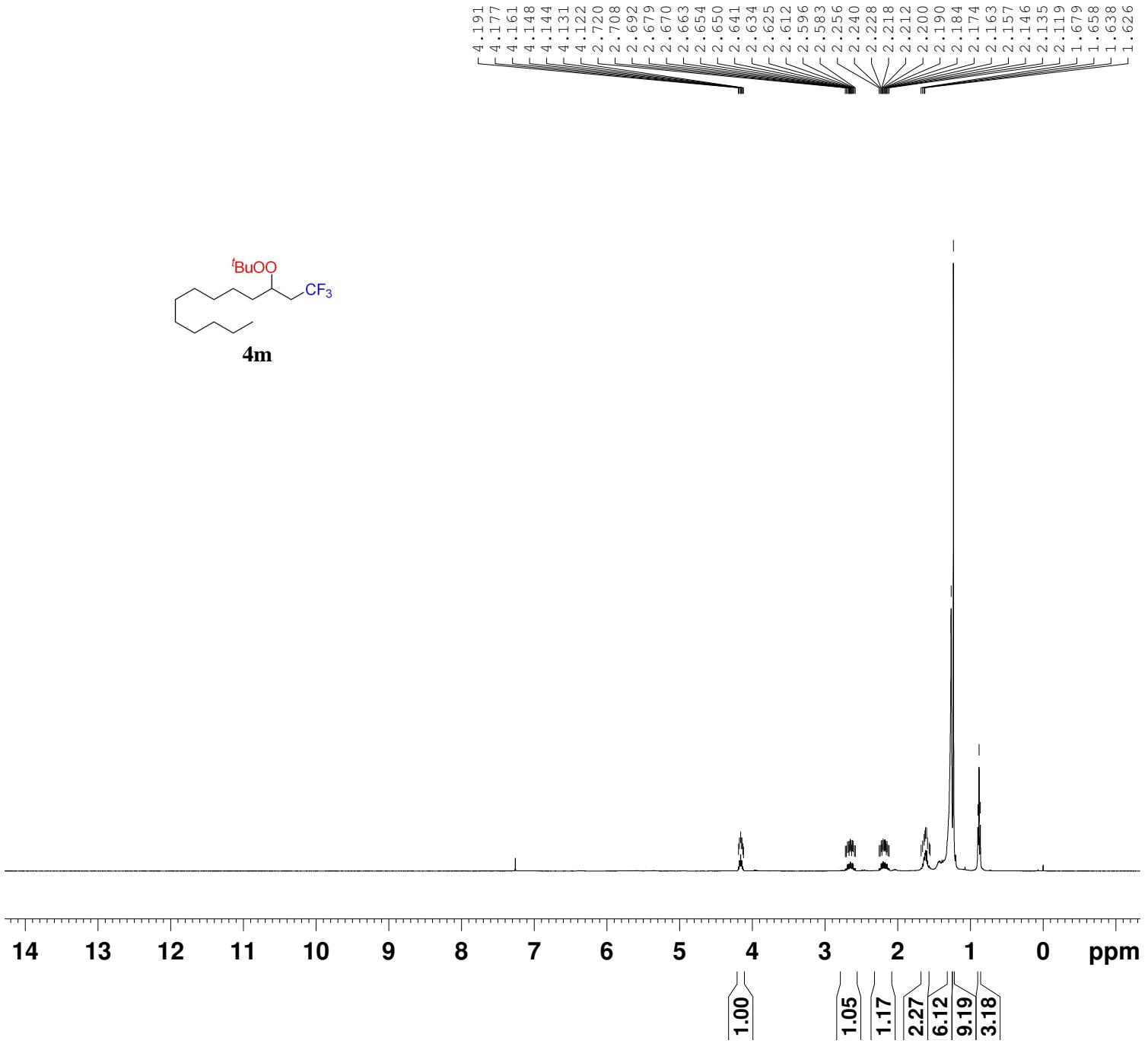
— -63.162

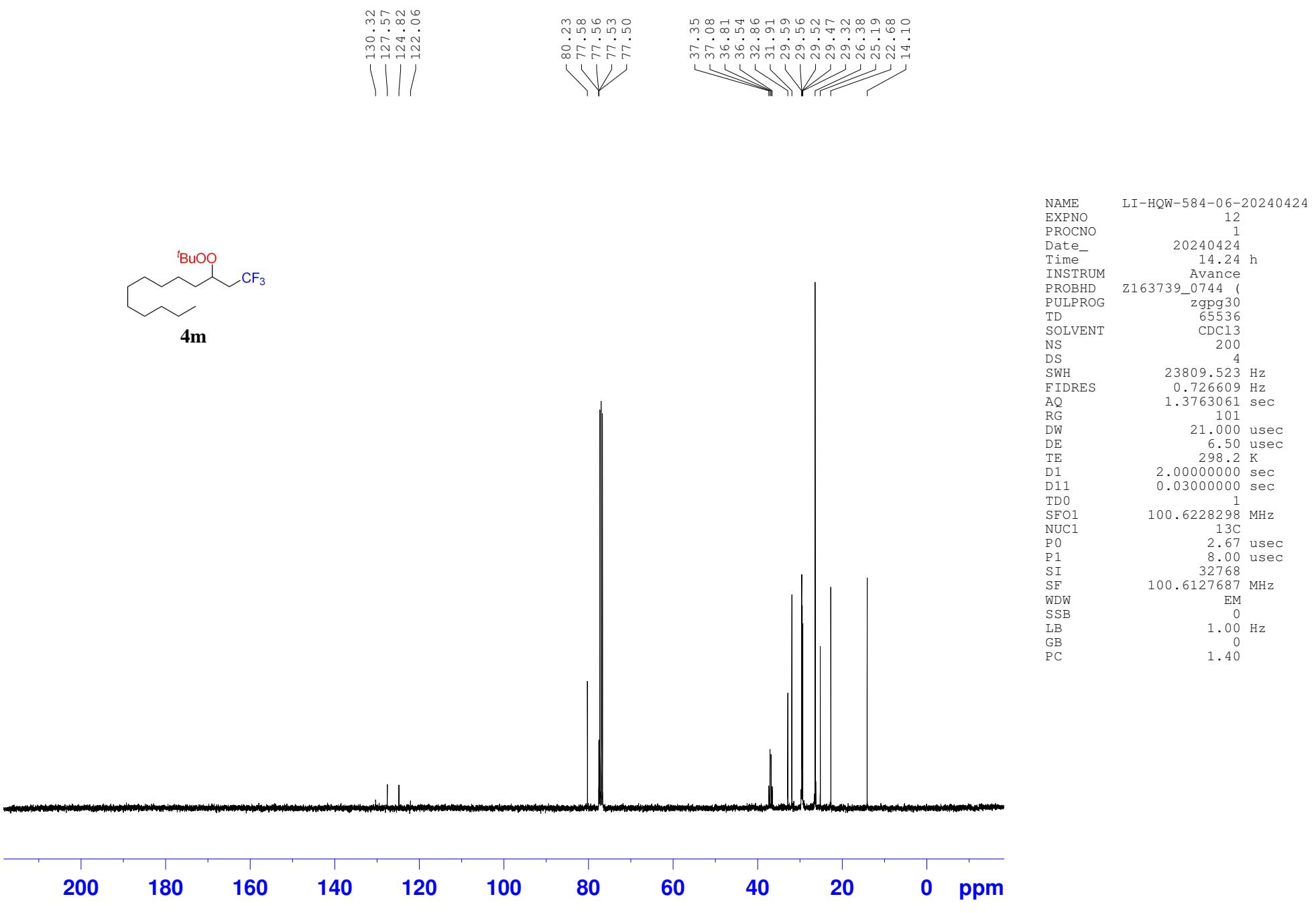


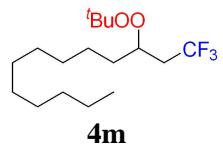
NAME	LV-HQW-699P-20240624
EXPNO	12
PROCNO	1
Date_	20240625
Time	0.27 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	303.3 K
D1	1.0000000 sec
D11	0.0300000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



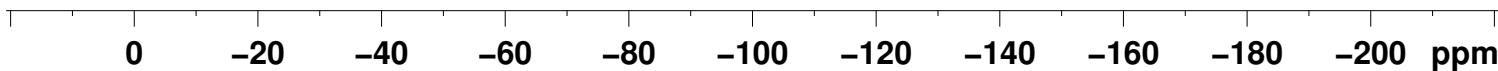
NAME	LI-HQW-584-06-20240424
EXPNO	10
PROCNO	1
Date_	20240424
Time	14.09 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zg30
TD	65536
SOLVENT	CDC13
NS	8
DS	0
SWH	6250.000 Hz
FIDRES	0.190735 Hz
AQ	5.2429299 sec
RG	45.2
DW	80.000 usec
DE	8.64 usec
TE	298.0 K
D1	1.0000000 sec
TD0	1
SFO1	400.1326008 MHz
NUC1	1H
P0	2.67 usec
P1	8.00 usec
SI	65536
SF	400.1300104 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



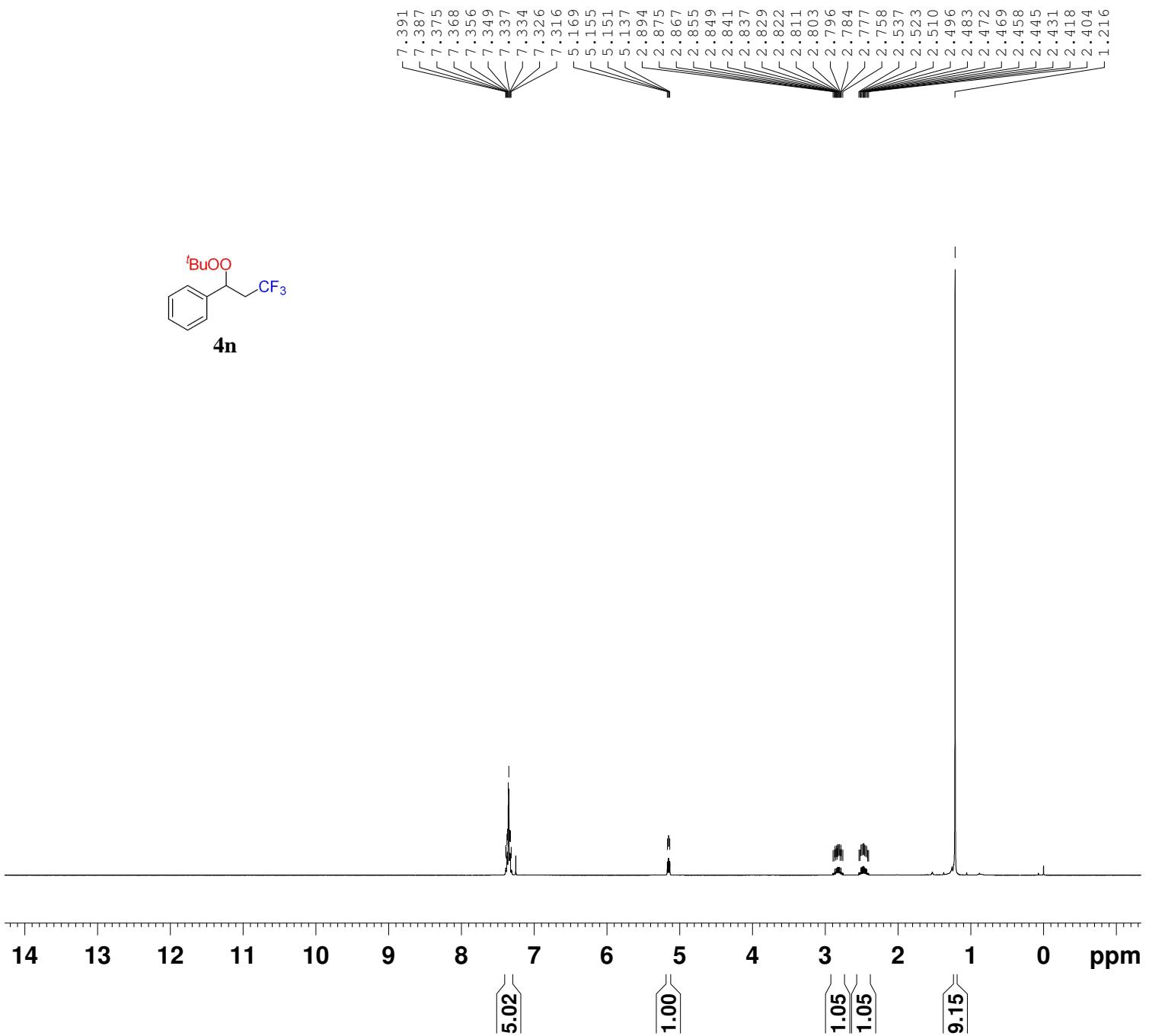
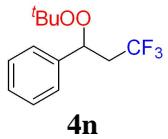




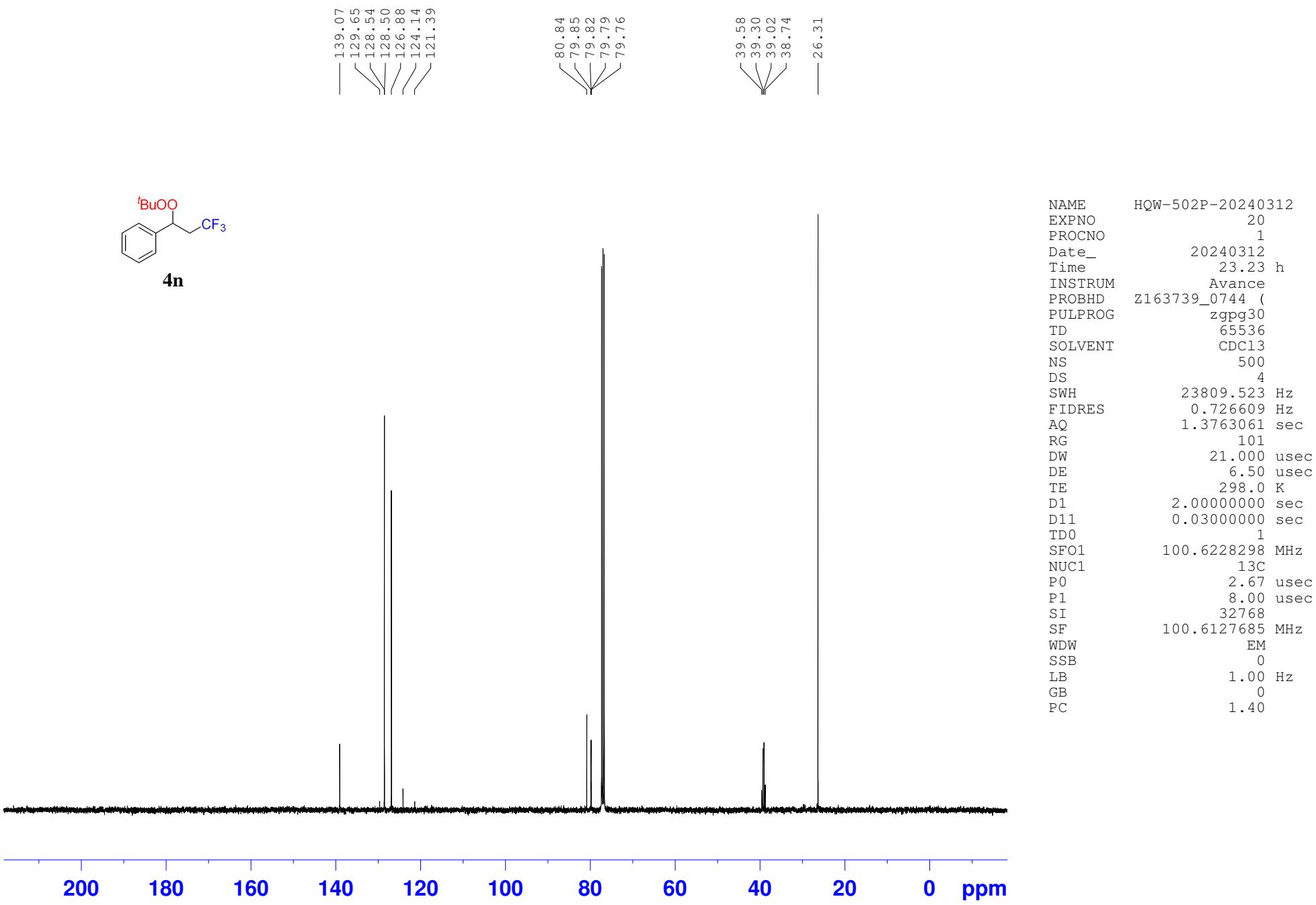
— -63.110

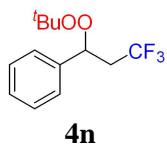


NAME	LI-HQW-584-06-20240424
EXPNO	11
PROCNO	1
Date_	20240424
Time	14.11 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.1 K
D1	1.0000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	¹⁹ F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

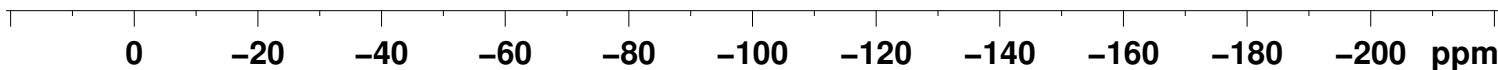


NAME	HQW-502P-20240312
EXPNO	10
PROCNO	1
Date_	20240312
Time	19.22 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zg30
TD	65536
SOLVENT	CDC13
NS	8
DS	0
SWH	6250.000 Hz
FIDRES	0.190735 Hz
AQ	5.2429299 sec
RG	101
DW	80.000 usec
DE	8.64 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
SFO1	400.1326008 MHz
NUC1	1H
P0	2.67 usec
P1	8.00 usec
SI	65536
SF	400.1300131 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

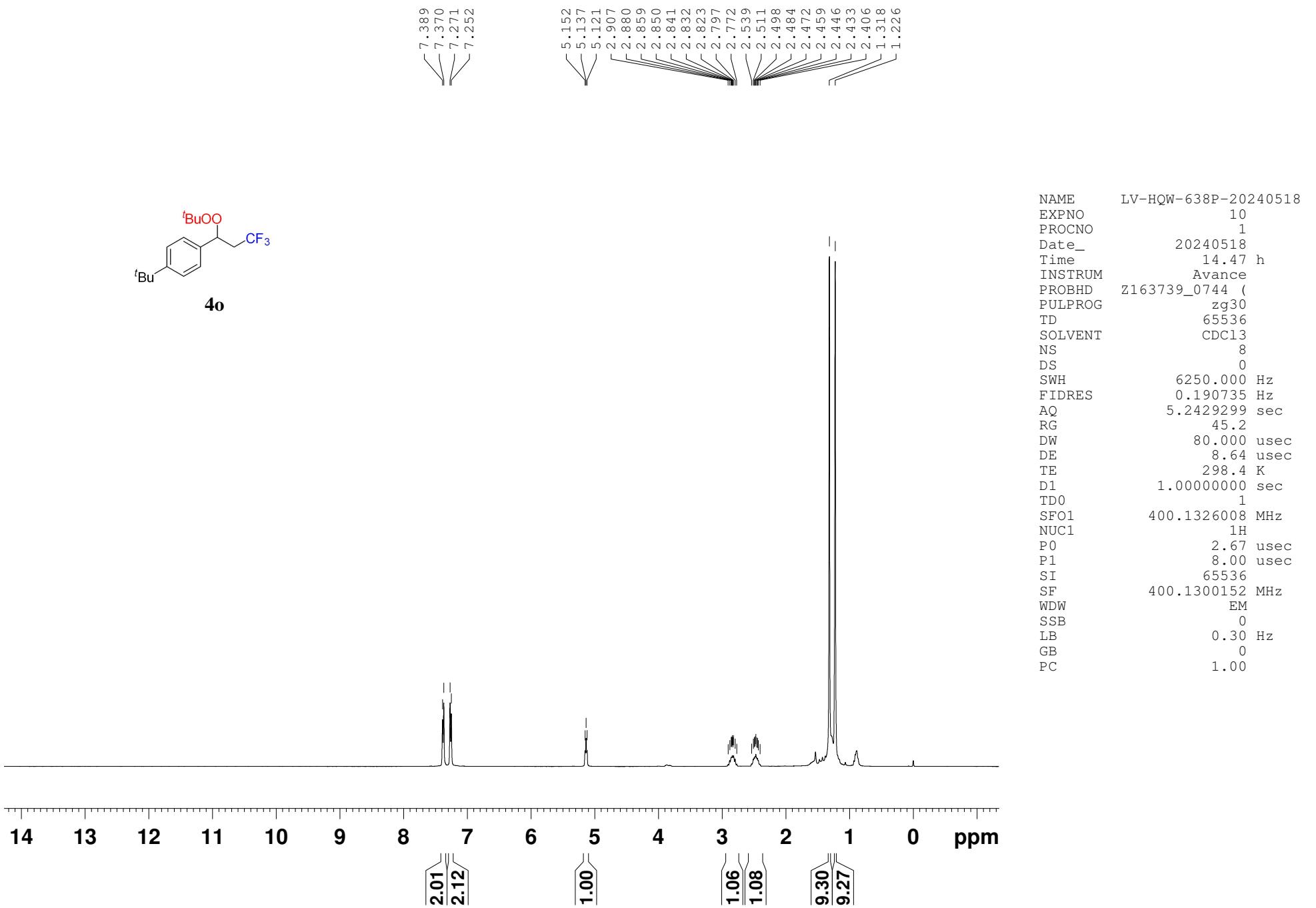


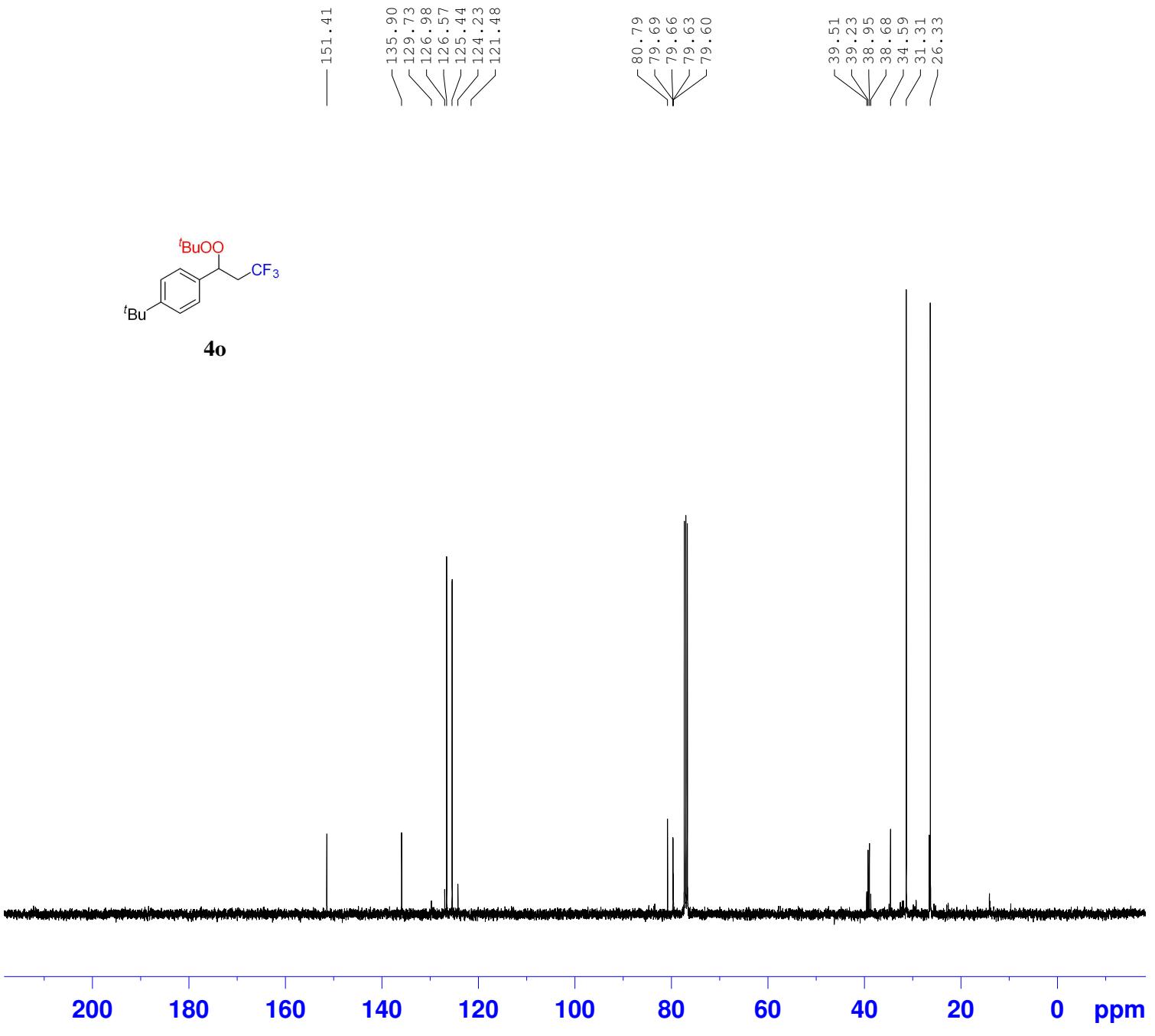


—63.279

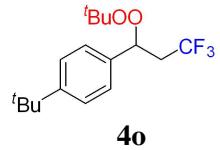


NAME	HQW-502P-20240312
EXPNO	12
PROCNO	1
Date_	20240312
Time	19.37 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDC13
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.1 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	¹⁹ F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





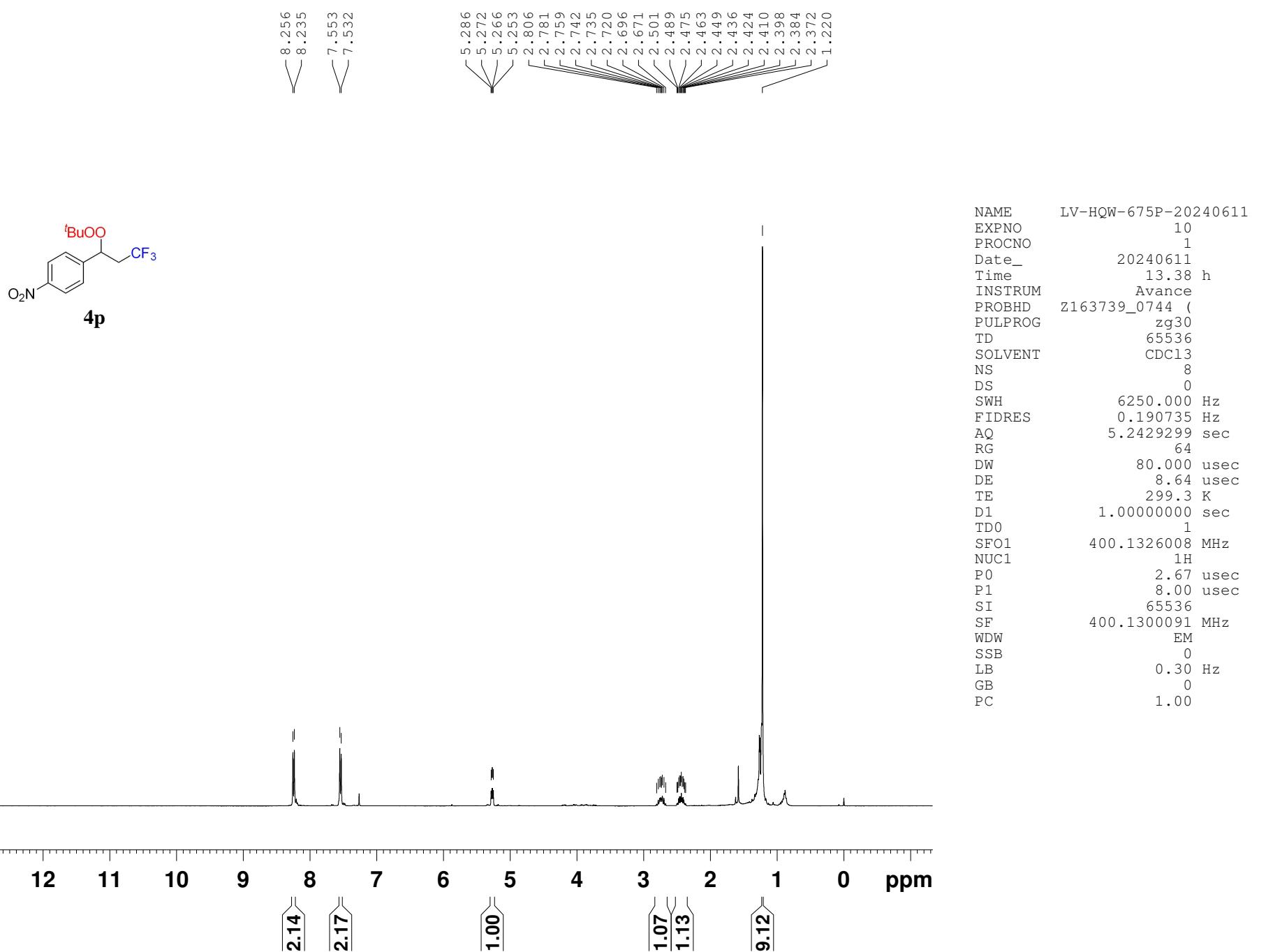
NAME LV-HQW-638P-20240518
 EXPNO 12
 PROCNO 1
 Date_ 20240518
 Time 15.00 h
 INSTRUM Avance
 PROBHD Z163739_0744 (zgpg30
 PULPROG 65536
 TD CDC13
 SOLVENT 160
 NS 4
 DS 23809.523 Hz
 SWH 0.726609 Hz
 FIDRES 1.3763061 sec
 AQ 101
 RG 21.000 usec
 DW 6.50 usec
 DE 299.7 K
 TE 2.00000000 sec
 D1 0.03000000 sec
 D11 1
 TDO 100.6228298 MHz
 SFO1 13C
 NUC1 2.67 usec
 P0 8.00 usec
 P1 32768
 SI 100.6127695 MHz
 SF EM
 WDW 0
 SSB 1.00 Hz
 LB 0
 GB 1.40
 PC

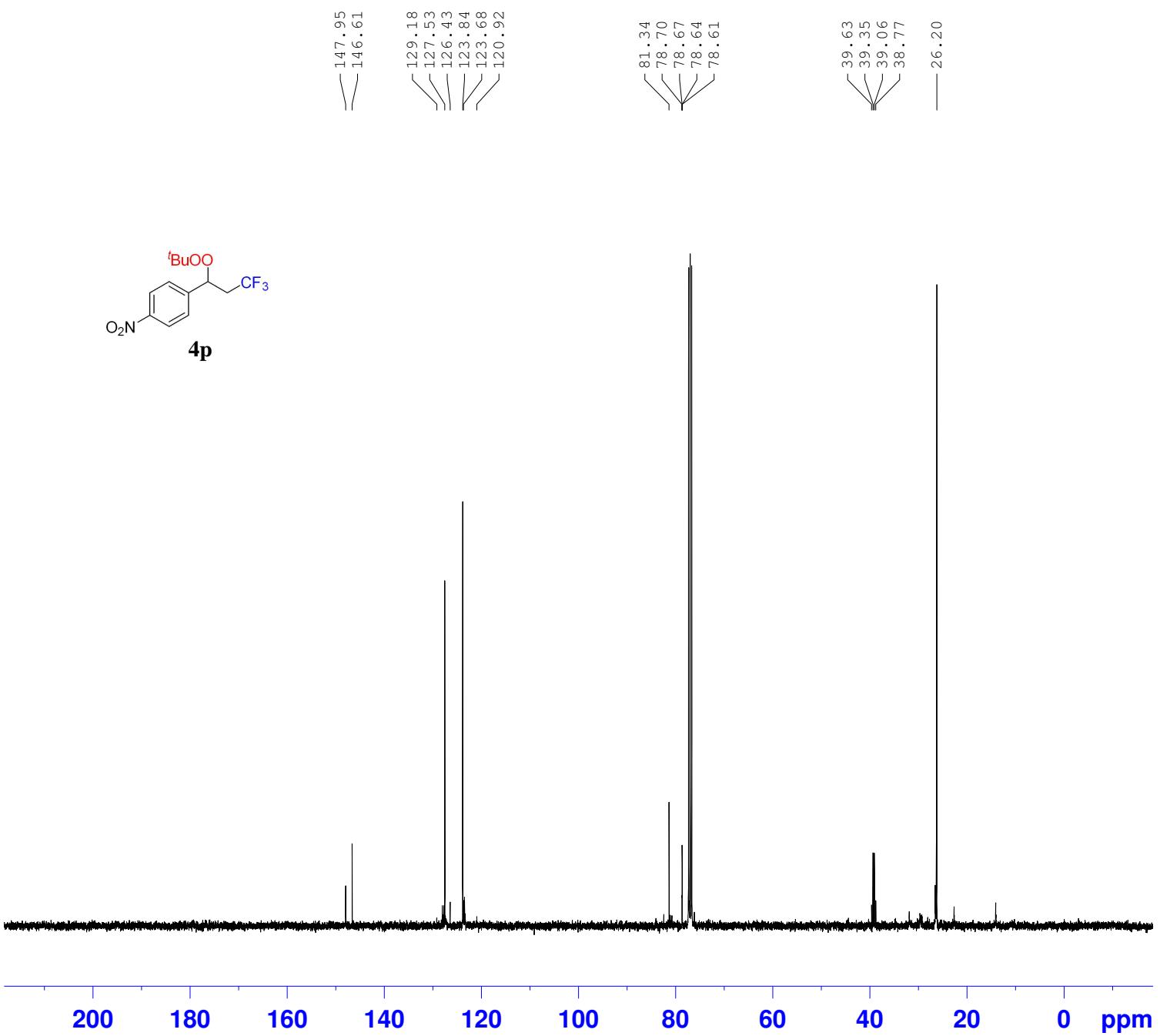


— -63.260

0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 ppm

NAME	LV-HQW-638P-20240518
EXPNO	11
PROCNO	1
Date_	20240518
Time	14.49 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	299.1 K
D1	1.0000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	¹⁹ F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

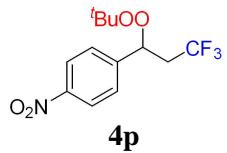




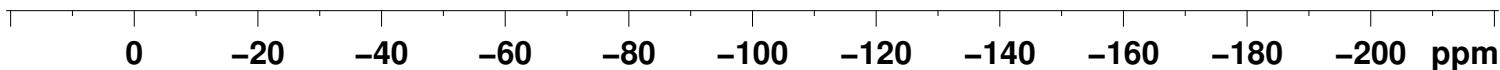
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NAME      LV-HQW-675P-20240611
EXPNO    12
PROCNO   1
Date_   20240611
Time   14.05 h
INSTRUM Avance
PROBHD Z163739_0744 (
PULPROG zgpg30
TD      65536
SOLVENT CDC13
NS      416
DS      4
SWH     23809.523 Hz
FIDRES  0.726609 Hz
AQ      1.3763061 sec
RG      101
DW      21.000 usec
DE      6.50 usec
TE      300.6 K
D1      2.0000000 sec
D11     0.03000000 sec
TD0      1
SFO1    100.6228298 MHz
NUC1    13C
P0      2.67 usec
P1      8.00 usec
SI      32768
SF      100.6127702 MHz
WDW
SSB
LB      1.00 Hz
GB
PC      1.40

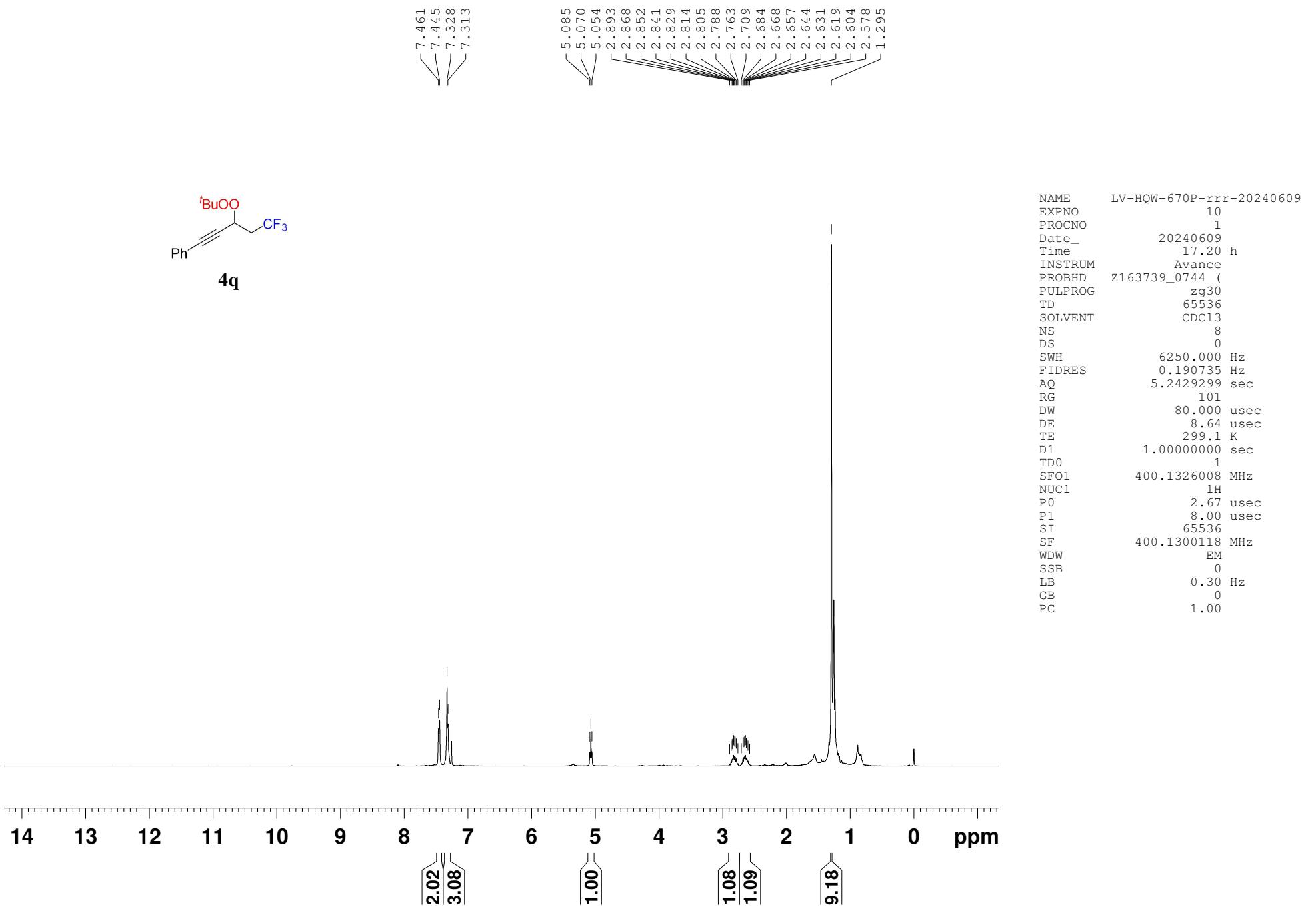
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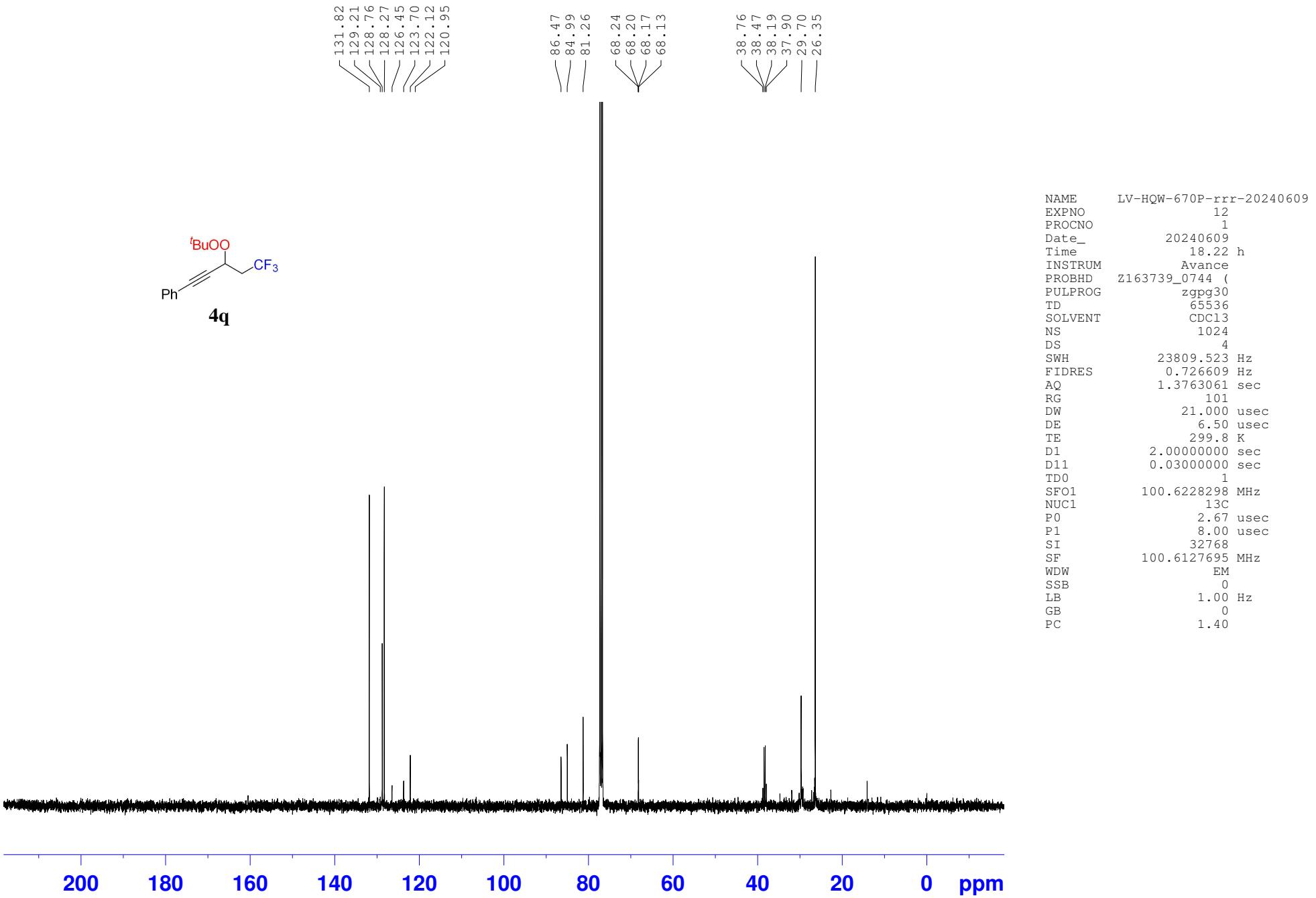


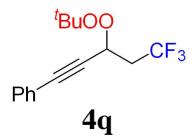
— -63.316



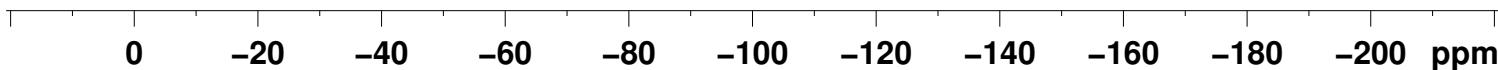
NAME	LV-HQW-675P-20240611
EXPNO	11
PROCNO	1
Date_	20240611
Time	13.40 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl3
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	299.6 K
D1	1.0000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



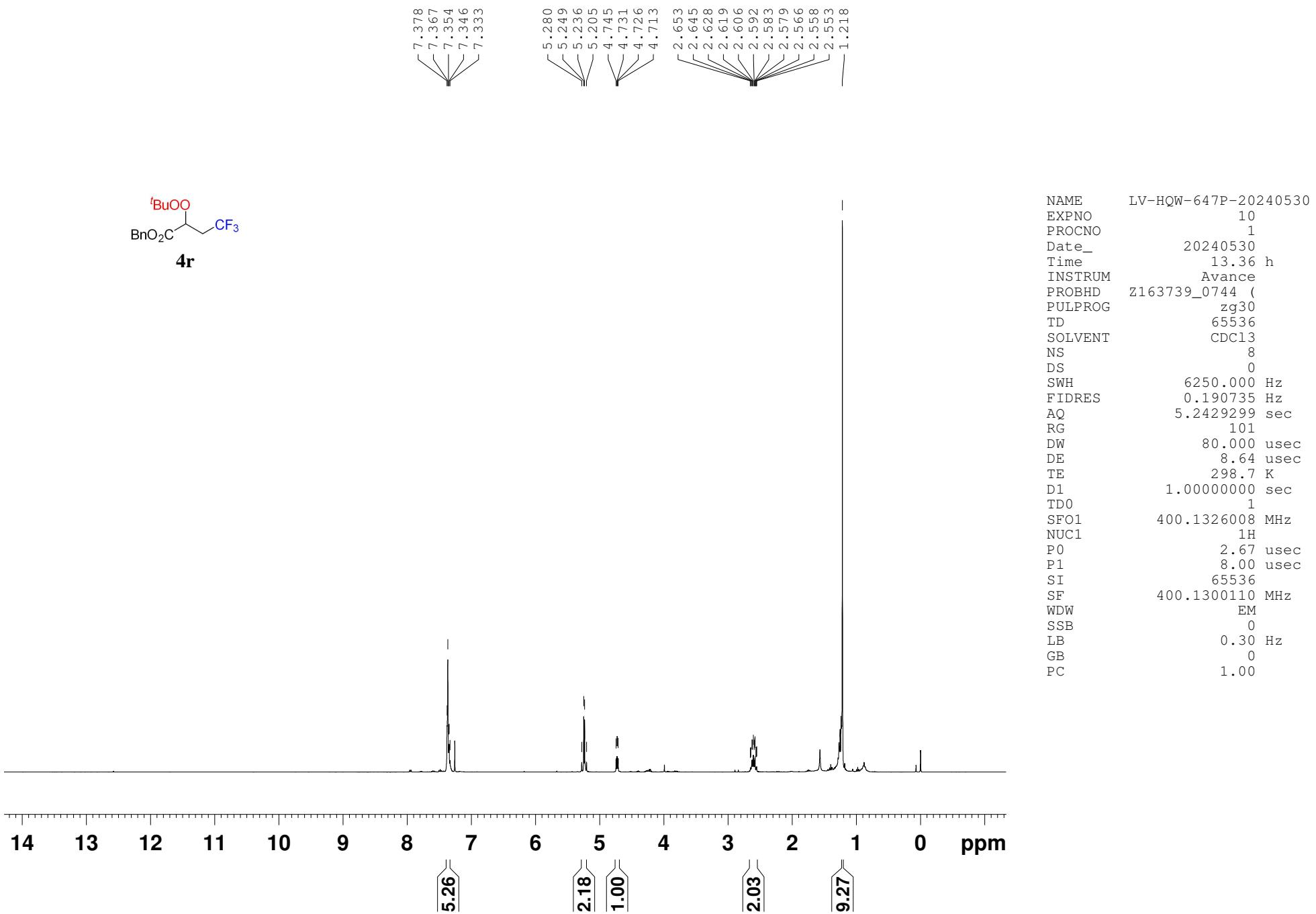


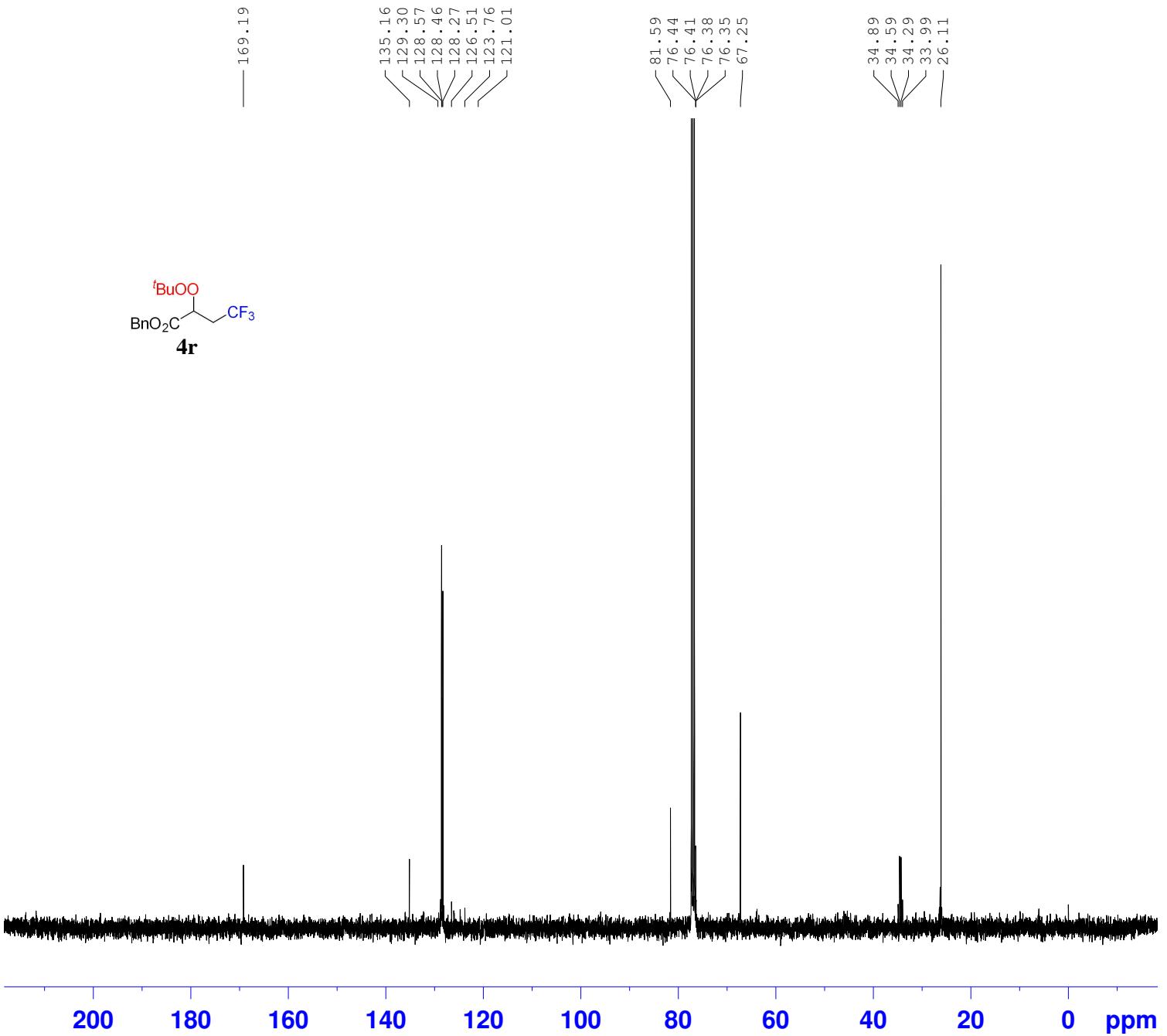


— -63.630 —



NAME	LV-HQW-670P-rrr-20240609
EXPNO	11
PROCNO	1
Date_	20240609
Time	17.21 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDC13
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	299.5 K
D1	1.0000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	¹⁹ F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

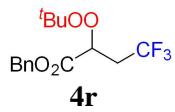




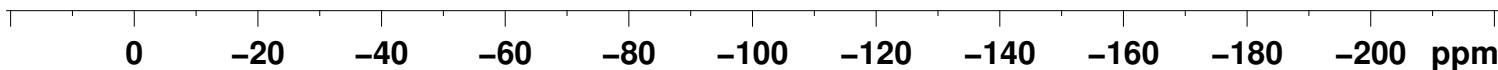
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NAME      LV-HQW-647P-20240530
EXPNO    12
PROCNO   1
Date_   20240530
Time    13.57 h
INSTRUM Avance
PROBHD Z163739_0744 (
PULPROG zgpg30
TD        65536
SOLVENT   CDCl3
NS         312
DS          4
SWH       23809.523 Hz
FIDRES   0.726609 Hz
AQ        1.3763061 sec
RG          101
DW        21.000 usec
DE          6.50 usec
TE        299.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0         1
SFO1     100.6228298 MHz
NUC1      13C
P0          2.67 usec
P1          8.00 usec
SI         32768
SF        100.6127709 MHz
WDW           EM
SSB            0
LB          1.00 Hz
GB            0
PC          1.40

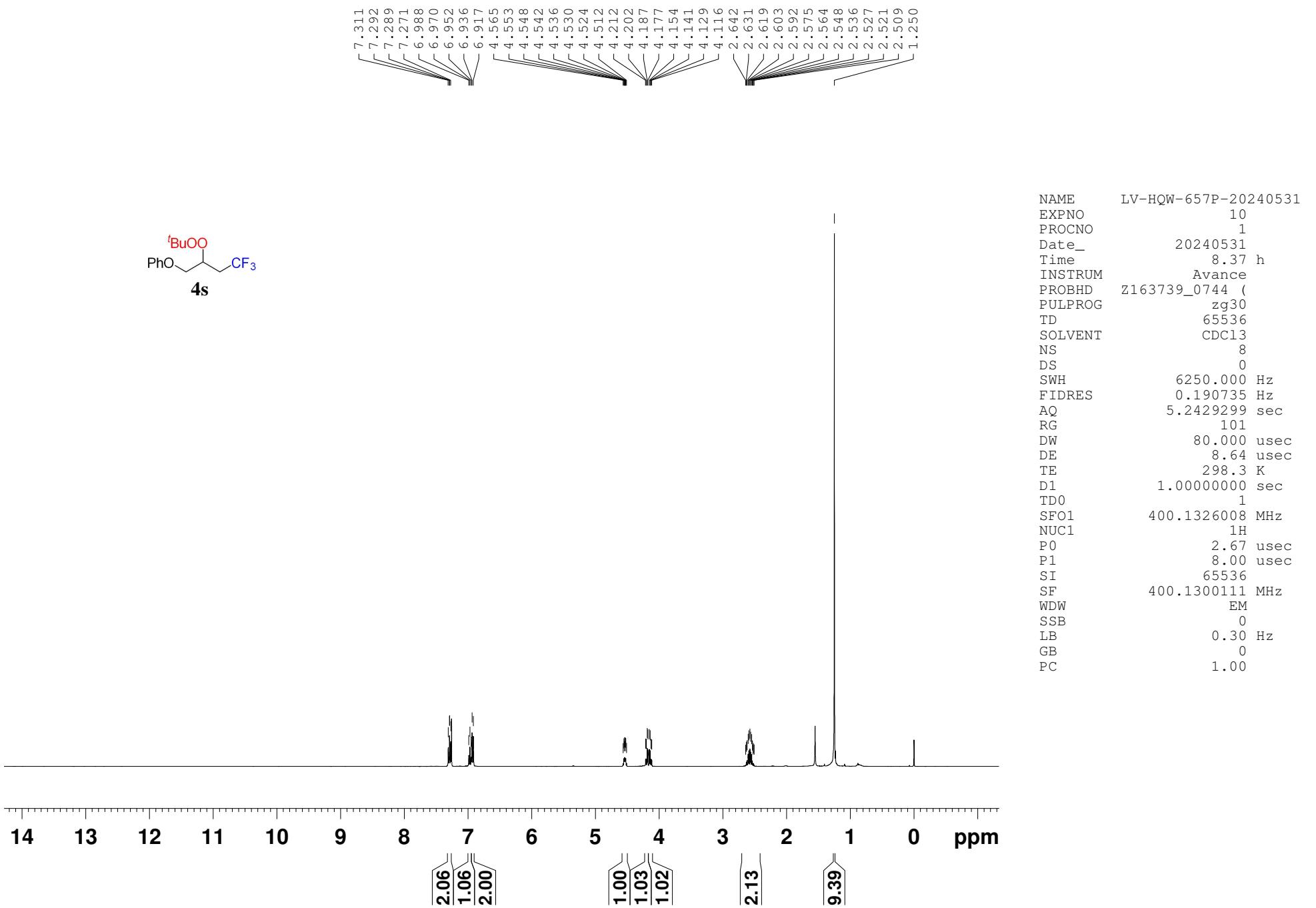
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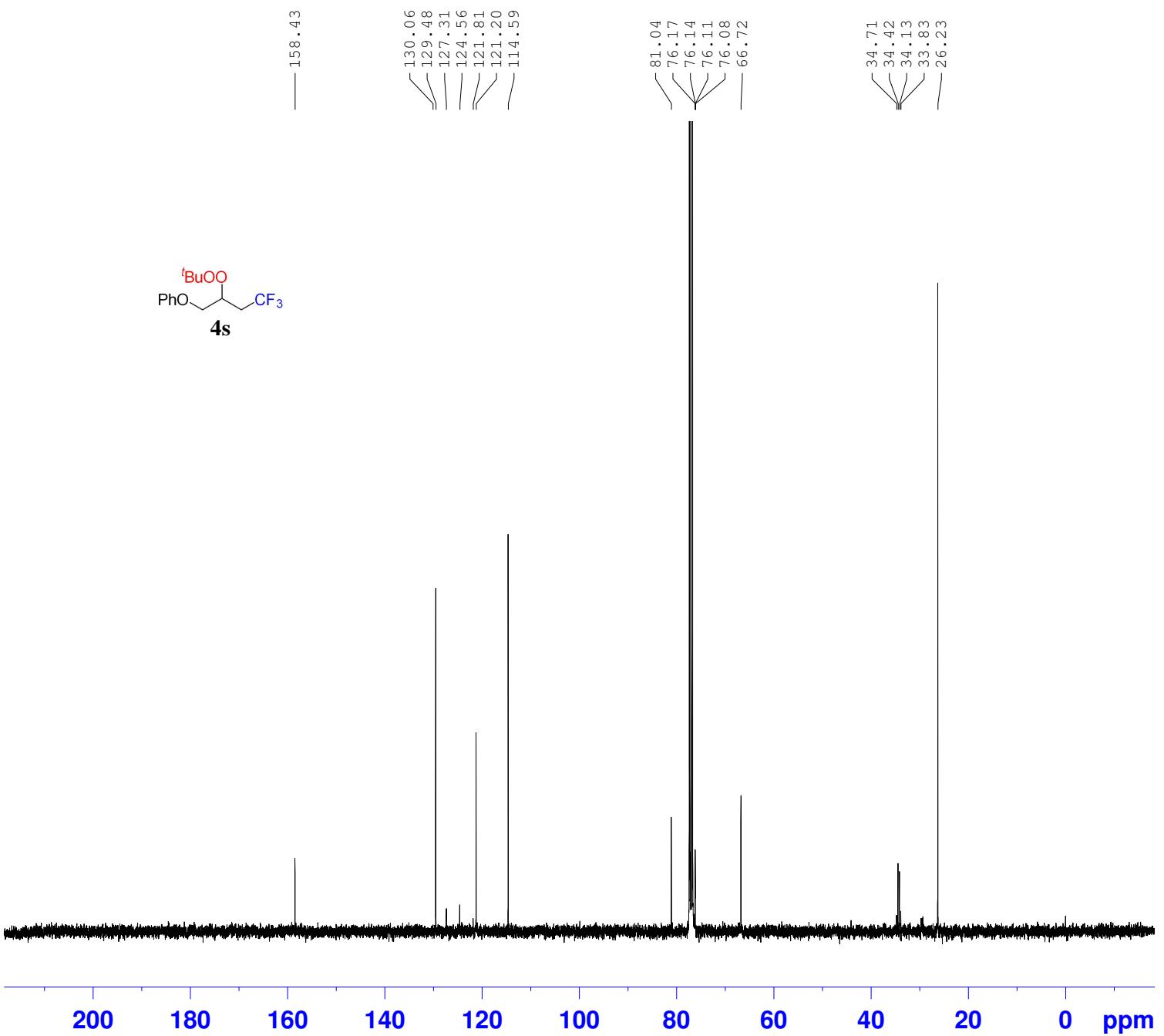


— -63.870

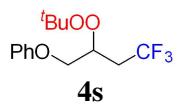


NAME	LV-HQW-647P-20240530
EXPNO	11
PROCNO	1
Date_	20240530
Time	13.38 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl3
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.9 K
D1	1.0000000 sec
D11	0.0300000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

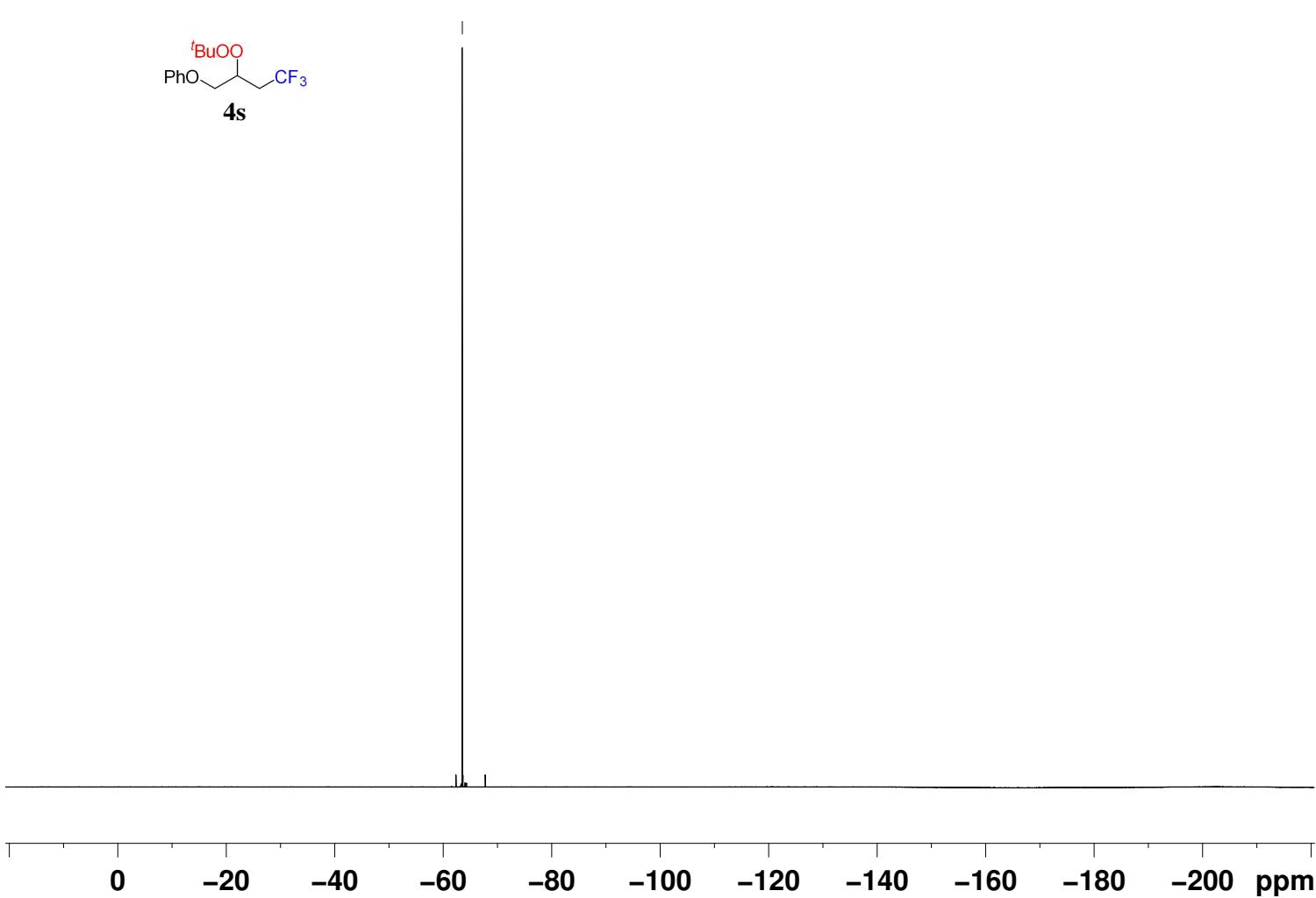




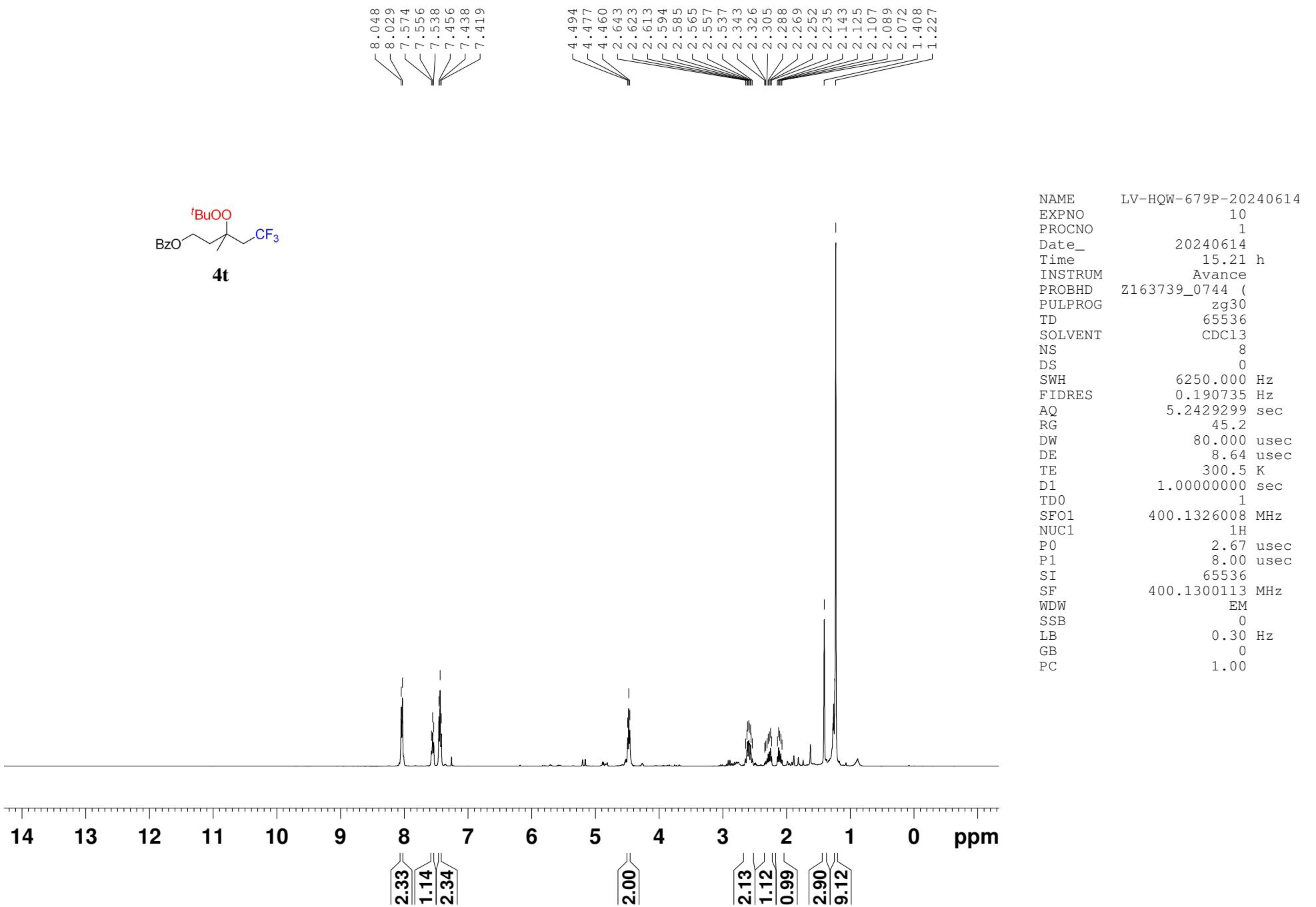
NAME LV-HQW-657P-20240607
 EXPNO 11
 PROCN0 1
 Date_ 20240607
 Time 18.13 h
 INSTRUM Avance
 PROBHD Z163739_0744 (zgpg30
 PULPROG 65536
 TD 512
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 23809.523 Hz
 FIDRES 0.726609 Hz
 AQ 1.3763061 sec
 RG 101
 DW 21.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1
 SFO1 100.6228298 MHz
 NUC1 13C
 P0 2.67 usec
 P1 8.00 usec
 SI 32768
 SF 100.6127702 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

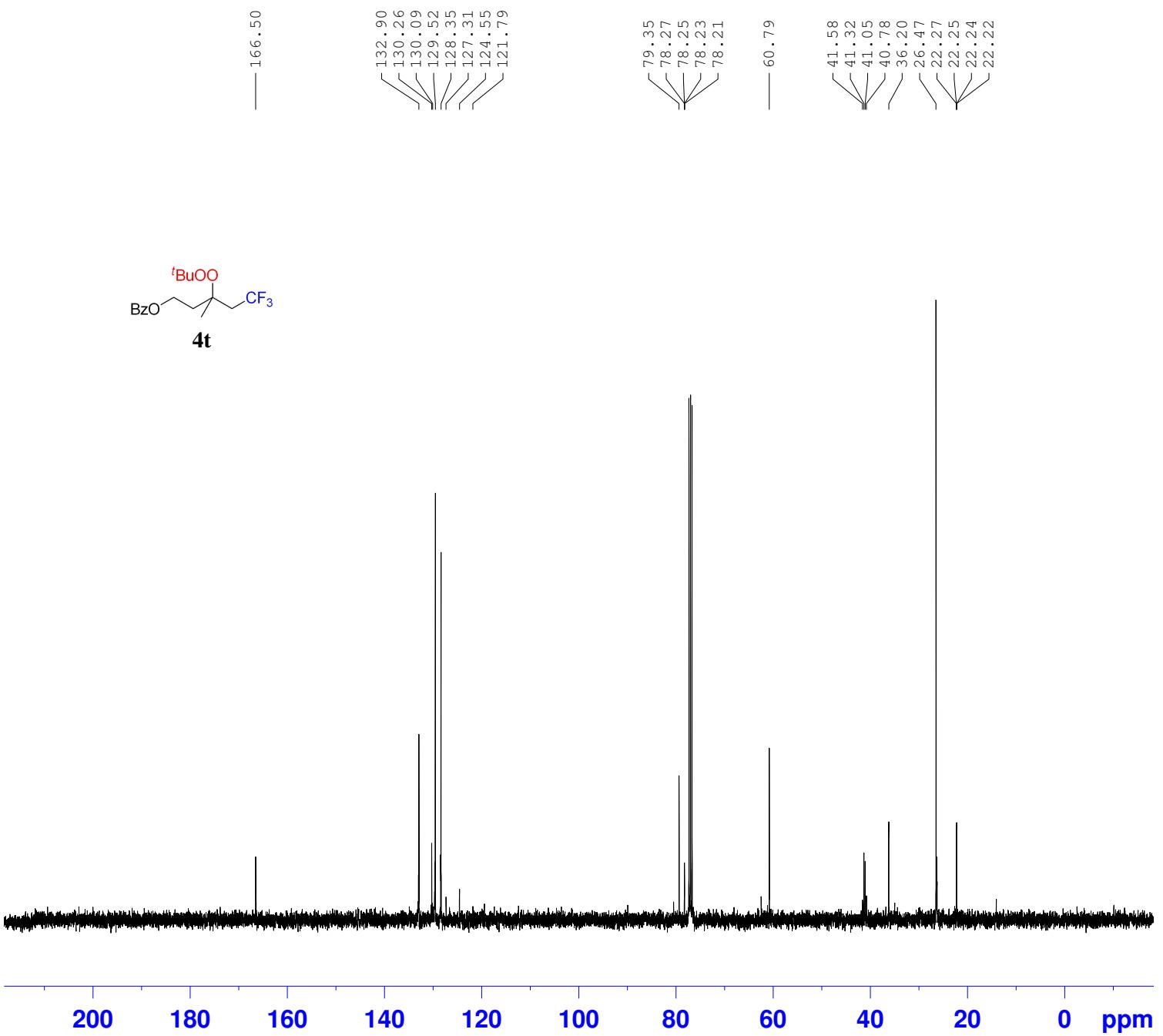


— — — — —63.519

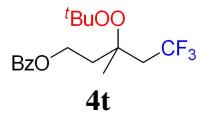


NAME	LV-HQW-657P-20240607
EXPNO	10
PROCNO	1
Date_	20240607
Time	17.13 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	299.2 K
D1	1.0000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

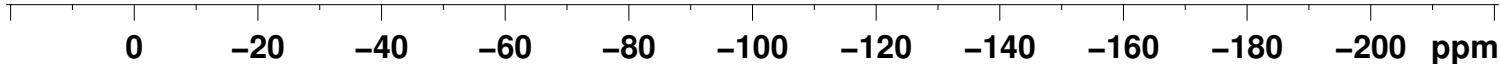




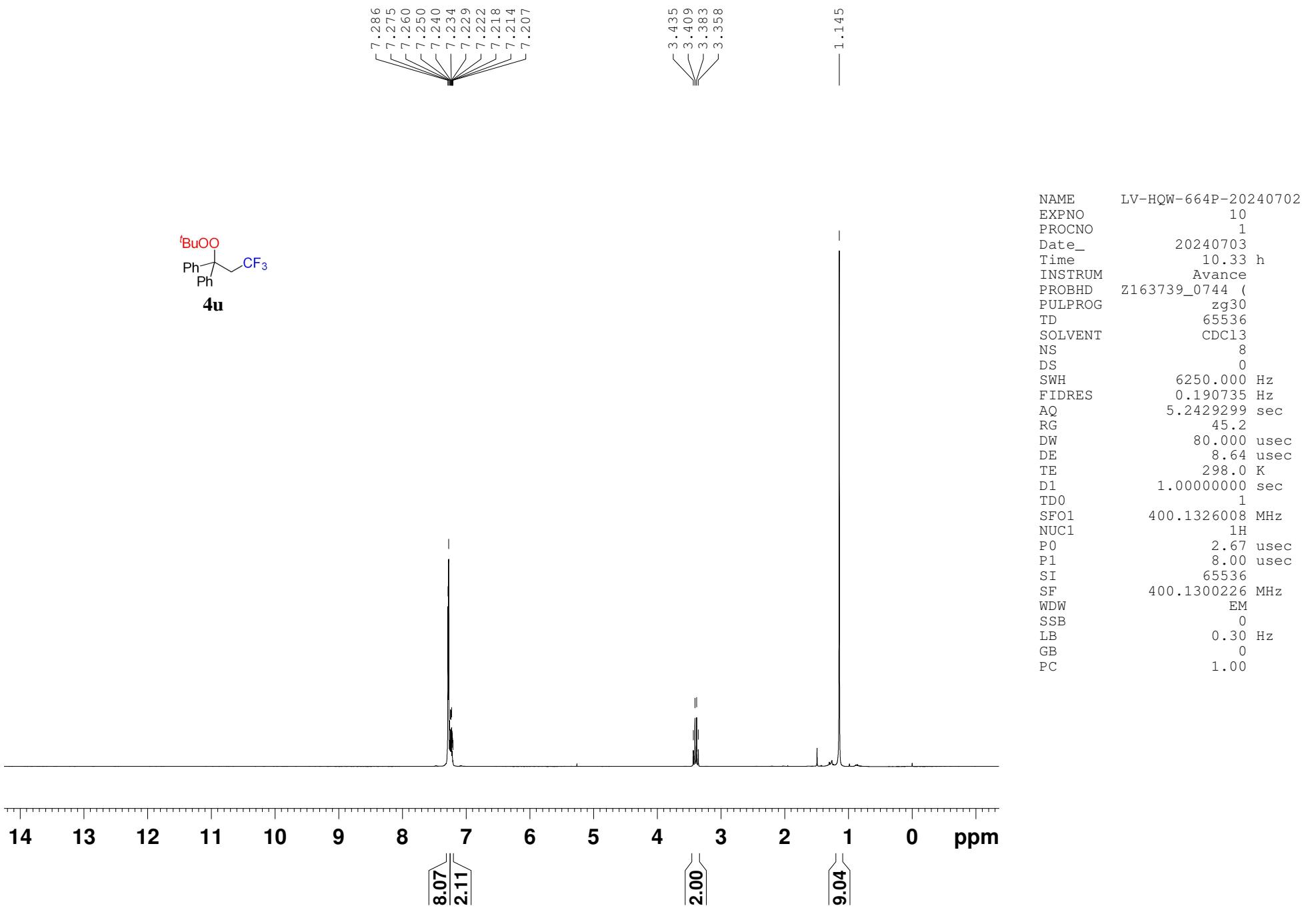
NAME LV-HQW-679P-20240614
 EXPNO 12
 PROCN0 1
 Date_ 20240614
 Time 15.28 h
 INSTRUM Avance
 PROBHD Z163739_0744 (
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 56
 DS 4
 SWH 23809.523 Hz
 FIDRES 0.726609 Hz
 AQ 1.3763061 sec
 RG 101
 DW 21.000 usec
 DE 6.50 usec
 TE 301.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1
 SFO1 100.6228298 MHz
 NUC1 13C
 P0 2.67 usec
 P1 8.00 usec
 SI 32768
 SF 100.6127709 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

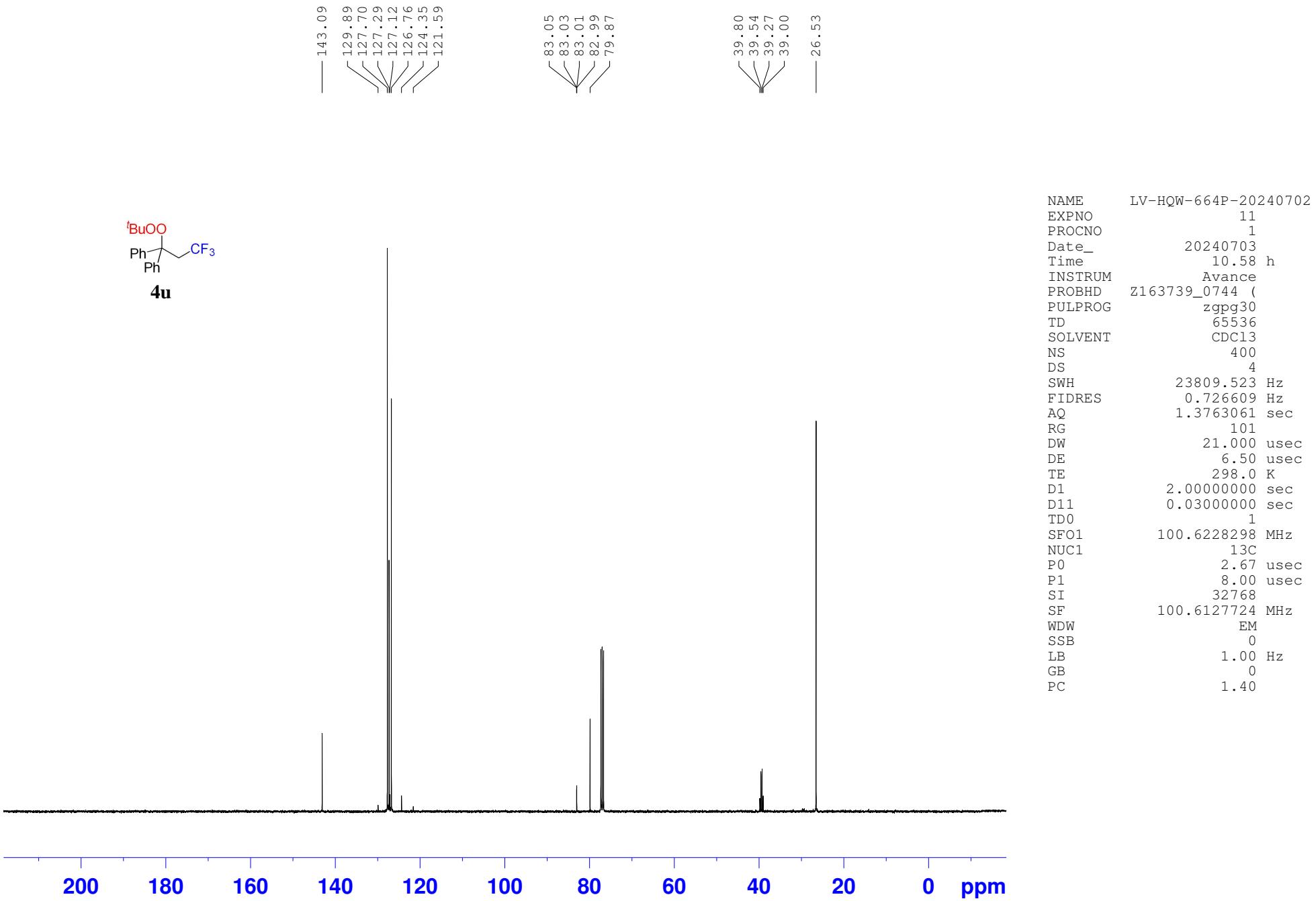


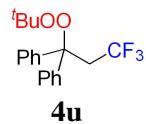
-59.926



NAME	LV-HQW-679P-20240614
EXPNO	11
PROCNO	1
Date_	20240614
Time	15.23 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	300.7 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



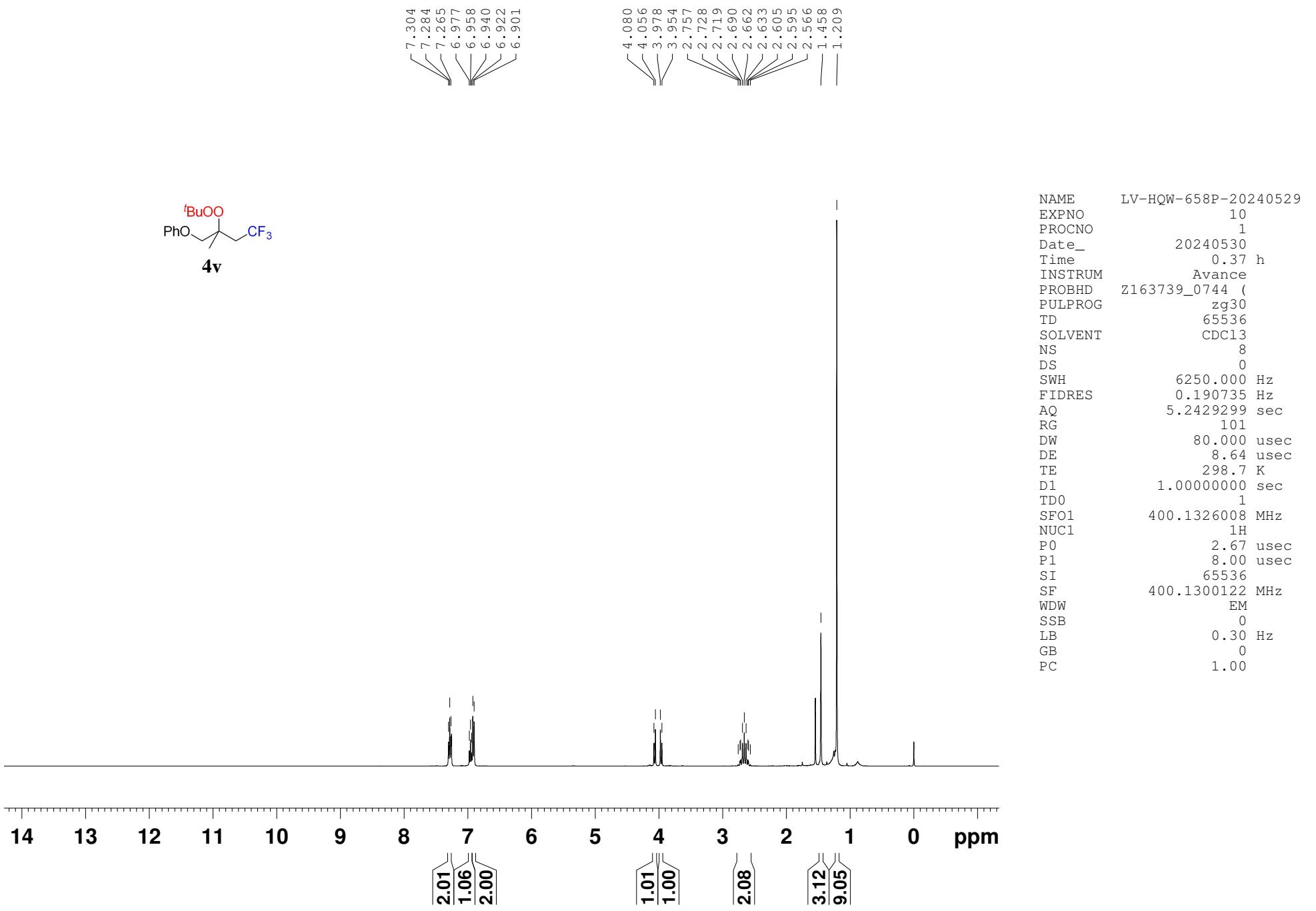


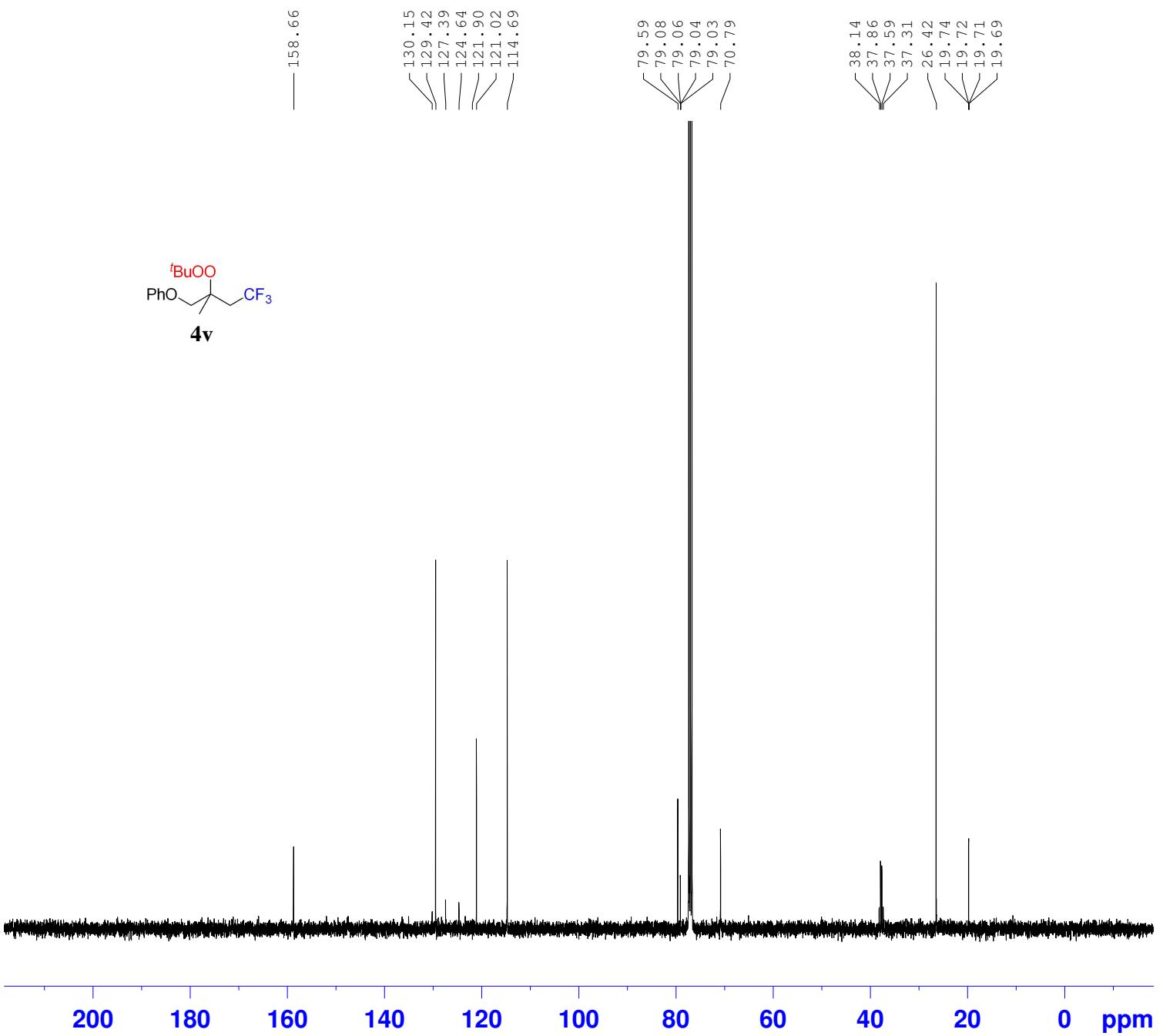


-58.891

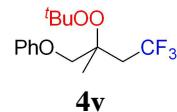


NAME	LV-HQW-664P-20240702
EXPNO	12
PROCNO	1
Date_	20240703
Time	11.00 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.1 K
D1	1.0000000 sec
D11	0.0300000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



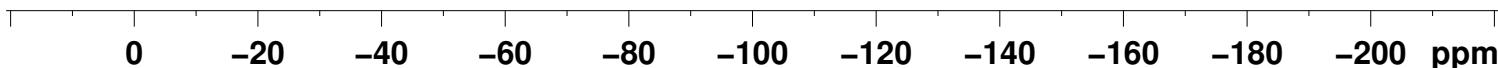


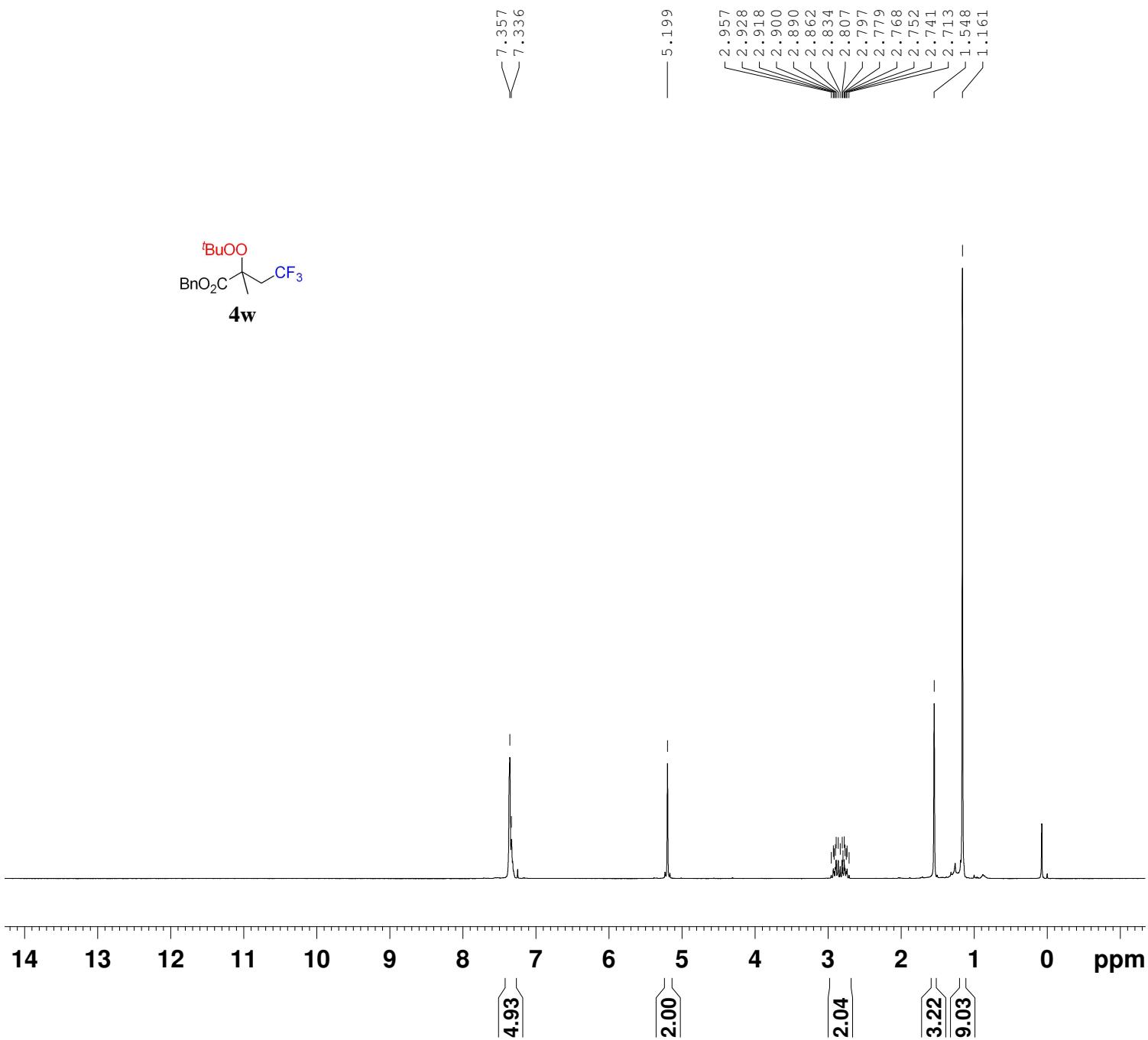
NAME	LV-HQW-658P-20240529
EXPNO	12
PROCNO	1
Date_	20240530
Time	1.09 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl ₃
NS	512
DS	4
SWH	23809.523 Hz
FIDRES	0.726609 Hz
AQ	1.3763061 sec
RG	101
DW	21.000 usec
DE	6.50 usec
TE	299.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TDO	1
SFO1	100.6228298 MHz
NUC1	¹³ C
P0	2.67 usec
P1	8.00 usec
SI	32768
SF	100.6127702 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



-59.909

NAME	LV-HQW-658P-20240529
EXPNO	11
PROCNO	1
Date_	20240530
Time	0.38 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl3
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.9 K
D1	1.0000000 sec
D11	0.0300000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





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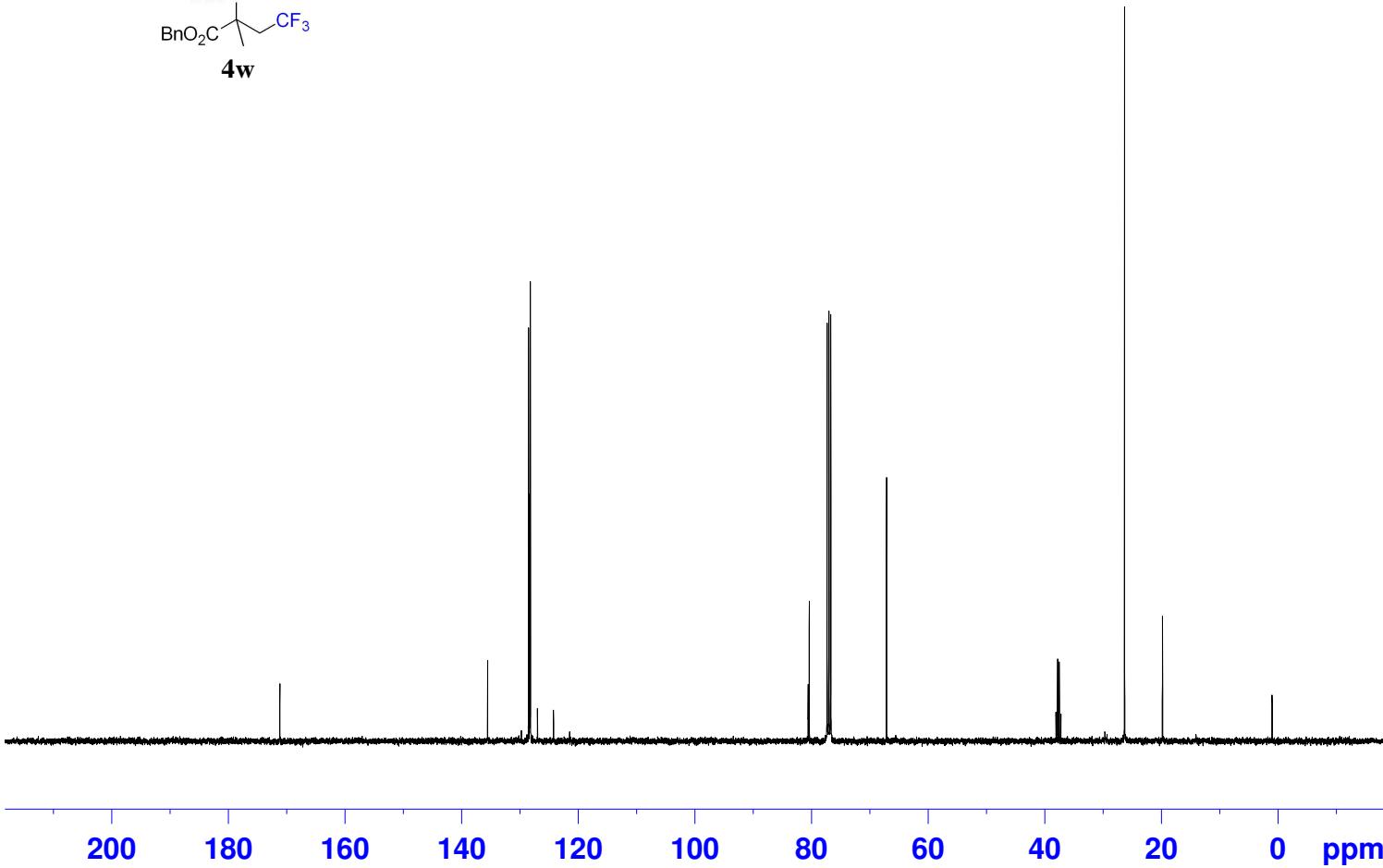
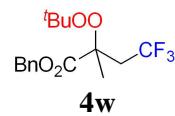
NAME      LV-HQW-4w-20240627
EXPNO        10
PROCNO       1
Date_   20240627
Time    17.35 h
INSTRUM Avance
PROBHD Z163739_0744 (
PULPROG zg30
TD        65536
SOLVENT   CDCl3
NS           8
DS           0
SWH       6250.000 Hz
FIDRES     0.190735 Hz
AQ        5.2429299 sec
RG          57
DW        80.000 usec
DE         8.64 usec
TE        305.9 K
D1        1.0000000 sec
TD0         1
SFO1      400.1326008 MHz
NUC1        1H
P0          2.67 usec
P1          8.00 usec
SI          65536
SF        400.1300146 MHz
WDW         EM
SSB          0
LB        0.30 Hz
GB          0
PC        1.00
  
```

— 171.16

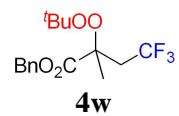
135.53
129.75
128.49
128.29
128.19
126.99
124.23
121.47

80.59
80.57
80.55
80.53
80.37
67.13

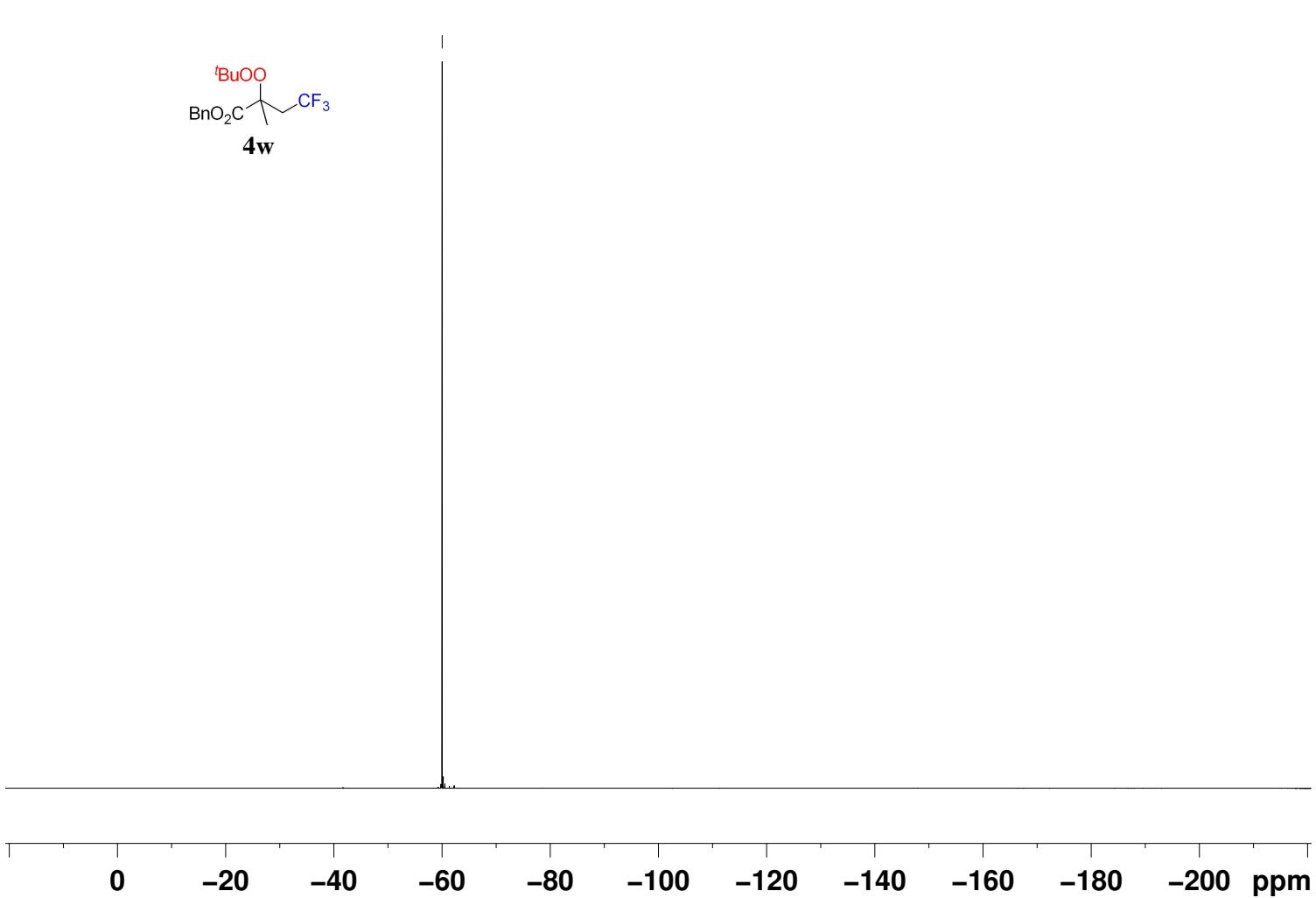
38.06
37.78
37.50
37.22
26.30
19.84
19.82
19.80
19.78



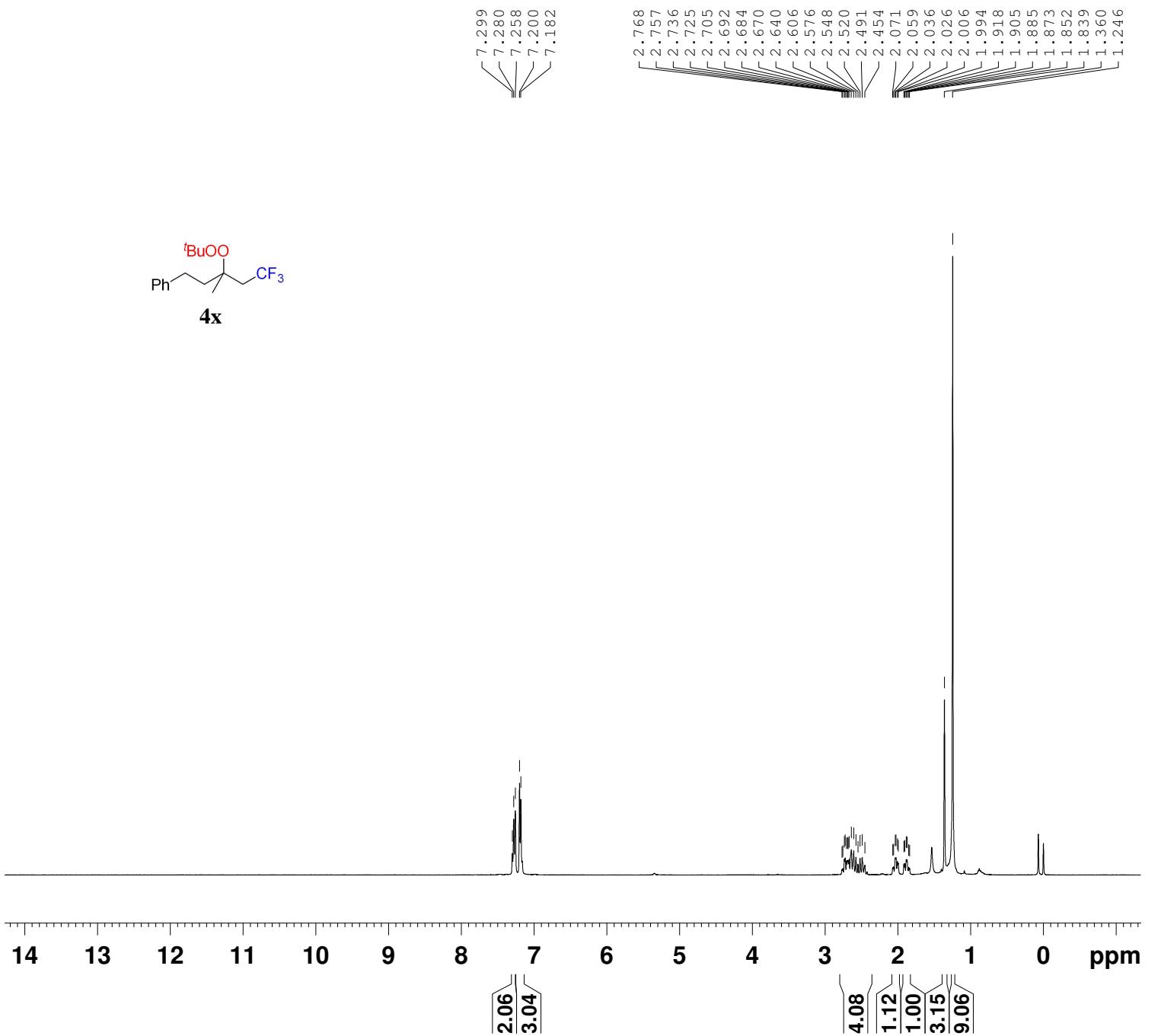
NAME	LV-HQW-4w-20240627
EXPNO	11
PROCNO	1
Date_	20240627
Time	18.00 h
INSTRUM	Avance
PROBHD	z163739_0744 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl ₃
NS	400
DS	4
SWH	23809.523 Hz
FIDRES	0.726609 Hz
AQ	1.3763061 sec
RG	101
DW	21.000 usec
DE	6.50 usec
TE	306.3 K
D1	2.0000000 sec
D11	0.0300000 sec
TD0	1
SFO1	100.6228298 MHz
NUC1	¹³ C
P0	2.67 usec
P1	8.00 usec
SI	32768
SF	100.6127687 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



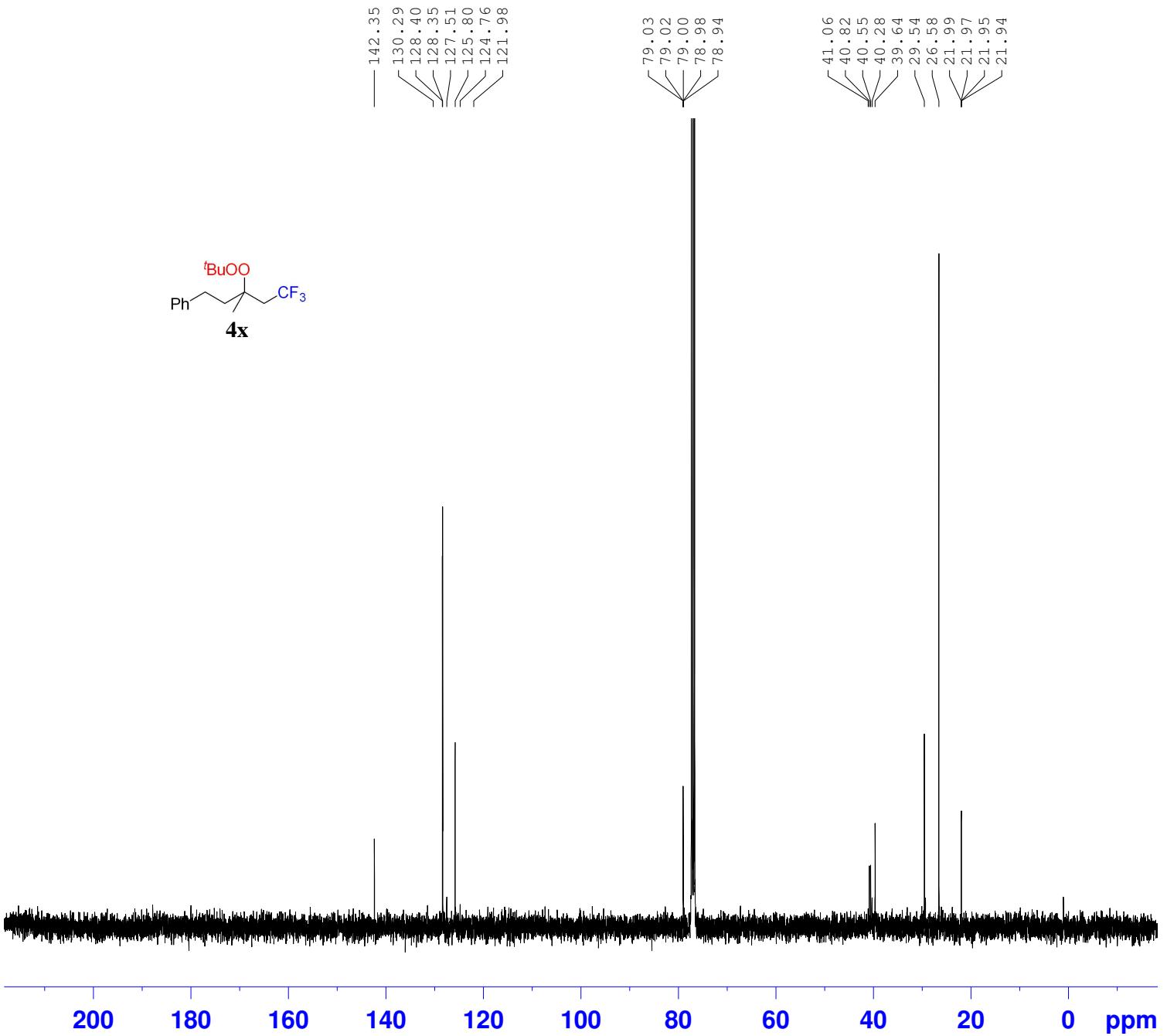
—60.023

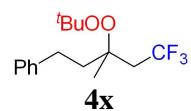


NAME	LV-HQW-4w-20240627
EXPNO	12
PROCNO	1
Date_	20240627
Time	18.02 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDC13
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	305.9 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	¹⁹ F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

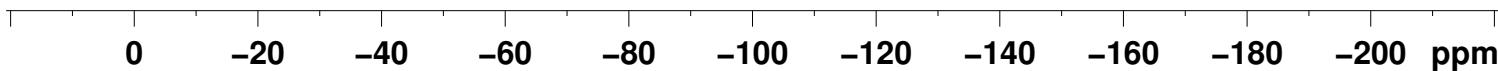


NAME LV-HQW-684P-20240617
 EXPNO 10
 PROCNO 1
 Date_ 20240617
 Time 19.01 h
 INSTRUM Avance
 PROBHD Z163739_0744 (zg30
 PULPROG 65536
 TD 65536
 SOLVENT CDC13
 NS 8
 DS 0
 SWH 6250.000 Hz
 FIDRES 0.190735 Hz
 AQ 5.2429299 sec
 RG 101
 DW 80.000 usec
 DE 8.64 usec
 TE 301.8 K
 D1 1.00000000 sec
 TDO 1
 SFO1 400.1326008 MHz
 NUC1 1H
 P0 2.67 usec
 P1 8.00 usec
 SI 65536
 SF 400.1300132 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

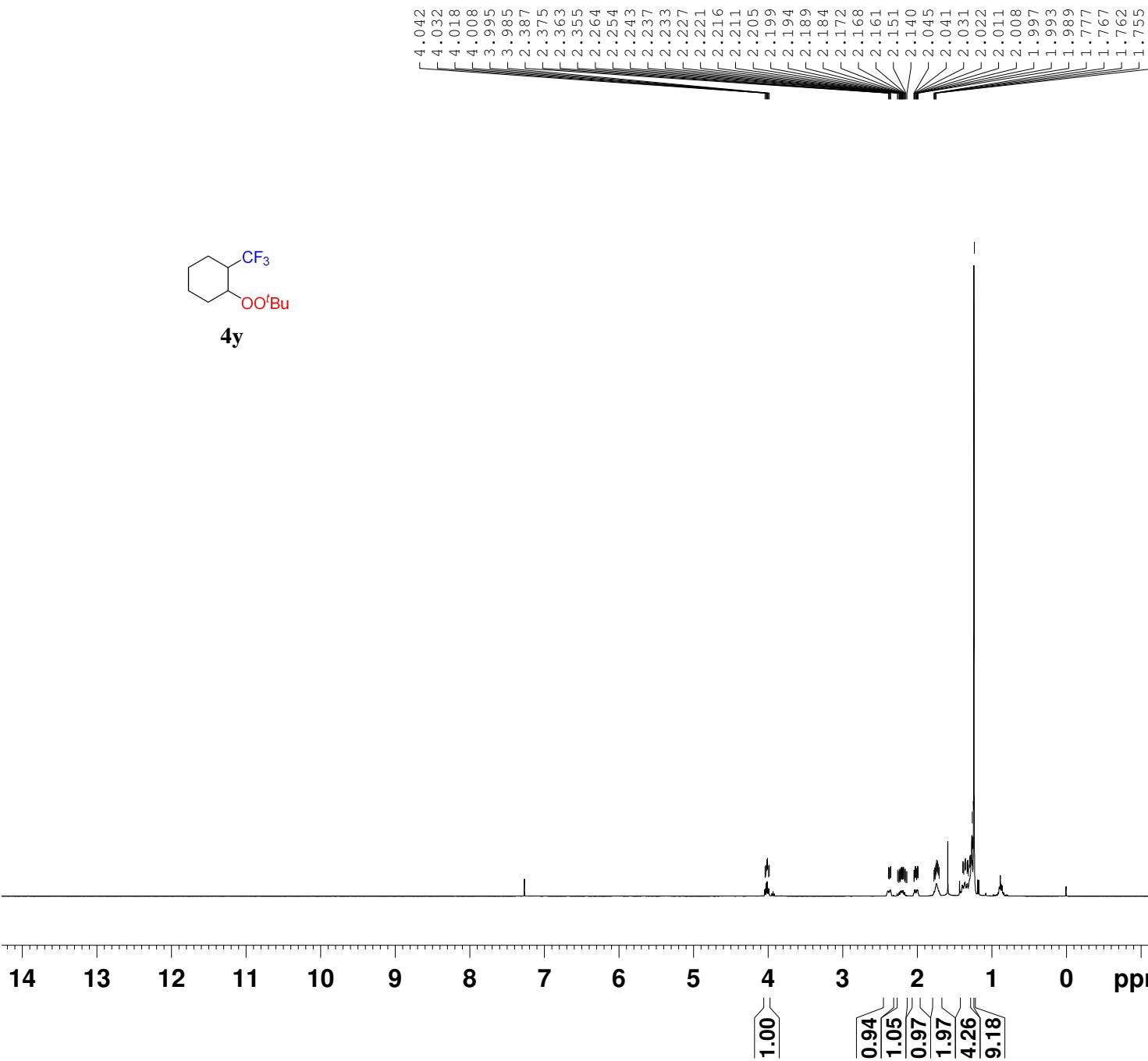
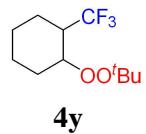




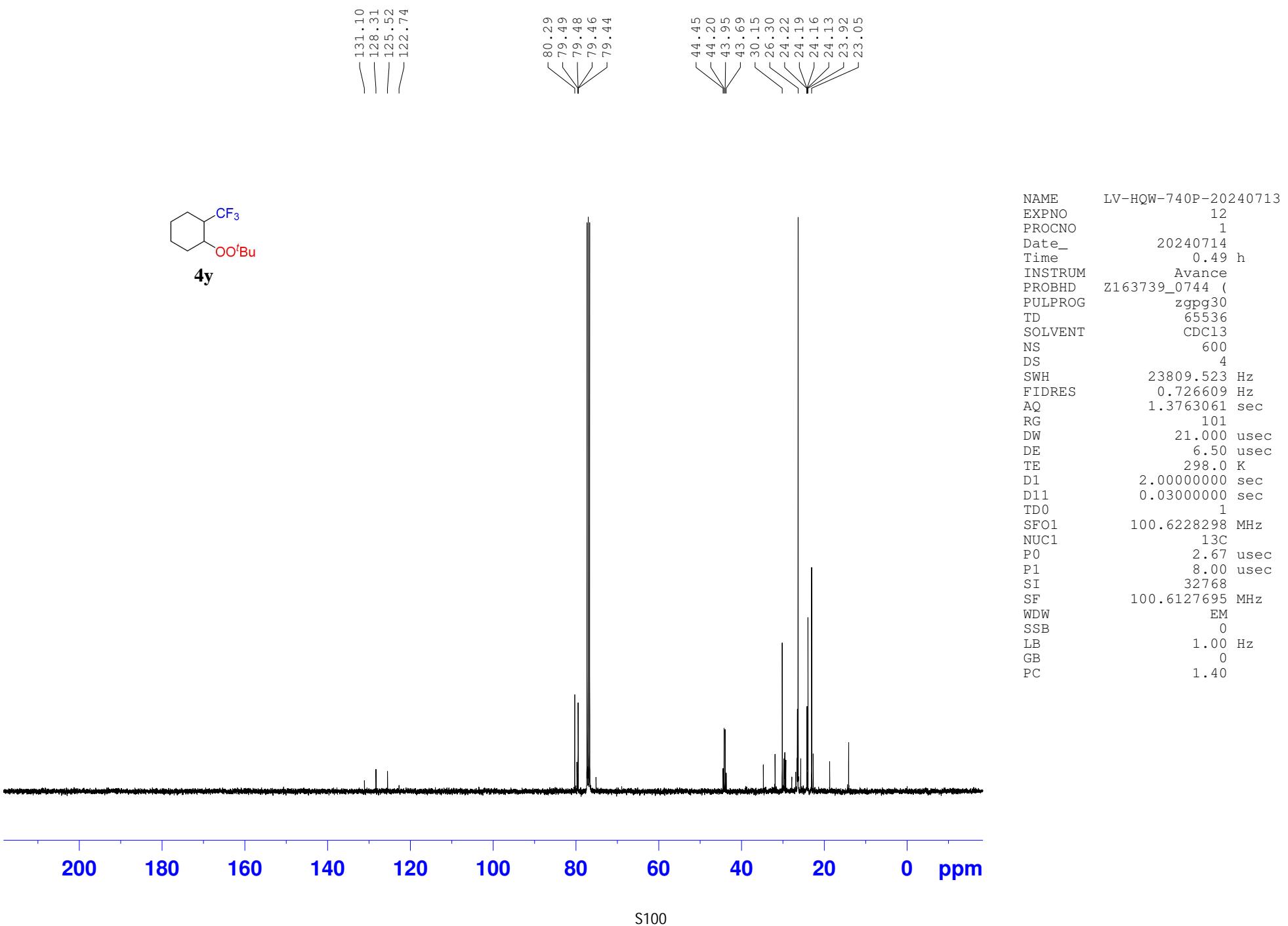
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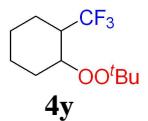


NAME	LV-HQW-684P-20240617
EXPNO	11
PROCNO	1
Date_	20240617
Time	19.03 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	302.2 K
D1	1.0000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME LV-HQW-740P-20240713
 EXPNO 10
 PROCNO 1
 Date_ 20240713
 Time 23.11 h
 INSTRUM Avance
 PROBHD Z163739_0744 (zg30
 PULPROG 65536
 TD 65536
 SOLVENT CDC13
 NS 8
 DS 0
 SWH 6250.000 Hz
 FIDRES 0.190735 Hz
 AQ 5.2429299 sec
 RG 71.8
 DW 80.000 usec
 DE 8.64 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1
 SFO1 400.1326008 MHz
 NUC1 1H
 P0 2.67 usec
 P1 8.00 usec
 SI 65536
 SF 400.1300089 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

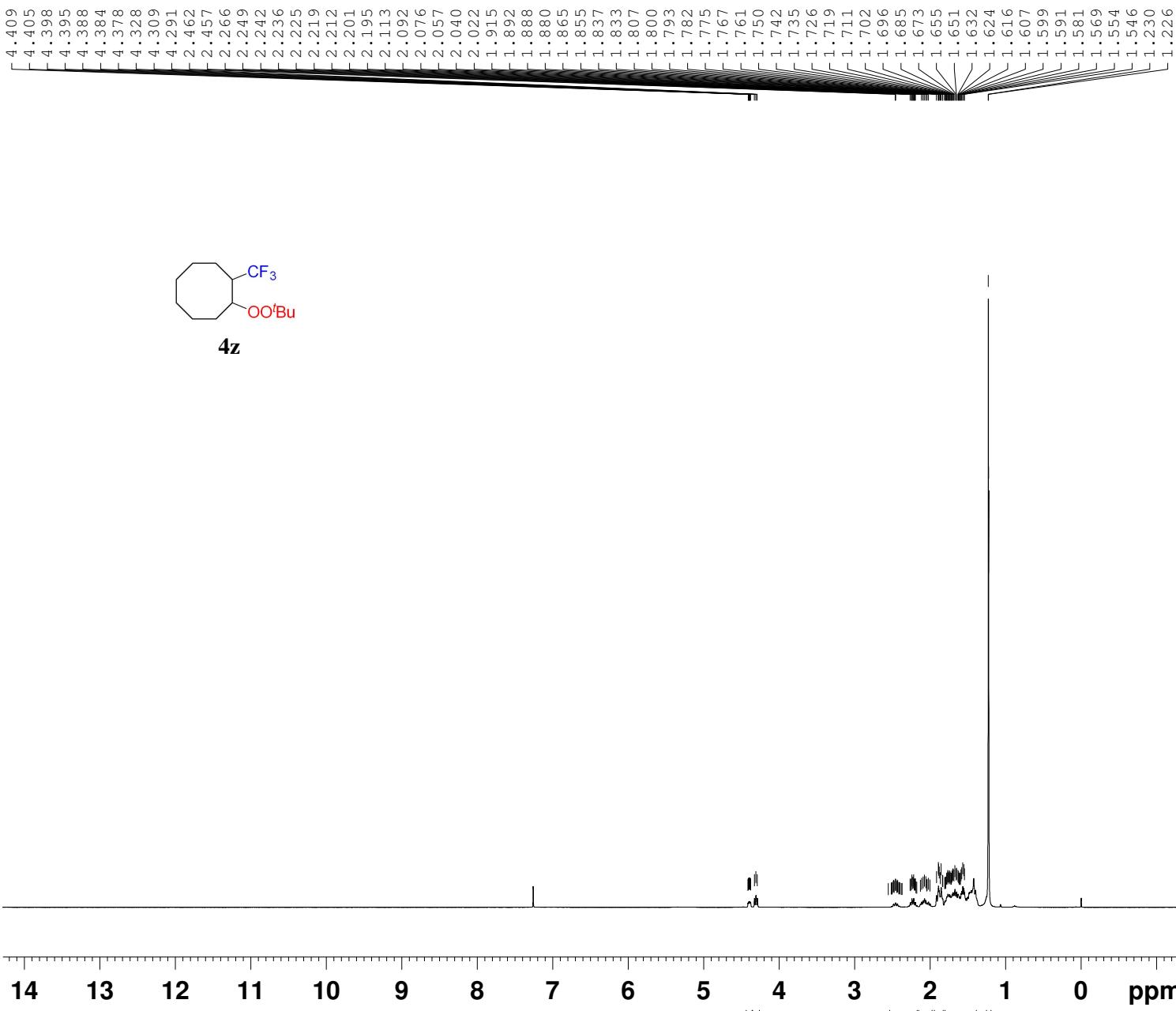


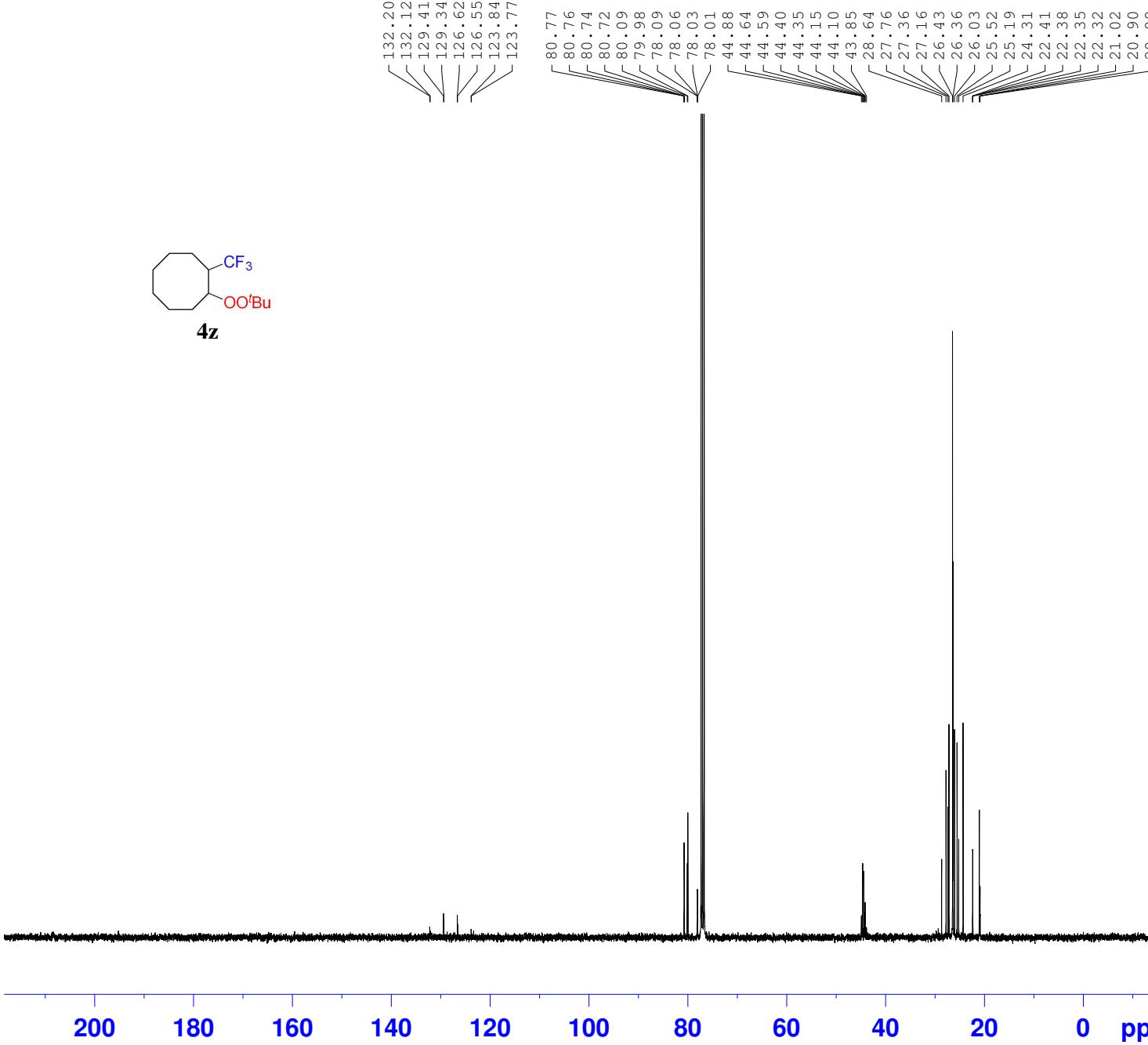


-68.169

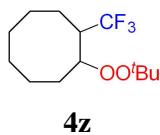
0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 ppm

NAME	LV-HQW-740P-20240713
EXPNO	11
PROCNO	1
Date_	20240713
Time	23.13 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl3
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.1 K
D1	1.0000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

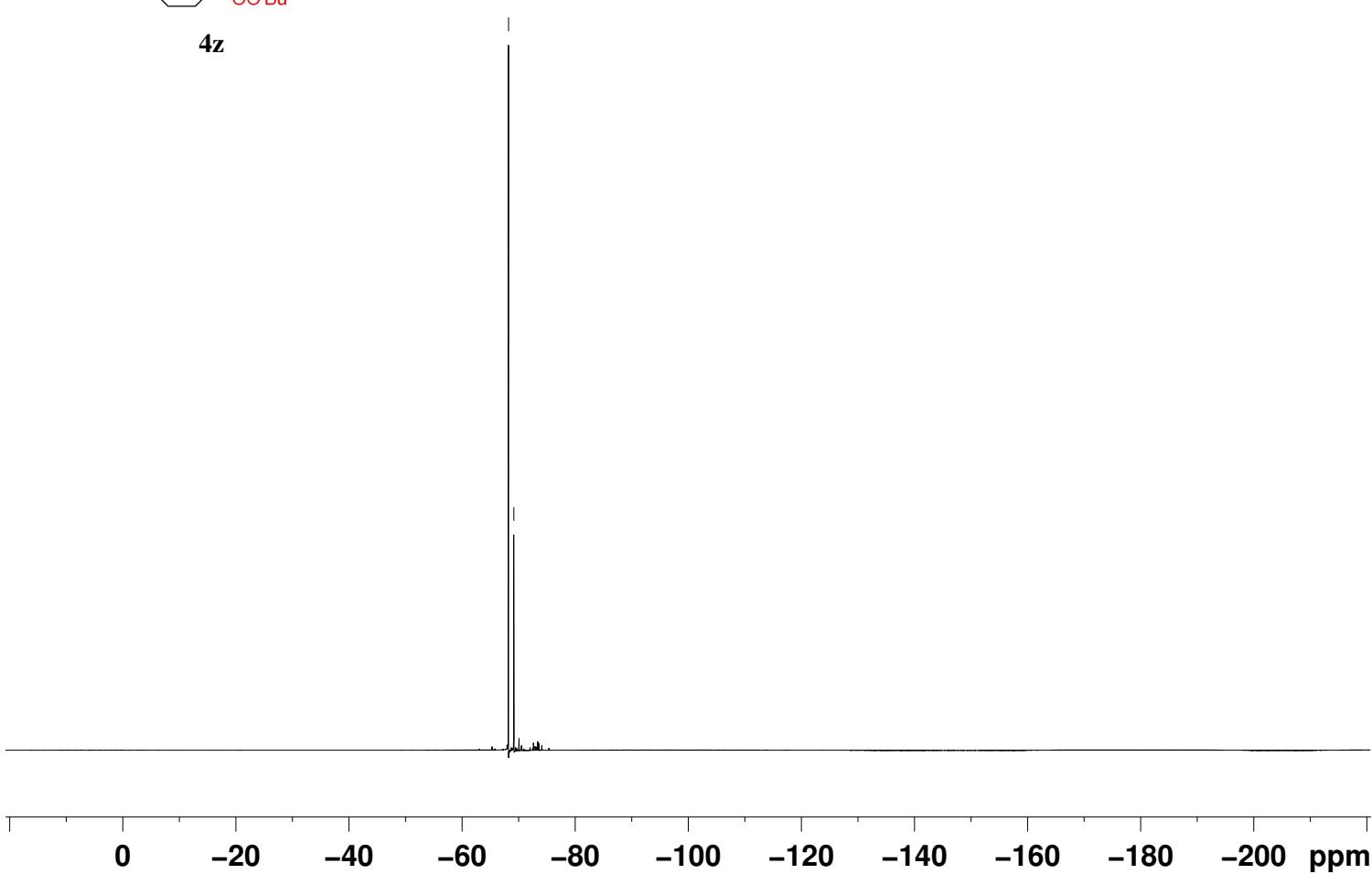




NAME	HQW-513P1-20240316
EXPNO	12
PROCNO	1
Date_	20240316
Time	17.16 h
INSTRUM	Avance
PROBHD	z163739_0744 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl ₃
NS	800
DS	4
SWH	23809.523 Hz
FIDRES	0.726609 Hz
AQ	1.3763061 sec
RG	101
DW	21.000 usec
DE	6.50 usec
TE	298.0 K
D1	2.0000000 sec
D11	0.0300000 sec
TDO	1
SFO1	100.6228298 MHz
NUC1	¹³ C
P0	2.67 usec
P1	8.00 usec
SI	32768
SF	100.6127695 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



-68.213
-69.152

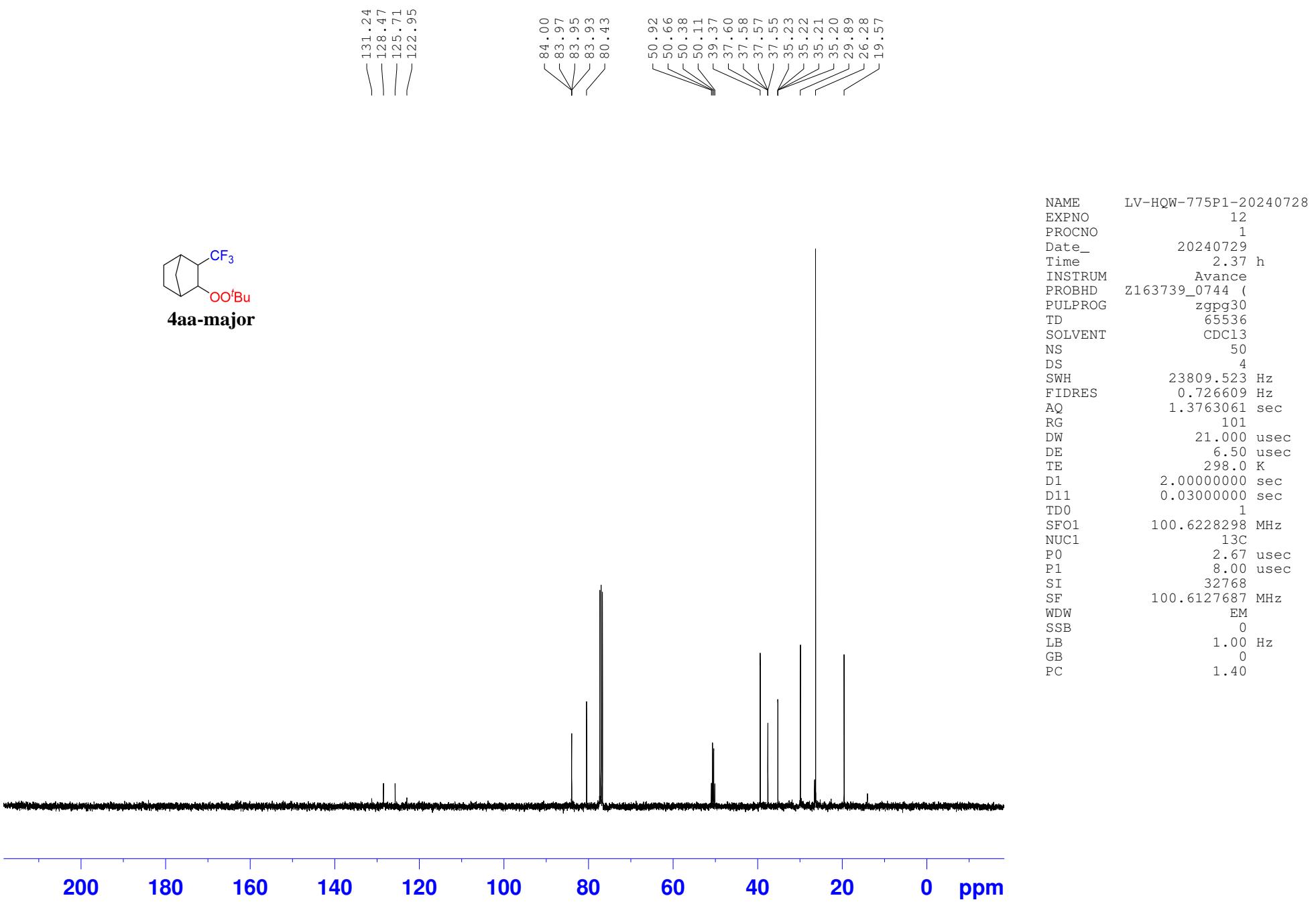


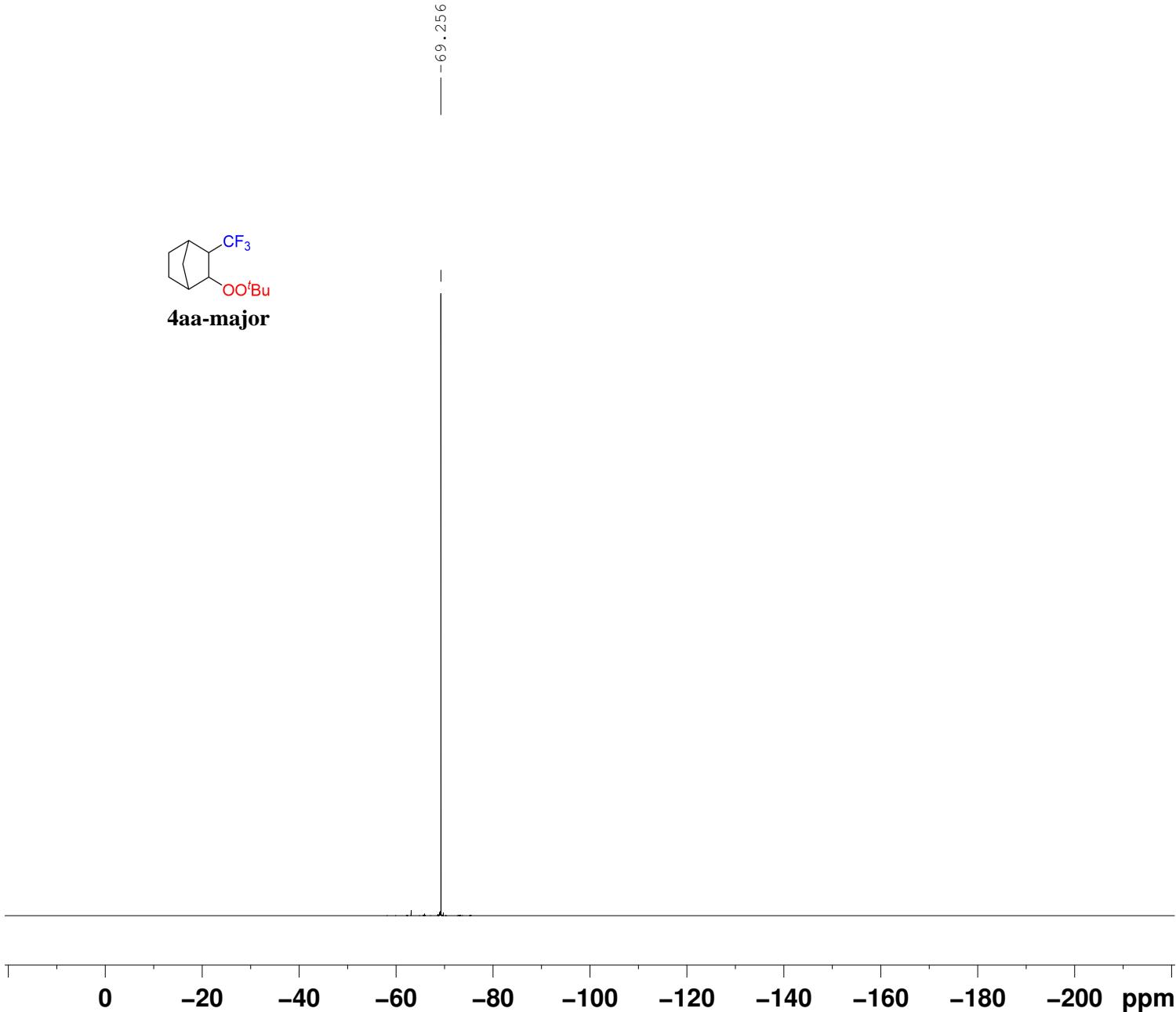
NAME	HQW-513P1-20240316
EXPNO	11
PROCNO	1
Date_	20240316
Time	16.28 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDC13
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.1 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	¹⁹ F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



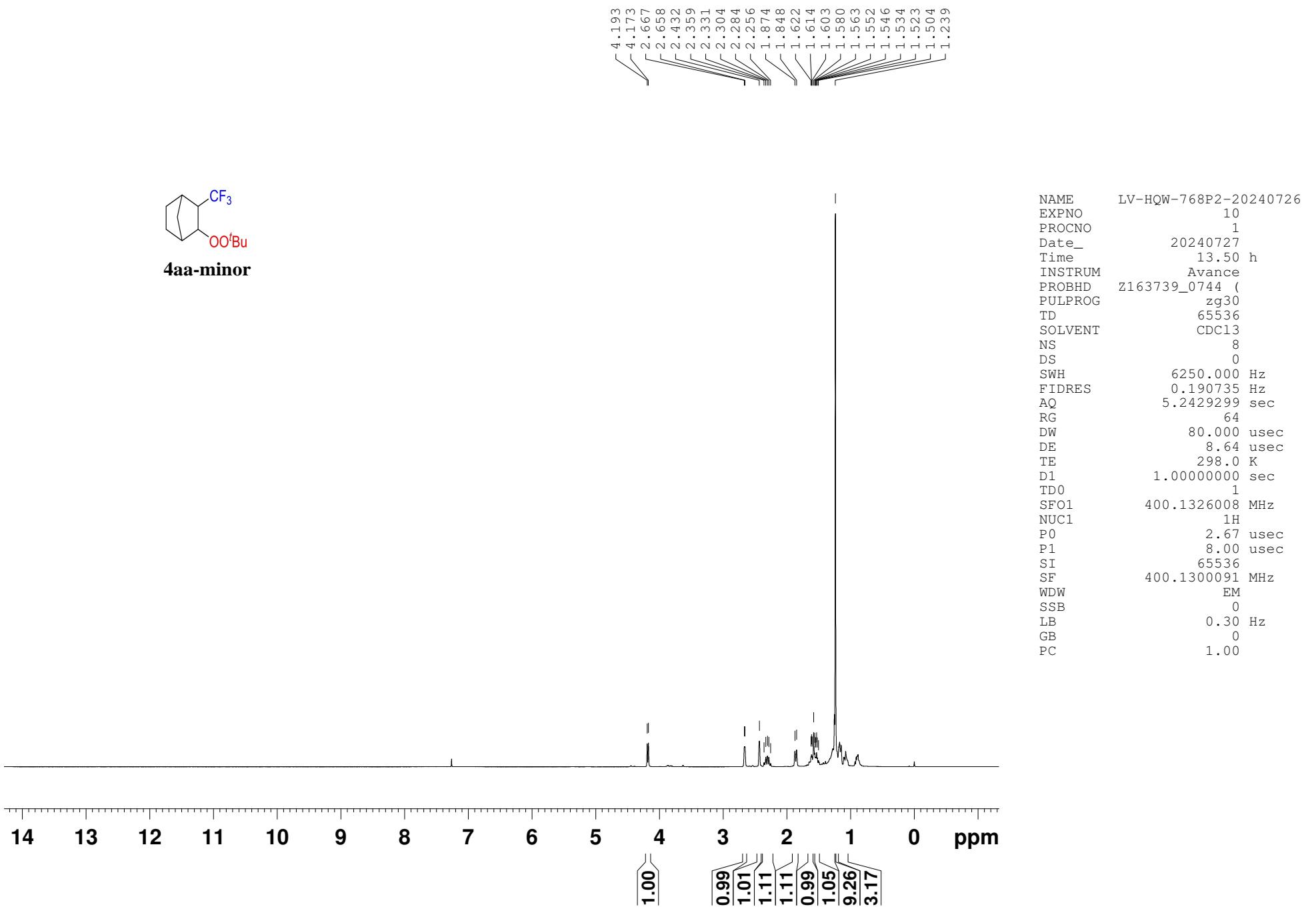
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm

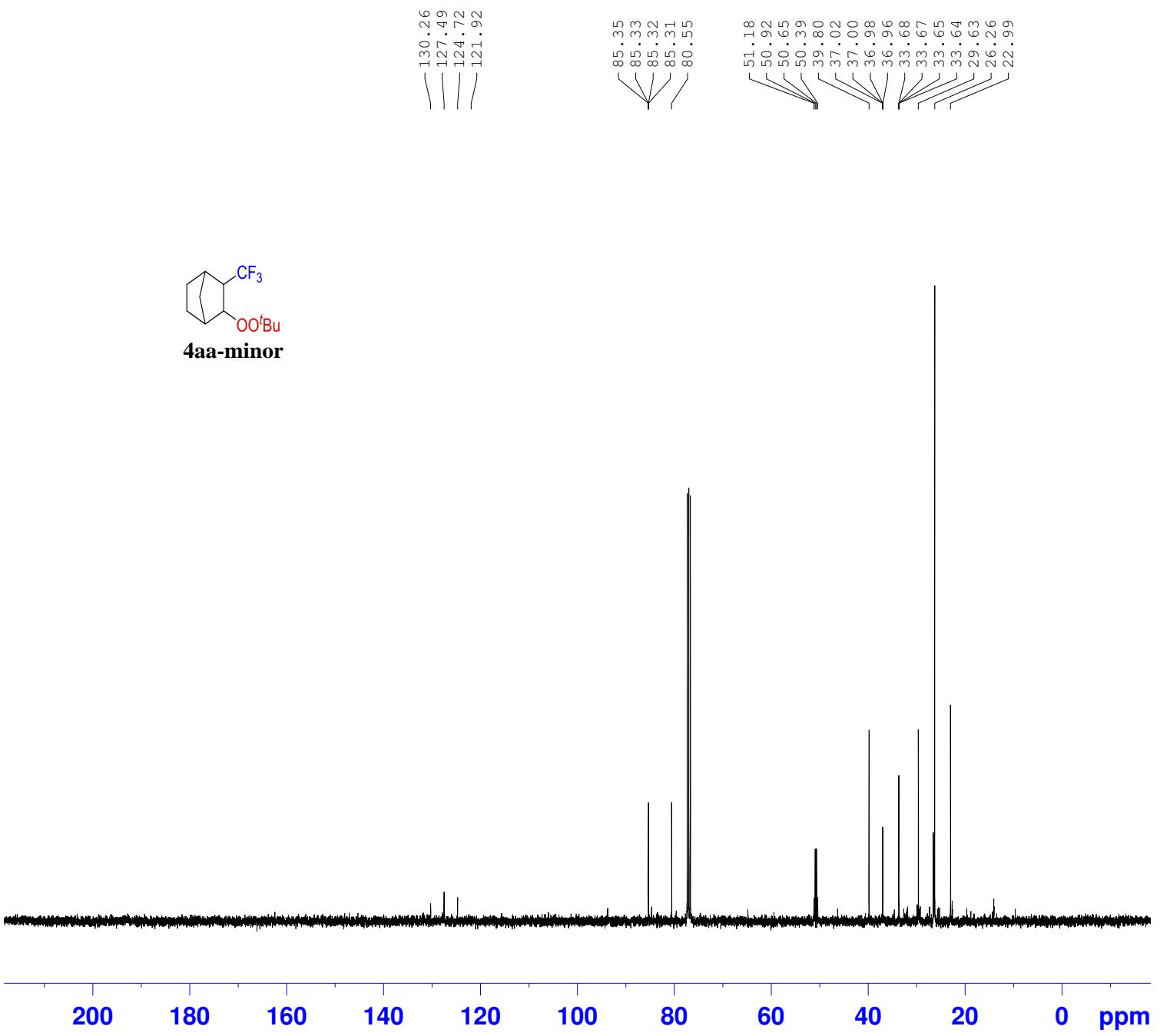
1.00
 1.01 2.12 2.24 1.16 2.03 9.36

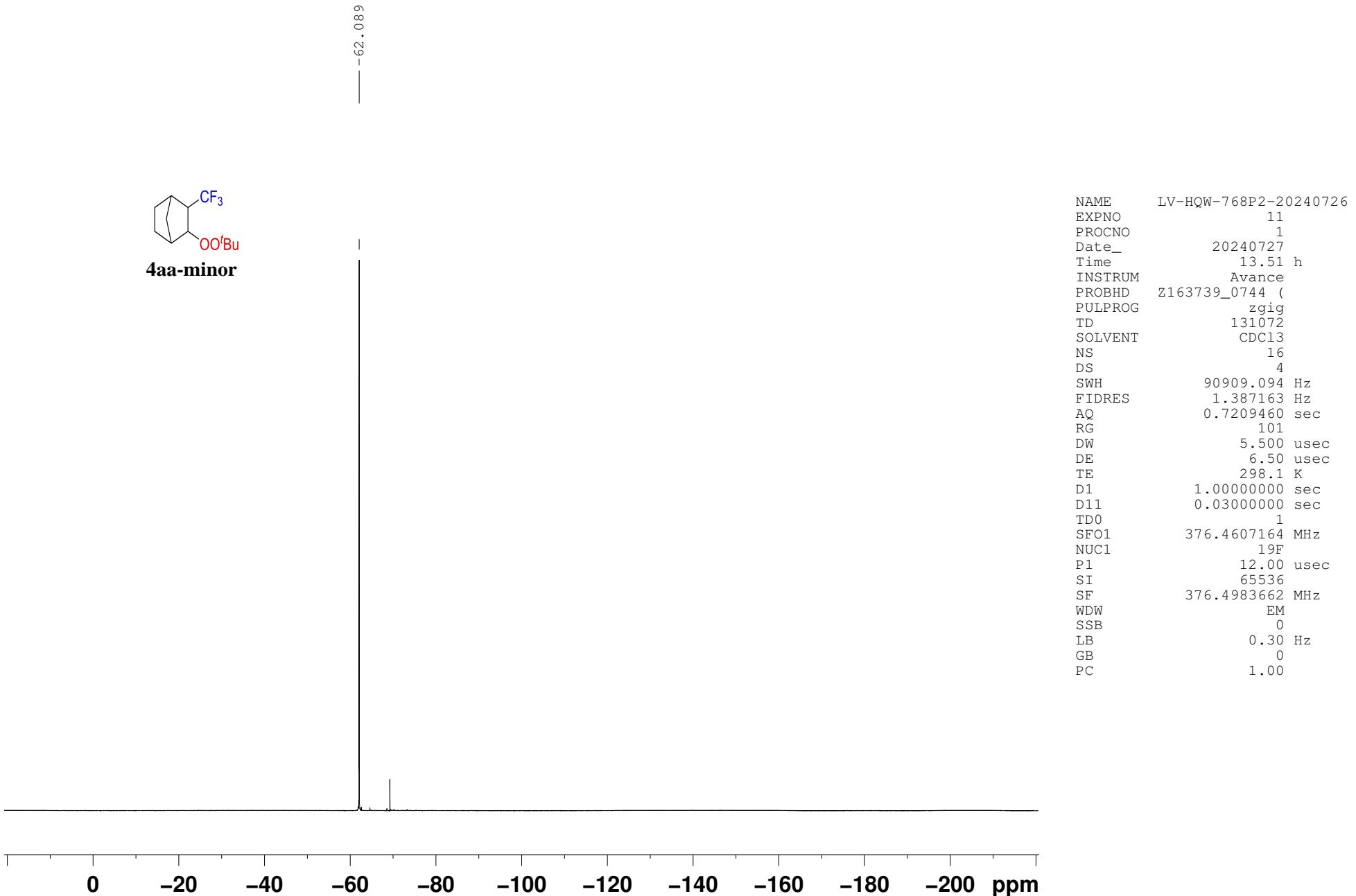


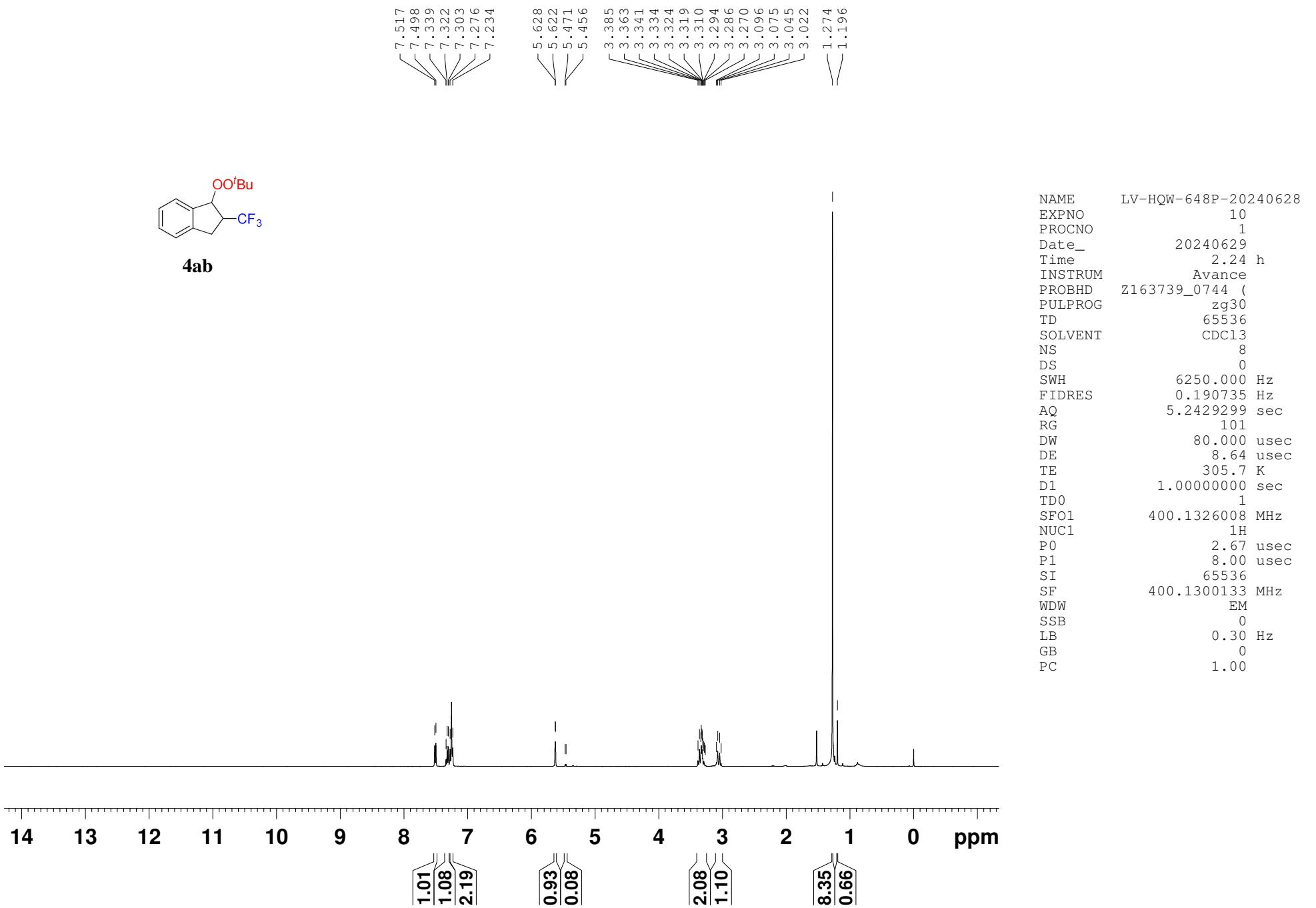


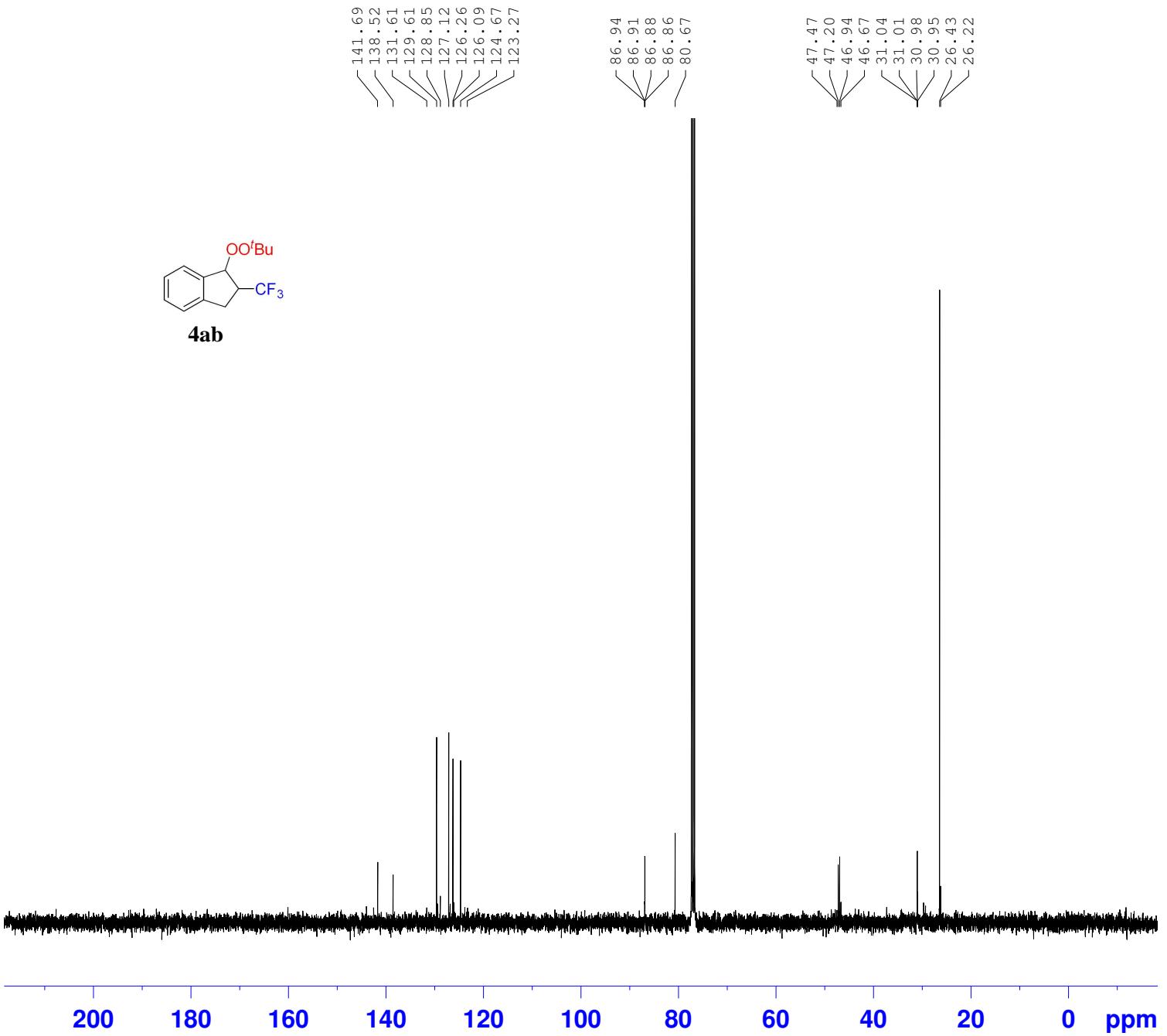
NAME	LV-HQW-775P1-20240728
EXPNO	11
PROCNO	1
Date_	20240729
Time	2.32 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDC ₁₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.1 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	¹⁹ F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



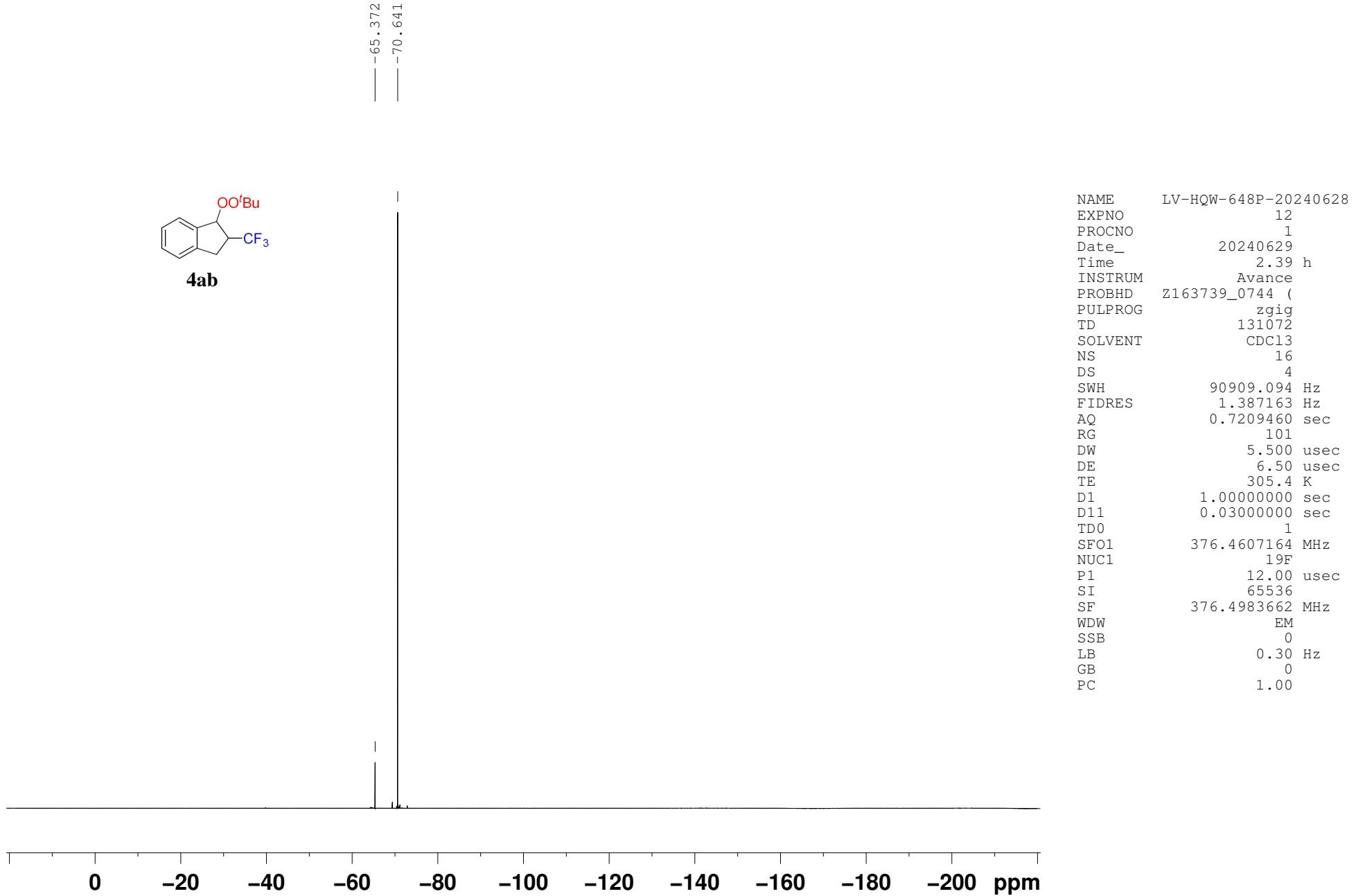


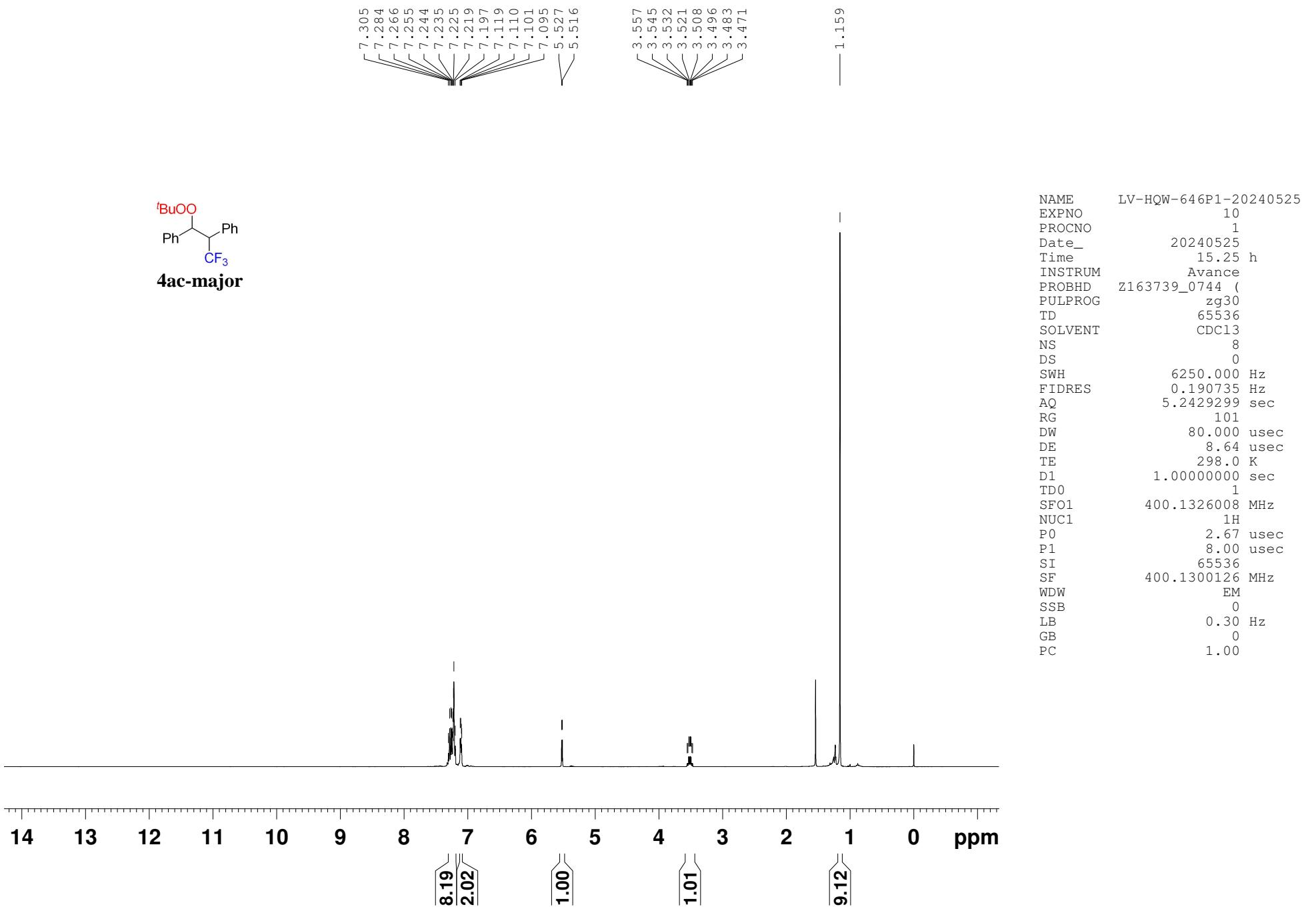


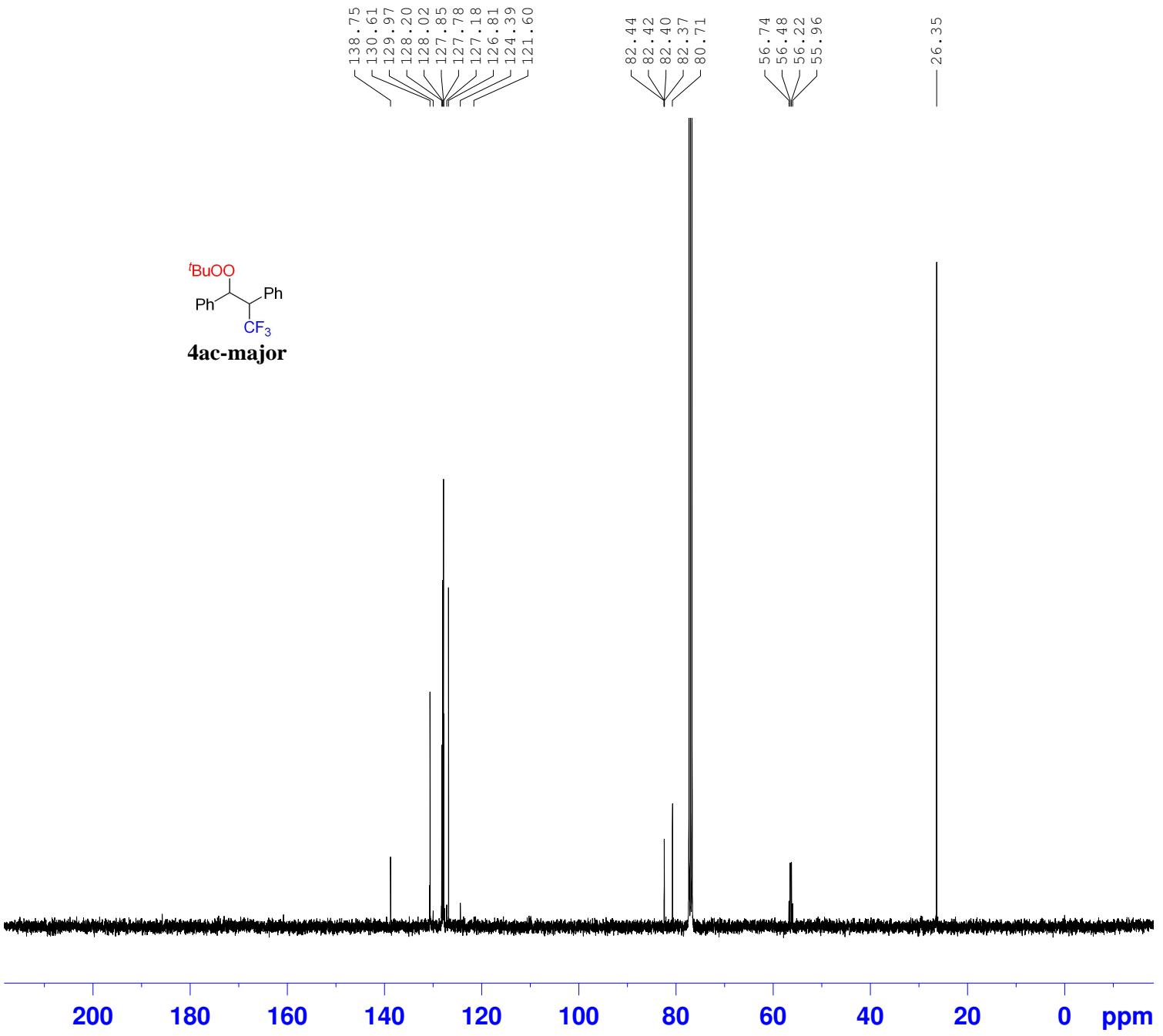




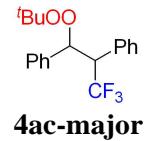
NAME	LV-HQW-648P-20240628
EXPNO	11
PROCNO	1
Date_	20240629
Time	2.37 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl ₃
NS	200
DS	4
SWH	23809.523 Hz
FIDRES	0.726609 Hz
AQ	1.3763061 sec
RG	101
DW	21.000 usec
DE	6.50 usec
TE	305.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TDO	1
SFO1	100.6228298 MHz
NUC1	¹³ C
P0	2.67 usec
P1	8.00 usec
SI	32768
SF	100.6127680 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40





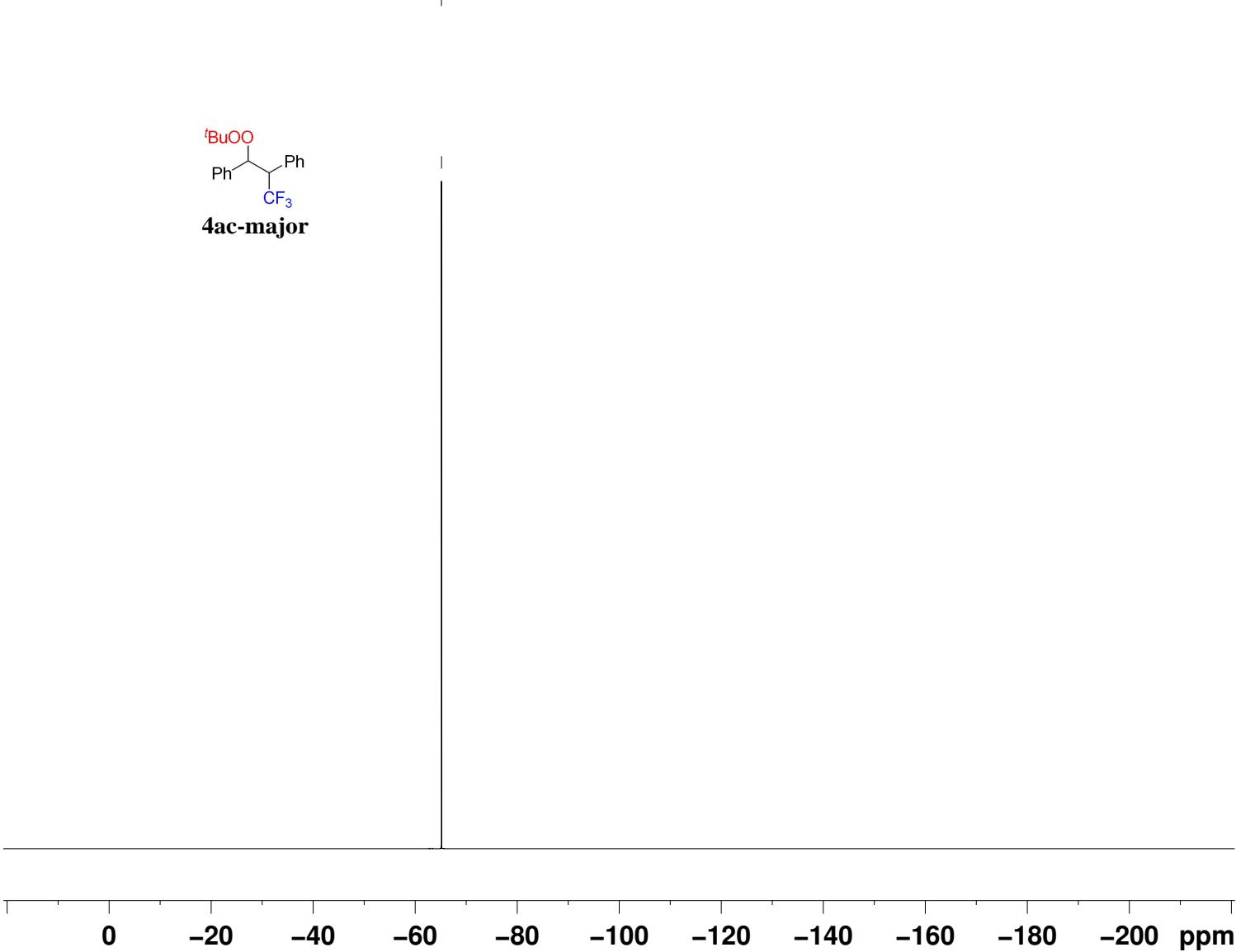


NAME	LV-HQW-646P1-20240525
EXPNO	12
PROCNO	1
Date_	20240525
Time	16.02 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl ₃
NS	584
DS	4
SWH	23809.523 Hz
FIDRES	0.726609 Hz
AQ	1.3763061 sec
RG	101
DW	21.000 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6228298 MHz
NUC1	¹³ C
P0	2.67 usec
P1	8.00 usec
SI	32768
SF	100.6127709 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

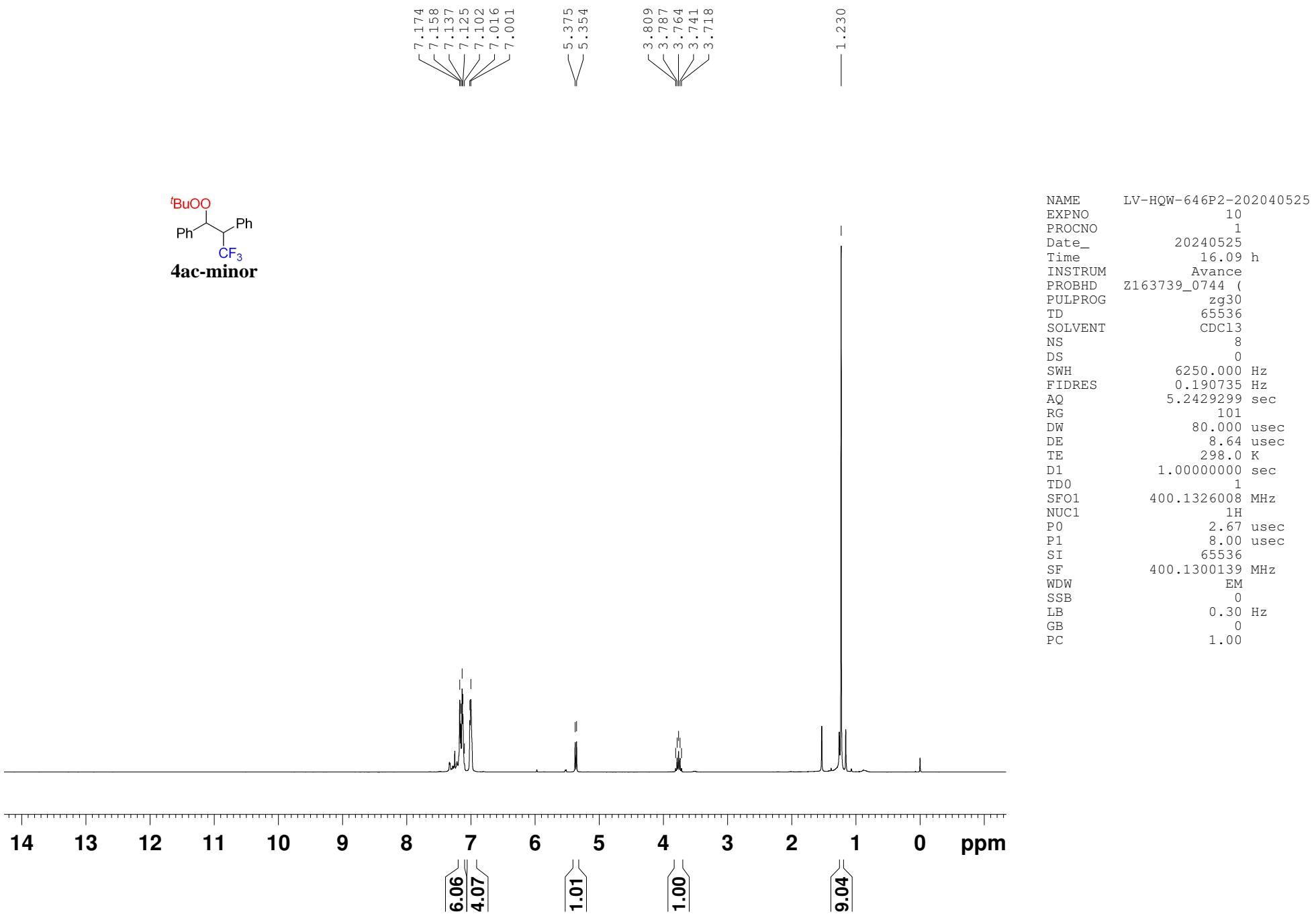


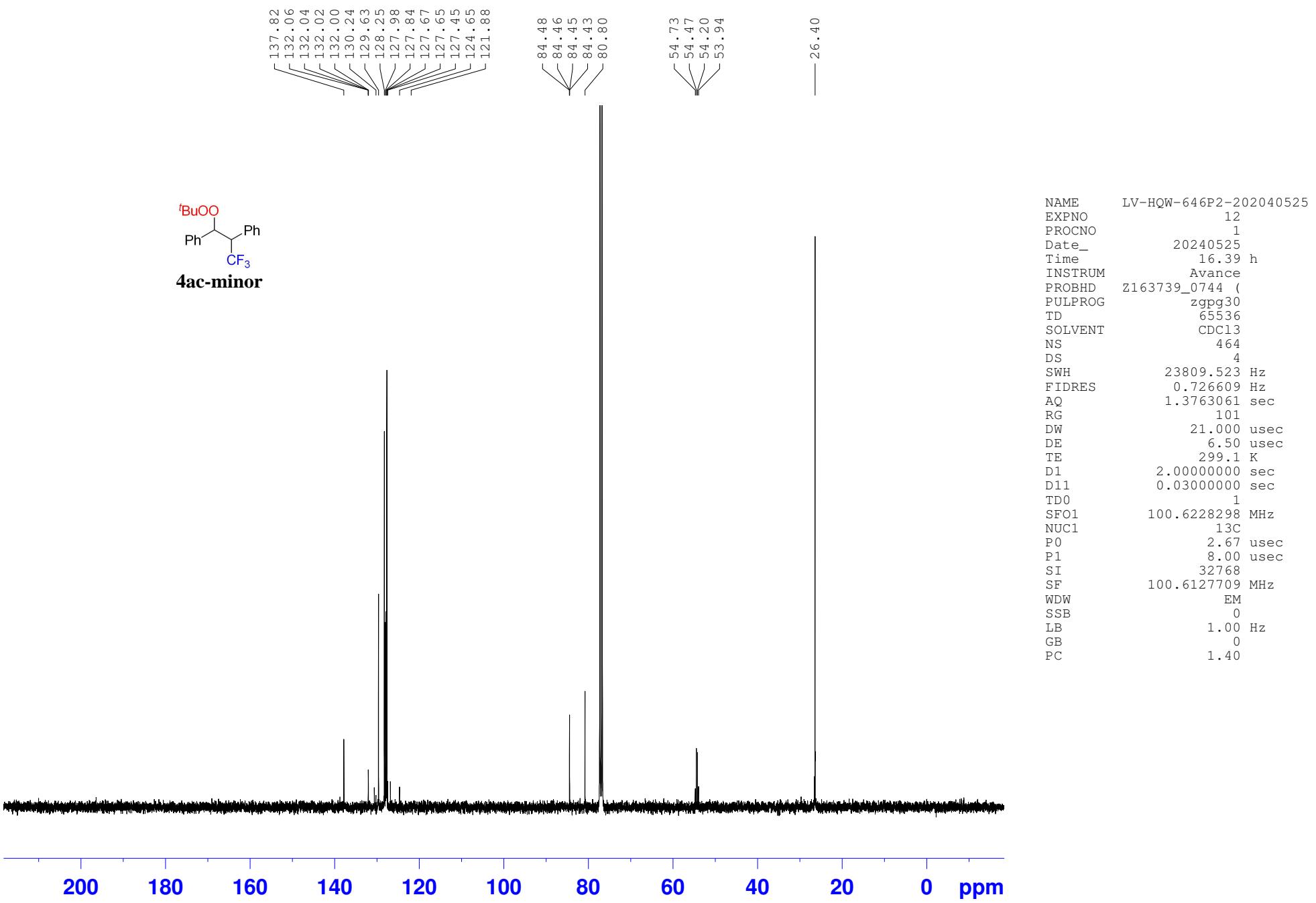
4ac-major

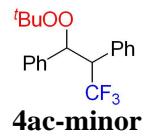
— -65.165



NAME	LV-HQW-646P1-20240525
EXPNO	11
PROCNO	1
Date_	20240525
Time	15.27 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDC13
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.1 K
D1	1.0000000 sec
D11	0.0300000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

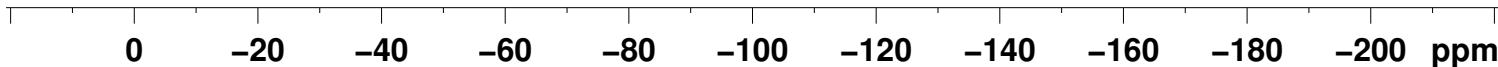


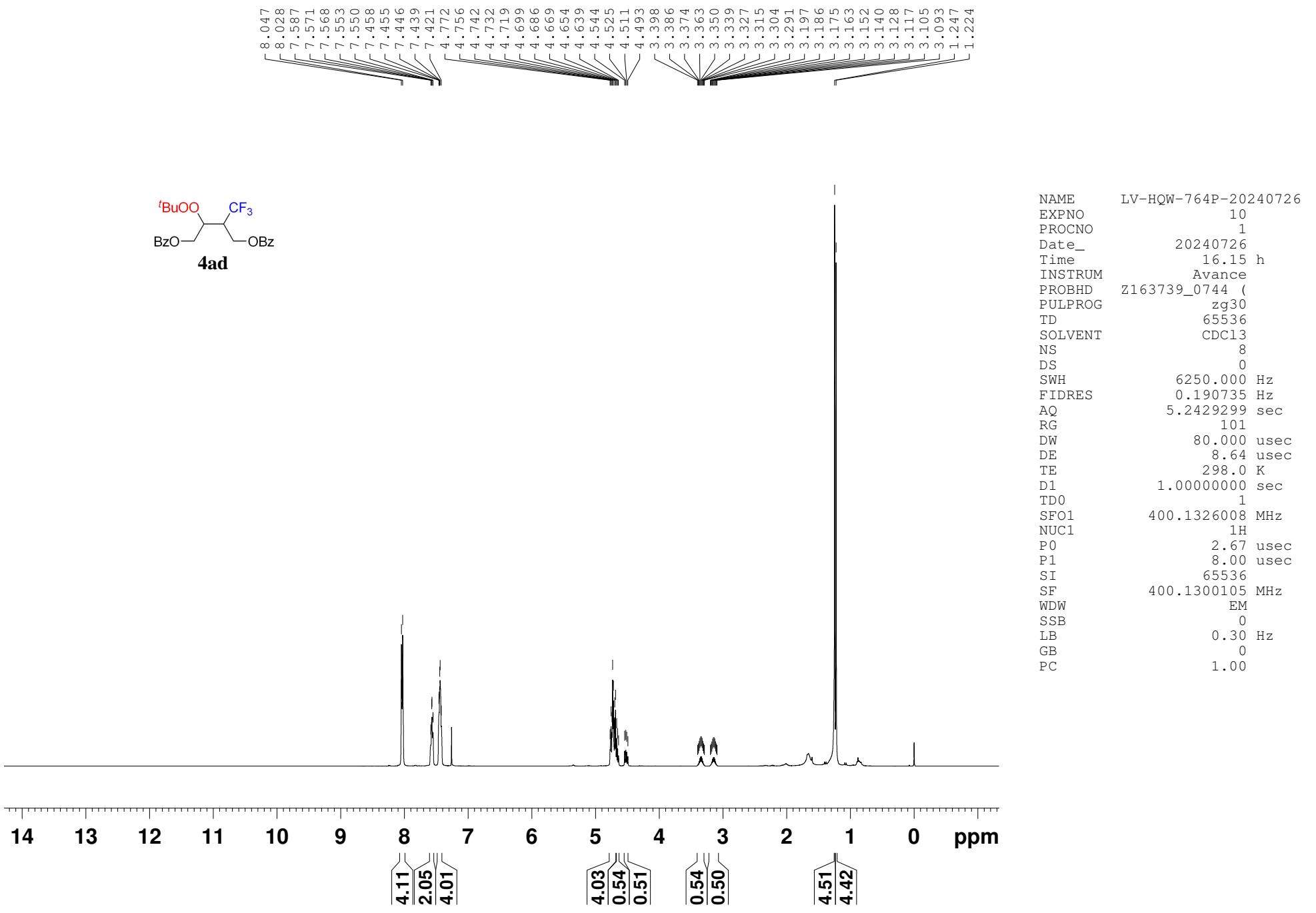


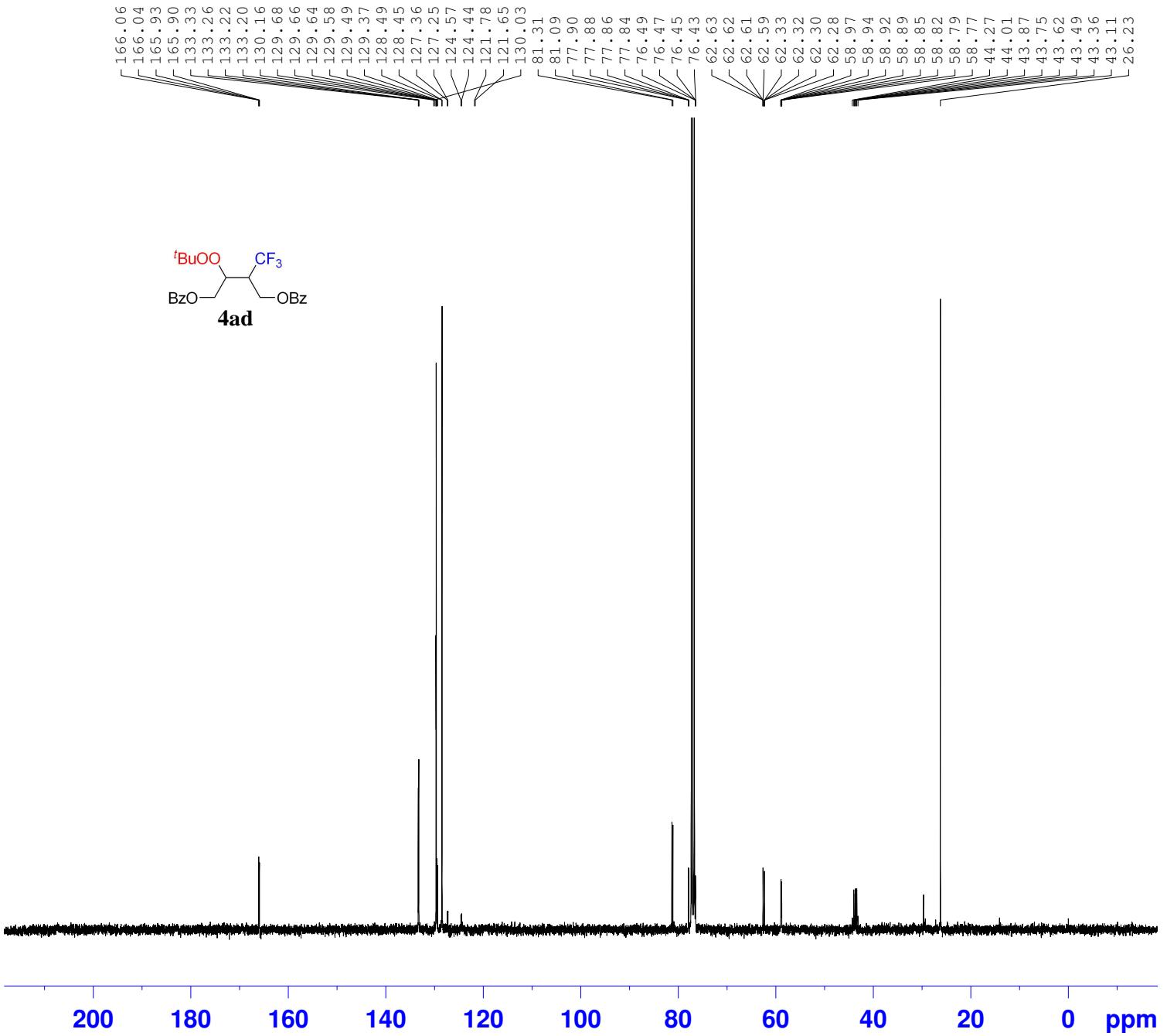


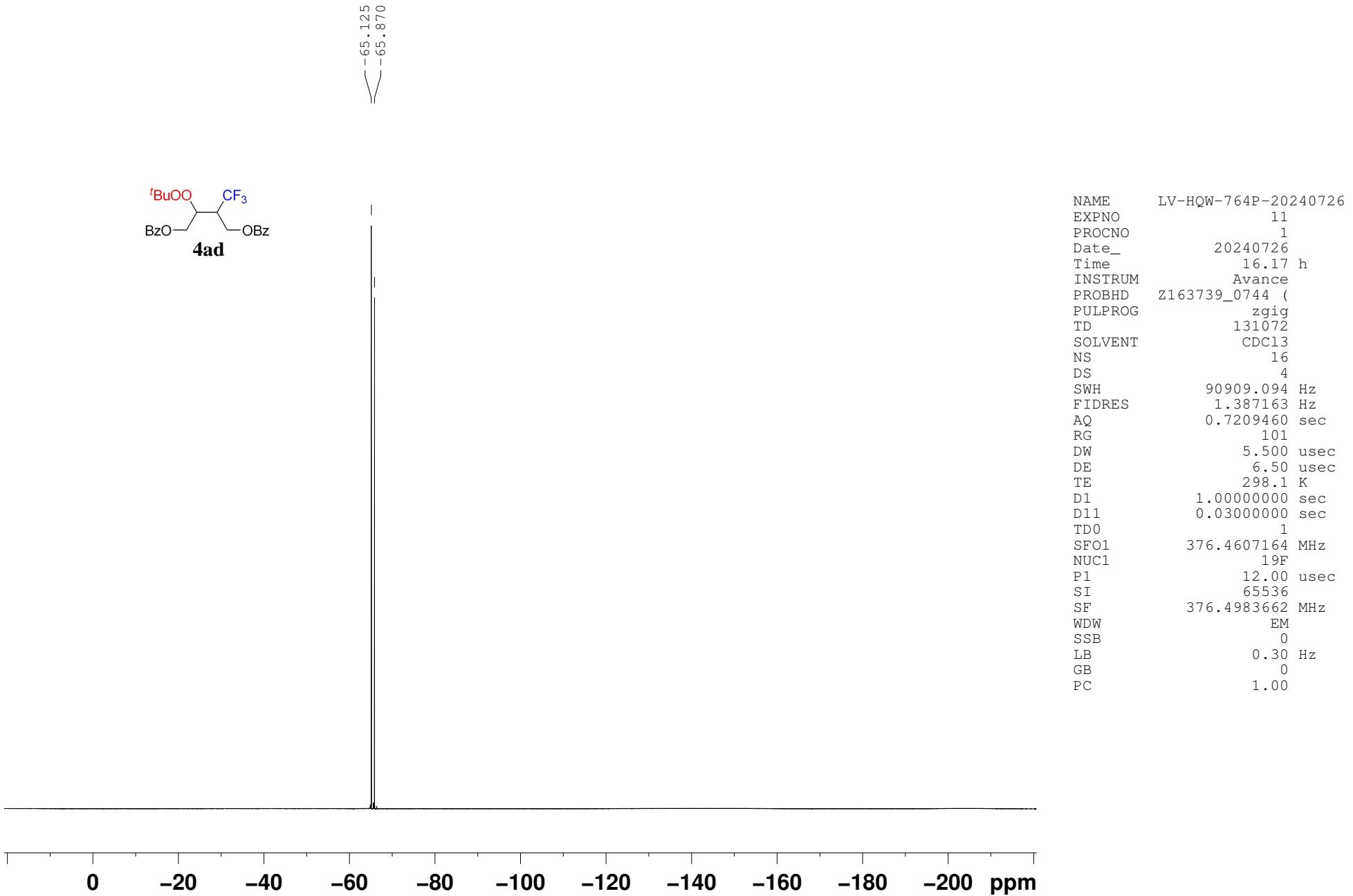
— -63.411

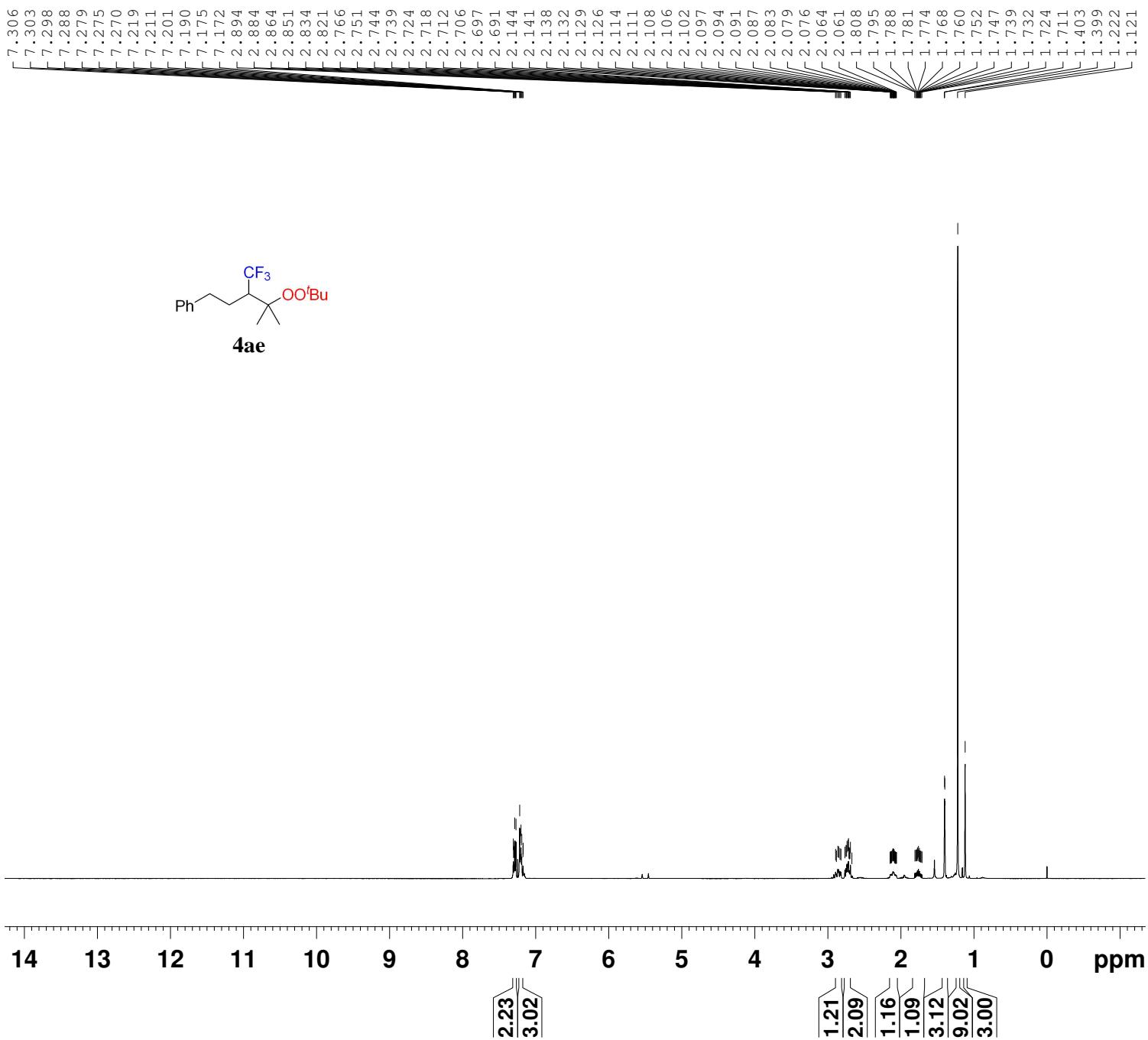
NAME	LV-HQW-646P2-202040525
EXPNO	11
PROCNO	1
Date_	20240525
Time	16.11 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.1 K
D1	1.0000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	¹⁹ F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



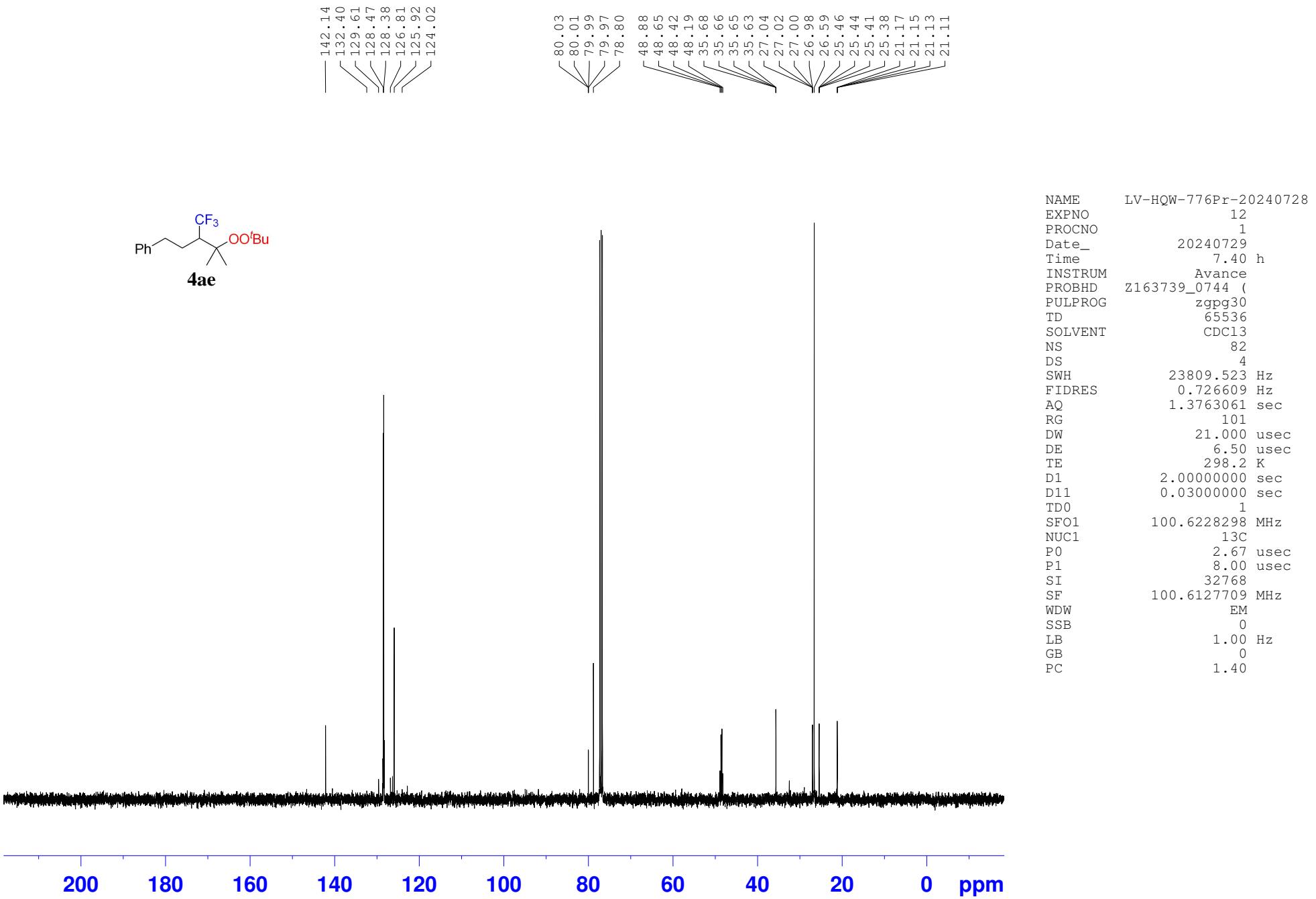


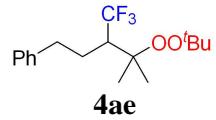




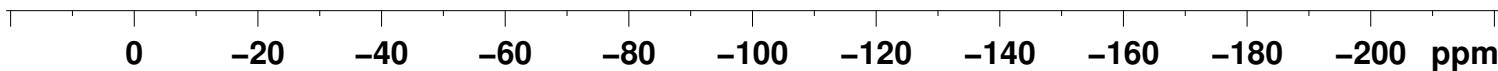


NAME LV-HQW-776Pr-20240728
 EXPNO 10
 PROCNO 1
 Date_ 20240729
 Time 7.32 h
 INSTRUM Avance
 PROBHD Z163739_0744 (zg30
 PULPROG 65536
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 6250.000 Hz
 FIDRES 0.190735 Hz
 AQ 5.2429299 sec
 RG 57
 DW 80.000 usec
 DE 8.64 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1
 SFO1 400.1326008 MHz
 NUC1 1H
 P0 2.67 usec
 P1 8.00 usec
 SI 65536
 SF 400.1300160 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

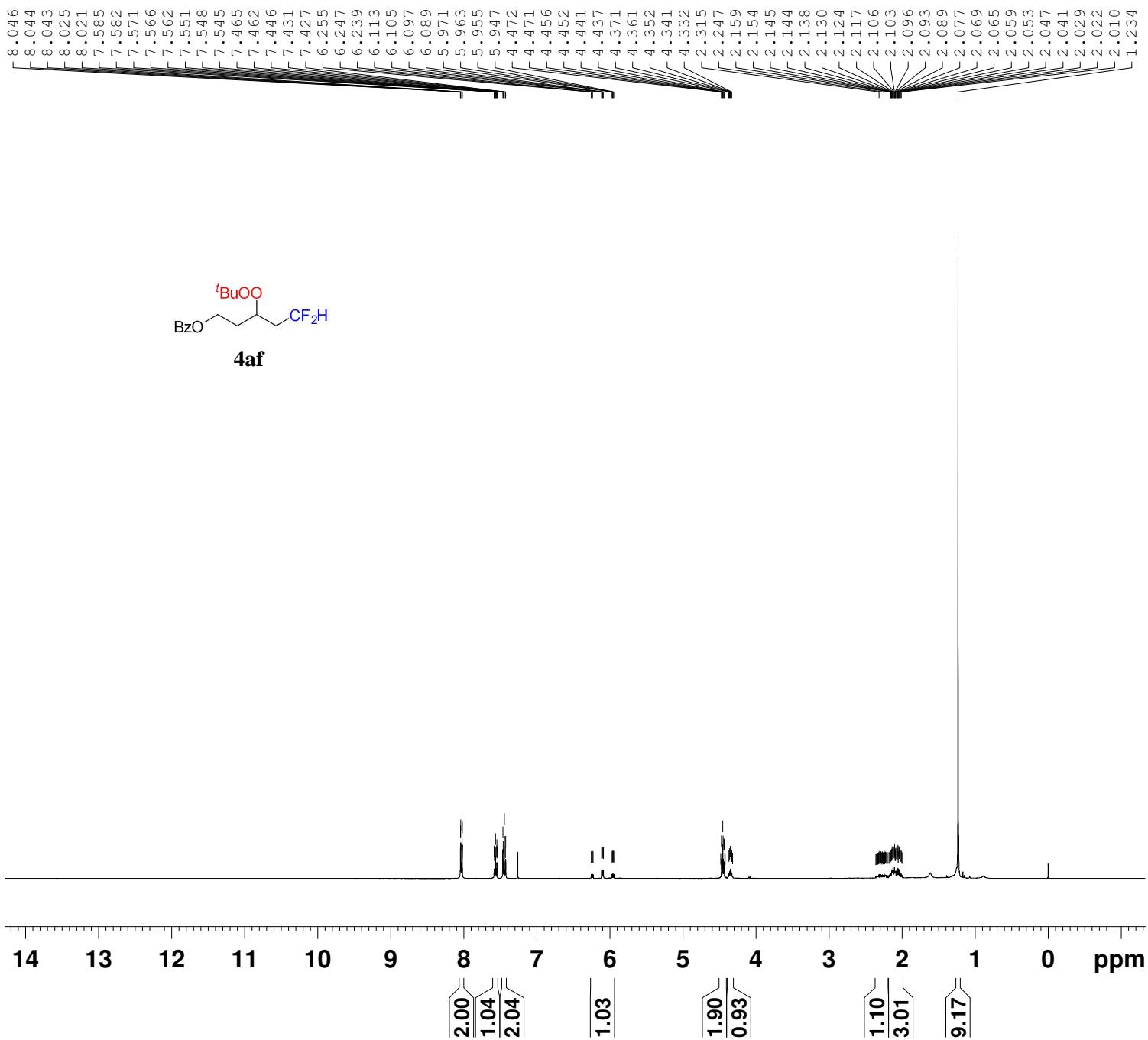




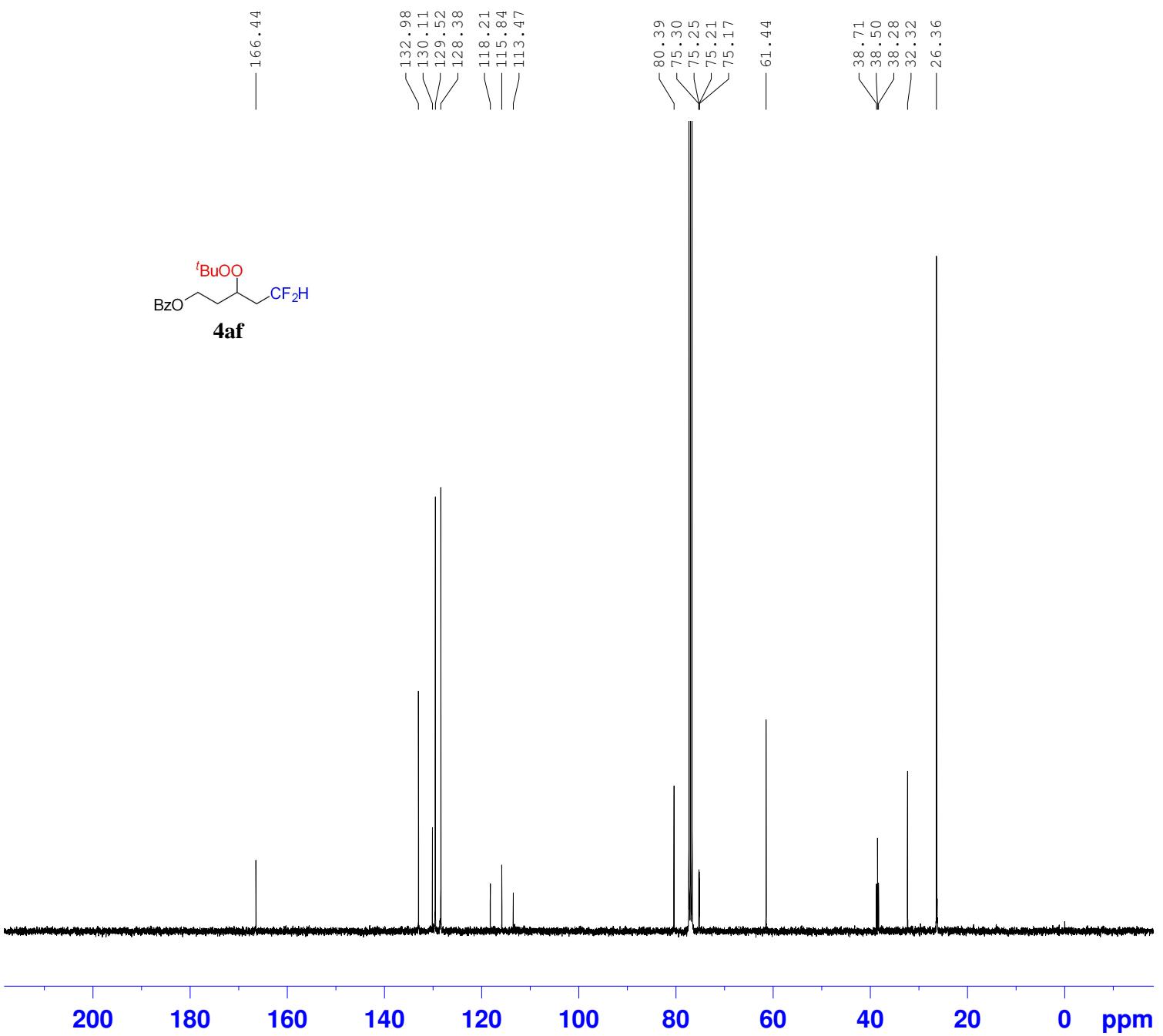
— -63.215 —



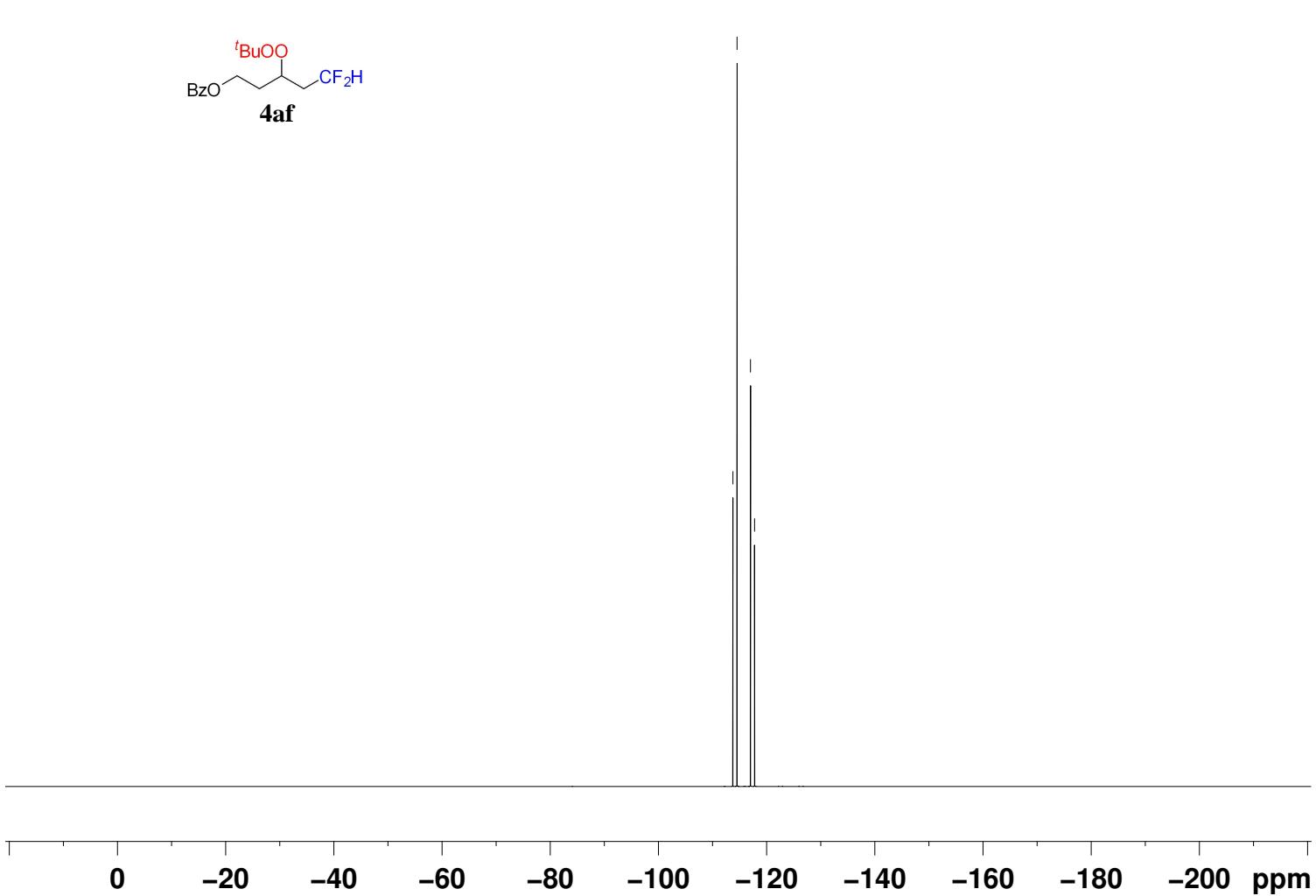
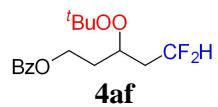
NAME	LV-HQW-776Pr-20240728
EXPNO	11
PROCNO	1
Date_	20240729
Time	7.33 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDC13
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.1 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



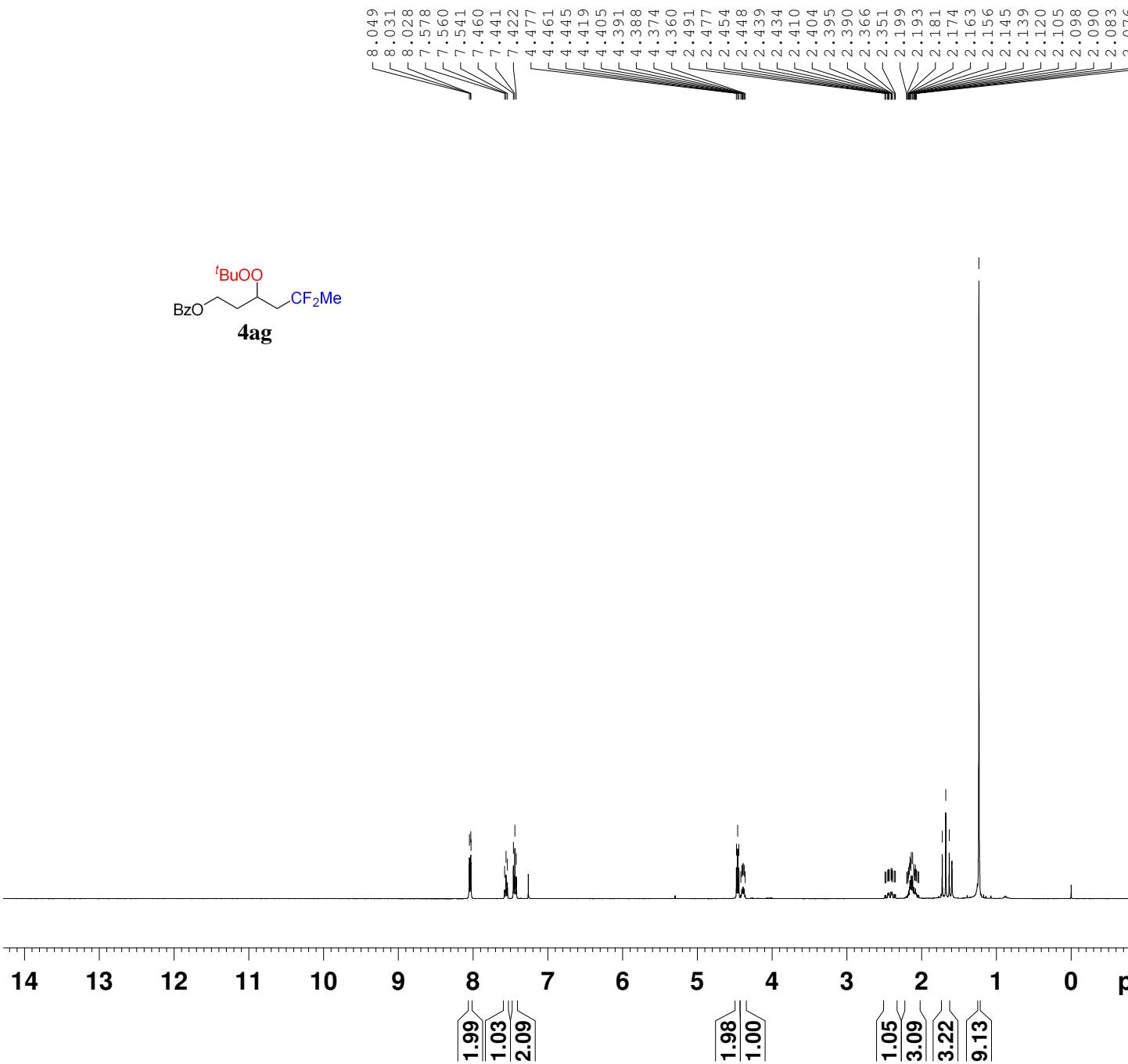
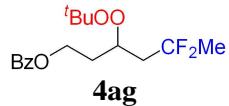
NAME LV-HQW-728P-20240708
 EXPNO 10
 PROCNO 1
 Date_ 20240709
 Time 0.19 h
 INSTRUM Avance
 PROBHD Z163739_0744 (zg30
 PULPROG 65536
 TD 65536
 SOLVENT CDC13
 NS 8
 DS 0
 SWH 6250.000 Hz
 FIDRES 0.190735 Hz
 AQ 5.2429299 sec
 RG 90.5
 DW 80.000 usec
 DE 8.64 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1
 SFO1 400.1326008 MHz
 NUC1 1H
 P0 2.67 usec
 P1 8.00 usec
 SI 65536
 SF 400.1300094 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



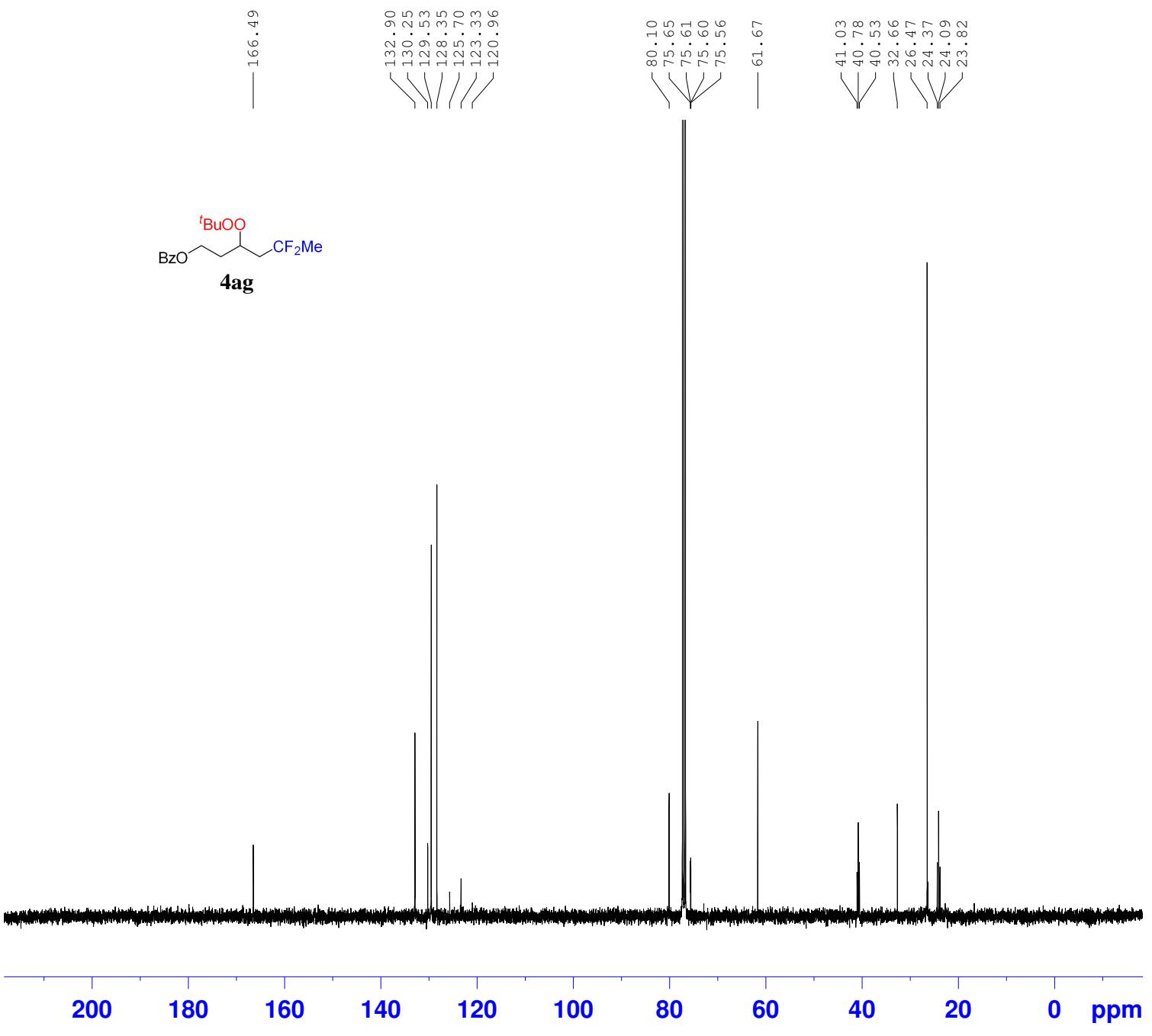
-113.770
-114.529
-117.014
-117.772



NAME LV-HQW-728P-20240708
EXPNO 11
PROCNO 1
Date_ 20240709
Time 0.21 h
INSTRUM Avance
PROBHD Z163739_0744 (
PULPROG zgig
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 90909.094 Hz
FIDRES 1.387163 Hz
AQ 0.7209460 sec
RG 101
DW 5.500 usec
DE 6.50 usec
TE 298.1 K
D1 1.0000000 sec
D11 0.03000000 sec
TD0 1
SFO1 376.4607164 MHz
NUC1 19F
P1 12.00 usec
SI 65536
SF 376.4983662 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



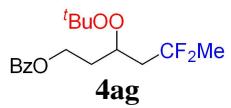
NAME LV-HQW-727P-20240708
 EXPNO 10
 PROCNO 1
 Date_ 20240708
 Time 18.20 h
 INSTRUM Avance
 PROBHD Z163739_0744 (zg30
 PULPROG
 TD 65536
 SOLVENT CDC13
 NS 8
 DS 0
 SWH 6250.000 Hz
 FIDRES 0.190735 Hz
 AQ 5.2429299 sec
 RG 90.5
 DW 80.000 usec
 DE 8.64 usec
 TE 298.0 K
 D1 1.0000000 sec
 TDO 1
 SFO1 400.1326008 MHz
 NUC1 1H
 P0 2.67 usec
 P1 8.00 usec
 SI 65536
 SF 400.1300098 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

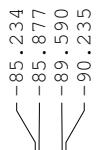


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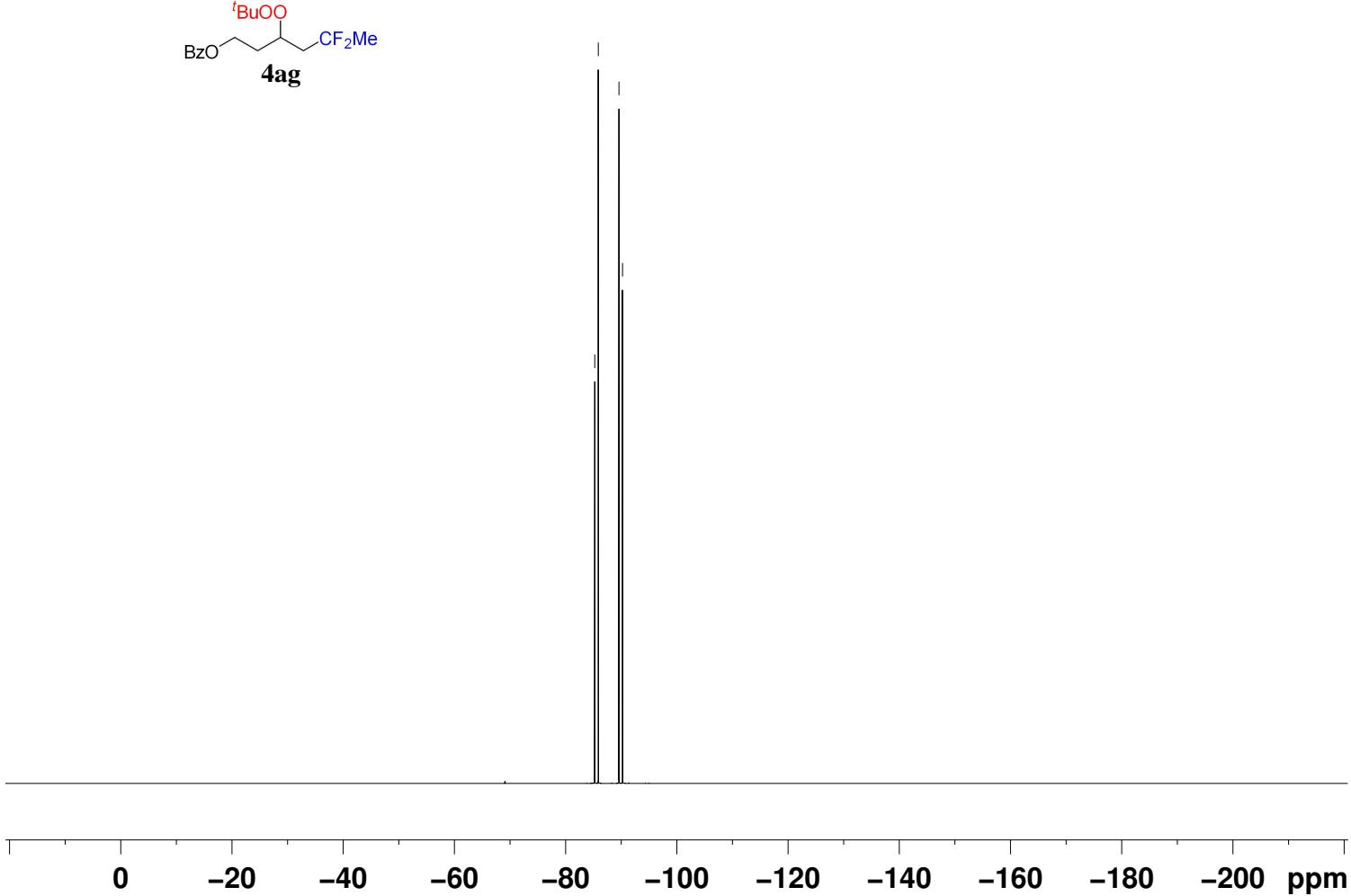
NAME      LV-HQW-727P-20240708
EXPNO    12
PROCNO   1
Date_   20240708
Time   18.36 h
INSTRUM Avance
PROBHD Z163739_0744 (
PULPROG zgpg30
TD      65536
SOLVENT CDC13
NS      216
DS      4
SWH     23809.523 Hz
FIDRES  0.726609 Hz
AQ      1.3763061 sec
RG      101
DW      21.000 usec
DE      6.50 usec
TE      298.0 K
D1      2.00000000 sec
D11     0.03000000 sec
TD0      1
SFO1    100.6228298 MHz
NUC1    13C
P0      2.67 usec
P1      8.00 usec
SI      32768
SF      100.6127710 MHz
WDW
SSB
LB      1.00 Hz
GB
PC      1.40

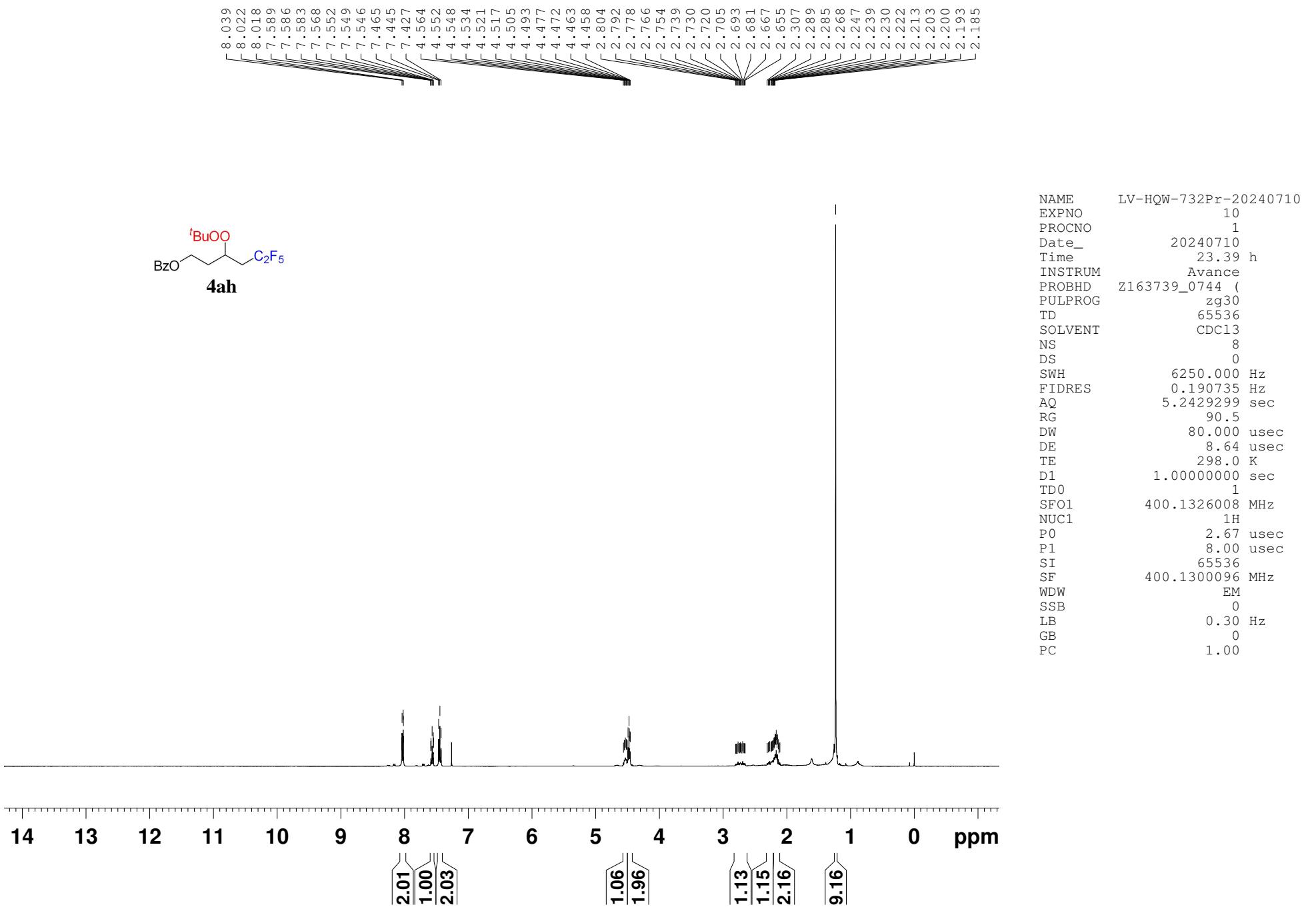
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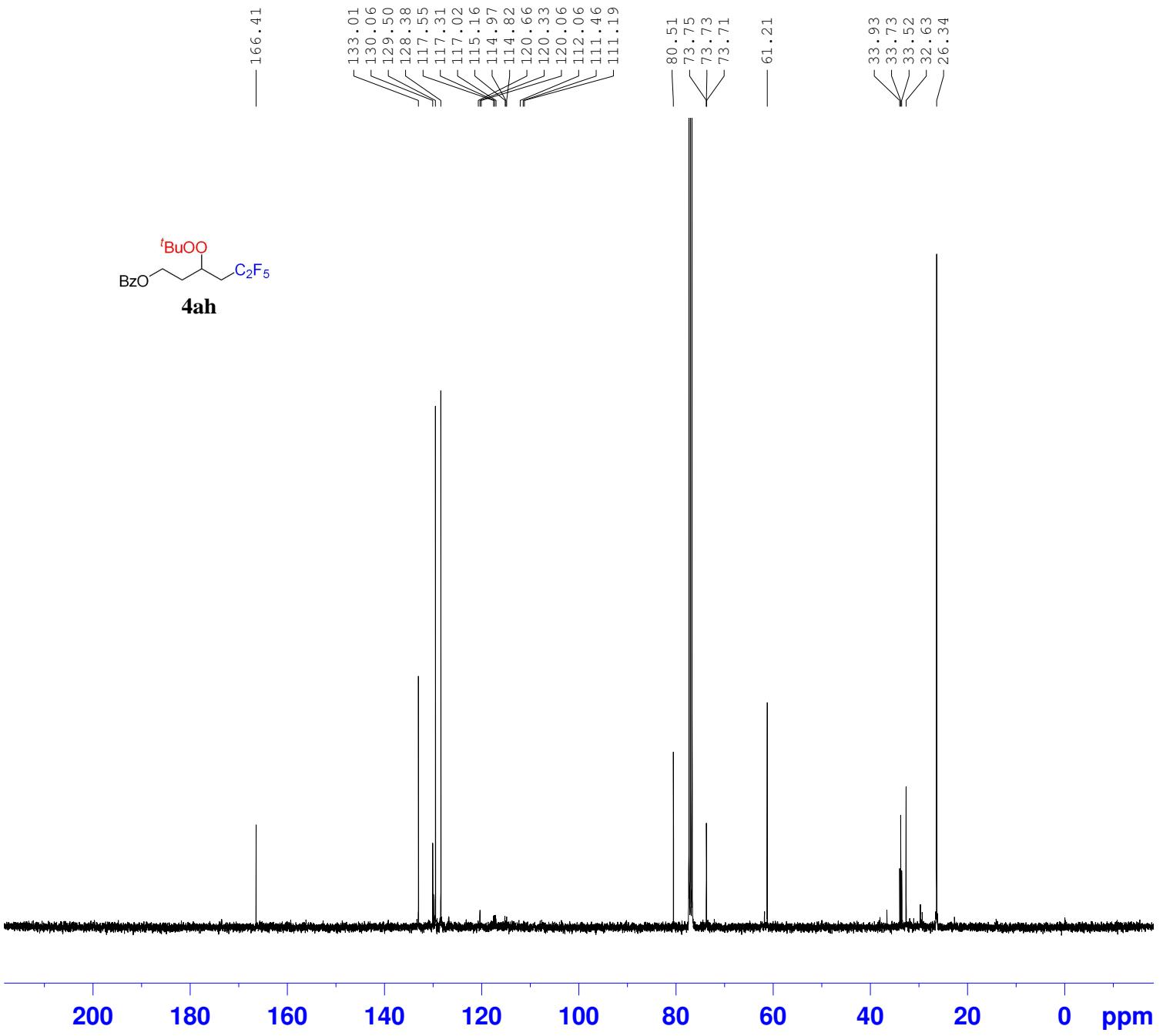




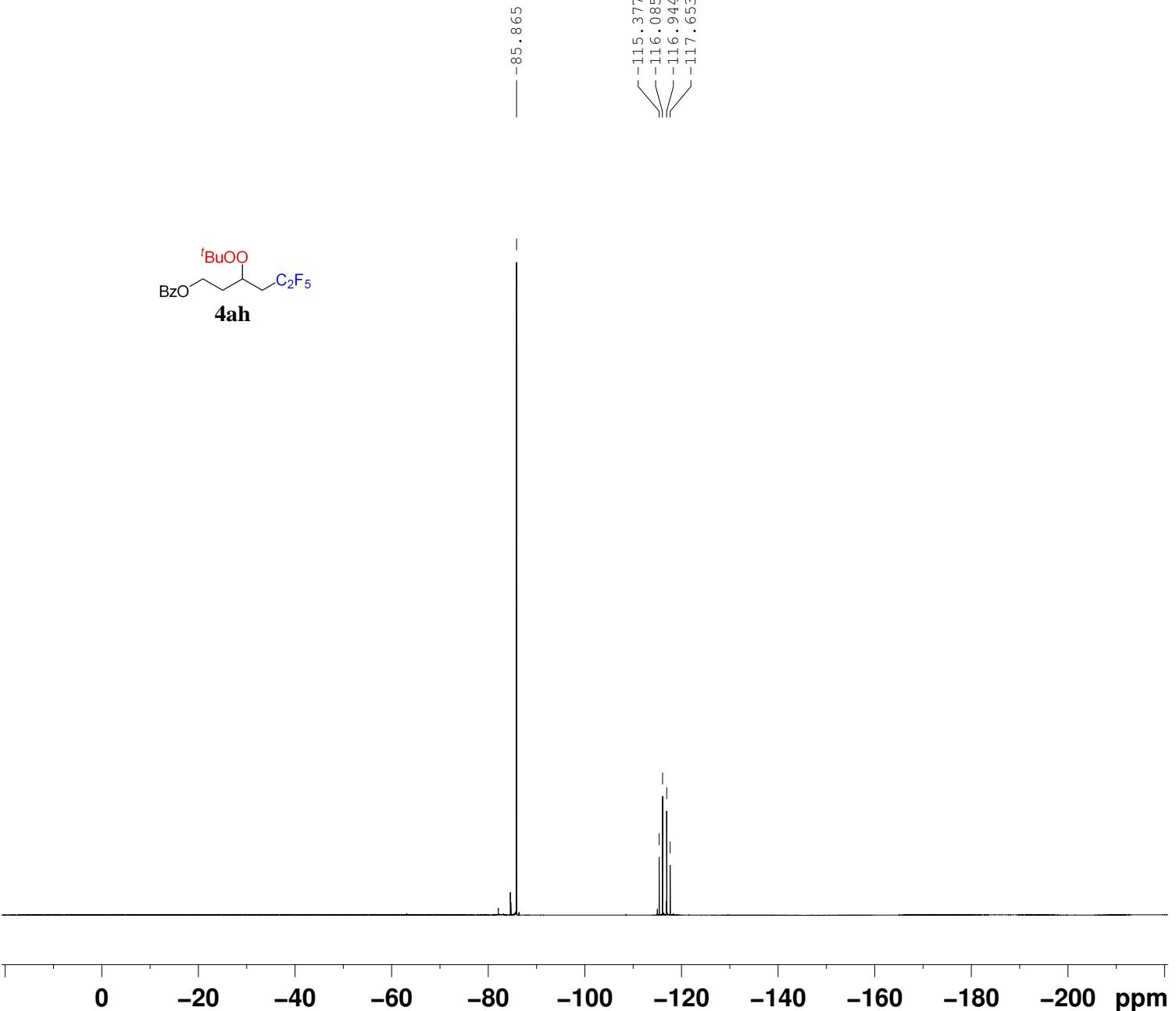
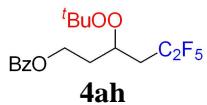
 -85.234
 -85.877
 -89.590
 -90.235







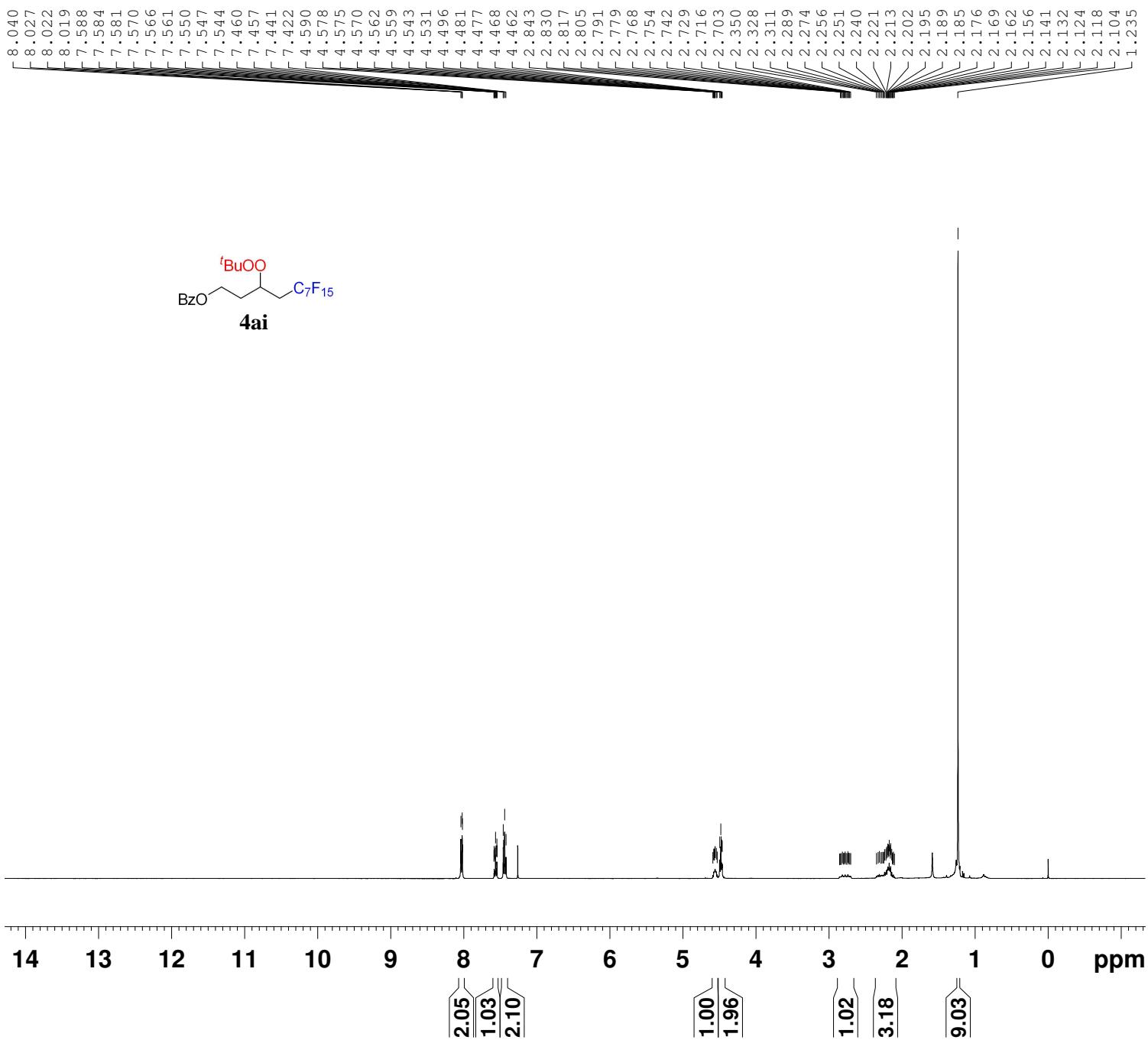
NAME	LV-HQW-732Pr-20240710
EXPNO	12
PROCNO	1
Date_	20240711
Time	0.28 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl ₃
NS	800
DS	4
SWH	23809.523 Hz
FIDRES	0.726609 Hz
AQ	1.3763061 sec
RG	101
DW	21.000 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6228298 MHz
NUC1	¹³ C
P0	2.67 usec
P1	8.00 usec
SI	32768
SF	100.6127709 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



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NAME      LV-HQW-732Pr-20240710
EXPNO     11
PROCNO    1
Date_   20240710
Time    23.41 h
INSTRUM Avance
PROBHD Z163739_0744 (
PULPROG zgig
TD        131072
SOLVENT   CDCl3
NS         16
DS          4
SWH       90909.094 Hz
FIDRES   1.387163 Hz
AQ        0.7209460 sec
RG        101
DW        5.500 usec
DE        6.50 usec
TE        298.1 K
D1        1.0000000 sec
D11       0.0300000 sec
TD0        1
SF01      376.4607164 MHz
NUC1      19F
P1        12.00 usec
SI        65536
SF        376.4983662 MHz
WDW
SSB
LB        0.30 Hz
GB
PC        1.00

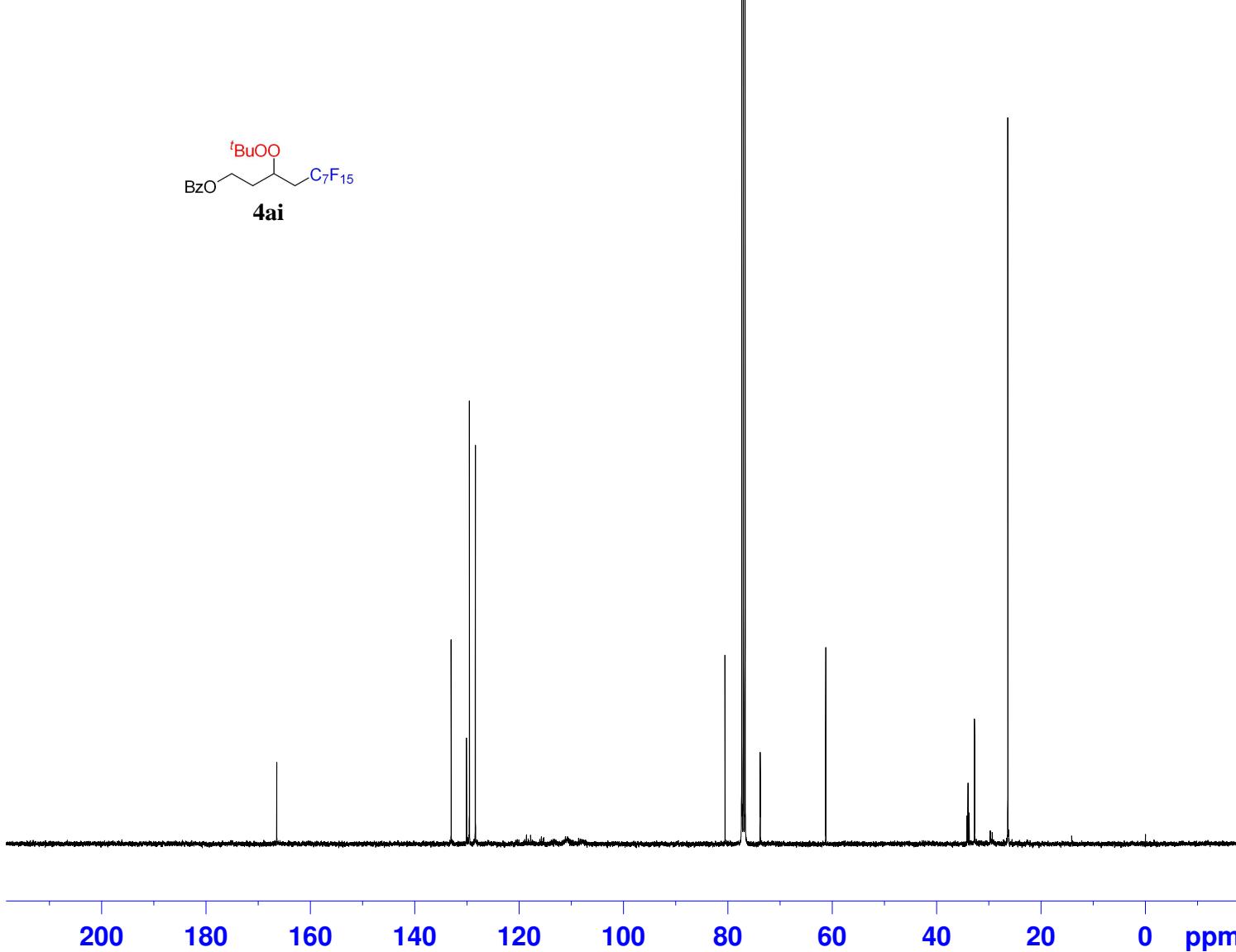
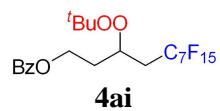
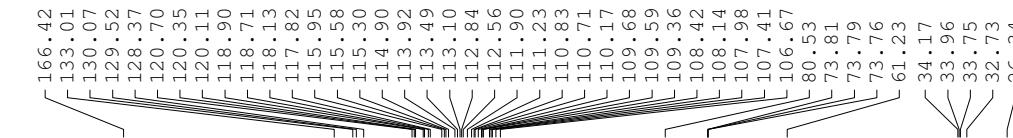
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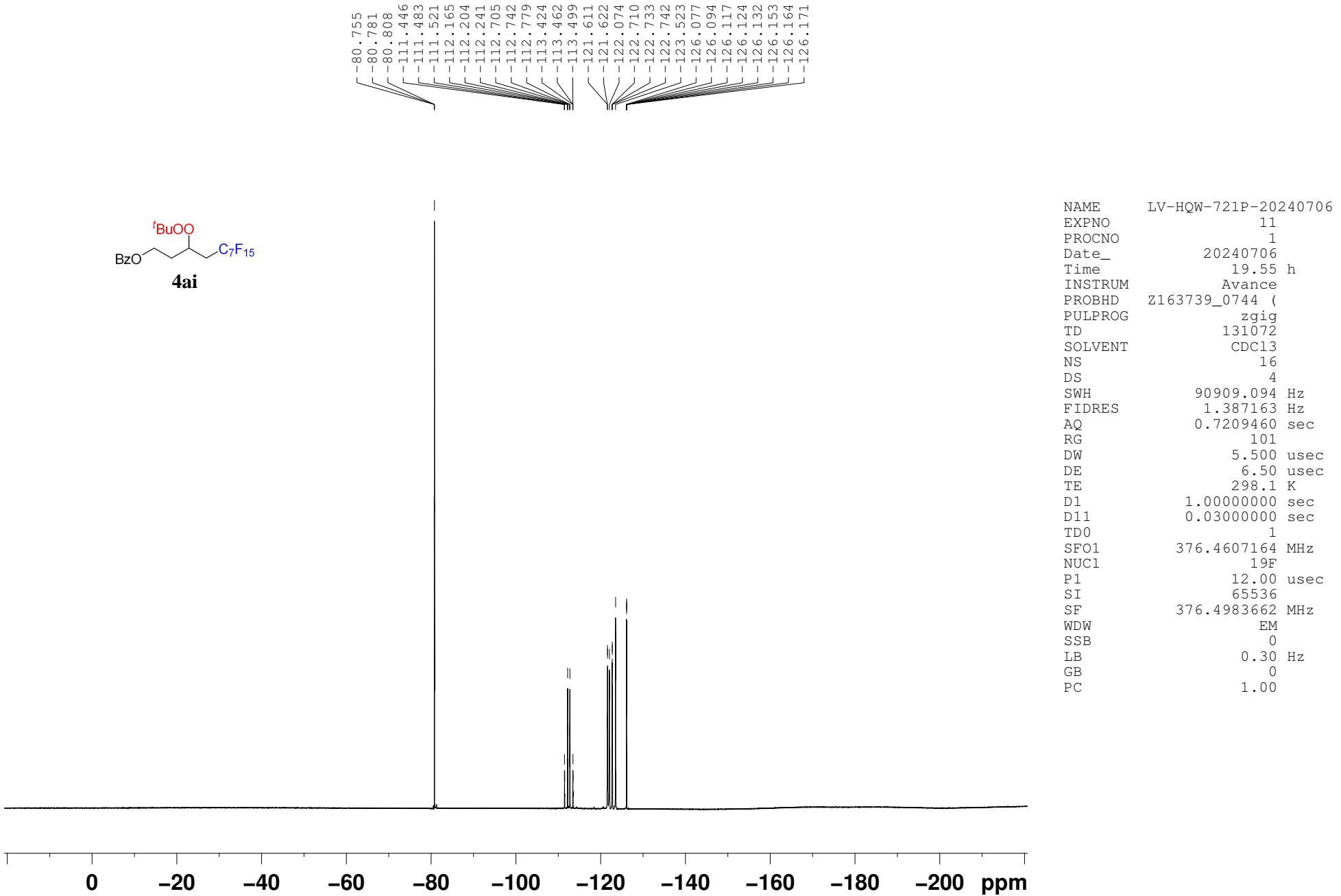


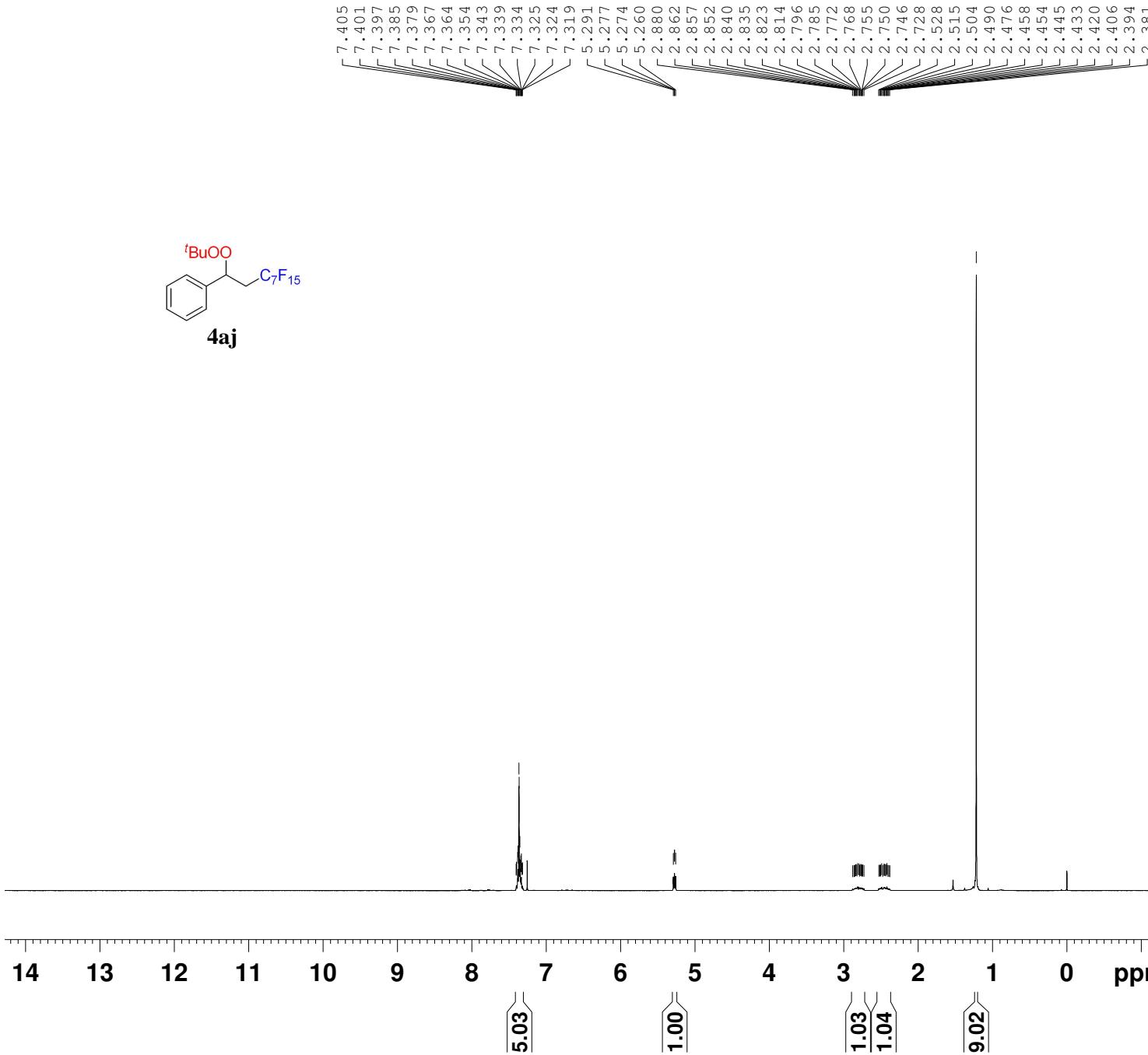
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NAME      LV-HQW-721P-20240706
EXPNO     10
PROCNO    1
Date_     20240706
Time      19.53 h
INSTRUM   Avance
PROBHD   Z163739_0744 (
PULPROG  zg30
TD        65536
SOLVENT   CDC13
NS         8
DS         0
SWH       6250.000 Hz
FIDRES   0.190735 Hz
AQ        5.2429299 sec
RG        101
DW        80.000 usec
DE        8.64 usec
TE        298.0 K
D1        1.0000000 sec
TD0       1
SFO1     400.1326008 MHz
NUC1      1H
P0        2.67 usec
P1        8.00 usec
SI        65536
SF        400.1300097 MHz
WDW      EM
SSB      0
LB        0.30 Hz
GB      0
PC        1.00

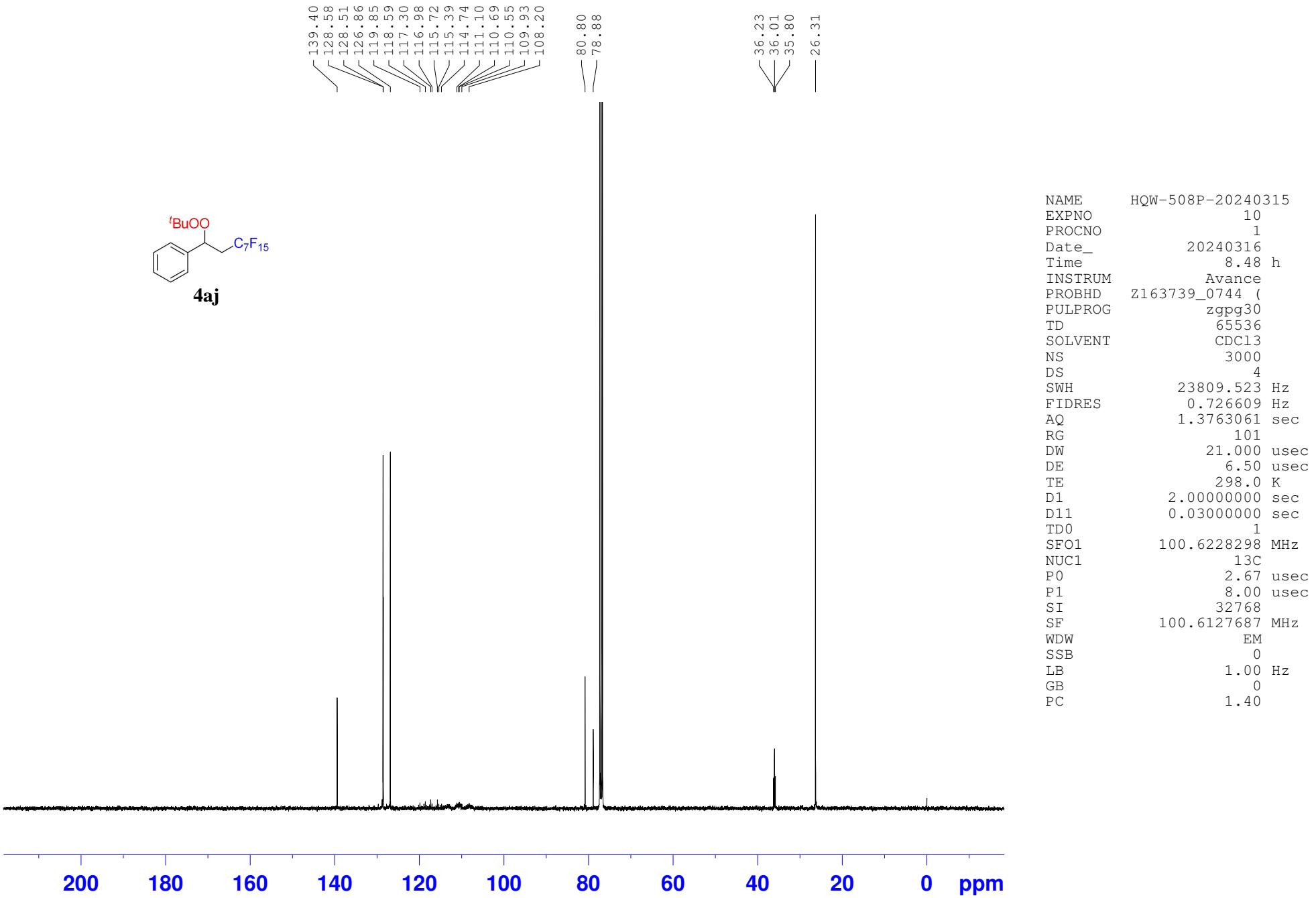
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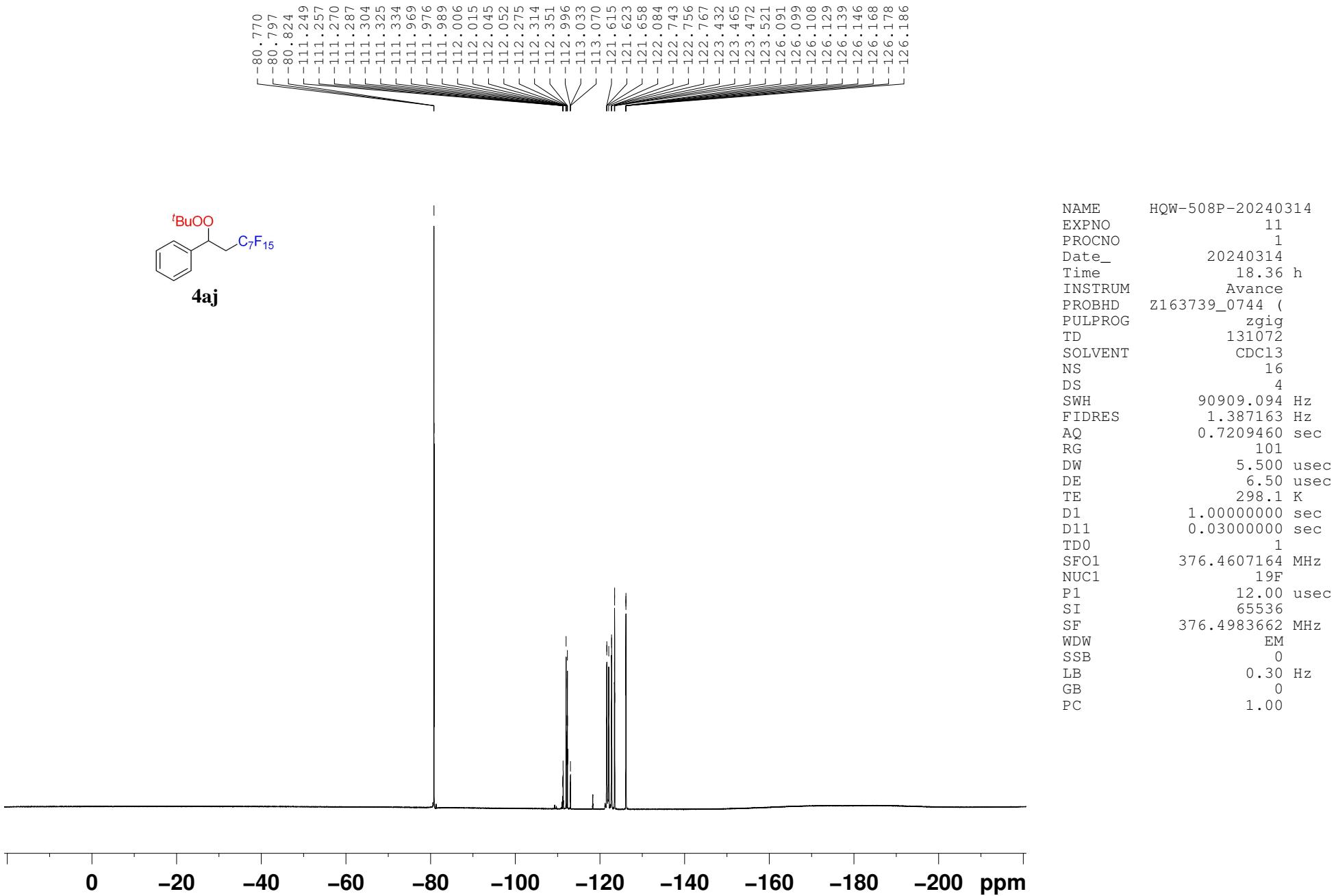


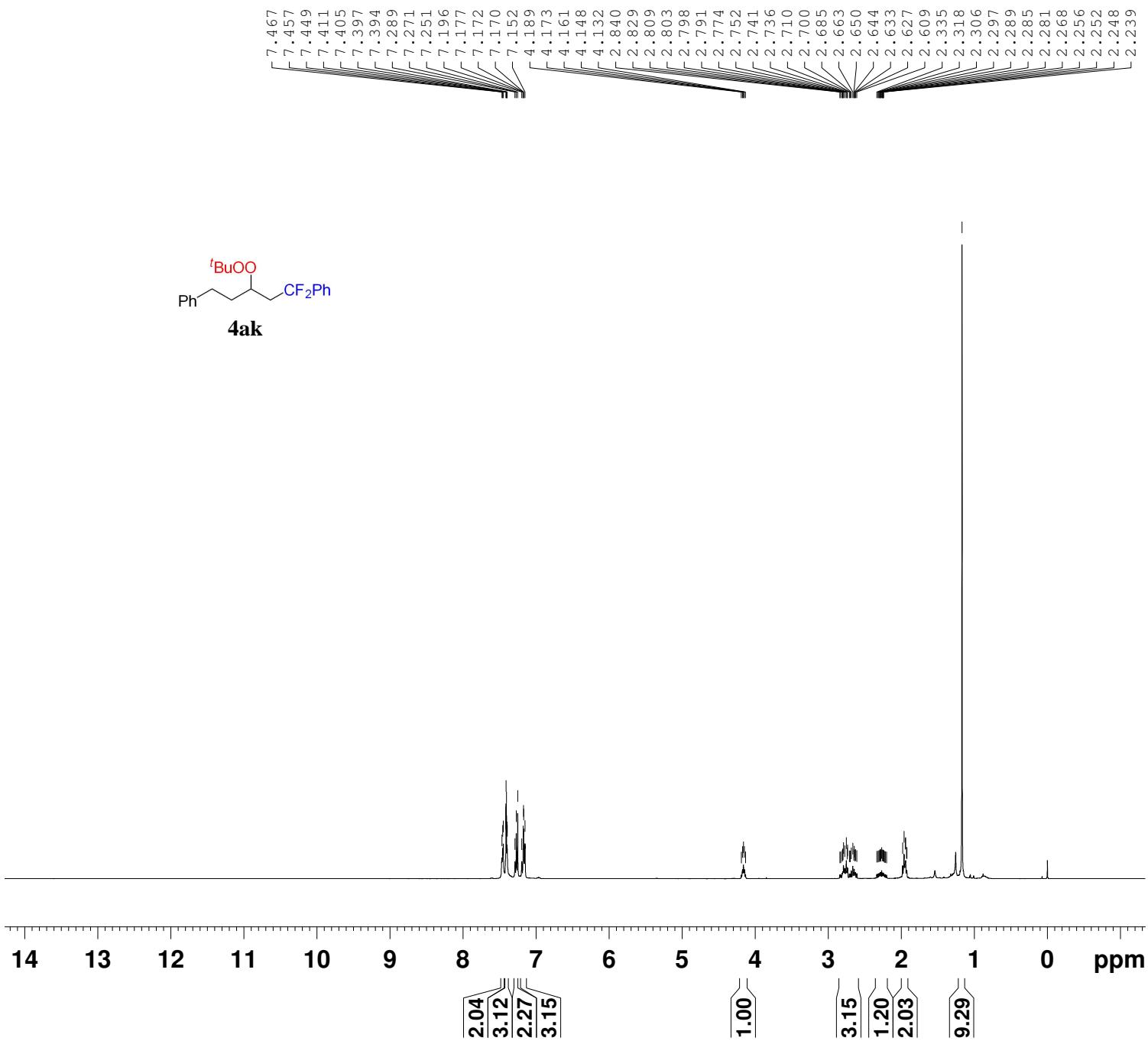




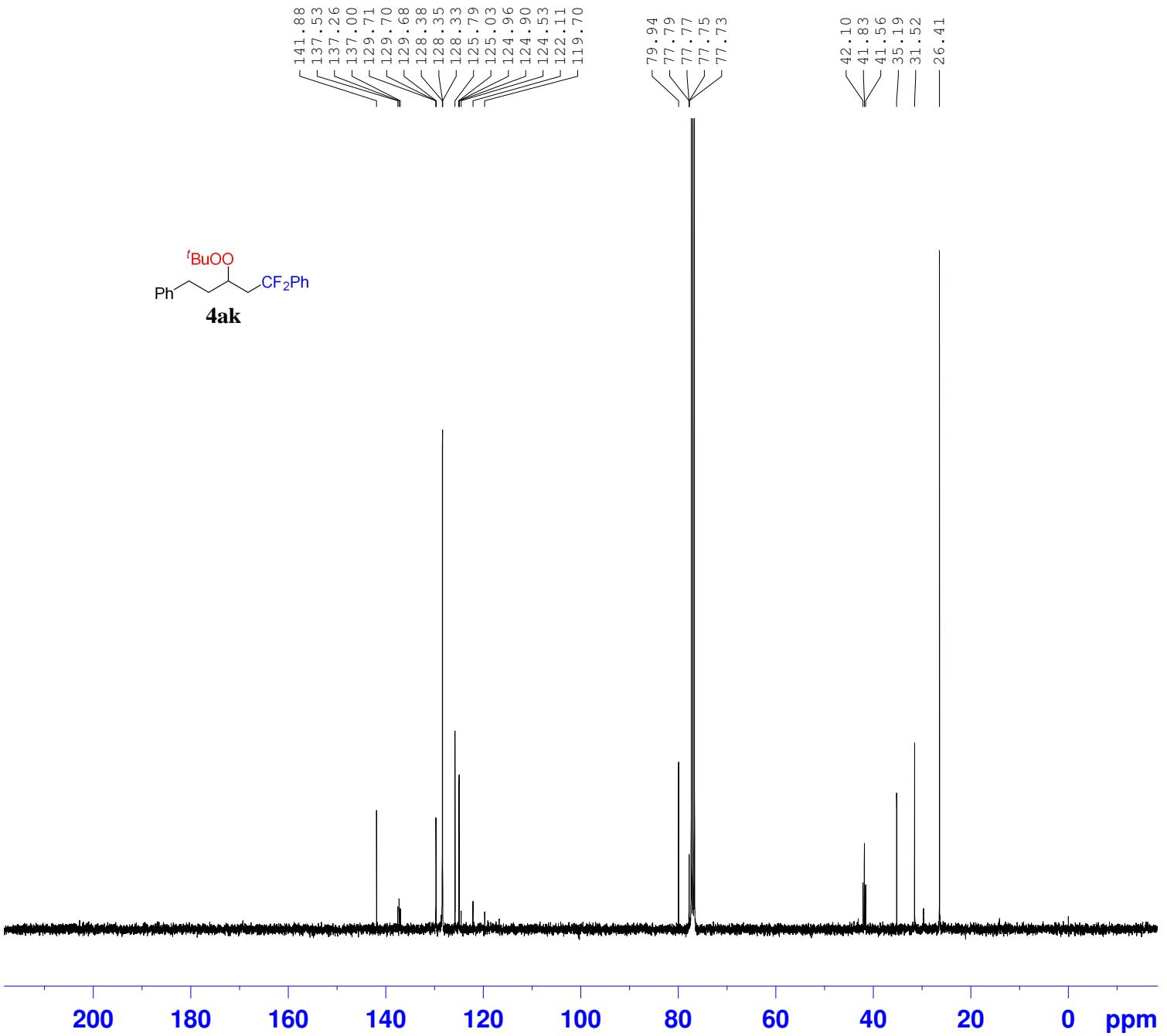
NAME HQW-508P-20240314
 EXPNO 10
 PROCNO 1
 Date_ 20240314
 Time 18.34 h
 INSTRUM Avance
 PROBHD Z163739_0744 (
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 8
 DS 0
 SWH 6250.000 Hz
 FIDRES 0.190735 Hz
 AQ 5.2429299 sec
 RG 101
 DW 80.000 usec
 DE 8.64 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1
 SFO1 400.1326008 MHz
 NUC1 1H
 P0 2.67 usec
 P1 8.00 usec
 SI 65536
 SF 400.1300121 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



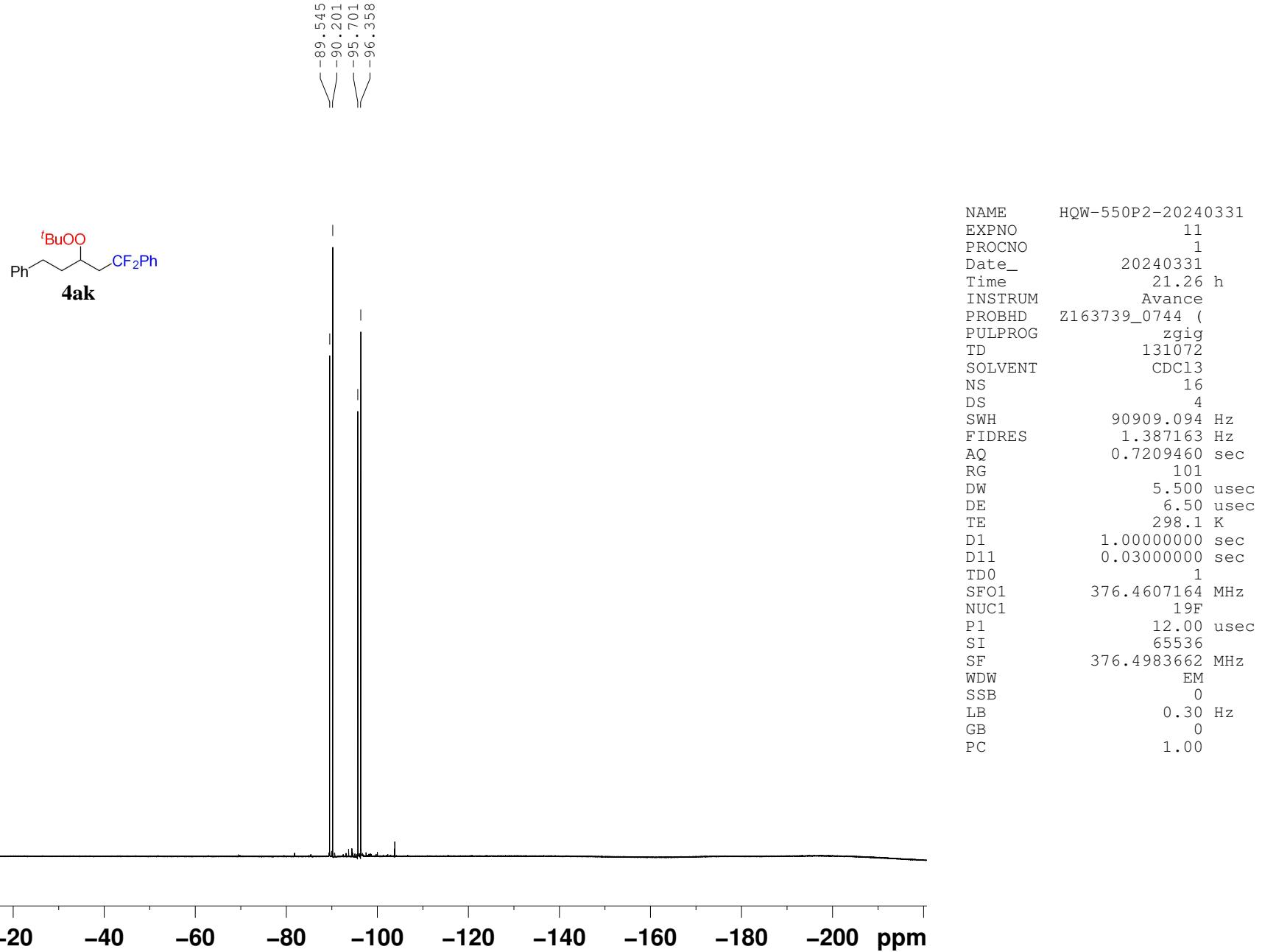


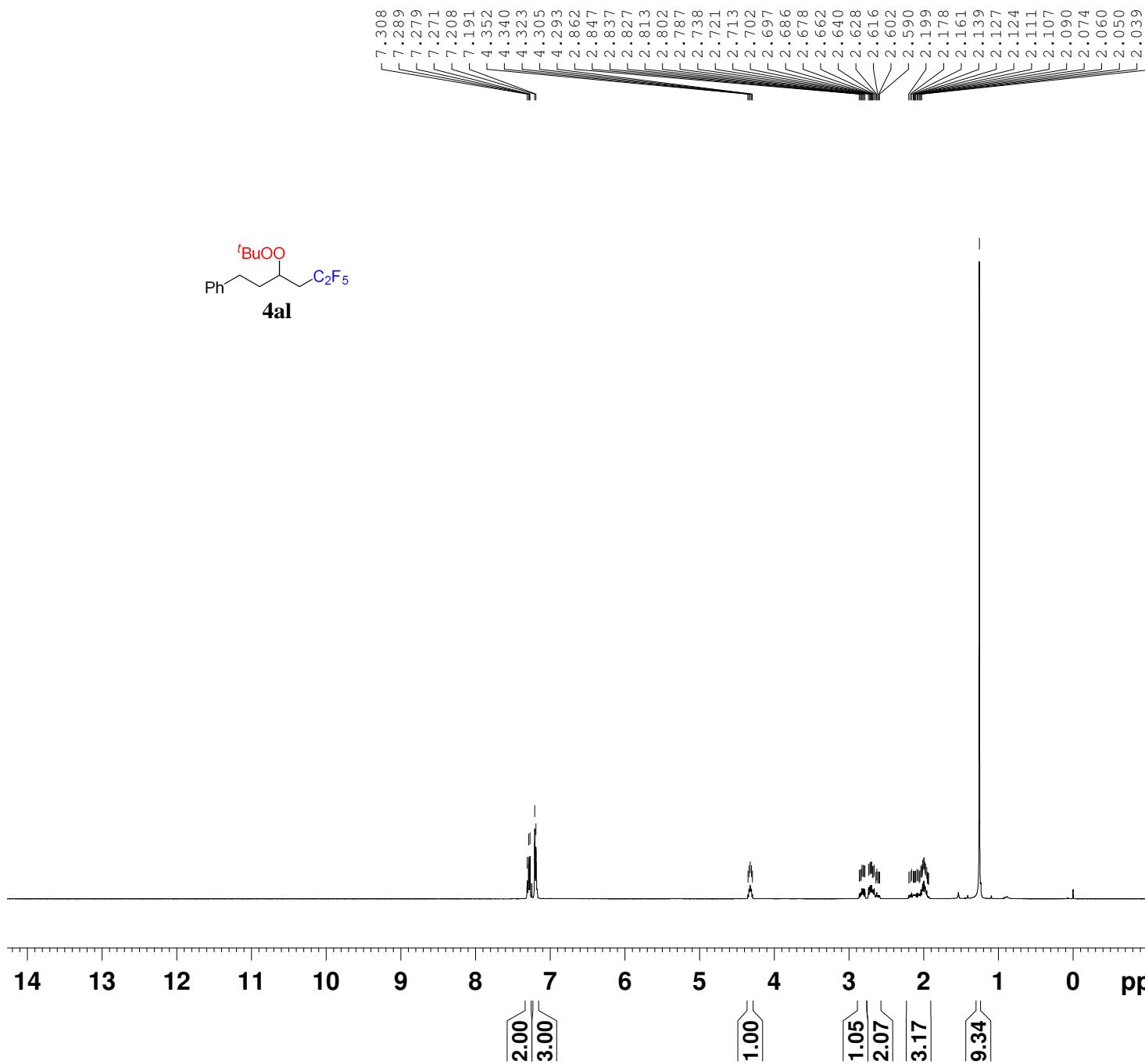


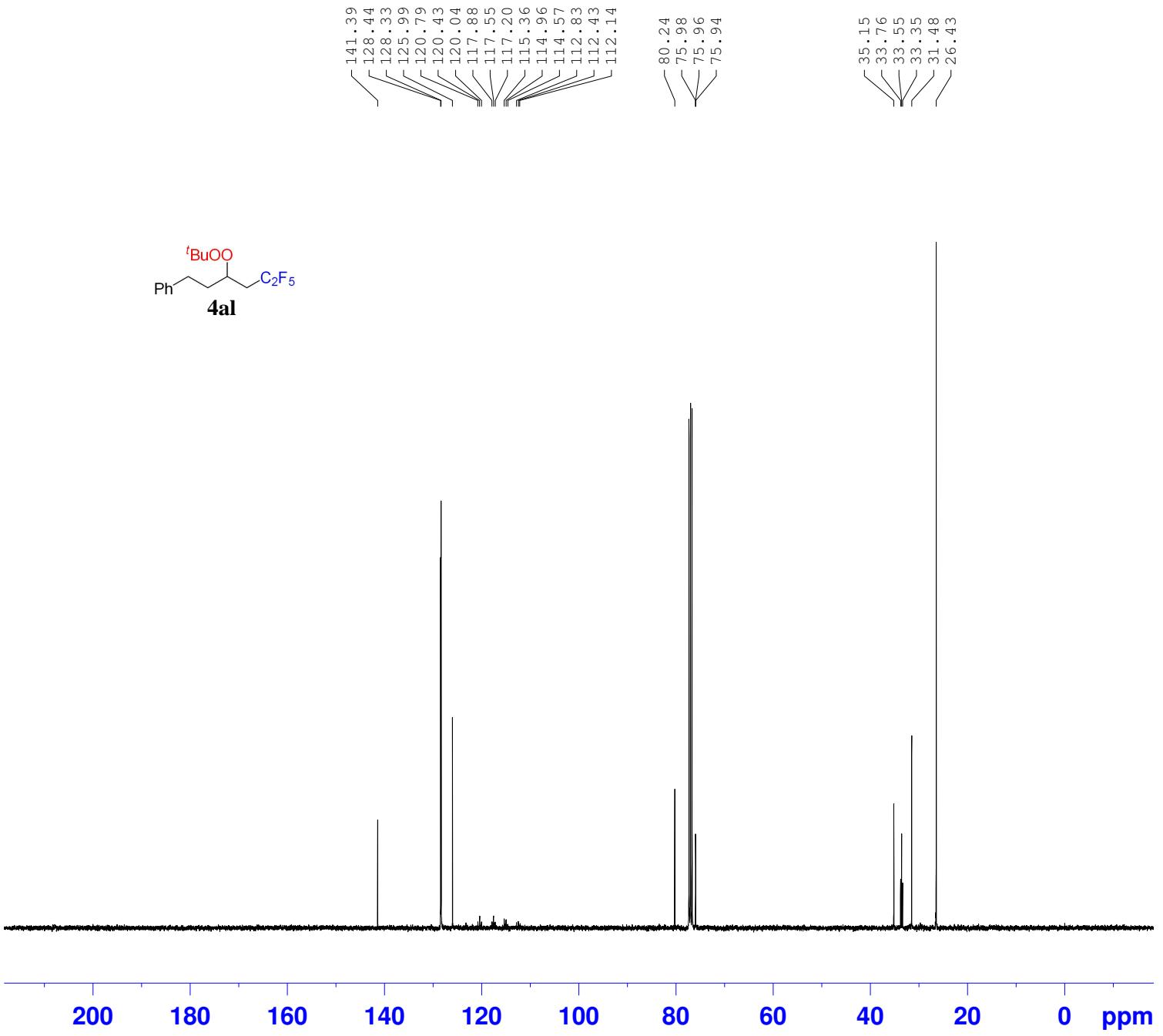
NAME HQW-550P2-20240331
EXPNO 10
PROCNO 1
Date_ 20240331
Time 21.24 h
INSTRUM Avance
PROBHD Z163739_0744 (zg30
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 6250.000 Hz
FIDRES 0.190735 Hz
AQ 5.2429299 sec
RG 101
DW 80.000 usec
DE 8.64 usec
TE 298.0 K
D1 1.0000000 sec
TD0 1
SFO1 400.1326008 MHz
NUC1 1H
P0 2.67 usec
P1 8.00 usec
SI 65536
SF 400.1300139 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

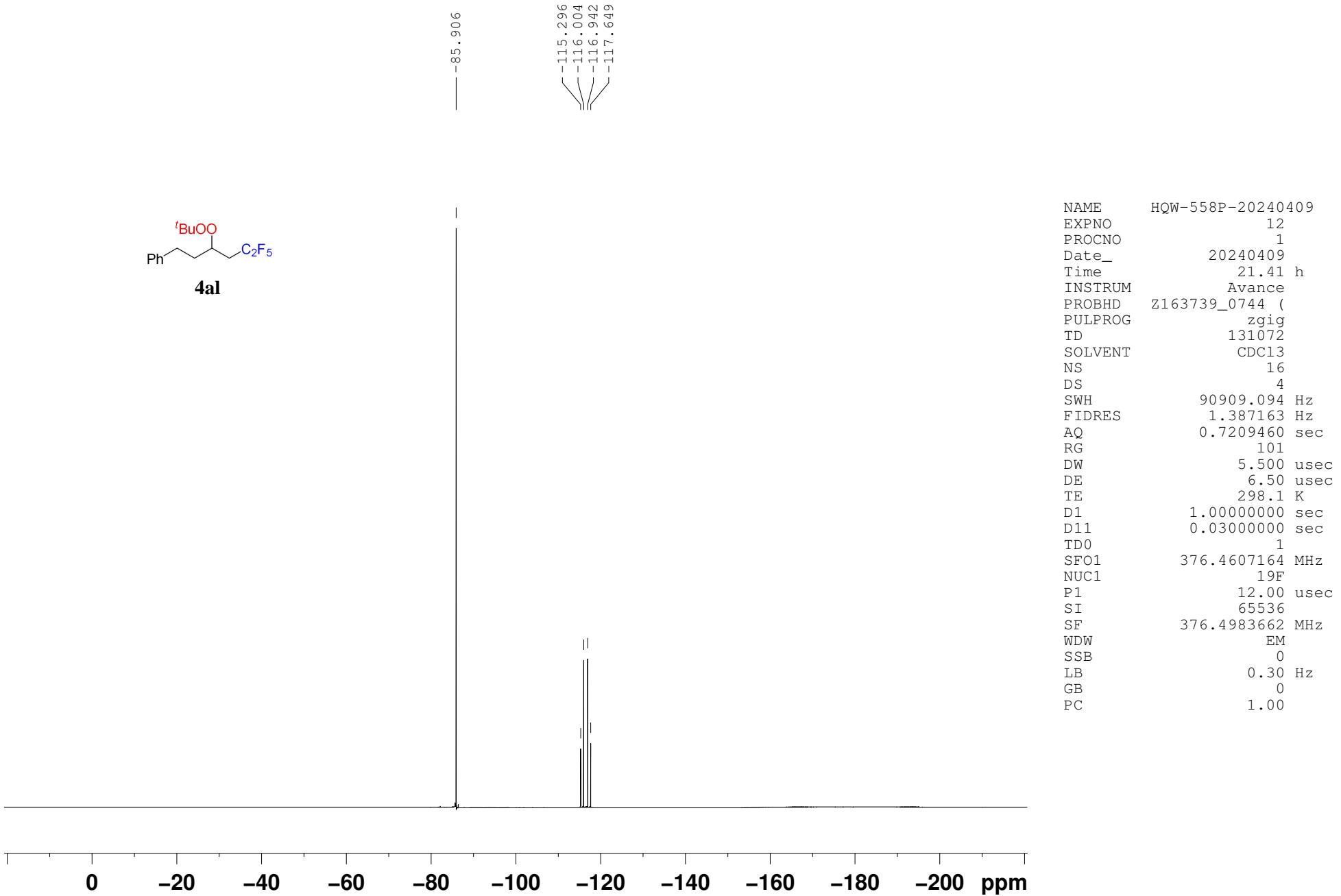


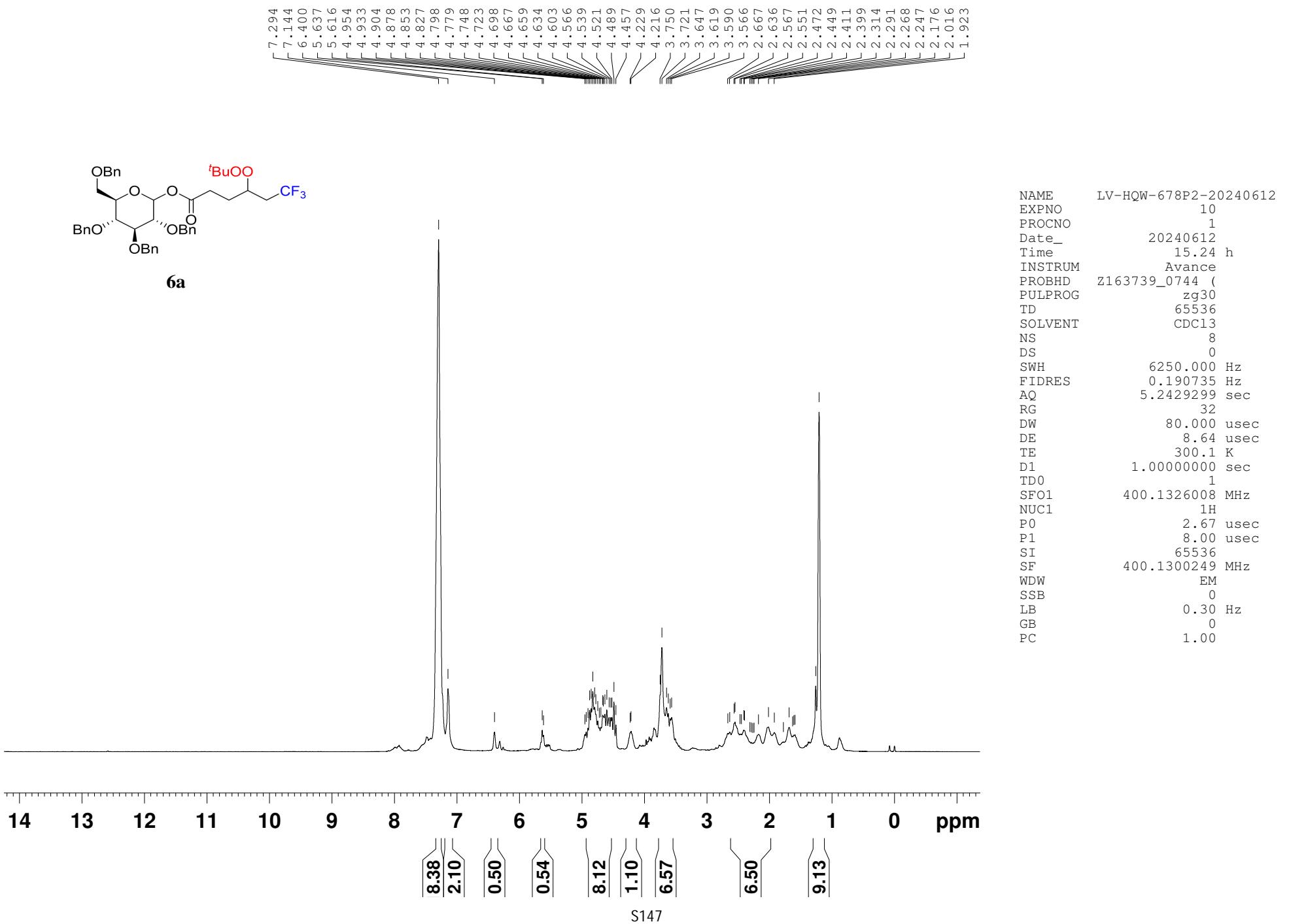
NAME	HQW-550P2-20240331
EXPNO	12
PROCNO	1
Date_	20240331
Time	22.10 h
INSTRUM	Avance
PROBHD	z163739_0744 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl ₃
NS	744
DS	4
SWH	23809.523 Hz
FIDRES	0.726609 Hz
AO	1.3763061 sec
RG	101
DW	21.000 usec
DE	6.50 usec
TE	298.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TDO	1
SFO1	100.6228298 MHz
NUC1	¹³ C
P0	2.67 usec
P1	8.00 usec
SI	32768
SF	100.6127717 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

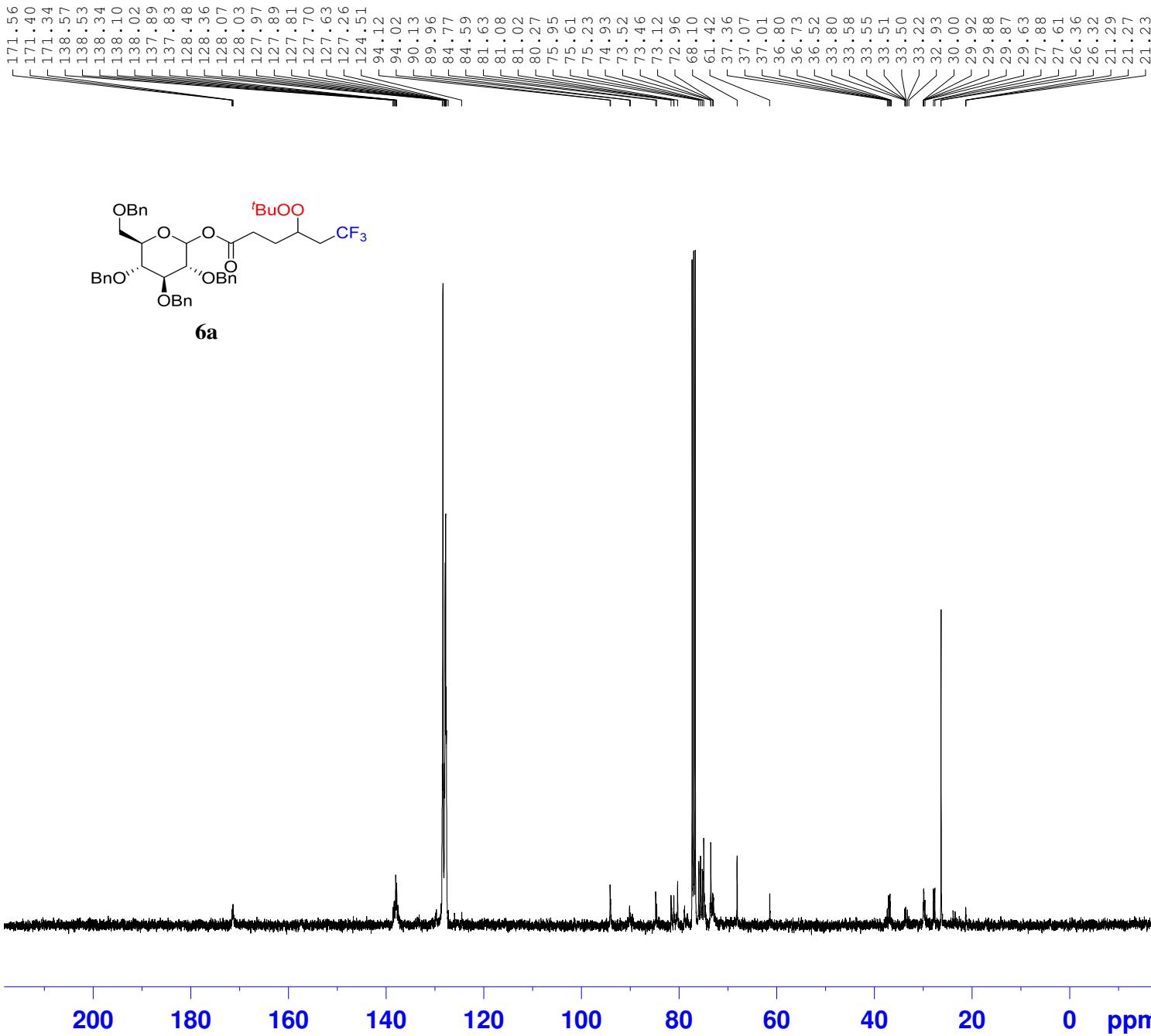


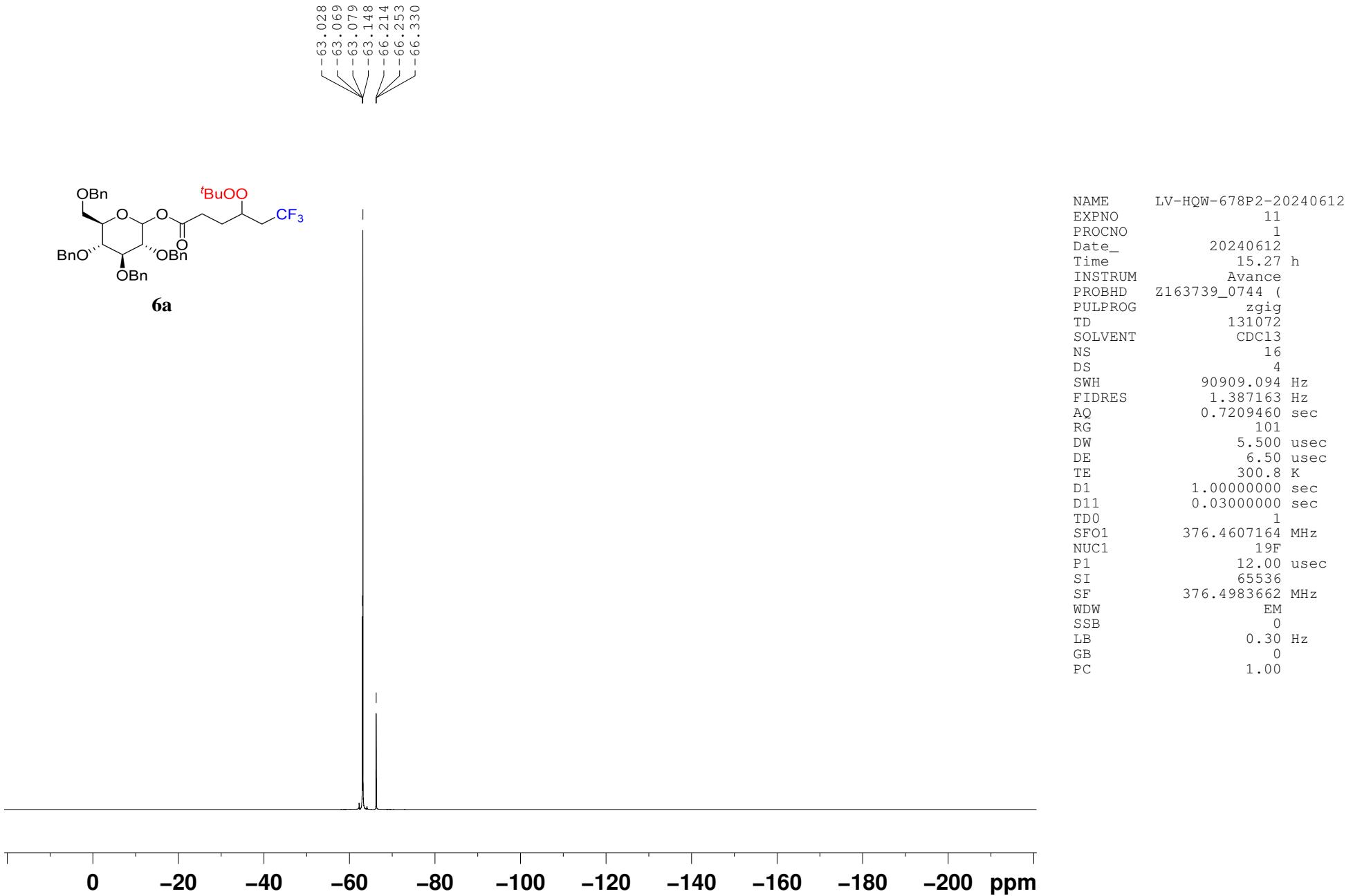


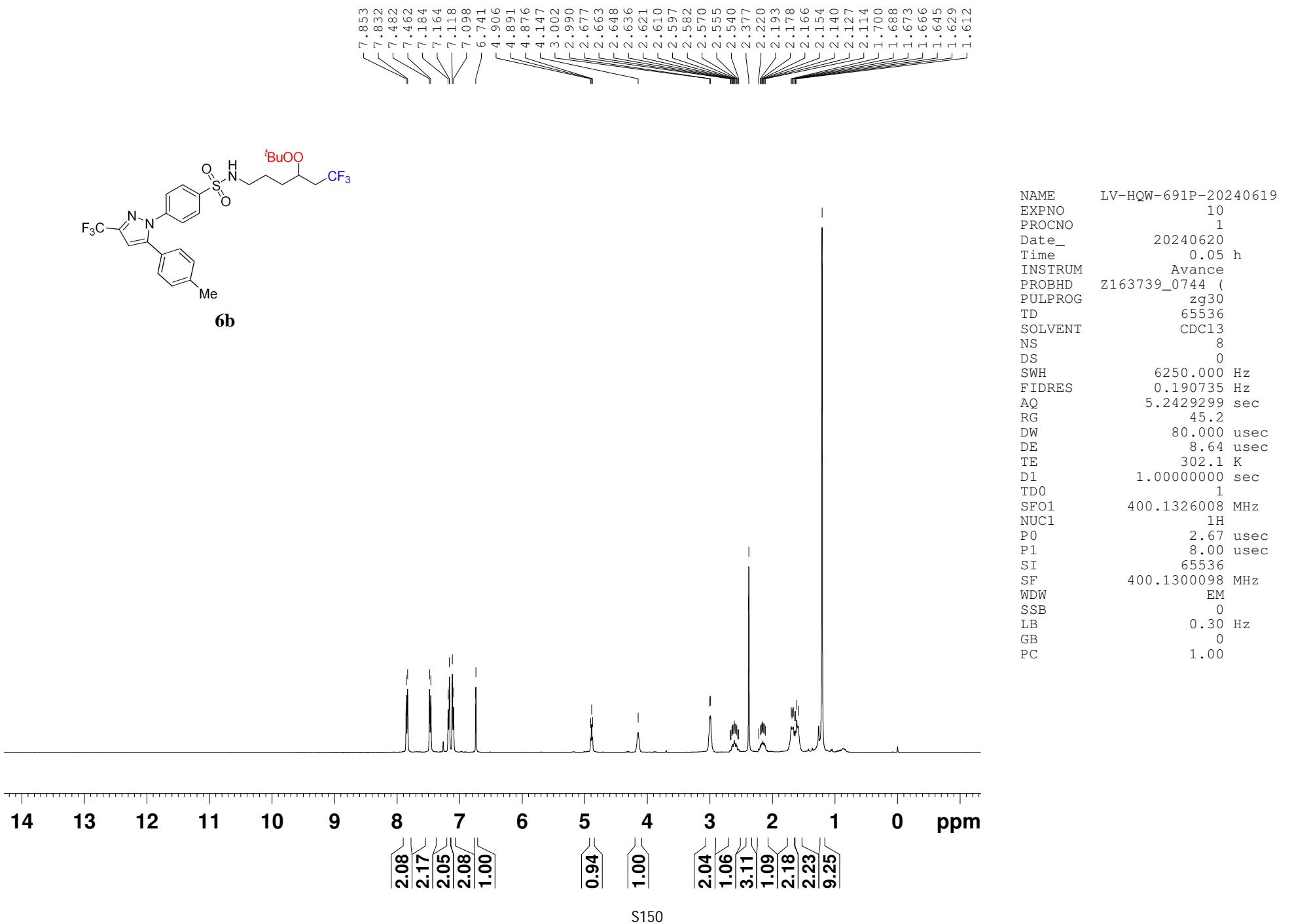


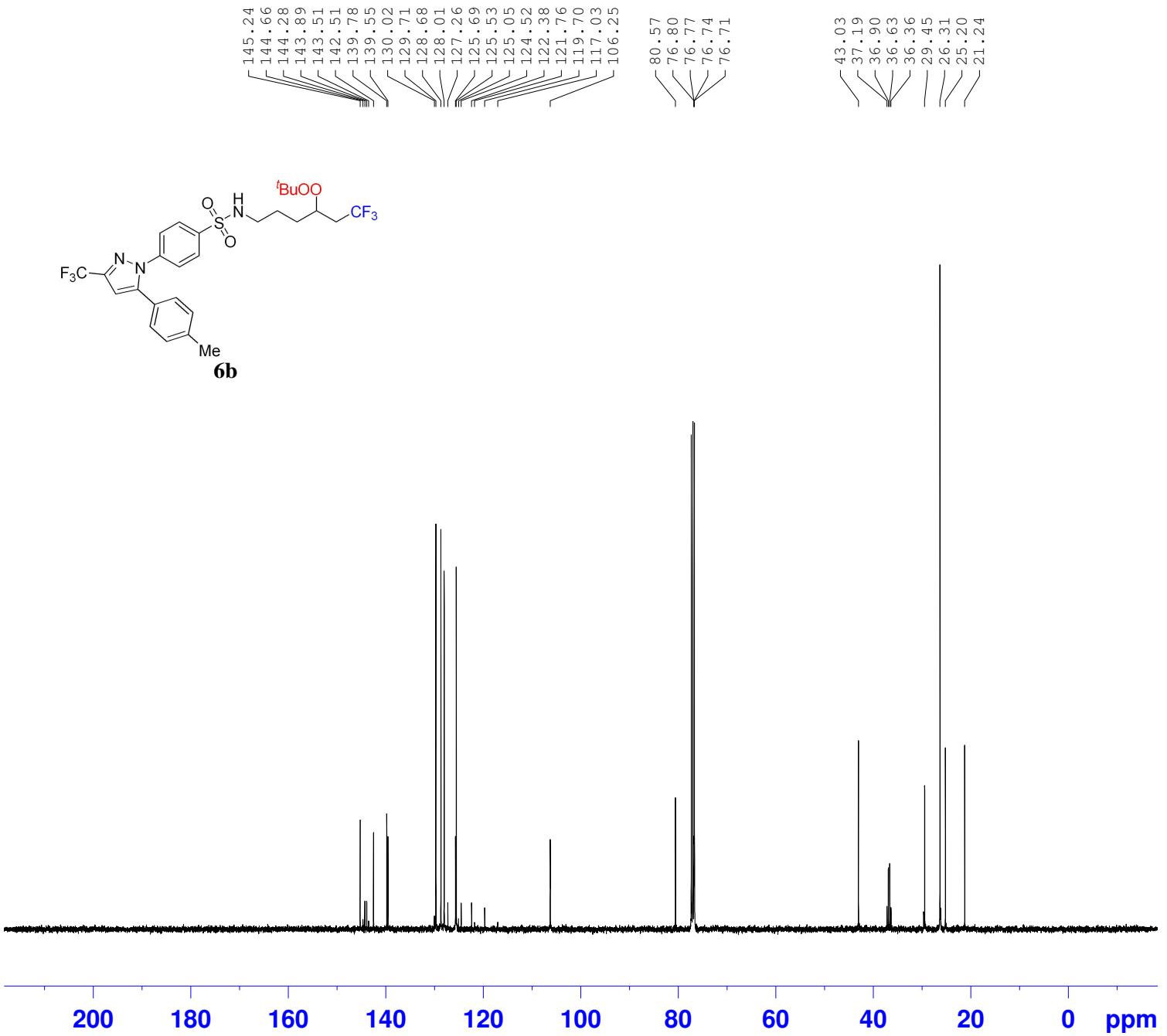




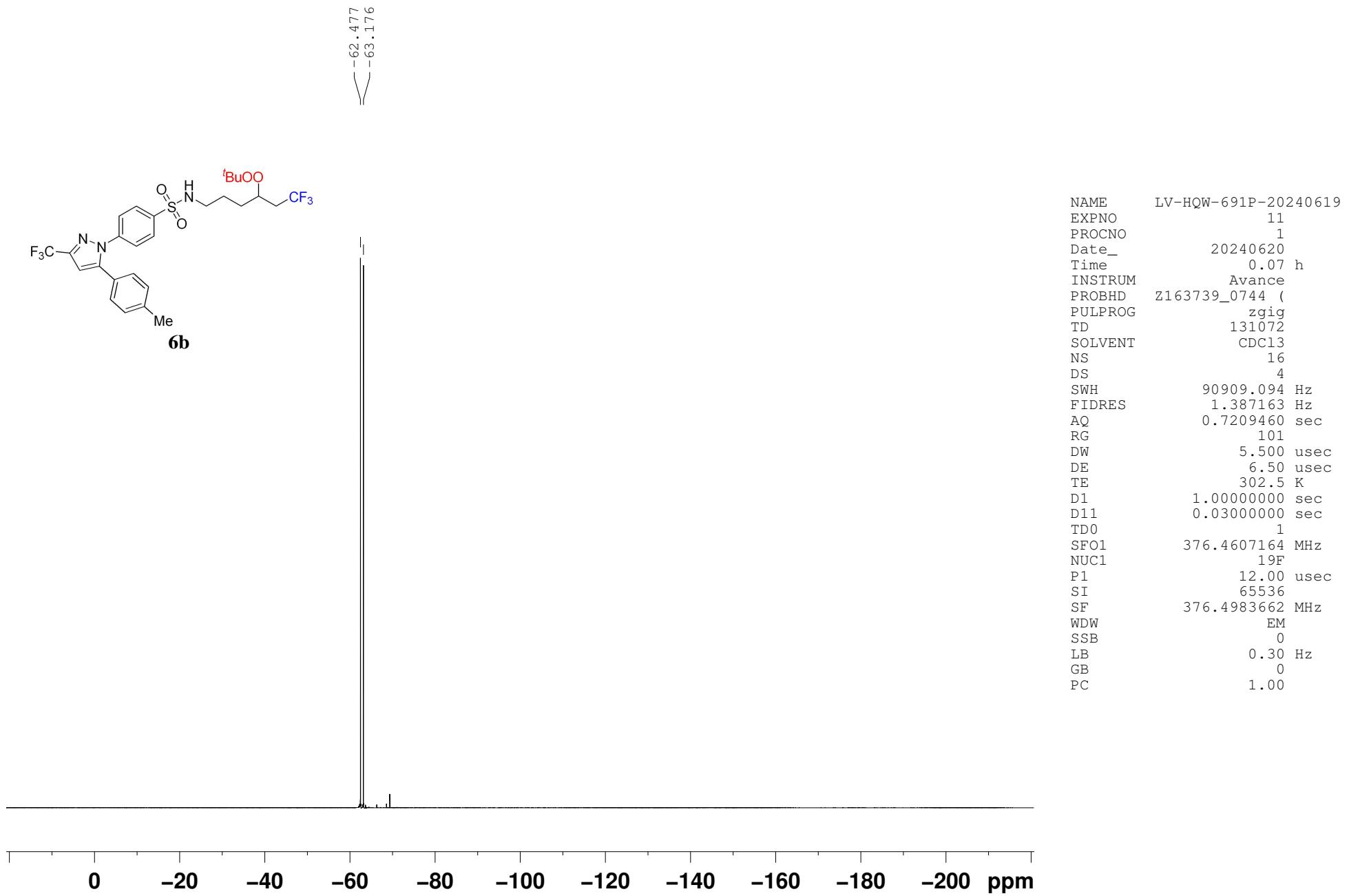


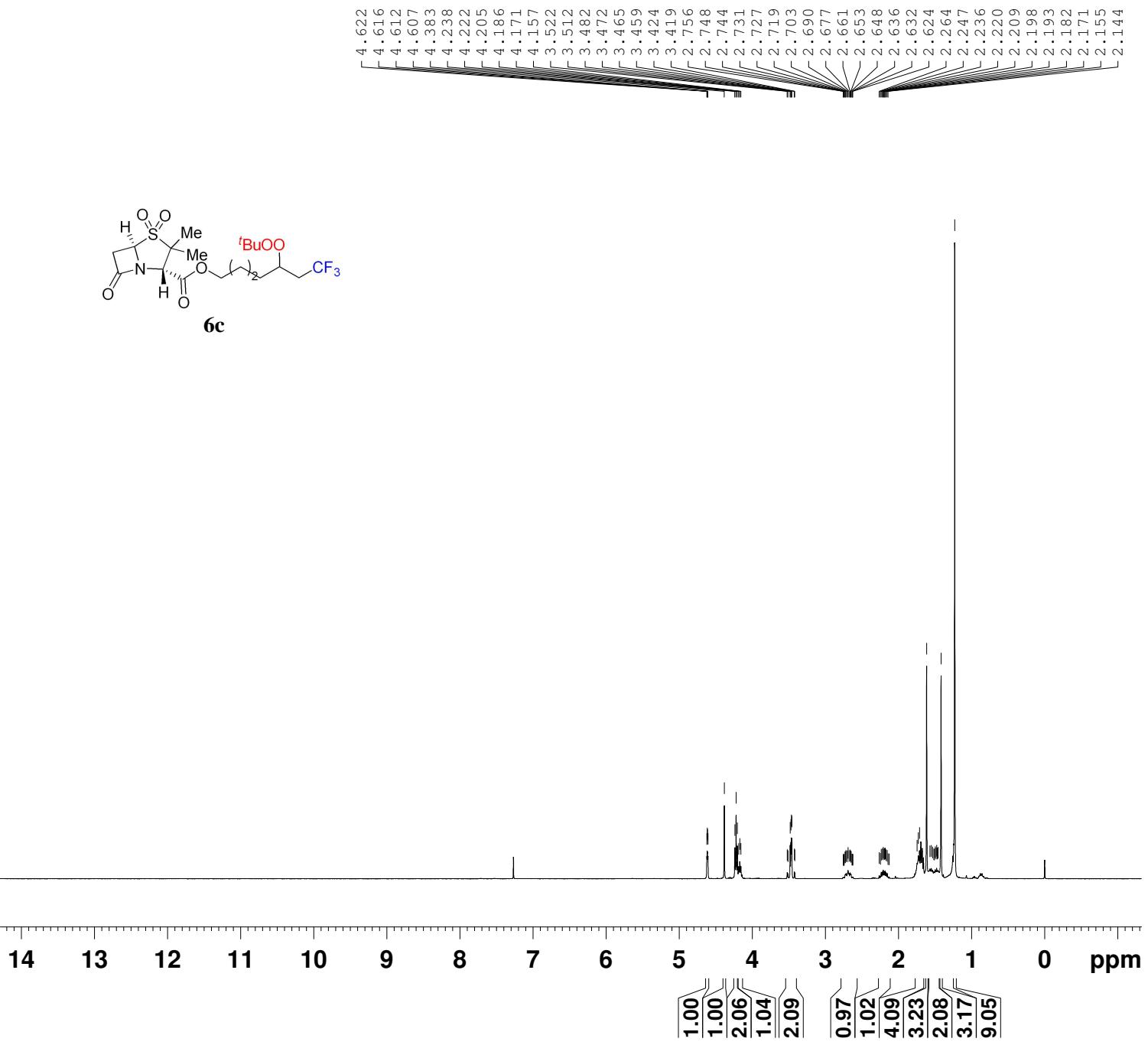




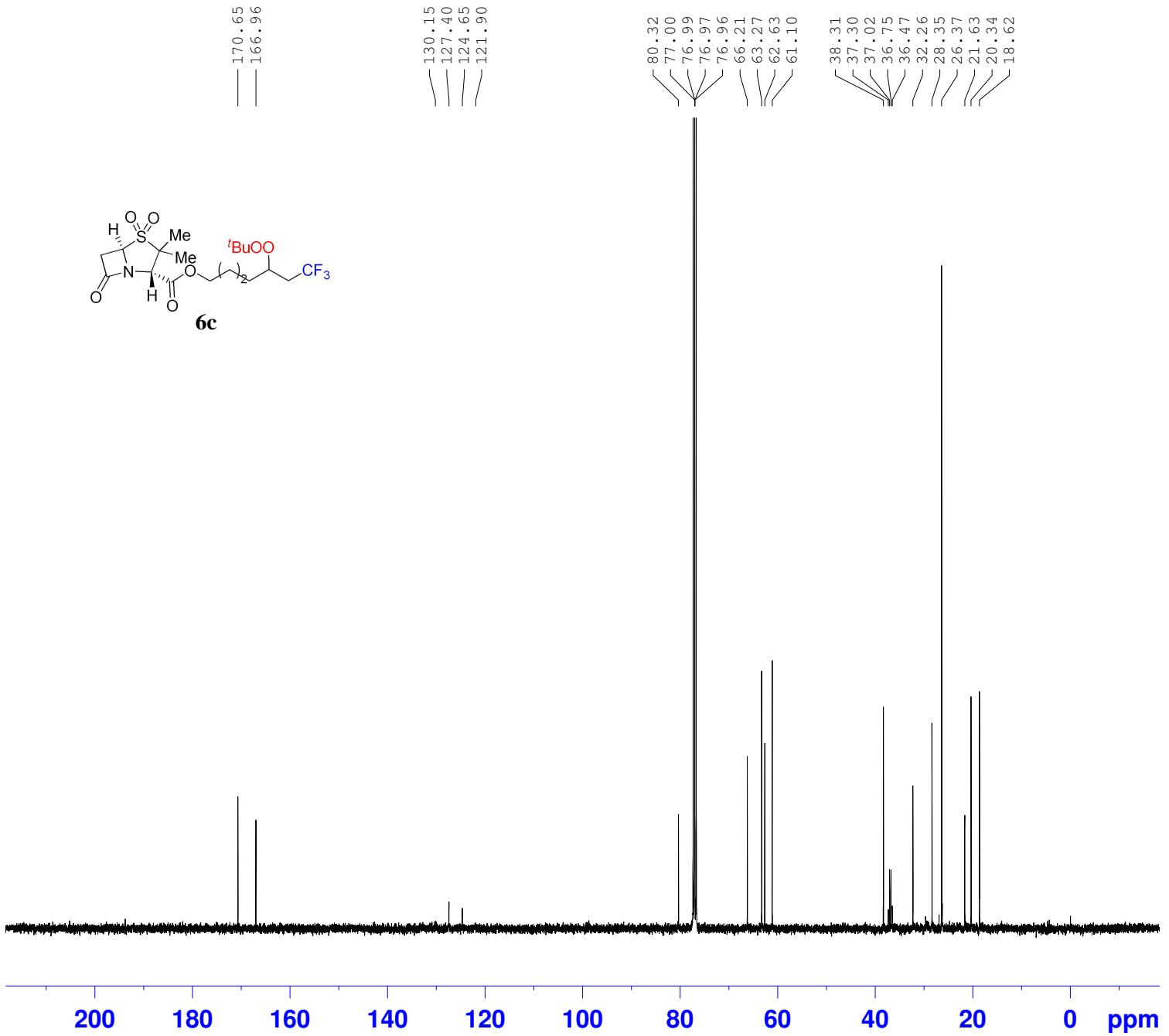
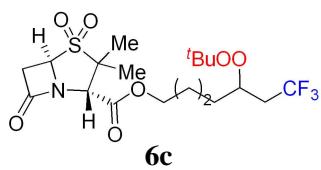


NAME LV-HQW-691P-20240619
 EXPNO 12
 PROCNO 1
 Date_ 20240620
 Time 0.43 h
 INSTRUM Avance
 PROBHD Z163739_0744 (zgpg30
 PULPROG 65536
 TD SOLVENT CDCl3
 NS 600
 DS 4
 SWH 23809.523 Hz
 FIDRES 0.726609 Hz
 AQ 1.3763061 sec
 RG 101
 DW 21.000 usec
 DE 6.50 usec
 TE 302.8 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1
 SFO1 100.6228298 MHz
 NUC1 13C
 P0 2.67 usec
 P1 8.00 usec
 SI 32768
 SF 100.6127716 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





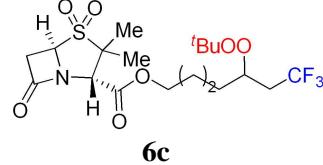
NAME LV-HQW-688P-20240616
 EXPNO 10
 PROCNO 1
 Date_ 20240617
 Time 0.33 h
 INSTRUM Avance
 PROBHD Z163739_0744 (zg30
 PULPROG 65536
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 6250.000 Hz
 FIDRES 0.190735 Hz
 AQ 5.2429299 sec
 RG 90.5
 DW 80.000 usec
 DE 8.64 usec
 TE 300.7 K
 D1 1.00000000 sec
 TDO 1
 SFO1 400.1326008 MHz
 NUC1 1H
 P0 2.67 usec
 P1 8.00 usec
 SI 65536
 SF 400.1300073 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



```

NAME      LV-HQW-688P-20240616
EXPNO     12
PROCNO    1
Date_     20240617
Time      1.17 h
INSTRUM   Avance
PROBHD   Z163739_0744 (
PULPROG  zgpg30
TD        65536
SOLVENT   CDC13
NS        600
DS        4
SWH       23809.523 Hz
FIDRES   0.726609 Hz
AQ        1.3763061 sec
RG        101
DW        21.000 usec
DE        6.50  usec
TE        301.3 K
D1        2.0000000 sec
D11       0.0300000 sec
TD0       1
SFO1      100.6228298 MHz
NUC1      13C
P0        2.67  usec
P1        8.00  usec
SI        32768
SF        100.6127709 MHz
WDW      EM
SSB      0
LB        1.00  Hz
GB      0
PC        1.40

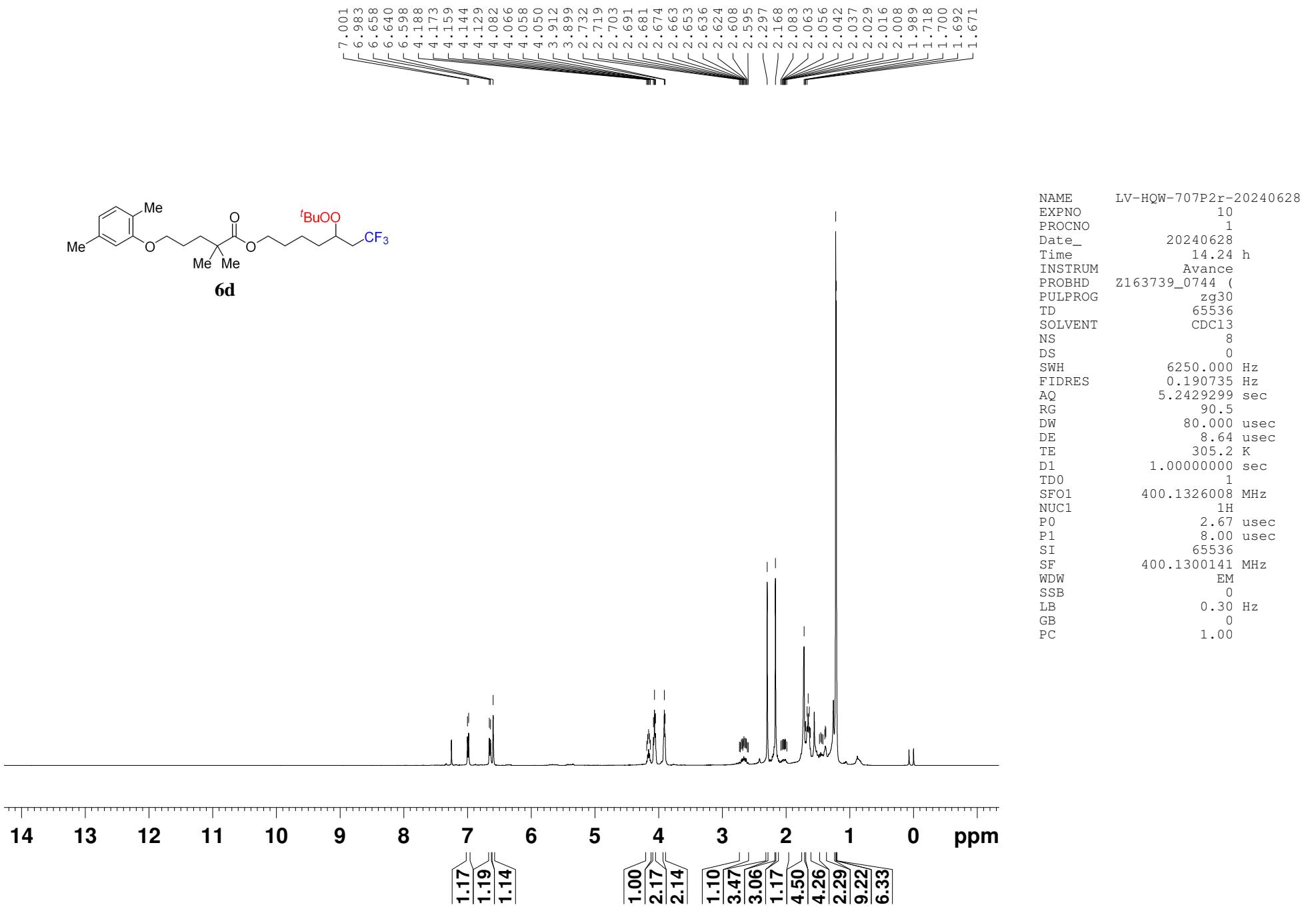
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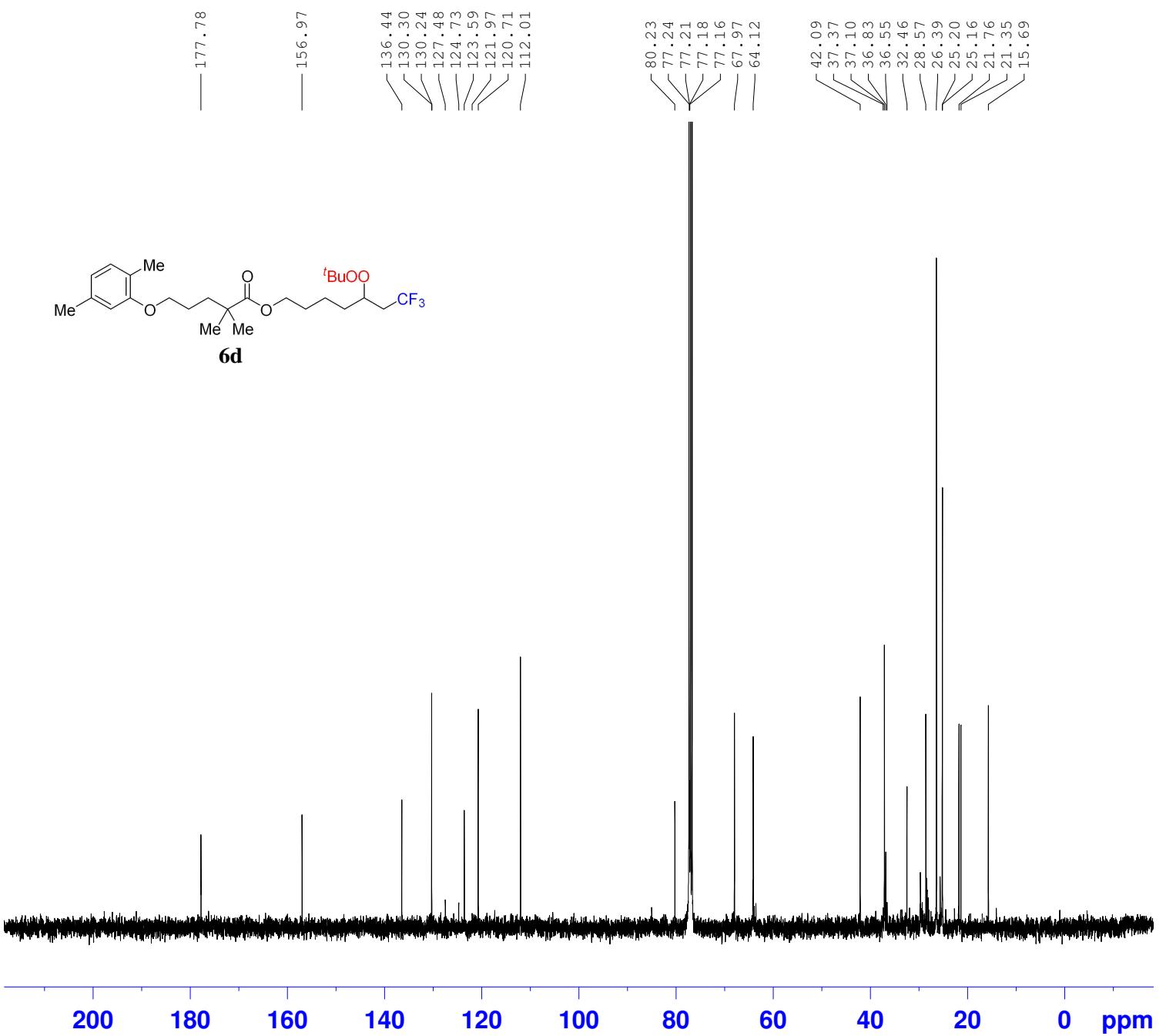


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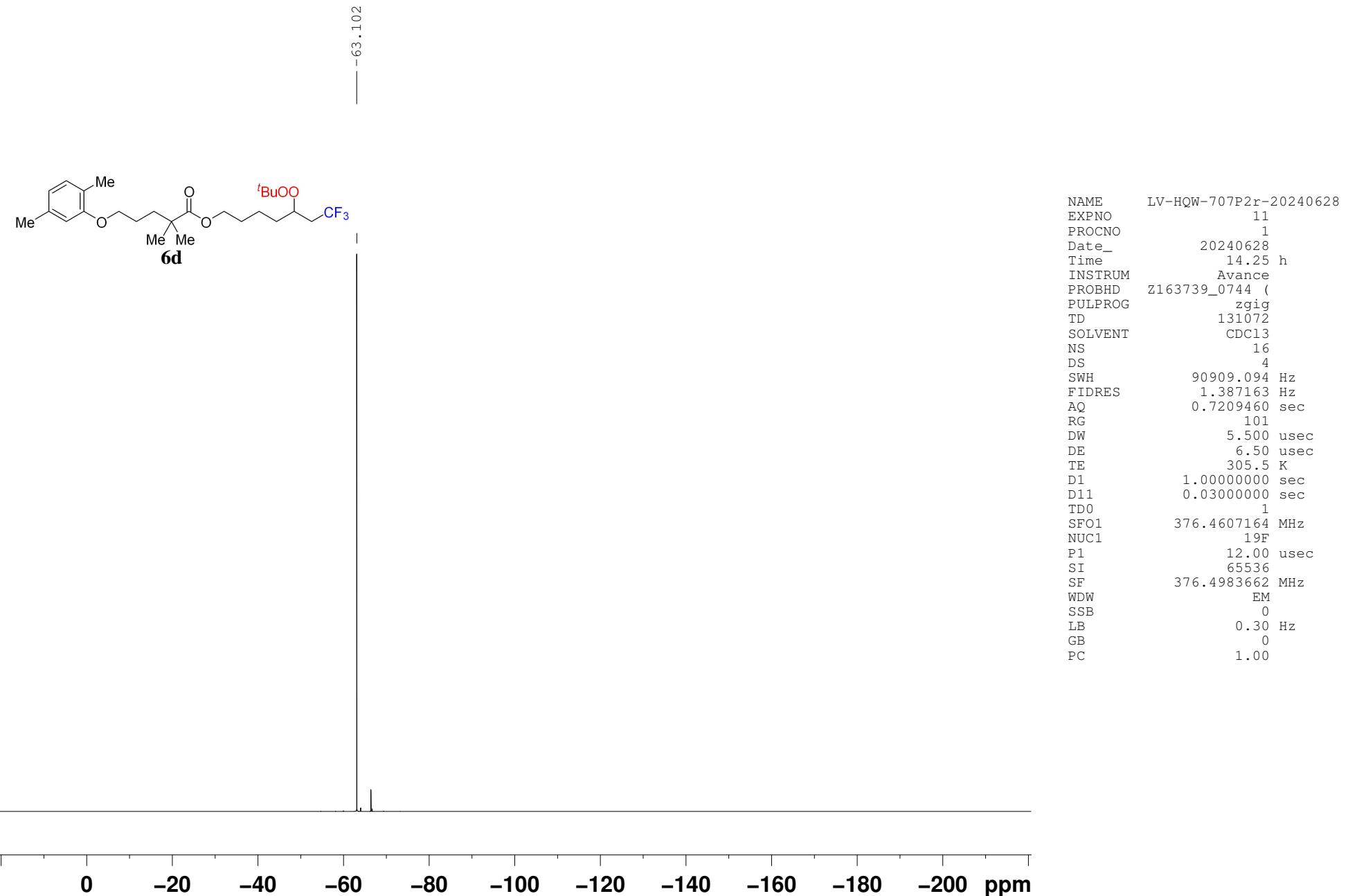
NAME LV-HQW-688P-20240616
 EXPNO 11
 PROCNO 1
 Date_ 20240617
 Time 0.41 h
 INSTRUM Avance
 PROBHD Z163739_0744 (zgig
 PULPROG 131072
 TD 16
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 90909.094 Hz
 FIDRES 1.387163 Hz
 AQ 0.7209460 sec
 RG 101
 DW 5.500 usec
 DE 6.50 usec
 TE 300.9 K
 D1 1.0000000 sec
 D11 0.0300000 sec
 TDO 1
 SFO1 376.4607164 MHz
 NUC1 19F
 P1 12.00 usec
 SI 65536
 SF 376.4983662 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

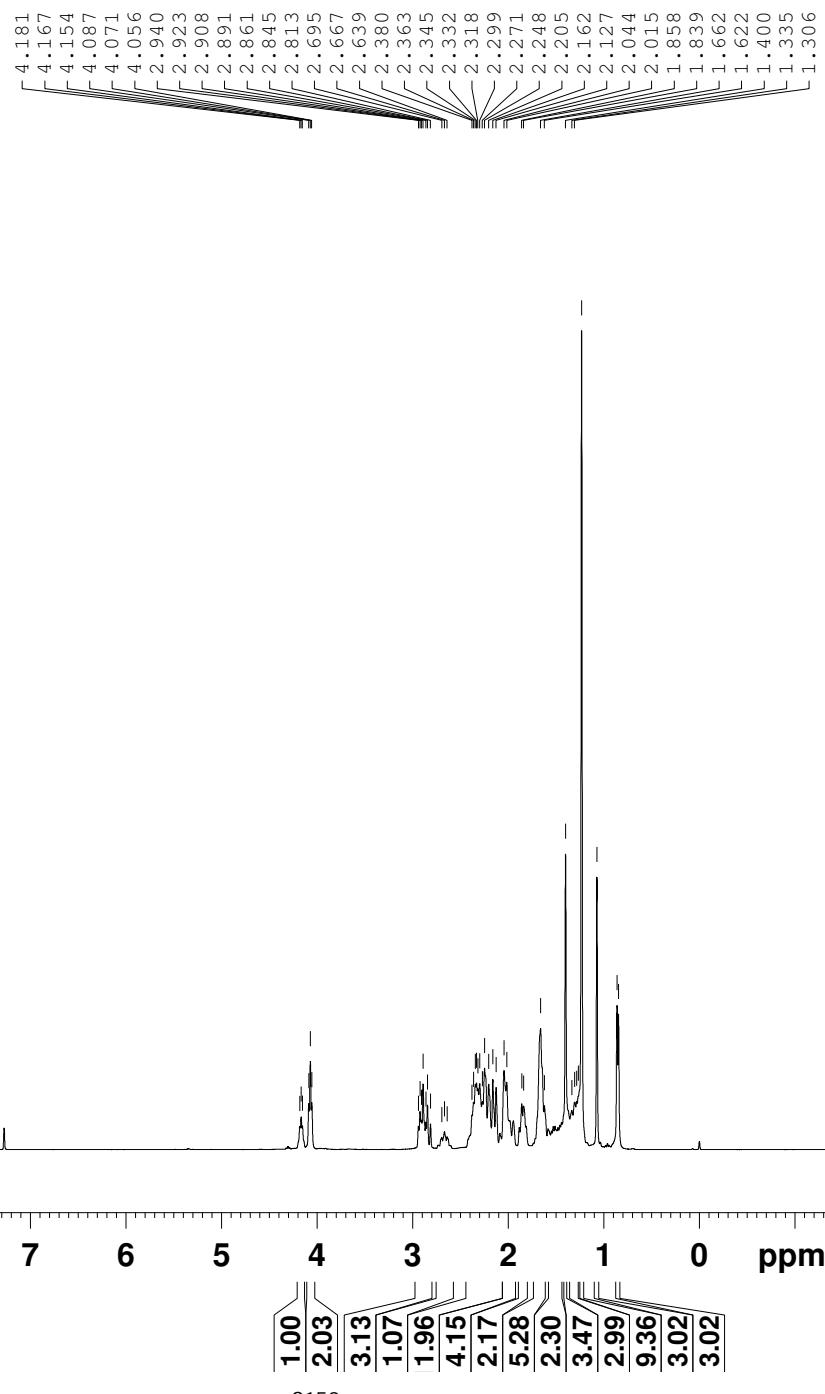
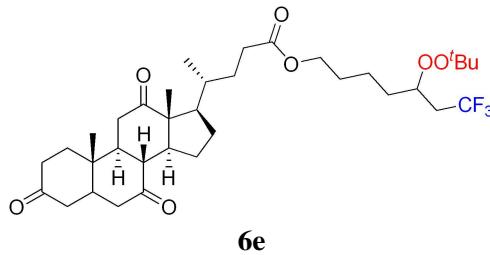
0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 ppm

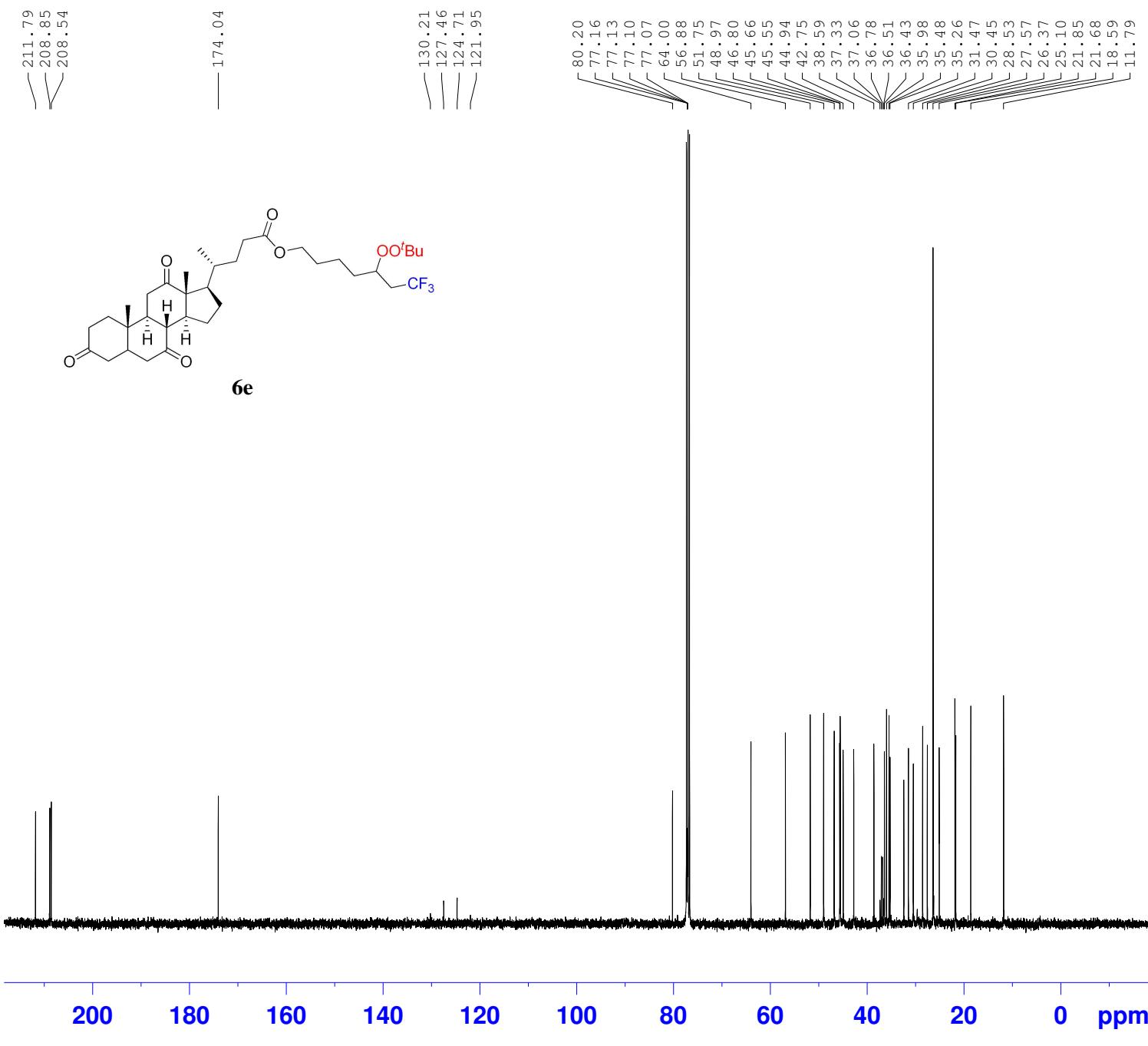




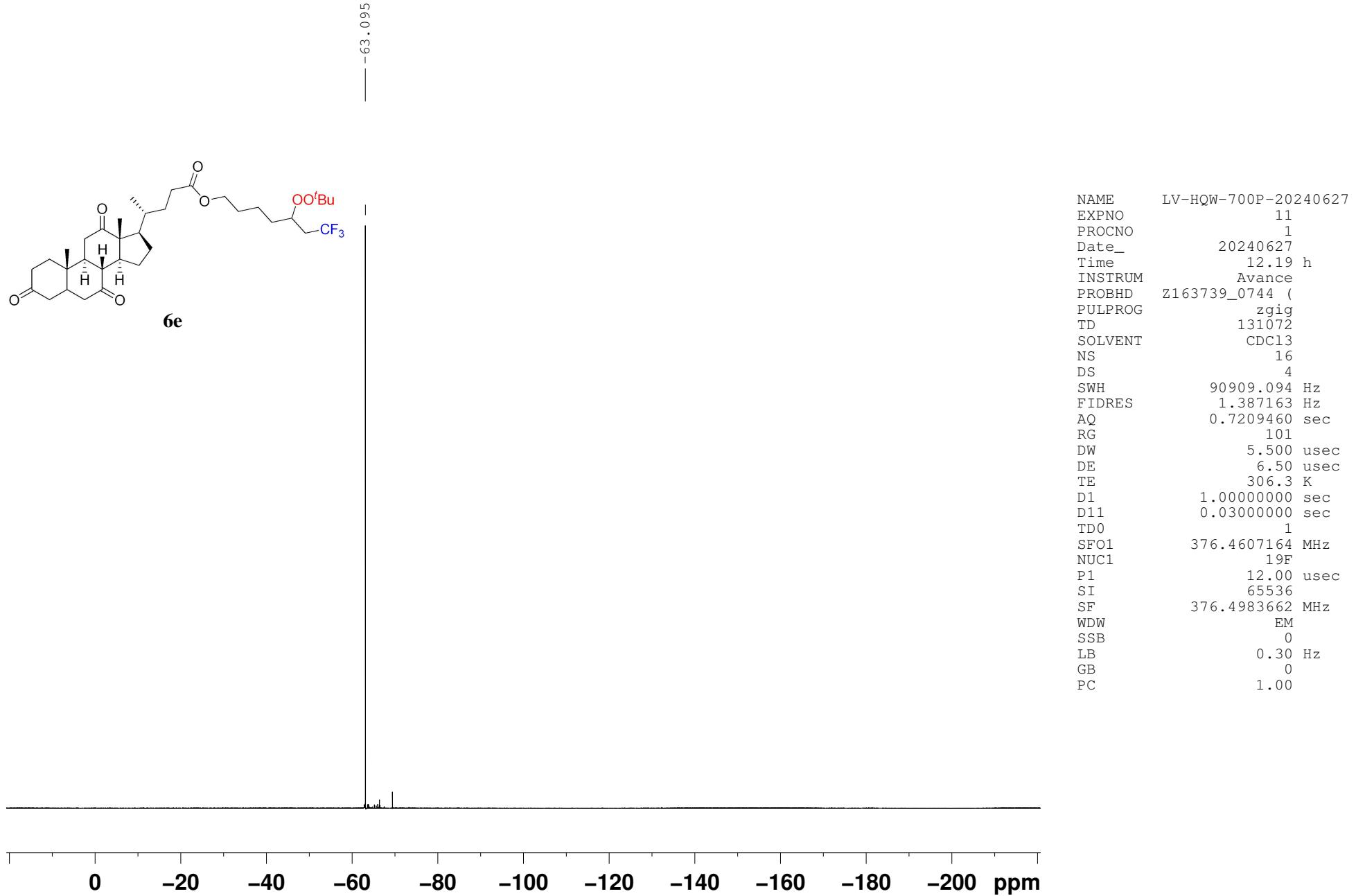
NAME	LV-HQW-707P2r-20240628
EXPNO	12
PROCNO	1
Date_	20240628
Time	15.01 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl ₃
NS	600
DS	4
SWH	23809.523 Hz
FIDRES	0.726609 Hz
AQ	1.3763061 sec
RG	101
DW	21.000 usec
DE	6.50 usec
TE	305.7 K
D1	2.0000000 sec
D11	0.03000000 sec
TDO	1
SFO1	100.6228298 MHz
NUC1	¹³ C
P0	2.67 usec
P1	8.00 usec
SI	32768
SF	100.6127687 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

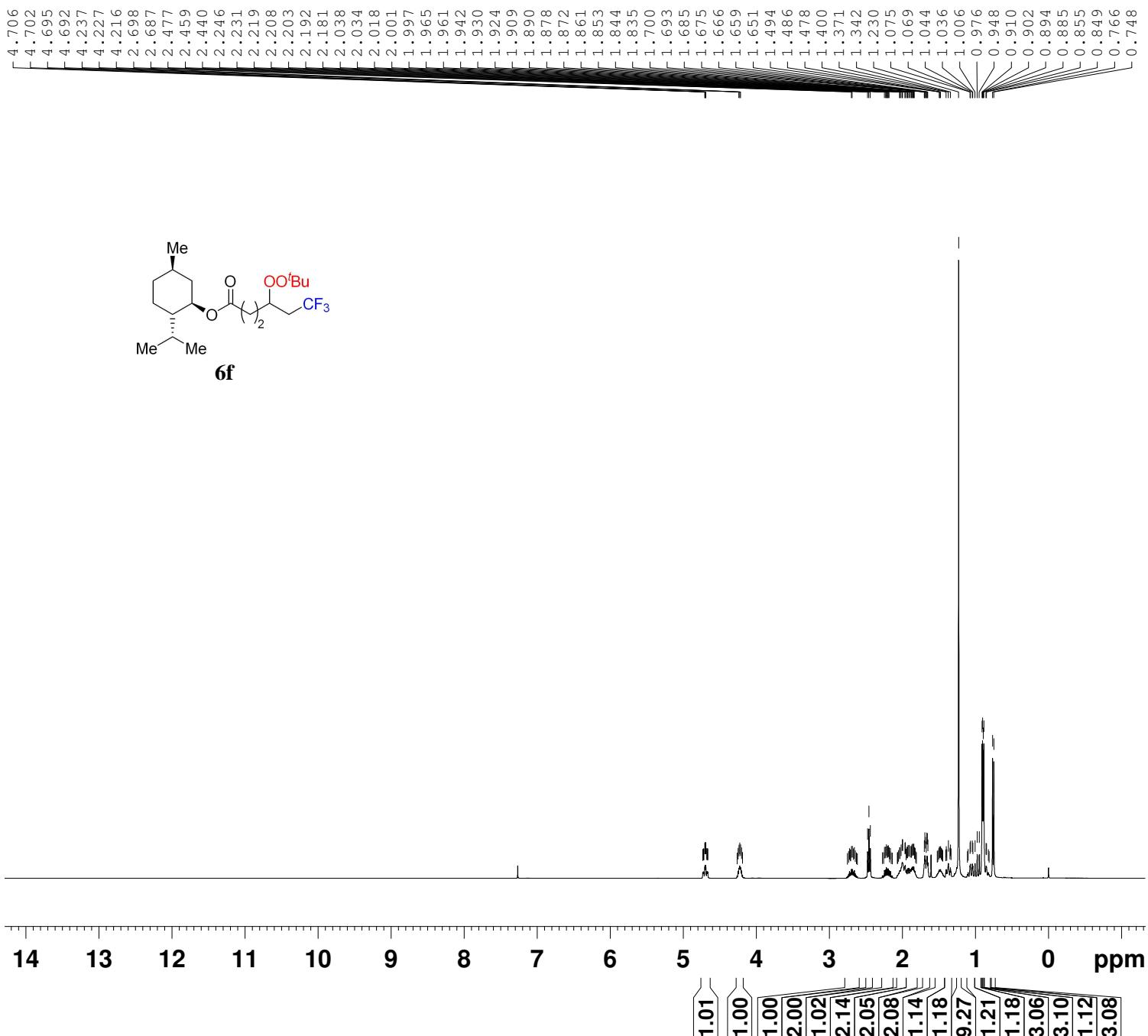




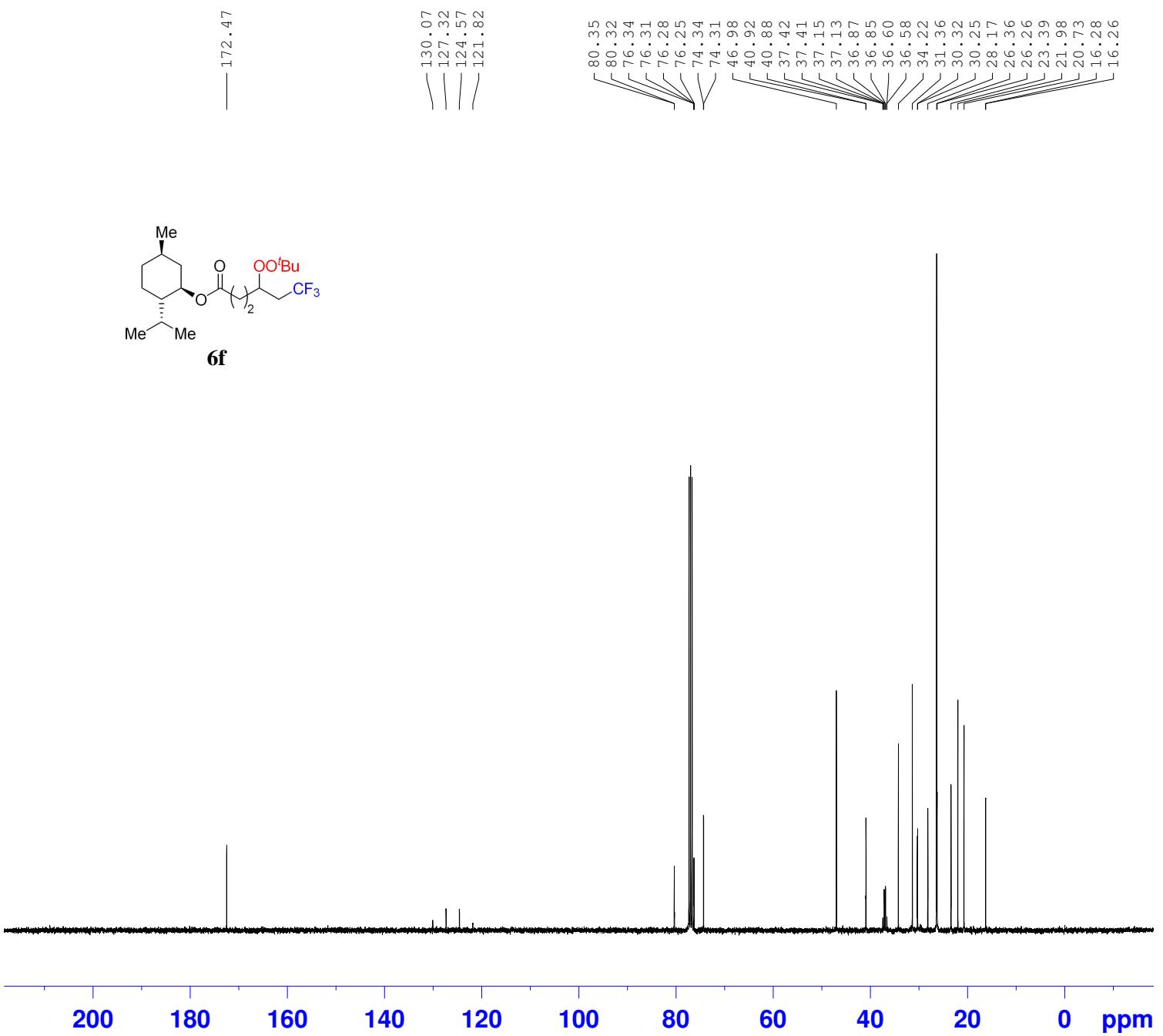


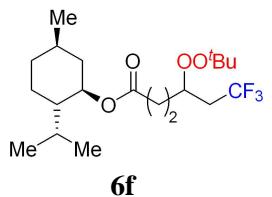
NAME LV-HQW-700P-20240627
 EXPNO 12
 PROCNO 1
 Date_ 20240627
 Time 12.55 h
 INSTRUM Avance
 PROBHD Z163739_0744 (zgpg30
 PULPROG 65536
 TD 500
 SOLVENT CDCl₃
 NS 600
 DS 4
 SWH 23809.523 Hz
 FIDRES 0.726609 Hz
 AQ 1.3763061 sec
 RG 101
 DW 21.000 usec
 DE 6.50 usec
 TE 307.5 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1
 SFO1 100.6228298 MHz
 NUC1 ¹³C
 P0 2.67 usec
 P1 8.00 usec
 SI 32768
 SF 100.6127709 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



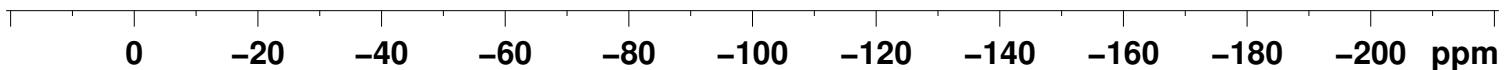


NAME LV-HQW-655P-20240528
 EXPNO 10
 PROCNO 1
 Date_ 20240528
 Time 13.16 h
 INSTRUM Avance
 PROBHD Z163739_0744 (zg30
 PULPROG 65536
 TD 8
 SOLVENT CDC13
 NS 0
 DS 0
 SWH 6250.000 Hz
 FIDRES 0.190735 Hz
 AQ 5.2429299 sec
 RG 45.2
 DW 80.000 usec
 DE 8.64 usec
 TE 298.6 K
 D1 1.00000000 sec
 TDO 1
 SFO1 400.1326008 MHz
 NUC1 1H
 P0 2.67 usec
 P1 8.00 usec
 SI 65536
 SF 400.1300077 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

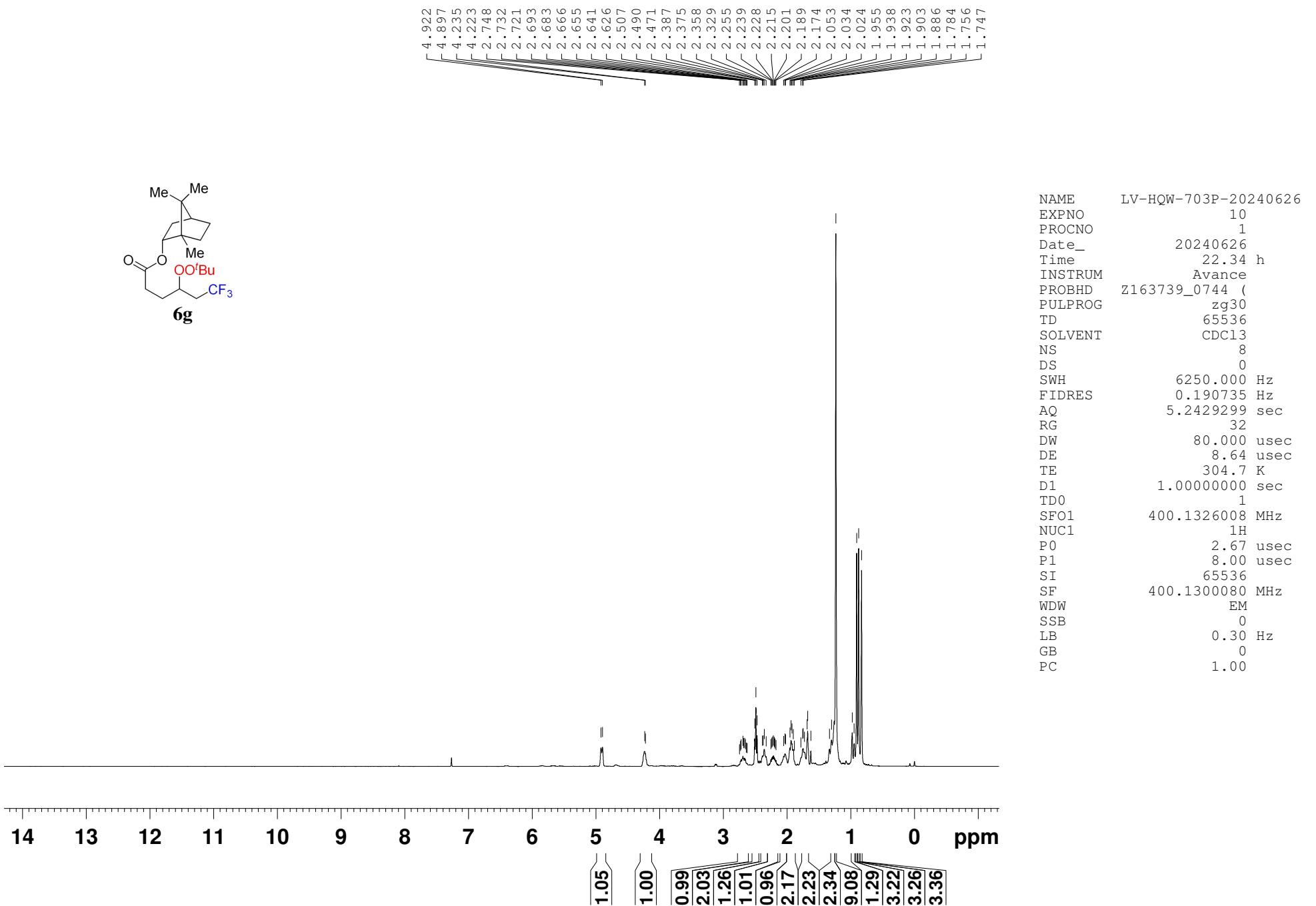


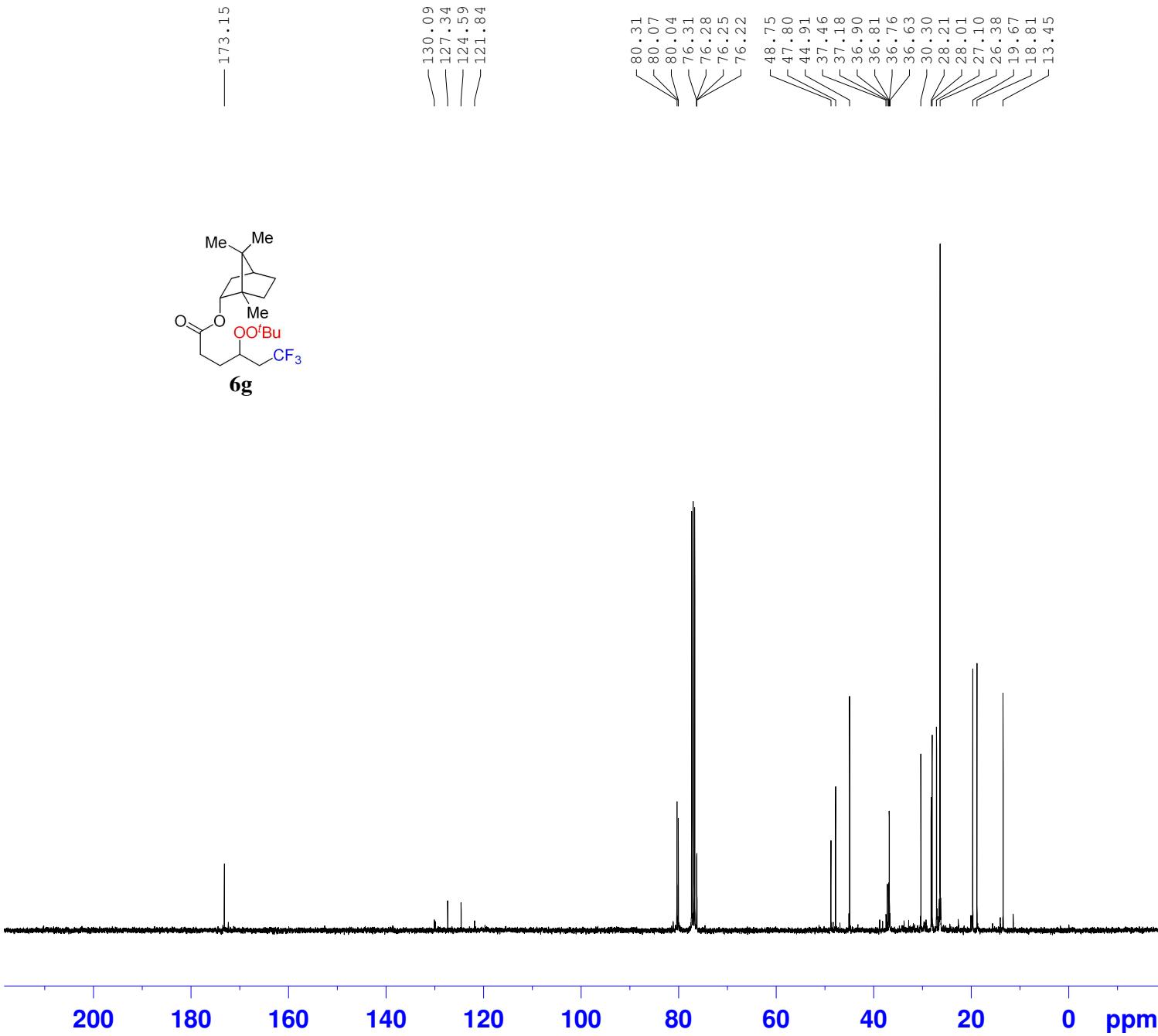
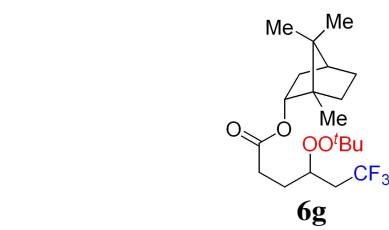


-63.217
-63.231

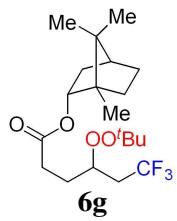


NAME	LV-HQW-655P-20240528
EXPNO	11
PROCNO	1
Date_	20240528
Time	13.18 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.5 K
D1	1.0000000 sec
D11	0.0300000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





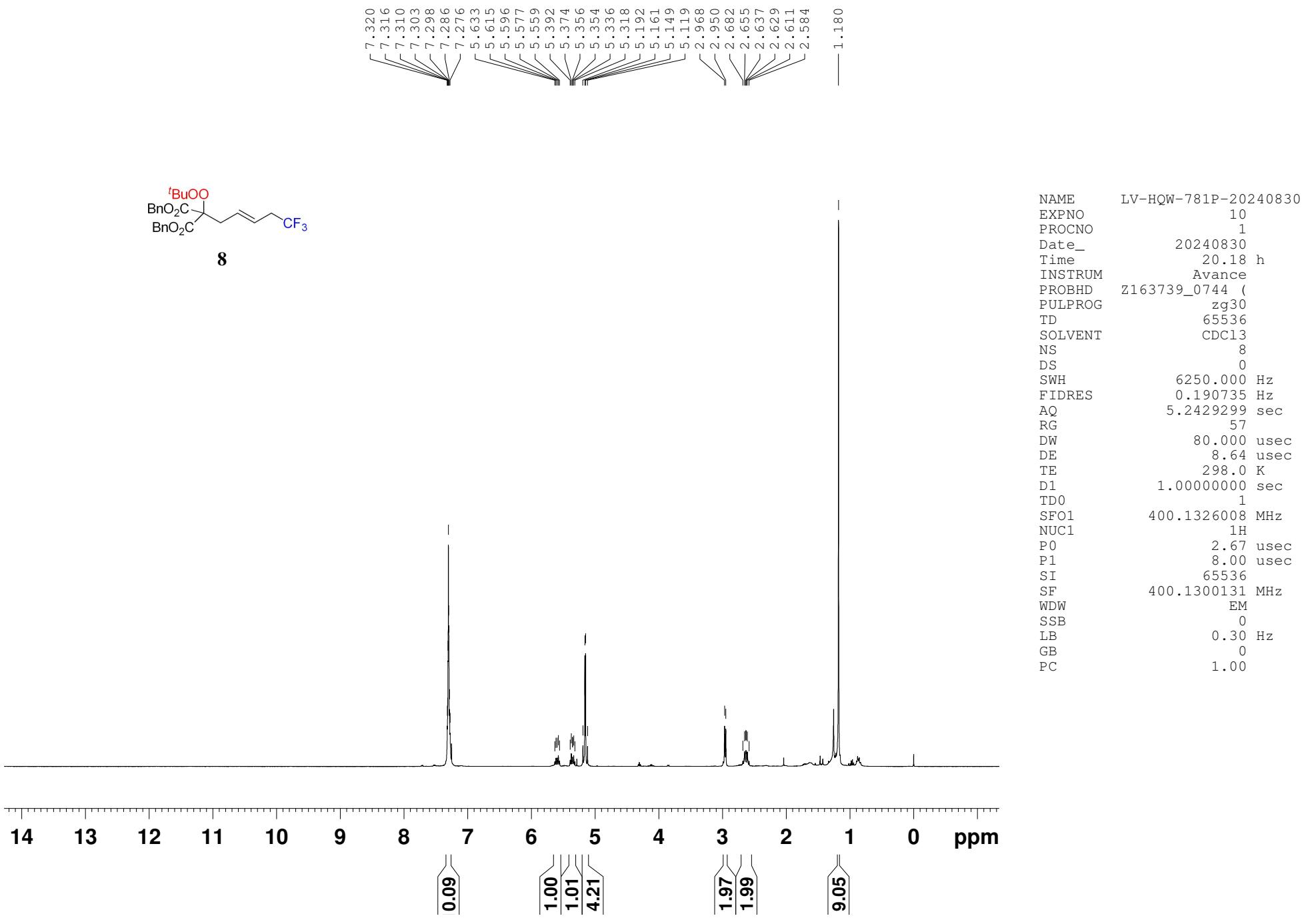
NAME LV-HQW-703P-20240626
 EXPNO 12
 PROCNO 1
 Date_ 20240626
 Time 23.06 h
 INSTRUM Avance
 PROBHD Z163739_0744 (zgpg30
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 464
 DS 4
 SWH 23809.523 Hz
 FIDRES 0.726609 Hz
 AQ 1.3763061 sec
 RG 101
 DW 21.000 usec
 DE 6.50 usec
 TE 306.5 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1
 SFO1 100.6228298 MHz
 NUC1 13C
 P0 2.67 usec
 P1 8.00 usec
 SI 32768
 SF 100.6127680 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

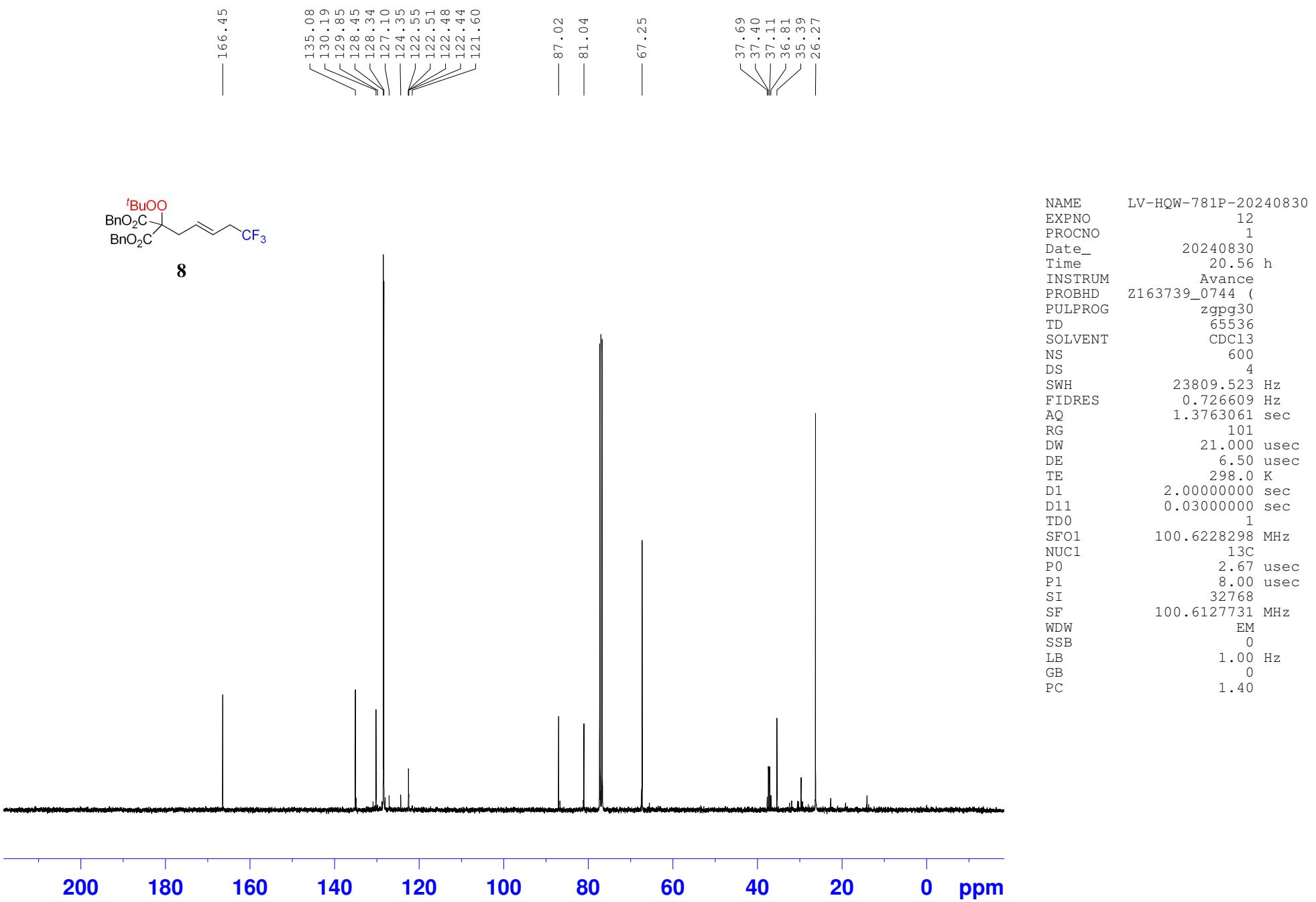


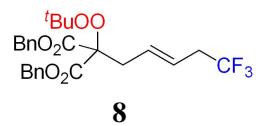
— -63.204

0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 ppm

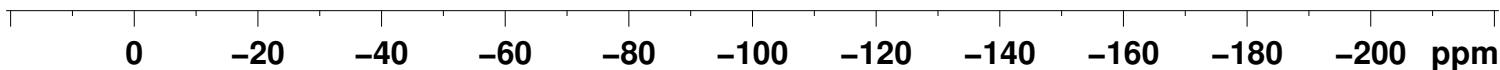
NAME	LV-HQW-703P-20240626
EXPNO	11
PROCNO	1
Date_	20240626
Time	22.37 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl ₃
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	305.3 K
D1	1.0000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00







—66.452



NAME	LV-HQW-781P-20240830
EXPNO	11
PROCNO	1
Date_	20240830
Time	20.20 h
INSTRUM	Avance
PROBHD	Z163739_0744 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl3
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	298.1 K
D1	1.0000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 MHz
NUC1	19F
P1	12.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00