Electronic Supplementary Material (ESI) for ChemComm.

Mn-Catalyzed Remote C(sp^3)–H Bond Peroxidation Triggered by Radical Trifluoromethylation of Unactivated Alkene

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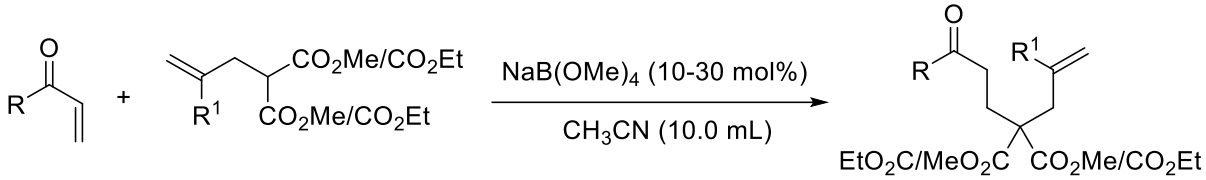
Contents

1. General information

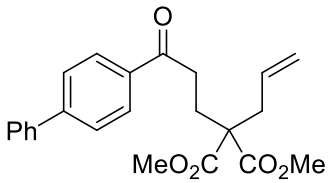
^{1}H NMR spectra were recorded on Bruker 400 MHz and 600 MHz spectrometer and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm) for $CDCl_3$. TGA analysis was carried out at thermogravimetric analyzer Q500 (TAInstrument company, USA). The peak patterns 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). ^{13}C NMR spectra were obtained at Bruker 100 MHz, 150 MHz and referenced to the internal solvent signals (central peak is 77.0 ppm in $CDCl_3$). ^{19}F NMR spectra were obtained at Bruker 376 MHz, 564 MHz. $CDCl_3$ was used as the NMR solvent. APEX II (Bruker Inc.) was used for ESI-MS and EI-MS. IR spectra were recorded by a Bruker Tensor 27 infrared spectrometer. 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, and J&K and used without further purification.

CAUTION-1: Mixing a metal salt and peroxide can cause explosion. See: Jones, A. K.; Wilson, T. E.; Nikam, S. S. *In Encyclopedia of Reagents for Organic Synthesis*, Paquette, L. A. Ed.; John Wiley & Sons, Inc. **1995**, *2*, 880.

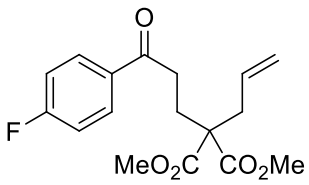
2. General procedure for synthesis of 1



The alkene substrates were synthesized according to the reported literatures. [1-5] To a solution of Michael donor (2.0 mmol) and $NaB(OMe)_4$ (10-30 mol %) in MeCN (10 mL) was added *α, β*-unsaturated ketone (2.0-4.0 mmol) at room temperature. The resulting solution was stirred at room temperature or 50 °C under air atmosphere and monitored by TLC. Upon completion, solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel (ethyl acetate/petroleum ether = 1/12-1/4) to give the desired products. (**1a**, **1b**, **1d**, **1e**, **1f**, **1h**, **1i**, **1l**, **1p**, **1q**, **1r**, **1s**, **1t**, **1u** were synthesized according to the reported literature [1-5])

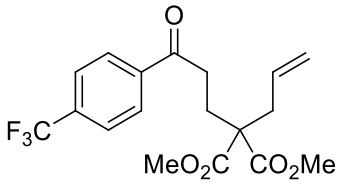


Dimethyl 2-allyl-2-(3-(biphenyl-4-yl)-3-oxopropyl)malonate (**1c**) Isolated by flash column chromatography (ethyl acetate/petroleum ether = 10:1, R_f = 0.3) in 74% yield (280 mg); White solid. ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.02 (d, J = 8.4 Hz, 2H), 7.67 (d, J = 8.4 Hz, 2H), 7.62 (d, J = 7.2 Hz, 2H), 7.46 (t, J = 7.4 Hz, 2H), 7.39 (t, J = 7.4 Hz, 1H), 5.75-5.68 (m, 1H), 5.17-5.12 (m, 2H), 3.73 (s, 6H), 3.04 (t, J = 7.7 Hz, 2H), 2.74 (d, J = 7.4 Hz, 2H), 2.35 (t, J = 7.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 198.4, 171.5, 145.8, 139.9, 135.4, 132.2, 129.0, 128.7, 128.3, 127.3, 127.26, 119.4, 57.1, 52.5, 38.5, 33.8, 27.4; HRMS (ESI) calcd for $C_{22}H_{29}F_3O_7Na$ $(M+Na)^+$: 403.1516; found: 403.1512.

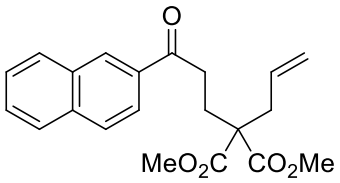


Dimethyl 2-allyl-2-(3-(4-fluorophenyl)-3-oxopropyl)malonate (**1g**) Isolated by flash column

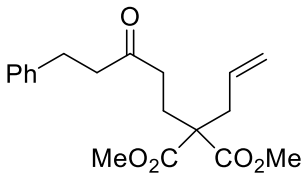
chromatography (ethyl acetate/petroleum ether = 10:1, R_f = 0.3) in 78% yield (250 mg); White solid. ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.99 (d, J = 5.6, 8.1 Hz, 2H), 7.60 (t, J = 8.5 Hz, 2H), 5.73-5.66 (m, 1H), 5.16-5.11 (m, 2H), 3.73 (s, 6H), 3.00 (t, J = 7.6 Hz, 2H), 2.72 (d, J = 7.4 Hz, 2H), 2.31 (t, J = 8.0 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 197.2, 171.3, 165.7 (d, J = 7.4 Hz), 133.1, 132.1, 130.7 (d, J = 7.4 Hz), 119.4, 115.6 (d, J = 7.4 Hz), 57.0, 52.5, 38.5, 33.7, 27.4; HRMS (ESI) calcd for $C_{17}H_{19}FO_5Na$ $(M+Na)^+$: 345.1109; found: 345.1108.



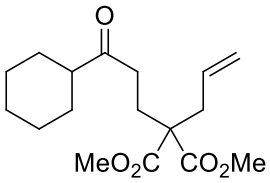
Dimethyl 2-allyl-2-(3-oxo-3-(4-(trifluoromethyl)phenyl)propyl)malonate (**1j**) Isolated by flash column chromatography (ethyl acetate/petroleum ether = 10:1, R_f = 0.3) in 72% yield (268 mg); Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.06 (d, J = 8.0 Hz, 2H), 7.73 (d, J = 8.0 Hz, 2H), 5.72-5.66 (m, 1H), 5.17-5.12 (m, 2H), 3.74 (s, 6H), 3.06 (t, J = 7.5 Hz, 2H), 2.73 (d, J = 7.4 Hz, 2H), 2.33 (t, J = 7.6 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 197.9, 171.3, 139.3, 134.4 (q, J = 32.5 Hz), 132.1, 128.4, 125.7 (q, J = 2.9 Hz), 123.6 (q, J = 271.1 Hz), 119.5, 56.9, 52.5, 38.6, 34.2, 27.3; HRMS (ESI) calcd for $C_{18}H_{19}F_3O_5Na$ $(M+Na)^+$: 395.1077; found: 395.1079.



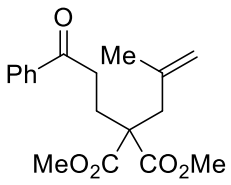
Dimethyl 2-allyl-2-(3-(naphthalen-2-yl)-3-oxopropyl)malonate (**1k**) Isolated by flash column chromatography (ethyl acetate/petroleum ether = 10:1, R_f = 0.3) in 71% yield (250 mg); White solid. ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.46 (s, 1H), 8.01 (d, J = 8.6 Hz, 1H), 7.96 (d, J = 8.1 Hz, 1H), 7.87 (t, J = 9.2 Hz, 2H), 7.59 (t, J = 7.0 Hz, 1H), 7.54 (t, J = 7.6 Hz, 1H), 5.76-5.69 (m, 2H), 5.18-5.12 (m, 1H), 3.74 (s, 6H), 3.15 (t, J = 7.6 Hz, 2H), 2.76 (d, J = 7.4 Hz, 2H), 2.39 (t, J = 7.9 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 198.7, 171.4, 135.5, 133.9, 132.5, 132.2, 129.7, 129.5, 128.4, 128.4, 127.7, 126.7, 123.8, 119.4, 57.1, 52.5, 38.5, 33.8, 27.5; HRMS (ESI) calcd for $C_{21}H_{22}O_5Na$ $(M+Na)^+$: 377.1359; found: 377.1353.



Dimethyl 2-allyl-2-(3-oxo-5-phenylpentyl)malonate (**1m**) Isolated by flash column chromatography (ethyl acetate/petroleum ether = 8:1, R_f = 0.3) in 66% yield (220 mg); Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.28-7.26 (m, 2H), 7.20-7.16 (m, 3H), 5.66-5.59 (m, 1H), 5.10-5.07 (m, 2H), 3.69 (s, 6H), 2.88 (t, J = 7.4 Hz, 2H), 2.72 (t, J = 7.9 Hz, 2H), 2.61 (d, J = 7.4 Hz, 2H), 2.40 (t, J = 7.4 Hz, 2H), 2.13 (t, J = 8.0 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.4, 171.4, 140.9, 132.1, 128.5, 128.3, 126.1, 119.4, 56.8, 52.5, 44.2, 38.2, 37.9, 29.8, 26.6; HRMS (ESI) calcd for $C_{19}H_{24}O_5Na$ $(M+Na)^+$: 355.1516; found: 355.1513.



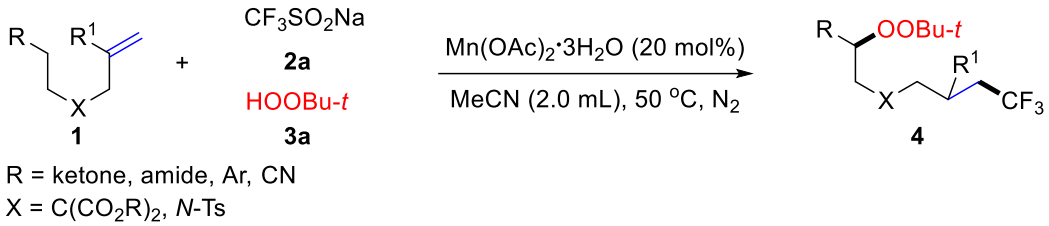
Dimethyl 2-allyl-2-(3-cyclohexyl-3-oxopropyl)malonate (**1n**) Isolated by flash column chromatography (ethyl acetate/petroleum ether = 8:1, R_f = 0.3) in 64% yield (200 mg); Colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 5.69-5.62 (m, 1H), 5.12-5.09 (m, 2H), 3.71 (s, 6H), 2.63 (d, J = 7.4 Hz, 2H), 2.46 (t, J = 7.5 Hz, 2H), 2.34-2.29 (m, 1H), 2.13 (t, J = 7.9 Hz, 2H), 1.82-1.76 (m, 4H), 1.66 (t, J = 12.7 Hz, 1H), 1.34-1.17 (m, 5H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 212.4, 171.4, 132.2, 119.2, 56.9, 52.4, 50.8, 38.2, 35.4, 28.5, 26.6, 25.8, 25.6; HRMS (ESI) calcd for $C_{17}H_{26}O_5Na$ $(M+Na)^+$: 333.1673; found: 333.1669.



Dimethyl 2-(2-methylallyl)-2-(3-oxo-3-phenylpropyl)malonate (**1o**) Isolated by flash column chromatography (ethyl acetate/petroleum ether = 10:1, R_f = 0.3) in 76% yield (241 mg); White solid. ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.95 (d, J = 8.0 Hz, 2H), 7.55 (t, J = 7.4 Hz, 1H), 7.45 (t, J = 7.5 Hz, 2H), 4.88 (s, 1H), 4.77 (s, 1H), 3.72 (s, 6H), 3.00 (t, J = 7.6 Hz, 2H), 2.79 (s, 2H), 2.34 (t, J = 7.8

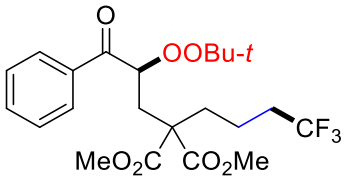
Hz, 2H), 1.67 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.6, 171.6, 140.1, 136.5, 132.1, 132.9, 128.4, 127.9, 115.8, 56.4, 52.3, 41.6, 33.7, 27.2, 22.9; HRMS (ESI) calcd for $C_{18}H_{22}O_5Na$ $(M+Na)^+$: 341.1359; found: 341.1353.

3 General procedure for synthesis of 4

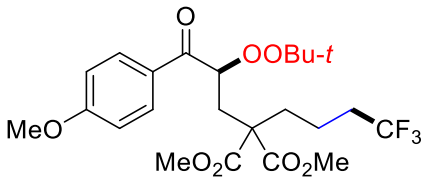


R = ketone, amide, Ar, CN
X = $C(CO_2R)_2$, *N*-Ts

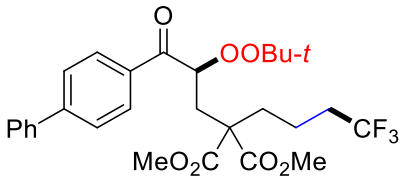
To a 25 mL Schlenk tube were added unactivated alkenes **1** (0.2 mmol), $Mn(OAc)_3{\cdot}H_2O$ (0.04 mmol), CF_3SO_2Na (0.4 mmol), MeCN (2.0 mL) and *t*-BuOOH (T-hydro, 70% in water, 1.0 mmol) under N_2 atmosphere at room temperature, and the resulting solution was stirred at 50°C for 3 h. The resulting mixture and the solvent were evaporated under vacuum. The residue was purified by flash column chromatography on silica gel (eluent: ethyl acetate/petroleum ether) to give the peroxides **4a**-**4u**.



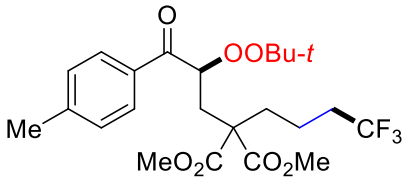
2-(2-(*tert*-butylperoxy)-3-oxo-3-phenylpropyl)-2-(4,4,4-trifluorobutyl)malonate (4a): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 7:1, R_f = 0.4) in 72% yield (67 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.05 (d, J = 7.9 Hz, 2H), 7.58 (t, J = 7.3 Hz, 1H), 7.47 (t, J = 7.6 Hz, 2H), 5.17 (dd, J =8.1, 4.2 Hz, 1H), 3.73 (s, 6H), 2.50-2.43 (m, 2H), 2.18-2.06 (m, 4H), 1.61-1.53 (m, 1H), 1.47-1.40 (m, 1H), 1.13 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 197.6, 171.0, 170.9, 135.2, 133.4, 129.0, 128.5, 126.8 (q, J = 274.7 Hz), 81.3, 80.8, 55.9, 52.7, 52.6, 33.9 (q, J = 28.8 Hz), 33.1, 32.2, 26.3, 17.2 (q, J = 2.6 Hz); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.3 (s, 3F); HRMS (ESI) calcd for $C_{22}H_{29}F_3O_7Na$ $(M+Na)^+$: 485.1758; found: 485.1760.



Dimethyl-2-(2-(*tert*-butylperoxy)-3-oxo-3-p-tolylpropyl)-2-(4,4,4-trifluorobutyl)malonate (4b): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 6:1, R_f = 0.4) in 72% yield (66 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.06 (d, J = 8.5 Hz, 2H), 6.95 (d, J = 8.5 Hz, 2H), 5.12 (t, J = 6.2 Hz, 1H), 3.88 (s, 3H), 3.73 (s, 6H), 2.45 (d, J = 6.1 Hz, 2H), 2.17-2.05 (m, 4H), 1.58-1.52 (m, 1H), 1.47-1.41 (m, 1H), 1.14 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 195.9, 171.0, 170.9, 163.7, 131.4, 128.1, 126.8 (q, J = 274.6 Hz), 113.7, 81.4, 80.8, 55.9, 55.4, 52.7, 52.6, 33.9 (q, J = 28.8 Hz), 33.3, 32.2, 26.4, 17.2 (q, J = 2.3 Hz). ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.3 (s, 3F); HRMS (ESI) calcd for $C_{23}H_{31}F_3O_8Na$ $(M+Na)^+$: 515.1863; found: 515.1865.

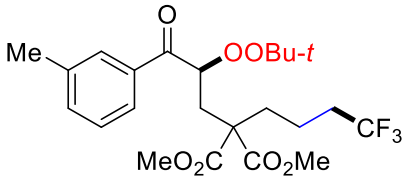


Dimethyl-2-(3-(biphenyl-4-yl)-2-(*tert*-butylperoxy)-3-oxopropyl)-2-(4,4,4-trifluorobutyl)malonate (4c): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 7:1, R_f = 0.4) in 70% yield (75 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.15 (d, J = 8.4 Hz, 2H), 7.70 (d, J = 8.5 Hz, 2H), 7.60 (d, J = 7.1 Hz, 2H), 7.48 (t, J = 7.4 Hz, 2H), 7.41 (t, J = 7.4 Hz, 1H), 5.19 (dd, J = 7.9, 4.4 Hz, 1H), 3.74 (s, 3H), 3.74 (s, 3H), 2.52-2.46 (m, 2H), 2.20-2.08 (m, 4H), 1.62-1.54 (m, 1H), 1.49-1.41 (m, 1H), 1.15 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 197.1, 171.0, 170.9, 146.0, 139.8, 133.8, 129.6, 129.0, 128.3, 127.3, 127.2, 126.8 (q, J = 274.6 Hz), 81.5, 80.9, 55.9, 52.8, 52.7, 33.9 (q, J = 28.5 Hz), 33.2, 32.2, 26.4, 17.2 (q, J = 2.9 Hz). ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.2 (s, 3F); HRMS (ESI) calcd for $C_{28}H_{33}F_3O_7Na$ $(M+Na)^+$: 561.2071; found: 561.2071.

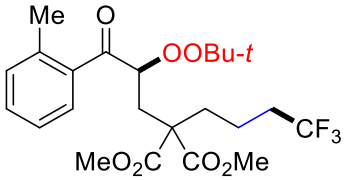


Dimethyl-2-(2-(*tert*-butylperoxy)-3-oxo-3-p-tolylpropyl)-2-(4,4,4-trifluorobutyl)malonate (4d): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 7:1, R_f = 0.4) in 75% yield (71 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.96 (d, J = 7.6 Hz, 2H), 7.27 (d, J = 8.0 Hz, 2H), 5.15 (dd, J = 7.2, 5.2 Hz, 1H), 3.72 (s, 6H), 2.45-2.40 (m, 5H), 2.17-2.05 (m, 4H), 1.60-1.52

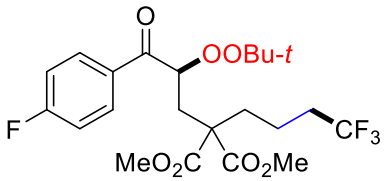
(m, 1H), 1.46-1.40 (m, 1H), 1.13 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 197.0, 171.0, 170.9, 144.2, 132.7, 129.2, 129.1, 126.8 (q, J = 274.9 Hz), 81.3, 80.8, 55.9, 52.7, 52.6, 33.9 (q, J = 28.5 Hz), 33.2, 32.2, 26.4, 21.7, 17.2 (q, J = 2.5 Hz); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.3 (s, 3F); HRMS (ESI) calcd for $C_{23}H_{31}F_3O_7Na$ $(M+Na)^+$: 499.1914; found: 499.1913.



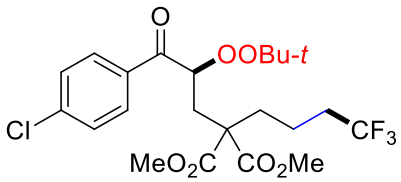
Dimethyl-2-(2-(*tert*-butylperoxy)-3-oxo-3-m-tolylpropyl)-2-(4,4,4-trifluorobutyl)malonate (4e): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 7:1, R_f = 0.4) in 70% yield (67 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.84 (d, J = 8.3 Hz, 2H), 7.40 (d, J = 7.4 Hz, 1H), 7.36 (t, J = 7.4 Hz, 1H), 5.16 (dd, J = 7.6, 4.9 Hz, 1H), 3.730 (s, 3H), 3.725 (s, 3H), 2.45-2.42 (m, 5H), 2.16-2.08 (m, 4H), 1.59-1.52 (m, 1H), 1.48-1.43 (m, 1H), 1.14 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 197.7, 171.0, 170.9, 138.4, 135.2, 134.2, 129.5, 128.4, 126.8 (q, J = 274.7 Hz), 126.2, 81.2, 80.8, 55.9, 52.8, 52.7, 33.9 (q, J = 28.5 Hz), 33.1, 32.2, 26.4, 21.4, 17.2(q, J = 2.9 Hz); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.3 (s, 3F); HRMS (ESI) calcd for $C_{23}H_{31}F_3O_7Na$ $(M+Na)^+$: 499.1914; found: 499.1914.



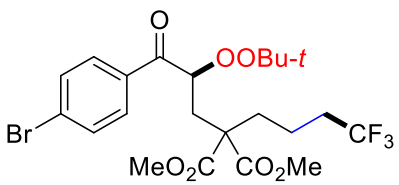
Dimethyl2-(2-(*tert*-butylperoxy)-3-oxo-3-o-tolylpropyl)-2-(4,4,4-trifluorobutyl)malonate (4f): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 7:1, R_f = 0.4) in 73% yield (69 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.65 (d, J = 7.6 Hz, 1H), 7.38 (t, J = 7.6 Hz, 1H), 7.26 (d, J = 8.5 Hz, 2H), 5.03 (dd, J = 8.9, 2.8 Hz, 1H), 3.73 (s, 3H), 3.67 (s, 3H), 2.48-2.44 (m, 4H), 2.48-2.44 (m, 4H), 2.39-2.35 (m, 1H), 1.58-1.51 (m, 1H), 1.41-1.34 (m, 1H), 1.09 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 201.8, 171.0, 170.9, 138.6, 136.3, 131.7, 131.3, 128.3, 126.8 (q, J = 274.7 Hz), 125.3, 81.8, 80.7, 56.0, 52.7, 52.6, 33.9 (q, J = 28.5 Hz), 32.5, 32.0, 26.3, 20.6, 17.1 (q, J = 2.9 Hz); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.3 (s, 3F); HRMS (ESI) calcd for $C_{23}H_{31}F_3O_7Na$ $(M+Na)^+$: 499.1914; found: 499.1916.



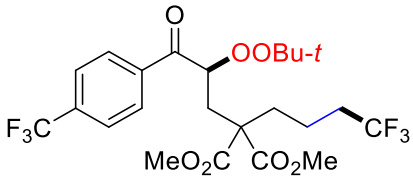
Dimethyl2-(2-(*tert*-butylperoxy)-3-(4-fluorophenyl)-3-oxopropyl)-2-(4,4,4-trifluorobutyl)malonate (4g): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 7:1, R_f = 0.4) in 69% yield (66 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.12 (dd, J = 8.7, 5.5 Hz, 2H), 7.15 (t, J = 8.5 Hz, 2H), 5.09 (dd, J = 8.1, 4.3 Hz, 1H), 3.74 (s, 6H), 2.49-2.42 (m, 2H), 2.17-2.05 (m, 4H), 1.60-1.53 (m, 1H), 1.47-1.41 (m, 1H), 1.12 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 196.0, 170.9, 170.8, 165.9 (d, J = 253.9 Hz), 131.8 (d, J = 9.0 Hz), 131.5 (d, J = 2.7 Hz), 126.8 (q, J = 275.0 Hz), 115.6 (d, J = 21.8 Hz), 81.8, 80.9, 55.9, 52.8, 52.7, 33.9 (q, J = 28.6 Hz), 33.1, 32.2, 26.3, 17.2 (q, J = 2.9 Hz); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.3 (s, 3F), -104.5 (s, 1F); HRMS (ESI) calcd for $C_{22}H_{28}F_4O_7Na$ $(M+Na)^+$: 503.1663; found: 503.1667.



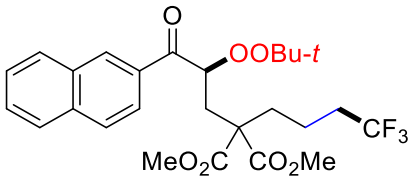
Dimethyl-2-(2-(*tert*-butylperoxy)-3-(4-chlorophenyl)-3-oxopropyl)-2-(4,4,4-trifluorobutyl)malonate (4h): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 7:1, R_f = 0.4) in 67% yield (66 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.03 (d, J = 8.5 Hz, 2H), 7.45 (d, J = 8.5 Hz, 2H), 5.08 (dd, J = 8.1, 4.1 Hz, 1H), 3.73 (s, 6H), 2.49-2.41 (m, 2H), 2.16-2.05 (m, 4H), 1.60-1.53 (m, 1H), 1.47-1.41 (m, 1H), 1.12 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 196.5, 170.9, 170.8, 139.8, 133.4, 130.5, 128.8, 126.8 (q, J = 274.6 Hz), 81.8, 81.0, 55.8, 52.8, 52.7, 33.8 (q, J = 28.6 Hz), 33.1, 32.2, 26.3, 17.2 (q, J = 2.4 Hz); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.2 (s, 3F); HRMS (ESI) calcd for $C_{22}H_{28}ClF_3O_7Na$ $(M+Na)^+$: 519.1368; found: 519.1373.



Dimethyl2-(3-(4-bromophenyl)-2-(*tert*-butylperoxy)-3-oxopropyl)-2-(4,4,4-trifluorobutyl)malonate (4i): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 7:1, R_f = 0.4) in 69% yield (74 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.95 (d, J = 8.3 Hz, 2H), 7.62 (d, J = 8.3 Hz, 2H), 5.07 (dd, J = 8.2, 4.1 Hz, 1H), 3.73 (s, 6H), 2.49-2.41 (m, 2H), 2.16-2.06 (m, 4H), 1.59-1.53 (m, 1H), 1.47-1.40 (m, 1H), 1.12 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 196.6, 170.9, 170.8, 133.8, 131.8, 130.6, 128.6, 126.8 (q, J = 274.6 Hz), 81.8, 81.0, 55.9, 52.8, 52.7, 33.8 (q, J = 28.6 Hz), 33.1, 32.3, 26.3, 17.2 (q, J = 3.0 Hz); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.2 (s, 3F); HRMS (ESI) calcd for $C_{22}H_{28}BrF_3O_7Na$ $(M+Na)^+$: 563.0863; found: 563.0870.

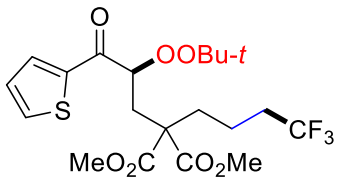


Dimethyl-2-(2-(*tert*-butylperoxy)-3-oxo-3-(4-(trifluoromethyl)phenyl)propyl)-2-(4,4,4-trifluorobutyl)malonate (4j): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 7:1, R_f = 0.4) in 59% yield (63 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.20 (d, J = 8.2 Hz, 2H), 7.74 (d, J = 8.3 Hz, 2H), 5.11 (dd, J = 8.3, 4.0 Hz, 1H), 3.74 (s, 6H), 2.52-2.43 (m, 2H), 2.17-2.05 (m, 4H), 1.61-1.53 (m, 1H), 1.48-1.41 (m, 1H), 1.11 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 196.9, 170.9, 137.8, 134.5 (q, J = 32.6 Hz), 129.4, 126.8 (q, J = 275.0 Hz), 125.5 (q, J = 3.4 Hz), 123.6 (q, J = 270.8 Hz), 82.1, 81.2, 55.8, 52.9, 52.8, 33.8 (q, J = 28.9 Hz), 33.0, 32.3, 26.3, 17.2 (q, J = 2.8 Hz); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -63.2 (s, 3F), -66.3 (s, 3F); HRMS (ESI) calcd for $C_{23}H_{28}F_6O_7Na$ $(M+Na)^+$: 553.1631; found: 553.1631.

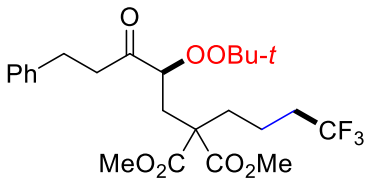


Dimethyl-2-(2-(*tert*-butylperoxy)-3-(naphthalen-2-yl)-3-oxopropyl)-2-(4,4,4-trifluorobutyl)malonate (4k): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 7:1, R_f = 0.4) in 36% yield (37 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.66 (s, 1H), 8.09 (dd, J = 8.6, 1.5 Hz, 1H), 7.97 (d, J = 8.1 Hz, 1H), 7.91 (d, J = 8.6 Hz, 1H), 7.88 (d, J = 8.1

Hz, 1H), 7.62 (t, *J* = 7.0 Hz, 1H), 7.57 (t, *J* = 7.9 Hz, 1H), 5.29 (dd, *J* = 7.3, 5.3 Hz, 1H), 3.74 (s, 3H), 3.73 (s, 3H), 2.56-2.50 (m, 2H), 2.21-2.09 (m, 4H), 1.63-1.56 (m, 1H), 1.50-1.43 (m, 1H), 1.14 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 197.4, 171.0, 170.9, 135.7, 132.5, 132.4, 131.0, 129.8, 128.7, 128.4, 127.8, 126.8 (q, *J* = 274.7 Hz), 126.8, 124.6, 81.5, 80.9, 55.9, 52.8, 52.7, 33.9 (q, *J* = 28.5 Hz), 33.3, 32.2, 26.4, 17.3 (q, *J* = 2.5 Hz); ^{19}F NMR (564 MHz, $CDCl_3$) δ -66.2 (s, 3F); HRMS (ESI) calcd for $C_{26}H_{31}F_3O_7Na$ $(M+Na)^+$: 535.1914; found: 535.1921.

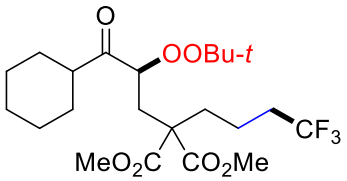


Dimethyl-2-(2-(*tert*-butylperoxy)-3-oxo-3-(thiophen-2-yl)propyl)-2-(4,4,4-trifluorobutyl)malonate (4l): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 7:1, R_f = 0.3) in 41% yield (38 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.99 (d, *J* = 3.8 Hz, 1H), 7.69 (d, *J* = 4.9 Hz, 1H), 7.16 (t, *J* = 4.1 Hz, 1H), 4.88 (dd, *J* = 9.0, 3.8 Hz, 1H), 3.76 (s, 3H), 3.74 (s, 3H), 2.51-2.42 (m, 2H), 2.19-2.05 (m, 4H), 1.59-1.51 (m, 1H), 1.49-1.43 (m, 1H), 1.17 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 191.0, 170.8, 140.7, 134.3, 133.6, 128.1, 126.8 (q, *J* = 274.7 Hz), 83.1, 81.2, 55.8, 52.8, 52.7, 33.9 (q, *J* = 28.6 Hz), 33.8, 31.9, 26.4, 17.2 (q, *J* = 2.6 Hz); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.2 (s, 3F); HRMS (ESI) calcd for $C_{20}H_{27}F_3O_7SNa$ $(M+Na)^+$: 491.1322; found: 491.1328.

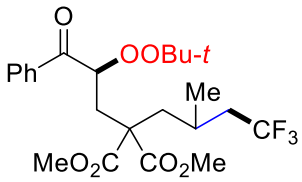


Dimethyl 2-(2-(*tert*-butylperoxy)-3-oxo-5-phenylpentyl)-2-(4,4,4-trifluorobutyl)malonate (4m): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 6:1, R_f = 0.4) in 58% yield (57 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.28 (t, *J* = 7.6 Hz, 2H), 7.22-7.18 (m, 3H), 4.24 (dd, *J* = 8.4, 4.2 Hz, 1H), 3.730 (s, 3H), 3.726 (s, 3H), 3.10-3.03 (m, 1H), 2.94-2.88 (m, 3H), 2.20-2.14 (m, 2H), 2.12-2.05 (m, 3H), 2.01-1.96 (m, 1H), 1.51-1.38 (m, 2H), 1.19 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 209.9, 170.8, 141.2, 128.5, 128.4, 126.8 (q, *J* = 274.9 Hz), 126.1, 84.5, 81.2, 55.6, 52.9, 52.7, 38.8, 33.8 (q, *J* = 28.6 Hz), 32.5, 31.6, 29.2, 26.4, 17.1 (q, *J* = 2.8 Hz); ^{19}F NMR (564

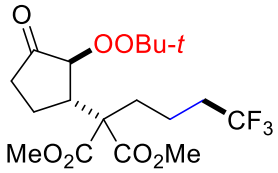
MHz, $CDCl_3$, ppm) δ -66.2 (s, 3F); HRMS (ESI) calcd for $C_{24}H_{33}F_3O_7Na$ $(M+Na)^+$: 513.2071; found: 513.2059.



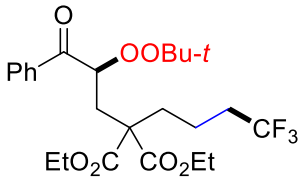
Dimethyl-2-(2-(*tert*-butylperoxy)-3-cyclohexyl-3-oxopropyl)-2-(4,4,4-trifluorobutyl)malonate (4n): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 6:1, R_f = 0.4) in 68% yield (64 mg); Colorless oil; ^1H NMR (600 MHz, $CDCl_3$, ppm) δ 4.34 (dd, J = 8.9, 3.4 Hz, 1H), 3.75 (s, 3H), 3.746 (s, 3H), 2.87-2.82 (m, 1H), 2.26-2.20 (m, 2H), 2.15-2.06 (m, 3H), 2.04-1.99 (m, 1H), 1.85-1.78 (m, 4H), 1.56-1.49 (m, 1H), 1.47-1.41 (m, 1H), 1.40-1.23 (m, 6H), 1.20 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 213.2, 171.0, 170.90, 126.8 (q, J = 274.7 Hz), 83.1, 80.8, 55.8, 52.8, 52.7, 45.8, 33.8 (q, J = 28.9 Hz), 32.5, 31.7, 29.2, 28.2, 26.4, 25.8, 25.5, 17.1 (q, J = 2.4 Hz); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.3 (s, 3F); HRMS (ESI) calcd for $C_{22}H_{35}F_3O_7Na$ $(M+Na)^+$: 491.2227; found: 491.2229.



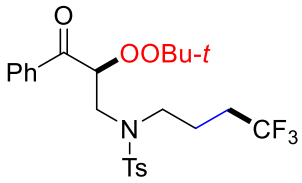
Dimethyl 2-(2-(*tert*-butylperoxy)-3-oxo-3-phenylpropyl)-2-(4,4,4-trifluoro-2-methylbutyl) malonate (4o): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 7:1, R_f = 0.4) in 68% yield (65 mg); Colorless oil (d:r = 1:1); ^1H NMR (600 MHz, $CDCl_3$, ppm) δ 8.07-8.05 (m, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.4 Hz, 2H), 5.19-5.15 (m, 1H), 3.72 (s, 3H), 3.71 (d, J = 1.6 Hz, 3H), 2.56-2.51 (m, 1H), 2.47-2.41 (m, 1H), 2.23-2.11 (m, 2H), 2.08-1.96 (m, 3H), 1.12 (d, J = 4.0 Hz, 9H), 0.99 (d, J = 5.4 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 197.7, 197.5, 171.5, 171.1, 171.0, 135.2, 135.1, 133.4, 133.3, 129.1, 129.0, 128.5, 128.4, 126.7 (q, J = 275.7 Hz), 81.4, 81.3, 80.9, 80.8, 55.2, 55.0, 52.8, 52.7, 52.6, 52.5, 41.2 (q, J = 26.6 Hz), 41.1 (q, J = 27.0 Hz), 40.3, 39.9, 33.9, 33.8, 26.4, 24.3 (q, J = 1.6 Hz), 20.4, 20.2; ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -63.0 (s, 3F), -66.1 (s, 3F); HRMS (ESI) calcd for $C_{23}H_{31}F_3O_7Na$ $(M+Na)^+$: 499.1914; found: 499.1914.



Dimethyl 2-((2R)-2-(*tert*-butylperoxy)-3-oxocyclopentyl)-2-(4,4,4-trifluorobutyl)malonate (4p): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 6:1, R_f = 0.4) in 52% yield (43 mg); Colorless oil (dr > 20:1); ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 4.25 (d, J = 8.4 Hz, 1H), 3.77 (s, 3H), 3.74 (s, 3H), 3.15-3.11 (m, 1H), 2.34-2.25 (m, 3H), 2.16-2.03 (m, 4H), 1.88-1.80 (m, 1H), 1.67-1.60 (m, 1H), 1.52-1.44 (m, 1H), 1.20 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 211.1, 169.8, 169.7, 125.8 (q, J = 274.7 Hz), 83.8, 79.7, 58.8, 51.4, 51.4, 41.0, 35.6, 32.9 (q, J = 28.5 Hz), 32.6, 25.3, 19.9, 16.5 (q, J = 2.5 Hz); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.1 (s, 3F); HRMS (ESI) calcd for $C_{18}H_{27}F_3O_7Na$ $(M+Na)^+$: 435.1601; found: 435.1604.

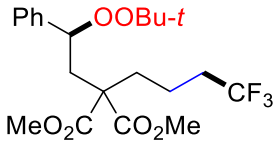


Diethyl-2-(2-(*tert*-butylperoxy)-3-oxo-3-phenylpropyl)-2-(4,4,4-trifluorobutyl)malonate (4q): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 7:1, R_f = 0.4) in 70% yield (69 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.06 (d, J = 7.9 Hz, 2H), 7.58 (t, J = 7.1 Hz, 1H), 7.46 (t, J = 7.6 Hz, 2H), 5.14 (dd, J = 7.7, 4.4 Hz, 1H), 4.23-4.15 (m, 4H), 2.49-2.42 (m, 2H), 2.17-2.05 (m, 4H), 1.63-1.55 (m, 1H), 1.49-1.41 (m, 1H), 1.25-1.21 (m, 6H), 1.12 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 197.7, 170.5, 170.4, 135.3, 133.3, 129.0, 128.5, 126.8 (q, J = 274.5 Hz), 81.6, 80.8, 61.7, 61.6, 56.0, 34.0 (q, J = 28.7 Hz), 32.9, 32.0, 26.3, 17.2 (q, J = 2.6 Hz), 13.9; ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.3 (s, 3F); HRMS (ESI) calcd for $C_{24}H_{33}F_3O_7Na$ $(M+Na)^+$: 513.2071; found: 513.2070.

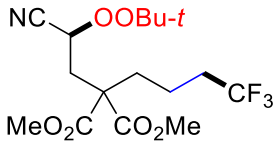


N-(2-(*tert*-butylperoxy)-3-oxo-3-phenylpropyl)-4-methyl-N-(4,4,4-

trifluorobutyl)benzenesulfonamide (4r): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 6:1, R_f = 0.4) in 42% yield (42 mg); Yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.06 (d, J = 7.2 Hz, 2H), 7.67 (d, J = 8.3 Hz, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 8.0 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 5.65 (dd, J = 8.0, 3.6 Hz, 1H), 3.73 (dd, J = 15.7, 3.5 Hz, 1H), 3.43-3.38 (m, 1H), 3.34 (dd, J = 15.7, 8.1 Hz, 1H), 3.24-3.19 (m, 1H), 2.42 (s, 3H), 2.13-2.03 (m, 2H), 1.92-1.85 (m, 2H), 1.10 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 196.4, 143.9, 135.8, 135.3, 133.8, 129.9, 129.0, 128.7, 127.3, 126.9 (q, J = 274.6 Hz), 83.2, 81.1, 49.4, 47.8, 31.2 (q, J = 29.2 Hz), 26.3, 21.5, 21.1 (q, J = 2.4 Hz); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.0 (s, 3F); HRMS (ESI) calcd for $C_{24}H_{30}F_3NO_5SNa$ $(M+Na)^+$: 524.1689; found: 524.1689.

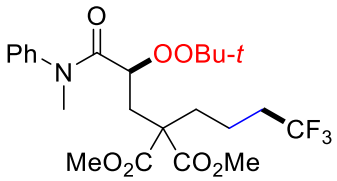


Dimethyl 2-(2-(*tert*-butylperoxy)-2-phenylethyl)-2-(4,4,4-trifluorobutyl)malonate (4s): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 10:1, R_f = 0.5) in 61% yield (53 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.35-7.28 (m, 5H), 4.78 (dd, J = 7.4, 5.6 Hz, 1H), 3.72 (s, 3H), 3.56 (s, 3H), 2.61 (dd, J = 15.2, 7.9 Hz, 1H), 2.37 (dd, J = 15.2, 5.2 Hz, 1H), 2.16-2.00 (m, 4H), 1.58-1.50 (m, 1H), 1.41-1.34 (m, 1H), 1.11 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 171.2, 171.1, 140.8, 128.2, 128.0, 127.3, 126.8 (q, J = 275.0 Hz), 81.9, 80.0, 55.9, 52.6, 52.5, 37.1, 33.9 (q, J = 28.5 Hz), 31.5, 26.5, 17.1 (q, J = 2.9 Hz); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.3 (s, 3F); HRMS (ESI) calcd for $C_{21}H_{29}F_3O_6Na$ $(M+Na)^+$: 457.1808; found: 457.1804.



Dimethyl 2-(2-(tert-butylperoxy)-2-cyanoethyl)-2-(4,4,4-trifluorobutyl)malonate (4t): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 10:1, R_f = 0.4) in 22% yield (17 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 4.75 (dd, J = 7.7, 4.9 Hz, 1H), 3.774 (s, 3H), 3.77 (s, 3H), 2.55-2.47 (m, 2H), 2.15-2.08 (m, 2H), 2.05-1.99 (m, 2H), 1.56-1.51 (m, 1H), 1.46-1.39 (m, 1H), 1.26 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 170.2 (d), 126.7 (q, J = 274.7 Hz), 117.7, 82.2, 68.6, 55.3, 53.1, 53.0, 51.9, 34.4, 33.7 (q, J = 28.8 Hz), 32.4, 26.3, 17.2 (q, J = 2.9 Hz); ^{19}F NMR (564

MHz, $CDCl_3$, ppm) δ -66.3 (s, 3F); HRMS (ESI) calcd for $C_{16}H_{24}F_3NO_6Na$ $(M+Na)^+$: 406.1448; found: 406.1446.



Dimethyl-2-(2-(tert-butylperoxy)-3-(methyl(phenyl)amino)-3-oxopropyl)-2-(4,4,4-trifluorobutyl)malonate (4u) Isolated by flash column chromatography (ethyl acetate/petroleum ether = 5:1, R_f = 0.4) in 43% yield (42 mg); Colorless oil; ^1H NMR (600 MHz, $CDCl_3$, ppm) δ 7.42 (t, J = 7.3 Hz, 2H), 7.36 (t, J = 7.4 Hz, 1H), 7.25 (d, J = 7.3 Hz, 2H), 4.45 (dd, J = 10.4, 2.8 Hz, 1H), 3.68 (s, 3H), 3.57 (s, 3H), 3.30 (s, 3H), 2.49 (dd, J = 15.6, 7.0 Hz, 1H), 2.19 (dd, J = 15.6, 2.8 Hz, 1H), 1.94-1.88 (m, 2H), 1.87-1.80 (m, 1H), 1.69-1.63 (m, 1H), 1.37-1.30 (m, 1H), 1.17 (s, 9H), 1.12-1.06 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 170.8 (d), 169.9, 143.1, 129.7, 128.0, 127.3, 127.2 (q, J = 275.1 Hz), 80.9, 75.4, 55.3, 52.64, 52.6, 37.9, 33.8 (q, J = 28.6 Hz), 33.4, 31.5, 26.3, 17.0 (q, J = 2.3 Hz); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.2 (s, 3F); HRMS (ESI) calcd for $C_{16}H_{24}F_3NO_6Na$ $(M+Na)^+$: 514.2023; found: 514.2022.

4. TGA of the compound 4a

In order to investigate the decomposition of the peroxide, we carried out the TGA (thermal gravimetric analysis) test of the peroxide **4a** (Figure S1). The sharp peak at 240 °C indicated that the decomposition temperature the peroxid **4a** is 240 °C.

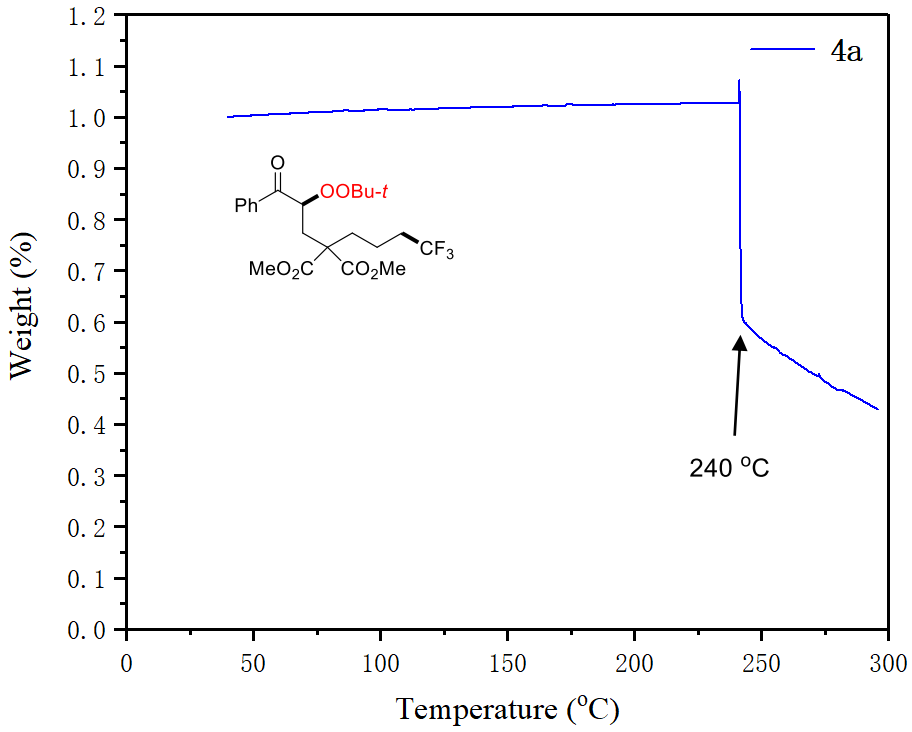
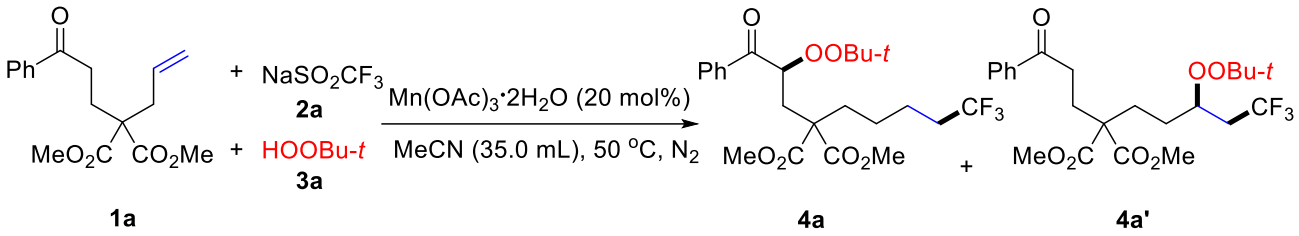
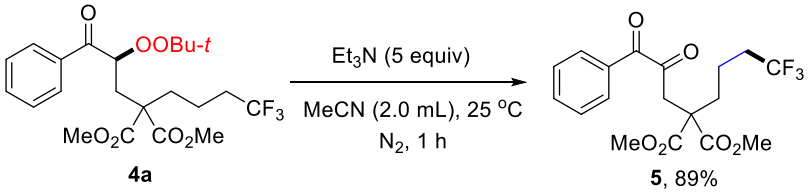


Figure S1. TGA test for the peroxide **4a**

5. Large scale synthesis and transformations

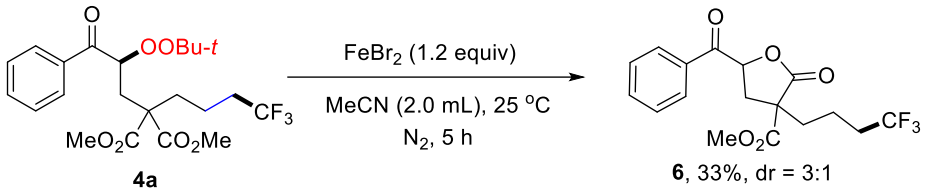


To a 50 mL Schlenk tube were added unactivated alkenes **1a** (3.5 mmol), $Mn(OAc)_3{\cdot}2H_2O$ (0.7 mmol), CF_3SO_2Na (7.0 mmol), MeCN (35.0 mL) and *t*-BuOOH (70% in water, 17.5 mmol) under N_2 atmosphere at room temperature, and the resulting solution was stirred at 50°C for 3 h. The resulting mixture and the solvent were evaporated under vacuum. The residue was purified by flash column chromatography on silica gel (eluent: ethyl acetate/petroleum ether) to give the peroxides **4a** and **4a′** in 52% yield (0.84 g, **4a**:**4a′** = 16:1).



To a 25 mL Schlenk tube with a magnetic stir bar were added **4a** (46.2 mg, 0.1 mmol), Et_3N (69 μL,

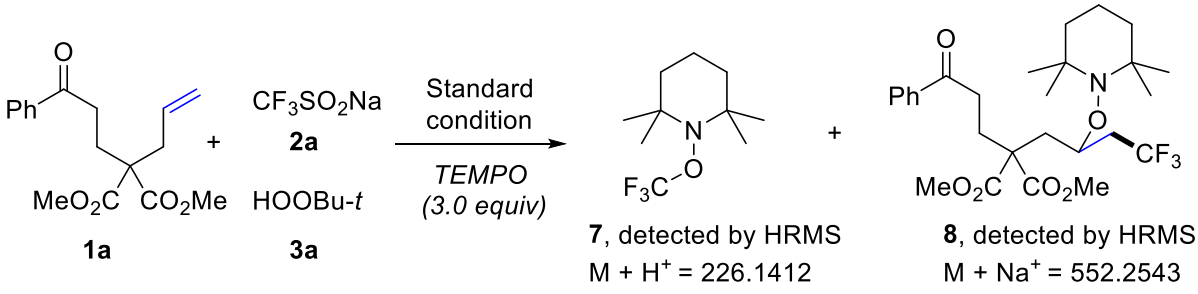
0.5 mmol) in MeCN (2.0 ml) and then allowed to stir at room temperature under N_2 atmosphere for 1 h. The resultant residue was purified by flash chromatography (ethyl acetate/petroleum ether = 1/10-1/8) to afford dimethyl 2-(2,3-dioxo-3-phenylpropyl)-2-(4,4,4-trifluorobutyl)malonate (**5**) (34.5 mg, 89% yield) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.00 (d, J = 7.9 Hz, 2H), 7.65 (t, J = 7.4 Hz, 1H), 7.50 (t, J = 7.9 Hz, 2H), 3.78 (s, 6H), 3.51 (s, 2H), 2.17-2.07 (m, 4H), 1.63-1.55 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 198.7, 190.7, 170.6, 134.7, 131.7, 130.5, 128.8, 126.7 (q, J = 274.8 Hz), 55.7, 53.0, 41.2, 33.8 (q, J = 28.8 Hz), 32.2, 17.7; HRMS (ESI) calcd for $C_{18}H_{19}F_3O_6Na$ $(M+Na)^+$: 411.1026; found: 411.1025.



To a 25 mL Schlenk tube equipped with a magnetic stir bar were added **4a** (46.2 mg, 0.1 mmol), $FeBr_2$ (25.9 mg, 0.12 mmol) in MeCN (2.0 ml) and then allowed to stir at room temperature under N_2 atmosphere for 5 h. The resultant residue was purified by flash chromatography (ethyl acetate/petroleum ether = 1/12-1/8) to afford methyl 5-benzoyl-2-oxo-3-(4,4,4-trifluorobutyl) tetrahydrofuran-3-carboxylate (**6**) (12 mg, 33% yield) as a colourless oil.[5] ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.02 (d, J = 7.4 Hz, 2H), 7.66 (d, J = 7.4 Hz, 1H), 7.50 (t, J = 7.8 Hz, 2H), 5.77 (dd, J = 9.0, 7.3 Hz, 1H), 3.86 (s, 3H), 3.04 (dd, J = 13.6, 7.2 Hz, 1H), 2.46 (dd, J = 13.6, 9.2 Hz, 1H), 2.15-2.08 (m, 2H), 1.72-1.47 (m, 4H). HRMS (ESI) calcd for $C_{17}H_{17}F_3O_5Na$ $(M+Na)^+$: 381.0920; found: 381.0919.

6. Mechanistic studies

Control experiments



To a 25 mL Schlenk tube were added **1a** (0.2 mmol), $Mn(OAc)_3{\cdot}2H_2O$ (0.04 mmol), CF_3SO_2Na (0.4 mmol), TEMPO (0.6 mmol), MeCN (2.0 mL) and *t*-BuOOH (70% in water, 1.0 mmol) under N_2 atmosphere at room temperature, and the resulting solution was stirred at 50℃ for 3 h. The model reaction was totally supressed where alkene **1a** was almost fully recovered, and we can detect the molecular weight of compounds **7** and **8**.

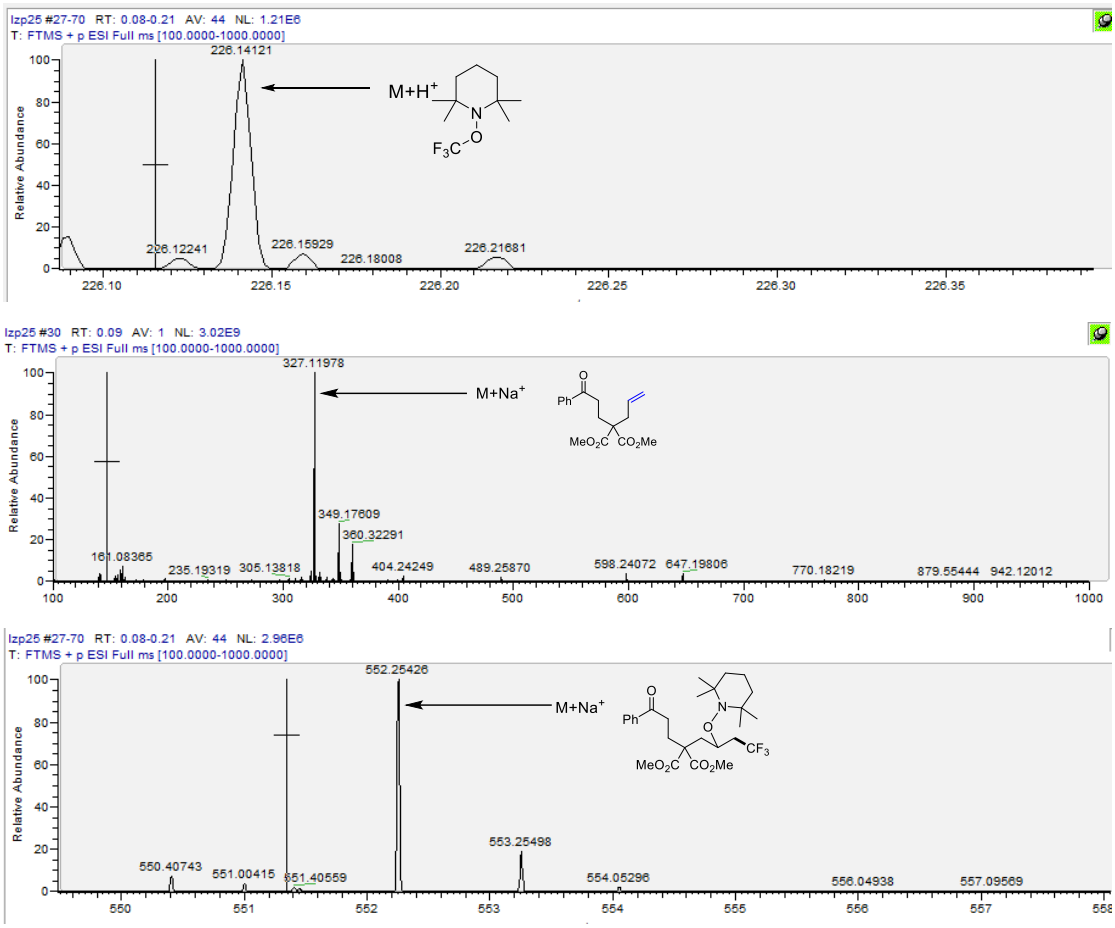
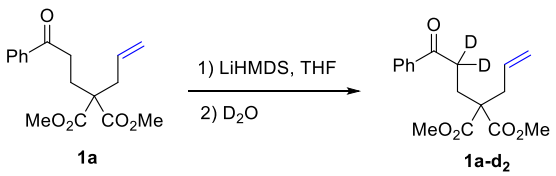


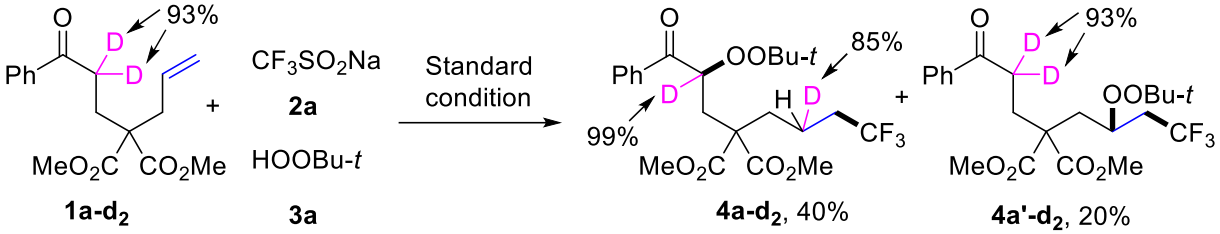
Figure S2. HRMS of the reaction solution

Deuterium labeling experiments



Experimental procedures for the synthesis of **1a-d$_2$**[5]: To a solution of **1a** (608.0 mg, 2.0 mmol) in THF (3.0 mL) at -78 °C was slowly added a solution of LiHMDS (5.0 mL, 1.0 M in THF) under N_2. After being stirred for 1 h at -78 °C, the reaction mixture was quenched by slowly sequential addition

of D_2O (2.0 mL). Then the mixture was warmed to room temperature, stirred for an additional 30 mins, and extracted with ethyl acetate (3 x 5.0 mL). The combined organic extracts were washed with brine (5.0 mL), dried over Na_2SO_4, filtered, and concentrated. The residue was repeated three times as the procedure described above and purified by flash chromatography on silica gel (ethyl acetate/petroleum ether = 1/12-1/6) to give the dimethyl 2-allyl-2-(3-(4-bromophenyl)-3-oxopropyl) malonate-d_2 (**1a-d$_2$**) (535.0 mg, 88% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.95 (d, J = 7.2 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.8 Hz, 2H), 5.75-5.65 (m, 1H), 5.17-5.11 (m, 2H), 3.73 (s, 6H), 3.03-2.92 (m, 0.14H), 2.72 (d, J = 7.4 Hz, 2H), 2.05 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 198.9, 171.4, 136.7, 133.1, 132.2, 128.6, 128.1, 119.4, 57.1, 52.5, 38.5, 27.3; HRMS (ESI) calcd for $C_{18}H_{19}F_3O_6Na$ $(M+Na)^+$: 411.1026; found: 411.1026.



To a 25 mL Schlenk tube were added unactivated alkenes **1** (0.2 mmol), $Mn(OAc)_3{\cdot}2H_2O$ (0.04 mmol), CF_3SO_2Na (0.4 mmol), MeCN (2.0 mL) and *t*-BuOOH (70% in water, 1.0 mmol) under N_2 atmosphere at room temperature, and the resulting solution was stirred at 50°C for 3 h. Solvent was removed under reduced pressure, and the crude residue was purified by silica gel column chromatography (ethyl acetate/petroleum ether = 1/20-1/15) to give **4a-d$_2$** and **4a′-d$_2$** in 40% and 20% yields, respectively.

4a-d$_2$: Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.05 (d, J = 7.9 Hz, 2H), 7.58 (t, J = 7.3 Hz, 1H), 7.47 (t, J = 7.6 Hz, 2H), 5.17 (dd, J = 8.1, 4.2 Hz, 0.016H), 3.73 (s, 6H), 2.50-2.43 (m, 2H), 2.18-2.06 (m, 4H), 1.61-1.53 (m, 1H), 1.47-1.40 (m, 1H), 1.13 (s, 9H); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -66.3 (s, 3F); HRMS (ESI) calcd for $C_{22}H_{27}D_2F_3O_7Na$ $(M+Na)^+$: 487.1883; found: 487.1880.

4a′-d$_2$: Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.95 (d, J = 7.9 Hz, 1.0H), 7.56 (t, J = 7.3 Hz, 0.5H), 7.46 (t, J = 7.6 Hz, 1.0H), 4.28-4.24 (m, 0.5H), 3.73 (d, J = 2.8 Hz, 3.0H), 2.89-2.80 (m, 0.5H), 2.44-2.32 (m, 2.0H), 2.28-2.23 (m, 0.5H), 1.16 (s, 4.5H); ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -62.9 (s, 3F); HRMS (ESI) calcd for $C_{22}H_{27}D_2F_3O_7Na$ $(M+Na)^+$: 487.1883; found: 487.1880.

7. References

[1] A. G. Campaña, N. Fuentes, E. Gómez-Bengoa, C. Mateo, J. E. Oltra, A. M. Echavarren and J. M. Cuerva, *J. Org. Chem.*, 2007, **72**, 8127-8130.

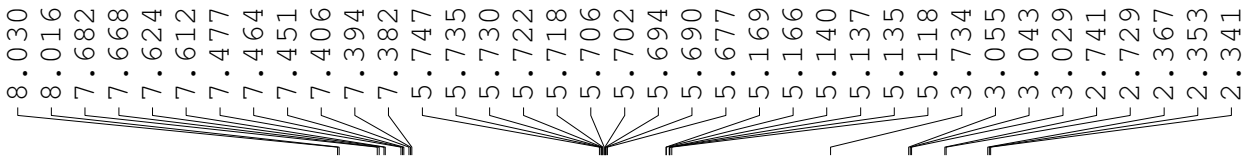
[2] Y.-P. Xiao, X.-Y. Liu and C.-M. Che, *Angew. Chem., Int. Ed.*, 2011, **50**, 4937-4941.

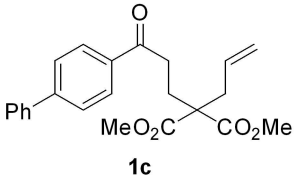
[3] D. Necas, M. Tusky and M. Kotora, *J. Am. Chem. Soc.*, 2004, **126**, 10222-10223.

[4] T. Azemi, M. Kitamura and K. Narasaka, *Tetrahedron*, 2004, **60**, 1339-1344.

[5] L. Huang, S. C. Zheng, B. Tan and X. Y. Liu, *Org. Lett.*, 2015, **17**, 1589-1592.

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





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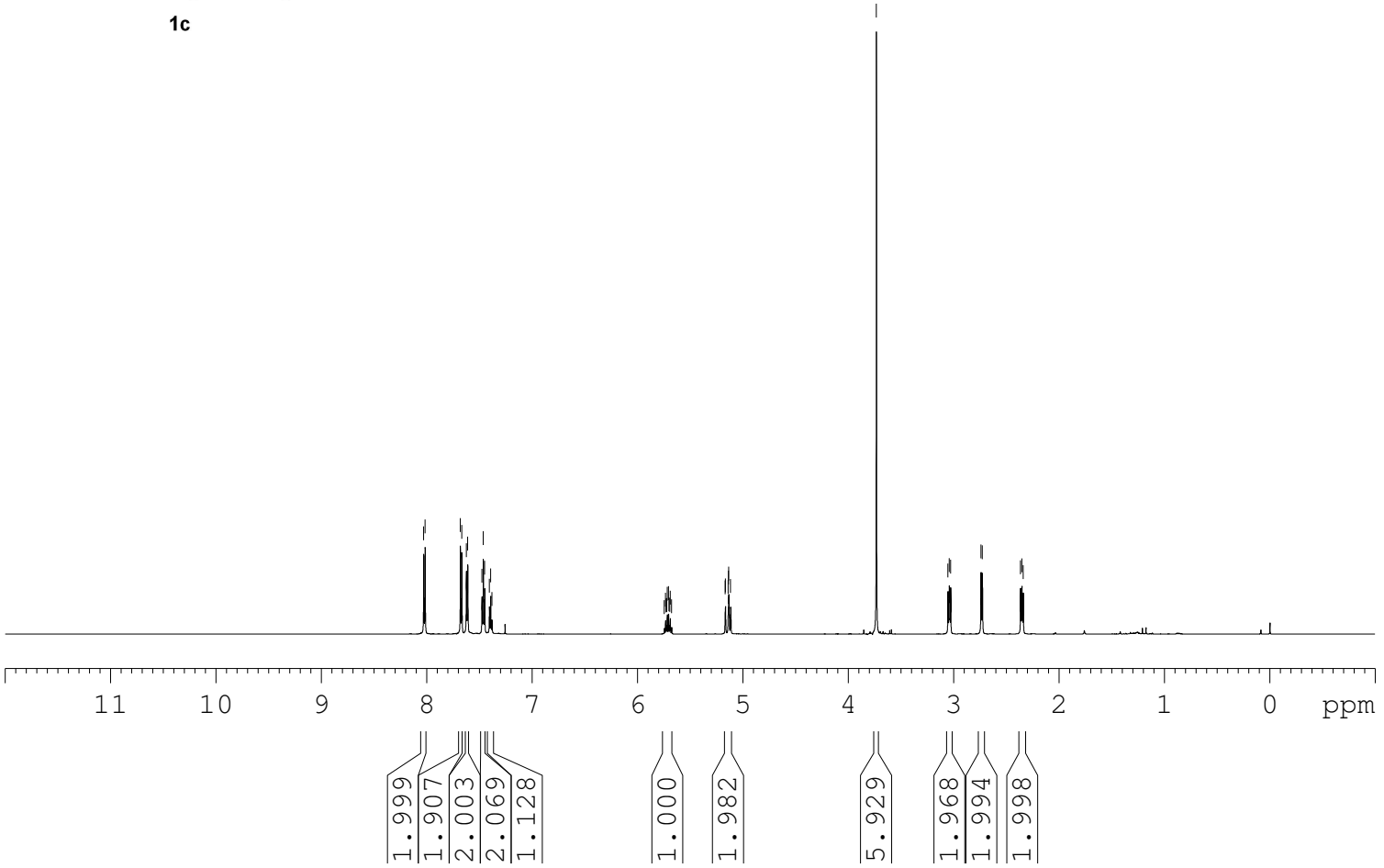


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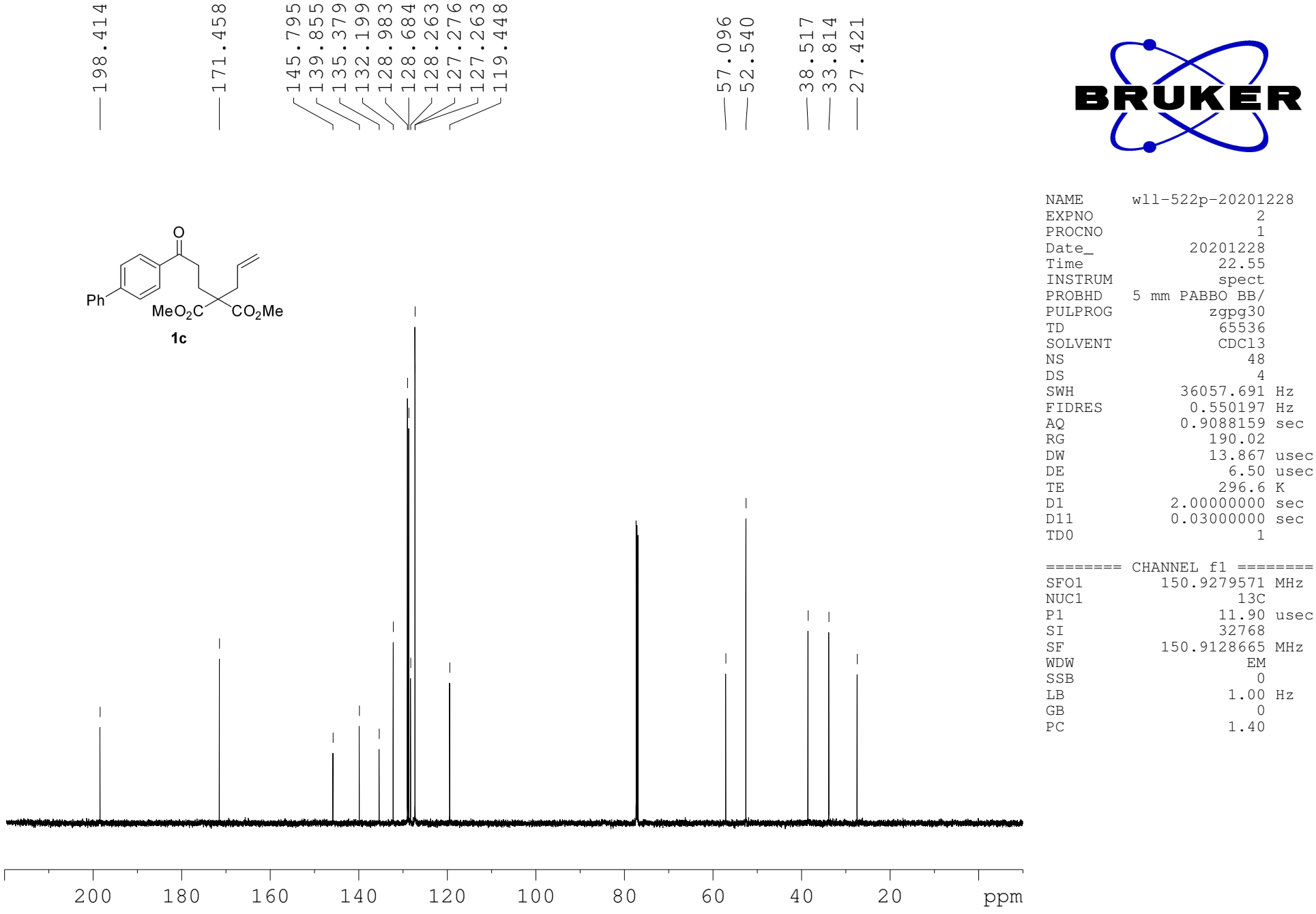

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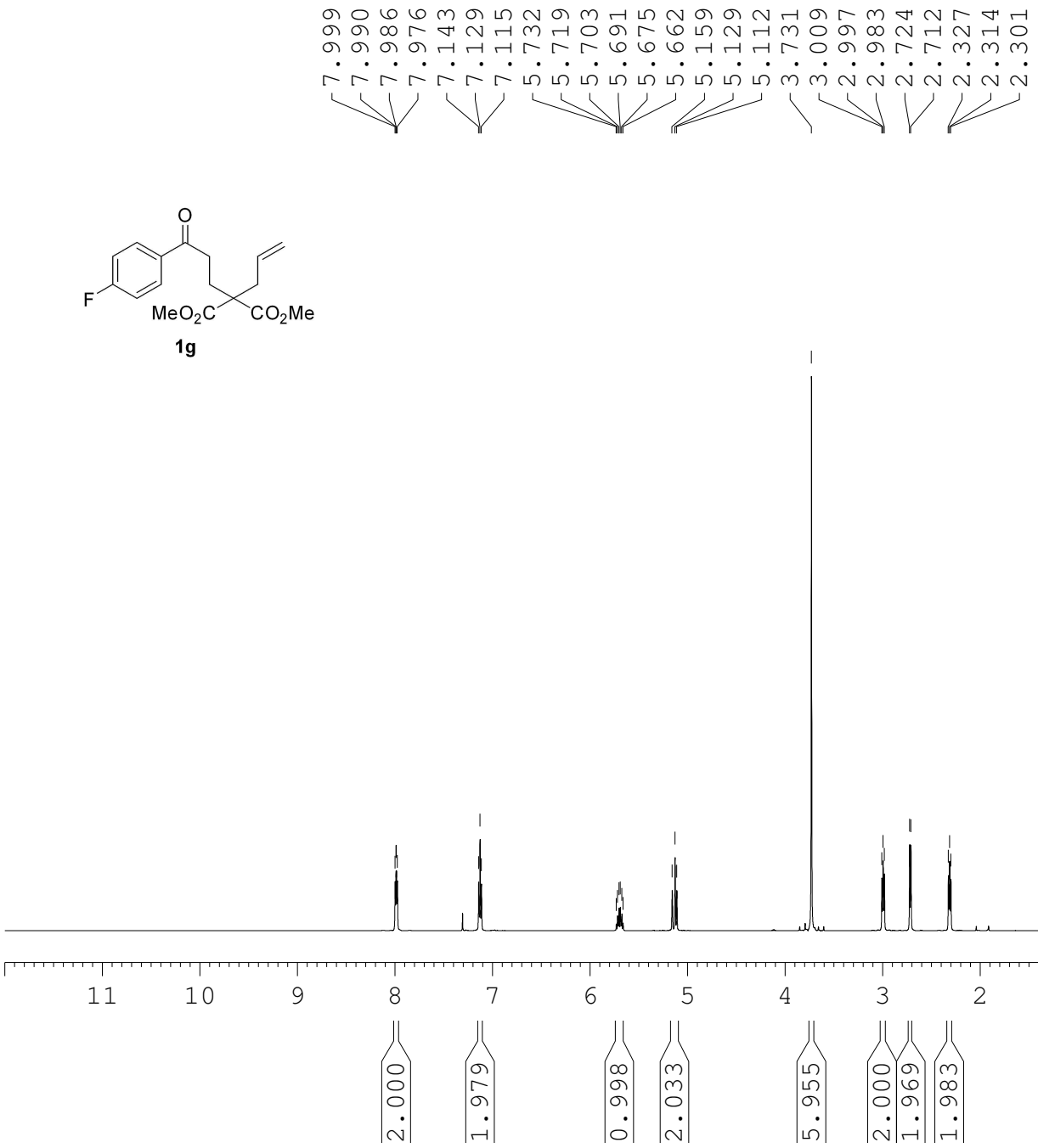

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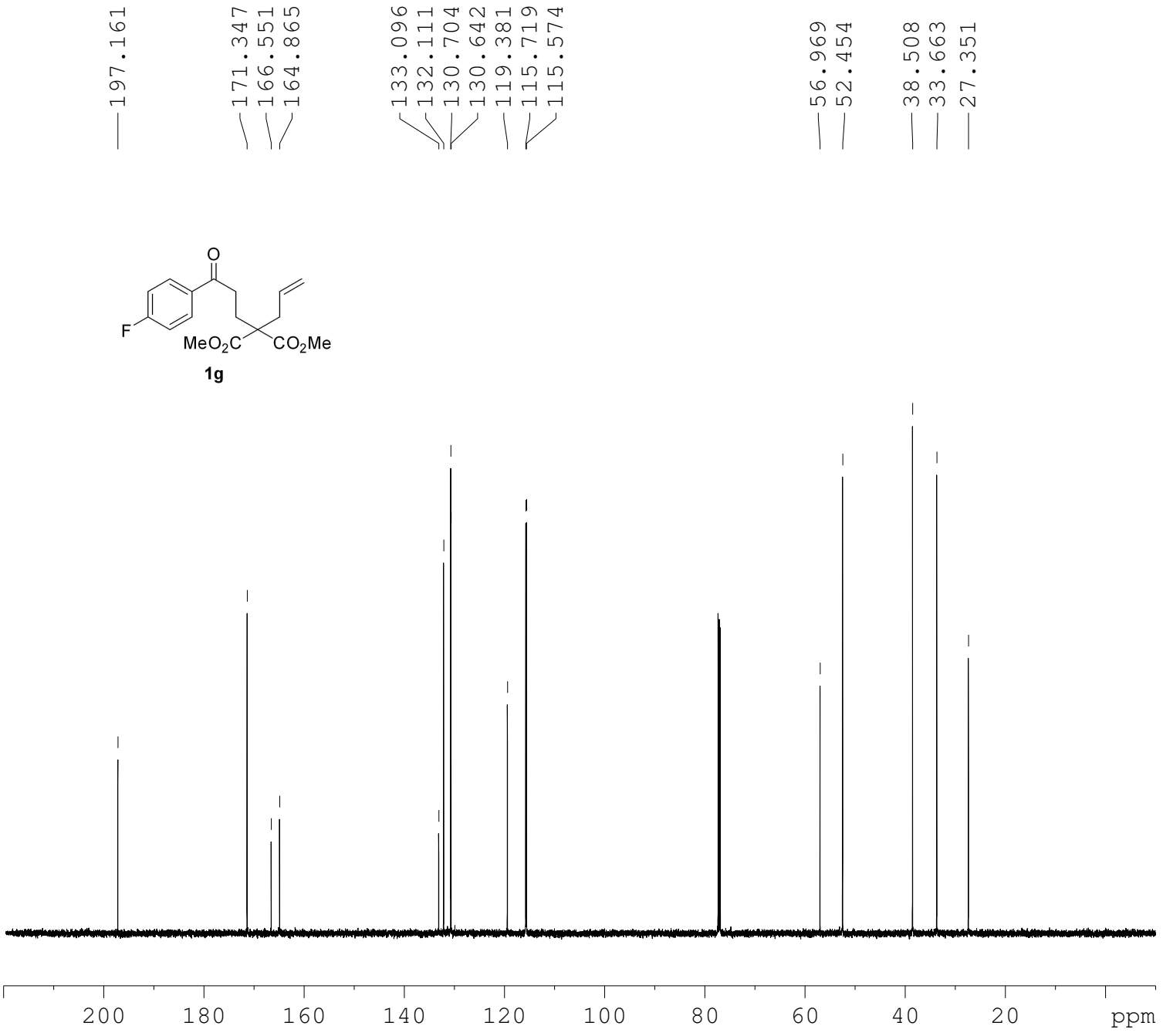

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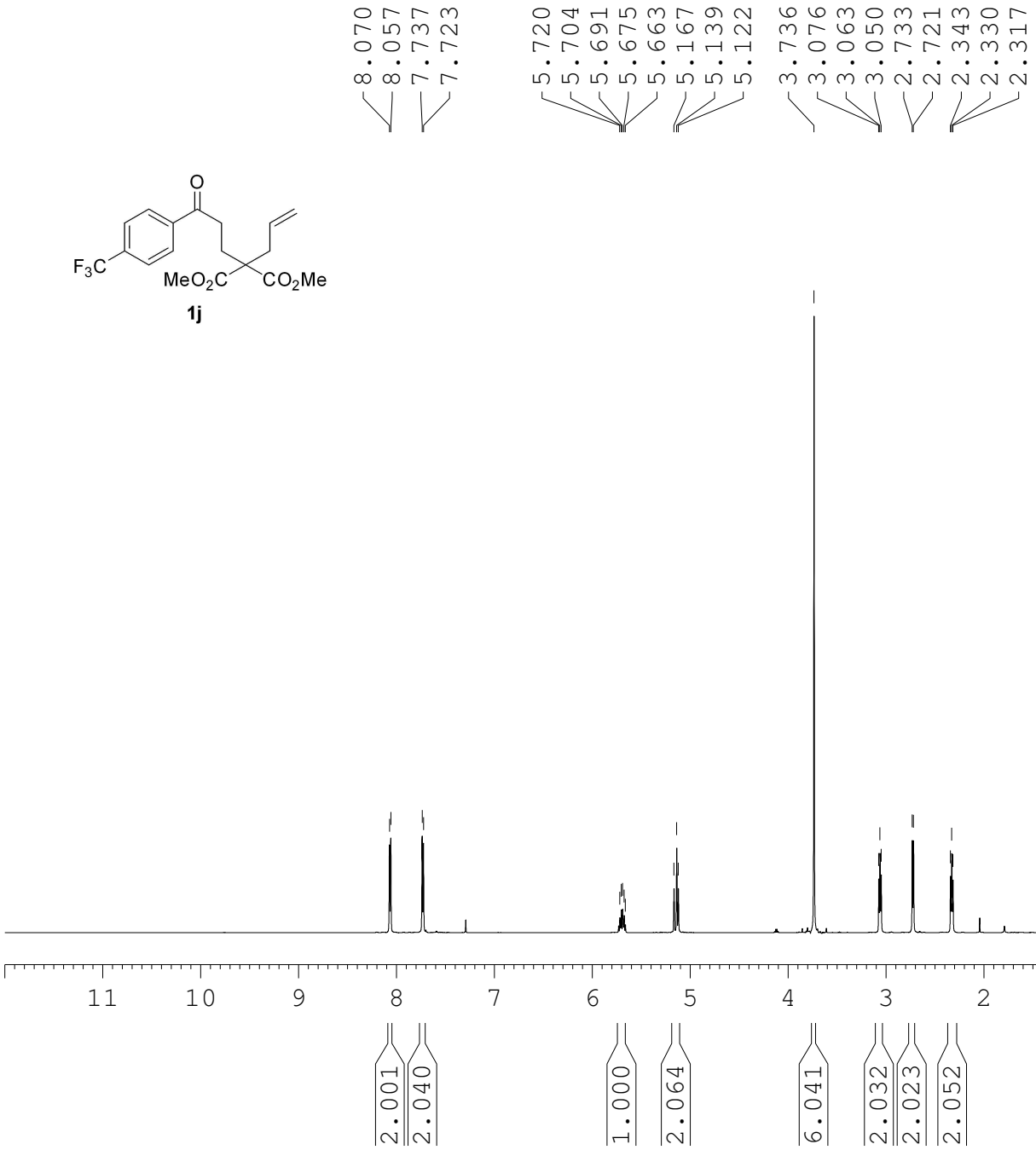

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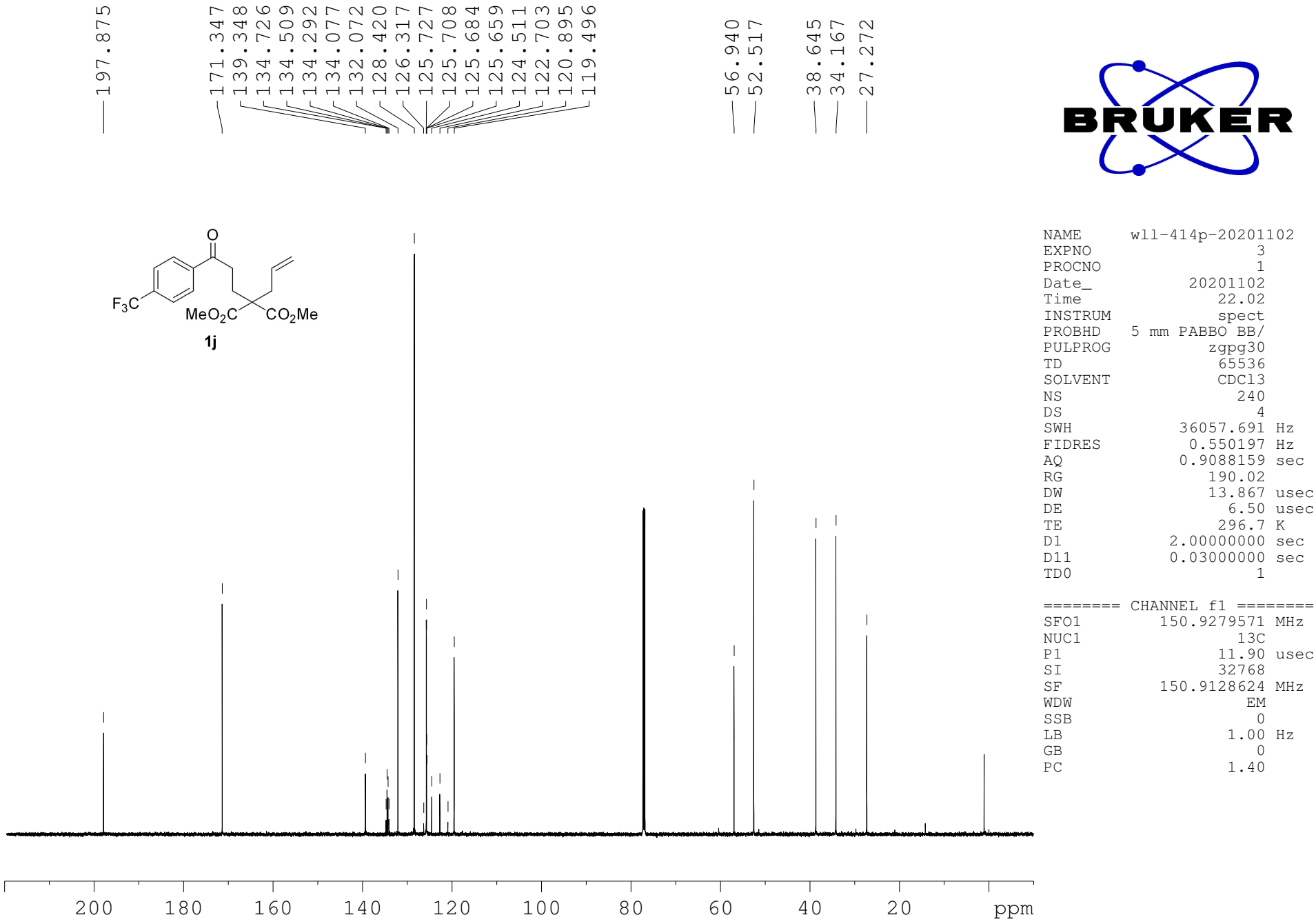

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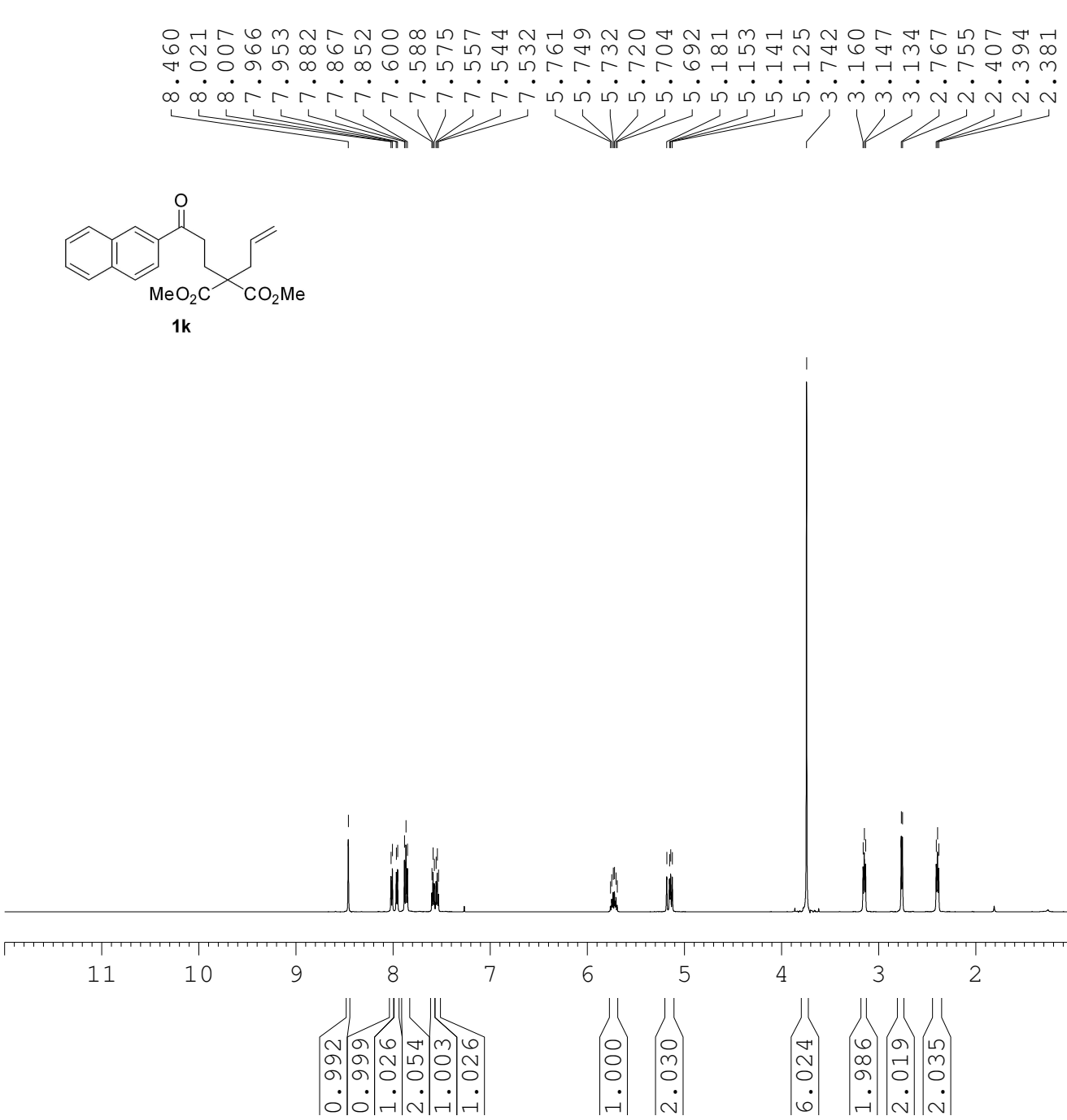
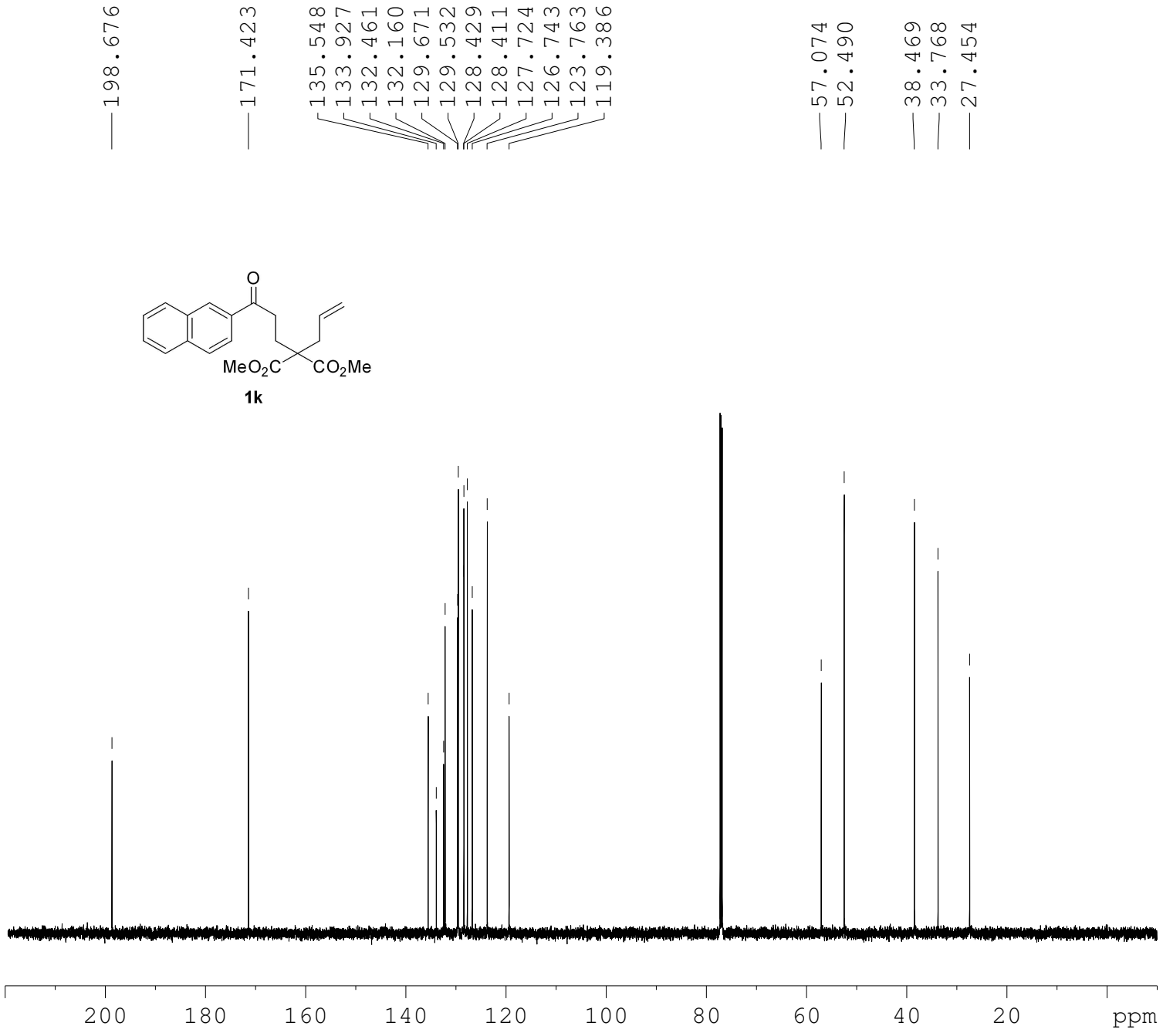
GB	0
PC	1.40

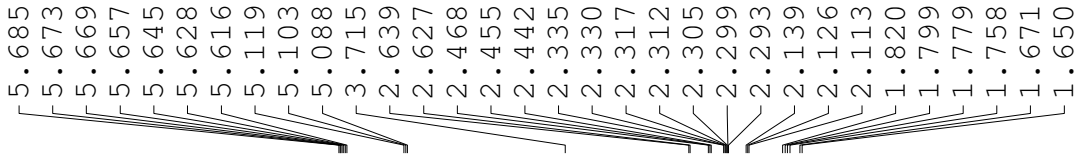


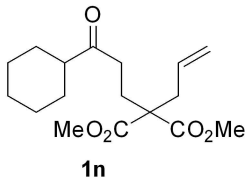

```
NAME      wll-372p-20201019
EXPNO                    1
PROCNO                   1
Date_             20201019
Time                 19.39
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               9615.385 Hz
FIDRES            0.146719 Hz
AQ                3.4079220 sec
RG                   28.69
DW                  52.000 usec
DE                   6.50 usec
TE                  295.8 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           600.1739011 MHz
NUC1                    1H
P1                    9.77 usec
SI                   65536
SF             600.1700118 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



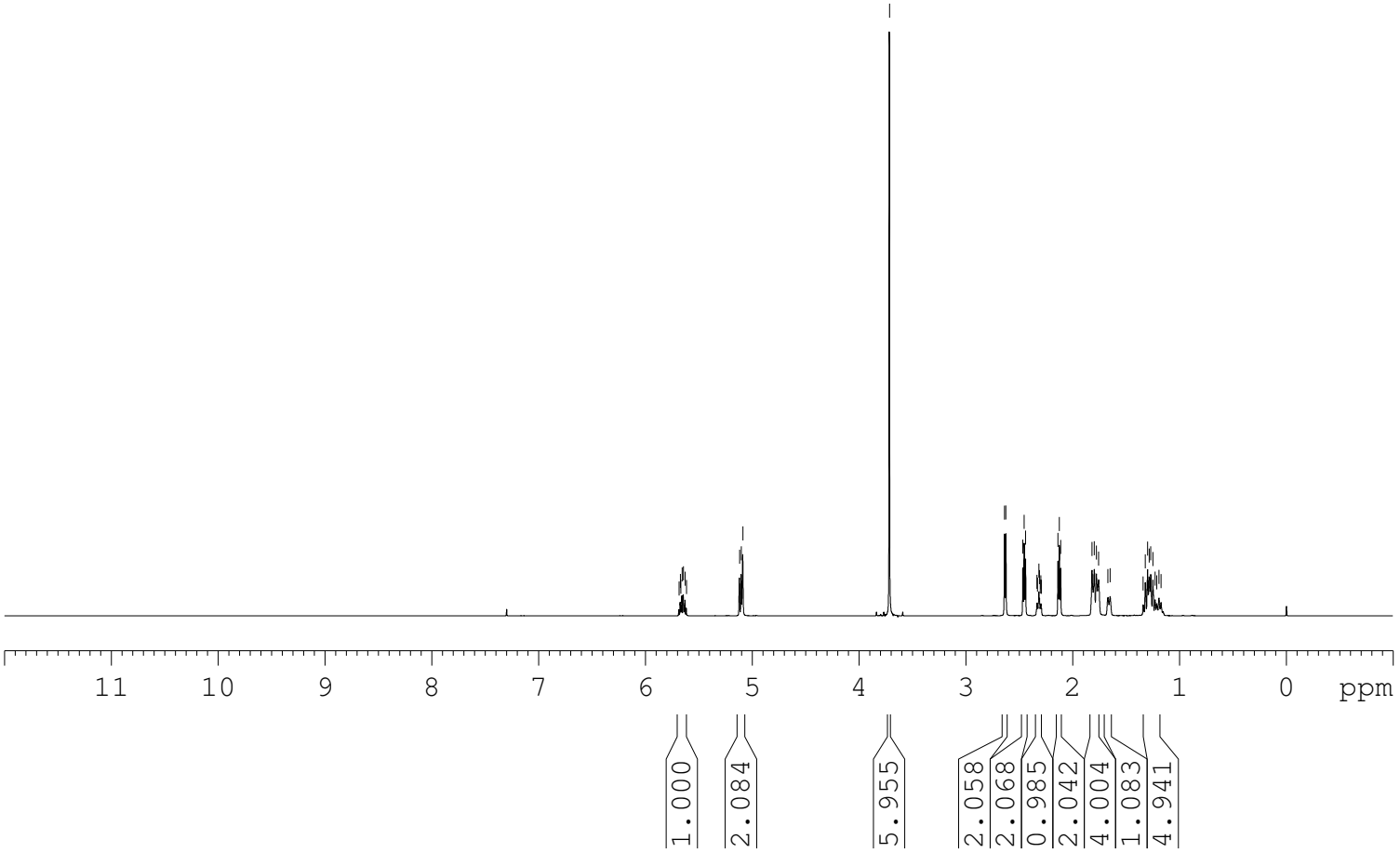
BRUKER

NAME	wll-372p-20201019
EXPNO	2
PROCNO	1
Date_	20201019
Time	19.41
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	48
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	295.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128750 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

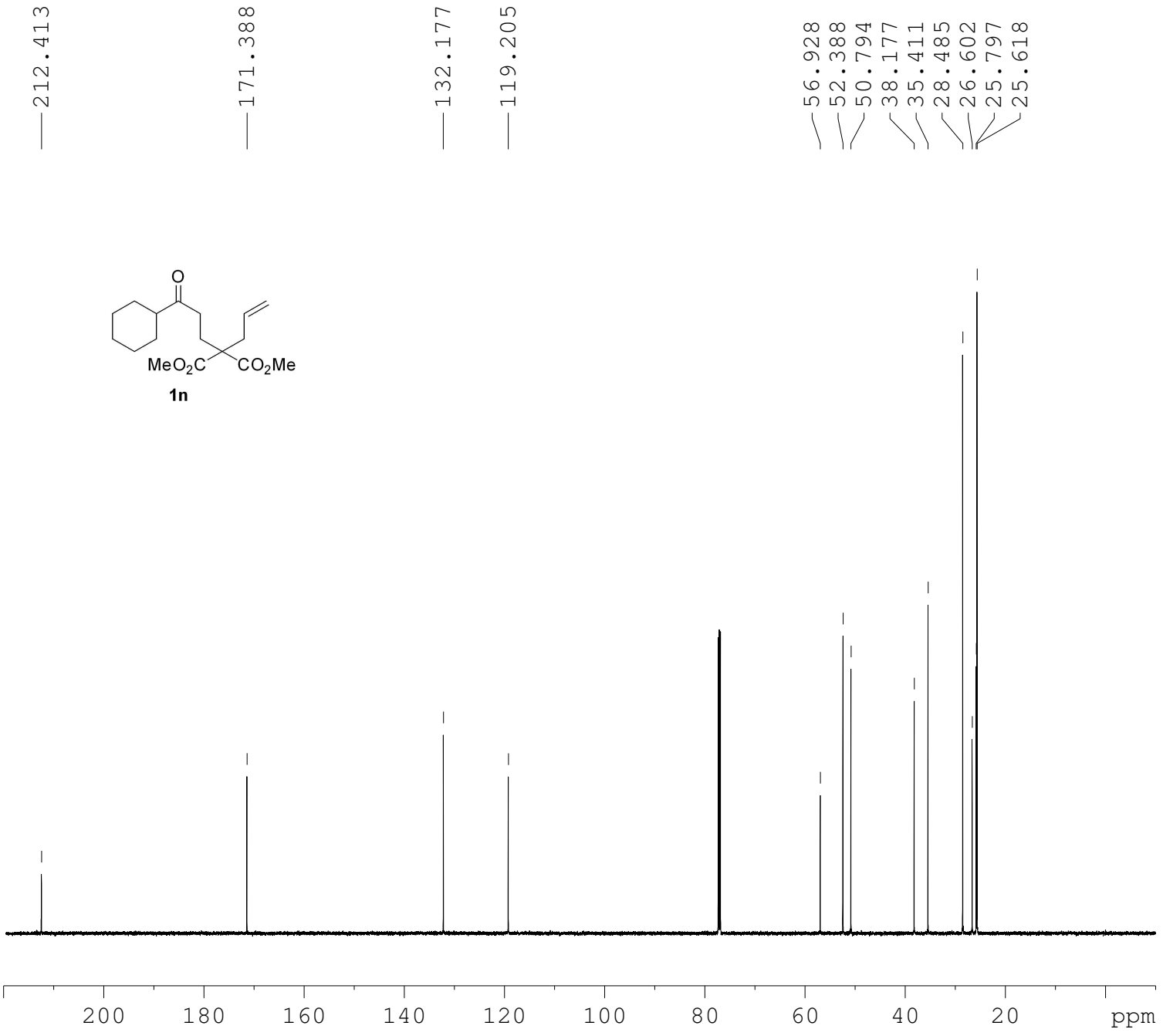




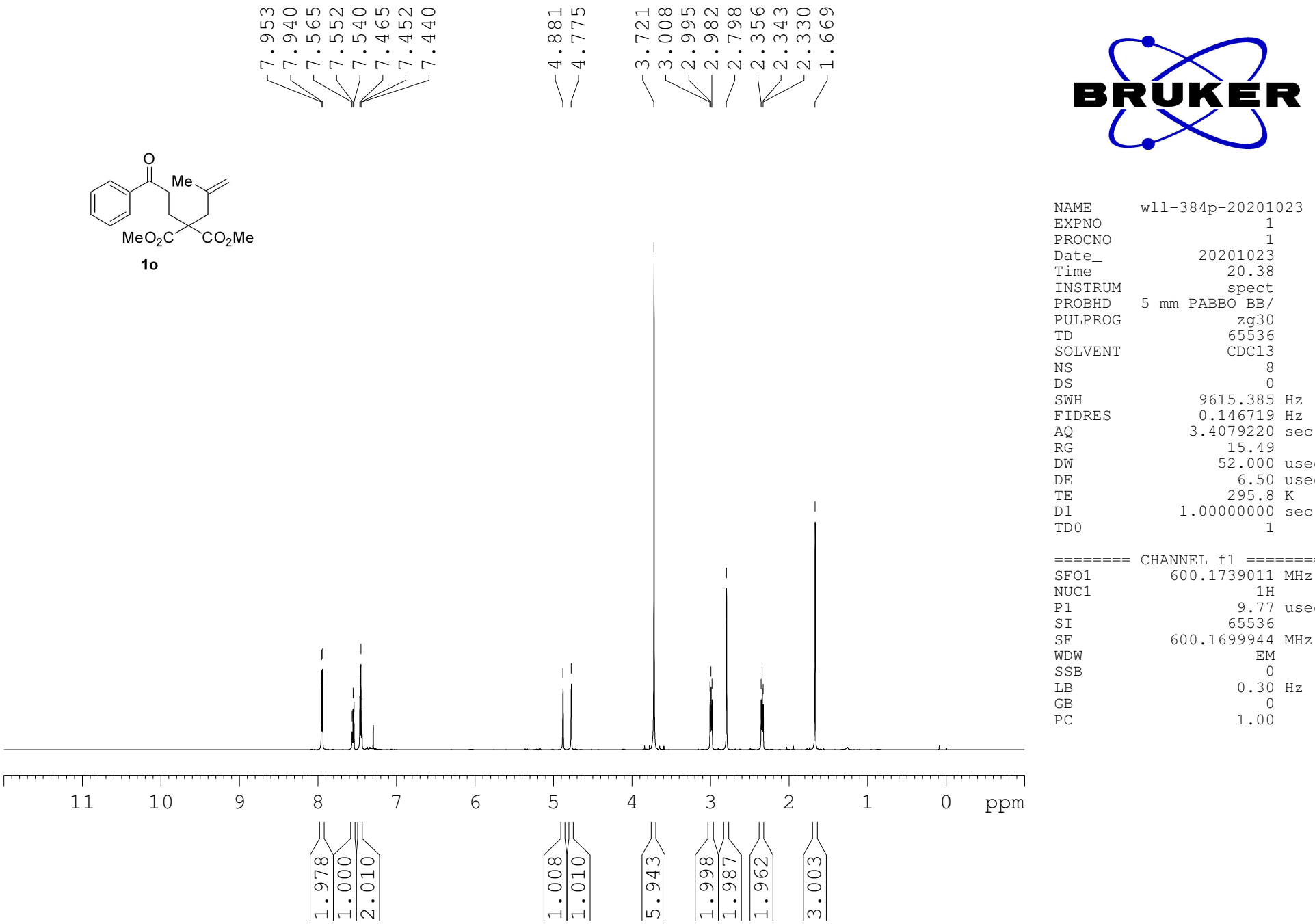
1n



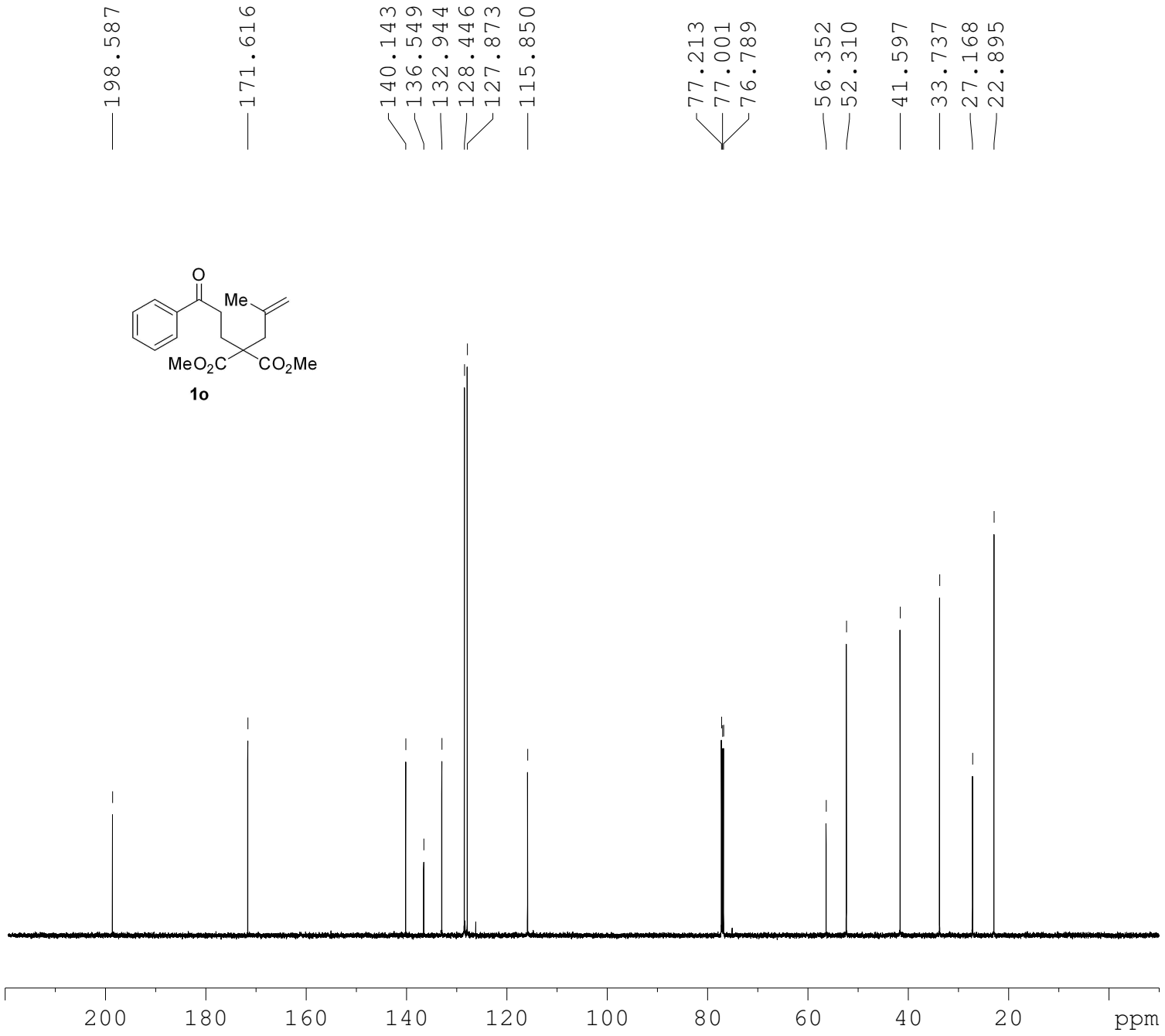
NAME	wll-381p-20201026
EXPNO	1
PROCNO	1
Date_	20201026
Time	15.58
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	9615.385 Hz
FIDRES	0.146719 Hz
AQ	3.4079220 sec
RG	15.49
DW	52.000 usec
DE	6.50 usec
TE	295.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	600.1739011 MHz
NUC1	1H
P1	9.77 usec
SI	65536
SF	600.1699929 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00




```
NAME      wll-381p-20201026
EXPNO                     2
PROCNO                    1
Date_              20201026
Time                  16.12
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      240
DS                        4
SWH              36057.691 Hz
FIDRES            0.550197 Hz
AQ               0.9088159 sec
RG                   190.02
DW                  13.867 usec
DE                    6.50 usec
TE                   296.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          150.9279571 MHz
NUC1                    13C
P1                    11.90 usec
SI                    32768
SF            150.9128665 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

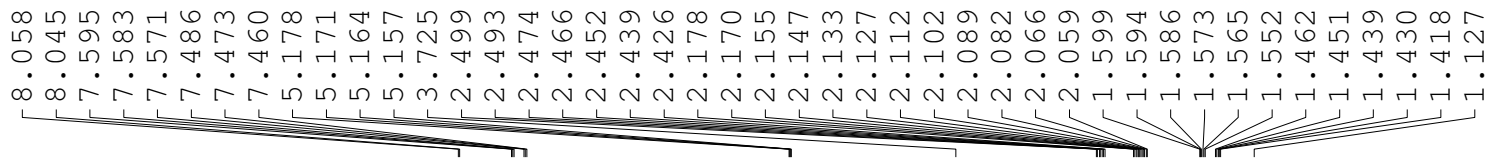


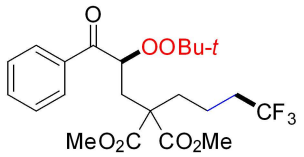

```
NAME      wll-384p-20201023
EXPNO                    1
PROCNO                   1
Date_             20201023
Time                 20.38
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               9615.385 Hz
FIDRES           0.146719 Hz
AQ               3.4079220 sec
RG                   15.49
DW                  52.000 usec
DE                   6.50 usec
TE                  295.8 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.77 usec
SI                   65536
SF            600.1699944 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



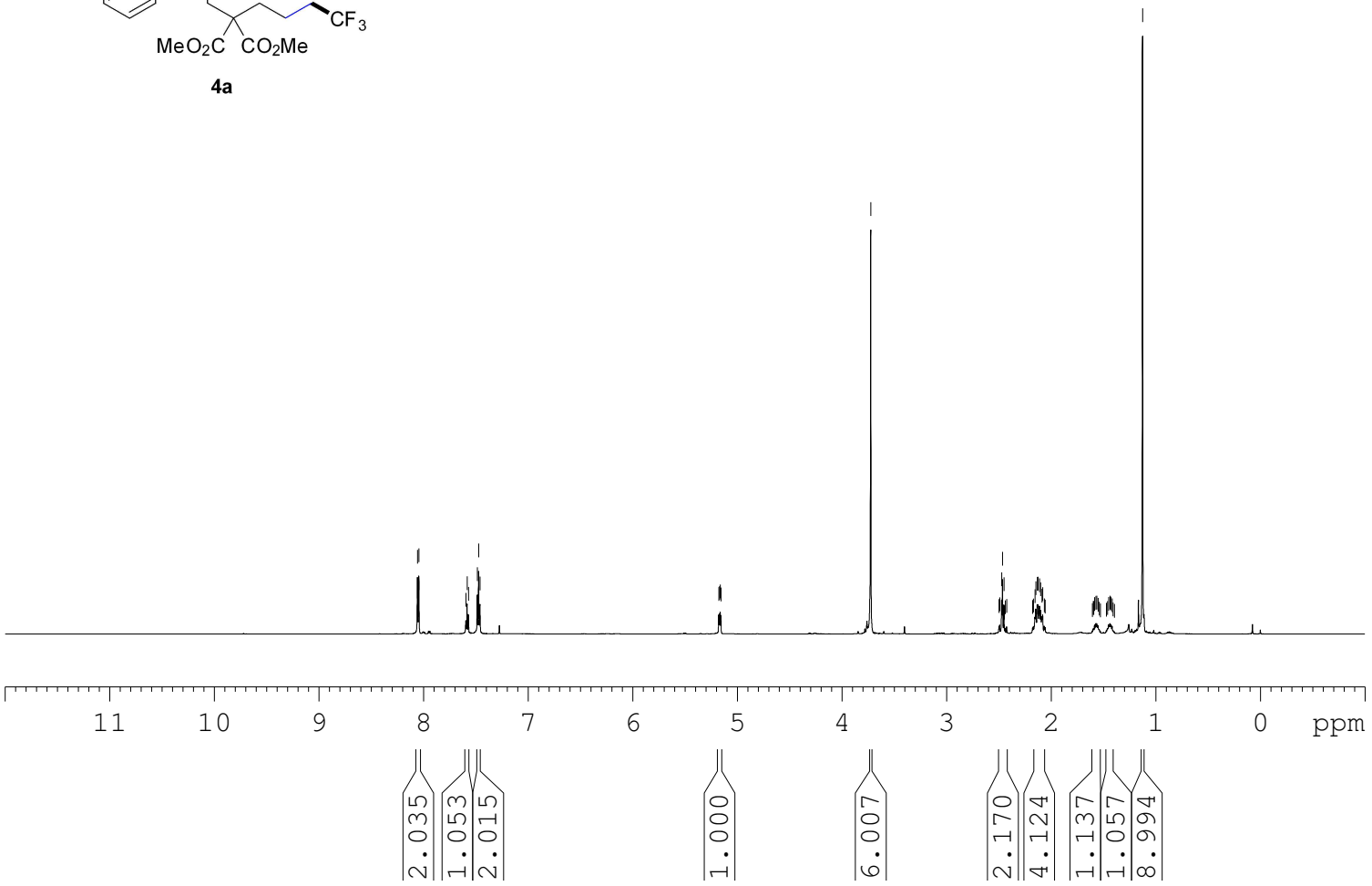
BRUKER

NAME	wll-384p-20201023
EXPNO	2
PROCNO	1
Date_	20201023
Time	20.41
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	48
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	296.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128879 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

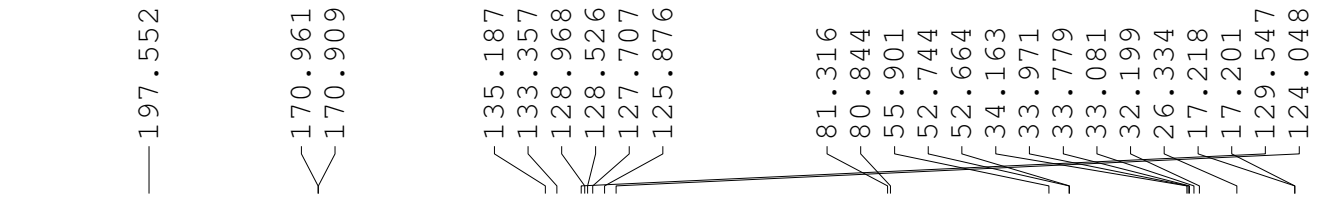


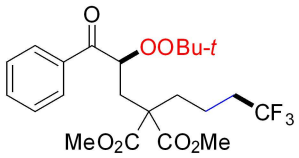


4a

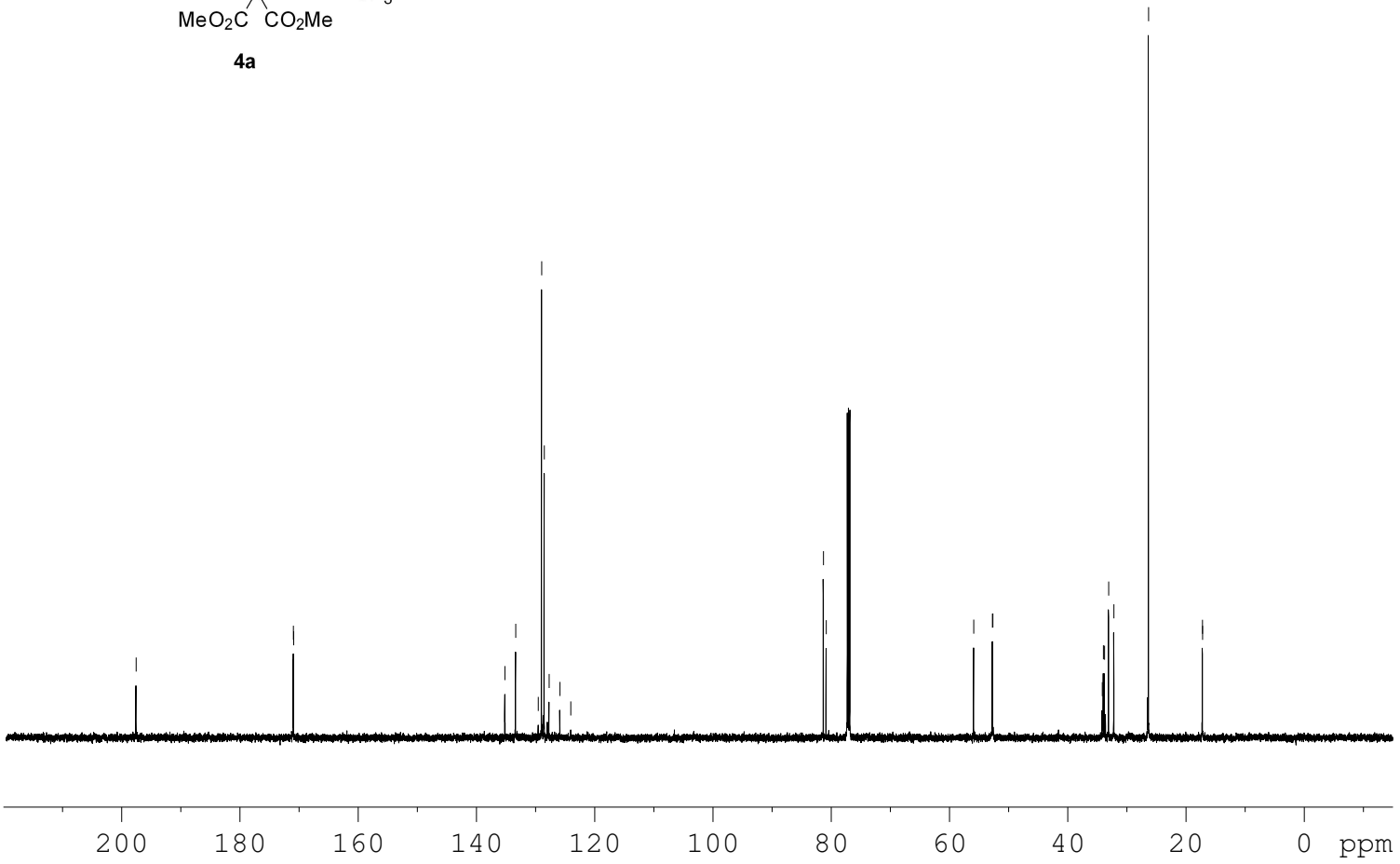



```
NAME      wll-313ap-20200923
EXPNO                      1
PROCNO                     1
Date_               20200923
Time                   14.27
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 9615.385 Hz
FIDRES              0.146719 Hz
AQ                  3.4079220 sec
RG                     36.09
DW                    52.000 usec
DE                     6.50 usec
TE                    296.7 K
D1                  1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             600.1739011 MHz
NUC1                       1H
P1                      9.77 usec
SI                     65536
SF               600.1700076 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

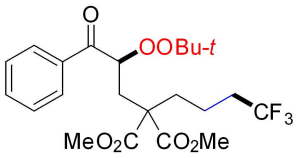




4a

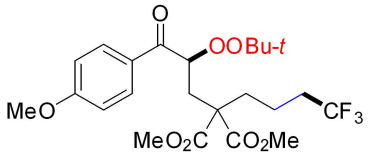


NAME	wll-313ap-20200924
EXPNO	1
PROCNO	1
Date_	20200924
Time	10.39
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	120
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	296.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128665 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



4a


```
NAME      wll-313ap-20200923
EXPNO                      2
PROCNO                     1
Date_               20200923
Time                   14.29
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG          zgfhigqn.2
TD                    131072
SOLVENT               CDCl3
NS                       16
DS                        4
SWH              133928.578 Hz
FIDRES             1.021794 Hz
AQ                 0.4893855 sec
RG                     15.49
DW                     3.733 usec
DE                      6.50 usec
TE                     296.6 K
D1                1.00000000 sec
D11               0.03000000 sec
D12               0.00002000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             564.6675534 MHz
NUC1                    19F
P1                     11.90 usec
SI                     65536
SF               564.7240258 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



4b


```
NAME      wll-326p-20201001
EXPNO                    1
PROCNO                   1
Date_             20201001
Time                 22.03
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               9615.385 Hz
FIDRES            0.146719 Hz
AQ                3.4079220 sec
RG                   30.73
DW                  52.000 usec
DE                   6.50 usec
TE                  297.9 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.77 usec
SI                   65536
SF            600.1700046 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

195.863
170.983
170.946
163.749
131.381
128.107
127.715
125.884
113.729
81.368
80.764
55.913
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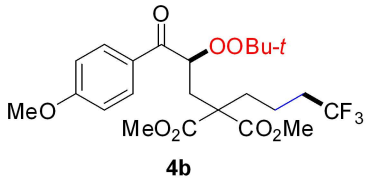
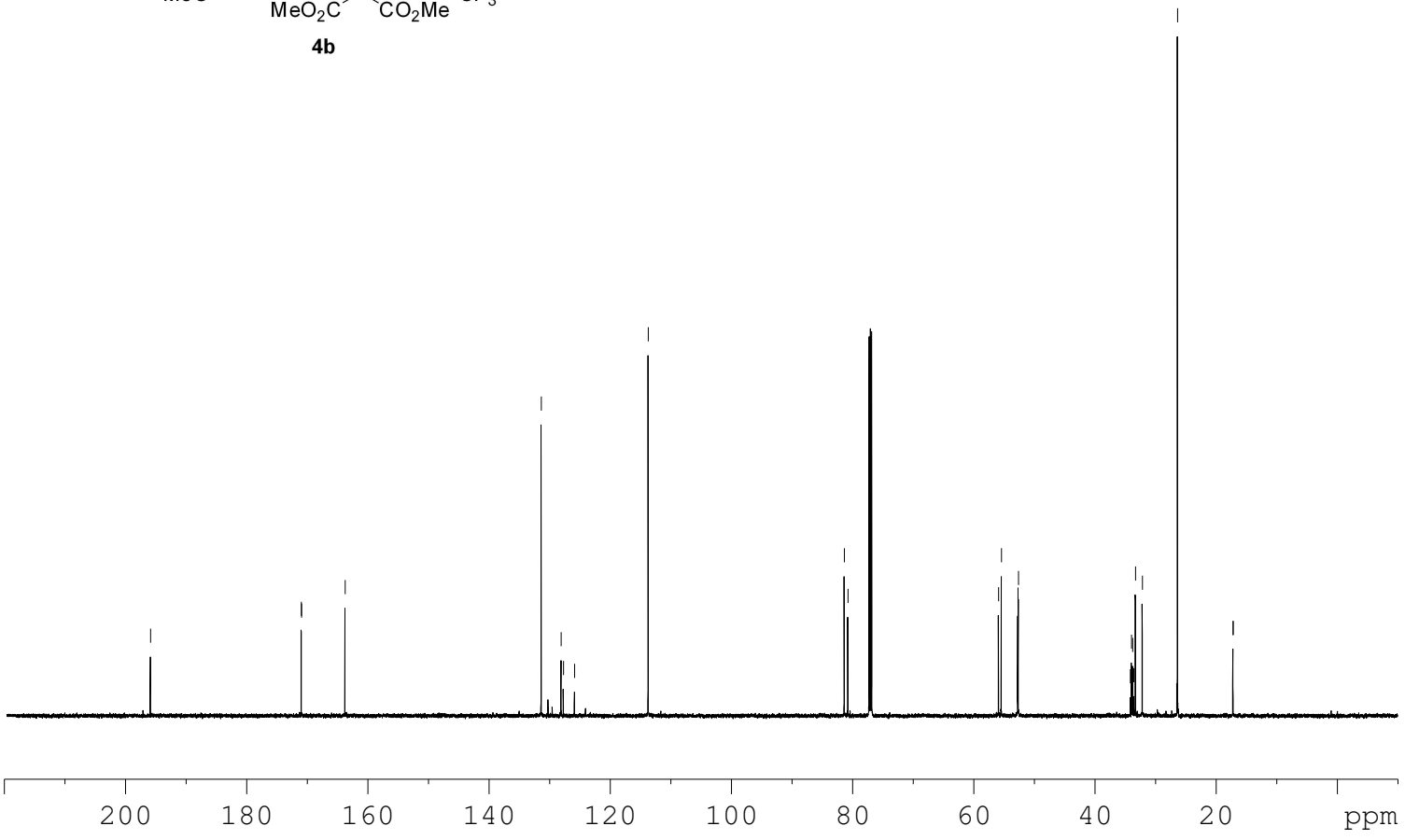
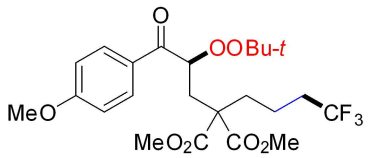
33.780
33.589
33.307
32.172
26.369
17.211
17.195



4b

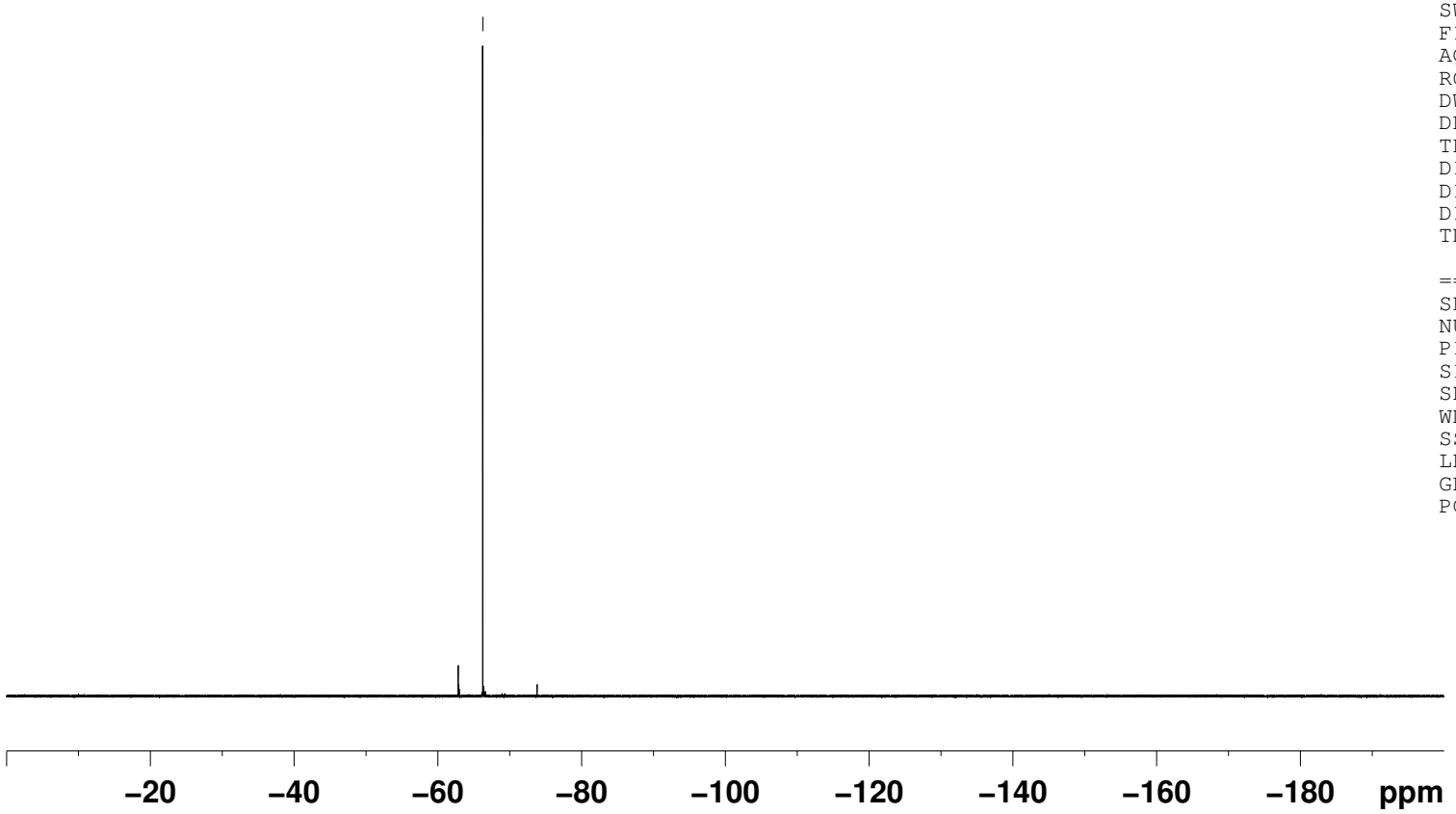


NAME	wll-326p-20201001
EXPNO	3
PROCNO	1
Date_	20201001
Time	22.22
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	300
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	298.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128665 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

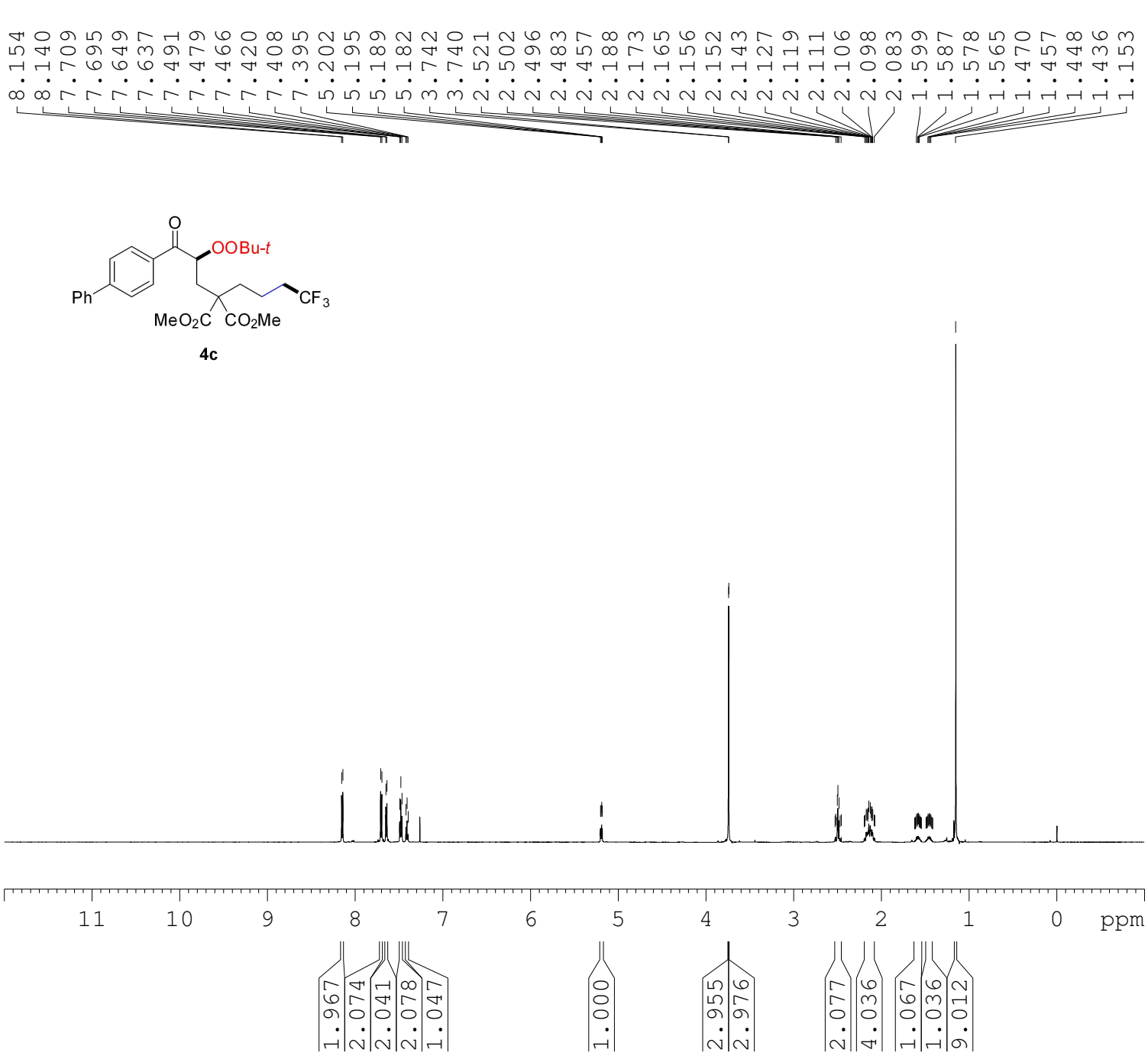


4b

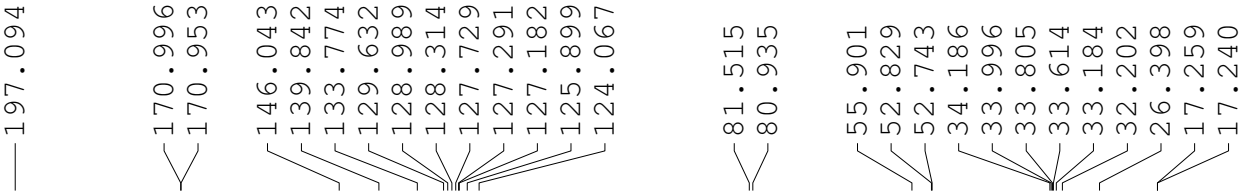
-66.261

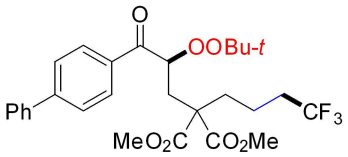



```
NAME      wll-326p-20201001
EXPNO                    2
PROCNO                   1
Date_             20201001
Time                 22.05
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                  131072
SOLVENT              CDCl3
NS                      16
DS                       4
SWH             133928.578 Hz
FIDRES            1.021794 Hz
AQ                0.4893855 sec
RG                    17.32
DW                    3.733 usec
DE                     6.50 usec
TE                    297.8 K
D1              1.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           564.6675534 MHz
NUC1                   19F
P1                   11.90 usec
SI                   65536
SF             564.7240258 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

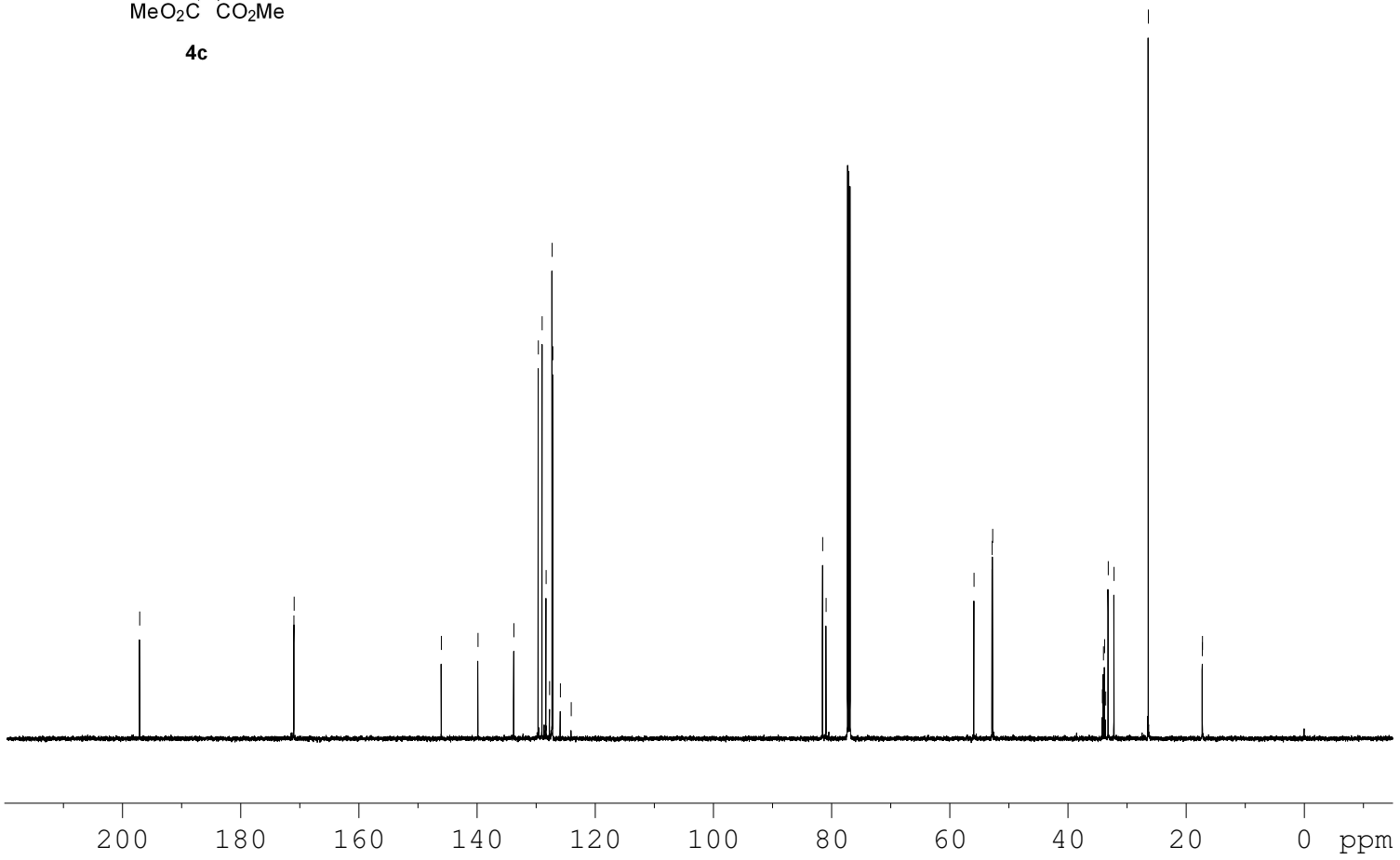



```
NAME      wll-526p-20201230
EXPNO                    1
PROCNO                   1
Date_             20201230
Time                 14.24
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               9615.385 Hz
FIDRES            0.146719 Hz
AQ                3.4079220 sec
RG                    38.1
DW                  52.000 usec
DE                   6.50 usec
TE                  294.5 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.77 usec
SI                   65536
SF            600.1700149 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

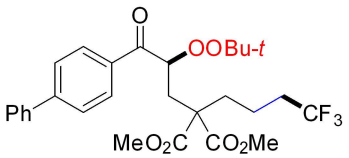




4c

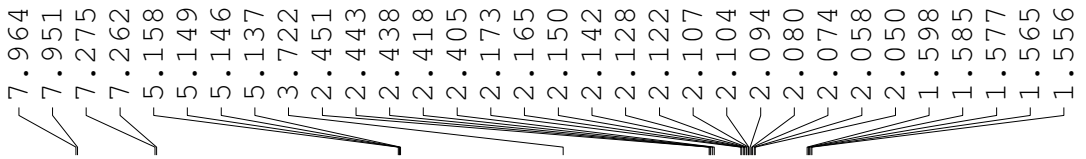


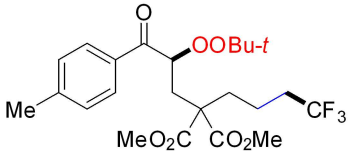
NAME	wll-526p-20201230
EXPNO	3
PROCNO	1
Date_	20201230
Time	14.47
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	400
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	295.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128665 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



4c


```
NAME      wll-526p-20201230
EXPNO                     2
PROCNO                    1
Date_              20201230
Time                  14.26
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                   131072
SOLVENT              CDCl3
NS                      16
DS                       4
SWH             133928.578 Hz
FIDRES            1.021794 Hz
AQ                0.4893855 sec
RG                    15.49
DW                   3.733 usec
DE                    6.50 usec
TE                   294.6 K
D1              1.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           564.6675534 MHz
NUC1                   19F
P1                   11.90 usec
SI                   65536
SF             564.7240258 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```





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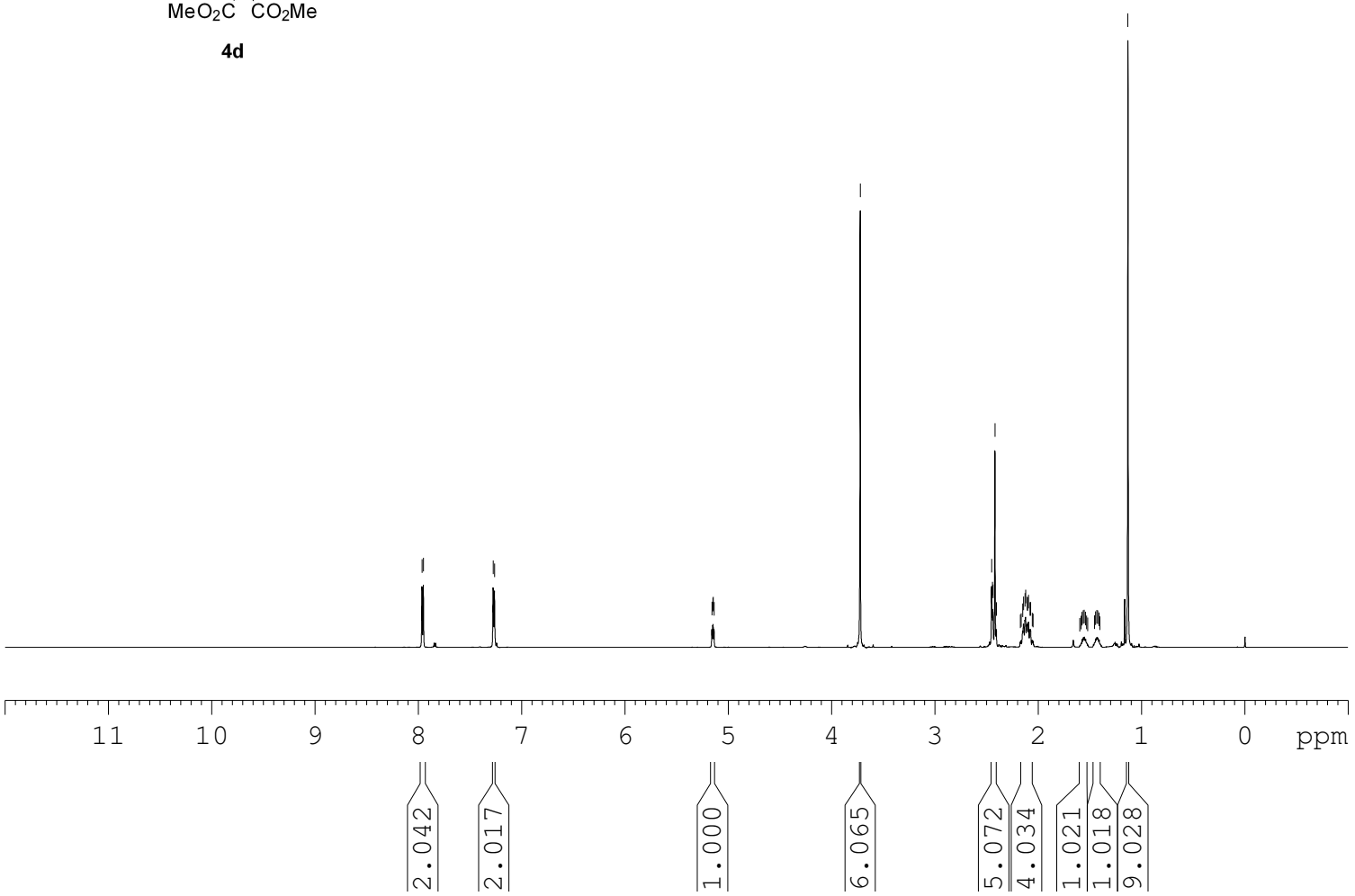
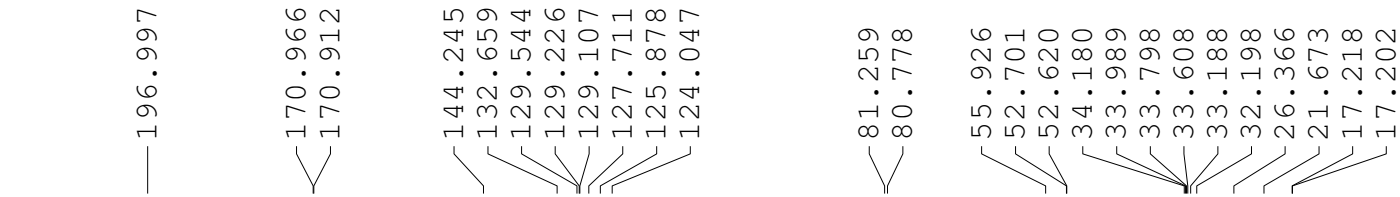
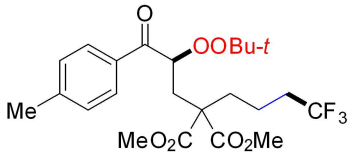
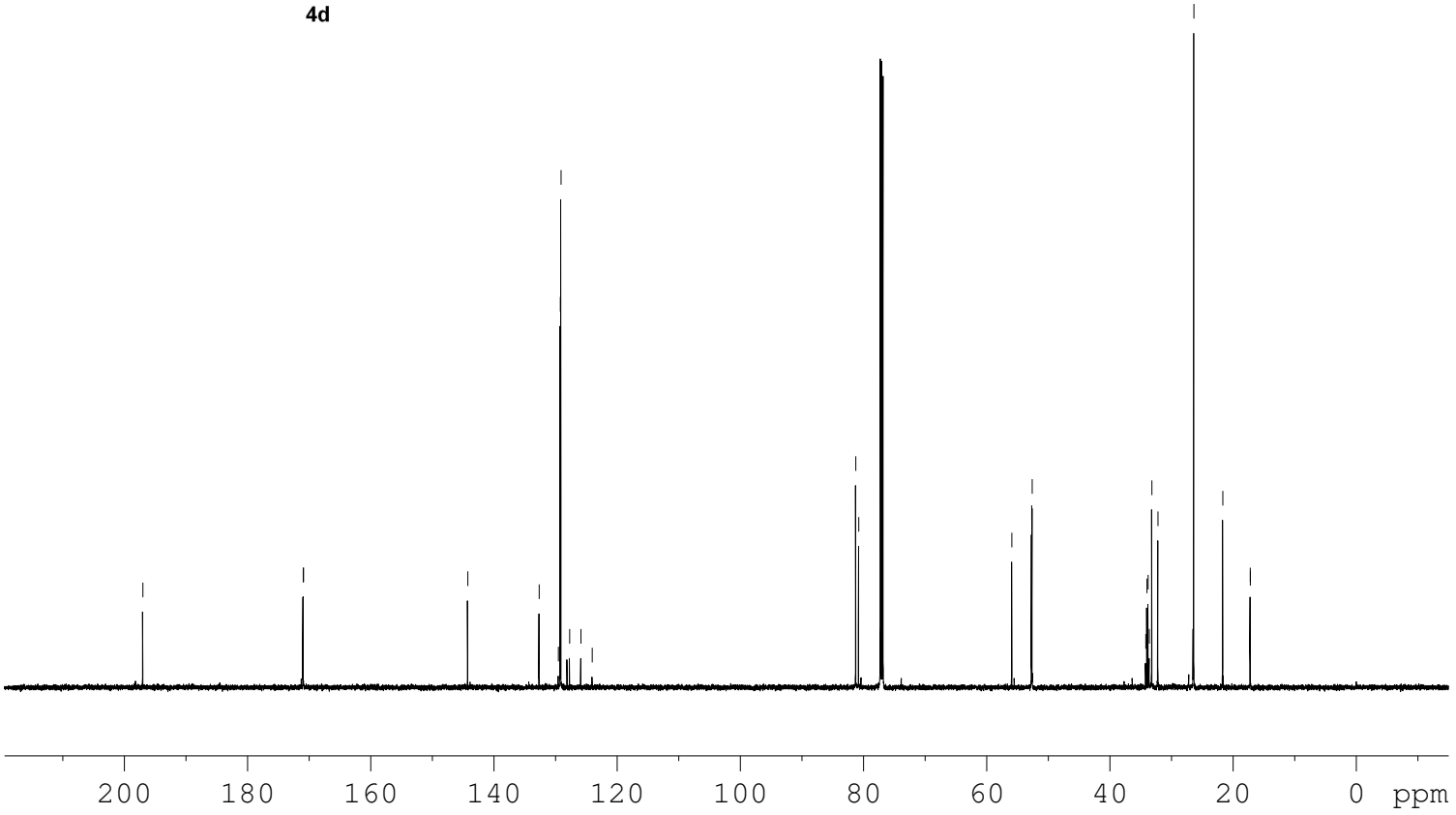


NAME	wll-415p-20201106
EXPNO	1
PROCNO	1
Date_	20201106
Time	15.52
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	9615.385 Hz
FIDRES	0.146719 Hz
AQ	3.4079220 sec
RG	38.1
DW	52.000 usec
DE	6.50 usec
TE	298.1 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
SFO1	600.1739011 MHz
NUC1	1H
P1	9.77 usec
SI	65536
SF	600.1700100 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

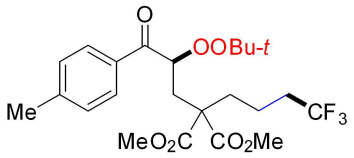




4d

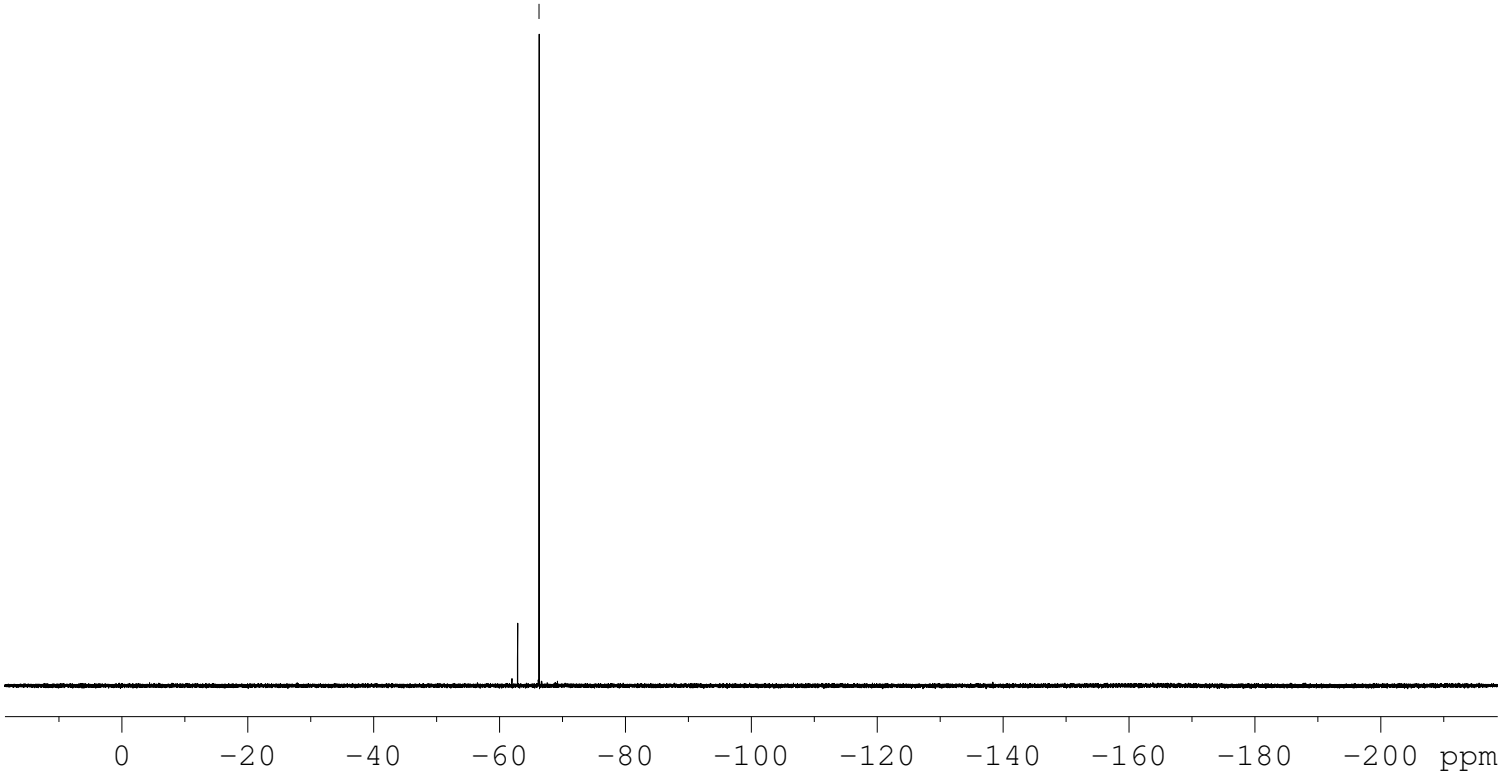



```
NAME      wll-415p-20201106
EXPNO                     3
PROCNO                    1
Date_              20201106
Time                  16.20
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       500
DS                         4
SWH               36057.691 Hz
FIDRES             0.550197 Hz
AQ                0.9088159 sec
RG                   190.02
DW                   13.867 usec
DE                     6.50 usec
TE                    299.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1           150.9279571 MHz
NUC1                    13C
P1                    11.90 usec
SI                    32768
SF             150.9128665 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

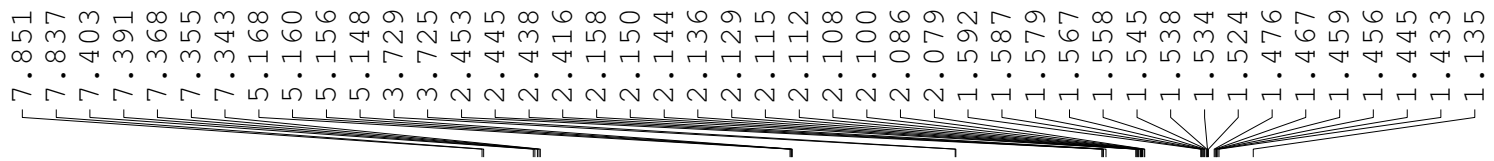


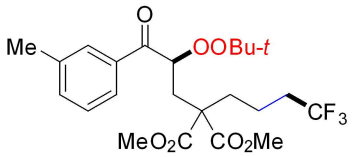
4d

-66.266



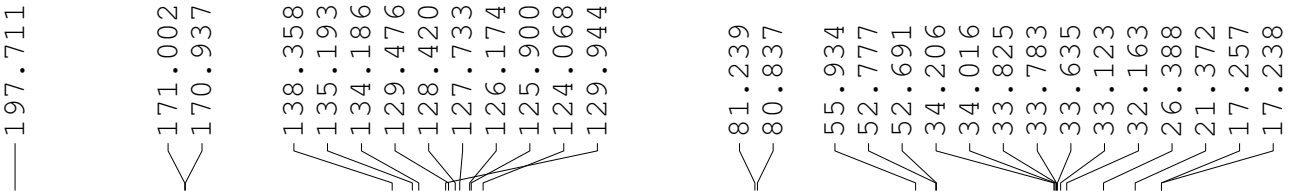

```
NAME      w11-415p-20201106
EXPNO                     2
PROCNO                    1
Date_              20201106
Time                  15.54
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                   131072
SOLVENT              CDCl3
NS                      16
DS                       4
SWH             133928.578 Hz
FIDRES            1.021794 Hz
AQ                0.4893855 sec
RG                    15.49
DW                   3.733 usec
DE                    6.50 usec
TE                   298.1 K
D1              1.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          564.6675534 MHz
NUC1                   19F
P1                   11.90 usec
SI                   65536
SF            564.7240258 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

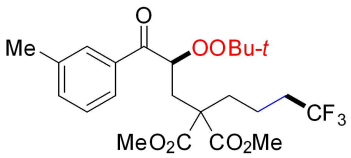




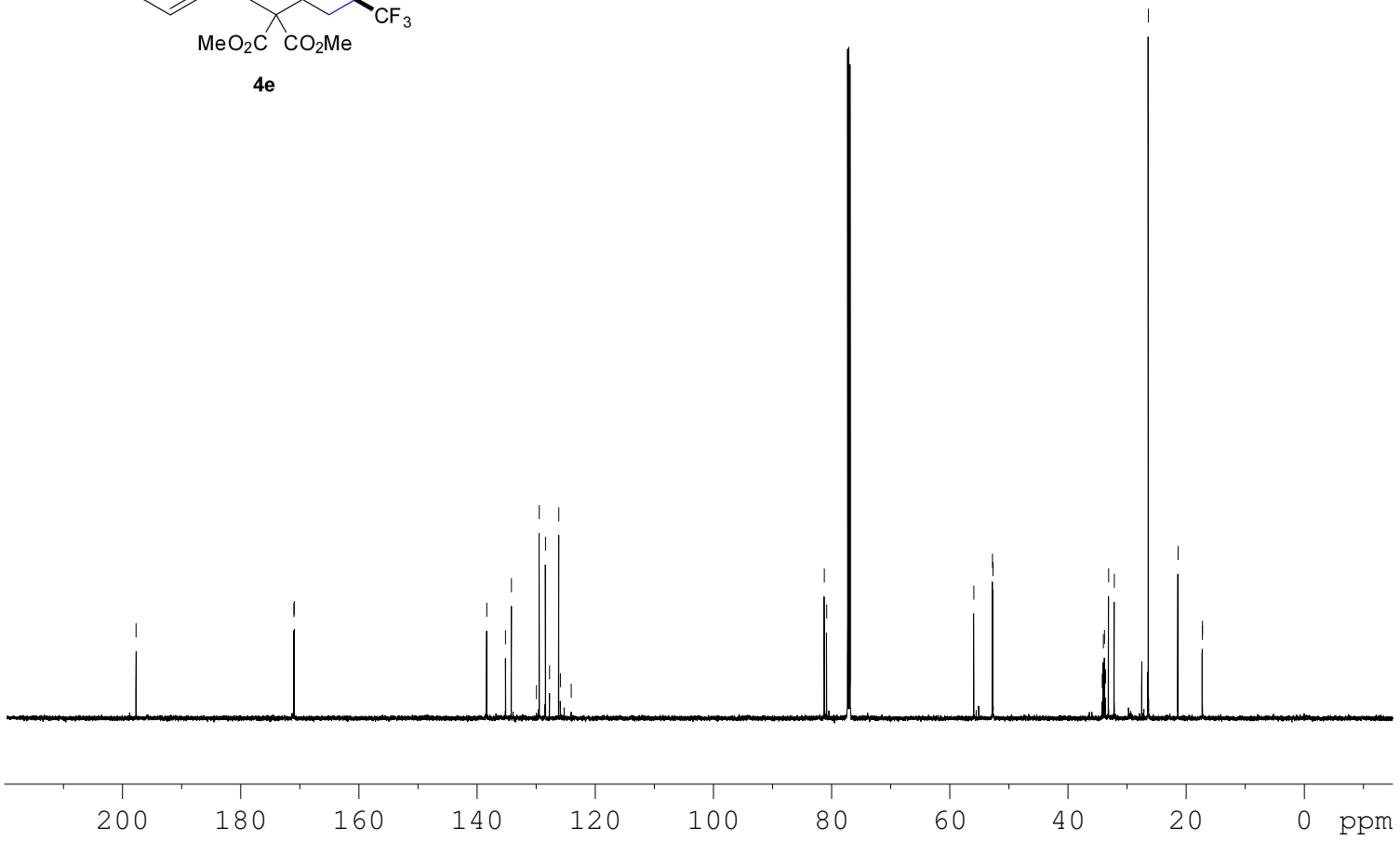
4e


```
NAME      wll-456p-again-20201118
EXPNO                   1
PROCNO                  1
Date_            20201118
Time                13.31
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH              9615.385 Hz
FIDRES           0.146719 Hz
AQ               3.4079220 sec
RG                   44.5
DW                 52.000 usec
DE                  6.50 usec
TE                 295.5 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         600.1739011 MHz
NUC1                   1H
P1                  9.77 usec
SI                 65536
SF           600.1700096 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

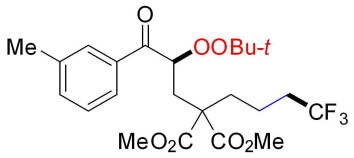




4e

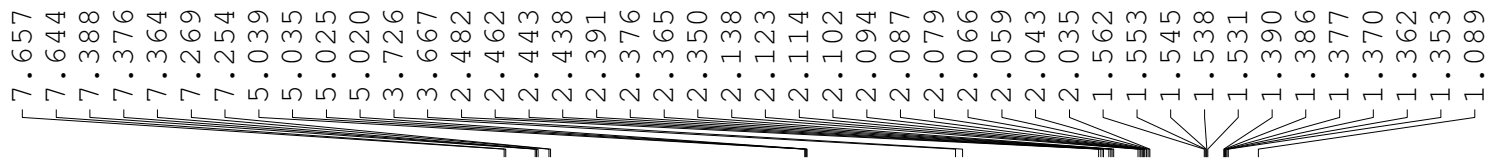


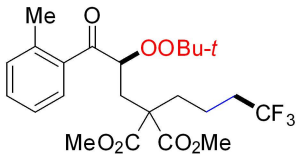

```
NAME      wll-456p-again-20201118
EXPNO                   3
PROCNO                  1
Date_            20201118
Time                14.00
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    500
DS                      4
SWH             36057.691 Hz
FIDRES           0.550197 Hz
AQ               0.9088159 sec
RG                 190.02
DW                13.867 usec
DE                 6.50 usec
TE                296.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          150.9279571 MHz
NUC1                  13C
P1                 11.90 usec
SI                 32768
SF            150.9128625 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



4e


```
NAME      wll-456p-again-20201118
EXPNO                           2
PROCNO                          1
Date_                    20201118
Time                        13.33
INSTRUM                      spect
PROBHD    5 mm PABBO BB/
PULPROG              zgfhigqn.2
TD                          131072
SOLVENT                     CDCl3
NS                             16
DS                              4
SWH                    133928.578 Hz
FIDRES                   1.021794 Hz
AQ                       0.4893855 sec
RG                          15.49
DW                          3.733 usec
DE                           6.50 usec
TE                          295.4 K
D1                     1.00000000 sec
D11                    0.03000000 sec
D12                    0.00002000 sec
TD0                             1

======== CHANNEL f1 ========
SFO1                 564.6675534 MHz
NUC1                        19F
P1                         11.90 usec
SI                          65536
SF                   564.7240258 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```

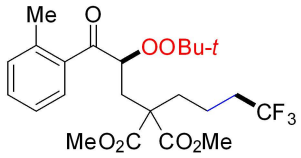




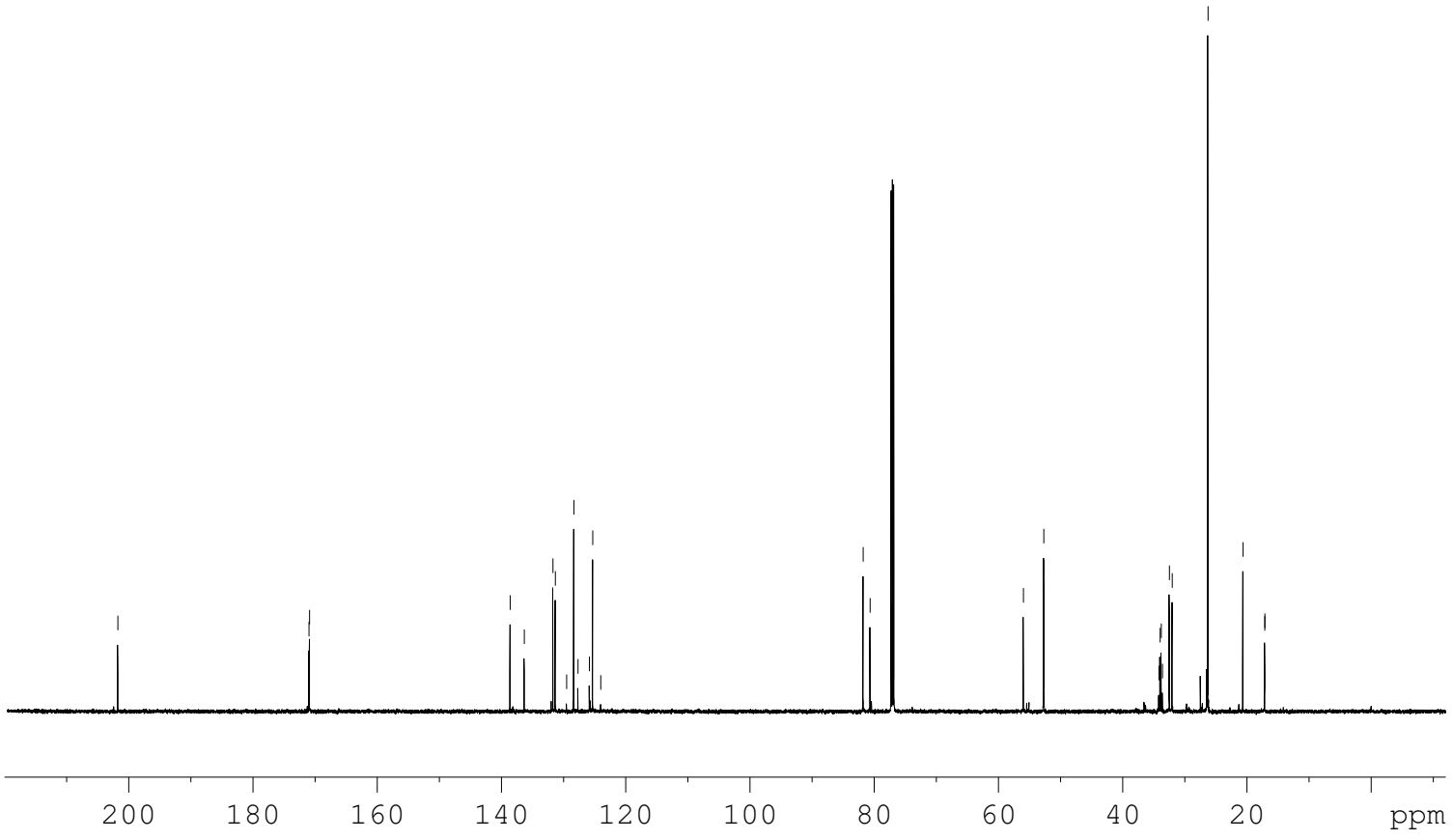
4f

```
NAME      wll-455p-again-20201118
EXPNO                  1
PROCNO                 1
Date_           20201118
Time               12.59
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             9615.385 Hz
FIDRES          0.146719 Hz
AQ              3.4079220 sec
RG                 36.09
DW                 52.000 usec
DE                  6.50 usec
TE                 295.1 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1739011 MHz
NUC1                   1H
P1                  9.77 usec
SI                 65536
SF           600.1700083 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

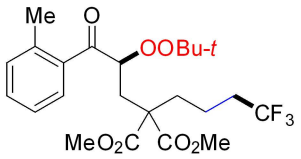
201.752
170.986
170.897
138.601
136.324
131.715
131.327
129.512
128.341
127.679
125.848
125.308
124.017
81.776
80.659
55.977
52.699
52.672
34.166
33.974
33.784
33.594
32.477
32.024
26.259
20.623
17.128
17.109



4f

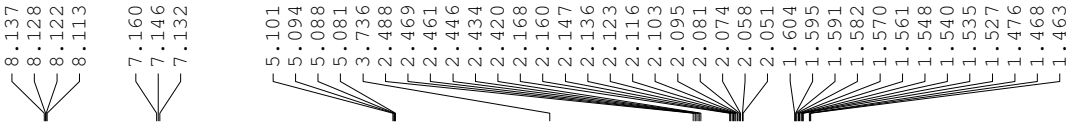


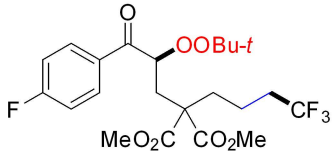

```
NAME      wll-455p-again-20201118
EXPNO                  4
PROCNO                 1
Date_           20201118
Time              13.27
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    4
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG               190.02
DW               13.867 usec
DE                 6.50 usec
TE               296.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        150.9279571 MHz
NUC1               13C
P1               11.90 usec
SI               32768
SF          150.9128665 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```



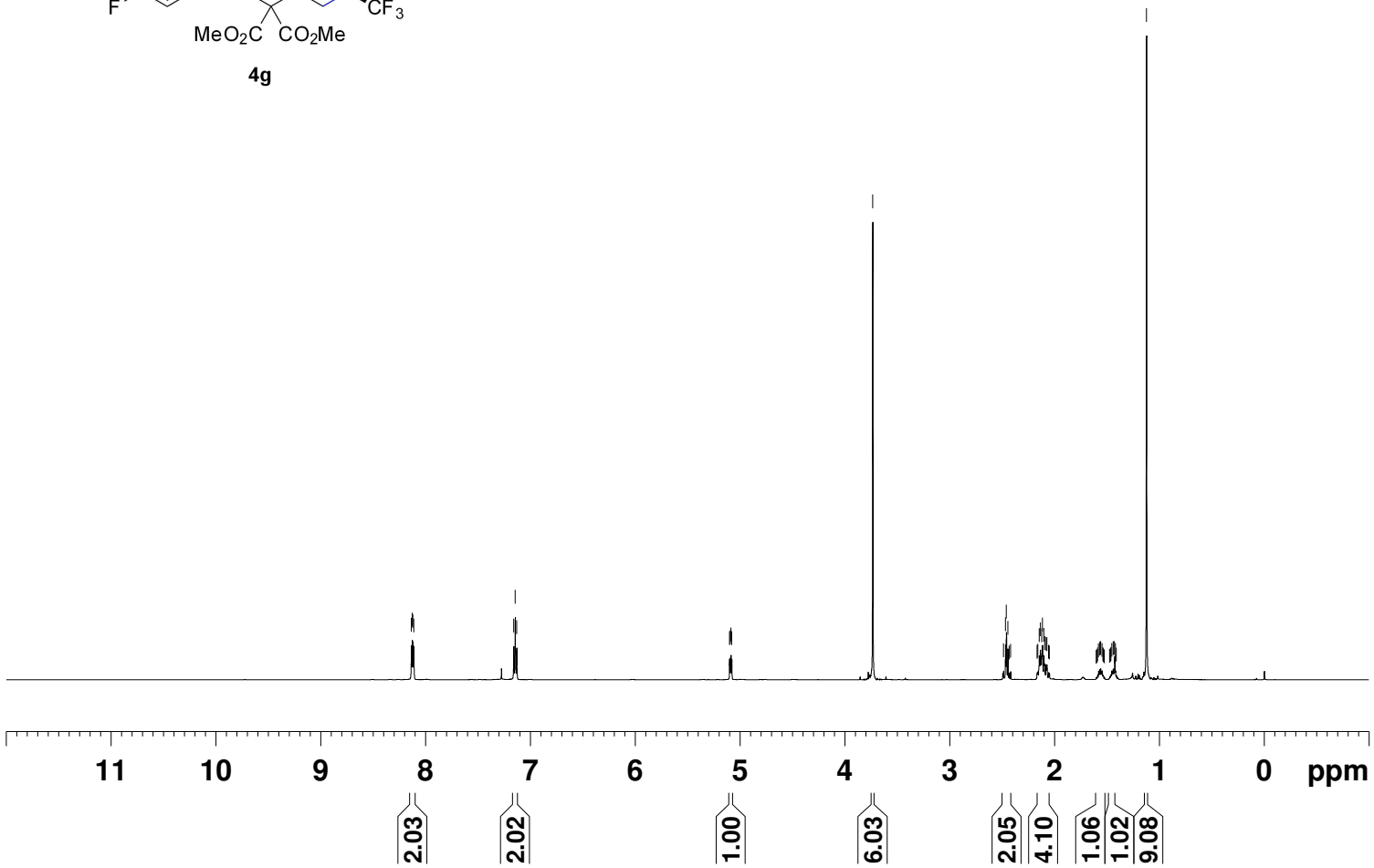
4f


```
NAME      wll-455p-again-20201118
EXPNO                  3
PROCNO                 1
Date_           20201118
Time               13.01
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                131072
SOLVENT            CDCl3
NS                    16
DS                     4
SWH           133928.578 Hz
FIDRES          1.021794 Hz
AQ             0.4893855 sec
RG                15.49
DW                3.733 usec
DE                 6.50 usec
TE                295.1 K
D1            1.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         564.6675534 MHz
NUC1                19F
P1                11.90 usec
SI                65536
SF           564.7240258 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

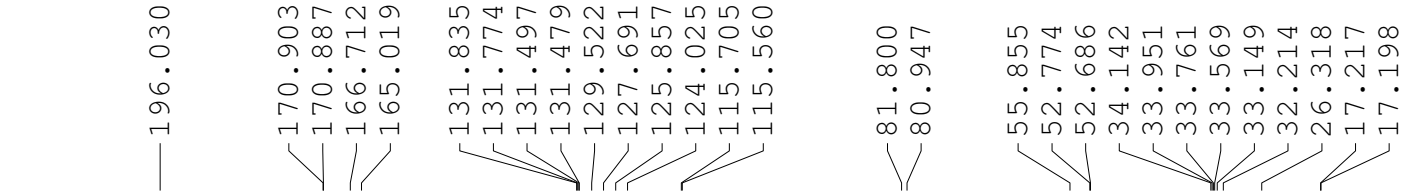


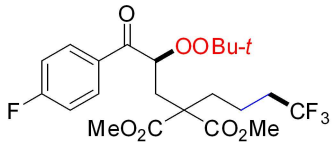


4g

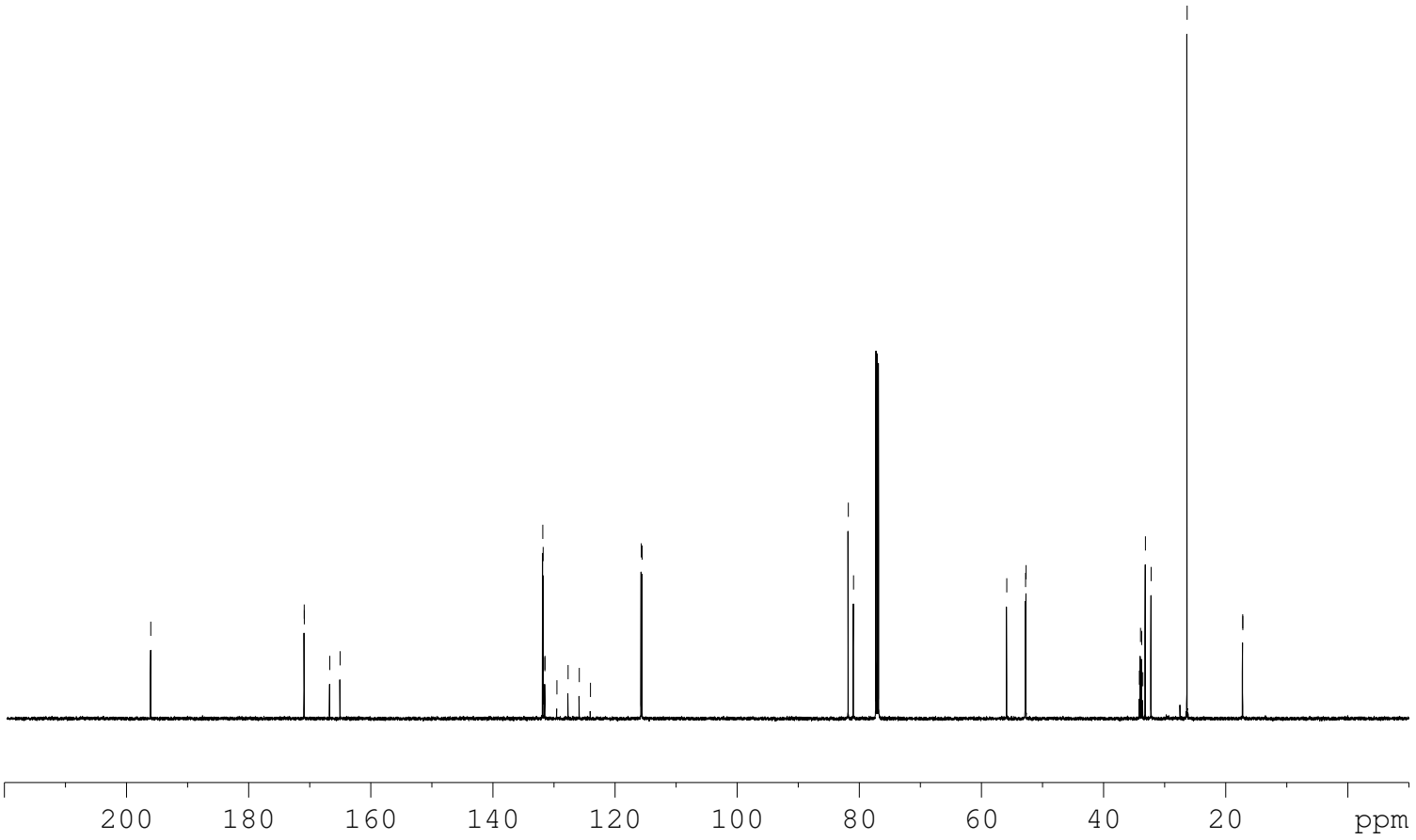



```
NAME      wll-459p-20201120
EXPNO                    1
PROCNO                   1
Date_             20201120
Time                 23.08
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               9615.385 Hz
FIDRES           0.146719 Hz
AQ               3.4079220 sec
RG                   36.09
DW                  52.000 usec
DE                    6.50 usec
TE                   294.7 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                    9.77 usec
SI                   65536
SF            600.1700050 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

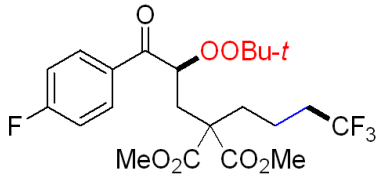




4g

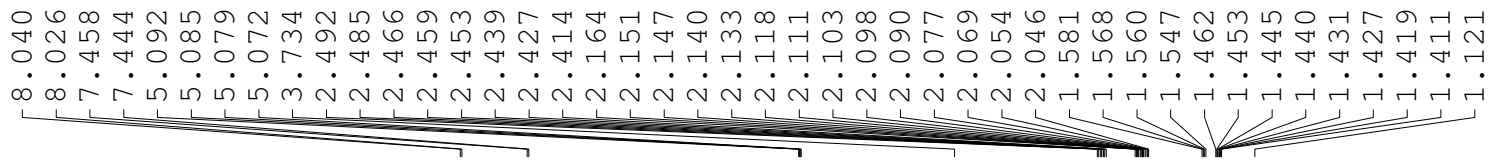


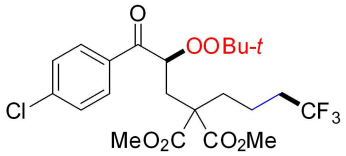
NAME	wll-459p-20201121
EXPNO	1
PROCNO	1
Date_	20201121
Time	11.51
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	400
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	296.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128665 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40


O
OOBu-t
F
CF3
MeO2C
CO2Me

4g

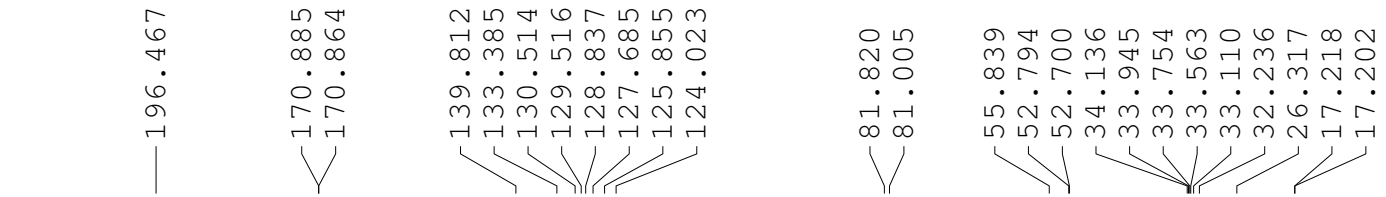
-66.2557
-104.4634
3.06
1.00
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

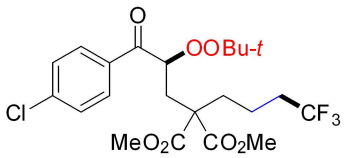




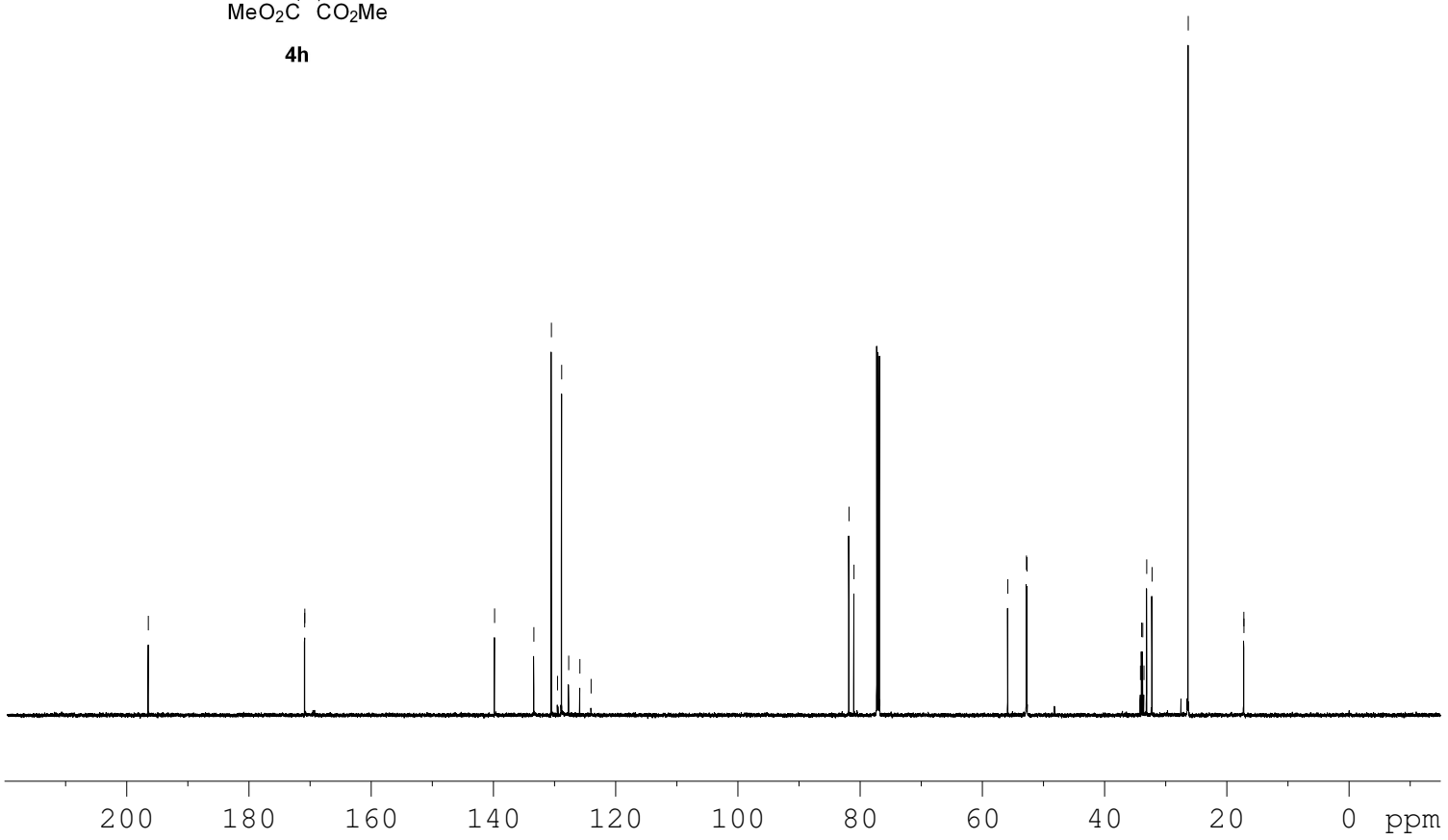
4h


```
NAME      wll-454p-20201118
EXPNO                     1
PROCNO                    1
Date_              20201118
Time                   9.36
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDC13
NS                        8
DS                        0
SWH               9615.385 Hz
FIDRES           0.146719 Hz
AQ               3.4079220 sec
RG                    30.73
DW                 52.000 usec
DE                  6.50 usec
TE                  294.5 K
D1             1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.77 usec
SI                    65536
SF            600.1700046 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    1.00
```

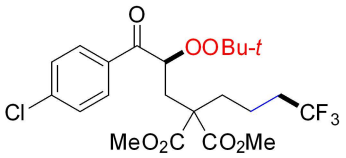




4h

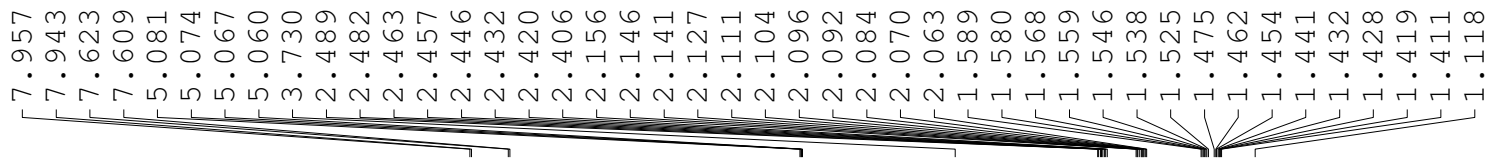


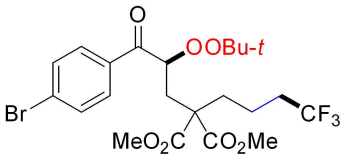
NAME	wll-454p-20201118
EXPNO	2
PROCNO	1
Date_	20201118
Time	14.18
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	300
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	296.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128665 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



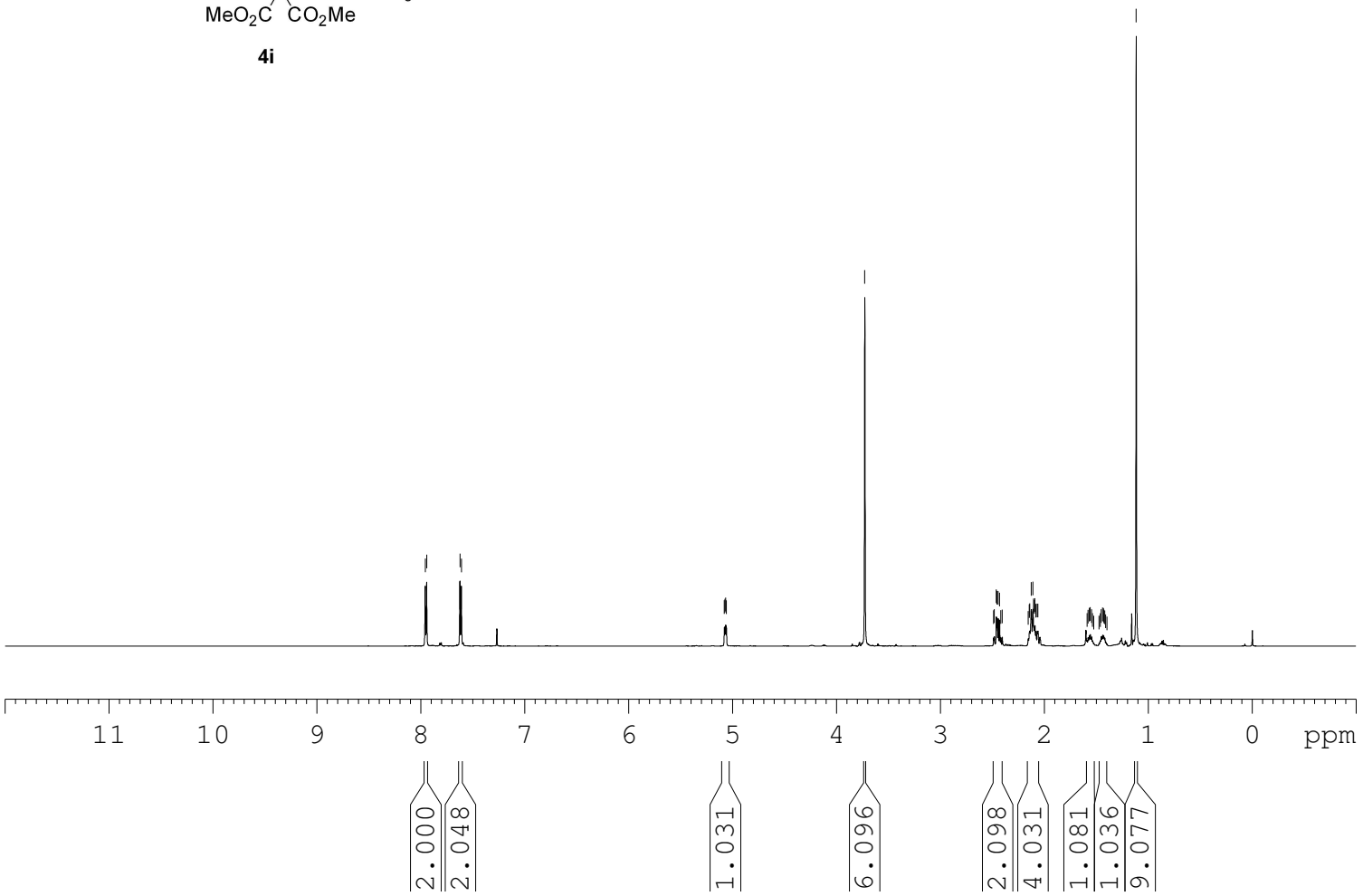
4h


```
NAME      wll-454p-20201118
EXPNO                     3
PROCNO                    1
Date_              20201118
Time                  14.25
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                   131072
SOLVENT              CDC13
NS                      16
DS                       4
SWH             133928.578 Hz
FIDRES            1.021794 Hz
AQ                0.4893855 sec
RG                    15.49
DW                   3.733 usec
DE                    6.50 usec
TE                   295.4 K
D1              1.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           564.6675534 MHz
NUC1                   19F
P1                   11.90 usec
SI                   65536
SF             564.7240258 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

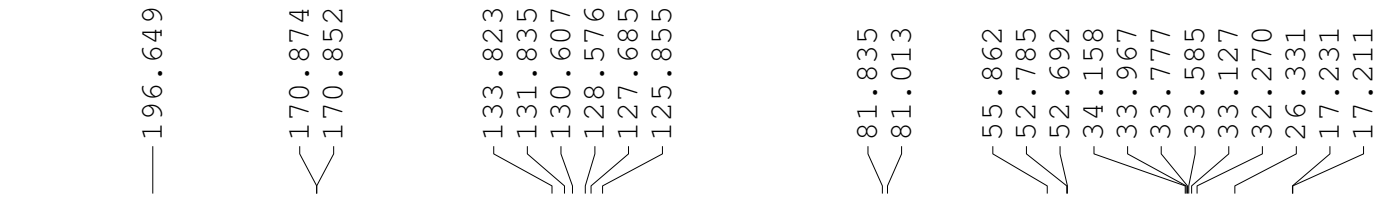


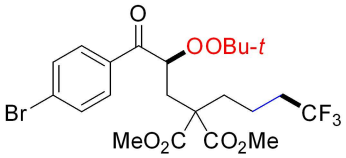


4i

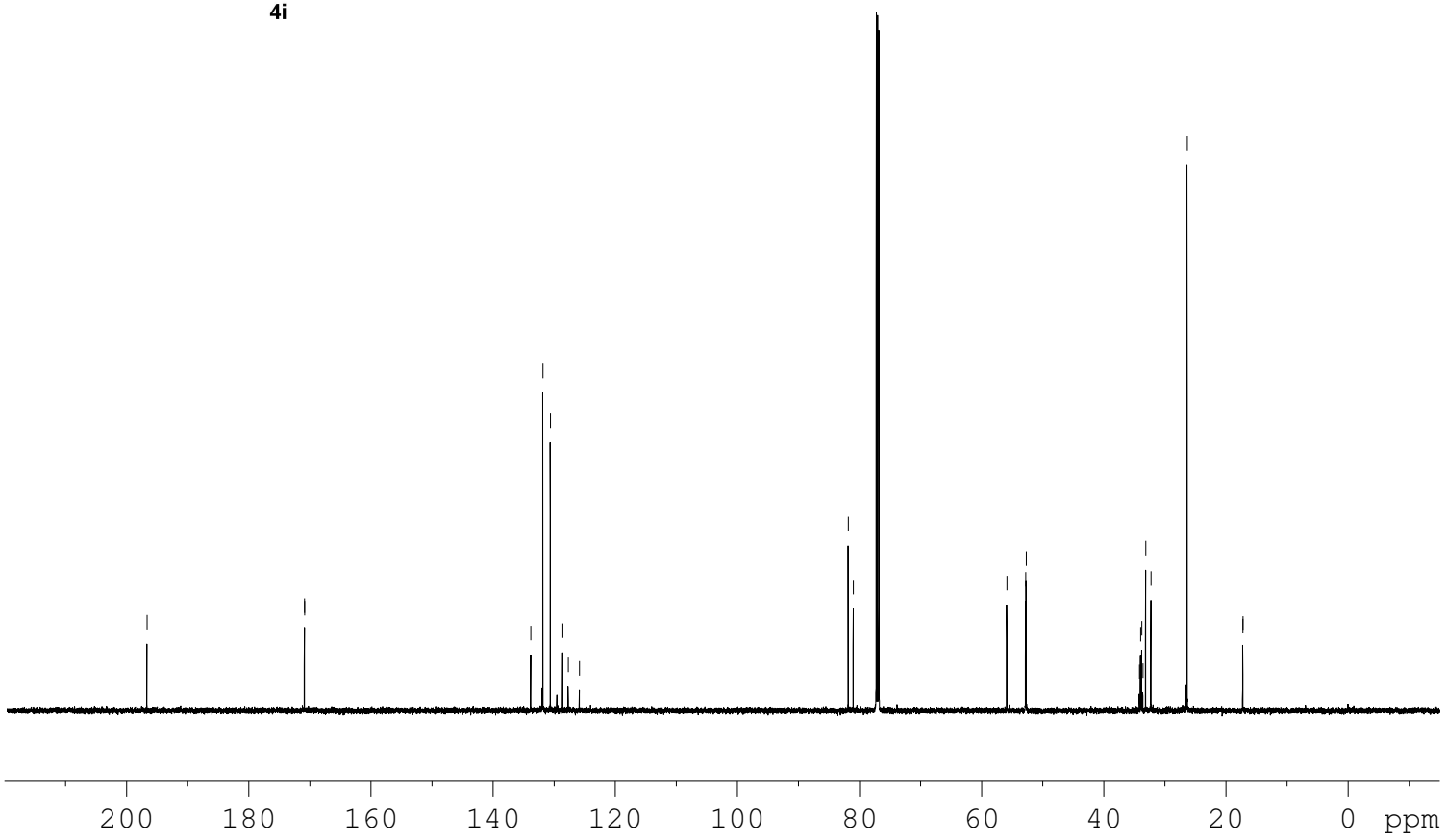



```
NAME      wll-416p-20201106
EXPNO                   1
PROCNO                  1
Date_            20201106
Time                16.24
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH              9615.385 Hz
FIDRES           0.146719 Hz
AQ               3.4079220 sec
RG                  62.22
DW                 52.000 usec
DE                  6.50 usec
TE                 298.6 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                   1H
P1                   9.77 usec
SI                  65536
SF            600.1700113 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

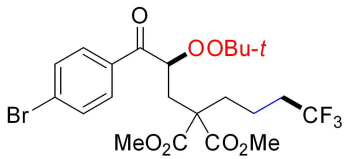




4i

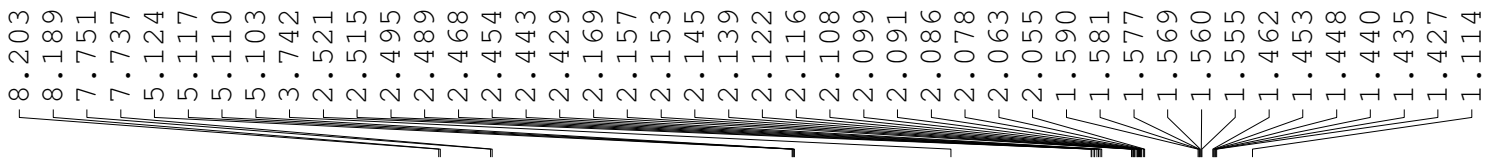


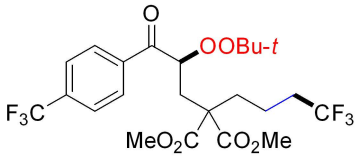
NAME	w11-416p-20201106
EXPNO	3
PROCNO	1
Date_	20201106
Time	16.52
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	500
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	299.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128665 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



4i

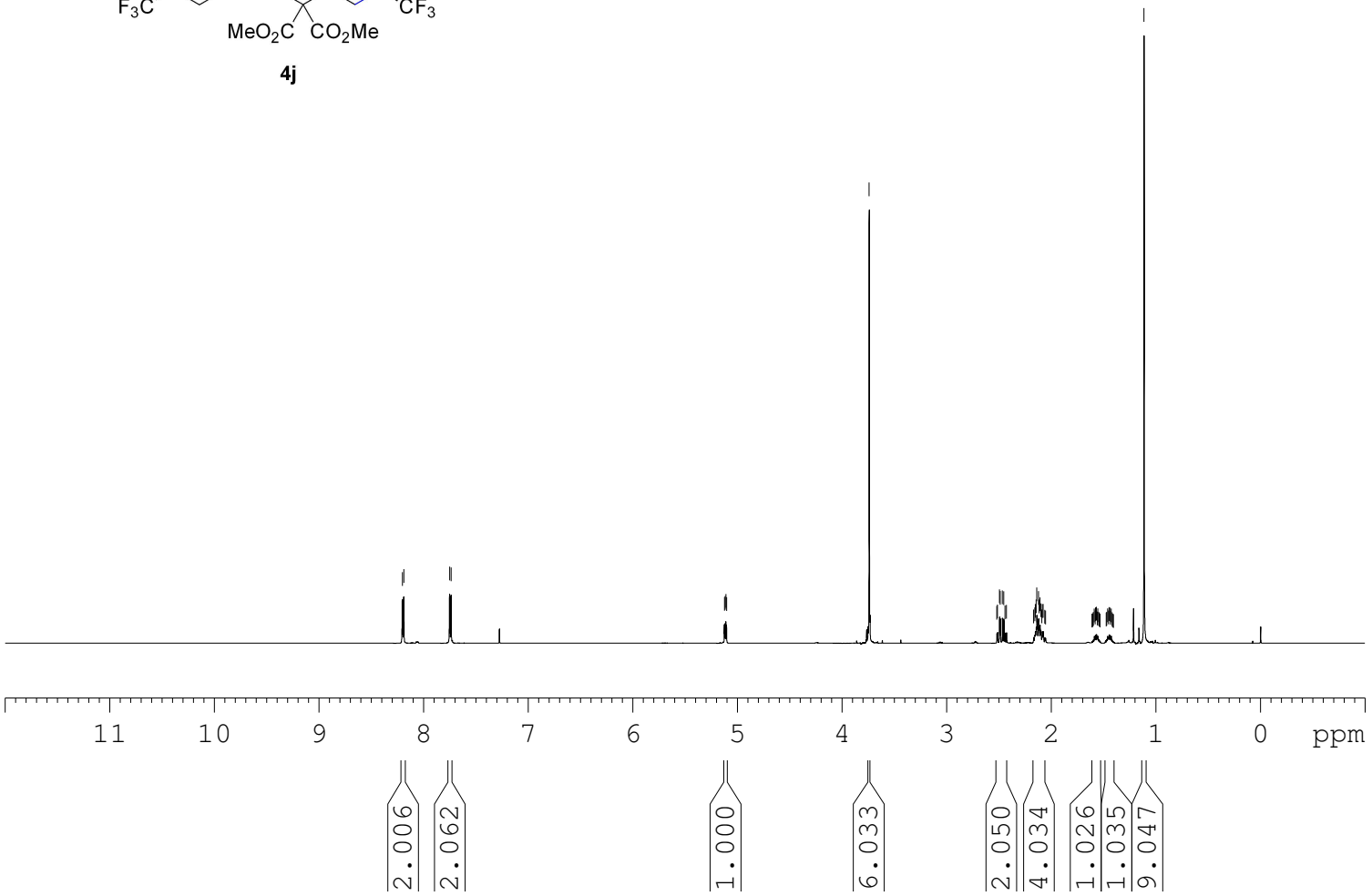

```
NAME      wll-416p-20201106
EXPNO                     2
PROCNO                    1
Date_              20201106
Time                  16.26
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                   131072
SOLVENT               CDCl3
NS                       16
DS                        4
SWH              133928.578 Hz
FIDRES             1.021794 Hz
AQ                 0.4893855 sec
RG                    15.49
DW                    3.733 usec
DE                     6.50 usec
TE                    298.5 K
D1              1.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            564.6675534 MHz
NUC1                    19F
P1                    11.90 usec
SI                    65536
SF              564.7240258 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

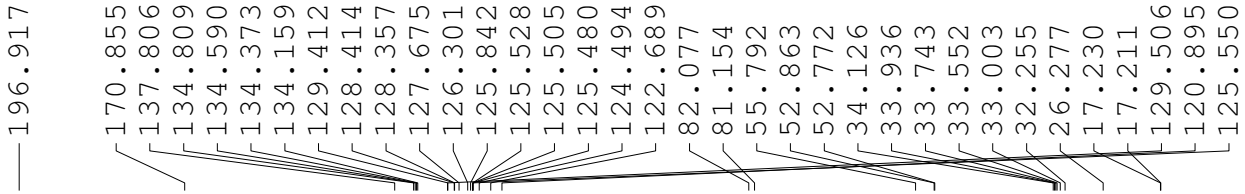


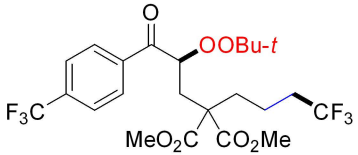


4j

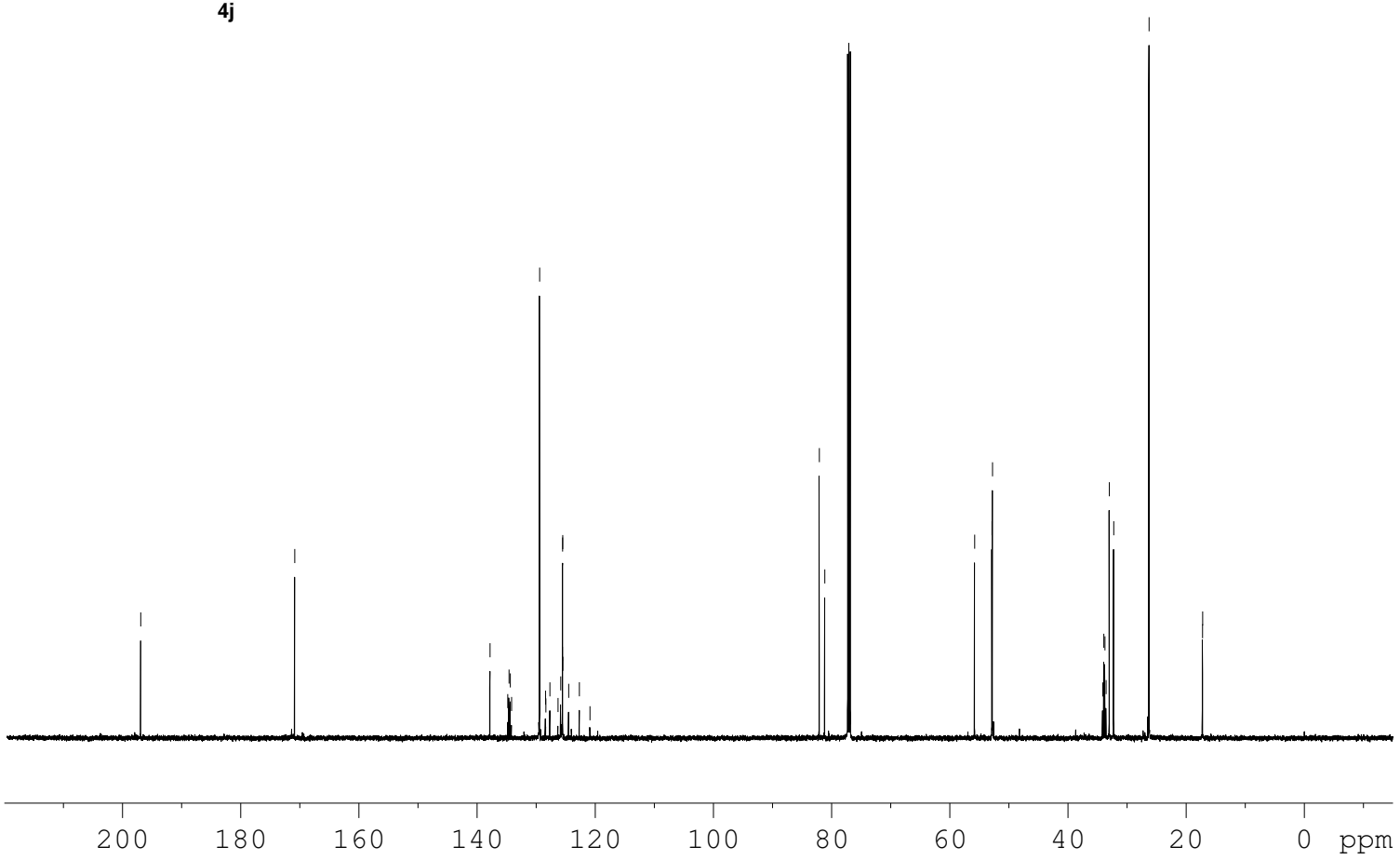
```
NAME      wll-523p-20201230
EXPNO                    1
PROCNO                   1
Date_             20201230
Time                 13.29
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               9615.385 Hz
FIDRES           0.146719 Hz
AQ               3.4079220 sec
RG                    38.1
DW                  52.000 usec
DE                   6.50 usec
TE                  294.1 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.77 usec
SI                   65536
SF            600.1700072 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



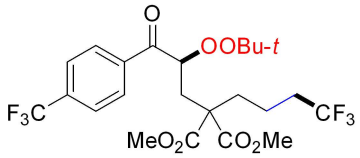




4j

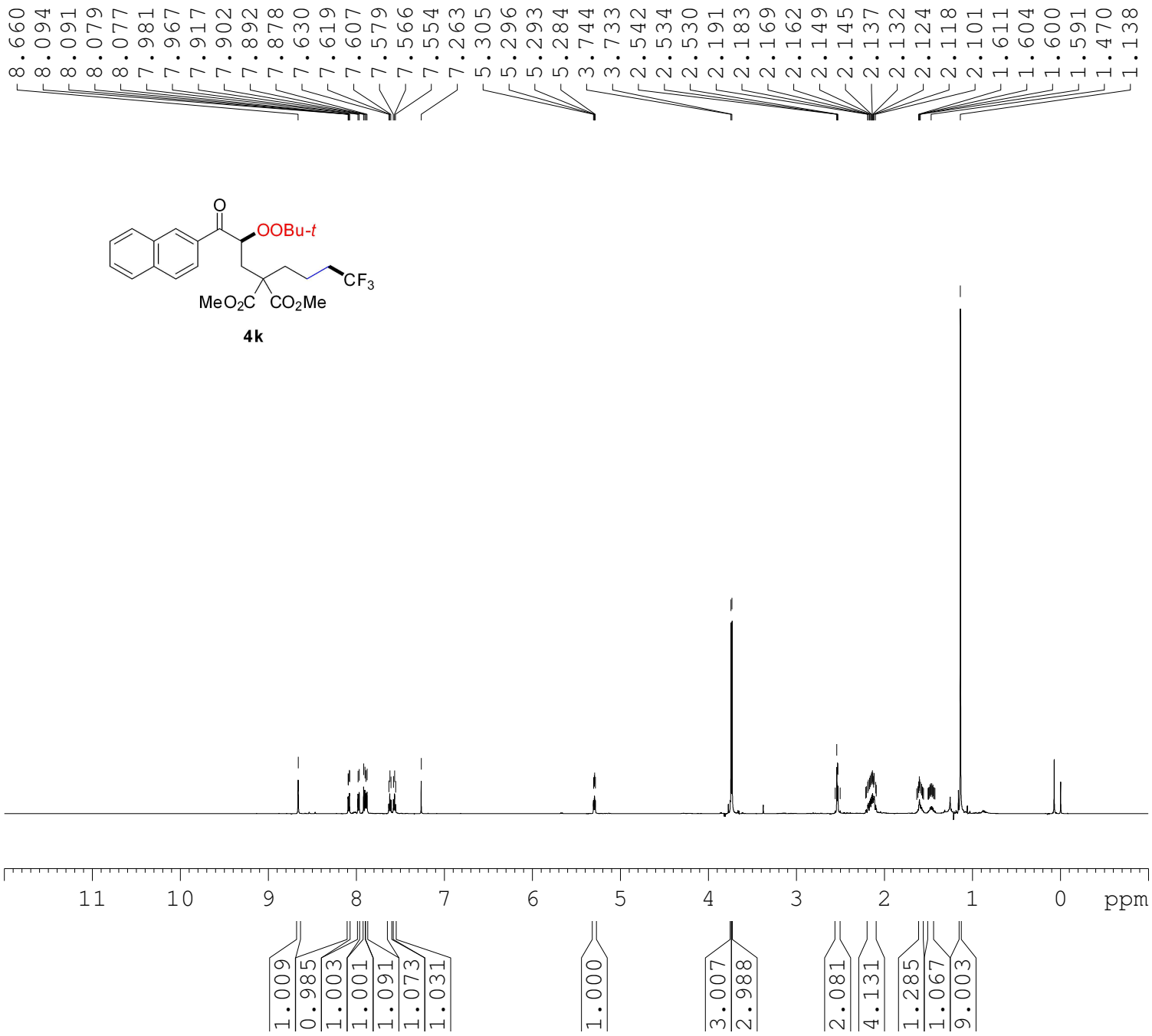



```
NAME      wll-523p-20201230
EXPNO                     2
PROCNO                    1
Date_              20201230
Time                  13.50
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      400
DS                        4
SWH               36057.691 Hz
FIDRES             0.550197 Hz
AQ                0.9088159 sec
RG                   190.02
DW                   13.867 usec
DE                     6.50 usec
TE                   295.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            150.9279571 MHz
NUC1                    13C
P1                    11.90 usec
SI                    32768
SF              150.9128665 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

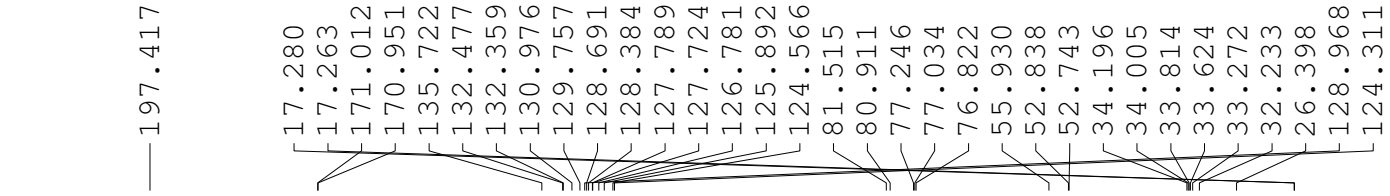


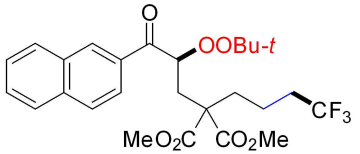
4j


```
NAME      wll-523p-20201230
EXPNO                     3
PROCNO                    1
Date_              20201230
Time                  13.52
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                   131072
SOLVENT              CDCl3
NS                      16
DS                       4
SWH             133928.578 Hz
FIDRES            1.021794 Hz
AQ                0.4893855 sec
RG                    15.49
DW                 3.733 usec
DE                 6.50 usec
TE                    294.7 K
D1              1.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           564.6675534 MHz
NUC1                    19F
P1                 11.90 usec
SI                    65536
SF             564.7240258 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    1.00
```



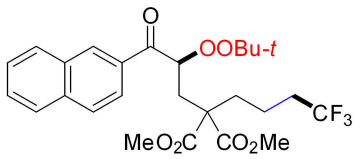

```
NAME      wll-525p-20210105
EXPNO                    1
PROCNO                   1
Date_             20210105
Time                 20.27
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               9615.385 Hz
FIDRES            0.146719 Hz
AQ               3.4079220 sec
RG                   62.22
DW                  52.000 usec
DE                    6.50 usec
TE                   294.6 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.77 usec
SI                   65536
SF            600.1700142 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```





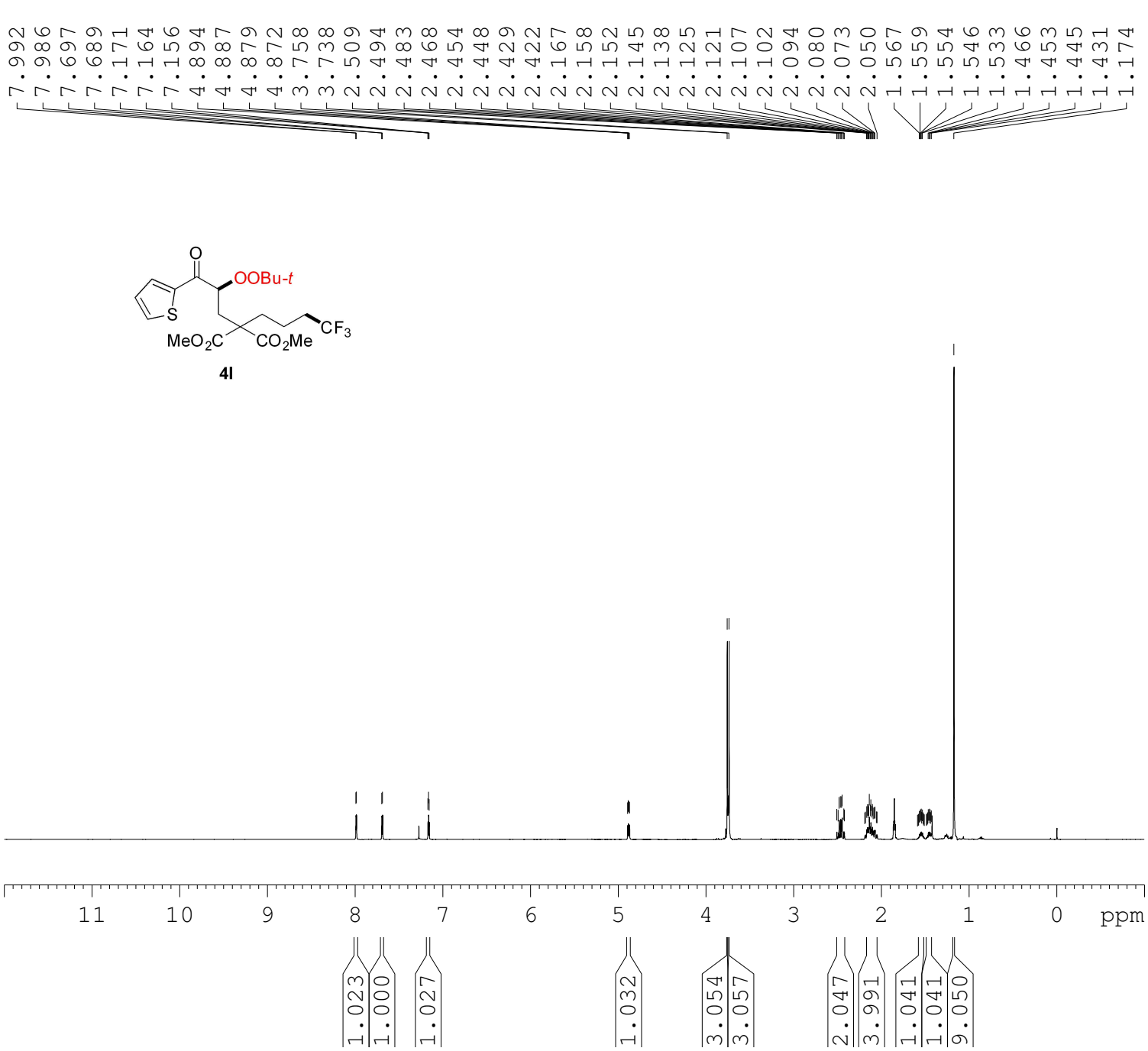
4k

NAME	wll-525p-20210105
EXPNO	3
PROCNO	1
Date_	20210105
Time	20.48
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	350
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	295.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128675 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



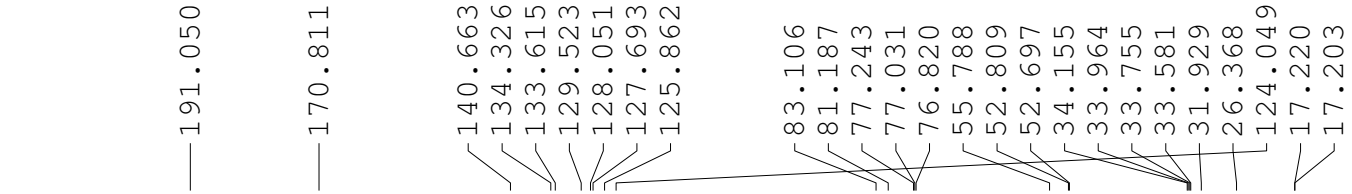
4k

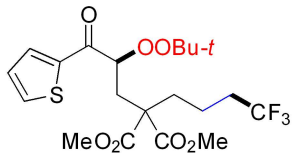

```
NAME      wll-525p-20210105
EXPNO                     2
PROCNO                    1
Date_              20210105
Time                  20.29
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                   131072
SOLVENT              CDCl3
NS                      16
DS                       4
SWH             133928.578 Hz
FIDRES            1.021794 Hz
AQ                0.4893855 sec
RG                    15.49
DW                   3.733 usec
DE                    6.50 usec
TE                   294.7 K
D1              1.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          564.6675534 MHz
NUC1                    19F
P1                   11.90 usec
SI                   65536
SF            564.7240258 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



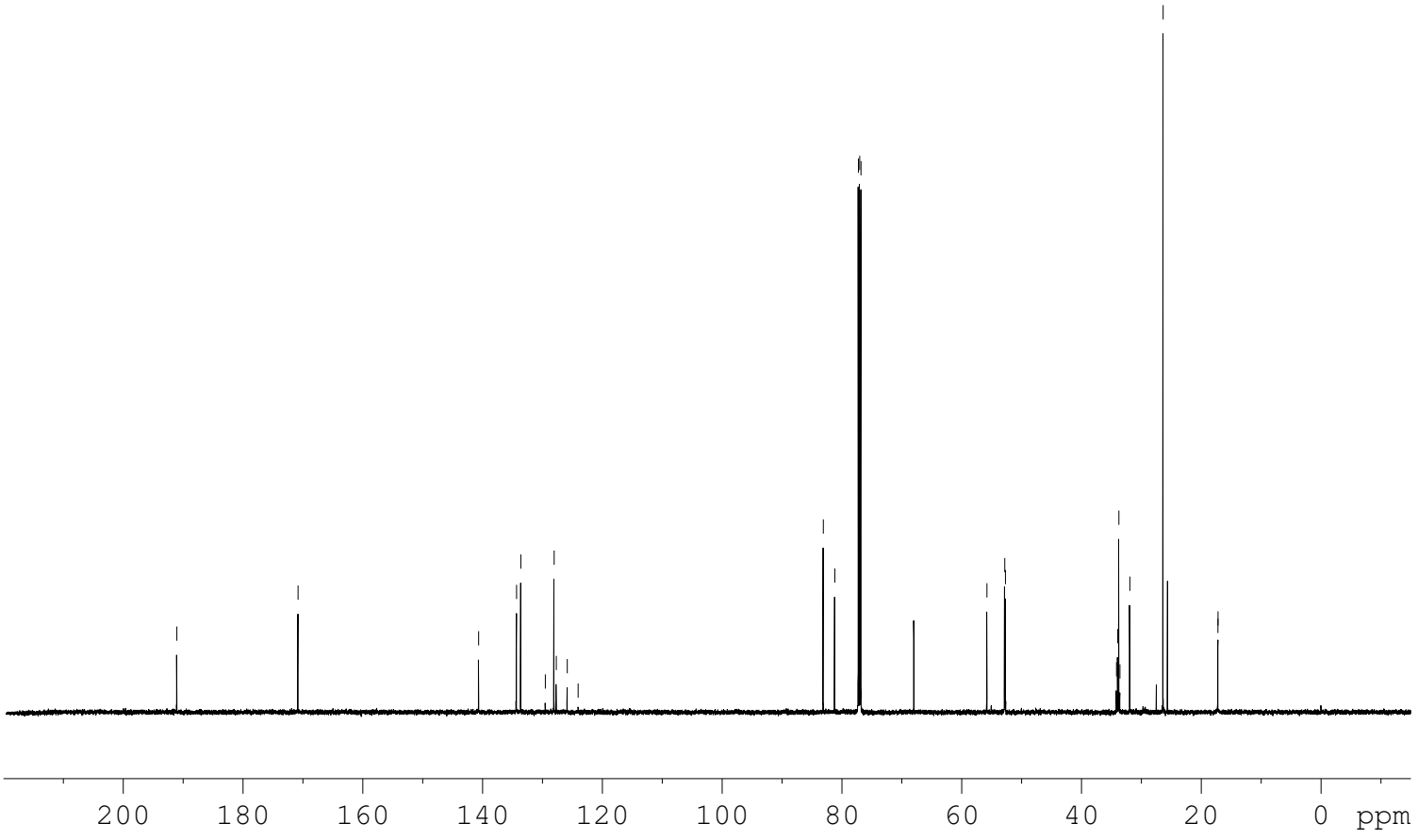
NAME	wll-460p-20201120
EXPNO	1
PROCNO	1
Date_	20201120
Time	23.14
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	9615.385 Hz
FIDRES	0.146719 Hz
AQ	3.4079220 sec
RG	44.5
DW	52.000 usec
DE	6.50 usec
TE	294.5 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========	
SFO1	600.1739011 MHz
NUC1	1H
P1	9.77 usec
SI	65536
SF	600.1700070 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

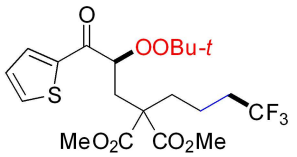




4l

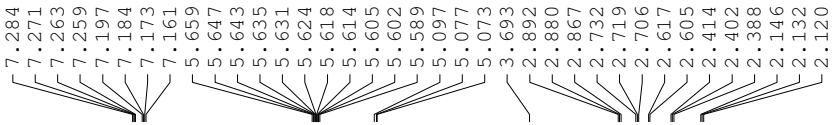


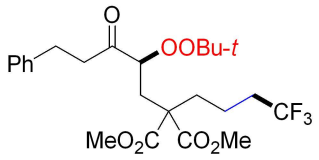

```
NAME      wll-460p-20201121
EXPNO                     1
PROCNO                    1
Date_              20201121
Time                  12.24
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      400
DS                        4
SWH              36057.691 Hz
FIDRES            0.550197 Hz
AQ               0.9088159 sec
RG                   190.02
DW                  13.867 usec
DE                    6.50 usec
TE                   296.6 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          150.9279571 MHz
NUC1                    13C
P1                    11.90 usec
SI                    32768
SF            150.9128665 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



4l

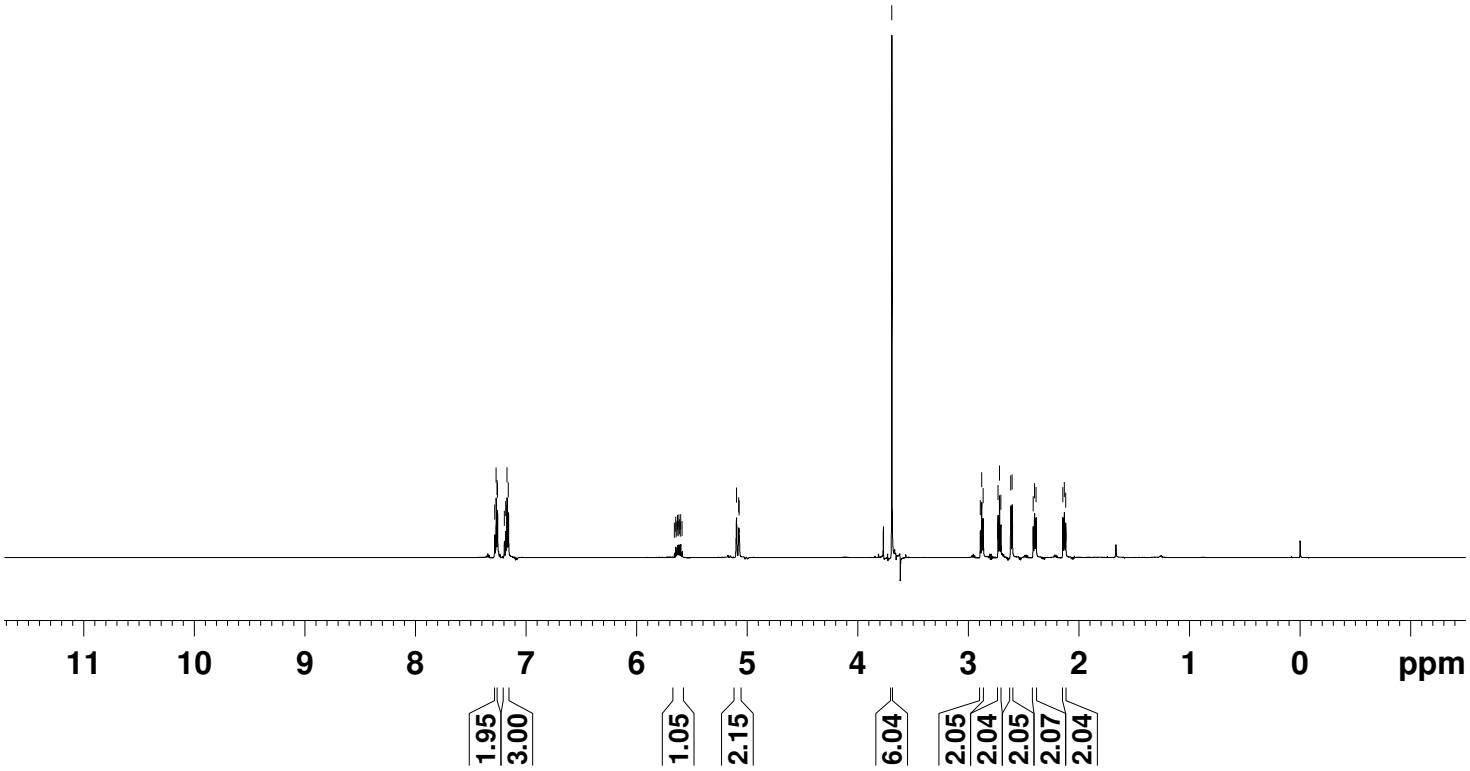

```
NAME      wll-460p-20201120
EXPNO                     2
PROCNO                    1
Date_              20201120
Time                  23.15
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                   131072
SOLVENT               CDCl3
NS                       16
DS                        4
SWH              133928.578 Hz
FIDRES             1.021794 Hz
AQ                 0.4893855 sec
RG                     15.49
DW                     3.733 usec
DE                      6.50 usec
TE                     294.5 K
D1               1.00000000 sec
D11              0.03000000 sec
D12              0.00002000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            564.6675534 MHz
NUC1                    19F
P1                    11.90 usec
SI                    65536
SF              564.7240258 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

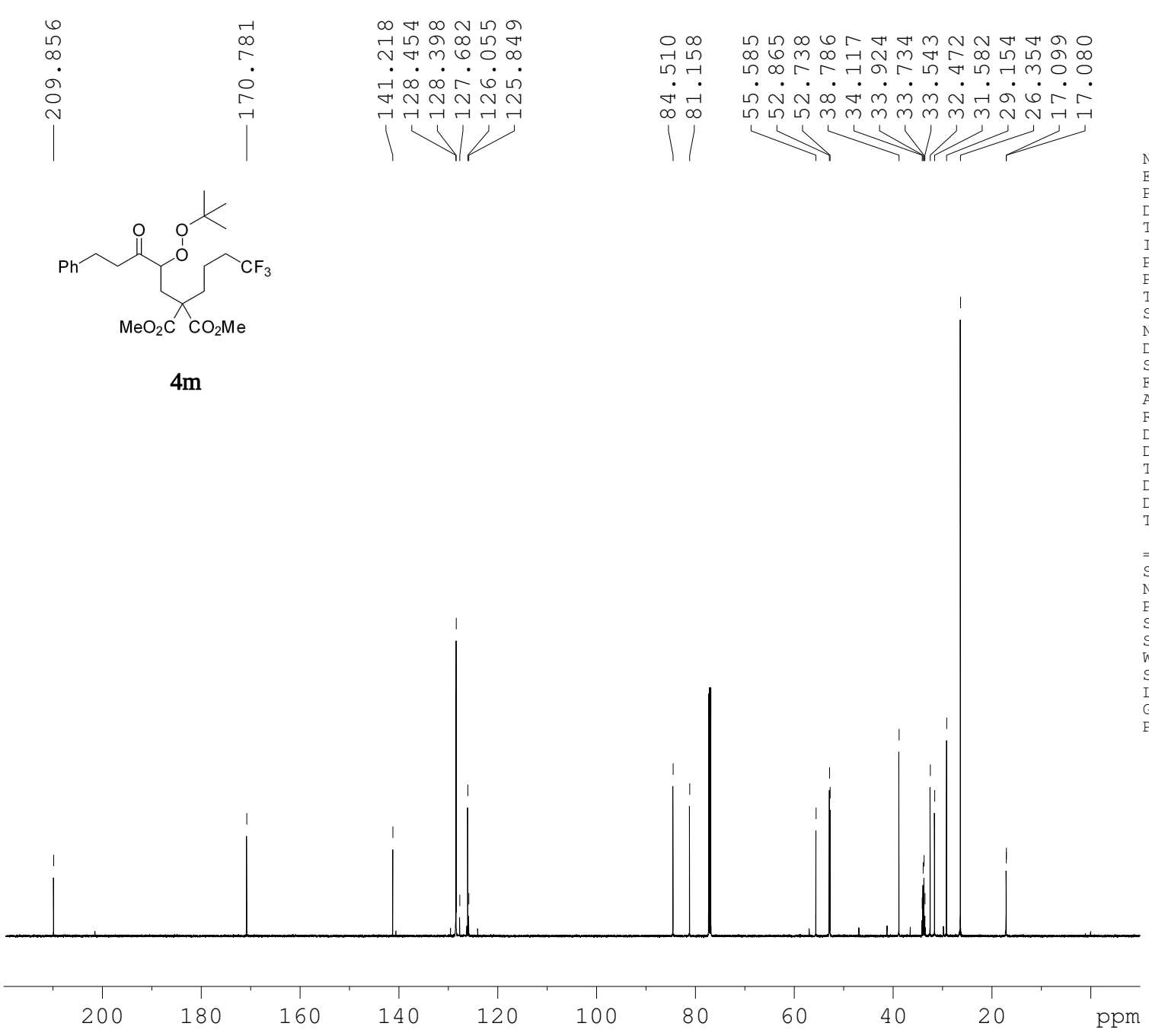




4m

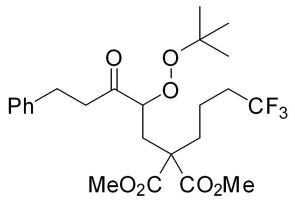
```
NAME      wll-4-16-1-20210612
EXPNO                       1
PROCNO                      1
Date_              20210612
Time                   11.15
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 9615.385 Hz
FIDRES              0.146719 Hz
AQ                  3.4079220 sec
RG                       44.5
DW                     52.000 usec
DE                      6.50 usec
TE                     296.1 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1            600.1739011 MHz
NUC1                      1H
P1                      9.96 usec
SI                     65536
SF              600.1700136 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```





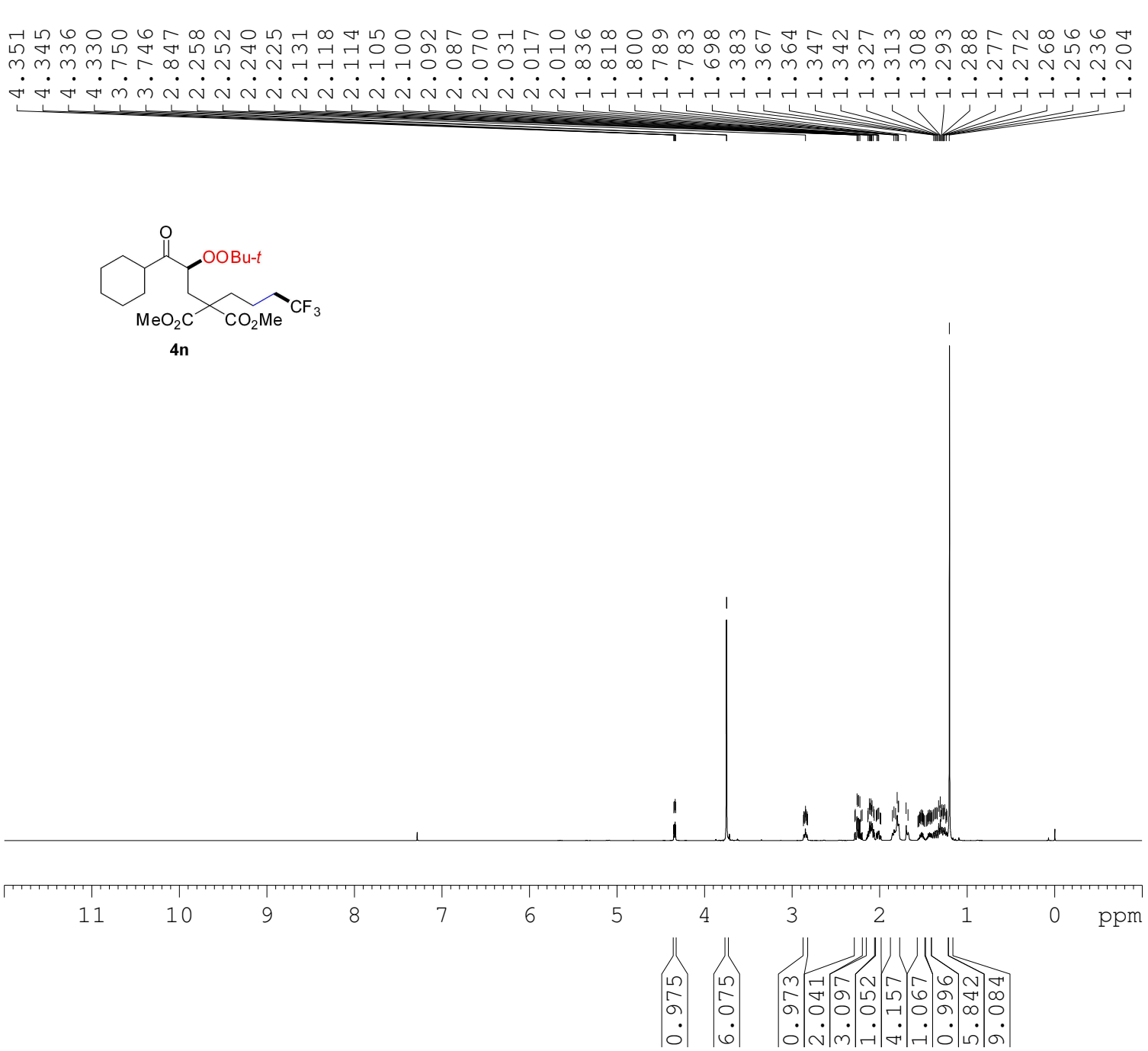
4m

NAME	wll-4-17-1p-20210420
EXPNO	3
PROCNO	1
Date_	20210420
Time	15.15
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	296.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	14.00 usec
SI	32768
SF	150.9128665 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



4m

NAME	wll-4-17-1p-20210420
EXPNO	2
PROCNO	1
Date_	20210420
Time	11.28
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgfhigqn.2
TD	131072
SOLVENT	CDCl3
NS	16
DS	4
SWH	133928.578 Hz
FIDRES	1.021794 Hz
AQ	0.4893855 sec
RG	15.49
DW	3.733 usec
DE	6.50 usec
TE	294.9 K
D1	1.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	564.6675534 MHz
NUC1	19F
P1	25.77 usec
SI	65536
SF	564.7240258 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



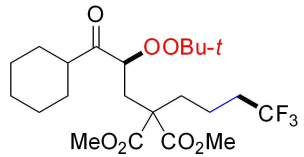
BRUKER

NAME	wll-535-1-20210112
EXPNO	1
PROCNO	1
Date_	20210113
Time	1.45
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	9615.385 Hz
FIDRES	0.146719 Hz
AQ	3.4079220 sec
RG	28.69
DW	52.000 usec
DE	6.50 usec
TE	294.5 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

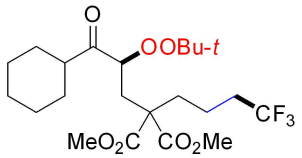
SFO1	600.1739011 MHz
NUC1	1H
P1	9.77 usec
SI	65536
SF	600.1700026 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

213.157
129.544
124.024
170.943
170.908
127.686
125.855
83.062
80.794
55.776
52.802
52.700
45.830
34.133
33.942
33.749
33.559
32.501
31.684
29.234
28.182
26.369
25.807
25.520
17.116
17.100



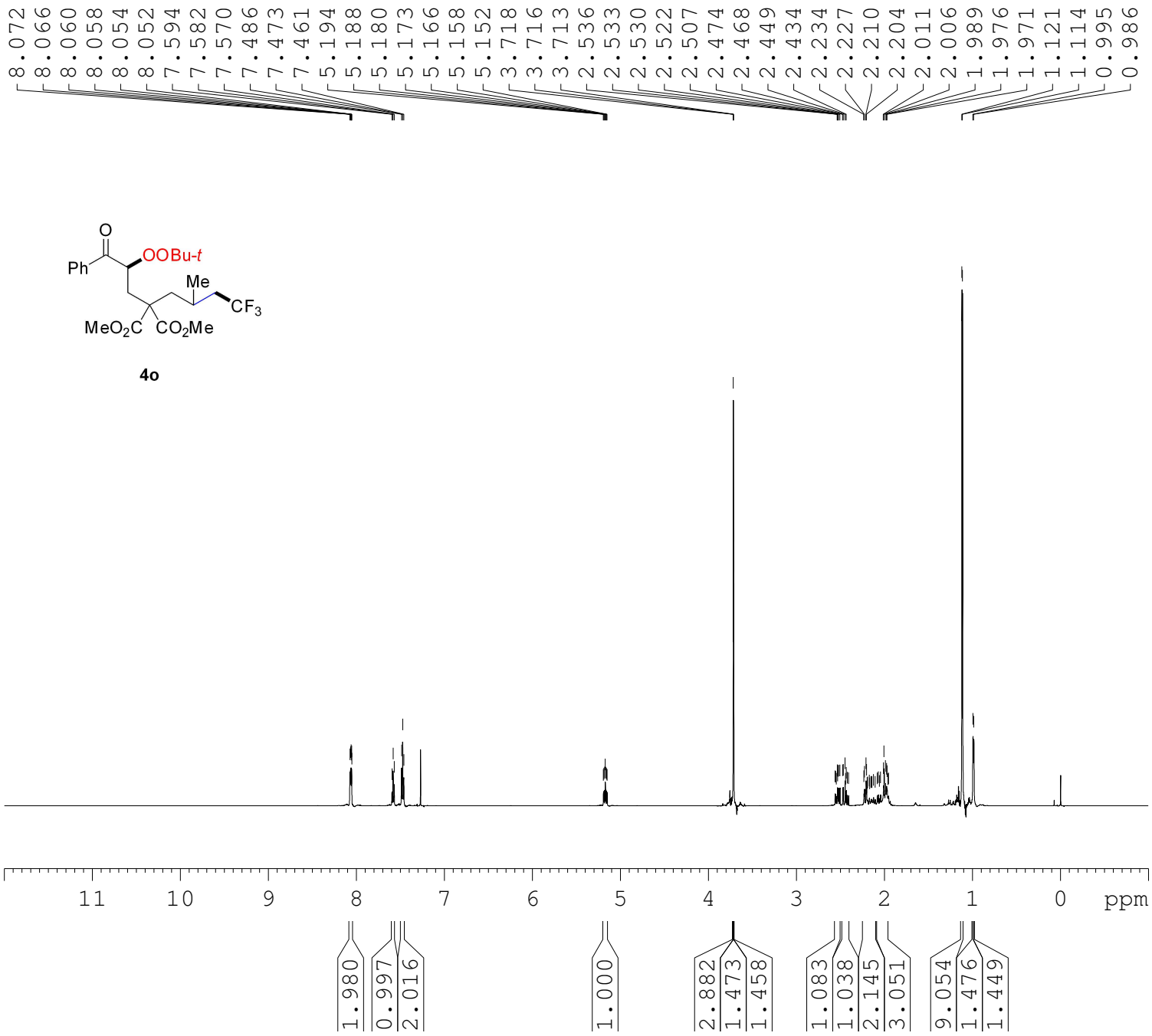
4n

NAME	wll-535-1-20210112
EXPNO	3
PROCNO	1
Date_	20210113
Time	9.31
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	300
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	295.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128665 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

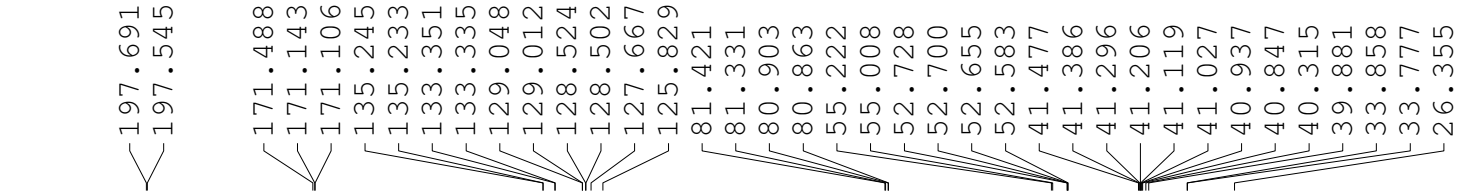


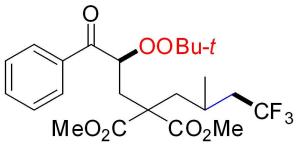
4n


```
NAME      wll-535-1-20210112
EXPNO                      2
PROCNO                     1
Date_               20210113
Time                    1.47
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                    131072
SOLVENT              CDCl3
NS                       16
DS                        4
SWH              133928.578 Hz
FIDRES             1.021794 Hz
AQ                0.4893855 sec
RG                    15.49
DW                   3.733 usec
DE                    6.50 usec
TE                   294.6 K
D1                1.00000000 sec
D11               0.03000000 sec
D12               0.00002000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             564.6675534 MHz
NUC1                     19F
P1                    11.90 usec
SI                    65536
SF               564.7240258 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00
```



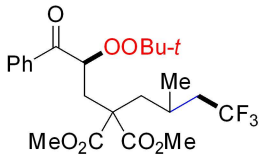

```
NAME      wll-521p-20201230
EXPNO                    1
PROCNO                   1
Date_             20201230
Time                14.51
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH              9615.385 Hz
FIDRES           0.146719 Hz
AQ               3.4079220 sec
RG                   44.5
DW                 52.000 usec
DE                  6.50 usec
TE                 294.6 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.77 usec
SI                  65536
SF            600.1700097 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```





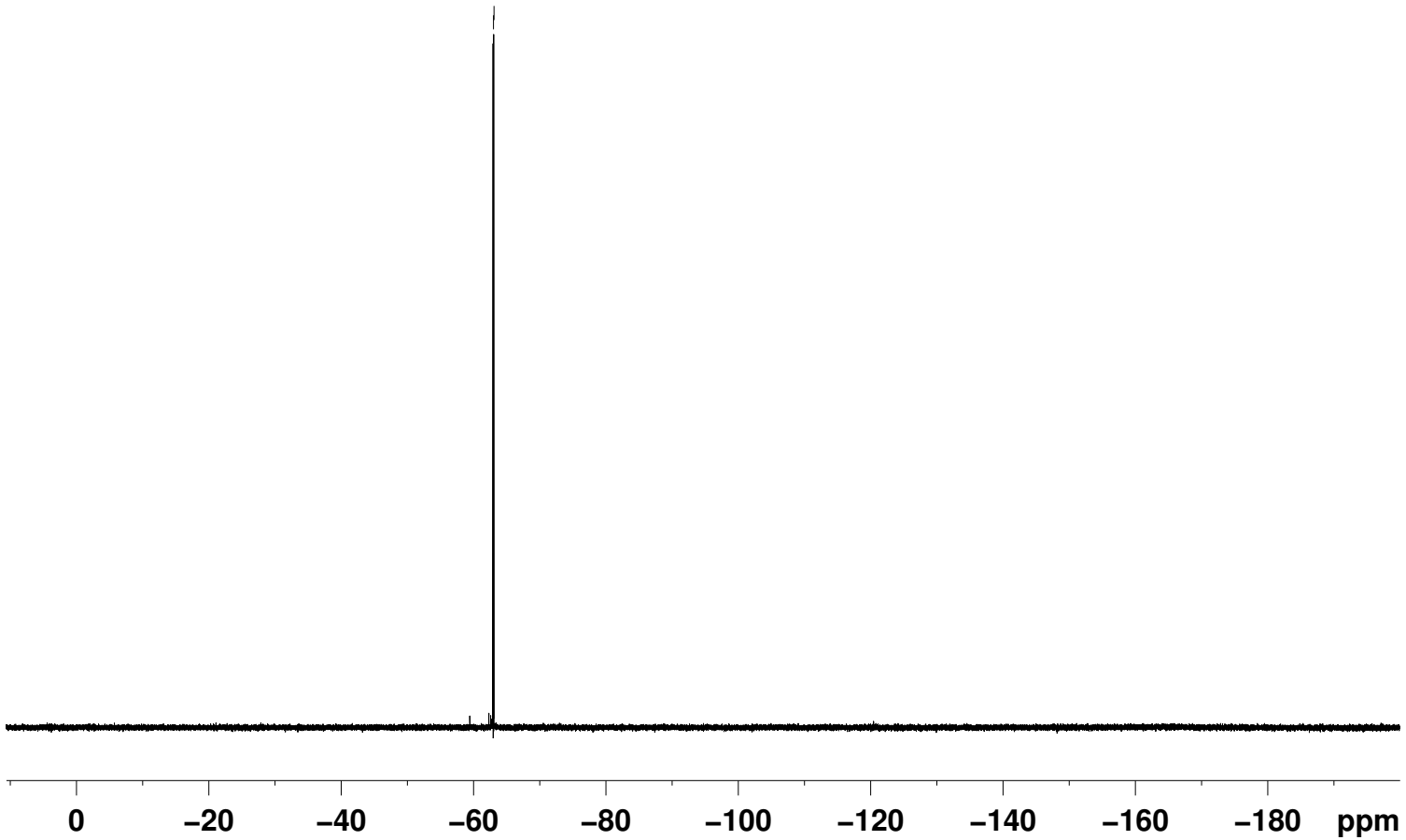
4o


```
NAME      wll-521p-20201230
EXPNO                     3
PROCNO                    1
Date_              20201230
Time                  17.34
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      300
DS                        4
SWH              36057.691 Hz
FIDRES            0.550197 Hz
AQ               0.9088159 sec
RG                   190.02
DW                 13.867 usec
DE                   6.50 usec
TE                  295.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          150.9279571 MHz
NUC1                    13C
P1                  11.90 usec
SI                     32768
SF            150.9128665 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                    1.40
```

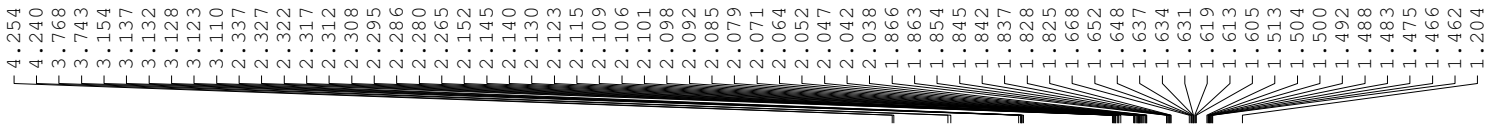


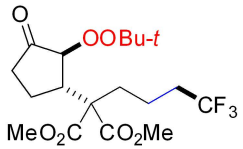
4o

-63.007
-63.091



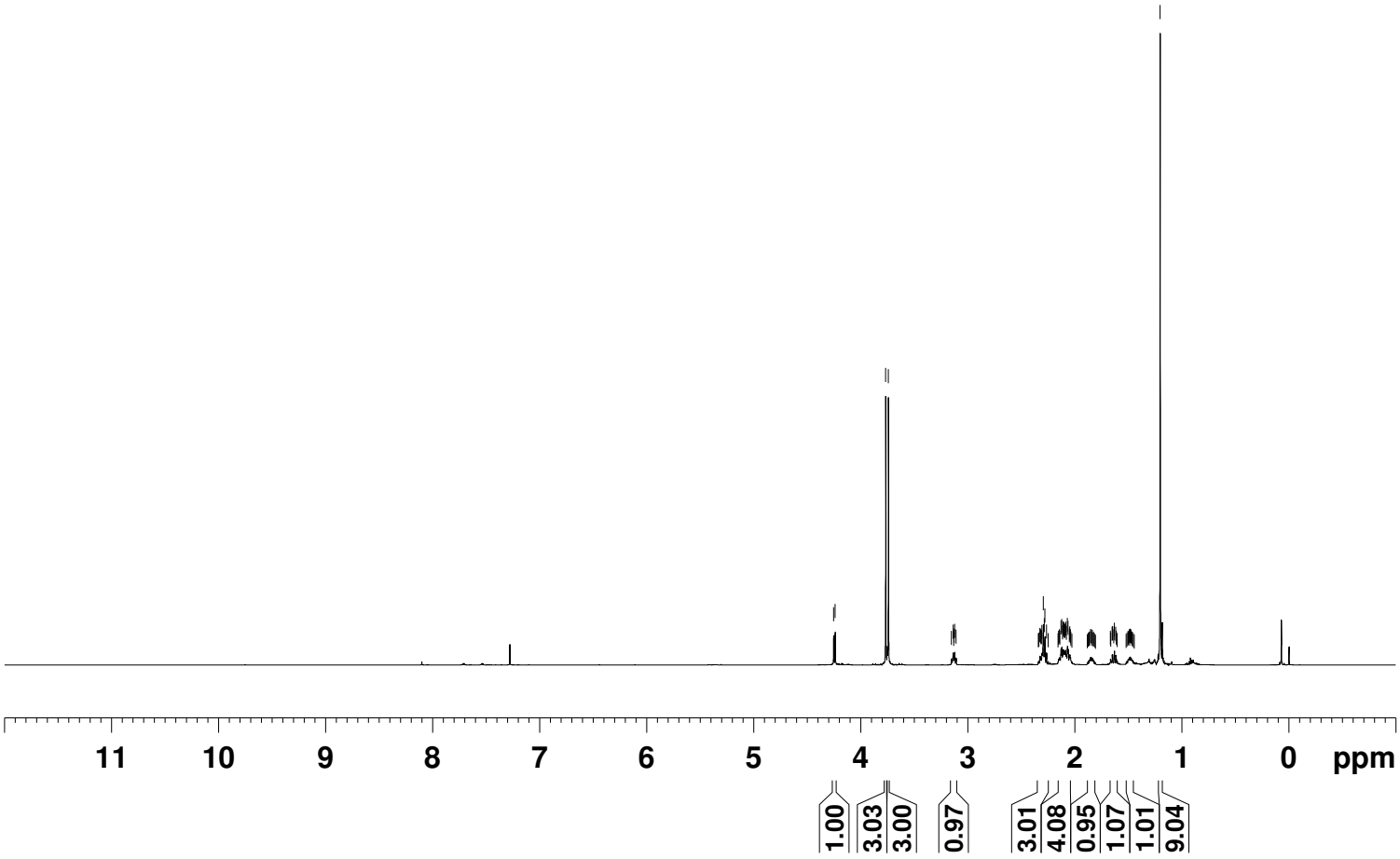

```
NAME      wll-521p-20201228
EXPNO                     2
PROCNO                    1
Date_              20201228
Time                  18.21
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                   131072
SOLVENT               CDCl3
NS                       16
DS                        4
SWH              133928.578 Hz
FIDRES             1.021794 Hz
AQ                 0.4893855 sec
RG                     15.49
DW                     3.733 usec
DE                      6.50 usec
TE                     296.1 K
D1               1.00000000 sec
D11              0.03000000 sec
D12              0.00002000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            564.6675534 MHz
NUC1                    19F
P1                    11.90 usec
SI                    65536
SF              564.7240258 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



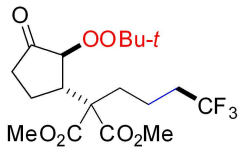


4p

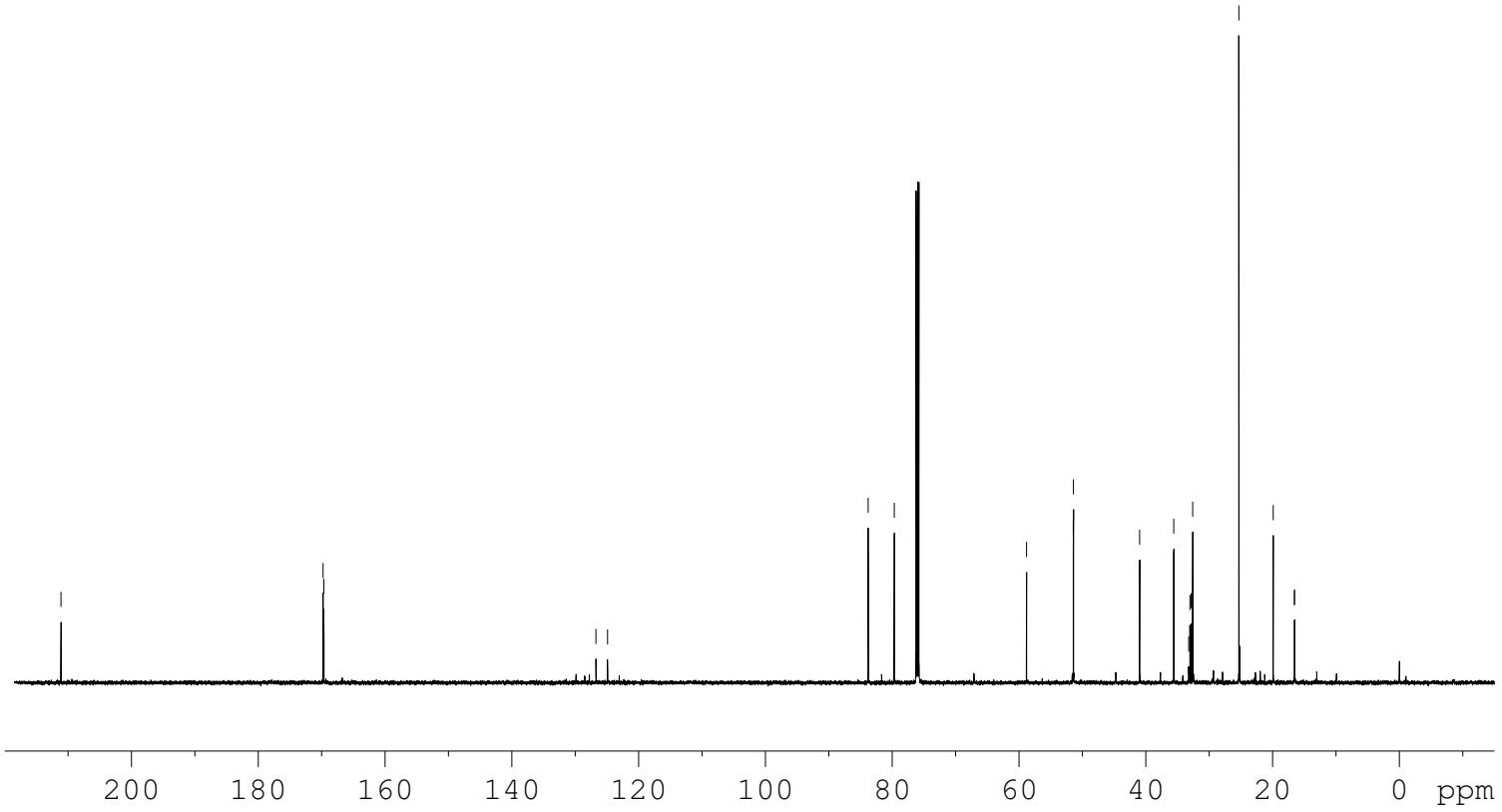
```
NAME      wll-524p-20201230
EXPNO                    1
PROCNO                   1
Date_             20201230
Time                 13.56
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               9615.385 Hz
FIDRES            0.146719 Hz
AQ                3.4079220 sec
RG                    38.1
DW                  52.000 usec
DE                   6.50 usec
TE                  294.3 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.77 usec
SI                  65536
SF            600.1700053 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



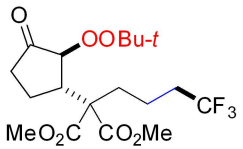
211.116
169.775
169.650
126.734
124.903
83.795
79.673
58.813
51.444
51.418
40.979
35.592
33.234
33.044
32.854
32.664
32.605
25.322
19.901
16.558
16.541



4p

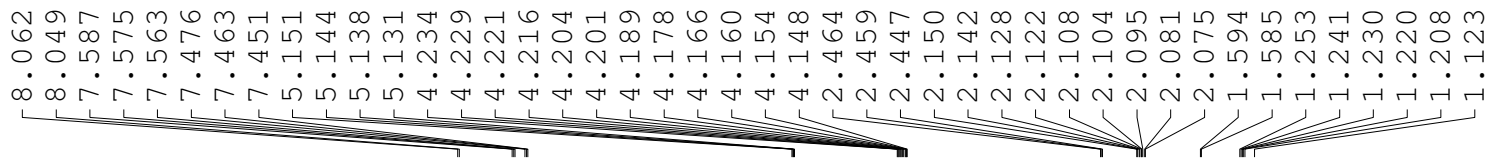


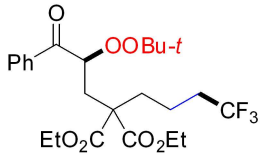

```
NAME      wll-524p-20201230
EXPNO                    2
PROCNO                   1
Date_             20201230
Time                 14.17
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      400
DS                        4
SWH              36057.691 Hz
FIDRES            0.550197 Hz
AQ                0.9088159 sec
RG                   190.02
DW                   13.867 usec
DE                     6.50 usec
TE                    295.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           150.9279571 MHz
NUC1                    13C
P1                   11.90 usec
SI                   32768
SF             150.9130212 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



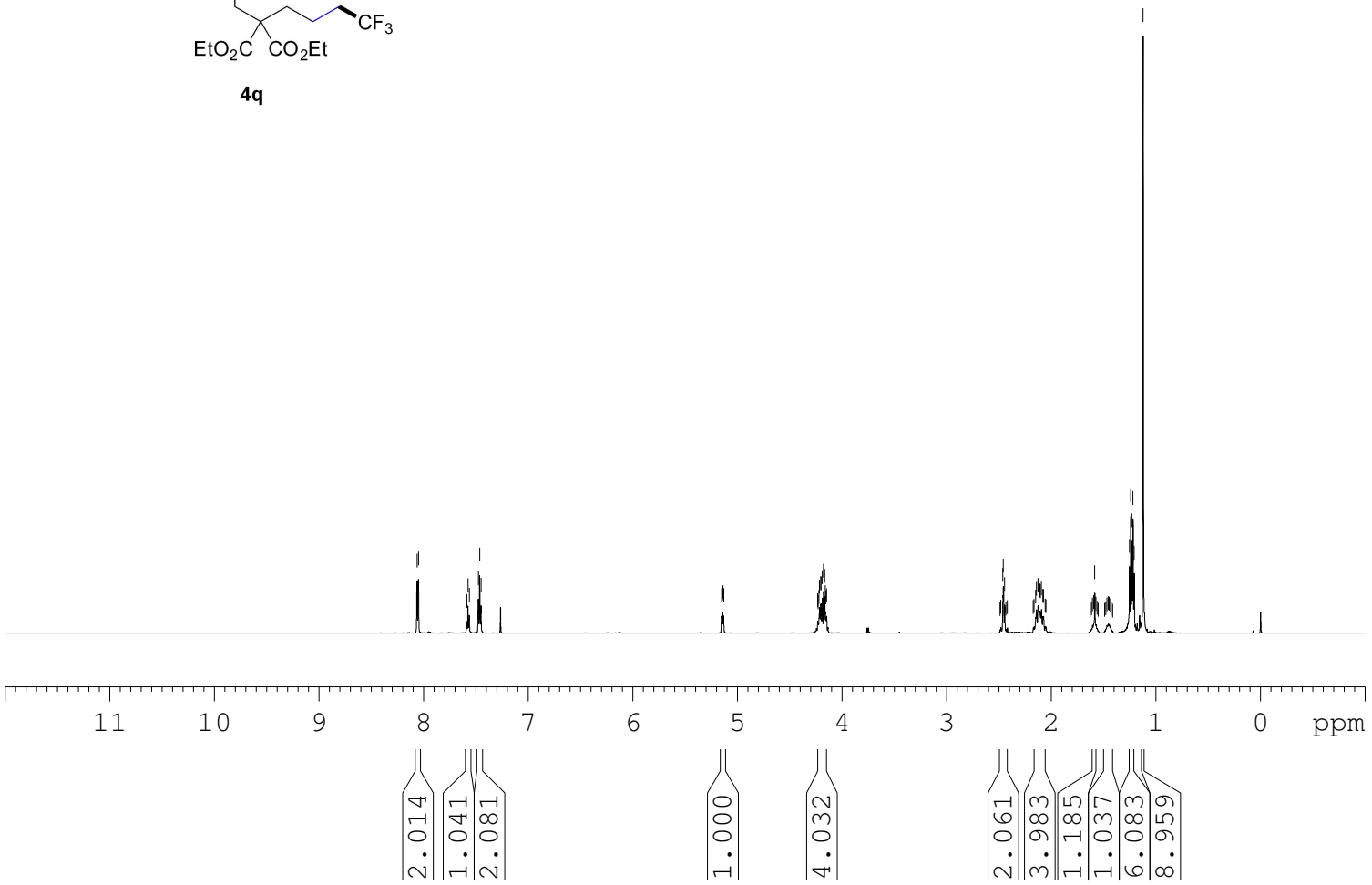
4p


```
NAME      wll-524p-20201230
EXPNO                     3
PROCNO                    1
Date_              20201230
Time                  14.19
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                   131072
SOLVENT              CDCl3
NS                      16
DS                       4
SWH             133928.578 Hz
FIDRES            1.021794 Hz
AQ                0.4893855 sec
RG                    15.49
DW                   3.733 usec
DE                    6.50 usec
TE                   294.9 K
D1              1.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          564.6675534 MHz
NUC1                   19F
P1                   11.90 usec
SI                   65536
SF            564.7240258 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

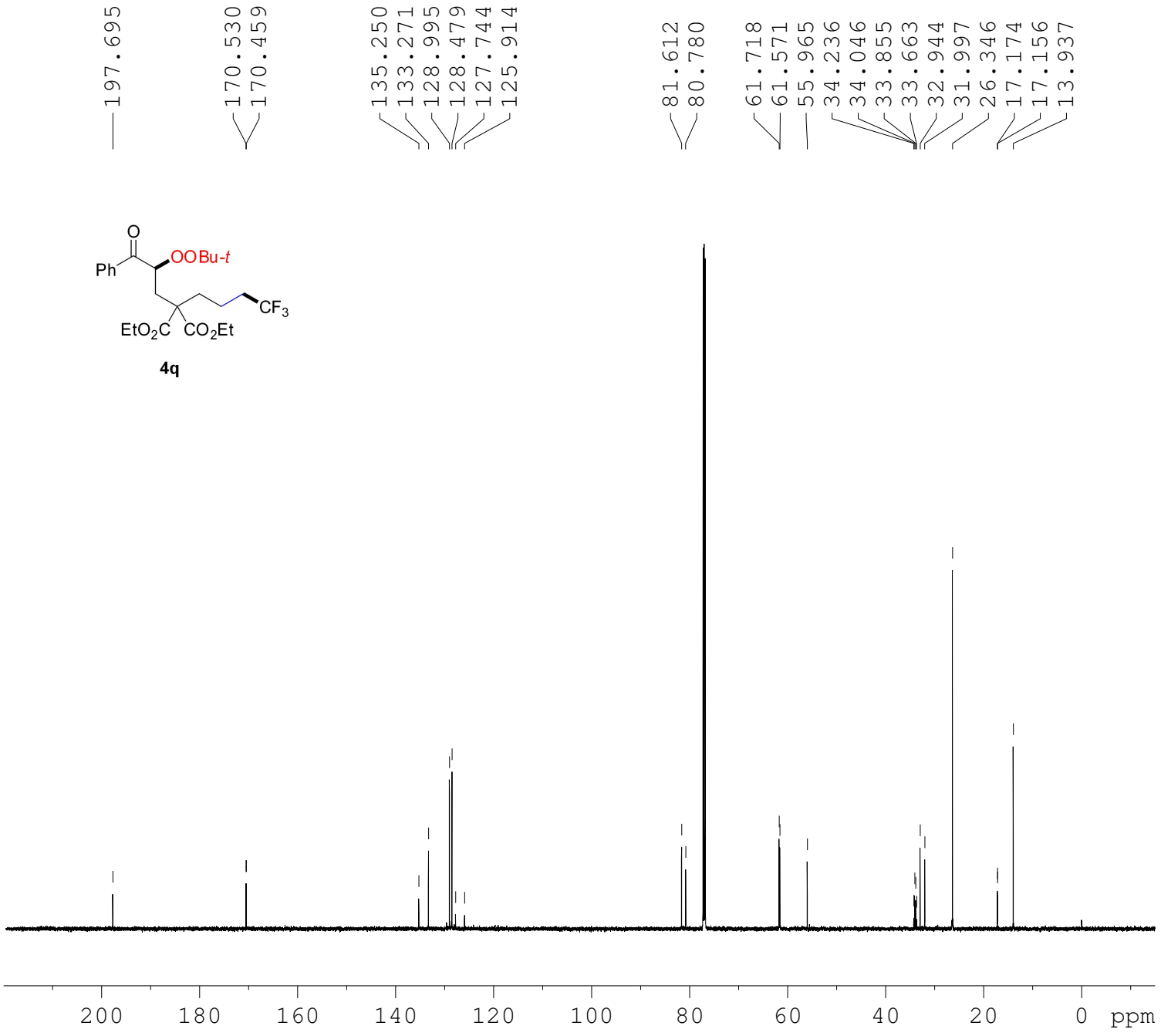




4q

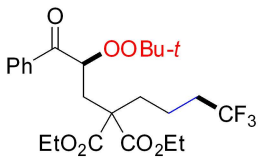



```
NAME      wll-418p-20201106
EXPNO                    1
PROCNO                   1
Date_             20201106
Time                 16.56
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               9615.385 Hz
FIDRES           0.146719 Hz
AQ               3.4079220 sec
RG                   69.87
DW                  52.000 usec
DE                   6.50 usec
TE                  298.7 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.77 usec
SI                   65536
SF            600.1700135 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



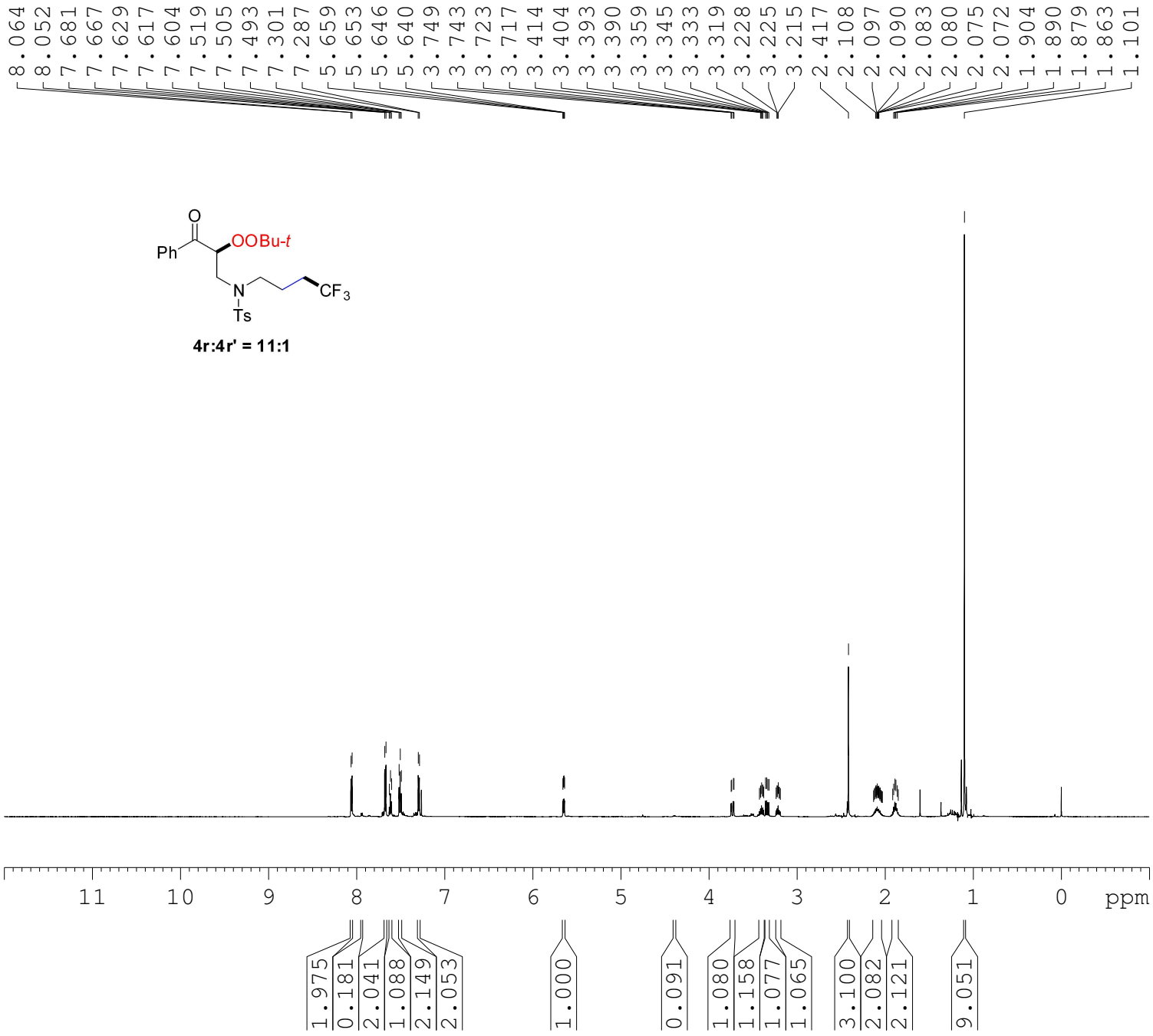
BRUKER

NAME	wll-418p-20201106
EXPNO	3
PROCNO	1
Date_	20201106
Time	17.39
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	800
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	299.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128665 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

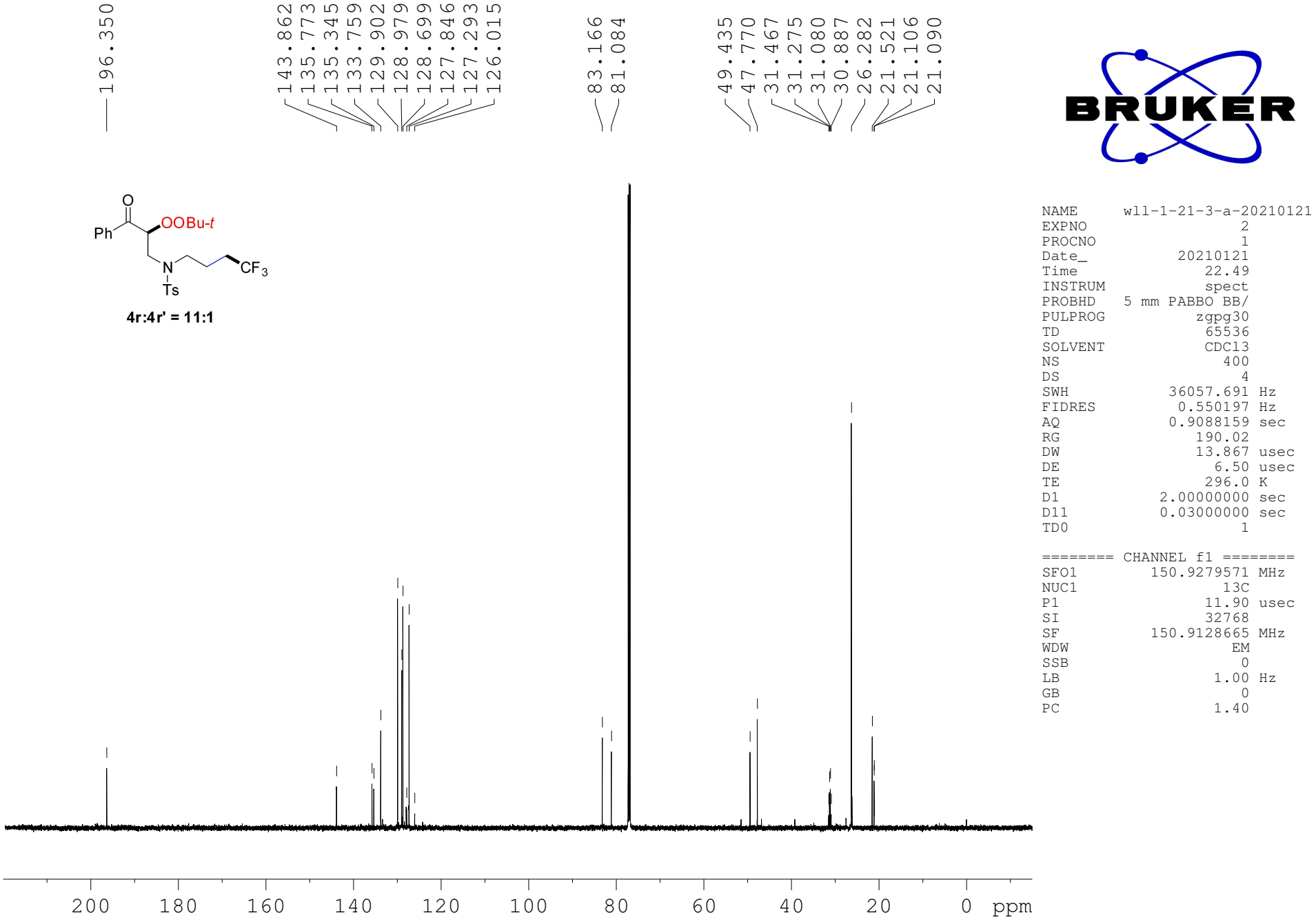


4q


```
NAME      wll-418p-20201106
EXPNO                     2
PROCNO                    1
Date_              20201106
Time                  16.58
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                   131072
SOLVENT               CDCl3
NS                       16
DS                        4
SWH              133928.578 Hz
FIDRES             1.021794 Hz
AQ                0.4893855 sec
RG                    15.49
DW                    3.733 usec
DE                     6.50 usec
TE                   298.6 K
D1             1.00000000 sec
D11            0.03000000 sec
D12            0.00002000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          564.6675534 MHz
NUC1                   19F
P1                   11.90 usec
SI                   65536
SF            564.7240258 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

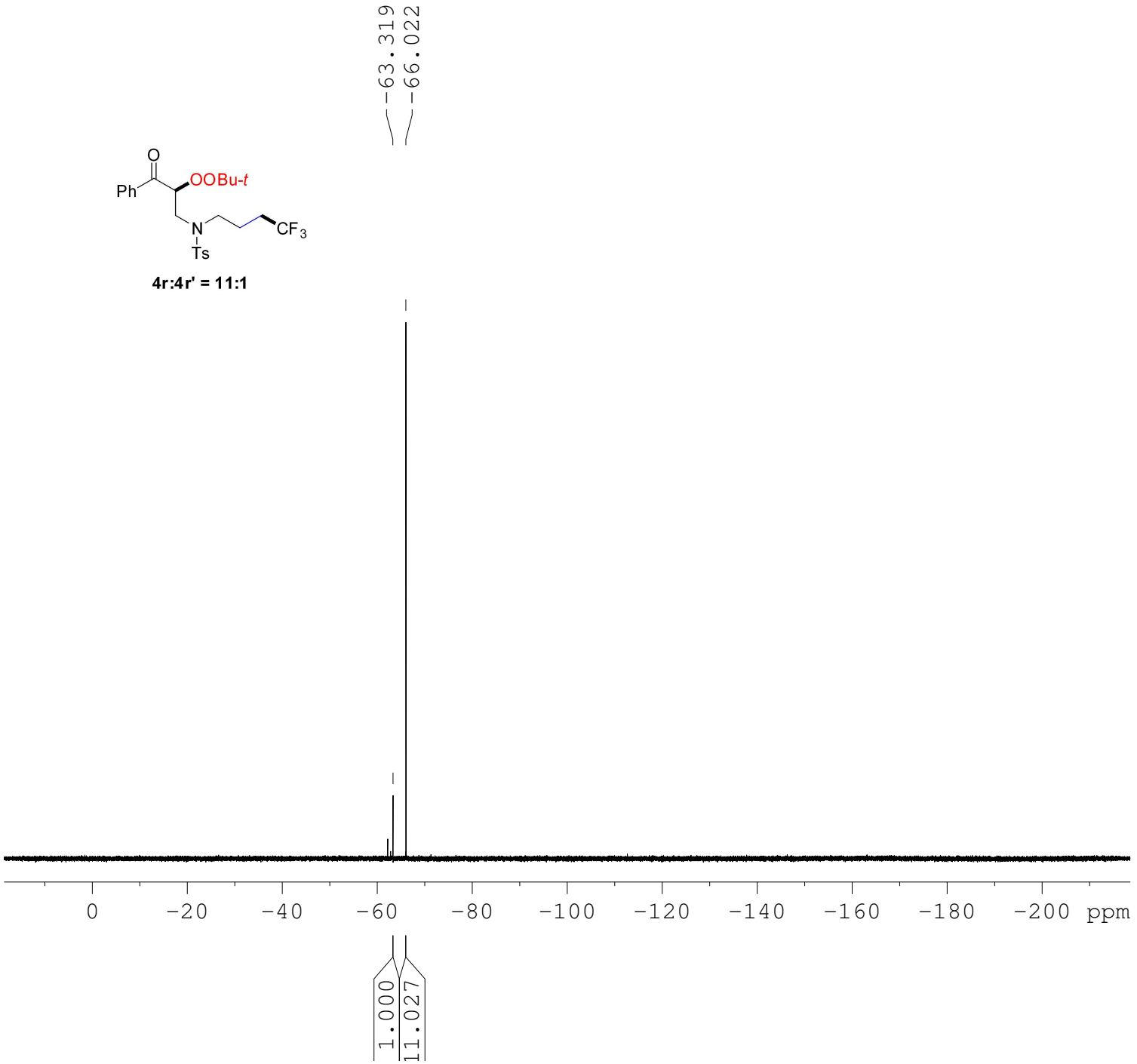



```
NAME      wll-1-21-3-a-20210121
EXPNO                         1
PROCNO                        1
Date_                  20210121
Time                      20.27
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                            8
DS                            0
SWH                    9615.385 Hz
FIDRES                 0.146719 Hz
AQ                     3.4079220 sec
RG                        62.22
DW                       52.000 usec
DE                        6.50 usec
TE                       294.5 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                600.1739011 MHz
NUC1                        1H
P1                        9.77 usec
SI                        65536
SF                  600.1700131 MHz
WDW                         EM
SSB                           0
LB                        0.30 Hz
GB                            0
PC                        1.00
```

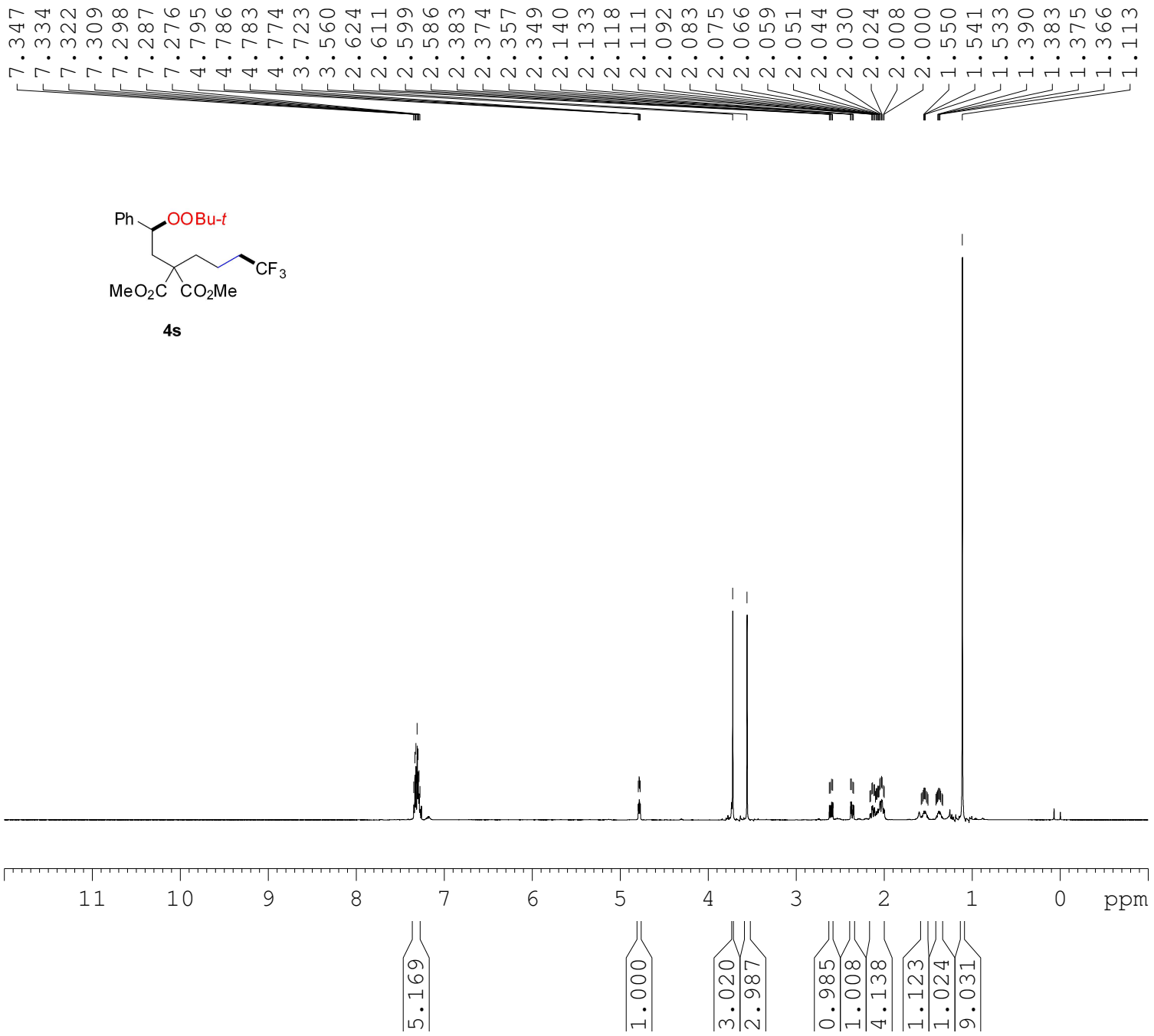


BRUKER

NAME	wll-1-21-3-a-20210121
EXPNO	2
PROCNO	1
Date_	20210121
Time	22.49
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	400
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	296.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128665 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

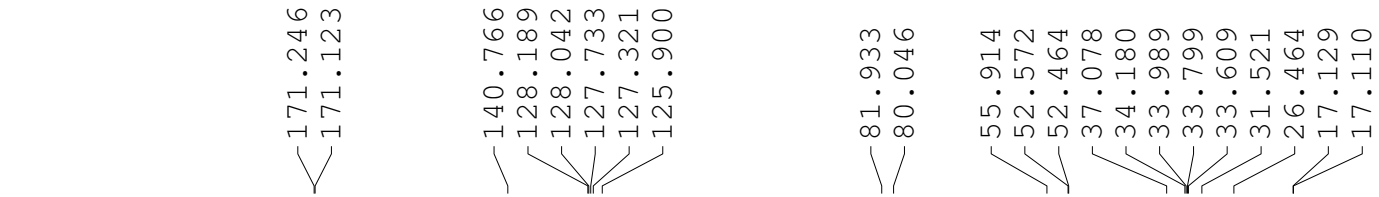


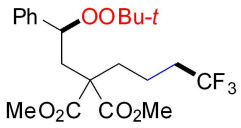

```
NAME      wll-1-21-3-a-20210121
EXPNO                         3
PROCNO                        1
Date_                  20210121
Time                      22.51
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                       131072
SOLVENT                   CDCl3
NS                           16
DS                            4
SWH                 133928.578 Hz
FIDRES                1.021794 Hz
AQ                    0.4893855 sec
RG                        15.49
DW                        3.733 usec
DE                         6.50 usec
TE                       295.3 K
D1                  1.00000000 sec
D11                 0.03000000 sec
D12                 0.00002000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               564.6675534 MHz
NUC1                       19F
P1                        11.90 usec
SI                        65536
SF                 564.7240258 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```



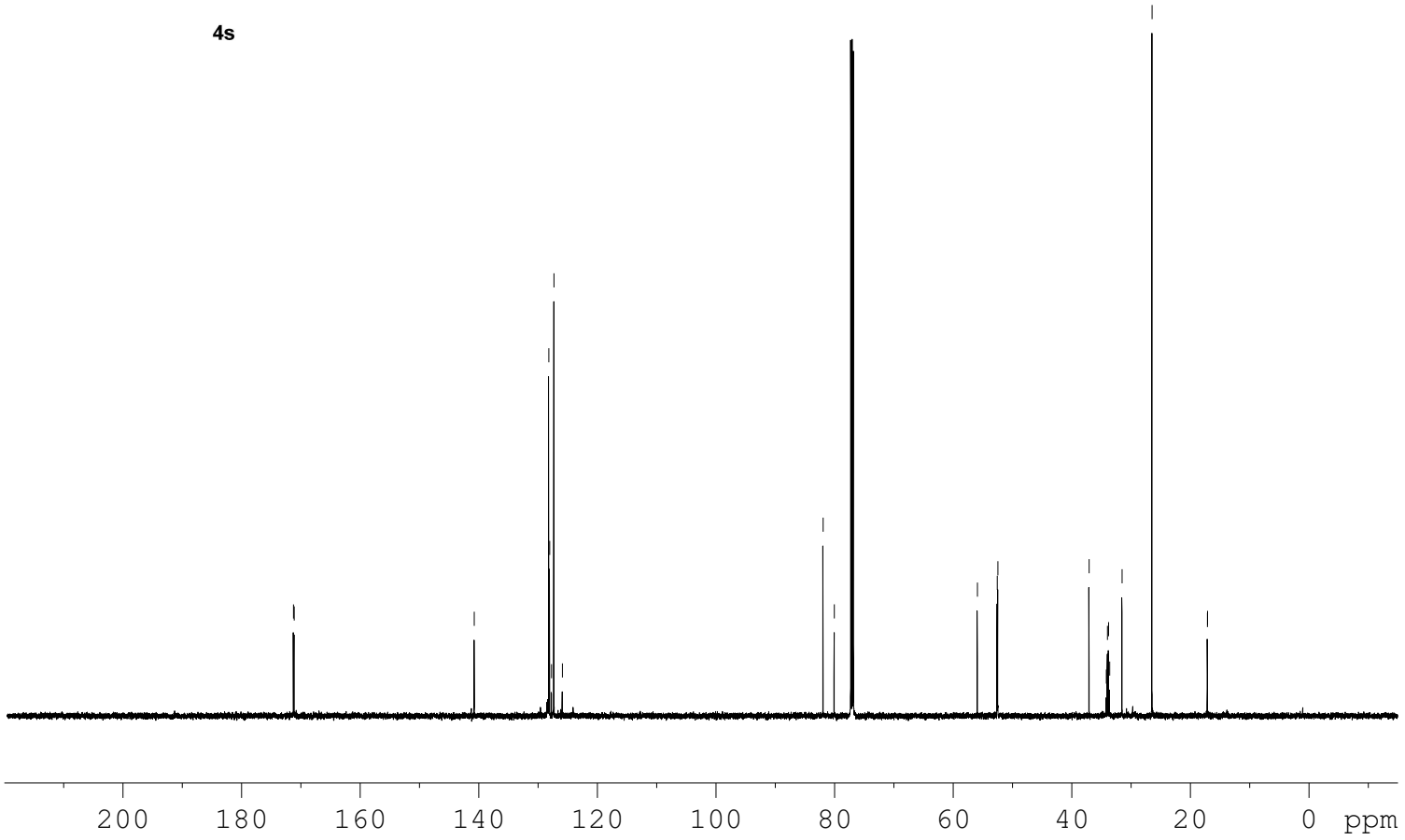
BRUKER

NAME	wll-301p-20200923
EXPNO	1
PROCNO	1
Date_	20200923
Time	14.33
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	9615.385 Hz
FIDRES	0.146719 Hz
AQ	3.4079220 sec
RG	56.75
DW	52.000 usec
DE	6.50 usec
TE	296.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	600.1739011 MHz
NUC1	1H
P1	9.77 usec
SI	65536
SF	600.1700147 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

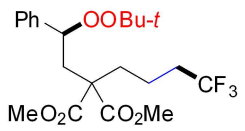




4s

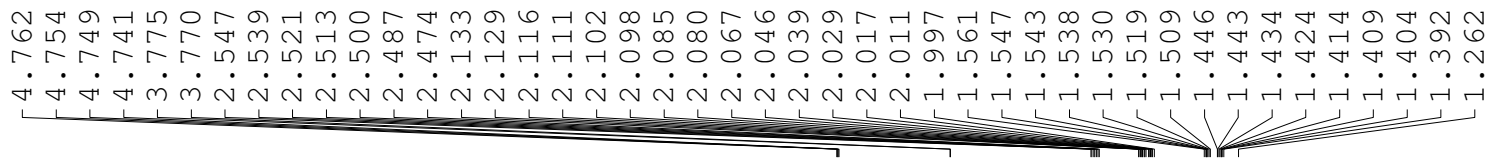


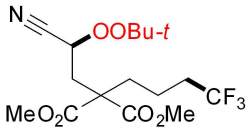

```
NAME      wll-301p-20200923
EXPNO                     3
PROCNO                    1
Date_              20200923
Time                  14.56
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     400
DS                       4
SWH              36057.691 Hz
FIDRES            0.550197 Hz
AQ                0.9088159 sec
RG                   190.02
DW                   13.867 usec
DE                     6.50 usec
TE                   297.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          150.9279571 MHz
NUC1                   13C
P1                   11.90 usec
SI                   32768
SF            150.9128665 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



4s

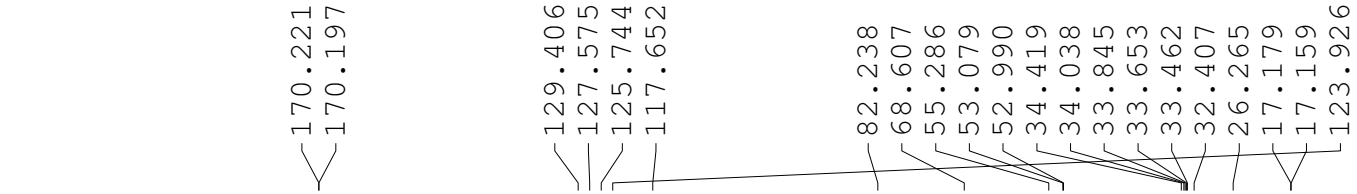

```
NAME      wll-301p-20200923
EXPNO                     2
PROCNO                    1
Date_              20200923
Time                  14.34
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                   131072
SOLVENT              CDCl3
NS                      16
DS                       4
SWH             133928.578 Hz
FIDRES            1.021794 Hz
AQ                0.4893855 sec
RG                   15.49
DW                   3.733 usec
DE                    6.50 usec
TE                   296.6 K
D1              1.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           564.6675534 MHz
NUC1                   19F
P1                   11.90 usec
SI                   65536
SF             564.7240258 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

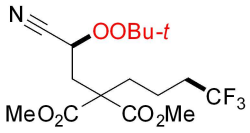




4t

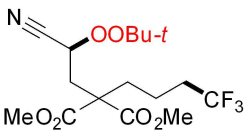
```
NAME      wll-3-8-2-a-20210319
EXPNO                        1
PROCNO                       1
Date_             20210319
Time                    18.16
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 9615.385 Hz
FIDRES              0.146719 Hz
AQ                3.4079220 sec
RG                     87.54
DW                    52.000 usec
DE                     6.50 usec
TE                    296.1 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1            600.1739011 MHz
NUC1                       1H
P1                      9.77 usec
SI                     65536
SF              600.1700125 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```





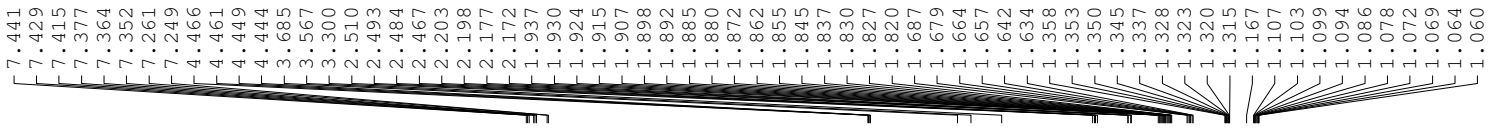
4t

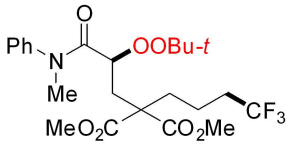
NAME	wll-3-8-2-a-20210319
EXPNO	3
PROCNO	1
Date_	20210319
Time	19.10
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	297.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128665 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



4t

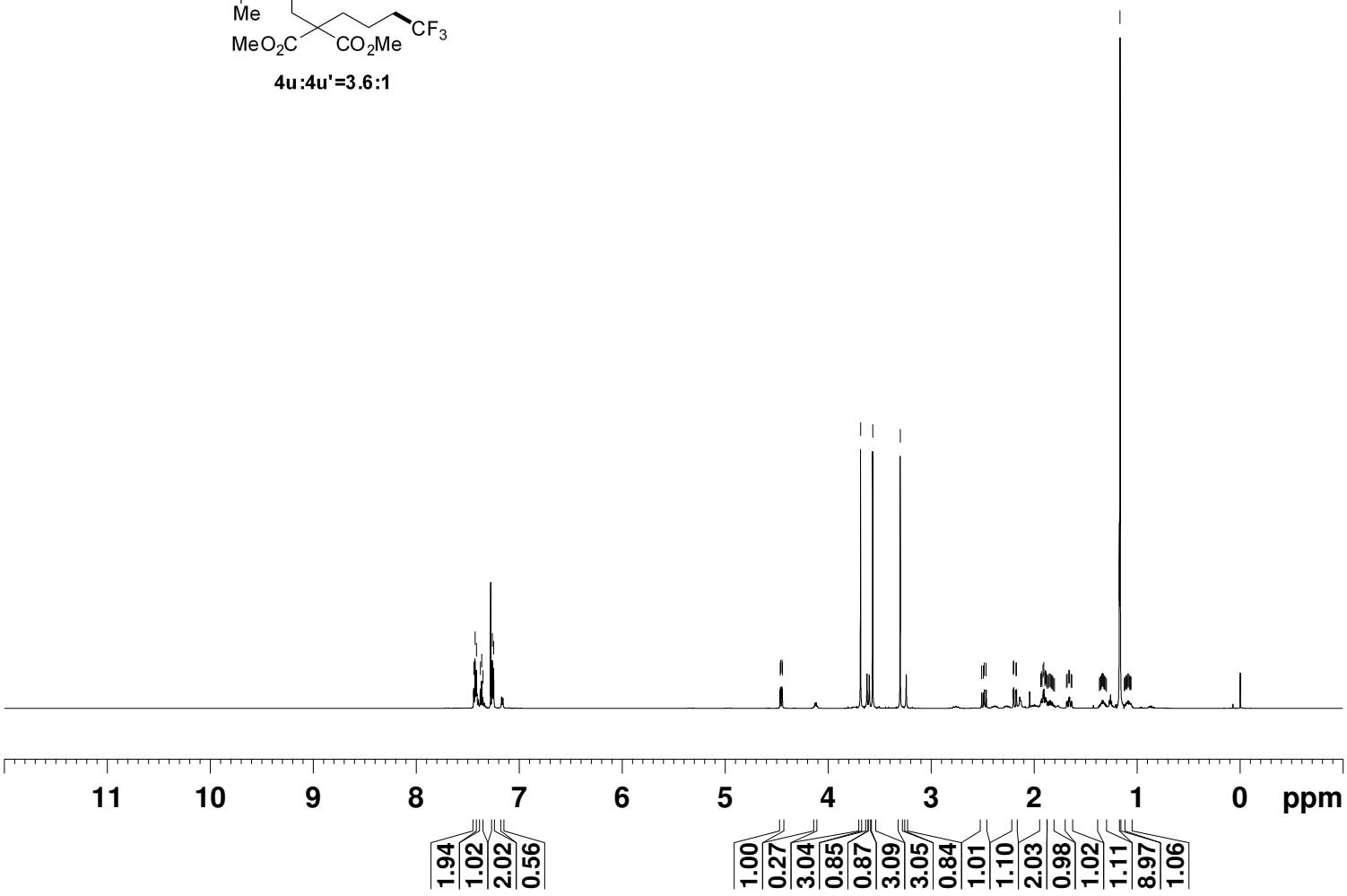

```
NAME      wll-3-8-2-a-20210319
EXPNO                        2
PROCNO                       1
Date_                 20210319
Time                     18.18
INSTRUM                  spect
PROBHD   5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                      131072
SOLVENT                 CDCl3
NS                         16
DS                          4
SWH                133928.578 Hz
FIDRES               1.021794 Hz
AQ                  0.4893855 sec
RG                      15.49
DW                      3.733 usec
DE                       6.50 usec
TE                      296.2 K
D1                 1.00000000 sec
D11                0.03000000 sec
D12                0.00002000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               564.6675534 MHz
NUC1                       19F
P1                       11.90 usec
SI                       65536
SF                 564.7240258 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```





4u:4u'=3.6:1

```
NAME      wll-519p-20201228
EXPNO                    1
PROCNO                   1
Date_             20201228
Time                 18.41
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               9615.385 Hz
FIDRES            0.146719 Hz
AQ                3.4079220 sec
RG                    44.5
DW                  52.000 usec
DE                   6.50 usec
TE                  296.0 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.77 usec
SI                   65536
SF            600.1700056 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



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143.058
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129.691
128.031
127.817
127.590
127.240
125.756
80.861
80.334
75.397
73.554
55.329
55.123
52.641
52.603
52.524
52.407
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33.695
33.503
33.363
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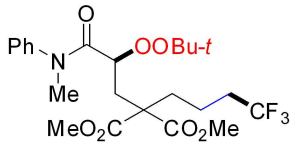
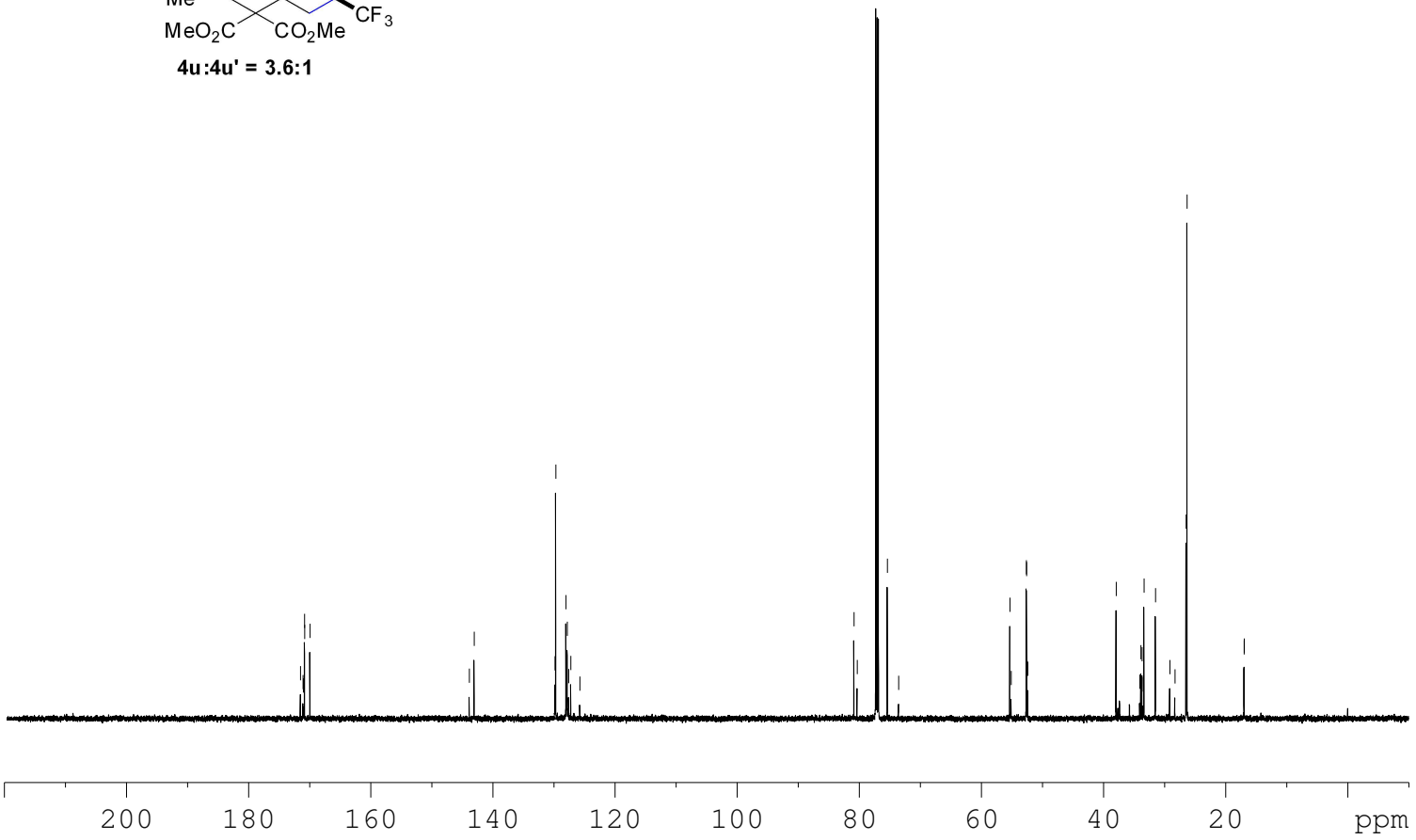
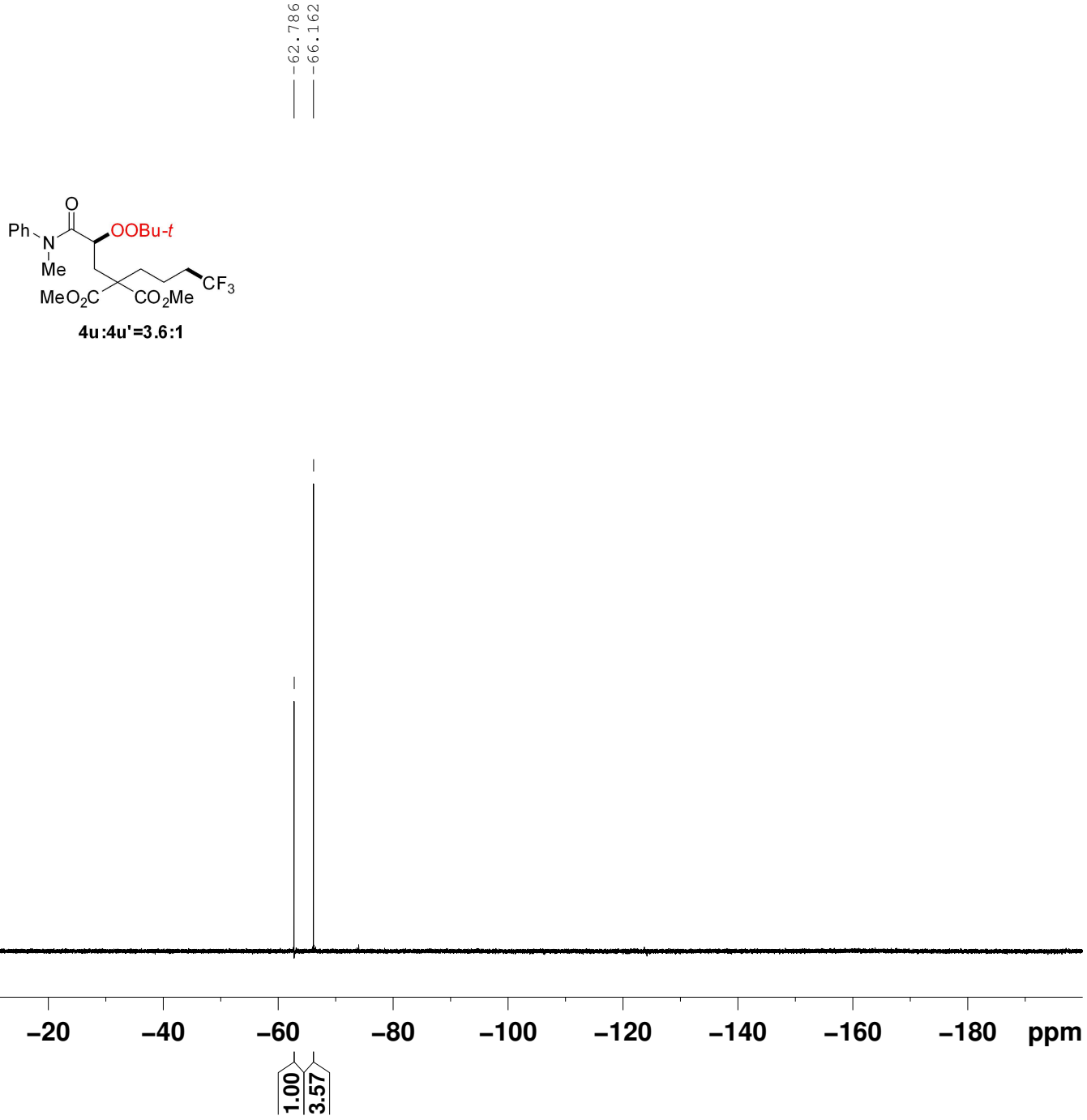
29.137
28.299
26.478
26.324



4u:4u' = 3.6:1

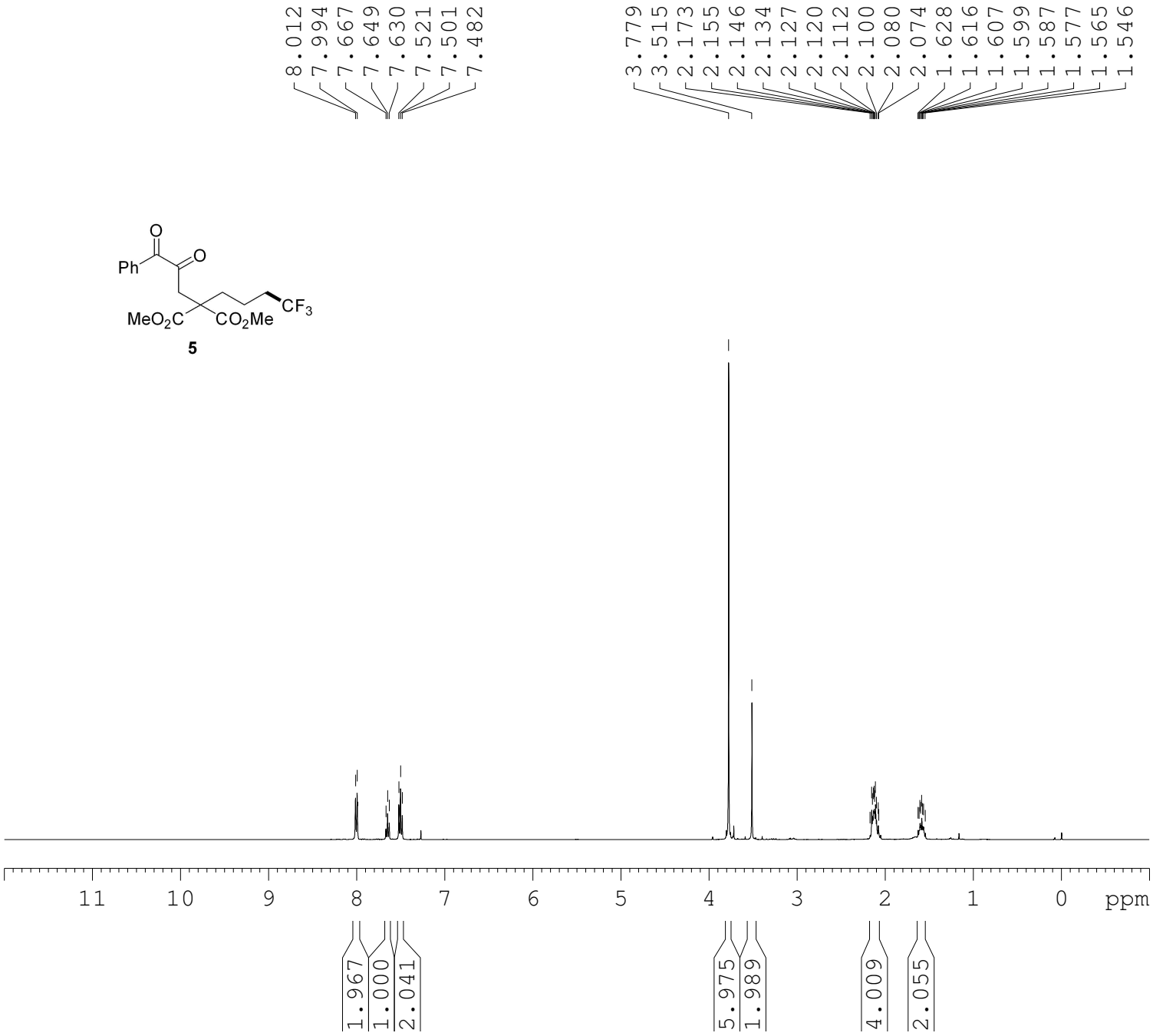


NAME	wll-519p-20201228
EXPNO	2
PROCNO	1
Date_	20201228
Time	18.57
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	300
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	297.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
========	CHANNEL f1 ========
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128665 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



BRUKER

NAME	wll-519p-20201228
EXPNO	3
PROCNO	1
Date_	20201228
Time	18.59
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgfhigqn.2
TD	131072
SOLVENT	CDCl3
NS	16
DS	4
SWH	133928.578 Hz
FIDRES	1.021794 Hz
AQ	0.4893855 sec
RG	15.49
DW	3.733 usec
DE	6.50 usec
TE	296.3 K
D1	1.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	564.6675534 MHz
NUC1	19F
P1	11.90 usec
SI	65536
SF	564.7240258 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	wll-3-25-4-20210327
EXPNO	1
PROCNO	1
Date_	20210327
Time	10.14
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	0
SWH	6393.862 Hz
FIDRES	0.195125 Hz
AQ	2.5625076 sec
RG	128
DW	78.200 usec
DE	6.50 usec
TE	295.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	10.40 usec
PL1	-1.00 dB
PL1W	17.01305389 W
SFO1	400.1326008 MHz
SI	32768
SF	400.1300061 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

198.677
190.672
170.609
134.669
131.656
130.454
128.804
128.098
125.351
77.337
77.020
76.702
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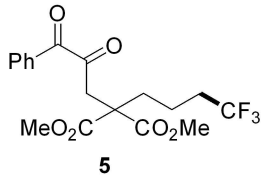
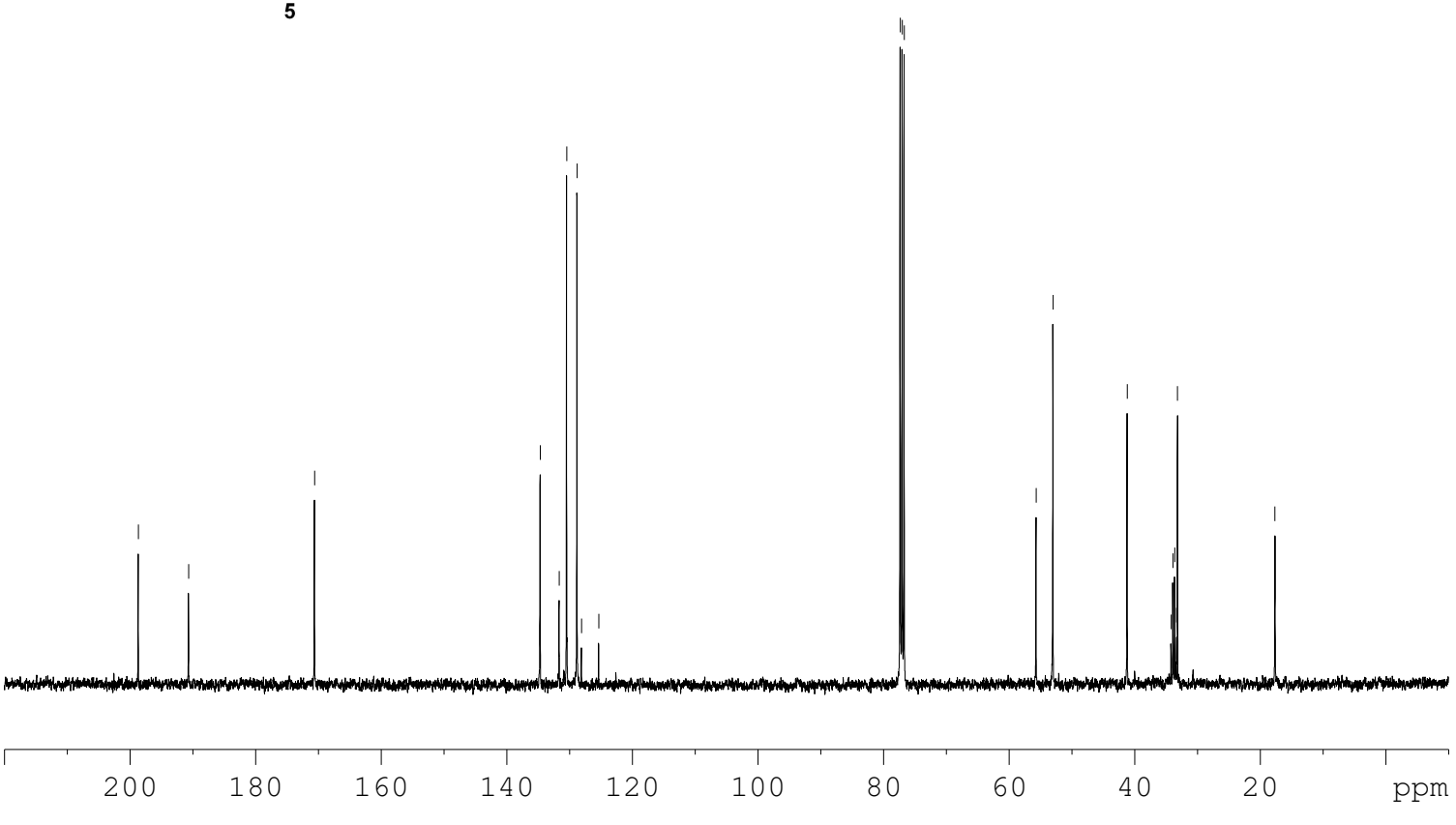
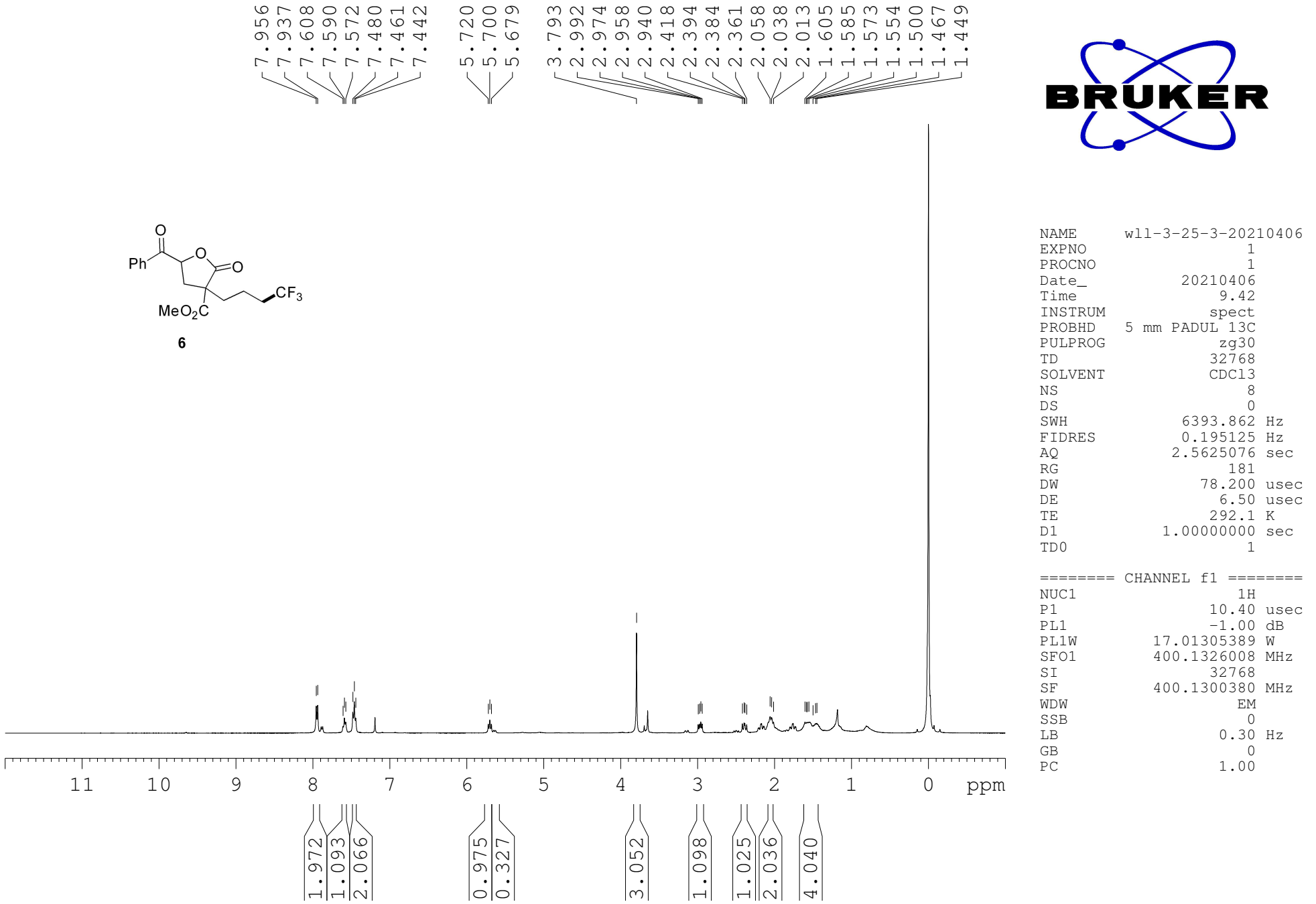
33.913
33.624
33.335
33.200
17.686



5

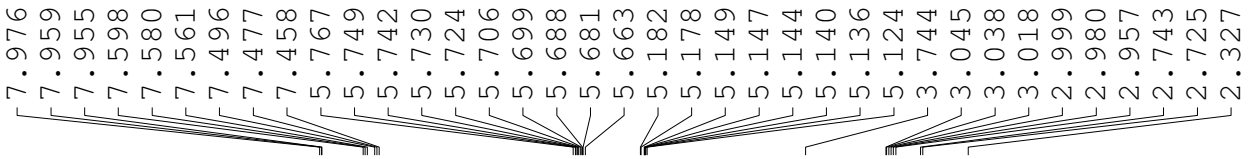


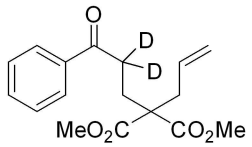
NAME	wll-3-25-4-20210327
EXPNO	2
PROCNO	1
Date_	20210327
Time	10.22
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	200
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	181
DW	19.800 usec
DE	6.50 usec
TE	296.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	3
======== CHANNEL f1 ========	
NUC1	13C
P1	15.00 usec
PL1	2.00 dB
PL1W	55.31277084 W
SFO1	100.6238364 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	16.72 dB
PL13	15.50 dB
PL2W	17.01305389 W
PL12W	0.28759566 W
PL13W	0.38087484 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127690 MHz



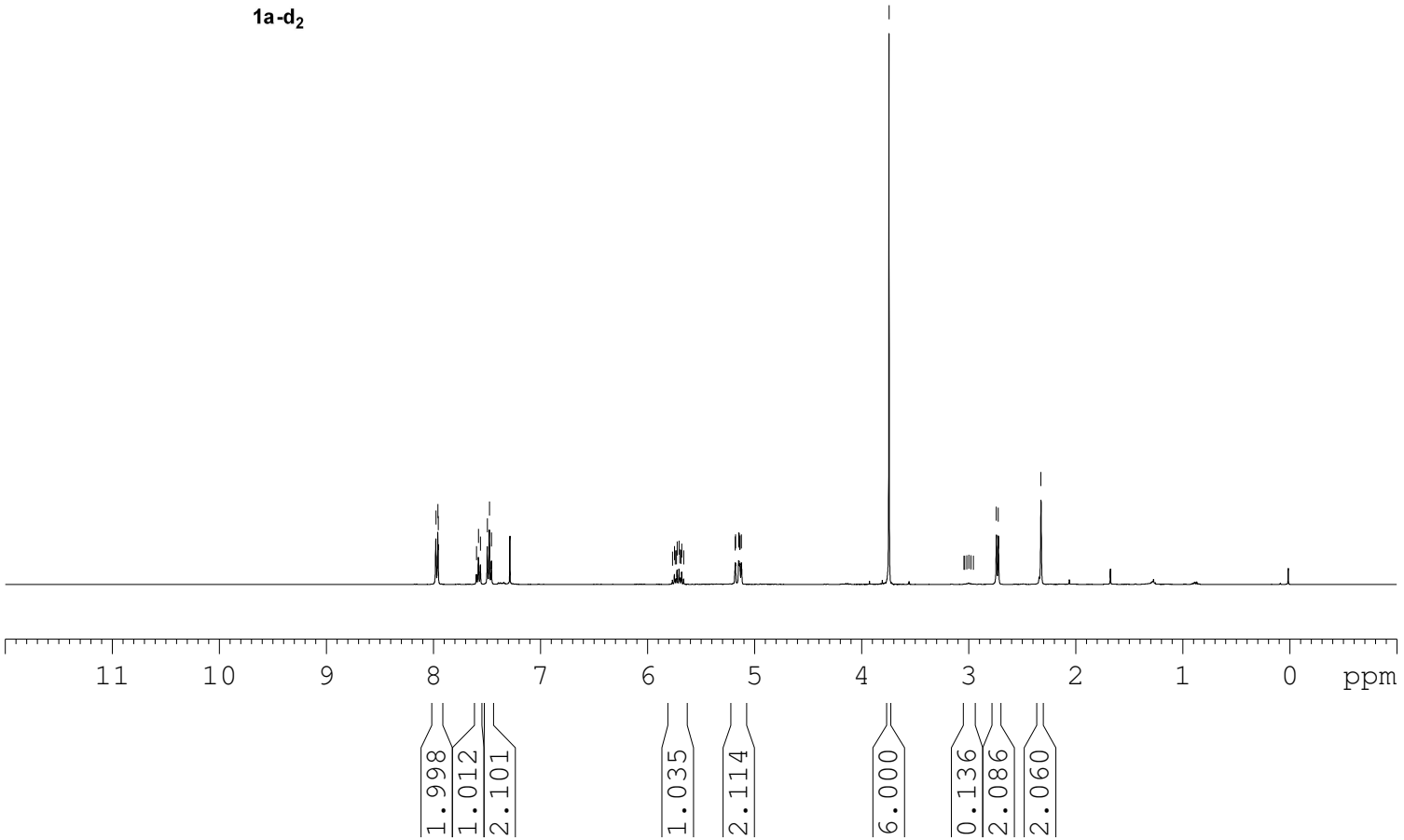
NAME	wll-3-25-3-20210406
EXPNO	1
PROCNO	1
Date_	20210406
Time	9.42
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	0
SWH	6393.862 Hz
FIDRES	0.195125 Hz
AQ	2.5625076 sec
RG	181
DW	78.200 usec
DE	6.50 usec
TE	292.1 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========	
NUC1	1H
P1	10.40 usec
PL1	-1.00 dB
PL1W	17.01305389 W
SFO1	400.1326008 MHz
SI	32768
SF	400.1300380 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

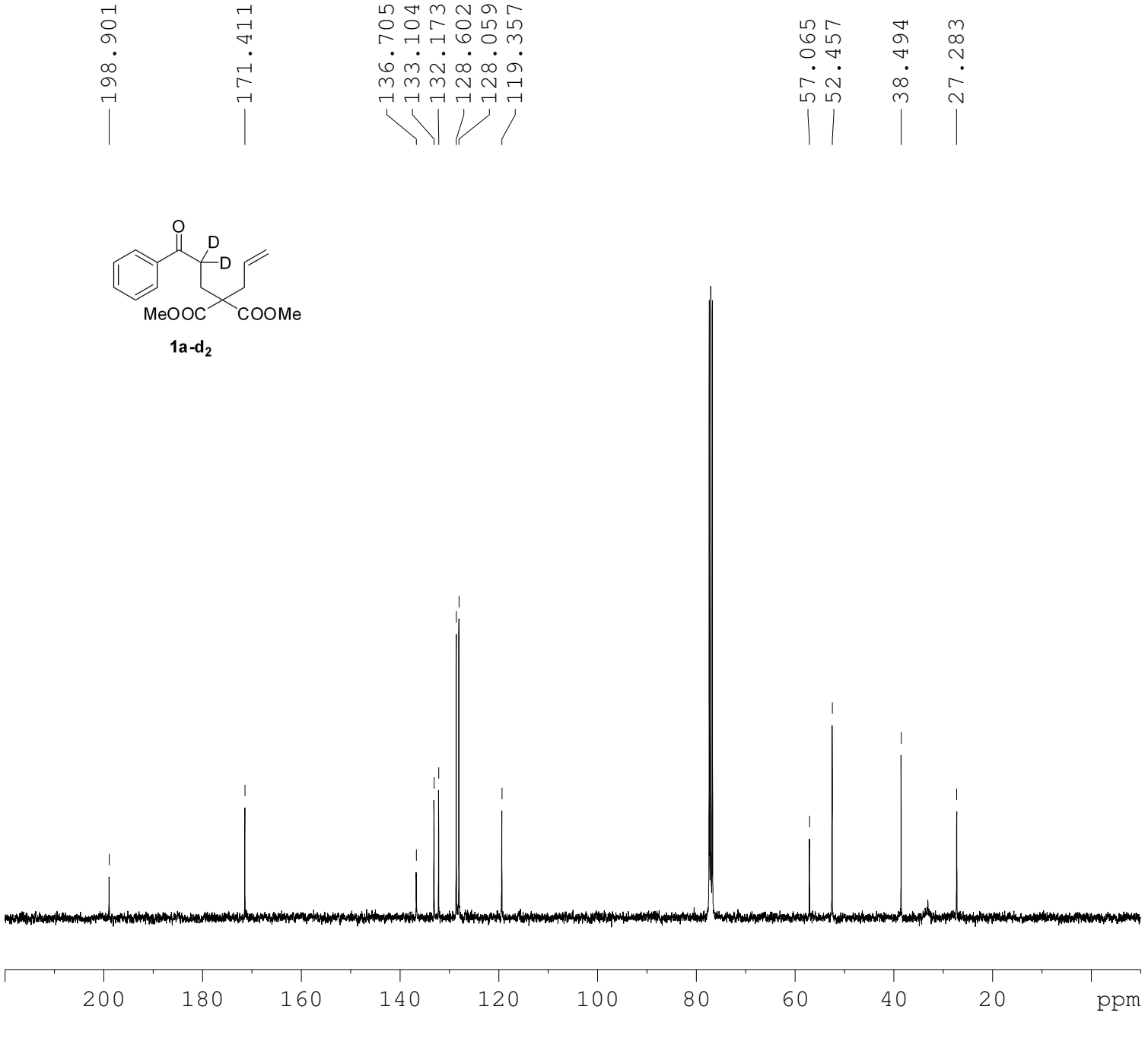




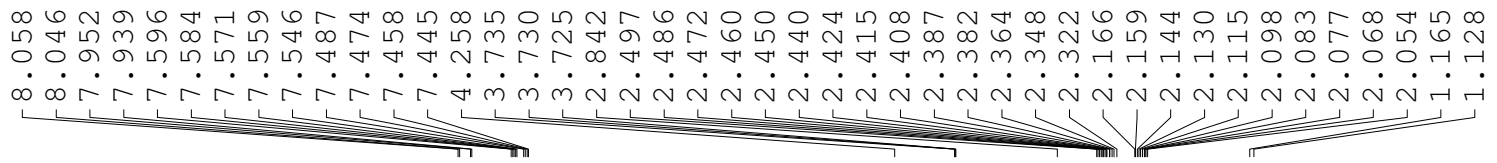
1a-d$_2$

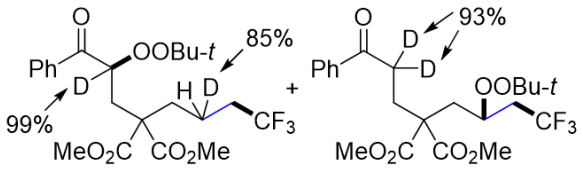



```
NAME      wll-3-22-1-20210323
EXPNO                       1
PROCNO                      1
Date_            20210323
Time                   15.14
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDC13
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       161
DW                    78.200 usec
DE                     6.50 usec
TE                    294.9 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     10.40 usec
PL1                    -1.00 dB
PL1W            17.01305389 W
SFO1            400.1326008 MHz
SI                     32768
SF              400.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

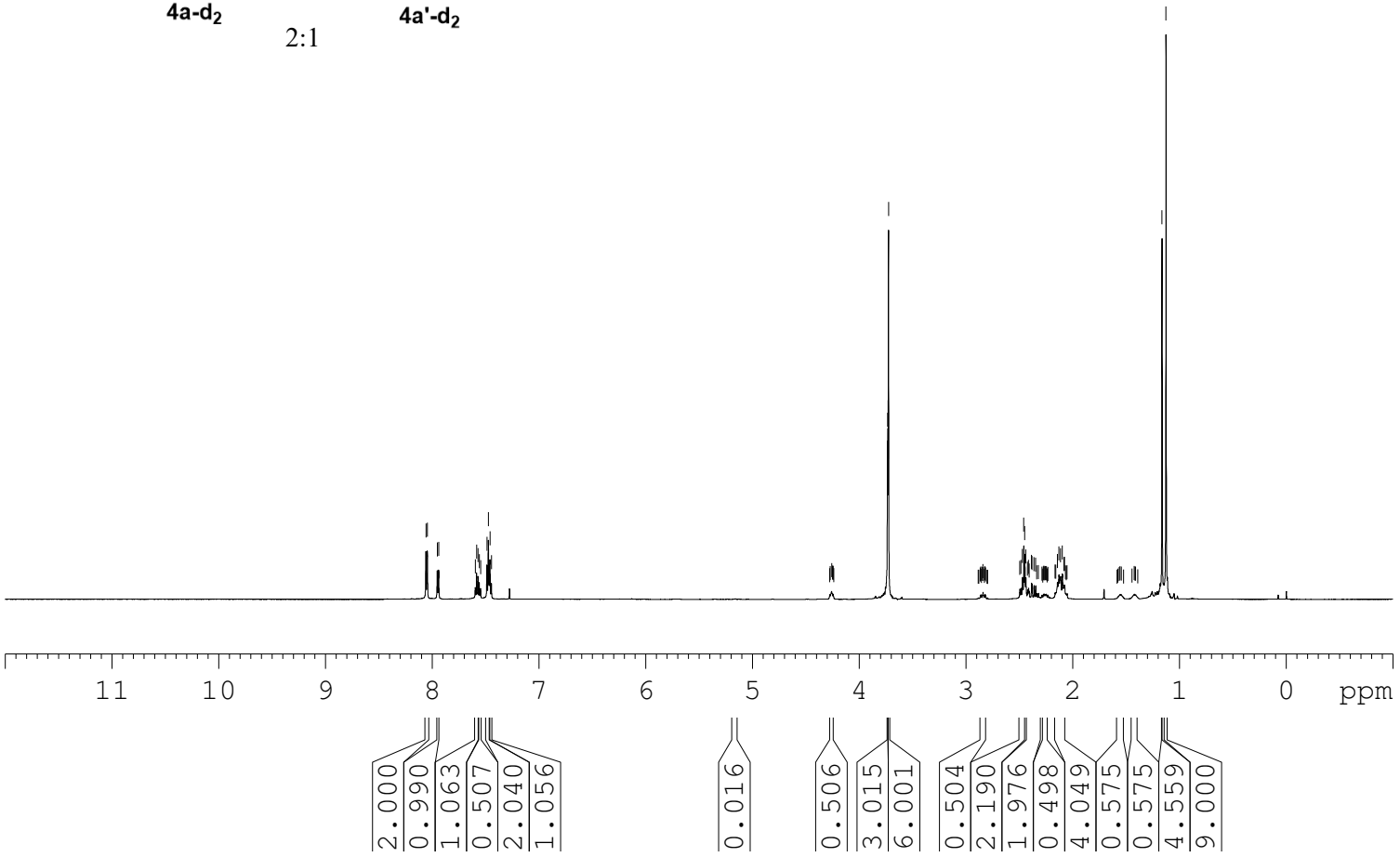



```
NAME      wll-3-22-1-20210323
EXPNO                       2
PROCNO                      1
Date_                20210323
Time                    15.20
INSTRUM                 spect
PROBHD   5 mm PADUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         150
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                         181
DW                      19.800 usec
DE                       6.50 usec
TE                      295.4 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          3

======== CHANNEL f1 ========
NUC1                       13C
P1                      15.00 usec
PL1                      2.00 dB
PL1W               55.31277084 W
SFO1              100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                   80.00 usec
PL2                     -1.00 dB
PL12                    16.72 dB
PL13                    15.50 dB
PL2W               17.01305389 W
PL12W               0.28759566 W
PL13W               0.38087484 W
SFO2              400.1316005 MHz
SI                       32768
```

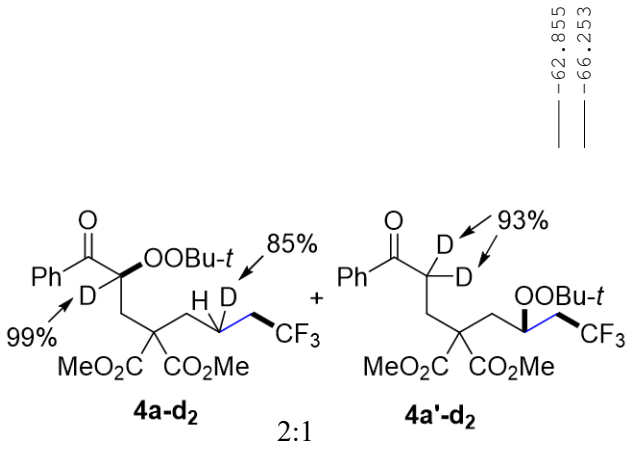




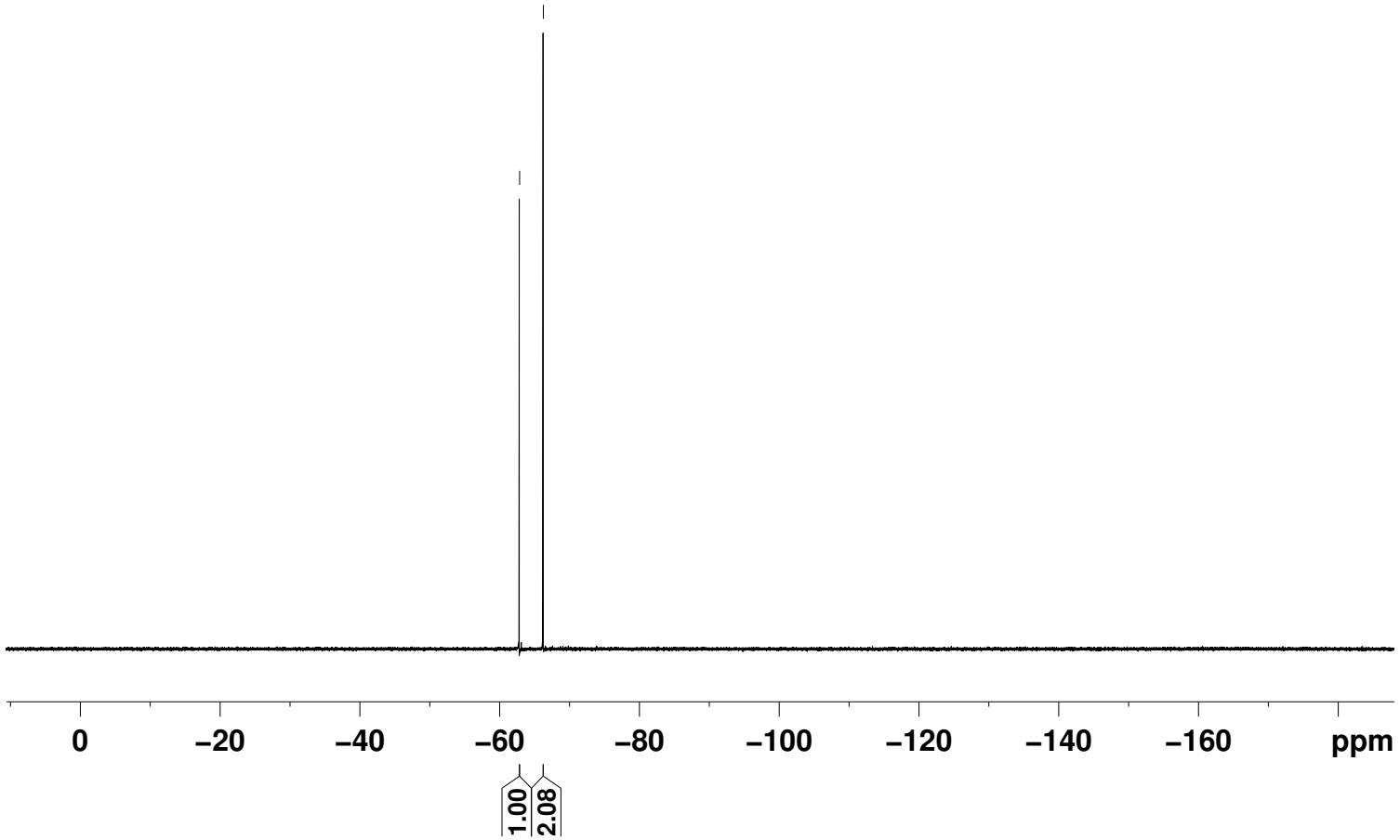
4a-d$_2$ 2:1 **4a'-d$_2$**




```
NAME      wll-3-23-1-20210324
EXPNO                   1
PROCNO                  1
Date_            20210324
Time                10.59
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH              9615.385 Hz
FIDRES           0.146719 Hz
AQ               3.4079220 sec
RG                  30.73
DW                 52.000 usec
DE                  6.50 usec
TE                 296.3 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         600.1739011 MHz
NUC1                   1H
P1                  9.77 usec
SI                 65536
SF           600.1700070 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



2:1




```
NAME      wll-3-23-1-20210324
EXPNO                       3
PROCNO                      1
Date_                20210324
Time                    11.22
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG          zgfhigqn.2
TD                     131072
SOLVENT                CDCl3
NS                        16
DS                         4
SWH                133928.578 Hz
FIDRES               1.021794 Hz
AQ                  0.4893855 sec
RG                       17.32
DW                       3.733 usec
DE                        6.50 usec
TE                       296.8 K
D1                  1.00000000 sec
D11                 0.03000000 sec
D12                 0.00002000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               564.6675534 MHz
NUC1                       19F
P1                       11.90 usec
SI                       65536
SF                 564.7240258 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```