Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Concomitant Functionalization of Two Different Ketones by Merging Brønsted Acid Catalysis and Radical Relay Coupling

Leilei Wang, Leiyang Lv*, Zhiping Li*

Key Laboratory of Advanced Light Conversion Materials and Biophotonics, Department of Chemistry, Renmin University of China, Beijing 100872, China

Email: lvleiyang2008@ruc.edu.cn, zhipingli@ruc.edu.cn

Contents

1. General information

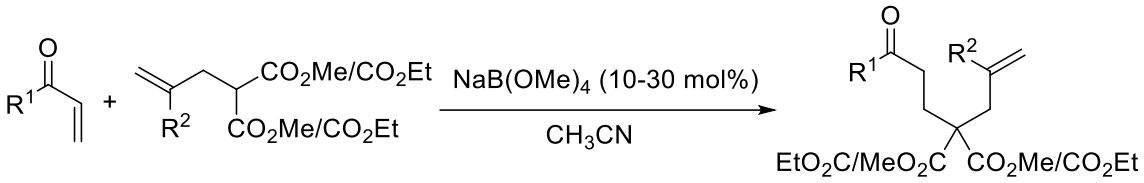
^{1}H NMR spectra were recorded on Bruker 400 MHz and 600 MHz spectrometer and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm) for $CDCl_3$. The peak patterns are indicated as follows: s, singlet; d, doublet; dd, doublet of doublet; t, triplet; m, 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 $CDCl_3$). $CDCl_3$ was used as the NMR solvent. APEX II (Bruker Inc.) was used for ESI-MS and EI-MS. 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, 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 start materials 1

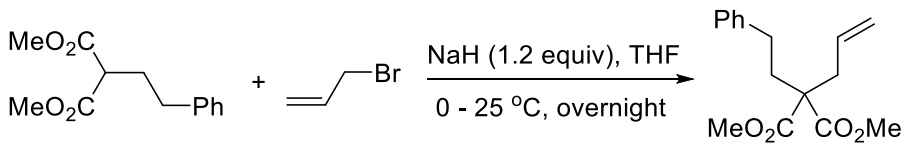
All the unactivated alkene substrate **1** was known compounds. Unactivated alkenes **1a-1t**[1, 2] and **1u**[3] were prepared as followed procedure.

(a) General procedure for the synthesis of **1a**-**1t**.



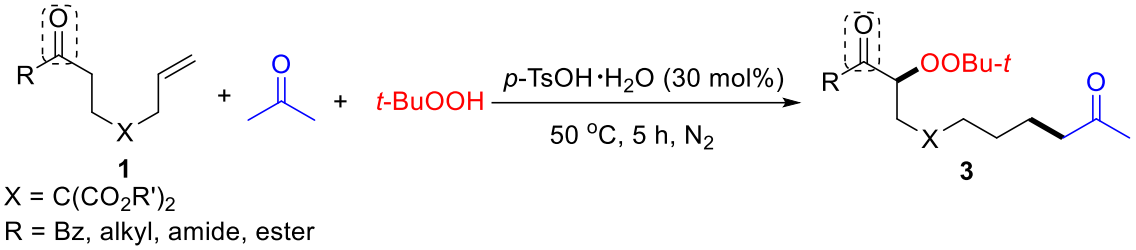
To a 25 mL round bottom flask with a magnetic stir bar a solution of Michael donor (1.0 mmol) and $NaB(OMe)_4$ (0.10-0.30 mmol, 10-30 mol %) in MeCN (3.0 mL) was added *α,β*-unsaturated ketone (1.0-2.0 mmol) at room temperature. The resulting solution was stirred at room temperature or 50 ^oC under air 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 in good yield.

(b) General procedure for the synthesis of **1u**.

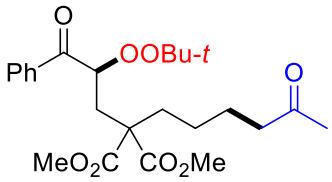


To a stirred suspension of NaH (60% in mineral oil, 2.4 mmol.) in dry THF (6.0 ml) at 0 ^oC was added a solution of dimethyl 2-phenethylmalonate (2.0 mmol) in THF (2.0 mL). The reaction mixture was kept for 0.5 h, and then a solution of allyl bromide (2.2 mmol) in THF (2.0 mL) was added dropwise, and then allowed to stir at room temperature for 12 h. Quenched with saturated NH_4Cl, extracted with ethyl acetate, and washed with brine. The organic phase was dried over anhydrous Na_2SO_4 and concentrated. The residue was purified by column chromatography on silica gel (EtOAc/petroleum ether = 1:15) to give disubstituted product **1u** as a colorless oil.

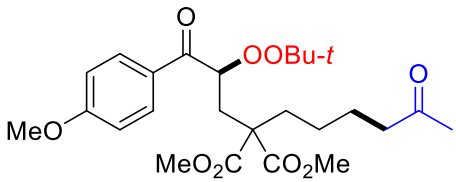
3. General procedure for synthesis of products 3



To a 10 mL sealable pressure tube was added unactivated alkenes **1** (0.1 mmol), *p*-TsOH·H_2O (0.03 mmol), acetone (0.6 mL) and *t*-BuOOH (5.5 M in decane, 0.5 mmol) under N_2 atmosphere at room temperature, and the resulting solution was stirred at 50 ℃ for 5 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 **3a**-**3u**.

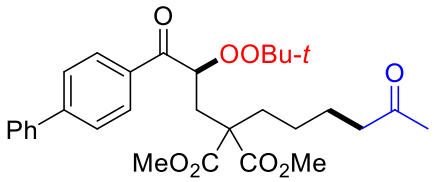


Dimethyl 2-(2-(*tert*-butylperoxy)-3-oxo-3-phenylpropyl)-2-(5-oxohexyl)malonate (3a): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.4) in 64% yield (28.7 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.07 (d, J = 7.2 Hz, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.8 Hz, 2H), 5.16 (dd, J = 7.0, 5.5 Hz, 1H), 3.72 (s, 3H), 3.71 (s, 3H), 2.45-2.43 (m, 4H), 2.13 (s, 3H), 2.12-2.06 (m, 1H), 2.04-1.99 (m, 1H), 1.63-1.58 (m, 2H), 1.27-1.19 (m, 2H), 1.13 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.5, 197.9, 171.3, 171.2, 135.2, 133.3, 129.0, 128.5, 81.6, 80.8, 55.9, 52.7, 52.6, 43.2, 33.2, 32.8, 29.9, 26.4, 23.8; HRMS (ESI) calcd for $C_{24}H_{34}O_8Na$ $(M+Na)^+$: 473.2146; found: 473.2141.

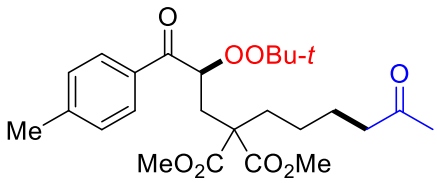


Dimethyl 2-(2-(*tert*-butylperoxy)-3-(4-methoxyphenyl)-3-oxopropyl)-2-(5-oxohexyl)malonate (3b): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.35) in 63% yield (30.2 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.97 (d, J = 8.2 Hz, 2H), 7.27 (d,

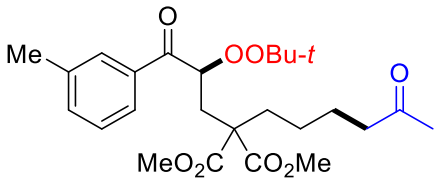
J = 8.6 Hz, 2H), 5.15 (t, J = 6.2 Hz, 1H), 3.72 (s, 3H), 3.71 (s, 3H), 2.45-2.41 (m, 7H), 2.13 (s, 3H), 2.11-2.05 (m, 1H), 2.03-1.98 (m, 1H), 1.63-1.58 (m, 2H), 1.26-1.18 (m, 2H), 1.14 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.6, 196.2, 171.4, 171.3, 163.7, 131.4, 128.1, 113.7, 81.6, 80.8, 55.9, 55.5, 52.7, 52.6, 43.2, 33.4, 32.8, 29.9, 26.4, 23.8; HRMS (ESI) calcd for $C_{25}H_{36}O_9Na$ $(M+Na)^+$: 503.2252; found: 503.2254.



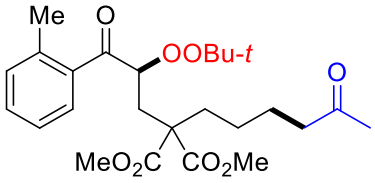
Dimethyl 2-(3-([1,1'-biphenyl]-4-yl)-2-(*tert*-butylperoxy)-3-oxopropyl)-2-(5-oxohexyl)malonate (3c): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.4) in 63% yield (32.9 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.16 (d, J = 8.4 Hz, 2H), 7.70 (d, J = 8.4 Hz, 2H), 7.65 (d, J = 7.3 Hz, 2H), 7.48 (t, J = 7.4 Hz, 2H), 7.41 (t, J = 7.4 Hz, 1H), 5.19 (t, J = 6.5 Hz, 1H), 3.73 (s, 3H), 3.72 (s, 3H), 2.47-2.44 (m, 4H), 2.13 (s, 3H), 2.10-2.08 (m, 1H), 2.06-2.01 (m, 1H), 1.64-1.59 (m, 2H), 1.29-1.19 (m, 2H), 1.15 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.5, 197.4, 171.4, 171.3, 145.9, 139.9, 133.8, 129.7, 129.0, 128.3, 127.3, 127.1, 81.8, 80.9, 55.9, 52.7, 52.6, 43.2, 33.3, 32.8, 30.0, 26.4, 23.8; HRMS (ESI) calcd for $C_{30}H_{38}O_8Na$ $(M+Na)^+$: 549.2459; found: 549.2455.



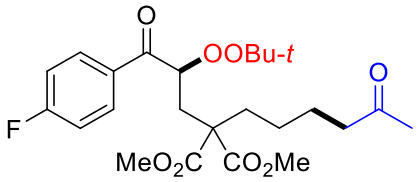
Dimethyl 2-(2-(*tert*-butylperoxy)-3-oxo-3-(p-tolyl)propyl)-2-(5-oxohexyl)malonate (3d): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.4) in 65% yield (30.1 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.97 (d, J = 8.2 Hz, 2H), 7.27 (d, J = 8.6 Hz, 2H), 5.15 (t, J = 6.2 Hz, 1H), 3.72 (s, 3H), 3.71 (s, 3H), 2.45-2.41 (m, 7H), 2.13 (s, 3H), 2.11-2.05 (m, 1H), 2.03-1.98 (m, 1H), 1.63-1.58 (m, 2H), 1.26-1.18 (m, 2H), 1.14 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.6, 197.4, 171.3, 171.2, 144.2, 132.6, 129.2, 129.1, 81.5, 80.8, 55.9, 52.6, 52.5, 43.2, 33.3, 32.8, 29.9, 26.4, 23.8, 23.7, 21.7; HRMS (ESI) calcd for $C_{25}H_{36}O_8Na$ $(M+Na)^+$: 487.2302; found: 487.2305.



Dimethyl 2-(2-(*tert*-butylperoxy)-3-oxo-3-(m-tolyl)propyl)-2-(5-oxohexyl)malonate (3e): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.4) in 61% yield (28.2 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.86 (d, J = 6.9 Hz, 2H), 7.40-7.34 (m, 2H) 5.15 (t, J = 6.3 Hz, 1H), 3.72 (s, 3H), 3.71 (s, 3H), 2.45-2.42 (m, 7H), 2.13 (s, 3H), 2.12-2.07 (m, 1H), 2.04-1.99 (m, 1H), 1.63-1.58 (m, 2H), 1.27-1.21 (m, 2H), 1.14 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.5, 198.0, 171.3, 171.2, 138.2, 135.2, 134.1, 129.5, 128.3, 126.2, 81.5, 80.8, 55.9, 52.6, 52.5, 43.2, 33.2, 32.7, 29.9, 26.4, 23.8, 21.4; HRMS (ESI) calcd for $C_{25}H_{36}O_8Na$ $(M+Na)^+$: 487.2302; found: 487.2304.

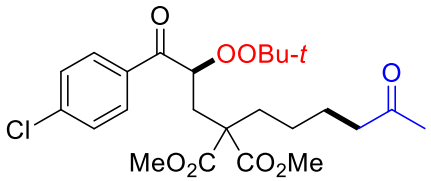


Dimethyl 2-(2-(*tert*-butylperoxy)-3-oxo-3-(o-tolyl)propyl)-2-(5-oxohexyl)malonate (3f): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.4) in 64% yield (29.5 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.67 (d, J = 7.2 Hz, 1H), 7.38-7.36 (m, 1H), 7.27-7.24 (m, 2H), 5.03 (dd, J = 9.0, 2.9 Hz, 1H), 3.71 (s, 3H), 3.66 (s, 3H), 2.46 (s, 3H), 2.44-2.33 (m, 4H), 2.12 (s, 3H), 2.09-2.04 (m, 1H), 2.02-1.97 (m, 1H), 1.61-1.55 (m, 2H), 1.24-1.20 (m, 1H), 1.09 (s, 9H), 1.08-1.03 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.5, 202.1, 171.3, 171.2, 138.6, 136.4, 131.7, 131.2, 128.4, 125.3, 82.0, 80.6, 56.0, 52.6, 52.5, 43.2, 32.6, 32.5, 29.9, 26.3, 23.8, 23.7, 20.7; HRMS (ESI) calcd for $C_{25}H_{36}O_8Na$ $(M+Na)^+$: 487.2302; found: 487.2303.

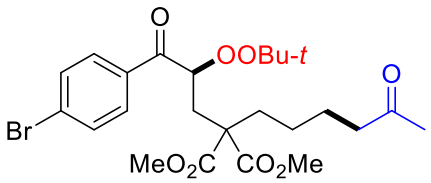


Dimethyl 2-(2-(*tert*-butylperoxy)-3-(4-fluorophenyl)-3-oxopropyl)-2-(5-oxohexyl)malonate (3g): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.4) in 63% yield

(29.3 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.15-8.13 (m, 2H), 7.15 (t, J = 8.5 Hz, 2H), 5.09 (t, J = 6.5 Hz, 1H), 3.73 (s, 3H), 3.72 (s, 3H), 2.46-2.42 (m, 4H), 2.14 (s, 3H), 2.11-2.05 (m, 1H), 2.03-1.98 (m, 1H), 1.63-1.58 (m, 2H), 1.26-1.19 (m, 2H), 1.13 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.5, 196.4, 171.3, 165.8 (d, J = 253.6 Hz), 131.8 (d, J = 9.1 Hz), 131.5 (d, J = 2.9 Hz), 115.6 (d, J = 21.6 Hz), 82.0, 80.9, 55.9, 52.7, 52.6, 43.2, 33.2, 32.8, 29.9, 26.4, 23.7; ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -104.62 (s, 1F); HRMS (ESI) calcd for $C_{24}H_{33}FO_8Na$ $(M+Na)^+$: 491.2052; found: 491.2054.

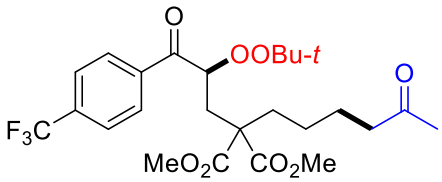


Dimethyl 2-(2-(*tert*-butylperoxy)-3-(4-chlorophenyl)-3-oxopropyl)-2-(5-oxohexyl)malonate (3h): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.4) in 61% yield (29.5 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.04 (d, J = 8.5 Hz, 2H), 7.45 (d, J = 8.5 Hz, 2H), 5.08 (dd, J = 7.2, 5.4 Hz, 1H), 3.72 (s, 3H), 3.72 (s, 3H), 2.46-2.41 (m, 4H), 2.14 (s, 3H), 2.10-2.05 (m, 1H), 2.03-1.98 (m, 1H), 1.63-1.58 (m, 2H), 1.25-1.18 (m, 2H), 1.12 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.5, 196.8, 171.2, 139.7, 133.4, 130.6, 128.8, 82.1, 81.0, 55.8, 52.7, 52.6, 43.2, 33.2, 32.8, 29.9, 26.4, 23.7; HRMS (ESI) calcd for $C_{24}H_{33}ClO_8Na$ $(M+Na)^+$: 507.1756; found: 507.1759.

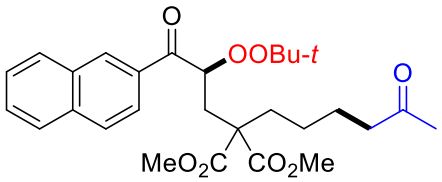


Dimethyl 2-(3-(4-bromophenyl)-2-(*tert*-butylperoxy)-3-oxopropyl)-2-(5-oxohexyl)malonate (3i): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 5:1, R_f = 0.4) in 58% yield (30.4 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.97 (d, J = 8.6 Hz, 2H), 7.62 (d, J = 8.6 Hz, 2H), 5.07 (dd, J = 7.4, 5.3 Hz, 1H), 3.72 (s, 3H), 3.72 (s, 3H), 2.46-2.41 (m, 4H), 2.14 (s, 3H), 2.10-2.05 (m, 1H), 2.03-1.97 (m, 1H), 1.63-1.58 (m, 2H), 1.26-1.18 (m, 2H), 1.12 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.5, 197.0, 171.2, 133.8, 131.8, 130.7, 128.5, 82.1, 81.0, 55.8, 52.7, 52.6, 43.2, 33.2, 32.8, 30.0, 26.4, 23.7; HRMS (ESI) calcd for $C_{24}H_{33}BrO_8Na$ $(M+Na)^+$:

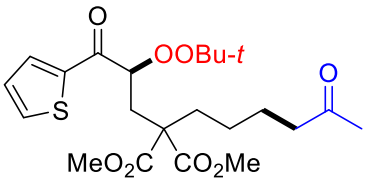
551.1251; found: 551.1254.



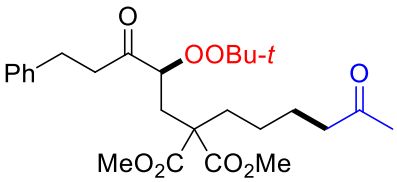
Dimethyl 2-(2-(*tert*-butylperoxy)-3-oxo-3-(4-(trifluoromethyl)phenyl)propyl)-2-(5-oxohexyl) malonate (3j): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.4) in 59% yield (30.7 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.21 (d, J = 8.2 Hz, 2H), 7.74 (d, J = 8.2 Hz, 2H), 5.11 (dd, J = 8.1, 4.4 Hz, 1H), 3.73 (s, 3H), 3.72 (s, 3H), 2.47-2.40 (m, 4H), 2.14 (s, 3H), 2.11-2.05 (m, 1H), 2.04-1.99 (m, 1H), 1.64-1.59 (m, 2H), 1.26-1.20 (m, 2H), 1.12 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.4, 197.2, 171.2, 137.9, 134.4 (q, J = 32.6 Hz), 129.4, 125.5 (q, J = 3.5 Hz), 123.6 (q, J = 270.6 Hz), 82.3, 81.1, 55.8, 52.7, 52.6, 43.1, 33.1, 32.8, 29.9, 26.3, 23.8, 23.7; ^{19}F NMR (564 MHz, $CDCl_3$, ppm) δ -63.18 (s, 3F); HRMS (ESI) calcd for $C_{25}H_{33}F_3O_8Na$ $(M+Na)^+$: 541.2020; found: 541.2022.



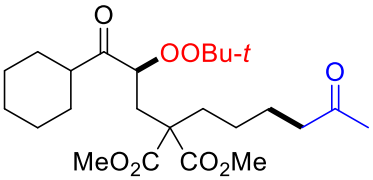
Dimethyl 2-(2-(*tert*-butylperoxy)-3-(naphthalen-2-yl)-3-oxopropyl)-2-(5-oxohexyl)malonate (3k): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.4) in 30% yield (14.9 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.68 (s, 1H), 8.10-8.08 (m, 1H), 7.99 (d, J = 8.1 Hz, 1H), 7.91-7.88 (m, 2H), 7.63-7.60 (m, 1H), 7.57-7.55 (m, 1H), 5.29 (dd, J = 7.2, 5.4 Hz, 1H), 3.74 (s, 3H), 3.72 (s, 3H), 2.54-2.48 (m, 2H), 2.44 (t, J = 7.4 Hz, 2H), 2.16-2.09 (m, 4H), 2.07-2.02 (m, 1H), 1.64-1.59 (m, 2H), 1.27-1.22 (m, 2H), 1.14 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.5, 197.8, 171.4, 171.3, 135.7, 132.5, 132.4, 131.0, 129.8, 128.6, 128.3, 127.8, 126.7, 124.6, 81.7, 80.9, 56.0, 52.7, 52.6, 43.2, 33.4, 32.8, 29.9, 26.4, 23.8; HRMS (ESI) calcd for $C_{28}H_{36}O_8Na$ $(M+Na)^+$: 523.2302; found: 523.2304.



Dimethyl 2-(2-(tert-butylperoxy)-3-oxo-3-(thiophen-2-yl)propyl)-2-(5-oxohexyl)malonate (3l): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.5) in 32% yield (14.7 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.01-8.00 (m, 1H), 7.69-7.68 (m, 1H), 7.17-7.15 (m, 1H), 4.88 (dd, J = 9.4, 3.6 Hz, 1H), 3.75 (s, 3H), 3.72 (s, 3H), 2.49-2.39 (m, 4H), 2.14 (s, 3H), 2.13-2.08 (m, 1H), 2.03-1.98 (m, 1H), 1.62-1.59 (m, 2H), 1.28-1.20 (m, 2H), 1.18 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.6, 191.4, 171.2, 171.1, 140.7, 134.2, 133.7, 128.0, 83.3, 81.2, 55.8, 52.7, 52.6, 43.3, 33.8, 32.4, 29.9, 26.4, 23.8, 23.7; HRMS (ESI) calcd for $C_{22}H_{32}O_8Na$ $(M+Na)^+$: 479.1710; found: 479.1708.

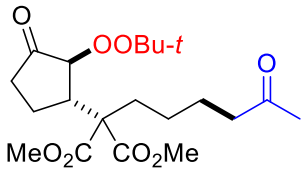


Dimethyl 2-(2-(tert-butylperoxy)-3-oxo-5-phenylpentyl)-2-(5-oxohexyl)malonate (3m): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.4) in 54% yield (25.8 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.28 (t, J = 7.6 Hz, 2H), 7.22-7.18 (m, 3H), 4.25 (dd, J = 7.2, 5.8 Hz, 1H), 3.72 (s, 3H), 3.71 (s, 3H), 3.11-3.04 (m, 1H), 2.95-2.87 (m, 3H), 2.44 (t, J = 7.3 Hz, 2H), 2.15 (d, J = 5.8 Hz, 2H), 2.13 (s, 3H), 2.06-2.00 (m, 1H), 1.96-1.91 (m, 1H), 1.61-1.56 (m, 2H), 1.19 (s, 9H), 1.15-1.11 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 210.2, 208.5, 171.1, 171.0, 141.3, 128.4, 128.3, 126.0, 84.6, 81.1, 55.6, 52.8, 52.6, 43.2, 38.7, 32.5, 32.1, 29.9, 29.2, 26.4, 23.8, 23.6; HRMS (ESI) calcd for $C_{26}H_{38}O_8Na$ $(M+Na)^+$: 501.2459; found: 501.2456.

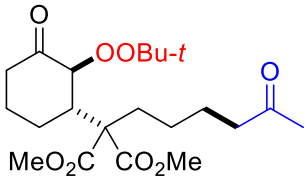


Dimethyl 2-(2-(tert-butylperoxy)-3-cyclohexyl-3-oxopropyl)-2-(5-oxohexyl)malonate (3n):

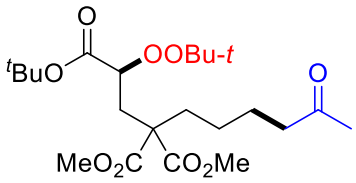
Isolated by flash column chromatography (ethyl acetate/petroleum ether = 4:1, R_f = 0.3) in 56% yield (25.4 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 4.34 (dd, J = 8.4, 4.1 Hz, 1H), 3.74 (s, 3H), 3.72 (s, 3H), 2.88-2.84 (m, 1H), 2.44 (t, J = 7.4 Hz, 2H), 2.24-2.18 (m, 2H), 2.13 (s, 3H), 2.06-2.01 (m, 1H), 1.97-1.92 (m, 1H), 1.85-1.78 (m, 4H), 1.62-1.57 (m, 2H), 1.41-1.25 (m, 6H), 1.20 (s, 9H), 1.18-1.09 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 213.6, 208.5, 171.3, 171.2, 83.3, 80.8, 55.8, 52.7, 52.6, 45.7, 43.2, 32.5, 29.9, 29.4, 28.2, 26.4, 25.8, 25.5, 23.8, 23.7; HRMS (ESI) calcd for $C_{24}H_{40}O_8Na$ $(M+Na)^+$: 479.2615; found: 479.2613.



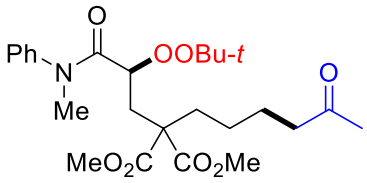
Dimethyl 2-((2R)-2-(tert-butylperoxy)-3-oxocyclopentyl)-2-(5-oxohexyl)malonate (3o): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 5:1, R_f = 0.4) in 41% yield (16.3 mg); Colorless oil (dr > 20:1); ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 4.26 (d, J = 7.9 Hz, 1H), 3.74 (s, 3H), 3.72 (s, 3H), 3.10-3.06 (m, 1H), 2.44 (t, J = 7.3 Hz, 2H), 2.30-2.26 (m, 3H), 2.13 (s, 3H), 2.07-1.97 (m, 2H), 1.66-1.50 (m, 5H), 1.21 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 212.5, 208.6, 171.1, 171.0, 84.9, 80.6, 60.0, 52.3, 52.2, 43.3, 42.4, 36.5, 34.4, 29.9, 26.4, 24.3, 23.9, 21.0; HRMS (ESI) calcd for $C_{20}H_{32}O_8Na$ $(M+Na)^+$: 423.1989; found: 499.1988.



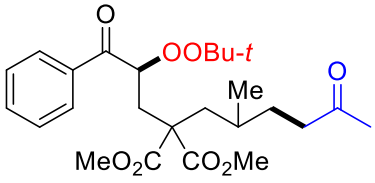
Dimethyl 2-((1S)-2-(tert-butylperoxy)-3-oxocyclohexyl)-2-(5-oxohexyl)malonate (3p): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 5:1, R_f = 0.4) in 46% yield (18.9 mg); Colorless oil (dr > 20:1); ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 4.27 (d, J = 4.6 Hz, 1H), 3.65 (s, 3H), 3.63 (s, 3H), 2.55-2.51 (m, 1H), 2.45-2.42 (m, 1H), 2.37 (t, J = 7.3 Hz, 2H), 2.17-2.12 (m, 1H), 2.06 (s, 3H), 1.90-1.78 (m, 5H), 1.52-1.47 (m, 2H), 1.34-1.29 (m, 1H), 1.28-1.20 (m, 2H), 1.17 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.6, 207.5, 170.7, 170.6, 86.3, 80.9, 60.2, 52.3, 52.0, 45.9, 43.2, 38.0, 34.3, 29.9, 26.4, 24.4, 24.1, 23.9, 21.6; HRMS (ESI) calcd for $C_{21}H_{34}O_8Na$ $(M+Na)^+$: 437.2146; found: 437.2143.



1-(tert-butyl) 3,3-dimethyl 1-(tert-butylperoxy)-8-oxononane-1,3,3-tricarboxylate (3q): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 5:1, R_f = 0.35) in 57% yield (25.3 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 4.23 (t, J = 6.3 Hz, 1H), 3.73 (s, 6H), 2.45 (t, J = 7.4 Hz, 2H), 2.29 (d, J = 6.4 Hz, 2H), 2.13 (s, 3H), 2.07-2.01 (m, 1H), 1.98-1.93 (m, 1H), 1.62-1.57 (m, 2H), 1.48 (s, 9H), 1.44 (d, J = 4.6 Hz, 2H), 1.21 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.5, 171.3, 171.2, 170.4, 81.5, 80.6, 79.2, 55.7, 52.7, 52.5, 43.3, 33.0, 32.1, 29.9, 28.0, 26.4, 23.8, 23.6; HRMS (ESI) calcd for $C_{22}H_{38}O_9Na$ $(M+Na)^+$: 469.2408; found: 469.2405.

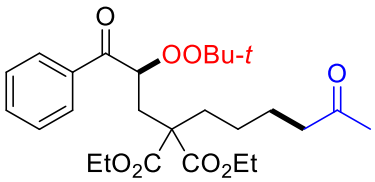


Dimethyl 2-(2-(tert-butylperoxy)-3-(methyl(phenyl)amino)-3-oxopropyl)-2-(5-oxohexyl) malonate (3r): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.3) in 33% yield (16.0 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.43 (t, J = 7.6 Hz, 2H), 7.35 (t, J = 7.4 Hz, 1H), 7.26 (d, J = 7.4 Hz, 2H), 4.47 (dd, J = 10.5, 2.8 Hz, 1H), 3.66 (s, 3H), 3.55 (s, 3H), 3.30 (s, 3H), 2.47-2.43 (m, 1H), 2.30 (t, J = 7.5 Hz, 2H), 2.18-2.15 (m, 1H), 2.11 (s, 3H), 1.89-1.84 (m, 1H), 1.62-1.57 (m, 1H), 1.39-1.34 (m, 2H), 1.17 (s, 9H), 1.04-0.99 (m, 1H), 0.80-0.73 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.5, 171.2, 170.2, 143.1, 129.6, 128.0, 127.9, 80.9, 75.6, 55.4, 52.5, 52.4, 43.2, 38.0, 33.4, 32.1, 29.9, 26.4, 23.6, 23.5; HRMS (ESI) calcd for $C_{25}H_{38}NO_8$ $(M+H)^+$: 480.2592; found: 480.2589.

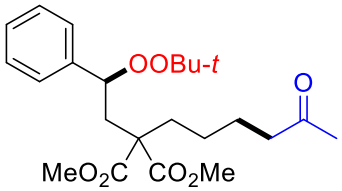


Dimethyl 2-(2-(tert-butylperoxy)-3-oxo-3-phenylpropyl)-2-(2-methyl-5-oxohexyl)malonate (3s): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.4) in 58% yield

(27.1 mg); Colorless oil (dr = 1:1); ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.07 (d, J = 7.6 Hz, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.47 (d, J = 7.6 Hz, 2H), 5.19 (dd, J = 8.8, 3.7 Hz, 0.5H), 5.12 (dd, J = 8.8, 3.8 Hz, 0.5H), 3.71 (s, 3H), 3.70 (s, 3H), 2.53-2.38 (m, 4H), 2.18-2.13 (m, 4H), 1.99-1.88 (m, 1H), 1.60-1.51 (m, 2H), 1.46-1.38 (m, 1H), 1.12 (d, J = 2.4 Hz, 9H), 0.83 (d, J = 5.6 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.7, 208.6, 198.0, 197.9, 171.8, 171.7, 171.5, 171.4, 135.3, 135.2, 133.3, 129.1, 129.0, 128.5, 81.9, 81.6, 80.9, 80.8, 55.1, 55.0, 52.6, 52.5, 52.4, 41.0, 40.1, 39.7, 33.8, 33.6, 31.6, 31.4, 30.0, 29.9, 28.4, 28.3, 26.4, 20.0, 19.9; HRMS (ESI) calcd for $C_{25}H_{36}O_8Na$ $(M+Na)^+$: 487.2302; found: 487.2299.

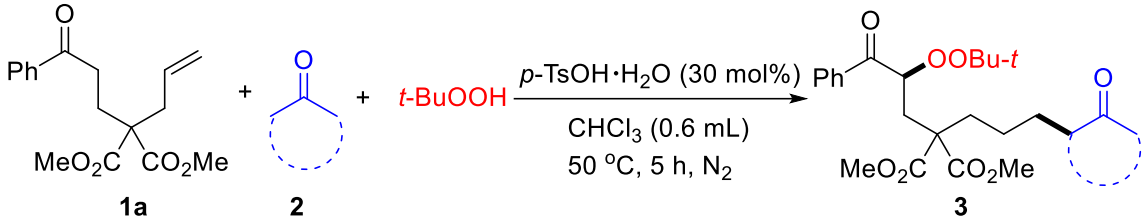


Diethyl 2-(2-(tert-butylperoxy)-3-oxo-3-phenylpropyl)-2-(5-oxohexyl)malonate (3t): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 5:1, R_f = 0.4) in 61% yield (29.0 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.07 (d, J = 7.4 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 5.14 (t, J = 6.1 Hz, 1H), 4.22-2.14 (m, 7H), 2.45-2.42 (m, 4H), 2.13 (s, 3H), 2.12-2.06 (m, 1H), 2.04-1.99 (m, 1H), 1.63-1.58 (m, 2H), 1.26-1.21 (m, 8H), 1.13 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.6, 198.0, 170.9, 170.8, 135.2, 133.2, 129.1, 128.5, 81.9, 80.8, 61.6, 61.4, 55.9, 43.3, 33.0, 32.5, 29.9, 26.4, 23.9, 23.7, 14.0; HRMS (ESI) calcd for $C_{26}H_{38}O_7Na$ $(M+Na)^+$: 501.2459; found: 501.2460.

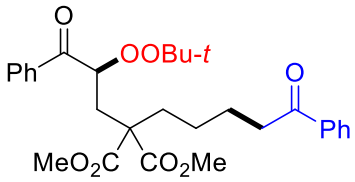


Dimethyl 2-(2-(tert-butylperoxy)-2-phenylethyl)-2-(5-oxohexyl)malonate (3u): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 5:1, R_f = 0.3) in 58% yield (24.6 mg); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 7.34-7.28 (m, 5H), 4.81 (dd, J = 8.0, 5.0 Hz, 1H), 3.71 (s, 3H), 3.55 (s, 3H), 2.58-2.54 (m, 1H), 2.41 (t, J = 7.4 Hz, 1H), 2.34-2.31 (m, 1H), 2.13 (s, 3H), 2.08-2.05 (m, 1H), 2.02-1.99 (m, 1H), 1.60-1.55 (m, 2H), 1.24-1.20 (m, 2H), 1.11 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.6, 171.6, 171.5, 141.1, 128.1, 127.9, 127.3, 81.9, 80.0, 55.9,

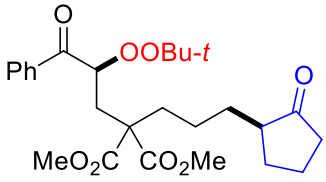
52.5, 52.4, 43.3, 37.3, 32.1, 29.9, 26.5, 23.8, 23.7; HRMS (ESI) calcd for $C_{23}H_{34}O_7Na$ $(M+Na)^+$: 445.2197; found: 445.2195.



To a 10 mL sealable pressure tube was added unactivated alkenes **1** (0.1 mmol), *p*-TsOH·H_2O (0.03 mmol), CH_3Cl (0.6 mL) and *t*-BuOOH (5.5 M in decane, 0.5 mmol) under N_2 atmosphere at room temperature, and the resulting solution was stirred at 50 ℃ for 5 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 **3v-3x**.

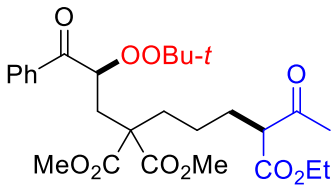


Dimethyl 2-(2-(tert-butylperoxy)-3-oxo-3-phenylpropyl)-2-(5-oxo-5-phenylpentyl)malonate (3v): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 4:1, R_f = 0.3) in 28% yield (14.3 mg); Colorless oil; ^1H NMR (600 MHz, $CDCl_3$, ppm) δ 8.36 (d, J = 7.3 Hz, 2H), 7.95 (d, J = 7.3 Hz, 2H), 7.58-7.55 (m, 2H), 7.48-7.45 (m, 4H), 5.19 (dd, J = 7.5, 5.0 Hz, 1H), 3.72 (s, 3H), 3.71 (s, 3H), 2.99 (t, J = 7.3 Hz, 2H), 2.48-2.42 (m, 2H), 2.18-2.13 (m, 1H), 2.11-2.05 (m, 1H), 1.81-1.76 (m, 2H), 1.39-1.33 (m, 1H), 1.31-1.23 (m, 1H), 1.13 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 199.8, 197.9, 171.4, 171.3, 137.0, 135.3, 133.3, 133.0, 129.1, 128.6, 128.5, 128.1, 81.6, 80.9, 56.0, 52.7, 52.6, 38.2, 33.2, 32.9, 26.4, 24.3, 24.0; HRMS (ESI) calcd for $C_{29}H_{36}O_8Na$ $(M+Na)^+$: 535.2302; found: 535.2303.



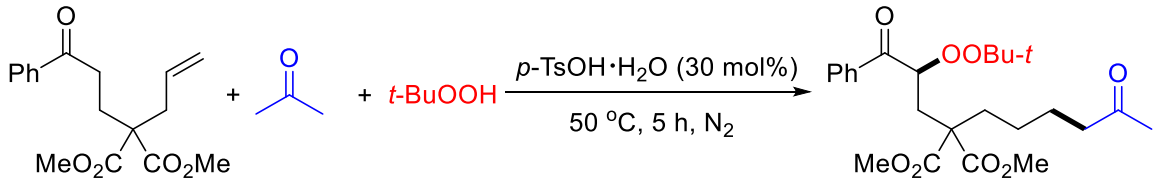
Dimethyl 2-(2-(tert-butylperoxy)-3-oxo-3-phenylpropyl)-2-(3-(2-oxocyclopentyl)propyl)-malonate (3w): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f =

0.3) in 16% yield (7.6 mg); Colorless oil (dr = 1:1); ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.05 (d, J = 7.3 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.9 Hz, 2H), 5.17-5.14 (m, 1H), 3.71 (s, 3H), 3.70 (s, 3H), 2.43-2.41 (m, 2H), 2.31-2.27 (m, 1H), 2.22-2.17 (m, 1H), 2.12-2.05 (m, 2H), 2.04-1.97 (m, 3H), 1.80-1.72 (m, 2H), 1.50-1.43 (m, 1H), 1.30-1.19 (m, 3H), 1.11 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 221.0, 220.9, 197.9, 171.4, 171.3, 135.3, 135.2, 133.3, 129.1, 128.6, 81.6, 81.5, 80.9, 80.8, 56.0, 55.9, 52.7, 52.6, 48.9, 48.8, 38.2, 38.1, 33.3, 33.2, 32.9, 32.8, 29.9, 29.8, 29.7, 29.6, 26.4, 22.4, 22.3, 20.8; HRMS (ESI) calcd for $C_{26}H_{36}O_8Na$ $(M+Na)^+$: 499.2302; found: 499.2303.



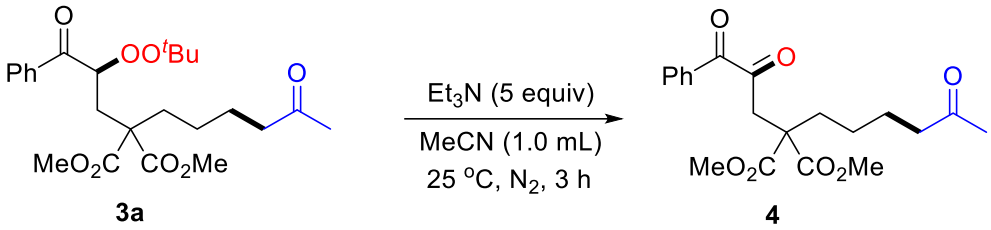
3-ethyl 7,7-dimethyl 9-(tert-butylperoxy)-2,10-dioxo-10-phenyldecane-3,7,7-tricarboxylate (3x): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.3) in 40% yield (20.9 mg); Colorless oil (dr = 1:1); ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.06 (d, J = 7.5 Hz, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.9 Hz, 2H), 5.17-5.14 (m, 1H), 4.22-4.17 (m, 2H), 3.71 (s, 3H), 3.71 (s, 3H), 3.42 (t, J = 7.3 Hz, 1H), 2.42 (d, J = 6.0 Hz, 2H), 2.23 (d, J = 0.8 Hz, 3H), 2.11-2.07 (m, 1H), 2.05-2.01 (m, 1H), 1.90-1.85 (m, 2H), 1.30-1.22 (m, 5H), 1.12 (d, J = 0.5 Hz, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 202.8, 197.9, 197.8, 171.2, 171.1, 169.6, 169.5, 135.3, 133.4, 129.1, 128.6, 81.5, 81.4, 61.5, 59.4, 59.3, 55.9, 52.7, 52.6, 33.2, 32.8, 32.7, 29.0, 28.9, 28.2, 26.5, 22.3, 22.2, 14.2; HRMS (ESI) calcd for $C_{27}H_{38}O_{10}Na$ $(M+Na)^+$: 545.2357; found: 545.2361.

4. Large scale synthesis and transformation



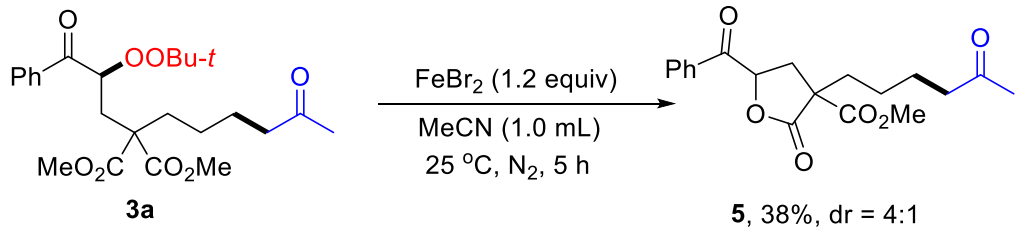
To a 25 mL sealable pressure tube was added unactivated alkenes **1a** (1 mmol), *p*-TsOH·H_2O (0.03 mmol), acetone (6.0 mL) and *t*-BuOOH (5.5 M in decane, 0.5 mmol) under N_2 atmosphere at room temperature, and the resulting solution was stirred at 50 ℃ for 5 h. The resulting mixture and the solvent were evaporated under vacuum. The residue was purified by flash column chromatography

on silica gel (eluent: ethyl acetate/petroleum ether =1/10-1/5) to give the peroxide **3a** in 57% yield (261 mg).



3a **4**

To a 25 mL Schlenk tube with a magnetic stir bar were added **3a** (45.0 mg, 0.1 mmol), Et_3N (70.0 vL, 0.5 mmol) in MeCN (1.0 ml) and then allowed to stir at room temperature under N_2 atmosphere for 3 h. The resultant residue was purified by flash chromatography (ethyl acetate/petroleum ether = 1/8-1/5) to afford dimethyl 2-(2,3-dioxo-3-phenylpropyl)-2-(5-oxohexyl)malonate (**4**) (31.7 mg, 84% yield) as a yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.00 (d, J = 7.2 Hz, 2H), 7.64 (t, J = 7.4 Hz, 1H), 7.50 (t, J = 7.8 Hz, 2H), 3.76 (s, 6H), 3.48 (s, 2H), 2.44 (t, J = 7.3 Hz, 2H), 2.12 (s, 3H), 2.06 (t, J = 8.4 Hz, 2H), 1.61-1.56 (m, 2H), 1.31-1.26 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.4, 199.0, 190.9, 171.0, 134.6, 131.7, 130.5, 128.8, 56.0, 52.9, 43.1, 41.2, 34.1, 29.9, 24.2, 23.6; HRMS (ESI) calcd for $C_{20}H_{24}O_7Na$ $(M+Na)^+$: 399.1414; found: 399.1405.

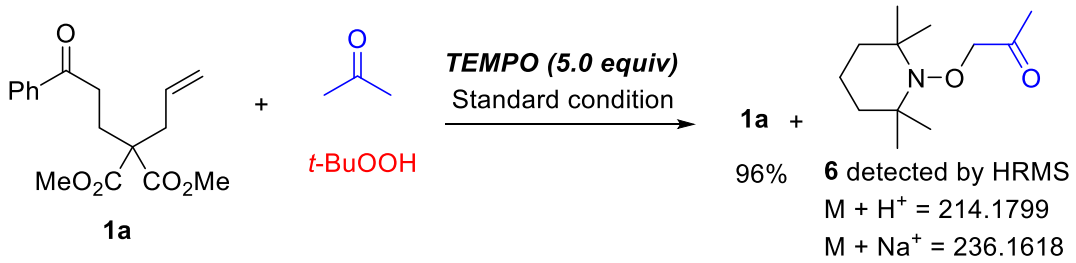


3a **5**, 38%, dr = 4:1

To a 25 mL Schlenk tube with a magnetic stir bar were added **3a** (90.0 mg, 0.2 mmol), $FeBr_2$ (51.8 mg, 0.24 mmol) in MeCN (1.0 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 = 1/5-1/2) to afford compound **5** with 4 : 1 diastereomer (26.1 mg, 38%, yellow oil). ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.01 (d, J = 8.0 Hz, 2H), 7.65 (t, J = 7.4 Hz, 1H), 7.52 (t, J = 7.8 Hz, 2H), 5.73 (dd, J = 9.2, 7.2 Hz, 1H), 3.84 (s, 3H), 3.04-3.01 (m, 1H), 2.47-2.41 (m, 4H), 2.18-2.12 (m, 5H), 1.81-1.76 (m, 1H), 1.62-1.57 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.4, 194.0, 193.3, 172.8, 169.6, 169.4, 134.4, 134.1, 133.9, 129.0, 128.9, 128.8, 128.7, 76.9, 76.6, 55.2, 53.5, 42.9, 42.8, 34.0, 29.9, 24.2, 23.4, 23.3; HRMS (ESI) calcd for $C_{19}H_{22}O_6Na$ $(M+Na)^+$: 369.1309; found: 369.1303.

5. Mechanistic studies

(a) Control experiments



To a 10 mL sealable pressure tube was added added unactivated alkenes **1a** (0.1 mmol), *p*-TsOH·H_2O (0.03 mmol), TEMPO (78.1 mg, 0.5 mmol), acetone (0.6 mL) and *t*-BuOOH (5.5 M in decane, 0.5 mmol) under N_2 atmosphere at room temperature, and the resulting solution was stirred at 50 ℃ for 5 h. The model reaction was totally supressed where alkene **1a** was almost fully recovered, and we can detect the molecular weight of compound **6** by HRMS (Figure 1).

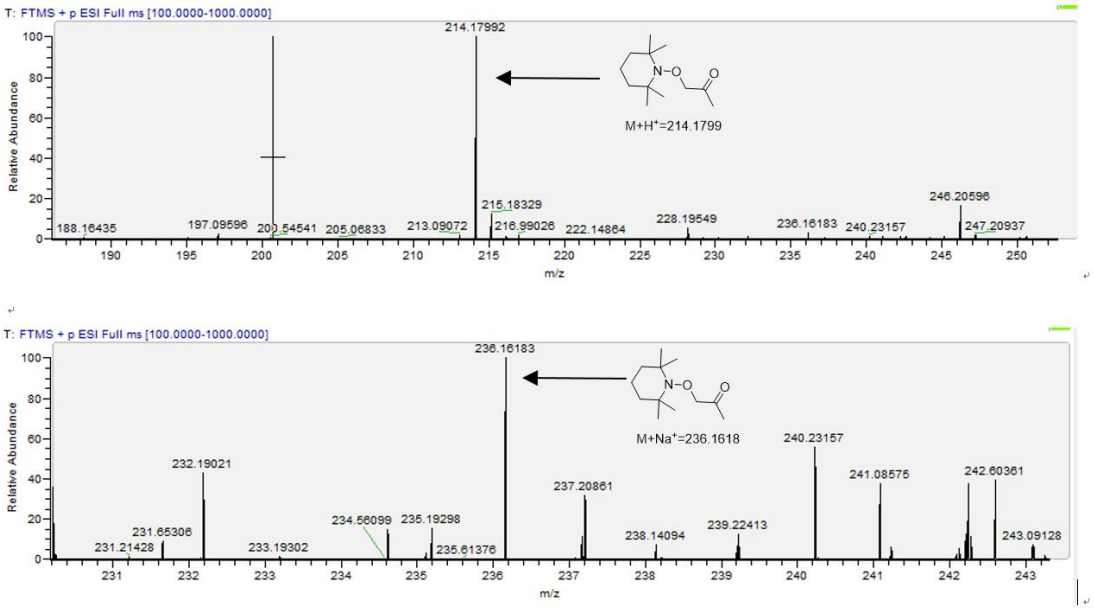
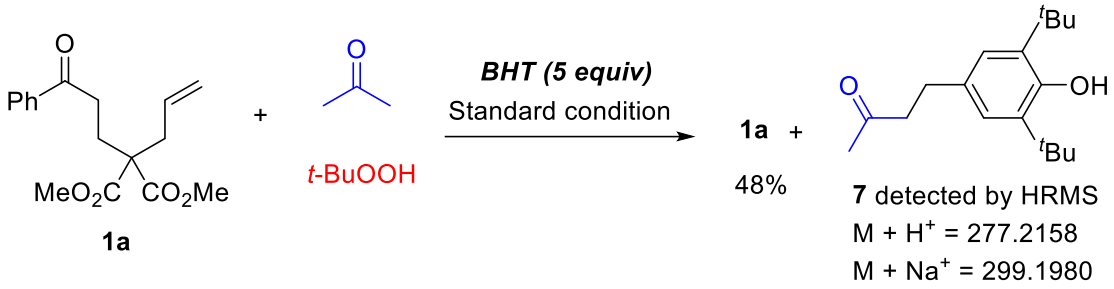


Figure 1. HRMS of the reaction solution



To a 10 mL sealable pressure tube was added added unactivated alkenes **1a** (0.1 mmol), *p*-TsOH·H_2O (0.03 mmol), BHT (110 mg, 0.5 mmol), acetone (0.6 mL) and *t*-BuOOH (5.5 M in decane, 0.5 mmol) under N_2 atmosphere at room temperature, and the resulting solution was stirred at 50 ℃ for 5 h. The model reaction was totally supressed where alkene **1a** was almost fully recovered, and we can detect the molecular weight of compound **7** by HRMS (Figure 2).

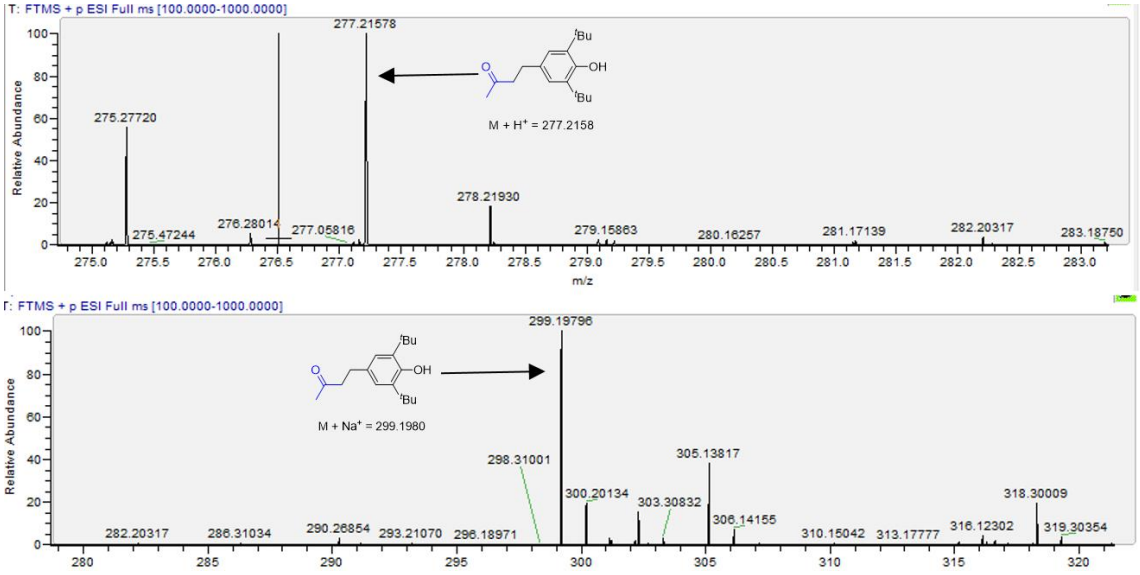
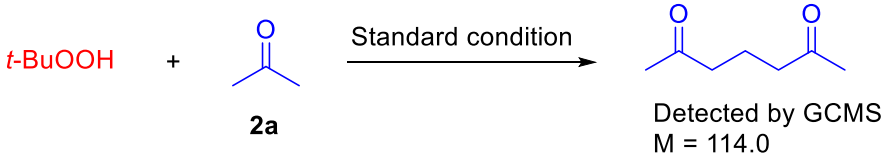


Figure 2. HRMS of the reaction solution



To a 10 mL sealable pressure tube was added added *p*-TsOH·H_2O (0.03 mmol), acetone (0.6 mL) and *t*-BuOOH (5.5 M in decane, 0.5 mmol) under N_2 atmosphere at room temperature, and the resulting solution was stirred at 50 ℃ for 5 h. We can detect the molecular weight of 2,5-hexadione by GC-MS. (Figure 3)

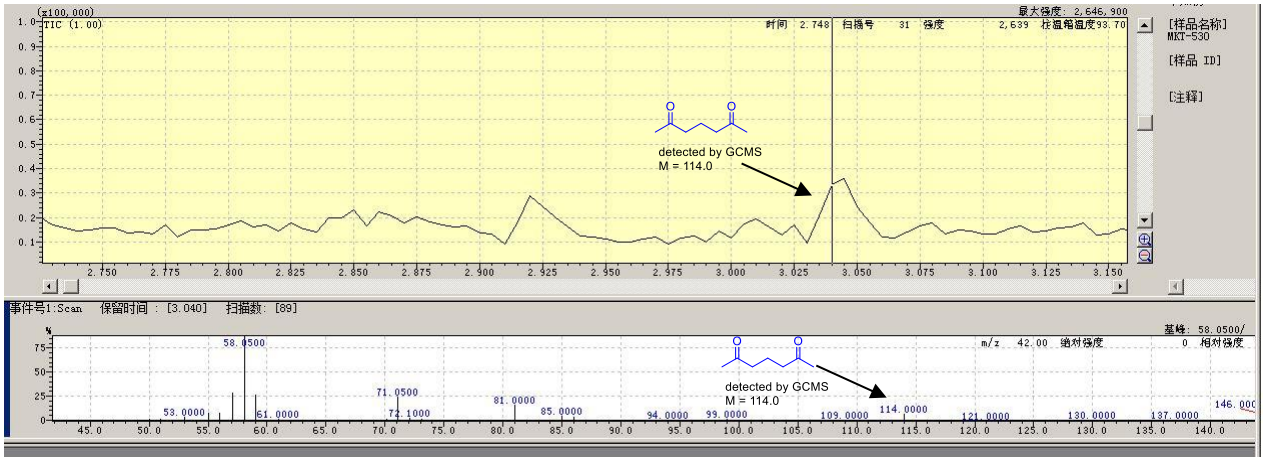
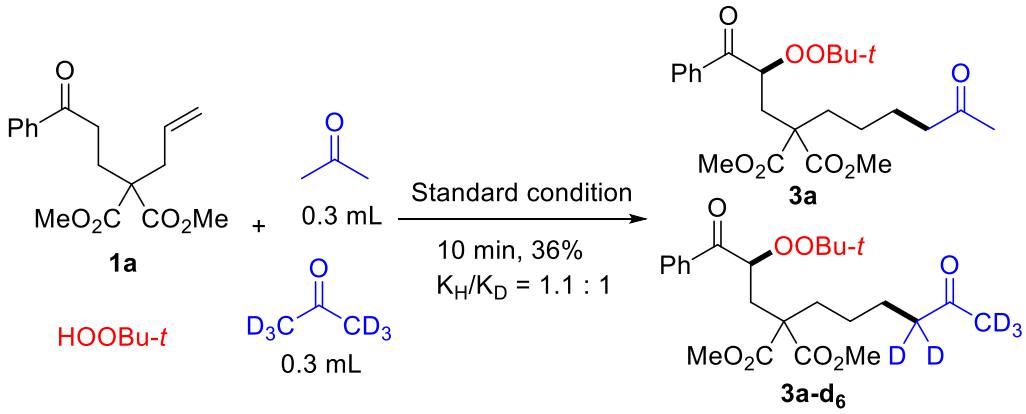


Figure 3. GC-MS of the reaction solution

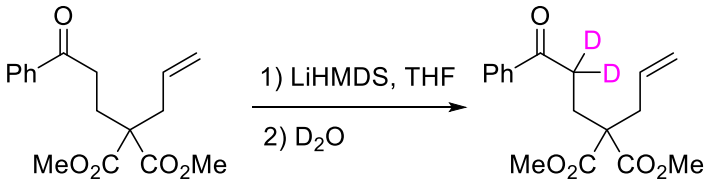
(b) Kinetic isotope effect (KIE) experiment



To a 10 mL sealable pressure tube was added unactivated alkenes **1a** (0.1 mmol), *p*-TsOH·H_2O (0.03 mmol), acetone (0.3 mL), acetone-d_6 (0.3 mL) and *t*-BuOOH (5.5 M in decane, 0.5 mmol) under N_2 atmosphere at room temperature, and the resulting solution was stirred at 50 °C for 10 minutes. The yield was 36% with a ratio of 1.1 : 1 (k_H : k_D), which suggested that the cleavage of the C(sp^3)-H bond was not the rate-determining steps in this transformation. ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.06 (d, J = 7.4 Hz, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.8 Hz, 2H), 5.16 (dd, J = 7.0, 5.2 Hz, 1H), 3.72 (s, 3H), 3.71 (s, 3H), 2.45-2.40 (m, 3.06H), 2.13 (s, 1.58H), 2.10-2.06 (m, 1H), 2.04-1.99 (m, 1H), 1.63-1.58 (m, 2H), 1.25-1.17 (m, 2H), 1.13 (s, 9H). HRMS (ESI) calcd for $C_{24}H_{29}D_5O_8Na$ $(M+Na)^+$: 478.2460; found: 478.2458.

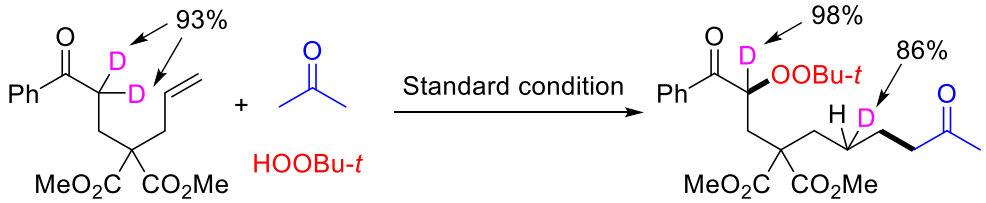
(c) Deuterium labeling experiment

The deuterium unactivated alkene substrate **1a** was known compound. Unactivated alkene **1a-d_2** was prepared as followed procedure.[1]



1a **1a-d_2**

Experimental procedures for the synthesis of **1a-d_2**: To a solution of **1a** (608.0 mg, 2.0 mmol) in THF (3.0 mL) at -78 ℃ 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 ℃, 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. **1a-d_2**[2]: Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 7.94 (d, J = 7.2 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.45 (t, J = 7.8 Hz, 2H), 5.74-5.64 (m, 1H), 5.16-5.11 (m, 2H), 3.72 (s, 6H), 3.02-2.91 (m, 0.14H), 2.71 (d, J = 7.4 Hz, 2H), 2.30 (s, 2H).



1a-d_2 **3a-d_2**, 57%

To a 10 mL sealable pressure tube was added unactivated alkenes **1a-d_2** (0.1 mmol), *p*-TsOH·H_2O (0.03 mmol), acetone (0.6 mL) and *t*-BuOOH (5.5 M in decane, 0.5 mmol) under N_2 atmosphere at room temperature, and the resulting solution was stirred at 50 ℃ for 5 h. The resulting mixture and the solvent were evaporated under vacuum. The residue was purified by flash column chromatography on silica gel (ethyl acetate/petroleum ether = 1/8-1/5) to give the peroxide **3a-d_2** in 54% yield (24.3 mg).

3a-d_2: Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$, ppm) δ 8.05 (d, J = 7.3 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.9 Hz, 2H), 5.15 (dd, J = 7.2, 5.2 Hz, 0.02H), 3.70 (s, 3H), 3.69 (s, 3H), 2.44-2.41 (m, 4H), 2.12 (s, 3H), 2.09-2.04 (m, 1H), 2.01-1.97 (m, 1H), 1.61-1.57 (m, 2H), 1.23-1.17 (m,

1.14H), 1.12 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$, ppm) δ 208.6, 197.9, 171.3, 171.2, 135.2, 133.3, 129.0, 128.5, 80.8, 55.9, 52.7, 52.6, 43.2, 33.1, 32.7, 29.9, 26.4, 23.7; HRMS (ESI) calcd for $C_{24}H_{32}D_2O_8Na$ $(M+Na)^+$: 475.2271; found: 475.2266.

6. References

(1) L. Huang, S. C. Zheng, B. Tan and X. Y. Liu, Metal-Free Direct 1,6- and 1,2-Difunctionalization Triggered by Radical Trifluoromethylation of Alkenes, *Org. Lett*., 2015, **17**, 1589-1592.

(2) L. Wang, Y. Ma, Y. Jiang, L. Lv and Z. Li, A Mn-catalyzed Remote C(sp3)-H Bond Peroxidation Triggered by Radical Trifluoromethylation of Unactivated Alkenes, *Chem. Commun*., 2021, **57**, 7846-7849.

(3) W. Yu, H. Jiang, L. Yan, Z. Feng, Y. Luo and P. Xu, Visible-Light induced Generation of Bifunctional Nitrogen-Centered Radicals: a Concise Synthetic Strategy to Construct Bicyclo[3.2.1] octane and Azepane cores, *Sci. China Chem*., 2021, **64**, 274-280.

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

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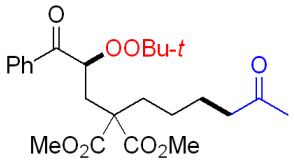
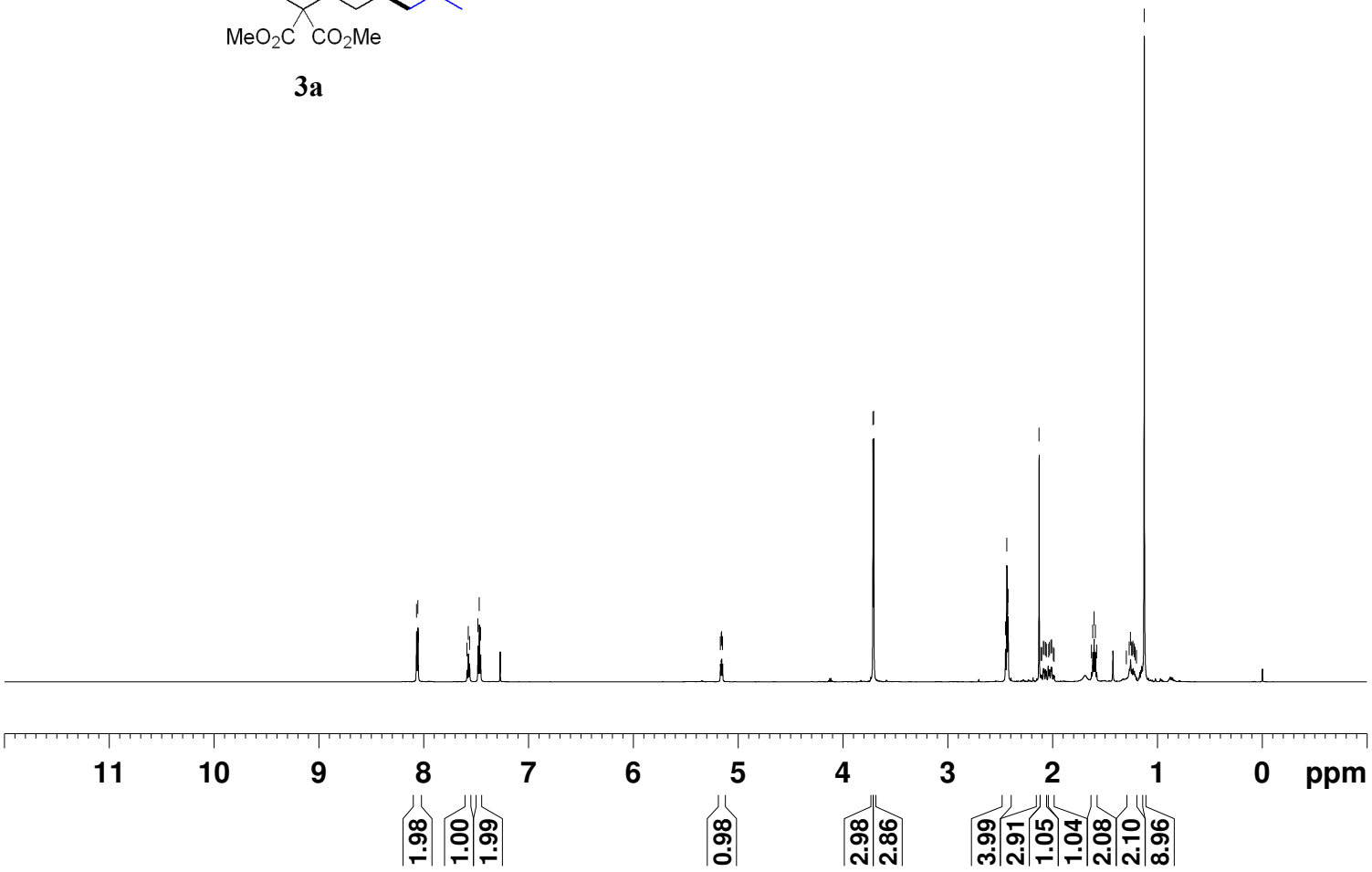


3a

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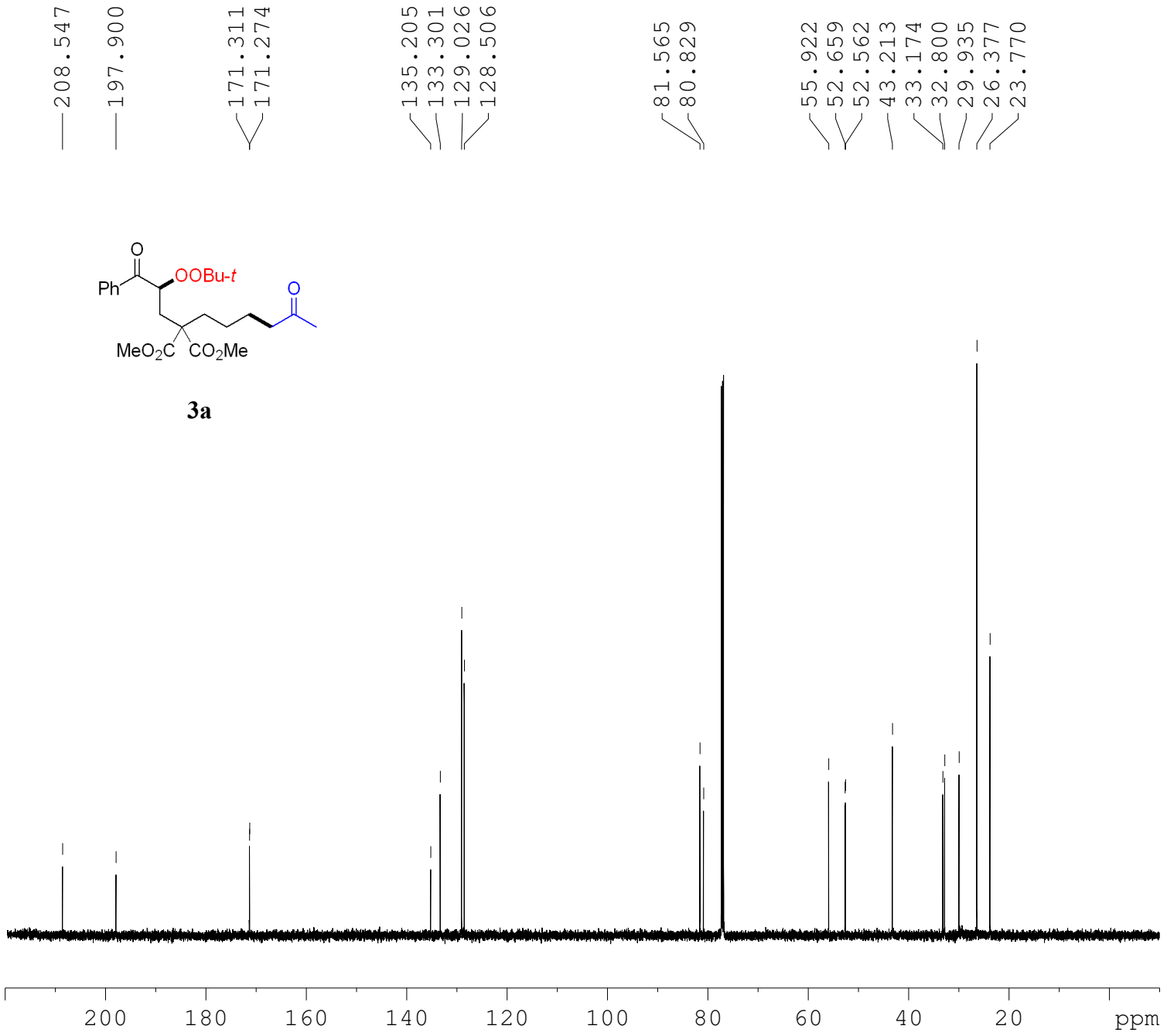

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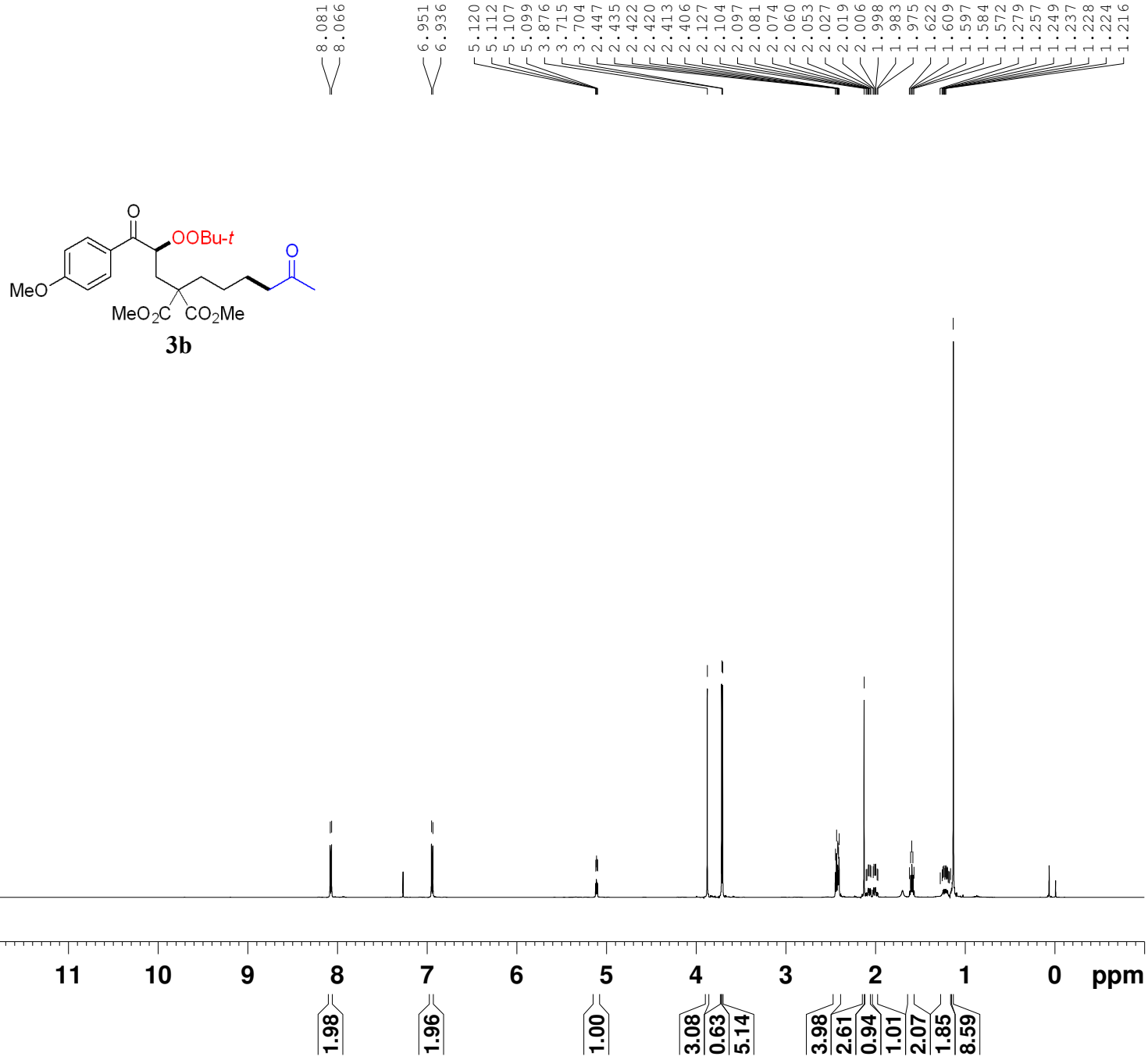


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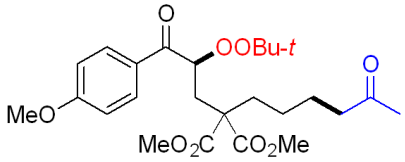

3b


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RG                  190.02
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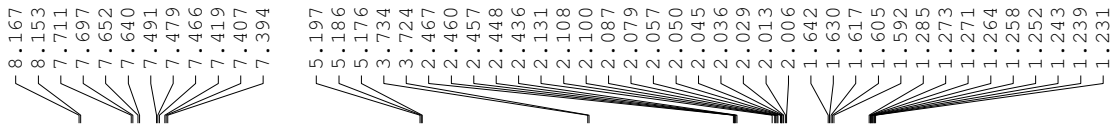

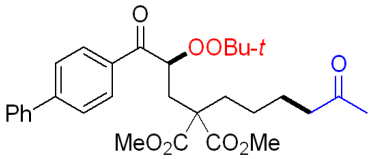

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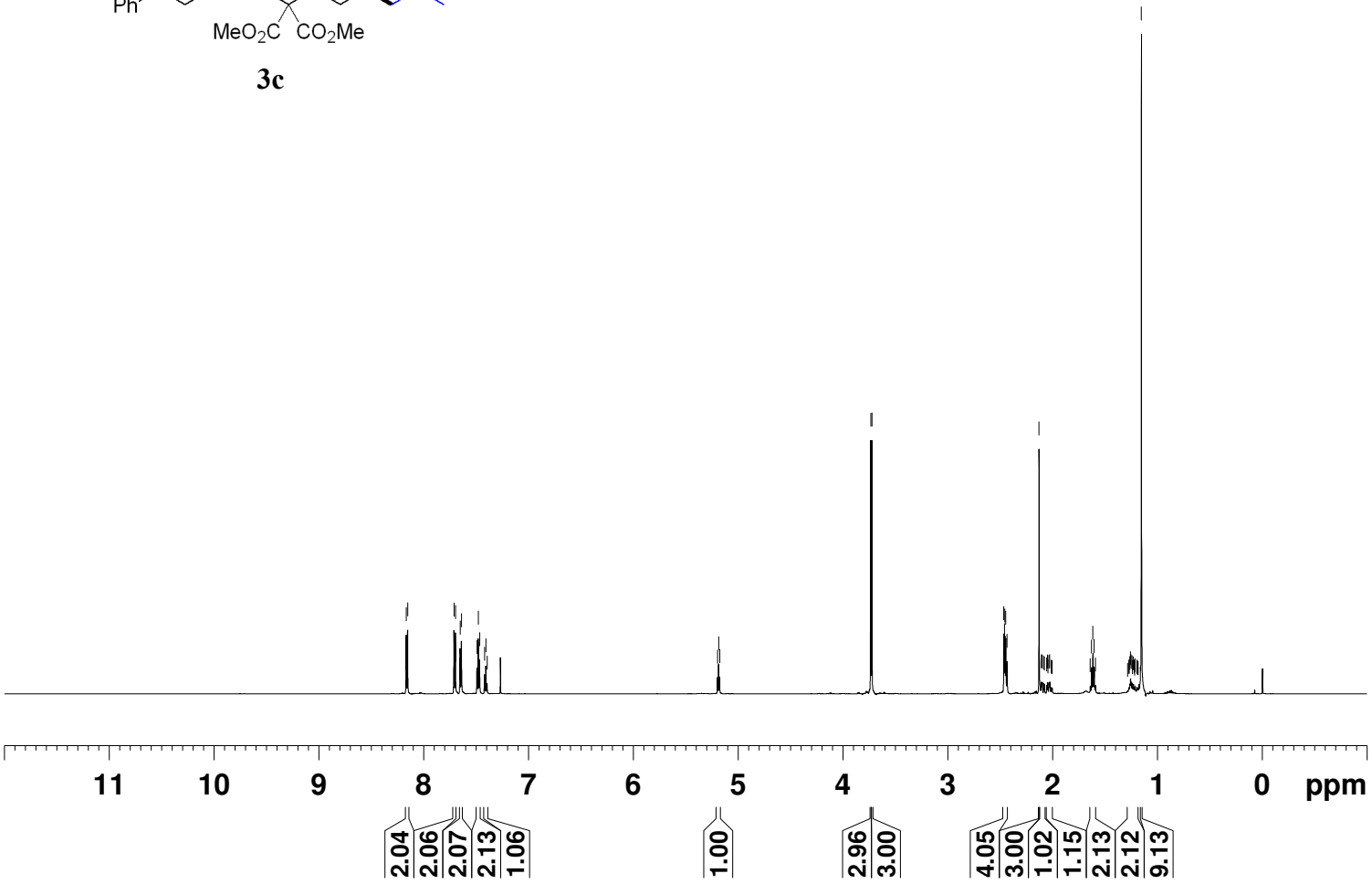

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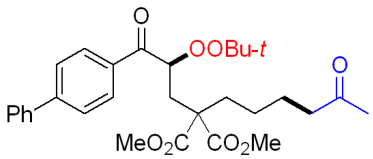

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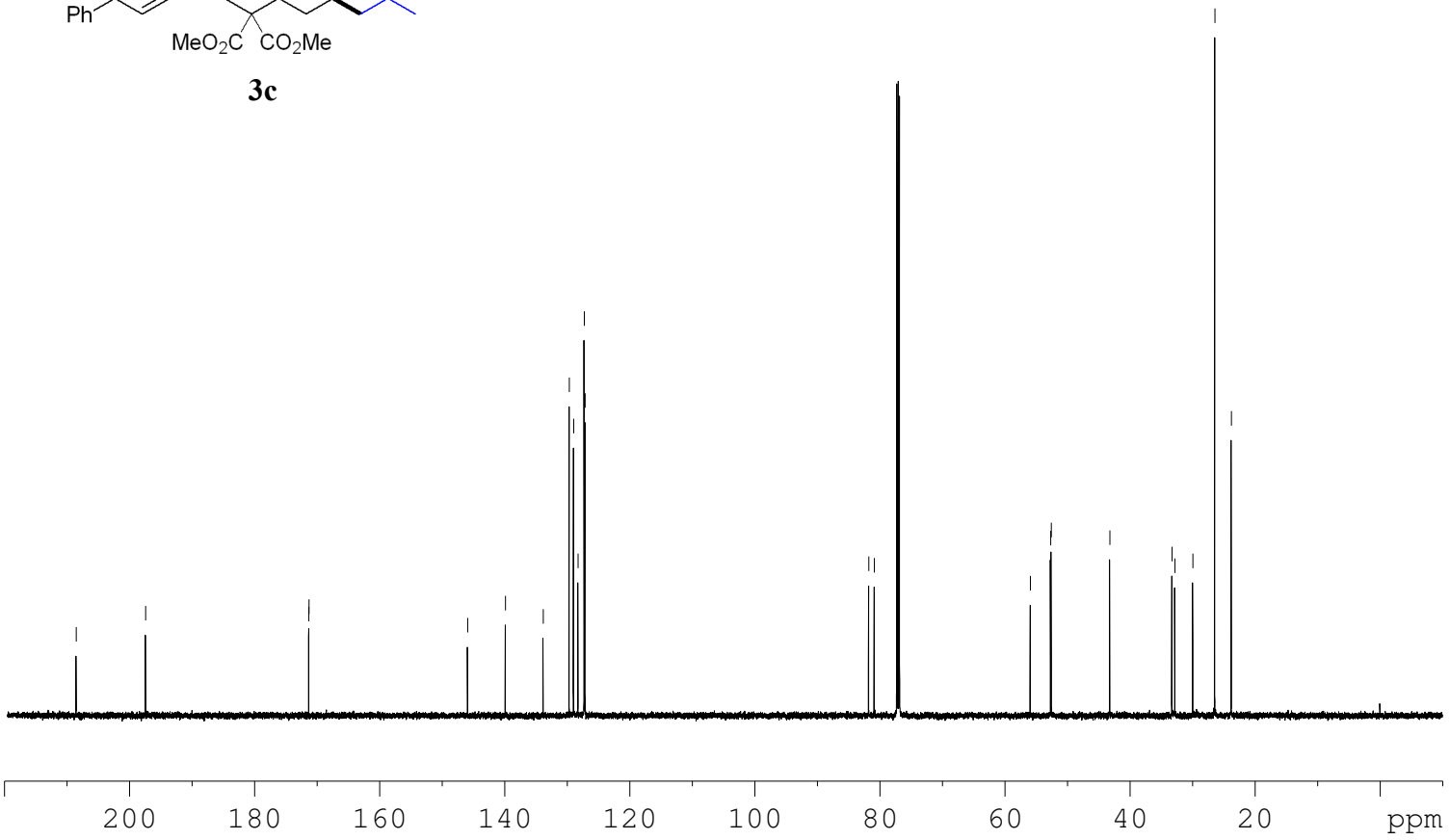

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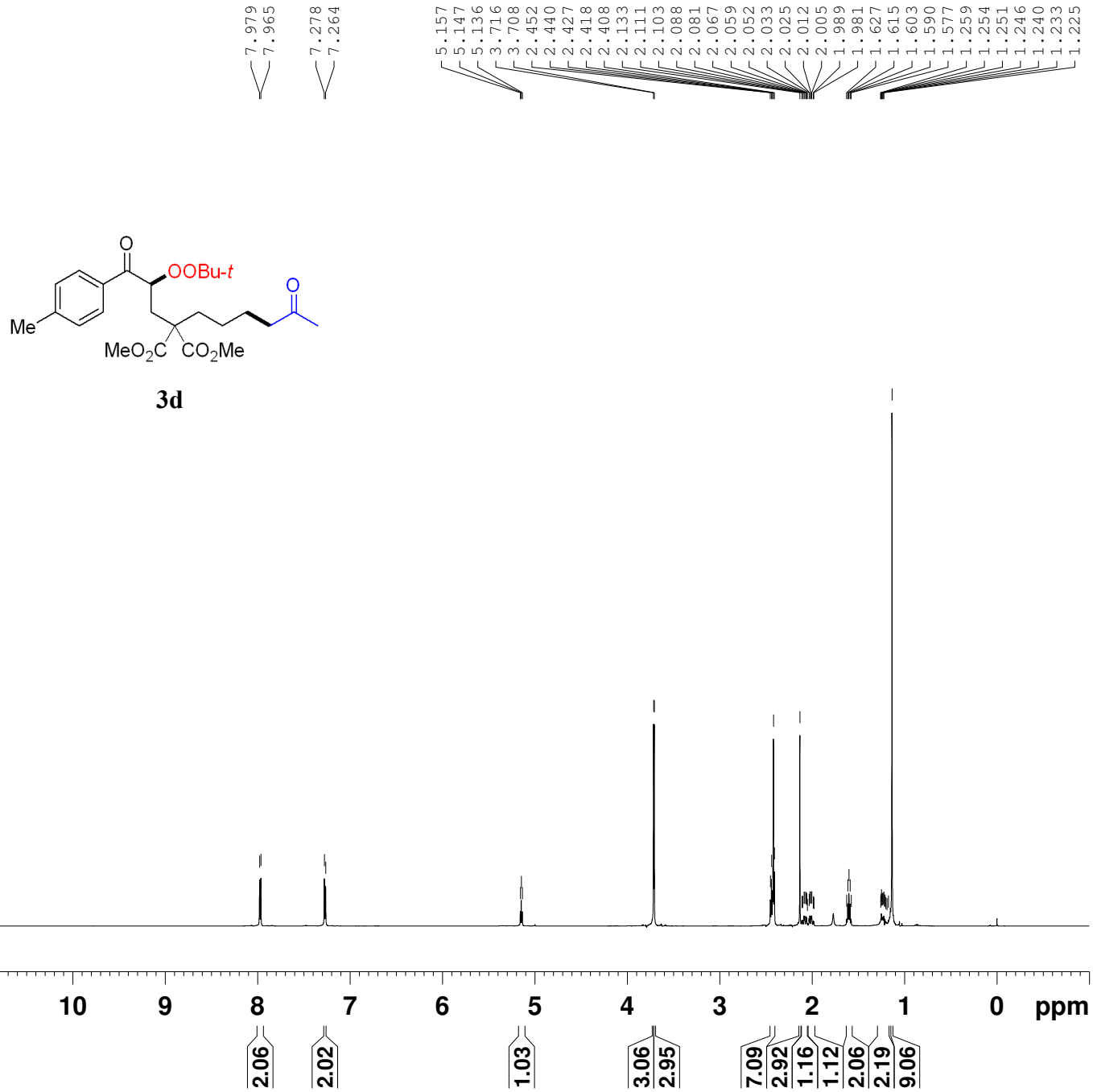


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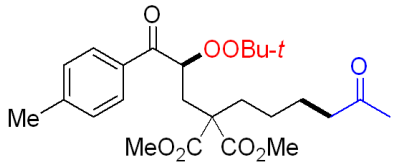
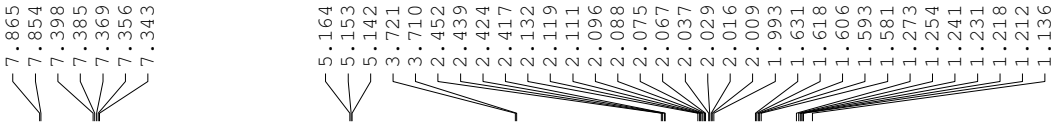
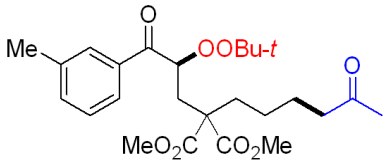
171.325
171.289
144.200
132.633
129.221
129.158
81.486
80.780
55.915
52.642
52.546
43.219
33.266
32.771
29.931
26.398
23.771
23.760
21.722



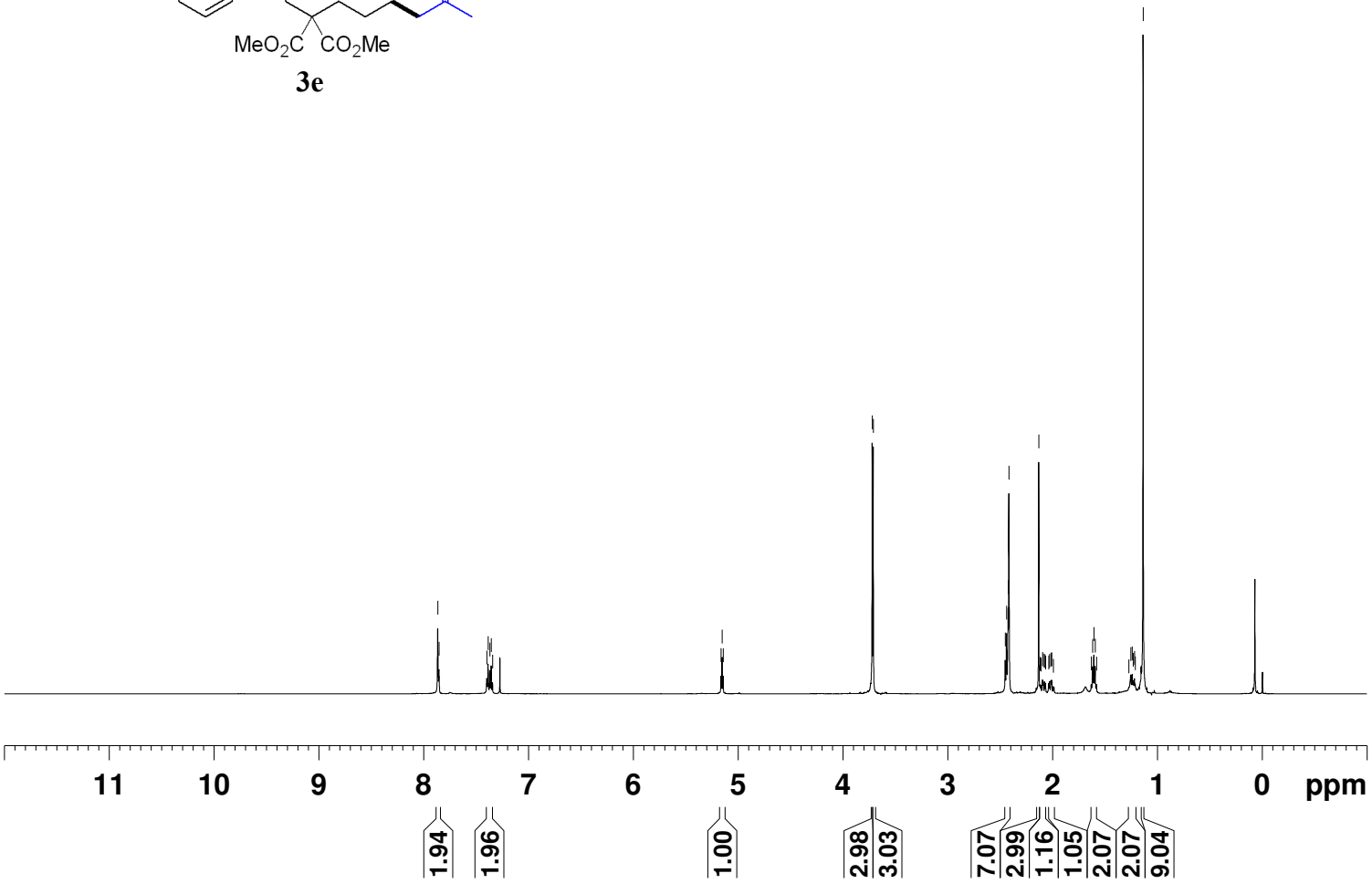
3d


```
NAME      wll-545p-20210608
EXPNO                     2
PROCNO                    1
Date_              20210609
Time                   1.14
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                   296.9 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
```



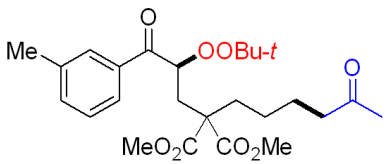


3e

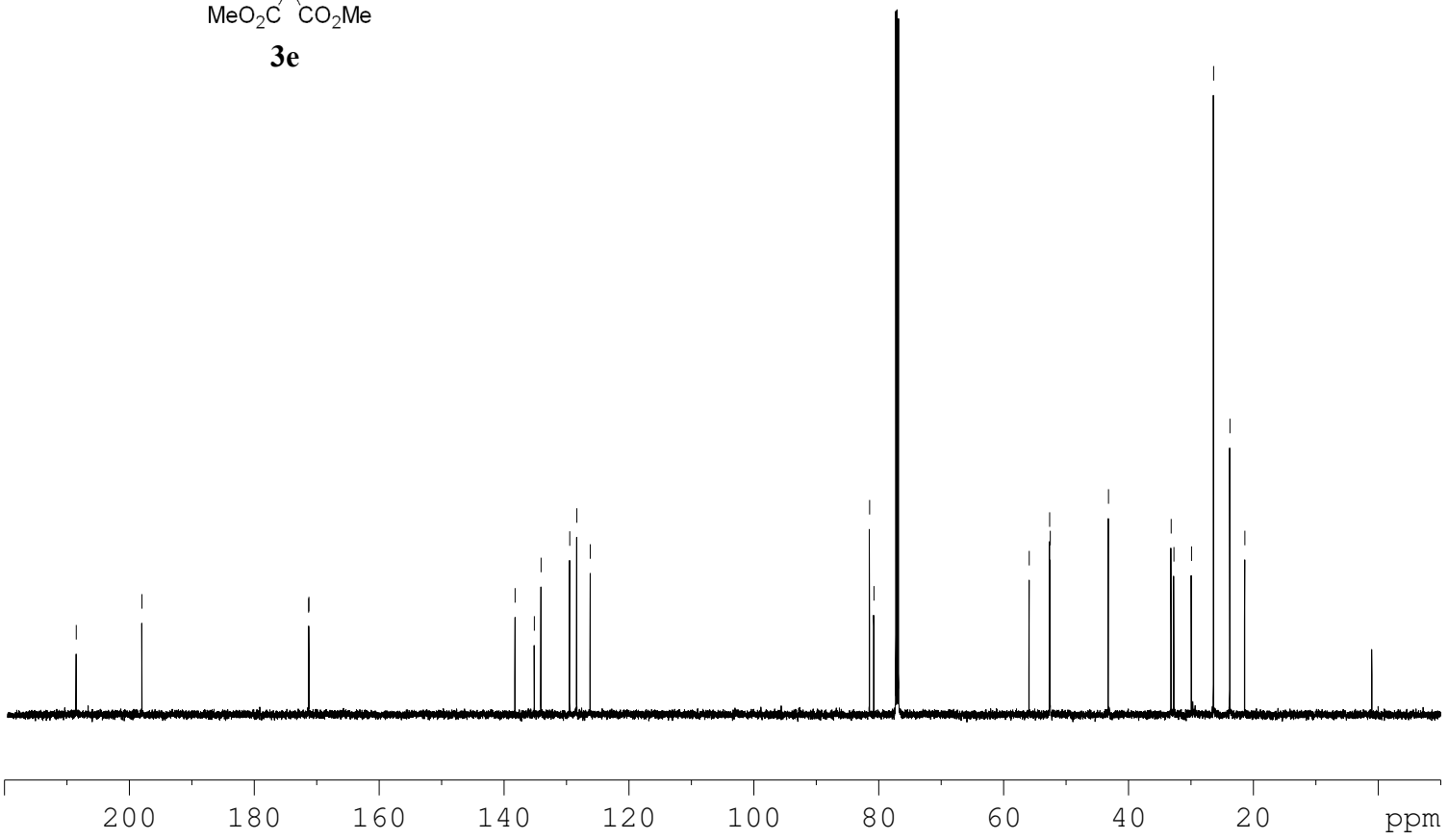



```
NAME      wll-552p-20210610
EXPNO                    1
PROCNO                   1
Date_             20210611
Time                  5.00
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH              9615.385 Hz
FIDRES           0.146719 Hz
AQ               3.4079220 sec
RG                   50.85
DW                  52.000 usec
DE                   6.50 usec
TE                  295.6 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.96 usec
SI                   65536
SF            600.1700088 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

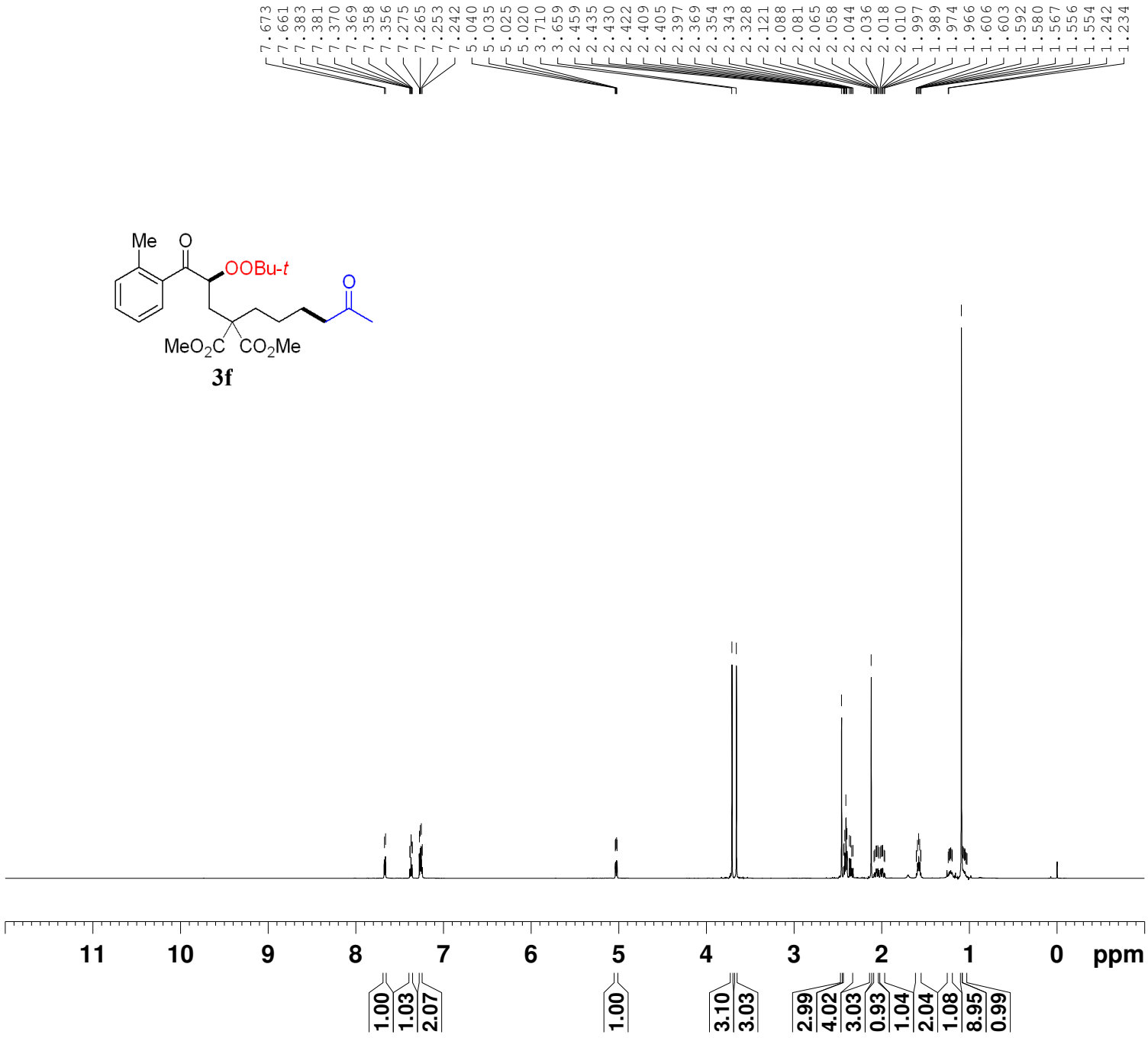
208.507
197.988
171.301
171.249
138.233
135.150
134.072
129.481
128.345
126.192
81.460
80.765
55.902
52.630
52.529
43.210
33.169
32.728
29.910
26.373
23.758
21.371



3e

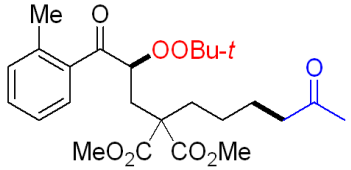



```
NAME      wll-552p-20210610
EXPNO                    2
PROCNO                   1
Date_             20210611
Time                  5.16
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   300
DS                     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                296.7 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           150.9128704 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

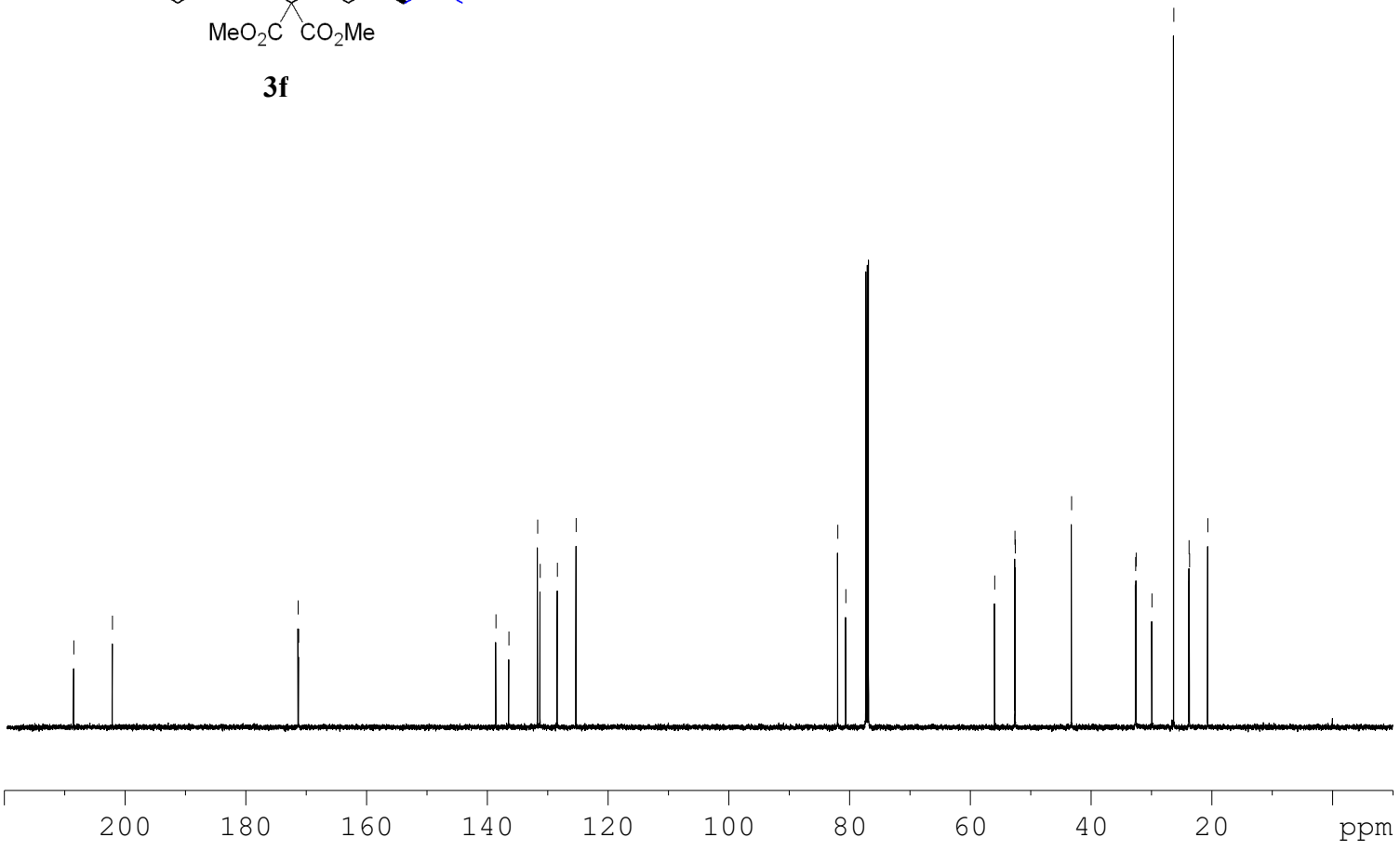



```
NAME      wll-549p-20210610
EXPNO                    1
PROCNO                   1
Date_             20210610
Time                 17.33
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               9615.385 Hz
FIDRES            0.146719 Hz
AQ                3.4079220 sec
RG                    44.5
DW                  52.000 usec
DE                   6.50 usec
TE                  295.6 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           600.1739011 MHz
NUC1                    1H
P1                    9.96 usec
SI                   65536
SF             600.1700066 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

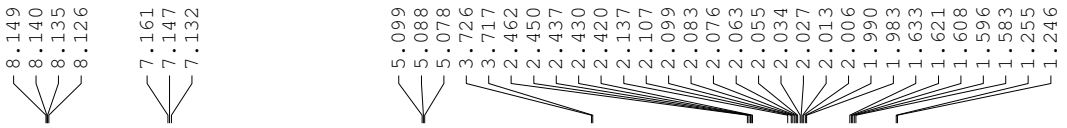
208.492
202.095
171.340
171.247
138.572
136.427
131.666
131.245
128.397
125.294
81.952
80.624
55.964
52.611
52.559
43.235
32.623
32.536
29.923
26.303
23.778
23.690
20.659

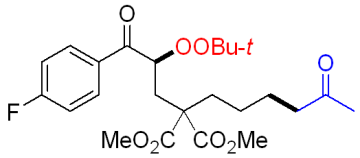


3f

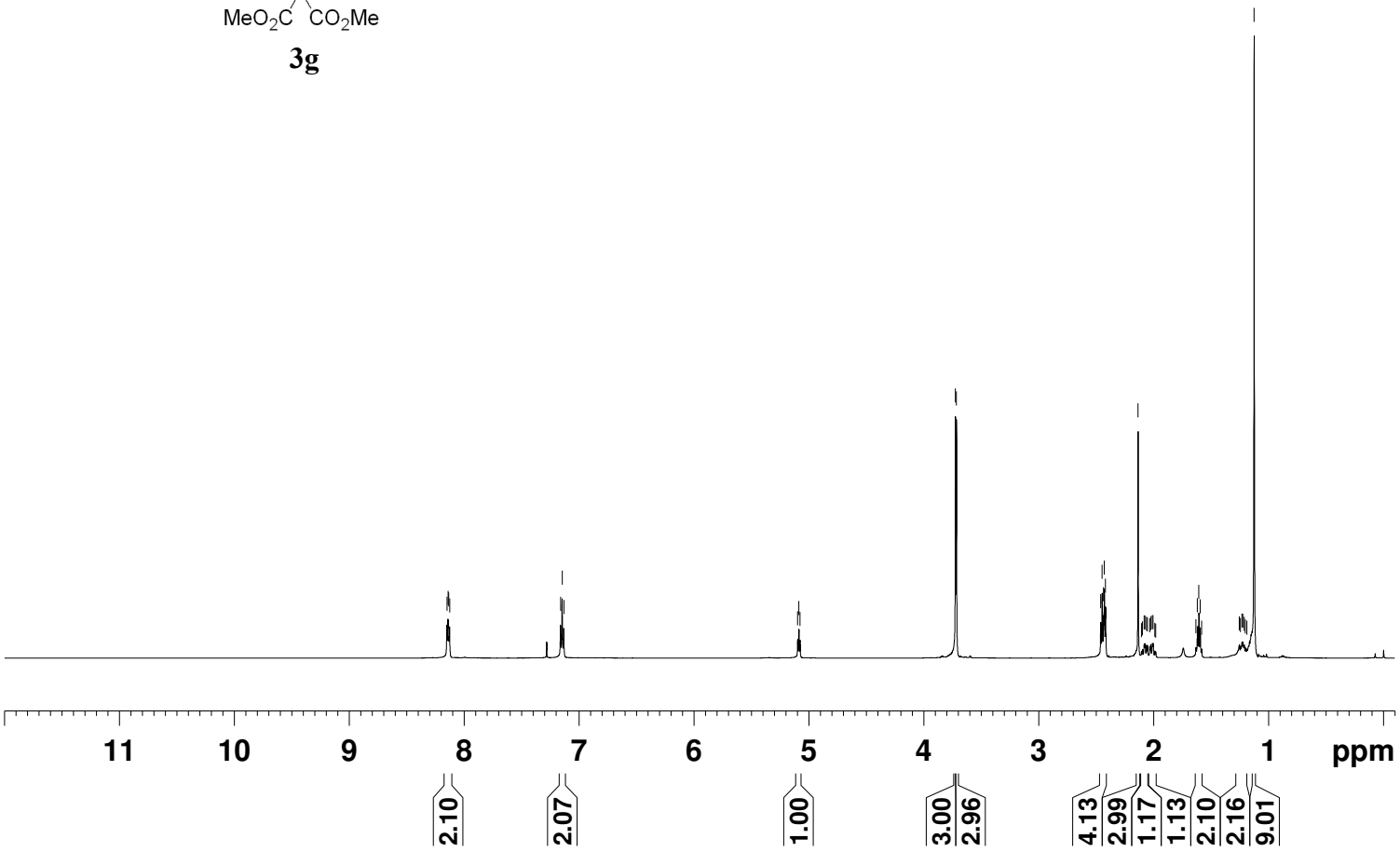



```
NAME      wll-549p-20210610
EXPNO                    2
PROCNO                   1
Date_             20210611
Time                  0.32
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     300
DS                       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                   296.9 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
```



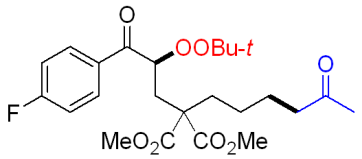


3g



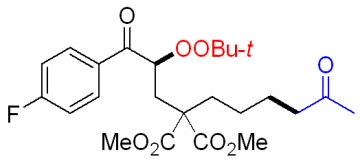

```
NAME      wll-546p-20210608
EXPNO                    1
PROCNO                   1
Date_             20210609
Time                 2.14
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH             9615.385 Hz
FIDRES          0.146719 Hz
AQ              3.4079220 sec
RG                   38.1
DW                52.000 usec
DE                 6.50 usec
TE                295.9 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         600.1739011 MHz
NUC1                   1H
P1                  9.96 usec
SI                 65536
SF           600.1700039 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

208.490
196.395
171.259
166.678
164.987
131.887
131.827
131.504
131.485
115.692
115.548
82.040
80.939
55.861
52.699
52.593
43.169
33.217
32.778
29.941
26.362
23.746



3g

NAME	wll-546p-20210608
EXPNO	2
PROCNO	1
Date_	20210609
Time	2.35
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	297.0 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

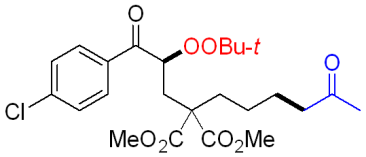


3g

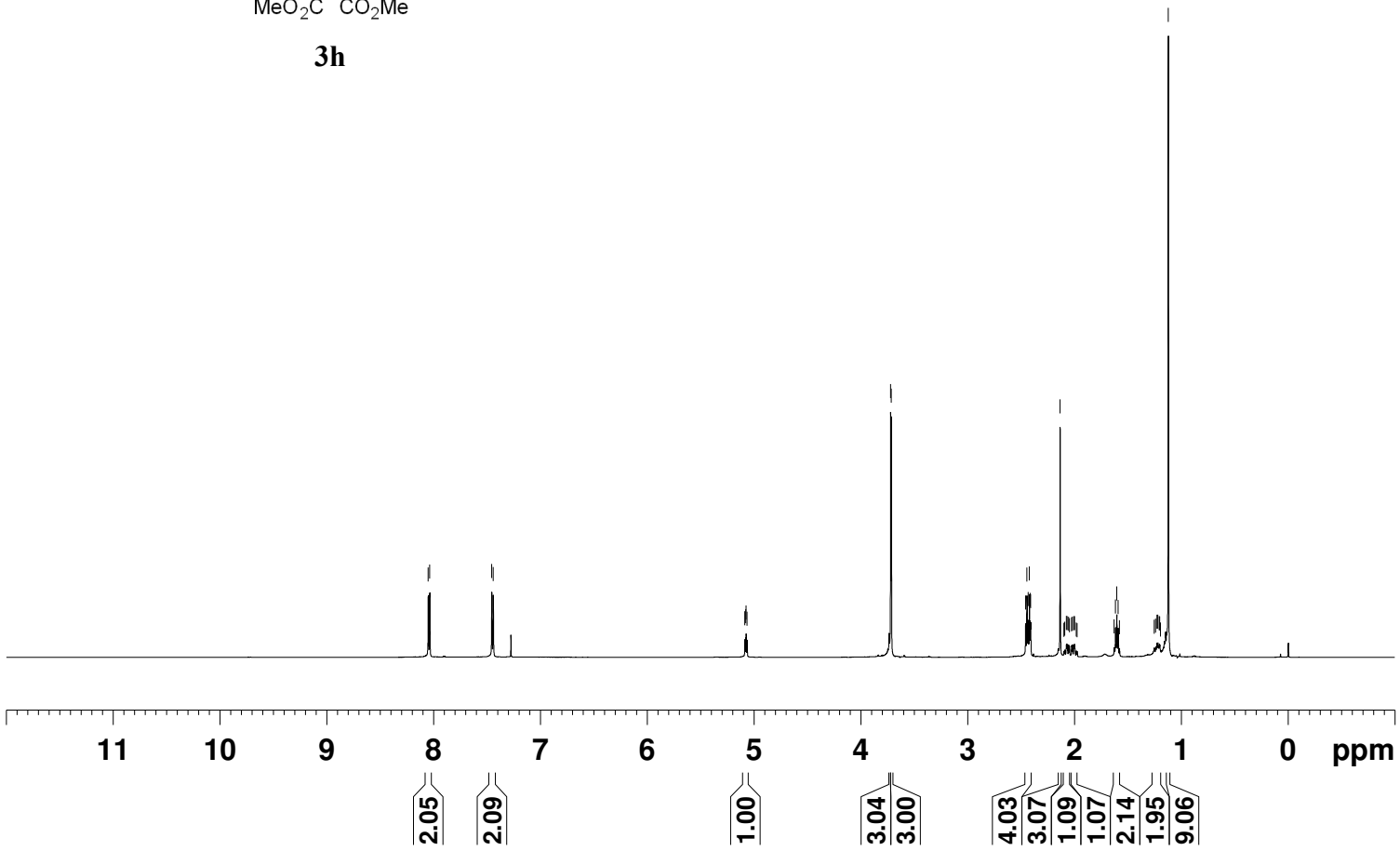
−104.6157

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)

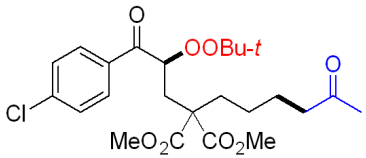


3h

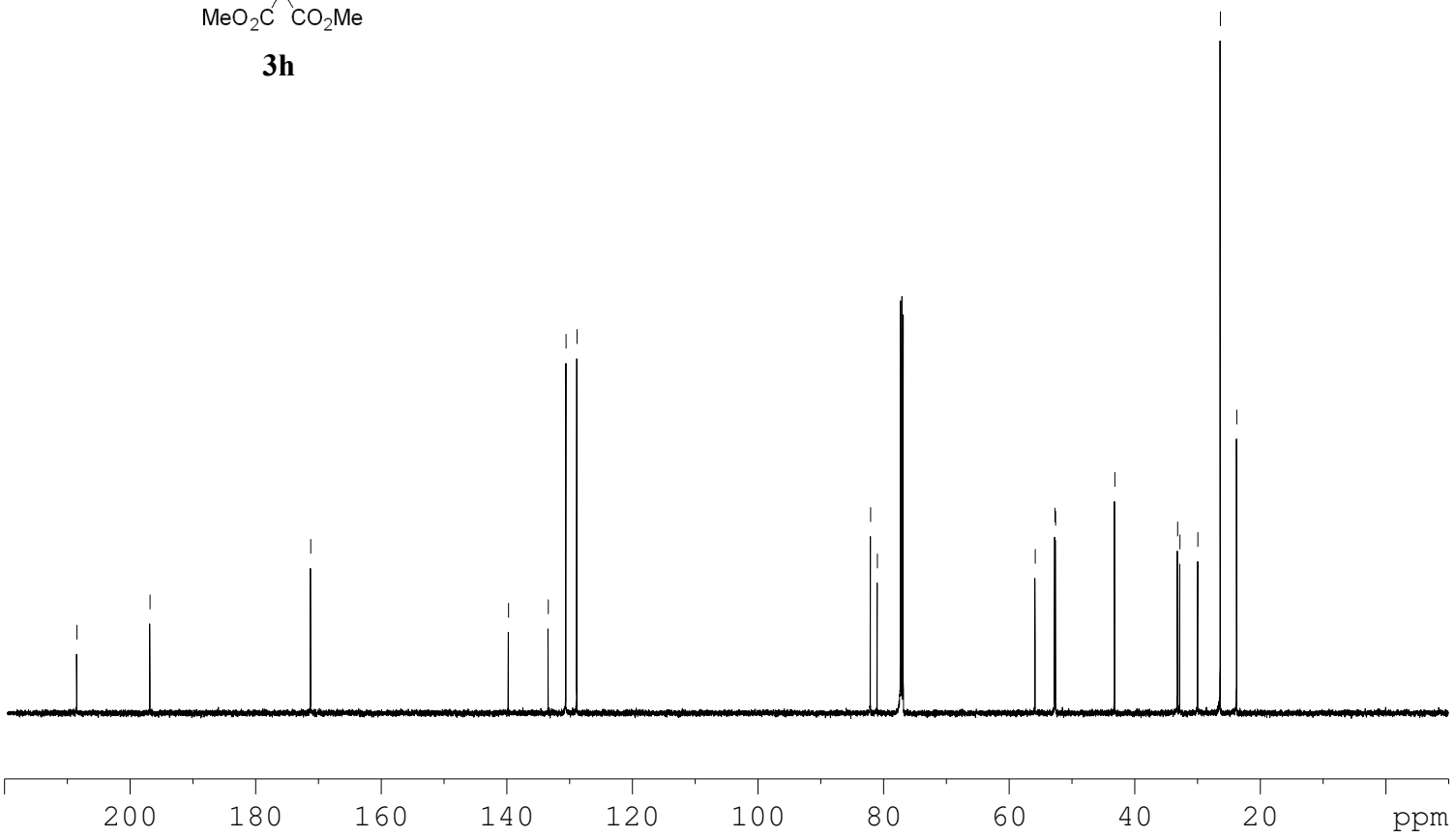



```
NAME      wll-547p-20210610
EXPNO                    1
PROCNO                   1
Date_             20210610
Time                 17.25
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       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                  295.6 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.96 usec
SI                   65536
SF            600.1700053 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

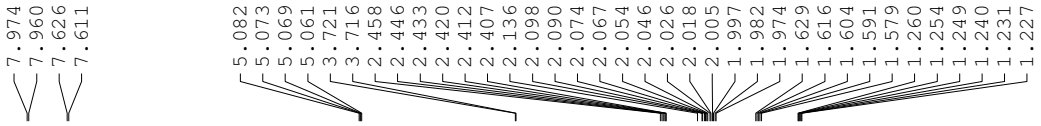
208.464
196.824
171.231
139.731
133.402
130.572
128.823
82.069
80.990
55.848
52.713
52.604
43.169
33.172
32.801
29.949
26.362
23.745

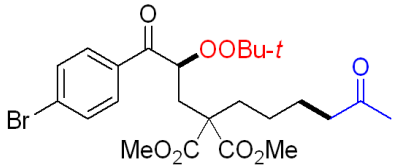


3h

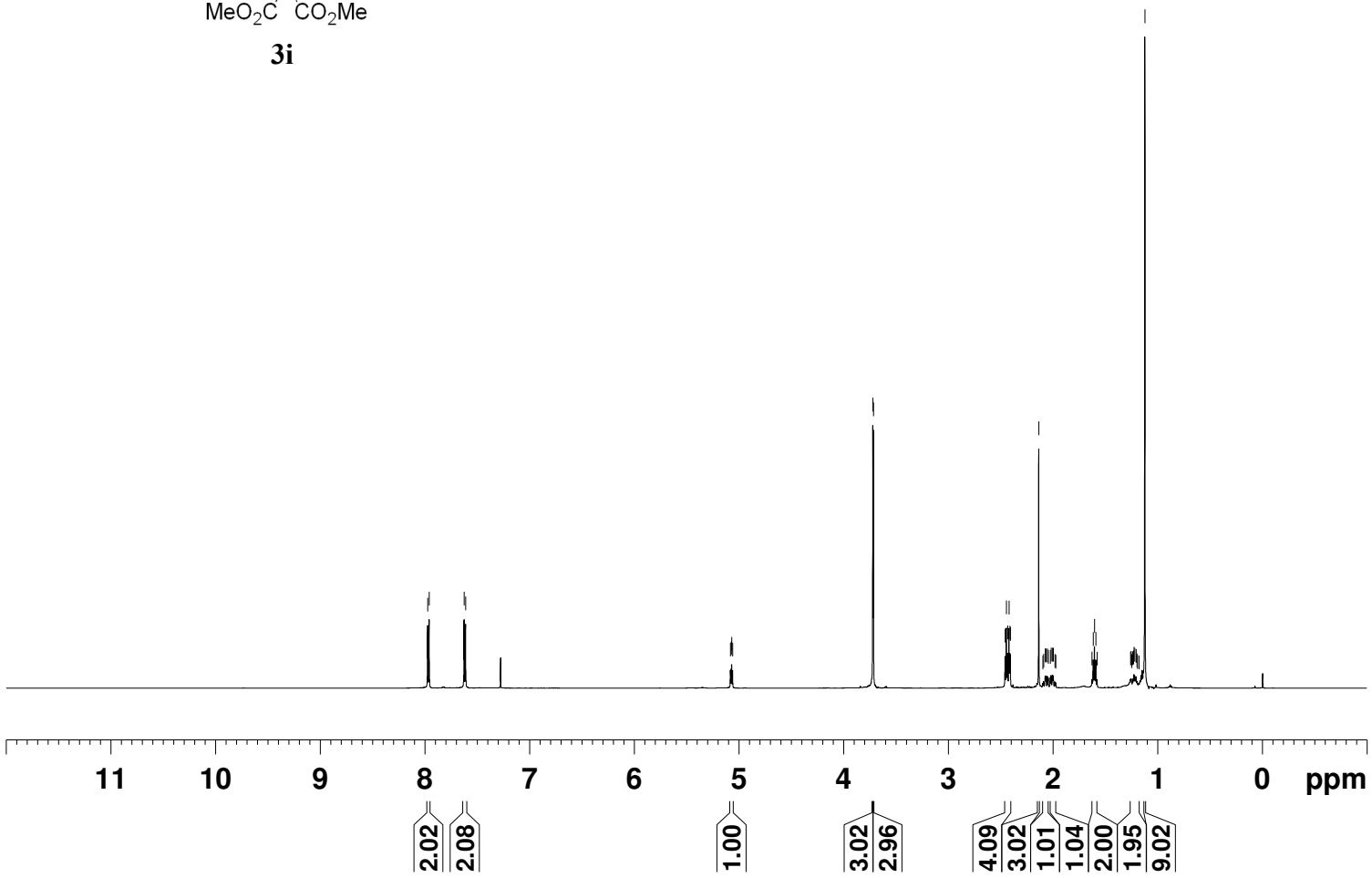



```
NAME      wll-547p-20210610
EXPNO                    2
PROCNO                   1
Date_             20210610
Time                 23.55
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      300
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                    296.8 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
```



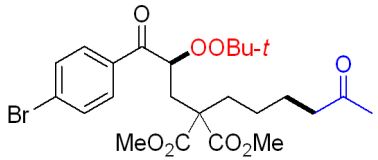


3i

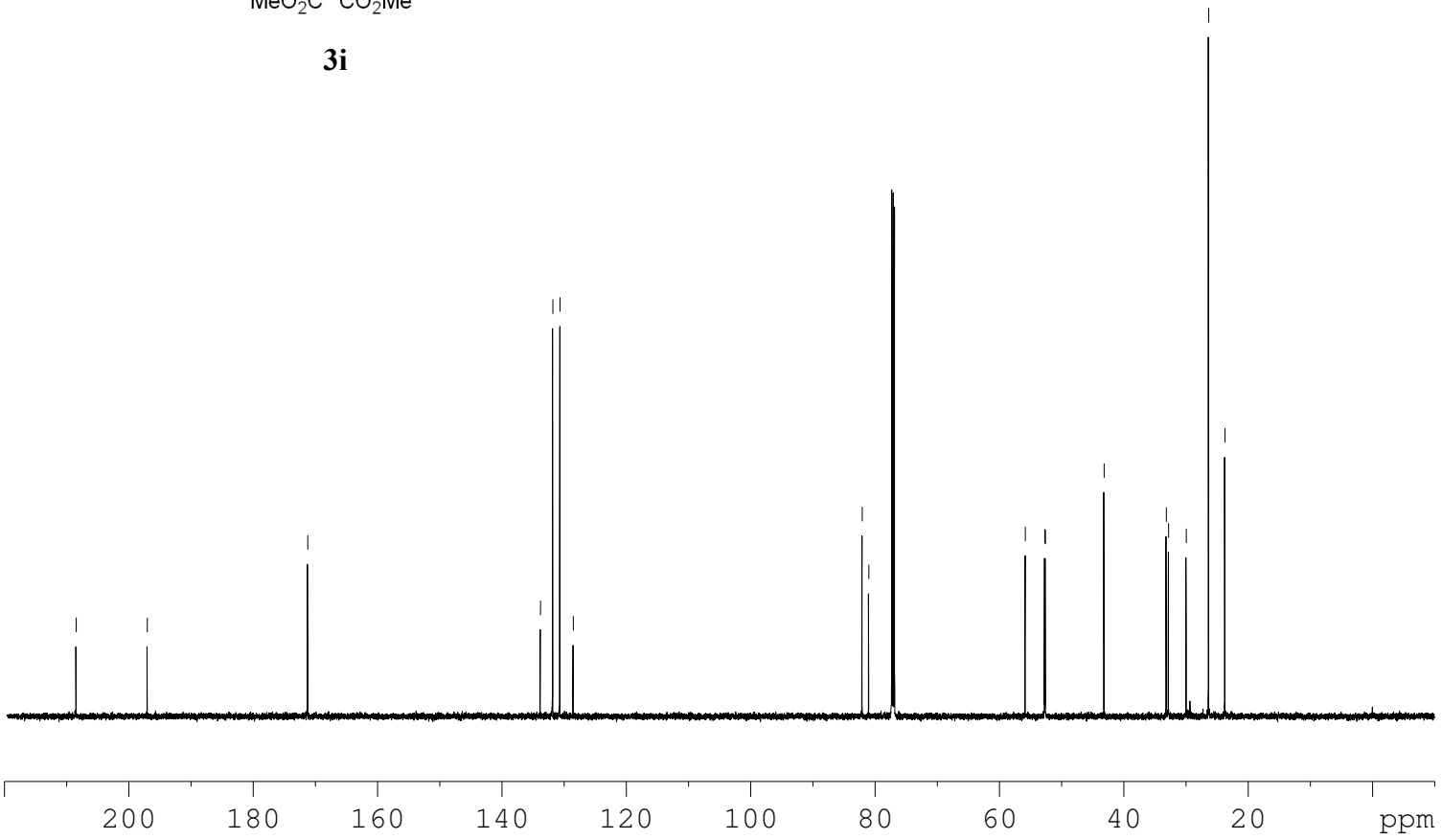



```
NAME      wll-553p-20210610
EXPNO                    1
PROCNO                   1
Date_             20210611
Time                  5.20
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       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                  295.6 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.96 usec
SI                   65536
SF            600.1700061 MHz
WDW                     EM
SSB                      0
LB                   0.30 Hz
GB                       0
PC                   1.00
```

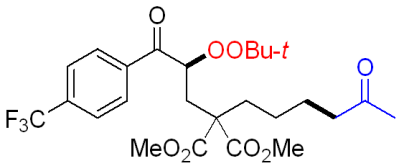
208.471
197.032
171.225
133.803
131.820
130.667
128.526
82.068
81.006
55.842
52.718
52.606
43.169
33.164
32.806
29.956
26.362
23.743



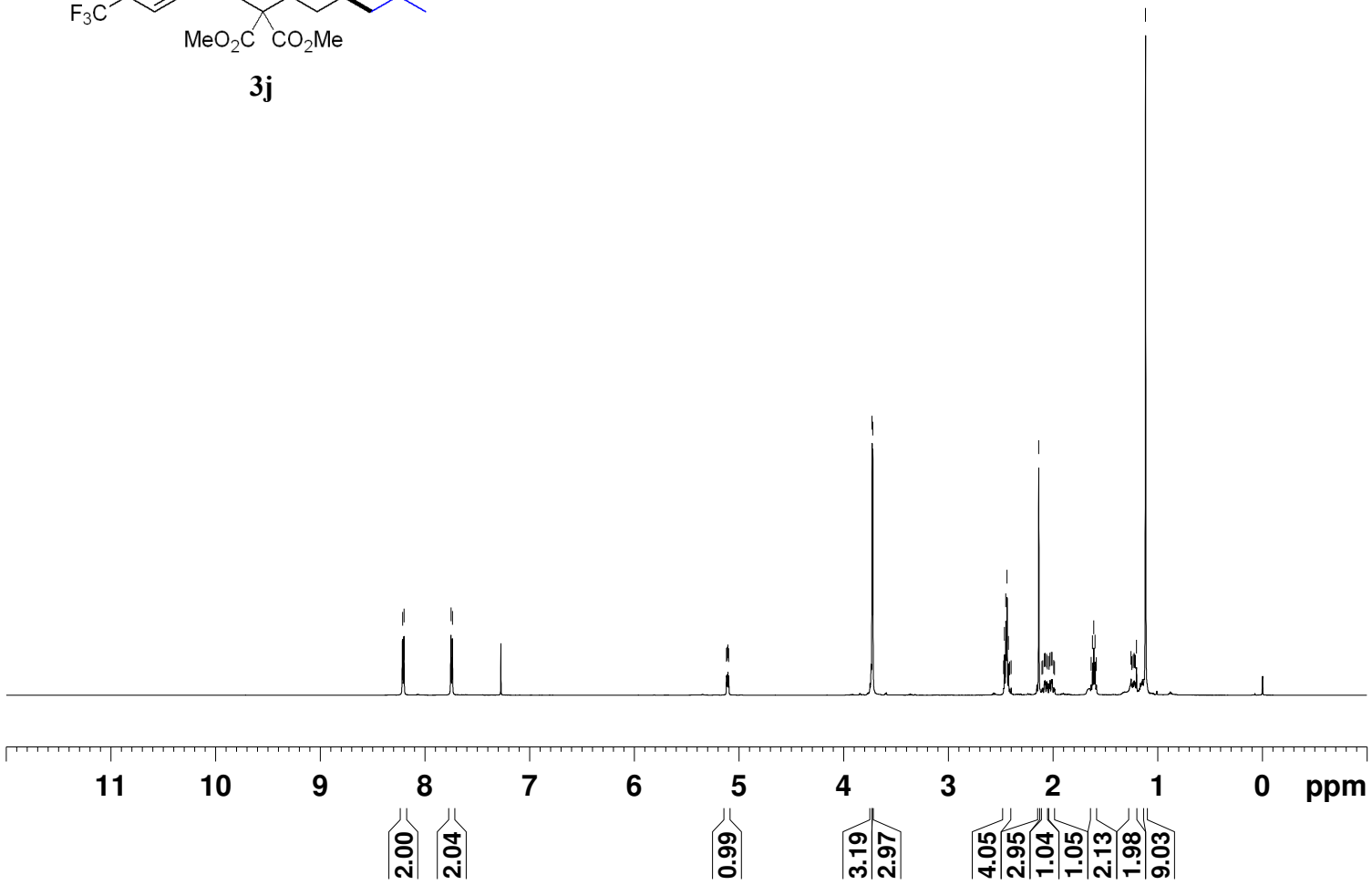
3i



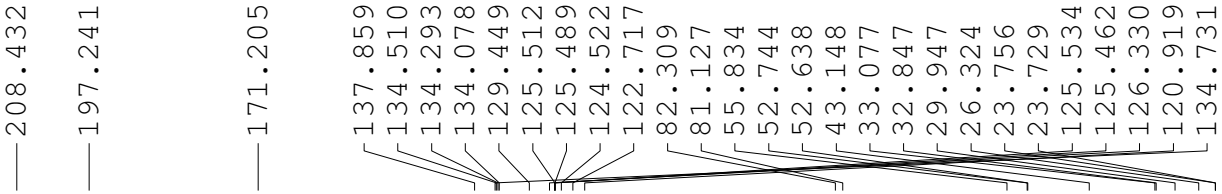
NAME	wll-553p-20210610
EXPNO	2
PROCNO	1
Date_	20210611
Time	5.36
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	300
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	296.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== 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

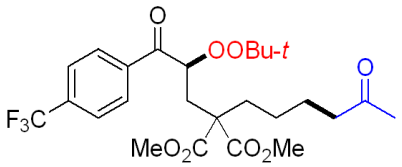


3j

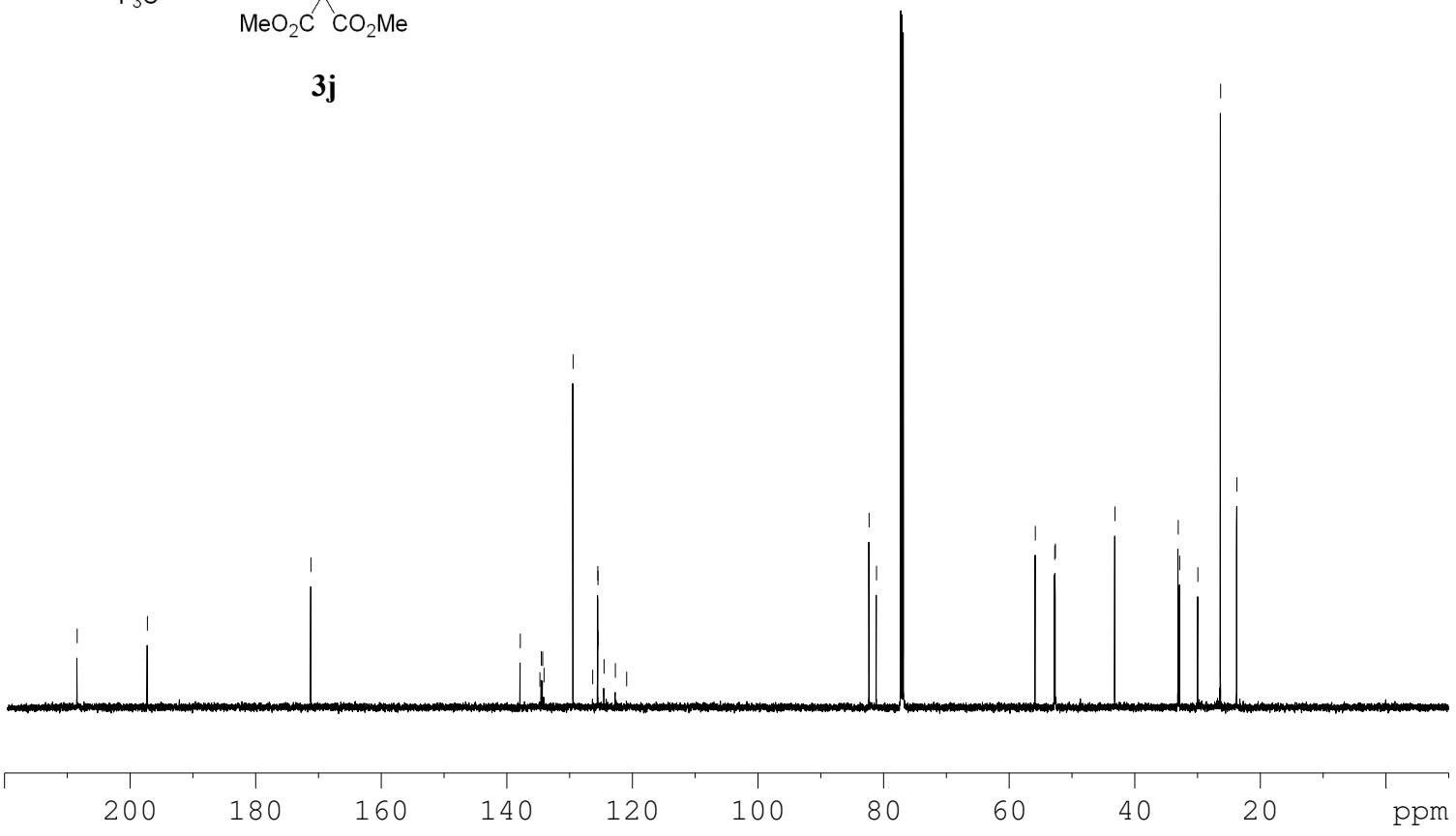



```
NAME      wll-557p-20210618
EXPNO                    1
PROCNO                   1
Date_             20210618
Time                 10.01
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       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                  295.2 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.96 usec
SI                   65536
SF            600.1700077 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

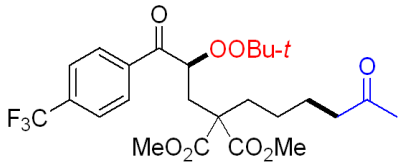




3j

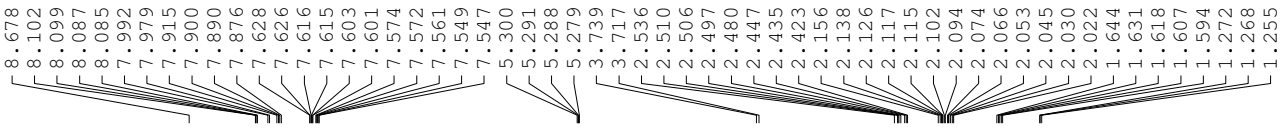


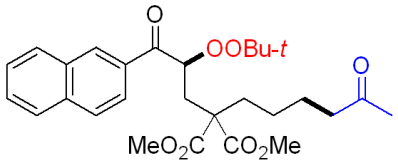
NAME	wll-557p-20210618
EXPNO	2
PROCNO	1
Date_	20210618
Time	12.52
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	300
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	296.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== 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



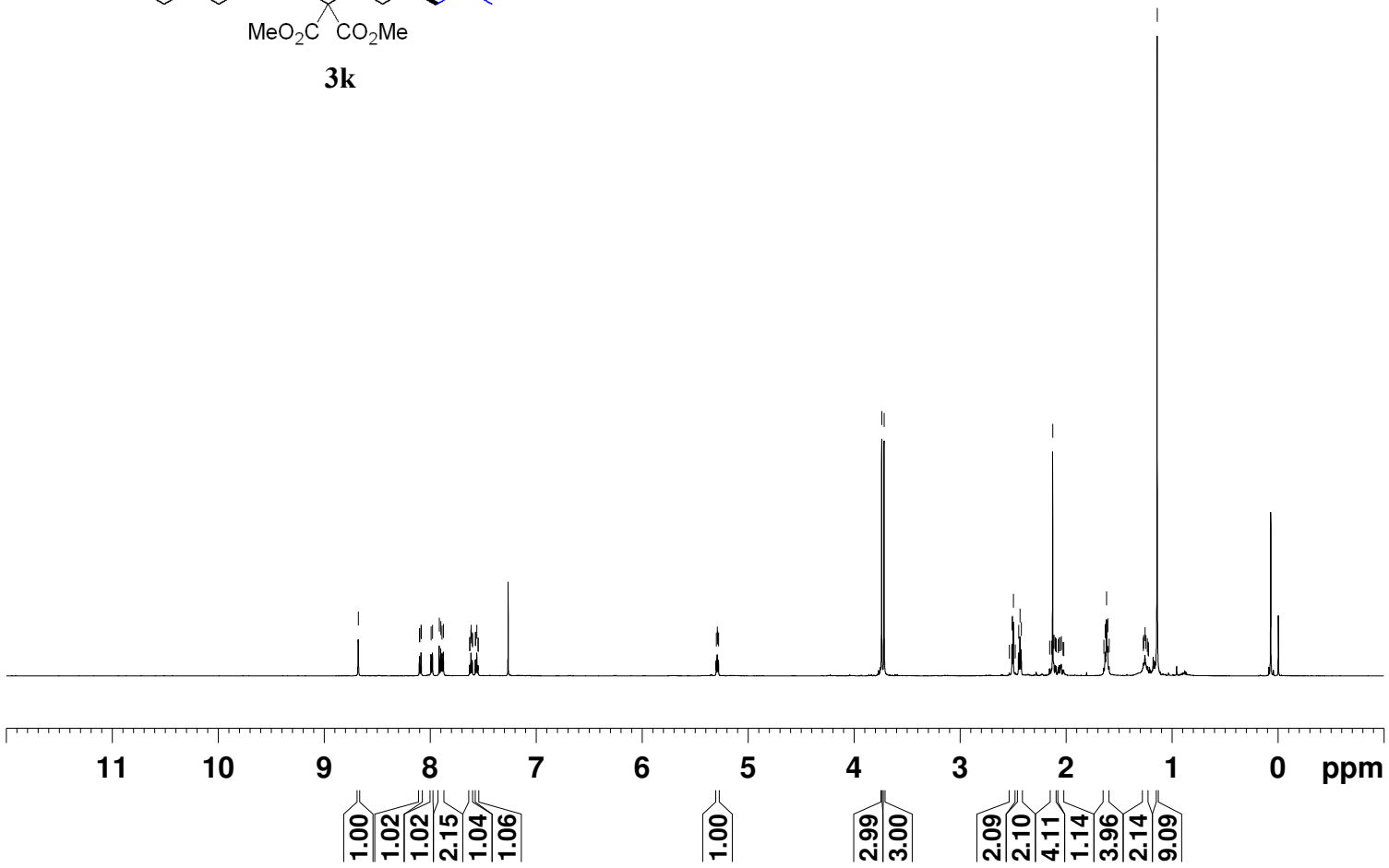
3j


```
NAME      wll-557p-20210730
EXPNO                    1
PROCNO                   1
Date_             20210730
Time                 10.52
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                   17.32
DW                   3.733 usec
DE                    6.50 usec
TE                  298.2 K
D1             1.00000000 sec
D11            0.03000000 sec
D12            0.00002000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          564.6675534 MHz
NUC1                   19F
P1                   25.77 usec
SI                   65536
SF            564.7240258 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

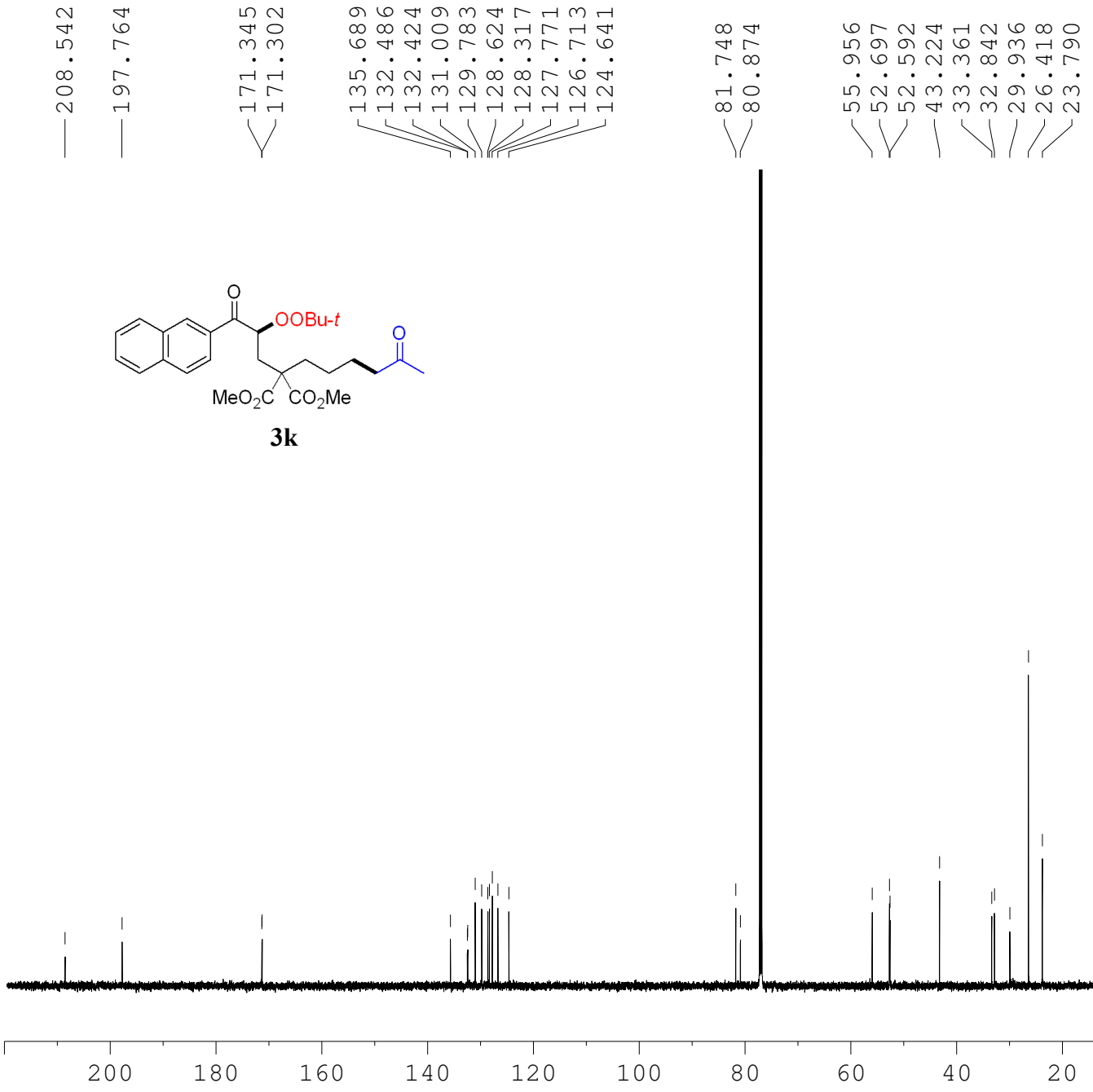




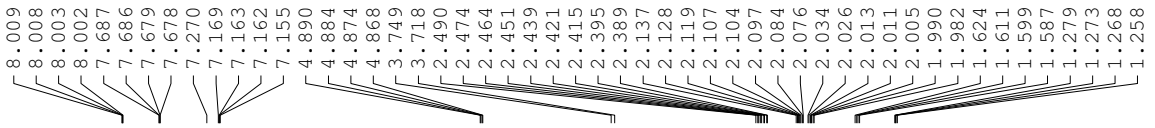
3k

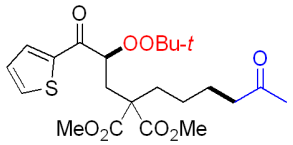



```
NAME      wll-551p-20210610
EXPNO                    1
PROCNO                   1
Date_             20210611
Time                  4.35
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       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                  295.4 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.96 usec
SI                  65536
SF            600.1700132 MHz
WDW                     EM
SSB                      0
LB                   0.30 Hz
GB                       0
PC                   1.00
```

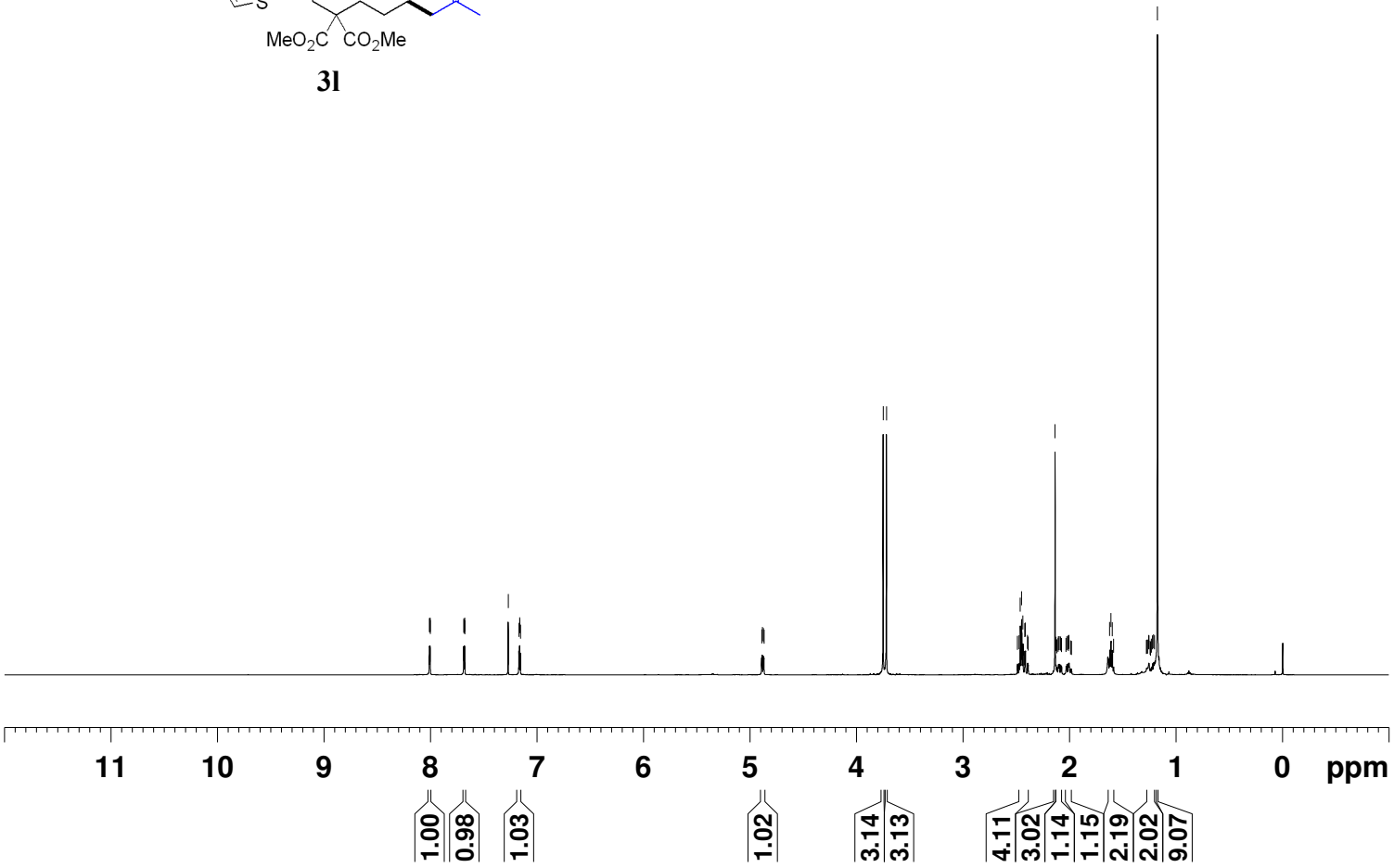



```
NAME      wll-551p-20210610
EXPNO                    2
PROCNO                   1
Date_             20210611
Time                 4.56
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   400
DS                     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                296.7 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           150.9128679 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



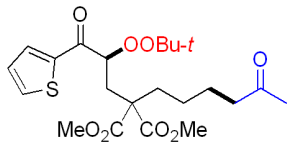


3l




```
NAME      wll-558p-20210618
EXPNO                    1
PROCNO                   1
Date_             20210618
Time                 10.05
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                  76.92
DW                 52.000 usec
DE                  6.50 usec
TE                 295.2 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                   1H
P1                  9.96 usec
SI                  65536
SF            600.1700100 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

208.522
191.445
171.187
171.173
140.672
134.267
133.714
128.072
83.330
81.188
55.778
52.736
52.603
43.226
33.790
32.450
29.956
26.412
23.771
23.748



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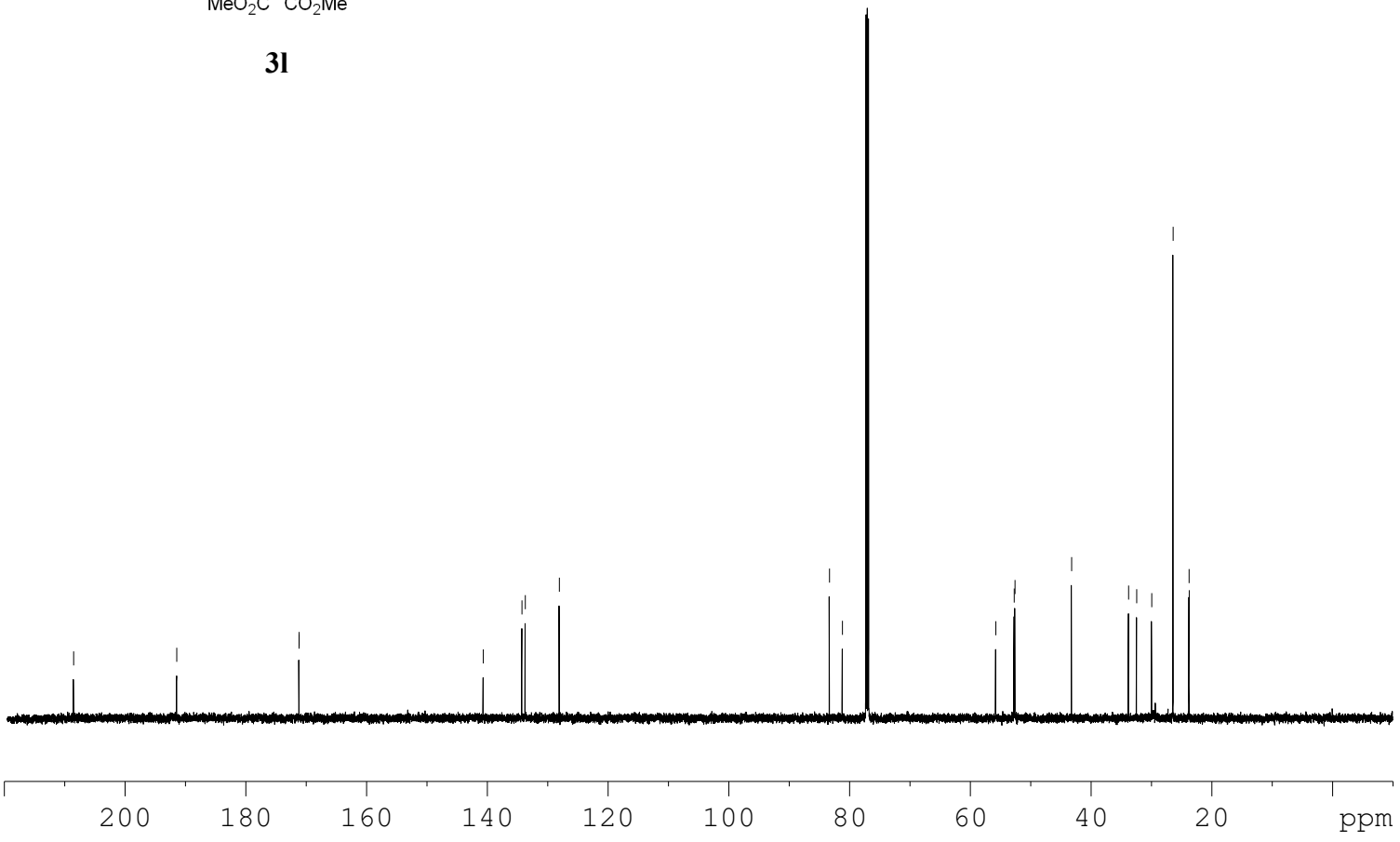
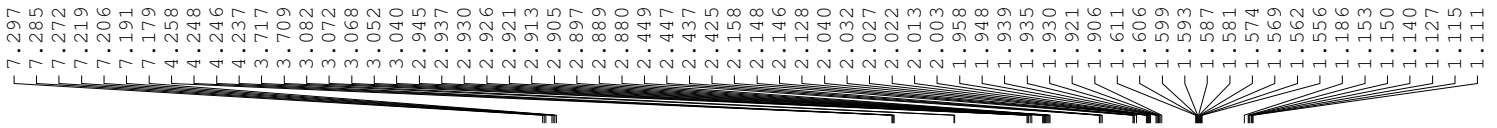
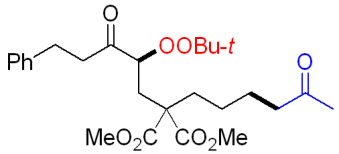
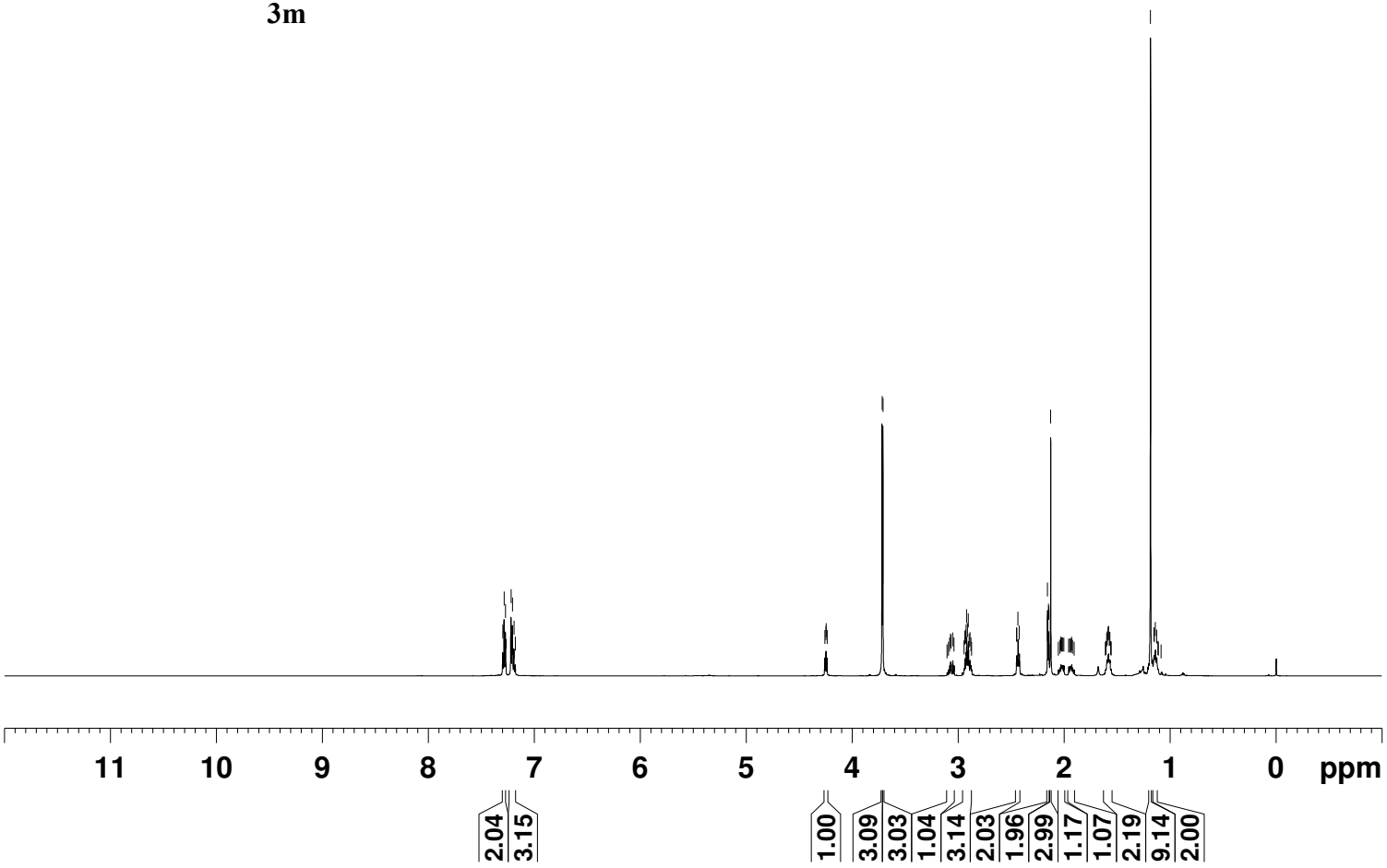


NAME	wll-558p-20210618
EXPNO	2
PROCNO	1
Date_	20210618
Time	13.05
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	200
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	296.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== 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

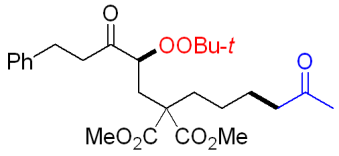




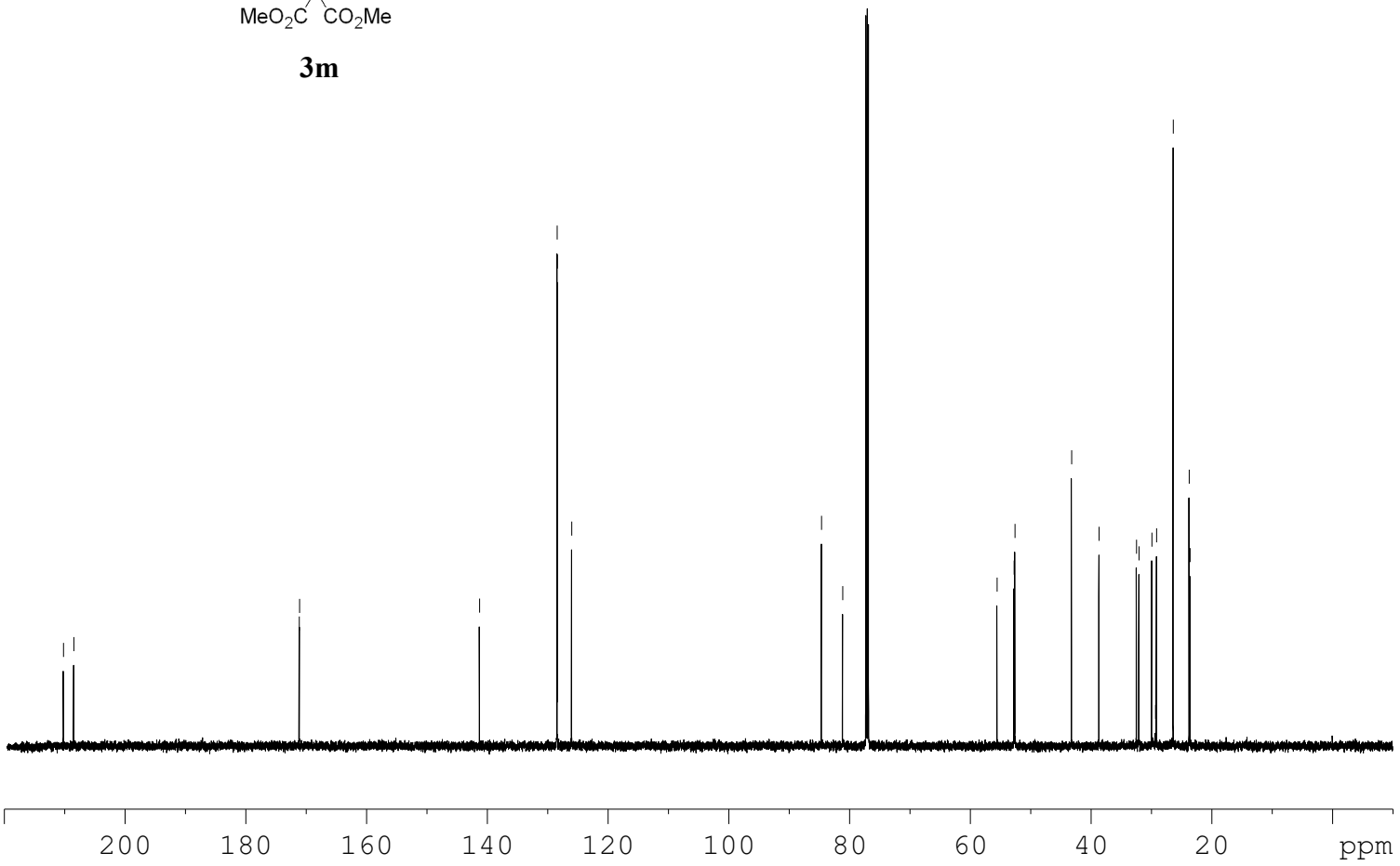
3m



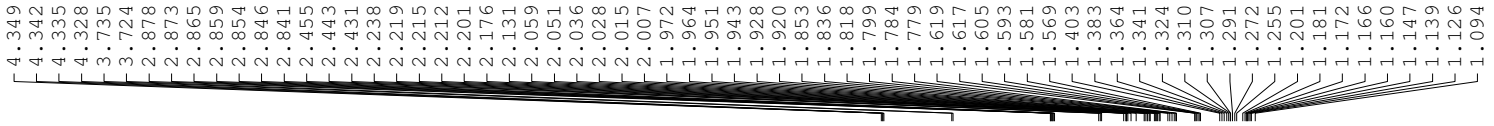

```
NAME      wll-559p-20210618
EXPNO                    1
PROCNO                   1
Date_             20210618
Time                 10.09
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               9615.385 Hz
FIDRES            0.146719 Hz
AQ                3.4079220 sec
RG                   50.85
DW                  52.000 usec
DE                   6.50 usec
TE                  295.3 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           600.1739011 MHz
NUC1                    1H
P1                   9.96 usec
SI                   65536
SF             600.1700110 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

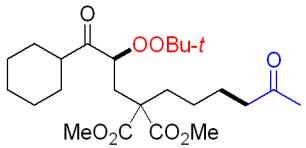


3m



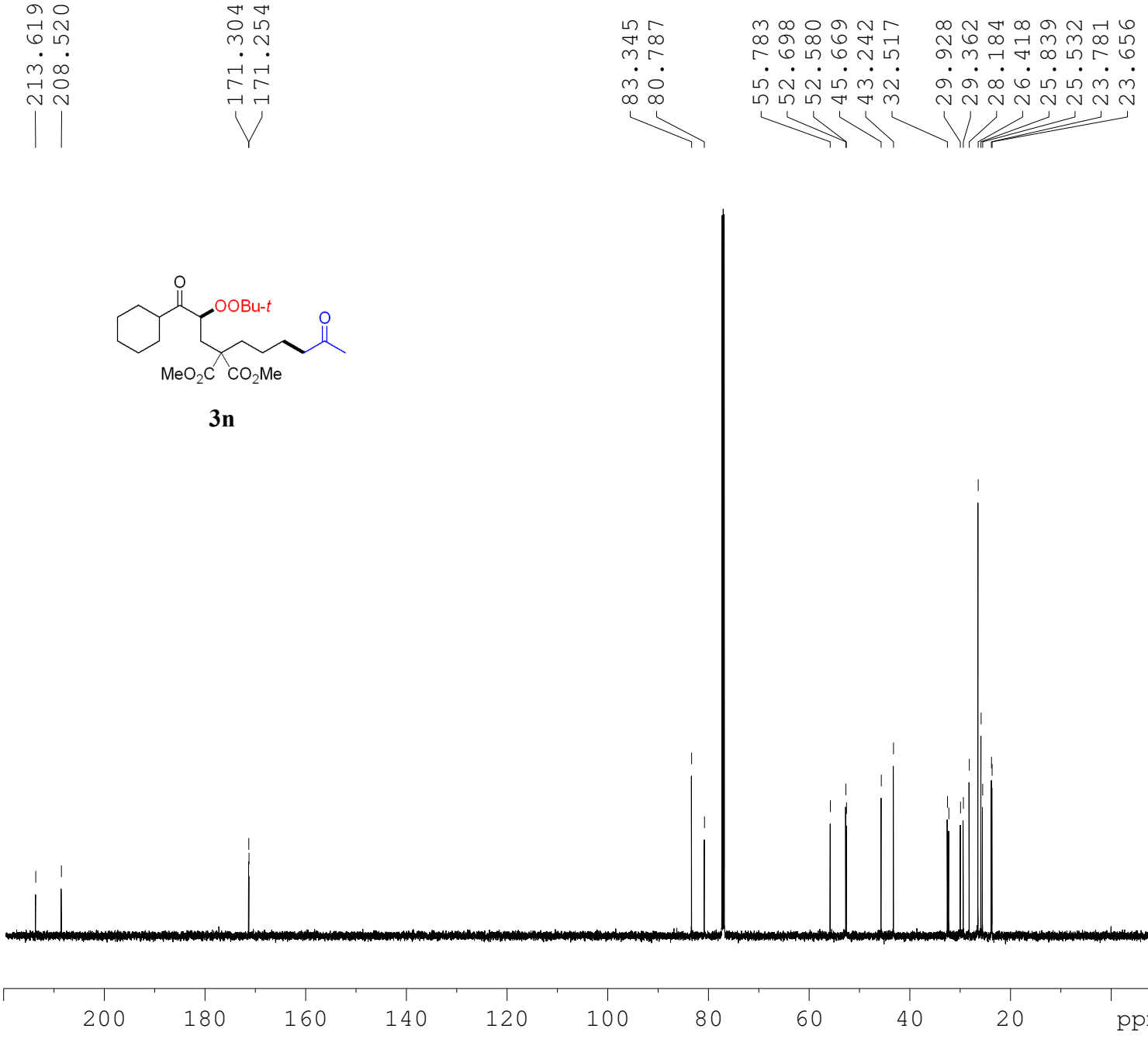

```
NAME      wll-559p-20210618
EXPNO                    2
PROCNO                   1
Date_             20210618
Time                13.19
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     200
DS                       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                   296.9 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
```



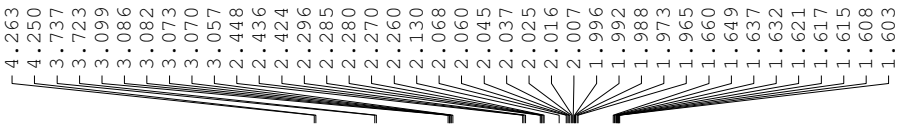


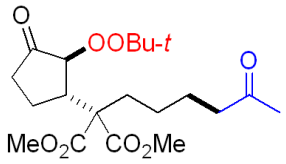
3n


```
NAME      wll-556p-20210618
EXPNO                    1
PROCNO                   1
Date_             20210618
Time                 9.57
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                 295.2 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.96 usec
SI                  65536
SF            600.1700078 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

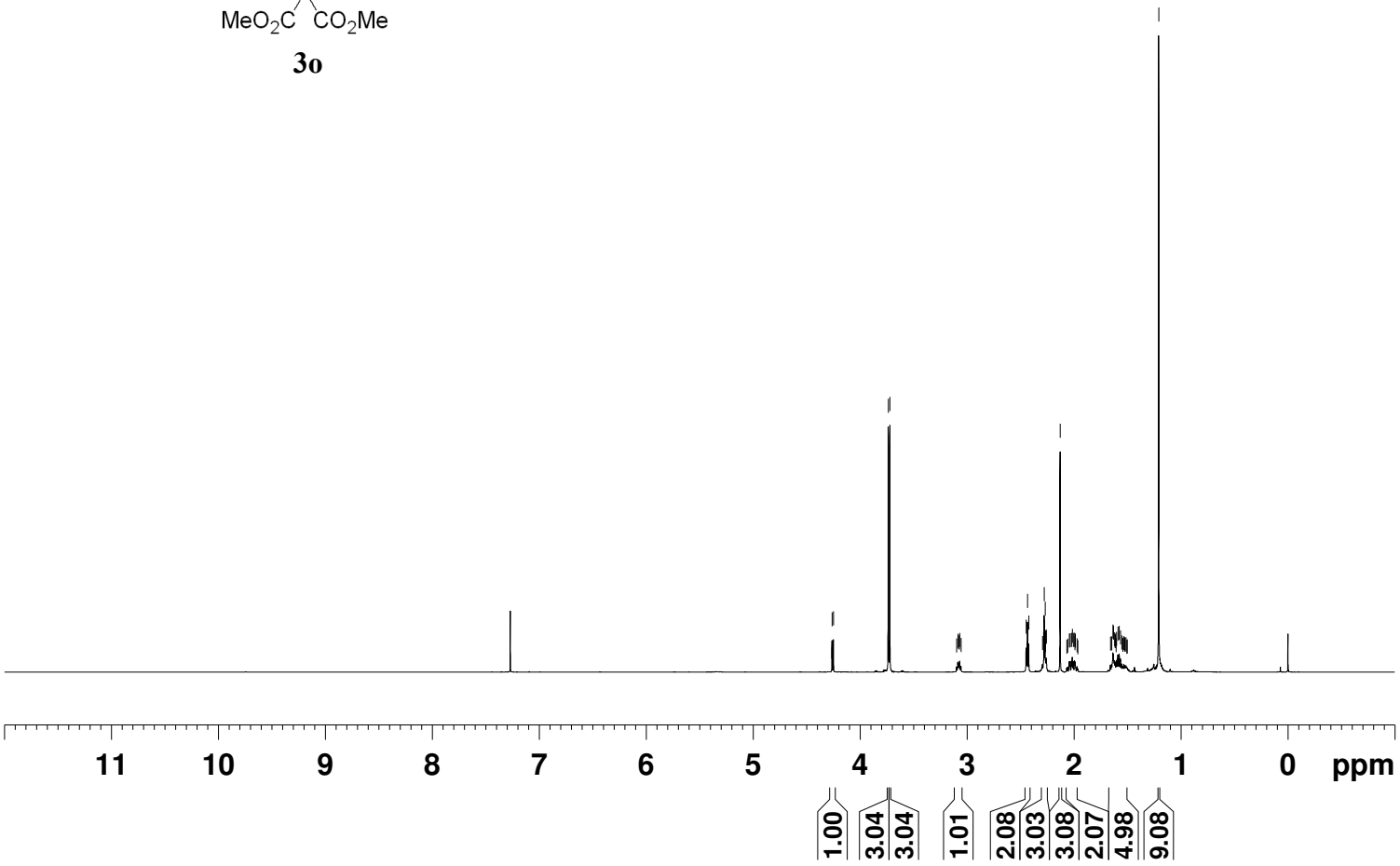



```
NAME      wll-556p-20210618
EXPNO                 2
PROCNO                1
Date_          20210618
Time              12.33
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  200
DS                    4
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG               190.02
DW               13.867 usec
DE                 6.50 usec
TE                296.7 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
```

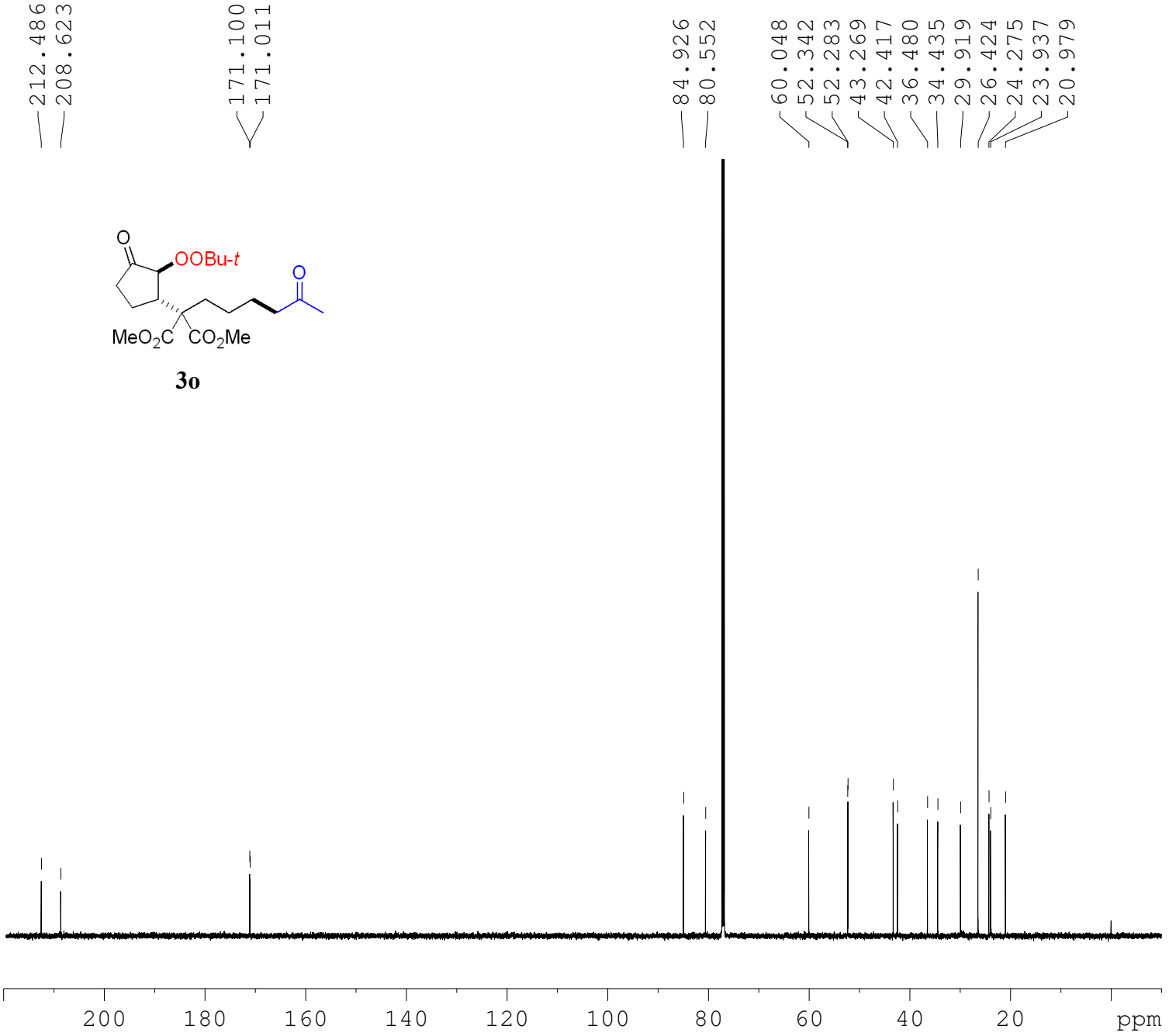




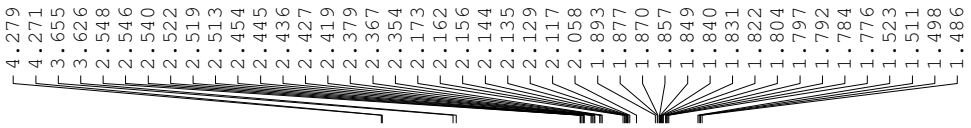
3o

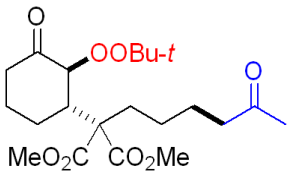



```
NAME      wll-554p-20210610
EXPNO                    1
PROCNO                   1
Date_             20210611
Time                  5.40
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH              9615.385 Hz
FIDRES           0.146719 Hz
AQ               3.4079220 sec
RG                   76.92
DW                  52.000 usec
DE                   6.50 usec
TE                  295.6 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.96 usec
SI                   65536
SF            600.1700095 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



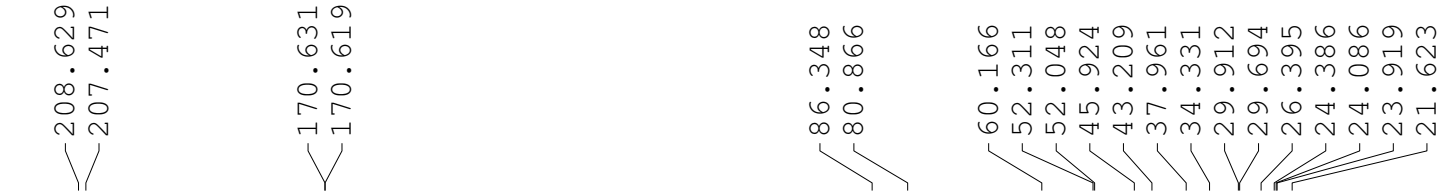
NAME	wll-554p-20210610
EXPNO	2
PROCNO	1
Date_	20210611
Time	6.11
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	600
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	296.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== 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

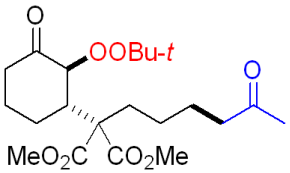




3p

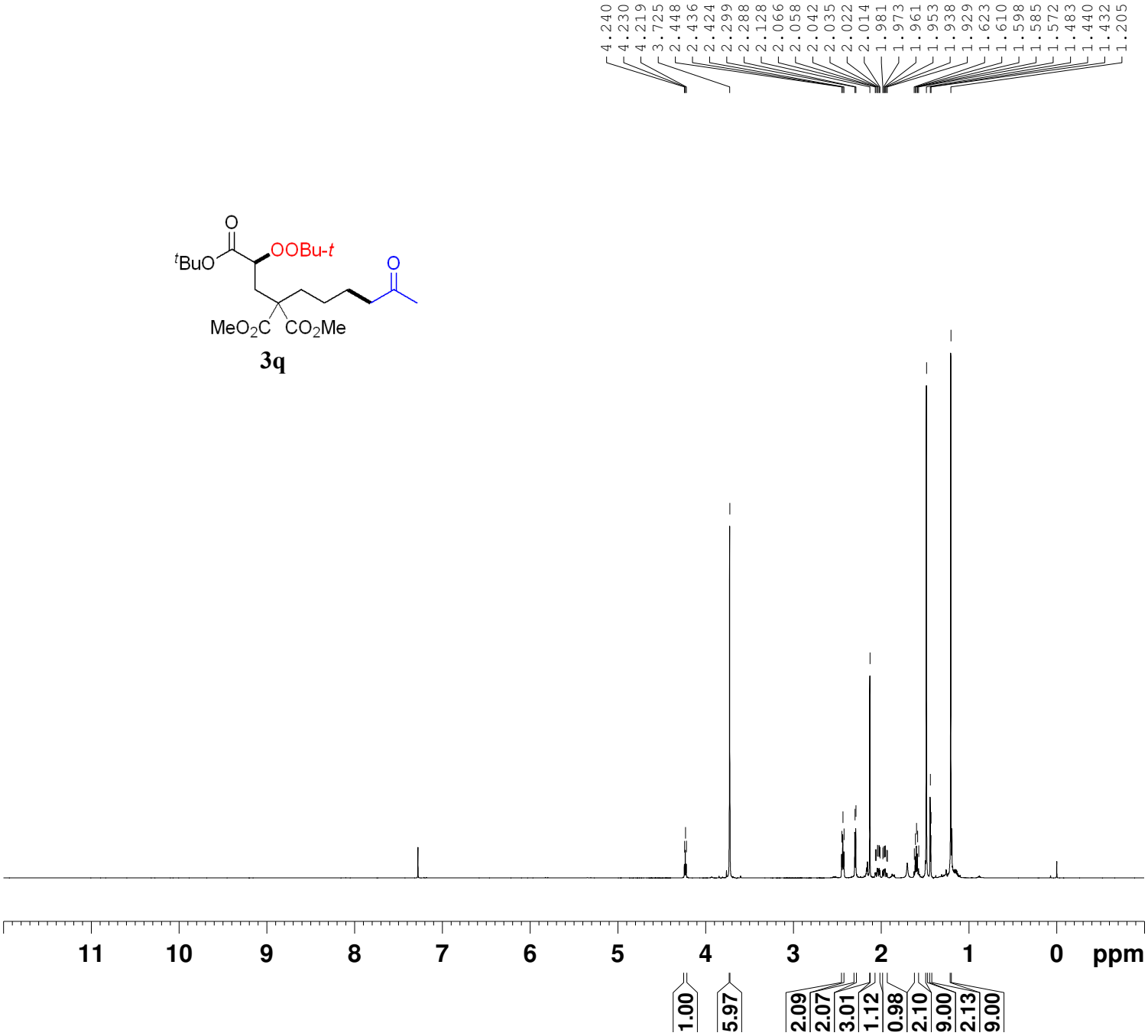

```
NAME      wll-592p-20210719
EXPNO                    1
PROCNO                   1
Date_             20210719
Time                 21.53
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       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                  297.0 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           600.1739011 MHz
NUC1                   1H
P1                   9.96 usec
SI                   65536
SF             600.1700488 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



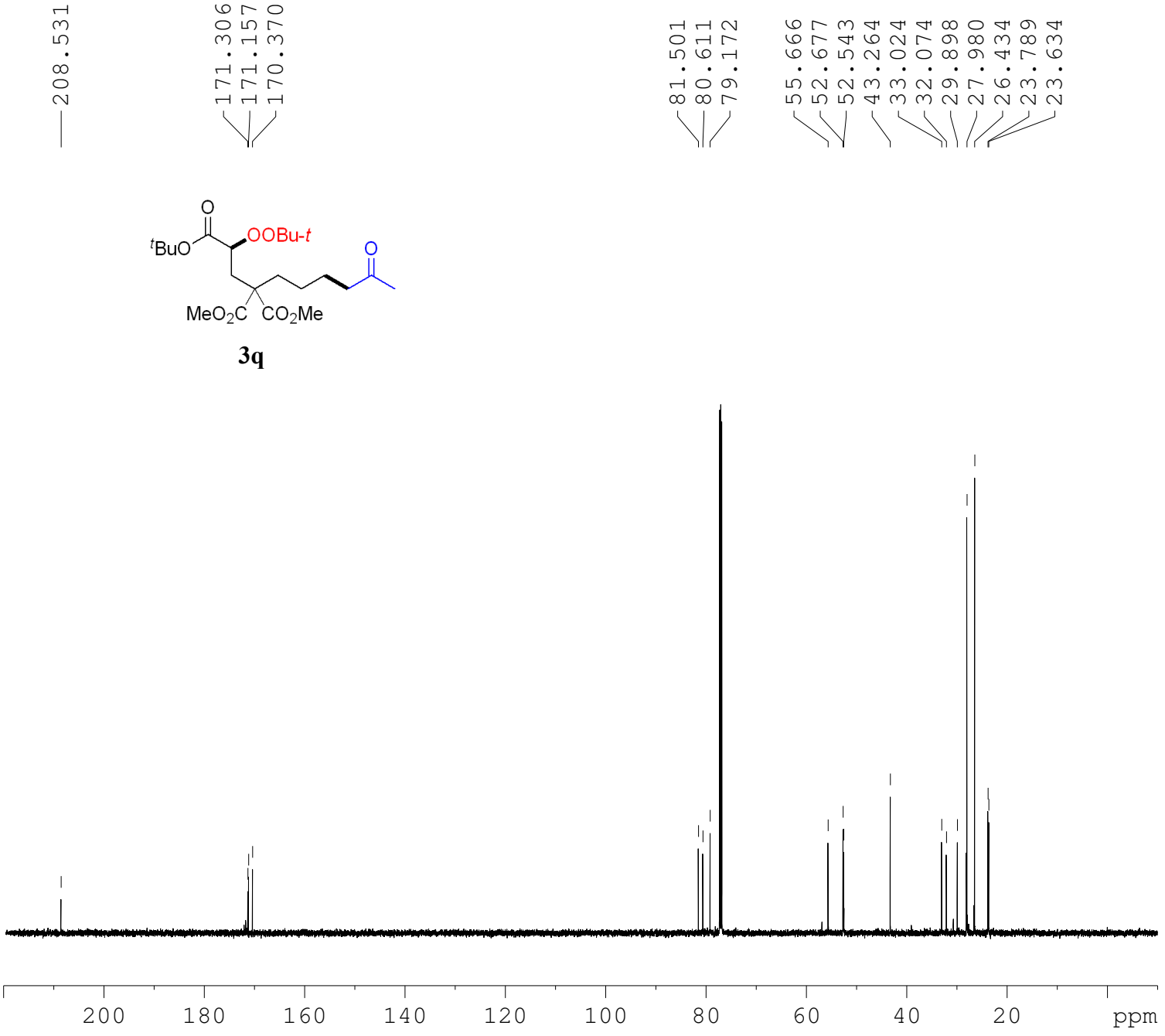


3p


```
NAME      wll-592p-20210719
EXPNO                    2
PROCNO                   1
Date_             20210719
Time                 22.56
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      200
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                    14.00 usec
SI                    32768
SF              150.9128665 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

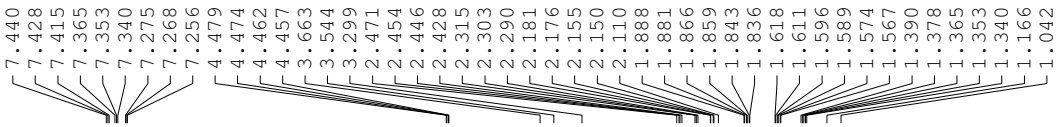


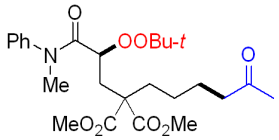

```
NAME      wll-564p-20210625
EXPNO                    1
PROCNO                   1
Date_             20210625
Time                 16.06
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       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                  296.5 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.96 usec
SI                   65536
SF            600.1700060 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



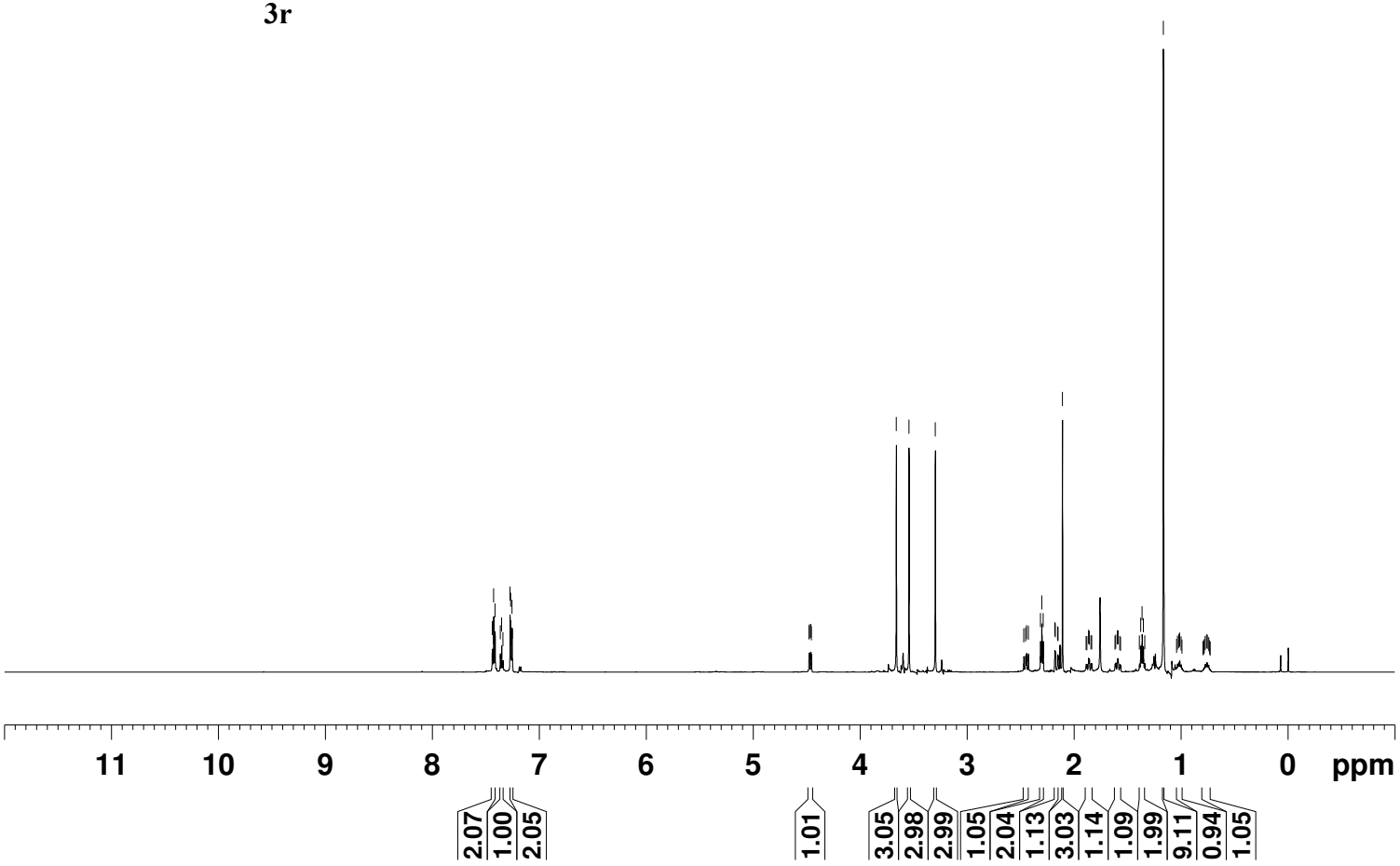
3q

NAME	wll-564p-20210625
EXPNO	2
PROCNO	1
Date_	20210625
Time	23.04
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	200
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	14.00 usec
SI	32768
SF	150.9128665 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

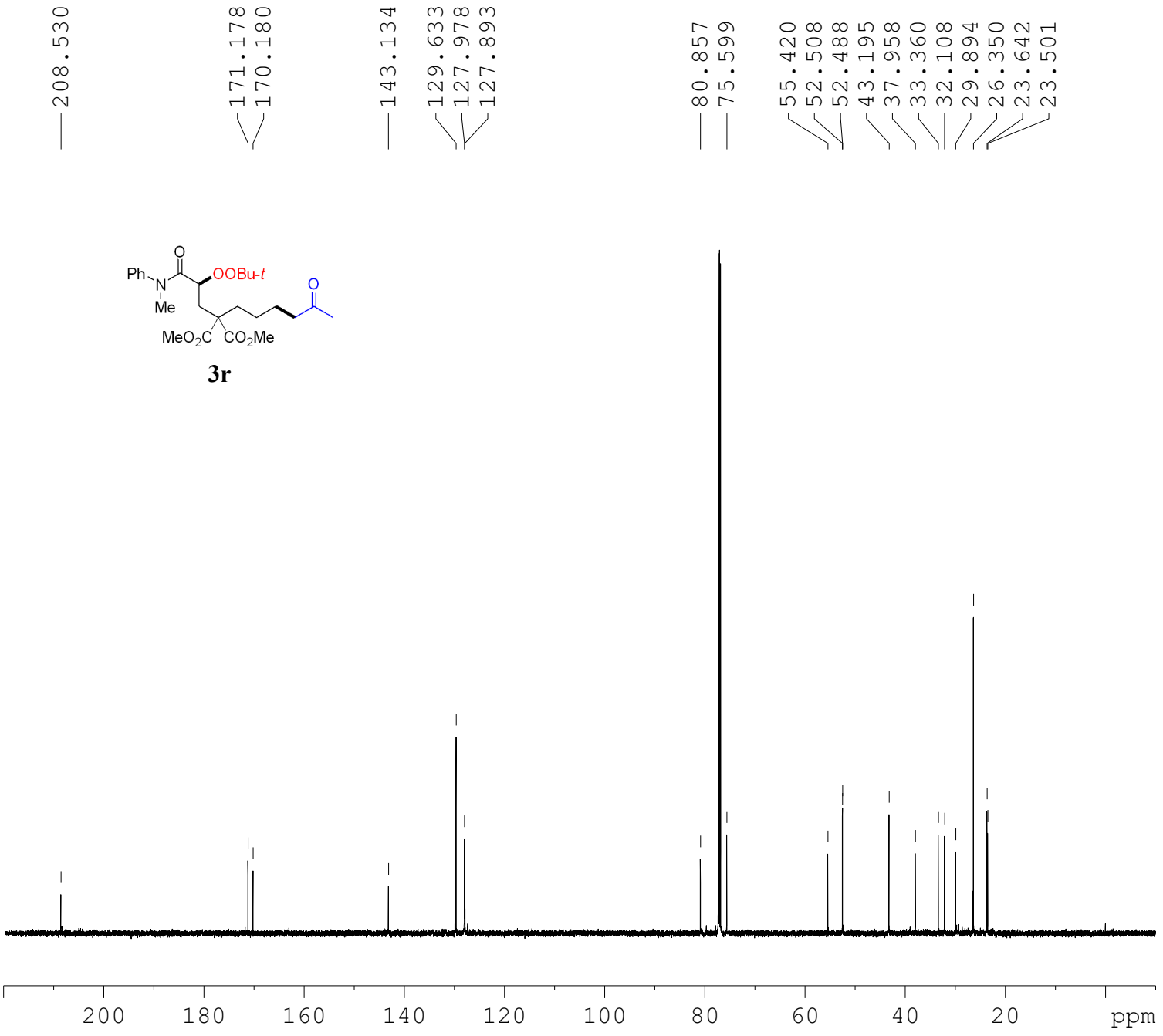




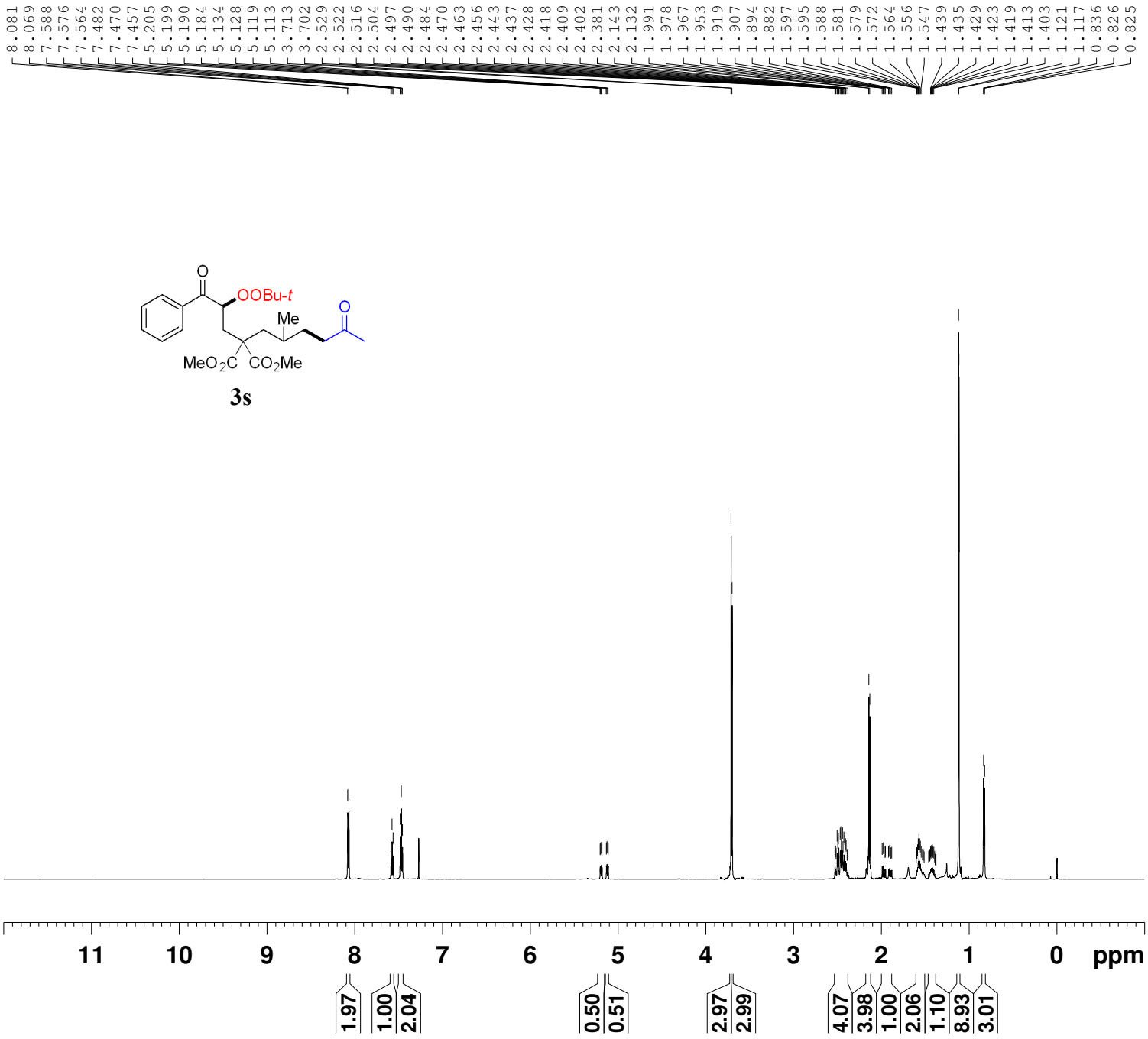
3r




```
NAME      wll-563p-20210625
EXPNO                    1
PROCNO                   1
Date_             20210625
Time                 16.02
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                   296.5 K
D1            1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                    9.96 usec
SI                   65536
SF            600.1700068 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

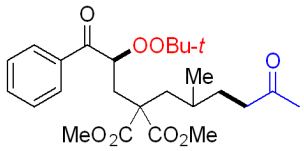



```
NAME      wll-563p-20210625
EXPNO                    2
PROCNO                   1
Date_             20210625
Time                 23.28
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      400
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                    14.00 usec
SI                    32768
SF            150.9128665 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

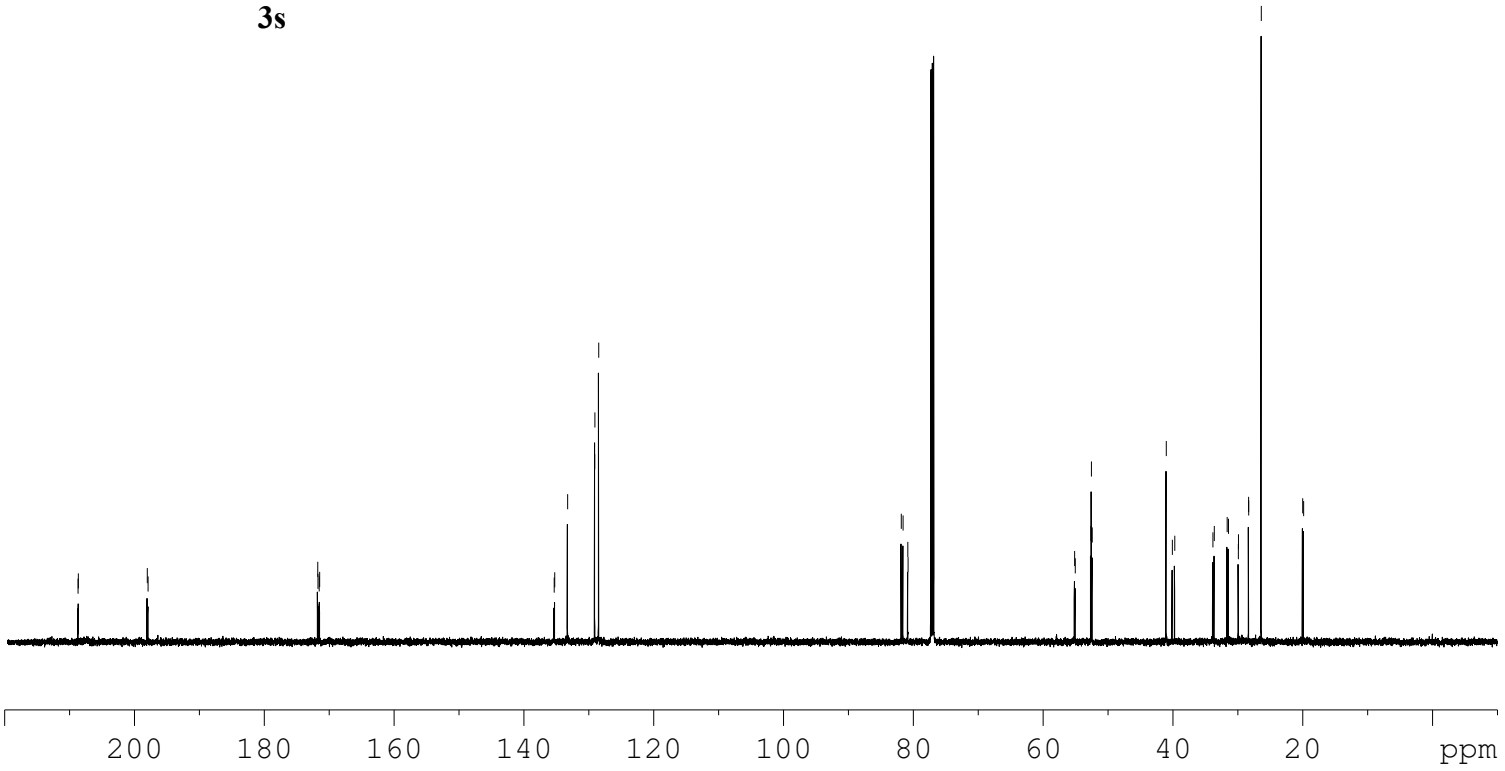



```
NAME      wll-560p-20210623
EXPNO                    1
PROCNO                   1
Date_             20210623
Time                 19.38
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               9615.385 Hz
FIDRES            0.146719 Hz
AQ                3.4079220 sec
RG                   50.85
DW                  52.000 usec
DE                   6.50 usec
TE                  297.4 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.96 usec
SI                   65536
SF            600.1700078 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

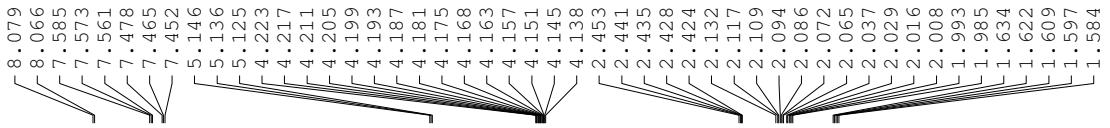
208.695
208.652
198.028
197.905
171.762
171.729
171.542
171.469
135.333
135.252
133.262
129.090
129.063
128.472
81.858
81.581
80.851
80.809
55.150
55.050
52.640
52.562
52.437
41.027
40.092
39.719
33.841
33.630
31.646
31.443
29.922
29.906
28.344
28.330
26.389

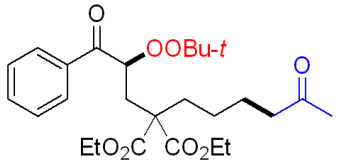


3s

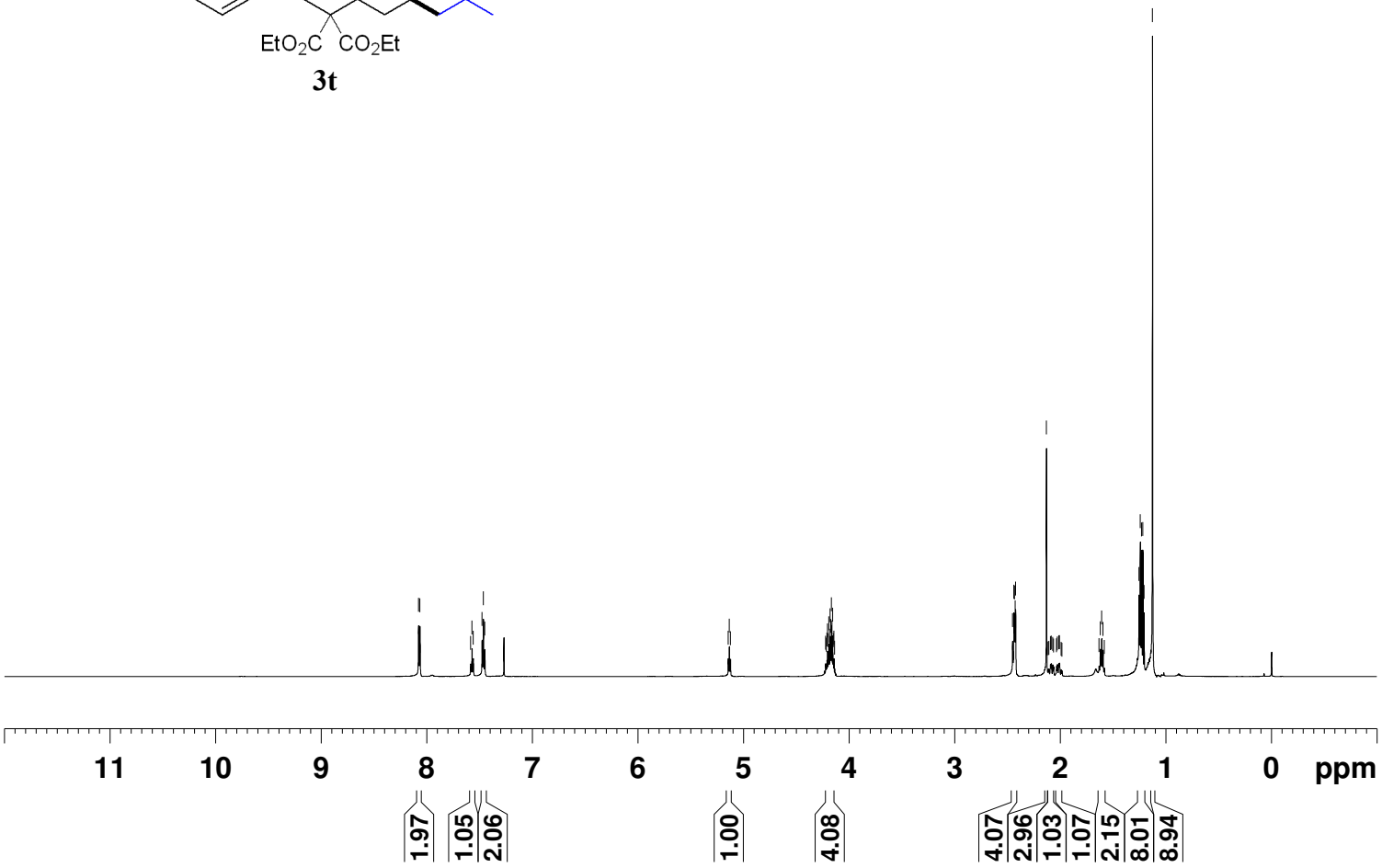


NAME	wll-560p-20210623
EXPNO	2
PROCNO	1
Date_	20210623
Time	20.24
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	240
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	298.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== 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



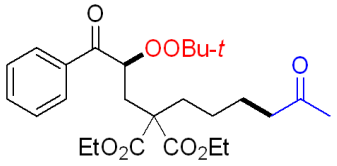


3t

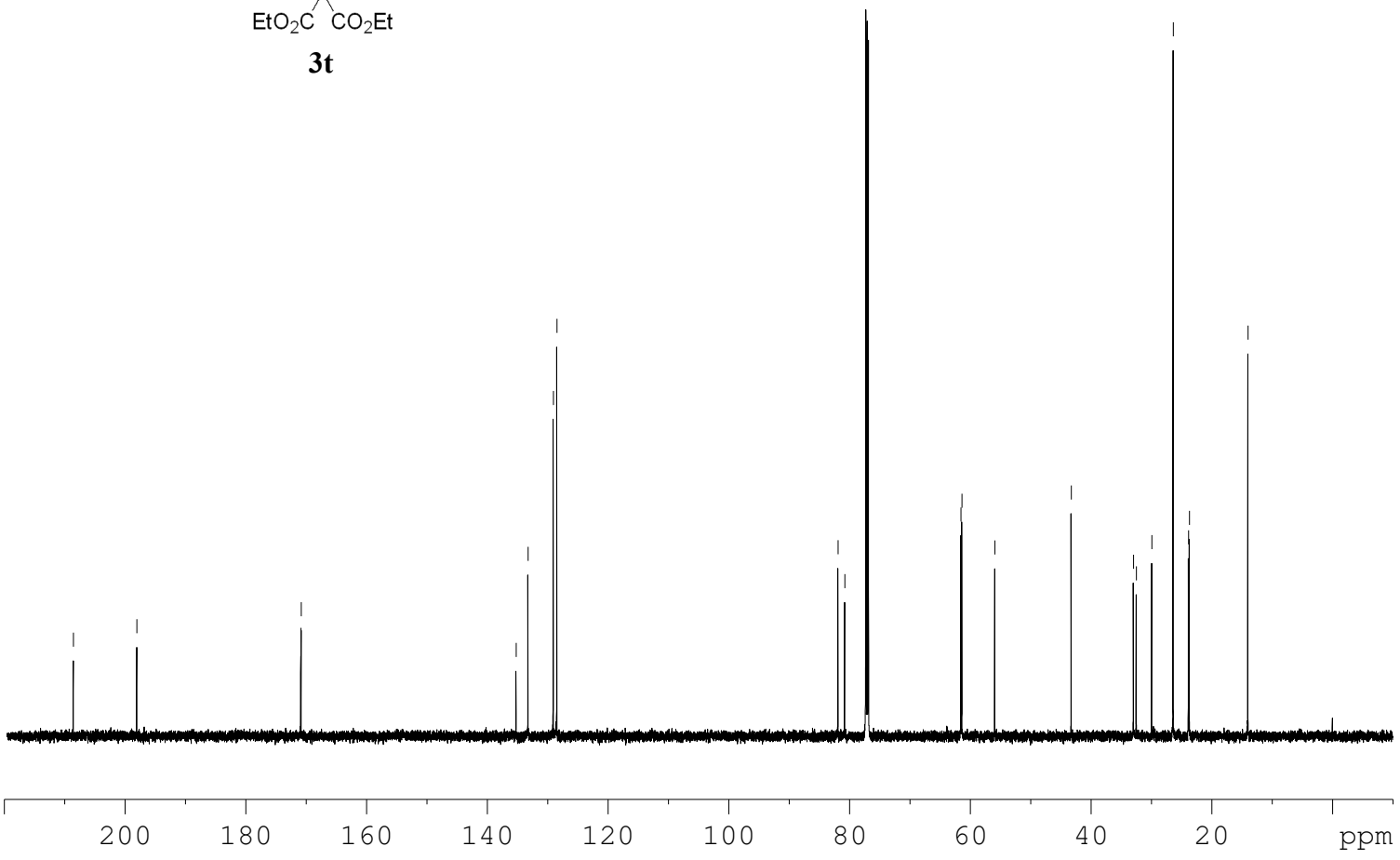



```
NAME      wll-550p-20210610
EXPNO                    1
PROCNO                   1
Date_             20210610
Time                 17.37
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       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                  295.6 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.96 usec
SI                   65536
SF            600.1700098 MHz
WDW                     EM
SSB                      0
LB                   0.30 Hz
GB                       0
PC                   1.00
```

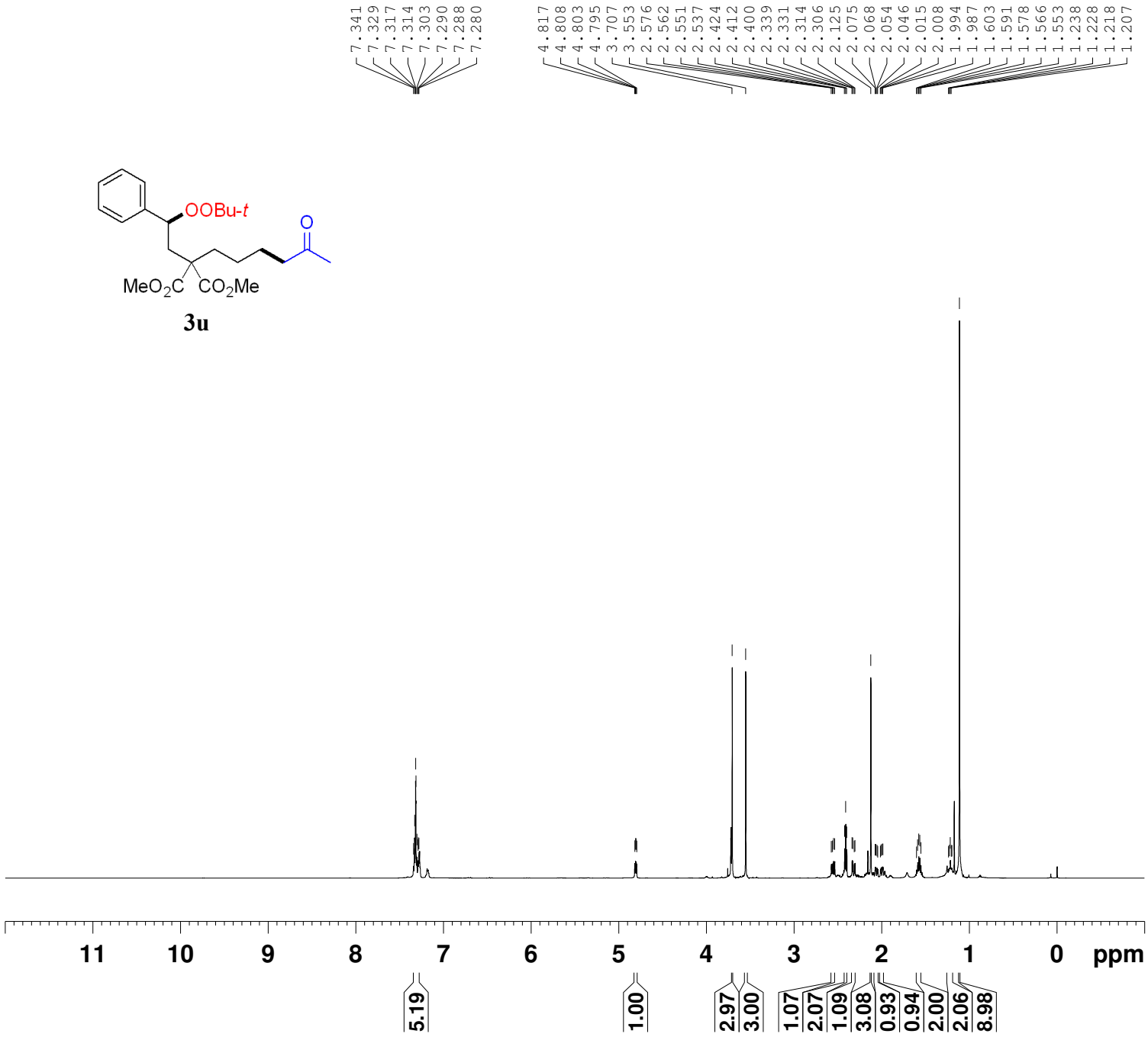
208.557
198.050
170.875
170.822
135.221
133.249
129.054
128.466
81.906
80.785
61.557
61.391
55.929
43.279
32.972
32.498
29.934
26.383
23.855
23.707
14.004



3t



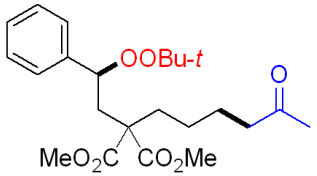
NAME	wll-550p-20210610
EXPNO	2
PROCNO	1
Date_	20210611
Time	0.51
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	300
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	296.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== 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



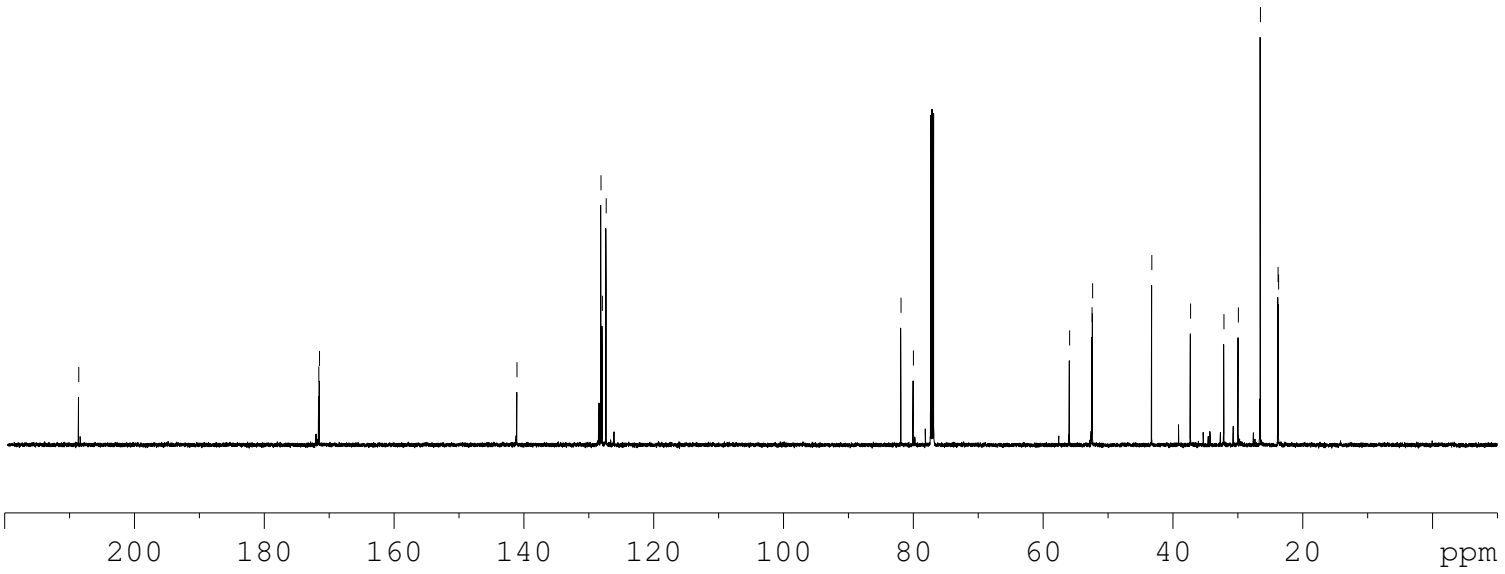
BRUKER

NAME	wll-540-7p-20210608
EXPNO	1
PROCNO	1
Date_	20210608
Time	15.05
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	295.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	600.1739011 MHz
NUC1	1H
P1	9.96 usec
SI	65536
SF	600.1700095 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

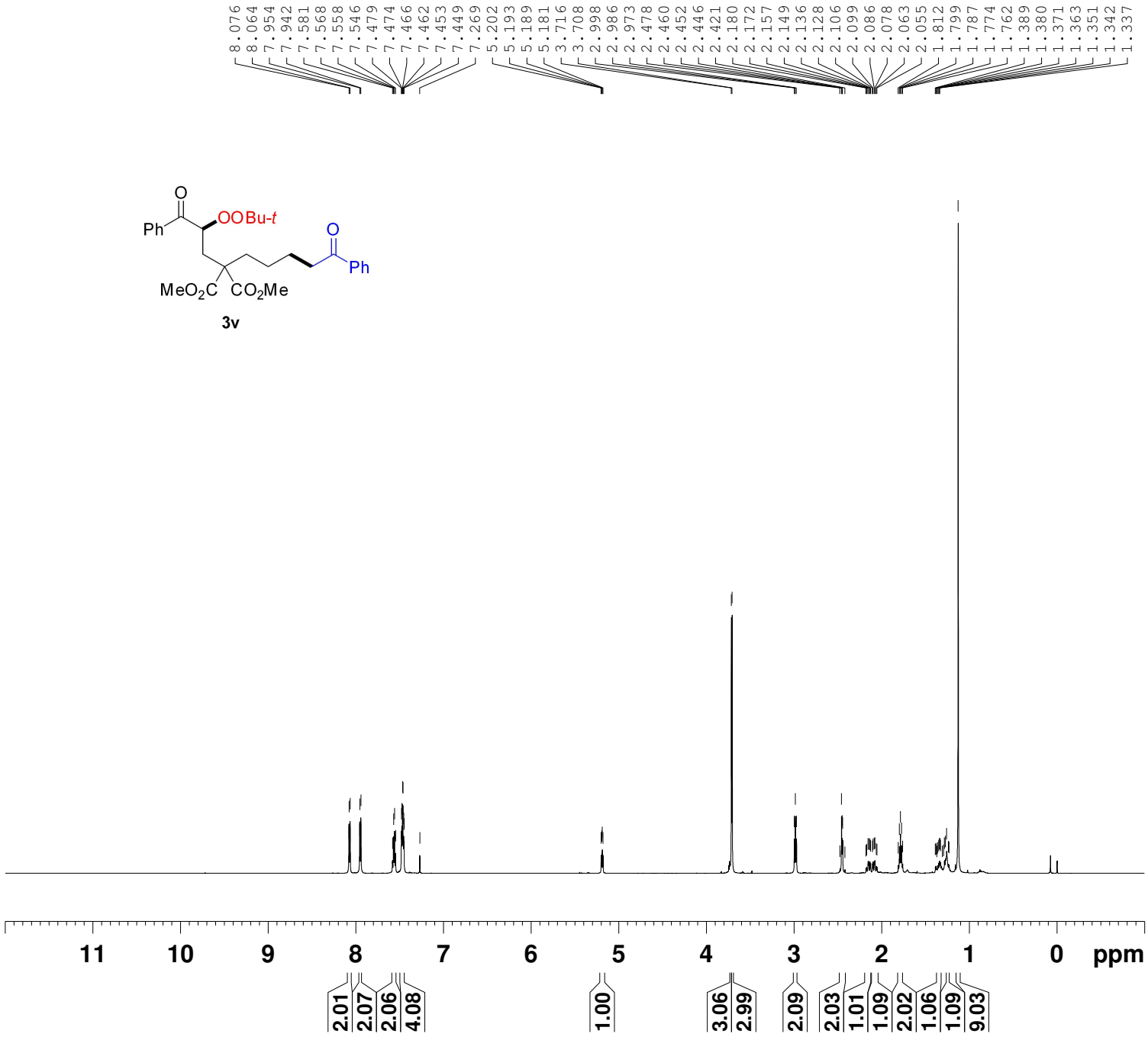
208.586
171.599
171.493
141.067
128.124
127.911
127.319
81.896
79.987
55.923
52.461
52.378
43.256
37.284
32.142
29.920
26.520
23.802
23.722



3u

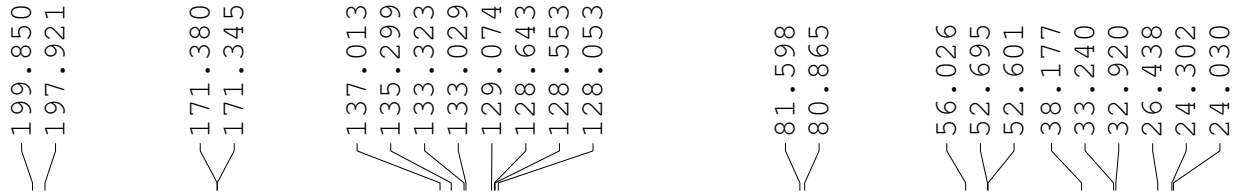


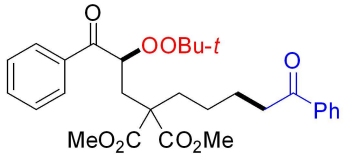
NAME	wll-540-7p-20210608
EXPNO	2
PROCNO	1
Date_	20210608
Time	15.21
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	300
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	296.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== 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



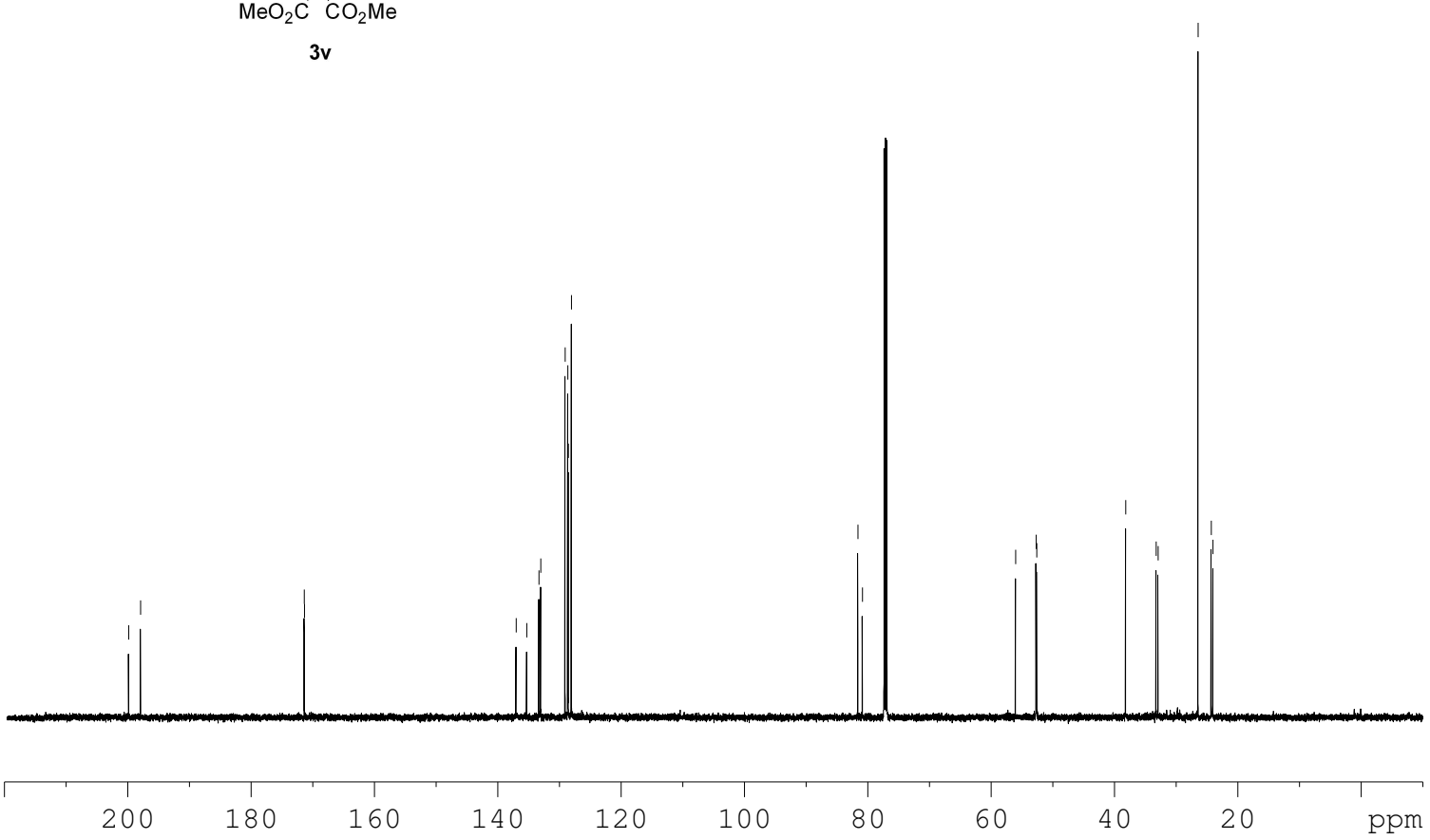
BRUKER

NAME	wll-695p-20211118
EXPNO	1
PROCNO	1
Date_	20211118
Time	21.33
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	44.5
DW	52.000 usec
DE	6.50 usec
TE	297.6 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
SFO1	600.1739011 MHz
NUC1	1H
P1	9.96 usec
SI	65536
SF	600.1700093 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

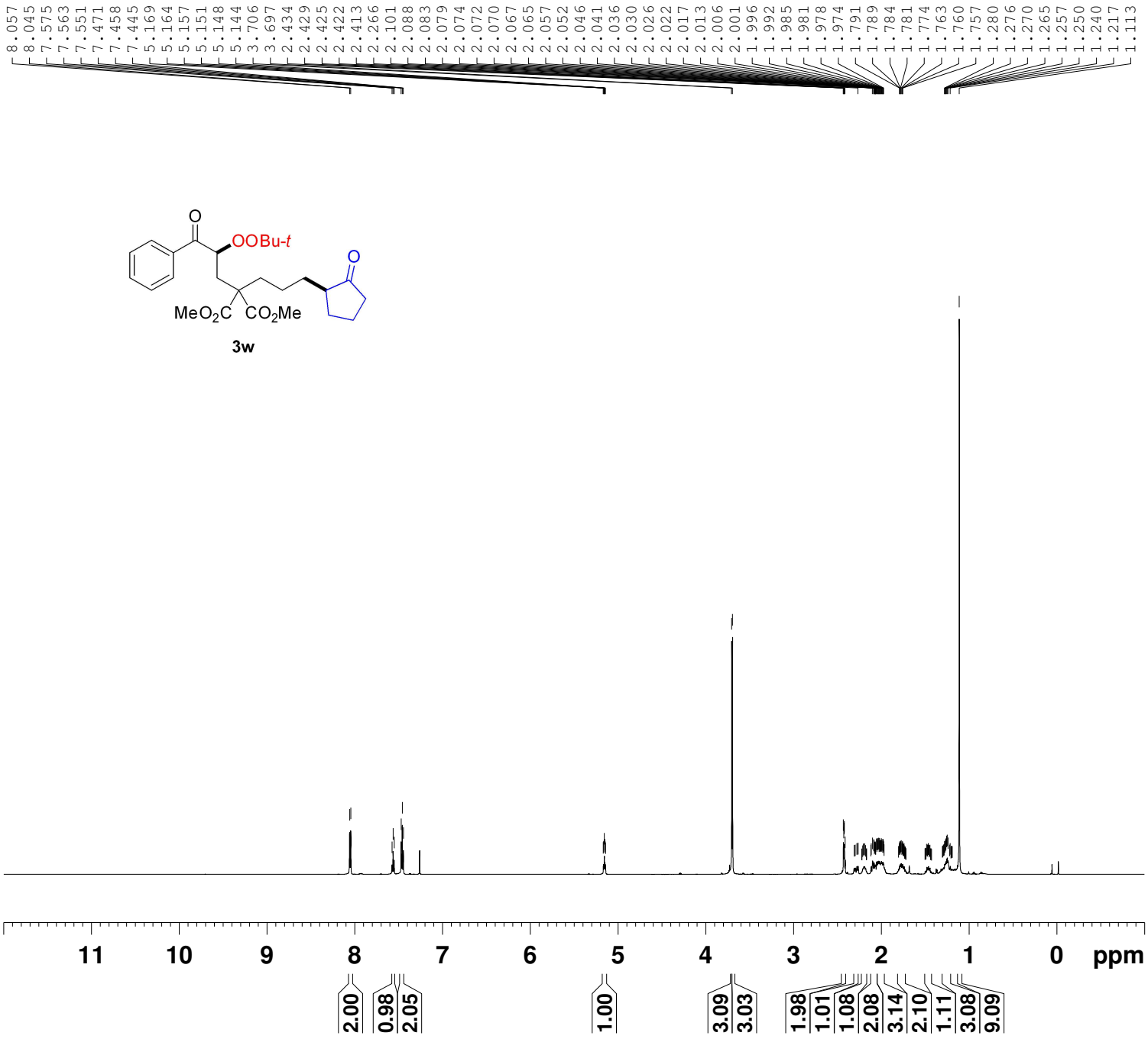




3v

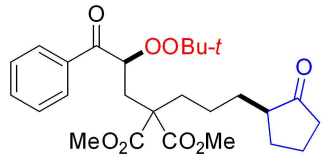



```
NAME        wll-695p-20211118
EXPNO                       2
PROCNO                      1
Date_            20211119
Time                     0.17
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      200
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                   298.7 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            150.9128594 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

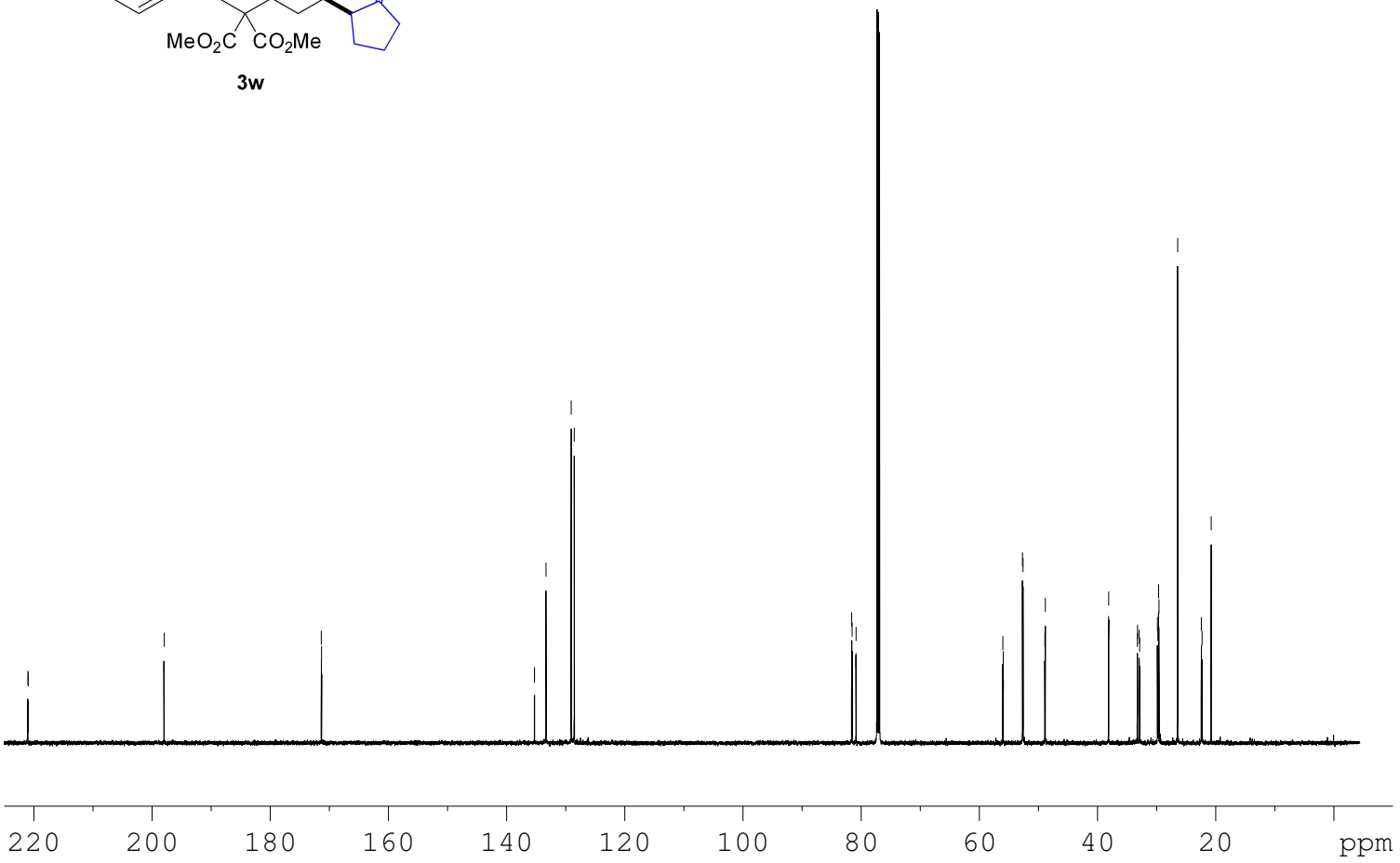



```
NAME      WLL-698P-20211122
EXPNO                    1
PROCNO                   1
Date_             20211122
Time                 10.26
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       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                   295.9 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.96 usec
SI                   65536
SF            600.1700151 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

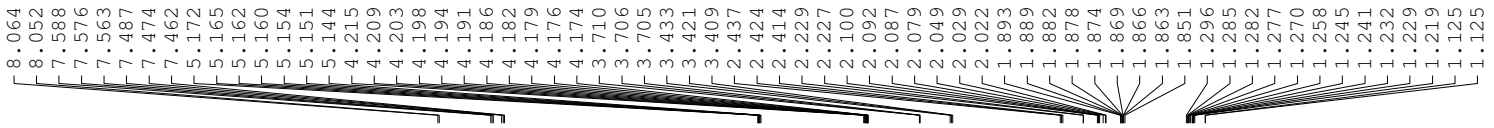
220.966
197.940
171.366
171.322
135.297
135.282
133.336
129.067
128.555
81.608
81.529
80.882
80.865
56.023
55.953
52.702
52.608
48.916
48.833
38.133
38.114
33.263
33.228
32.945
32.877
29.861
29.789
29.670
29.604
26.438
22.437

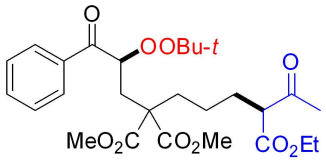


3w

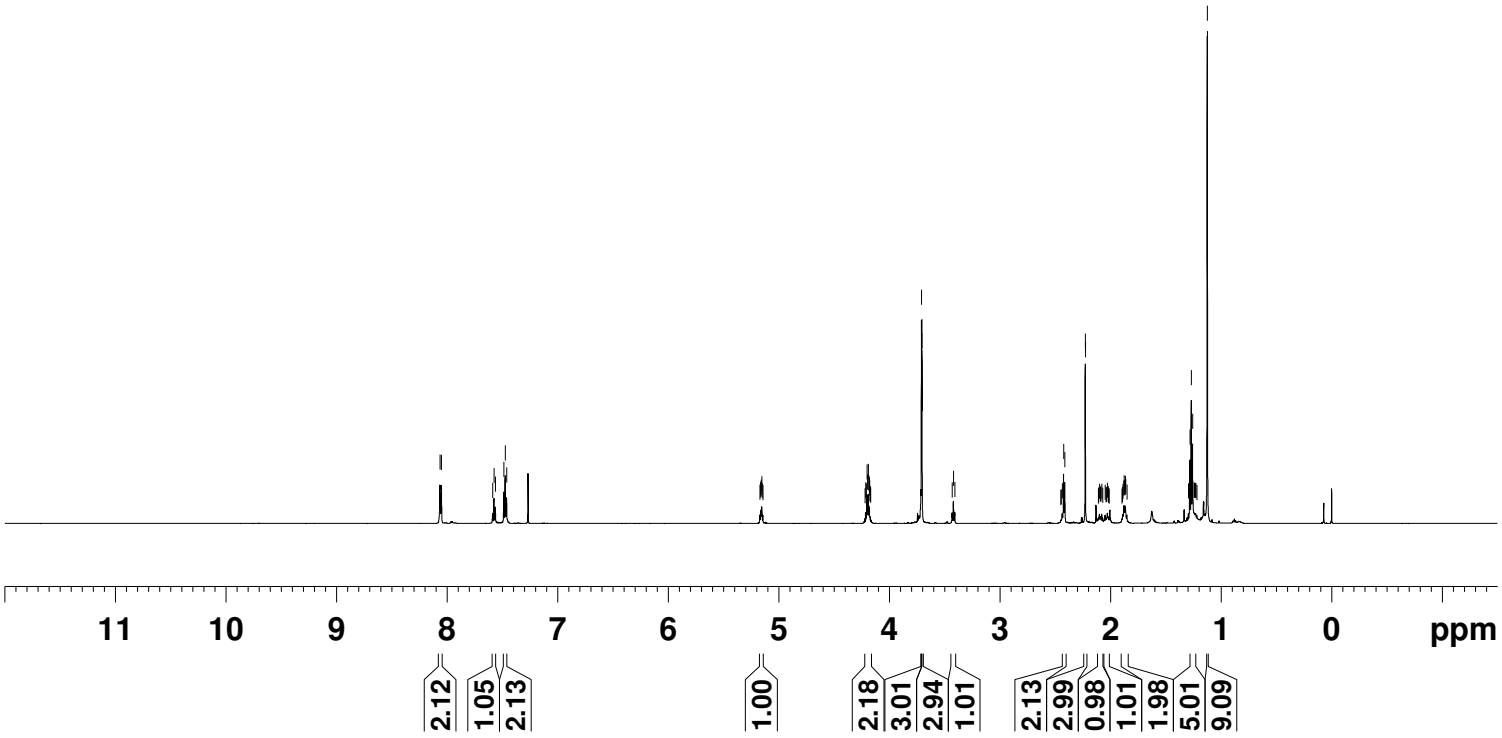



```
NAME      WLL-698P-20211122
EXPNO                    2
PROCNO                   1
Date_             20211122
Time                 11.23
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDC13
NS                    1024
DS                       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                    297.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          150.9302215 MHz
NUC1                   13C
P1                   11.90 usec
SI                   32768
SF            150.9128588 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

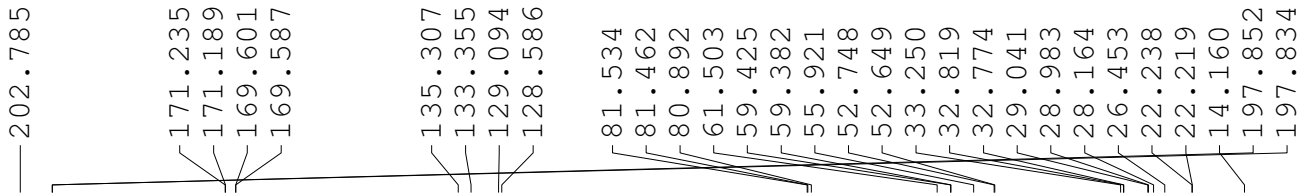


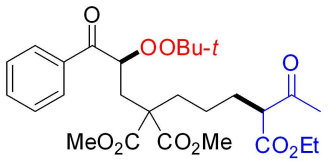


3x



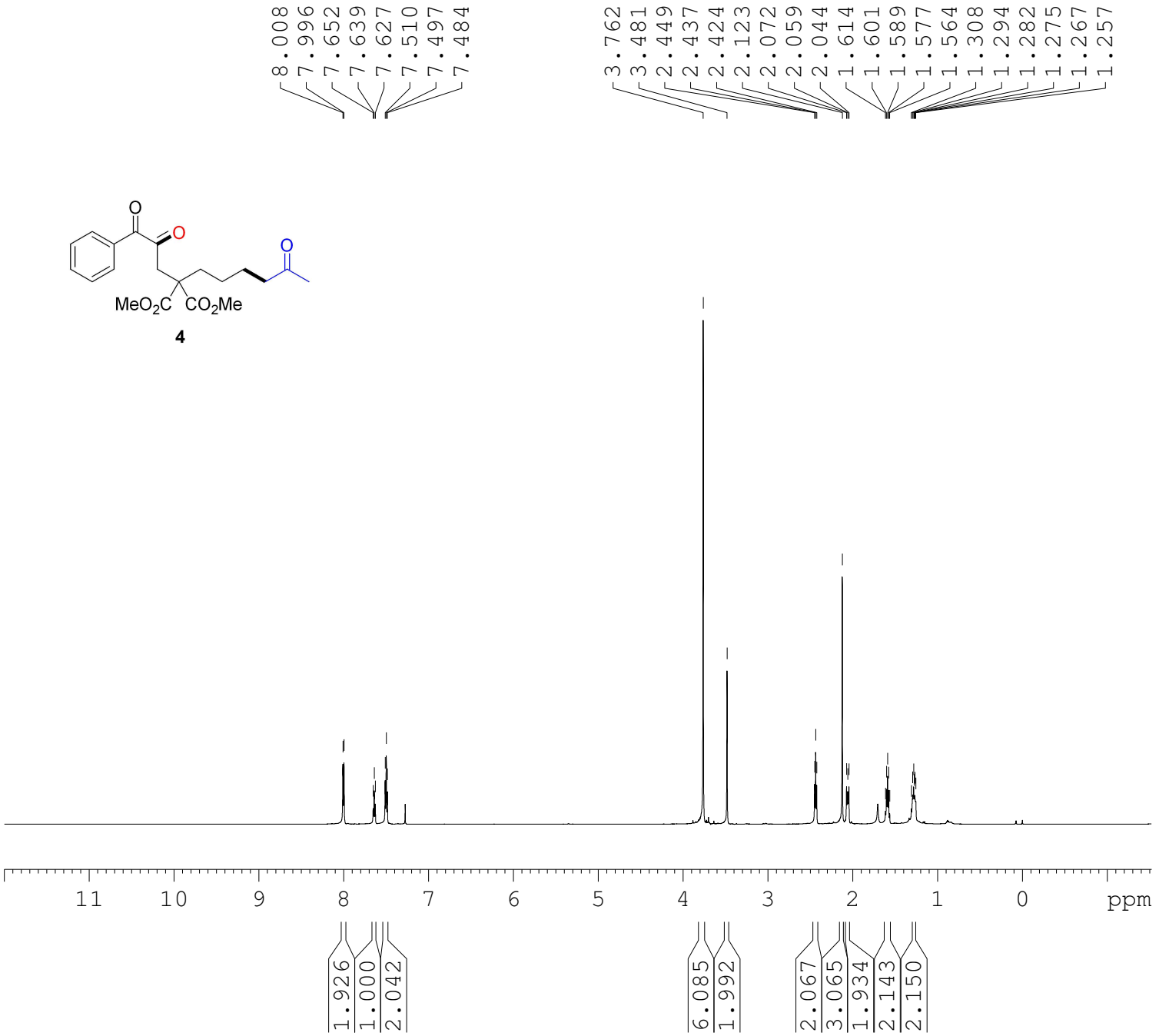
NAME	wll-697p-20211118
EXPNO	1
PROCNO	1
Date_	20211118
Time	21.28
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDC13
NS	8
DS	0
SWH	9615.385 Hz
FIDRES	0.146719 Hz
AQ	3.4079220 sec
RG	69.87
DW	52.000 usec
DE	6.50 usec
TE	297.6 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
SFO1	600.1739011 MHz
NUC1	1H
P1	9.96 usec
SI	65536
SF	600.1700107 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





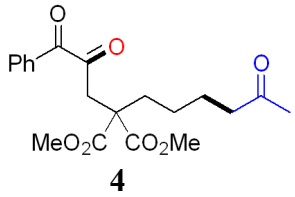
3x

NAME	wll-697p-20211118
EXPNO	2
PROCNO	1
Date_	20211119
Time	0.32
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	200
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	298.7 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	150.9128560 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

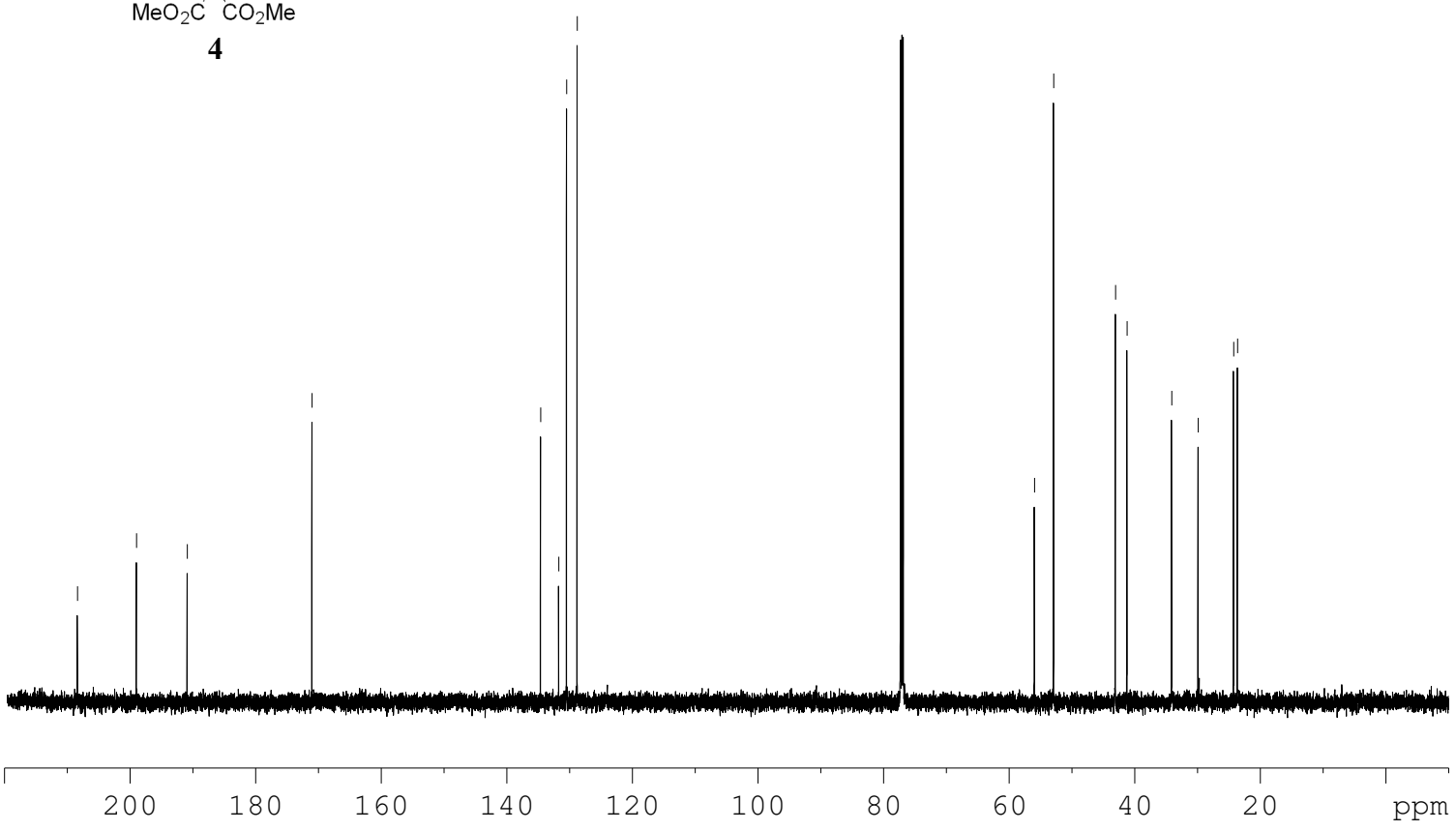


NAME	WLL-578PP-20210709
EXPNO	1
PROCNO	1
Date_	20210709
Time	15.52
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	9615.385 Hz
FIDRES	0.146719 Hz
AQ	3.4079220 sec
RG	56.75
DW	52.000 usec
DE	6.50 usec
TE	301.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	600.1739011 MHz
NUC1	1H
P1	9.96 usec
SI	65536
SF	600.1700073 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

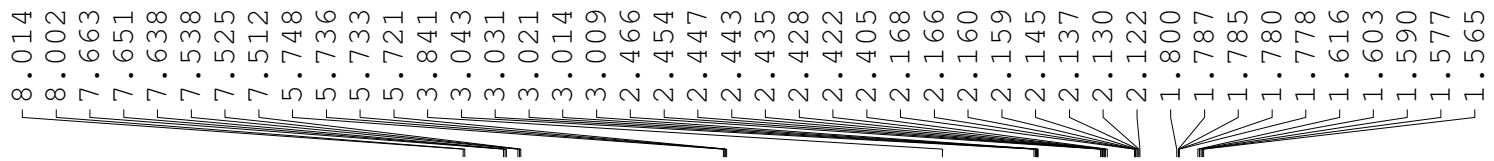
208.370
198.970
190.888
171.020
134.607
131.723
130.473
128.786
55.955
52.903
43.051
41.215
34.101
29.892
24.238
23.618

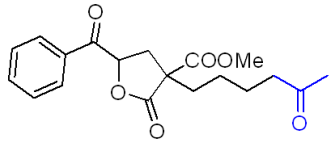


4

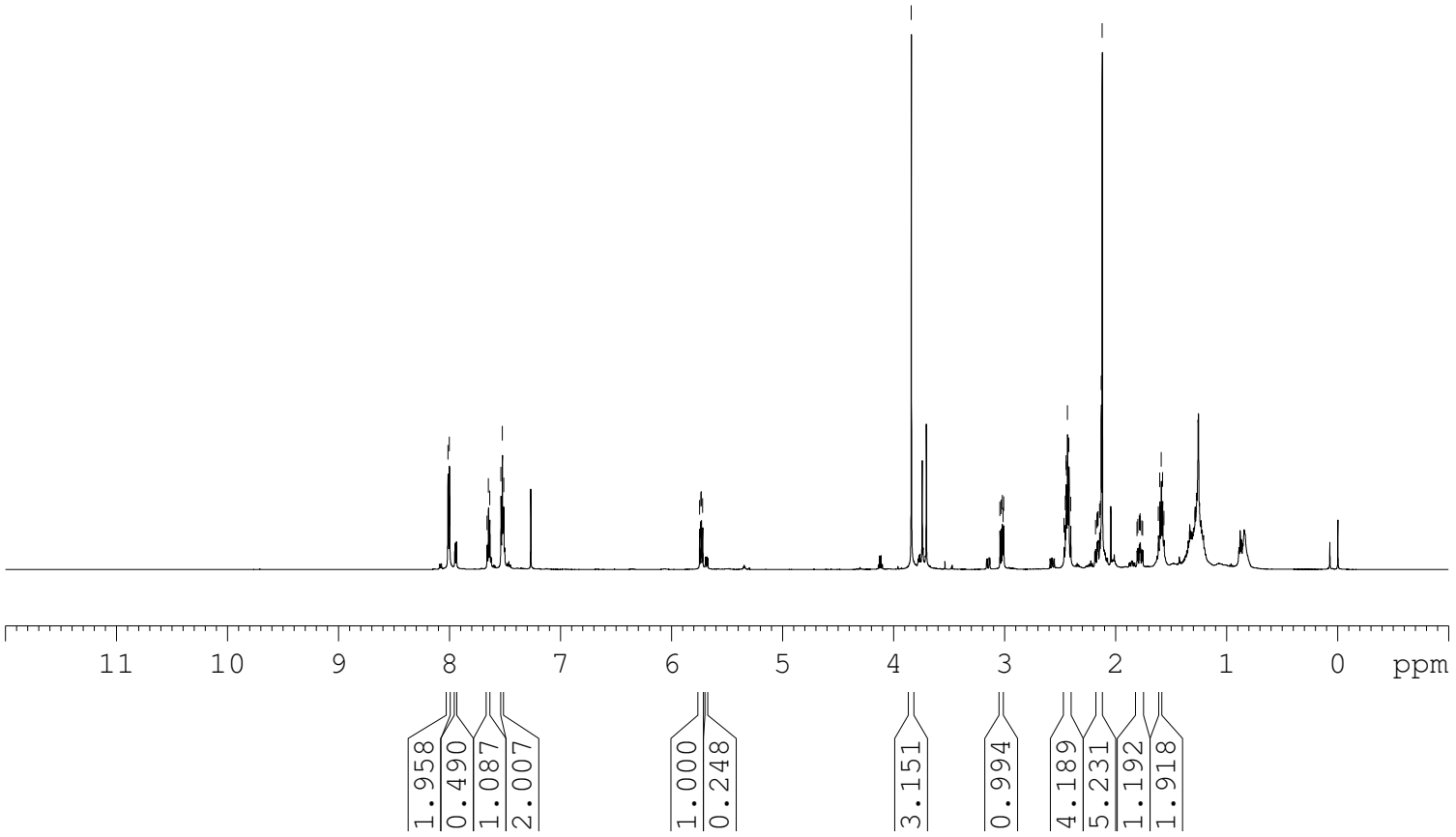


NAME	WLL-578PP-20210709
EXPNO	2
PROCNO	1
Date_	20210709
Time	15.58
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	100
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	302.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
========	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

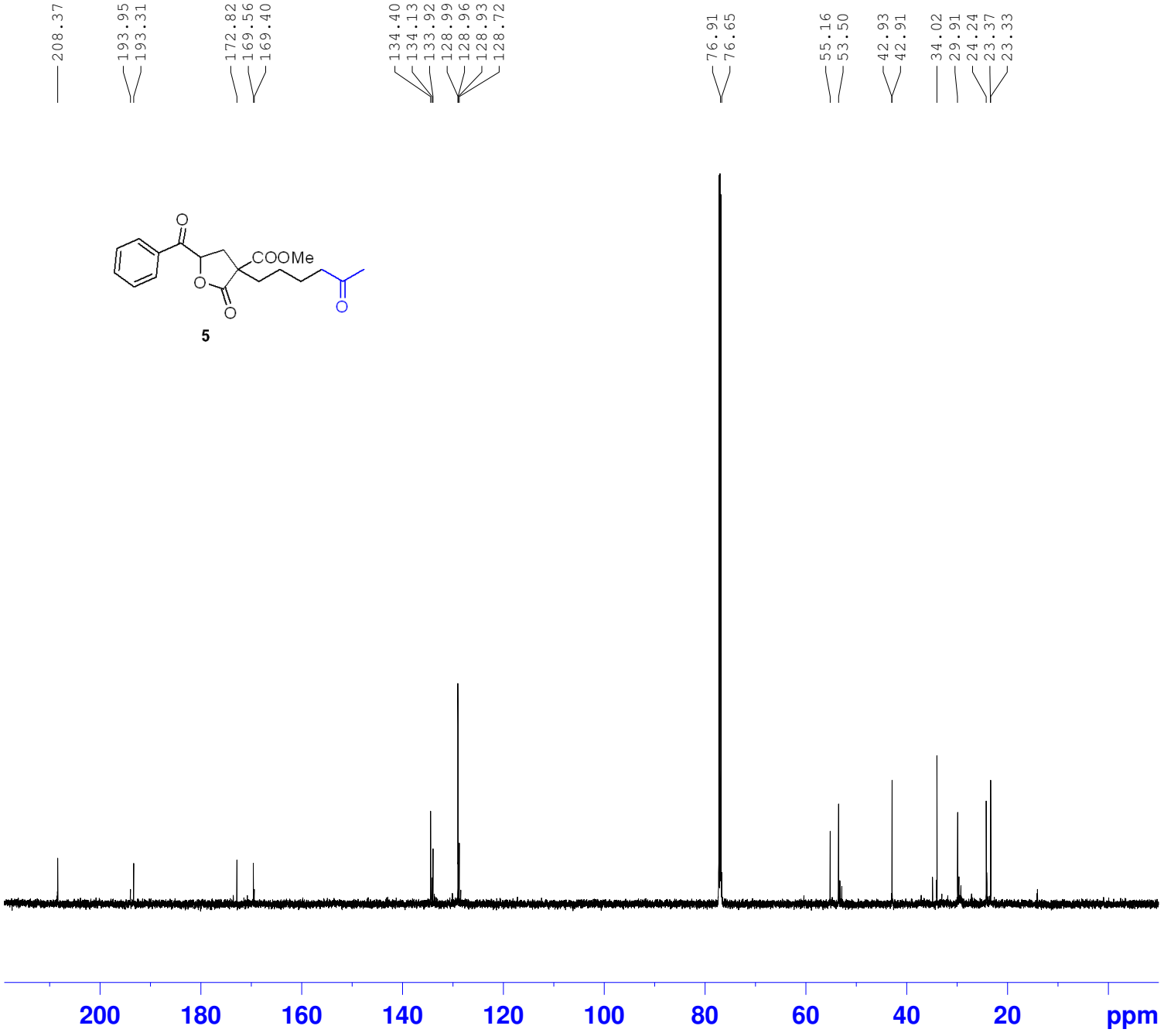




5

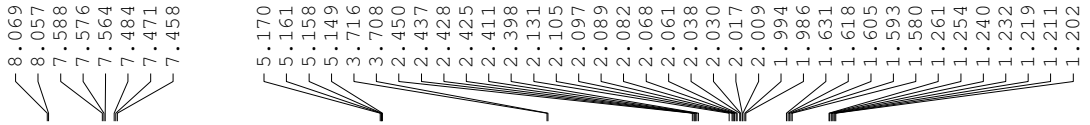


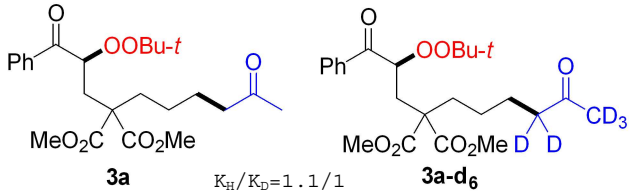
NAME	WLL-600-PRO-20211224
EXPNO	1
PROCNO	1
Date_	20211224
Time	16.17
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	9615.385 Hz
FIDRES	0.146719 Hz
AQ	3.4079220 sec
RG	62.22
DW	52.000 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	600.1739011 MHz
NUC1	1H
P1	9.96 usec
SI	65536
SF	600.1700092 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

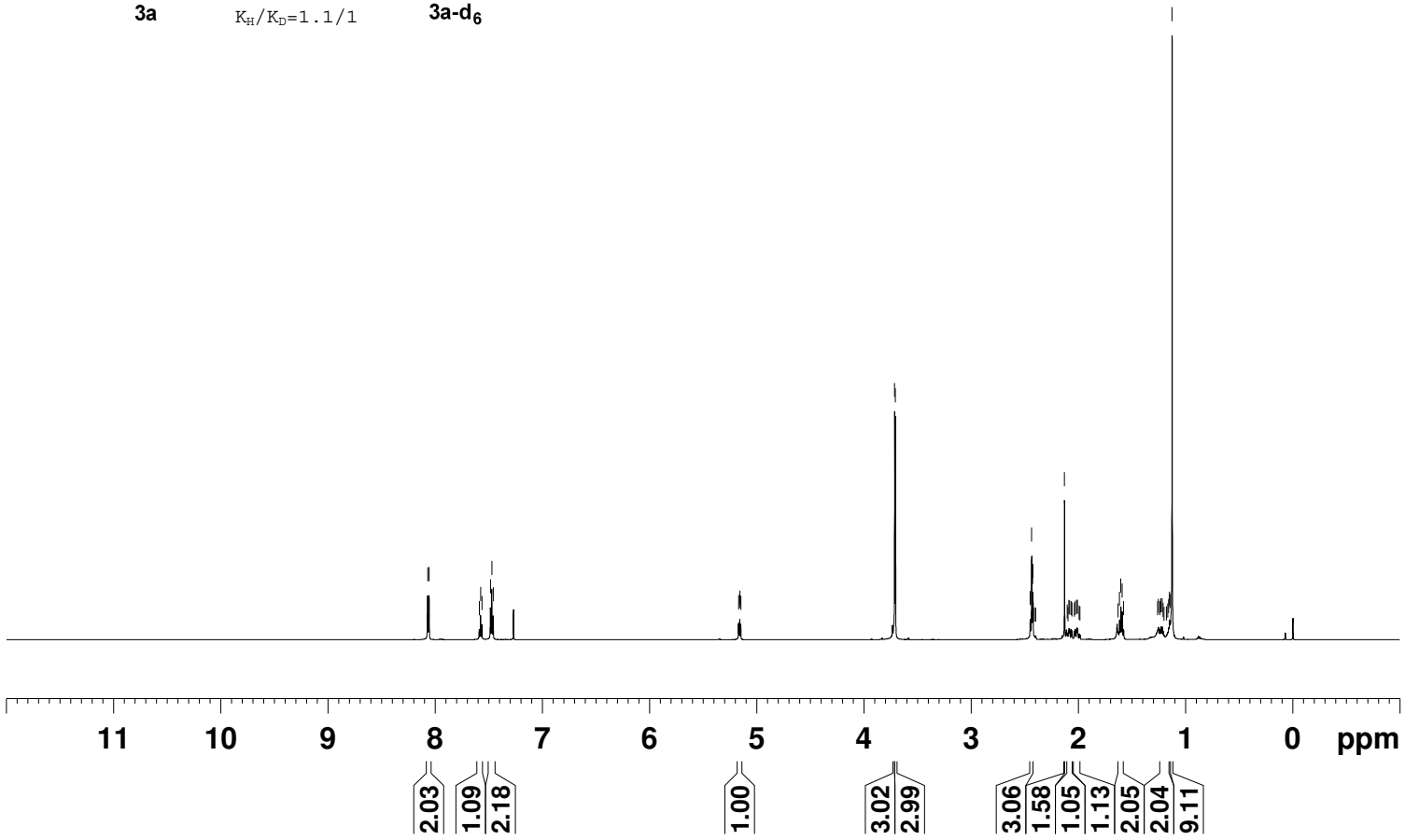


BRUKER

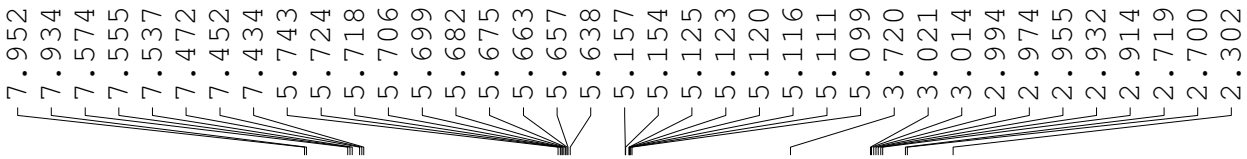
NAME	WLL-600-PRO-20211224
EXPNO	2
PROCNO	1
Date_	20211224
Time	16.52
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	300
DS	4
SWH	36057.691 Hz
FIDRES	0.550197 Hz
AQ	0.9088159 sec
RG	190.02
DW	13.867 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	150.9279571 MHz
NUC1	13C
P1	11.90 usec
SI	32768
SF	150.9128726 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

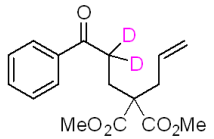




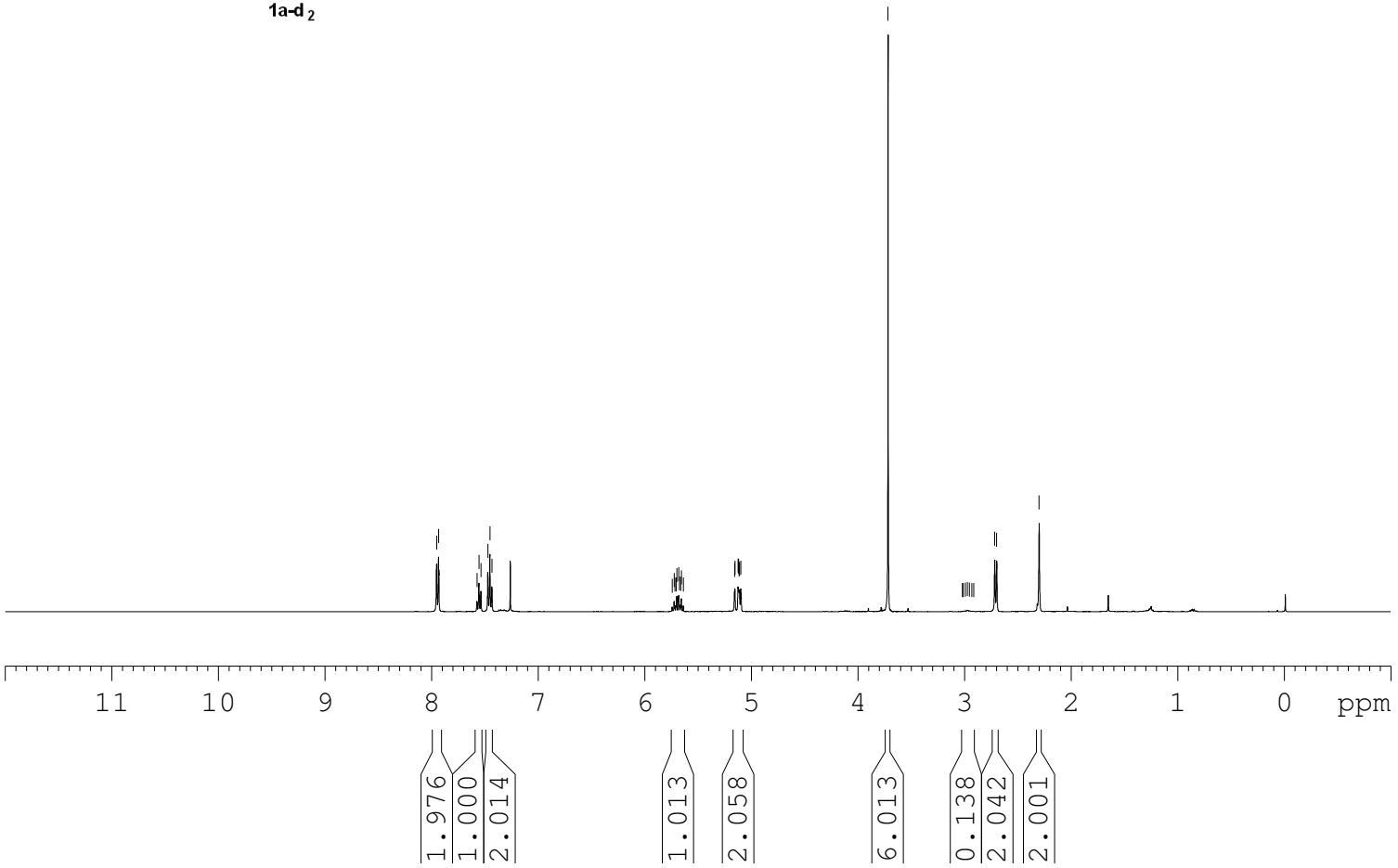



```
NAME      wll-614p-20211118
EXPNO                    1
PROCNO                   1
Date_             20211118
Time                 21.23
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      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                 297.7 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1           600.1739011 MHz
NUC1                   1H
P1                   9.96 usec
SI                  65536
SF             600.1700098 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

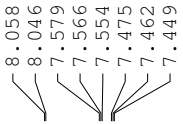


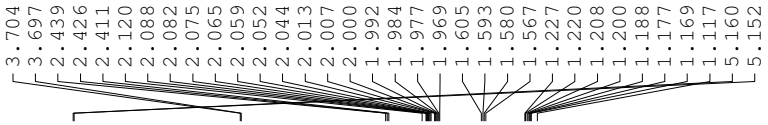


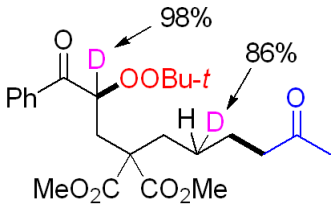
1a-d 2



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	CDCl3
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	400.1300098 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

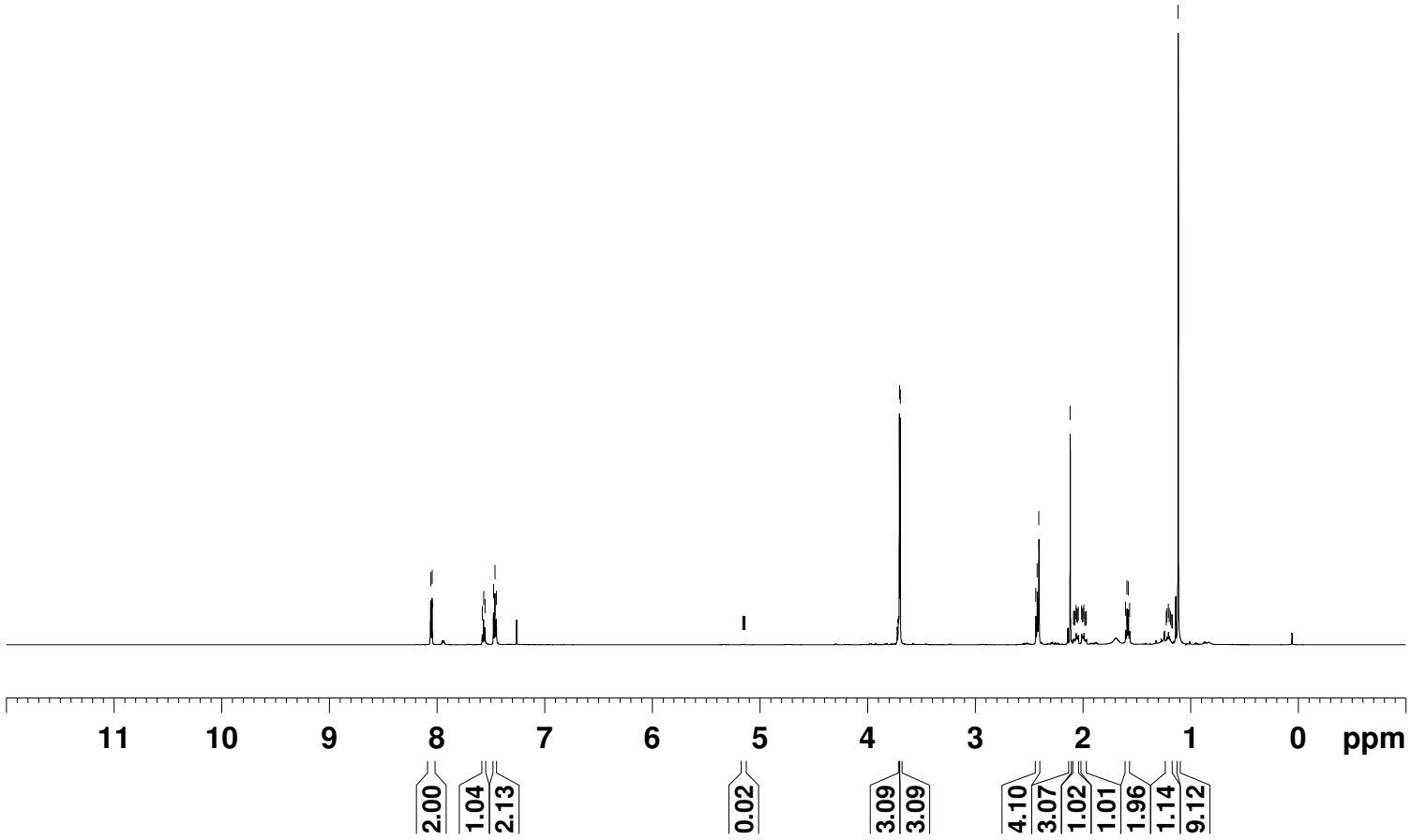


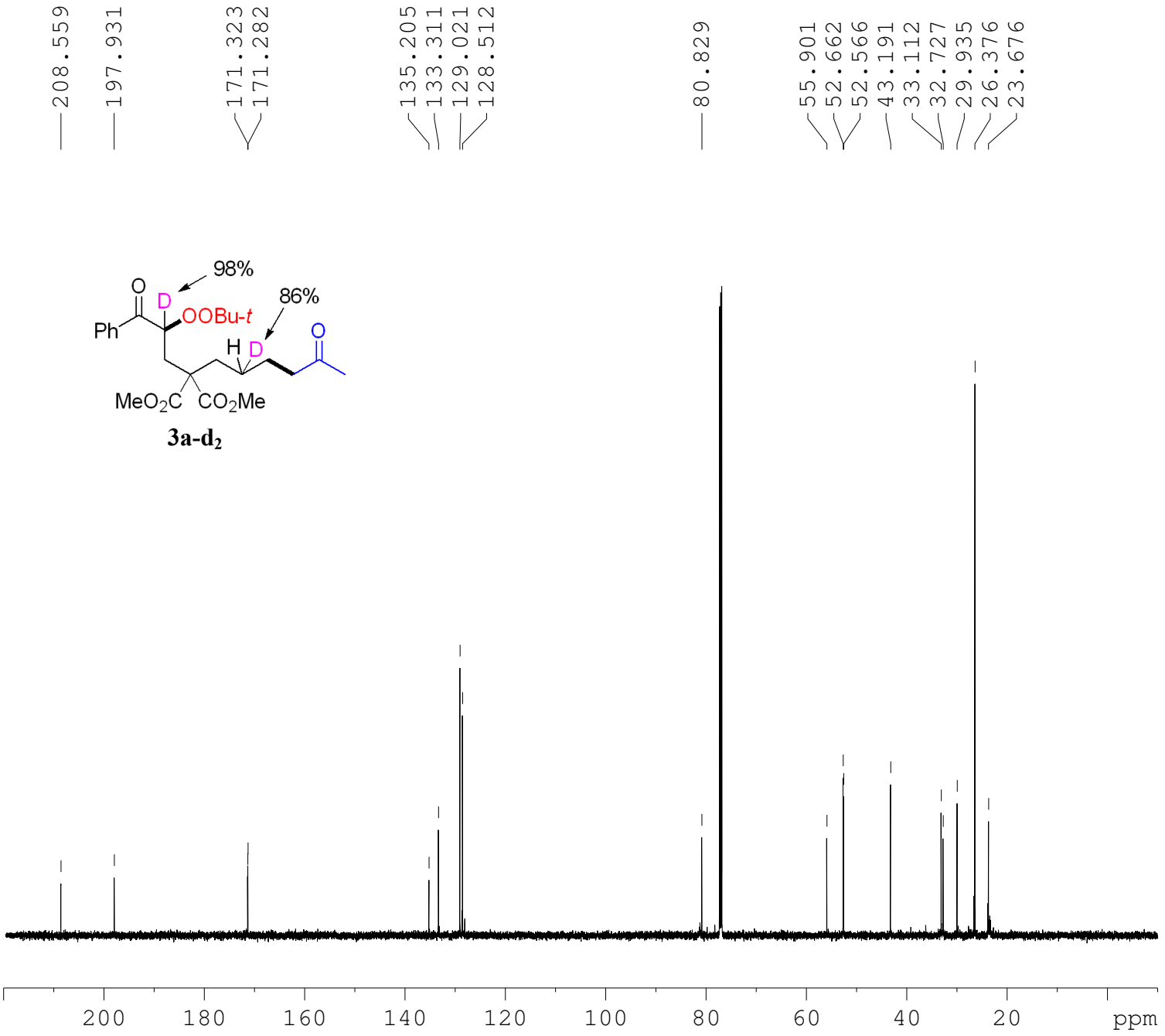




3a-d$_2$

```
NAME      wll-599p-20210727
EXPNO                    1
PROCNO                   1
Date_             20210727
Time                 19.53
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                  298.1 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          600.1739011 MHz
NUC1                    1H
P1                   9.96 usec
SI                    65536
SF            600.1700154 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```






```
NAME      wll-599p-20210727
EXPNO                    2
PROCNO                   1
Date_             20210727
Time                 20.09
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                   298.2 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
```