

Supporting Information

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

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1. General information

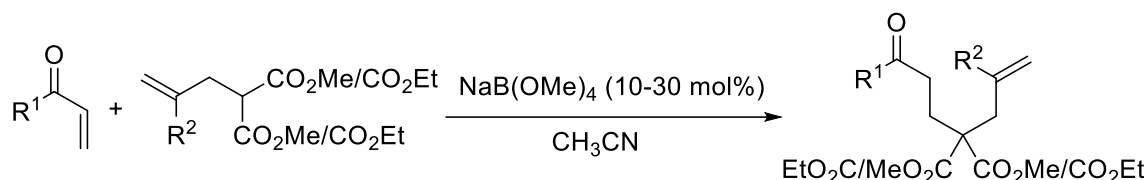
¹H NMR spectra were recorded on Bruker 400 MHz and 600 MHz spectrometer and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm) for CDCl₃. The peak patterns are indicated as follows: s, singlet; d, doublet; dd, doublet of doublet; t, triplet; m, multiplet; q, quartet. The coupling constants, *J*, are reported in Hertz (Hz). ¹³C NMR spectra were obtained at Bruker 100 MHz, 150 MHz and referenced to the internal solvent signals (central peak is 77.0 ppm in CDCl₃). CDCl₃ 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

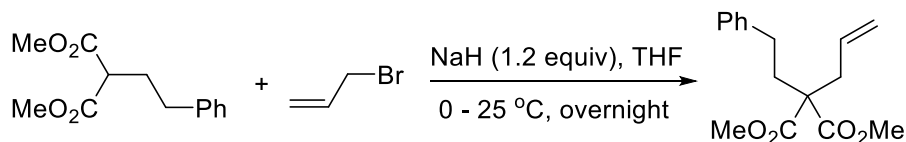
All the unactivated alkene substrate **1** was known compounds. Unactivated alkenes **1a-1t**^{1,2} and **1u**³ were prepared as followed procedure.

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



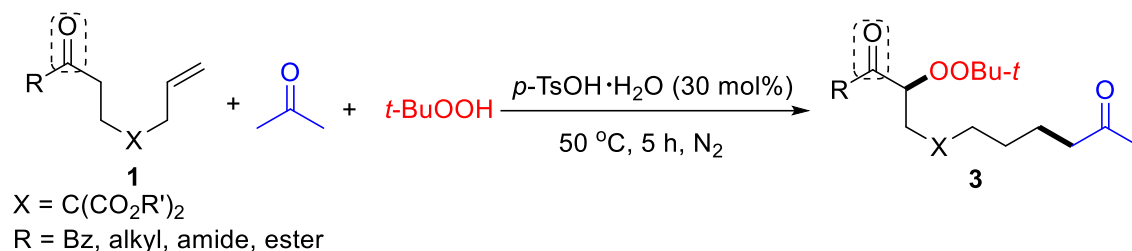
To a 25 mL round bottom flask with a magnetic stir bar a solution of Michael donor (1.0 mmol) and $NaB(OMe)_4$ (0.10-0.30 mmol, 10-30 mol %) in MeCN (3.0 mL) was added α,β -unsaturated ketone (1.0-2.0 mmol) at room temperature. The resulting solution was stirred at room temperature or 50 °C under air atmosphere and monitored by TLC. Upon completion, solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel (ethyl acetate/petroleum ether = 1/12-1/4) to give the desired products in good yield.

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

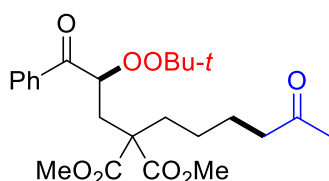


To a stirred suspension of NaH (60% in mineral oil, 2.4 mmol.) in dry THF (6.0 ml) at 0 °C was added a solution of dimethyl 2-phenethylmalonate (2.0 mmol) in THF (2.0 mL). The reaction mixture was kept for 0.5 h, and then a solution of allyl bromide (2.2 mmol) in THF (2.0 mL) was added dropwise, and 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.

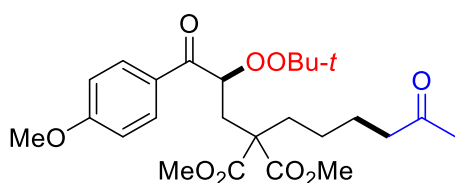
3. General procedure for synthesis of products 3



To a 10 mL sealable pressure tube was added unactivated alkenes **1** (0.1 mmol), *p*-TsOH·H₂O (0.03 mmol), acetone (0.6 mL) and *t*-BuOOH (5.5 M in decane, 0.5 mmol) under N₂ atmosphere at room temperature, and the resulting solution was stirred at 50 °C 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**.

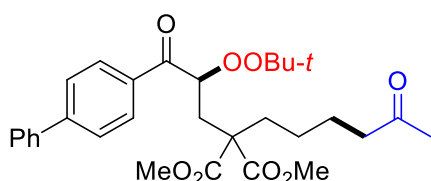


Dimethyl 2-(2-(*tert*-butylperoxy)-3-oxo-3-phenylpropyl)-2-(5-oxohexyl)malonate (3a): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, $R_f = 0.4$) in 64% yield (28.7 mg); Colorless oil; ¹H NMR (600 MHz, CDCl₃, ppm) δ 8.07 (d, $J = 7.2$ Hz, 2H), 7.58 (t, $J = 7.4$ Hz, 1H), 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); ¹³C NMR (150 MHz, CDCl₃, 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 C₂₄H₃₄O₈Na (M+Na)⁺: 473.2146; found: 473.2141.

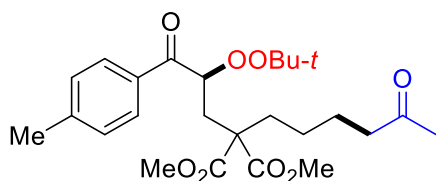


Dimethyl 2-(2-(*tert*-butylperoxy)-3-(4-methoxyphenyl)-3-oxopropyl)-2-(5-oxohexyl)malonate (3b): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, $R_f = 0.35$) in 63% yield (30.2 mg); Colorless oil; ¹H NMR (600 MHz, CDCl₃, ppm) δ 7.97 (d, $J = 8.2$ Hz, 2H), 7.27 (d,

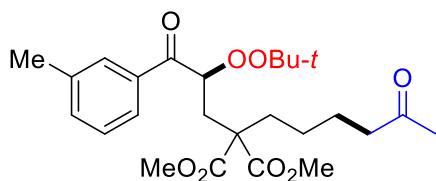
$J = 8.6$ Hz, 2H), 5.15 (t, $J = 6.2$ Hz, 1H), 3.72 (s, 3H), 3.71 (s, 3H), 2.45-2.41 (m, 7H), 2.13 (s, 3H), 2.11-2.05 (m, 1H), 2.03-1.98 (m, 1H), 1.63-1.58 (m, 2H), 1.26-1.18 (m, 2H), 1.14 (s, 9H); ^{13}C NMR (150 MHz, CDCl_3 , ppm) δ 208.6, 196.2, 171.4, 171.3, 163.7, 131.4, 128.1, 113.7, 81.6, 80.8, 55.9, 55.5, 52.7, 52.6, 43.2, 33.4, 32.8, 29.9, 26.4, 23.8; HRMS (ESI) calcd for $\text{C}_{25}\text{H}_{36}\text{O}_9\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 503.2252; found: 503.2254.



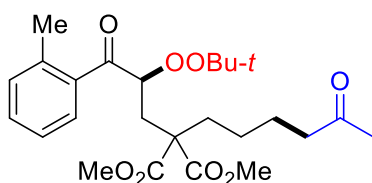
Dimethyl 2-(3-([1,1'-biphenyl]-4-yl)-2-(*tert*-butylperoxy)-3-oxopropyl)-2-(5-oxohexyl)malonate (3c): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, $R_f = 0.4$) in 63% yield (32.9 mg); Colorless oil; ^1H NMR (600 MHz, CDCl_3 , ppm) δ 8.16 (d, $J = 8.4$ Hz, 2H), 7.70 (d, $J = 8.4$ 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 $\text{C}_{30}\text{H}_{38}\text{O}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 549.2459; found: 549.2455.



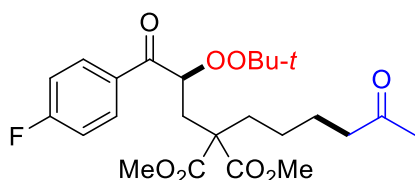
Dimethyl 2-(2-(*tert*-butylperoxy)-3-oxo-3-(*p*-tolyl)propyl)-2-(5-oxohexyl)malonate (3d): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, $R_f = 0.4$) in 65% yield (30.1 mg); Colorless oil; ^1H NMR (600 MHz, CDCl_3 , ppm) δ 7.97 (d, $J = 8.2$ Hz, 2H), 7.27 (d, $J = 8.6$ 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 $\text{C}_{25}\text{H}_{36}\text{O}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 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; ^1H 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 $\text{C}_{25}\text{H}_{36}\text{O}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 487.2302; found: 487.2304.

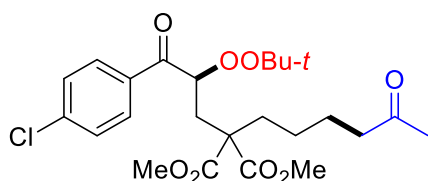


Dimethyl 2-(2-(*tert*-butylperoxy)-3-oxo-3-(*o*-tolyl)propyl)-2-(5-oxohexyl)malonate (3f): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, $R_f = 0.4$) in 64% yield (29.5 mg); Colorless oil; ^1H NMR (600 MHz, CDCl_3 , ppm) δ 7.67 (d, $J = 7.2$ Hz, 1H), 7.38-7.36 (m, 1H), 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 $\text{C}_{25}\text{H}_{36}\text{O}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 487.2302; found: 487.2303.



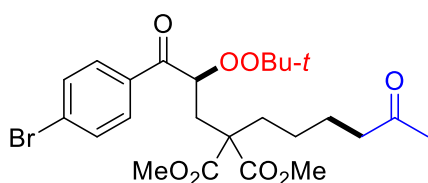
Dimethyl 2-(2-(*tert*-butylperoxy)-3-(4-fluorophenyl)-3-oxopropyl)-2-(5-oxohexyl)malonate (3g): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, $R_f = 0.4$) in 63% yield

(29.3 mg); Colorless oil; ^1H 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 $\text{C}_{24}\text{H}_{33}\text{FO}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 491.2052; found: 491.2054.



Dimethyl 2-(2-(*tert*-butylperoxy)-3-(4-chlorophenyl)-3-oxopropyl)-2-(5-oxohexyl)malonate (3h):

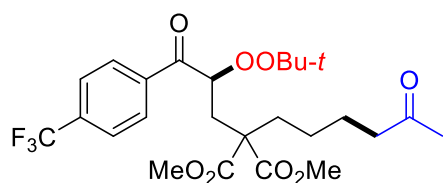
Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, $R_f = 0.4$) in 61% yield (29.5 mg); Colorless oil; ^1H NMR (600 MHz, CDCl_3 , ppm) δ 8.04 (d, $J = 8.5$ Hz, 2H), 7.45 (d, $J = 8.5$ Hz, 2H), 5.08 (dd, $J = 7.2, 5.4$ Hz, 1H), 3.72 (s, 3H), 3.72 (s, 3H), 2.46-2.41 (m, 4H), 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 $\text{C}_{24}\text{H}_{33}\text{ClO}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 507.1756; found: 507.1759.



Dimethyl 2-(3-(4-bromophenyl)-2-(*tert*-butylperoxy)-3-oxopropyl)-2-(5-oxohexyl)malonate (3i):

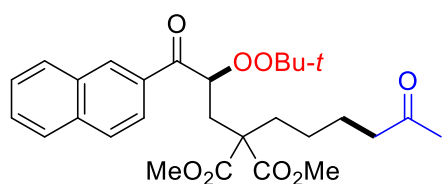
Isolated by flash column chromatography (ethyl acetate/petroleum ether = 5:1, $R_f = 0.4$) in 58% yield (30.4 mg); Colorless oil; ^1H NMR (600 MHz, CDCl_3 , ppm) δ 7.97 (d, $J = 8.6$ Hz, 2H), 7.62 (d, $J = 8.6$ Hz, 2H), 5.07 (dd, $J = 7.4, 5.3$ Hz, 1H), 3.72 (s, 3H), 3.72 (s, 3H), 2.46-2.41 (m, 4H), 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 $\text{C}_{24}\text{H}_{33}\text{BrO}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$:

551.1251; found: 551.1254.



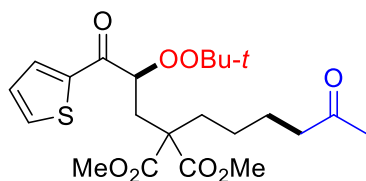
Dimethyl 2-(2-(*tert*-butylperoxy)-3-oxo-3-(4-(trifluoromethyl)phenyl)propyl)-2-(5-oxohexyl)

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; ^1H 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 $\text{C}_{25}\text{H}_{33}\text{F}_3\text{O}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 541.2020; found: 541.2022.



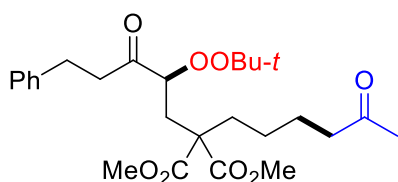
Dimethyl 2-(2-(*tert*-butylperoxy)-3-(naphthalen-2-yl)-3-oxopropyl)-2-(5-oxohexyl)malonate (3k):

Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.4) in 30% yield (14.9 mg); Colorless oil; ^1H 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 $\text{C}_{28}\text{H}_{36}\text{O}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 523.2302; found: 523.2304.



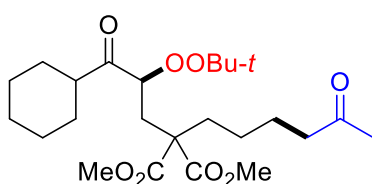
Dimethyl 2-(2-(tert-butylperoxy)-3-oxo-3-(thiophen-2-yl)propyl)-2-(5-oxohexyl)malonate (3l):

Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, $R_f = 0.5$) in 32% yield (14.7 mg); Colorless oil; $^1\text{H NMR}$ (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); $^{13}\text{C NMR}$ (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 $\text{C}_{22}\text{H}_{32}\text{O}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 479.1710; found: 479.1708.



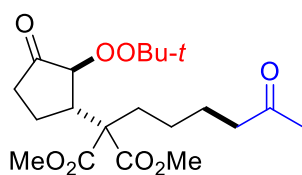
Dimethyl 2-(2-(tert-butylperoxy)-3-oxo-5-phenylpentyl)-2-(5-oxohexyl)malonate (3m):

Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, $R_f = 0.4$) in 54% yield (25.8 mg); Colorless oil; $^1\text{H NMR}$ (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); $^{13}\text{C NMR}$ (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 $\text{C}_{26}\text{H}_{38}\text{O}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 501.2459; found: 501.2456.

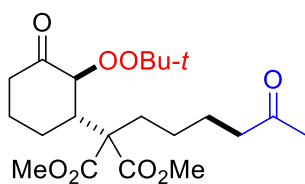


Dimethyl 2-(2-(tert-butylperoxy)-3-cyclohexyl-3-oxopropyl)-2-(5-oxohexyl)malonate (3n):

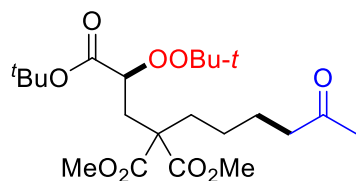
Isolated by flash column chromatography (ethyl acetate/petroleum ether = 4:1, $R_f = 0.3$) in 56% yield (25.4 mg); Colorless oil; ^1H NMR (600 MHz, CDCl_3 , ppm) δ 4.34 (dd, $J = 8.4, 4.1$ Hz, 1H), 3.74 (s, 3H), 3.72 (s, 3H), 2.88-2.84 (m, 1H), 2.44 (t, $J = 7.4$ Hz, 2H), 2.24-2.18 (m, 2H), 2.13 (s, 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 $\text{C}_{24}\text{H}_{40}\text{O}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 479.2615; found: 479.2613.



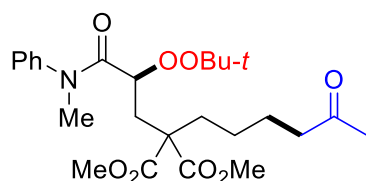
Dimethyl 2-((2R)-2-(tert-butylperoxy)-3-oxocyclopentyl)-2-(5-oxohexyl)malonate (3o): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 5:1, $R_f = 0.4$) in 41% yield (16.3 mg); Colorless oil (dr > 20:1); ^1H NMR (600 MHz, CDCl_3 , ppm) δ 4.26 (d, $J = 7.9$ Hz, 1H), 3.74 (s, 3H), 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 $\text{C}_{20}\text{H}_{32}\text{O}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 423.1989; found: 499.1988.



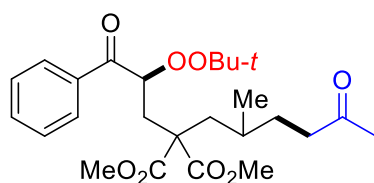
Dimethyl 2-((1S)-2-(tert-butylperoxy)-3-oxocyclohexyl)-2-(5-oxohexyl)malonate (3p): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 5:1, $R_f = 0.4$) in 46% yield (18.9 mg); Colorless oil (dr > 20:1); ^1H NMR (600 MHz, CDCl_3 , ppm) δ 4.27 (d, $J = 4.6$ Hz, 1H), 3.65 (s, 3H), 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 $\text{C}_{21}\text{H}_{34}\text{O}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 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; ^1H 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 $\text{C}_{22}\text{H}_{38}\text{O}_9\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 469.2408; found: 469.2405.

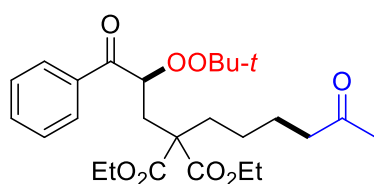


Dimethyl 2-(2-(tert-butylperoxy)-3-(methyl(phenyl)amino)-3-oxopropyl)-2-(5-oxohexyl)malonate (3r): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, $R_f = 0.3$) in 33% yield (16.0 mg); Colorless oil; ^1H NMR (600 MHz, CDCl_3 , ppm) δ 7.43 (t, $J = 7.6$ Hz, 2H), 7.35 (t, $J = 7.4$ 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 $\text{C}_{25}\text{H}_{38}\text{NO}_8$ ($\text{M}+\text{H}$) $^+$: 480.2592; found: 480.2589.

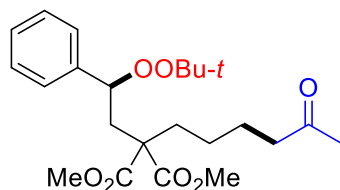


Dimethyl 2-(2-(tert-butylperoxy)-3-oxo-3-phenylpropyl)-2-(2-methyl-5-oxohexyl)malonate (3s): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, $R_f = 0.4$) in 58% yield

(27.1 mg); Colorless oil (dr = 1:1); ^1H 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 $\text{C}_{25}\text{H}_{36}\text{O}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 487.2302; found: 487.2299.

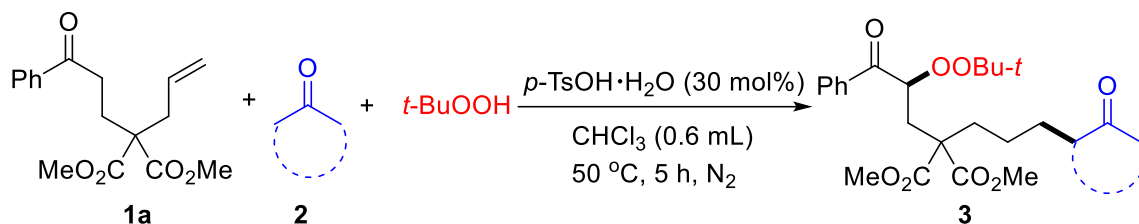


Diethyl 2-(2-(tert-butylperoxy)-3-oxo-3-phenylpropyl)-2-(5-oxohexyl)malonate (3t): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 5:1, $R_f = 0.4$) in 61% yield (29.0 mg); Colorless oil; ^1H NMR (600 MHz, CDCl_3 , ppm) δ 8.07 (d, $J = 7.4$ Hz, 2H), 7.57 (t, $J = 7.4$ Hz, 1H), 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 $\text{C}_{26}\text{H}_{38}\text{O}_7\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 501.2459; found: 501.2460.

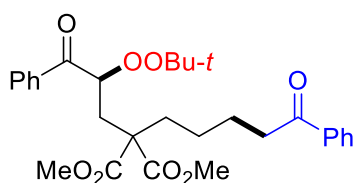


Dimethyl 2-(2-(tert-butylperoxy)-2-phenylethyl)-2-(5-oxohexyl)malonate (3u): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 5:1, $R_f = 0.3$) in 58% yield (24.6 mg); Colorless oil; ^1H NMR (600 MHz, CDCl_3 , ppm) δ 7.34-7.28 (m, 5H), 4.81 (dd, $J = 8.0, 5.0$ Hz, 1H), 3.71 (s, 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 C₂₃H₃₄O₇Na (M+Na)⁺: 445.2197; found: 445.2195.

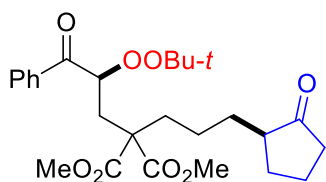


To a 10 mL sealable pressure tube was added unactivated alkenes **1** (0.1 mmol), *p*-TsOH·H₂O (0.03 mmol), CH₃Cl (0.6 mL) and *t*-BuOOH (5.5 M in decane, 0.5 mmol) under N₂ atmosphere at room temperature, and the resulting solution was stirred at 50 °C for 5 h. The resulting mixture and the solvent 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):

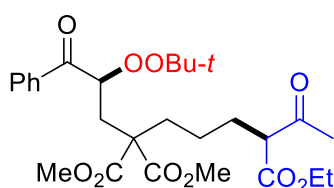
Isolated by flash column chromatography (ethyl acetate/petroleum ether = 4:1, *R_f* = 0.3) in 28% yield (14.3 mg); Colorless oil; ¹H NMR (600 MHz, CDCl₃, ppm) δ 8.36 (d, *J* = 7.3 Hz, 2H), 7.95 (d, *J* = 7.3 Hz, 2H), 7.58-7.55 (m, 2H), 7.48-7.45 (m, 4H), 5.19 (dd, *J* = 7.5, 5.0 Hz, 1H), 3.72 (s, 3H), 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); ¹³C NMR (150 MHz, CDCl₃, 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 C₂₉H₃₆O₈Na (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* =

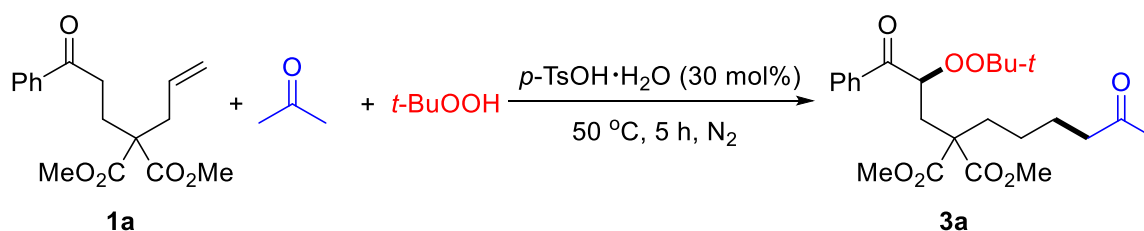
0.3) in 16% yield (7.6 mg); Colorless oil (dr = 1:1); ^1H NMR (600 MHz, CDCl_3 , ppm) δ 8.05 (d, J = 7.3 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.9 Hz, 2H), 5.17-5.14 (m, 1H), 3.71 (s, 3H), 3.70 (s, 3H), 2.43-2.41 (m, 2H), 2.31-2.27 (m, 1H), 2.22-2.17 (m, 1H), 2.12-2.05 (m, 2H), 2.04-1.97 (m, 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 $\text{C}_{26}\text{H}_{36}\text{O}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 499.2302; found: 499.2303.



3-ethyl 7,7-dimethyl 9-(tert-butylperoxy)-2,10-dioxo-10-phenyldecane-3,7,7-tricarboxylate (**3x**):

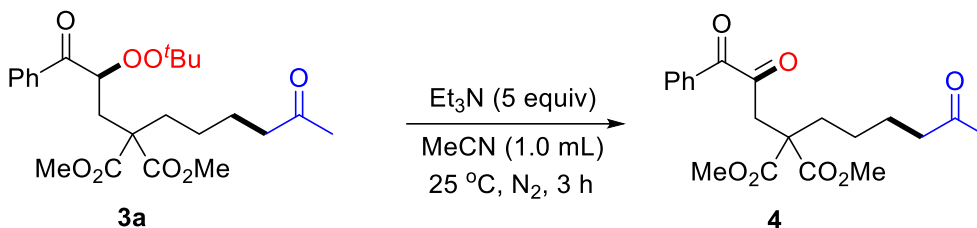
Isolated by flash column chromatography (ethyl acetate/petroleum ether = 3:1, R_f = 0.3) in 40% yield (20.9 mg); Colorless oil (dr = 1:1); ^1H NMR (600 MHz, CDCl_3 , ppm) δ 8.06 (d, J = 7.5 Hz, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.9 Hz, 2H), 5.17-5.14 (m, 1H), 4.22-4.17 (m, 2H), 3.71 (s, 3H), 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 $\text{C}_{27}\text{H}_{38}\text{O}_{10}\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 545.2357; found: 545.2361.

4. Large scale synthesis and transformation

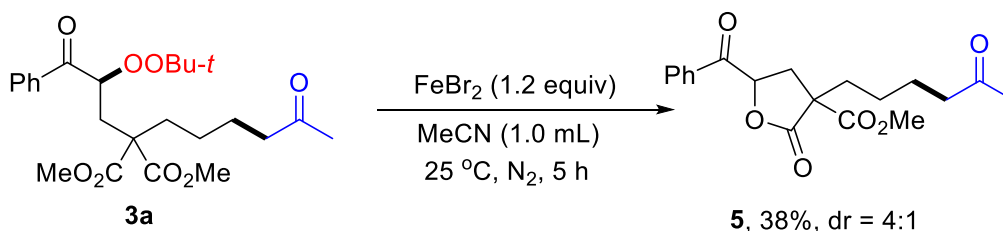


To a 25 mL sealable pressure tube was added unactivated alkenes **1a** (1 mmol), p -TsOH \cdot H $_2$ O (0.03 mmol), acetone (6.0 mL) and t -BuOOH (5.5 M in decane, 0.5 mmol) under N_2 atmosphere at room temperature, and the resulting solution was stirred at 50 $^\circ\text{C}$ for 5 h. The resulting 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).



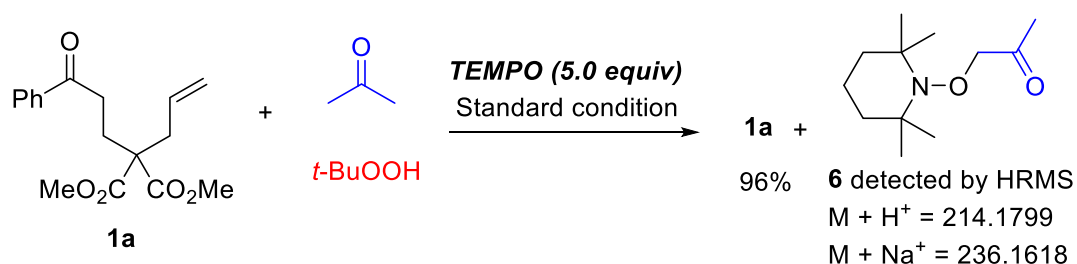
To a 25 mL Schlenk tube with a magnetic stir bar were added **3a** (45.0 mg, 0.1 mmol), Et₃N (70.0 vL, 0.5 mmol) in MeCN (1.0 ml) and then allowed to stir at room temperature under N₂ atmosphere for 3 h. The resultant residue was purified by flash chromatography (ethyl acetate/petroleum ether = 1/8-1/5) to afford dimethyl 2-(2,3-dioxo-3-phenylpropyl)-2-(5-oxohexyl)malonate (**4**) (31.7 mg, 84% yield) as a yellow oil. ¹H NMR (600 MHz, CDCl₃, 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); ¹³C NMR (150 MHz, CDCl₃, 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 C₂₀H₂₄O₇Na (M+Na)⁺: 399.1414; found: 399.1405.



To a 25 mL Schlenk tube with a magnetic stir bar were added **3a** (90.0 mg, 0.2 mmol), FeBr₂ (51.8 mg, 0.24 mmol) in MeCN (1.0 ml) and then allowed to stir at room temperature under N₂ 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). ¹H NMR (600 MHz, CDCl₃, 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); ¹³C NMR (150 MHz, CDCl₃, 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 C₁₉H₂₂O₆Na (M+Na)⁺: 369.1309; found: 369.1303.

5. Mechanistic studies

(a) Control experiments



To a 10 mL sealable pressure tube was added unactivated alkenes **1a** (0.1 mmol), *p*-TsOH·H₂O (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 N₂ atmosphere at room temperature, and the resulting solution was stirred at 50 °C for 5 h. The model reaction was totally suppressed where alkene **1a** was almost fully recovered, and we can detect the molecular weight of compound **6** by HRMS (Figure 1).

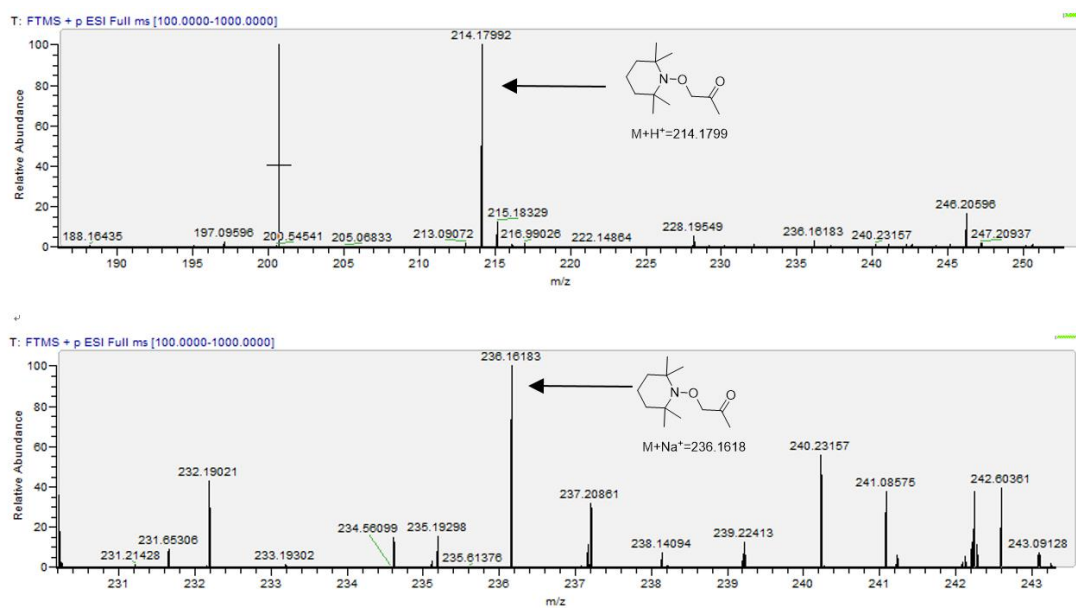
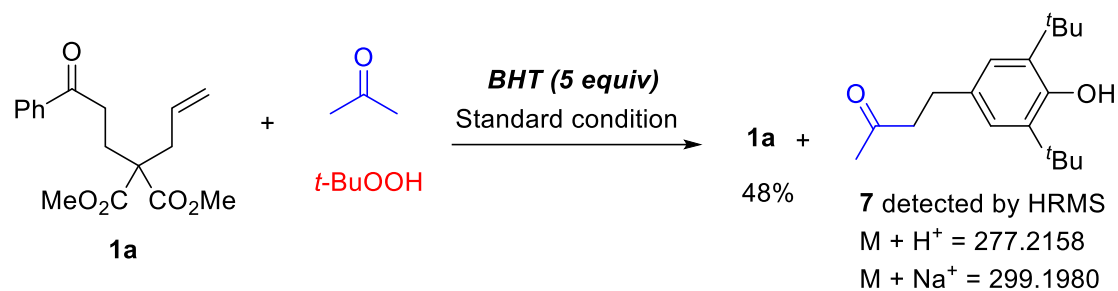


Figure 1. HRMS of the reaction solution



To a 10 mL sealable pressure tube was added unactivated alkenes **1a** (0.1 mmol), *p*-TsOH·H₂O (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 N₂ atmosphere at room temperature, and the resulting solution was stirred at 50 °C for 5 h. The model reaction was totally suppressed where alkene **1a** was almost fully recovered, and we can detect the molecular weight of compound **7** by HRMS (Figure 2).

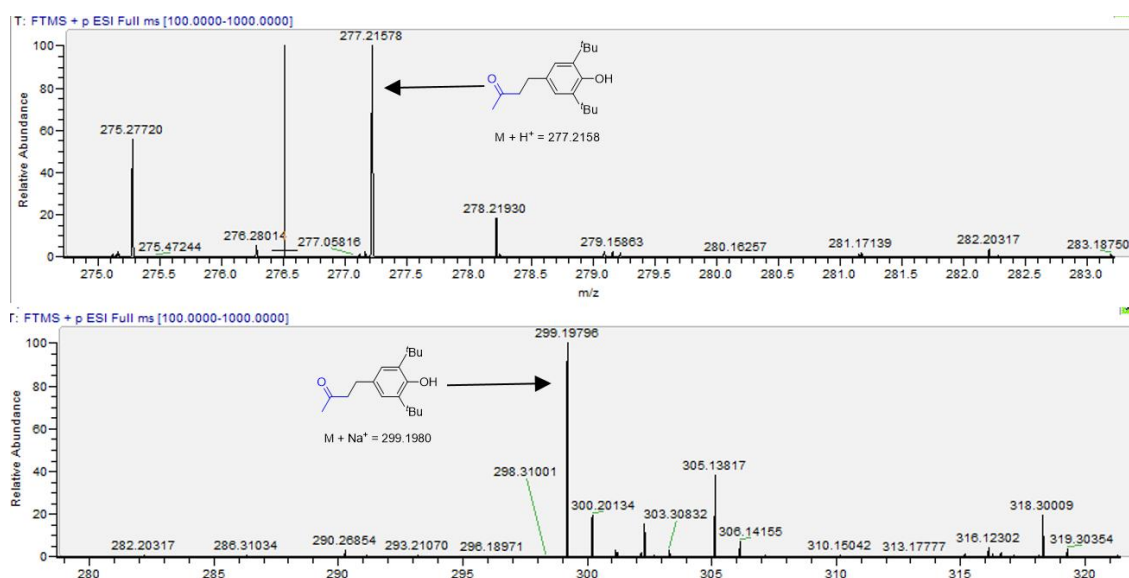
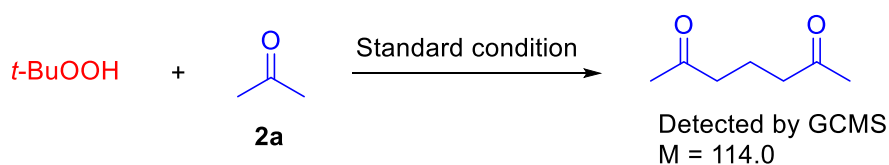


Figure 2. HRMS of the reaction solution



To a 10 mL sealable pressure tube was added *p*-TsOH·H₂O (0.03 mmol), acetone (0.6 mL) and *t*-BuOOH (5.5 M in decane, 0.5 mmol) under N₂ atmosphere at room temperature, and the resulting solution was stirred at 50 °C for 5 h. We can detect the molecular weight of 2,5-hexadione by GC-MS. (Figure 3)

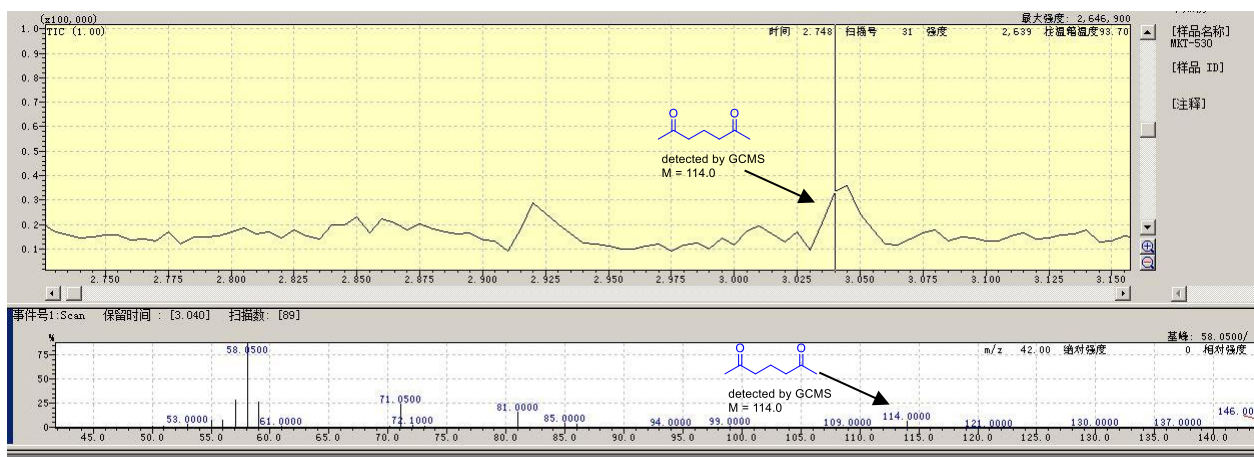
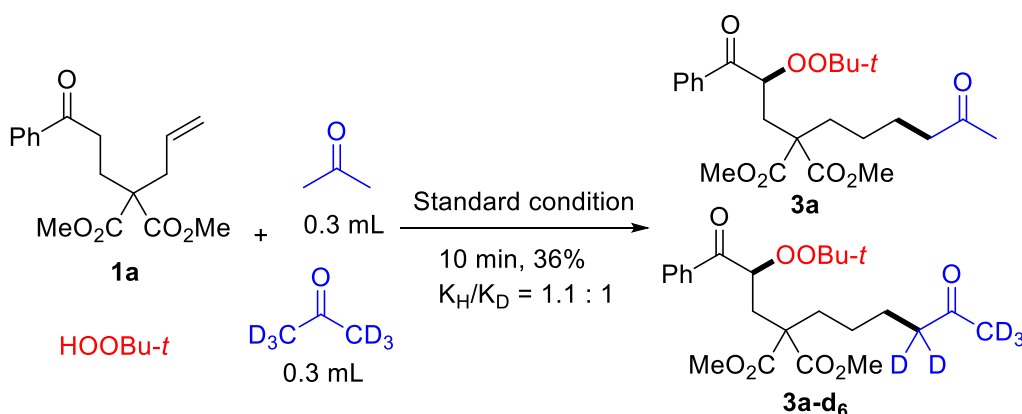


Figure 3. GC-MS of the reaction solution

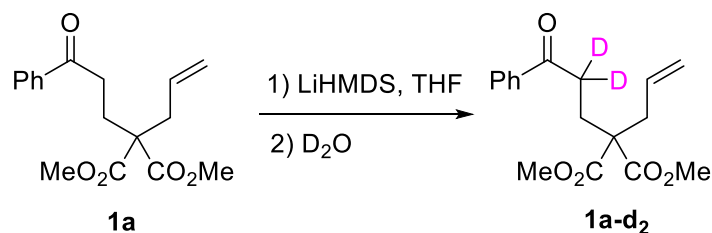
(b) Kinetic isotope effect (KIE) experiment



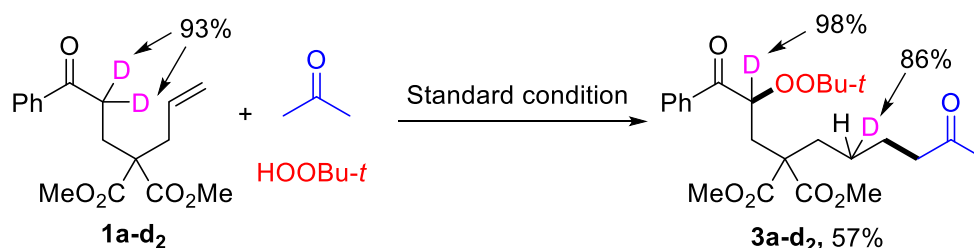
To a 10 mL sealable pressure tube was added unactivated alkenes **1a** (0.1 mmol), *p*-TsOH·H₂O (0.03 mmol), acetone (0.3 mL), acetone-d₆ (0.3 mL) and *t*-BuOOH (5.5 M in decane, 0.5 mmol) under N₂ atmosphere at room temperature, and the resulting solution was stirred at 50 °C for 10 minutes. The yield was 36% with a ratio of 1.1 : 1 (*k_H* : *k_D*), which suggested that the cleavage of the C(sp³)-H bond was not the rate-determining steps in this transformation. ¹H NMR (600 MHz, CDCl₃, 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 C₂₄H₂₉D₅O₈Na (M+Na)⁺: 478.2460; found: 478.2458.

(c) Deuterium labeling experiment

The deuterium unactivated alkene substrate **1a** was known compound. Unactivated alkene **1a-d₂** was prepared as followed procedure.¹



Experimental procedures for the synthesis of **1a-d₂**: To a solution of **1a** (608.0 mg, 2.0 mmol) in THF (3.0 mL) at -78 °C was slowly added a solution of LiHMDS (5.0 mL, 1.0 M in THF) under N₂. After being stirred for 1 h at -78 °C, the reaction mixture was quenched by slowly sequential addition of D₂O (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 Na₂SO₄, 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-**d₂** (**1a-d₂**) (535.0 mg, 88% yield) as a colorless oil. **1a-d₂**²: Colorless oil; ¹H NMR (400 MHz, CDCl₃, 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).



To a 10 mL sealable pressure tube was added unactivated alkenes **1a-d₂** (0.1 mmol), *p*-TsOH·H₂O (0.03 mmol), acetone (0.6 mL) and *t*-BuOOH (5.5 M in decane, 0.5 mmol) under N₂ atmosphere at room temperature, and the resulting solution was stirred at 50 °C 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-d₂** in 54% yield (24.3 mg).

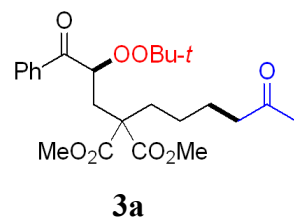
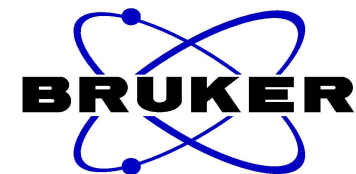
3a-d₂: Colorless oil; ¹H NMR (600 MHz, CDCl₃, ppm) δ 8.05 (d, *J* = 7.3 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.9 Hz, 2H), 5.15 (dd, *J* = 7.2, 5.2 Hz, 0.02H), 3.70 (s, 3H), 3.69 (s, 3H), 2.44-2.41 (m, 4H), 2.12 (s, 3H), 2.09-2.04 (m, 1H), 2.01-1.97 (m, 1H), 1.61-1.57 (m, 2H), 1.23-1.17 (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 $\text{C}_{24}\text{H}_{32}\text{D}_2\text{O}_8\text{Na}$ ($\text{M}+\text{Na}$) $^+$: 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 ^1H NMR and ^{13}C NMR, ^{19}F NMR spectra for all products

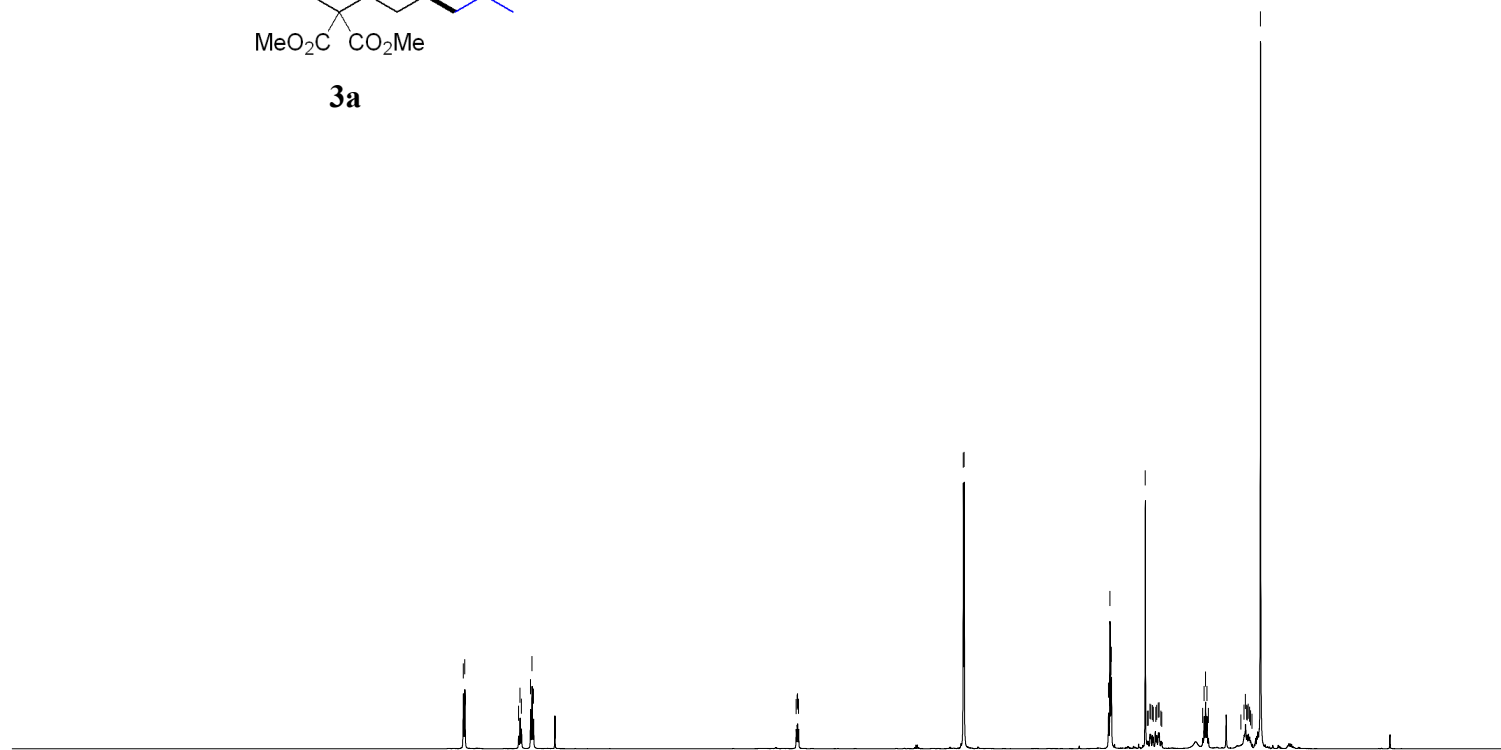


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LB 0.30 Hz
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PC 1.00



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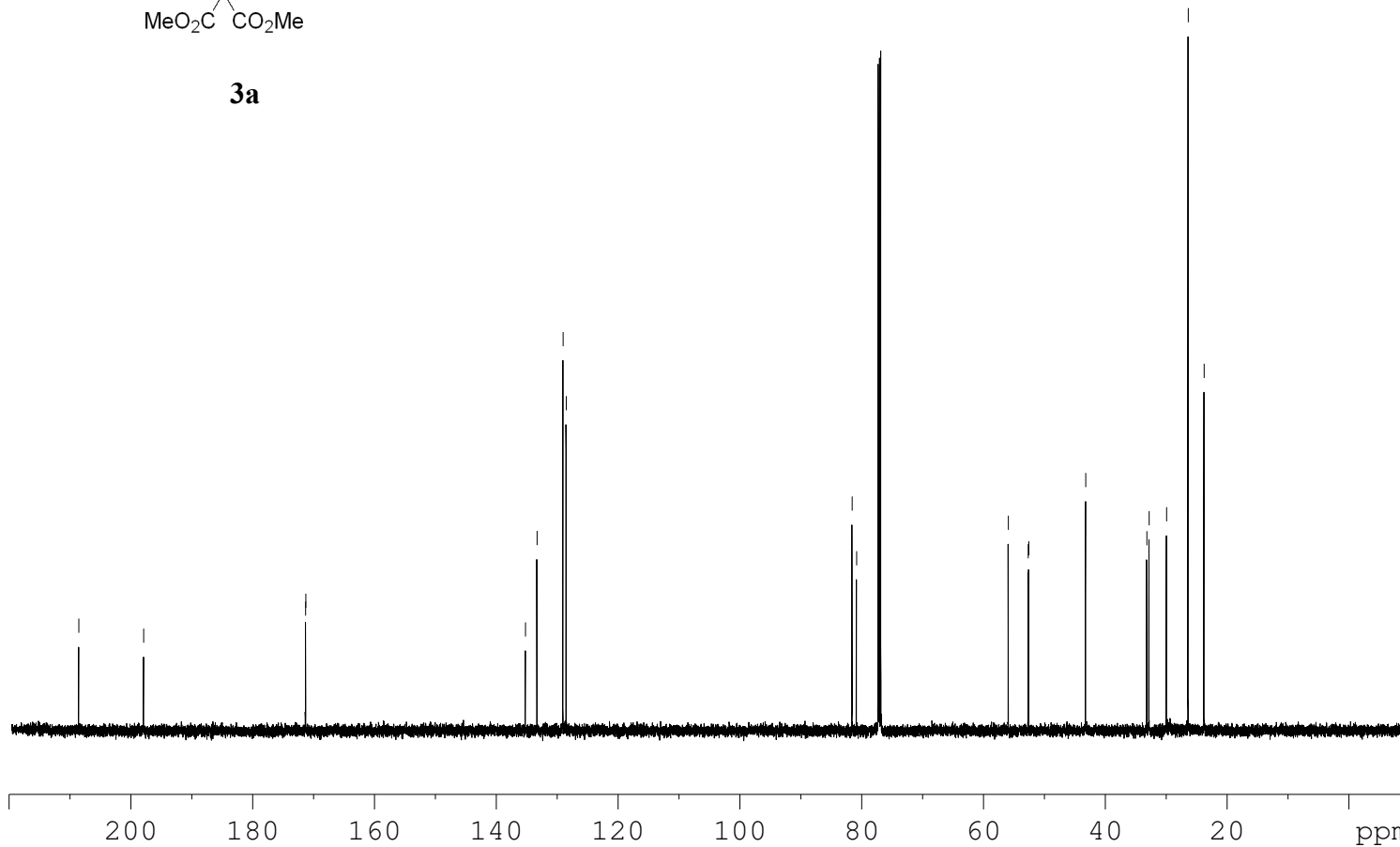
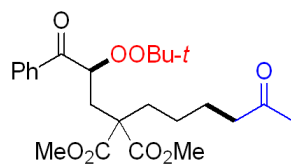
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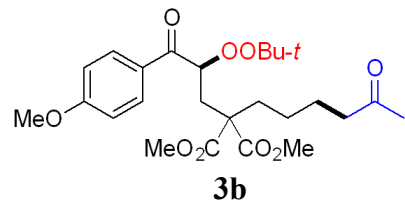
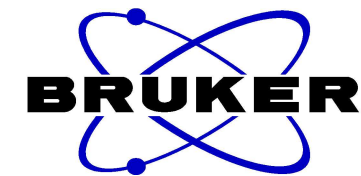
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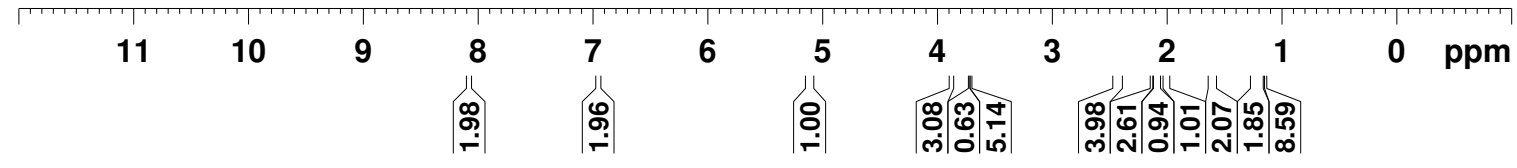
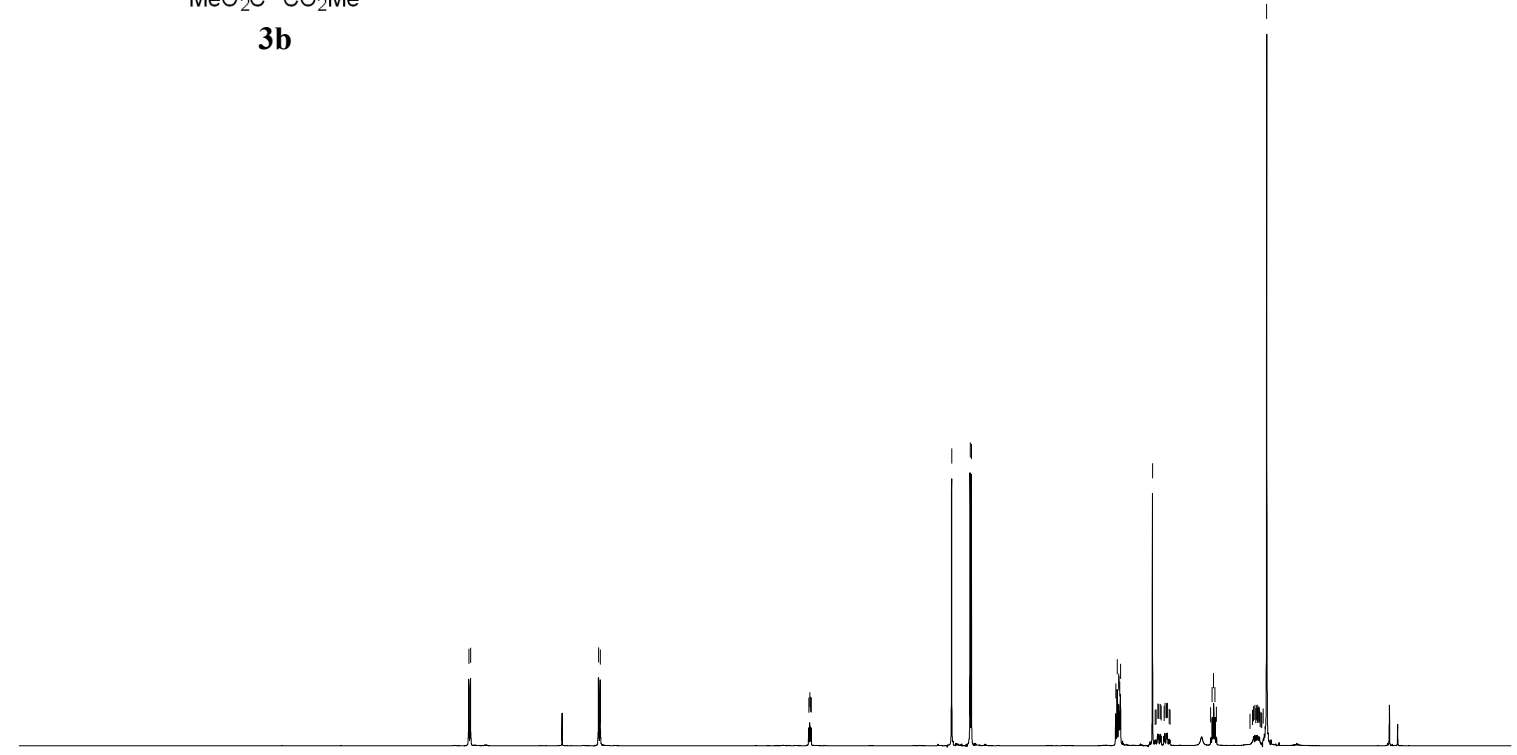
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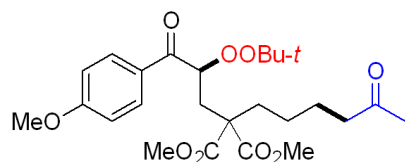
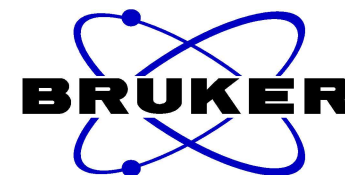
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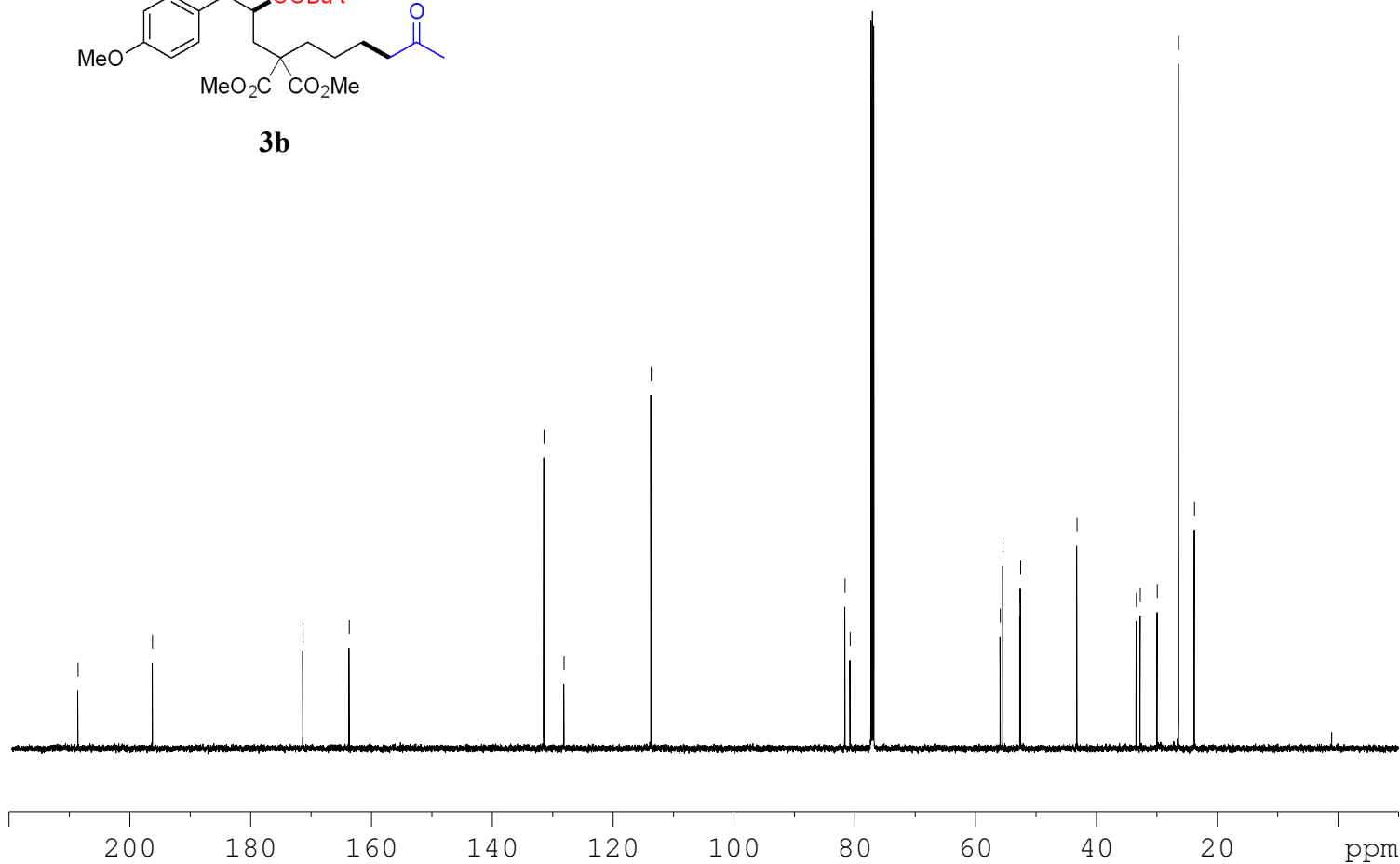
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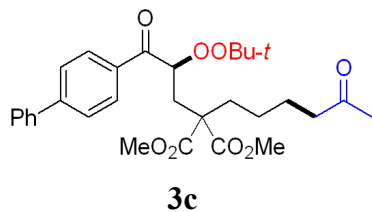
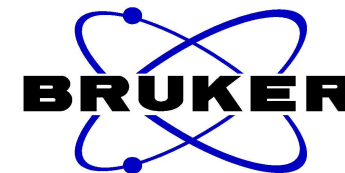


3b



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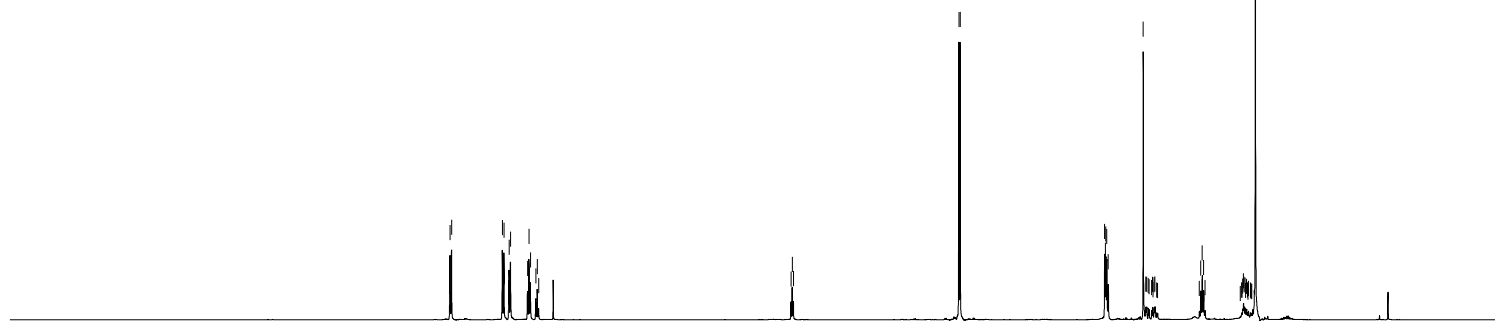
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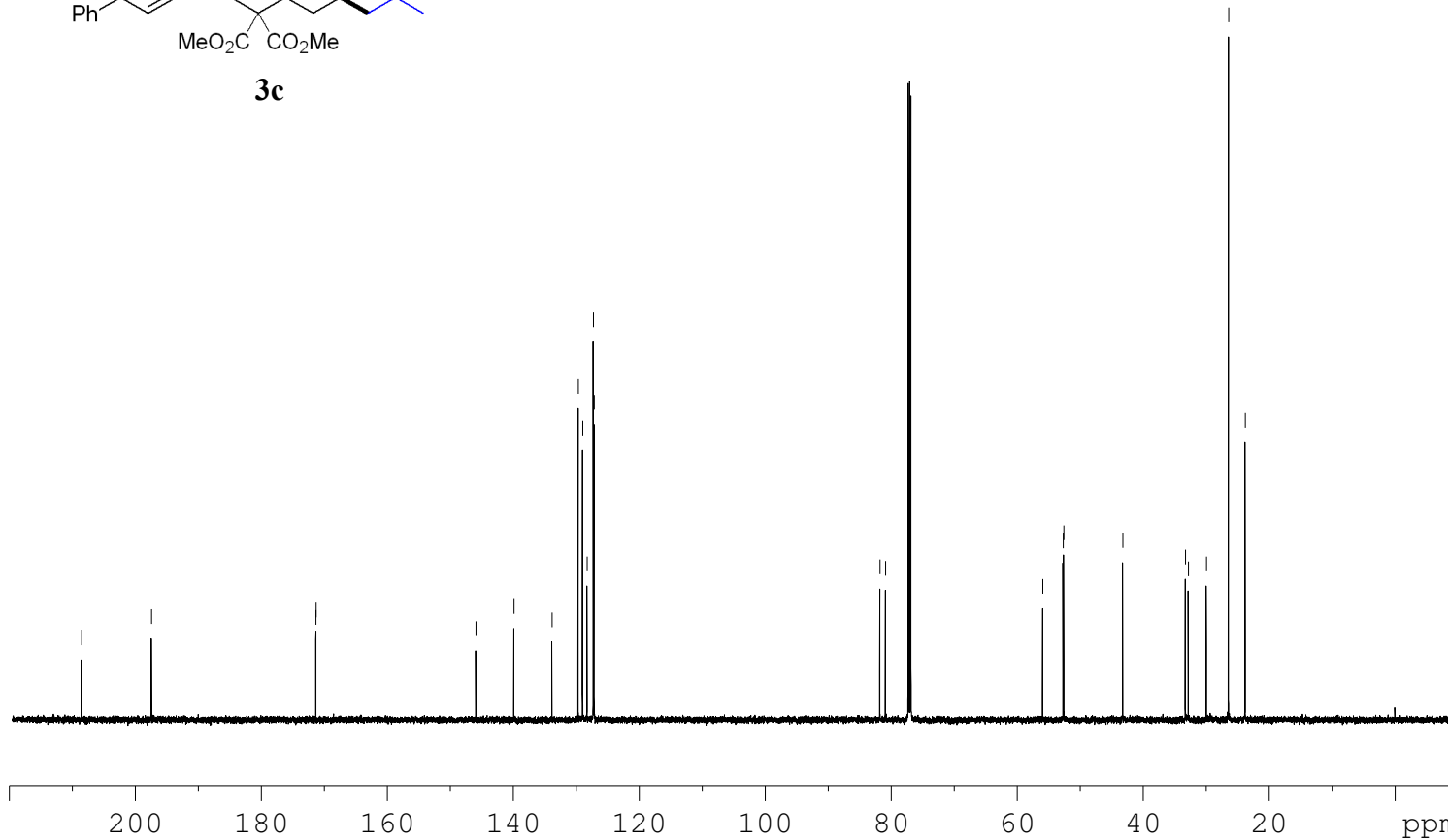
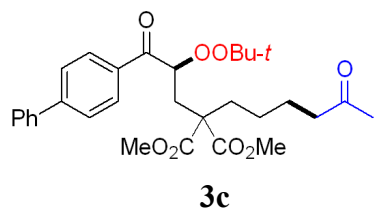
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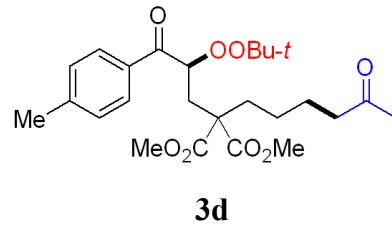
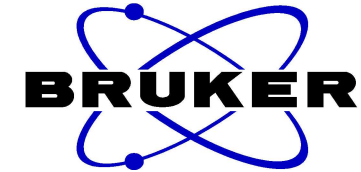
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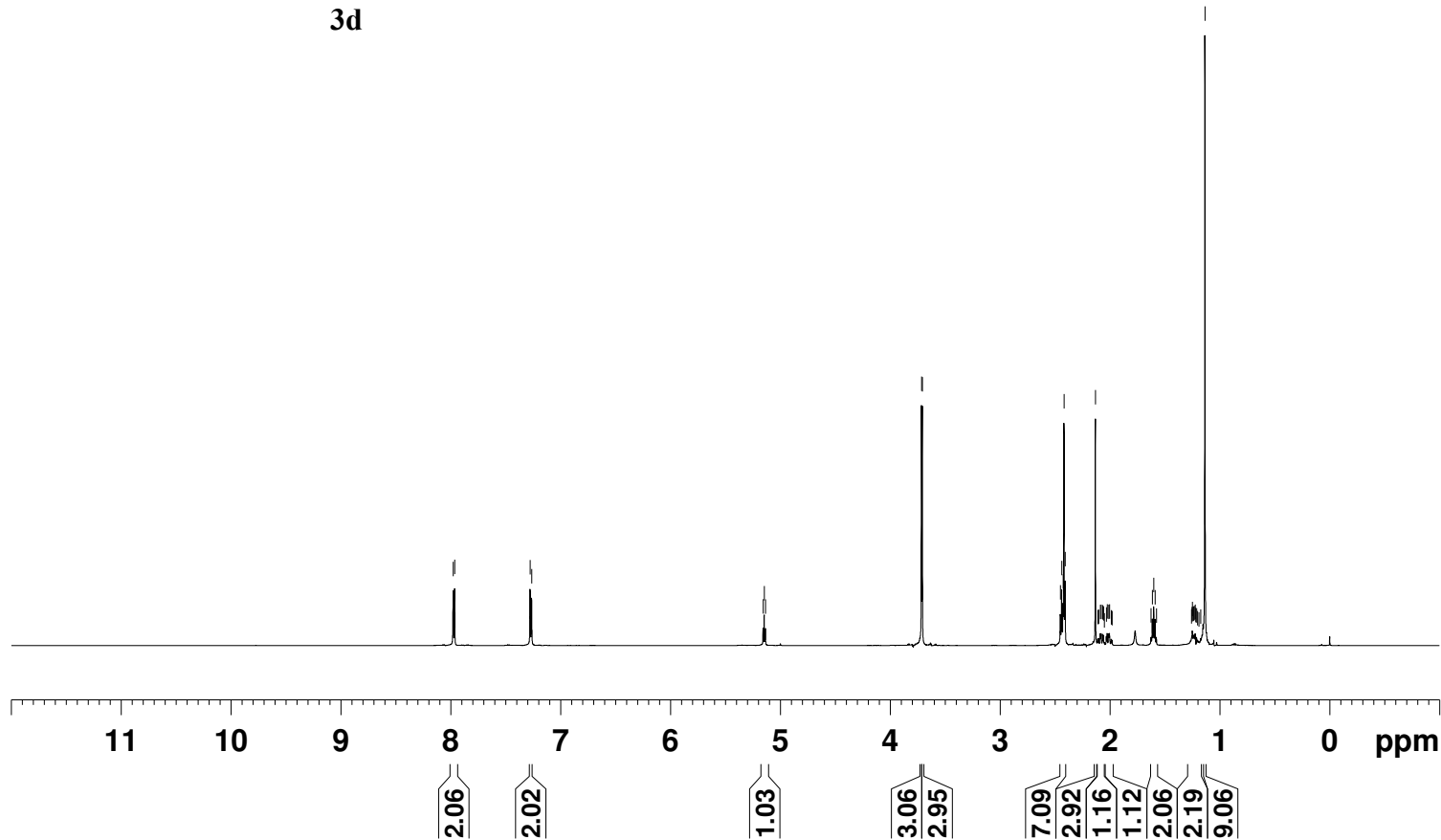
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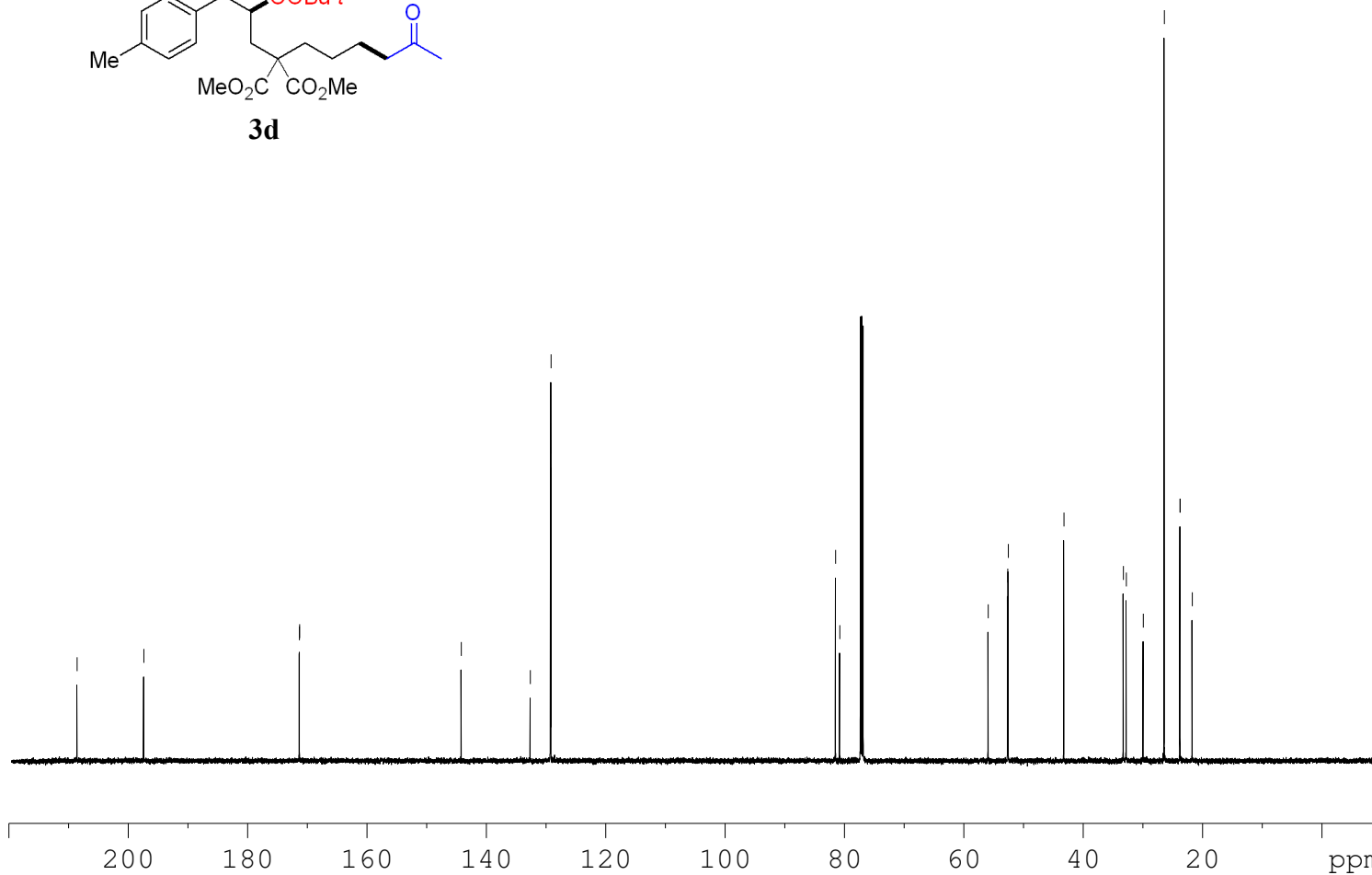
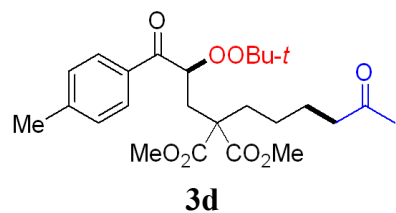
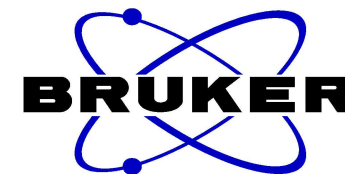
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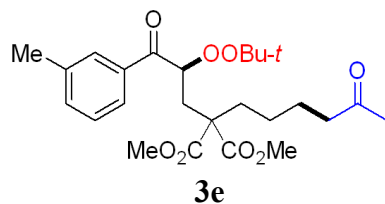
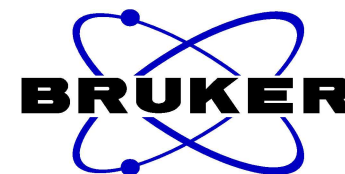
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< 29.931
< 26.398
< 23.771
< 23.760
< 21.722



```
NAME      w11-545p-20210608
EXPNO      2
PROCNO     1
Date_      20210609
Time       1.14
INSTRUM    spect
PROBHD     5 mm PABBO BB/
PULPROG    zgpg30
TD         65536
SOLVENT    CDC13
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         2.0000000 sec
D11        0.0300000 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
```

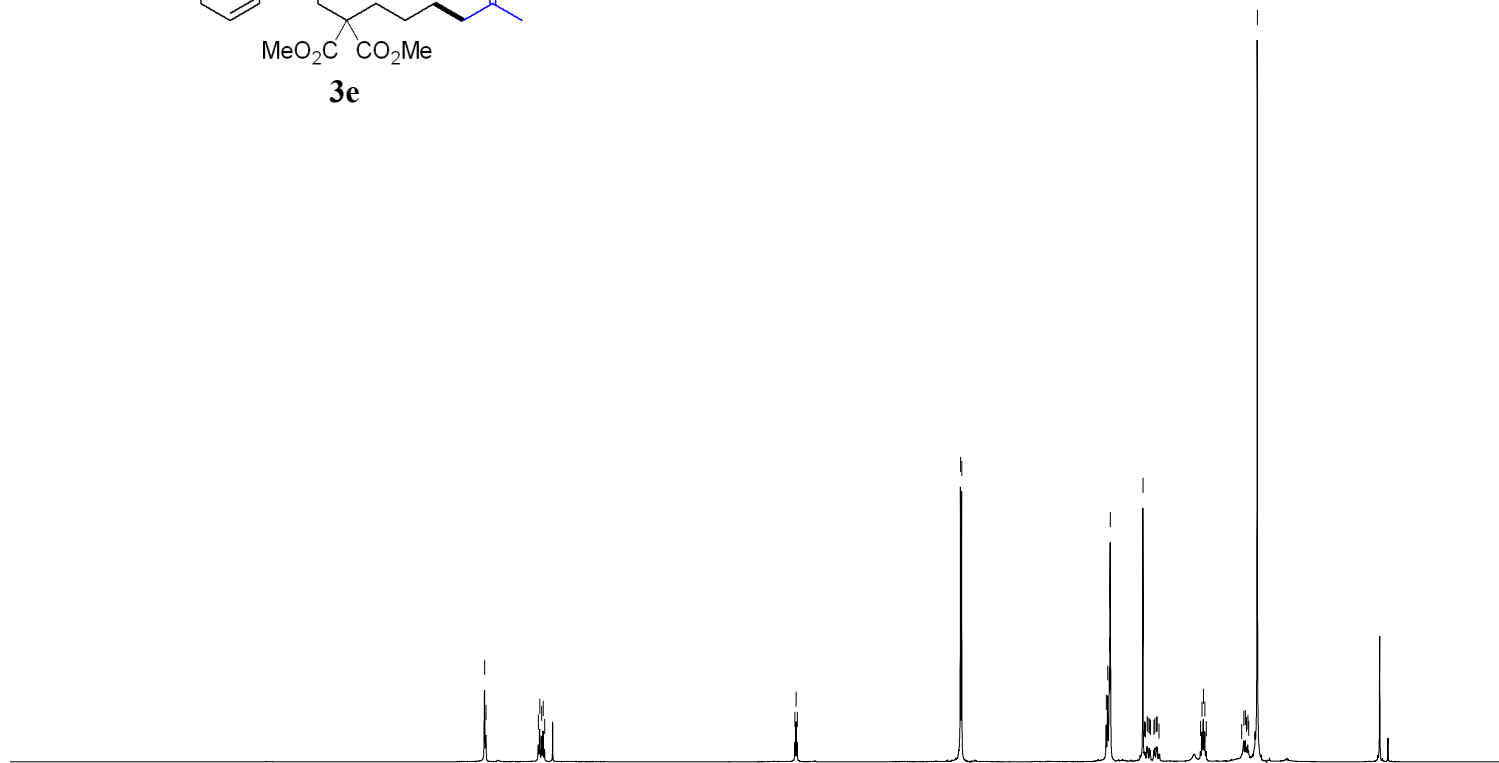


7.865
7.854
7.398
7.385
7.369
7.356
7.343

5.164
5.153
5.142
3.721
3.710
2.452
2.439
2.424
2.417
2.132
2.119
2.111
2.096
2.088
2.075
2.067
2.037
2.029
2.016
2.009
1.993
1.631
1.618
1.606
1.593
1.581
1.273
1.254
1.241
1.231
1.218
1.212
1.136

NAME w11-552p-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



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

1.94
 1.96
 1.00
 2.98
 3.03
 7.07
 2.99
 1.16
 1.05
 2.07
 2.07
 9.04

— 208.507

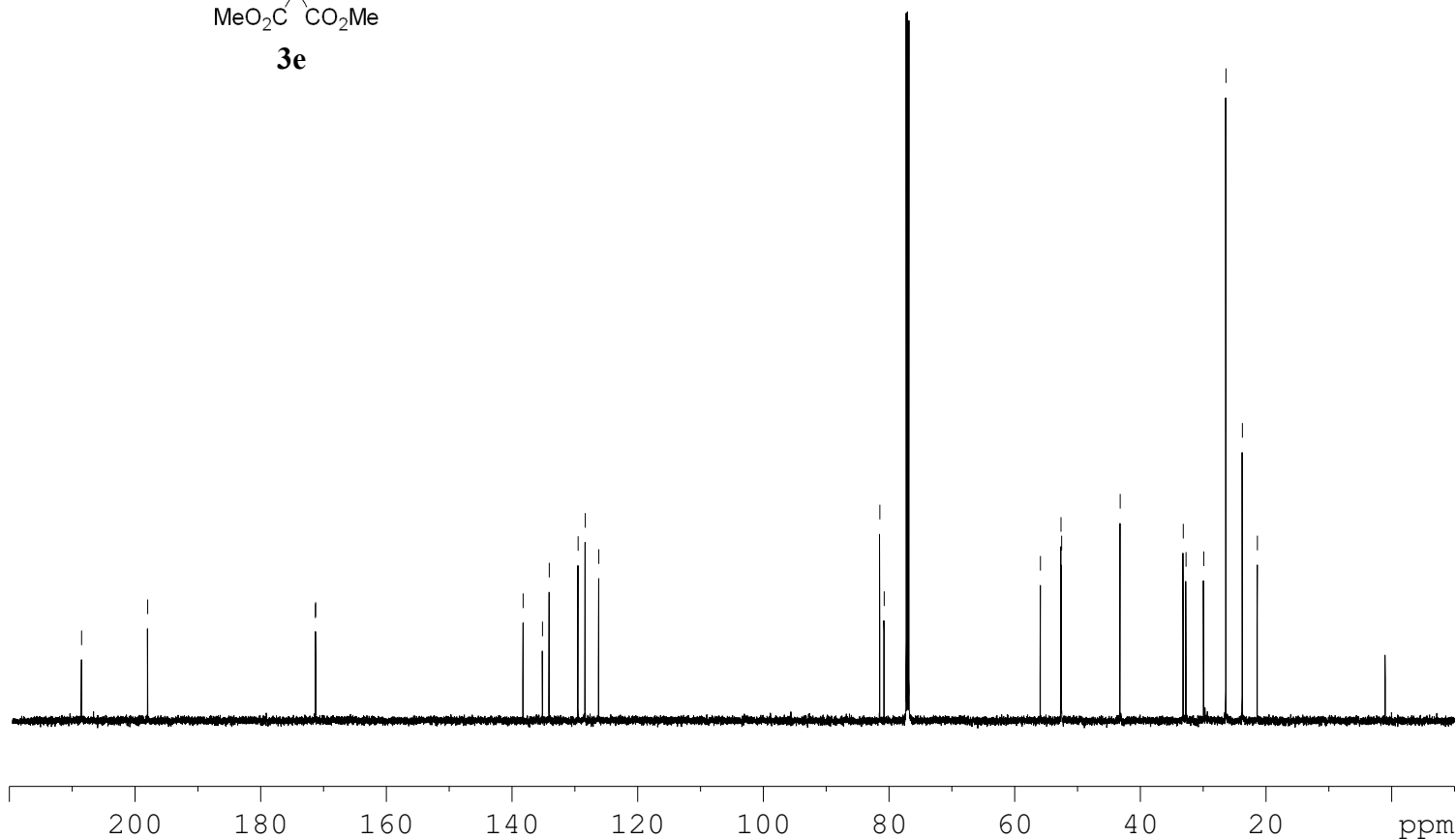
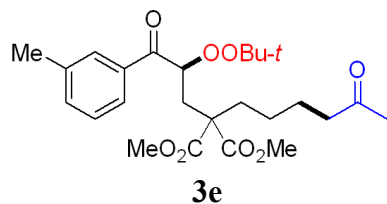
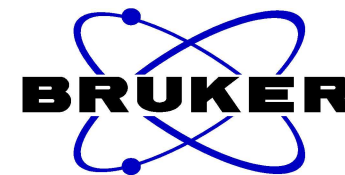
— 197.988

< 171.301
< 171.249

< 138.233
< 135.150
< 134.072
< 129.481
< 128.345
< 126.192

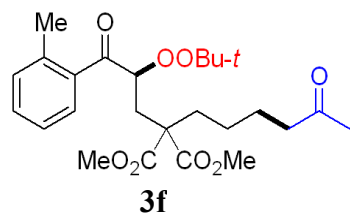
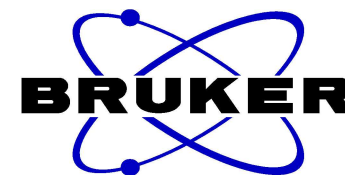
< 81.460
< 80.765

< 55.902
< 52.630
< 52.529
< 43.210
< 33.169
< 32.728
< 29.910
< 26.373
< 23.758
< 21.371



NAME w11-552p-20210610
EXPNO 2
PROCNO 1
Date_ 20210611
Time 5.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
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

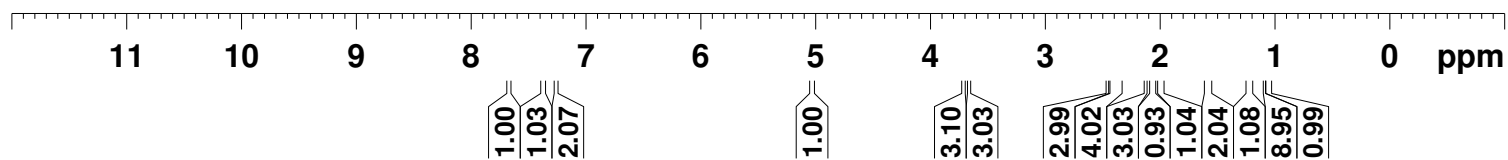
==== CHANNEL f1 =====
SF01 150.9279571 MHz
NUC1 13C
P1 14.00 usec
SI 32768
SF 150.9128704 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



7.673
7.661
7.383
7.381
7.370
7.369
7.358
7.356
7.275
7.265
7.253
7.242
5.040
5.035
5.025
5.020
3.710
3.659
2.459
2.435
2.430
2.422
2.409
2.405
2.397
2.369
2.354
2.343
2.328
2.121
2.088
2.081
2.065
2.058
2.044
2.036
2.018
2.010
1.997
1.989
1.974
1.966
1.606
1.603
1.592
1.580
1.567
1.556
1.554
1.242
1.234

NAME w11-549p-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



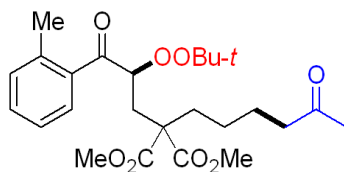
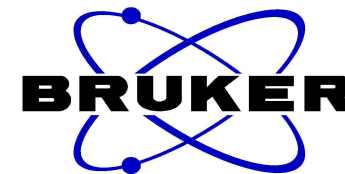
— 208.492
— 202.095

< 171.340
< 171.247

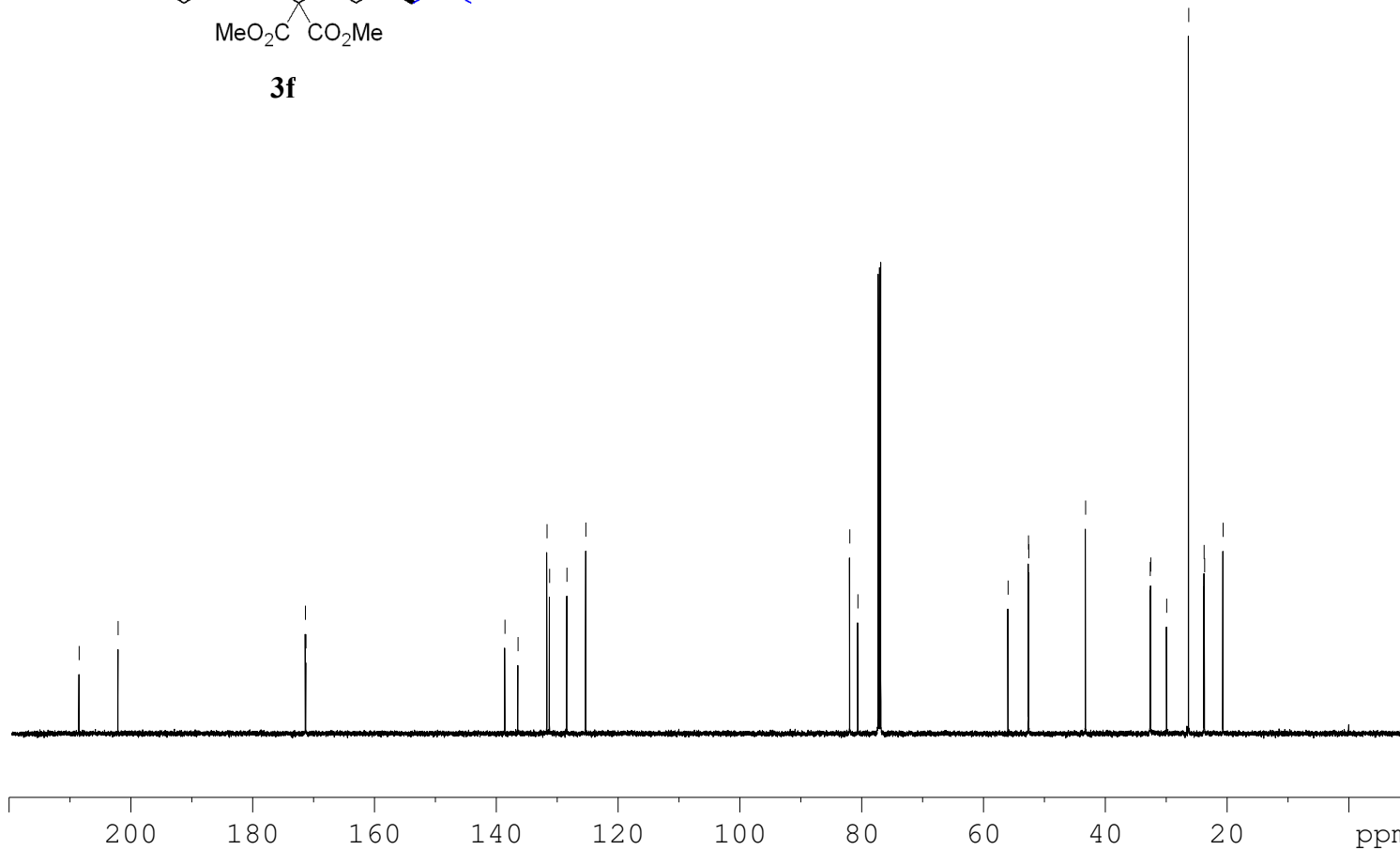
< 138.572
< 136.427
< 131.666
< 131.245
< 128.397
< 125.294

< 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

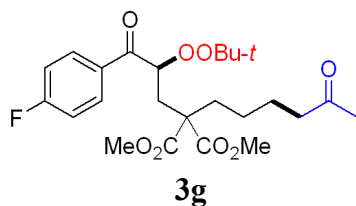


3f



NAME w11-549p-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

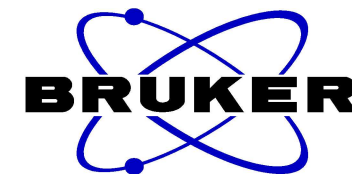
==== CHANNEL f1 =====
SFO1 150.9279571 MHz
NUC1 13C
P1 14.00 usec
SI 32768
SF 150.9128665 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



8.149
8.140
8.135
8.126

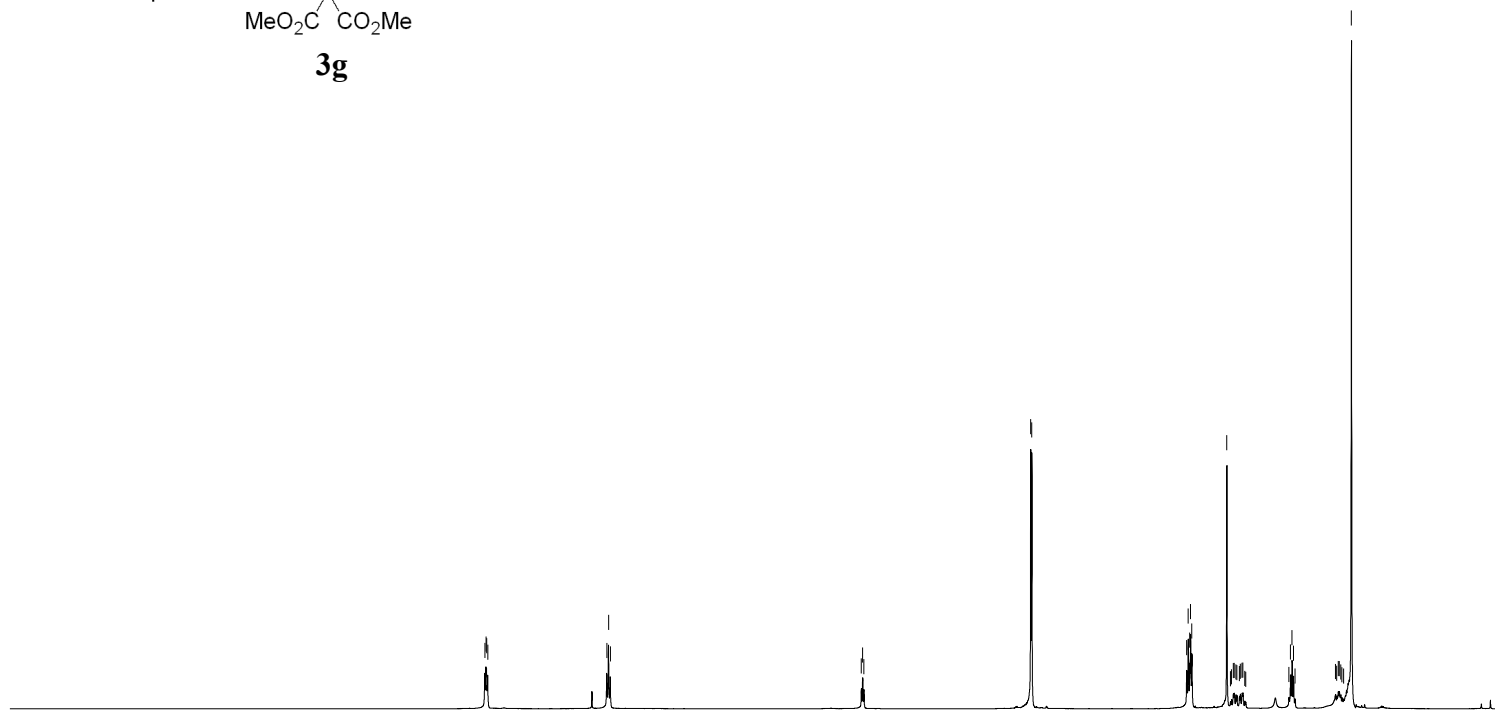
7.161
7.147
7.132

5.099
5.088
5.078
3.726
3.717
2.462
2.450
2.437
2.430
2.420
2.137
2.107
2.099
2.083
2.076
2.063
2.055
2.034
2.027
2.013
2.006
1.990
1.983
1.633
1.621
1.608
1.596
1.583
1.255
1.246



NAME w11-546p-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



11 10 9 8 7 6 5 4 3 2 1 ppm

2.10

2.07

1.00

3.00
2.96

4.13

2.99

1.17

1.13

2.10

2.16

9.01

— 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

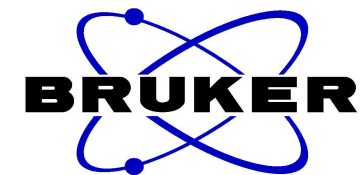
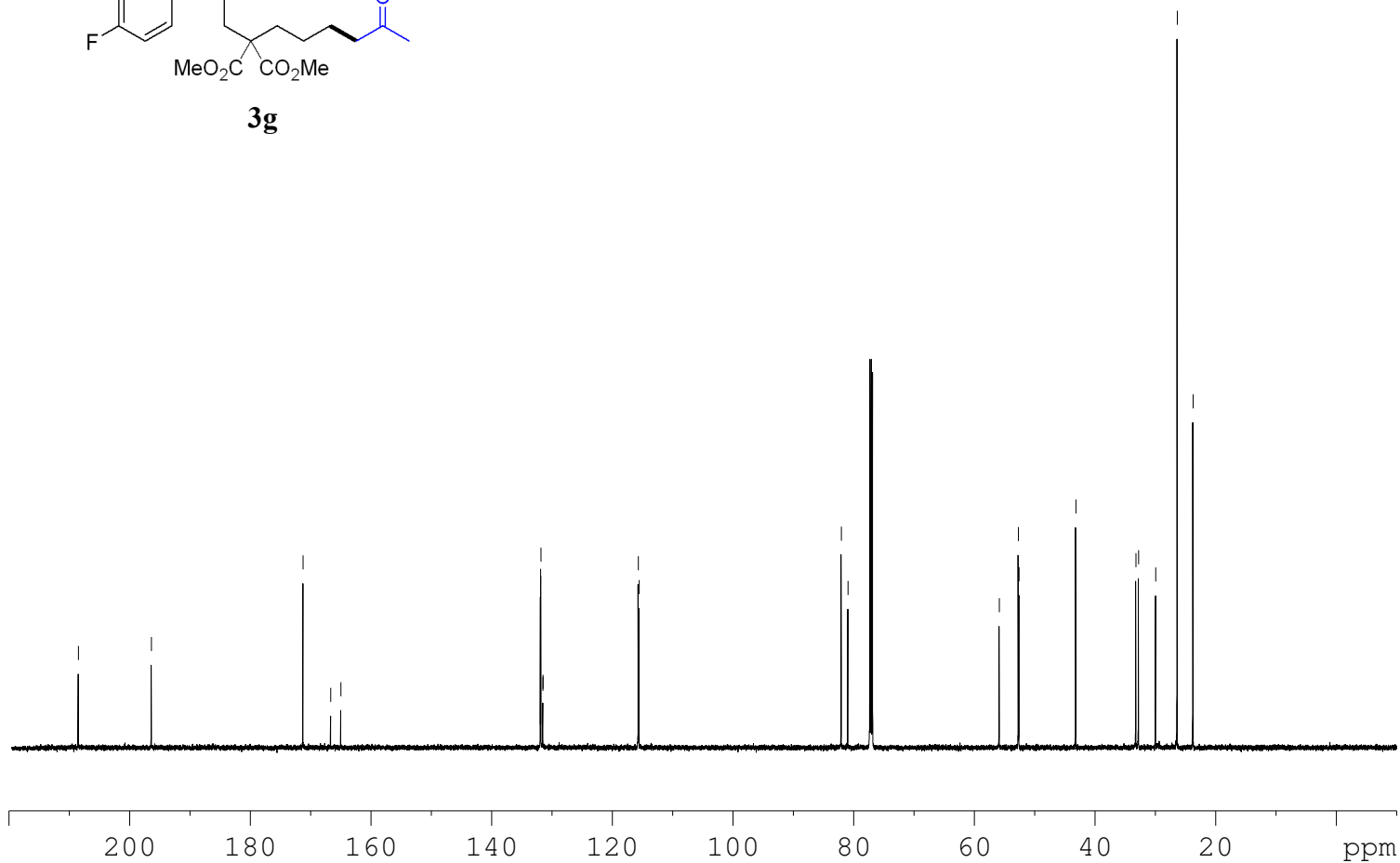
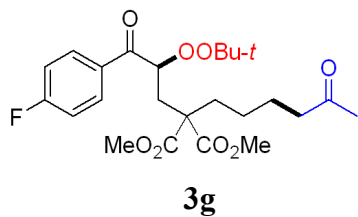
33.217

32.778

29.941

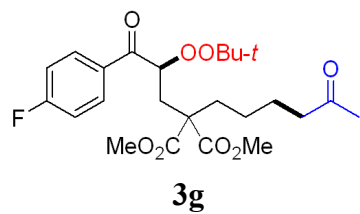
26.362

23.746

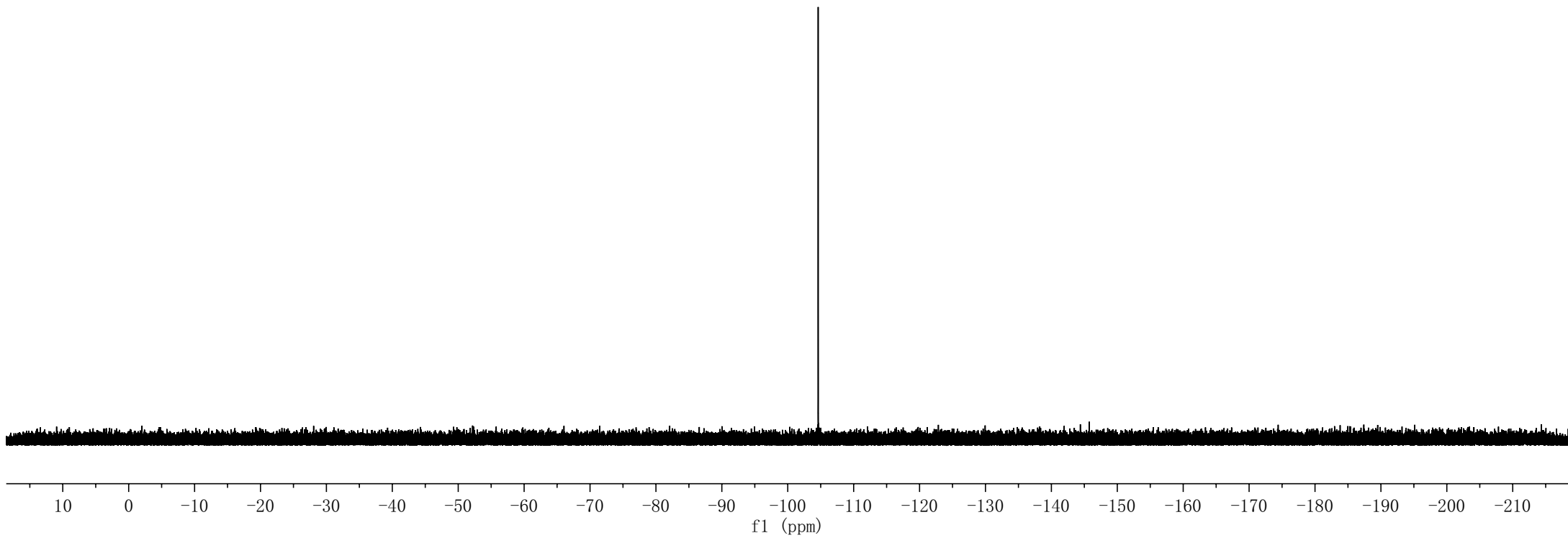


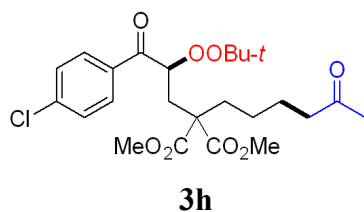
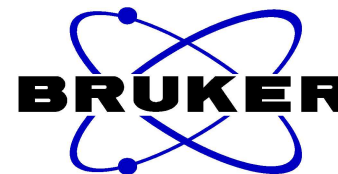
```
NAME      w11-546p-20210608
EXPNO     2
PROCNO    1
Date_     20210609
Time      2.35
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        400
DS        4
SWH       36057.691 Hz
FIDRES    0.550197 Hz
AQ        0.9088159 sec
RG        190.02
DW        13.867 usec
DE        6.50 usec
TE        297.0 K
D1        2.0000000 sec
D11       0.03000000 sec
TD0       1

===== CHANNEL f1 =====
SF01     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
```



—104.6157



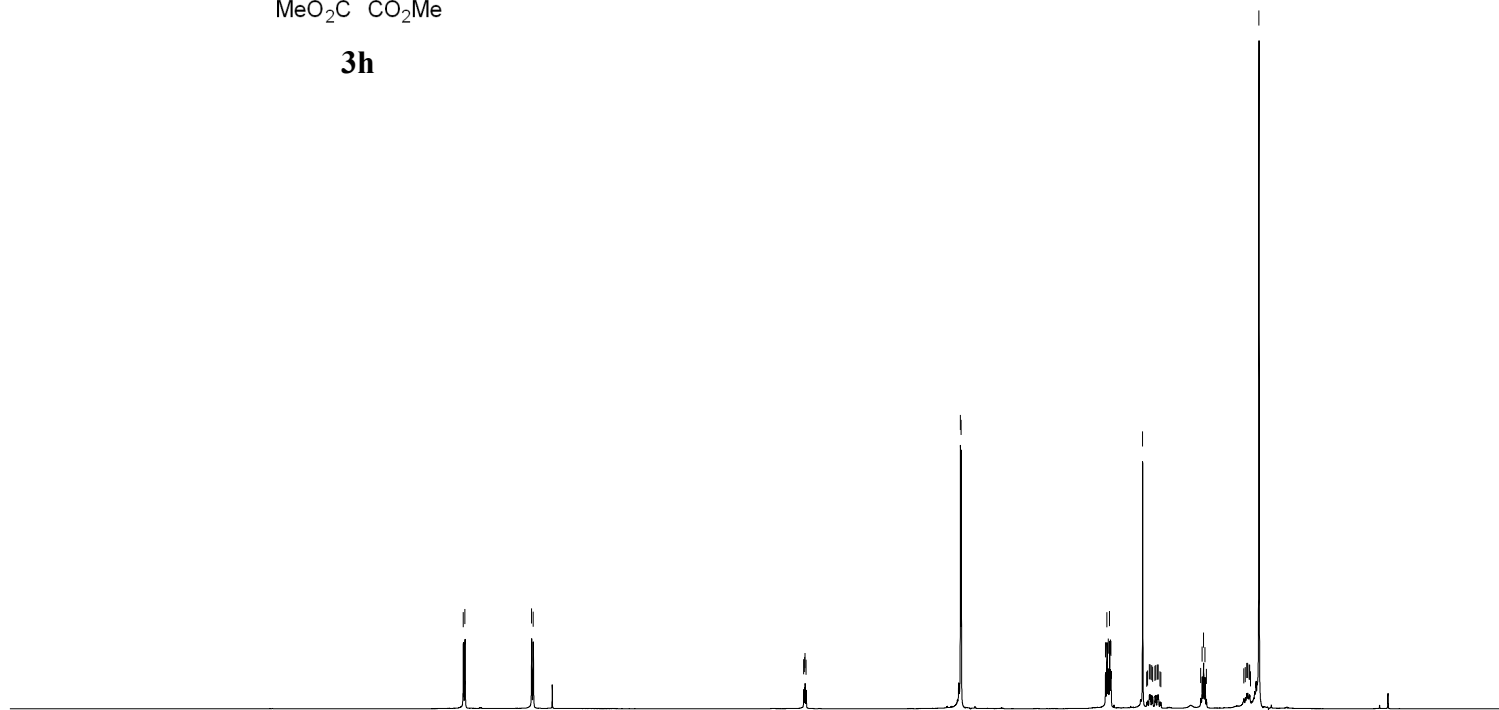


8.052
8.038
7.457
7.443

5.088
5.079
5.076
5.067
3.723
3.716
2.459
2.447
2.435
2.424
2.416
2.412
2.136
2.100
2.092
2.077
2.070
2.056
2.048
2.029
2.021
2.007
2.000
1.984
1.977
1.630
1.618
1.605
1.593
1.580
1.254
1.242
1.229
1.221
1.208

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 CDC13
 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



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

2.05
2.09

1.00

3.04
3.00

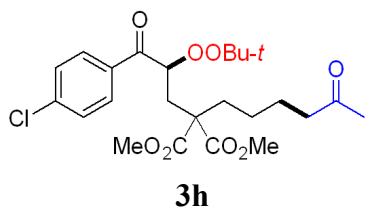
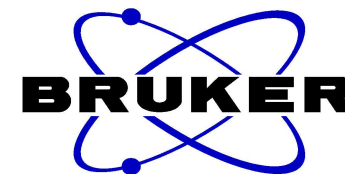
4.03
3.07
1.09
1.07
2.14
1.95
9.06

— 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

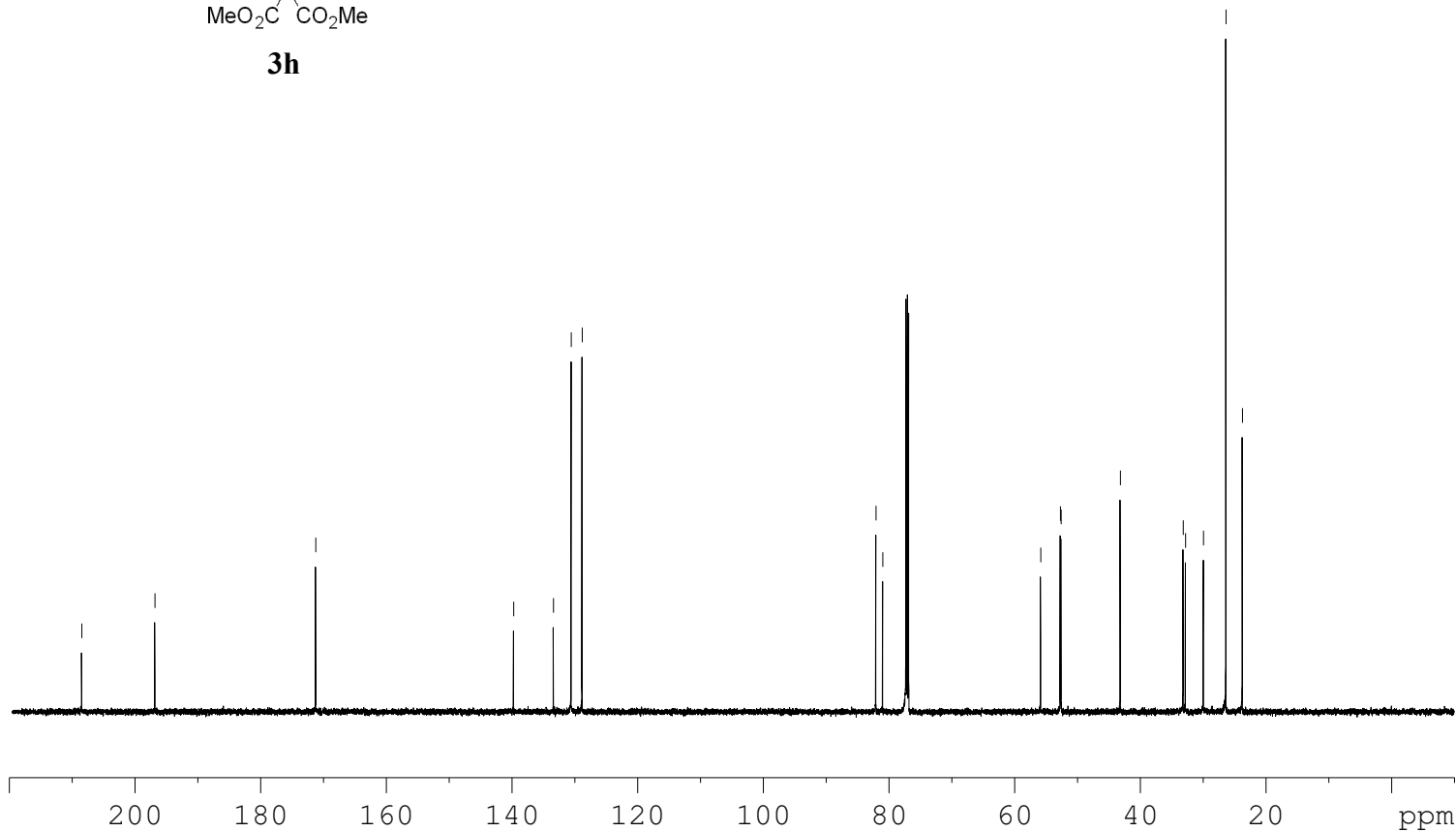


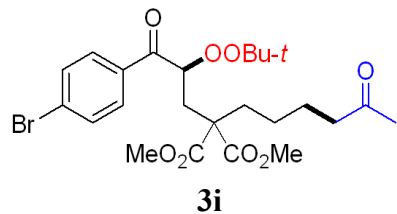
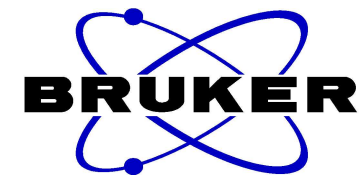
```

NAME      w11-547p-20210610
EXPNO     2
PROCNO    1
Date_     20210610
Time      23.55
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDC13
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
  
```

```

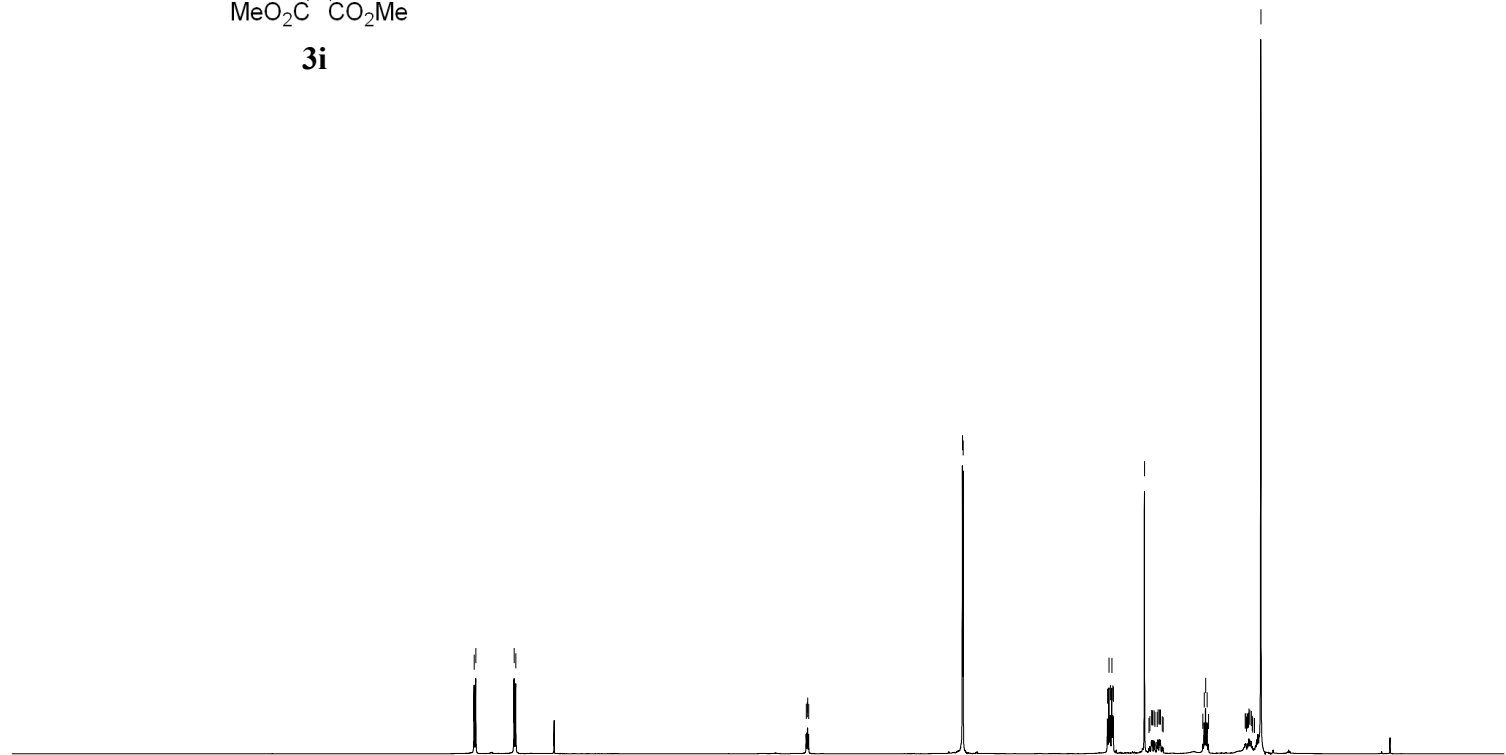
===== CHANNEL f1 =====
SFO1     150.9279571 MHz
NUC1     13C
P1       14.00 usec
SI       32768
SF       150.9128665 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
  
```





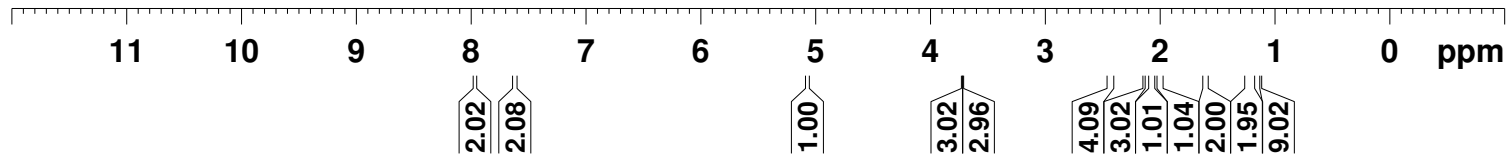
7.974
7.960
7.626
7.611

5.082
5.073
5.069
5.061
3.721
3.716
2.458
2.446
2.433
2.420
2.412
2.407
2.136
2.098
2.090
2.074
2.067
2.054
2.046
2.026
2.018
2.005
1.997
1.982
1.974
1.629
1.616
1.604
1.591
1.579
1.260
1.254
1.249
1.240
1.231
1.227



NAME w11-553p-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



2.02
2.08
1.00
3.02
2.96
4.09
3.02
1.01
1.04
2.00
1.95
9.02

— 208.471

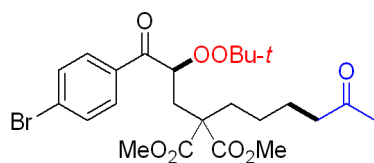
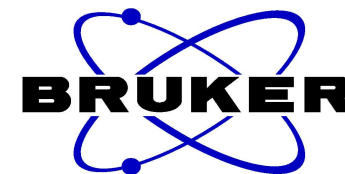
— 197.032

— 171.225

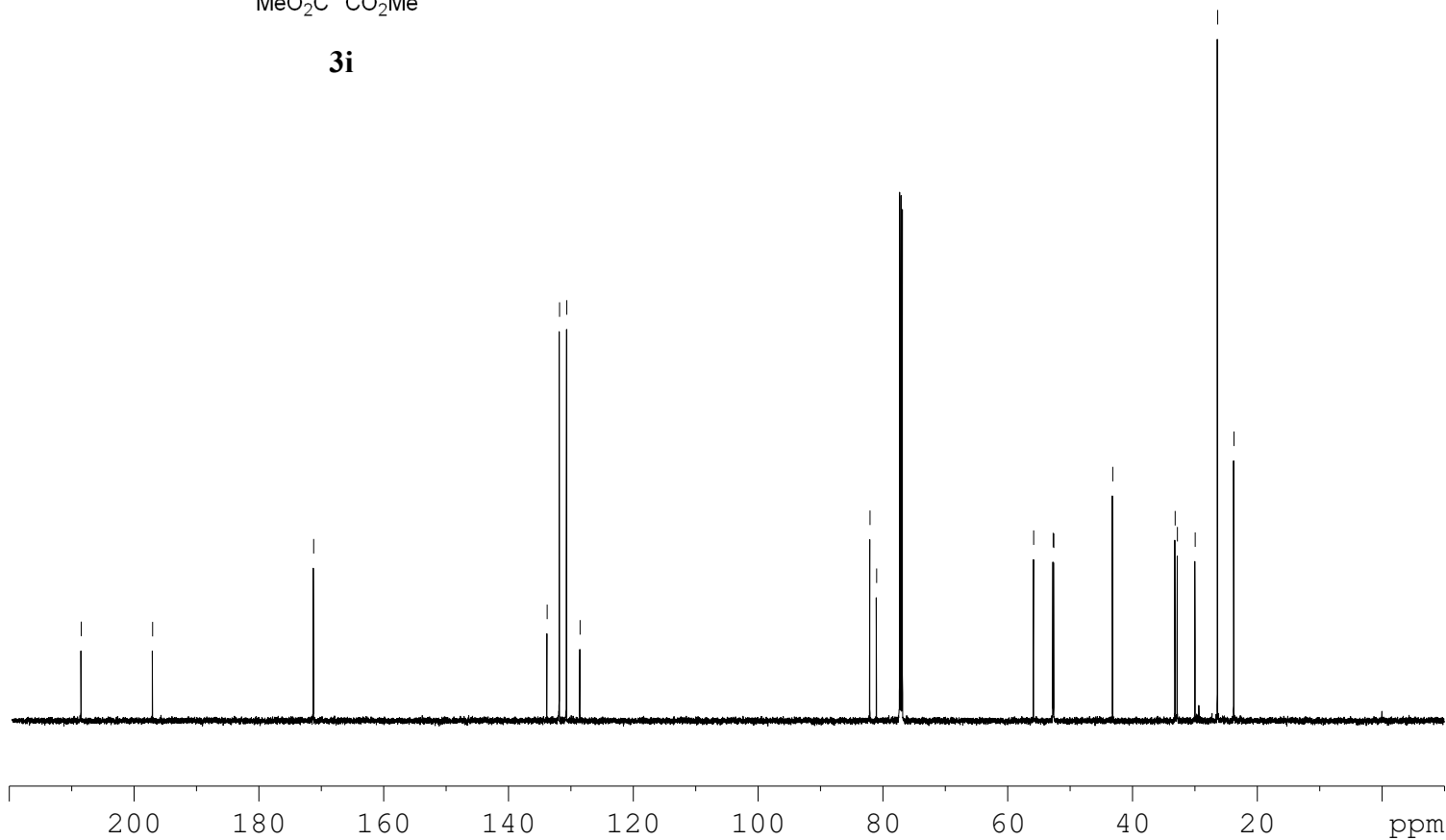
133.803
131.820
130.667
128.526

82.068
81.006

55.842
52.718
52.606
43.169
33.164
32.806
29.956
26.362
23.743

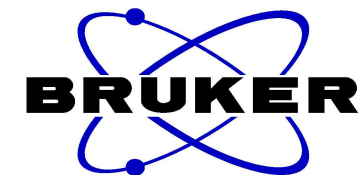


3i



NAME w11-553p-20210610
EXPNO 2
PROCNO 1
Date_ 20210611
Time 5.36
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
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

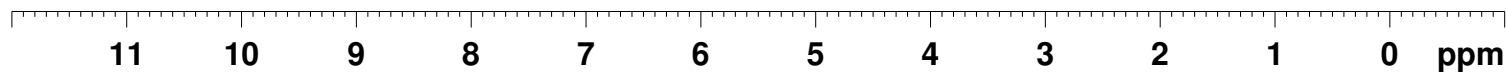
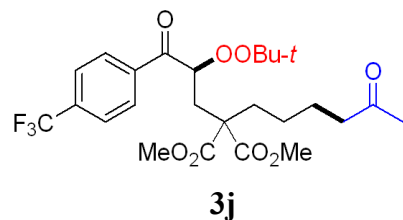


NAME w11-557p-20210618
EXPNO 1
PROCNO 1
Date_ 20210618
Time 10.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 0
SWH 9615.385 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 62.22
DW 52.000 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1

==== 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

8.213
8.200
7.751
7.737

5.120
5.113
5.107
5.099
3.730
3.723
2.473
2.465
2.452
2.440
2.427
2.415
2.401
2.138
2.107
2.099
2.083
2.076
2.063
2.055
2.038
2.030
2.017
2.010
1.994
1.986
1.637
1.624
1.612
1.599
1.587
1.256
1.248
1.235
1.226



2.00
2.04

0.99

3.19
2.97

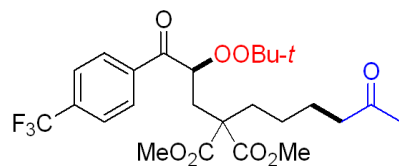
4.05
2.95
1.04
1.05
2.13
1.98
9.03

— 208.432

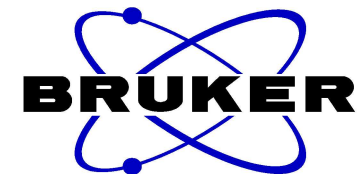
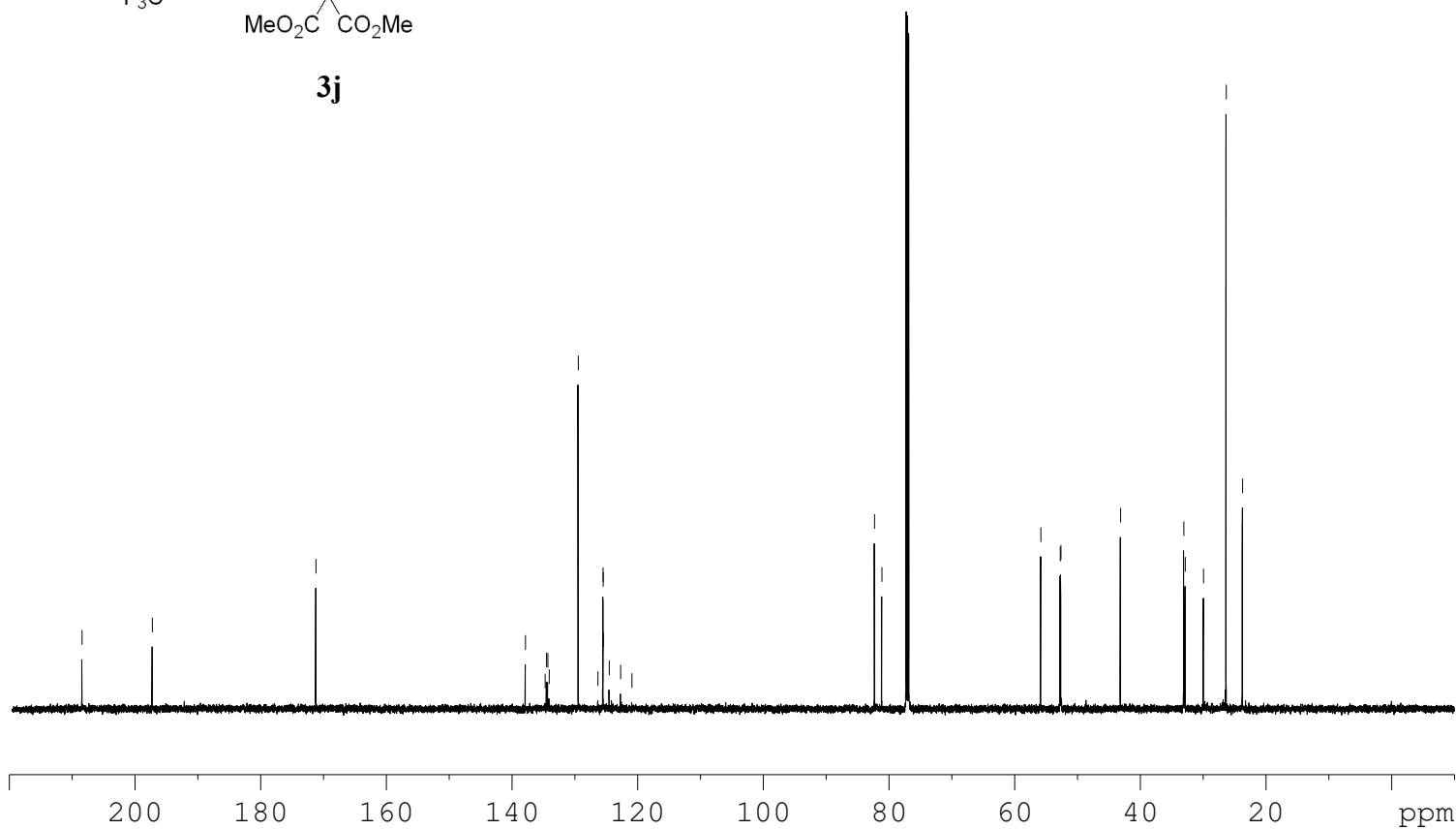
— 197.241

— 171.205

137.859
134.510
134.293
134.078
129.449
125.512
125.489
124.522
122.717
82.309
81.127
55.834
52.744
52.638
43.148
33.077
32.847
29.947
26.324
23.756
23.729
125.534
125.462
126.330
120.919
134.731

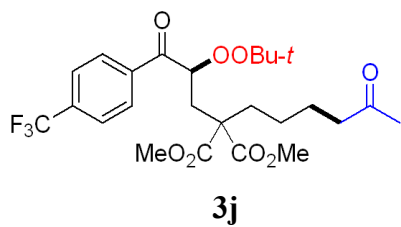


3j

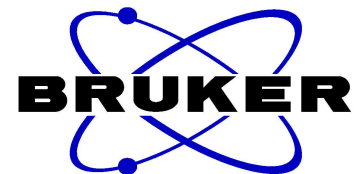


NAME w11-557p-20210618
EXPNO 2
PROCNO 1
Date_ 20210618
Time 12.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 190.02
DW 13.867 usec
DE 6.50 usec
TE 296.9 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== 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



--63.180

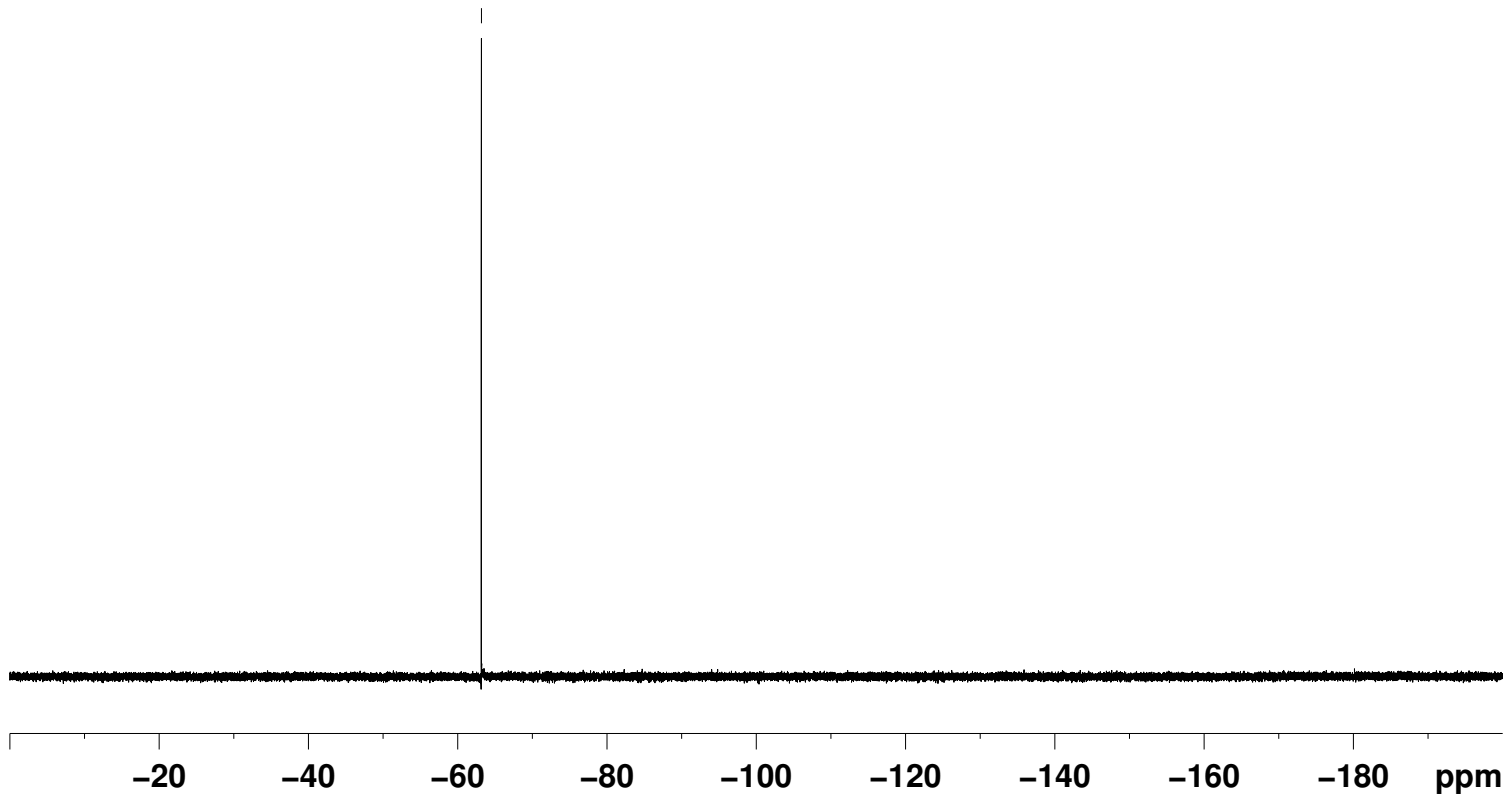


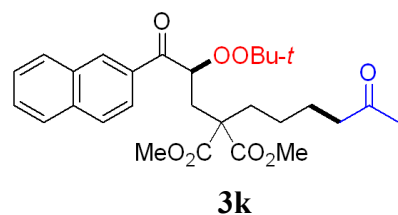
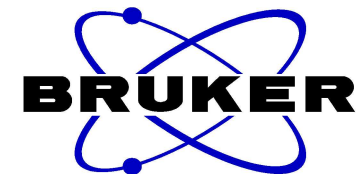
```

NAME      w11-557p-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
  
```

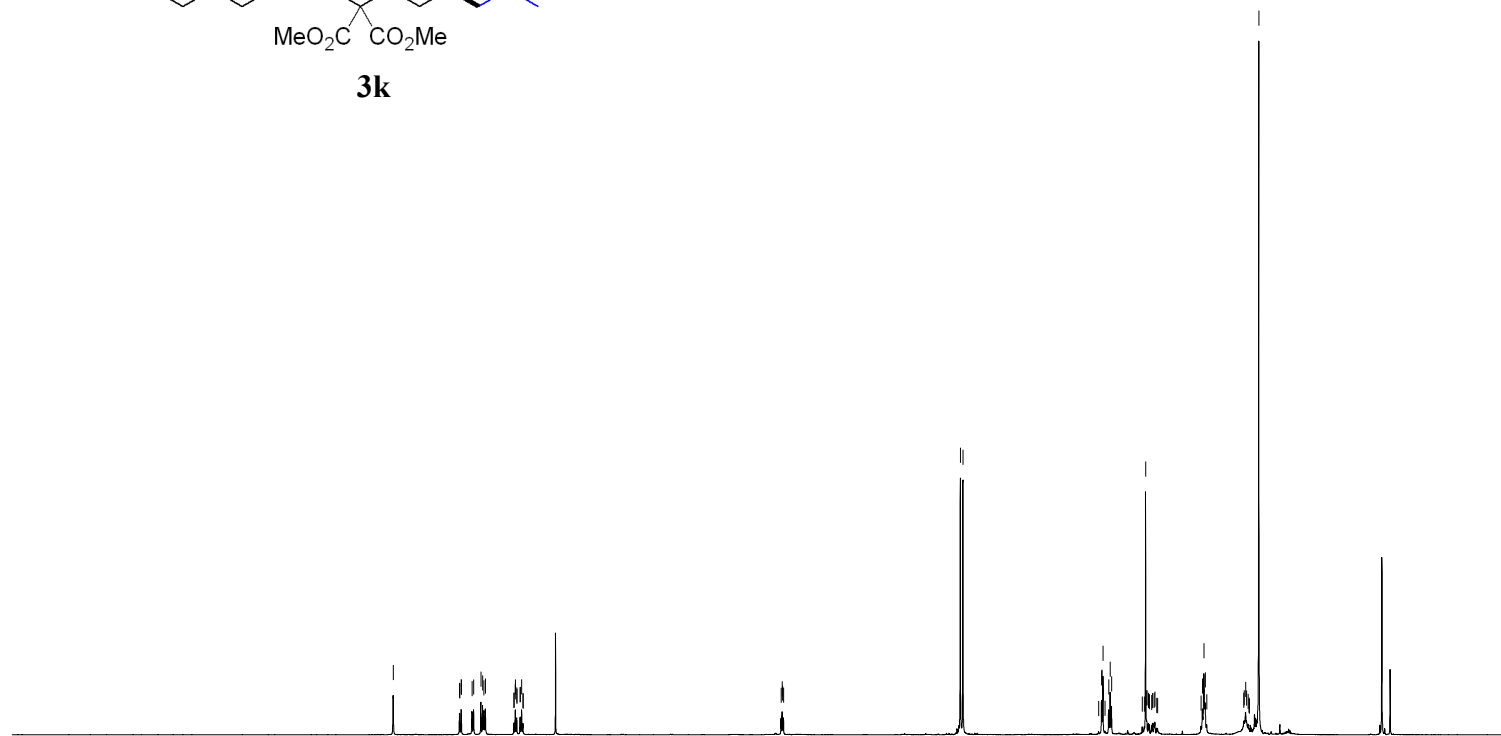




8.678
8.102
8.099
8.087
8.085
7.992
7.979
7.915
7.900
7.890
7.876
7.628
7.626
7.616
7.615
7.603
7.601
7.574
7.572
7.561
7.549
7.547
5.300
5.291
5.288
5.279
3.739
3.717
2.536
2.510
2.506
2.497
2.480
2.447
2.435
2.423
2.156
2.138
2.126
2.117
2.115
2.102
2.094
2.074
2.066
2.053
2.045
2.030
2.022
1.644
1.631
1.618
1.607
1.594
1.272
1.268
1.255

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



1.00
1.02
1.02
2.15
1.04
1.06
1.00
2.99
3.00
2.09
2.10
4.11
1.14
3.96
2.14
9.09

— 208.542

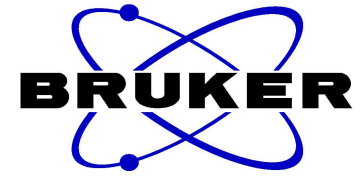
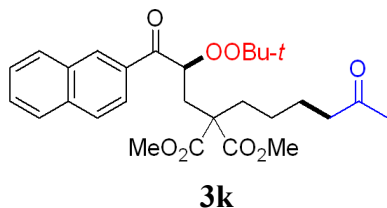
— 197.764

171.345
171.302

135.689
132.486
132.424
131.009
129.783
128.624
128.317
127.771
126.713
124.641

81.748
80.874

55.956
52.697
52.592
43.224
33.361
32.842
29.936
26.418
23.790

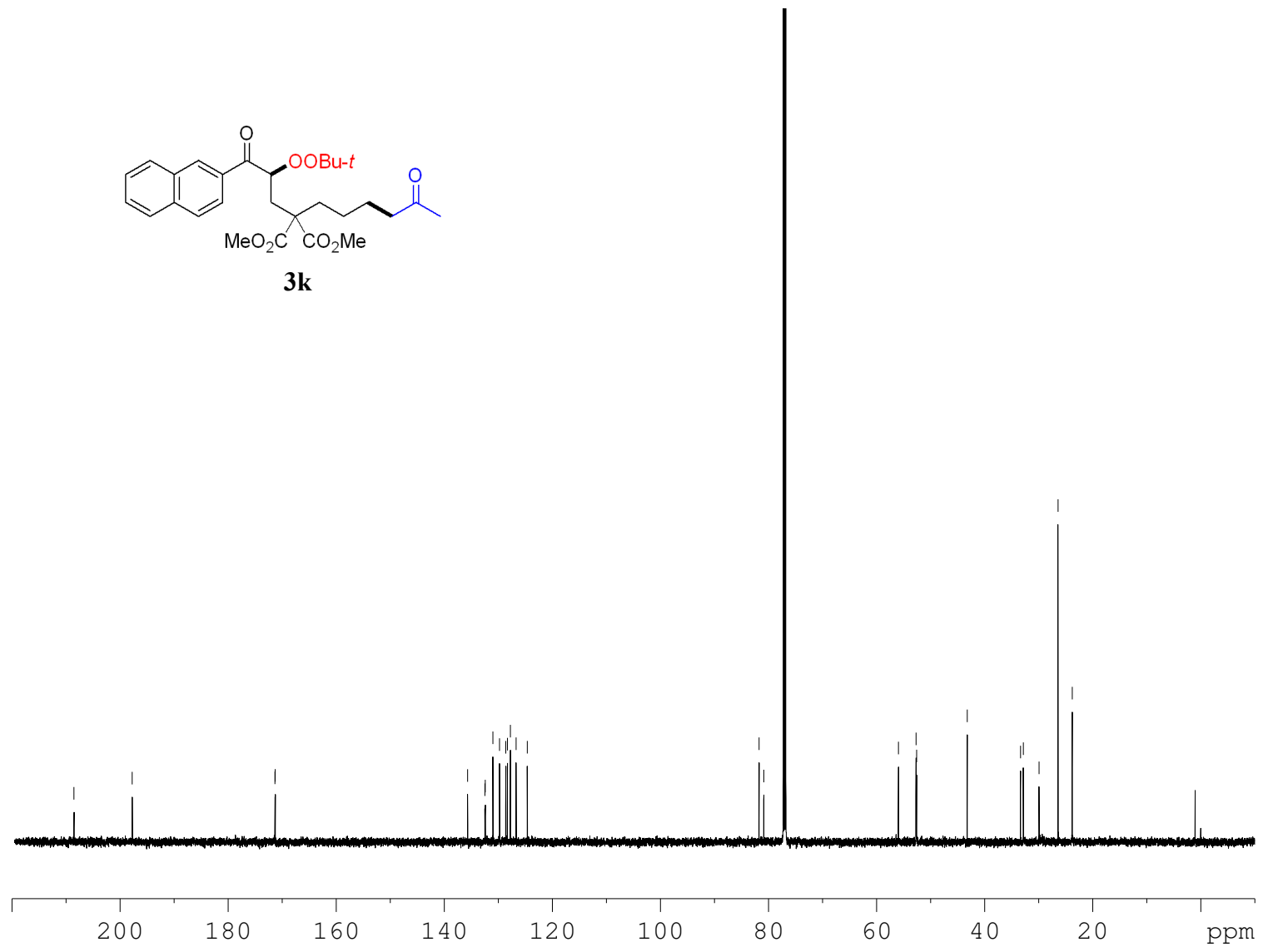


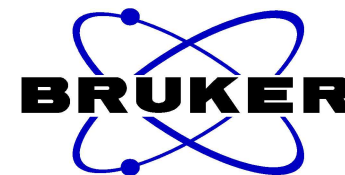
```

NAME      w11-551p-20210610
EXPNO     2
PROCNO    1
Date_     20210611
Time      4.56
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDC13
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         2.0000000 sec
D11        0.0300000 sec
TD0        1
  
```

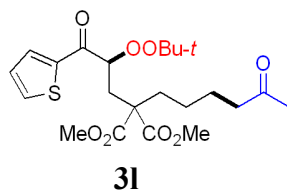
```

===== 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
  
```



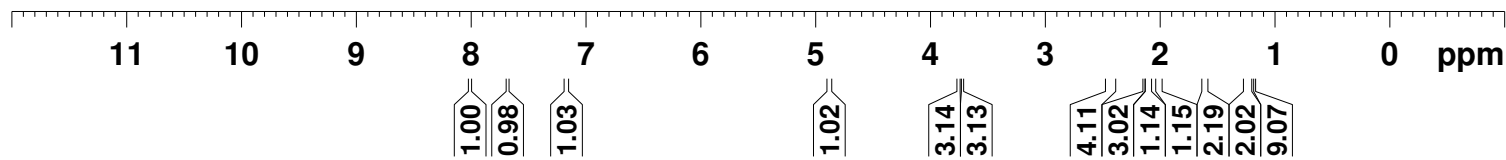
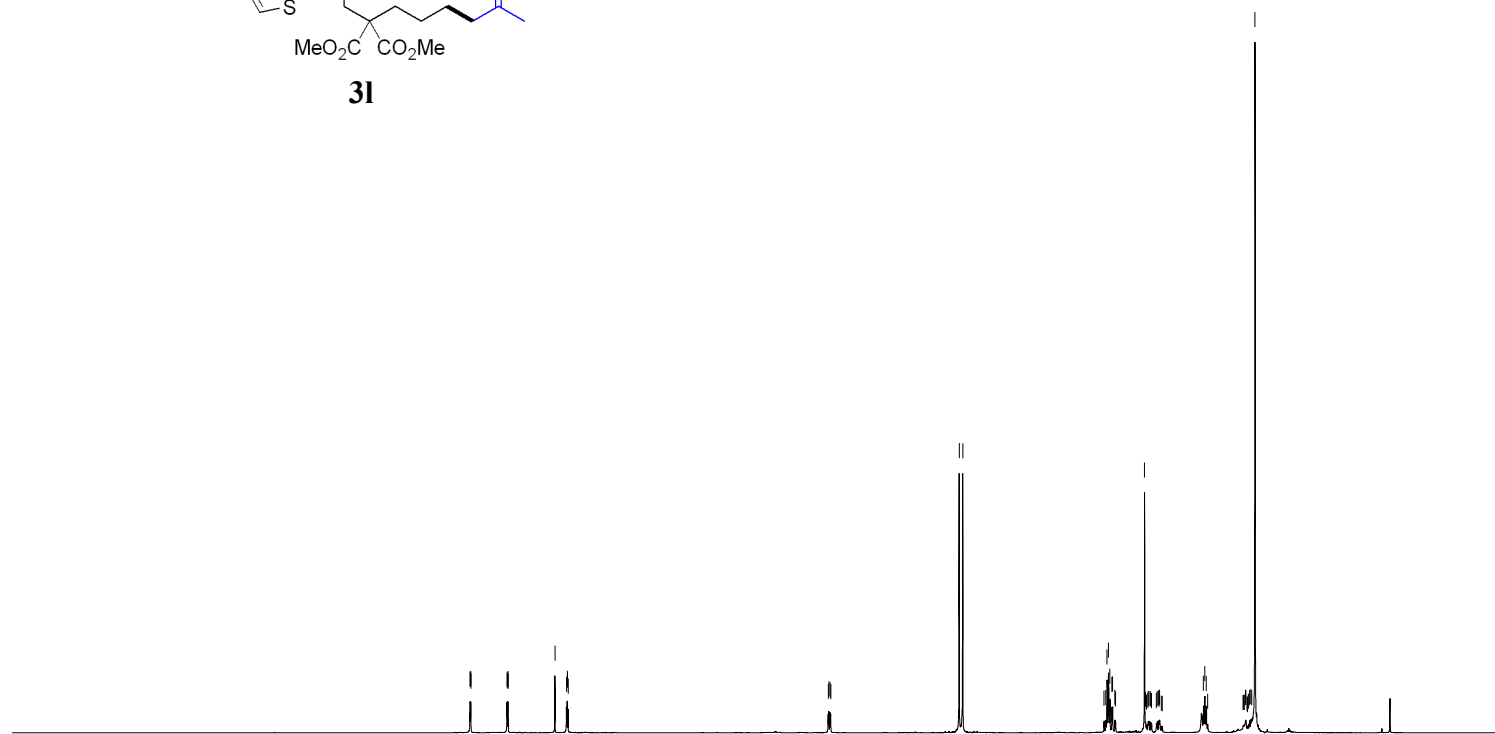


8.009
8.008
8.003
8.002
7.687
7.686
7.679
7.678
7.270
7.169
7.163
7.162
7.155
4.890
4.884
4.874
4.868
3.749
3.718
2.490
2.474
2.464
2.451
2.439
2.421
2.415
2.395
2.389
2.137
2.128
2.119
2.107
2.104
2.097
2.084
2.076
2.034
2.026
2.013
2.011
2.005
1.990
1.982
1.624
1.611
1.599
1.587
1.279
1.273
1.268
1.258



NAME w11-558p-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

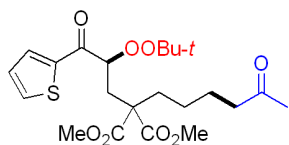
— 191.445

< 171.187
< 171.173

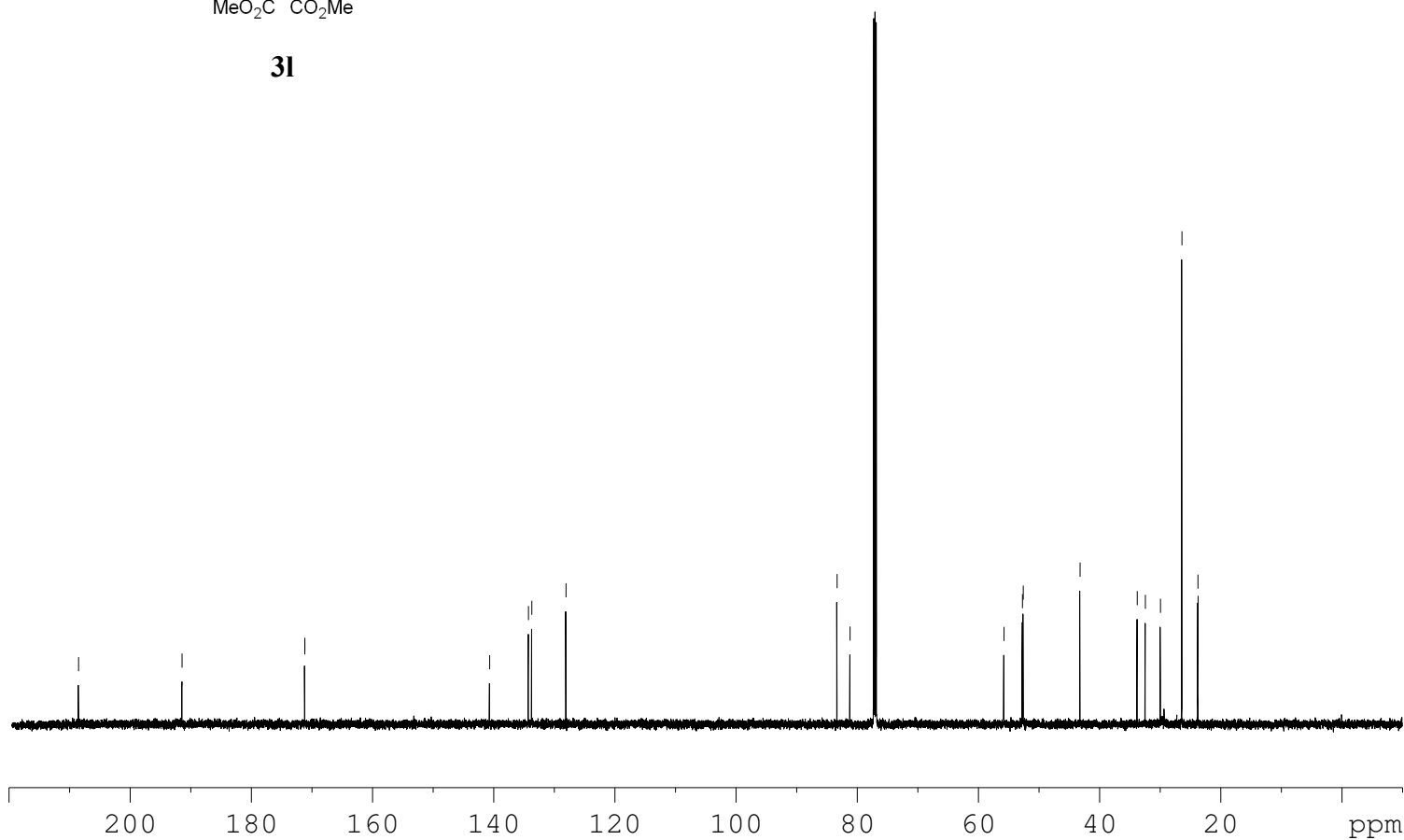
< 140.672
< 134.267
< 133.714
< 128.072

< 83.330
< 81.188

< 55.778
< 52.736
< 52.603
< 43.226
< 33.790
< 32.450
< 29.956
< 26.412
< 23.771
< 23.748



31



```

NAME      w11-558p-20210618
EXPNO     2
PROCNO    1
Date_     20210618
Time      13.05
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDC13
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        2.0000000 sec
D11       0.03000000 sec
TD0       1

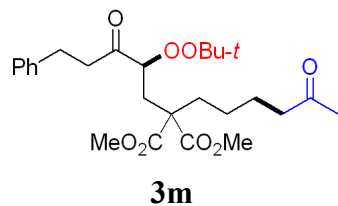
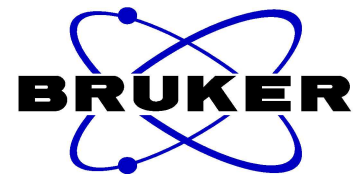
```

```

===== 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

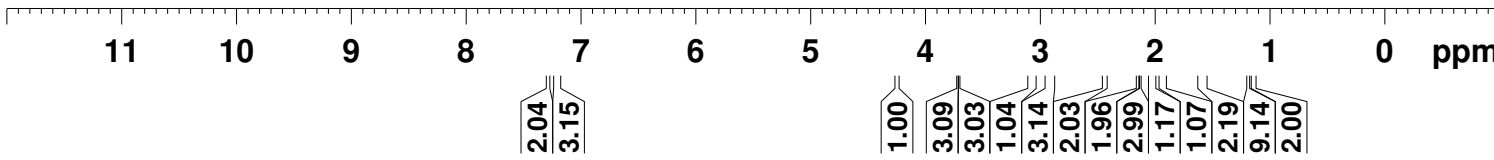
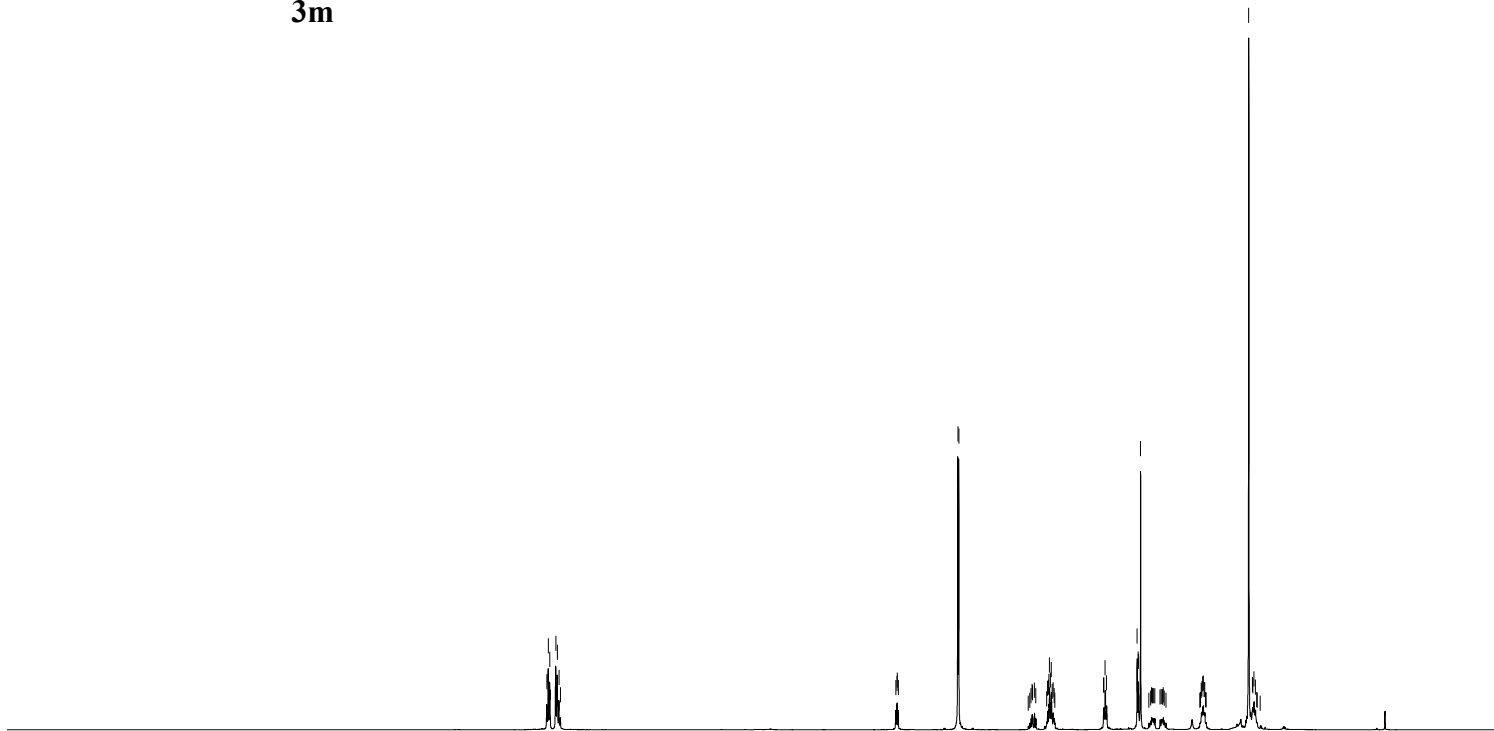
```

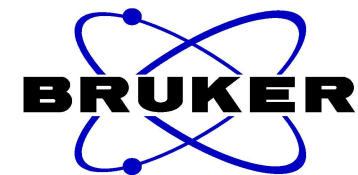
7.297
7.285
7.272
7.219
7.206
7.191
7.179
4.258
4.248
4.246
4.237
3.717
3.709
3.082
3.072
3.068
3.052
3.040
2.945
2.937
2.930
2.926
2.921
2.913
2.905
2.897
2.889
2.880
2.449
2.447
2.437
2.425
2.158
2.148
2.146
2.128
2.040
2.032
2.027
2.022
2.013
2.003
1.958
1.948
1.939
1.935
1.930
1.921
1.906
1.611
1.606
1.599
1.593
1.587
1.581
1.574
1.569
1.562
1.556
1.186
1.153
1.150
1.140
1.127
1.115
1.111



NAME w11-559p-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





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 CDC13
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 2.0000000 sec
D11 0.0300000 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

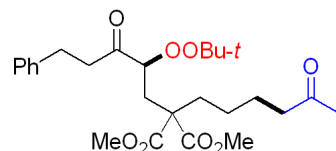
210.201
208.484

171.149
171.100

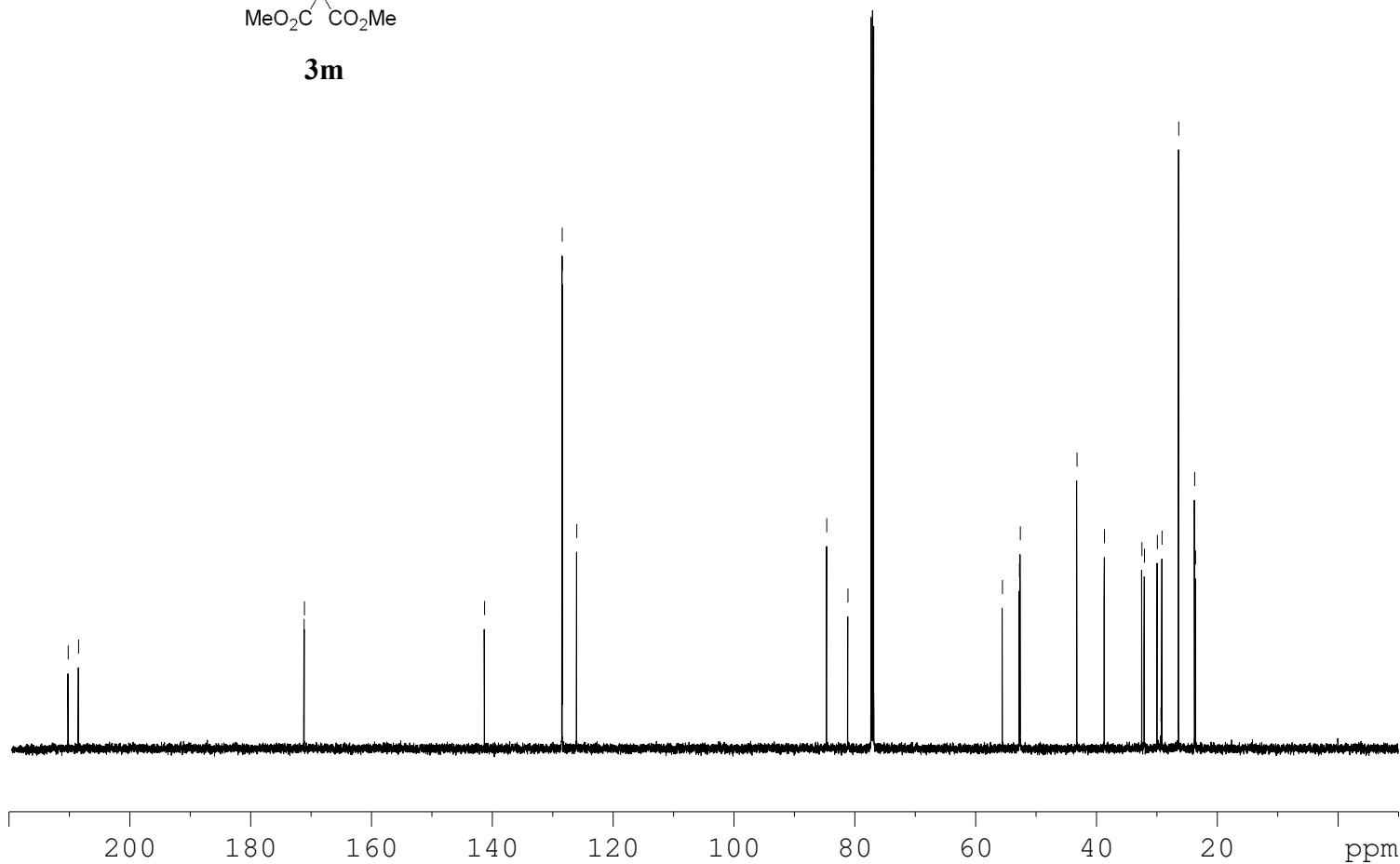
141.284
128.436
128.392
126.021

84.649
81.138

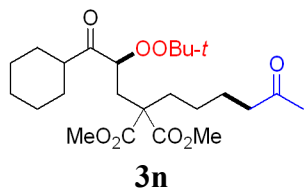
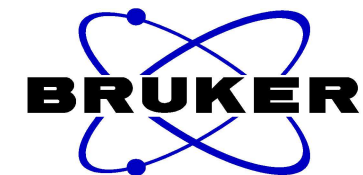
55.570
52.753
52.605
43.219
38.679
32.477
32.063
29.927
29.328
29.151
26.388
23.751
23.602



3m



4.349
4.342
4.335
4.328
3.735
3.724
2.878
2.873
2.865
2.859
2.854
2.846
2.841
2.455
2.443
2.431
2.238
2.219
2.215
2.212
2.201
2.176
2.131
2.059
2.051
2.036
2.028
2.015
2.007
1.972
1.964
1.951
1.943
1.928
1.920
1.853
1.836
1.818
1.799
1.784
1.779
1.619
1.617
1.605
1.593
1.581
1.569
1.403
1.383
1.364
1.341
1.324
1.310
1.307
1.291
1.272
1.255
1.201
1.181
1.172
1.166
1.160
1.147
1.139
1.126
1.094

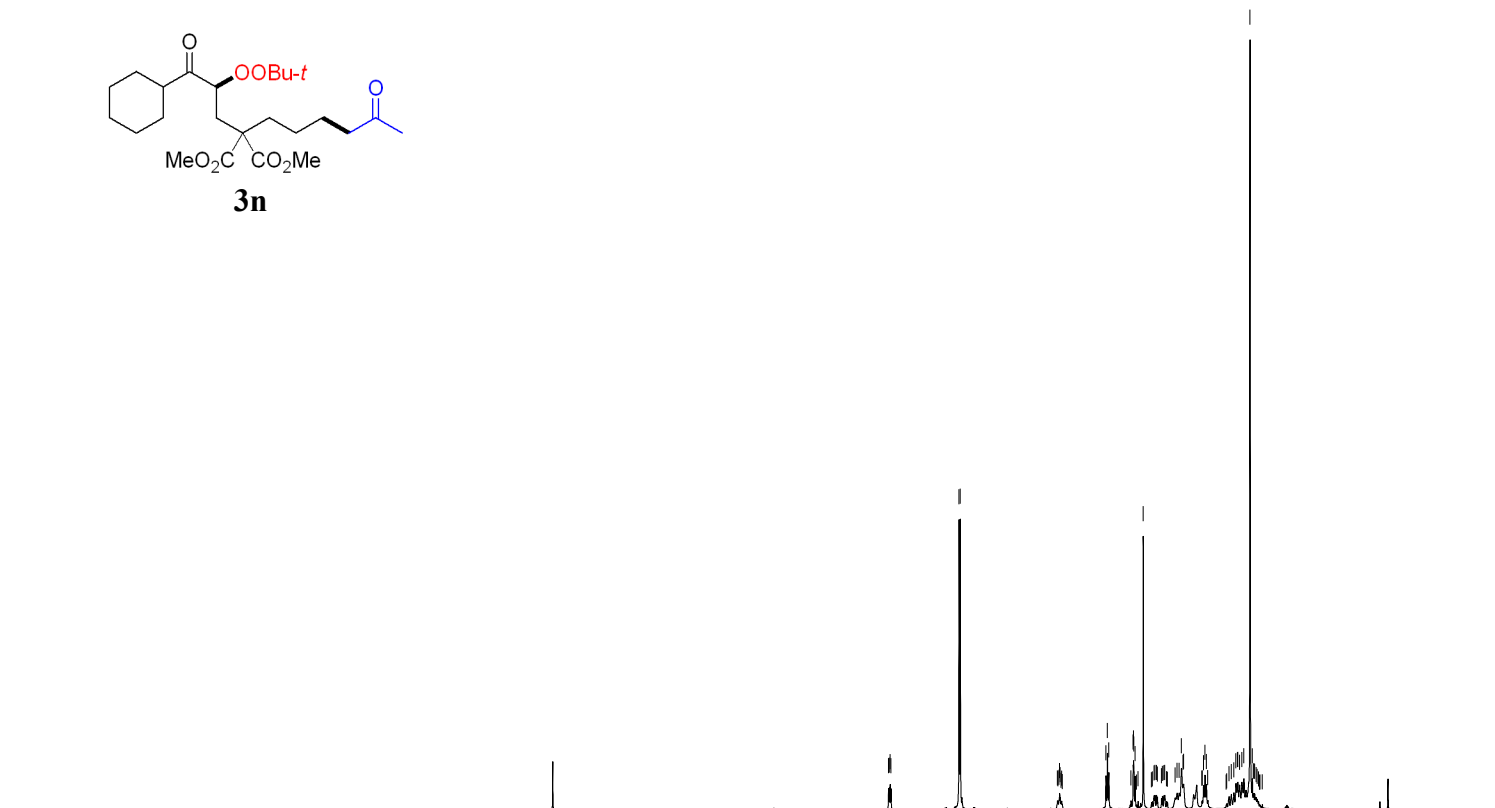


```

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   CDC13
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
  
```



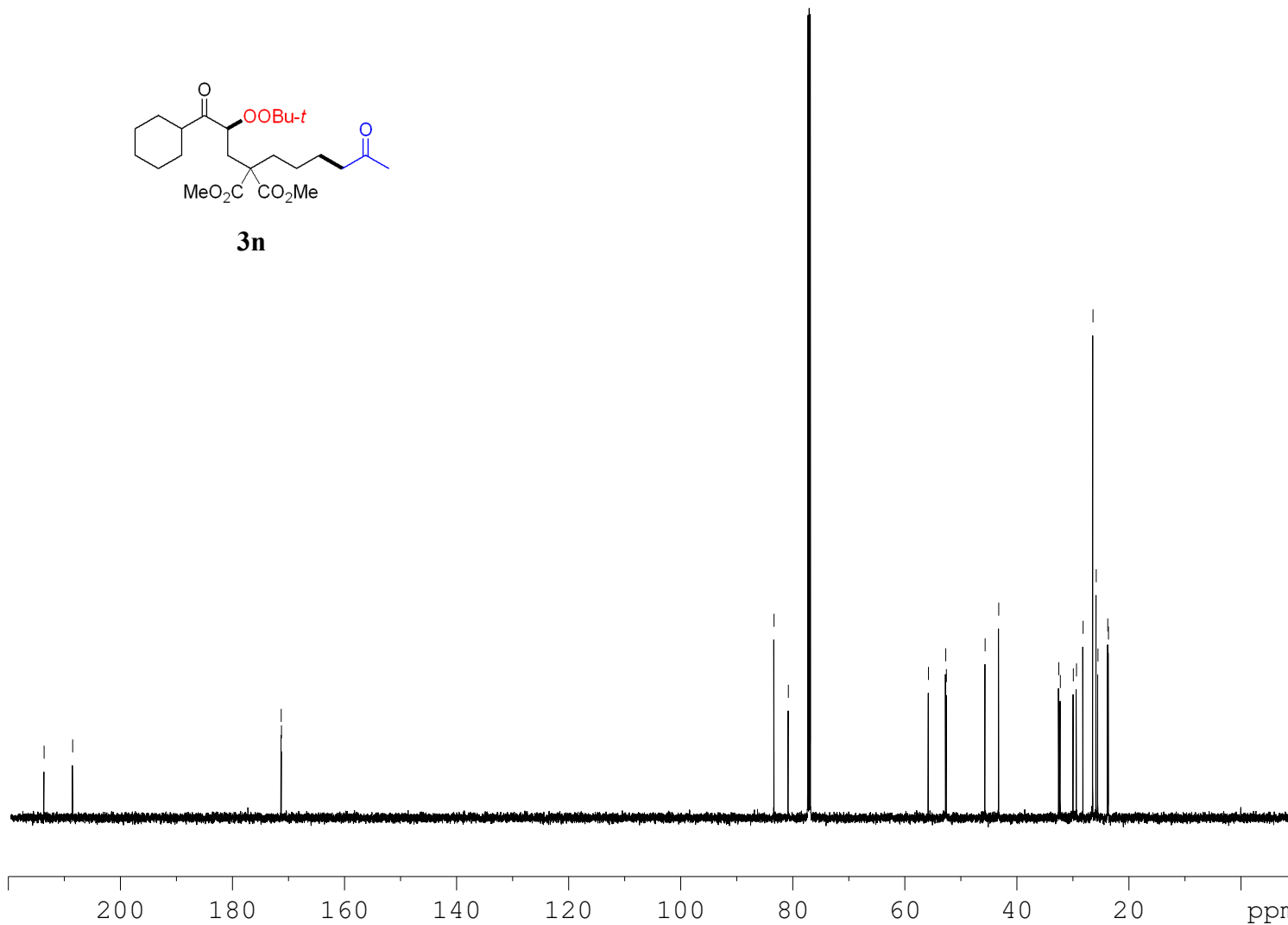
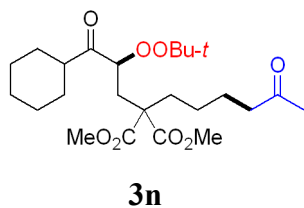
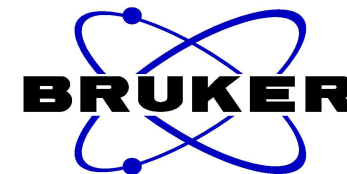
1.00
3.03
2.96
1.02
2.05
2.09
3.02
1.03
1.03
4.04
2.00
6.07
9.13
1.97

— 213.619
— 208.520

< 171.304
< 171.254

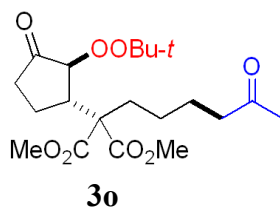
83.345
80.787

55.783
52.698
52.580
45.669
43.242
32.517
29.928
29.362
28.184
26.418
25.839
25.532
23.781
23.656

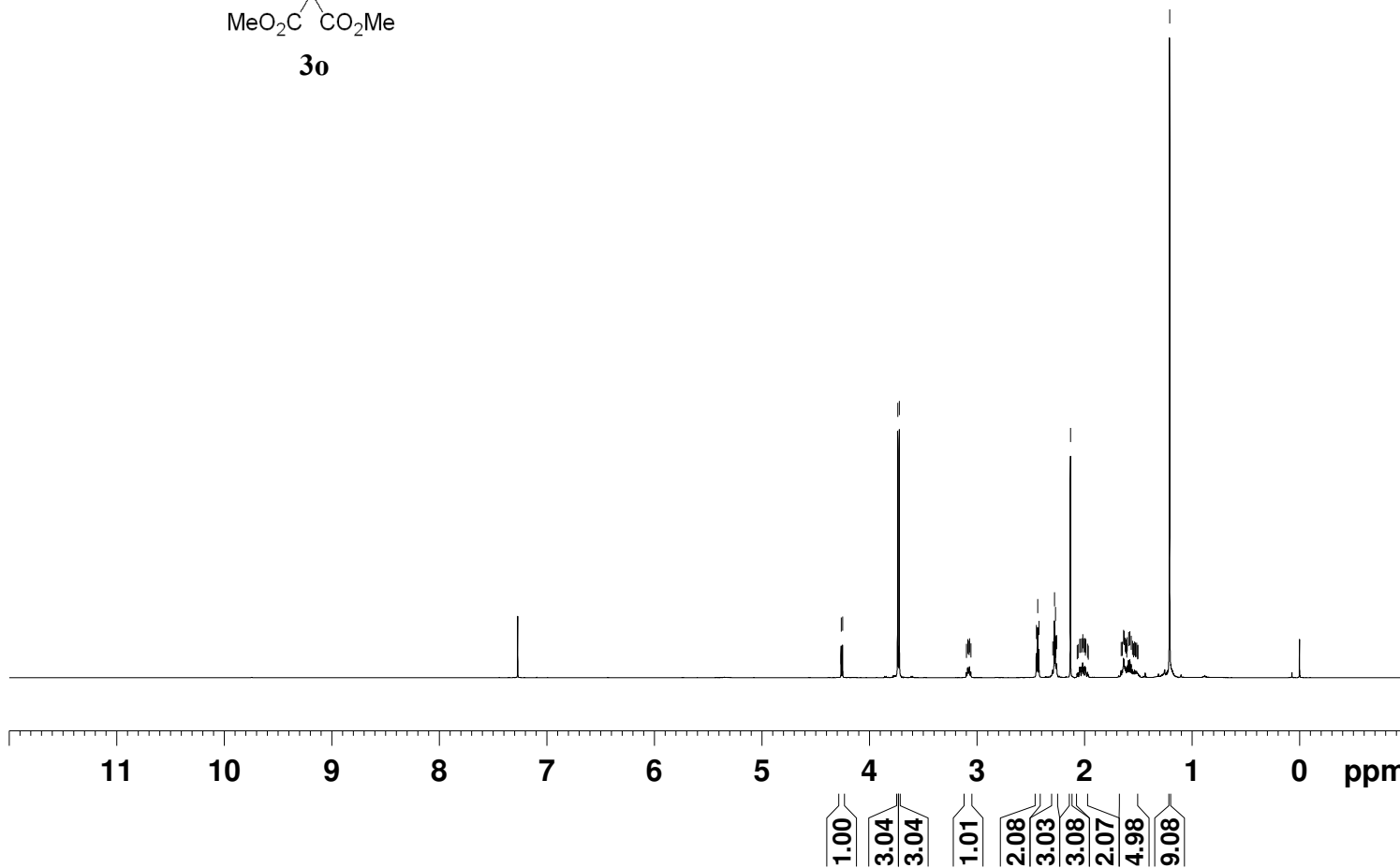


NAME w11-556p-20210618
EXPNO 2
PROCNO 1
Date_ 20210618
Time 12.33
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 200
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 190.02
DW 13.867 usec
DE 6.50 usec
TE 296.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== 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



4.263
4.250
3.737
3.723
3.099
3.086
3.082
3.073
3.070
3.057
2.448
2.436
2.424
2.296
2.285
2.280
2.270
2.260
2.130
2.068
2.060
2.045
2.037
2.025
2.016
2.007
1.996
1.992
1.988
1.973
1.965
1.660
1.649
1.637
1.632
1.621
1.617
1.615
1.608
1.603



NAME w11-554p-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 w11-554p-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 2.0000000 sec
D11 0.03000000 sec
TD0 1

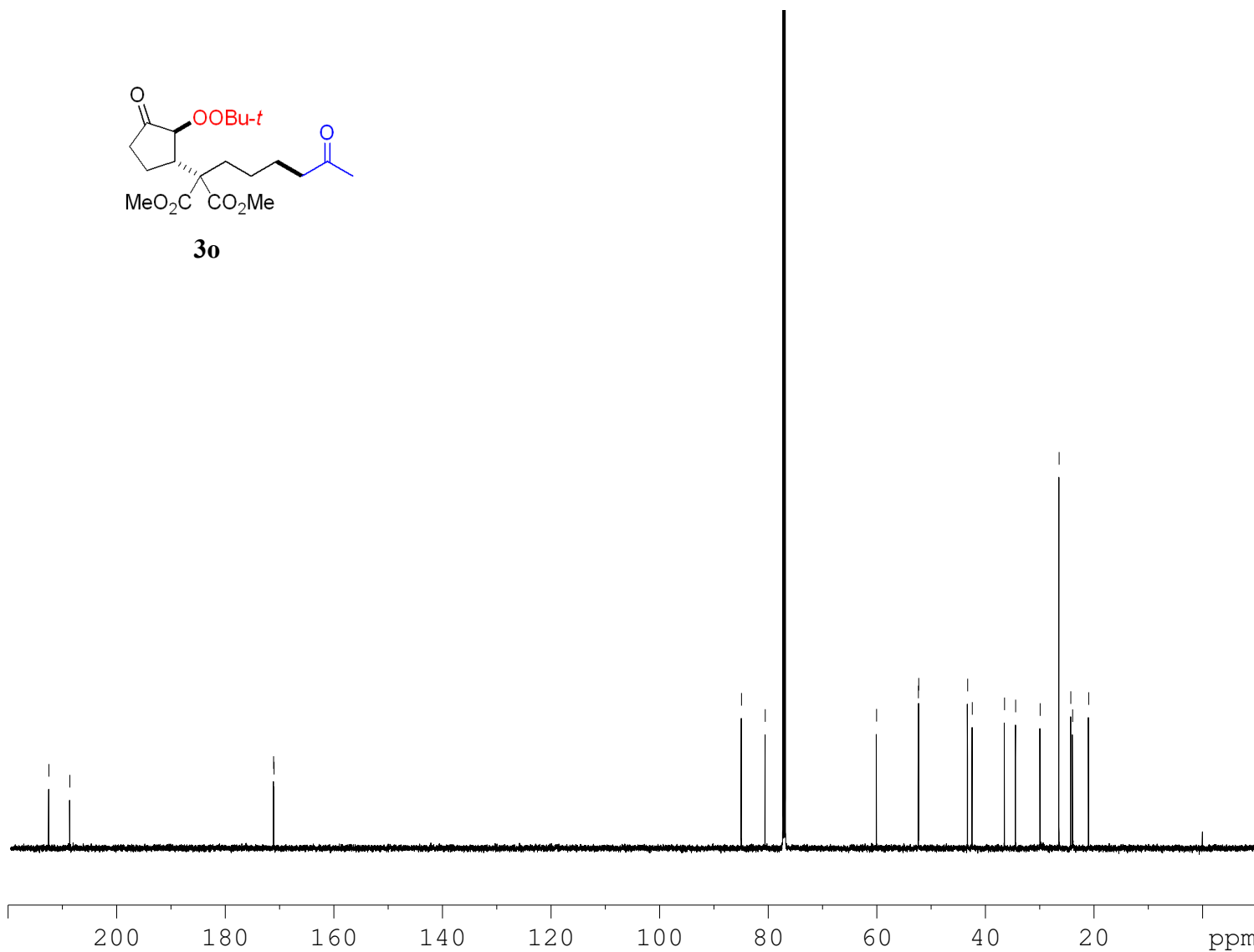
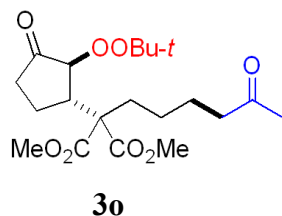
===== CHANNEL f1 =====
SFO1 150.9279571 MHz
NUC1 13C
P1 14.00 usec
SI 32768
SF 150.9128665 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

212.486
208.623

171.100
171.011

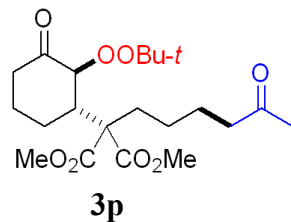
84.926
80.552

60.048
52.342
52.283
43.269
42.417
36.480
34.435
29.919
26.424
24.275
23.937
20.979





4.279
4.271
3.655
3.626
2.548
2.546
2.540
2.522
2.519
2.513
2.454
2.445
2.436
2.427
2.419
2.379
2.367
2.354
2.173
2.162
2.156
2.144
2.135
2.129
2.117
2.058
1.893
1.877
1.870
1.857
1.849
1.840
1.831
1.822
1.804
1.797
1.792
1.784
1.776
1.523
1.511
1.498
1.486

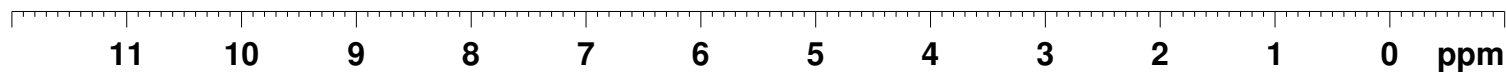


```

NAME      w11-592p-20210719
EXPNO     1
PROCNO    1
Date_     20210719
Time      21.53
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        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
  
```



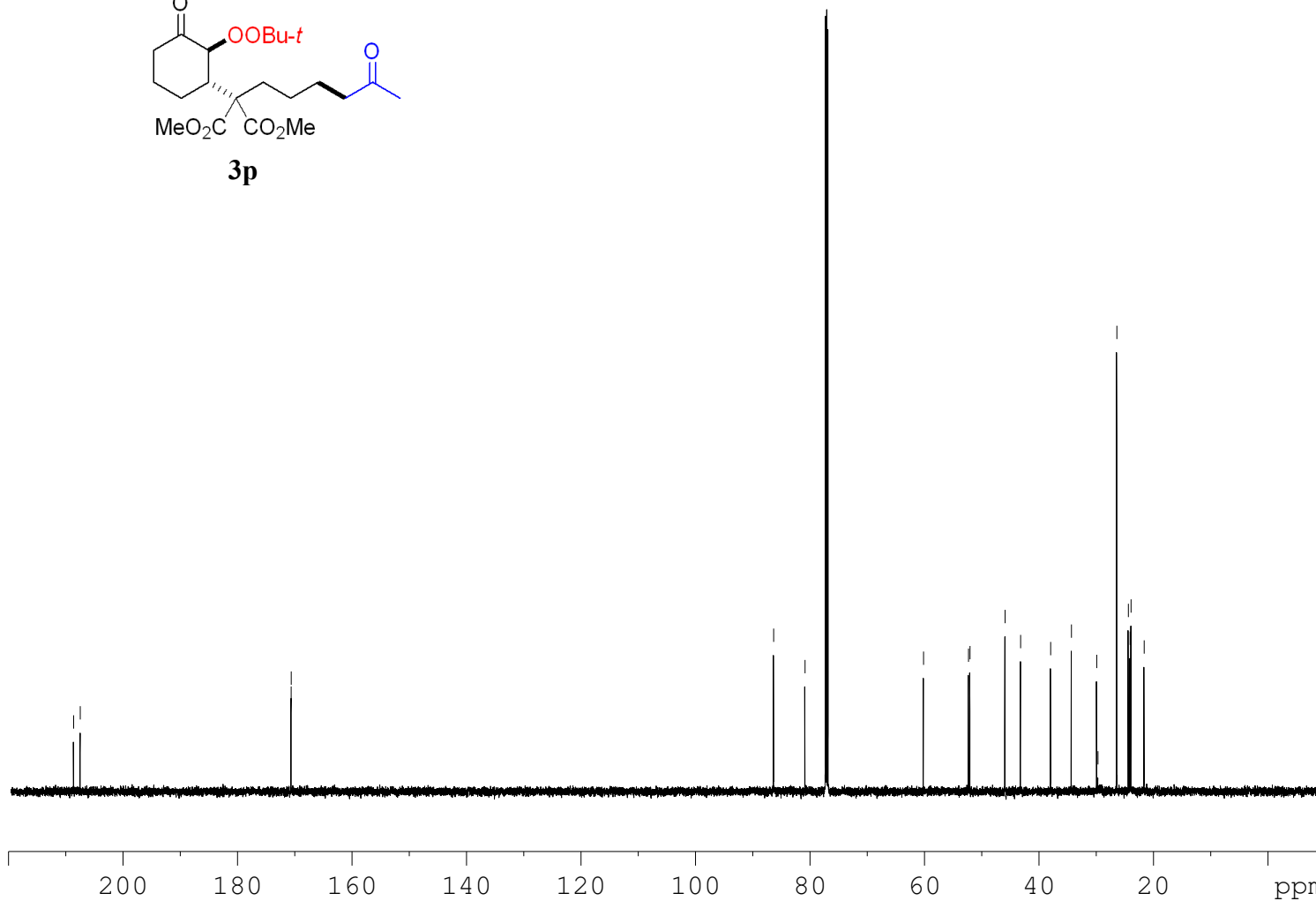
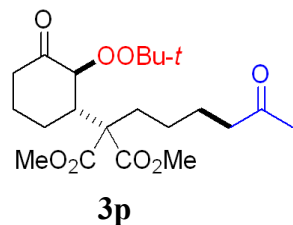
1.00
3.05
2.97
1.01
1.00
2.11
1.06
3.14
5.14
2.07
0.98
1.92
9.10

208.629
207.471

170.631
170.619

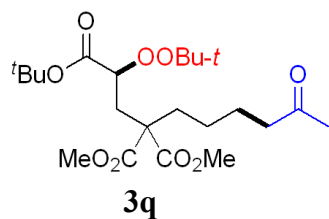
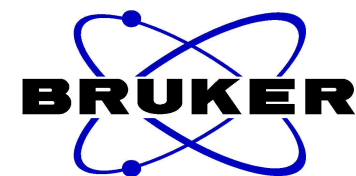
86.348
80.866

60.166
52.311
52.048
45.924
43.209
37.961
34.331
29.912
29.694
26.395
24.386
24.086
23.919
21.623

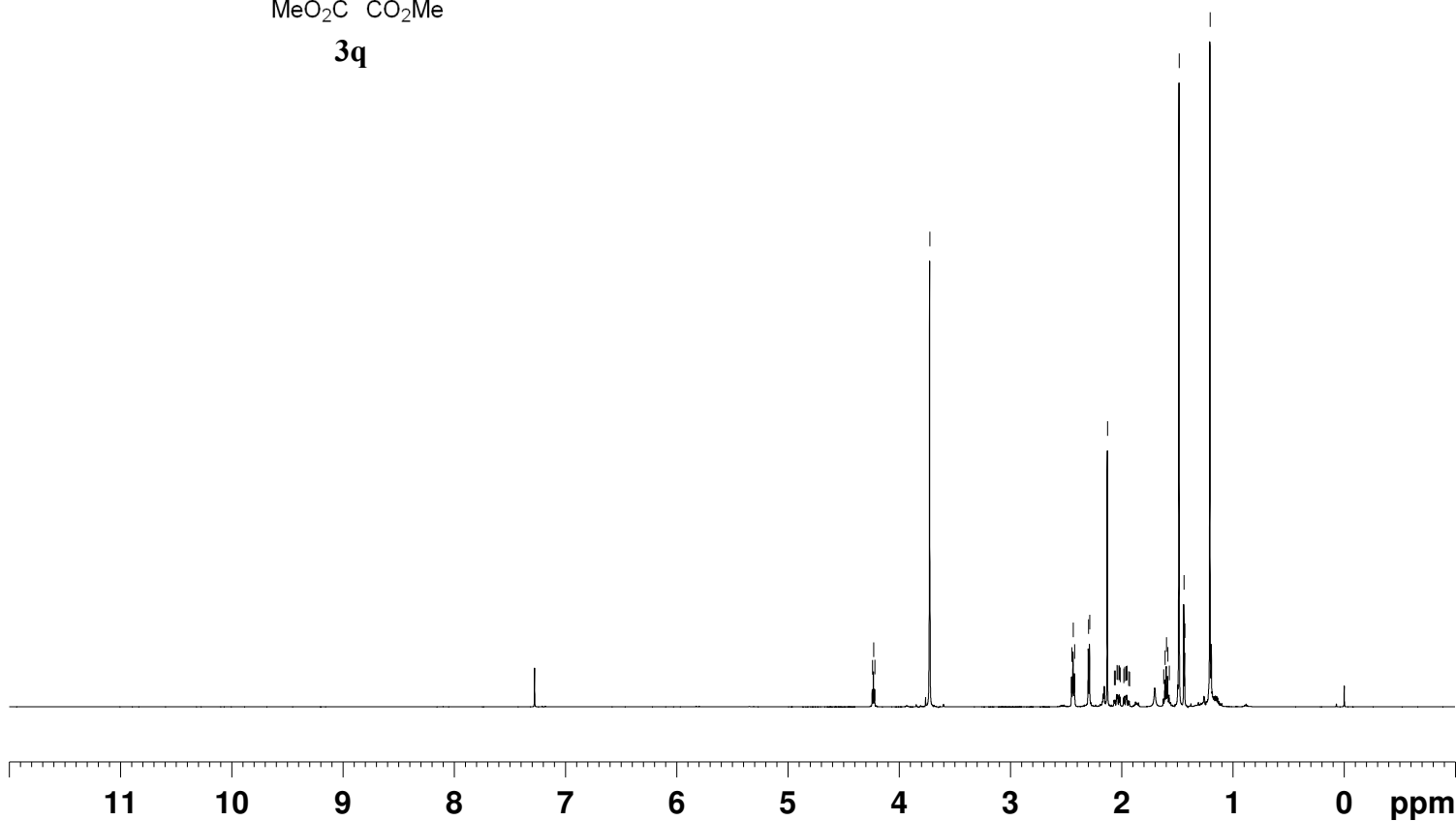


NAME w11-592p-20210719
EXPNO 2
PROCNO 1
Date_ 20210719
Time 22.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 190.02
DW 13.867 usec
DE 6.50 usec
TE 298.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SF01 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



4.240
4.230
4.219
3.725
2.448
2.436
2.424
2.299
2.288
2.128
2.066
2.058
2.042
2.035
2.022
2.014
1.981
1.973
1.961
1.953
1.938
1.929
1.623
1.610
1.598
1.585
1.572
1.483
1.440
1.432
1.205



1.00
5.97
2.09
2.07
3.01
1.12
0.98
2.10
9.00
2.13
9.00

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

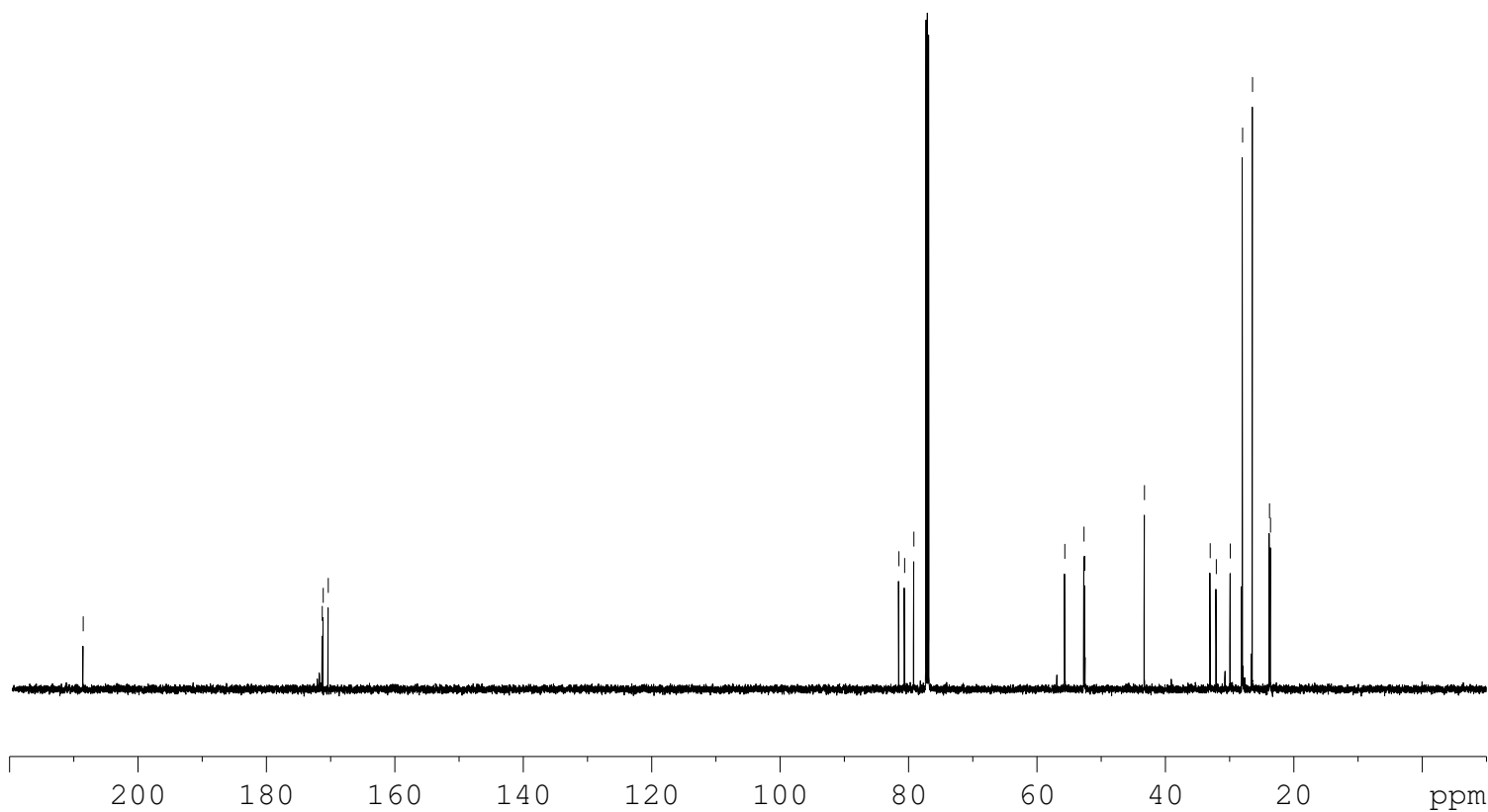
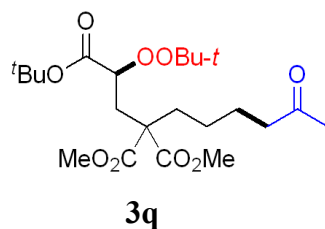
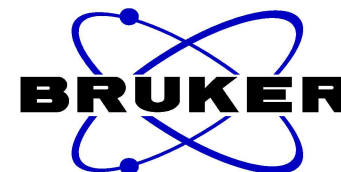
===== CHANNEL f1 =====
SFO1 600.1739011 MHz
NUC1 1H
P1 9.96 usec
SI 65536
SF 600.1700060 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

— 208.531

171.306
171.157
170.370

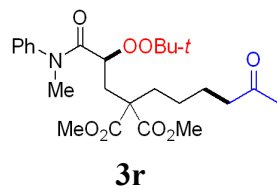
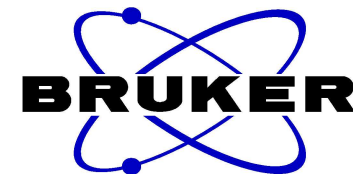
81.501
80.611
79.172

55.666
52.677
52.543
43.264
33.024
32.074
29.898
27.980
26.434
23.789
23.634



```
NAME      w11-564p-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         2.0000000 sec
D11        0.03000000 sec
TD0        1
```

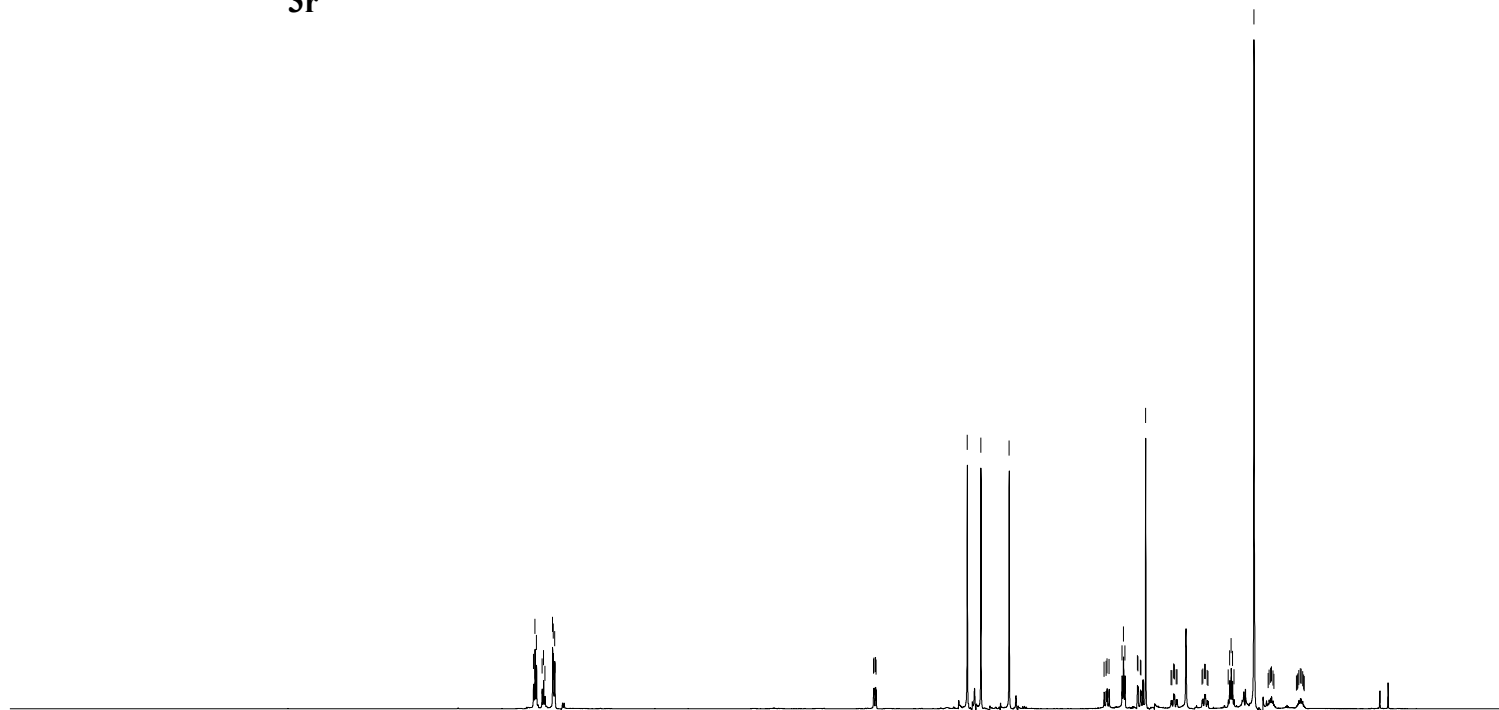
```
===== CHANNEL f1 =====
SFO1      150.9279571 MHz
NUC1       13C
P1         14.00 usec
SI         32768
SF         150.9128665 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
```

7.440
7.428
7.415
7.365
7.353
7.340
7.275
7.268
7.256
4.479
4.474
4.462
4.457
3.663
3.544
3.299
2.471
2.454
2.446
2.428
2.315
2.303
2.290
2.181
2.176
2.155
2.150
2.110
1.888
1.881
1.866
1.859
1.843
1.836
1.618
1.611
1.596
1.589
1.574
1.567
1.390
1.378
1.365
1.353
1.340
1.166
1.042

NAME w11-563p-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



2.07
1.00
2.05

1.01

3.05

2.98

2.99

1.05

2.04

1.13

3.03

1.14

1.09

1.99

9.11

0.94

1.05

— 208.530

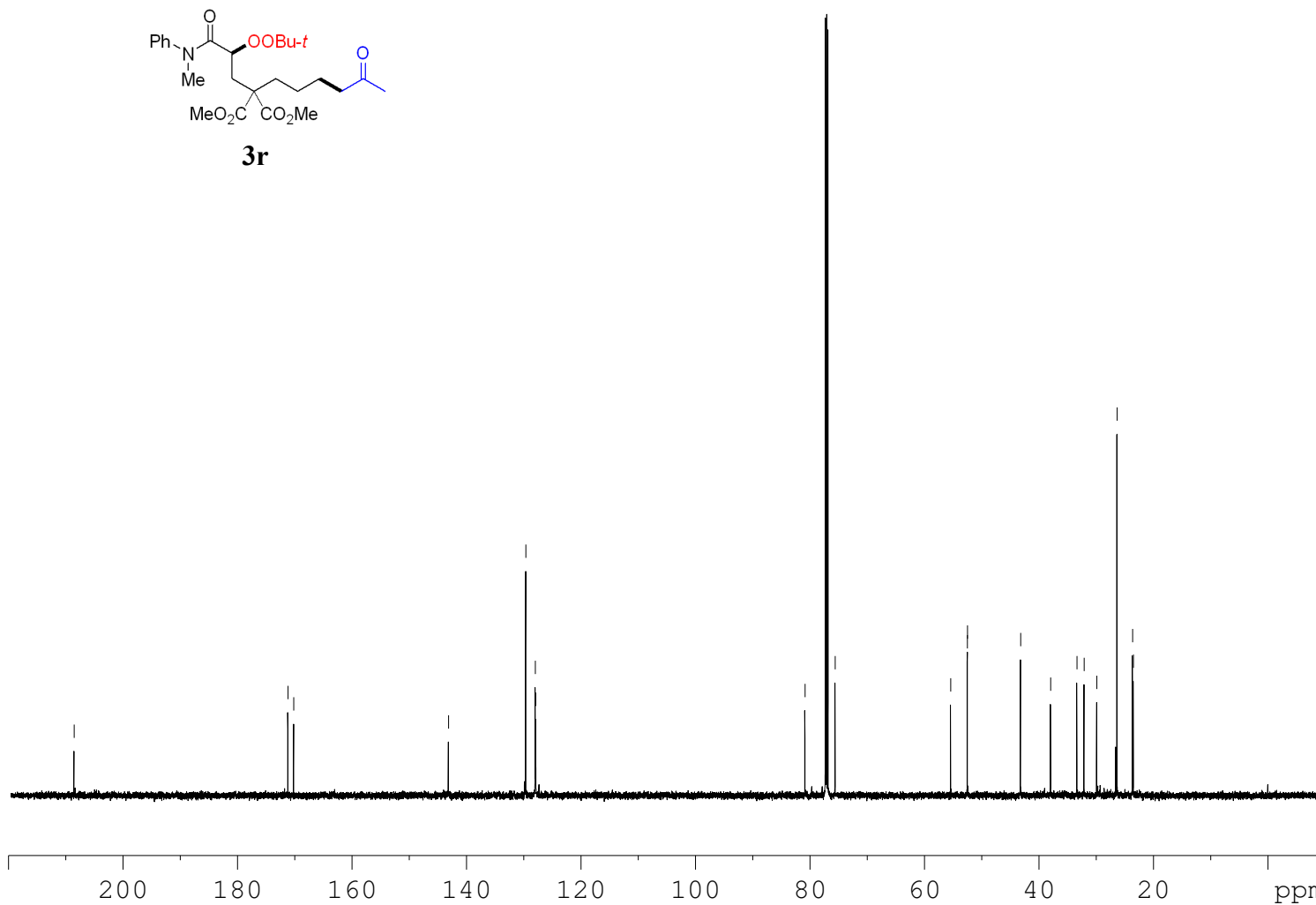
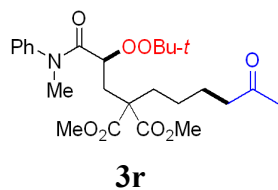
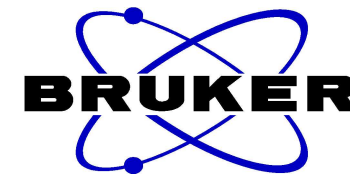
171.178
170.180

— 143.134

129.633
127.978
127.893

— 80.857
— 75.599

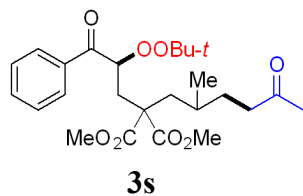
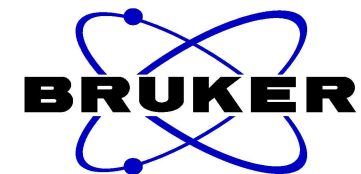
55.420
52.508
52.488
43.195
37.958
33.360
32.108
29.894
26.350
23.642
23.501



NAME w11-563p-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

8.081
8.069
7.588
7.576
7.564
7.482
7.470
7.457
5.205
5.199
5.190
5.184
5.134
5.128
5.119
5.113
3.713
3.702
2.529
2.522
2.516
2.504
2.497
2.490
2.484
2.470
2.470
2.463
2.456
2.443
2.437
2.428
2.418
2.409
2.402
2.381
2.143
2.132
1.991
1.978
1.967
1.953
1.919
1.907
1.894
1.882
1.597
1.595
1.588
1.581
1.579
1.572
1.564
1.556
1.547
1.439
1.435
1.429
1.423
1.419
1.413
1.403
1.121
1.117
0.836
0.826
0.825



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

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

1.97
1.00
2.04
0.50
0.51
2.97
2.99
4.07
3.98
1.00
2.06
1.10
8.93
3.01

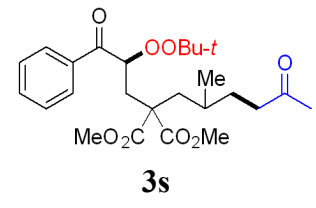
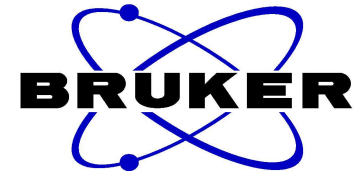
208.695
208.652
198.028
197.905

171.762
171.729
171.542
171.469

135.333
135.252
133.262
129.090
129.063
128.472

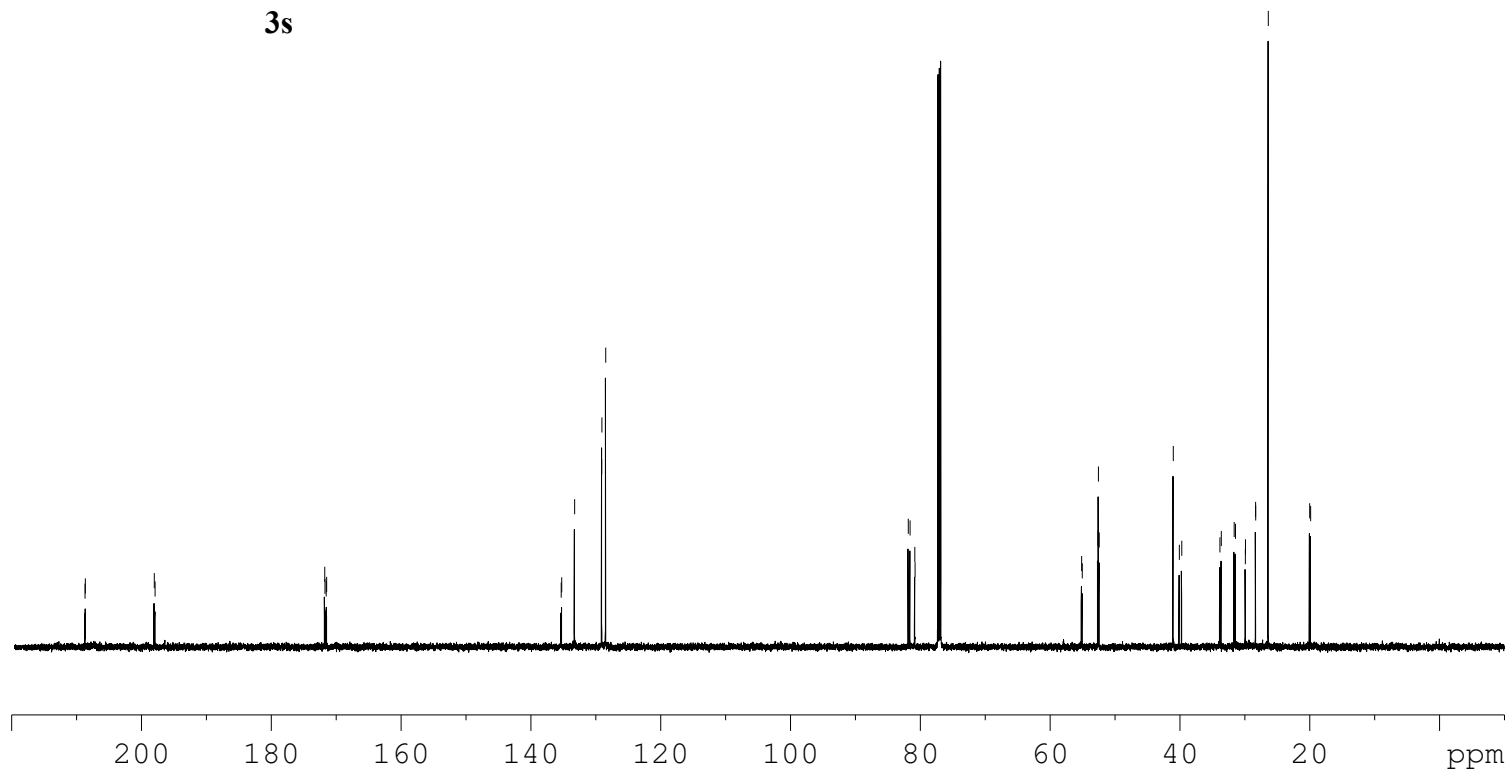
81.858
81.581
80.851
80.809

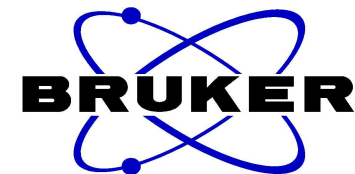
55.150
55.050
52.640
52.562
52.437
41.027
40.092
39.719
33.841
33.630
31.646
31.443
29.922
29.906
28.344
28.330
26.389



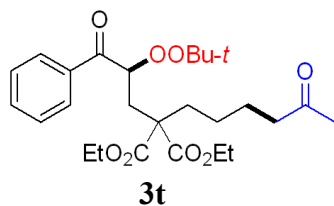
NAME w11-560p-20210623
EXPNO 2
PROCNO 1
Date_ 20210623
Time 20.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 240
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 190.02
DW 13.867 usec
DE 6.50 usec
TE 298.5 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== 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



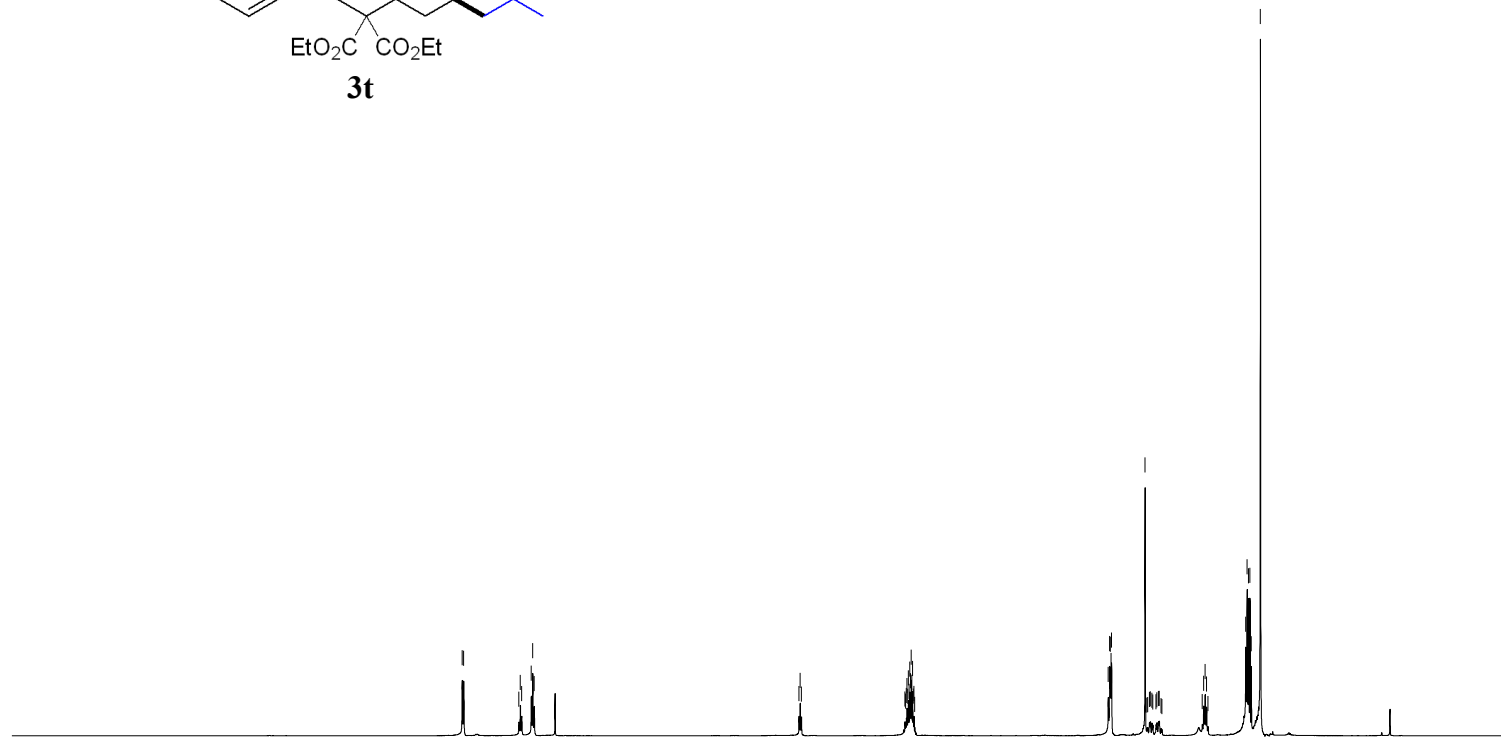


8.079
8.066
7.585
7.573
7.561
7.478
7.465
7.452
5.146
5.136
5.125
4.223
4.217
4.211
4.205
4.199
4.193
4.187
4.181
4.175
4.168
4.163
4.157
4.151
4.145
4.138
2.453
2.441
2.435
2.428
2.424
2.132
2.117
2.109
2.094
2.086
2.072
2.065
2.037
2.029
2.016
2.008
1.993
1.985
1.634
1.622
1.609
1.597
1.584



NAME w11-550p-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



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

1.97
1.05
2.06
1.00
4.08
4.07
2.96
1.03
1.07
2.15
8.01
8.94

— 208.557

— 198.050

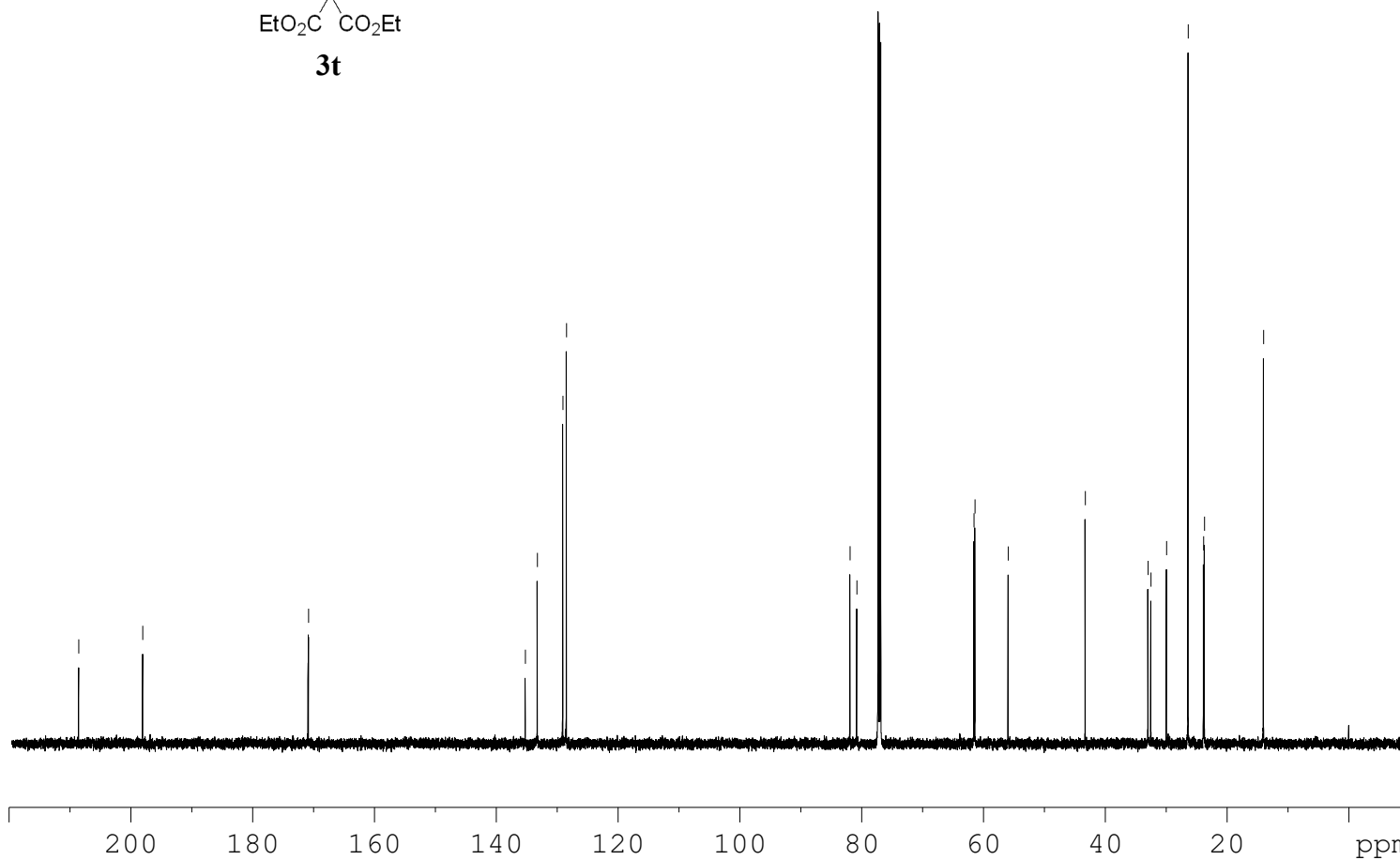
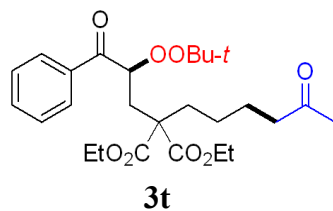
< 170.875
< 170.822

< 135.221
< 133.249
< 129.054
< 128.466

< 81.906
< 80.785

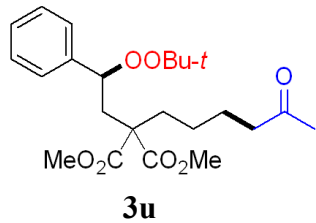
< 61.557
< 61.391
< 55.929

< 43.279
< 32.972
< 32.498
< 29.934
< 26.383
< 23.855
< 23.707
< 14.004



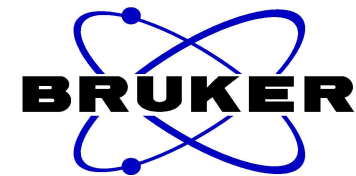
NAME w11-550p-20210610
EXPNO 2
PROCNO 1
Date_ 20210611
Time 0.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 300
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 190.02
DW 13.867 usec
DE 6.50 usec
TE 296.9 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

===== 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



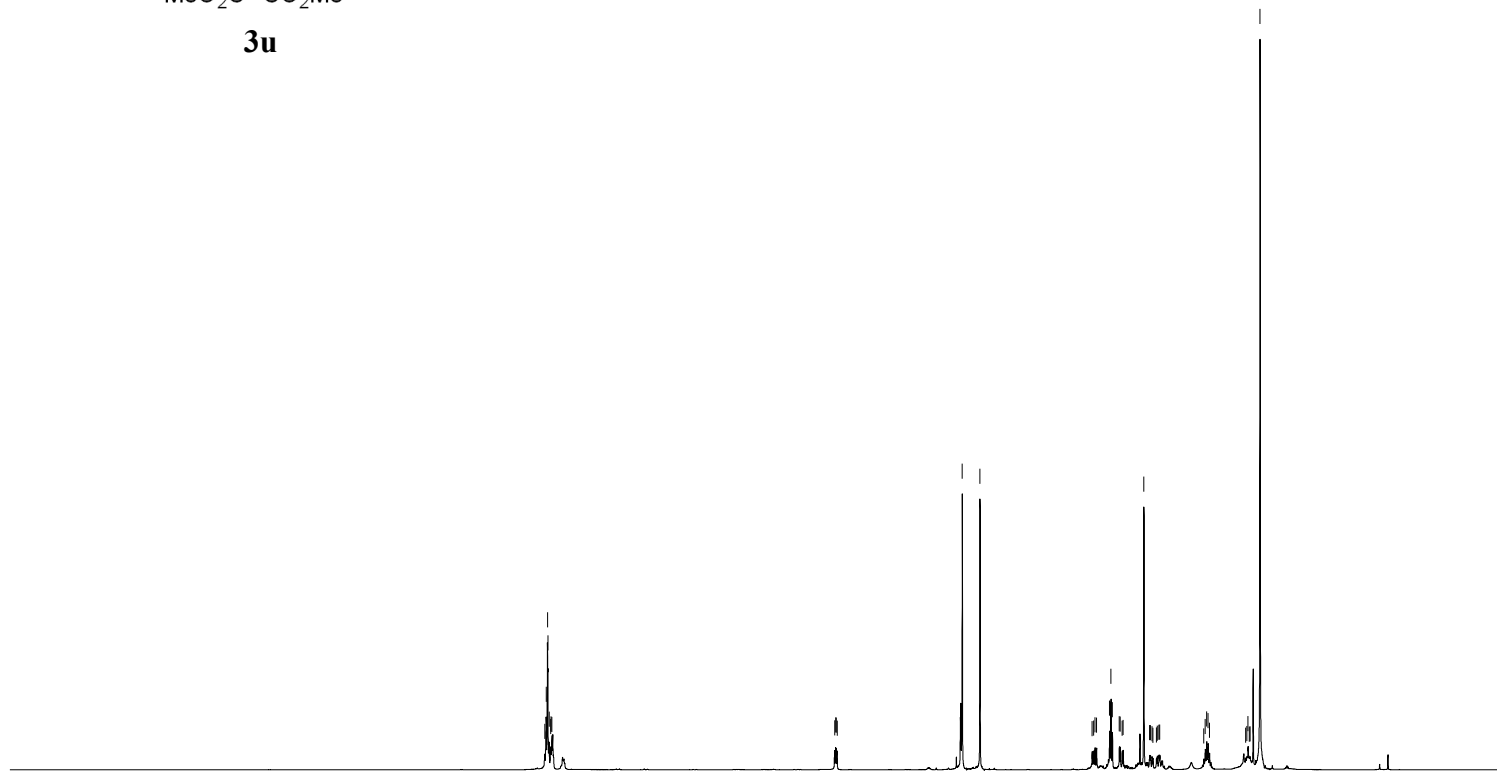
7.341
7.329
7.317
7.314
7.303
7.290
7.288
7.280

4.817
4.808
4.803
4.795
3.707
3.553
2.576
2.562
2.551
2.537
2.424
2.412
2.400
2.339
2.331
2.314
2.306
2.125
2.075
2.068
2.054
2.046
2.015
2.008
1.994
1.987
1.603
1.591
1.578
1.566
1.553
1.238
1.228
1.218
1.207



NAME w11-540-7p-20210608
 EXPNO 1
 PROCNO 1
 Date_ 20210608
 Time_ 15.05
 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 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



5.19
1.00
2.97
3.00
1.07
2.07
1.09
3.08
0.93
0.94
2.00
2.06
8.98

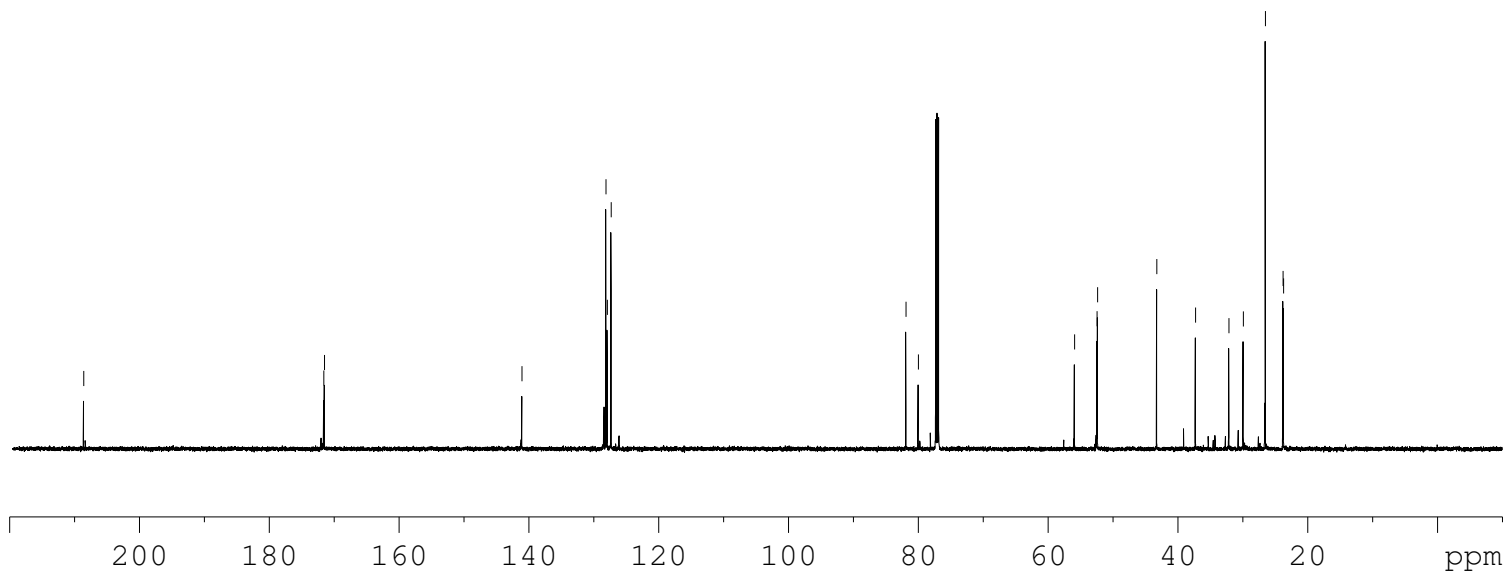
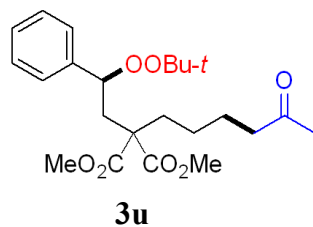
— 208.586

< 171.599
< 171.493

— 141.067
< 128.124
< 127.911
< 127.319

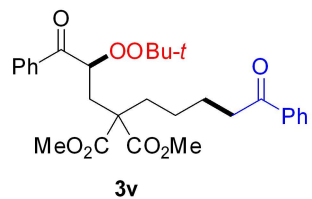
< 81.896
< 79.987

< 55.923
< 52.461
< 52.378
< 43.256
< 37.284
< 32.142
< 29.920
< 26.520
< 23.802
< 23.722

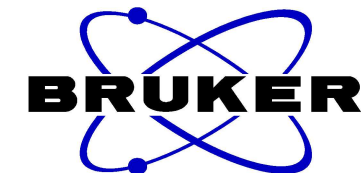


NAME w11-540-7p-20210608
EXPNO 2
PROCNO 1
Date_ 20210608
Time 15.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 300
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 190.02
DW 13.867 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== 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

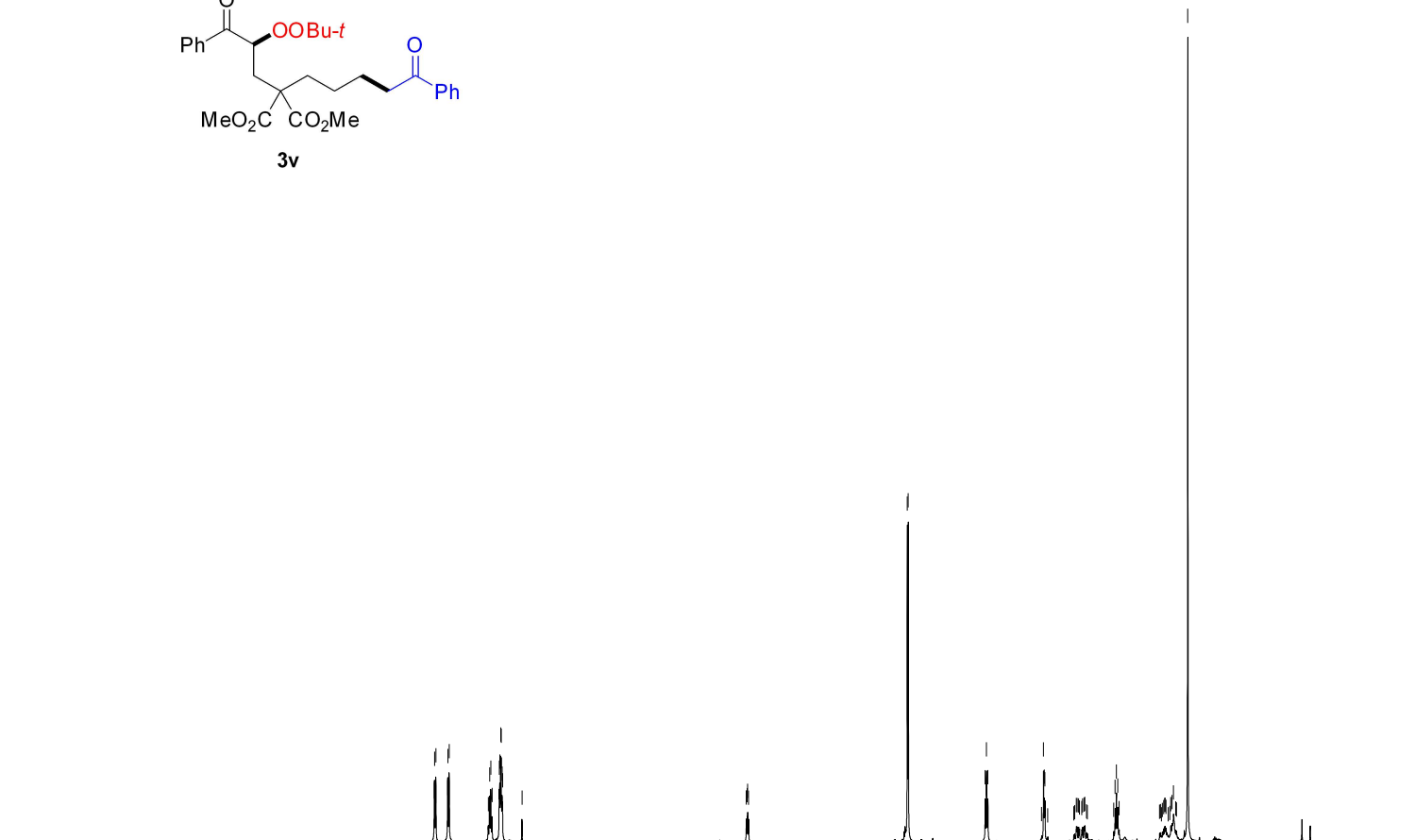


8.076
8.064
7.954
7.942
7.581
7.568
7.558
7.546
7.479
7.474
7.466
7.453
7.449
7.269
5.202
5.193
5.189
5.181
3.716
3.708
2.998
2.986
2.973
2.478
2.460
2.452
2.446
2.421
2.180
2.172
2.157
2.149
2.136
2.128
2.106
2.099
2.086
2.063
2.055
1.812
1.799
1.787
1.774
1.762
1.389
1.380
1.371
1.363
1.351
1.342
1.337



NAME w11-695p-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



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

2.01
2.07
2.06
4.08
1.00
3.06
2.99
2.09
2.03
1.01
1.09
2.02
1.06
1.09
9.03

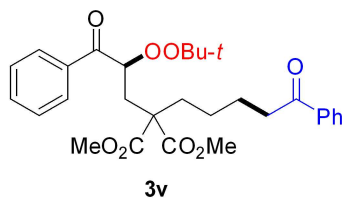
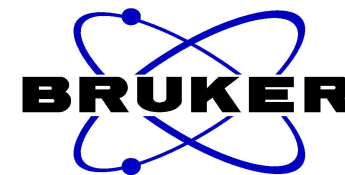
199.850
197.921

171.380
171.345

137.013
135.299
133.323
133.029
129.074
128.643
128.553
128.053

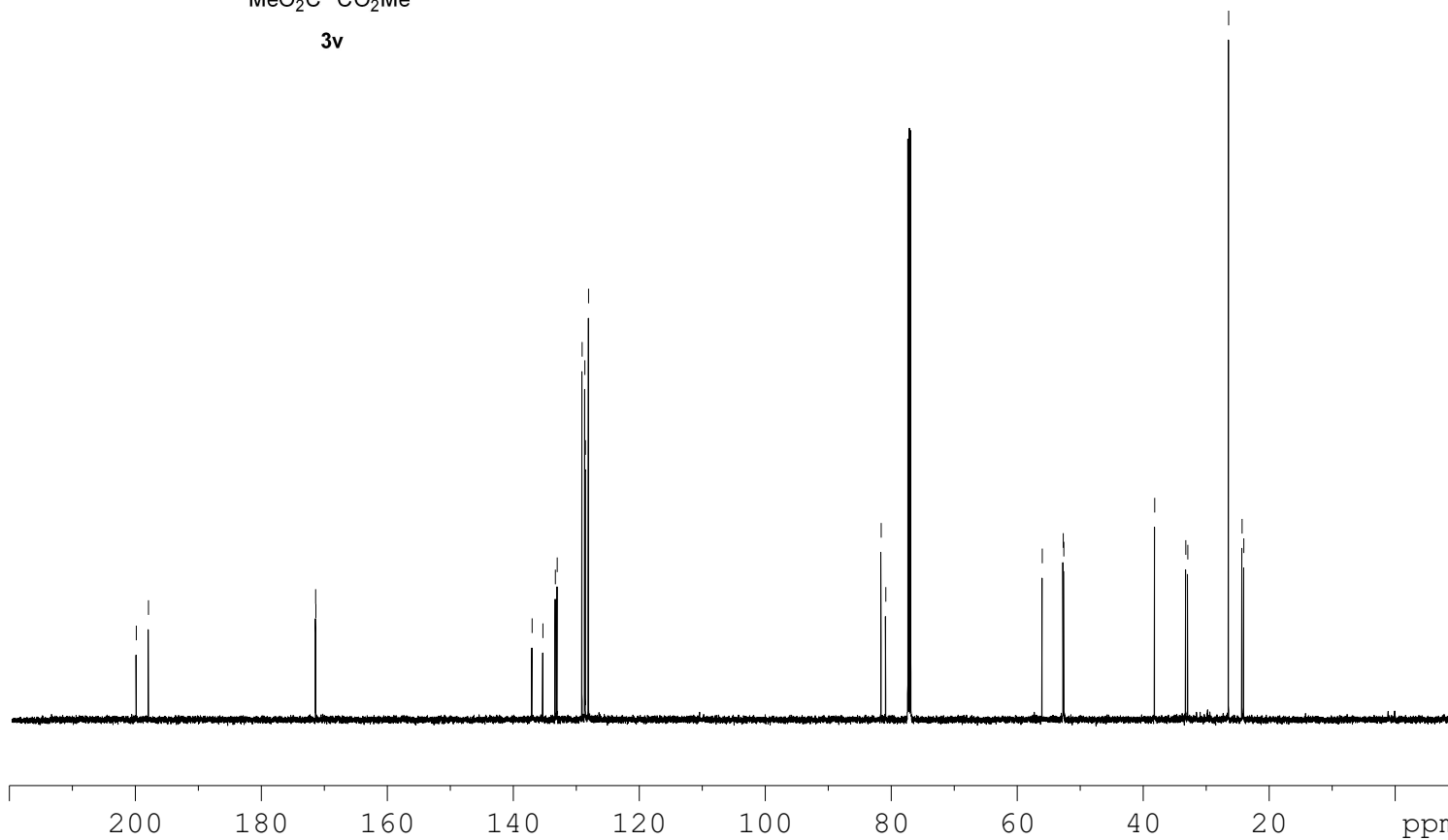
81.598
80.865

56.026
52.695
52.601
38.177
33.240
32.920
26.438
24.302
24.030

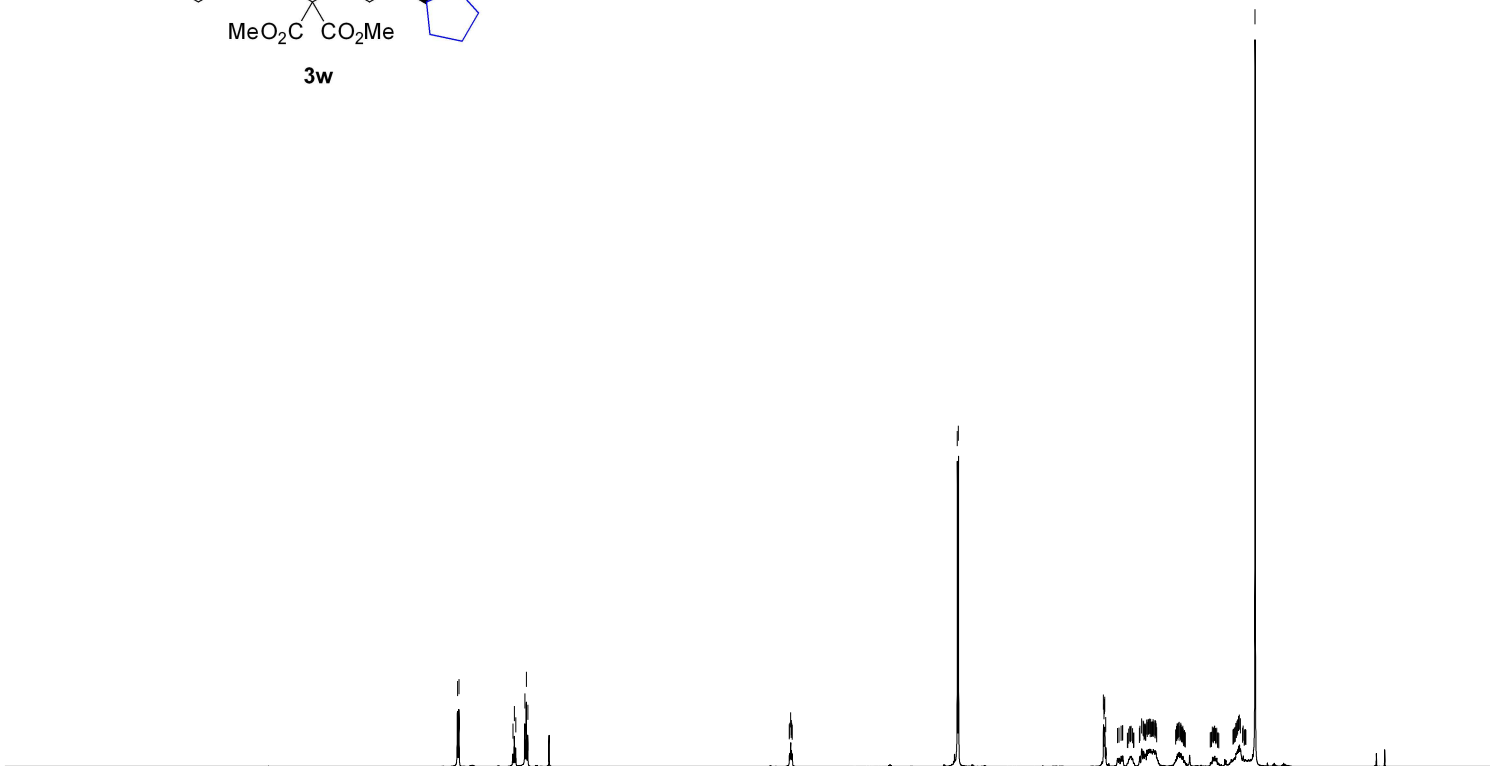
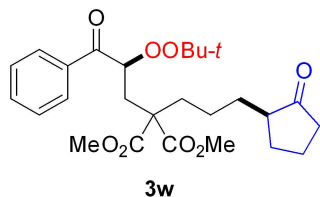
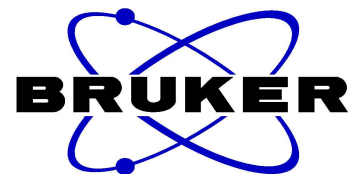


NAME w11-695p-20211118
EXPNO 2
PROCNO 1
Date_ 20211119
Time 0.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 190.02
DW 13.867 usec
DE 6.50 usec
TE 298.7 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

==== 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



8.057
8.045
7.575
7.563
7.551
7.471
7.458
7.445
5.169
5.164
5.157
5.151
5.148
5.144
3.706
3.697
2.434
2.429
2.425
2.422
2.413
2.266
2.101
2.088
2.083
2.079
2.074
2.072
2.070
2.067
2.065
2.057
2.052
2.046
2.041
2.036
2.030
2.026
2.022
2.017
2.013
2.006
2.001
1.996
1.992
1.985
1.981
1.978
1.974
1.971
1.789
1.784
1.781
1.774
1.763
1.760
1.757
1.280
1.276
1.270
1.265
1.257
1.250
1.240
1.217
1.113



```

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   CDC13
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
  
```

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

2.00 0.98 2.05 1.00 3.09 3.03 1.98 1.01 1.08 2.08 3.14 2.10 1.11 3.08 9.09

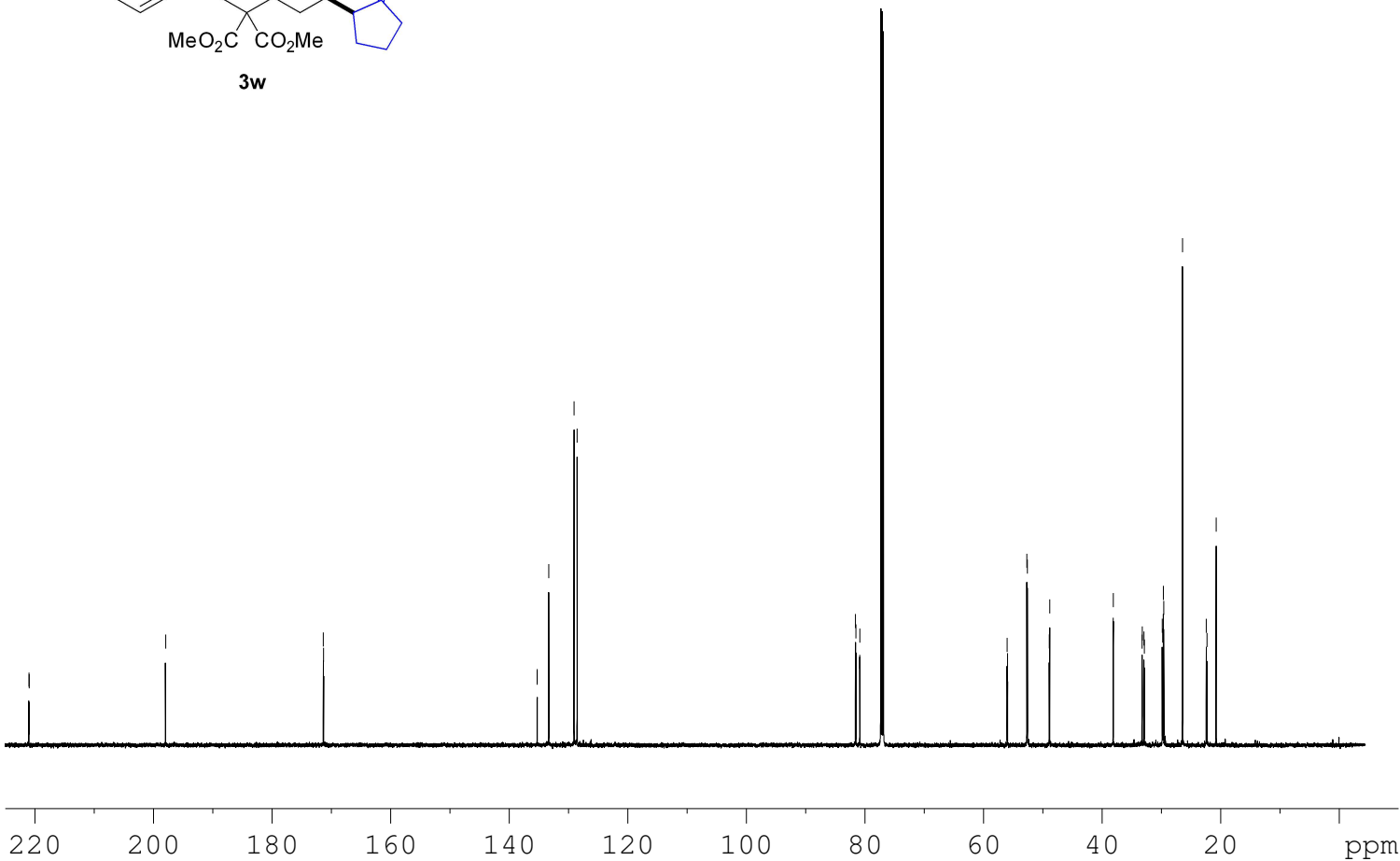
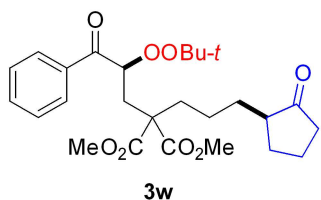
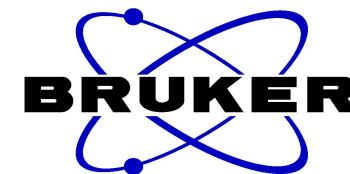
220.966

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



```

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

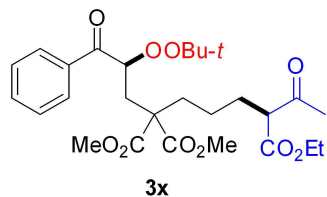
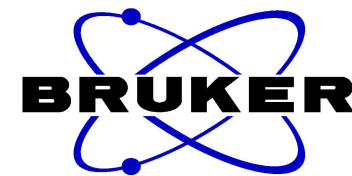
```

```

===== 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

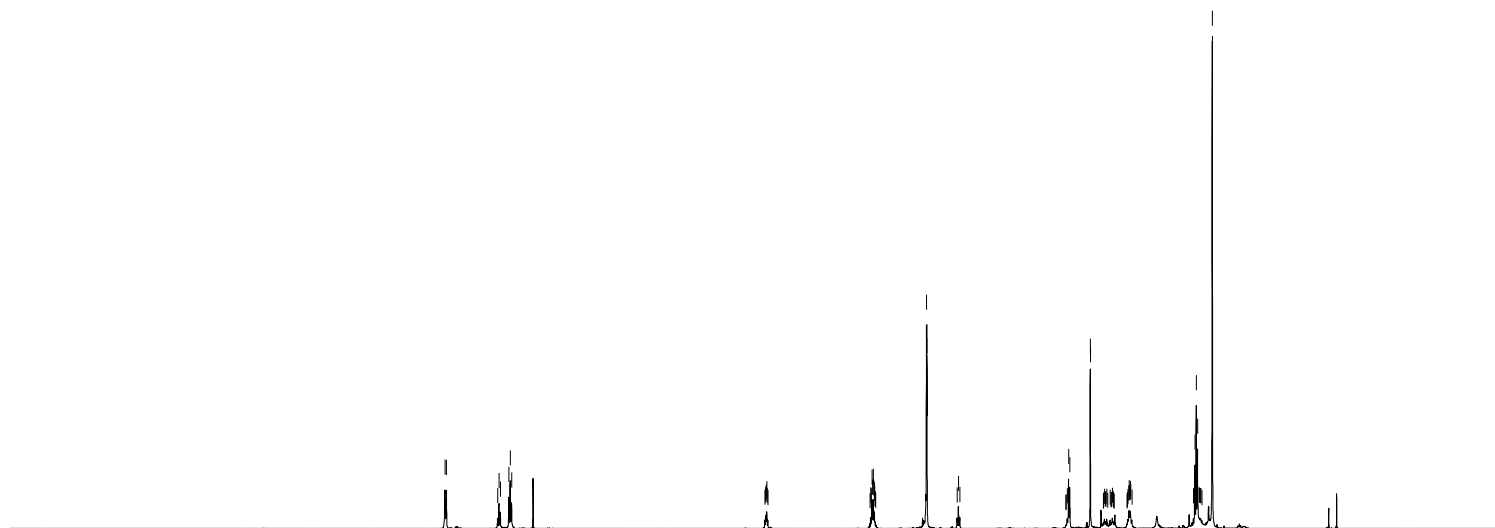
```

8.064
8.052
7.588
7.576
7.563
7.487
7.474
7.462
5.172
5.165
5.162
5.160
5.154
5.151
5.144
4.215
4.209
4.203
4.198
4.194
4.191
4.186
4.182
4.176
4.174
3.710
3.706
3.705
3.433
3.421
3.409
2.437
2.424
2.414
2.229
2.227
2.100
2.092
2.087
2.079
2.049
2.029
2.022
1.893
1.889
1.882
1.878
1.874
1.869
1.866
1.863
1.851
1.296
1.285
1.282
1.277
1.270
1.258
1.245
1.241
1.232
1.229
1.219
1.125
1.125



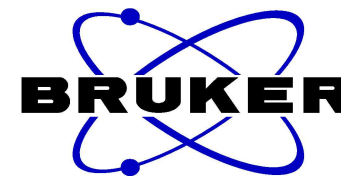
NAME w11-697p-20211118
EXPNO 1
PROCNO 1
Date_ 20211118
Time 21.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 9615.385 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 69.87
DW 52.000 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1

===== 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



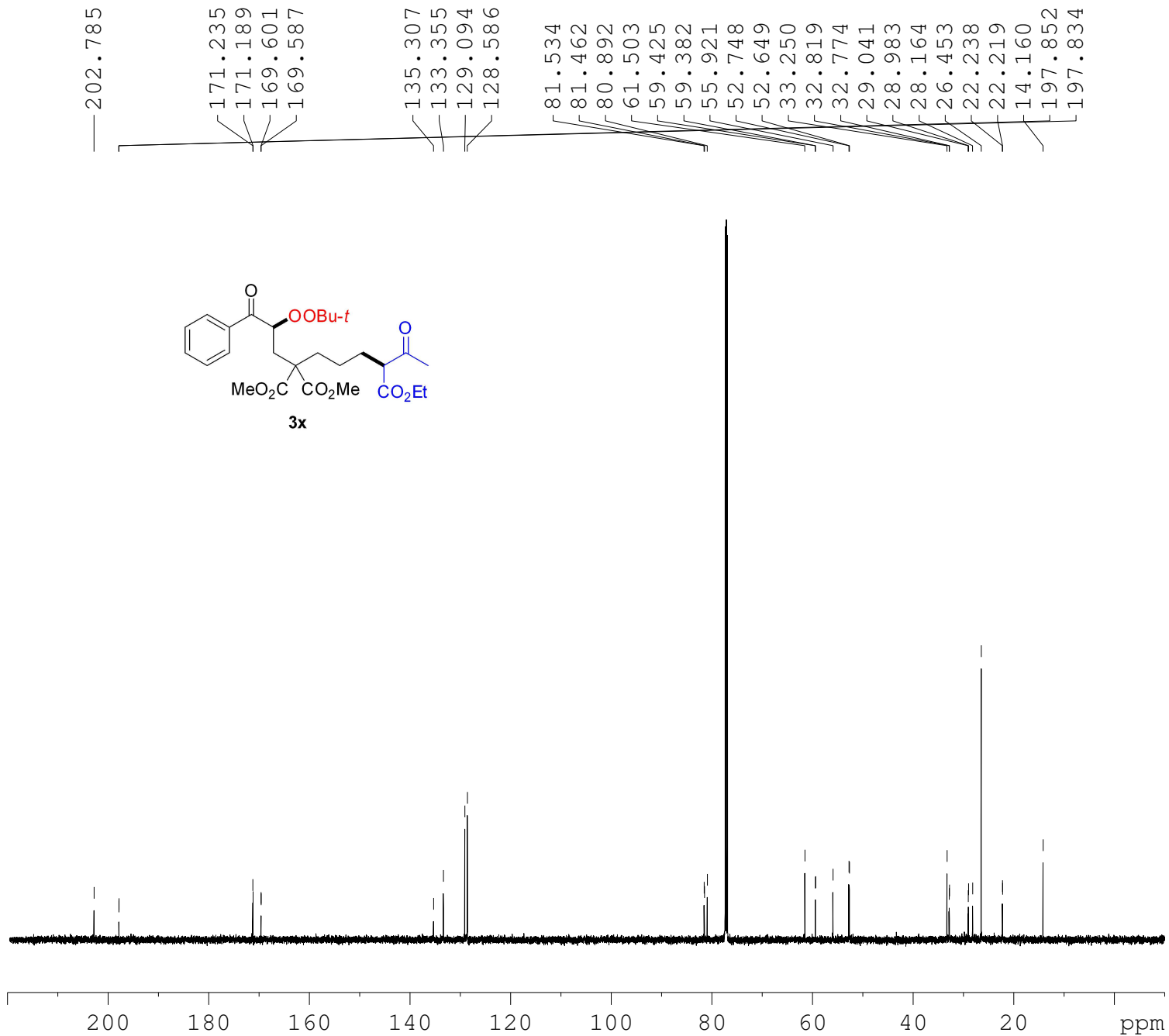
11 10 9 8 7 6 5 4 3 2 1 0 ppm

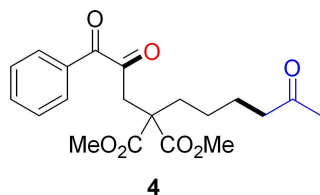
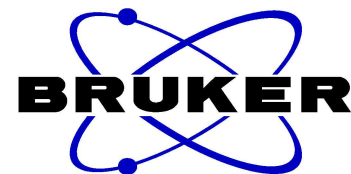
2.12
1.05
2.13
1.00
2.18
3.01
2.94
1.01
2.13
2.99
0.98
1.01
1.98
5.01
9.09



NAME w11-697p-20211118
EXPNO 2
PROCNO 1
Date_ 20211119
Time_ 0.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
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 2.0000000 sec
D11 0.03000000 sec
TD0 1

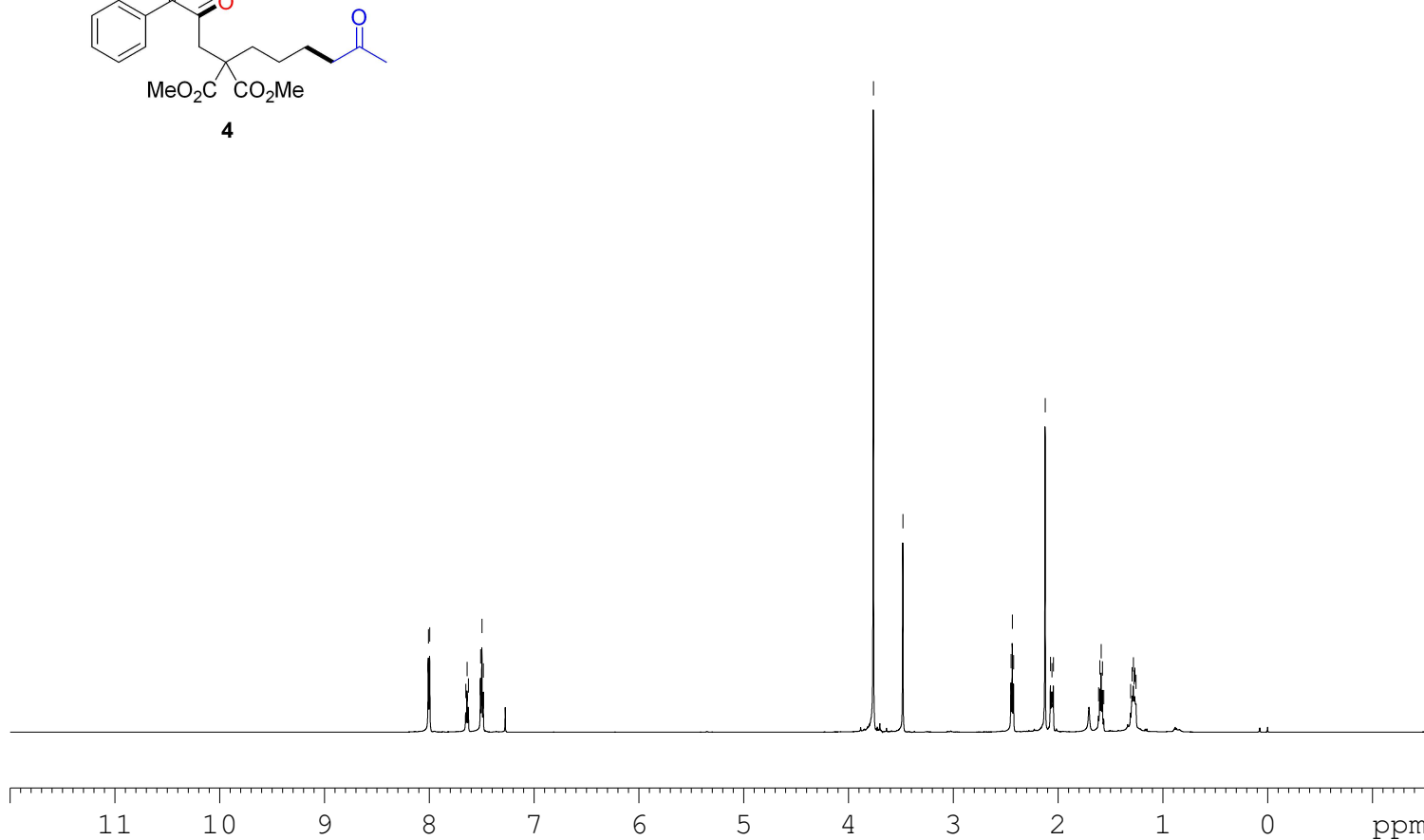
===== CHANNEL f1 =====
SF01 150.9279571 MHz
NUC1 13C
P1 11.90 usec
SI 32768
SF 150.9128560 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





8.008
7.996
7.652
7.639
7.627
7.510
7.497
7.484

3.762
3.481
2.449
2.437
2.424
2.123
2.072
2.059
2.044
1.614
1.601
1.589
1.577
1.564
1.308
1.294
1.282
1.275
1.267
1.257



1.926
1.000
2.042

6.085
1.992

2.067
3.065
1.934
2.143
2.150

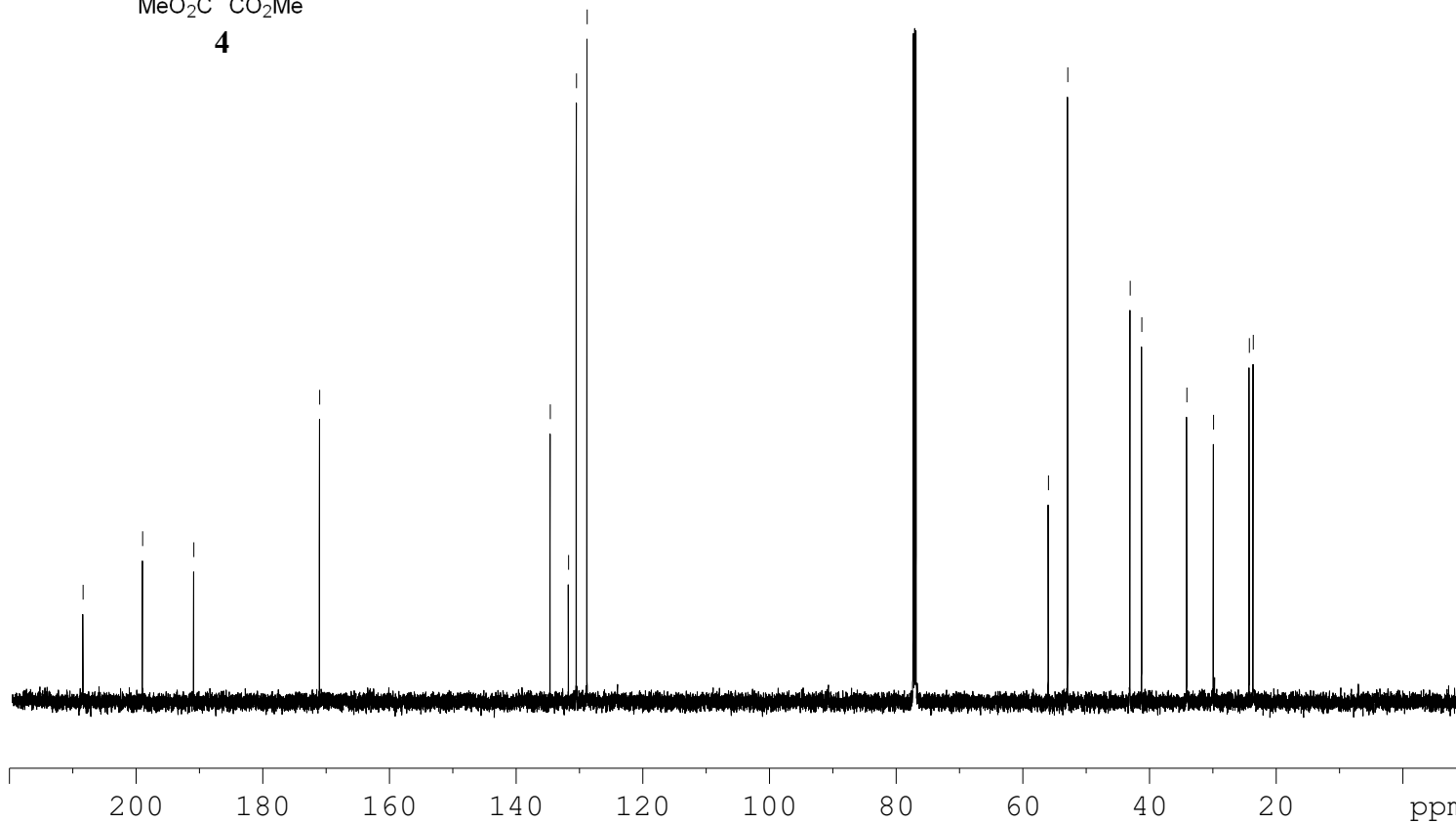
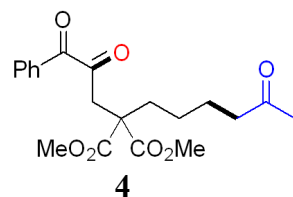
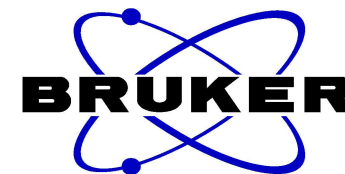
NAME WLL-578PP-20210709
EXPNO 1
PROCNO 1
Date_ 20210709
Time 15.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 0
SWH 9615.385 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 56.75
DW 52.000 usec
DE 6.50 usec
TE 301.1 K
D1 1.00000000 sec
TD0 1

===== 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

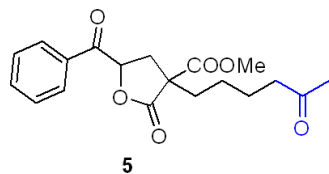
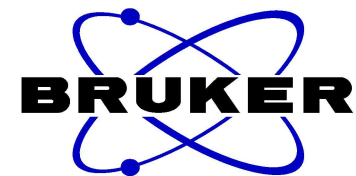
55.955
52.903
43.051
41.215
34.101
29.892
24.238
23.618



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

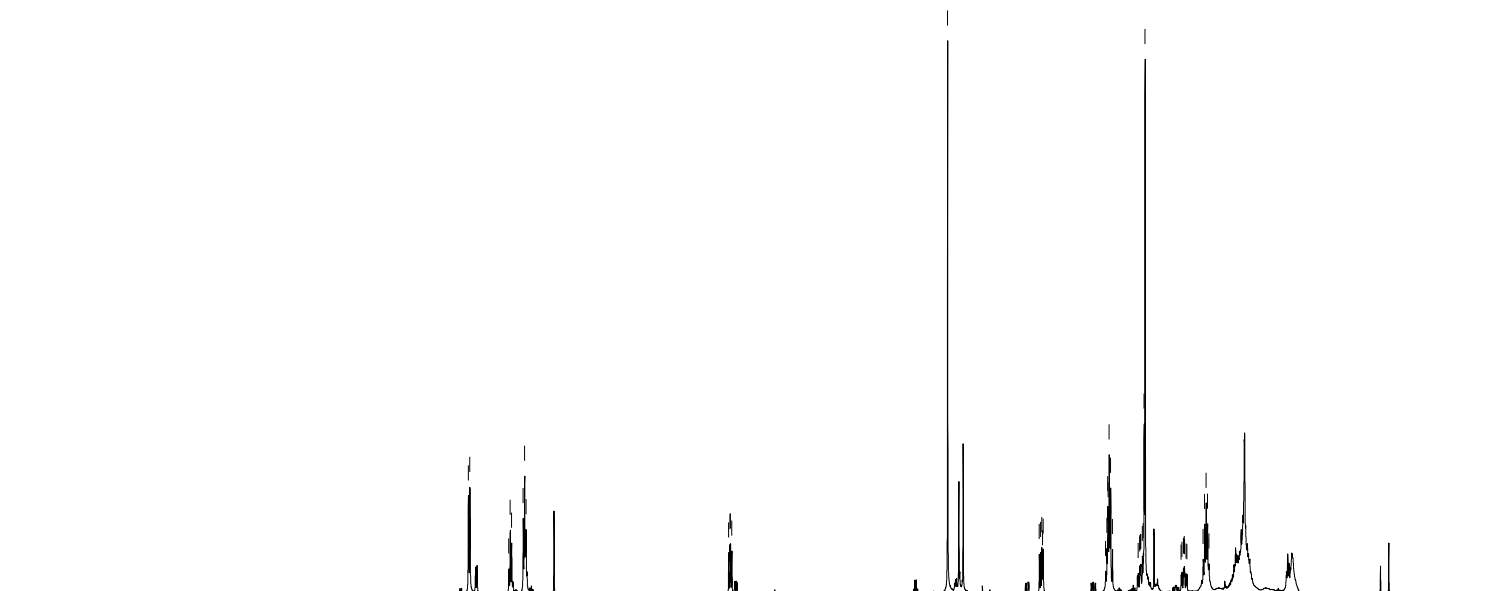
8.014
8.002
7.663
7.651
7.638
7.538
7.525
7.512
5.748
5.736
5.733
5.721
3.841
3.043
3.031
3.021
3.014
3.009
2.466
2.454
2.447
2.443
2.435
2.428
2.422
2.405
2.168
2.166
2.160
2.159
2.145
2.137
2.130
2.122
1.800
1.787
1.785
1.780
1.778
1.616
1.603
1.590
1.577
1.565



```

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   CDC13
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

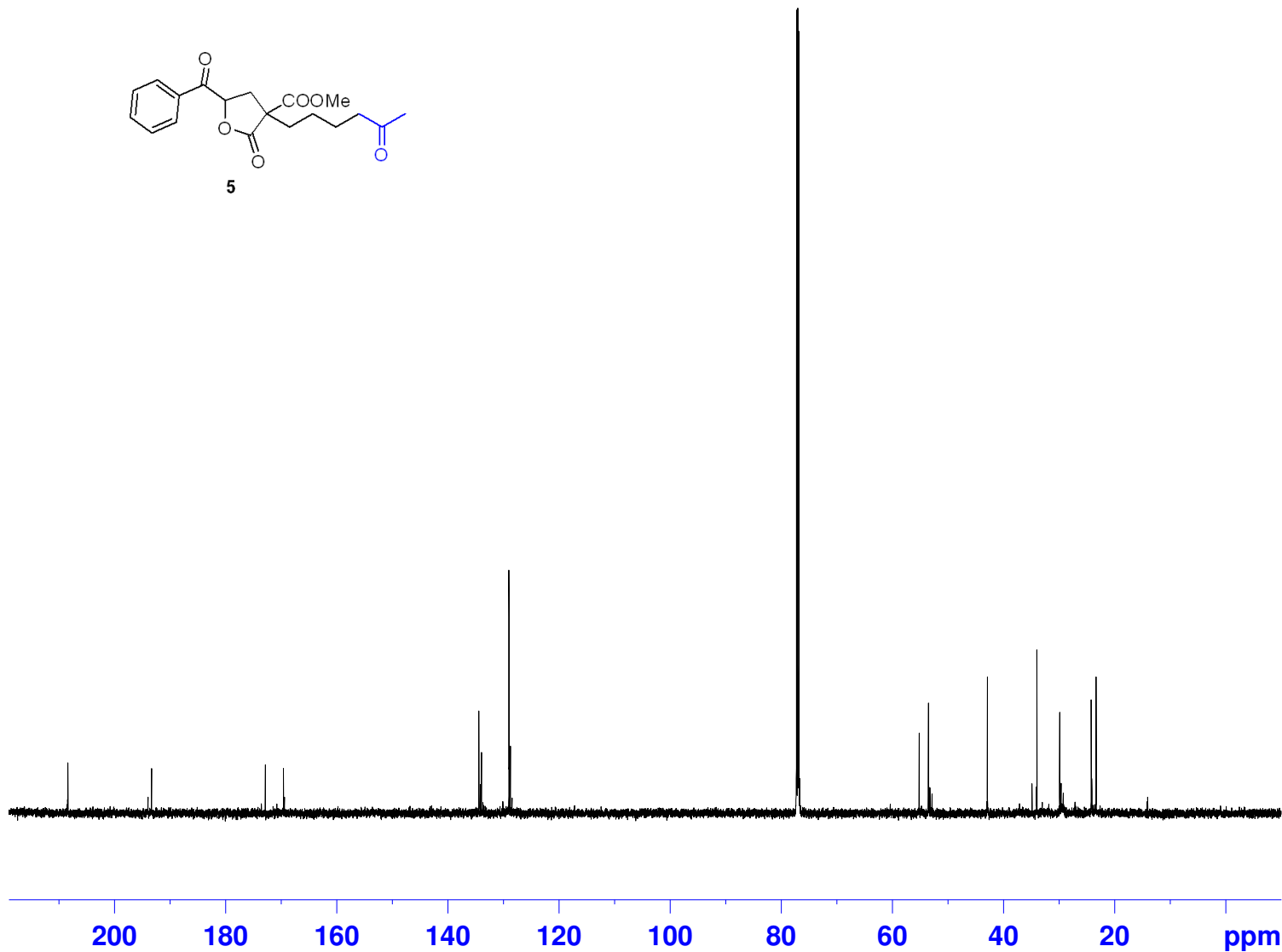
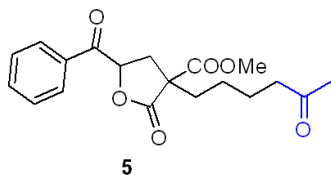
===== CHANNEL f1 =====
SFO1      600.1739011 MHz
NUC1      1H
P1        9.96 usec
SI        65536
SF        600.1700092 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



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

1.958
0.490
1.087
2.007
1.000
0.248
3.151
0.994
4.189
5.231
1.192
1.918

— 208.37
 < 193.95
 < 193.31
 < 172.82
 < 169.56
 < 169.40
 < 134.40
 < 134.13
 < 133.92
 < 128.99
 < 128.96
 < 128.93
 < 128.72
 < 76.91
 < 76.65
 < 55.16
 < 53.50
 < 42.93
 < 42.91
 — 34.02
 — 29.91
 — 24.24
 < 23.37
 < 23.33



```

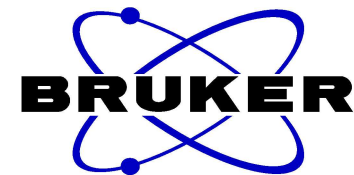
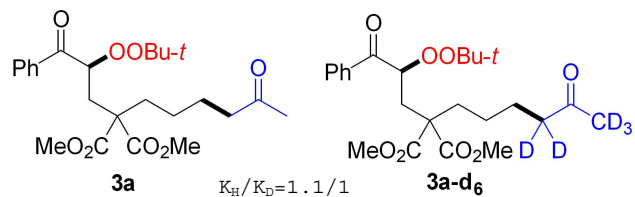
NAME      WLL-600-PRO-20211224
EXPNO     2
PROCNO    1
Date_     20211224
Time      16.52
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDC13
NS         300
DS         4
SWH       36057.691 Hz
FIDRES    0.550197 Hz
AQ         0.9088159 sec
RG         190.02
DW         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
  
```

8.069
8.057
7.588
7.576
7.564
7.484
7.471
7.458

5.170
5.161
5.158
5.149
3.716
3.708
2.450
2.437
2.428
2.425
2.411
2.398
2.131
2.105
2.097
2.089
2.082
2.068
2.061
2.038
2.030
2.017
2.009
1.994
1.986
1.631
1.618
1.605
1.593
1.580
1.261
1.254
1.240
1.232
1.219
1.211
1.202

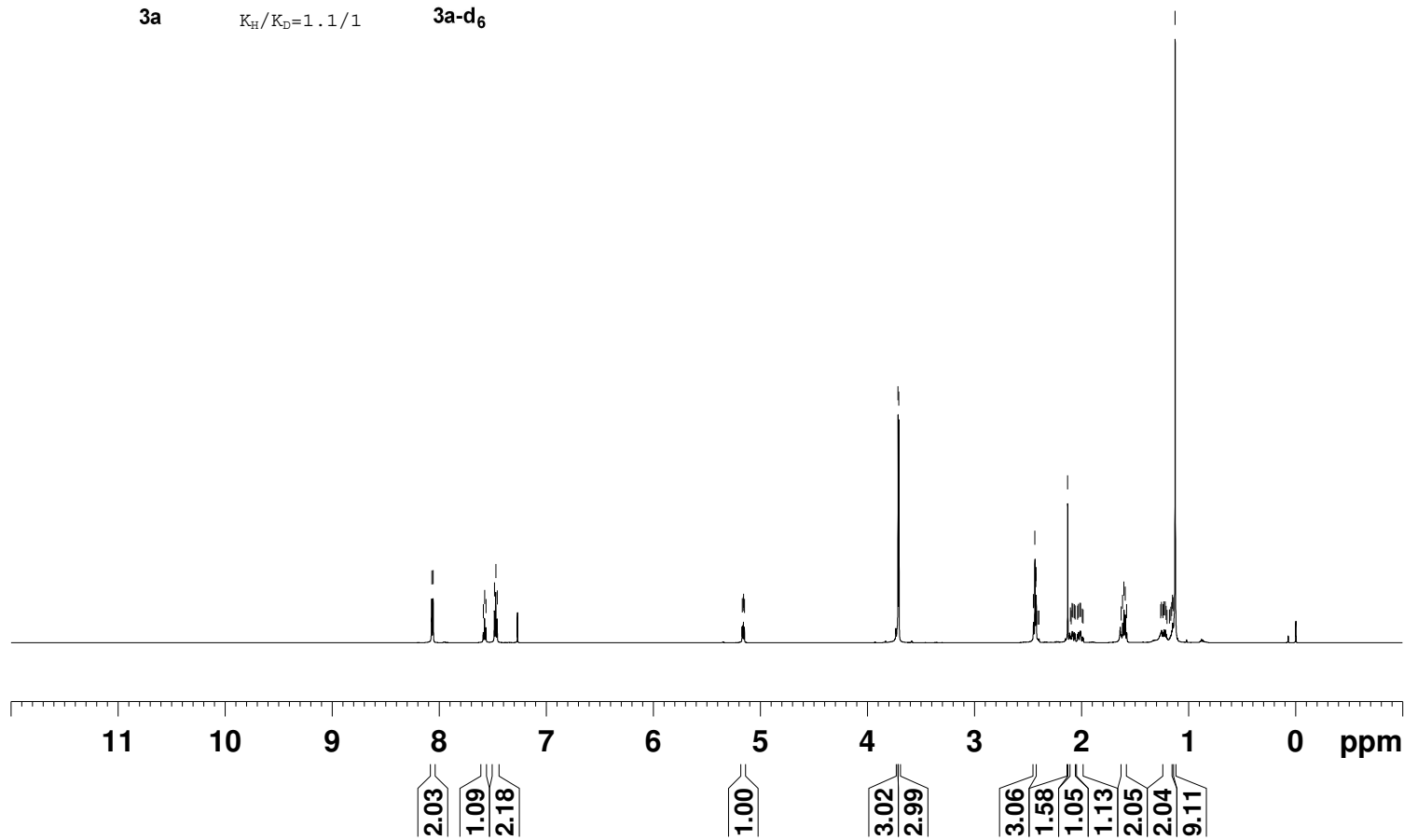


```

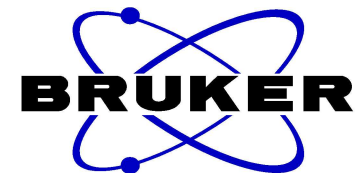
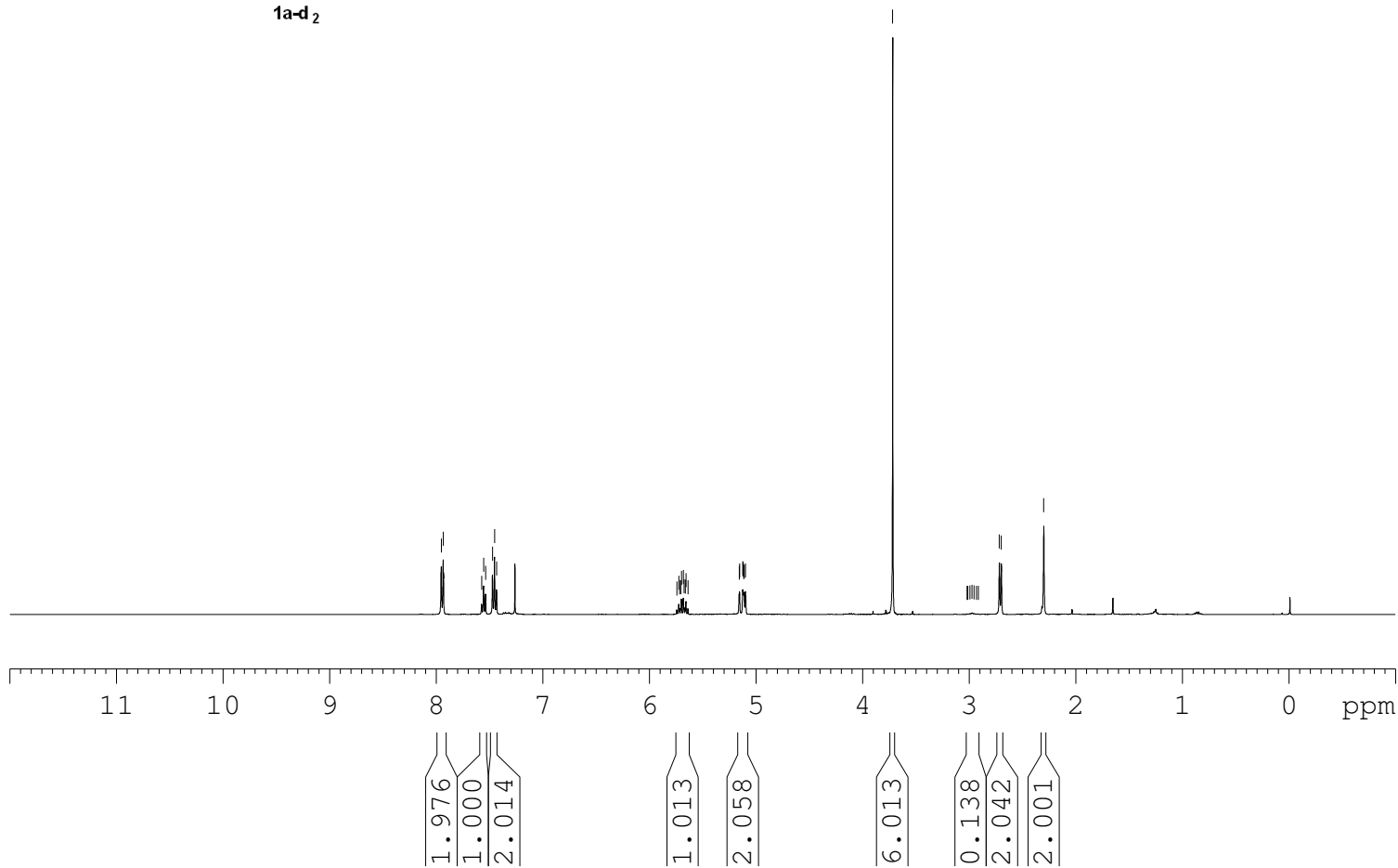
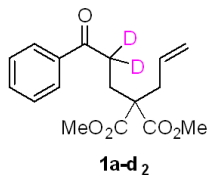
NAME      wll-614p-20211118
EXPNO     1
PROCNO    1
Date_     20211118
Time      21.23
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDC13
NS         8
DS         0
SWH       9615.385 Hz
FIDRES    0.146719 Hz
AQ         3.4079220 sec
RG         69.87
DW         52.000 usec
DE         6.50 usec
TE         297.7 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
  
```

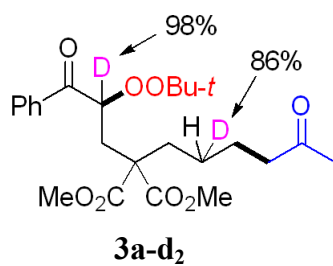


7.952
7.934
7.574
7.555
7.537
7.472
7.452
7.434
5.743
5.724
5.718
5.706
5.699
5.682
5.675
5.663
5.657
5.638
5.157
5.154
5.125
5.123
5.120
5.116
5.111
5.099
3.720
3.021
3.014
2.994
2.974
2.955
2.932
2.914
2.719
2.700
2.302



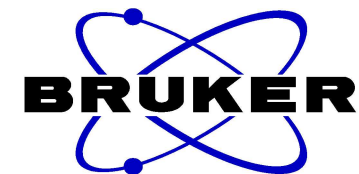
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EXPNO 1
PROCNO 1
Date_ 20210323
Time 15.14
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 161
DW 78.200 usec
DE 6.50 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1

===== 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



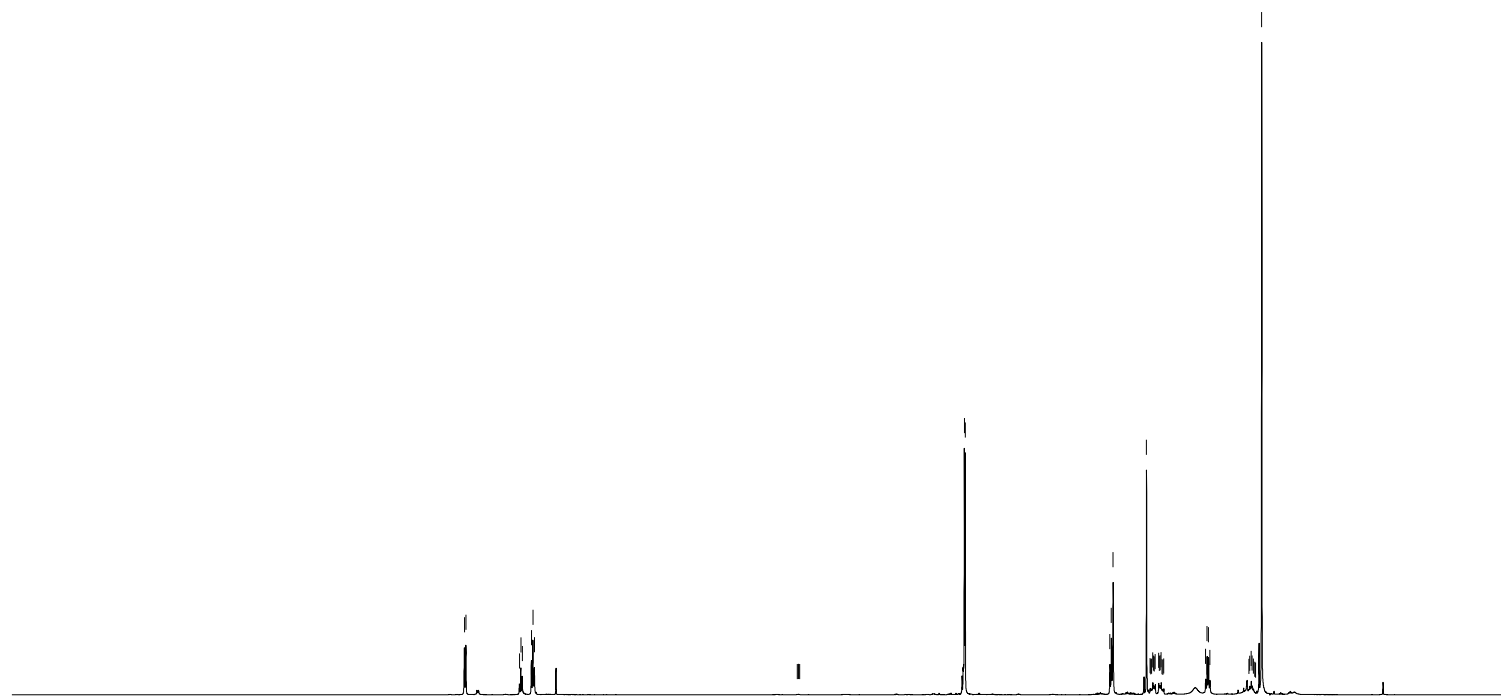
8.058
 8.046
 7.579
 7.566
 7.554
 7.475
 7.462
 7.449

3.704
 3.697
 2.439
 2.426
 2.411
 2.120
 2.088
 2.082
 2.075
 2.065
 2.059
 2.052
 2.044
 2.013
 2.007
 2.000
 1.992
 1.984
 1.977
 1.969
 1.605
 1.593
 1.580
 1.567
 1.227
 1.220
 1.208
 1.200
 1.188
 1.177
 1.169
 1.117
 1.160
 5.152



NAME w11-599p-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



2.00
 1.04
 2.13

0.02

3.09
 3.09

4.10
 3.07
 1.02
 1.01
 1.96
 1.14
 9.12

— 208.559

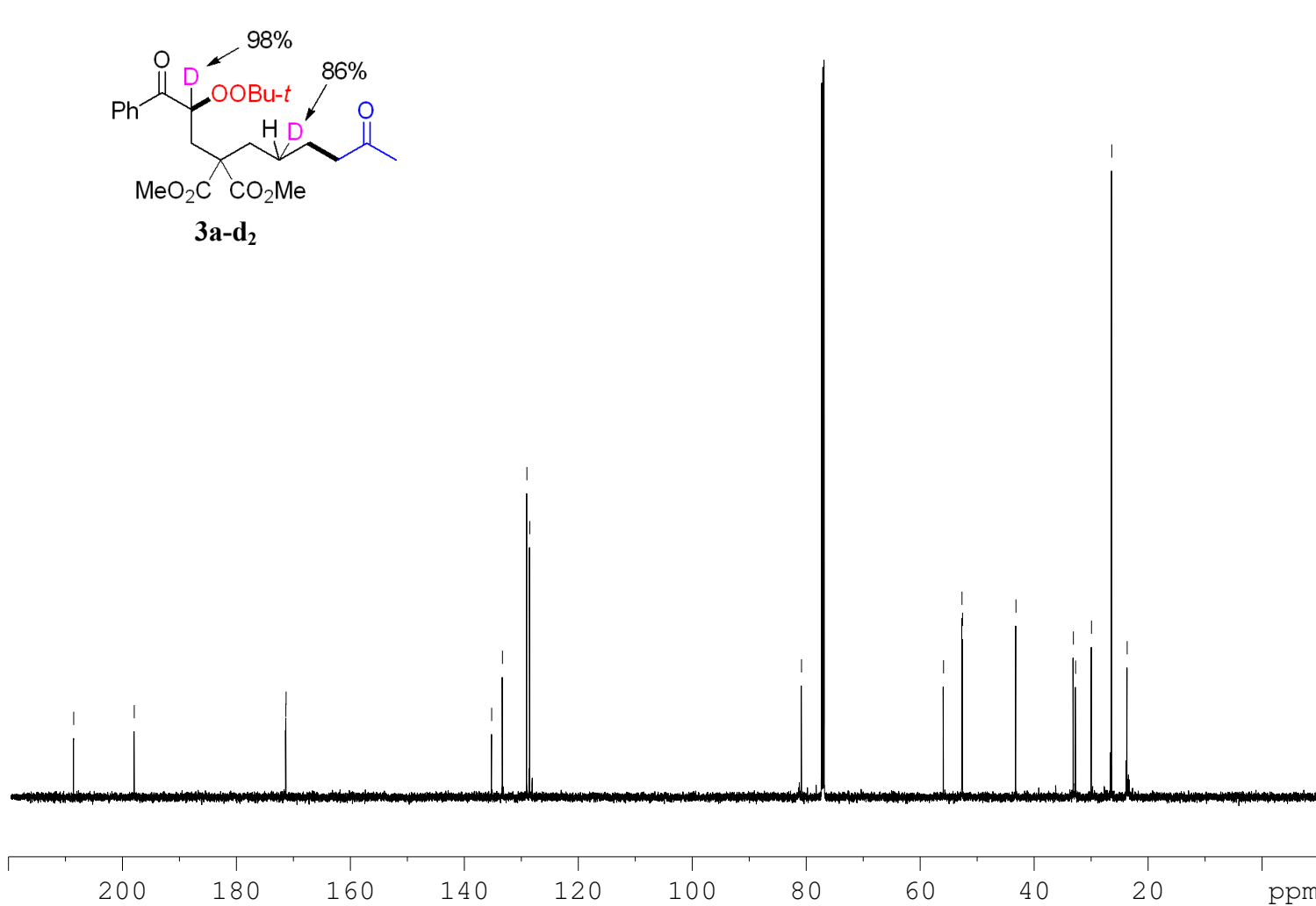
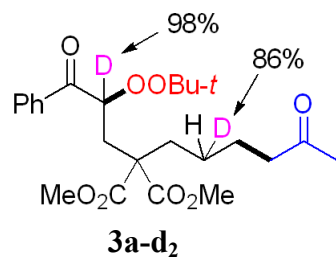
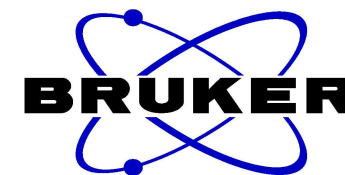
— 197.931

< 171.323
< 171.282

< 135.205
< 133.311
< 129.021
< 128.512

— 80.829

< 55.901
< 52.662
< 52.566
— 43.191
< 33.112
< 32.727
< 29.935
< 26.376
< 23.676



NAME w11-599p-20210727
EXPNO 2
PROCNO 1
Date_ 20210727
Time 20.09
INSTRUM spect
PROBHD 5 mm PABBO BE/
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