

Supporting Information

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Synthesis of pyrrolidine-3-carboxylic acid derivatives via asymmetric Michael addition reactions of carboxylate-substituted enones

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General

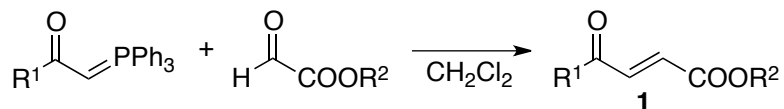
For thin layer chromatography (TLC), Merck silica gel 60 F254 aluminum sheets were used. Flash column chromatography was performed using Merck silica gel 60 (230-400 mesh). ^1H NMR and ^{13}C NMR were recorded on a Bruker Avance 400. Proton chemical shifts are reported in ppm downfield from tetramethylsilane or from the residual solvent as internal standard in CDCl_3 (δ 7.26 ppm) and in CD_3OD (δ 3.31 ppm). Carbon chemical shifts were internally referenced to the deuterated solvent signals in CDCl_3 (δ 77.0 ppm) and in CD_3OD (δ 49.0 ppm). High-resolution mass spectra were recorded on a Thermo Scientific LTQ Orbitrap ESI ion trap mass spectrometer. Optical rotations were measured on a Jasco P2200 polarimeter.

Note

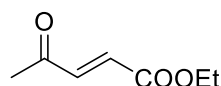
This corrected version was prepared based on the correct structure of **3**, determined from the X-ray crystal structure of a derivative of **3**, which will be reported separately in the future.

1. Synthesis of enones

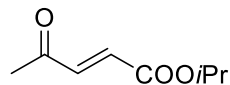
General procedure for the synthesis of enones



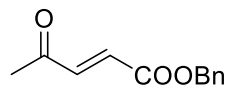
Enones were synthesized according to the reported procedures.¹ To a solution of glyoxylate ester or its derivative (10 mmol) in CH₂Cl₂ (30 mL), 1-(triphenylphosphoranylidene)-2-propanone or its derivative (33 mmol) was added and the mixture was stirred at room temperature (25°C) for 24 h. The mixture was concentrated and purified by flash column chromatography (hexane/EtOAc) to give enone **1**.



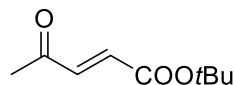
Known compound.² Pale yellow oil. ¹H NMR (400 MHz, CDCl₃): δ 7.01 (d, *J* = 16.1 Hz, 1H), 6.64 (d, *J* = 16.1 Hz, 1H), 4.26 (q, *J* = 7.1 Hz, 2H), 2.35 (s, 3H), 1.32 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 197.6, 165.4, 139.9, 131.6, 61.4, 28.0, 14.1.



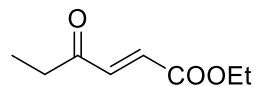
Known compound.³ Pale yellow oil. ¹H NMR (400 MHz, CDCl₃): δ 6.96 (d, *J* = 16.1 Hz, 1H), 6.59 (d, *J* = 16.1 Hz, 1H), 5.09 (septet, *J* = 6.2 Hz, 1H), 2.32 (s, 3H), 1.27 (d, *J* = 6.2 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃): δ 197.6, 164.9, 139.7, 132.1, 69.0, 27.9, 21.6.



Known compound.⁴ Pale yellow oil. ¹H NMR (400 MHz, CDCl₃): δ 7.51-7.31 (m, 5H), 7.05 (d, *J* = 16.1 Hz, 1H), 6.69 (d, *J* = 16.1 Hz, 1H), 5.25 (s, 2H), 2.35 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 197.4, 165.3, 140.3, 135.2, 128.7, 128.6, 128.4, 67.2, 28.1.

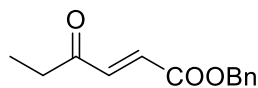


Known compound.⁵ Pale yellow oil. ¹H NMR (400 MHz, CDCl₃): δ 6.91 (d, *J* = 16.1 Hz, 1H), 6.57 (d, *J* = 16.1 Hz, 1H), 2.34 (s, 3H), 1.51 (s, 9H). ¹³C NMR (100 MHz, CDCl₃): δ 197.9, 164.6, 139.2, 133.7, 82.1, 28.0, 27.9.

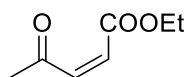


Known compound.⁴ Pale yellow oil. ¹H NMR (400 MHz, CDCl₃): δ 7.04 (d, *J* = 16.0 Hz, 1H), 6.66 (d, *J* = 16.0 Hz, 1H), 4.24 (q, *J* = 7.1 Hz, 2H), 2.65 (q, *J* = 7.2 Hz, 2H), 1.30 (t, *J* = 7.0 Hz, 3H), 1.11 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 200.1, 165.5, 139.1, 130.6, 61.3,

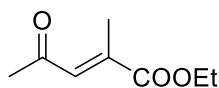
34.7, 14.1, 7.6.



Known compound.⁴ Pale yellow oil. ¹H NMR (400 MHz, CDCl₃): δ 7.48-7.31 (m, 5H), 7.10 (d, *J* = 16.0 Hz, 1H), 6.72 (d, *J* = 16.0 Hz, 1H), 5.24 (s, 2H), 2.66 (q, *J* = 7.2 Hz, 2H), 1.13 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 200.0, 165.4, 139.6, 135.2, 130.2, 128.6, 128.5, 128.4, 67.1, 34.8, 7.6.



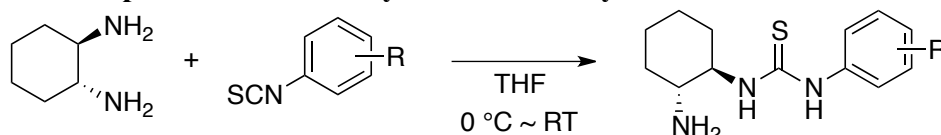
Known compound.⁶ This (*Z*)-isomer was generated during the synthesis of the (*E*)-isomer and was purified by flash column chromatography. Pale yellow oil. ¹H NMR (400 MHz, CDCl₃): δ 6.45 (d, *J* = 12.2 Hz, 1H), 6.02 (d, *J* = 12.2 Hz, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 2.36 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 201.3, 165.2, 141.6, 124.7, 61.2, 30.0, 14.0.



Known compound.⁷ Pale yellow oil. ¹H NMR (400 MHz, CDCl₃): δ 7.07 (s, 1H), 4.25 (q, *J* = 7.1 Hz, 2H), 2.30 (s, 3H), 2.21 (s, 3H), 1.32 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 199.4, 167.6, 140.9, 132.3, 61.6, 32.1, 14.3, 14.1.

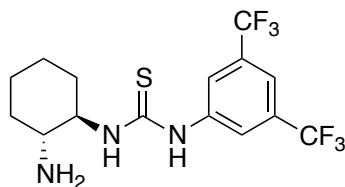
2. Synthesis of catalysts

General procedure for the synthesis of catalysts F and G



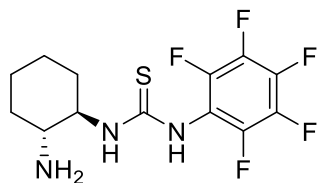
Catalysts **F** and **G** were synthesized according to the reported procedure.⁸ To a solution of (1*R*,2*R*)-cyclohexanediamine (154.0 mg, 0.64 mmol) in dry THF (5.0 mL), the corresponding isocyanate (0.70 mmol) was added dropwise at 0 °C. The mixture was stirred at 0 °C for 30 min and at room temperature (24 °C) for 16 h. The mixture was concentrated under reduced pressure and purified by flash column chromatography (CH₂Cl₂/MeOH = 9:1) to give the catalyst.

Catalyst F: 1-((1*R*,2*R*)-2-aminocyclohexyl)-3-(3,5-bis(trifluoromethyl)phenyl)thiourea⁸



Known compound.⁸

Catalyst G



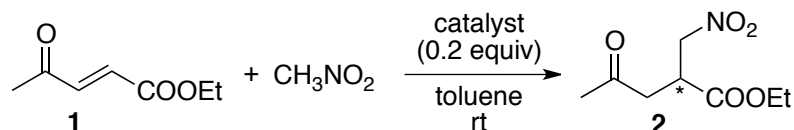
Colorless Solid. ^1H NMR (400 MHz, CDCl_3): δ 6.71 (bs, 1H), 3.60-3.30 (m, 1H), 3.02-2.57 (m, 1H), 2.39-1.64 (m, 4H), 1.44-1.07 (m, 4H). ^{13}C NMR (100 MHz, CD_3OD): δ 185.0, 148.5 ($J_{\text{C,F}} = 246$ Hz), 139.2 ($J_{\text{C,F}} = 246$ Hz), 116.7, 61.9, 56.0, 34.5, 32.5, 25.9, 25.7. ESI-HRMS: calcd for $\text{C}_{13}\text{H}_{15}\text{N}_3\text{F}_5\text{S}$ ($[\text{M}+\text{H}]^+$) 340.0901, found 340.0897.

3. Screening of catalysts and conditions

Procedure for the catalyst screening (Table 1)

To a mixture of catalyst (0.04 mmol) and additive (if used, 0.04 mmol) in solvent (0.5 mL), enone **1** (0.2 mmol) and nitromethane (1.0 mmol) were added at room temperature (24 °C) and the mixture (initially often suspension) was stirred at the same temperature. The progress of the reaction was monitored by TLC. After 48 h (except noted), the mixture was poured into aqueous 1 M HCl solution (1 mL) and extracted with CH_2Cl_2 . Organic layers were combined, dried over Na_2SO_4 , concentrated, and purified by flash column chromatography (hexane/EtOAc = 4:1) to afford **2**. The ee was determined by chiral-phase HPLC.

Table S1. Additional catalyst screening.

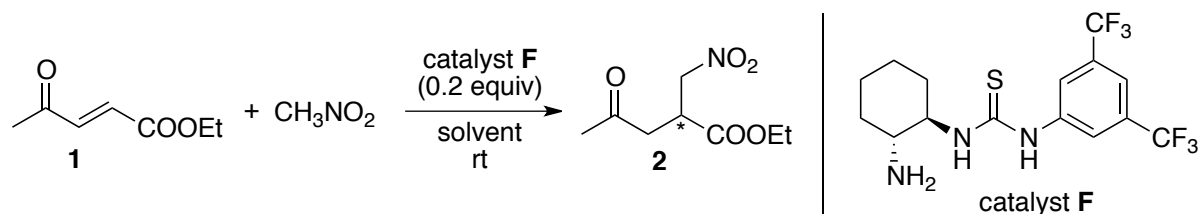


entry	catalyst	yield (%)	ee (%)
1		0	-
2		24	23
3		0	-

entry	catalyst	yield (%)	ee (%)
4		3	-
5		33	-

a Conditions: Enone **1** (0.2 mmol), nitromethane (1.0 mmol), and catalyst (0.04 mmol) in toluene (0.5 mL) at 24 °C for 48 h.

Table S2. Solvent screening in the catalyst **F**-catalyzed reaction.



entry	solvent	yield (%)	ee (%)
1	toluene	51	82
2	CH_2Cl_2	70	85
3	CHCl_3	62	82
4	CH_3CN	34	-
5	THF	42	-
6	<i>o</i> -xylene	58	89
7	EtOAc	42	-
8	<i>i</i> -PrOH	40	-

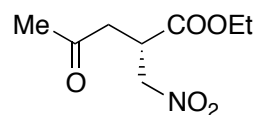
a Conditions: Enone **1** (0.2 mmol), nitromethane (1.0 mmol), and catalyst **F** (0.04 mmol) in solvent (0.5 mL) at 24 °C for 48 h.

4. Michael addition reactions to afford **2**

General procedure for the Michael addition reactions to afford **2** (Table 2 and Scheme 2)

To a mixture of catalyst **F** (0.04 mmol) and additive (if used, 0.04 mmol) in CH_2Cl_2 (0.2 mL), enone (0.2 mmol) and nitroalkane (1.0 mmol) were added at room temperature (24 °C) and the mixture (initially often suspension) was stirred at the same temperature. The progress of the reaction was monitored by TLC. After indicated time, the mixture was poured into aqueous 1M HCl aqueous solution (1 mL) and extracted with CH_2Cl_2 . Organic layers were combined, dried over Na_2SO_4 , concentrated, and purified by flash column chromatography (hexane/EtOAc) to give **2**. The ee was determined by chiral-phase HPLC. Racemic standards of the Michael Addition product were synthesized using racemic catalyst **F**.

Ethyl 2-(nitromethyl)-4-oxopentanoate (Compound **2a**)



To a mixture of 1-((1*R*,2*R*)-2-aminocyclohexyl)-3-(3,5-bis(trifluoromethyl)phenyl)thiourea (catalyst **F**) (15.4 mg, 0.04 mmol) and acetic acid (1.8 μL , 0.04 mmol) in CH_2Cl_2 (0.2 mL), (*E*)-ethyl 4-oxopent-2-enoate (**1a**) (28.4 mg, 0.2 mmol) and nitromethane (54.2 μL , 1.0 mmol) were added at 10 °C and the mixture (initially suspension) was stirred at the same temperature. The progress of the reaction was monitored by TLC. After 5 days, the mixture was poured into

aqueous 1M HCl solution (1.0 mL) and extracted with CH₂Cl₂. Organic layers were combined, dried over Na₂SO₄, concentrated, and purified by flash column chromatography (hexane/ EtOAc = 4:1) to afford **2** (30.9 mg, 76 %, 94 % ee).

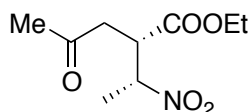
Pale yellow oil. ¹H NMR (400 MHz, CDCl₃): δ 4.74 (dd, *J* = 14.4 Hz, 6.0 Hz, 1H), 4.69 (dd, *J* = 14.4 Hz, 5.6 Hz, 1H), 4.23-4.15 (m, 2H), 3.57-3.49 (m, 1H), 3.04 (dd, *J* = 18.6 Hz, 5.6 Hz, 1H), 2.81 (dd, *J* = 18.6 Hz, 6.6 Hz, 1H), 2.20 (s, 3H), 1.25 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 204.9, 170.5, 61.9, 41.4, 38.2, 29.9, 13.9. ESI-HRMS: calcd for C₈H₁₄O₅N ([M+H]⁺) 204.0872, found 204.0849. HPLC (Daicel Chiralpak IA, hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 220 nm): *t*_R (major enantiomer) = 27.0 min, *t*_R (minor enantiomer) = 24.4 min.

A 2 gram-scale synthesis of 2a. A mixture of catalyst **F** (819.5 mg, 2.13 mmol), enone **1a** (2.14 g, 15.1 mmol), and nitromethane (4.0 mL, 75 mmol) in CH₂Cl₂ (10 mL) was stirred at room temperature (24 °C). The progress of the reaction was monitored by TLC. After 4 days, the mixture was poured into aqueous 1M HCl solution (15 mL) and extracted with CH₂Cl₂. Organic layers were combined, dried over Na₂SO₄, concentrated, and purified by flash column chromatography (hexane/EtOAc = 4:1) to afford **2a** (2.1 g, 69%, 90% ee).

Compound 2b

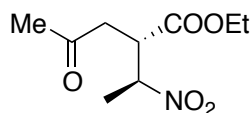
The dr was determined by ¹H NMR analysis before purification. The diastereomers (compounds **2b-1** and **2b-2**) were separately purified by flash column chromatography. Relative stereochemistries were tentatively assigned based on the NOESY experiments.

Compound 2b-1



Rf 0.35 (hexane/EtOAc = 4:1). Pale yellow oil, 21.7 mg, 50%, 90% ee. ¹H NMR (400 MHz, CDCl₃): δ 4.95 (qd, *J* = 8.9 Hz, 5.6 Hz, 1H), 4.26-4.13 (m, 2H), 3.44 (ddd, *J* = 9.4 Hz, 5.6 Hz, 3.6 Hz, 1H), 3.05 (dd, *J* = 17.9 Hz, 9.4 Hz, 1H), 2.53 (dd, *J* = 17.9 Hz, 3.6 Hz, 1H), 2.21 (s, 3H), 1.57 (d, *J* = 6.9 Hz, 3H), 1.27 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 204.9, 170.6, 82.5, 61.8, 44.2, 40.5, 29.9, 16.8, 14.0. ESI-HRMS: calcd for C₉H₁₆O₅N ([M+H]⁺) 218.1028, found 218.1004. HPLC (Daicel Chiralpak AS, hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 220 nm): *t*_R (major enantiomer) = 38.4 min, *t*_R (minor enantiomer) = 44.8 min.

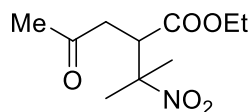
Compound 2b-2



Rf 0.32 (hexane/EtOAc = 4:1). Pale yellow oil, 21.7 mg, 50%, 93% ee. ¹H NMR (400 MHz, CDCl₃): δ 4.92 (qd, *J* = 6.8 Hz, 5.6 Hz, 1H), 4.18 (q, *J* = 7.2 Hz, 2H), 3.62 (ddd, *J* = 8.5 Hz, 5.6 Hz, 4.4 Hz, 1H), 3.01 (dd, *J* = 18.0 Hz, 8.5 Hz, 1H), 2.67 (dd, *J* = 18.0 Hz, 4.4 Hz, 1H), 2.21 (s, 3H), 1.55 (d, *J* = 6.8 Hz, 3H), 1.26 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 204.8,

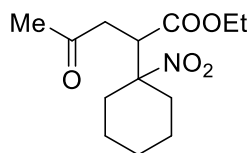
170.5, 82.0, 61.8, 43.6, 40.3, 30.0, 16.2, 14.0. ESI-HRMS: calcd for $C_9H_{16}O_5N$ ($[M+H]^+$) 218.1028, found 218.1004. HPLC (Daicel Chiralpak AS, hexane/*i*-PrOH = 75/25, flow rate 0.5 mL/min, $\lambda = 220$ nm): t_R (major enantiomer) = 14.1 min, t_R (minor enantiomer) = 13.6 min.

Compound 2c



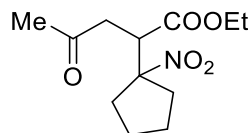
Pale yellow oil, 41.6 mg, 90%, 92% ee. 1H NMR (400 MHz, $CDCl_3$): δ 4.17 (q, $J = 7.1$ Hz, 2H), 3.66 (dd, $J = 11.2$ Hz, 2.4 Hz, 1H), 3.04 (dd, $J = 17.8$ Hz, 11.2 Hz, 1H), 2.41 (dd, $J = 17.8$ Hz, 2.4 Hz, 1H), 2.17 (s, 3H), 1.63 (s, 3H), 1.59 (s, 3H), 1.27 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 204.9, 170.8, 88.3, 61.6, 48.3, 41.4, 29.8, 25.5, 23.1, 14.0. ESI-HRMS: calcd for $C_{10}H_{18}O_5N$ ($[M+H]^+$) 232.1179, found 232.1173. HPLC (Daicel Chiralpak AS, hexane/*i*-PrOH = 97/3, flow rate 0.5 mL/min, $\lambda = 220$ nm): t_R (major enantiomer) = 28.4 min, t_R (minor enantiomer) = 27.2 min.

Compound 2d



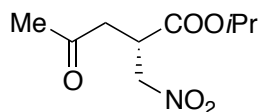
Pale yellow oil, 35.3 mg, 65%, 92% ee. 1H NMR (400 MHz, $CDCl_3$): δ 4.17 (q, $J = 7.1$ Hz, 2H), 3.30 (dd, $J = 11.4$ Hz, 3.0 Hz, 1H), 3.04 (dd, $J = 18.0$ Hz, 11.4 Hz, 1H), 2.57-2.42 (m, 3H), 2.15 (s, 3H), 1.77-1.52 (m, 4H), 1.46-1.12 (m, 4H), 1.28 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 205.3, 170.6, 91.8, 61.5, 49.2, 40.9, 33.3, 31.4, 29.9, 24.4, 22.2, 22.1, 14.0. ESI-HRMS: calcd for $C_{13}H_{22}O_5N$ ($[M+H]^+$) 272.1498, found 272.1470. HPLC (Daicel Chiralpak IA, hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, $\lambda = 220$ nm): t_R (major enantiomer) = 20.1 min, t_R (minor enantiomer) = 17.9 min.

Compound 2e



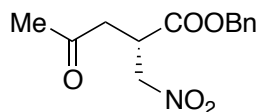
Pale yellow oil, 43.7 mg, 85%, 93% ee. 1H NMR (400 MHz, $CDCl_3$): δ 4.21-4.12 (m, 2H), 3.56 (dd, $J = 10.8$ Hz, 2.9 Hz, 1H), 3.07 (dd, $J = 18.0$ Hz, 10.8 Hz, 1H), 2.70-2.60 (m, 1H), 2.58-2.49 (m, 1H), 2.50 (dd, $J = 18.0$ Hz, 2.9 Hz, 1H), 2.17 (s, 3H), 2.12-2.00 (m, 1H), 1.96-1.84 (m, 1H), 1.80-1.64 (m, 4H), 1.26 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 205.3, 170.7, 100.3, 61.5, 47.3, 42.1, 36.8, 35.3, 29.8, 24.0, 23.6, 14.0. ESI-HRMS: calcd for $C_{12}H_{20}O_5N$ ($[M+H]^+$) 258.1341, found 258.1319. HPLC (Daicel Chiralpak AS, hexane/*i*-PrOH = 98/2, flow rate 0.5 mL/min, $\lambda = 220$ nm): t_R (major enantiomer) = 28.4 min, t_R (minor enantiomer) = 27.2 min.

Compound 2f



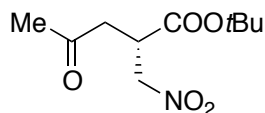
Pale yellow oil, 32.6 mg, 75%, 94% ee. ^1H NMR (400 MHz, CDCl_3): δ 5.05 (septet, $J = 6.2$ Hz, 1H), 4.73 (dd, $J = 14.4$ Hz, 5.6 Hz, 1H), 4.69 (dd, $J = 14.4$ Hz, 5.6 Hz, 1H), 3.54-3.46 (m, 1H), 3.03 (dd, $J = 18.5$ Hz, 5.6 Hz, 1H), 2.80 (dd, $J = 18.5$ Hz, 6.6 Hz, 1H), 2.21 (s, 3H), 1.24 (d, $J = 6.2$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 204.9, 170.0, 74.8, 69.7, 41.4, 38.4, 29.9, 21.6, 21.5. ESI-HRMS: calcd for $\text{C}_9\text{H}_{16}\text{O}_5\text{N}$ ($[\text{M}+\text{H}]^+$) 218.1028, found 218.1004. HPLC (Daicel Chiralpak AS, hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, $\lambda = 220$ nm): t_R (major enantiomer) = 45.1 min, t_R (minor enantiomer) = 43.2 min.

Compound 2g



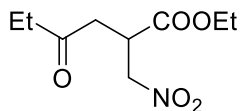
Pale yellow oil, 38.2 mg, 72%, 94% ee. ^1H NMR (400 MHz, CDCl_3): δ 7.73-7.28 (m, 5H), 5.16 (s, 2H), 4.80-4.67 (m, 2H), 3.64-3.55 (m, 1H), 3.03 (dd, $J = 18.6$ Hz, 5.5 Hz, 1H), 2.82 (dd, $J = 18.6$ Hz, 6.5 Hz, 1H), 2.17 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 204.8, 170.4, 135.0, 128.7, 128.6, 128.3, 74.6, 67.6, 41.4, 38.3, 29.8. ESI-HRMS: calcd for $\text{C}_{13}\text{H}_{16}\text{O}_5\text{N}$ ($[\text{M}+\text{H}]^+$) 266.1028, found 266.1003. HPLC (Daicel Chiralpak AS, hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, $\lambda = 220$ nm): t_R (major enantiomer) = 44.8 min, t_R (minor enantiomer) = 39.0 min.

Compound 2h



Pale yellow oil, 36.5 mg, 79%, 93% ee. ^1H NMR (400 MHz, CDCl_3): δ 4.69 (dd, $J = 14.0$ Hz, 6.0 Hz, 1H), 4.65 (dd, $J = 14.0$ Hz, 5.6 Hz, 1H), 3.49-3.41 (m, 1H), 2.99 (dd, $J = 18.5$ Hz, 5.7 Hz, 1H), 2.76 (dd, $J = 18.5$ Hz, 6.6 Hz, 1H), 2.20 (s, 3H), 1.44 (s, 9H). ^{13}C NMR (100 MHz, CDCl_3): δ 205.2, 169.5, 82.6, 74.9, 41.5, 39.0, 29.9, 27.8. ESI-HRMS: calcd for $\text{C}_{10}\text{H}_{18}\text{O}_5\text{N}$ ($[\text{M}+\text{H}]^+$) 232.1179, found 232.1173. HPLC (Daicel Chiralpak IA, hexane/*i*-PrOH = 99/1, flow rate 0.5 mL/min, $\lambda = 220$ nm): t_R (major enantiomer) = 34.3 min, t_R (minor enantiomer) = 32.3 min.

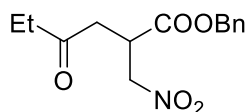
Compound 2i



Pale yellow oil, 30.4 mg, 70%, 96% ee. ^1H NMR (400 MHz, CDCl_3): δ 4.75 (dd, $J = 14.0$ Hz, 6.0 Hz, 1H), 4.70 (dd, $J = 14.0$ Hz, 5.6 Hz, 1H), 4.25-4.15 (m, 2H), 3.60-3.51 (m, 1H), 3.00 (dd, $J =$

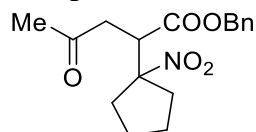
18.3 Hz, 5.6 Hz, 1H), 2.77 (dd, $J = 18.3$ Hz, 6.5 Hz, 1H), 2.57- 2.42 (m, 2H), 1.26 (t, $J = 7.1$ Hz, 3H), 1.08 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 207.9, 170.6, 74.8, 61.8, 40.1, 38.2, 36.0, 14.0, 7.6. ESI-HRMS: calcd for $\text{C}_9\text{H}_{16}\text{O}_5\text{N}$ ($[\text{M}+\text{H}]^+$) 218.1028, found 218.1004. HPLC (Daicel Chiralpak AS, hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, $\lambda = 220$ nm): t_{R} (major enantiomer) = 40.4 min, t_{R} (minor enantiomer) = 37.1 min.

Compound 2j



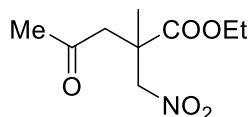
Pale yellow oil, 33.5 mg, 60%, 95% ee. ^1H NMR (400 MHz, CDCl_3): δ 7.46 (dd, $J = 14.4$ Hz, 6.0 Hz, 1H), 4.72 (dd, $J = 14.4$ Hz, 5.6 Hz, 1H), 5.16 (s, 2H), 4.82-4.68 (m, 2H), 3.67-3.57 (m, 1H), 3.00 (dd, $J = 18.3$ Hz, 5.6 Hz, 1H), 2.78 (dd, $J = 18.3$ Hz, 6.5 Hz, 1H), 2.51-2.36 (m, 2H), 1.05 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 207.8, 170.5, 135.0, 128.7, 128.6, 128.3, 74.7, 67.6, 40.1, 38.2, 36.0, 7.6. ESI-HRMS: calcd for $\text{C}_{14}\text{H}_{18}\text{O}_5\text{N}$ ($[\text{M}+\text{H}]^+$) 280.1179, found 280.1173. HPLC (Daicel Chiralpak AS, hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, $\lambda = 220$ nm): t_{R} (major enantiomer) = 56.7 min, t_{R} (minor enantiomer) = 48.8 min.

Compound 2k



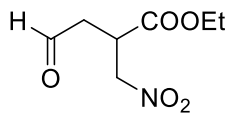
Colorless oil, 46.0 mg, 72%, 90% ee. ^1H NMR (400 MHz, CDCl_3): δ 7.42-7.29 (m, 5H), 5.16 (d, $J = 12.2$ Hz, 1H), 5.12 (d, $J = 12.2$ Hz, 1H), 3.63 (dd, $J = 10.8$ Hz, 2.9 Hz, 1H), 3.09 (dd, $J = 18.0$ Hz, 10.8 Hz, 1H), 2.69-2.58 (m, 1H), 2.56-2.43 (m, 2H), 2.14 (s, 3H), 2.09-1.97 (m, 1H), 1.93-1.81 (m, 1H), 1.78-1.50 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3): δ 205.3, 170.6, 135.1, 128.6, 128.5, 128.4, 100.2, 67.4, 47.3, 42.1, 36.9, 35.2, 29.9, 24.0, 23.6. ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{22}\text{O}_5\text{N}$ ($[\text{M}+\text{H}]^+$) 320.1492, found 320.1497. HPLC (Daicel Chiralpak AS, hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, $\lambda = 220$ nm): t_{R} (major enantiomer) = 23.3 min, t_{R} (minor enantiomer) = 28.3 min.

Compound 2l



Reaction of (*E*)-ethyl 2-methyl-4-oxopent-2-enoate with nitromethane was performed according to the general procedure but in toluene at 45 °C. Pale yellow oil, 18.7 mg, 43 %. ^1H NMR (400 MHz, CDCl_3): δ 4.90 (d, $J = 12.0$ Hz, 1H), 4.80 (d, $J = 12.0$ Hz, 1H), 4.25-4.15 (m, 2H), 3.04 (d, $J = 18.6$ Hz, 1H), 2.90 (d, $J = 18.6$ Hz, 1H), 2.17 (s, 3H), 1.36 (s, 3H), 1.26 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 205.6, 173.0, 79.2, 61.8, 47.3, 43.5, 30.3, 22.1, 13.9. ESI-HRMS: calcd for $\text{C}_9\text{H}_{16}\text{O}_5\text{N}$ ($[\text{M}+\text{H}]^+$) 218.1028, found 218.1004.

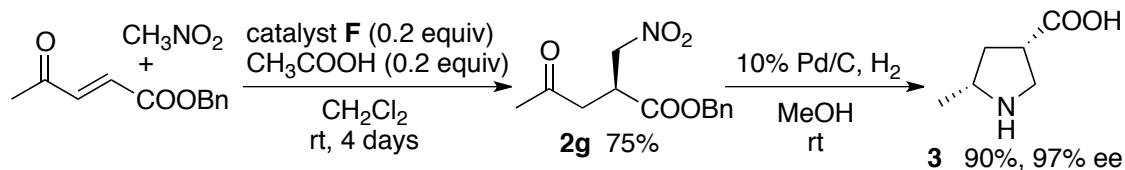
Compound 2m



Reaction of (*E*)-ethyl 4-oxobut-2-enoate with nitromethane was performed according to the general procedure to afford **2**. Pale yellow oil, 17.8 mg, 47%. ¹H NMR (400 MHz, CDCl₃): δ 9.78 (s, 1H), 4.77 (dd, *J* = 14.4 Hz, 6.1 Hz, 1H), 4.69 (dd, *J* = 14.4 Hz, 6.1 Hz, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 3.64-3.56 (m, 1H), 3.10 (dd, *J* = 19.1 Hz, 5.7 Hz, 1H), 2.88 (dd, *J* = 19.1 Hz, 5.7 Hz, 1H), 1.27 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 197.9, 170.1, 74.4, 62.1, 42.0, 37.0, 14.0. ESI-HRMS: calcd for C₇H₁₂O₅N ([M+H]⁺) 190.0710, found 190.0704.

5. Transformations of **2** to pyrrolidine-3-carboxylic acid and β²-amino acid derivatives

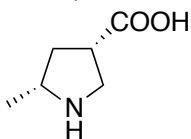
Synthesis of (3*S*,5*R*)-5-methylpyrrolidine-3-carboxylic acid (**3**) via **2g** (Scheme 3)



To a mixture of catalyst **F** (291.3 mg, 0.76 mmol) and acetic acid (40.0 μL, 0.70 mmol) in CH₂Cl₂ (3.0 mL), (*E*)-benzyl 4-oxopent-2-enoate (902.5 mg, 4.42 mmol) and nitromethane (1.0 mL, 18 mmol) were added at room temperature (24 °C) and the mixture (initially suspension) was stirred at the same temperature for 4 days (the reaction progress was monitored by TLC). The mixture was poured into 1 M HCl aqueous solution (15 mL) and extracted with CH₂Cl₂. Organic layers were combined, dried over Na₂SO₄, concentrated, and purified by flash column chromatography (hexane/ EtOAc = 4:1) to afford product **2g** (879.5 mg, 75 %).

A mixture of compound **2g** (346.5 mg, 1.31 mmol) and 10% Pd on charcoal (258.2 mg) in anhydrous MeOH (10 mL) was stirred under H₂ (balloon) at room temperature (24 °C) for 2 days. The mixture was filtered through celite and the filtrate was concentrated to remove the solvent to give **3** (151 mg, 90%, 97% ee).

(3*S*,5*R*)-5-Methylpyrrolidine-3-carboxylic acid (Compound **3**)

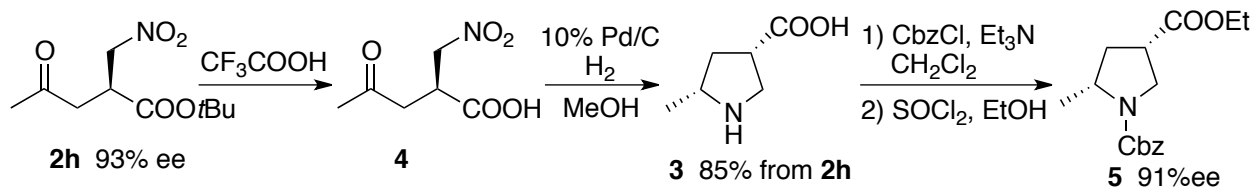


¹H NMR (400 MHz, CD₃OD): δ 3.68-3.58 (m, 1H), 3.54 (dd, *J* = 11.6 Hz, 7.2 Hz, 1H), 3.36 (dd, *J* = 11.6 Hz, 8.8 Hz, 1H), 3.11-3.02 (m, 1H), 2.47 (ddd, *J* = 13.4 Hz, 8.0 Hz, 6.8 Hz, 1H), 1.79 (ddd, *J* = 13.4 Hz, 10.0 Hz, 8.8 Hz, 1H), 1.41 (d, *J* = 6.4 Hz, 3H). ¹³C NMR (100 MHz, CD₃OD): δ 179.3, 57.7, 49.1, 46.4, 37.9, 17.5. HPLC (Daicel Chiralpak ZWIX (+), MeOH/MeCN/H₂O = 49/49/2, flow rate 0.5 mL/min, ELSD): *t*_R (major enantiomer) = 19.6 min, *t*_R (minor enantiomer)

= 16.5 min.

NMR chemical shifts of compound **3** were altered in the presence of acids (see below).

Transformations of **3a** to **4**

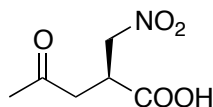


A mixture of compound **2h** (46.2 mg, 0.20 mmol, 93% ee) and trifluoroacetic acid (2 mL) was stirred for 1 hour at room temperature (24 °C). The mixture was concentrated under vacuum to afford compound **4**, which was directly used for the next step.

A mixture of compound **4**, 10% Pd on charcoal (21.8 mg) in anhydrous MeOH (5 mL) was stirred under H₂ (balloon) at room temperature (24 °C) for 2 days. The mixture was filtered through celite and the filtrate was concentrated under vacuum to afford compound **3** (21.9 mg, 85 % from **2h**).

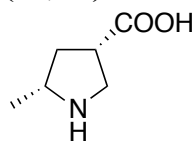
To a mixture of compound **3** (21.9 mg) and triethylamine (57.2 μL, 0.40 mmol) in CH₂Cl₂ (5 mL), benzyl chloroformate (42.3 μL, 0.30 mmol) was added dropwise over 30 min at room temperature (24 °C) and the mixture was stirred for 10 h at the same temperature. The mixture was diluted with CH₂Cl₂ and washed with saturated aqueous solution of NaHCO₃ (5 mL). The organic phase was dried over anhydrous Na₂SO₄, concentrated, and purified by flash column chromatography (hexane/EtOAc = 2:1) to afford the Cbz-protected product. This was dissolved in anhydrous EtOH (2 mL). To this solution, thionyl chloride (21.8 μL, 0.30 mmol) was added at room temperature (24 °C) and the mixture was stirred for 16 h at the same temperature. The mixture was concentrated and diluted with CH₂Cl₂. The mixture was washed with saturated aqueous NaHCO₃. The organic phase was dried over anhydrous Na₂SO₄, concentrated, and purified by flash column chromatography (hexane/EtOAc = 4:1) to afford compound **5**.

Compound **4**



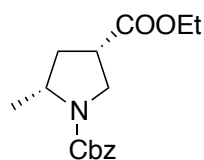
Pale yellow oil, 35 mg, 99%. ¹H NMR (400 MHz, CDCl₃): δ 4.77 (dd, *J* = 14.6 Hz, 5.8 Hz, 1H), 4.70 (dd, *J* = 14.6 Hz, 5.5 Hz, 1H), 3.66-3.56 (m, 1H), 3.06 (dd, *J* = 18.6 Hz, 4.6 Hz, 1H), 2.86 (dd, *J* = 18.6 Hz, 6.4 Hz, 1H), 2.22 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 205.1, 175.8, 74.2, 41.2, 37.8, 27.8. ESI-HRMS: calcd for C₆H₁₀O₅N ([M+H]⁺) 176.0553, found 176.0583.

(3*S*,5*R*)-5-Methylpyrrolidine-3-carboxylic acid (Compound 3) obtained from 2h



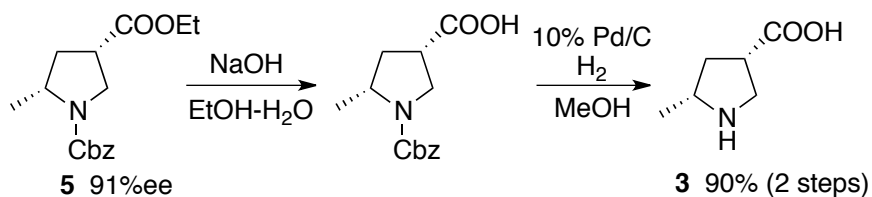
Colorless solid, 21.9 mg, 85% for two steps from **2h**. $[\alpha]_D^{20} +9.6$ (c 0.52, MeOH, 91% ee). Lit. $[\alpha]_D^{25} +10.3$ (c 0.58, MeOH).⁹ Compound **3** obtained from compound **2h** possibly included trace CF₃COOH. ¹H NMR (400 MHz, CD₃OD): δ 3.78-3.70 (m, 1H), 3.62 (dd, $J = 12.0$ Hz, 6.8 Hz, 1H), 3.50 (dd, $J = 12.0$ Hz, 9.2 Hz, 1H), 3.41-3.32 (m, 1H), 2.61-2.53 (m, 1H), 1.89-1.80 (m, 1H), 1.44 (d, $J = 6.4$ Hz, 3H). ¹³C NMR (100 MHz, CD₃OD): δ 182.8, 57.1, 51.2, 48.6, 40.1, 19.3.

Compound 5



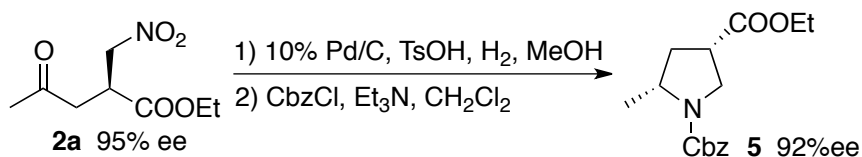
Colorless oil. ¹H NMR (400 MHz, CDCl₃): δ 7.49-7.28 (m, 5H), 5.24-5.03 (m, 2H), 4.19-4.13 (m, 2H), 4.04-3.77 (m, 2H), 3.64-3.52 (m, 1H), 3.03-2.88 (m, 1H), 2.49-2.33 (m, 2H), 2.02-1.77 (m, 1H), 1.43-1.14 (m, 6H). ¹³C NMR (100 MHz, CDCl₃): δ 172.8, 154.5, 136.8, 128.5, 127.94, 127.91, 66.7, 61.0, 53.7, 53.0, 48.6, 48.2, 42.2, 41.8, 37.2, 36.6, 21.3, 20.3, 14.1. ESI-HRMS: calcd for C₁₆H₂₂O₄N ([M+H]⁺) 292.1543, found 292.1544. HPLC (Daicel Chiralpak AS, hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, $\lambda = 254$ nm): t_R (major enantiomer) = 25.3 min, t_R (minor enantiomer) = 24.3 min.

Transformation of 5 to 3



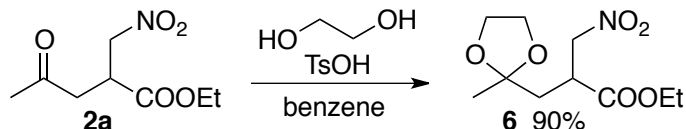
To a solution of compound **5** (91% ee, 29.1 mg, 0.10 mmol) in EtOH (1.0 mL)-H₂O (1.0 mL), 1 M NaOH aqueous solution (0.15 mL) was added at room temperature and the mixture was stirred for 16 h. The mixture was poured into ice-1 M HCl (10 mL), and the mixture was extracted with CH₂Cl₂. The organic phase was dried over anhydrous Na₂SO₄, concentrated under vacuum. The residue was dissolved in MeOH (5 mL), and 10% Pd on charcoal (39.7 mg) was added. The mixture was stirred under H₂ (balloon) at room temperature (24 °C) for 2 days. The mixture was filtered through celite and the filtrate was concentrated under vacuum to afford compound **3** (11.6 mg, 90% yield for two steps from **5**).

Transformation of **2a** to **5**



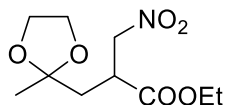
A mixture of compound **2a** (95% ee, 203 mg, 1.0 mmol) was dissolved in anhydrous MeOH (10 mL). To this solution, *p*-toluenesulfonic acid (187 mg, 1.0 mmol) and 10% Pd on charcoal (173 mg) were added and the mixture was stirred under H₂ (balloon) at room temperature (24 °C) for 2 days. The mixture was filtered through celite and the filtrate was concentrated under vacuum. The residue was dissolved in CH₂Cl₂ (20 mL), and triethylamine (530 μL, 3.8 mmol) was added. To the mixture, benzyl chloroformate (270 μL, 1.92 mmol) was added dropwise over 30 min at room temperature (24 °C). The mixture was stirred at the same temperature for 10 h. The mixture was diluted with CH₂Cl₂ and washed with saturated aqueous NaHCO₃ solution. The organic phase was dried over anhydrous Na₂SO₄, concentrated, and purified by flash column chromatography (hexane/EtOAc = 2:1) to give **5** (92% ee).

Transformation of **2a** to **6**



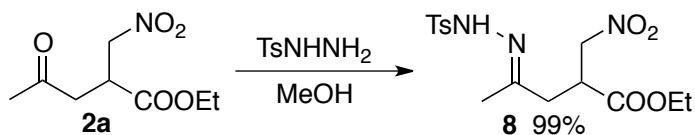
A mixture of **2a** (81.2 mg, 0.40 mmol), ethylene glycol (2 mL), and *p*-toluenesulfonic acid monohydrate (15.0 mg, 0.08 mmol) in benzene (10 mL) was heated at reflux with a Dean-Stark apparatus for 24 h. After being cooled to room temperature, the mixture was diluted with EtOAc, washed with saturated aqueous NaHCO₃. The organic phase was dried over MgSO₄, filtered, concentrated, and purified by flash column chromatography (hexane/EtOAc = 4:1) to give **6** (89.0 mg, 90%).

Compound **6**



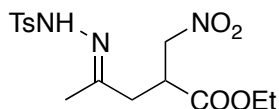
Colorless oil. ¹H NMR (400 MHz, CDCl₃): δ 4.74 (dd, *J* = 14.4 Hz, 8.8 Hz, 1H), 4.66 (dd, *J* = 14.4 Hz, 4.6 Hz, 1H), 4.26-4.12 (m, 2H), 4.05-3.88 (m, 4H), 3.40-3.30 (m, 1H), 2.24 (dd, *J* = 14.8 Hz, 5.3 Hz, 1H), 1.94 (dd, *J* = 14.8 Hz, 7.4 Hz, 1H), 1.33 (s, 3H), 1.27 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 172.2, 108.6, 75.4, 64.6, 64.4, 61.5, 38.9, 37.4, 24.0, 14.0. ESI-HRMS: calcd for C₁₀H₁₈O₆N ([M+H]⁺) 248.1129, found 248.1099.

Transformation of 2a to 8



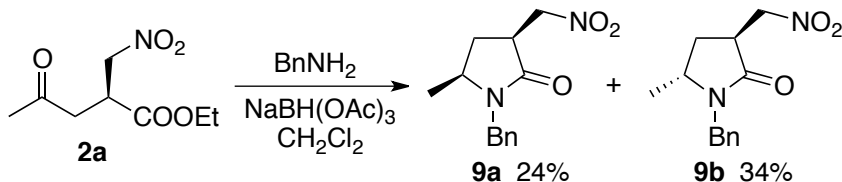
A solution of **2a** (46.7 mg, 0.23 mmol) and *p*-toluenesulfonyl hydrazide (42.8 mg, 0.23 mmol) in MeOH (2.3 mL) was refluxed for 2 h. After being cooled to room temperature, generated precipitate was collected by filtration and washed with hexane/EtOAc (10:1) to give **7** (99%, 85.4 mg, 99%).

Compound 8



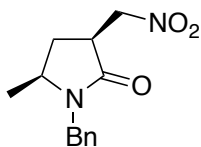
Colorless solid. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.80 (d, $J = 8.1$ Hz, 2H), 7.33 (d, $J = 8.1$ Hz, 2H), 4.52 (dd, $J = 14.6$ Hz, 7.8 Hz, 1H), 4.32 (dd, $J = 14.6$ Hz, 4.6 Hz, 1H), 4.23-4.05 (m, 2H), 3.53-3.43 (m, 1H), 2.71 (dd, $J = 17.7$ Hz, 4.4 Hz, 1H), 2.52 (dd, $J = 17.7$ Hz, 8.4 Hz, 1H), 2.44 (s, 3H), 1.81 (s, 3H), 1.22 (t, $J = 7.2$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 171.0, 153.2, 144.6, 134.9, 129.7, 128.1, 73.9, 61.7, 39.1, 36.4, 21.6, 16.5, 13.9. ESI-HRMS: calcd for $\text{C}_{15}\text{H}_{22}\text{O}_6\text{N}_3\text{S}$ ($[\text{M}+\text{H}]^+$) 372.1224, found 372.1230.

Transformation of 2a to 9



To a solution of **2a** (40.6 mg, 0.20 mmol) in CH_2Cl_2 (1.5 mL), benzylamine (43.7 μL , 0.40 mmol) and $\text{NaBH}(\text{OAc})_3$ (85.4 mg, 0.40 mmol) were added at room temperature (24 $^\circ\text{C}$) and the mixture was stirred for 48 h at the same temperature. To the mixture, aqueous 1 N NaOH (1.5 mL) was added and the mixture was extracted with CH_2Cl_2 . Organic layers were combined, washed with brine, dried over Na_2SO_4 , filtered, concentrated, and purified by flash column chromatography (hexane/EtOAc = 4:1) to give **9a** (11.9 mg, 24%) and **9b** (16.9 mg, 34%). Relative stereochemistry was determined by NOESY experiments.

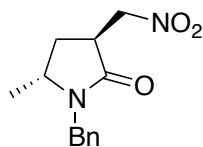
Compound 9a



Rf 0.40 (hexane/EtOAc = 3:1), colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.37-7.18 (m, 5H), 4.96 (d, $J = 15.0$ Hz, 1H), 4.93 (dd, $J = 13.8$ Hz, 3.8 Hz, 1H), 4.50 (dd, $J = 13.8$ Hz, 9.2 Hz, 1H),

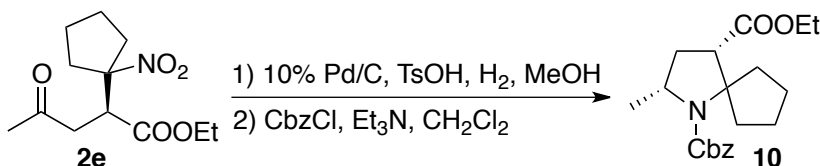
4.08 (d, $J = 15.0$ Hz, 1H), 3.59-3.48 (m, 1H), 3.30-3.19 (m, 1H), 2.53 (ddd, $J = 12.8$ Hz, 8.8 Hz, 6.6 Hz, 1H), 1.44 (ddd, $J = 12.8$ Hz, 10.8 Hz, 8.9 Hz, 1H), 1.22 (d, $J = 6.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 171.8, 136.1, 128.8, 127.9, 127.7, 76.3, 51.1, 44.3, 40.8, 32.8, 19.9. ESI-HRMS: calcd for $\text{C}_{13}\text{H}_{17}\text{O}_3\text{N}_2$ ($[\text{M}+\text{H}]^+$) 249.1234, found 249.1227.

Compound 9b



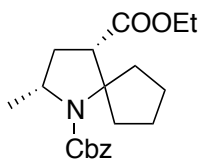
Rf 0.35 (hexane/EtOAc = 3:1), colorless oil. ^1H NMR (400 MHz, CDCl_3): δ 7.41-7.19 (m, 5H), 4.99 (d, $J = 14.9$ Hz, 1H), 4.86 (dd, $J = 13.8$ Hz, 3.8 Hz, 1H), 4.53 (dd, $J = 13.8$ Hz, 8.8 Hz, 1H), 3.99 (d, $J = 14.9$ Hz, 1H), 3.62-3.50 (m, 1H), 3.41-3.29 (m, 1H), 2.02 (dd, $J = 8.8$ Hz, 5.8 Hz, 2H), 1.19 (d, $J = 6.4$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 171.0, 136.0, 128.8, 128.0, 127.8, 75.9, 50.8, 44.5, 39.3, 31.2, 19.0. ESI-HRMS: calcd for $\text{C}_{13}\text{H}_{17}\text{O}_3\text{N}_2$ ($[\text{M}+\text{H}]^+$) 249.1234, found 249.1227.

Transformation 3e to 10



A mixture of compound **2a** (95% ee, 115.0 mg, 0.57 mmol) was dissolved in anhydrous MeOH (10 mL). To this solution, *p*-toluenesulfonic acid (91.4 mg, 0.53 mmol) and 10% Pd on charcoal (62.0 mg) were added and the mixture was stirred under H_2 (balloon) at room temperature (24 °C) for 2 days. The mixture was filtered through celite and the filtrate was concentrated under vacuum. The residue was dissolved in CH_2Cl_2 (20 mL), and triethylamine (226 μL , 1.58 mmol) was added. To the mixture, benzyl chloroformate (270 μL , 1.92 mmol) was added dropwise over 30 min at room temperature (24 °C). The mixture was stirred at the same temperature for 10 h. The mixture was diluted with CH_2Cl_2 and washed with saturated aqueous NaHCO_3 solution. The organic phase was dried over anhydrous Na_2SO_4 , concentrated, and purified by flash column chromatography (hexane/EtOAc = 2:1) to give **10**.

Compound 10

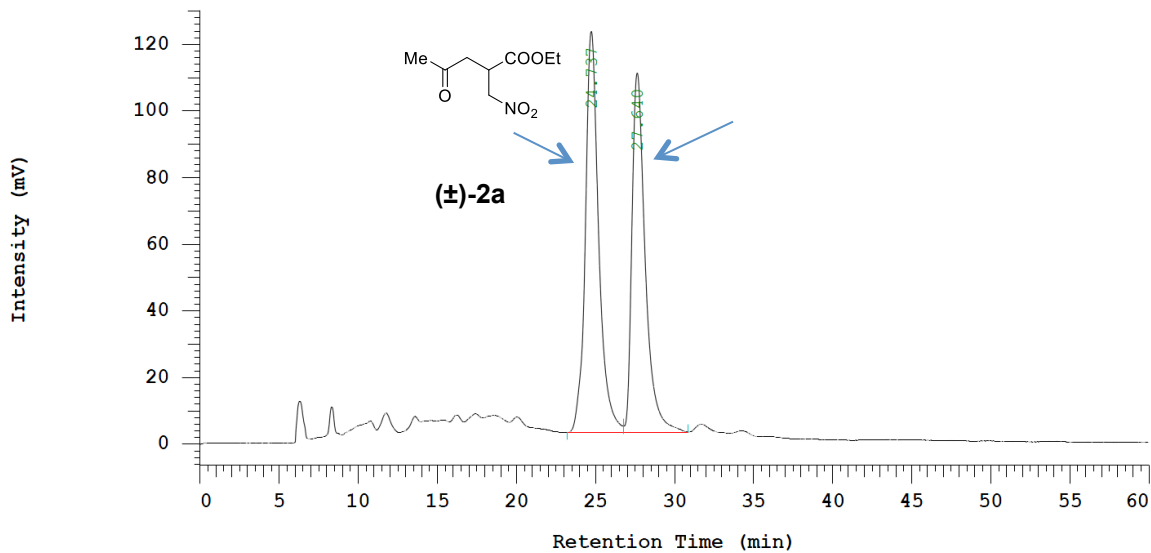


Colorless oil. ^1H NMR (400 MHz, CDCl_3): δ 7.45-7.28 (m, 5H), 5.23-5.05 (m, 2H), 4.26-4.06 (m, 2H), 3.93-3.82 (m, 1H), 2.86-2.71 (m, 1H), 2.27-2.12 (m, 1H), 2.03-1.10 (m, 12H), 1.27 (t, $J =$

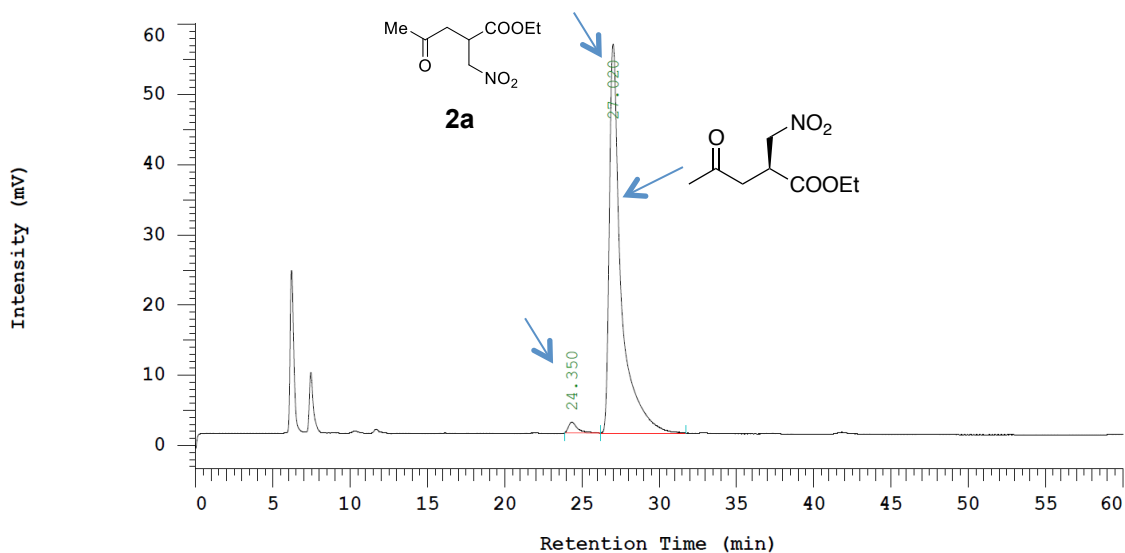
7.2 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 171.7, 153.8, 136.7, 128.4, 128.1, 128.0, 73.4, 66.4, 60.7, 54.2, 53.3, 35.9, 35.4, 34.4, 29.7, 26.9, 25.5, 24.0, 22.6, 14.1. ESI-HRMS: calcd for $\text{C}_{20}\text{H}_{28}\text{O}_4\text{N}$ ($[\text{M}+\text{H}]^+$) 346.2013, found 346.2016.

6. References

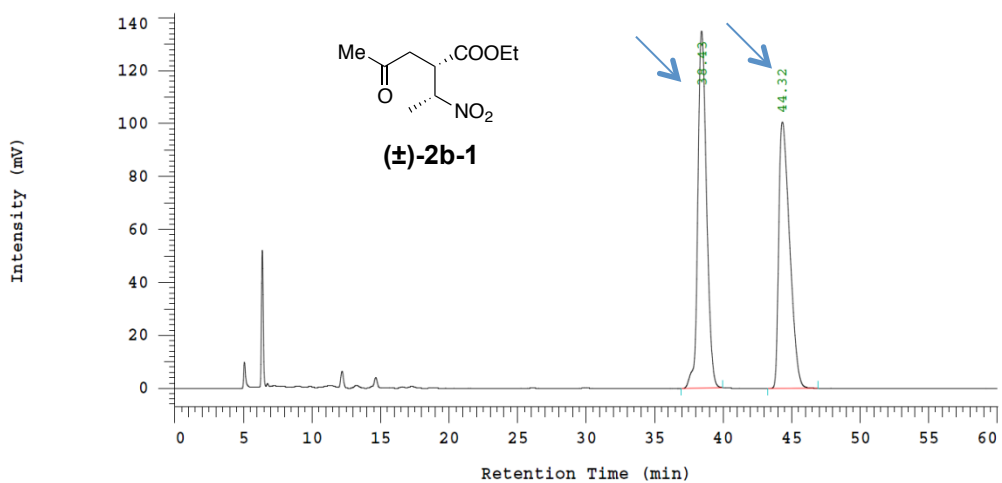
- (1) H.-L. Cui, F. Tanaka, *Chem. Eur. J.* **2013**, *19*, 6213.
- (2) X. Zhao, K. E. Ruhl, T. Rovis, *Angew. Chem. Int. Ed.* **2012**, *51*, 12330.
- (3) A. Takahashi, H. Yanai, M. Zhang, T. Sonoda, M. Mishima, T. Taguchi, *J. Org. Chem.*, **2010**, *75*, 1259.
- (4) J. Wang, A. Ma, D. Ma, *Org. Lett.*, **2008**, *10*, 5425.
- (5) M. Ronsheim, C. Zercher, *J. Org. Chem.*, **2003**, *68*, 4535.
- (6) W. Baratta, A. Zotto, *Chem. Comm.* **1997**, *22*, 2163.
- (7) D. Lyzwa, K. Dudzinski, P. Kwiatkowski, *Org. Lett.*, **2012**, *14*, 1540.
- (8) K. Dudzinski, A. Pakulska, P. Kwiatkowski, *Org. Lett.*, **2012**, *14*, 4222.
- (9) H. Zhang, M. Mifsud, F. Tanaka, C. F. Barbas, III, *J. Am. Chem. Soc.* **2006**, *128*, 9630.



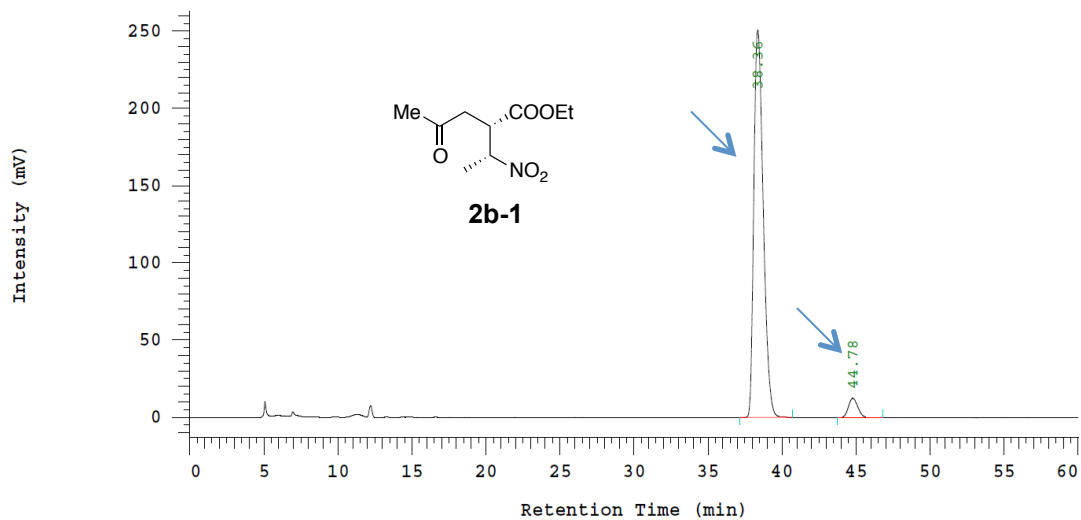
No.	RT	Area	Conc 1
1	24.737	6973274	52.940
2	27.640	6198704	47.060
		13171978	100.000



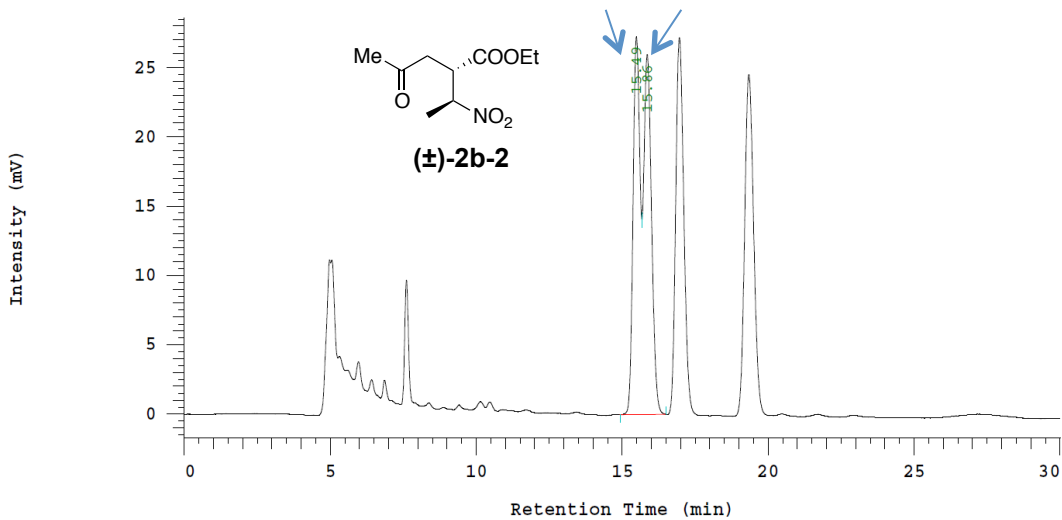
No.	RT	Area	Conc 1
1	24.350	62894	2.136
2	27.020	2880925	97.864
		2943819	100.000



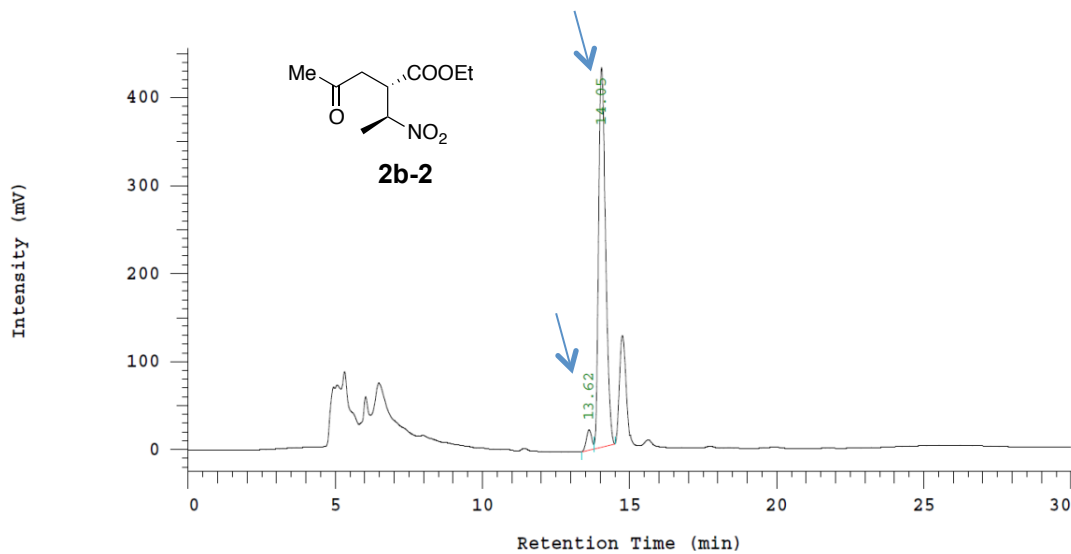
No.	RT	Area	Area %
1	38.43	5838327	51.722
2	44.32	5449663	48.278
		11287990	100.000



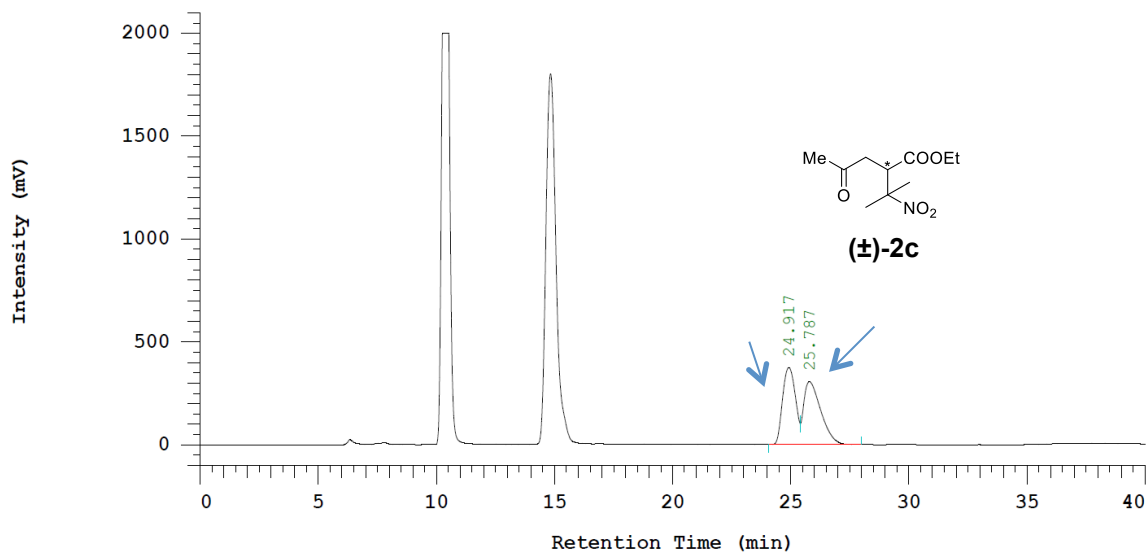
No.	RT	Area	Area %
1	38.36	10875166	95.192
2	44.78	549296	4.808
		11424462	100.000



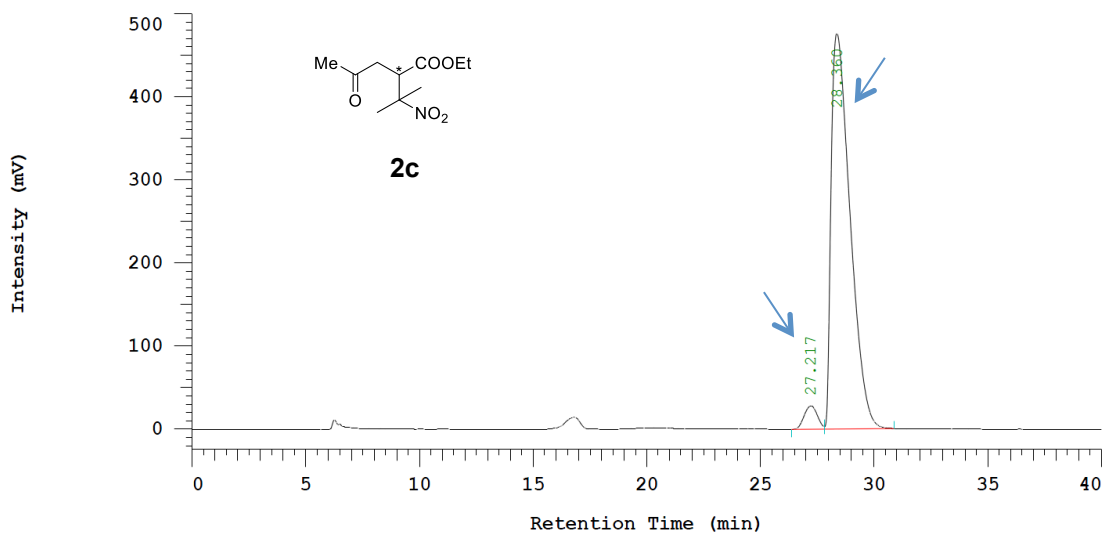
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2	15.86	489562	52.351
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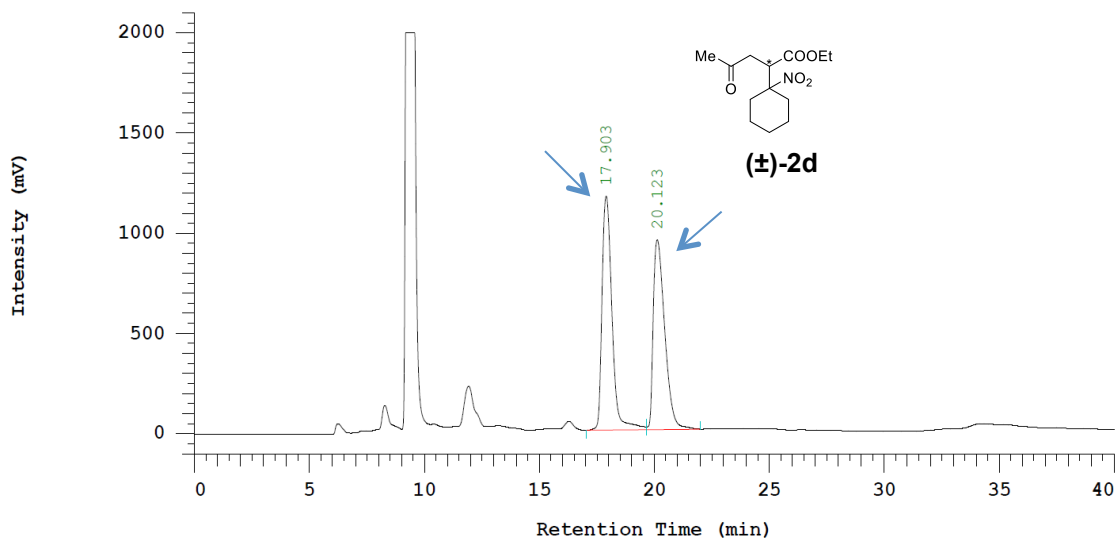
No.	RT	Area	Area %
1	13.62	281303	3.764
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		7473438	100.000



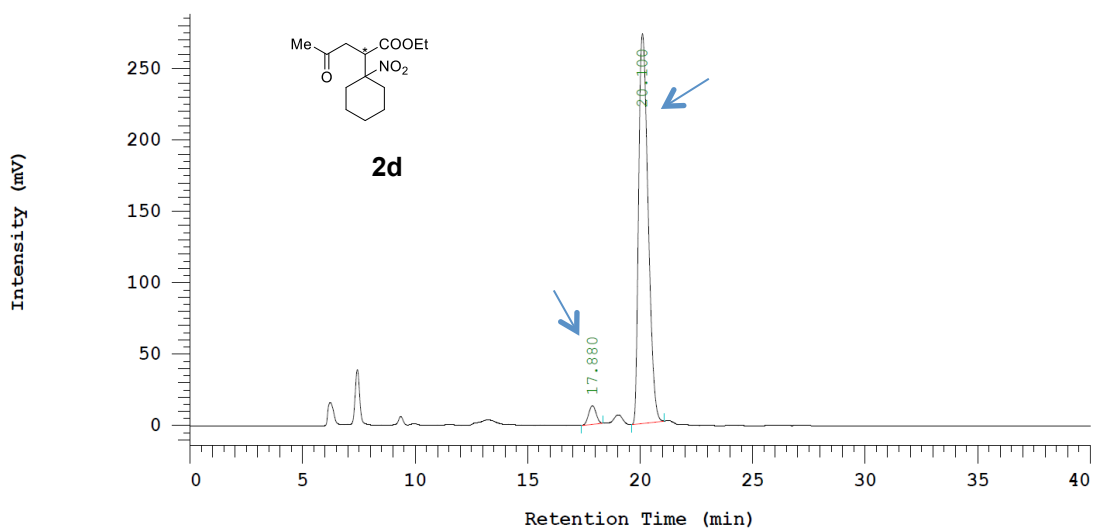
No.	RT	Area	Area %
1	24.917	13988504	47.609
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		29382119	100.000



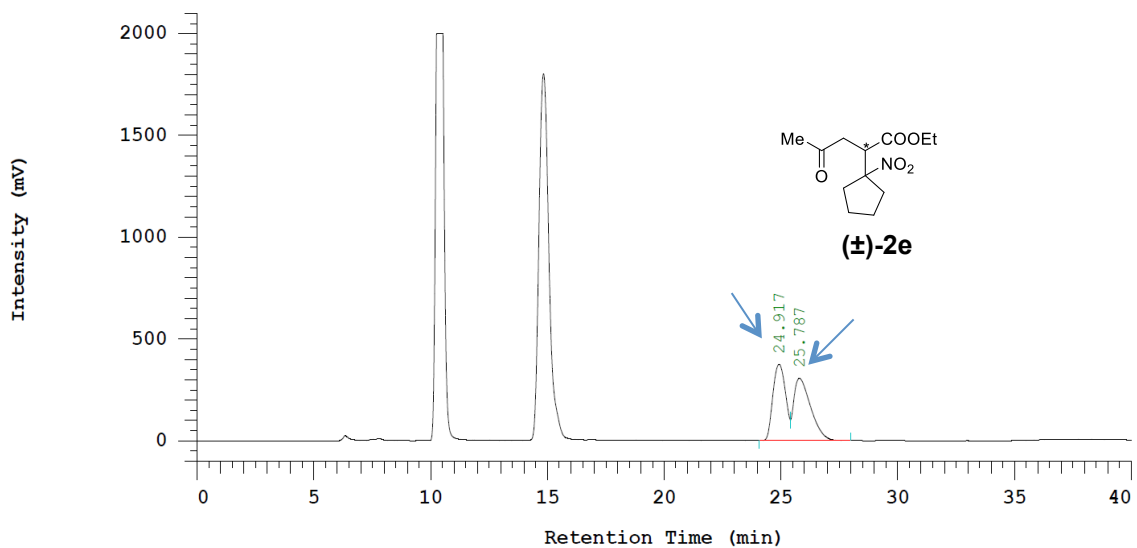
No.	RT	Area	Area %
1	27.217	1121048	3.945
2	28.360	27293166	96.055
		28414214	100.000



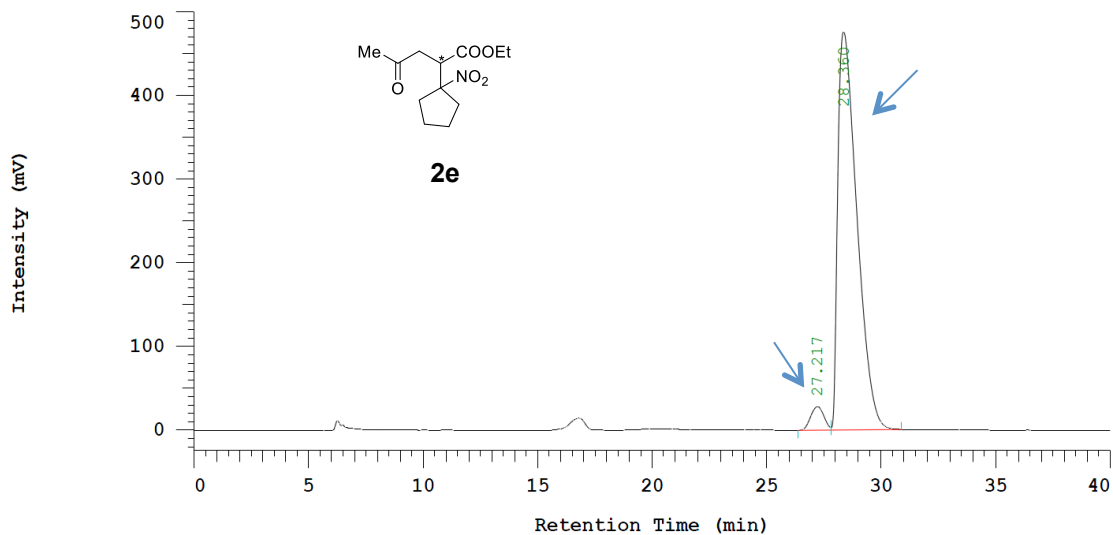
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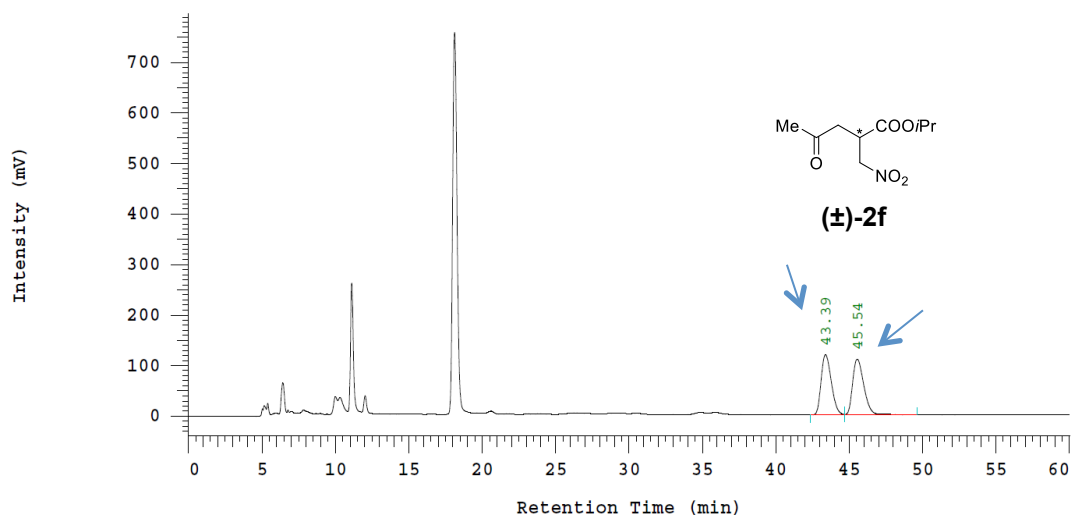
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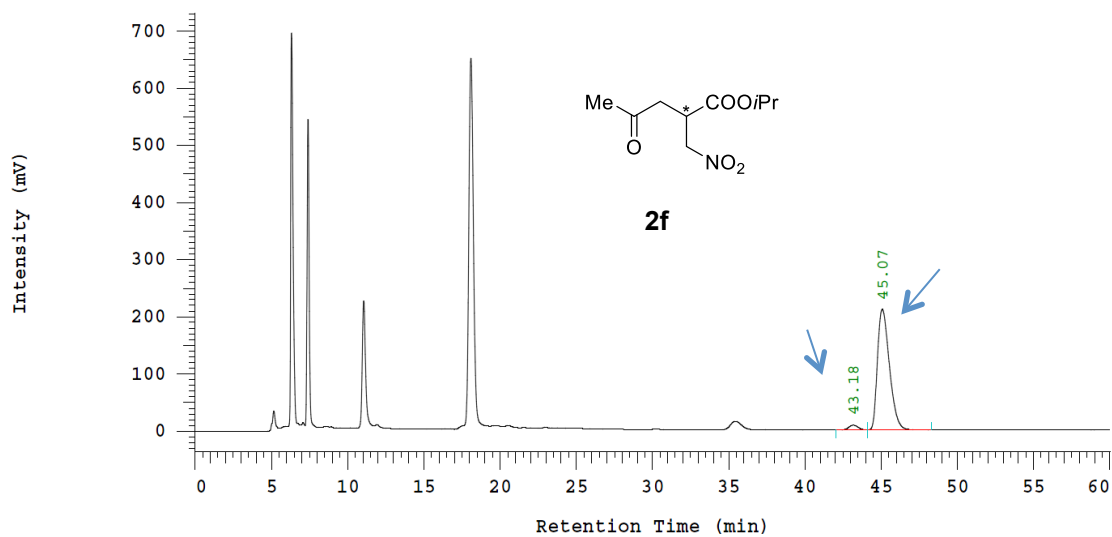
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1	24.917	13988504	47.609
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		29382119	100.000



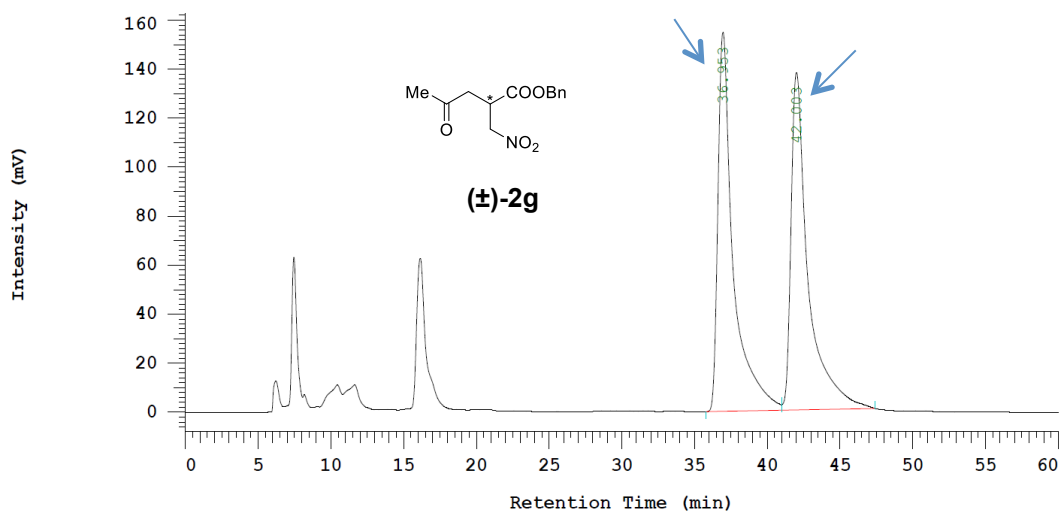
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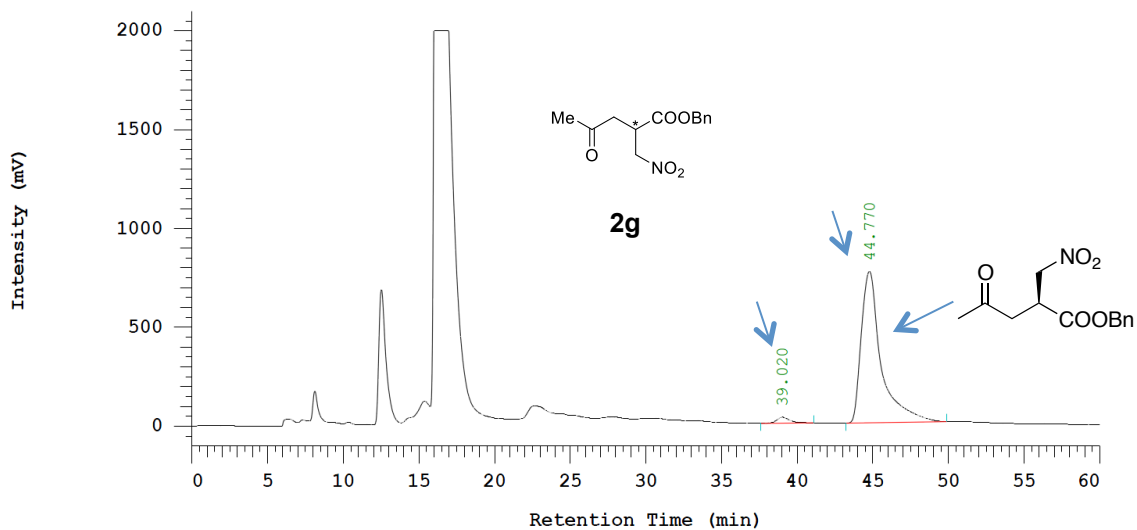
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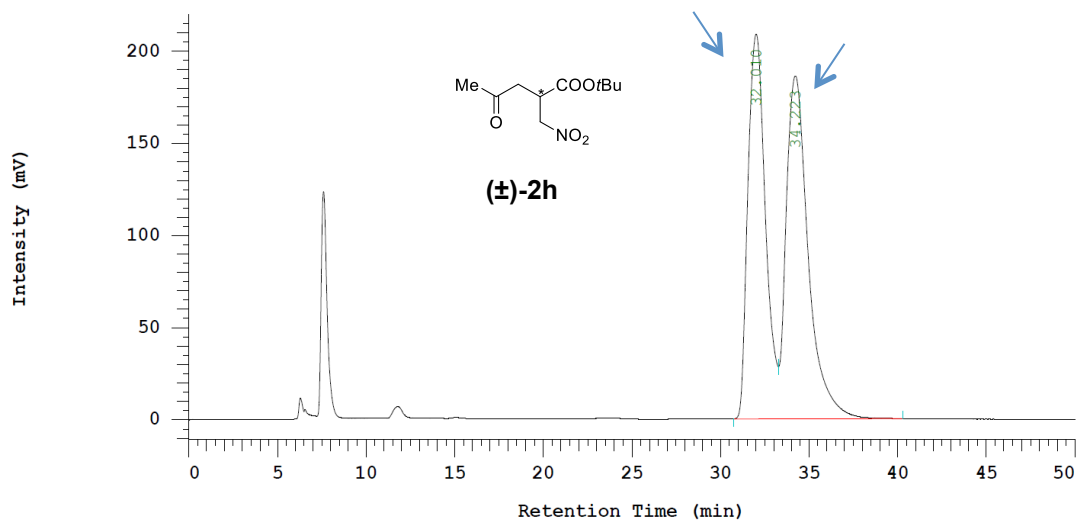
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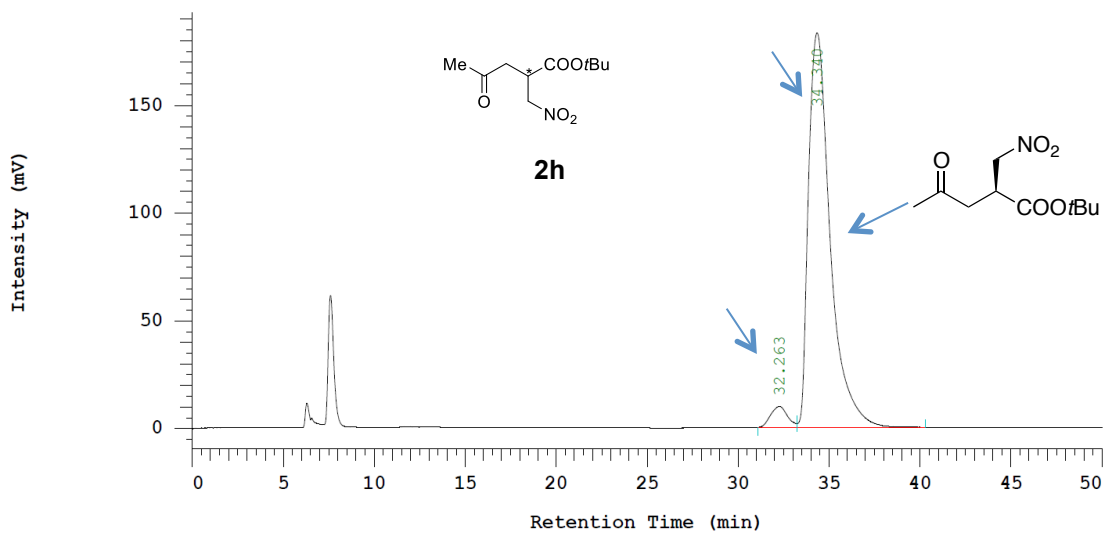
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1	36.953	11153224	50.075
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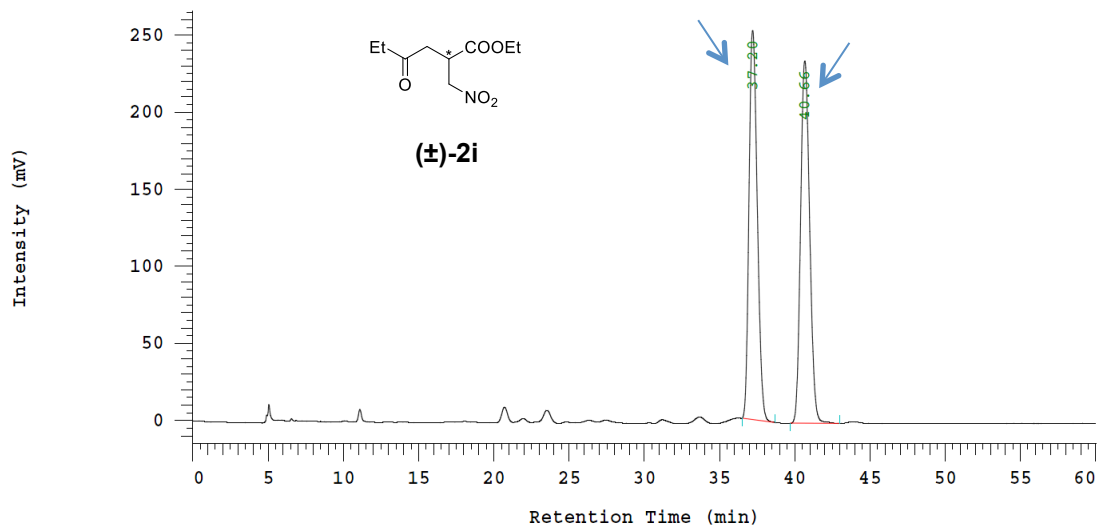
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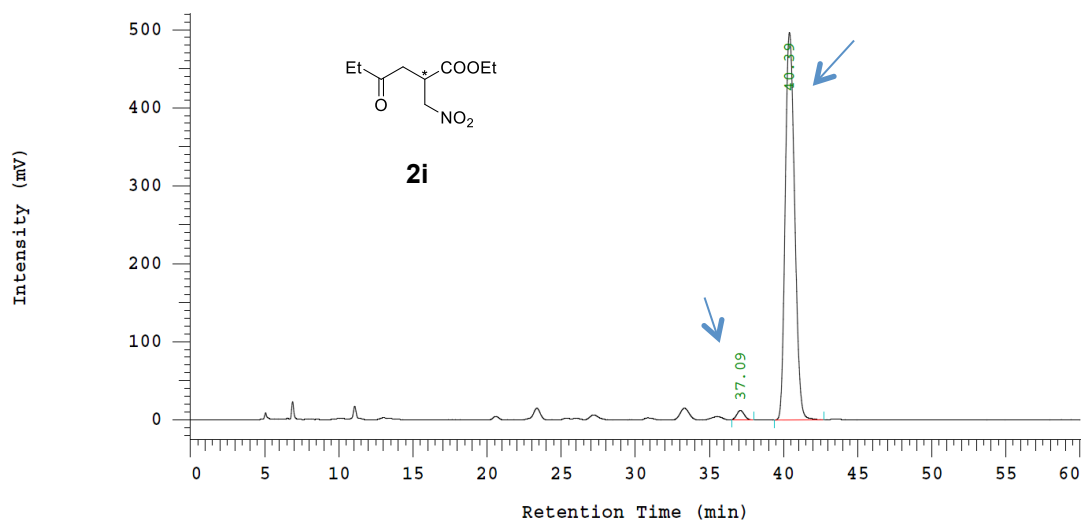
No.	RT	Area	Conc 1
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			100.000



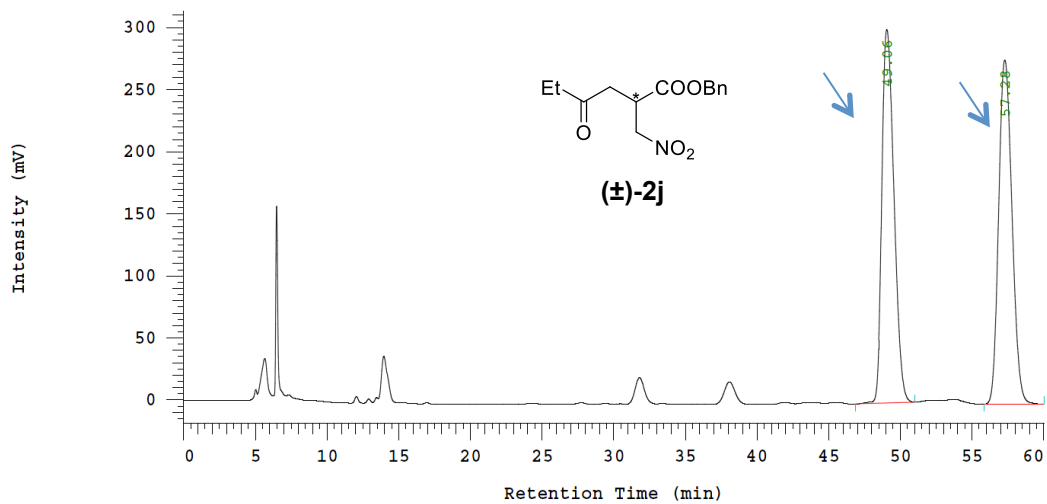
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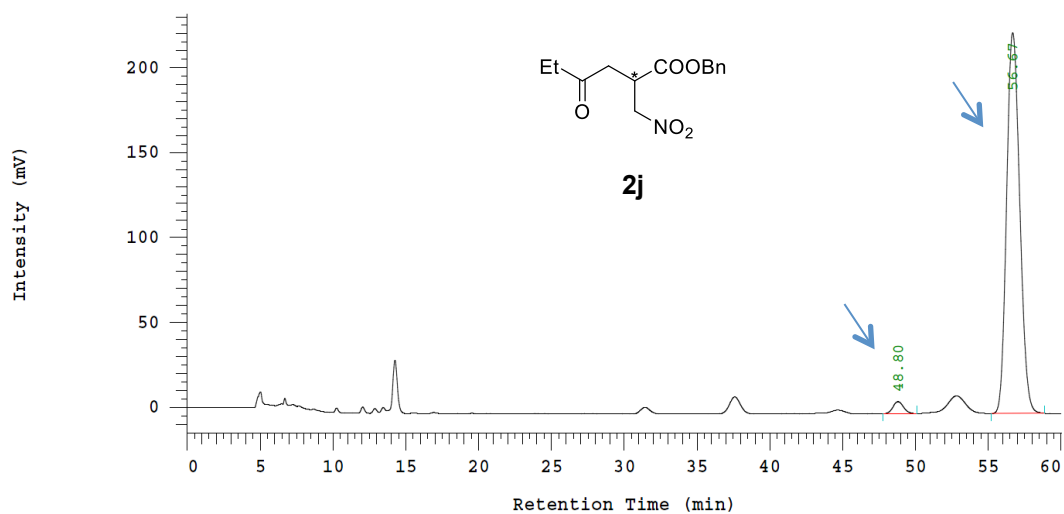
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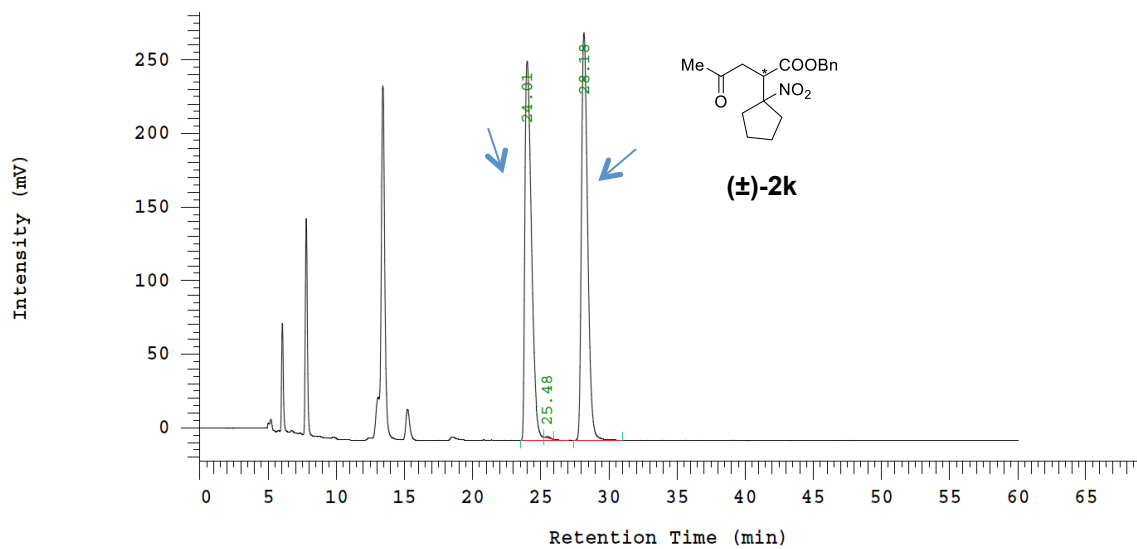
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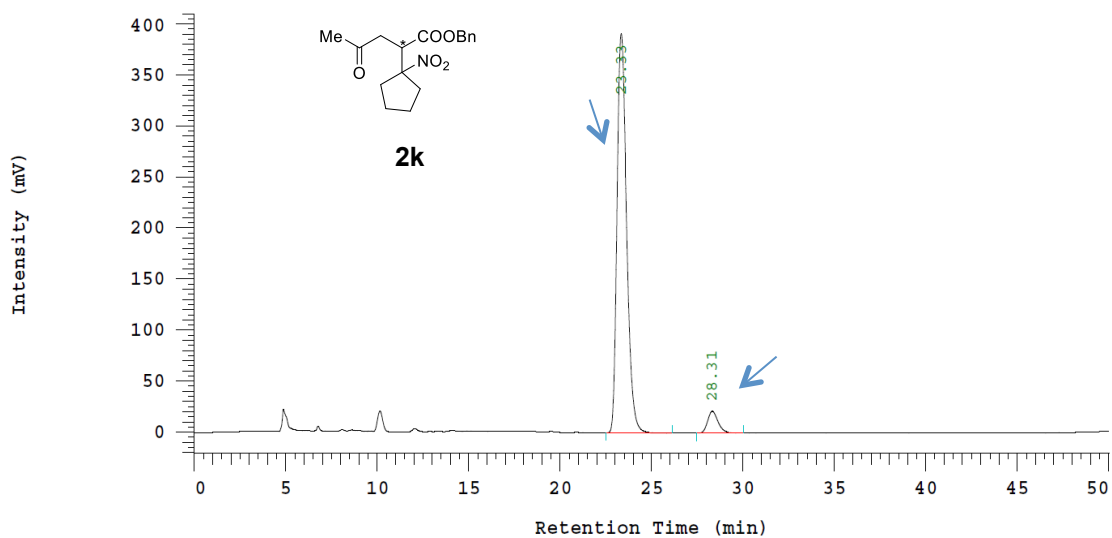
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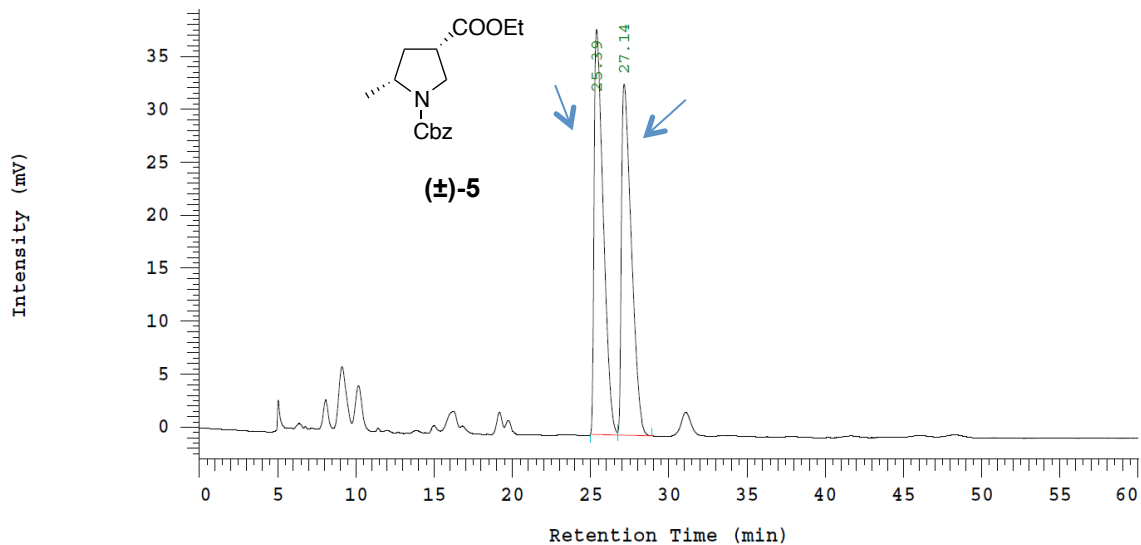
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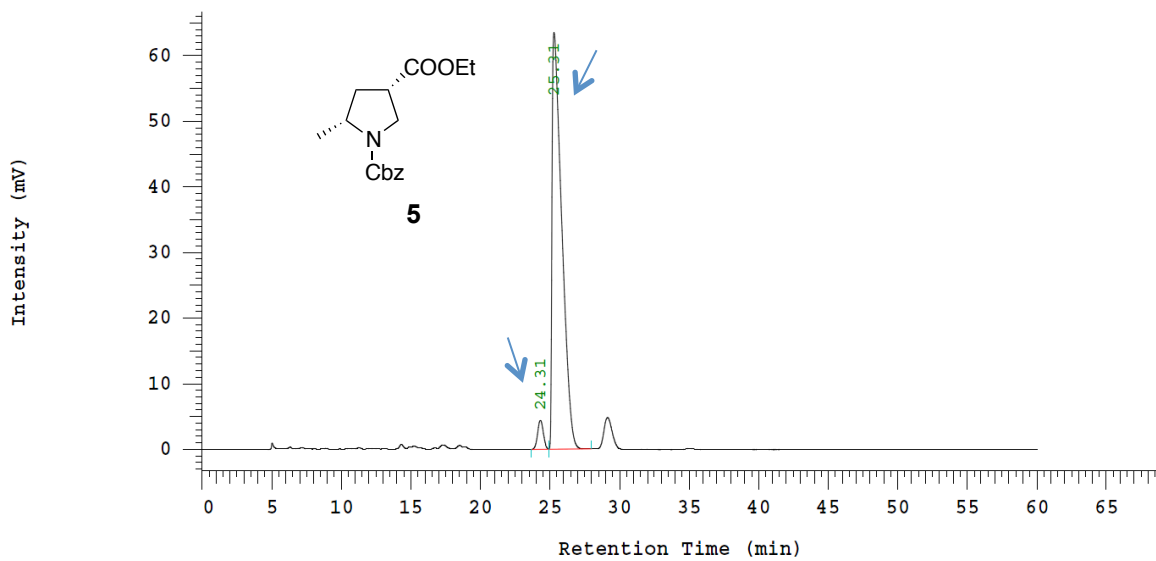
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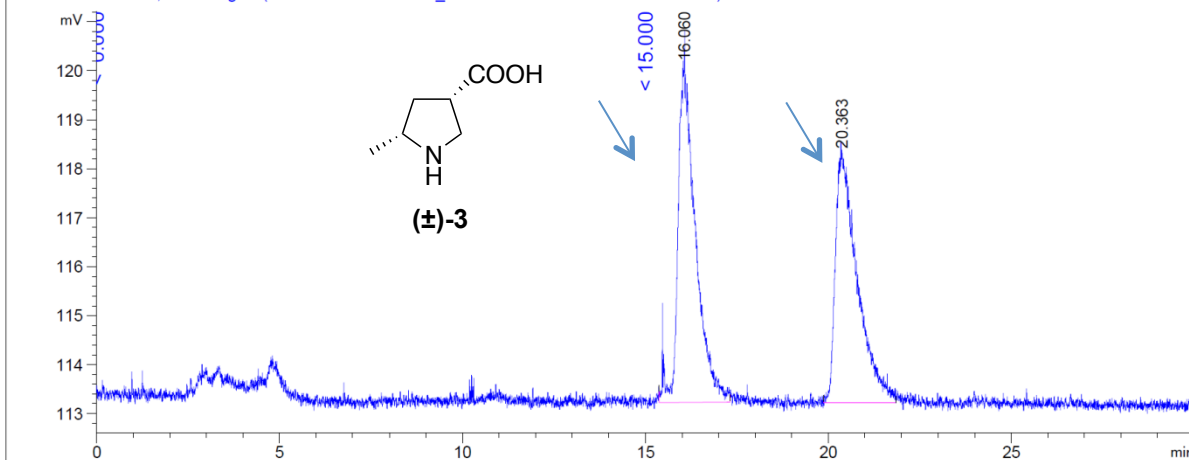
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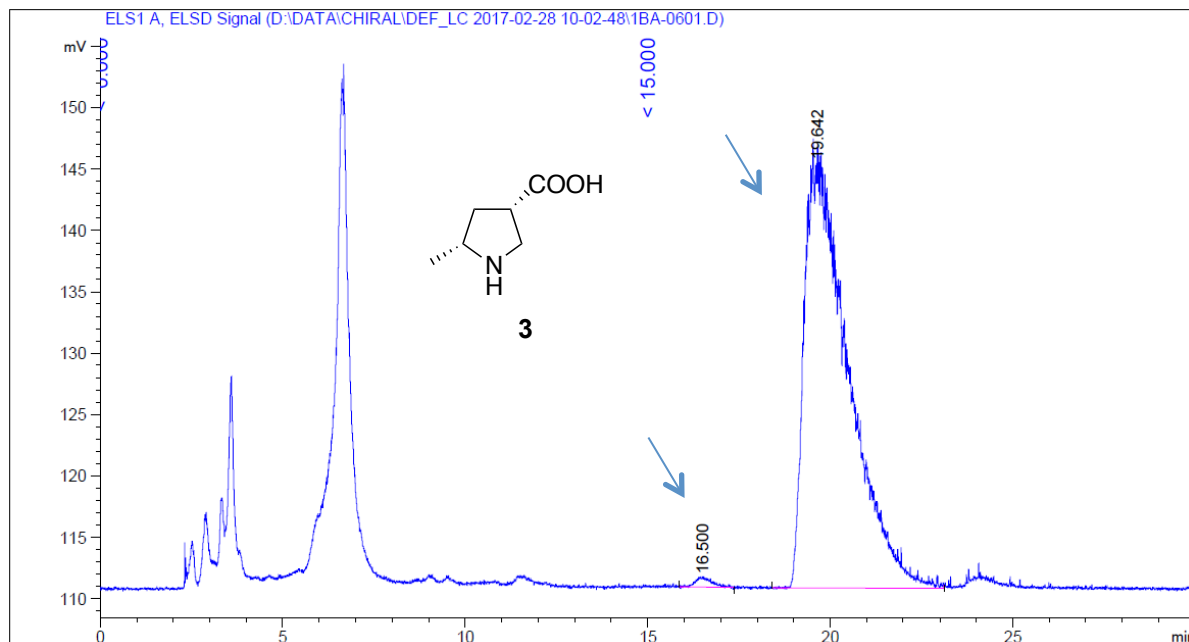
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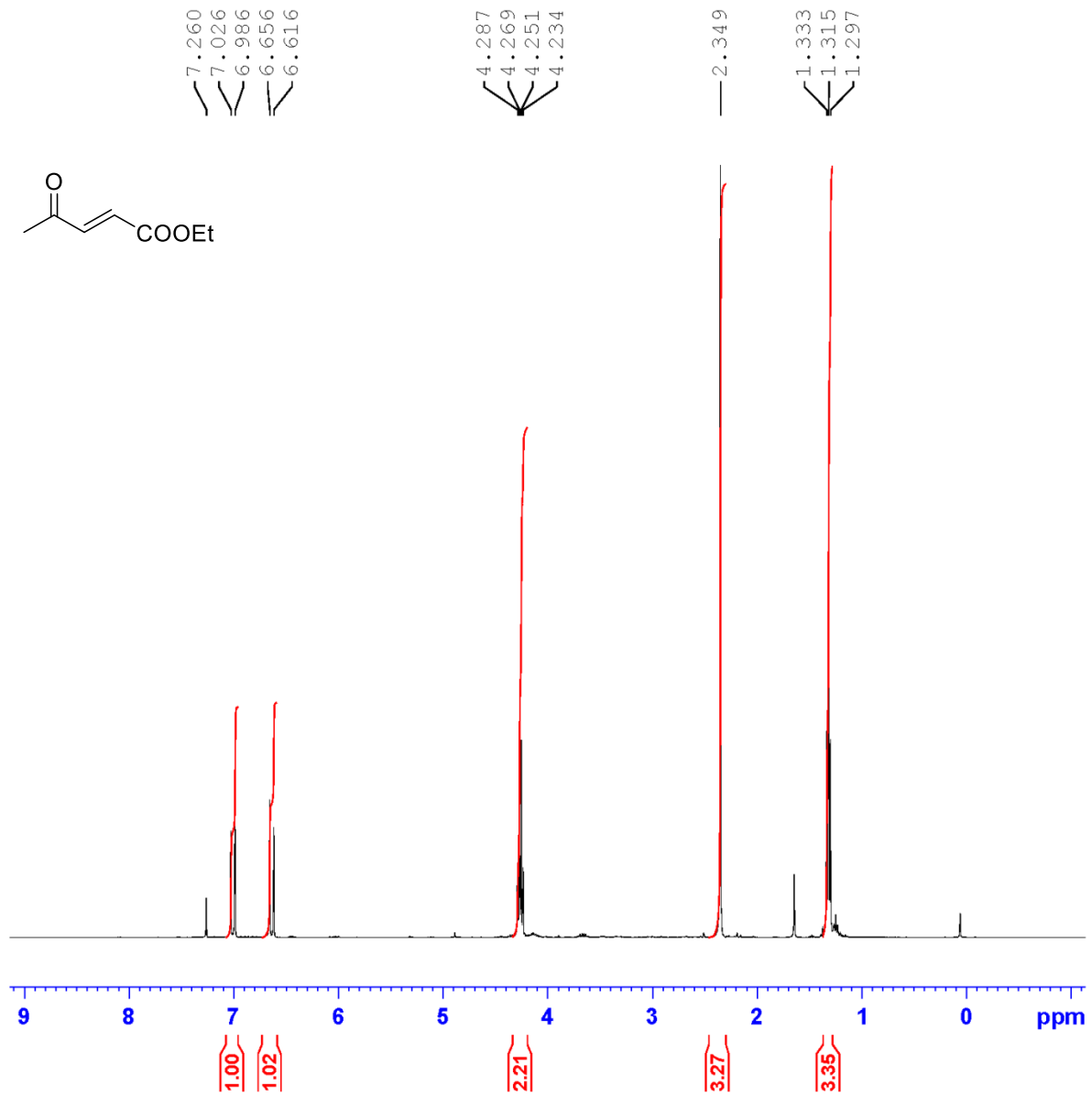
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1	16.060	BB	0.4718	233.04874	6.93982	52.3282
2	20.363	BB	0.6059	212.31113	5.09763	47.6718



Peak #	RetTime [min]	Type	Width [min]	Area [mV*s]	Height [mV]	Area %
1	16.500	BB	0.5274	25.88804	7.56224e-1	0.8698
2	19.642	BB	1.2513	2950.44043	34.58559	99.1302



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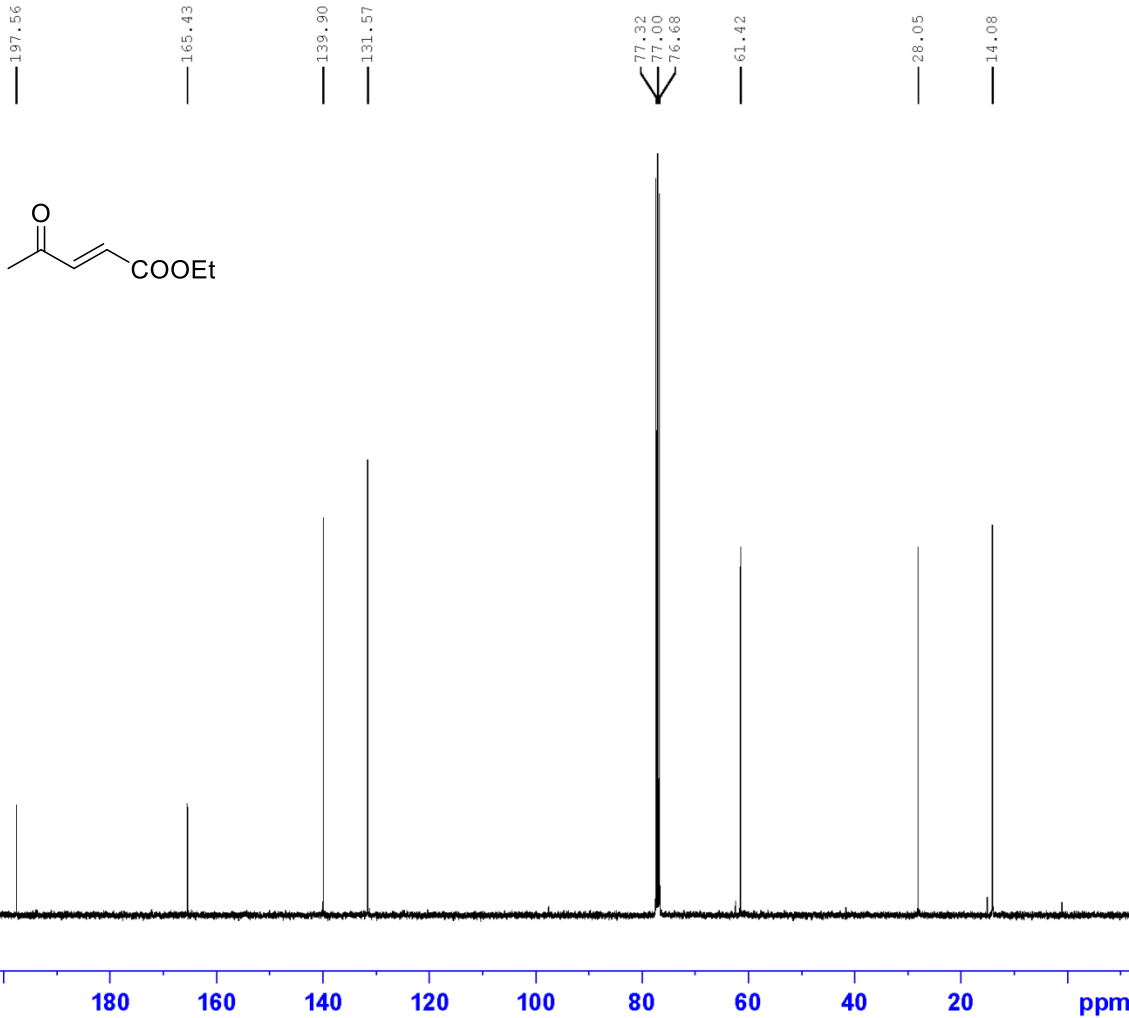
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EXPNO     10
PROCNO    1

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Time      13.58
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894465 sec
RG         49.09
DW         62.400 usec
DE         6.50 usec
TE         298.7 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      400.1324710 MHz
NUC1       1H
P1         15.00 usec
PLW1       8.00000000 W

F2 - Processing parameters
SI         65536
SF         400.1300090 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

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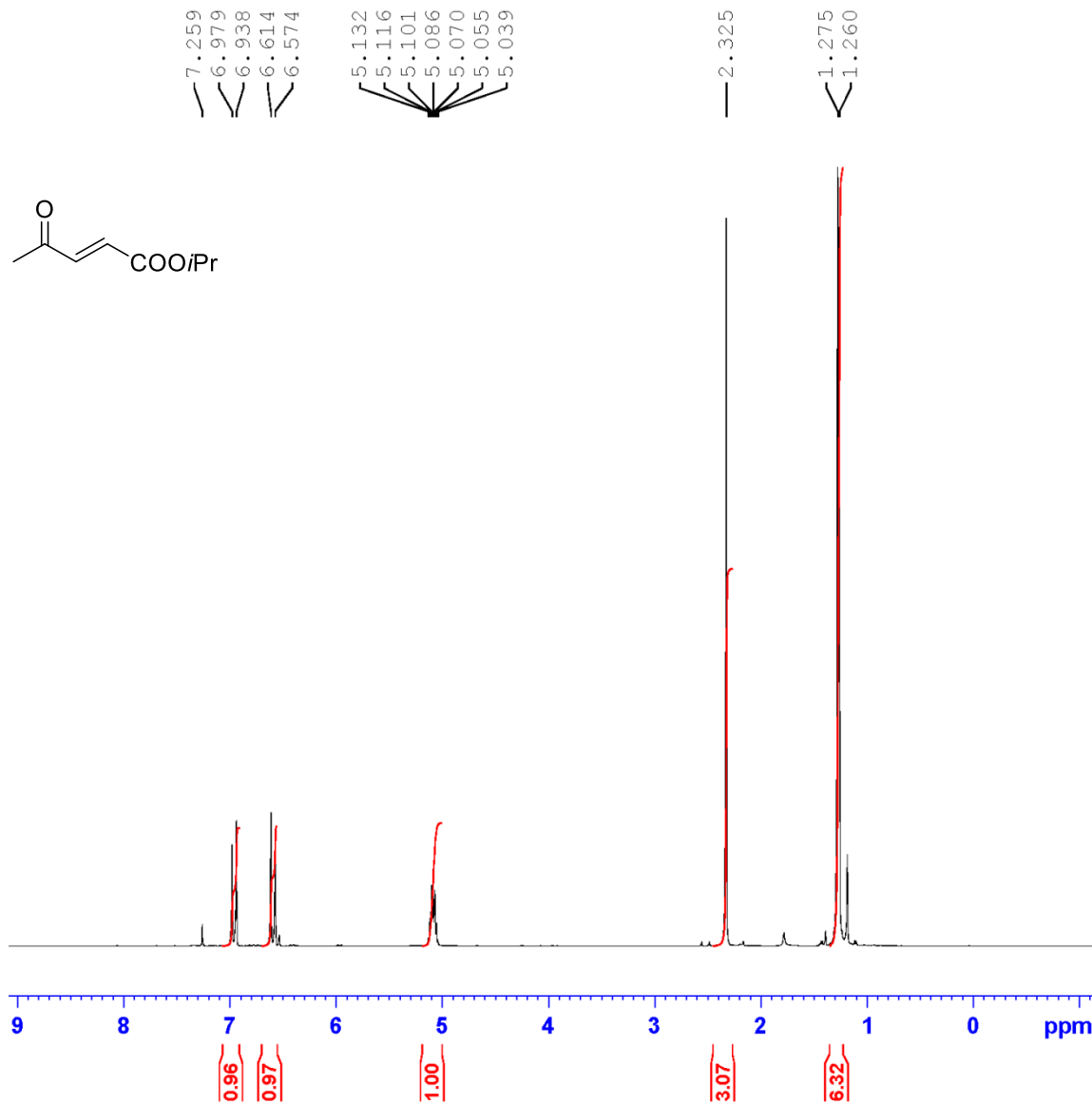
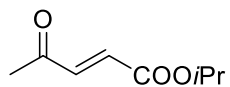
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 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160816
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 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.9 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.0000000 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 8.0000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
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 SSB 0
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 GB 0
 PC 1.40

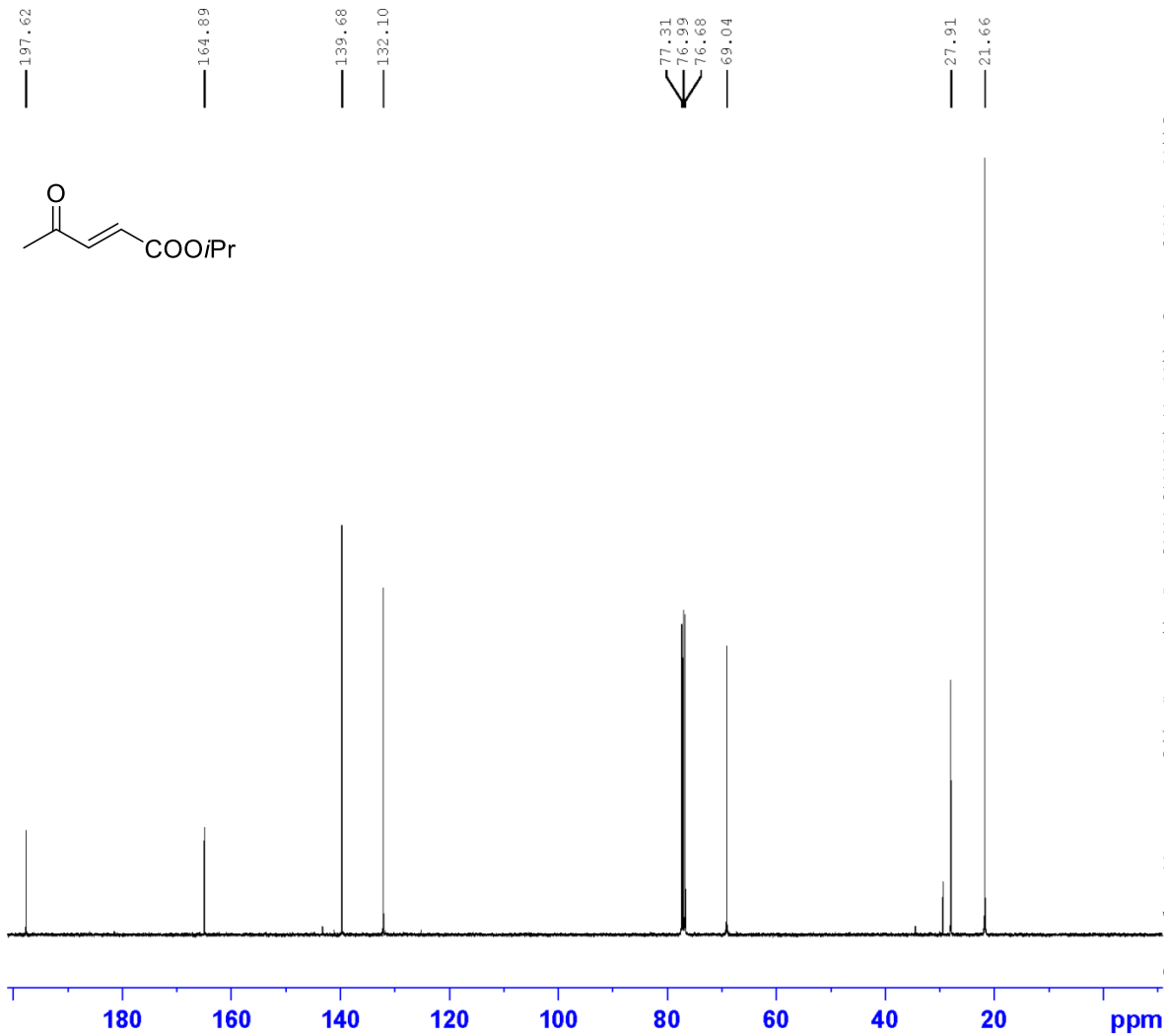


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 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
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 INSTRUM spect
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 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 31.13
 DW 62.400 usec
 DE 6.50 usec
 TE 298.7 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
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 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



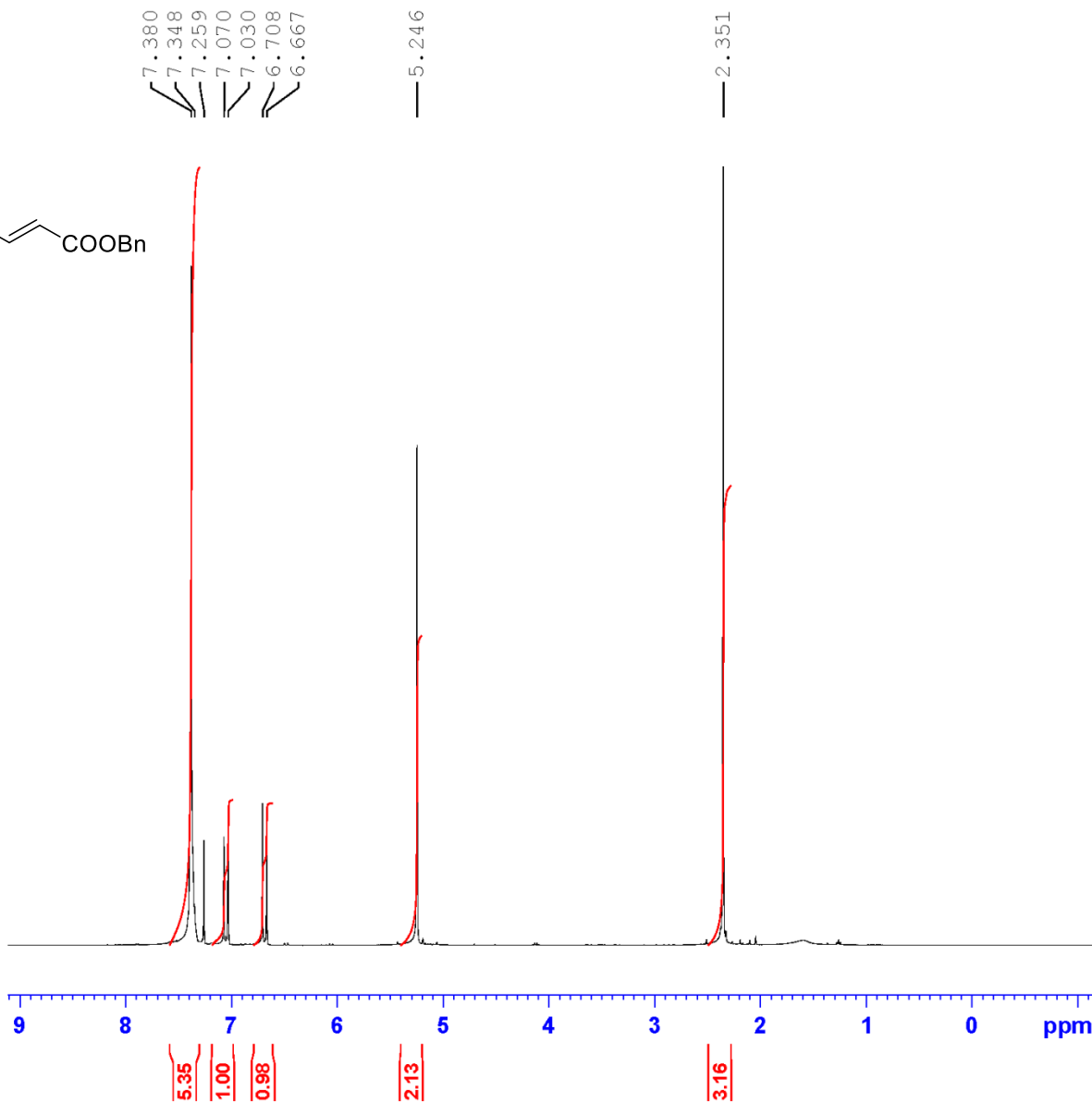
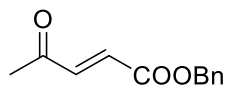
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 EXPNO 10
 PROCNO 1

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 PULPROG zgpg30
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 SOLVENT CDCl3
 NS 1300
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.8 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
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 NUC1 13C
 P1 10.00 usec
 PLW1 70.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 8.00000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

F2 - Processing parameters
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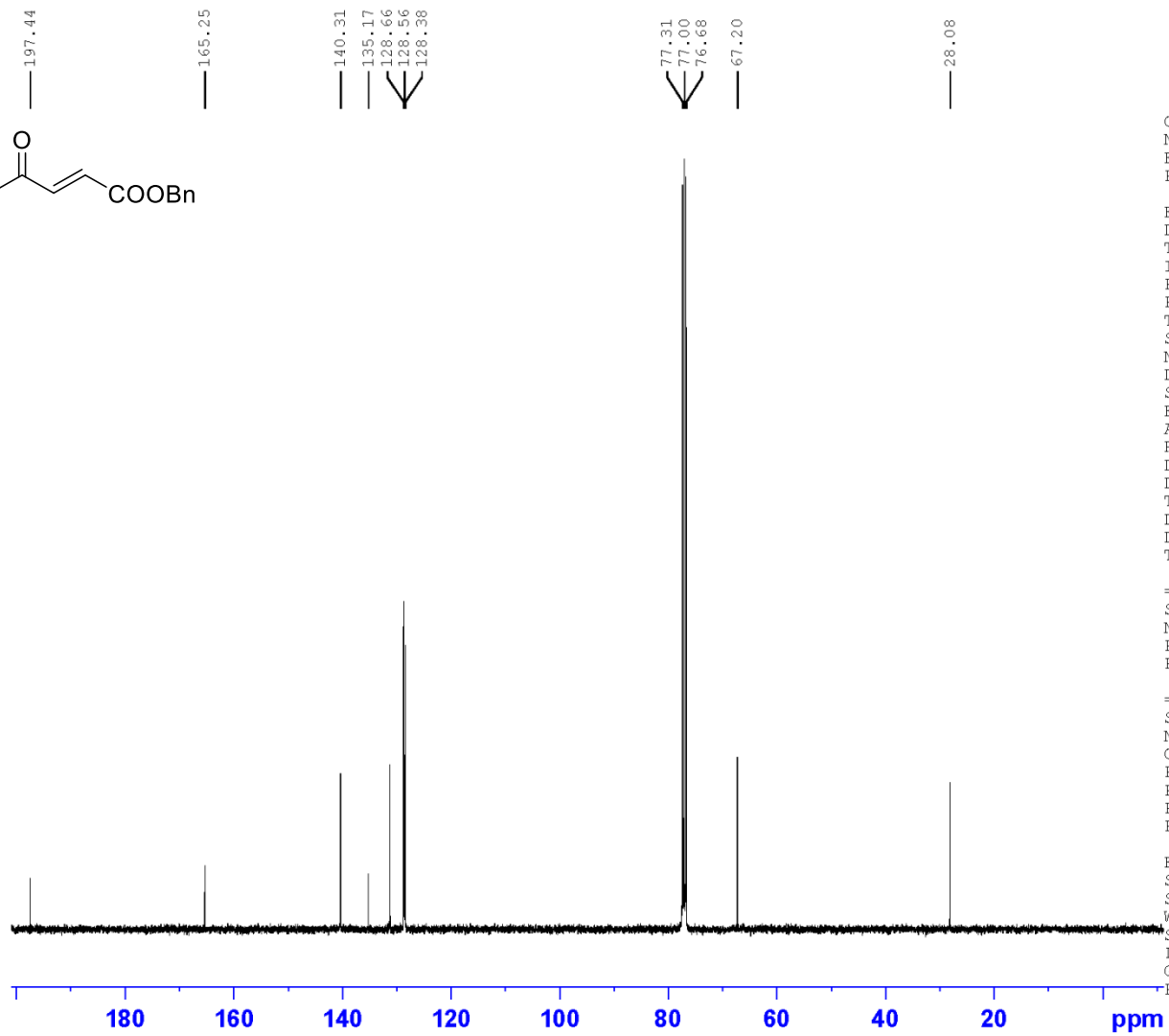
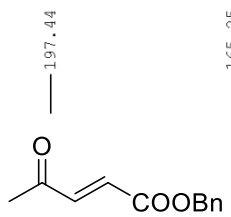


Current Data Parameters
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 EXPNO 10
 PROCNO 1

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 Time_ 14.06
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 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 71.01
 DW 62.400 usec
 DE 6.50 usec
 TE 298.7 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300095 MHz
 WDW EM
 SSB 0
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 GB 0
 FC 1.00



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Current Data Parameters
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EXPNO     10
PROCNO    1

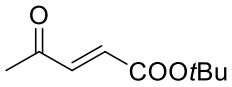
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PULPROG   zgpg30
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SOLVENT   CDCl3
NS         1024
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631488 sec
RG         195.88
DW         20.800 usec
DE         6.50 usec
TE         299.8 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      100.6228293 MHz
NUC1       13C
P1         10.00 usec
PLW1       70.00000000 W

===== CHANNEL f2 =====
SFO2      400.1316005 MHz
NUC2       1H
CPDPRG2   waltz16
PCPD2     80.00 usec
PLW2       8.00000000 W
PLW12     0.28125000 W
PLW13     0.28125000 W

F2 - Processing parameters
SI         32768
SF         100.6127713 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

```



7.259
6.931
6.891
6.588
6.547

2.339
1.506

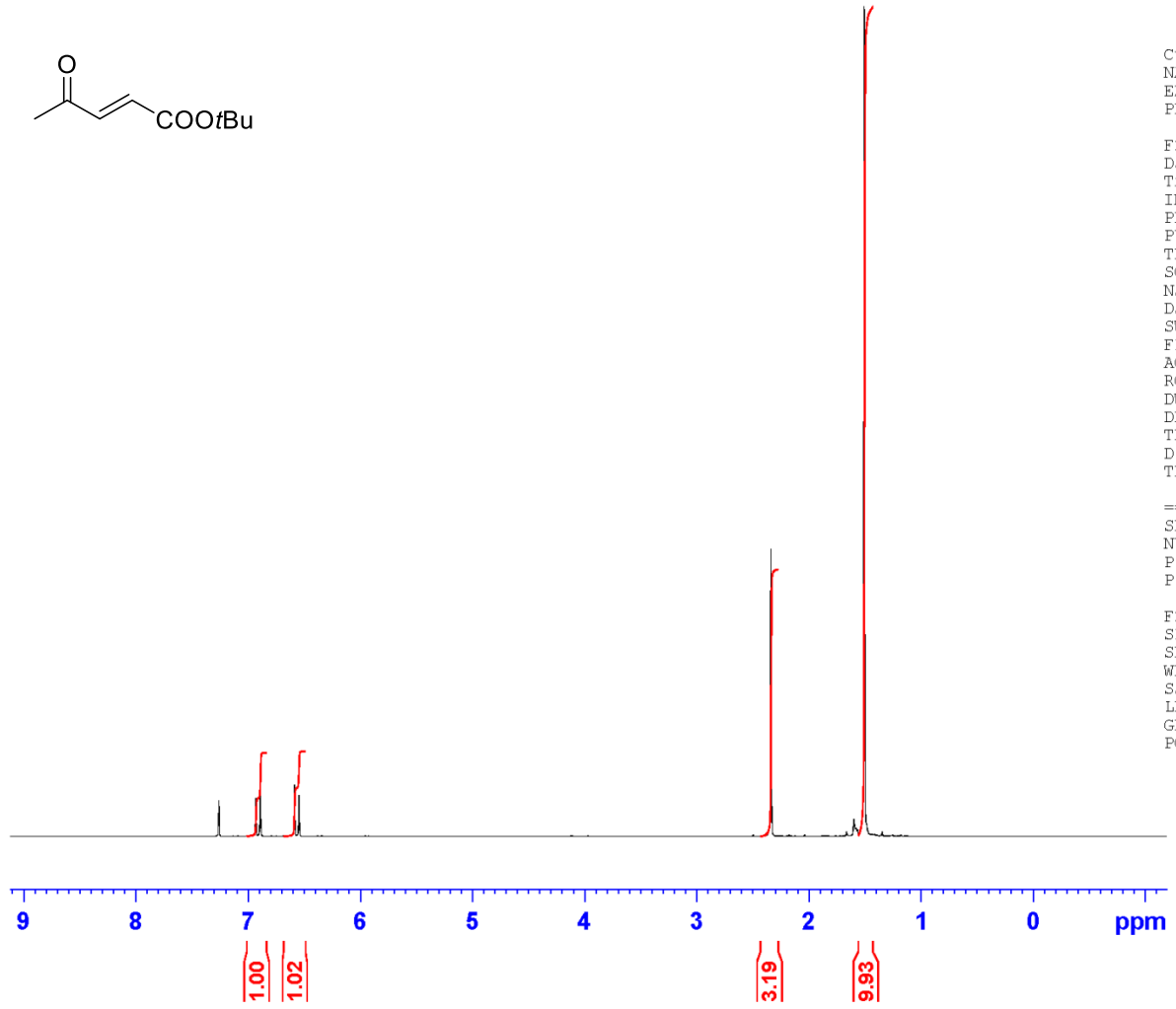
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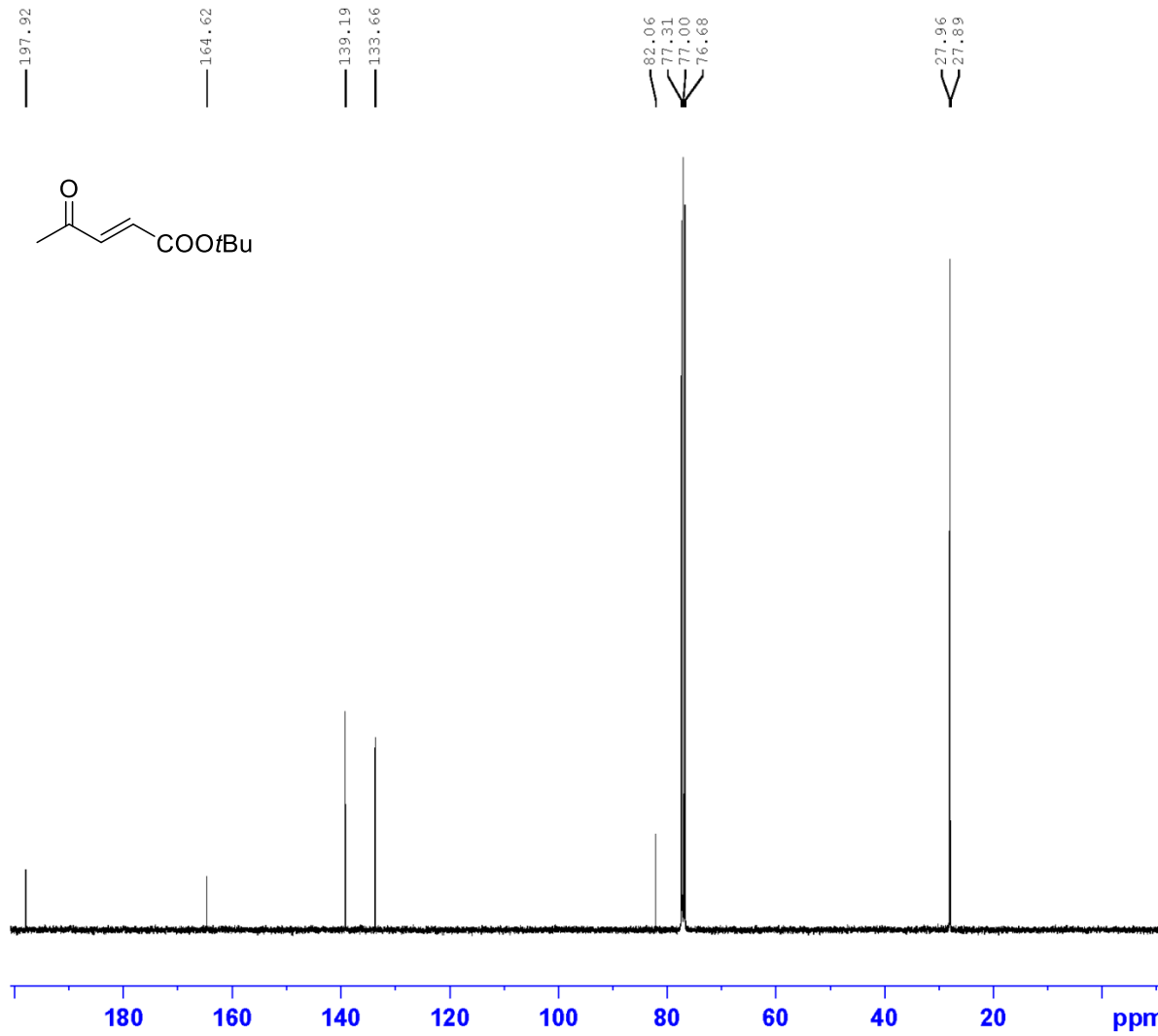
Current Data Parameters
NAME      yf-16-0816-s4
EXPNO     10
PROCNO    1

F2 - Acquisition Parameters
Date_     20160816
Time      14.11
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ         4.0894465 sec
RG         62.88
DW         62.400 usec
DE         6.50 usec
TE         298.7 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      400.1324710 MHz
NUC1       1H
P1         15.00 usec
PLW1       8.00000000 W

F2 - Processing parameters
SI         65536
SF         400.1300096 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```





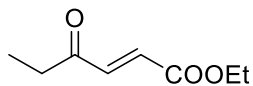
Current Data Parameters
 NAME YF-16-0816-S4-C
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160817
 Time_ 11.02
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.8 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 8.00000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127706 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

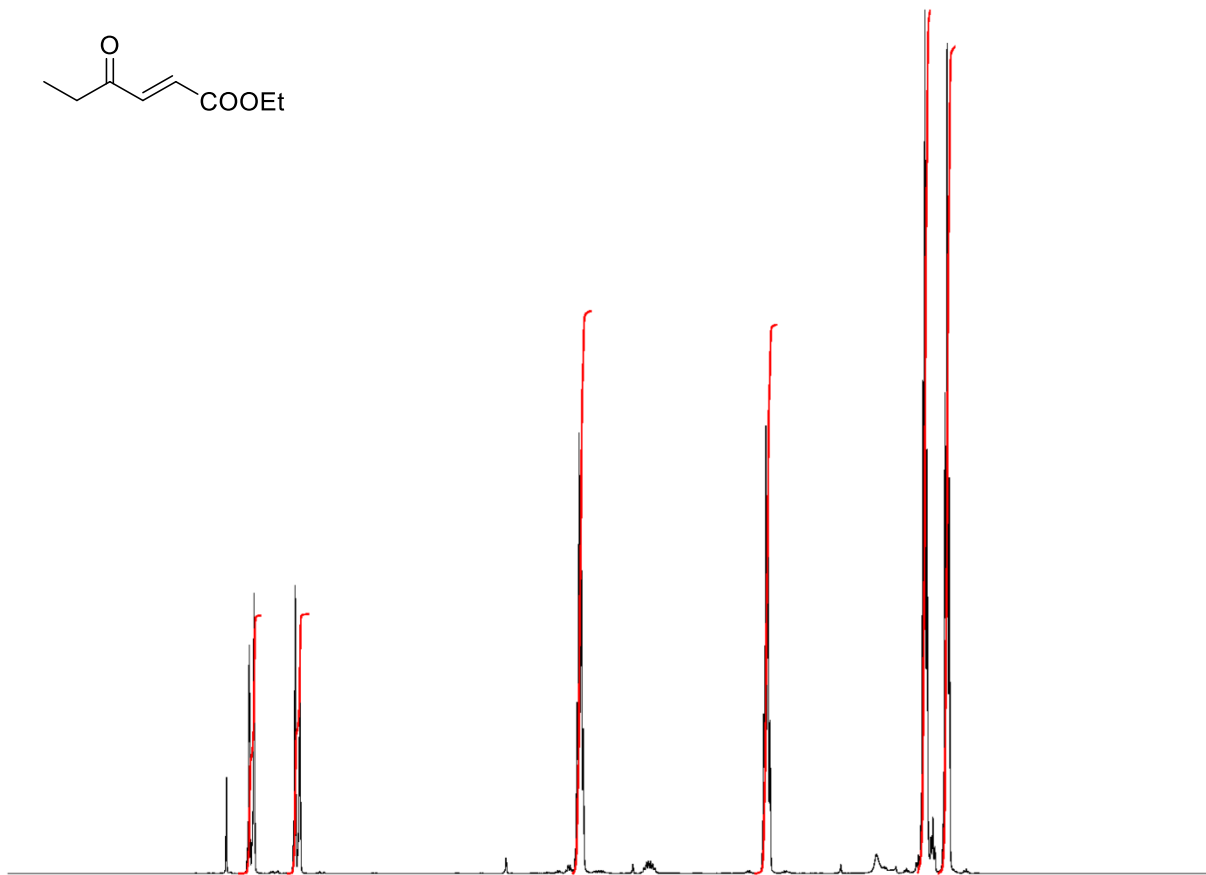


7.260
7.065
7.025
6.675
6.635

4.271
4.253
4.236
4.218

2.678
2.661
2.643
2.625

1.321
1.303
1.285
1.131
1.113
1.095



9 8 7 6 5 4 3 2 1 0 ppm

1.00
1.01

2.18

2.13

3.35
3.20

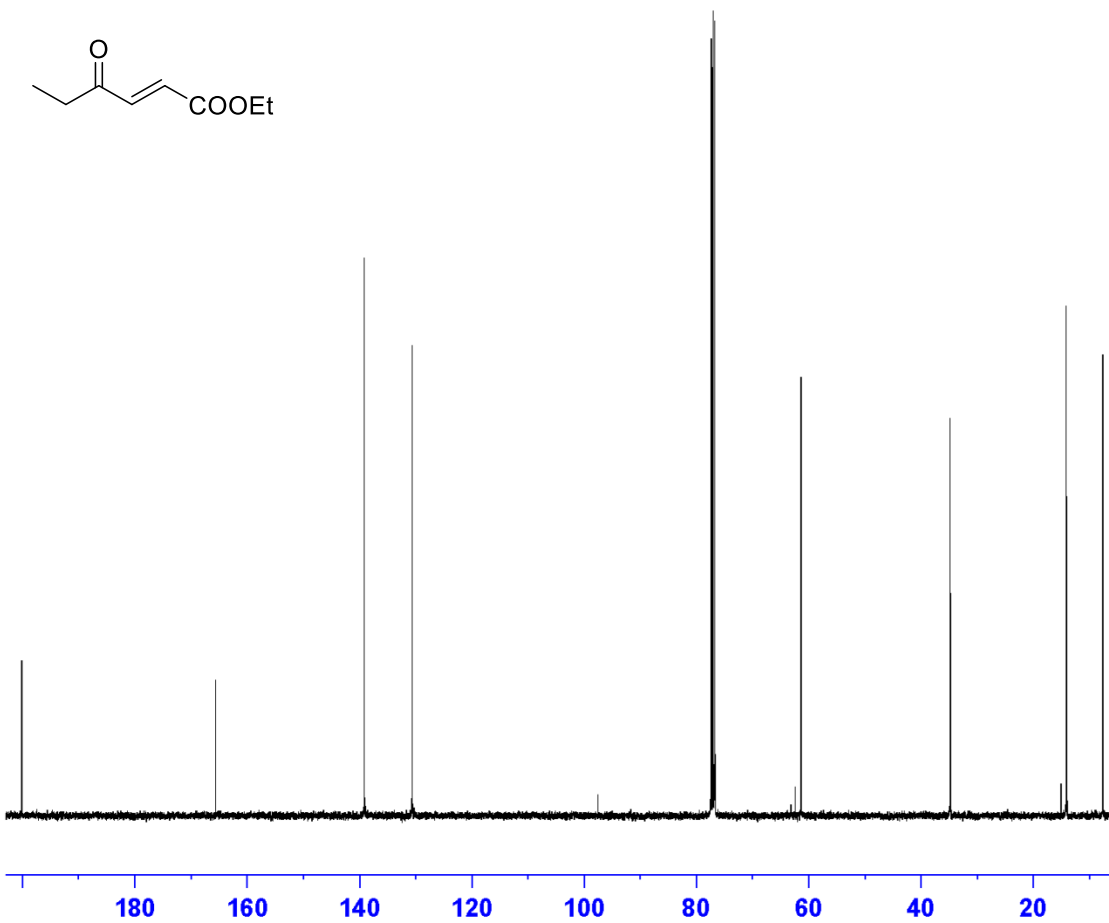
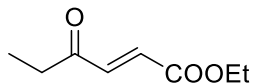
Current Data Parameters
NAME yf-16-0816-s5
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160816
Time_ 14.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.13
DW 62.400 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

200.09
 165.54
 139.12
 130.58
 77.31
 76.99
 76.68
 61.32
 34.75
 14.07
 7.55



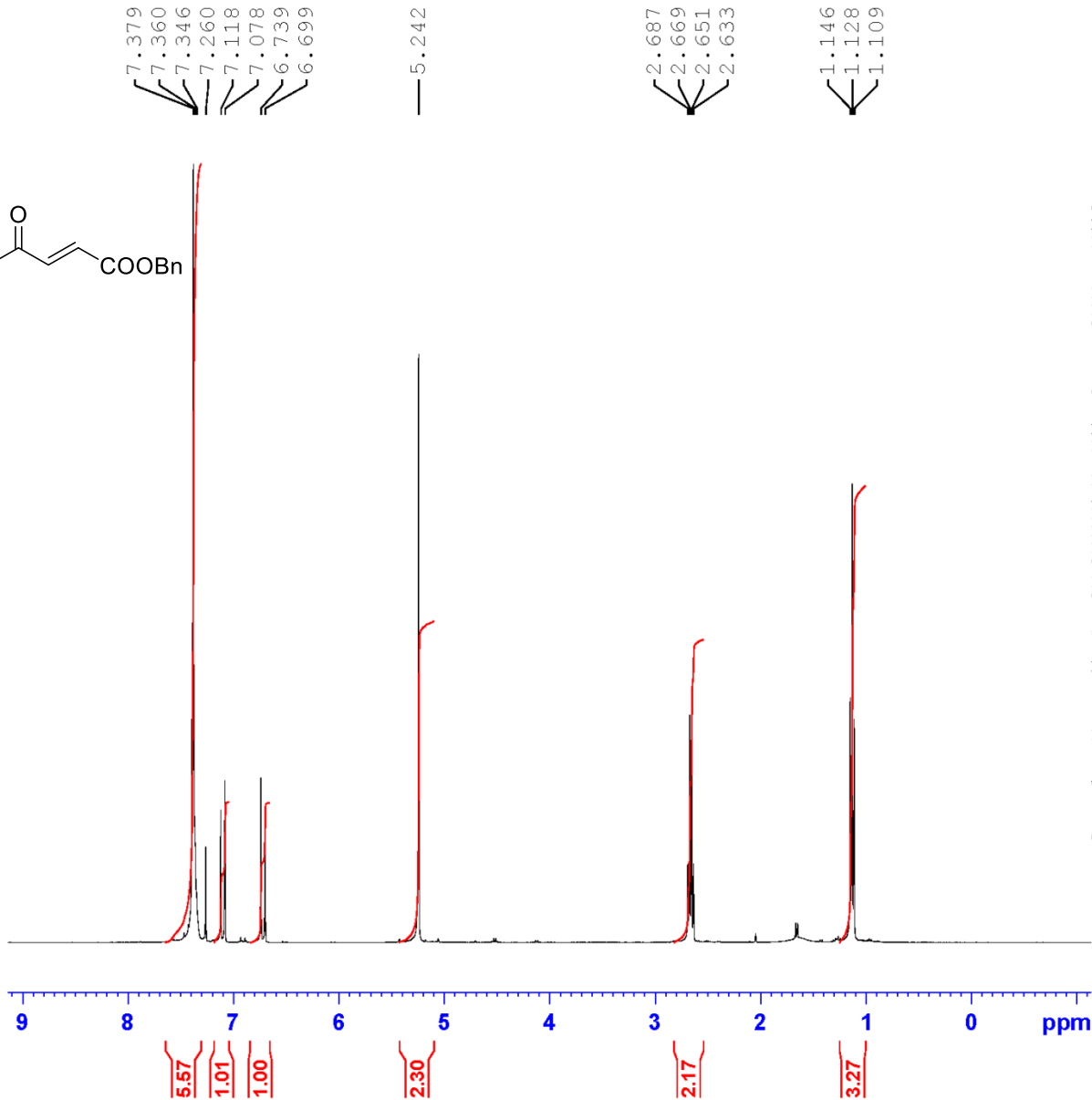
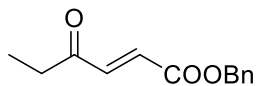
Current Data Parameters
 NAME YF-16-0816-S5-C
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160817
 Time_ 12.12
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.9 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 8.00000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127721 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

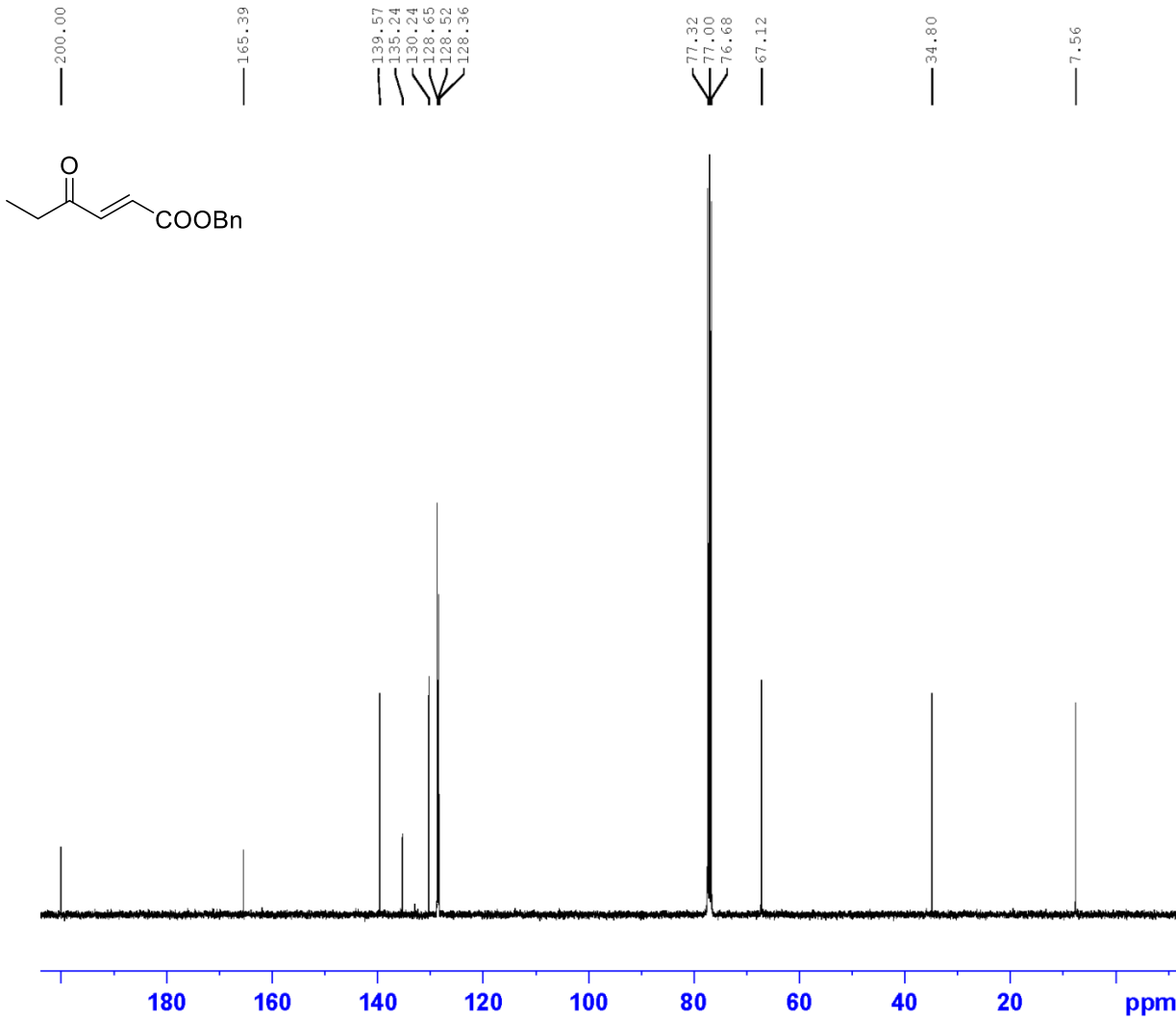


Current Data Parameters
 NAME yf-16-0816-s6
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160816
 Time_ 14.19
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 62.88
 DW 62.400 usec
 DE 6.50 usec
 TE 298.8 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300090 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



```

Current Data Parameters
NAME      YF-16-0816-S6-C
EXPNO     10
PROCNO    1

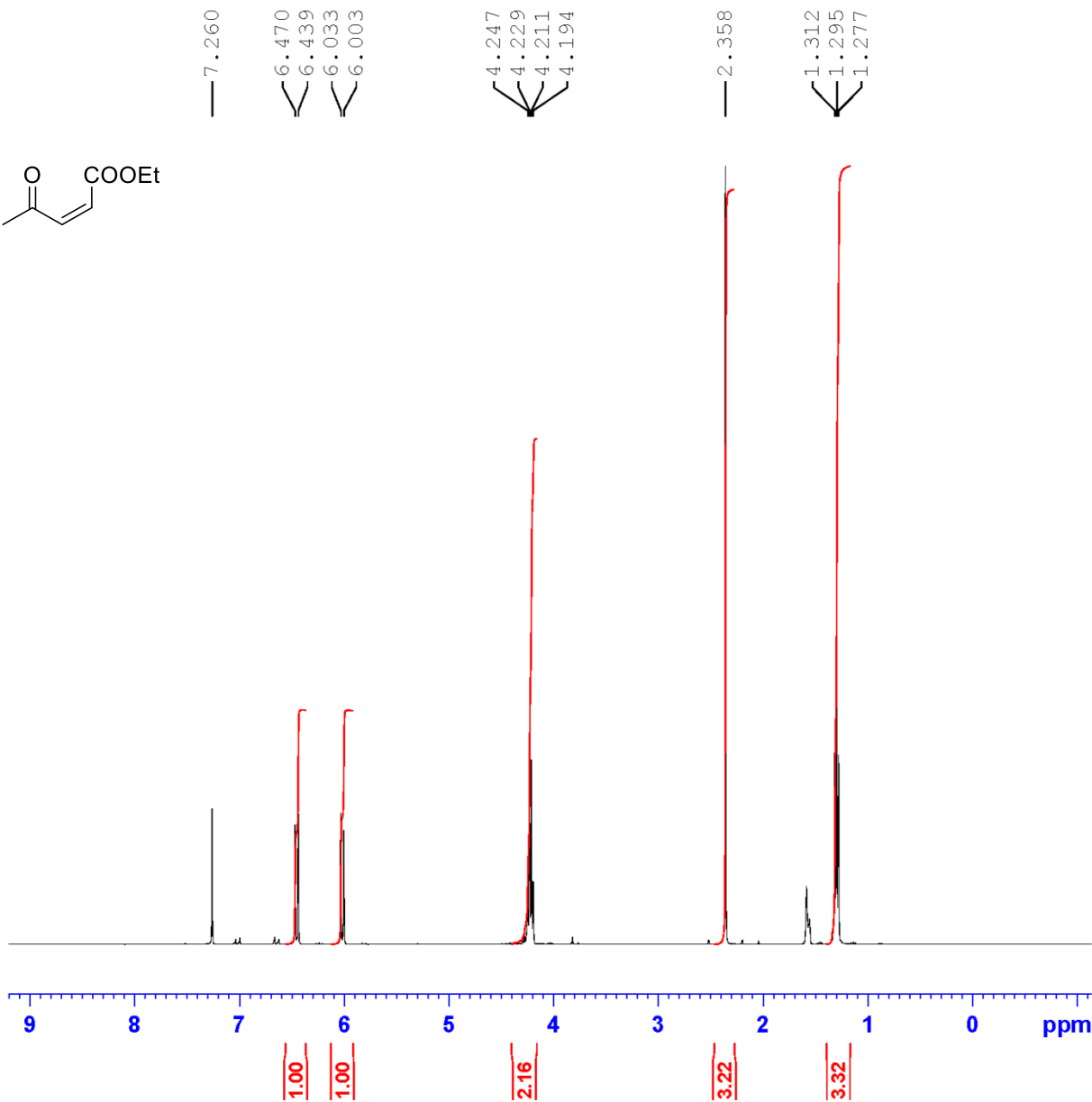
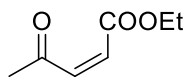
F2 - Acquisition Parameters
Date_     20160817
Time_     13.13
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDC13
NS         1024
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631488 sec
RG         195.88
DW         20.800 usec
DE         6.50 usec
TE         299.9 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      100.6228293 MHz
NUC1       13C
P1         10.00 usec
PLW1       70.00000000 W

===== CHANNEL f2 =====
SFO2      400.1316005 MHz
NUC2       1H
CPDPRG[2] waltz16
PCPD2      80.00 usec
PLW2       8.00000000 W
PLW12      0.28125000 W
PLW13      0.28125000 W

F2 - Processing parameters
SI         32768
SF         100.6127713 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

```

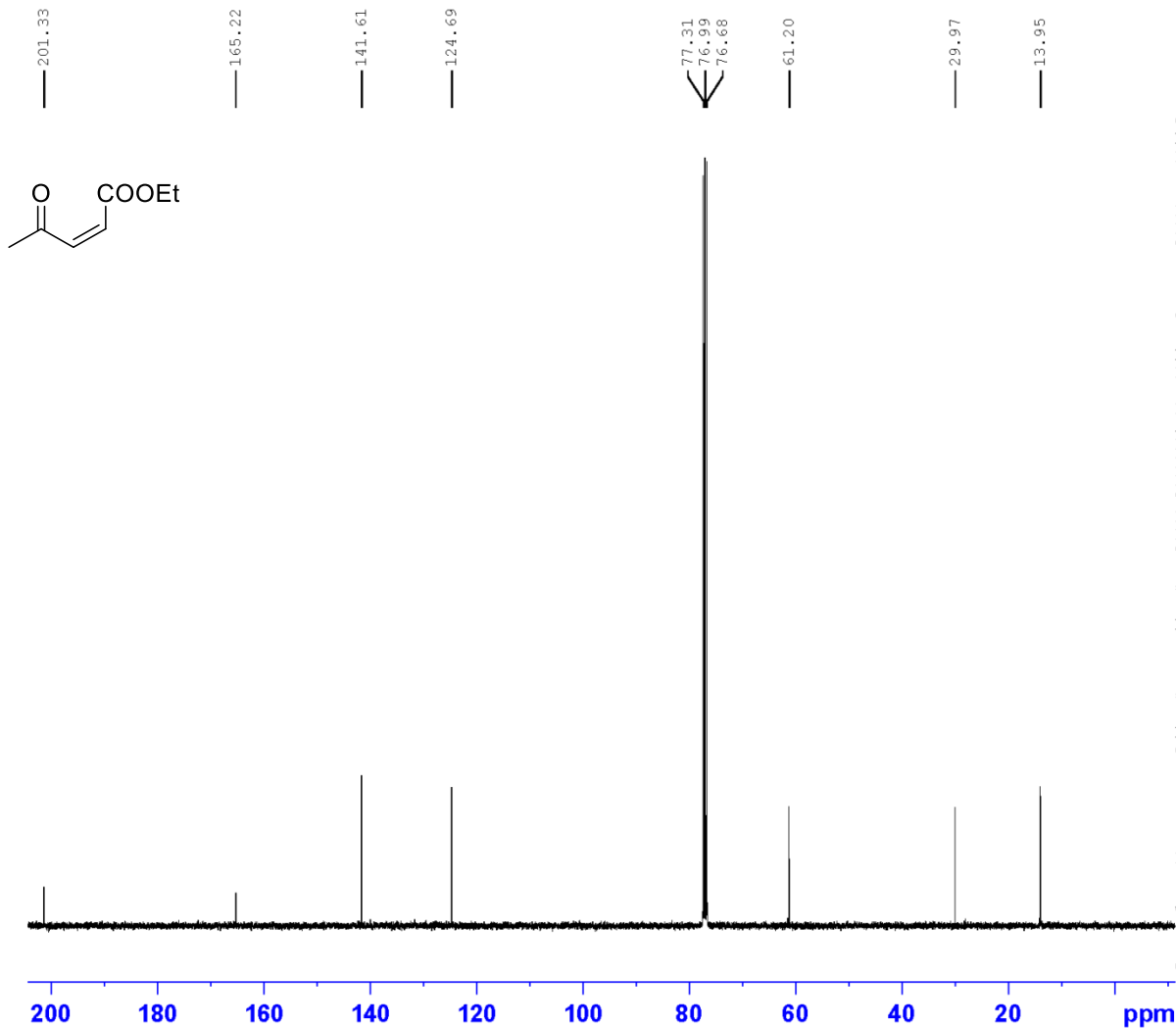


Current Data Parameters
 NAME yf-16-0816-s7-1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160816
 Time_ 15.42
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 77.81
 DW 62.400 usec
 DE 6.50 usec
 TE 299.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300092 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



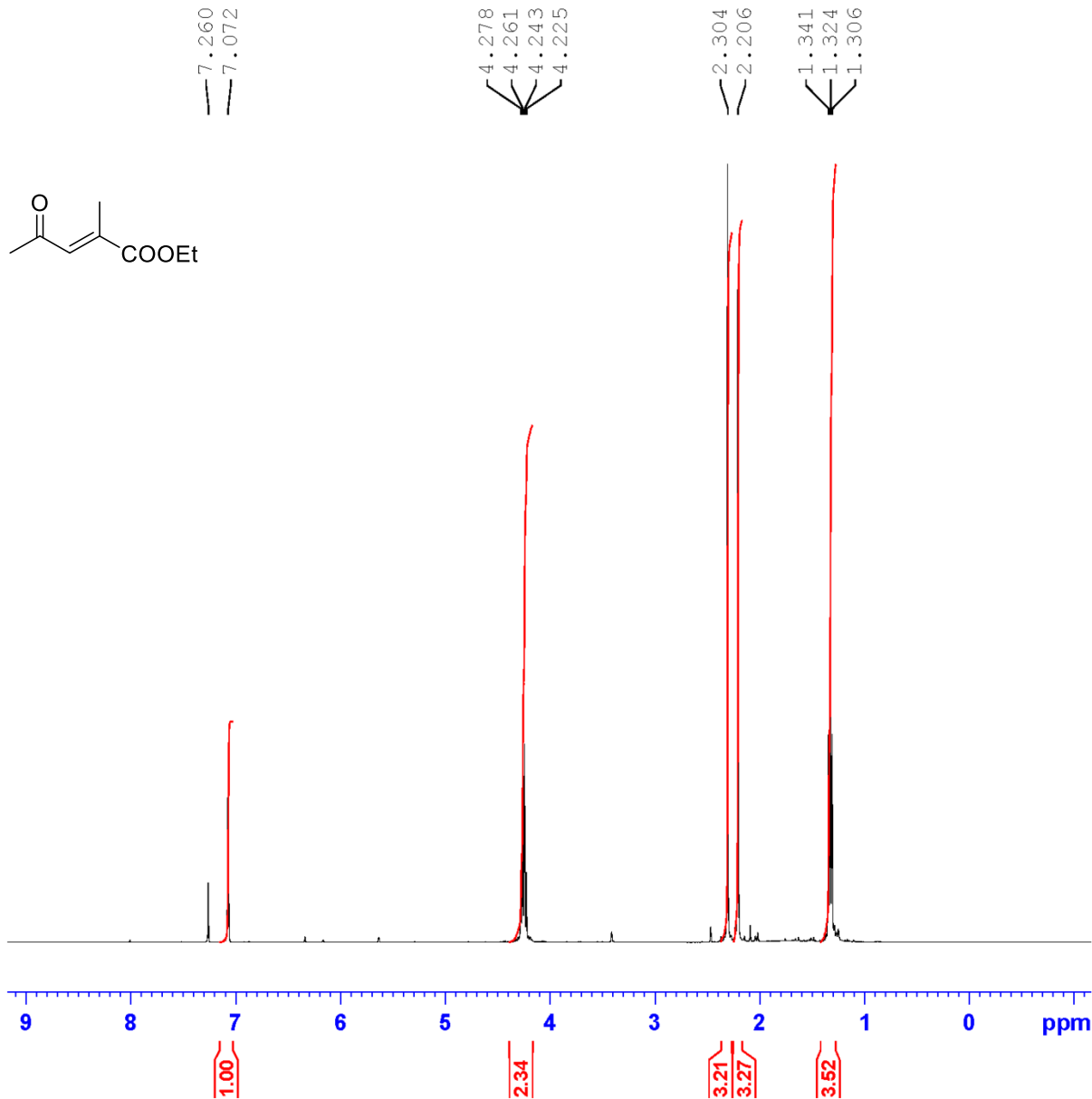
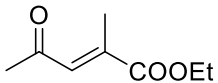
Current Data Parameters
 NAME YF-16-0816-S7-C
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date 20160817
 Time 14.15
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.9 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 FCPD2 80.00 usec
 PLW2 8.00000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127706 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

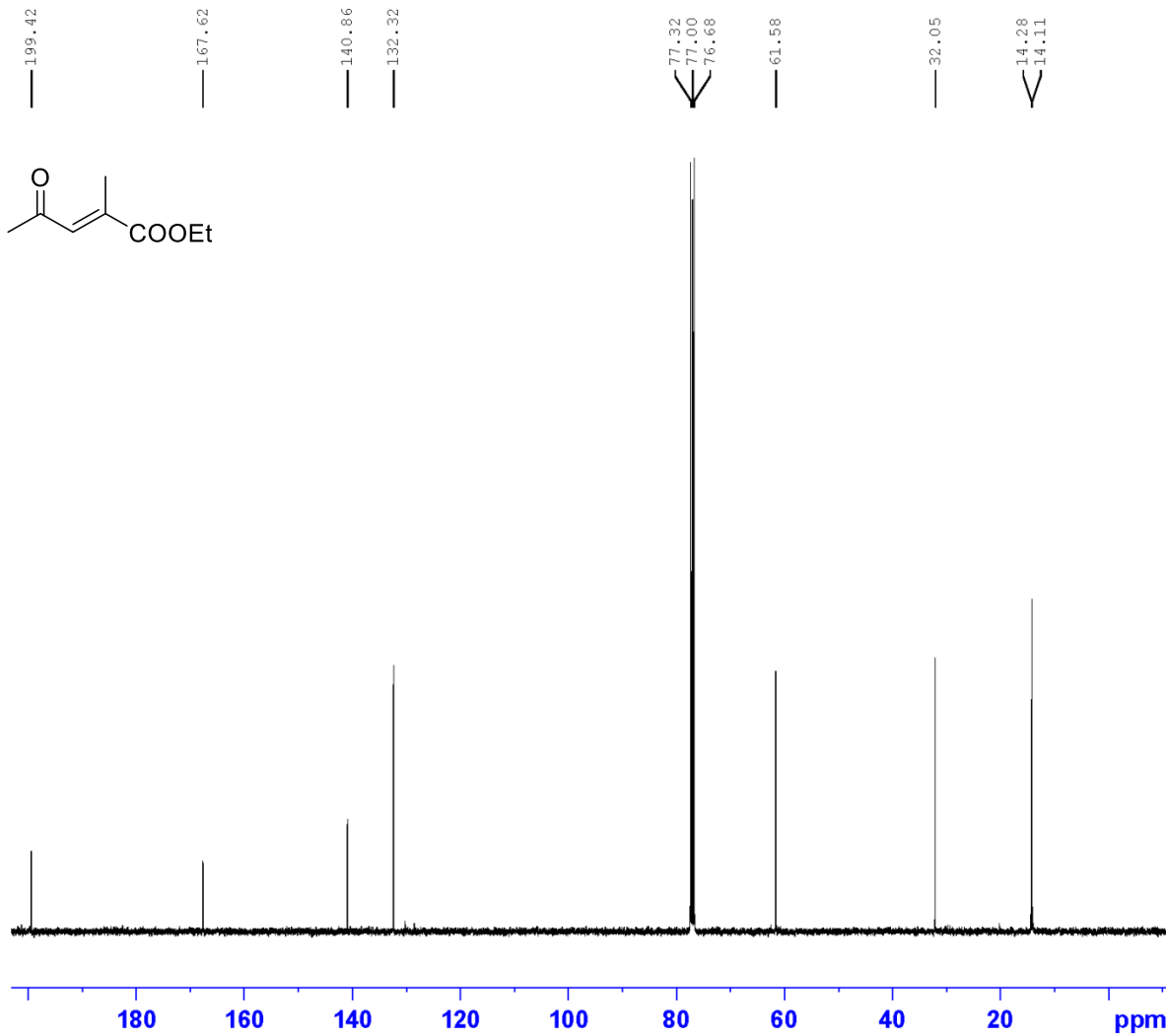


Current Data Parameters
 NAME yf-16-0816-s8-1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160816
 Time_ 15.47
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 54.59
 DW 62.400 usec
 DE 6.50 usec
 TE 299.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300091 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



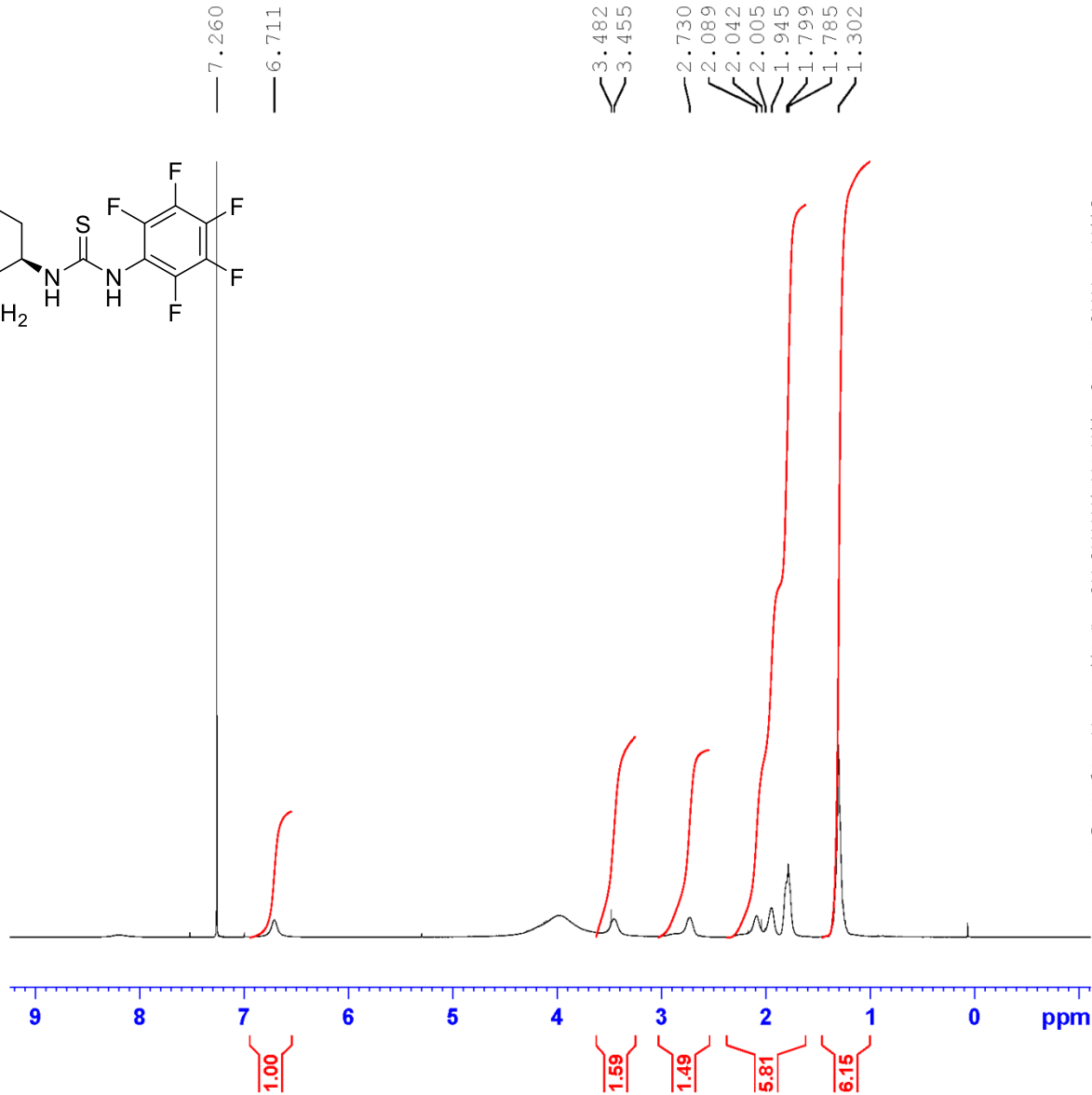
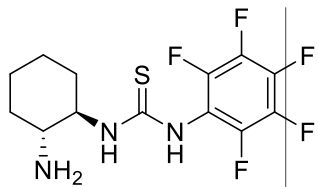
Current Data Parameters
 NAME YF-16-0816-S8-C
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160817
 Time_ 15.16
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.9 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 8.00000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127706 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

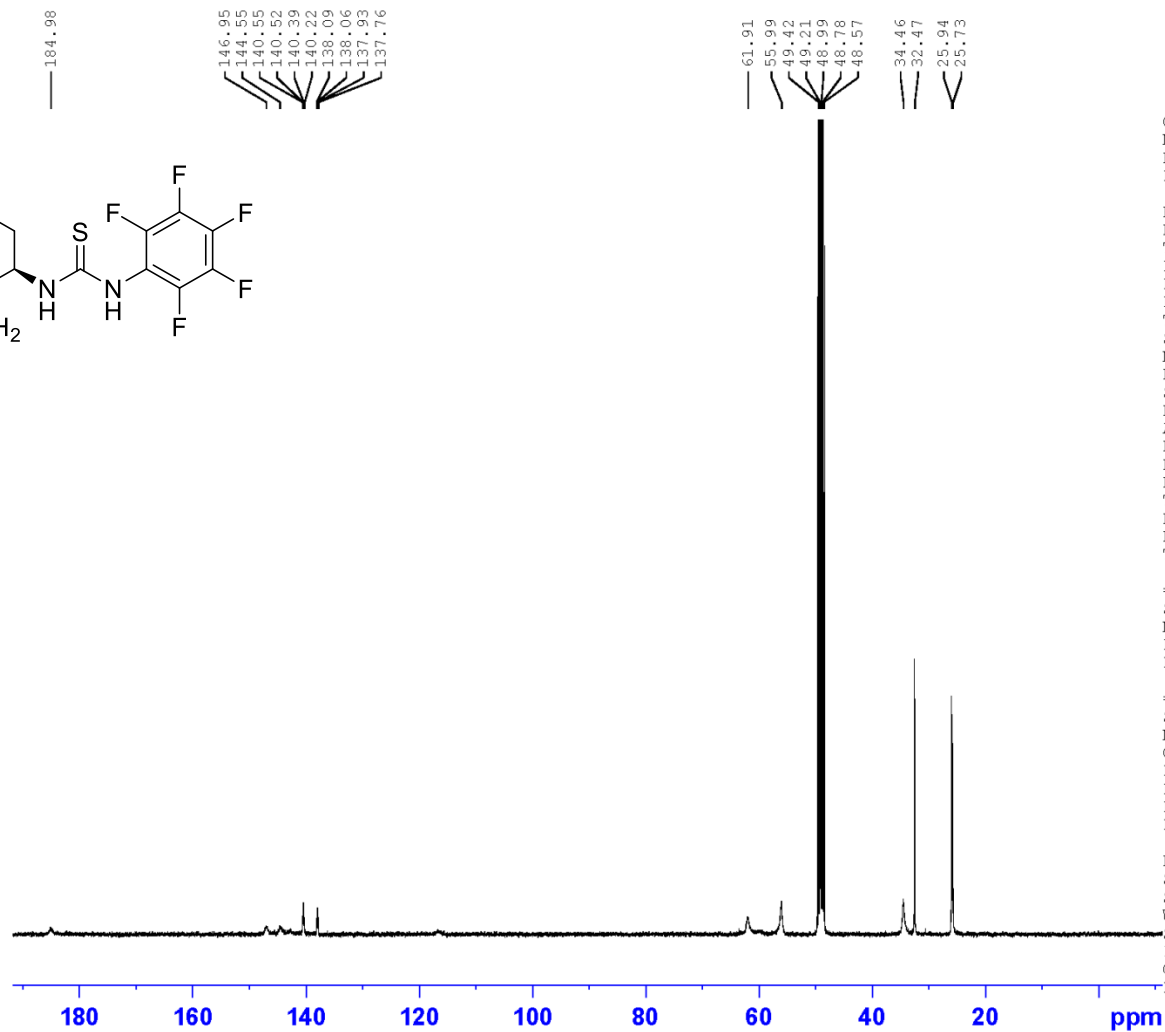
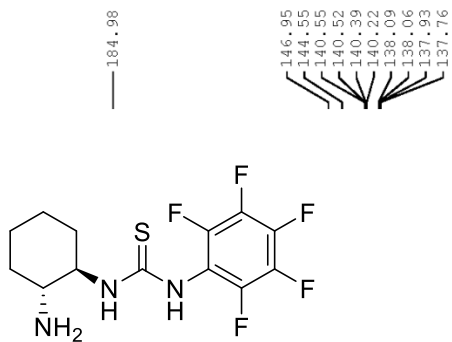


Current Data Parameters
 NAME yf-16-1122-c7
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161122
 Time_ 16.10
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 77.81
 DW 62.400 usec
 DE 6.50 usec
 TE 298.4 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 7.50000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300096 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



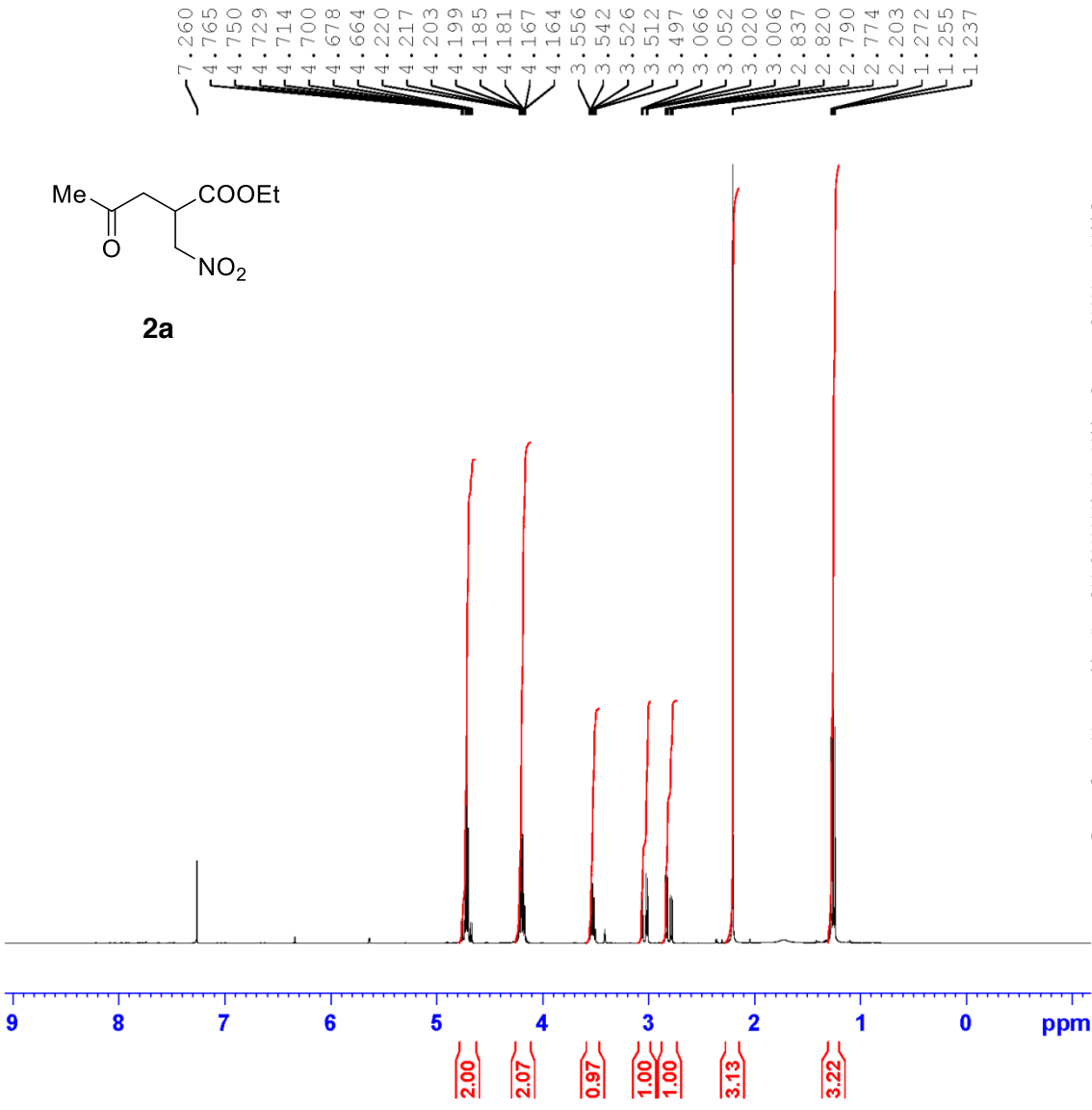
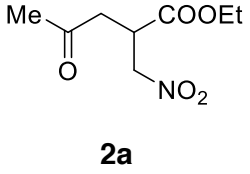
Current Data Parameters
 NAME YF-17-0125-C7-C
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170125
 Time_ 8.35
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CD3OD_SFE
 NS 8000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.4 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 72.0000000 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 7.5000000 W
 PLW12 0.26367000 W
 PLW13 0.16633999 W

F2 - Processing parameters
 SI 32768
 SF 100.6126284 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



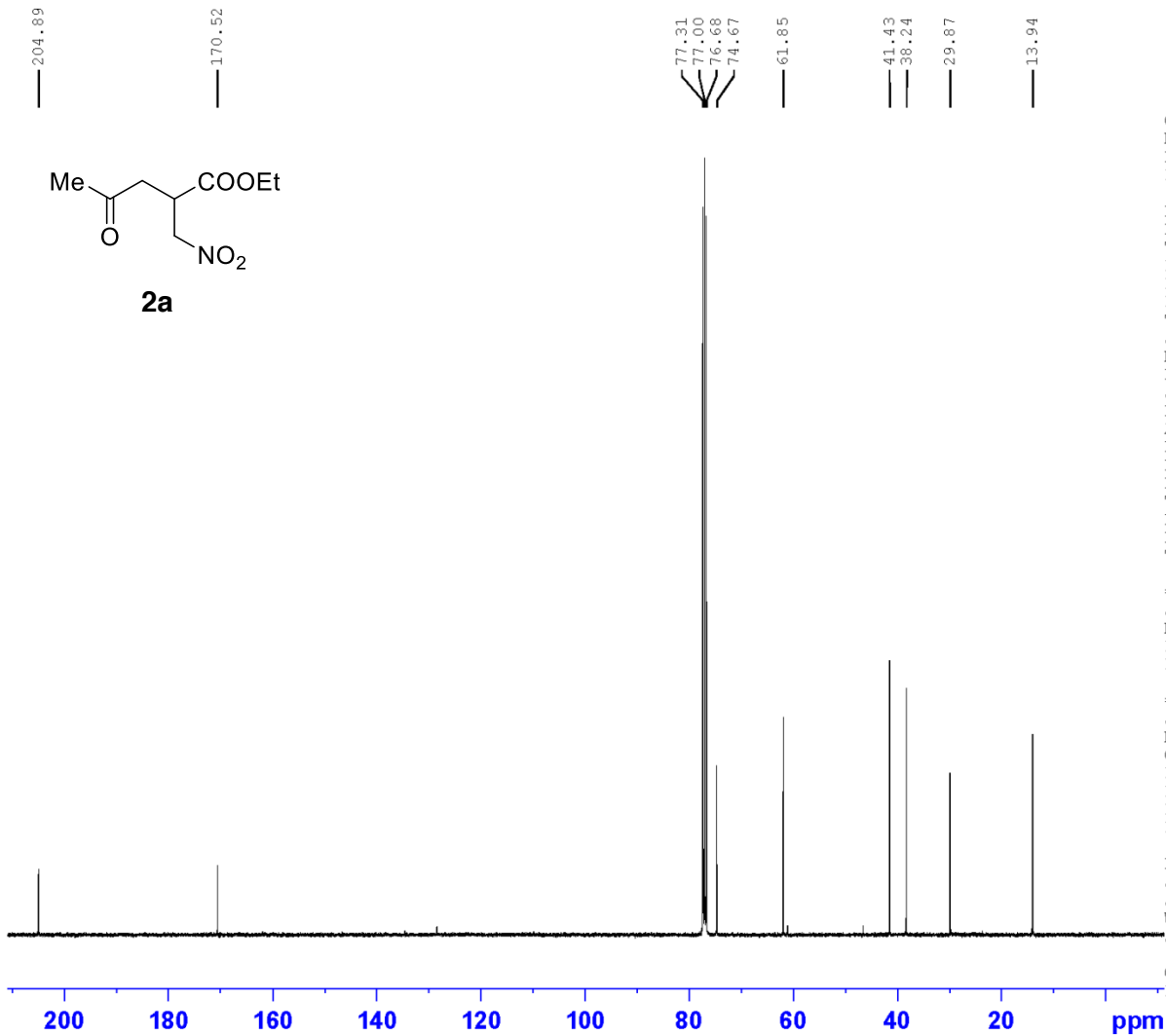
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Current Data Parameters
NAME      yf-16-0222-414
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20160222
Time      14.49
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDC13
NS         16
DS         2
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.089465 sec
RG         54.59
DW         62.400 usec
DE         6.50 usec
TE         300.2 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      400.1324710 MHz
NUC1       1H
P1         15.00 usec
PLW1       8.00000000 W

F2 - Processing parameters
SI         65536
SF         400.1300095 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



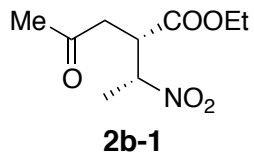
Current Data Parameters
 NAME yf-16-0303-414c
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160304
 Time_ 2.33
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 4096
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 302.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

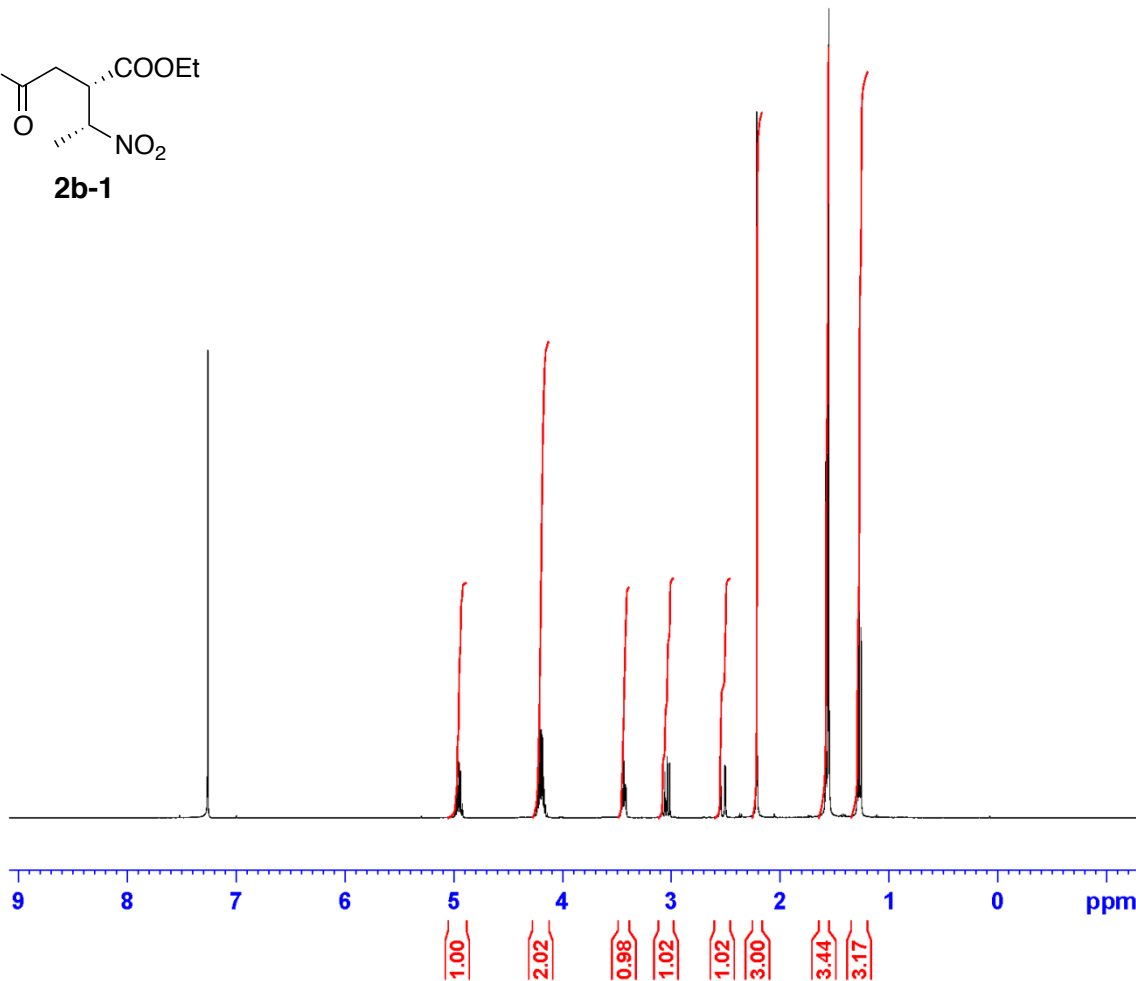
==== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 8.00000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127685 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



7.260
 4.985
 4.968
 4.953
 4.937
 4.920
 4.237
 4.228
 4.219
 4.210
 4.201
 4.192
 4.184
 4.174
 4.166
 4.157
 3.459
 3.450
 3.445
 3.435
 3.426
 3.421
 3.412
 3.081
 3.057
 3.036
 3.012
 2.552
 2.543
 2.507
 2.498
 2.212
 2.112
 1.577
 1.560
 1.288
 1.270
 1.253



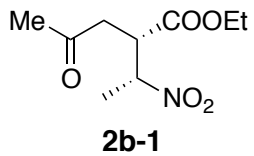
Current Data Parameters
 NAME yf-16-0407-521-9
 EXPNO 10
 PROCNO 1

 F2 - Acquisition Parameters
 Date_ 20160407
 Time_ 16.05
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 88.94
 DW 62.400 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

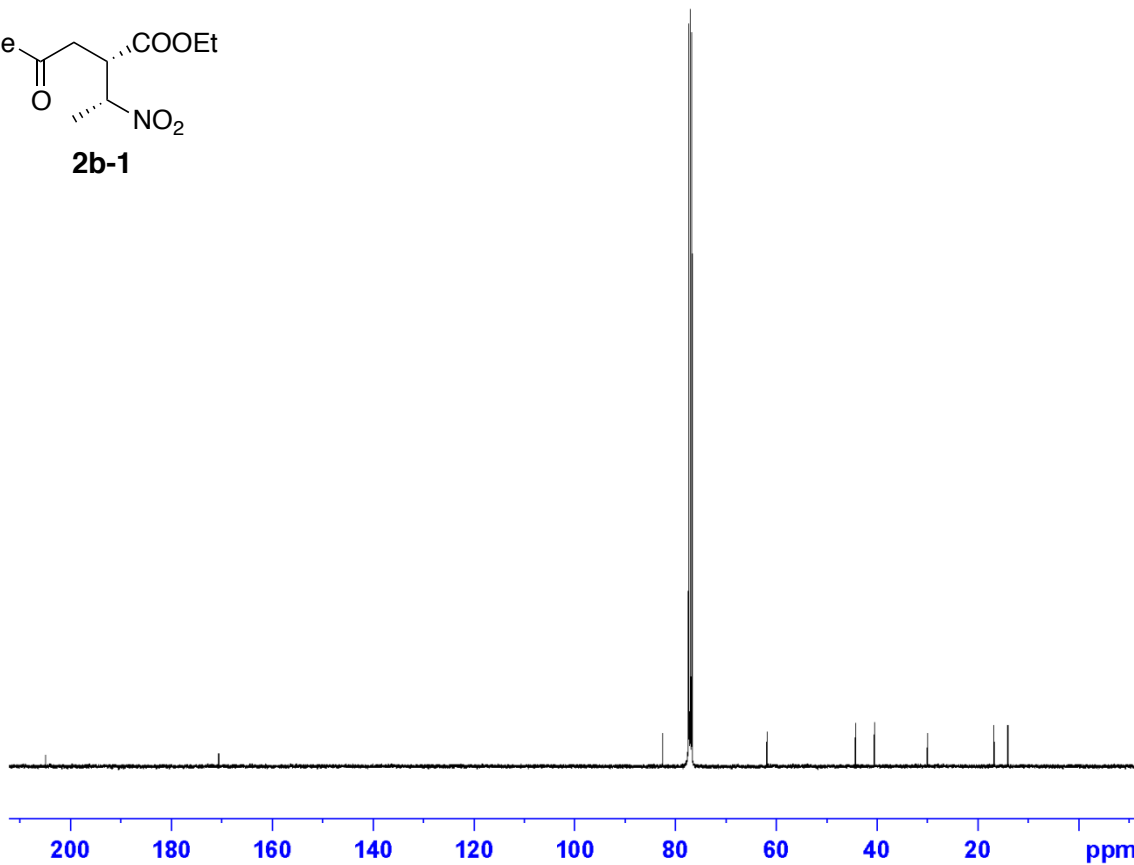
 ===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

 F2 - Processing parameters
 SI 65536
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

204.88
170.55



82.48
77.31
77.00
76.68
61.77
44.20
40.46
29.93
16.76
13.98



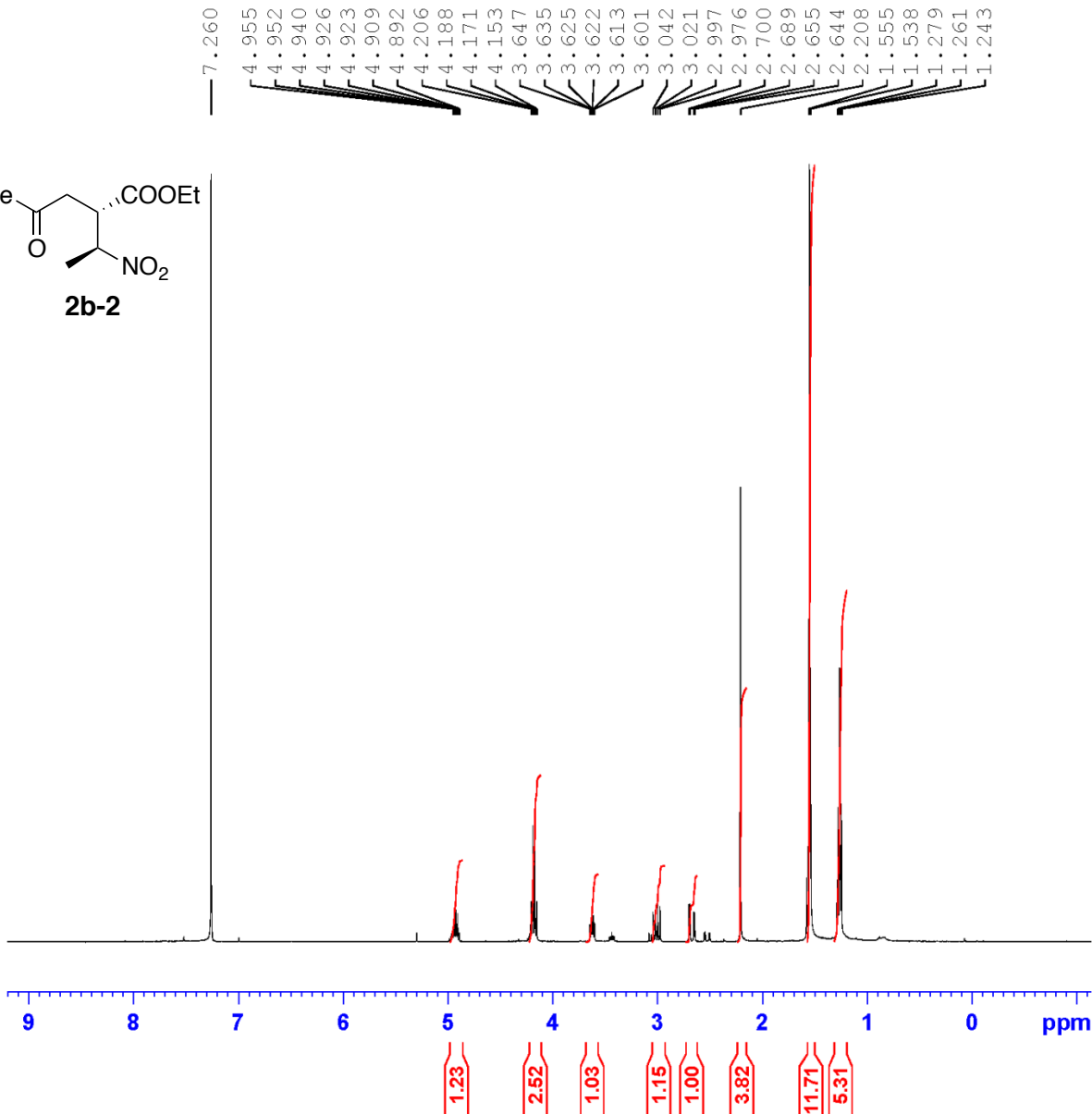
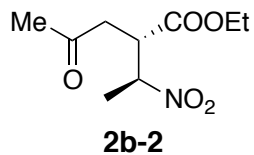
Current Data Parameters
 NAME YF-16-0407-521-9-C
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160408
 Time_ 4.06
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 6000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 301.9 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

=====
 CHANNEL f1
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.00000000 W

=====
 CHANNEL f2
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 8.00000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127670 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

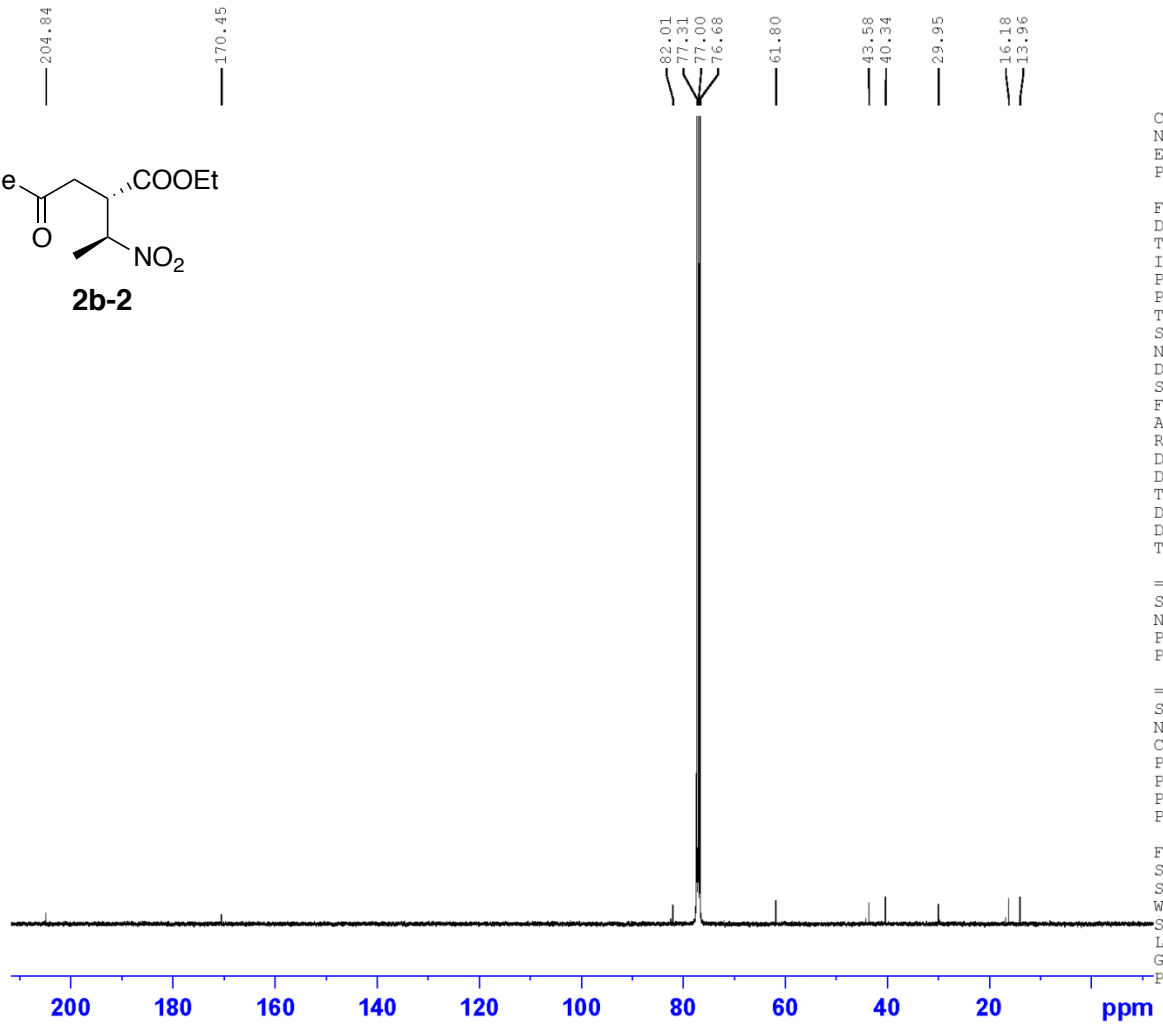
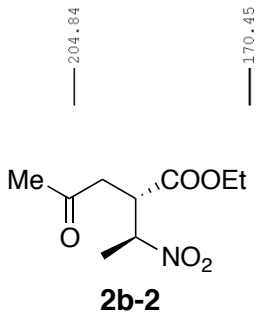


Current Data Parameters
 NAME yf-16-0620-e3-11
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160620
 Time_ 11.58
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 98.39
 DW 62.400 usec
 DE 6.50 usec
 TE 298.7 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



```

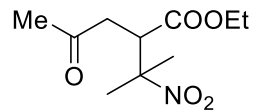
Current Data Parameters
NAME      YF-16-0707-E3-11C
EXPNO     10
PROCNO    1

F2 - Acquisition Parameters
Date_     20160708
Time      8.02
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         10000
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631488 sec
RG         195.88
DW         20.800 usec
DE         6.50 usec
TE         300.4 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1

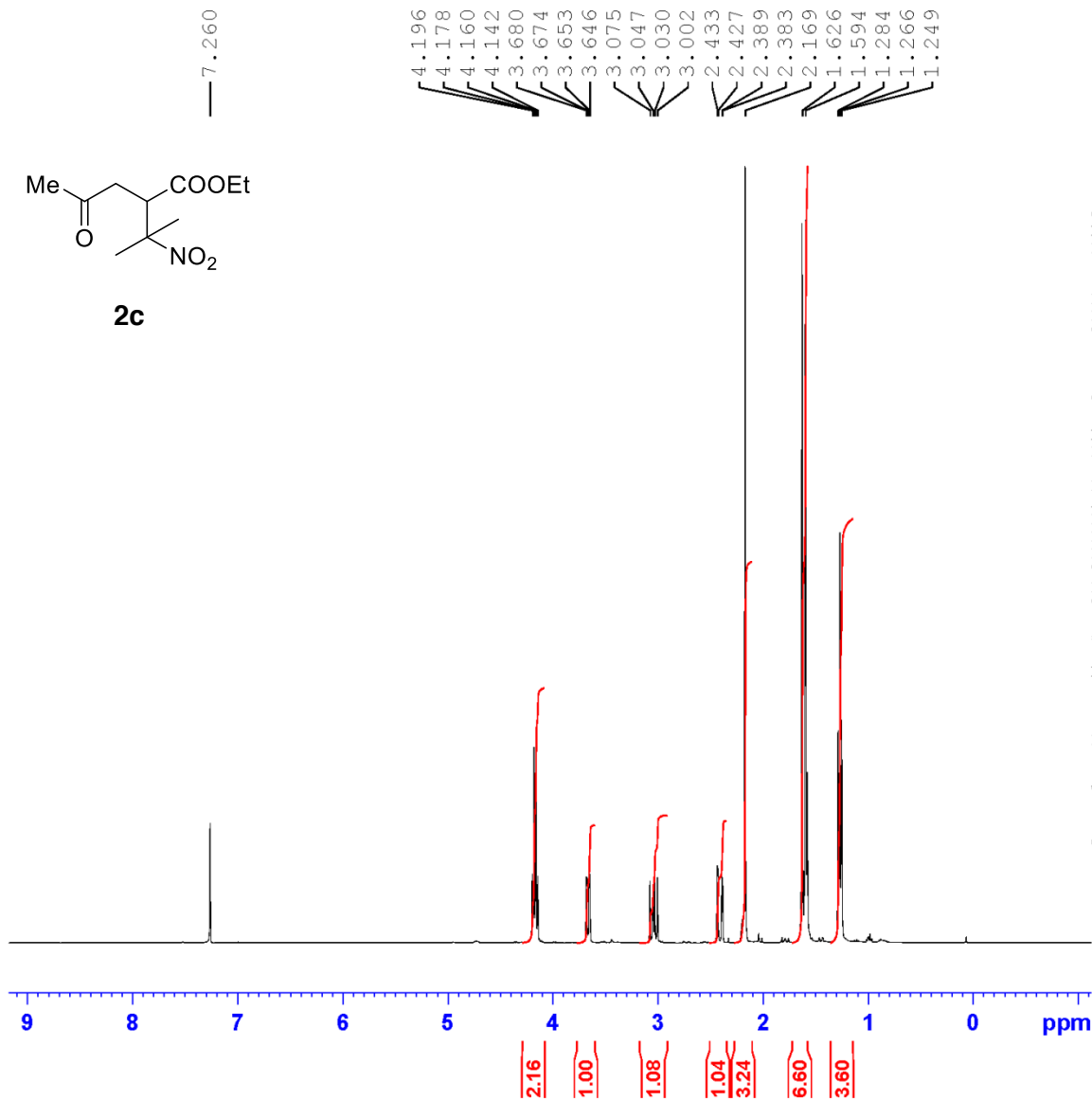
===== CHANNEL f1 =====
SFO1      100.6228293 MHz
NUC1       13C
P1         10.00 usec
PLW1       70.0000000 W

===== CHANNEL f2 =====
SFO2      400.1316005 MHz
NUC2       1H
CPDPRG[2] waltz16
PCPD2      80.00 usec
PLW2       8.0000000 W
PLW12      0.2812500 W
PLW13      0.2812500 W

F2 - Processing parameters
SI         32768
SF         100.6127697 MHz
WDW        EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```



2c

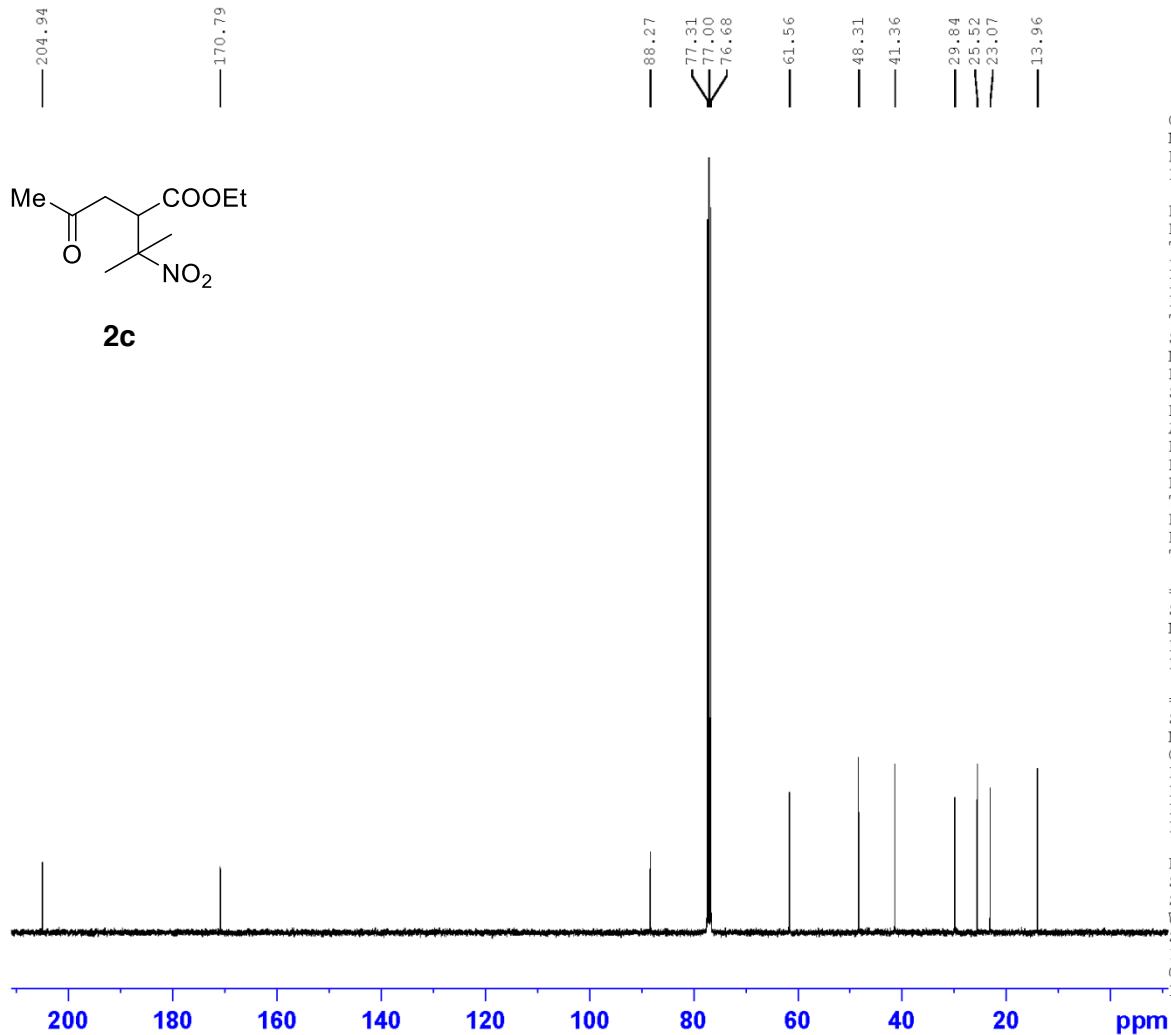


Current Data Parameters
 NAME YF-16-0818-640P
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date 20160818
 Time 18.01
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 62.88
 DW 62.400 usec
 DE 6.50 usec
 TE 298.8 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300092 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



```

Current Data Parameters
NAME      YF-16-0818-640P-C
EXPNO     10
PROCNO    1

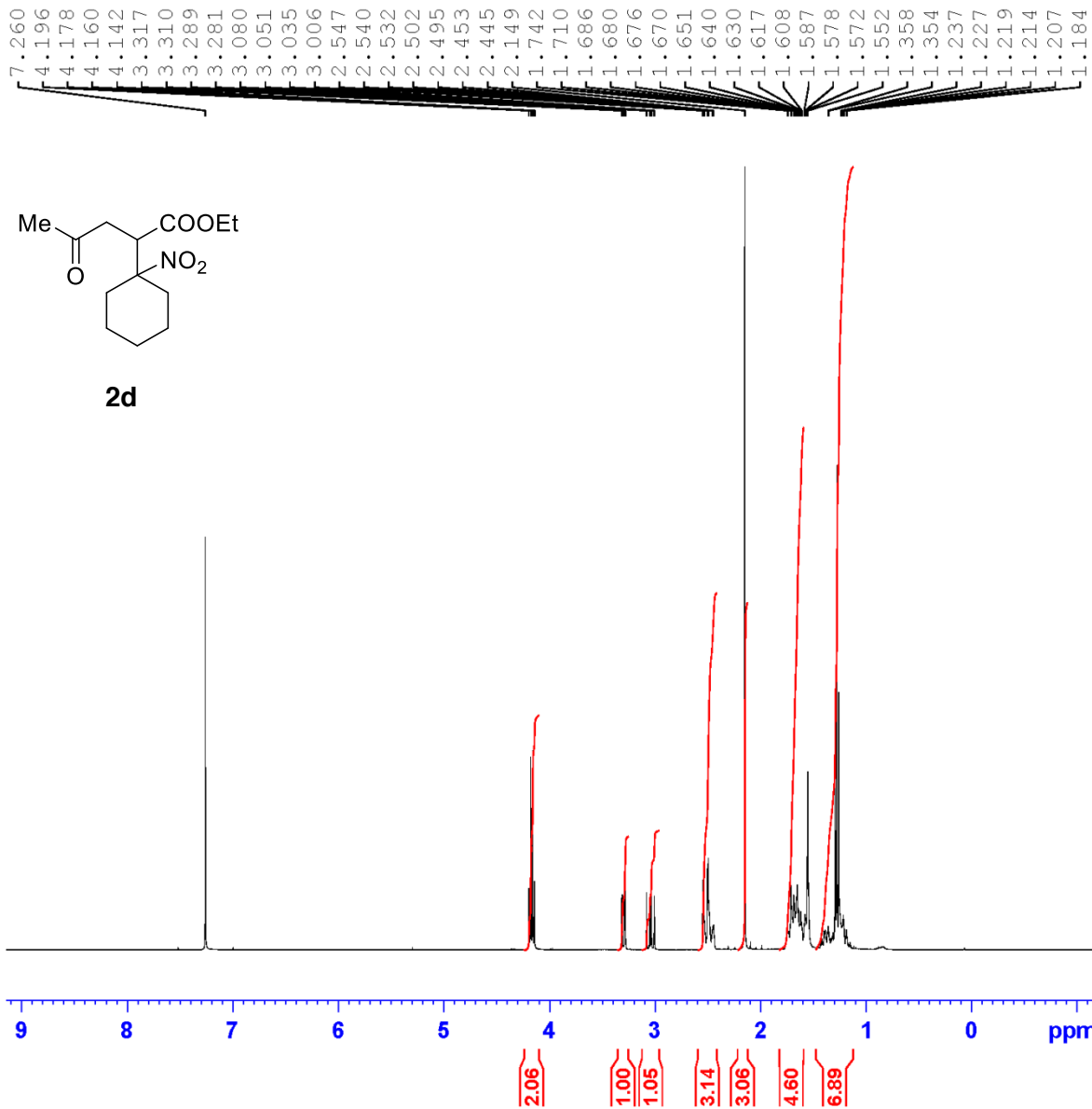
F2 - Acquisition Parameters
Date_     20160818
Time      21.48
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1200
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631488 sec
RG         195.88
DW         20.800 usec
DE         6.50 usec
TE         299.8 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      100.6228293 MHz
NUC1       13C
P1         10.00 usec
PLW1       70.00000000 W

===== CHANNEL f2 =====
SFO2      400.1316005 MHz
NUC2       1H
CPDPRG[2] waltz16
PCPD2     80.00 usec
PLW2       8.00000000 W
PLW12     0.28125000 W
PLW13     0.28125000 W

F2 - Processing parameters
SI         32768
SF         100.6127706 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

```

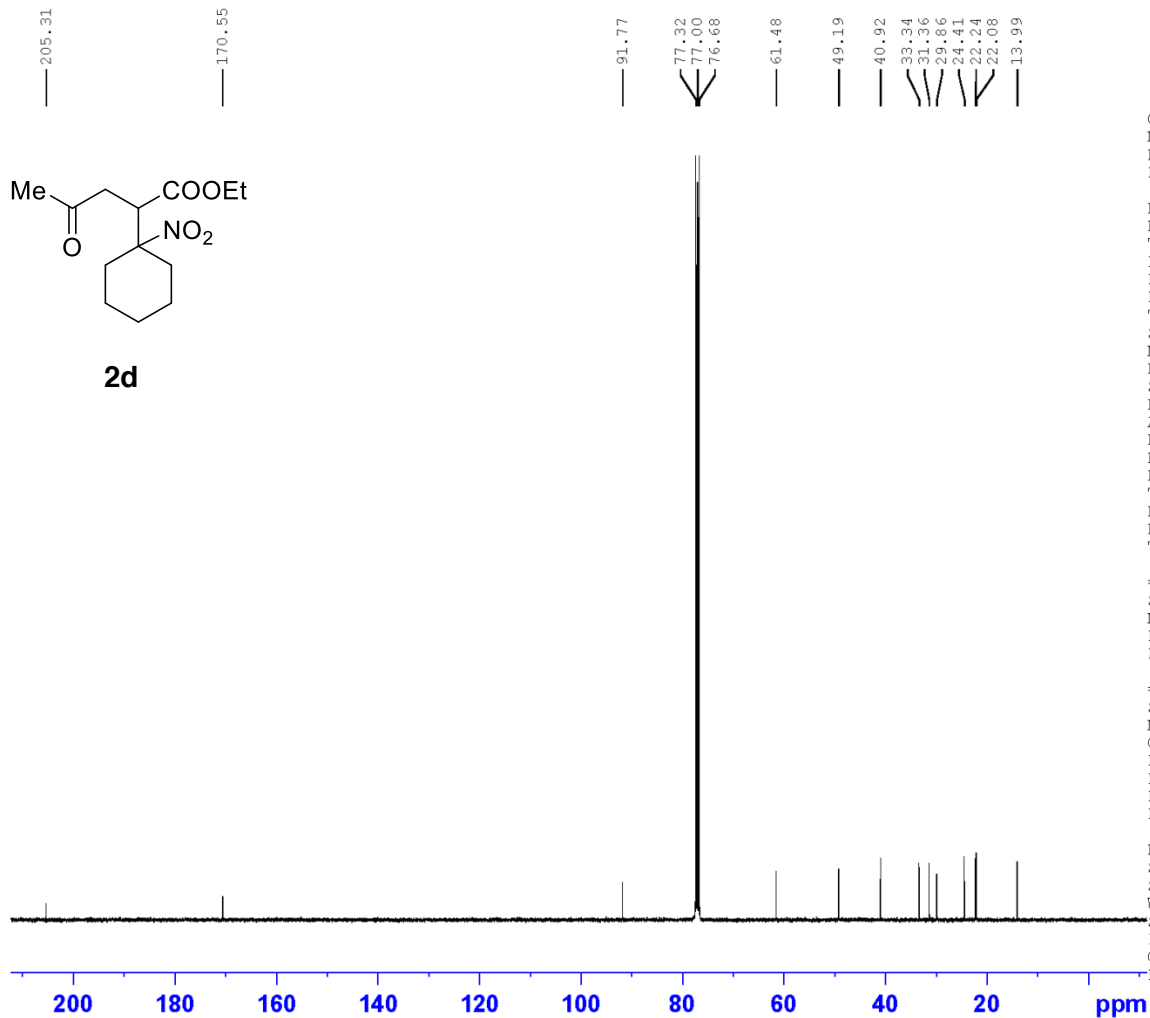



Current Data Parameters
 NAME yf-16-0222-394
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date 20160222
 Time 14.41
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 77.81
 DW 62.400 usec
 DE 6.50 usec
 TE 300.3 K
 D1 1.00000000 sec
 TD0 1

=====
 CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 FC 1.00



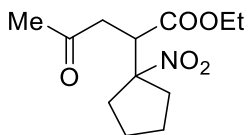
Current Data Parameters
 NAME yf-16-0223-394c
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160224
 Time_ 3.07
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 4096
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 302.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

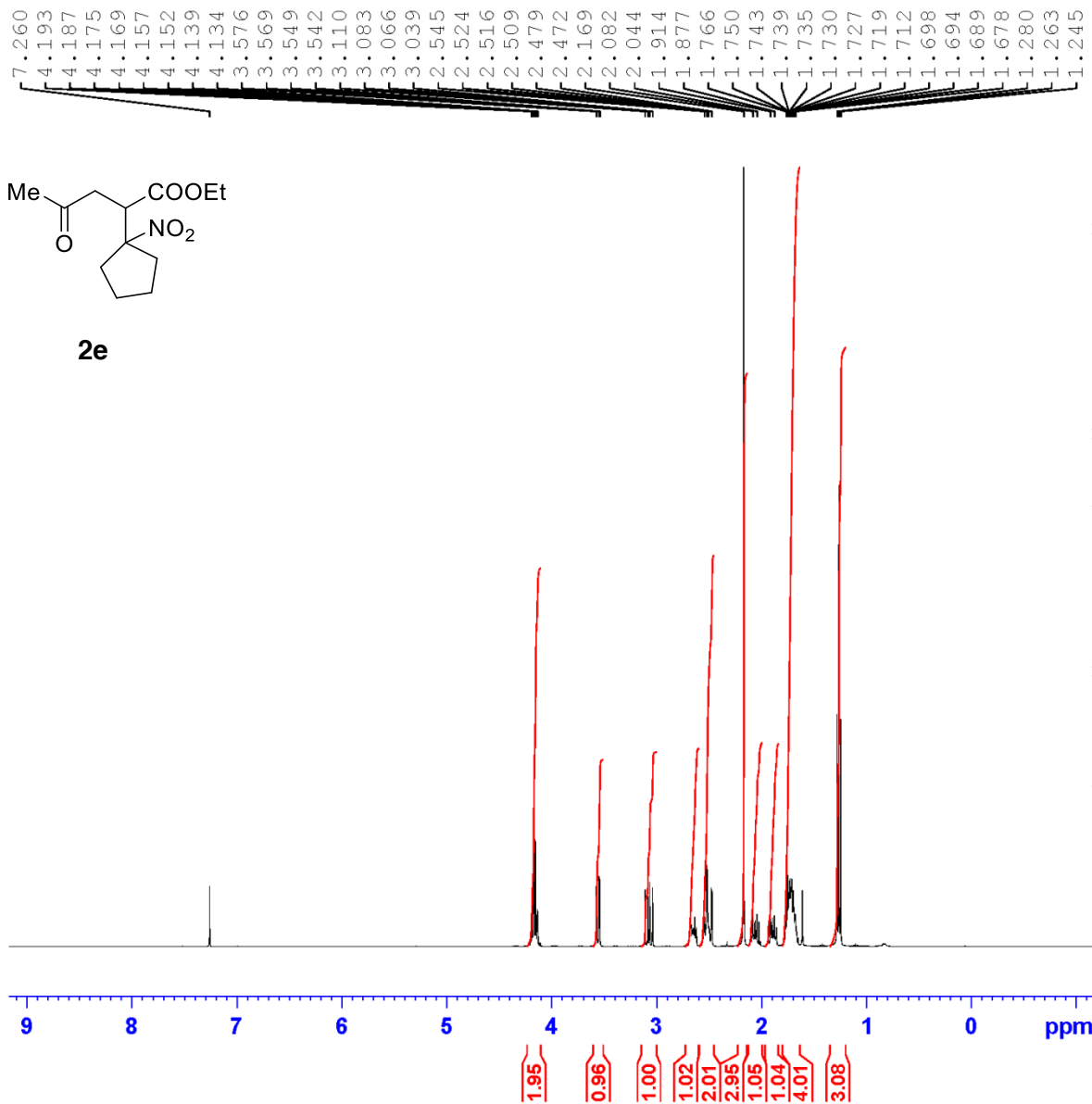
===== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 8.00000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127669 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



2e



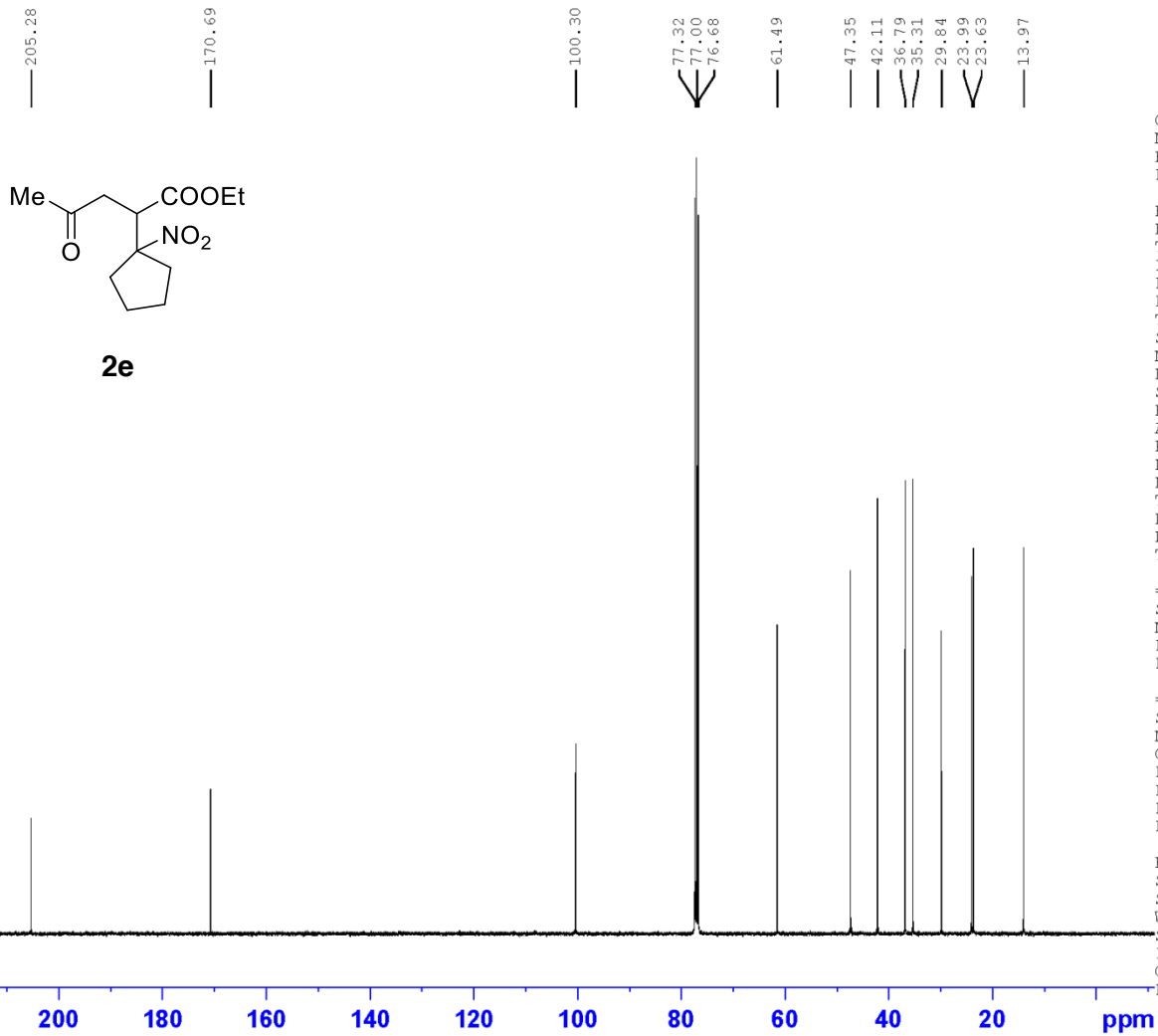
```

Current Data Parameters
NAME      yf-16-0222-400
EXPNO     10
PROCNO    1

F2 - Acquisition Parameters
Date_     20160222
Time      14.45
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDC13
NS         16
DS         2
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894465 sec
RG         31.13
DW         62.400 usec
DE         6.50 usec
TE         300.2 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
SF01      400.1324710 MHz
NUC1       1H
P1         15.00 usec
PLW1       8.00000000 W

F2 - Processing parameters
SI         65536
SF         400.1300094 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



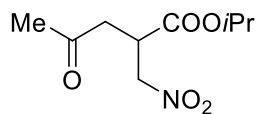
Current Data Parameters
 NAME yf-16-0226-400c
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160227
 Time_ 2.16
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 4096
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 302.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

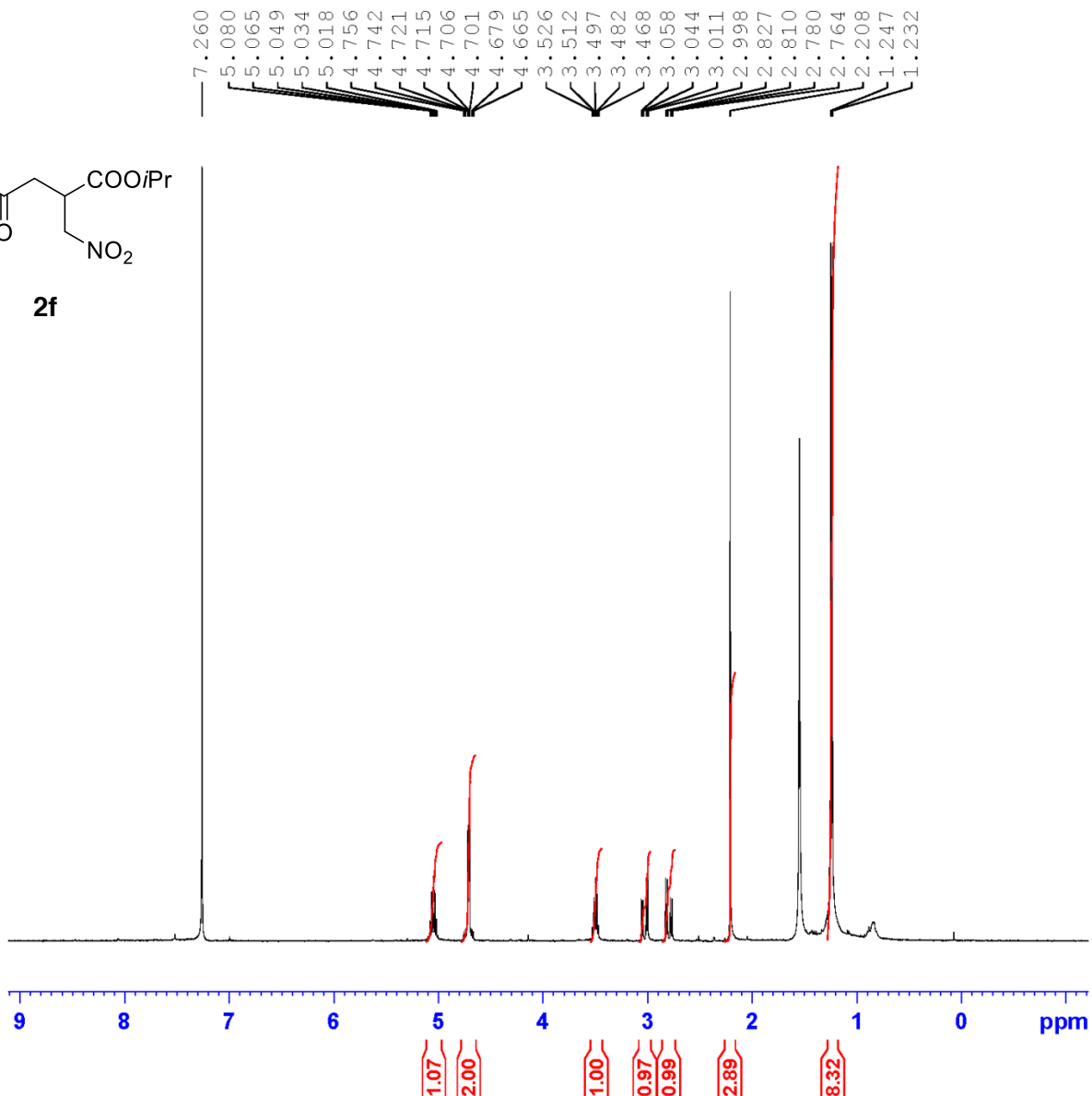
==== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 8.00000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127691 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



2f

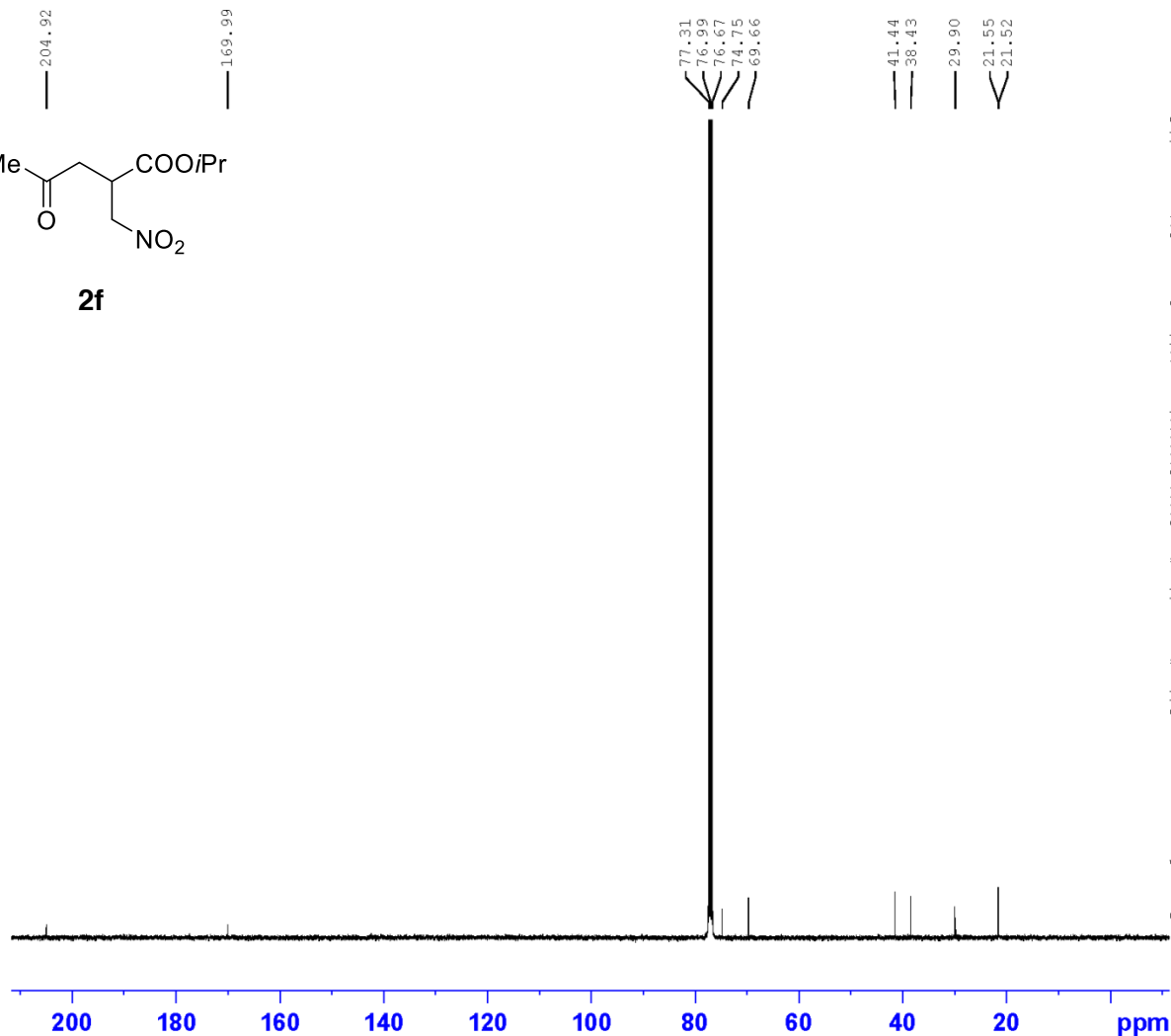
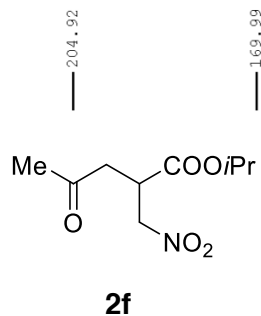


Current Data Parameters
 NAME YF-16-0401-463P-H
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160401
 Time_ 14.42
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 88.94
 DW 62.400 usec
 DE 6.50 usec
 TE 300.3 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300096 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



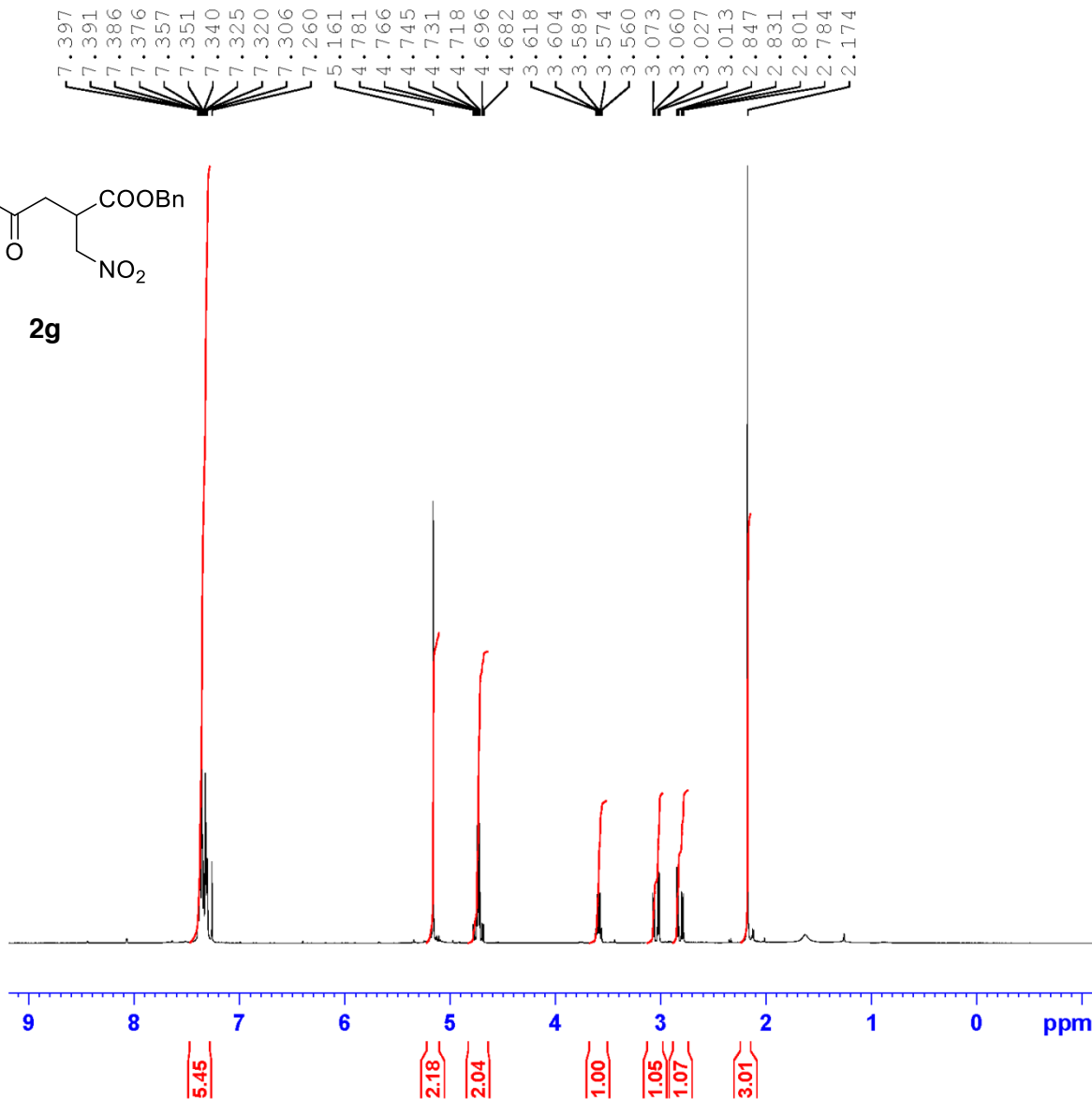
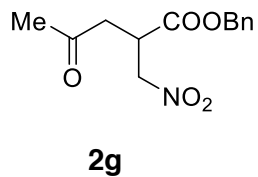
Current Data Parameters
 NAME YF-16-0401-463IPR-P-C
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160402
 Time_ 7.53
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 10000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 302.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 8.00000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127676 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

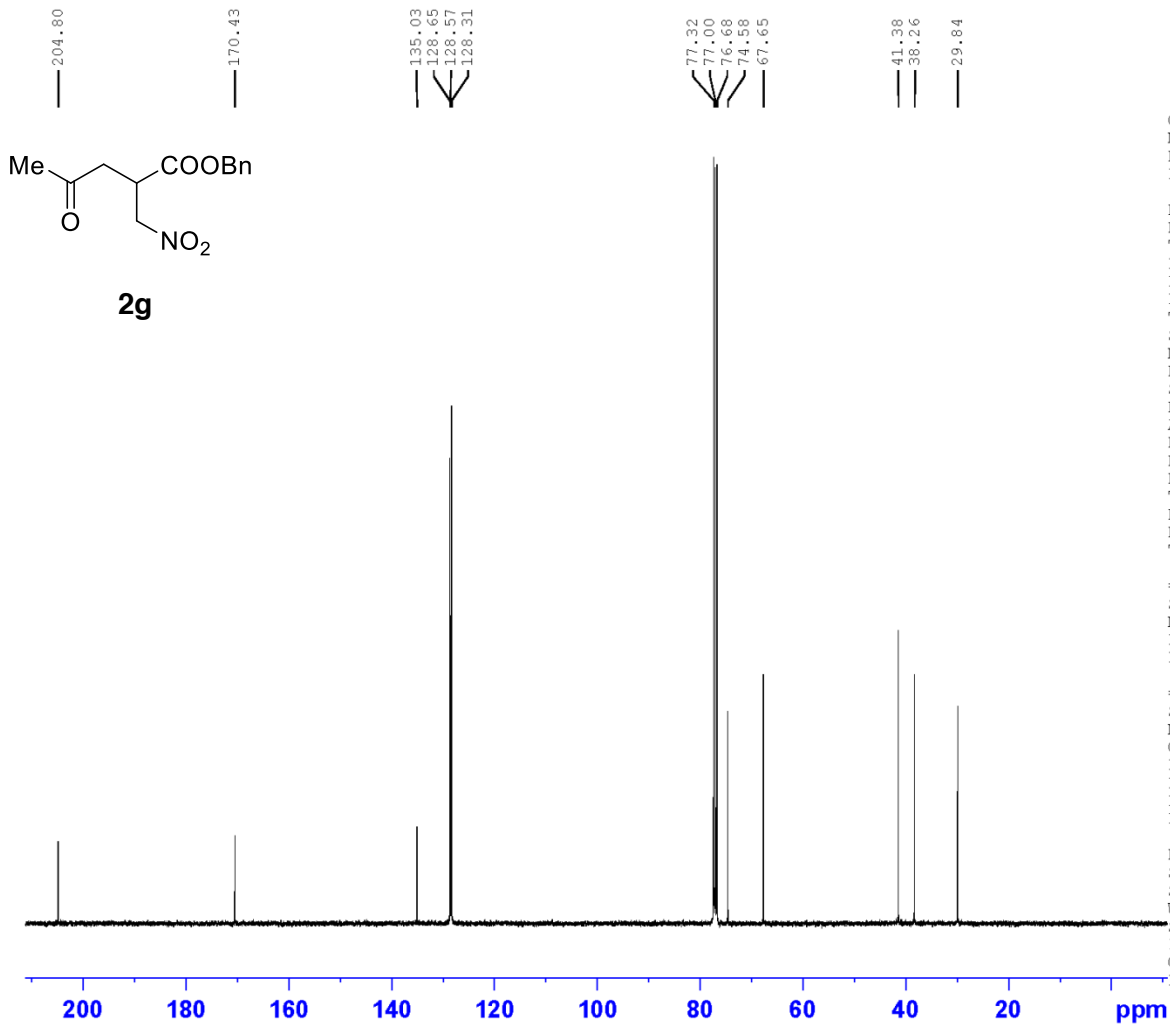


Current Data Parameters
 NAME YF-16-0401-BN-P-H
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160401
 Time_ 14.46
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 54.59
 DW 62.400 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300094 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



```

Current Data Parameters
NAME      yf-16-0402-BN-P-C
EXPNO     10
PROCNO    1

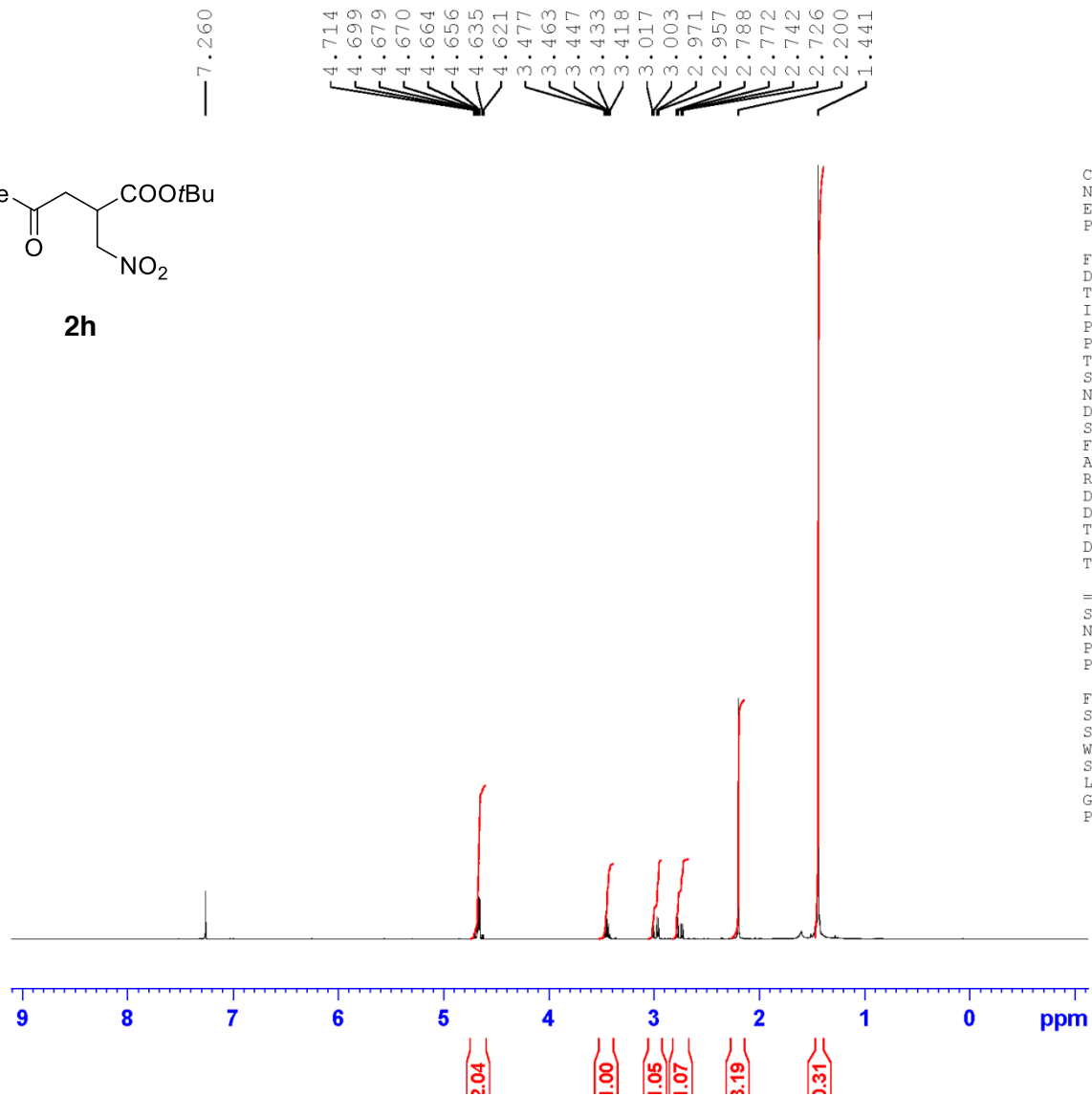
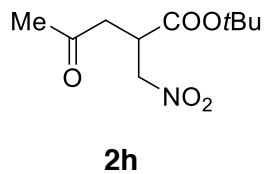
F2 - Acquisition Parameters
Date_     20160402
Time      20.13
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         3000
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631488 sec
RG         195.88
DW         20.800 usec
DE         6.50 usec
TE         302.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      100.6228293 MHz
NUC1       13C
P1         10.00 usec
PLW1       70.00000000 W

===== CHANNEL f2 =====
SFO2      400.1316005 MHz
NUC2       1H
CPDPRG[2] waltz16
PCPD2      80.00 usec
PLW2       8.00000000 W
PLW12      0.28125000 W
PLW13      0.28125000 W

F2 - Processing parameters
SI         32768
SF         100.6127691 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

```

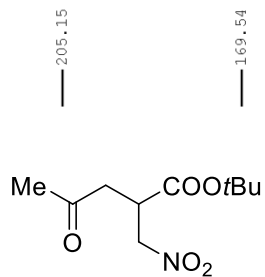



Current Data Parameters
 NAME yf-16-0222-494
 EXPNO 10
 PROCNO 1

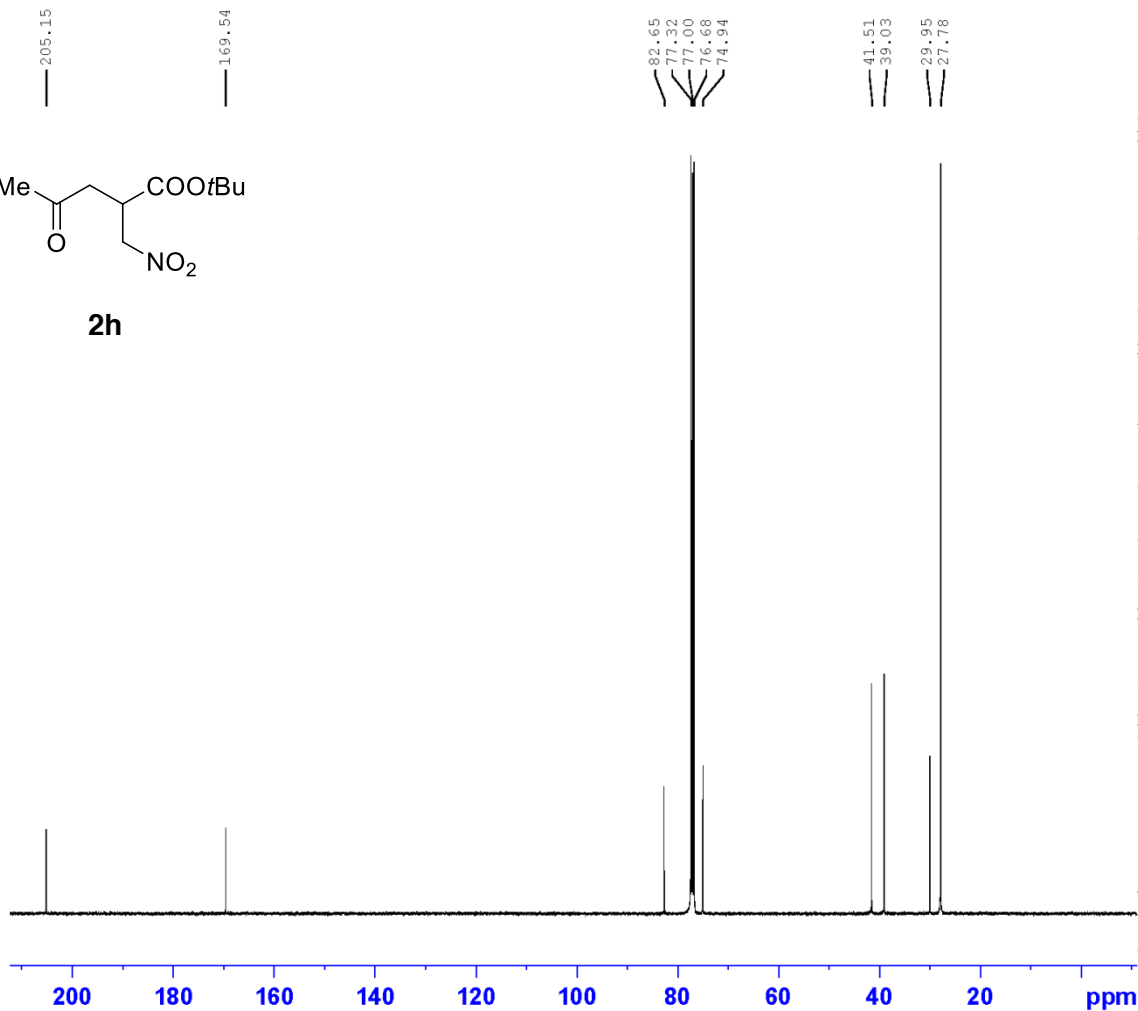
F2 - Acquisition Parameters
 Date_ 20160222
 Time_ 14.53
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.089465 sec
 RG 62.88
 DW 62.400 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



2h



Current Data Parameters
NAME YF-16-0815-494P-C
EXPNO 10
PROCNO 1

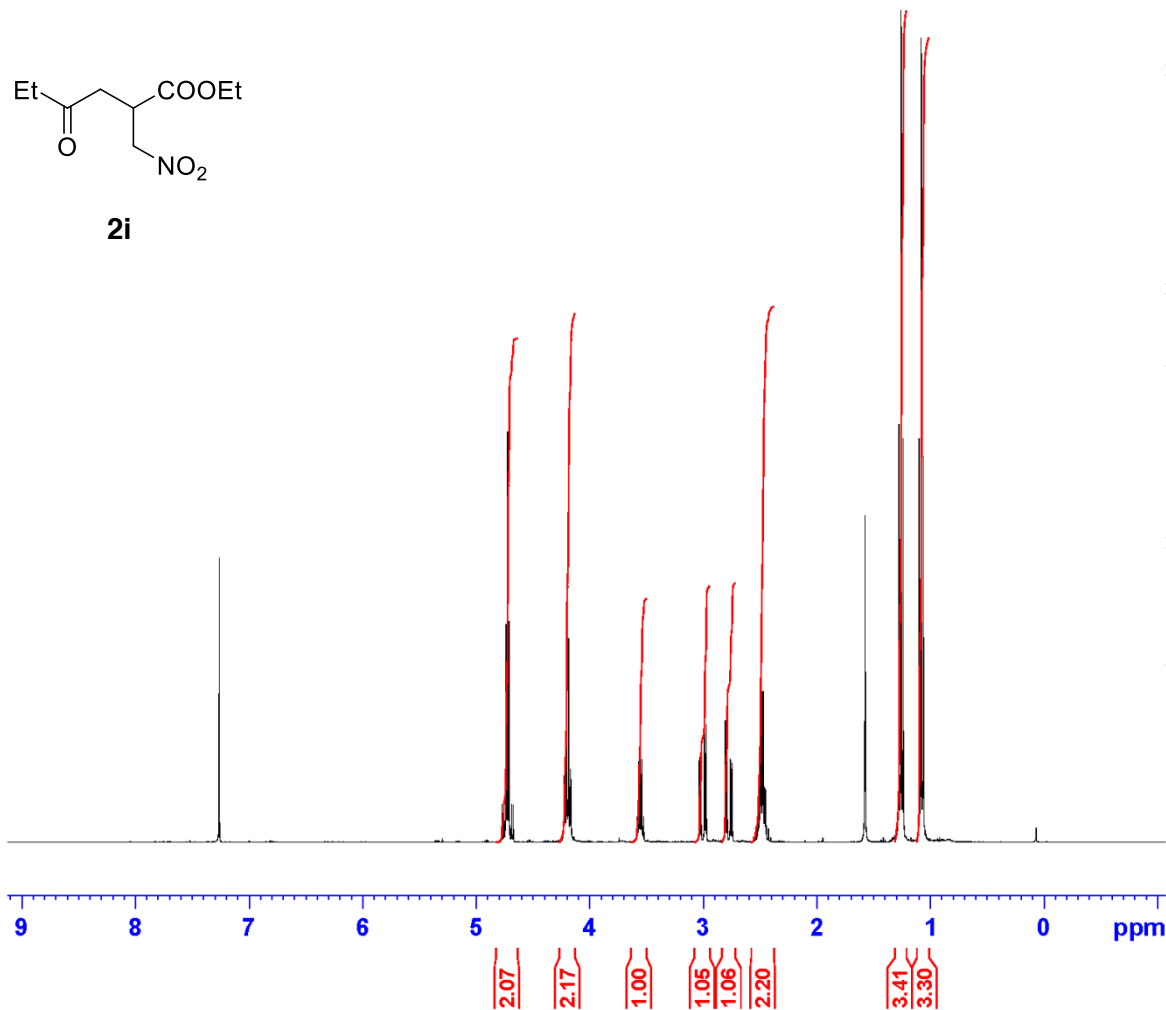
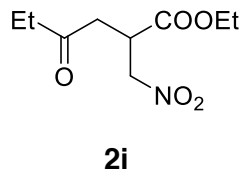
F2 - Acquisition Parameters
Date_ 20160816
Time 6.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 7000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 195.88
DW 20.800 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 8.00000000 W
PLW12 0.28125000 W
PLW13 0.28125000 W

F2 - Processing parameters
SI 32768
SF 100.6127706 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

7.260
4.772
4.757
4.736
4.722
4.708
4.686
4.673
4.222
4.219
4.213
4.204
4.201
4.186
4.184
4.168
4.166
3.582
3.568
3.553
3.538
3.523
3.033
3.019
2.987
2.973
2.805
2.789
2.760
2.743
2.513
2.504
2.494
2.486
2.476
2.468
2.457
2.450
1.273
1.256
1.238
1.096
1.078
1.060



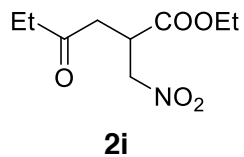
Current Data Parameters
NAME yf-16-0711-600p
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160711
Time_ 16.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.089465 sec
RG 71.01
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 1.0000000 sec
TD0 1

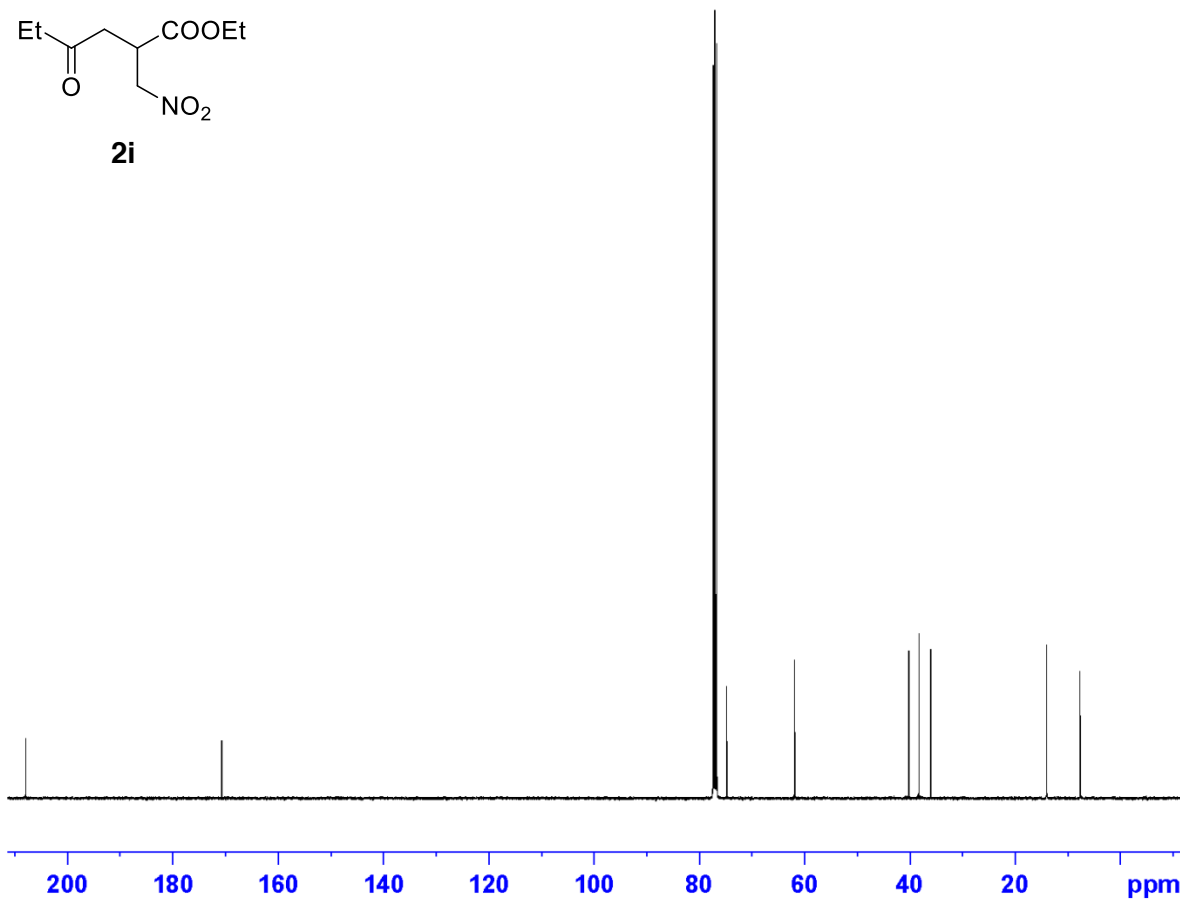
==== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

207.88
170.63



77.31
77.00
76.68
74.75
61.84
40.14
38.21
35.99
13.96
7.61



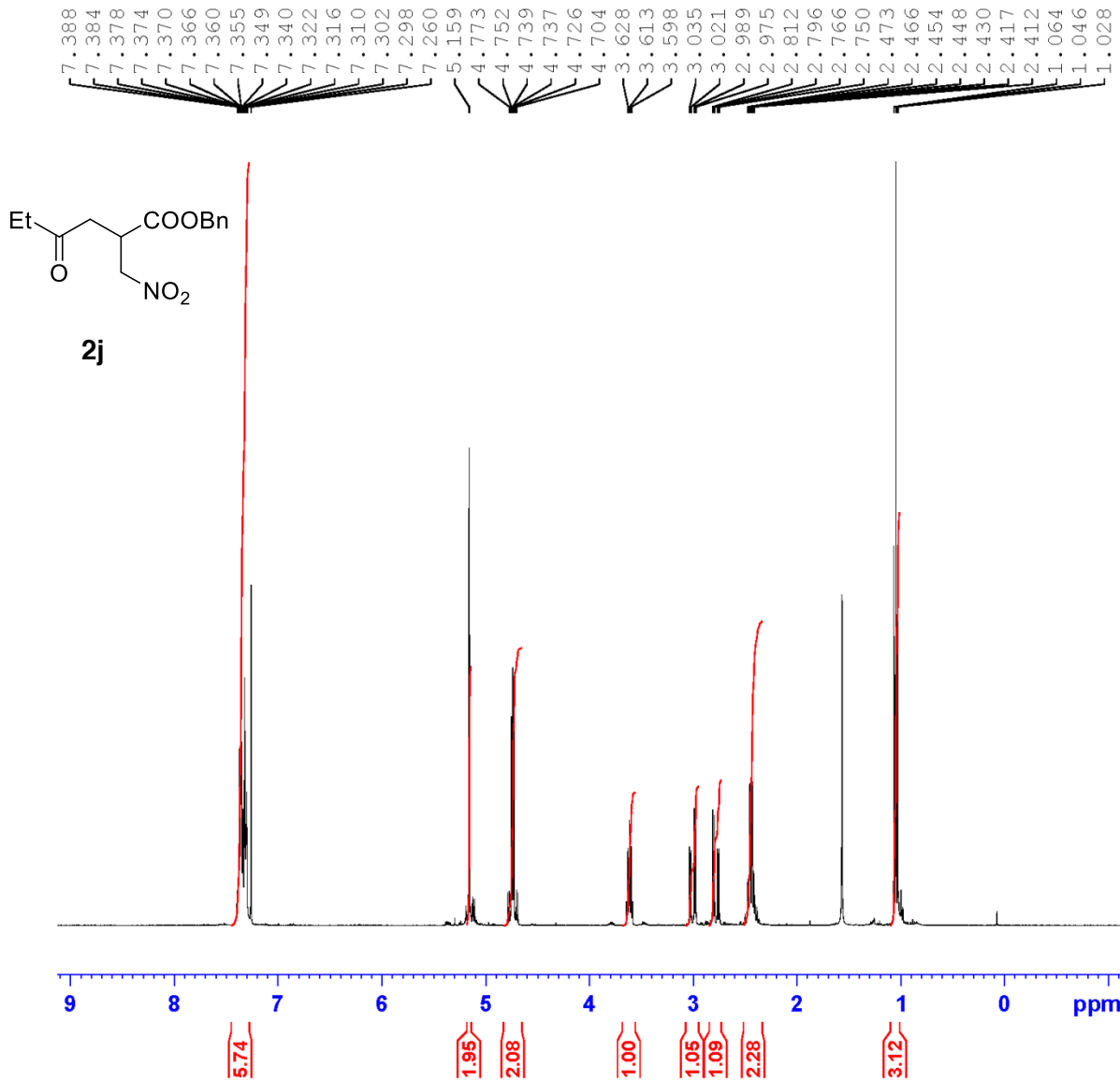
Current Data Parameters
NAME YF-16-0711-600P-C
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160712
Time_ 6.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 195.88
DW 20.800 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

==== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 8.00000000 W
PLW12 0.28125000 W
PLW13 0.28125000 W

F2 - Processing parameters
SI 32768
SF 100.6127706 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

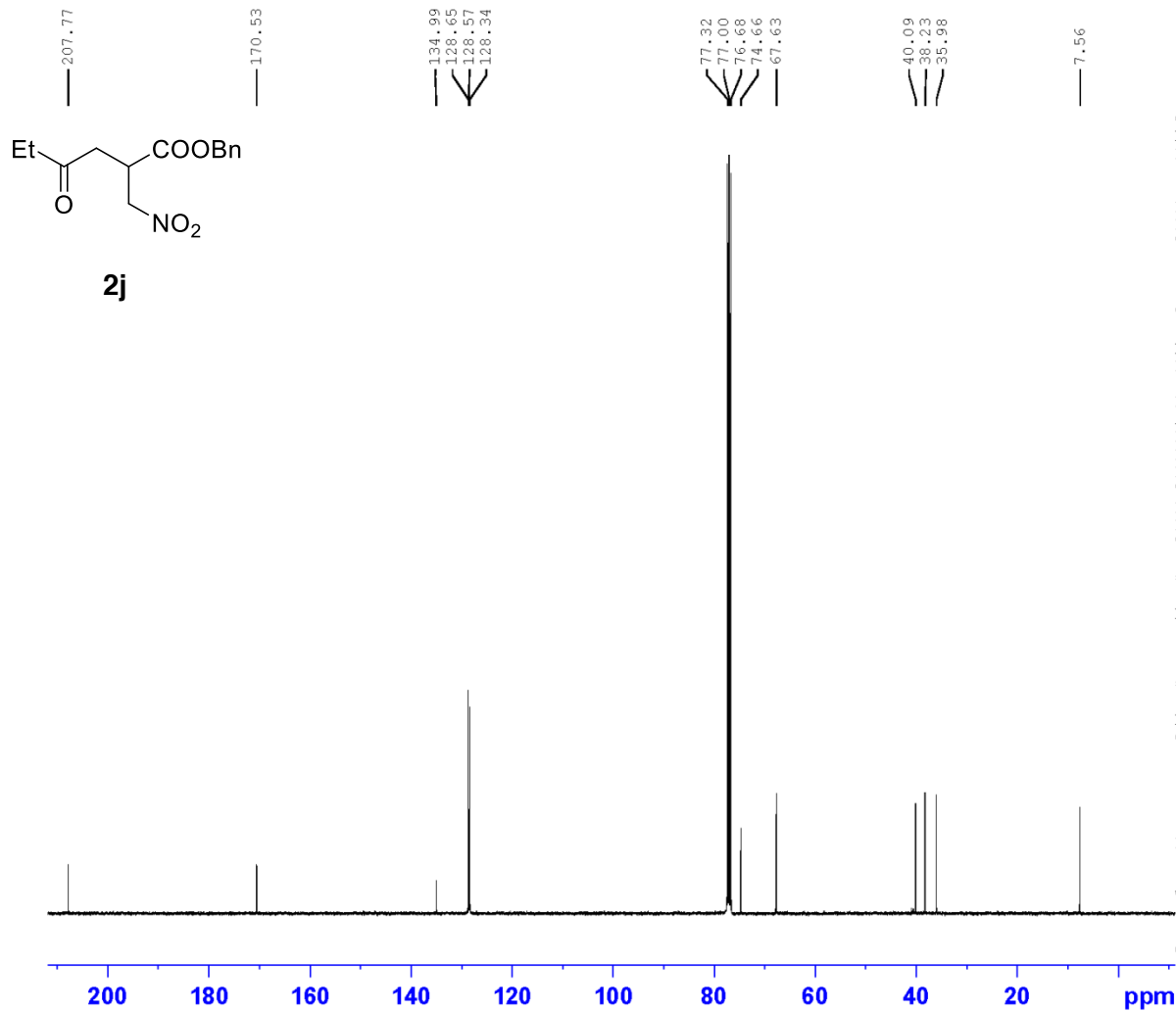


Current Data Parameters
 NAME YF-16-0720-607P
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160720
 Time_ 16.40
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 71.01
 DW 62.400 usec
 DE 6.50 usec
 TE 299.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



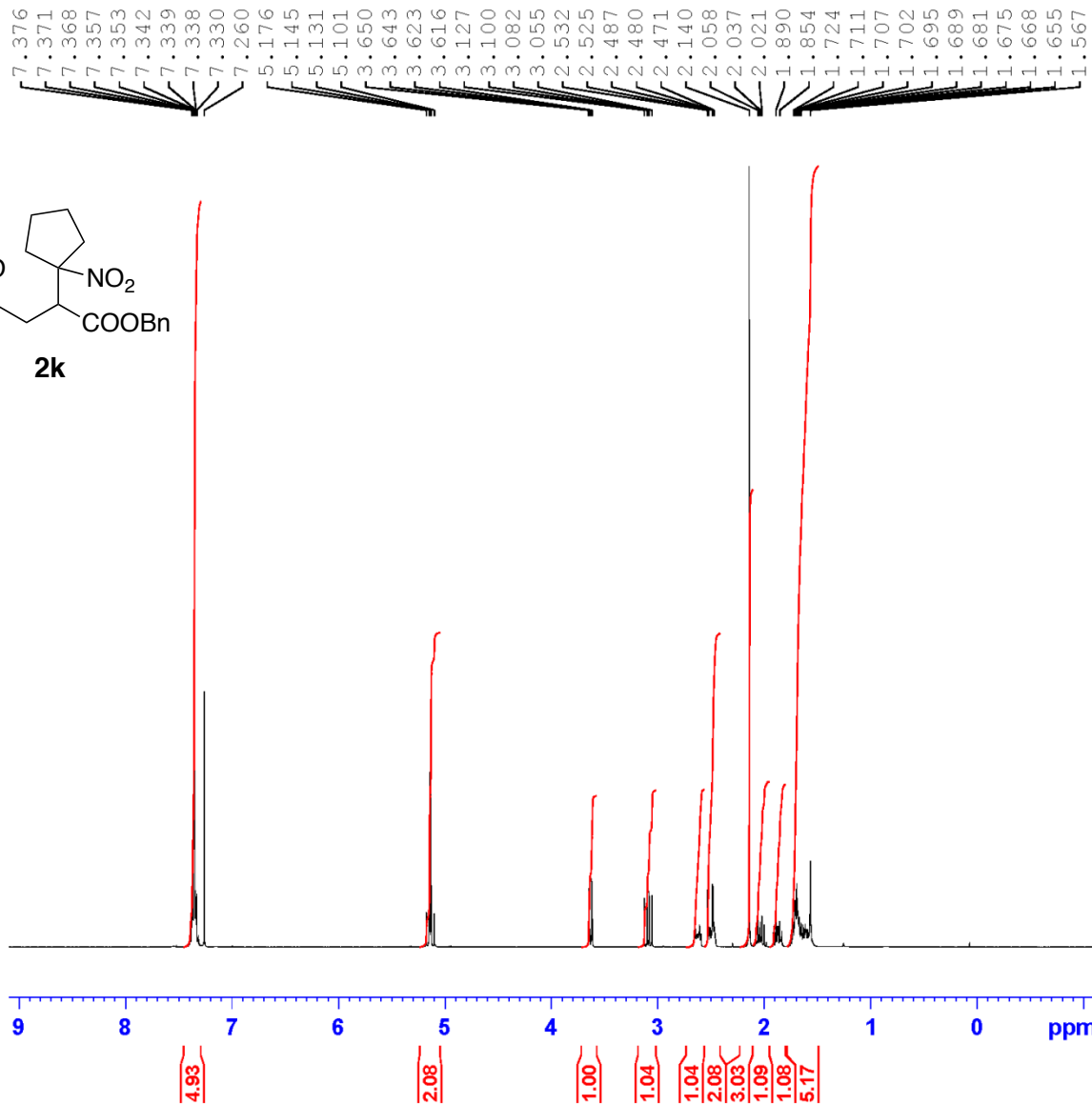
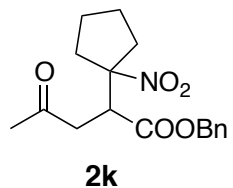
Current Data Parameters
 NAME YF-16-0727-607P-C
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date 20160728
 Time 3.59
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 6000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.9 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.0000000 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 FCPD2 80.00 usec
 PLW2 8.0000000 W
 PLW12 0.2812500 W
 PLW13 0.2812500 W

F2 - Processing parameters
 SI 32768
 SF 100.6127706 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



```

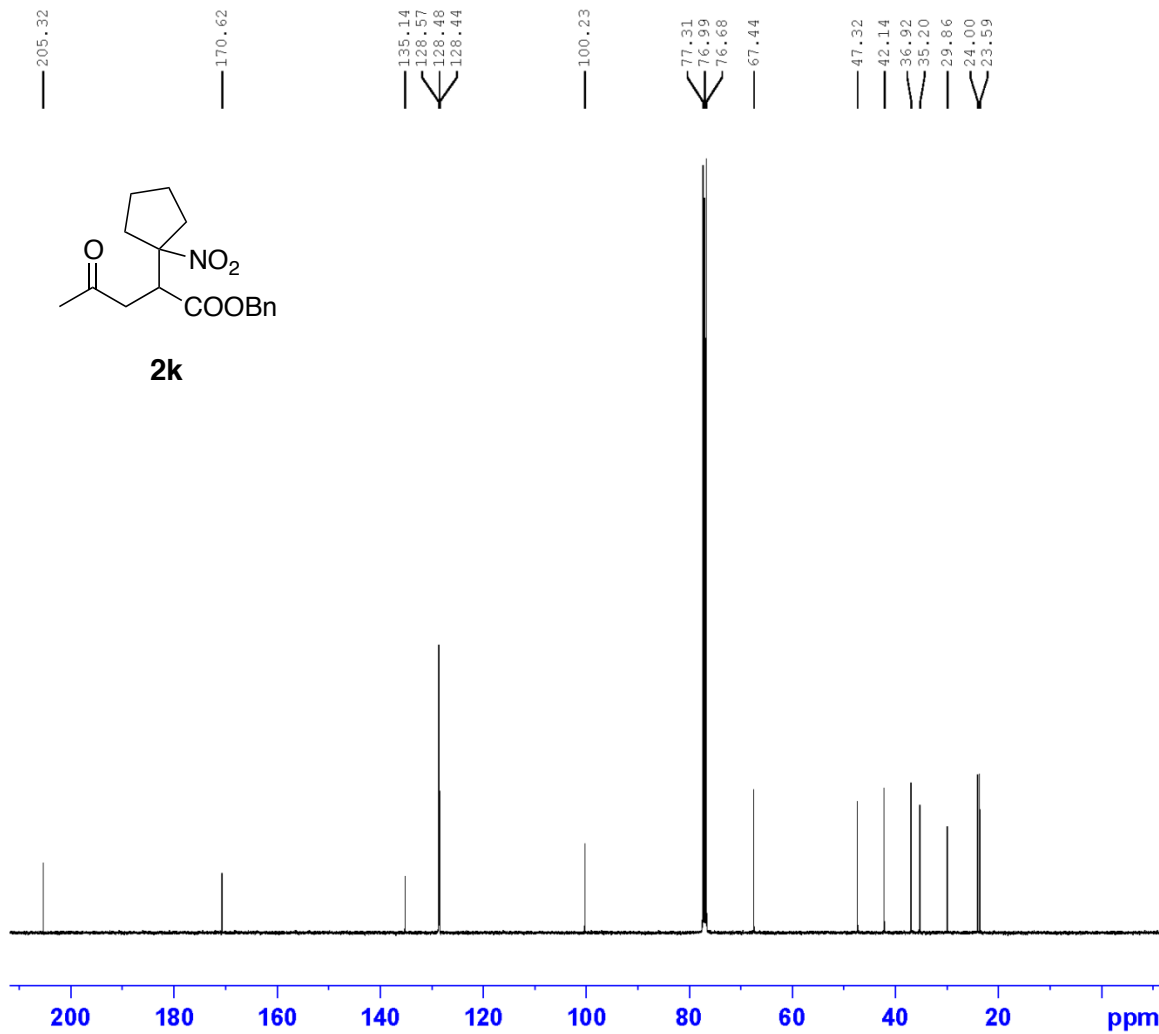
Current Data Parameters
NAME      YF-17-0105-701P
EXPNO     10
PROCNO    1

F2 - Acquisition Parameters
Date_     20170105
Time      11.25
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894465 sec
RG         71.01
DW         62.400 usec
DE         6.50 usec
TE         298.5 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      400.1324710 MHz
NUC1       1H
P1         15.00 usec
PLW1       7.50000000 W

F2 - Processing parameters
SI         65536
SF         400.1300095 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

```



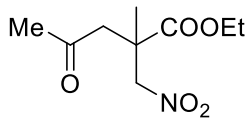
Current Data Parameters
 NAME yf-17-0105-701p-c
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170106
 Time_ 3.45
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 6000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.5 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

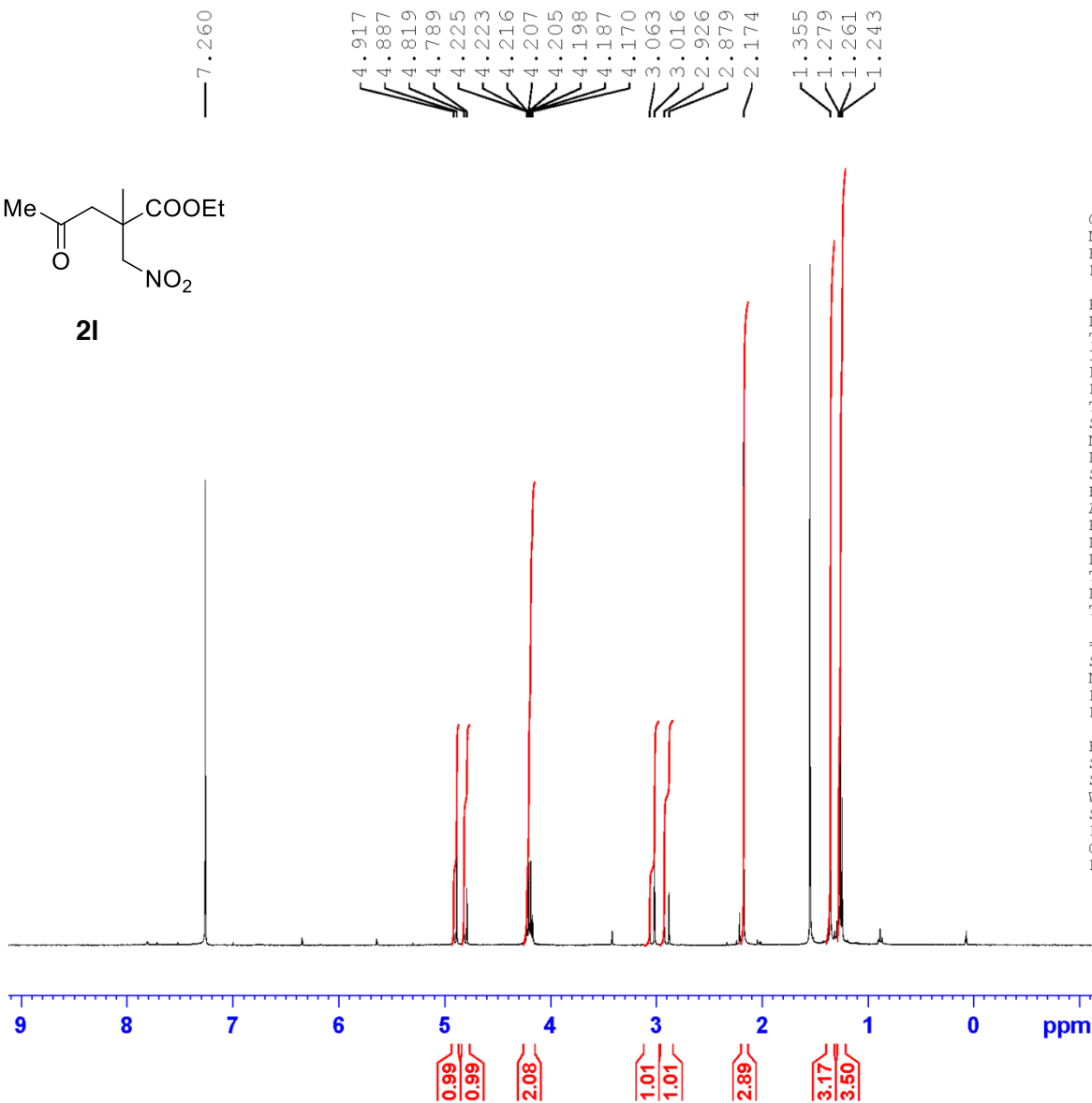
===== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 72.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 7.50000000 W
 PLW12 0.26367000 W
 PLW13 0.16633999 W

F2 - Processing parameters
 SI 32768
 SF 100.6127713 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



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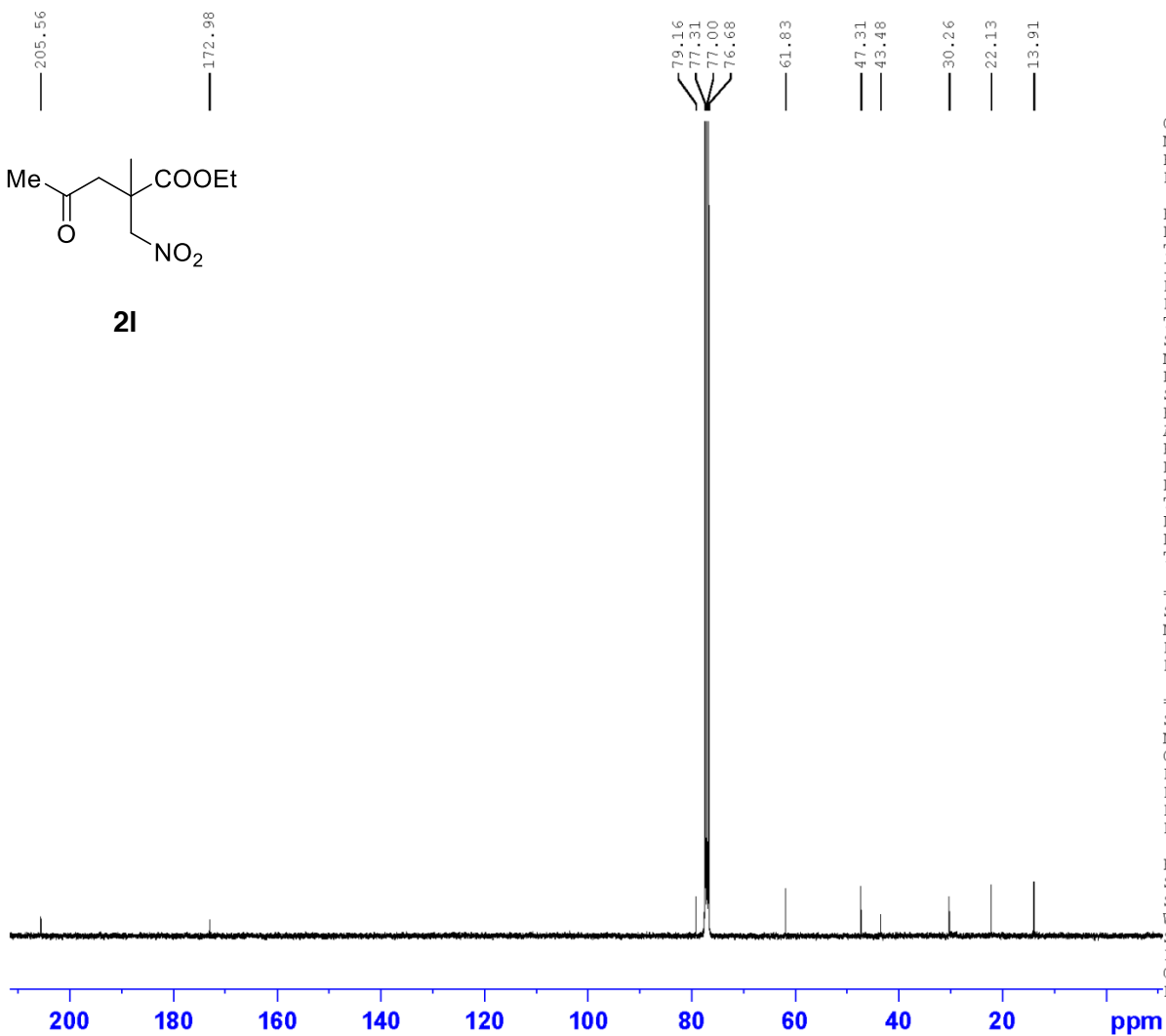
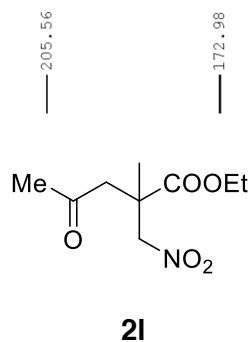


Current Data Parameters
 NAME yf-16-0405-527p-2
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160405
 Time_ 22.59
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 88.94
 DW 62.400 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



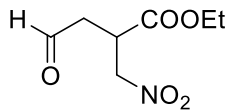
Current Data Parameters
 NAME yf-16-0406-527p-c
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160407
 Time_ 5.12
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 7000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 302.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

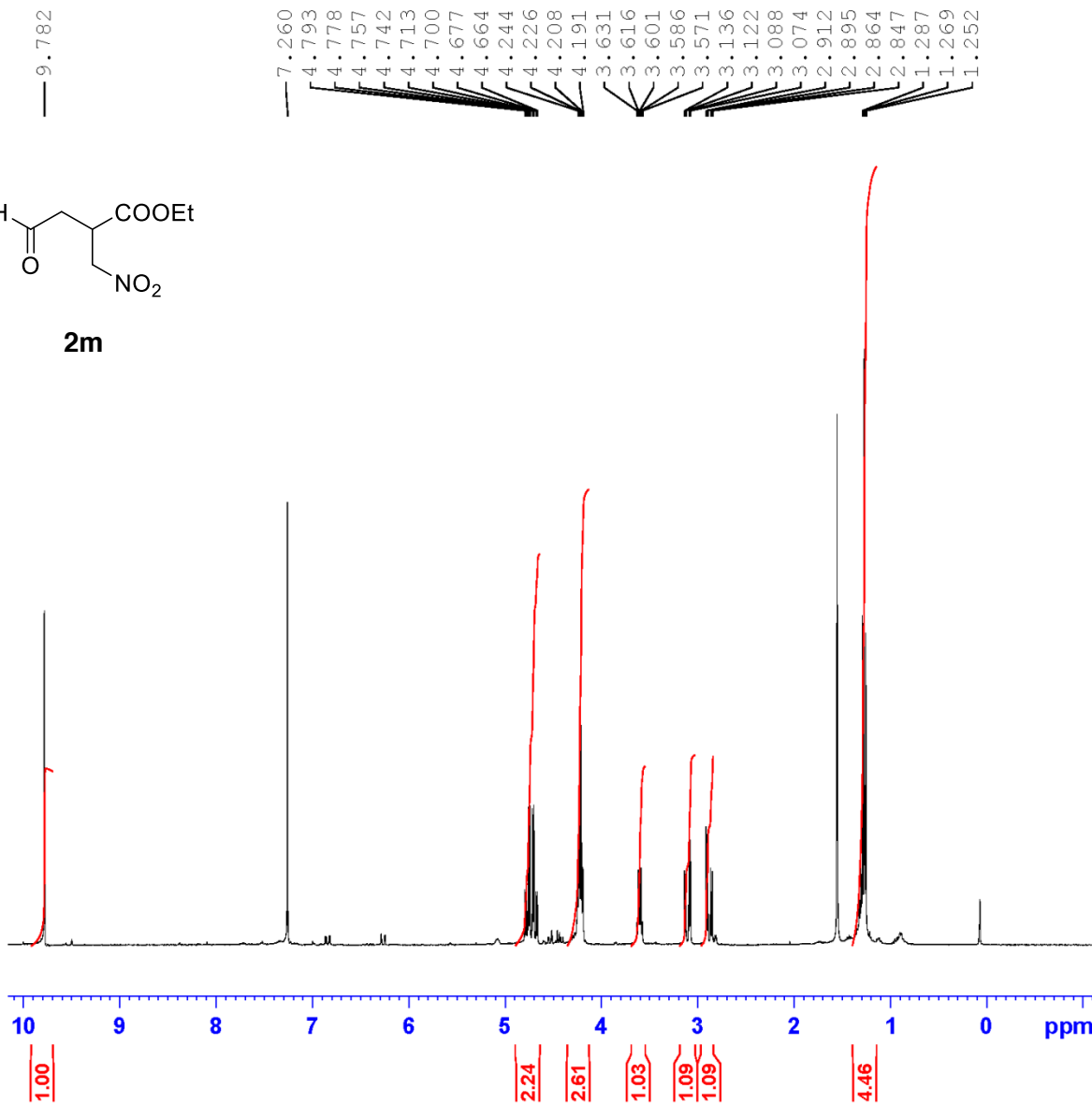
===== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 8.00000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127669 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



2m

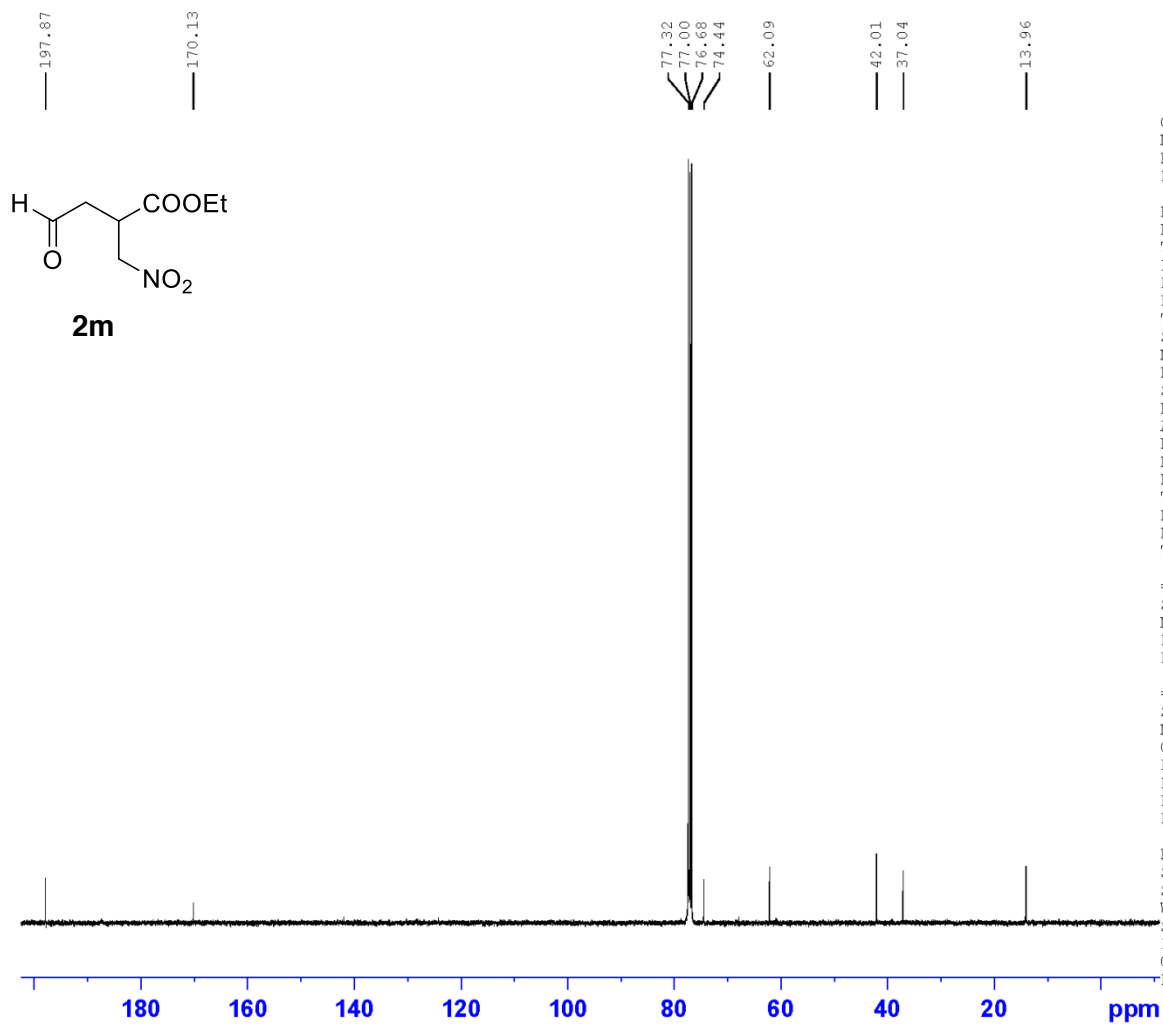


Current Data Parameters
 NAME yf-16-0403-525p-h
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160403
 Time_ 18.35
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 77.81
 DW 62.400 usec
 DE 6.50 usec
 TE 300.3 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300096 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



```

Current Data Parameters
NAME      yf-16-0403-525p-c
EXPNO     10
PROCNO    1

F2 - Acquisition Parameters
Date_     20160403
Time      21.33
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         3000
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631488 sec
RG         195.88
DW         20.800 usec
DE         6.50 usec
TE         302.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

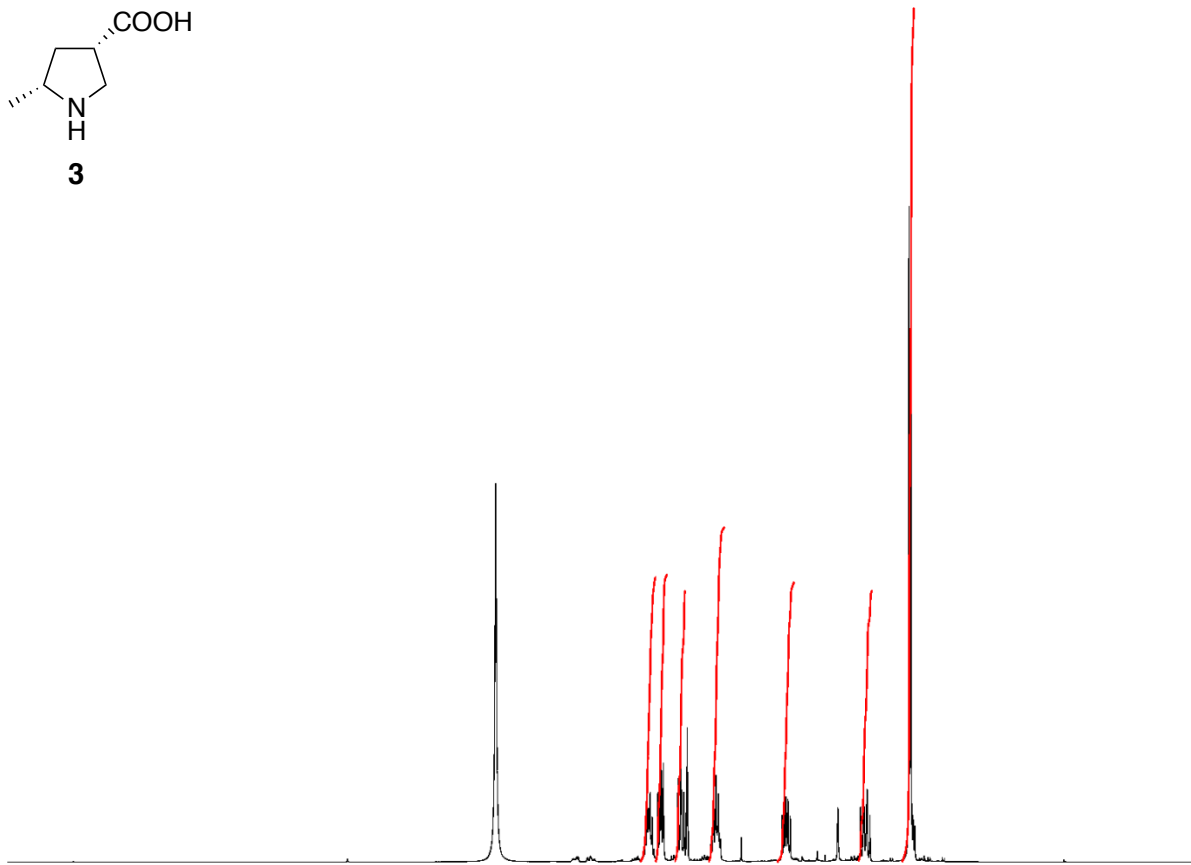
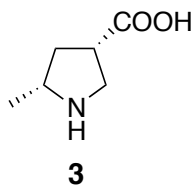
===== CHANNEL f1 =====
SFO1      100.6228293 MHz
NUC1       13C
P1         10.00 usec
PLW1       70.00000000 W

===== CHANNEL f2 =====
SFO2      400.1316005 MHz
NUC2       1H
CPDPRG[2] waltz16
PCPD2      80.00 usec
PLW2       8.00000000 W
PLW12      0.28125000 W
PLW13      0.28125000 W

F2 - Processing parameters
SI         32768
SF         100.6127669 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

```

4.941
 3.667
 3.657
 3.650
 3.642
 3.634
 3.625
 3.618
 3.609
 3.560
 3.542
 3.531
 3.513
 3.389
 3.367
 3.360
 3.338
 3.318
 3.314
 3.310
 3.306
 3.302
 3.107
 3.085
 3.065
 3.046
 3.031
 3.024
 2.501
 2.484
 2.481
 2.468
 2.465
 2.451
 2.448
 2.431
 1.833
 1.809
 1.800
 1.785
 1.777
 1.752
 1.420
 1.404



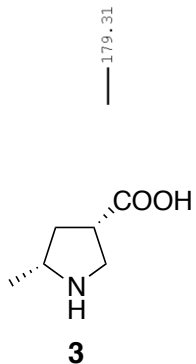
```

Current Data Parameters
NAME      yf-16-1110-688cp
EXPNO     10
PROCNO    1

F2 - Acquisition Parameters
Date_     20161110
Time_     15.23
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CD3OD_SPE
NS        16
DS        2
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ        4.0894465 sec
RG        31.13
DW        62.400 usec
DE        6.50 usec
TE        298.5 K
D1        1.00000000 sec
TD0       1

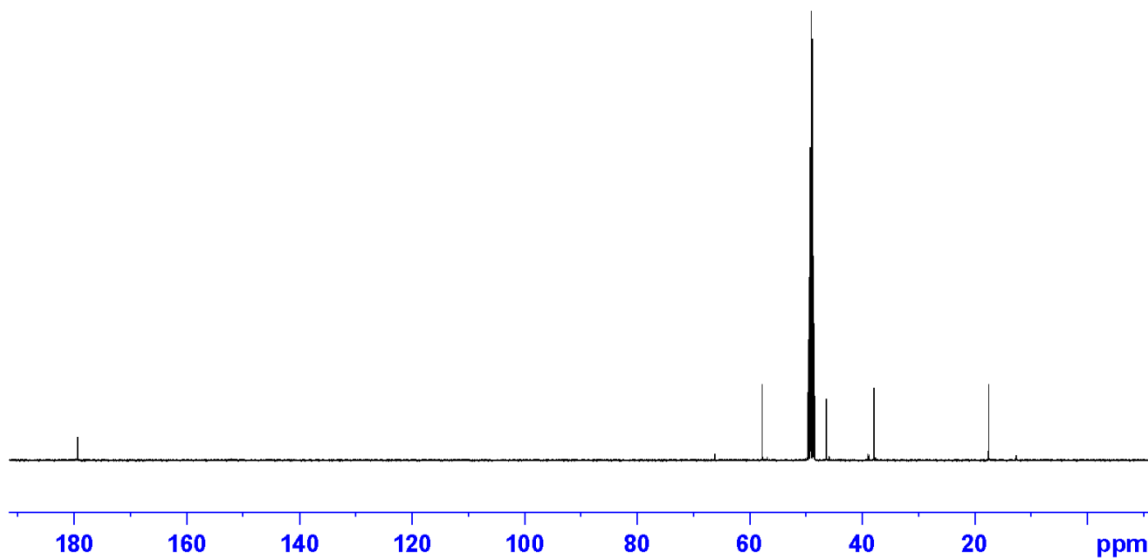
===== CHANNEL f1 =====
SFO1      400.1324710 MHz
NUC1      1H
P1        15.00 usec
PLW1      7.50000000 W

F2 - Processing parameters
SI        65536
SF        400.1300076 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



57.72
49.64
49.43
49.21
49.11
49.00
48.79
48.57
48.36
46.33
37.86

17.49



```

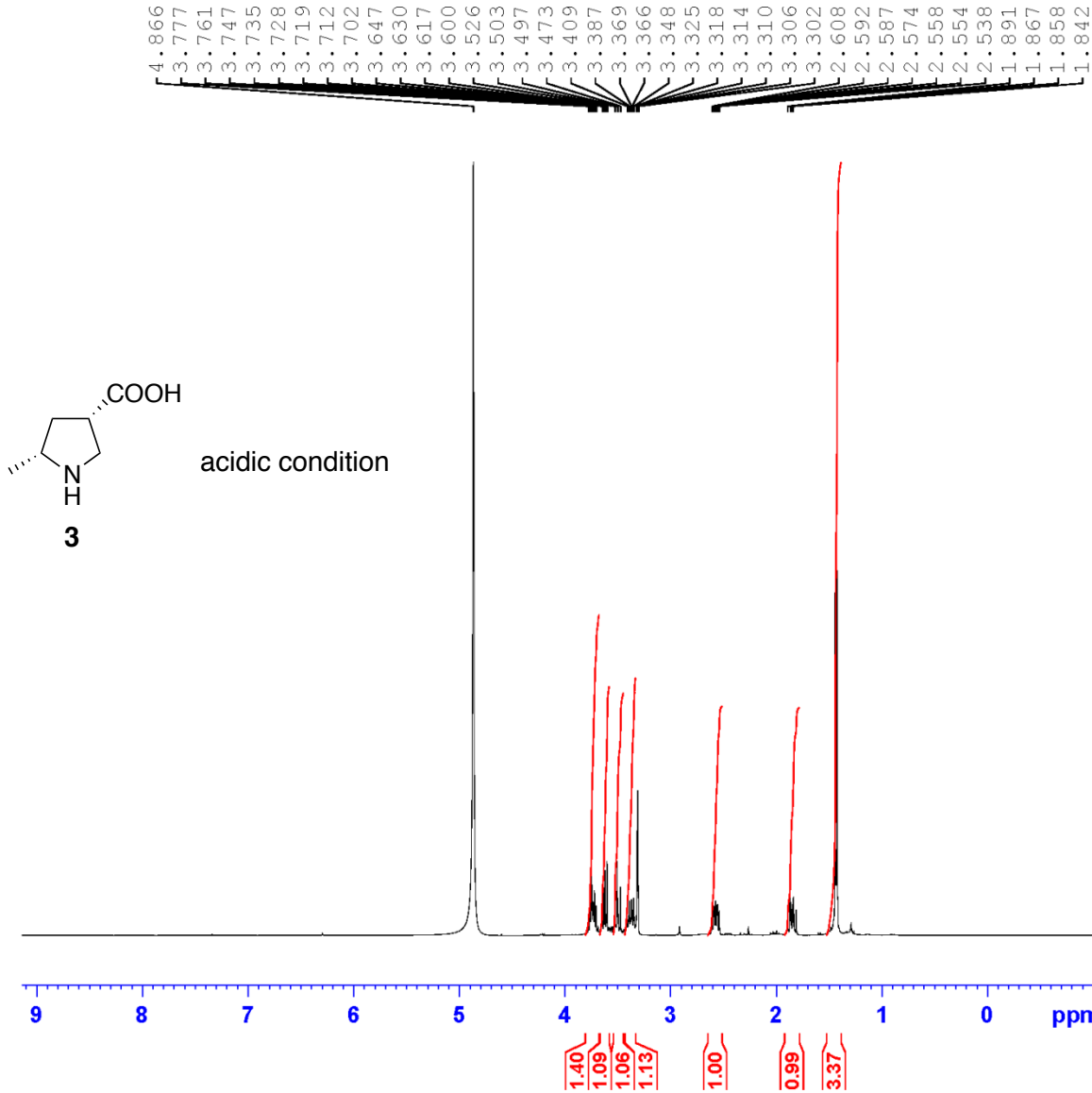
Current Data Parameters
NAME      yf-16-1110-688cp
EXPNO     20
PROCNO    1

F2 - Acquisition Parameters
Date_     20161110
Time      15.58
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CD3OD_SPE
NS         512
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631488 sec
RG         195.88
DW         20.800 usec
DE         6.50 usec
TE         299.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1       100.6228293 MHz
NUC1        13C
P1          10.00 usec
PLW1        72.00000000 W

===== CHANNEL f2 =====
SFO2       400.1316005 MHz
NUC2         1H
CPDPRG[2]   waltz16
PCPD2        80.00 usec
PLW2         7.50000000 W
PLW12        0.26367000 W
PLW13        0.16633999 W

F2 - Processing parameters
SI          32768
SF          100.6126284 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

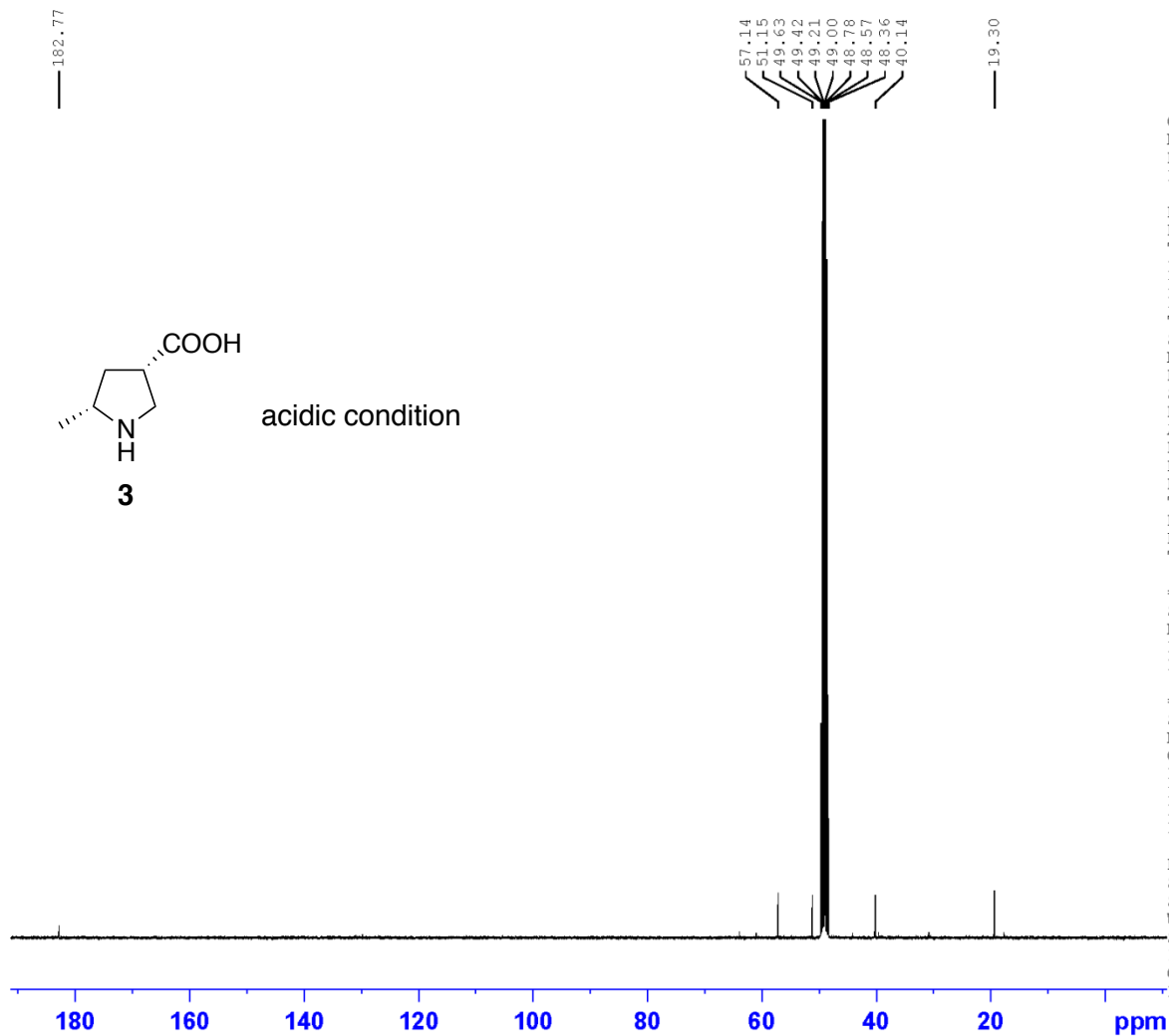


Current Data Parameters
 NAME YF-16-1109-689-1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161109
 Time_ 15.48
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CD3OD_SPE
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 62.88
 DW 62.400 usec
 DE 6.50 usec
 TE 298.6 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 7.50000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300074 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



```

Current Data Parameters
NAME      yf-16-0814-637-w
EXPNO     11
PROCNO    1

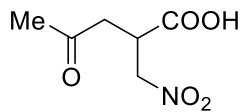
F2 - Acquisition Parameters
Date_     20160814
Time      21.56
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CD3OD_SPE
NS         3000
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631488 sec
RG         195.88
DW         20.800 usec
DE         6.50 usec
TE         299.9 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      100.6228293 MHz
NUC1       13C
P1         10.00 usec
PLW1       70.00000000 W

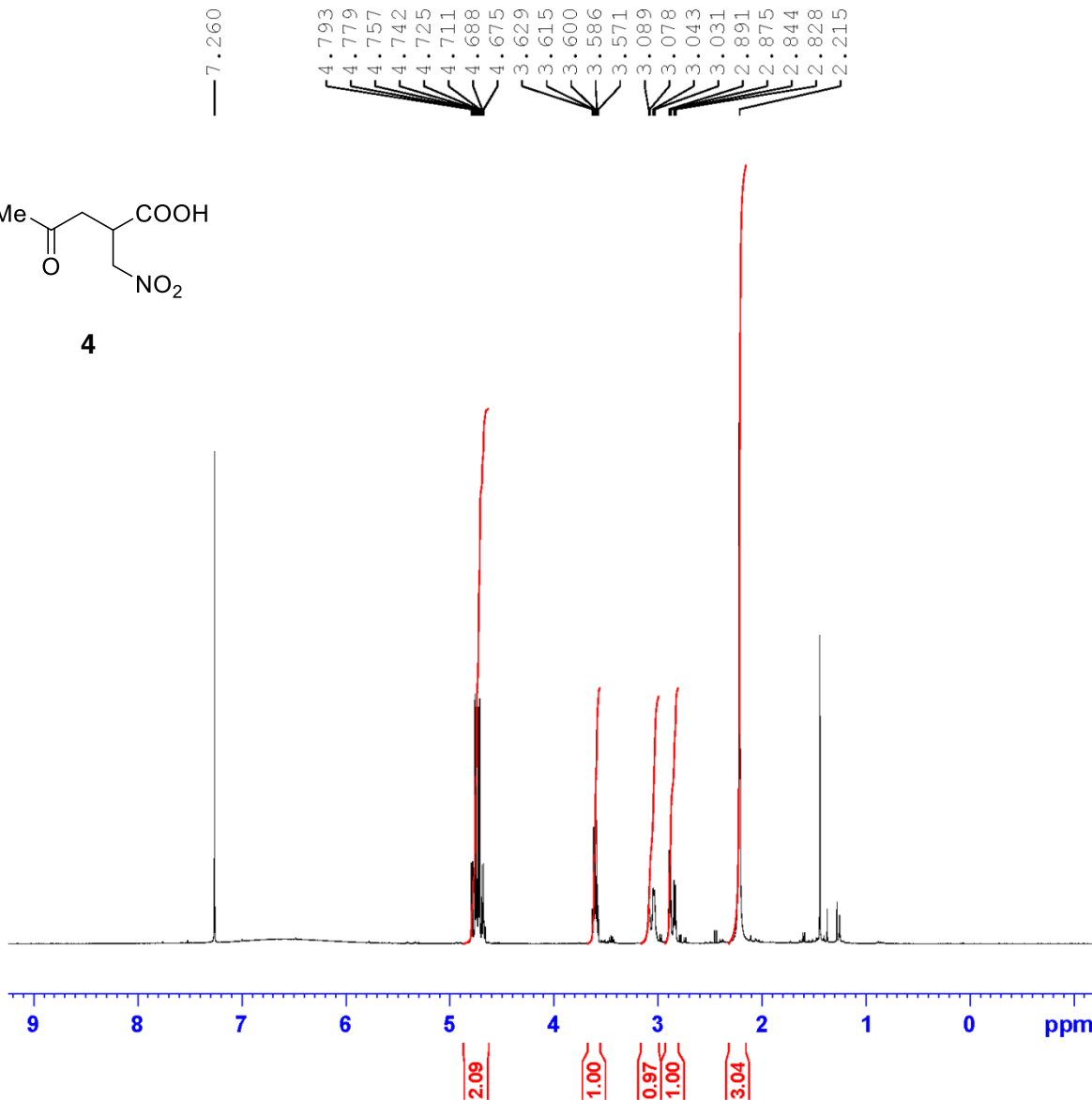
===== CHANNEL f2 =====
SFO2      400.1316005 MHz
NUC2       1H
CPDPRG[2] waltz16
PCPD2      80.00 usec
PLW2       8.00000000 W
PLW12      0.28125000 W
PLW13      0.28125000 W

F2 - Processing parameters
SI         32768
SF         100.6126277 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

```

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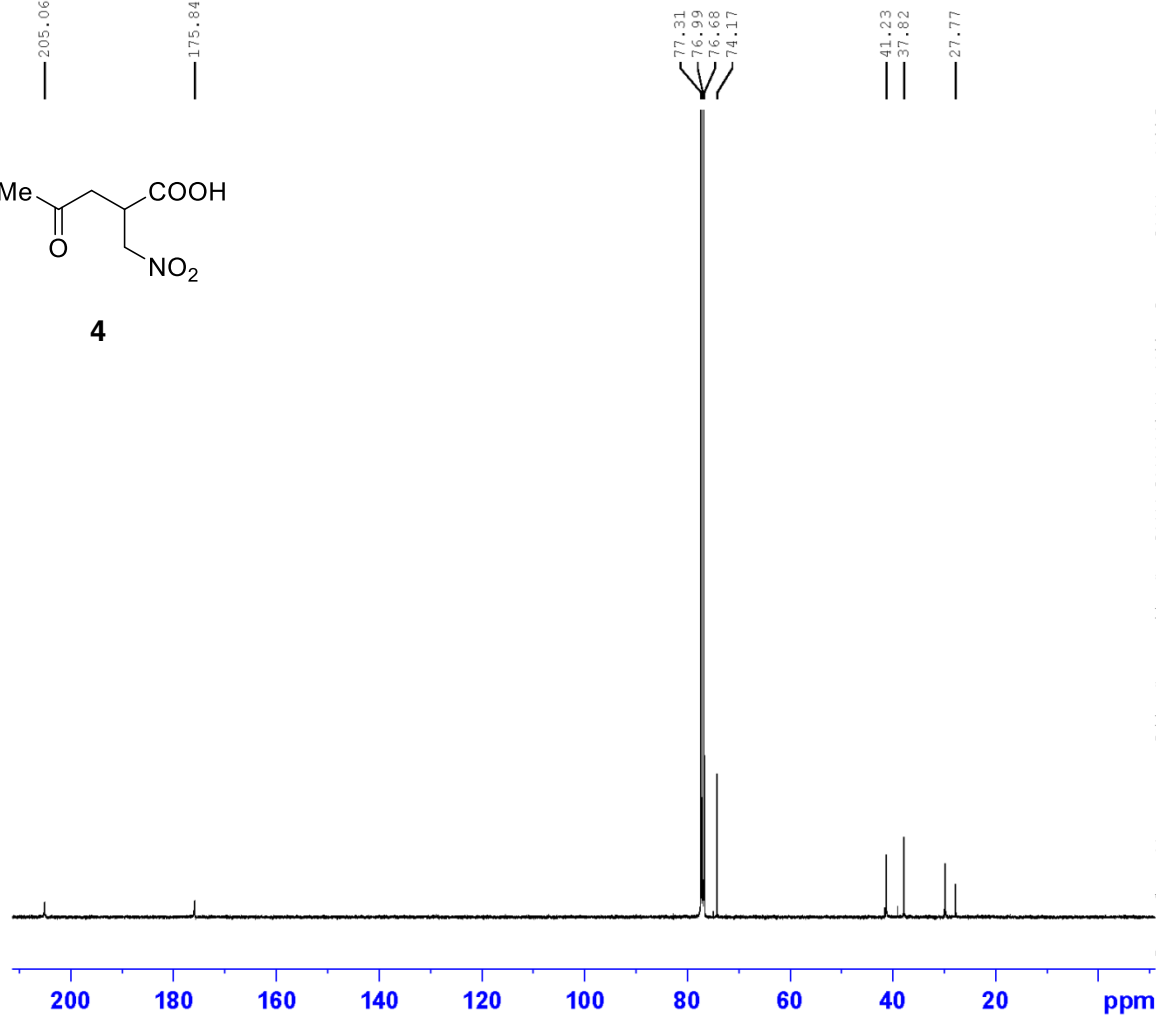
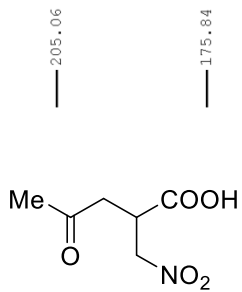


Current Data Parameters
 NAME YF-17-0112-678
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170112
 Time_ 22.33
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 77.81
 DW 62.400 usec
 DE 6.50 usec
 TE 298.7 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 7.50000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



```

Current Data Parameters
NAME      YF-17-0112-678-C
EXPNO     10
PROCNO    1

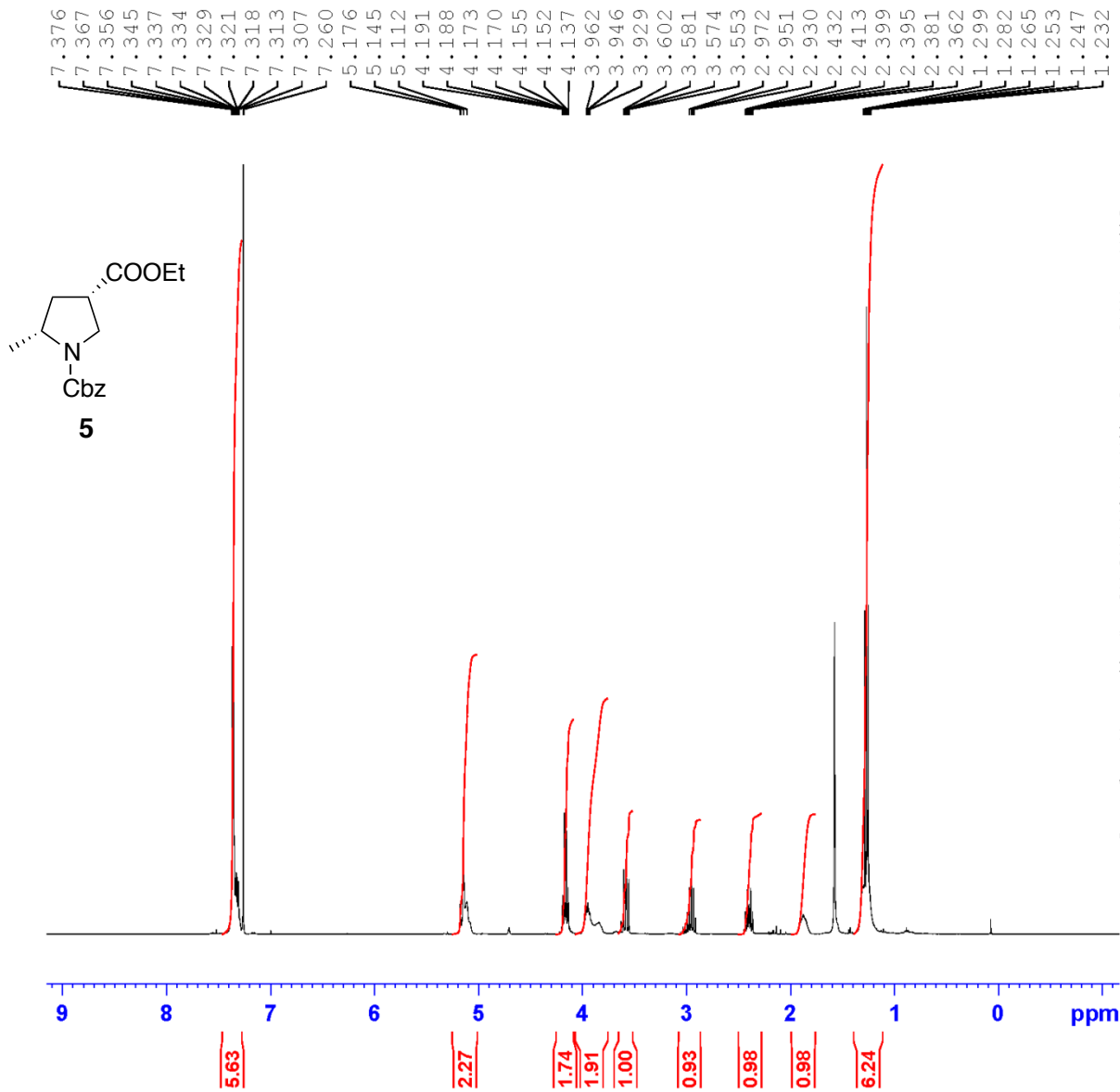
F2 - Acquisition Parameters
Date_     20170113
Time      6.28
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        8000
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631488 sec
RG        195.88
DW        20.800 usec
DE        6.50 usec
TE        299.4 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1

===== CHANNEL f1 =====
SFO1      100.6228293 MHz
NUC1      13C
P1        10.00 usec
PLW1      72.00000000 W

===== CHANNEL f2 =====
SFO2      400.1316005 MHz
NUC2      1H
CPDPRG[2] waltz16
PCPD2     80.00 usec
PLW2      7.50000000 W
PLW12     0.26367000 W
PLW13     0.16633999 W

F2 - Processing parameters
SI        32768
SF        100.6127713 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

```

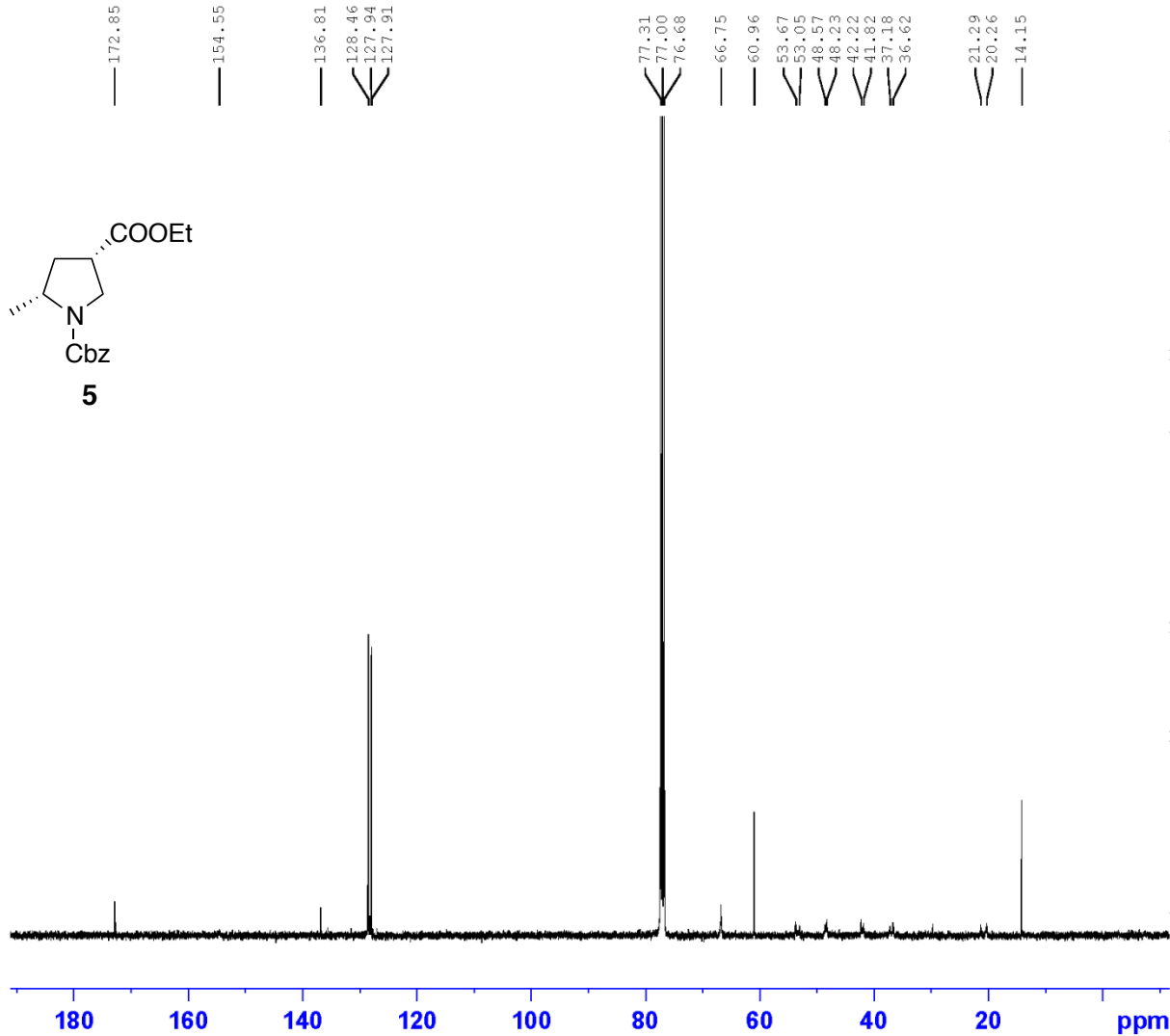


Current Data Parameters
 NAME YF-17-0115-698-1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170115
 Time_ 22.42
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 77.81
 DW 62.400 usec
 DE 6.50 usec
 TE 298.7 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 7.50000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



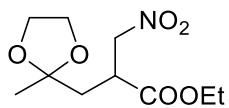
Current Data Parameters
 NAME YF-17-0115-698-1-C
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170116
 Time_ 6.56
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 8000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.3 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

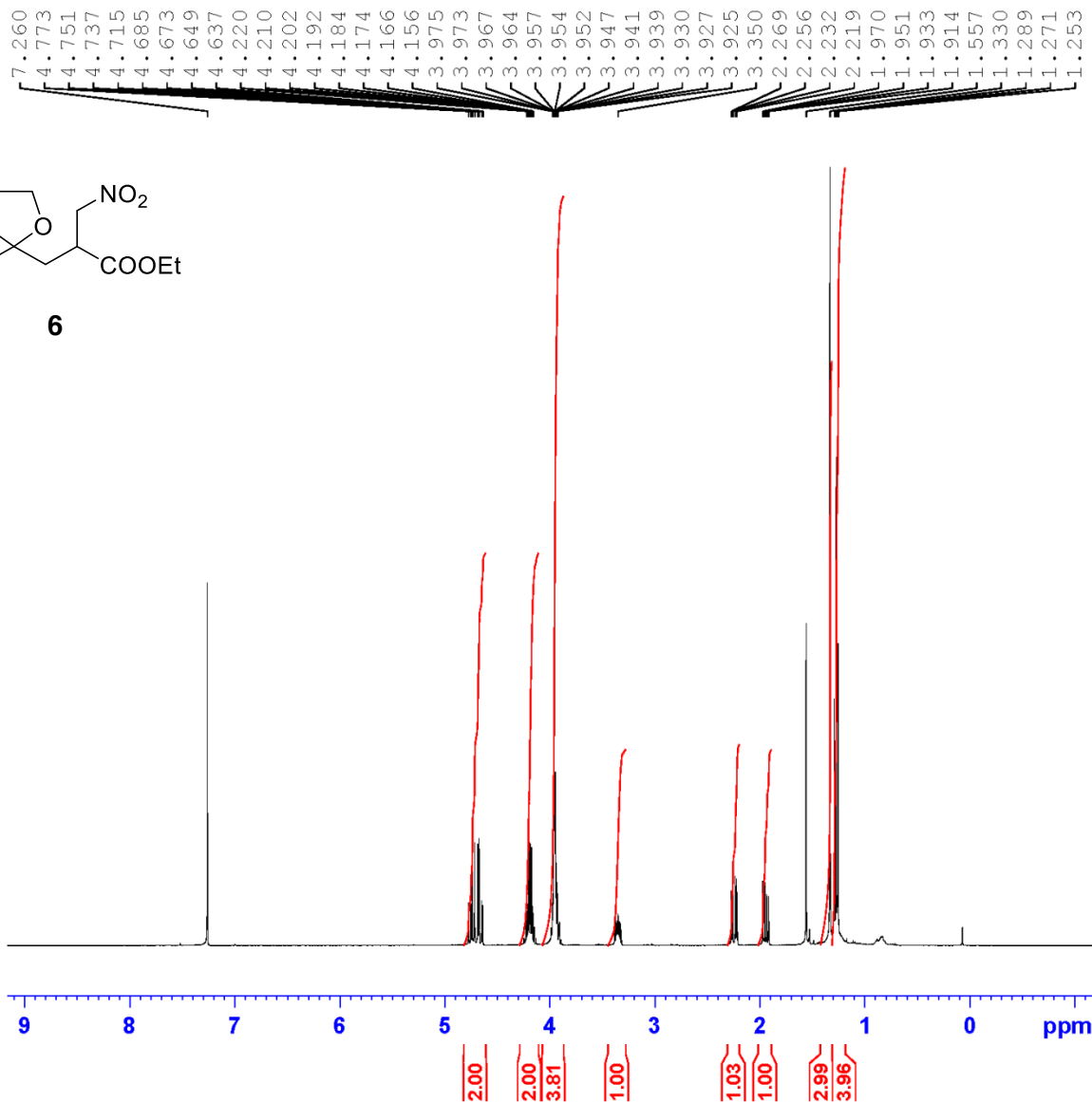
==== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 72.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 7.50000000 W
 PLW12 0.26367000 W
 PLW13 0.16633999 W

F2 - Processing parameters
 SI 32768
 SF 100.6127706 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



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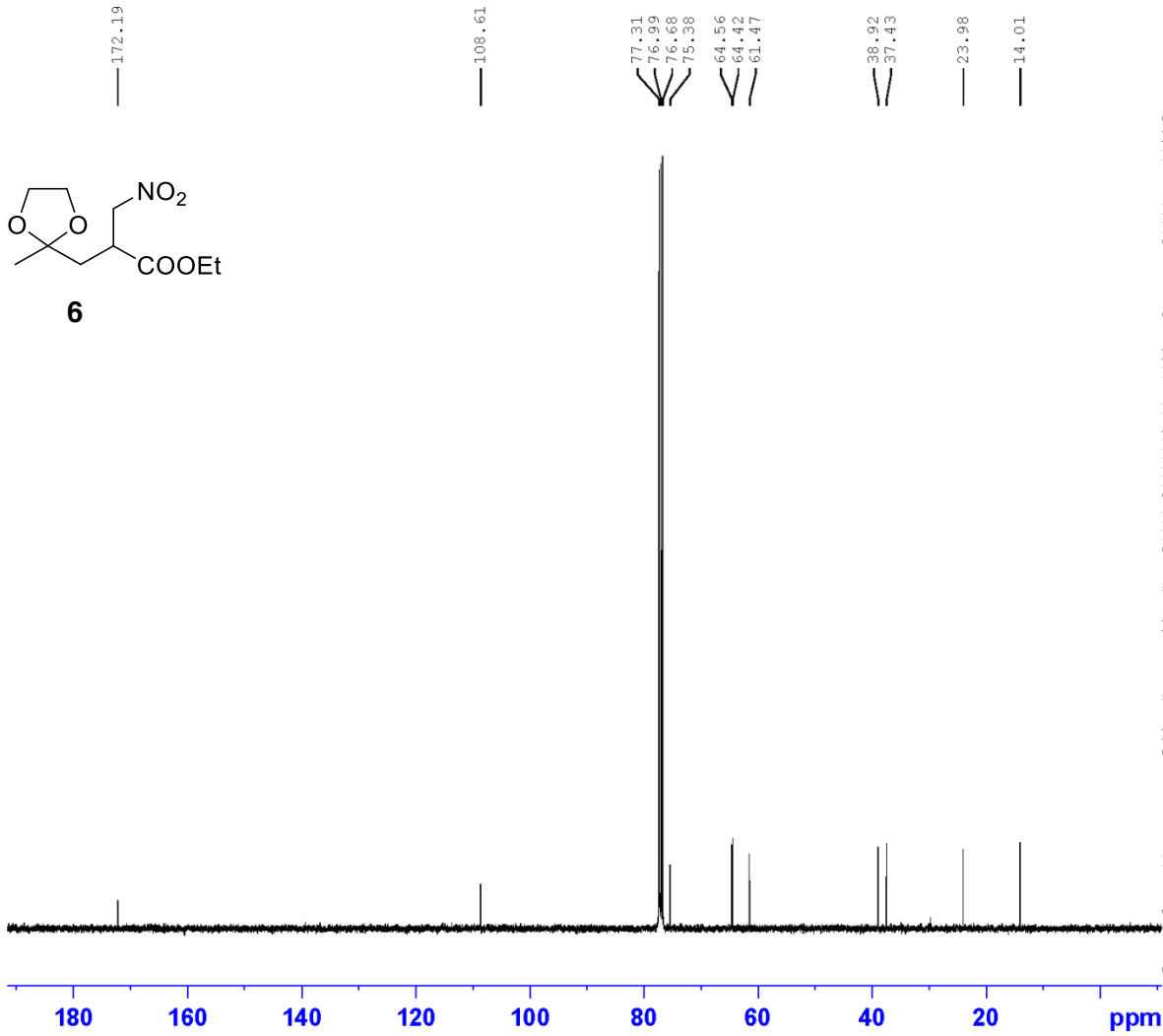
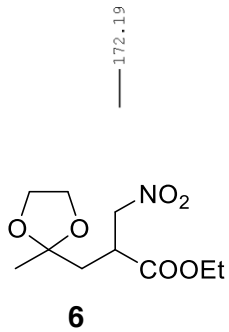
```

Current Data Parameters
NAME      YF-16-1208-AG16C
EXPNO     10
PROCNO    1

F2 - Acquisition Parameters
Date_     20161208
Time      11.44
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894465 sec
RG         77.81
DW         62.400 usec
DE         6.50 usec
TE         298.5 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      400.1324710 MHz
NUC1       1H
P1         15.00 usec
PLW1      7.50000000 W

F2 - Processing parameters
SI         65536
SF         400.1300095 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



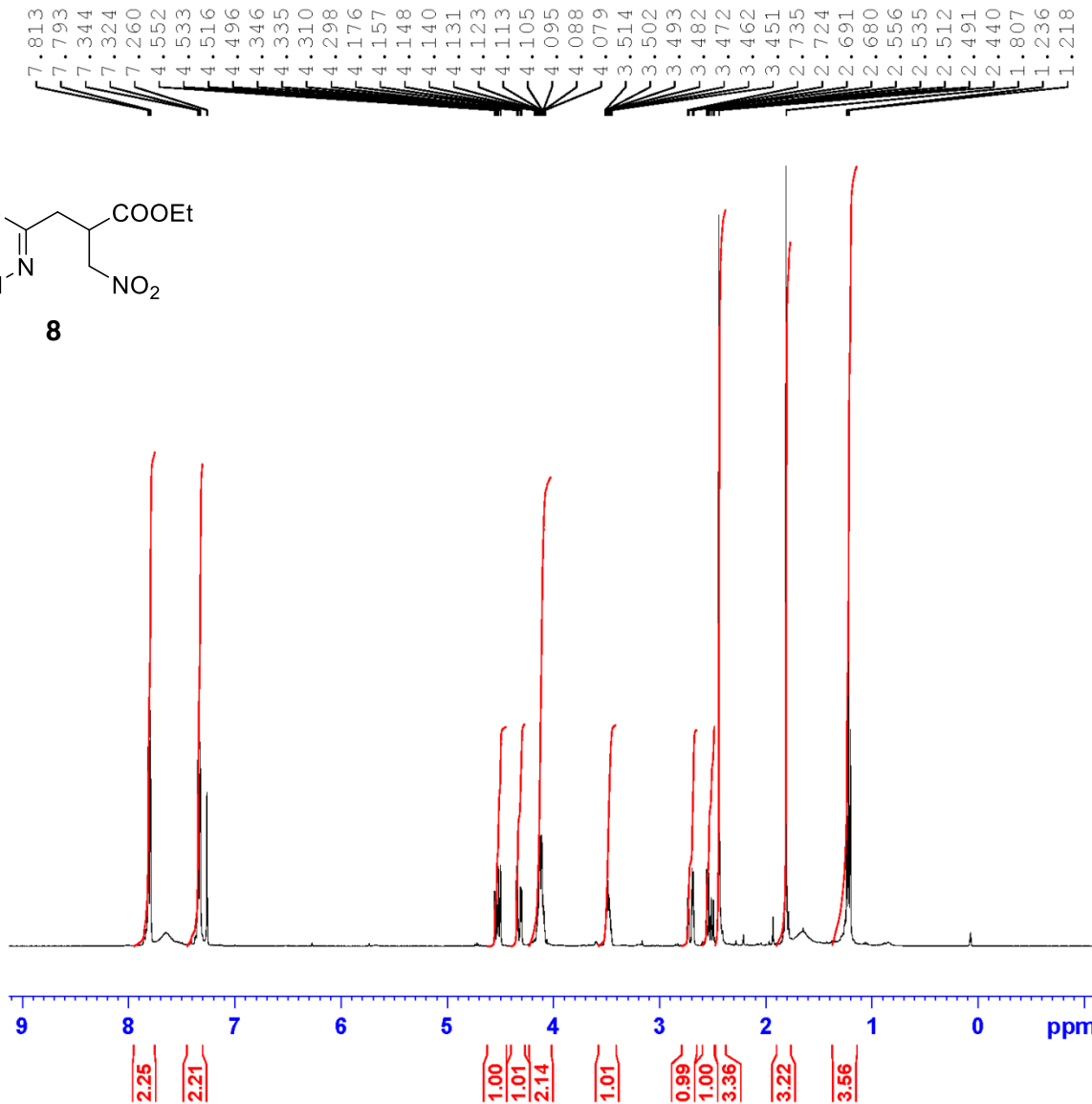
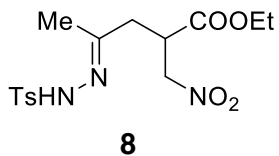
Current Data Parameters
 NAME YF-16-1208-AG16C-C
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161208
 Time_ 12.40
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.3 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 72.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 7.50000000 W
 PLW12 0.26367000 W
 PLW13 0.16633999 W

F2 - Processing parameters
 SI 32768
 SF 100.6127706 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

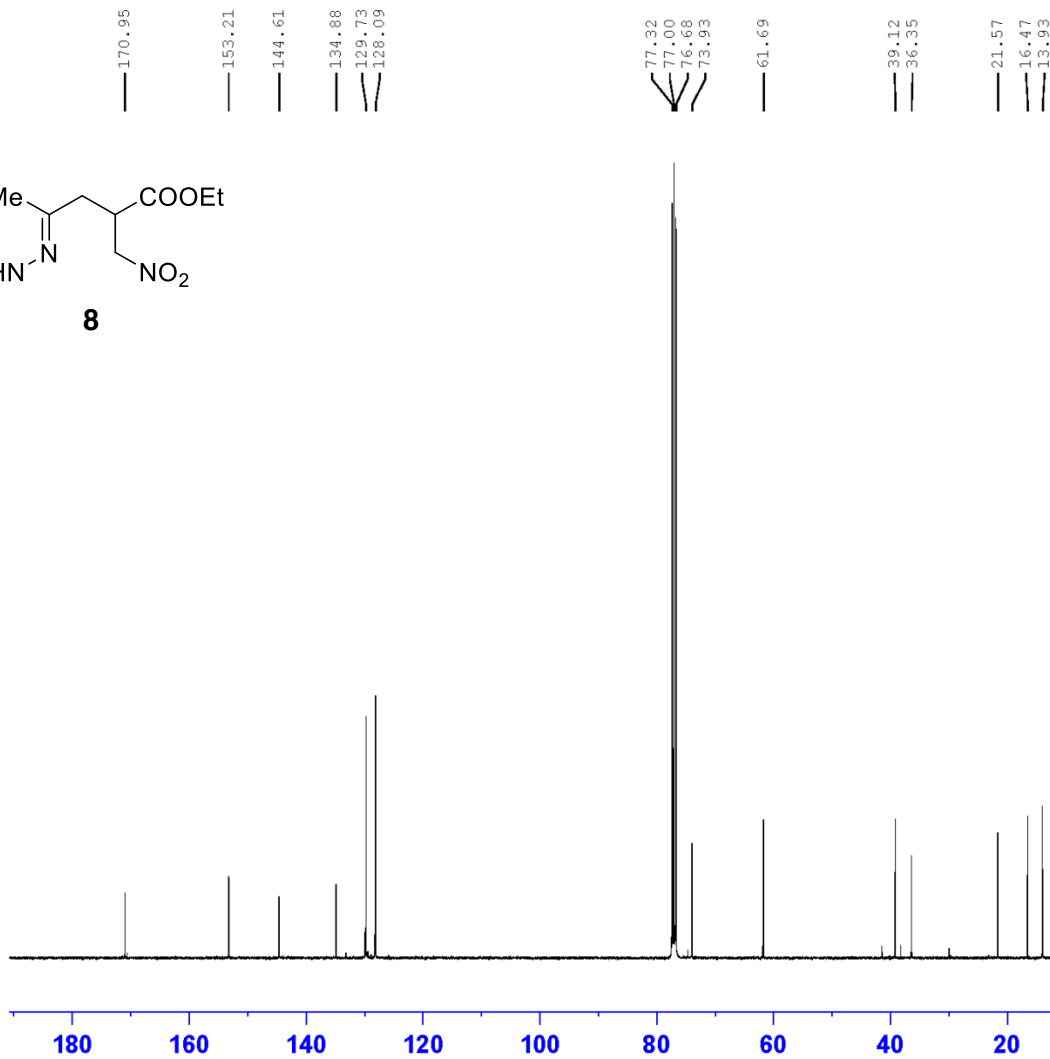
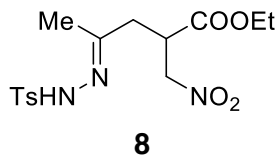


Current Data Parameters
 NAME yf-16-0805-625p
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160805
 Time_ 10.29
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 62.88
 DW 62.400 usec
 DE 6.50 usec
 TE 298.7 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300093 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



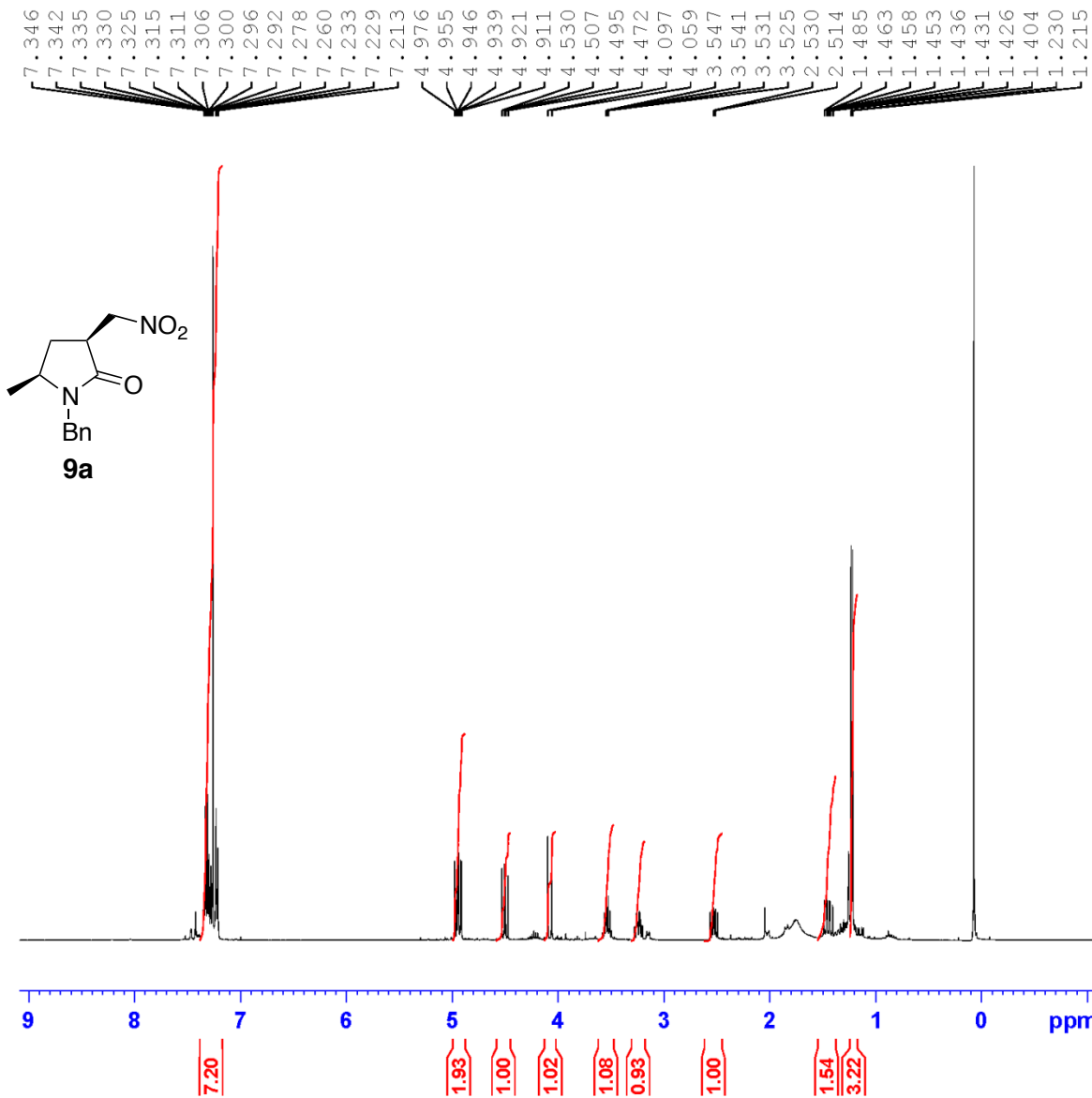
Current Data Parameters
 NAME yf-16-0807-616p-C
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160808
 Time_ 6.24
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 10000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.9 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 8.00000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

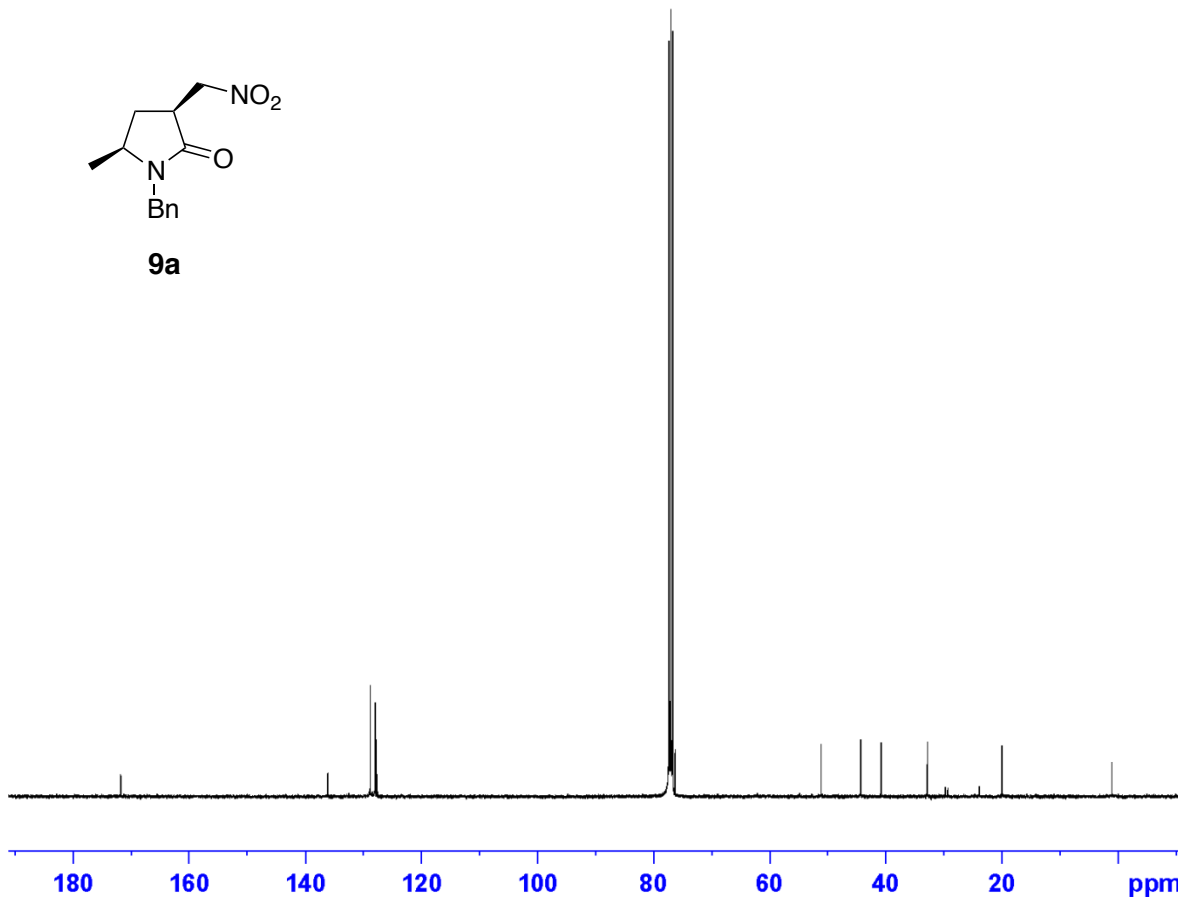
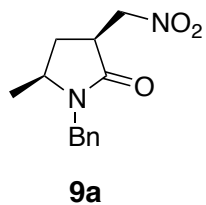
F2 - Processing parameters
 SI 32768
 SF 100.6127713 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



171.75
136.08
128.77
127.93
127.69

77.31
76.99
76.67
76.25

51.08
44.29
40.75
32.78
19.93



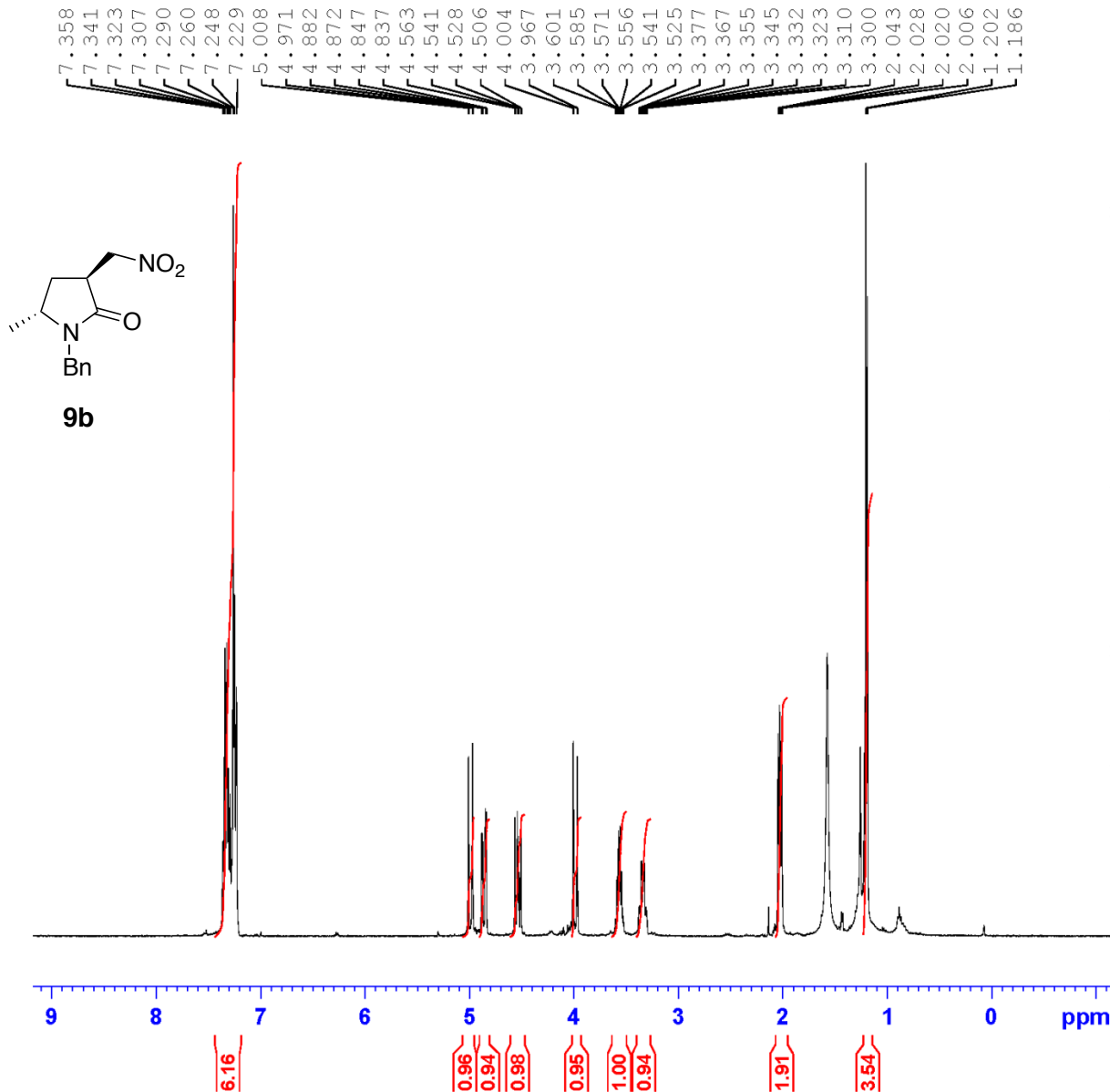
Current Data Parameters
NAME YF-16-1018-658-2-C
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161019
Time_ 6.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 195.88
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.00000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 8.00000000 W
PLW12 0.28125000 W
PLW13 0.28125000 W

F2 - Processing parameters
SI 32768
SF 100.6127708 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

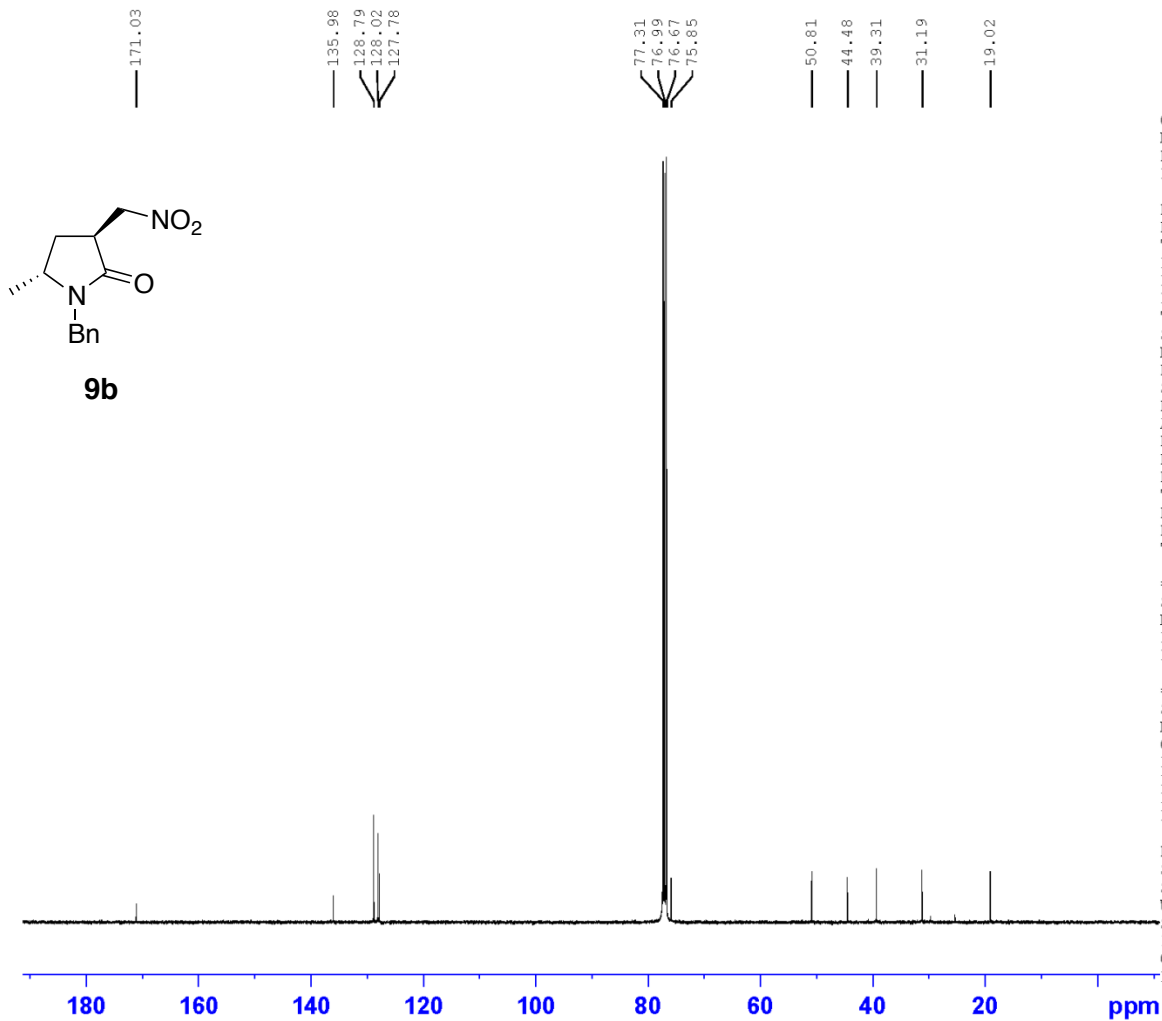


Current Data Parameters
 NAME YF-16-0805-626-2
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160805
 Time_ 22.15
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.089465 sec
 RG 88.94
 DW 62.400 usec
 DE 6.50 usec
 TE 298.6 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300091 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



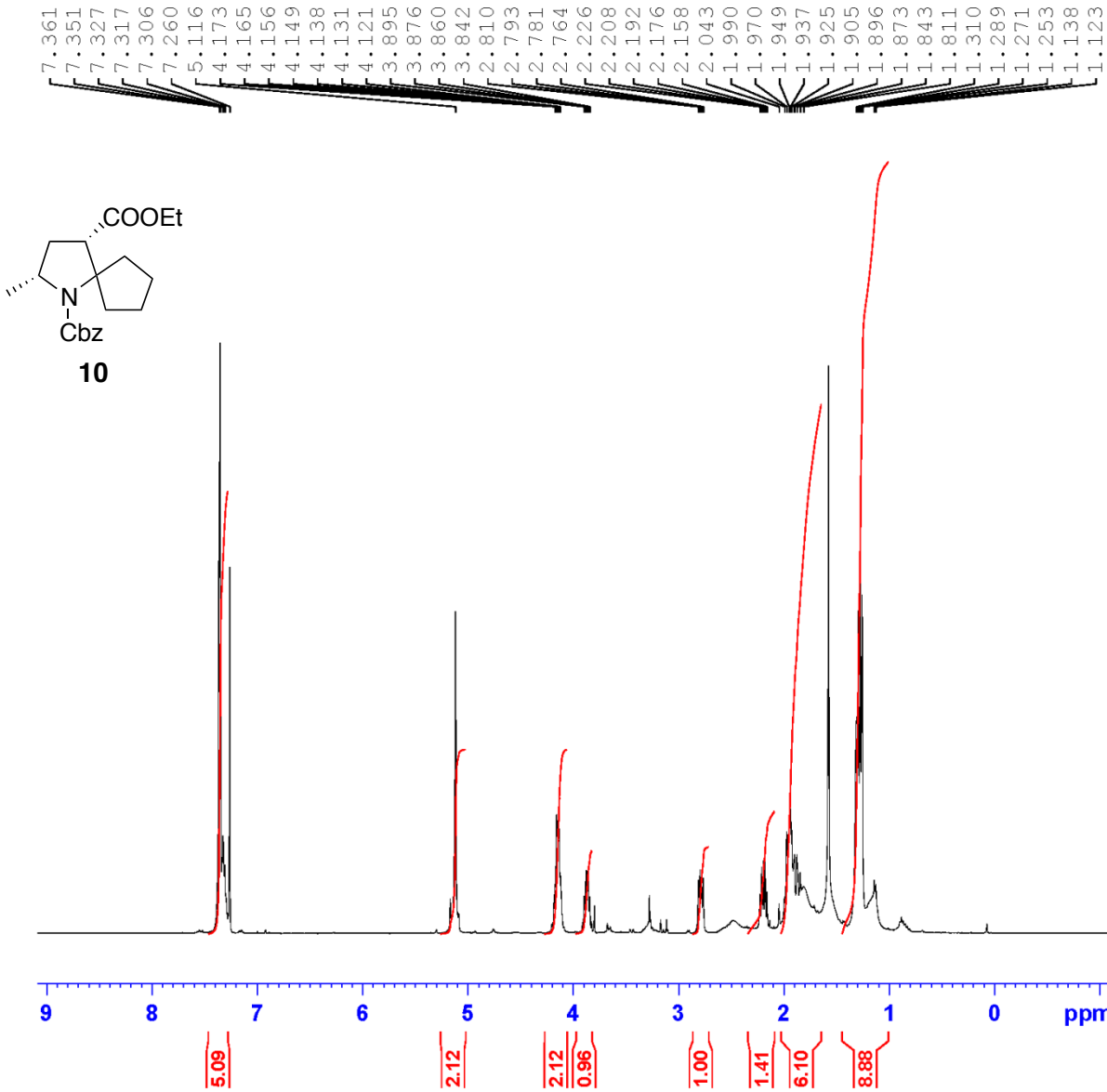
Current Data Parameters
 NAME yf-16-0817-626-2-c
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160818
 Time_ 6.23
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 8000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 FCPD2 80.00 usec
 PLW2 8.00000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127706 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

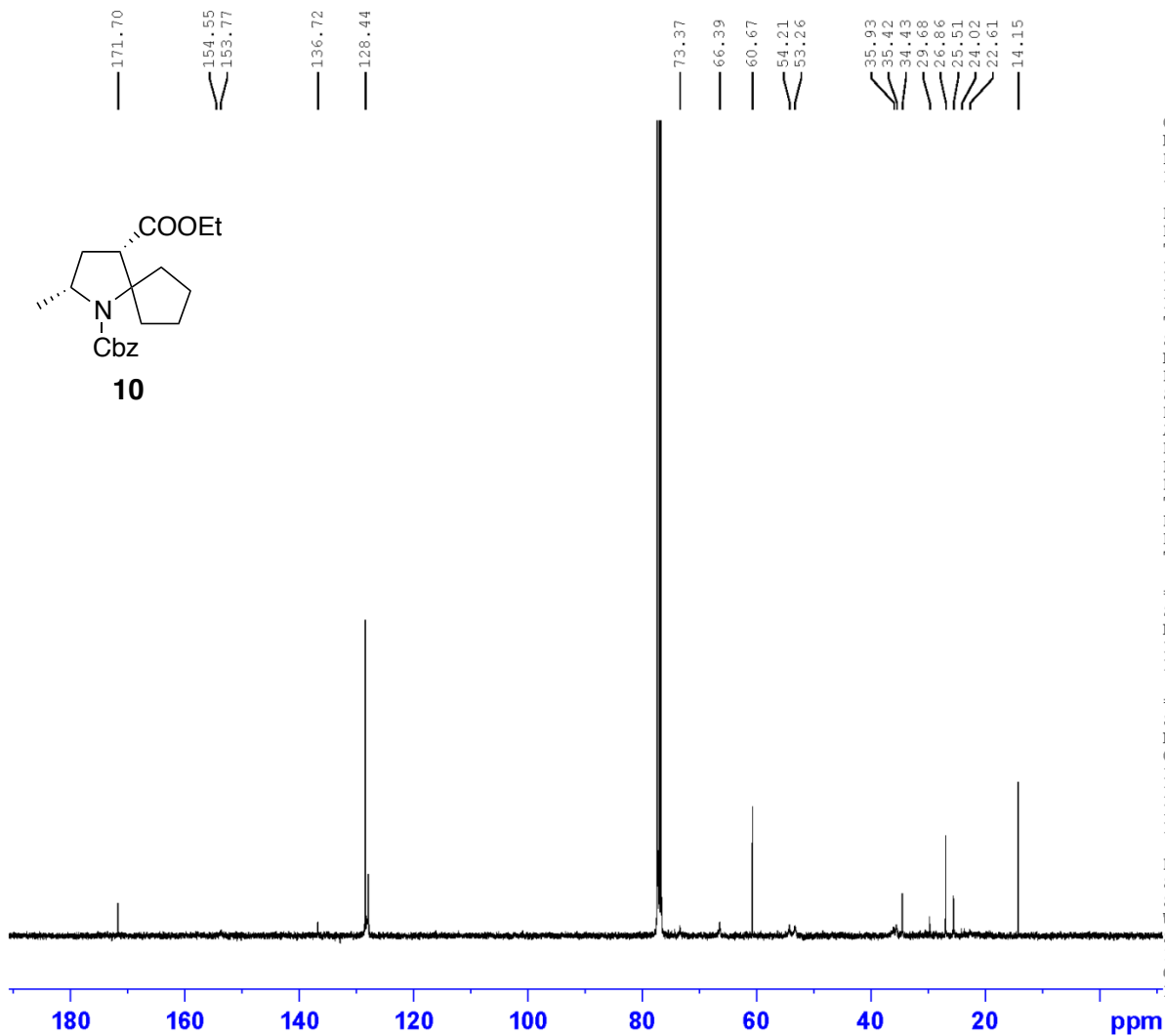


Current Data Parameters
 NAME YF-16-0828-647-1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160828
 Time_ 21.58
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 71.01
 DW 62.400 usec
 DE 6.50 usec
 TE 298.9 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300091 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME YF-16-0828-647-1-C
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160829
 Time_ 6.22
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 8000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.9 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 70.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 8.00000000 W
 PLW12 0.28125000 W
 PLW13 0.28125000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127706 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40