Supporting Information

Intramolecular Oxa-Michael Reactions of Aldols Generated from Enones and Isatins to Afford Spirooxindole Tetrahydropyrans

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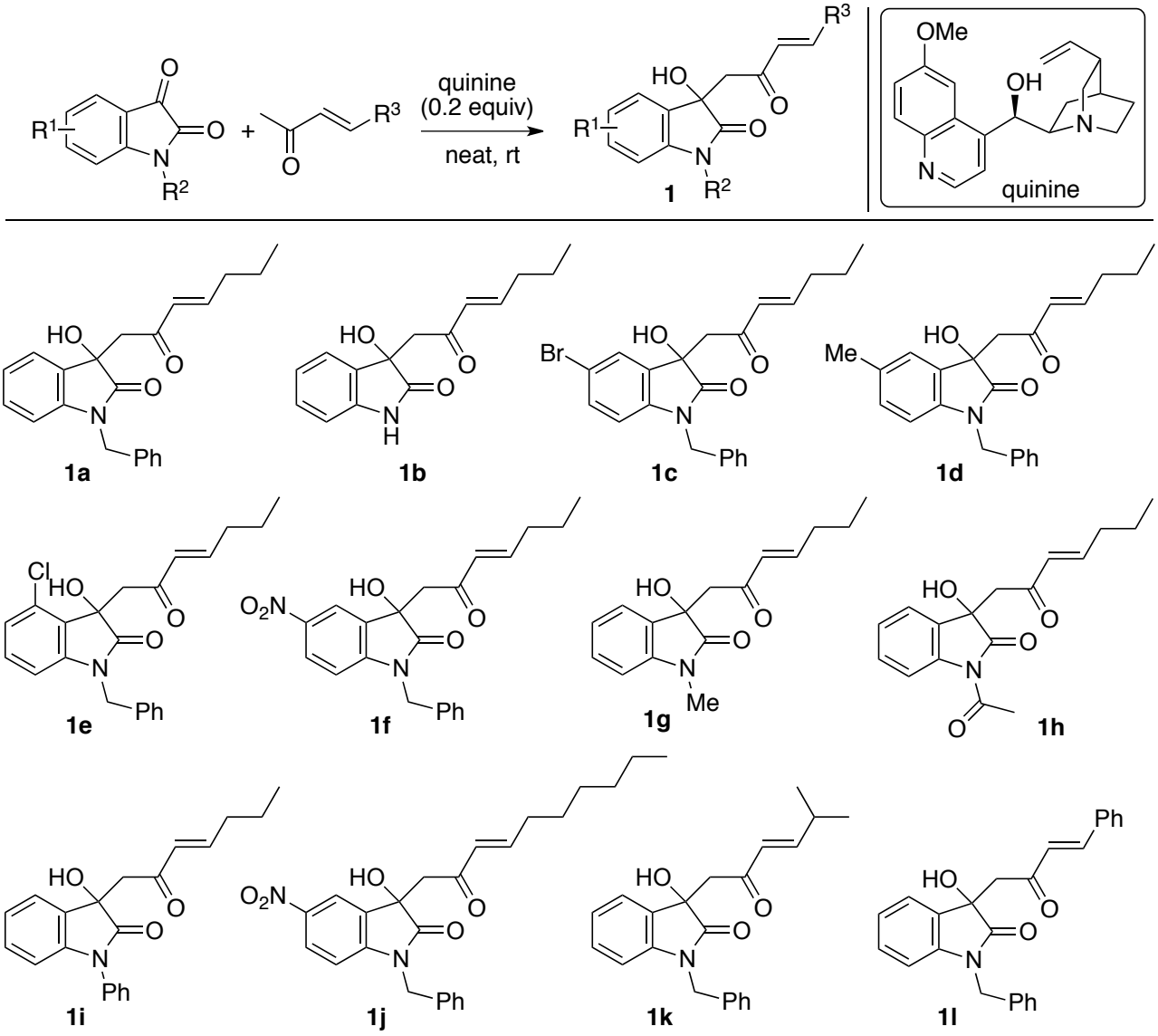
General

For thin layer chromatography (TLC), Merck silica gel 60 F254 aluminum sheets were used and the compounds were visualized by irradiation with UV light. Flash column chromatography was performed using Merck silica gel 60 (230-400 mesh) or Yamazen flash column (60 Å, 40 μm). ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance 400 or a Bruker Avance 500 with CryoProbe. Proton chemical shifts are reported in ppm downfield from tetramethylsilane (δ 0.00 ppm) or relative to the residual proton signal of the deuterated solvent in $CDCl_3$ (δ 7.26 ppm). Carbon chemical shifts were internally referenced to the deuterated solvent signals in $CDCl_3$ (δ 77.0 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.

1. Synthesis of 1

1.1. Synthesis of (±)-1

Compounds (±)-**1** were synthesized by previously reported method.[1,2] Compounds **1a**,[1] **1b**,[2] **1c**,[1] **1d**,[1] and **1g**,[1] **1h**,[1] **1i**,[1] **1j**,[1] **1k**,[1] and **1l**[3] were previously reported.

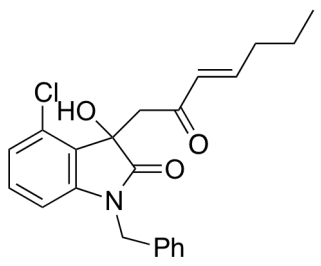


Synthesis of (±)-1a[1]

A mixture of 3-hepten-2-one (2.7 mL, 21 mmol, 5.0 equiv), *N*-benzylisatin (1.0 g, 4.2 mmol, 1.0 equiv), and quinine (270 mg, 0.84 mmol, 0.2 equiv) was stirred at room temperature for 1 day.[1] The mixture was concentrated and purified by flash column chromatography (hexane:EtOAc = 4:1) to give **1a** (600 mg, 41%) as a colorless solid. ^{1}H and ^{13}C NMR data of **1a** were consistent with those previously reported.[1]

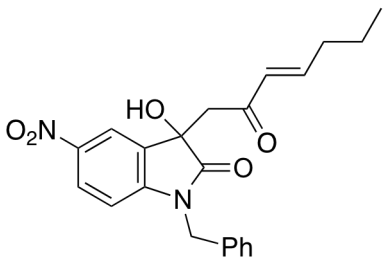
Compound 1e

Synthesized from corresponding enone (1.17 mL, 9.2 mmol) and *N*-benzyl-4-chloroisatin (500 mg, 1.84 mmol) in the presence of quinine (119 mg, 0.37 mmol), flash column chromatography



(hexane/EtOAc = 4:1), 350 mg, 50%. R_f 0.32 (hexane/EtOAc = 3:1), pale yellow gum. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.38-7.25 (m, 5H), 7.10 (t, *J* = 8.1 Hz, 1H), 6.93 (d, *J* = 8.1 Hz, 1H), 6.85 (dt, *J* = 15.9 Hz, 6.9 Hz, 1H), 6.58 (d, *J* = 8.1 Hz, 1H), 6.03 (d, *J* = 15.9 Hz, 1H), 4.95 (d, *J* = 15.8 Hz, 1H), 4.90 (d, *J* = 15.8 Hz, 1H), 3.92 (d, *J* = 16.7 Hz, 1H), 3.80 (brs, 1H, OH), 3.50 (d, *J* = 16.7 Hz, 1H), 2.21-2.15 (m, 2H), 1.51-1.43 (m, 2H), 0.92 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$: δ 196.8, 176.2, 149.3, 145.1, 135.0, 131.0, 131.01, 130.9, 130.0, 128.8, 127.6, 127.1, 125.6, 123.9, 108.2, 74.9, 44.3, 44.1, 34.5, 21.1, 13.6. ESI-HRMS: *m*/*z* calcd for $C_{22}H_{23}O_3NCl$ ($[M+H]^+$) 384.1361, found 384.1343.

Compound 1f

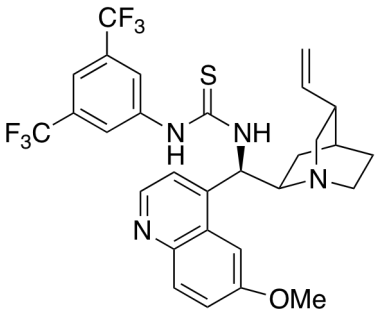


Synthesized from corresponding enone (1.37 mL, 10.5 mmol) and *N*-benzyl-5-nitroisatin (600 mg, 2.10 mmol) in the presence of quinine (136 mg, 0.42 mmol), flash column chromatography (hexane/EtOAc = 4:1), 580 mg, 70%. R_f 0.27 (hexane/EtOAc = 3:1), pale yellow gum. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.22 (d, *J* = 2.1 Hz, 1H), 8.13 (dd, *J* = 8.6 Hz, 2.1 Hz, 1H), 7.37-7.26 (m, 5H), 6.88 (dt, *J* = 15.9 Hz, 7.0 Hz, 1H), 6.76 (d, *J* = 8.6 Hz, 1H), 6.06 (d, *J* = 15.9 Hz, 1H), 5.00 (d, *J* = 15.8 Hz, 1H), 4.92 (d, *J* = 15.8 Hz, 1H), 4.46 (brs, 1H, OH), 3.53 (d, *J* = 17.4 Hz, 1H), 3.30 (d, *J* =17.4 Hz, 1H), 2.23-2.15 (m, 2H), 1.52-1.44 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 197.1, 176.77, 176.75, 150.5, 148.6, 143.5, 134.2, 130.8, 129.7, 129.0, 128.0, 127.1, 119.8, 109.3, 73.6, 45.7, 44.2, 34.5, 21.1, 13.6. ESI-HRMS: *m*/*z* calcd for $C_{22}H_{23}O_5N_2$ ($[M+H]^+$) 395.1601, found 395.1581.

1.2. Synthesis of (*R*)-1

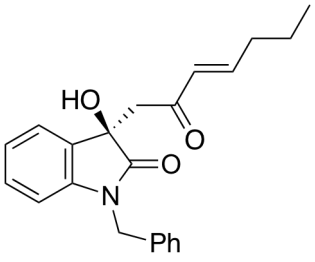
Synthesis of (*R*)-1a

Compound (*R*)-**1a** was synthesized by the reported method.[3] A mixture of 3-hepten-2-one (660 μL, 5.0 mmol, 10 equiv), *N*-benzylisatin (118.5 mg, 0.5 mmol, 1.0 equiv), and catalyst **A** (21.0 mg, 0.05 mmol, 0.1 equiv) in THF (10 mL) was stirred at 0 °C for 4 days. The mixture was concentrated and purified by flash column chromatography (hexane/EtOAc = 4:1) to give (*R*)-**1a** (70.0 mg, 40%, er 89:11). This was dissolved in hexane-EtOAc and generated precipitates were removed by filtration. The filtrate was concentrated to give (*R*)-**1a** (er 94:6) as colorless gum.



catalyst **A**

Compound (*R*)-1a

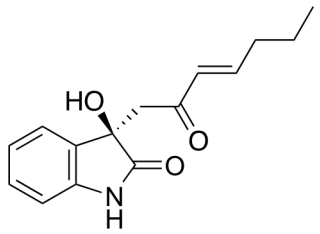


^{1}H and ^{13}C NMR data of (*R*)-**1a** were consistent with those previously reported.[1] HPLC (Daicel Chiralpak IC-3, hexane/*i*-PrOH = 80:20, 1.0 mL/min, λ = 254 nm): t_R 30 min (major enantiomer, (*R*)-**1a**), 44 min (minor enantiomer, (*S*)-**1a**).

Synthesis of (*R*)-1b

Compound (*R*)-**1b** was synthesized by the reported method.[3] A mixture of 3-hepten-2-one (897 μL, 6.8 mmol, 10 equiv), isatin (100 mg, 0.68 mmol, 1.0 equiv), and catalyst **A** (28.6 mg, 0.68 mmol, 0.1 equiv) in THF (1- mL) was stirred at 5 °C for 4 days. The mixture was concentrated, purified by flash column chromatography (hexane:EtOAc = 7:3), and crystallized from hexane-EtOAc to give (*R*)-**1b** (57.0 mg, 32%, er >99.5:0.5) as colorless crystals.

Compound (*R*)-1b



^{1}H and ^{13}C NMR data of (*R*)-**1b** were consistent with those previously reported.[2] HPLC (Daicel Chiralpak IC-3, hexane/*i*-PrOH = 70:30, 1.0 mL/min, λ = 254 nm): t_R 15 min (major enantiomer, (*R*)-**1b**), 29 min (minor enantiomer, (*S*)-**1b**).

2. Evaluations of Catalysts and Conditions (Tables 1 and 2)

2.1. Procedure for Table 1

To a solution of **1a** (69.8 mg, 0.2 mmol, 1.0 equiv) in the indicated solvent (1.0 mL), acid (0.02 mmol, 0.1 equiv) was added at room temperature (25 °C), and the mixture was stirred at the same temperature (monitored by TLC). At the indicated time point, a portion of the mixture was diluted with $CDCl_3$, except the reaction in toluene (the reaction in toluene was diluted with toluene-d8), which was analyzed by ^{1}H NMR to determine the conversion and the dr (**2a**:**3a**).

2.2. Procedure for Table 2

To a solution of **1a** (69.8 mg, 0.2 mmol, 1.0 equiv) in $CHCl_3$ (super dehydrated, indicated volume), trifluoromethanesulfonic acid (TfOH, indicated amount, either directly or stock solution, see below) was added at room temperature (25 °C), and the mixture was stirred at the same temperature. The reaction progress was monitored by TLC analyses and by ^{1}H NMR; a portion of the mixture was diluted with $CDCl_3$ and was analyzed by ^{1}H NMR to determine the conversion and the dr (**2a**:**3a**). The data at the time when the conversion reached >80% are listed in the Table, except where noted.

For the reaction with TfOH (0.1, 0.2, or 0.4 equiv), TfOH (1.76 μL, 3.5 μL, or 7.0 μL, respectively) was directly added. For the reaction with TfOH (0.05 equiv), a stock solution of TfOH (0.56 M in THF, 17.6 μL, 0.01 mmol) was added. For the reaction with TfOH (0.02 equiv), a stock solution of TfOH (0.56 M in THF, 7.0 μL, 0.004 mmol) was added. For the reaction with TfOH (0.01 equiv), a stock solution of TfOH (0.28 M in THF, 7.0 μL, 0.002 mmol) was added. For the reaction with TfOH (0.005 equiv), a stock solution of TfOH (0.28 M in THF, 3.5 μL, 0.001 mmol) was added.

Preparation of a stock solution of TfOH 0.56 M in THF: TfOH (10.0 μL, 0.113 mmol) was dissolved in THF (200 μL). Preparation of TfOH 0.28 M in THF: a stock solution of TfOH (10.0 μL, 0.113 mmol) was dissolved in THF (400 μL).

3. Intramolecular Oxa-Michael Reactions of 1 (Table 3)

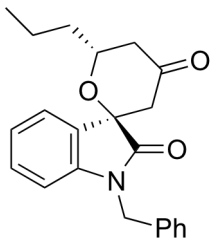
General procedure for the intramolecular oxa-Michael reactions of 1 to afford 2 and 3 (Table 3)
To a solution of **1** (0.2 mmol, 1.0 equiv) in $CHCl_3$ (super dehydrated, 4.0 mL), TfOH (0.01, 0.02, or 0.05 equiv as indicated in the Table, stock solution in THF as described in section 2.2) was added at room temperature (25 °C), and the mixture was stirred at the same temperature until **1** is completely or almost completely consumed (monitored by TLC). A portion of the mixture was diluted with $CDCl_3$ and was analyzed by ^{1}H NMR to determine the dr (**2**:**3**). The mixture was purified by flash column chromatography (hexane/EtOAc = 95:5 to 90:10) to give **2** and **3**.

A 0.2 mmol-scal reaction of 1a to afford 2a (TfOH 0.01 equiv)
To a solution of **1a** (69.8 mg, 0.20 mmol, 1.0 equiv) in $CHCl_3$ (super dehydrated, 4.0 mL), TfOH (0.28 M in THF, 7.0 μL, 0.002 mmol, 0.01 equiv) was added at room temperature (25 °C), and the mixture was stirred for 72 h (monitored by TLC). The dr (**2a**:**3a**) was determined by ^{1}H NMR analysis to be 7:1. The mixture was purified by flash column chromatography (hexane/EtOAc = 95:5 to 90:10) to give **2a** (56.0 mg, 80%) and **3a** (3.6 mg, 5%).

A 0.2 mmol-scal reaction of 1a to afford 2a (TfOH 0.05 equiv)
To a solution of **1a** (69.8 mg, 0.20 mmol, 1.0 equiv) in $CHCl_3$ (super dehydrated, 4.0 mL), TfOH (0.56 M in THF, 17.6 μL, 0.001 mmol, 0.05 equiv) was added at room temperature (25 °C), and the mixture was stirred for 16 h (monitored by TLC). The dr (**2a**:**3a**) was determined by ^{1}H NMR analysis to be 7:1. The mixture was purified by flash column chromatography (hexane/EtOAc = 95:5 to 90:10) to give **2a** (55.0 mg, 79%) and **3a** (6.4 mg, 9%).

A 1.0 mmol-scale reaction of 1a to afford 2a (TfOH 0.01 equiv)
To a solution of **1a** (349.1 mg, 1.00 mmol, 1.0 equiv) in $CHCl_3$ (super dehydrated, 20.0 mL), TfOH (0.88 μL, 0.01 mmol, 0.01 equiv) was added at room temperature (25 °C), and the mixture was stirred at the same temperature for 96 h (until **1a** is completely consumed, monitored by TLC). The mixture was purified by flash column chromatography (hexane/EtOAc = 95:5 to 90:10) to give **2a** (285.0 mg, 82%) and **3a** (29.8 mg, 9%).

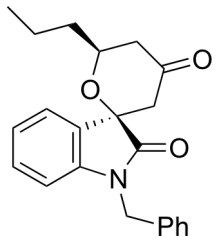
Compound 2a (major diastereomer)
(2'*R,6'*R**)-1-Benzyl-6'-propyl-5',6'-dihydrospiro[indoline-3,2'-pyran]-2,4'(3'*H*)-dione**



R_f 0.30 (hexane/EtOAc = 4:1), colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36 (dd, *J* = 7.5 Hz, 0.8 Hz, 1H), 7.34-7.29 (m, 2H), 7.29-7.24 (m, 3H), 7.23 (td, *J* = 7.5 Hz, 1.2 Hz, 1H), 7.08 (td, *J* = 7.5 Hz, 0.8 Hz, 1H), 6.69 (d, *J* = 7.8 Hz, 1H), 4.99-4.93 (m, 1H), 4.86 (d, *J* = 15.7 Hz, 1H), 4.83 (d, *J* = 15.7 Hz, 1H), 2.81 (dd, *J* = 14.9 Hz, 0.6 Hz, 1H), 2.65 (ddd, *J* = 14.9 Hz, 2.7 Hz, 1.8 Hz, 1H), 2.53 (dd, *J* = 14.9 Hz, 1.8 Hz, 1H), 2.41 (dd, *J* = 14.9 Hz, 11.1 Hz, 1H), 1.75-1.64 (m, 1H), 1.60-1.50 (m, 1H), 1.50-1.30 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 204.4, 174.7, 142.2, 135.2, 130.2, 128.8, 127.7, 127.1, 123.9, 123.3, 109.6, 77.7, 71.5, 46.8, 45.5, 43.3, 38.4, 18.1, 13.8. ESI-HRMS: *m*/*z* calcd for $C_{22}H_{24}O_3N$ ($[M+H]^+$) 350.1751, found 350.1747.

Compound 3a[4] (minor diastereomer)

(2'*R,6'*S**)-1-Benzyl-6'-propyl-5',6'-dihydrospiro[indoline-3,2'-pyran]-2,4'(3'*H*)-dione**[4]



R_f 0.25 (hexane/EtOAc = 4:1), colorless solid. ^{1}H and ^{13}C NMR data were consistent with the previously reported data.[4] ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.34-7.25 (m, 5H), 7.23 (td, *J* = 7.5 Hz, 1.1 Hz, 1H), 7.19 (d, *J* = 7.5 Hz, 1H), 6.99 (td, *J* = 7.5 Hz, 0.8 Hz, 1H), 6.73 (d, *J* = 7.5 Hz, 1H), 4.97 (d, *J* = 15.8 Hz, 1H), 4.88 (d, *J* = 15.8 Hz, 1H), 4.36-4.30 (m, 1H), 3.16 (d, *J* = 14.5 Hz, 1H), 2.68 (dd, *J* = 14.5 Hz, 11.1 Hz, 1H), 2.62 (ddd, *J* = 14.5 Hz, 3.0 Hz, 1.5 Hz, 1H), 2.43 (dd, *J* = 14.5 Hz, 1.5 Hz, 1H), 1.80-1.70 (m, 1H), 1.62-1.50 (m, 1H), 1.46-1.20 (m, 2H), 0.84 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 205.6, 173.5, 142.4, 135.0, 130.1, 128.8, 128.0, 127.7, 127.0, 125.1, 122.6, 110.1, 78.4, 73.0, 47.7, 45.5, 43.8, 38.3, 18.2, 13.7. ESI-HRMS: *m*/*z* calcd for $C_{22}H_{24}O_3N$ ($[M+H]^+$) 350.1751, found 350.1760.

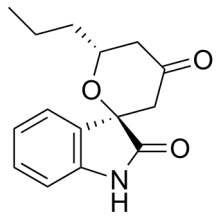
Reaction of 1b to give 2b/3b

Synthesized from **1b** (51.8 mg, 0.20 mmol) in the presence of TfOH (0.02 equiv), 96 h, **2b** (45.0 mg, 87%) and **3b** (3.1 mg, 6%).

Synthesized from **1b** (51.8 mg, 0.20 mmol) in the presence of TfOH (0.05 equiv), 32 h, **2b** (40.8 mg, 79%) and **3b** (9.6 mg, 18%).

Compound 2b (major diastereomer)

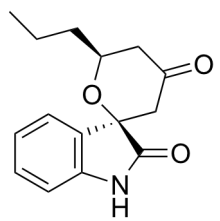
(2'*R,6'*R**)-6'-propyl-5',6'-dihydrospiro[indoline-3,2'-pyran]-2,4'(3'*H*)-dione**



R_f 0.25 (hexane/EtOAc = 3:1), colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.64 (s, 1H, NH), 7.33 (d, *J* = 7.6 Hz, 1H), 7.28 (td, *J* = 7.6 Hz, 1.1 Hz, 1H), 7.08 (td, *J* = 7.6 Hz, 0.7 Hz, 1H), 6.89 (d, *J* = 7.6 Hz, 1H), 4.95-4.85 (m, 1H), 2.77 (d, *J* = 14.8 Hz, 1H), 2.62 (ddd, *J* = 14.8 Hz, 3.0 Hz, 1.7 Hz, 1H), 2.52 (dd, *J* = 14.8 Hz, 1.7 Hz, 1H), 2.40 (dd, *J* = 14.8 Hz, 11.1 Hz, 1H), 1.72-1.60 (m, 1H), 1.60-1.50 (m, 1H), 1.50-1.30 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 204.8, 176.9, 140.3, 130.2, 129.2, 124.1, 123.2, 110.5, 78.0, 71.5, 46.8, 45.5, 38.4, 18.0, 13.8. ESI-HRMS: *m*/*z* calcd for $C_{15}H_{18}O_3N$ ($[M+H]^+$) 260.1281, found 260.1282.

Compound 3b[2] (minor diastereomer)

(2'*R,6'*S**)-6'-propyl-5',6'-dihydrospiro[indoline-3,2'-pyran]-2,4'(3'*H*)-dione**[2]



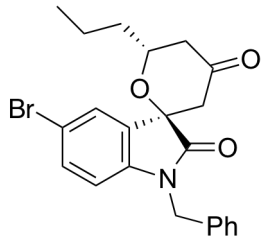
R_f 0.22 (hexane/EtOAc = 3:1, colorless solid. ^{1}H and ^{13}C NMR data were consistent with the previously reported data.[2] ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.39 (s, 1H, NH), 7.30 (td, *J* = 7.5 Hz, 1.1 Hz, 1H), 7.17 (d, *J* = 7.5 Hz, 1H), 7.01 (td, *J* = 7.5 Hz, 0.9 Hz, 1H), 6.94 (d, *J* = 7.5 Hz, 1H), 4.39-4.29 (m, 1H), 3.08 (d, *J* = 14.2 Hz, 1H), 2.69-2.59 (m, 2H), 2.42 (dd, *J* = 14.2 Hz, 1.4 Hz, 1H), 1.80-1.70 (m, 1H), 1.60-1.50 (m, 1H), 1.47-1.20 (m, 2H), 0.83 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 205.4, 175.1, 140.3, 130.3, 128.2, 125.4, 122.7, 110.9, 78.7, 73.0, 47.6, 45.4, 38.3, 18.2, 13.7. ESI-HRMS: *m*/*z* calcd for $C_{15}H_{18}O_3N$ ($[M+H]^+$) 260.1281, found 260.1289.

Reaction of 1c to give 2c/3c

Synthesized from **1c** (85.4 mg, 0.20 mmol) in the presence of TfOH (0.02 equiv), 72 h, **2c** (59.4 mg, 70%) and **3c** (9.0 mg, 11%).

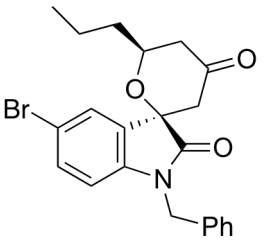
Synthesized from **1c** (85.4 mg, 0.20 mmol) in the presence of TfOH (0.05 equiv), 30 h, **2c** (56.0 mg, 66%) and **3c** (19.3 mg, 23%).

Compound 2c



R_f 0.34 (hexane/EtOAc = 4:1), colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.48 (d, *J* = 1.9 Hz, 1H), 7.35-7.25 (m, 4H), 7.23-7.20 (m, 2H), 6.56 (d, *J* = 8.3 Hz, 1H), 4.97-4.90 (m, 1H), 4.84 (d, *J* = 15.8 Hz, 1H), 4.81 (d, *J* = 15.8 Hz, 1H), 2.77 (d, *J* = 14.8 Hz, 1H), 2.60 (ddd, *J* = 14.8 Hz, 2.6 Hz, 1.7 Hz, 1H), 2.47 (dd, *J* = 14.8 Hz, 1.7 Hz, 1H), 2.37 (dd, *J* = 14.8 Hz, 11.0 Hz, 1H), 1.72-1.63 (m, 1H), 1.60-1.50 (m, 1H), 1.50-1.34 (m, 2H), 0.91 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 203.7, 174.1, 141.2, 134.7, 133.0, 130.6, 128.9, 127.8, 127.3, 127.0, 116.0, 111.1, 77.5, 71.7, 46.8, 45.4, 43.4, 38.4, 18.0, 13.8. ESI-HRMS: *m*/*z* calcd for $C_{22}H_{23}O_3NBr$ ($[M+H]^+$) 428.0856, found 428.0853.

Compound 3c



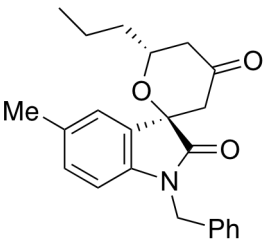
R_f 0.28 (hexane/EtOAc = 4:1), colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36-7.26 (m, 5H), 7.24-7.21 (m, 2H), 6.60 (d, *J* = 8.3 Hz, 1H), 4.93 (d, *J* = 15.8 Hz, 1H), 4.86 (d, *J* = 15.8 Hz, 1H), 4.31-4.25 (m, 1H), 3.12 (d, *J* = 14.5 Hz, 1H), 2.70 (dd, *J* = 14.7 Hz, 11.3 Hz, 1H), 2.63 (ddd, *J* = 14.7 Hz, 2.7 Hz, 1.5 Hz, 1H), 2.45 (dd, *J* = 14.5 Hz, 1.5 Hz, 1H), 1.80-1.70 (m, 1H), 1.64-1.55 (m, 1H), 1.50-1.25 (m, 2H), 0.86 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 204.9, 173.0, 141.4, 134.5, 133.0, 130.0, 128.9, 128.0, 127.9, 115.4, 111.5, 78.3, 73.4, 47.4, 45.1, 43.9, 38.2, 18.26, 13.7. ESI-HRMS: *m*/*z* calcd for $C_{22}H_{23}O_3NBr$ ($[M+H]^+$) 428.0856, found 428.0865.

Reaction of 1d to give 2d/3d

Synthesized from **1d** (72.6 mg, 0.20 mmol) in the presence of TfOH (0.02 equiv), 72 h, **2d** (65.8 mg, 91%) and **3d** (4.2 mg, 5%).

Synthesized from **1d** (72.6 mg, 0.20 mmol) in the presence of TfOH (0.05 equiv), 30 h, **2d** (58.7 mg, 80%) and **3d** (11.0 mg, 15%).

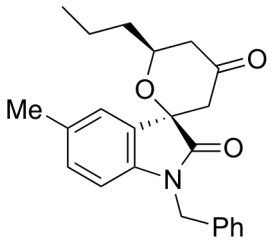
Compound 2d



R_f 0.30 (hexane/EtOAc = 4:1), colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32-7.23 (m, 5H), 7.20-7.18 (m, 1H), 7.03-7.00 (m, 1H), 6.58 (d, *J* = 7.9 Hz, 1H), 5.00-4.93 (m, 1H), 4.85 (d, *J* = 15.7 Hz, 1H), 4.80 (d, *J* = 15.7 Hz, 1H), 2.79 (d, *J* = 14.9 Hz, 1H), 2.64 (ddd, *J* = 14.9 Hz, 2.7 Hz, 1.7 Hz, 1H), 2.52 (dd, *J* = 14.9 Hz, 1.7 Hz, 1H), 2.40 (dd, *J* = 14.9 Hz. 11.1 Hz, 1H), 2.31 (s, 3H), 1.72-1.60 (m, 1H), 1.60-1.50 (m, 1H), 1.50-1.34 (m, 2H), 0.90 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (125 MHz,

$CDCl_3$): δ 204.5, 174.6, 139.7, 135.3, 133.0, 130.4, 128.7, 127.6, 127.1, 124.7, 109.3, 77.8, 71.4, 46.8, 45.6, 43.3, 38.4, 21.0, 18.1, 13.8. ESI-HRMS: *m/z* calcd for $C_{23}H_{26}O_3N$ ($[M+H]^+$) 364.1907, found 364.1903

Compound 3d

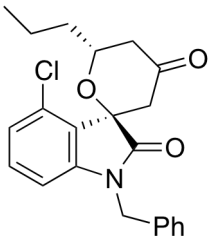


R_f 0.23 (hexane/EtOAc = 4:1), colorless solid. ^1H NMR (500 MHz, $CDCl_3$): δ 7.33-7.23 (m, 5H), 7.03-7.00 (m, 1H), 6.99-6.98 (m, 1H), 6.61 (d, *J* = 7.9 Hz, 1H), 4.94 (d, *J* = 15.7 Hz, 1H), 4.86 (d, *J* = 15.7 Hz, 1H), 4.36-4.30 (m, 1H), 3.15 (d, *J* = 14.2 Hz, 1H), 2.68 (dd, *J* = 14.6 Hz, 10.9 Hz, 1H), 2.64 (ddd, *J* = 14.6 Hz, 3.2 Hz, 1.5 Hz, 1H), 2.42 (dd, *J* = 14.2 Hz, 1.5 Hz, 1H), 2.27 (s, 3H), 1.80-1.70 (m, 1H), 1.60-1.53 (m, 1H), 1.48-1.25 (m, 2H), 0.85 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 205.8, 173.4, 140.0, 135.1, 132.3, 130.3, 128.8, 128.0, 127.7, 127.0, 125.9, 109.8, 78.5, 73.0, 47.6, 45.6, 43.8, 38.3, 21.0, 18.2, 13.7. ESI-HRMS: calcd for $C_{23}H_{26}O_3N$ ($[M+H]^+$) 364.1907, found 364.1914

Reaction of 1e to give 2e/3e

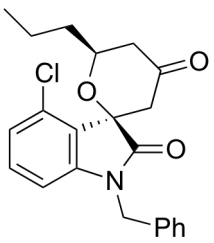
Synthesized from **1e** (76.6 mg, 0.20 mmol) in the presence of TfOH (0.05 equiv), 120 h, **2e** (38.7 mg, 51%) and **3e** (34.5 mg, 45%).

Compound 2e



R_f 0.31 (hexane/EtOAc = 4:1), colorless solid. ^1H NMR (500 MHz, $CDCl_3$): δ 7.34-7.22 (m, 5H), 7.14 (*t*, *J* = 8.1 Hz, 1H), 7.00 (dd, *J* = 8.1 Hz, 0.5 Hz, 1H), 6.59 (d, *J* = 8.1 Hz, 1H), 4.99-4.93 (m, 1H), 4.85 (d, *J* = 15.7 Hz, 1H), 4.82 (d, *J* = 15.7 Hz, 1H), 3.42 (d, *J* = 15.0 Hz, 1H), 2.62 (ddd, *J* = 15.0 Hz, 2.6 Hz, 1.7 Hz, 1H), 2.49 (dd, *J* = 15.0 Hz, 11.0 Hz, 1H), 2.48 (dd, *J* = 15.0 Hz, 1.7 Hz, 1H), 1.74-1.65 (m, 1H), 1.64-1.52 (m, 1H), 1.50-1.20 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 204.4, 174.3, 144.0, 134.8, 131.9, 131.2, 128.9, 127.9, 127.0, 124.8, 124.6, 108.1, 78.3, 71.4, 46.5, 43.5, 42.2, 38.2, 17.8, 13.9. ESI-HRMS: *m/z* calcd for $C_{22}H_{23}O_3NCl$ ($[M+H]^+$) 384.1361, found 384.1359.

Compound 3e

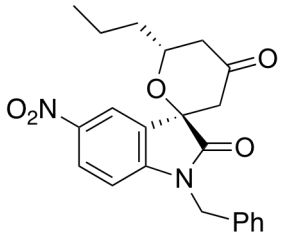


R_f 0.25 (hexane/EtOAc = 4:1), colorless solid. ^1H NMR (500 MHz, $CDCl_3$): δ 7.34-7.22 (m, 5H), 7.14 (t, *J* = 8.0 Hz, 1H) 7.01 (d, *J* = 8.0 Hz, 1H), 6.60 (d, *J* = 8.0 Hz, 1H), 4.89 (d, *J* = 15.7 Hz, 1H), 4.75 (d, *J* = 15.7 Hz, 1H), 4.71-4.65 (m, 1H), 3.57 (d, *J* = 16.8 Hz, 1H), 2.98 (dd, *J* = 18.1 Hz, 12.2 Hz, 1H), 2.58 (dd, *J* = 18.1 Hz, 1.9 Hz, 1H), 2.42 (d, *J* = 16.8 Hz, 1H), 1.78-1.64 (m, 1H), 1.64-1.40 (m, 3H), 0.93 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$: δ 205.7, 175.6, 144.0, 134.8, 131.3, 131.1, 128.9, 127.8, 127.1, 124.2, 108.3, 78.0, 72.5, 45.8, 43.9, 40.8, 37.6, 18.1, 13.9. ESI-HRMS: calcd for *m/z* $C_{22}H_{23}O_3NCl$ ($[M+H]^+$) 384.1361, found 384.1366.

Reaction of 1f to give 2f/3f

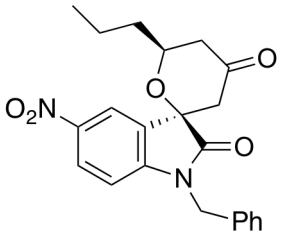
Synthesized from **1f** (78.8 mg, 0.20 mmol) in the presence of TfOH (0.02 equiv), 96 h, **2f** (44.6 mg, 57%) and **3f** (21.0 mg, 27%).

Compound 2f



R_f 0.18 (hexane/EtOAc = 4:1), pale yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.27 (d, *J* = 2.2 Hz, 1H), 8.19 (dd, *J* = 8.7 Hz, 2.2 Hz, 1H), 7.36-7.28 (m, 3H), 7.24-7.21 (m, 2H), 6.79 (d, *J* = 8.7 Hz, 1H), 4.97-4.87 (m, 3H), 2.87 (d, *J* = 14.9 Hz, 1H), 2.68 (ddd, *J* = 14.8 Hz, 3.0 Hz, 1.5 Hz, 1H), 2.53 (dd, *J* = 14.9 Hz, 1.5 Hz, 1H), 2.47 (dd, *J* = 14.8 Hz, 11.1 Hz, 1H), 1.75-1.63 (m, 1H), 1.60-1.50 (m, 1H), 1.50-1.34 (m, 2H), 0.91 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 202.9, 174.7, 147.8, 143.9, 134.0, 129.5, 129.1, 128.2, 127.1, 127.0, 120.2, 109.4, 77.1, 72.1, 46.8, 45.1, 43.7, 38.3, 18.1, 13.8. ESI-HRMS: *m*/*z* calcd for $C_{22}H_{23}O_5N_2$ ($[M+H]^+$) 395.1601, found 395.1597.

Compound 3f



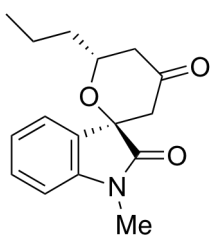
R_f 0.13 (hexane/EtOAc = 4:1), pale yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.21 (dd, *J* = 8.7 Hz, 2.2 Hz, 1H), 8.08 (d, *J* = 2.2 Hz, 1H), 7.37-7.28 (m, 3H), 7.25-7.22 (m, 2H), 6.83 (d, *J* = 8.7 Hz, 1H), 4.99 (d, *J* = 15.8 Hz, 1H), 4.93 (d, *J* = 15.8 Hz, 1H), 4.39-4.33 (m, 1H), 3.10 (d, *J* = 14.7 Hz, 1H), 2.78 (dd, *J* = 15.0 Hz, 11.4 Hz, 1H), 2.70 (ddd, *J* = 15.0 Hz, 2.5 Hz, 1.1 Hz, 1H), 2.56 (dd, *J* = 14.7 Hz, 1.1 Hz, 1H), 1.80-1.70 (m, 1H), 1.77-1.58 (m, 1H) 1.50-1.25 (m, 2H), 0.87 (t, *J* = 7.3Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$: δ 204.0, 173.9, 147.9, 143.3, 133.9, 129.1, 129.0, 128.3, 127.1, 127.0, 120.4, 109.7, 77.7, 73.8, 47.2, 44.7, 44.3, 38.1, 18.1, 13.7. ESI-HRMS: *m*/*z* calcd for $C_{22}H_{23}O_5N_2$ ($[M+H]^+$) 395.1601, found 395.1607.

Reaction of 1g to give 2g/3g

Synthesized from **1g** (54.6 mg, 0.20 mmol) in the presence of TfOH (0.02 equiv), 48 h, **2g** (43.2 mg, 80%) and **3g** (6.8 mg, 12%).

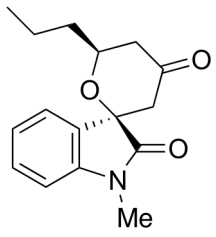
Synthesized from **1g** (54.6 mg, 0.20 mmol) in the presence of TfOH (0.05 equiv), 48 h, **2g** (43.0 mg, 79%) and **3g** (9.9 mg, 18%).

Compound 2g



R_f 0.21 (hexane/EtOAc = 4:1), colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.37-7.33 (m, 2H), 7.12 (td, *J* = 7.8 Hz, 0.6 Hz, 1H), 6.82 (dd, *J* = 7.8 Hz, 0.6 Hz, 1H), 4.98-4.87 (m, 1H), 3.14 (s, 3H), 2.75 (dd, *J* = 14.8 Hz, 0.5 Hz, 1H), 2.62 (ddd, *J* = 14.8 Hz, 2.8 Hz, 1.8 Hz, 1H), 2.45 (dd, *J* = 14.8 Hz, 1.8 Hz, 1H), 2.38 (dd, *J* = 14.8 Hz, 11.1 Hz, 1H), 1.70-1.60 (m, 1H), 1.59-1.49 (m, 1H), 1.48-1.30 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 204.4, 174.6, 143.0, 130.2, 128.7, 123.8, 123.3, 108.5, 77.7, 71.5, 46.7, 45.6, 38.5, 25.9, 18.0, 13.9. ESI-HRMS: *m*/*z* calcd for $C_{16}H_{20}O_3N$ ($[M+H]^+$) 274.1438, found 274.1437.

Compound 3g[4]

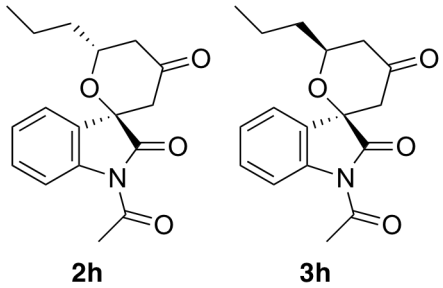


R_f 0.13 (hexane/EtOAc = 4:1), colorless solid. ^{1}H and ^{13}C NMR data were consistent with the previously reported data.[4] ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36 (td, *J* = 7.8 Hz, 1.1 Hz, 1H), 7.18 (d, *J* = 7.8 Hz, 1H), 7.04 (td, *J* = 7.8 Hz, 0.8 Hz, 1H), 6.88 (d, *J* = 7.8 Hz, 1H), 4.37-4.29 (m, 1H), 3.22 (s, 3H), 3.09 (d, *J* = 14.3 Hz, 1H), 2.66 (dd, *J* = 14.6 Hz, 11.0 Hz, 1H), 2.60 (ddd, *J* = 14.6 Hz, 3.1 Hz, 1.6 Hz, 1H), 2.36 (dd, *J* = 14.3 Hz, 1.6 Hz, 1H), 1.79-1.69 (m, 1H), 1.60-1.50 (m, 1H), 1.40-1.20 (m, 2H), 0.83 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 205.6, 173.2, 143.3, 130.2, 127.9, 125.0, 122.6, 109.0, 78.4, 73.0, 47.6, 45.5, 38.3, 26.4, 18.1, 13.7. ESI-HRMS: *m*/*z* calcd for $C_{16}H_{20}O_3N$ ($[M+H]^+$) 274.1438, found 274.1443.

Reaction of 1h to give 2h/3h

Synthesized from **1h** (60.2 mg, 0.20 mmol) in the presence of TfOH (0.05 equiv), 72 h, **2h**/**3h** (48.5 mg, 80% as a mixture of **2h**/**3h** = 3:1).

Compound 2h/3h (3:1)



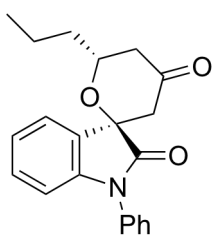
2h **3h**

R_f 0.35 (hexane/EtOAc = 4:1), colorless gum. ^{1}H NMR (500 MHz, $CDCl_3$): **2h**/**3h** = 3:1, δ 8.31 (d, *J* = 8.0 Hz, 1H x 1/4), 8.25-8.21 (m, 1H x 3/4), 7.44-7.39 m, 2H x 3/4 + 1H x 1/4), 7.28 (td, *J* = 7.5 Hz, 1.0 Hz, 1H x 3/4), 7.25-7.18 (m, 2H x 1/4), 4.77-4.70 (m, 1H x 3/4), 4.35-4.25 (m, 1H x 1/4), 3.05 (d, *J* = 15.0 Hz, 1H x 1/4), 2.79 (d, *J* = 15.0 Hz, 1H x 3/4), 2.71 (s, 3H x 1/4), 2.68-2.60 (m, 1H x 3/4 + 2H x 1/4), 2.64 (s, 3H x 3/4), 1.73-1.23 (m, 4H), 0.89 (t, *J* = 7.4 Hz, 3H x 3/4), 0.83 (t, *J* = 7.4 Hz, 3H x 1/4). ^{13}C NMR (125 MHz, $CDCl_3$): **2h**/**3h** = 3:1, δ 204.7, 203.6, 175.0, 174.1, 170.5, 170.4, 139.8, 139.6, 130.8, 130.7, 127.9, 127.3, 125.9, 125.2, 124.5, 123.7, 117.3, 116.9, 78.0, 77.7, 72.5, 72.2, 47.3, 46.7, 45.8, 45.1, 38.4, 38.1, 26.6, 26.5, 18.07, 18.05, 13.8, 13.6. ESI-HRMS: *m*/*z* calcd for $C_{17}H_{20}O_4N$ ($[M+H]^+$) 302.1387, found 302.1384.

Reaction of 1i to give 2i/3i

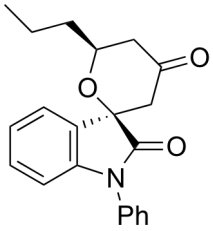
Synthesized from **1i** (67.0 mg, 0.20 mmol) in the presence of TfOH (0.05 equiv), 24 h, **2i** (52.3 mg, 78%) and **3i** (10.5 mg, 16%).

Compound 2i



R_f 0.31 (hexane/EtOAc = 4:1), pale yellow gum. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.52-7.49 (m, 2H), 7.44 (dd, *J* = 7.5 Hz, 0.6 Hz, 1H), 7.42-7.38 (m, 3H), 7.29 (td, *J* = 7.5 Hz, 1.0 Hz, 1H), 7.16 (td, *J* = 7.5 Hz, 0.5 Hz, 1H), 6.85 (d, *J* = 7.5 Hz, 1H), 5.00-4.90 (m, 1H), 2.85 (d, *J* = 15.0 Hz, 1H), 2.64 (ddd, *J* = 15.0 Hz, 2.5 Hz, 1.6 Hz, 1H), 2.63 (dd, *J* = 15.0 Hz, 1.6 Hz, 1H), 2.42 (dd, *J* = 15.0 Hz, 11.0 Hz, 1H), 1.75-1.65 (m, 1H), 1.60-1.50 (m, 1H), 1.50-1.35 (m, 2H), 0.91 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 204.2, 173.8, 143.0, 133.4, 130.2, 129.5, 128.6, 128.1, 126.1, 124.2, 123.8, 109.8, 77.7, 71.5, 46.6, 45.7, 38.4, 18.0, 13.8. ESI-HRMS: *m*/*z* calcd for $C_{21}H_{22}O_3N$ ($[M+H]^+$) 336.1594, found 336.1596.

Compound 3i

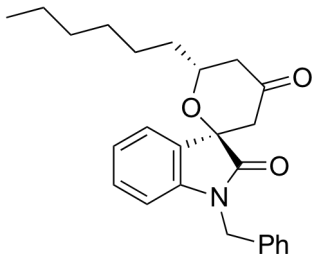


R_f 0.25 (hexane/EtOAc = 4:1), pale yellow gum. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.56-7.51 (m, 2H), 7.45-7.41 (m, 3H), 7.31-7.24 (m, 2H), 7.07 (t, *J* = 7.8 Hz, 1H), 6.86 (d, *J* = 7.8 Hz, 1H), 4.44-4.35 (m, 1H), 3.16 (d, *J* = 14.4 Hz, 1H), 2.70 (dd, *J* = 14.7 Hz, 11.2 Hz, 1H), 2.64 (ddd, *J* = 14.7 Hz, 2.5 Hz, 1.5 Hz, 1H), 2.53 (dd, *J* = 14.4 Hz, 1.5 Hz, 1H), 1.80-1.70 (m, 1H), 1.60-1.55 (m, 1H), 1.48-1.25 (m, 2H), 0.86 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 205.6, 172.6, 143.4, 133.7, 130.1, 129.6, 128.3, 127.9, 126.5, 125.2, 123.1, 110.3, 78.4, 73.0, 47.5, 45.6, 38.3, 18.2, 13.7. ESI-HRMS: *m*/*z* calcd for $C_{21}H_{22}O_3N$ ($[M+H]^+$) 336.1594, found 336.1602

Reaction of 1j to give 2j/3j

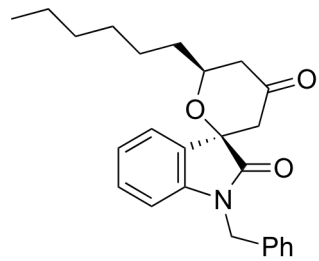
Synthesized from **1j** (78.2 mg, 0.20 mmol) in the presence of TfOH (0.05 equiv), 48 h, **2g** (65.1 mg, 83%) and **3g** (11.2 mg, 14%).

Compound 2j



R_f 0.33 (hexane/EtOAc = 4:1), colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.37 (dd, *J* = 7.5 Hz, 0.9 Hz, 1H), 7.33-7.23 (m, 5H), 7.21 (td, *J* = 7.5 Hz, 1.2 Hz, 1H), 7.07 (td, *J* = 7.5 Hz, 0.8 Hz, 1H), 6.69 (d, *J* = 7.5 Hz, 1H), 4.97-4.91 (m, 1H), 4.87 (d, *J* = 15.7 Hz, 1H), 4.82 (d, *J* = 15.7 Hz, 1H), 2.81 (d, *J* = 15.0 Hz, 1H), 2.65 (ddd, *J* = 15.0 Hz, 2.5 Hz, 1.5 Hz, 1H), 2,57 (dd, *J* = 15.0 Hz, 1.5 Hz, 1H), 2.41 (dd, *J* = 15.0 Hz, 11.0 Hz, 1H), 1.72-1.65 (m, 1H), 1.60-1.50 (m, 1H), 1.45-1.20 (m, 8H), 0.85 (t, *J* = 6.8Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 204.4, 174.6, 142.2, 135.2, 130.2, 128.8, 127.7, 127.1, 123.9, 123.3, 109.5, 77.7, 71.7, 46.8, 45.5, 43.3, 36.3, 31.6, 29.0, 24.8, 22.4, 14.0. ESI-HRMS: *m*/*z* calcd for $C_{25}H_{30}O_3N$ ($[M+H]^+$) 392.2220, found 392.2213.

Compound 3j



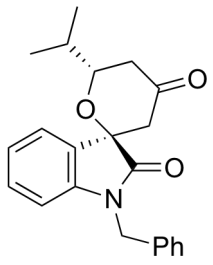
R_f 0.28 (hexane/EtOAc = 4:1), colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.34-7.24 (m, 5H), 7.23 (t, *J* = 7.5 Hz, 1H), 7.18 (d, *J* = 7.5 Hz, 1H), 6.99 (t, *J* = 7.5 Hz, 1H), 6.74 (d, *J* = 7.5 Hz, 1H), 4.97 (d, *J* = 15.8 Hz, 1H), 4.87 (d, *J* = 15.8 Hz, 1H), 4.34-4.27 (m, 1H), 3.16 (d, *J* = 14.3 Hz, 1H), 2.68 (dd, *J* = 15.0 Hz, 11.0 Hz, 1H), 2.65-2.60 (m, 1H), 2.43 (d, *J* = 14.3 Hz, 1H), 1.80-1.70 (m, 1H), 1.65-1.50 (m, 1H), 1.40-1.15 (m, 8H), 0.81 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 205.6, 173.4, 142.4, 135.0, 130.1, 128.8, 128.0, 127.7, 127.0, 125.1, 122.6, 110.0, 78.4, 73.3, 47.7, 45.5, 43.8, 36.3, 31.5, 28.8, 24.8, 22.4, 13.9. ESI-HRMS: *m*/*z* calcd for $C_{25}H_{30}O_3N$ ($[M+H]^+$) 392.2220, found 392.2225.

Reaction of 1k to give 2k/3k

Synthesized from **1k** (34.9 mg, 0.10 mmol) in the presence of TfOH (0.02 equiv) in $CHCl_3$ (2.0 mL), 144 h, **2k** (23.0 mg, 66%) and **3k** (4.9 mg, 14%).

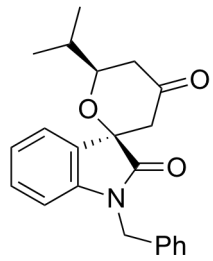
Synthesized from **1k** (69.8 mg, 0.20 mmol) in the presence of TfOH (0.05 equiv), 96 h, **2k** (53.0 mg, 76%) and **3k** (16.6 mg, 24%).

Compound 2k



R_f 0.25 (hexane/EtOAc = 4:1), colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36 (d, *J* = 7.5 Hz, 1H), 7.33-7.29 (m, 2H), 7.28-7.21 (m, 4H), 7.08 (t, *J* = 7.5 Hz, 1H), 6.69 (d, *J* = 7.5 Hz, 1H), 4.89 (d, *J* = 15.7 Hz, 1H), 4.81 (d, *J* = 15.7 Hz, 1H), 4.71 (ddd, *J* = 11.0 Hz, 6.0 Hz, 3.0 Hz, 1H), 2.80 (d, *J* = 15.0 Hz, 1H), 2.64 (ddd, *J* = 15.0 Hz, 3.0 Hz, 1.5 Hz, 1H), 2.54 (dd, *J* = 15.0 Hz, 1.5 Hz, 1H), 2.44 (dd, *J* = 15.0 Hz, 11.0 Hz, 1H), 1.90-1.80 (m, 1H), 0.95 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (125 MHz, $CDCl_3$: δ 204.9, 174.7, 142.2, 135.2, 130.1, 128.9, 128.8, 127.7, 127.0, 123.9, 123.3, 109.5, 77.5, 76.1, 45.5, 43.6, 43.3, 33.0, 18.0, 17.3. ESI-HRMS: calcd for *m*/*z* $C_{22}H_{24}O_3N$ ($[M+H]^+$) 350.1751, found 350.1759.

Compound 3k

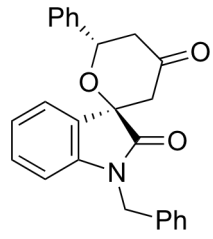


R_f 0.21 (hexane/EtOAc = 4:1), colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35-7.25 (m, 5H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.20 (d, *J* = 7.6 Hz, 1H), 7.00 (t, *J* = 7.6 Hz, 1H), 6.72 (d, *J* = 7.6 Hz, 1H), 4.97 (d, *J* = 15.8 Hz, 1H), 4.86 (d, *J* = 15.8 Hz, 1H), 4.03 (ddd, *J* = 11.0 Hz, 6.3 Hz, 3.0 Hz, 1H), 3.13 (d, *J* = 14.3 Hz, 1H), 2.72 (dd, *J* = 14.5 Hz, 11.0 Hz, 1H), 2.66-2.61 (m, 1H), 2.44 (dd, *J* = 14.3 Hz, 1.3 Hz, 1H), 1.99-1.87 (m, 1H), 0.93 (d, *J* = 6.8 Hz, 3H), 0.91 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 206.1, 173.5, 142.4, 135.0, 130.1, 128.8, 128.0, 127.7, 127.0, 125.0, 122.6, 110.0, 78.2, 77.9, 45.4, 44.7, 43.8, 33.2, 18.3, 17.5. ESI-HRMS: *m*/*z* calcd for $C_{22}H_{24}O_3N$ ($[M+H]^+$) 350.1751, found 350.1746.

Reaction of 1l to give 2l

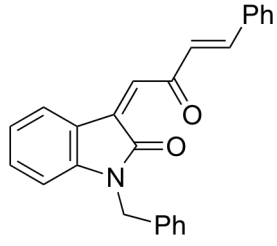
Synthesized from **1l** (63.0 mg, 0.16 mmol) in the presence of TfOH (0.05 equiv) in $CHCl_3$ (3.2 mL), 192 h, **2l** (29.0 mg, 46%). Byproduct **4l**[1] (12.6 mg, 20%) was also isolated.

Compound 2l



R_f 0.22 (hexane/EtOAc = 4:1), colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.48 (d, *J* = 7.5 Hz, 1H), 7.45-7.42 (m, 2H), 7.40-7.21 (m, 9H), 7.11 (t, *J* = 7.5 Hz, 1H), 6.70 (d, *J* = 7.5 Hz, 1H), 6.03 (dd, *J* = 11.0 Hz, 3.2 Hz, 1H), 4.92 (d, *J* = 15.7 Hz, 1H), 4.79 (d, *J* = 15.7 Hz, 1H), 2.98 (d, *J* = 14.9 Hz, 1H), 2.89 (ddd, *J* = 14.7 Hz, 3.2 Hz, 1.7 Hz, 1H), 2.76 (dd, *J* = 14.7 Hz, 11.0 Hz, 1H), 2.60 (dd, *J* = 14.9 Hz, 1.7 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 203.3, 174.5, 142.3, 140.6, 135.1, 130.4, 128.9, 128.6, 128.5, 128.2, 127.8, 127.2, 126.0, 124.0, 123.5, 109.7, 78.1, 73.6, 48.8, 45.8, 43.5. ESI-HRMS: *m*/*z* calcd for $C_{25}H_{22}O_3N$ ($[M+H]^+$) 384.1594, found 384.1589

Compound 4l[1]



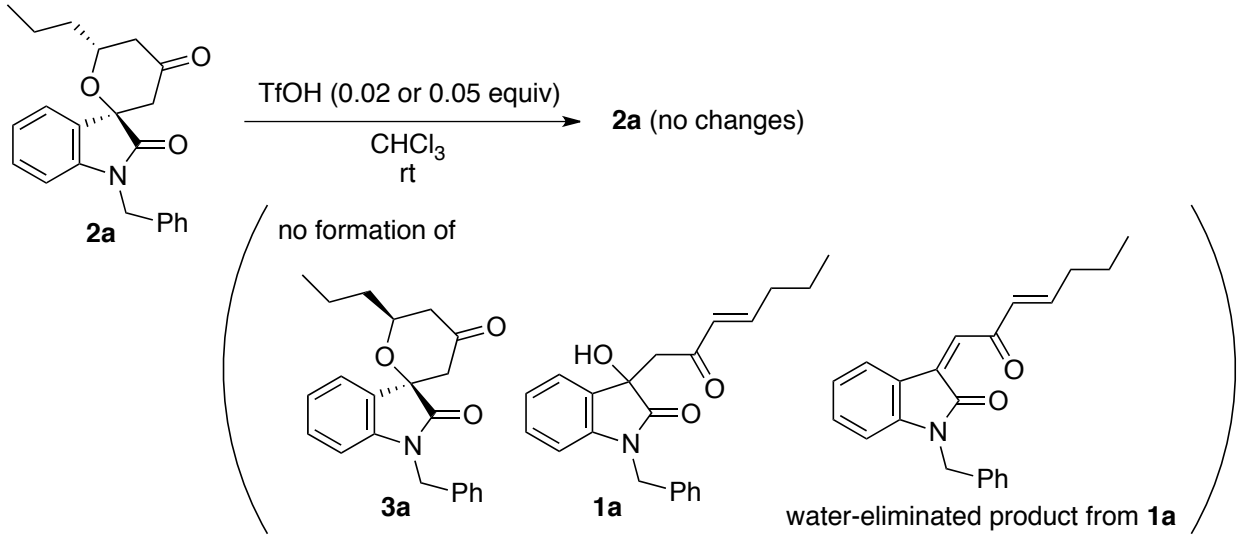
R_f 0.38 (hexane/EtOAc = 4:1), red solid. ^{1}H and ^{13}C NMR data were consistent with the previously reported data.[1] ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.46 (d, *J* = 7.7 Hz, 1H), 7.74 (d, *J* = 16.2 Hz, 1H), 7.59-7.54 (m, 3H), 7.40-7.36 (m, 3H), 7.29-7.18 (m, 6H), 7.02 (d, *J* = 16.2 Hz, 1H),

6.97 (t, J = 7.7 Hz, 1H), 6.64 (d, J = 7.7 Hz, 1H), 4.91 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 189.9, 168.3, 145.2, 145.1, 135.9, 135.4, 134.2, 132.6, 131.0, 129.0, 128.8, 128.6, 128.1, 128.0, 127.7, 127.5, 127.2, 122.9, 120.4, 109.1, 43.8.

4. Stability Evaluations of Products 2 and 3

Treatment of 2a under the TfOH-catalyzed oxa-Michael reaction conditions

To a solution of **2a** (11.0 mg, 0.031 mmol, 1.0 equiv) in $CHCl_3$ (super dehydrated, 620 μL), TfOH (0.56 M in THF, 1.1 μL, 0.00062 mmol, 0.02 equiv) was added at room temperature (25 °C), and the mixture was stirred at the same temperature for 24 h. The mixture was diluted with $CDCl_3$ and analyzed by ^{1}H NMR. No changes of **2a** were detected.



To a solution of **2a** (11.0 mg, 0.031 mmol, 1.0 equiv) in $CHCl_3$ (super dehydrated, 620 μL), TfOH (0.56 M in THF, 2.8 μL, 0.0016 mmol, 0.05 equiv) was added at room temperature (25 °C), and the mixture was stirred at the same temperature for 24 h. The mixture was diluted with $CDCl_3$ and analyzed by ^{1}H NMR. No changes of **2a** were detected.

Treatment of 2a/3a (3:1) under the TfOH-catalyzed oxa-Michael reaction conditions

To a solution of **2a**/**3a** (3:1, 32.0 mg, 0.091 mmol, 1.0 equiv) in $CHCl_3$ (super dehydrated, 1.82 mL), TfOH (0.56 M in THF, 3.3 μL, 0.0018 mmol, 0.02 equiv) was added at room temperature (25 °C), and the mixture was stirred at the same temperature for 24 h. The mixture was diluted with $CDCl_3$ and analyzed by ^{1}H NMR. No changes of the ratio of **2a**/**3a** and no decompositions were detected.

To a solution of **2a**/**3a** (3:1, 30.0 mg, 0.085 mmol, 1.0 equiv) in $CHCl_3$ (super dehydrated, 1.70 mL), TfOH (0.56 M in THF, 7.5 μL, 0.0042 mmol, 0.05 equiv) was added at room temperature (25 °C), and the mixture was stirred at the same temperature for 24 h. The mixture was diluted with $CDCl_3$ and analyzed by ^{1}H NMR. No changes of the ratio of **2a**/**3a** and no decompositions were detected.

Treatment of 3a under the TfOH-catalyzed oxa-Michael reaction conditions

To a solution of **3a** (2.0 mg, 0.0057 mmol, 1.0 equiv) in $CHCl_3$ (super dehydrated, 114 μL), TfOH (0.28 M in THF, 0.4 μL, 0.0001 mmol, 0.02 equiv) was added at room temperature (25 °C), and the mixture was stirred at the same temperature for 24 h. The mixture was diluted with $CDCl_3$ and analyzed by ^{1}H NMR. No changes of **3a** were detected.

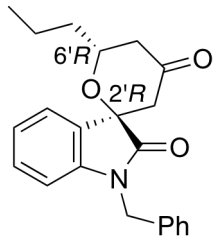
5. Intramolecular Oxa-Michael Reactions of (*R*)-1 (Scheme 2)

Reactions of (*R*)-1a

To a solution of (*R*)-**1a** (er 94:6, 50.0 mg, 0.14 mmol, 1.0 equiv) in $CHCl_3$ (super dehydrated, 2.8 mL), TfOH (0.56 M in THF, 12.2 μL, 0.007 mmol, 0.05 equiv) was added at room temperature (25 °C), and the mixture was stirred for 16 h (monitored by TLC). The dr (**2a**:**3a**) was determined by ^{1}H NMR analysis to be 7:1. The mixture was purified by flash column chromatography (hexane/EtOAc = 95:5 then 90:10) to give (2'*R*,6'*R*)-**2a** (40.0 mg, 80%, er 94:6) and (2'*R*,6'*S*)-**3** (5.5 mg, 11%, er 94:6).

Compound (2'*R*,6'*R*)-2a (major diastereomer)

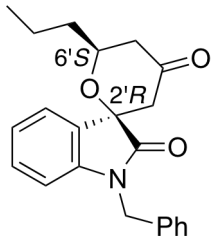
(2'*R*,6'*R*)-1-Benzyl-6'-propyl-5',6'-dihydrospiro[indoline-3,2'-pyran]-2,4'(3'*H*)-dione



Colorless solid. HPLC (Daicel Chiralpak IC-3, hexane/*i*-PrOH = 90:10, 1.0 mL/min, λ = 254 nm): t_R 37 min (major enantiomer, (2'*R*,6'*R*)-**2a**), 43 min (minor enantiomer, (2'*S*,6'*S*)-**2a**).

Compound (2'*R*,6'*S*)-3a (minor diastereomer)

(2'*R*,6'*S*)-1-Benzyl-6'-propyl-5',6'-dihydrospiro[indoline-3,2'-pyran]-2,4'(3'*H*)-dione



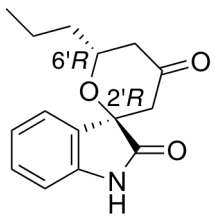
Colorless solid. HPLC (Daicel Chiralpak IC-3, hexane/*i*-PrOH = 90:10, 1.0 mL/min, λ = 254 nm): t_R 52 min (major enantiomer, (2'*R*,6'*S*)-**3a**), 79 min (minor enantiomer, (2'*S*,6'*R*)-**3a**).

Reactions of (*R*)-1b

To a solution of (*R*)-**1b** (er >99.5:0.5, 42.0 mg, 0.16 mmol, 1.0 equiv) in $CHCl_3$ (super dehydrated, 3.2 mL), TfOH (0.56 M in THF, 14.3 μL, 0.008 mmol, 0.05 equiv) was added at room temperature (25 °C), and the mixture was stirred for 30 h (monitored by TLC). The dr (**2b**:**3b**) was determined by ^{1}H NMR analysis to be 4:1. The mixture was purified by flash column chromatography (hexane/EtOAc = 95:5 then 90:10) to give (2'*R*,6'*R*)-**2b** (32.4 mg, 77%, er >99.5:0.5) and (2'*R*,6'*S*)-**3b** (7.9 mg, 19%, er >99.5:0.5).

Compound (2'*R*,6'*R*)-2b (major diastereomer)

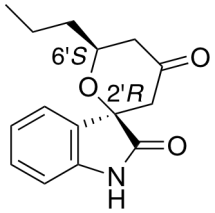
(2'*R*,6'*R*)-1-Benzyl-6'-propyl-5',6'-dihydrospiro[indoline-3,2'-pyran]-2,4'(3'*H*)-dione



Colorless solid. $[\alpha]^{24}_{D}$ +11 (c 0.40, CH_2Cl_2). HPLC (Daicel Chiralpak IC-3, hexane/*i*-PrOH = 70:30, 1.0 mL/min, λ = 254 nm): t_R 18 min (minor enantiomer, (2'*S*,6'*S*)-**2b**), 21 min (major enantiomer, (2'*R*,6'*R*)-**2b**). HPLC (Daicel Chiralpak IB-3, hexane/*i*-PrOH = 90:10, 0.6 mL/min, λ = 254 nm): t_R 17.3 min (minor enantiomer, (2'*S*,6'*S*)-**2b**), 18.2 min (major enantiomer, (2'*R*,6'*R*)-**2b**).

Compound (2'*R*,6'*S*)-3b (minor diastereomer)

(2'*R*,6'*S*)-1-Benzyl-6'-propyl-5',6'-dihydrospiro[indoline-3,2'-pyran]-2,4'(3'*H*)-dione



Colorless solid. HPLC (Daicel Chiralpak IC-3, hexane/*i*-PrOH = 70:30, 1.0 mL/min, λ = 254 nm): t_R 12 min (major enantiomer, (2'*R*,6'*S*)-**3b**), 16 min (minor enantiomer, (2'*S*,6'*R*)-**3b**). HPLC (Daicel Chiralpak IB-3, hexane/*i*-PrOH = 90:10, 0.6 mL/min, λ = 254 nm): t_R 18.5 min (minor enantiomer, (2'*R*,6'*S*)-**3b**), 20.3 min (major enantiomer, (2'*S*,6'*R*)-**3b**).

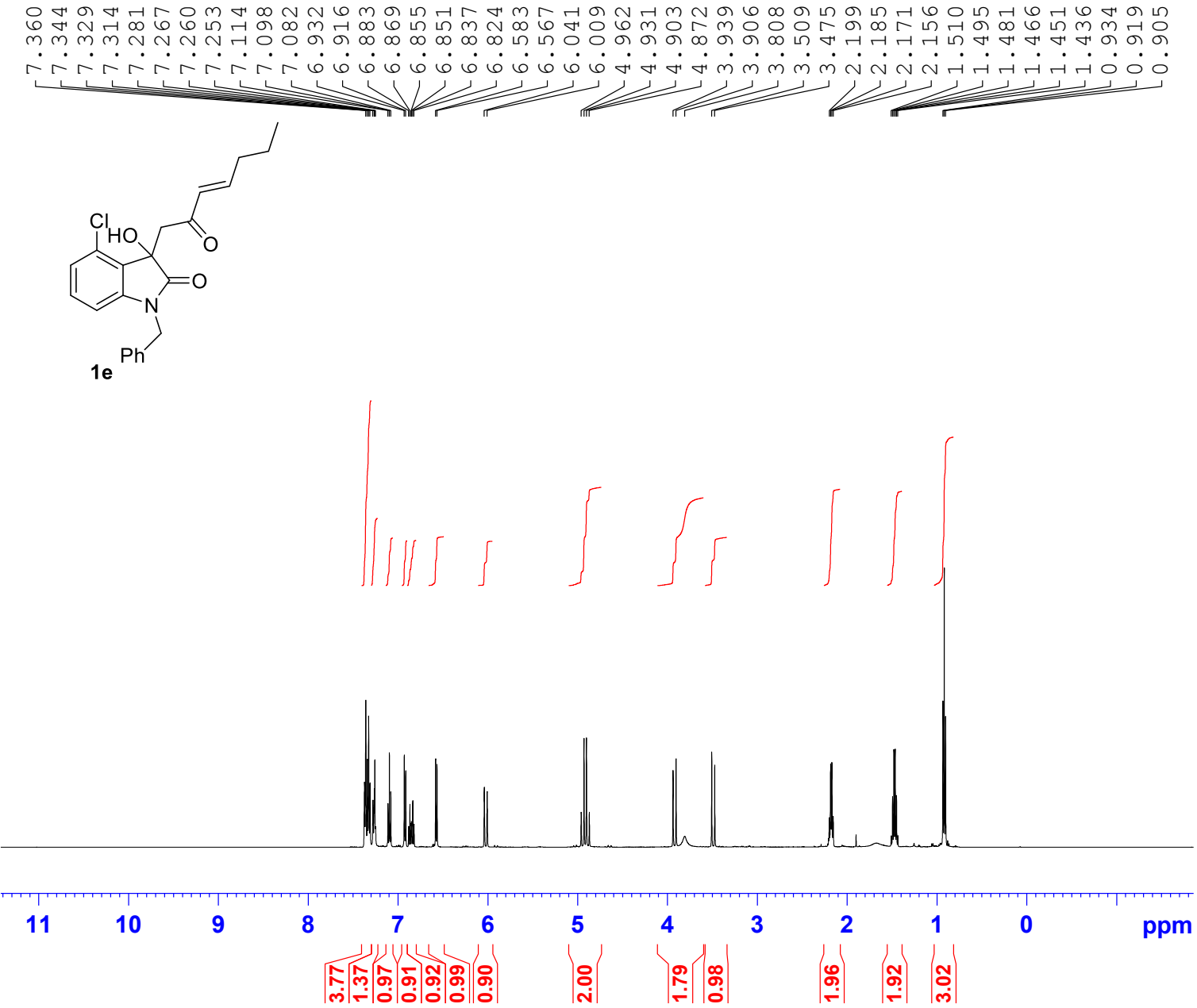
6. References

(1) J. R. Huang, M. Sohail, T. Taniguchi, K. Monde, and F. Tanaka, *Angew. Chem. Int. Ed.* 2017, **56**, 5853.

(2) H.-L. Cui and F. Tanaka, *Chem. Eur. J.* 2013, **19**, 6213.

(3) Q. Guo, M. Bhanushali, and C. G. Zhao, *Angew. Chem. Int. Ed,* 2010, **49**, 9460.

(4) H.-L. Cui, P. V. Chouthaiwale, F. Yin, and F. Tanaka, *Organic. Biomol. Chem.* 2016, **14**, 1777.

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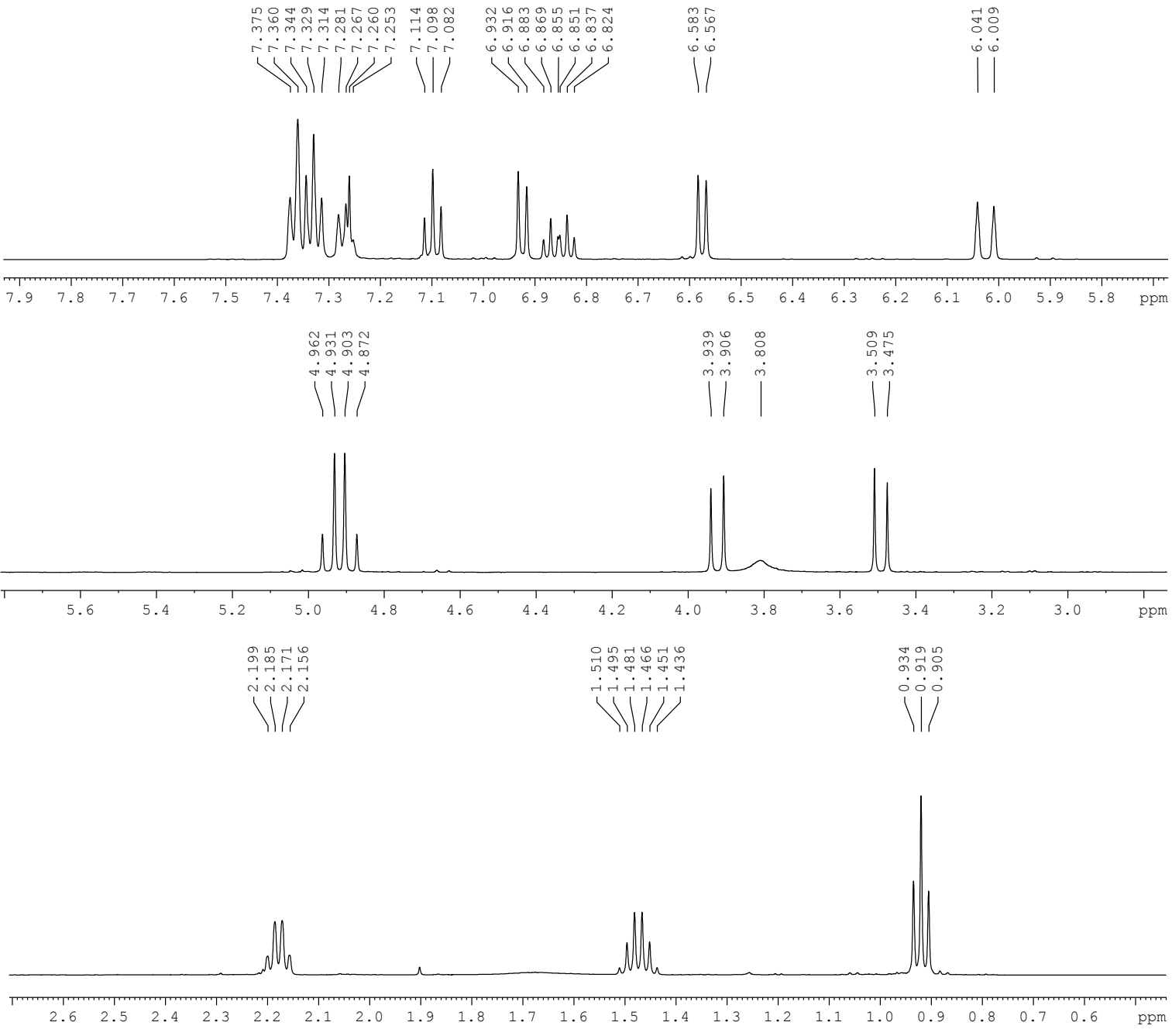


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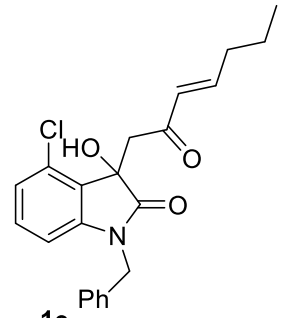

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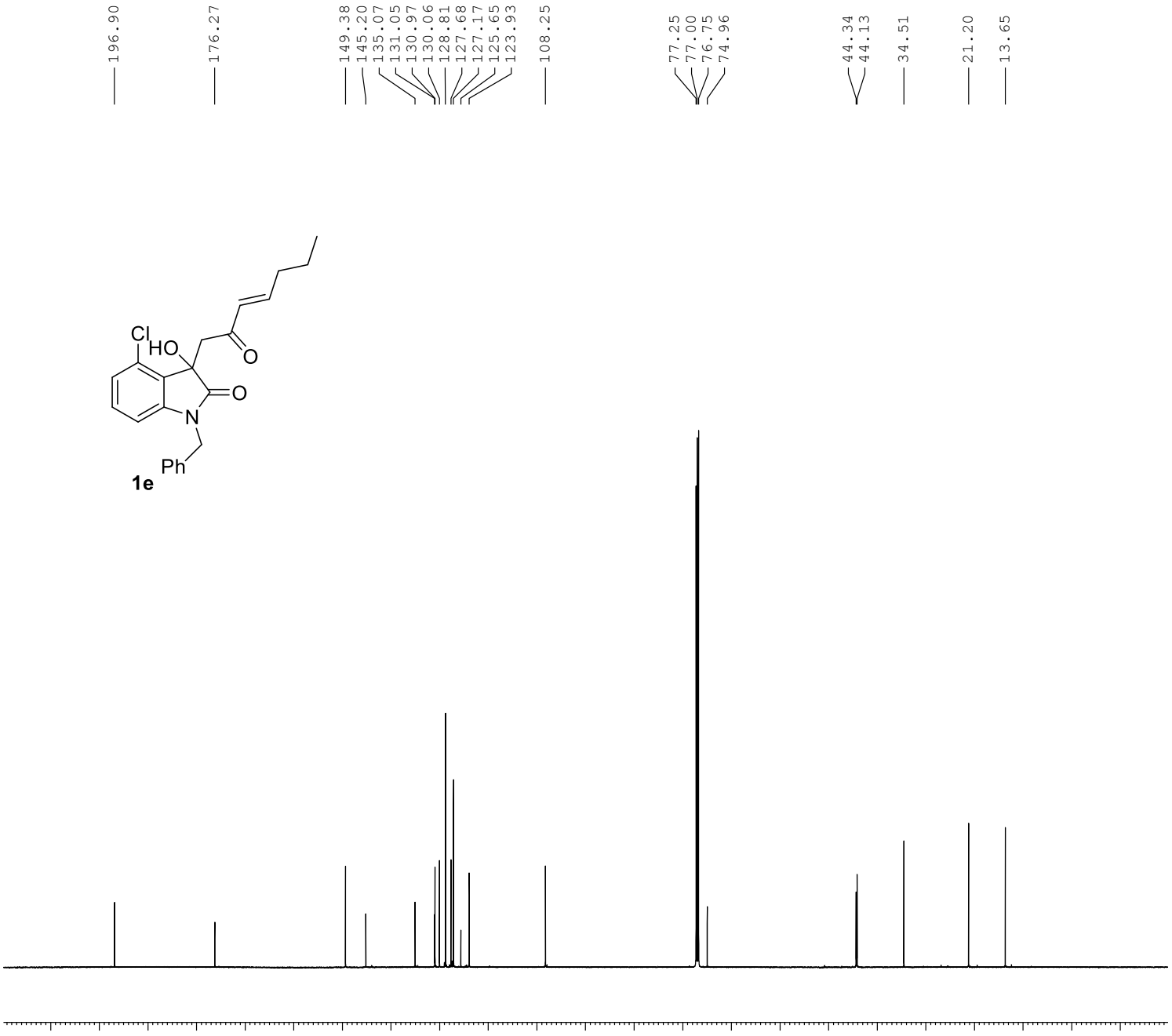


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PC                 1.40
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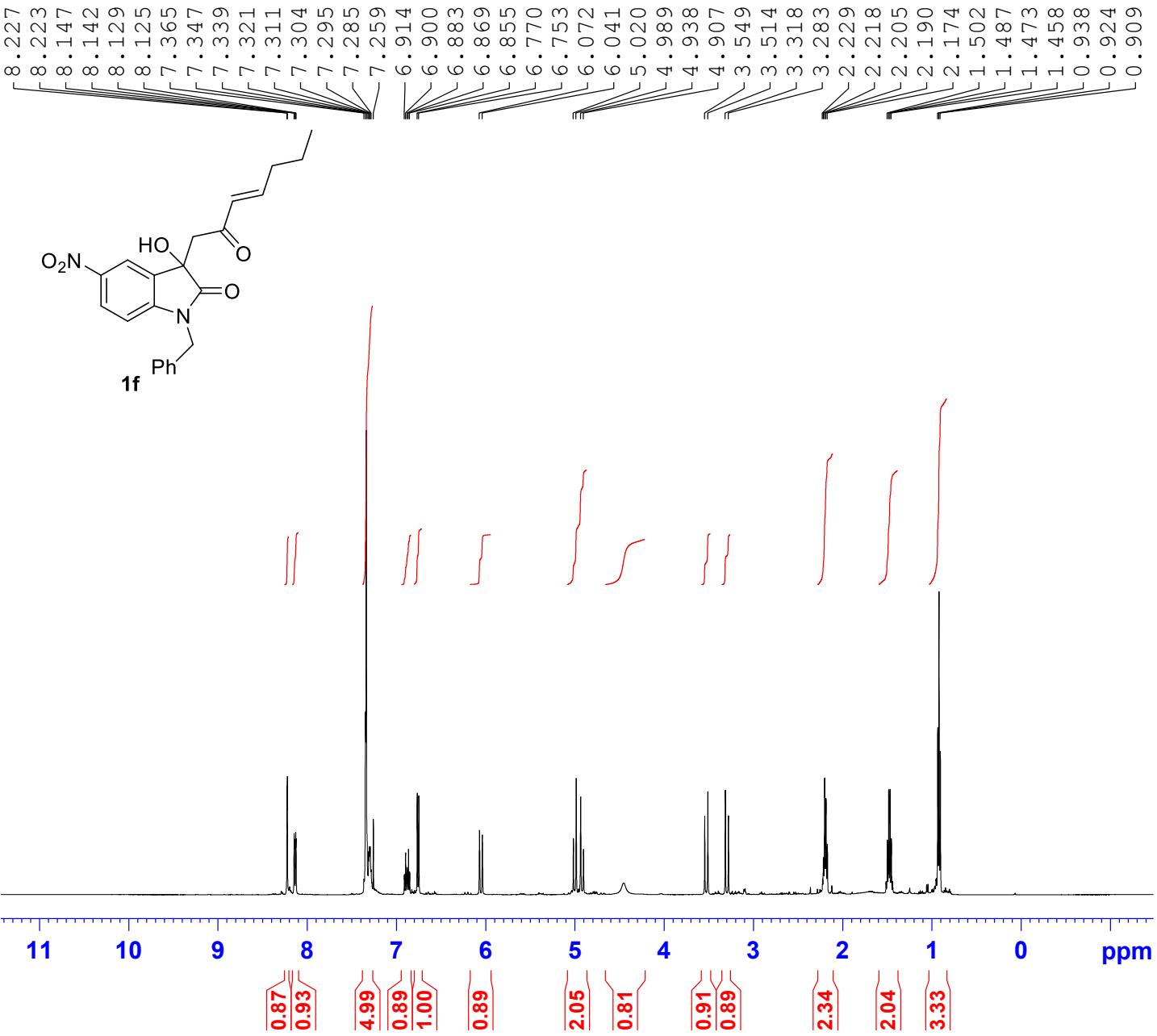
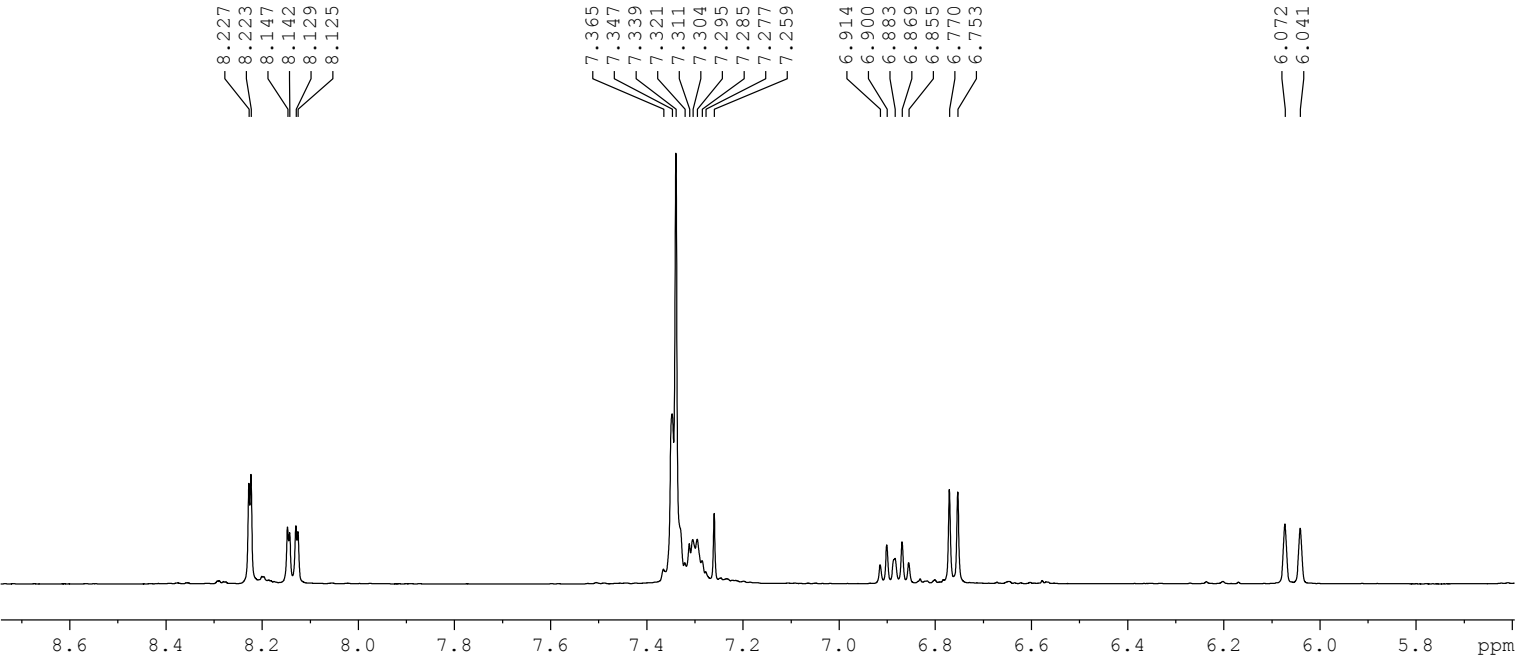
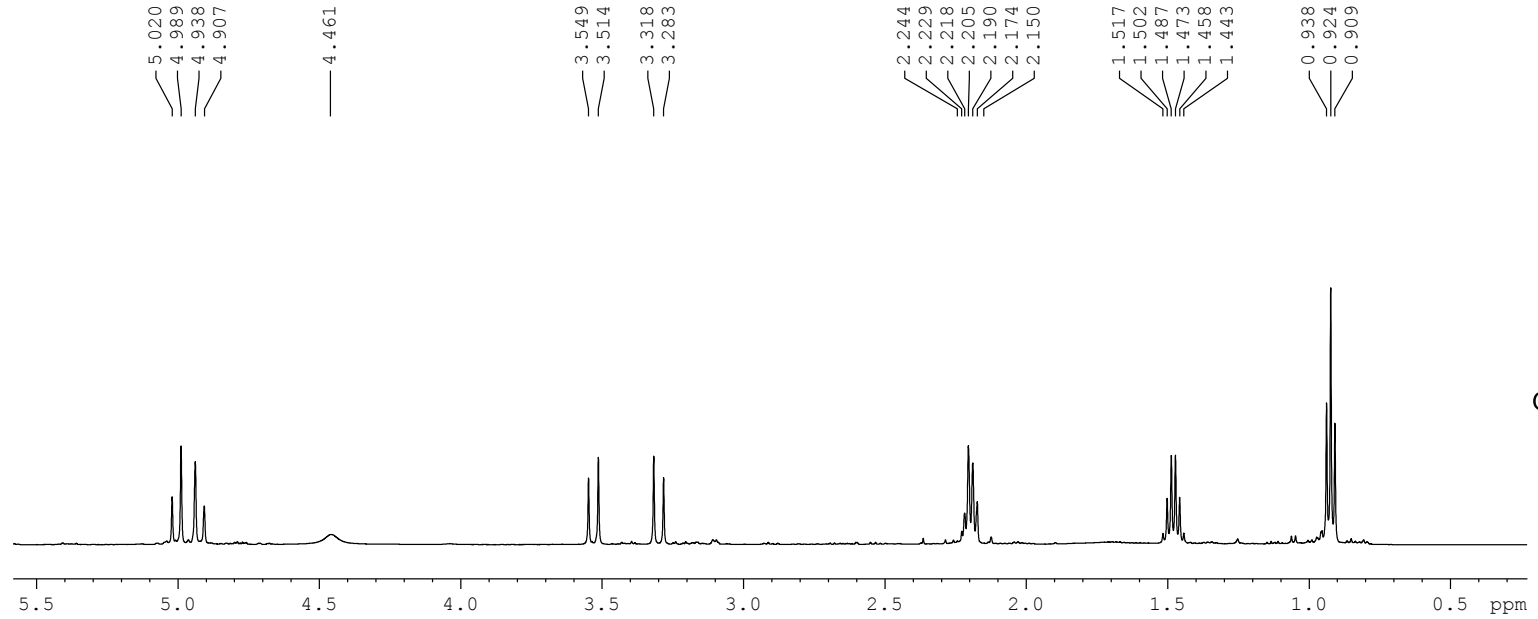



```
Current Data Parameters
NAME      MH-81-Clean spectera
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20190402
Time                18.17 h
INSTRUM             Avance
PROBHD    Z167889_0002 (
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH             10000.000 Hz
FIDRES          0.305176 Hz
AQ              3.2767999 sec
RG                 8.54834
DW                50.000 usec
DE                10.45 usec
TE                298.2 K
D1             1.00000000 sec
TD0                      1
SFO1          500.1330883 MHz
NUC1                    1H
P0                 4.00 usec
P1                12.00 usec
PLW1           7.84999990 W

F2 - Processing parameters
SI                   65536
SF            500.1300126 MHz
WDW                     EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

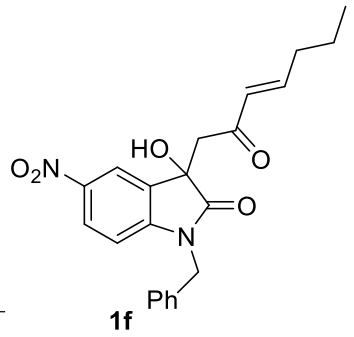




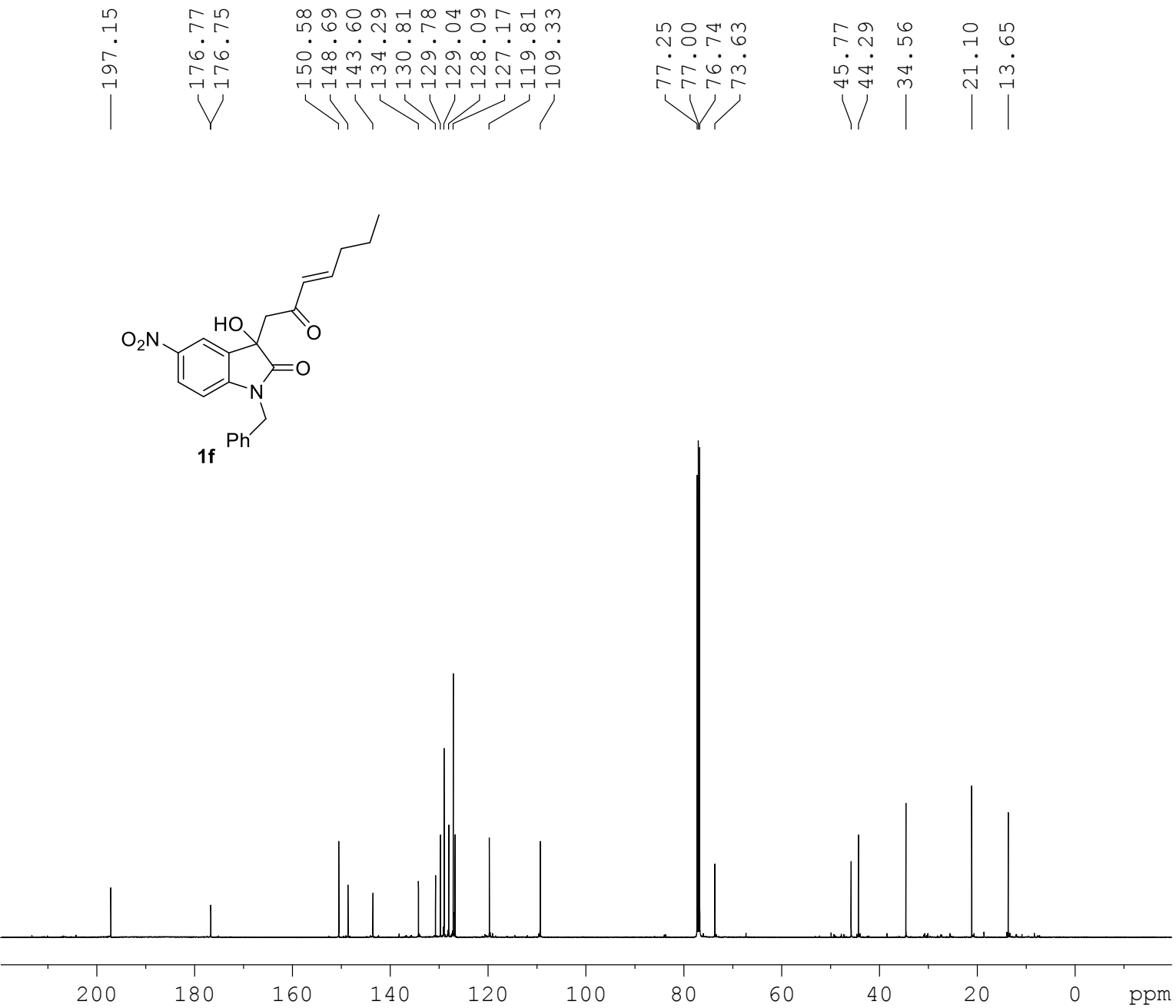

```
Current Data Parameters
NAME      MH-81-Clean spectera
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20190402
Time                18.17 h
INSTRUM             Avance
PROBHD    Z167889_0002 (
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH             10000.000 Hz
FIDRES           0.305176 Hz
AQ               3.2767999 sec
RG                  8.54834
DW                 50.000 usec
DE                 10.45 usec
TE                 298.2 K
D1              1.00000000 sec
TD0                      1
SFO1          500.1330883 MHz
NUC1                    1H
P0                 4.00 usec
P1                12.00 usec
PLW1           7.84999990 W

F2 - Processing parameters
SI                   65536
SF            500.1300126 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



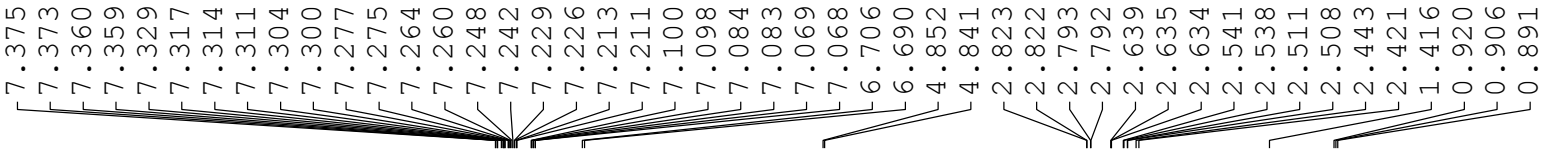
1f

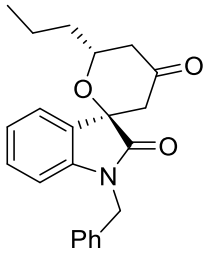



```
Current Data Parameters
NAME      MH-81-Clean spectera- C13
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_           20190402
Time              19.21 h
INSTRUM           Avance
PROBHD   Z167889_0002 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ            1.0878977 sec
RG                  101
DW               16.600 usec
DE               30.00 usec
TE                298.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1                13C
P0                3.33 usec
P1               10.00 usec
PLW1        27.29999924 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2            80.00 usec
PLW2         7.84999990 W
PLW12        0.17662001 W
PLW13        0.08870100 W

F2 - Processing parameters
SI                32768
SF          125.7577955 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



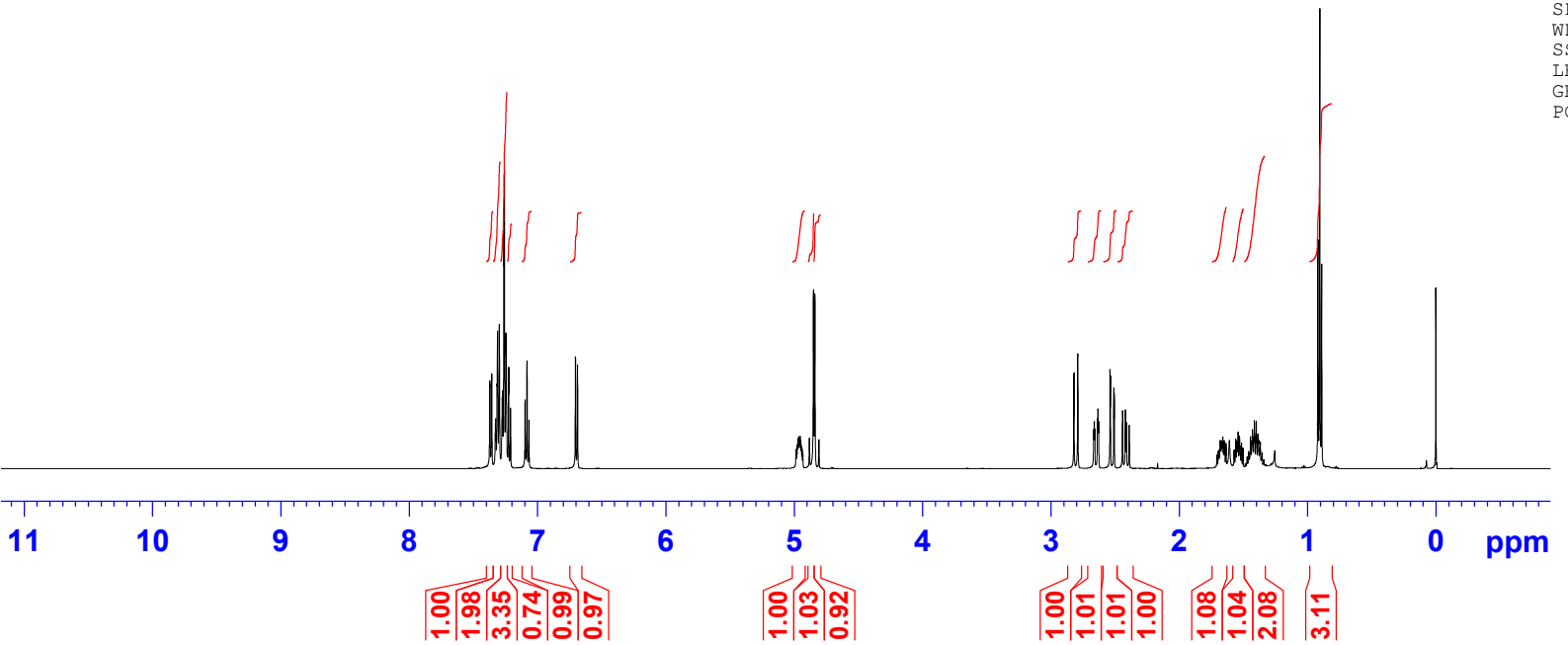


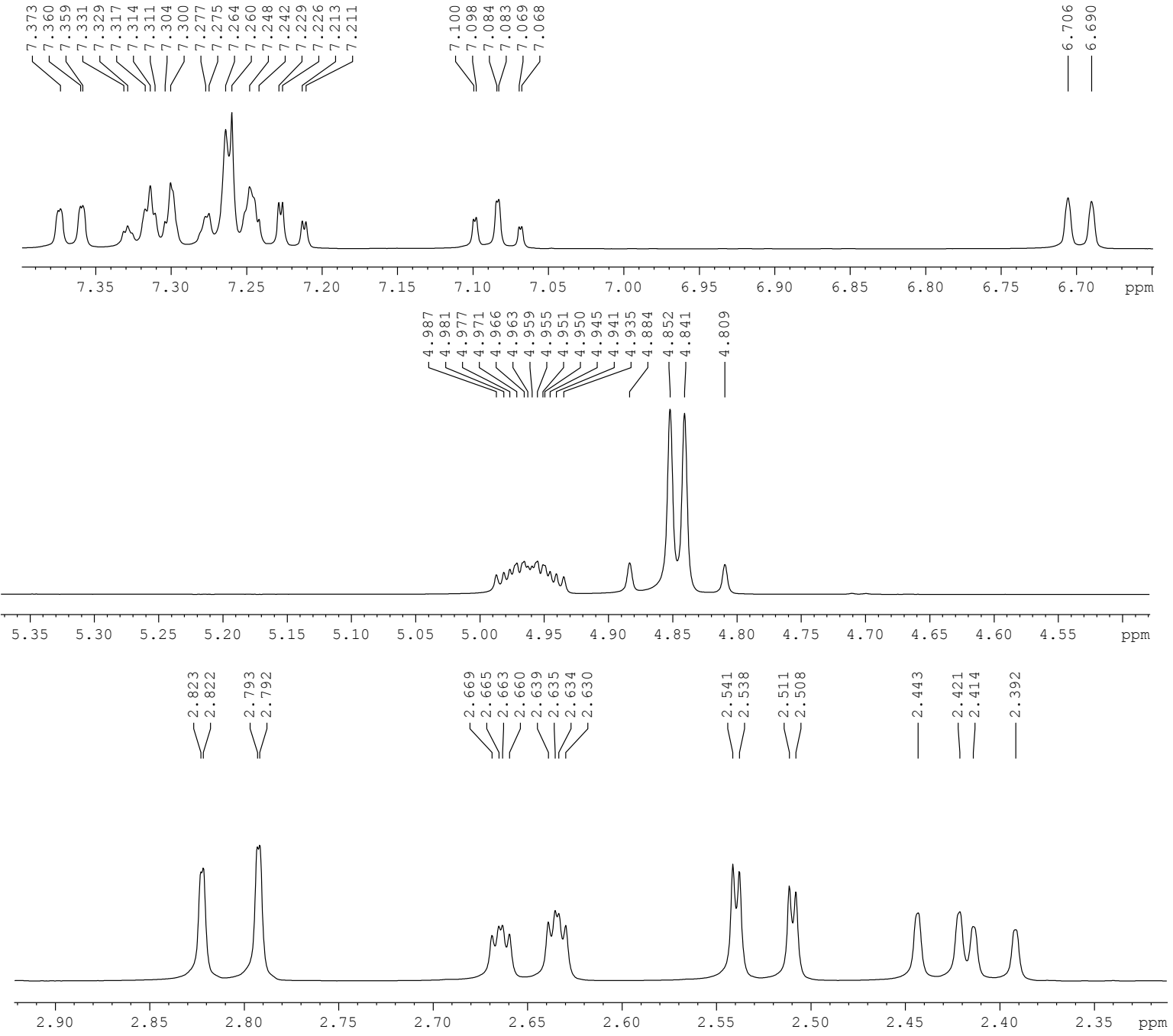
2a

```
Current Data Parameters
NAME      MH-83-MAJOR-CLEAN
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_           20190125
Time              11.47 h
INSTRUM           Avance
PROBHD    Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES        0.305176 Hz
AQ            3.2767999 sec
RG                    32
DW               50.000 usec
DE               11.14 usec
TE               298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                2.67 usec
P1                8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300122 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

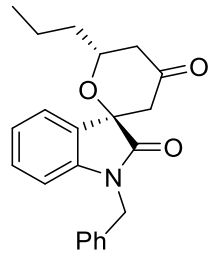




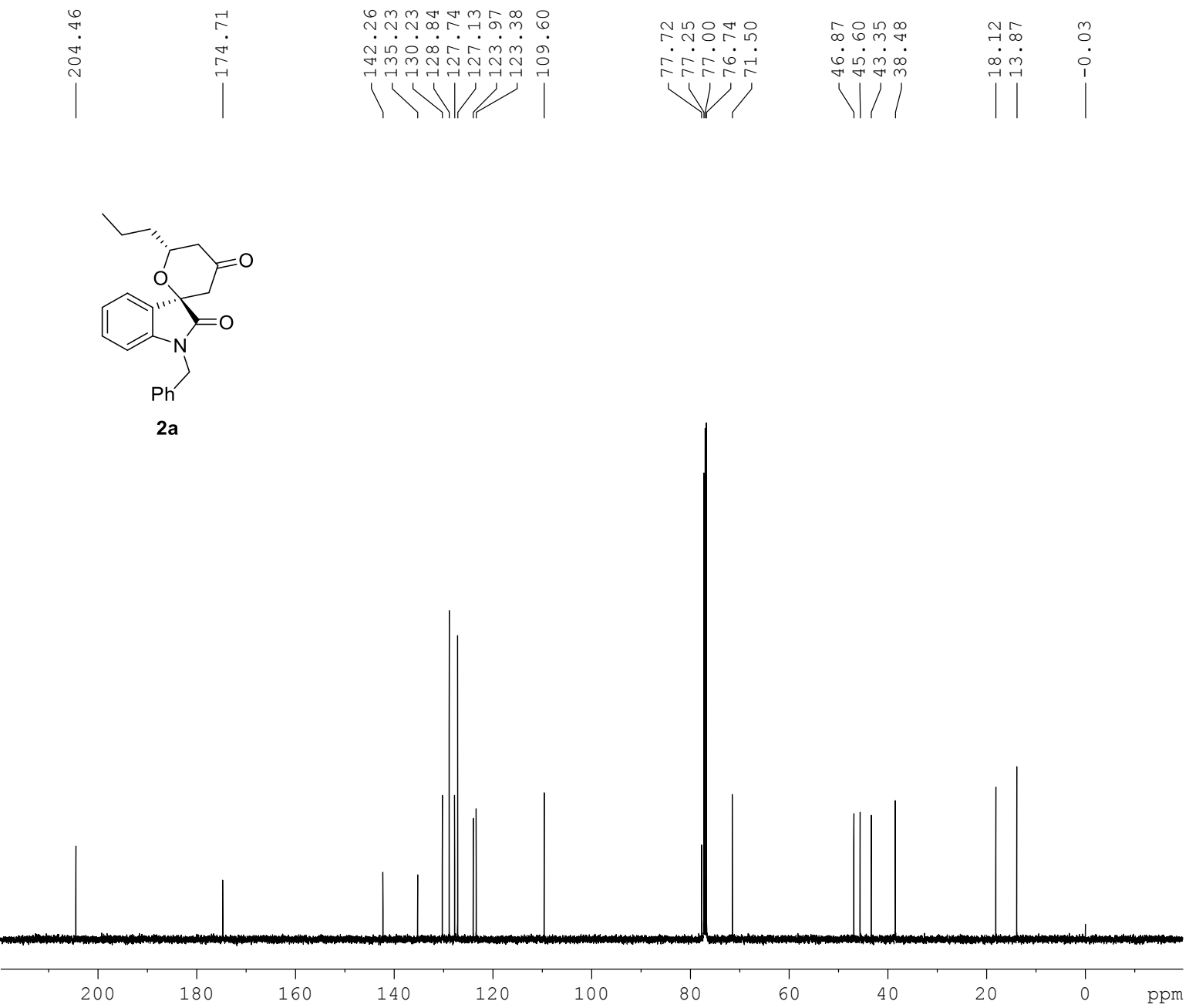

```
Current Data Parameters
NAME      MH-83-MAJOR-CLEAN
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_           20190125
Time              11.47 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          23.68499947 W

F2 - Processing parameters
SI                 65536
SF            500.1300122 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



2a

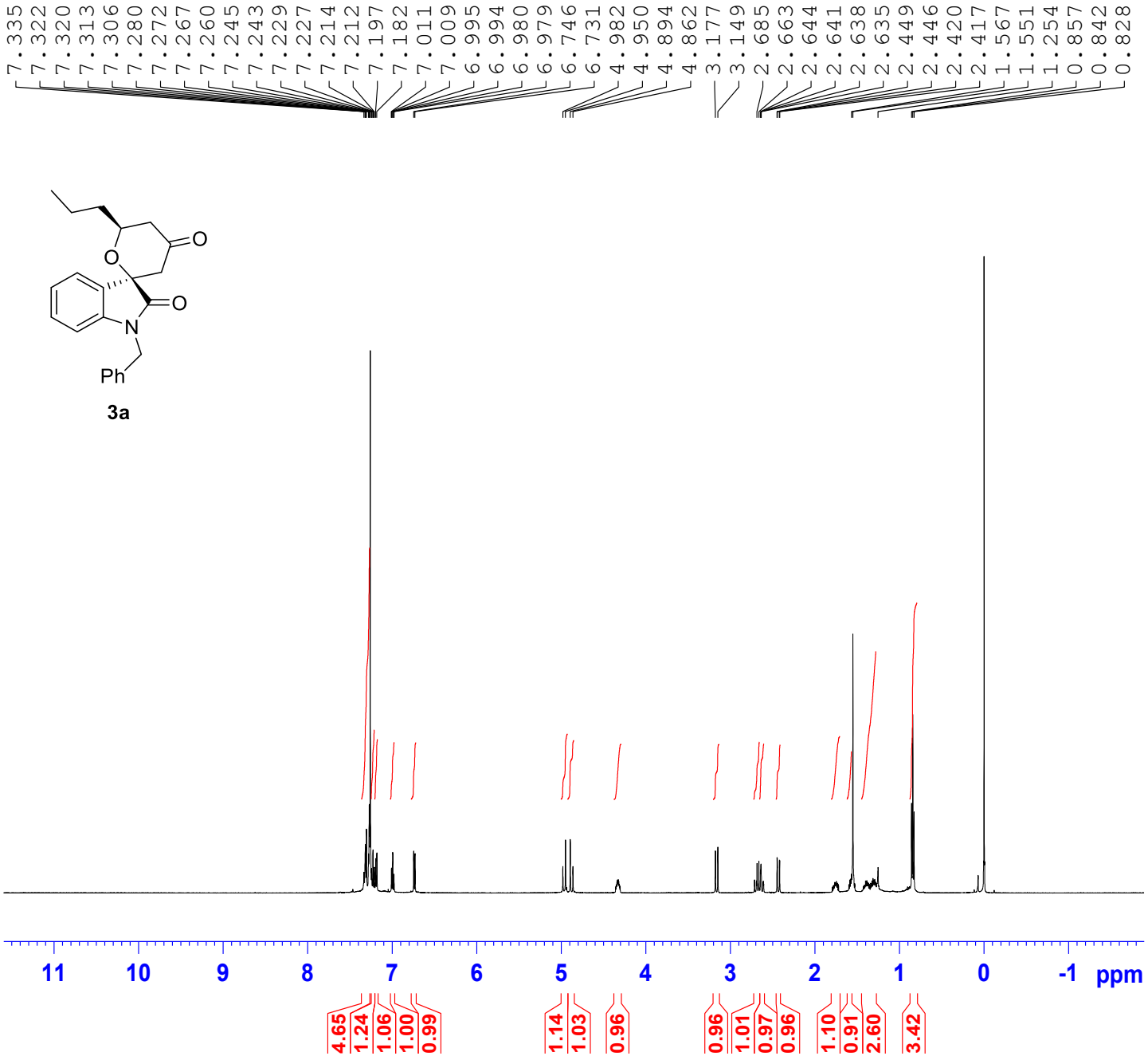


2a

```
Current Data Parameters
NAME      MH-83-MAJOR-CLEAN-C13
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_              20190125
Time                 12.03 h
INSTRUM              Avance
PROBHD    Z151574_0027 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      172
DS                        4
SWH              30120.482 Hz
FIDRES            0.919204 Hz
AQ               1.0878977 sec
RG                      101
DW                   16.600 usec
DE                    6.50 usec
TE                   298.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1
SFO1           125.7703643 MHz
NUC1                   13C
P0                    3.33 usec
P1                   10.00 usec
PLW1           88.26000214 W
SFO2           500.1320005 MHz
NUC2                   1H
CPDPRG[2           waltz65
PCPD2                80.00 usec
PLW2           23.68499947 W
PLW12           0.23014790 W
PLW13           0.11535020 W

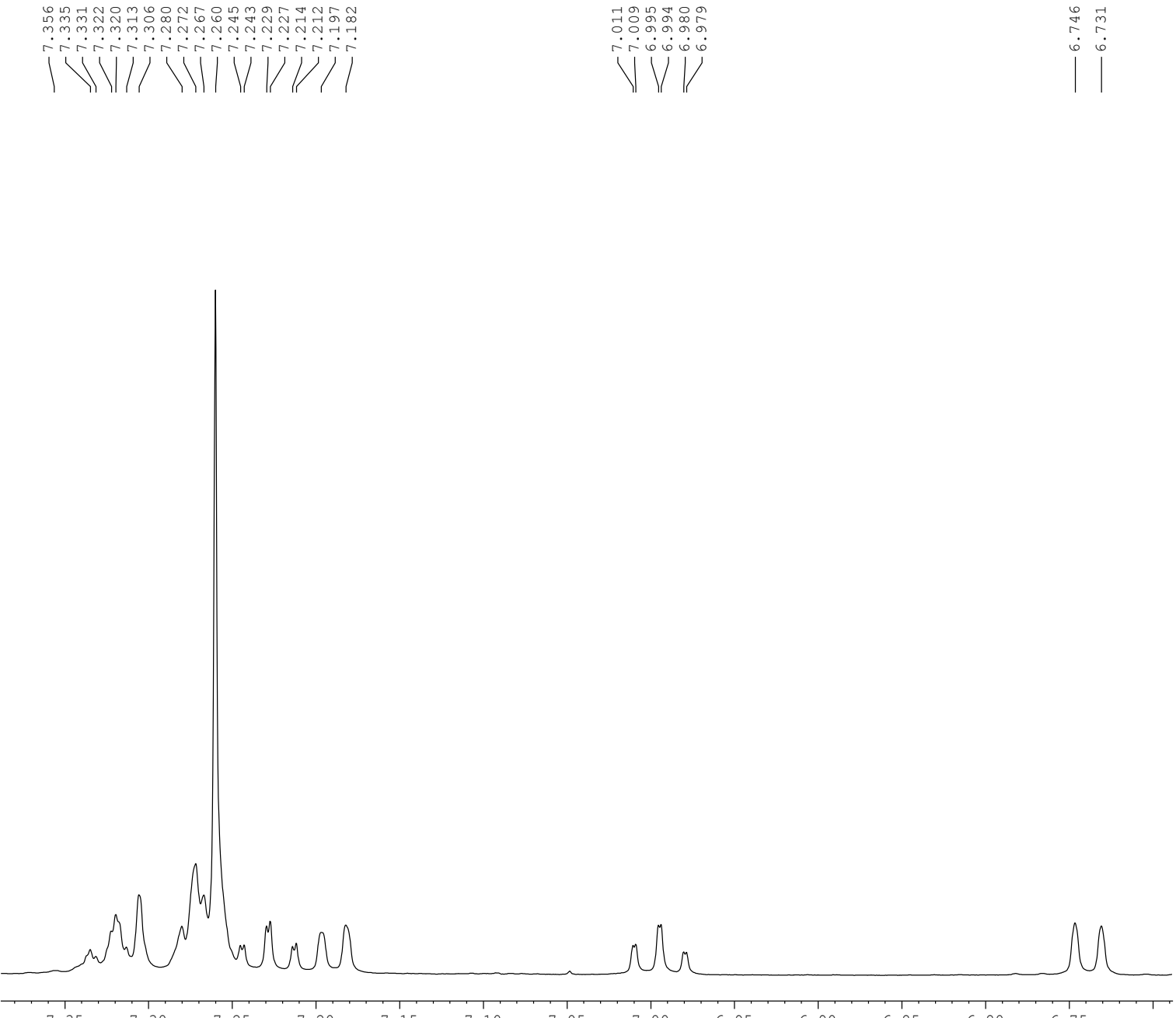
F2 - Processing parameters
SI                    32768
SF             125.7577946 MHz
WDW                      EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
```




```
Current Data Parameters
NAME      MH-83-Minor-clean spectra
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20190126
Time              14.03 h
INSTRUM           Avance
PROBHD    Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES        0.305176 Hz
AQ            3.2767999 sec
RG                    32
DW               50.000 usec
DE               11.14 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                2.67 usec
P1                8.00 usec
PLW1         23.68499947 W

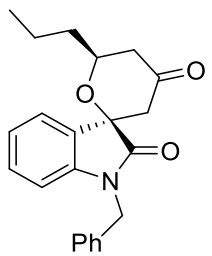
F2 - Processing parameters
SI                 65536
SF           500.1300124 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



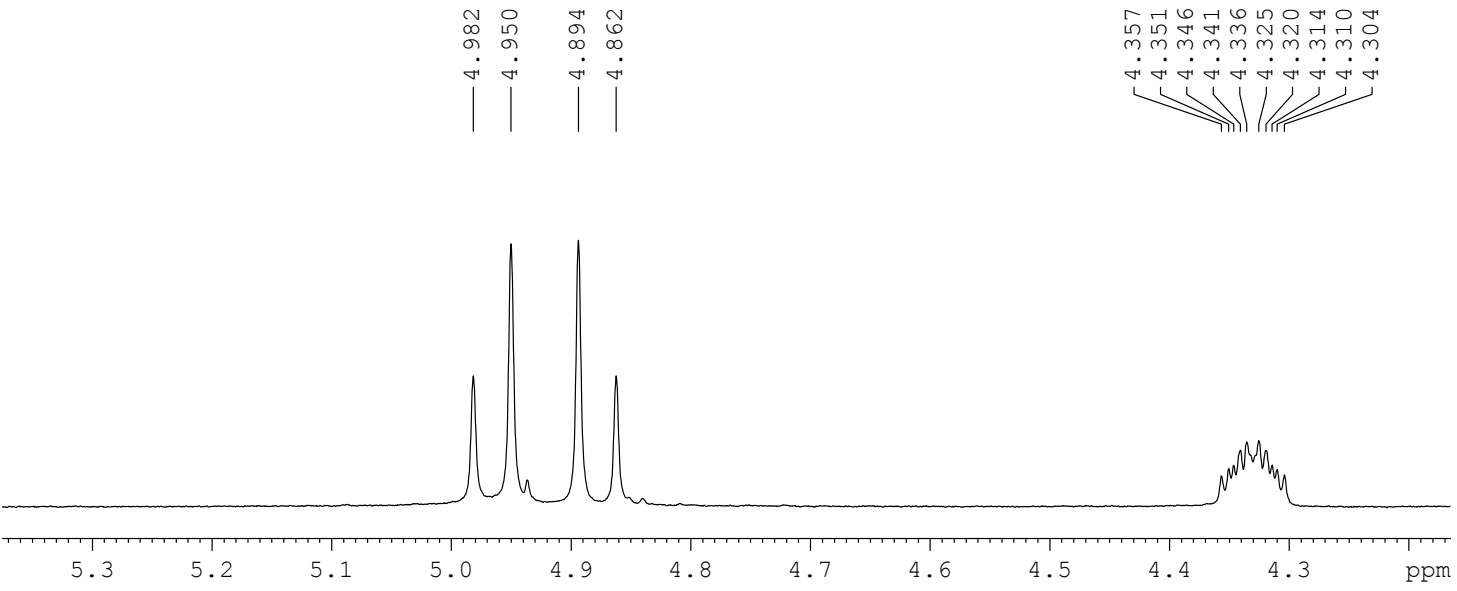

```
Current Data Parameters
NAME      MH-83-Minor-clean spectra
EXPNO                  1
PROCNO                 1

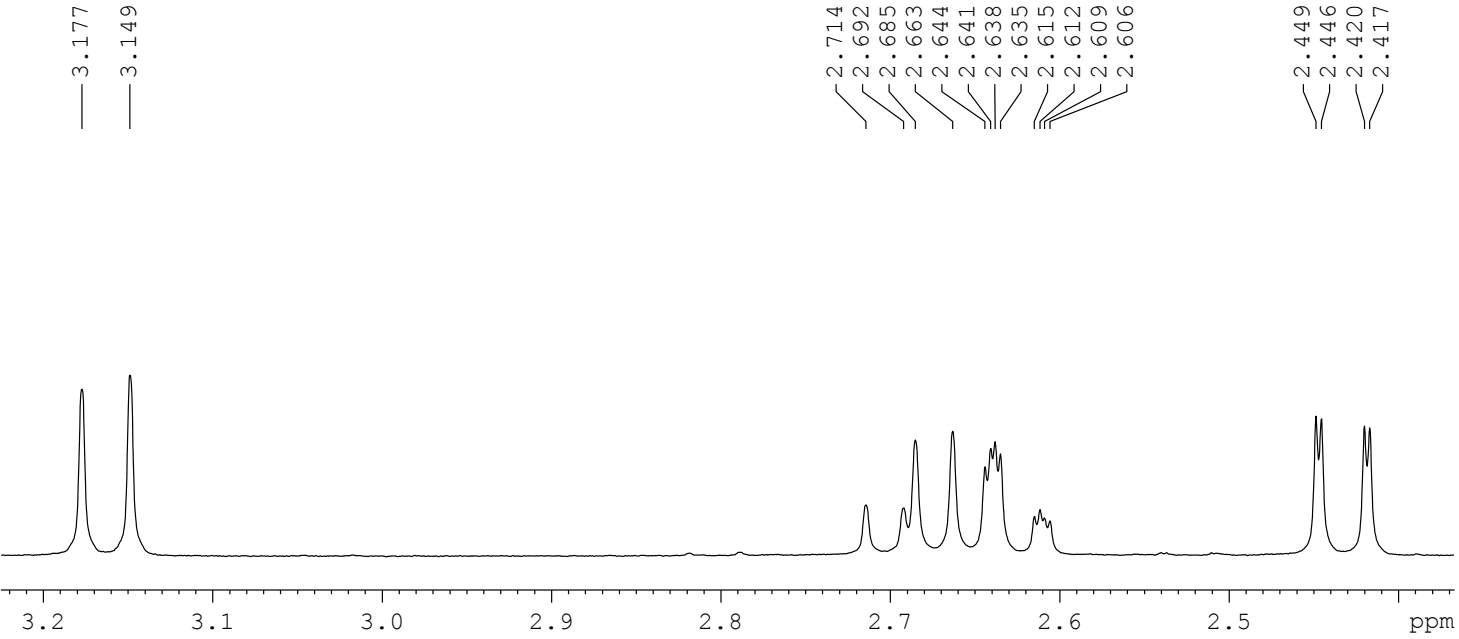
F2 - Acquisition Parameters
Date_           20190126
Time              14.03 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW               50.000 usec
DE               11.14 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                2.67 usec
P1                8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300124 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



3a

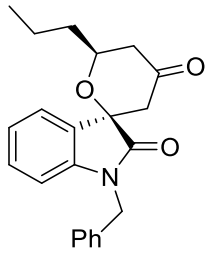




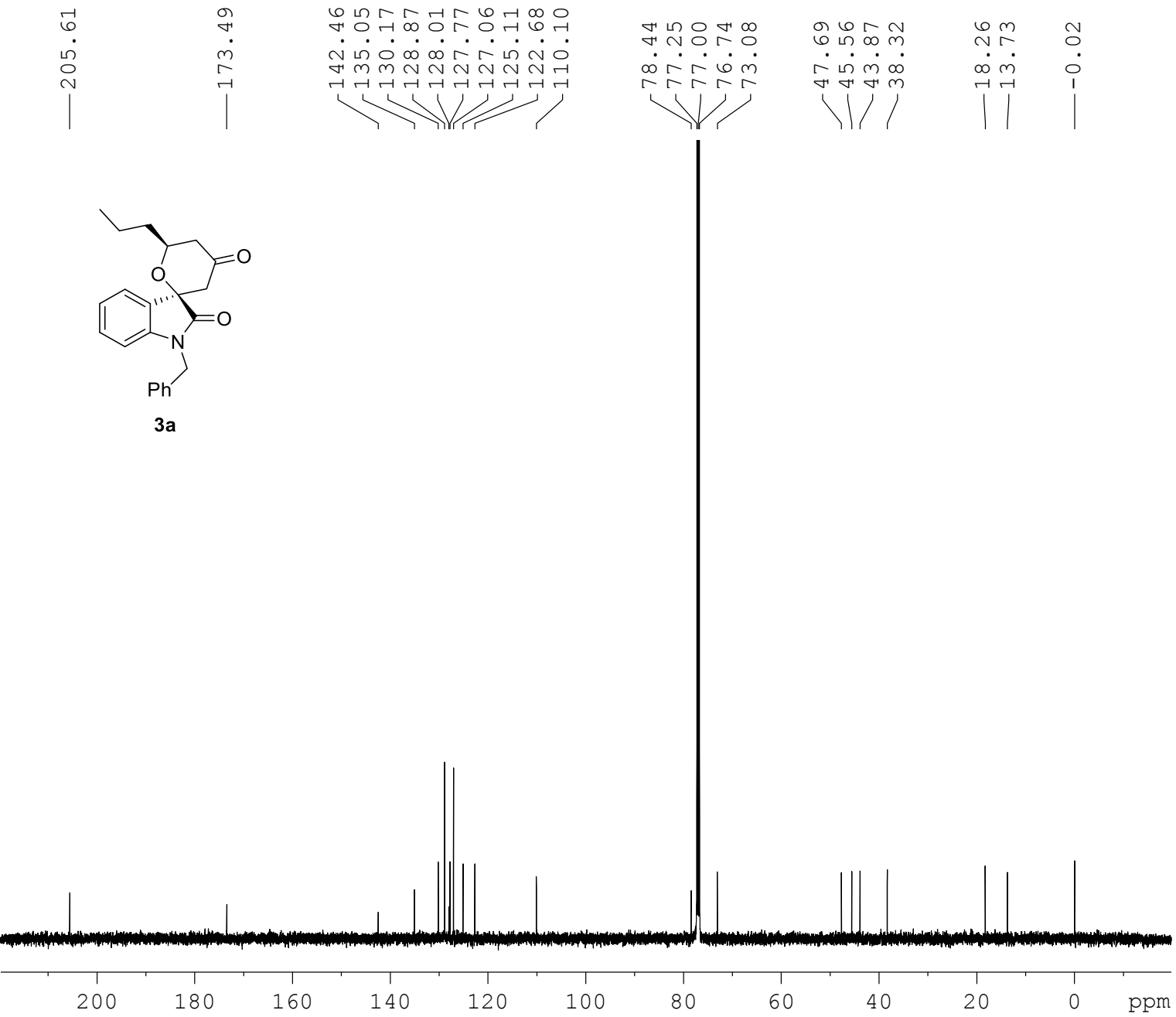

```
Current Data Parameters
NAME      MH-83-Minor-clean spectra
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20190126
Time               14.03 h
INSTRUM            Avance
PROBHD    Z151574_0027 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH            10000.000 Hz
FIDRES             0.305176 Hz
AQ              3.2767999 sec
RG                     32
DW                 50.000 usec
DE                 11.14 usec
TE                 298.0 K
D1              1.00000000 sec
TD0                     1
SFO1           500.1330883 MHz
NUC1                   1H
P0                 2.67 usec
P1                 8.00 usec
PLW1           23.68499947 W

F2 - Processing parameters
SI                  65536
SF             500.1300125 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



3a

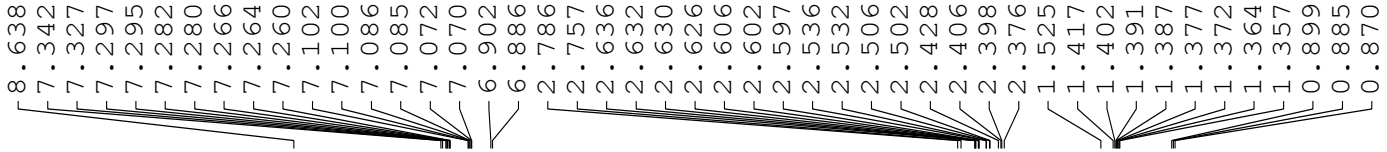


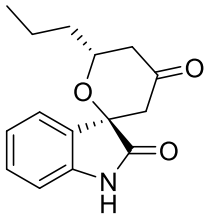
3a

```
Current Data Parameters
NAME      MH-83-Minor-Clean spectra-again-C13
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20190220
Time              12.44 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ            1.0878977 sec
RG                  101
DW               16.600 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1                13C
P0                3.33 usec
P1               10.00 usec
PLW1        88.26000214 W
SFO2        500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz65
PCPD2             80.00 usec
PLW2        23.68499947 W
PLW12        0.23014790 W
PLW13        0.11535020 W

F2 - Processing parameters
SI                32768
SF          125.7577919 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



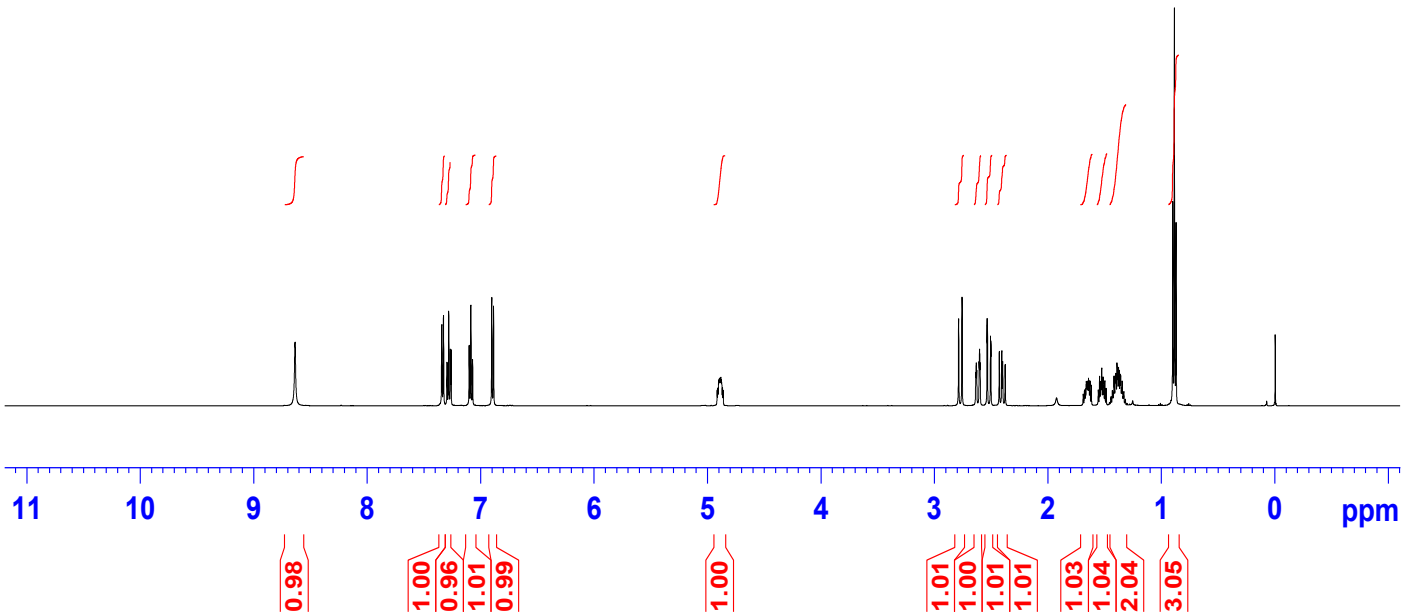


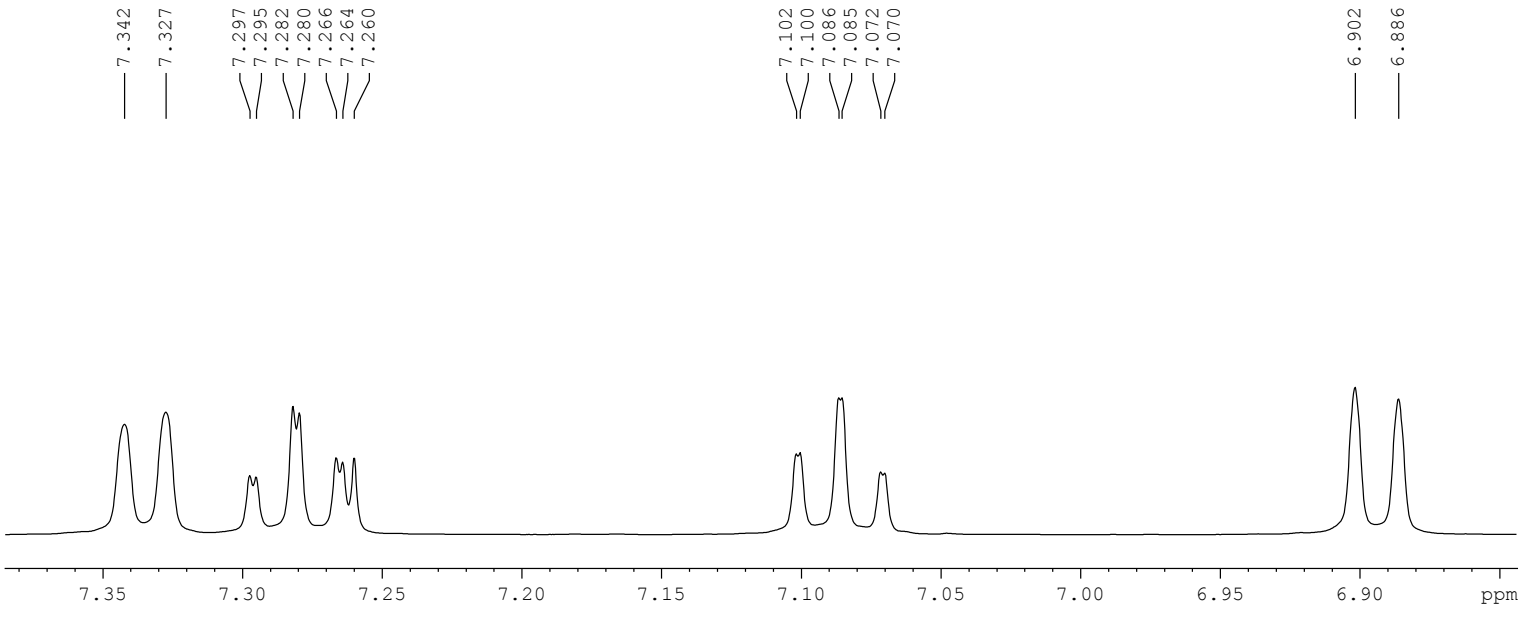
2b

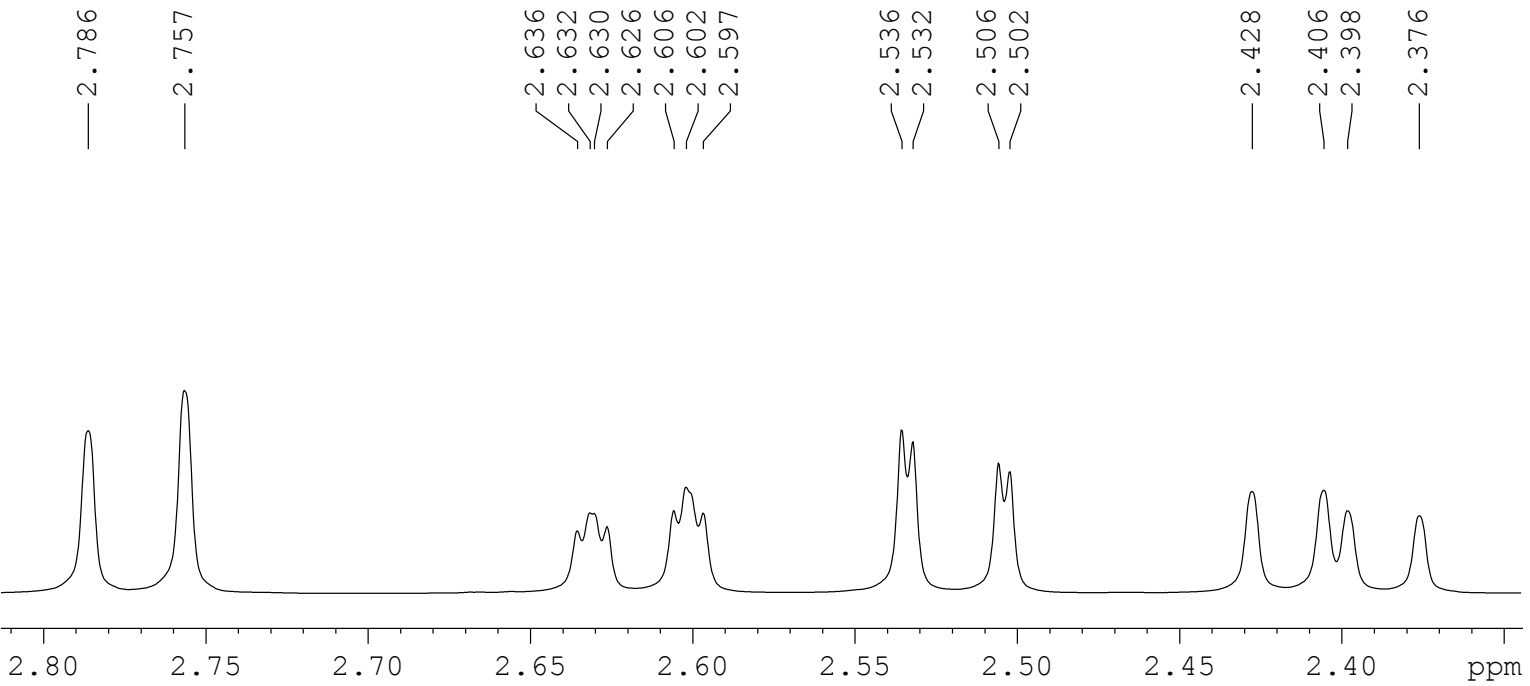
```
Current Data Parameters
NAME      MH-84-Major-Clean spectra
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20190201
Time              18.45 h
INSTRUM          Avance
PROBHD   Z151574_0027 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH          10000.000 Hz
FIDRES         0.305176 Hz
AQ           3.2767999 sec
RG                  32
DW               50.000 usec
DE               11.14 usec
TE               298.0 K
D1           1.00000000 sec
TD0                  1
SFO1         500.1330883 MHz
NUC1                1H
P0               2.67 usec
P1               8.00 usec
PLW1        23.68499947 W

F2 - Processing parameters
SI               65536
SF         500.1300120 MHz
WDW                 EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```



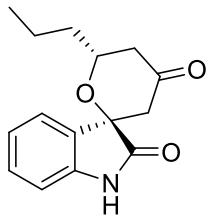




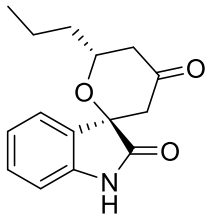

```
Current Data Parameters
NAME      MH-84-Major-Clean spectra
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190201
Time              18.45 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG            zg30
TD                65536
SOLVENT            CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                   32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                   1
SFO1         500.1330883 MHz
NUC1                 1H
P0                2.67 usec
P1                8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                65536
SF           500.1300120 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



2b

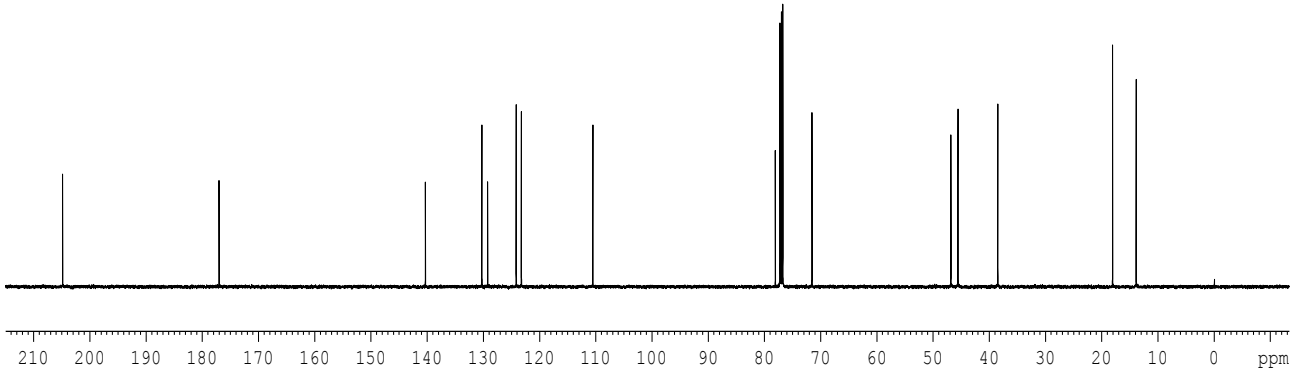


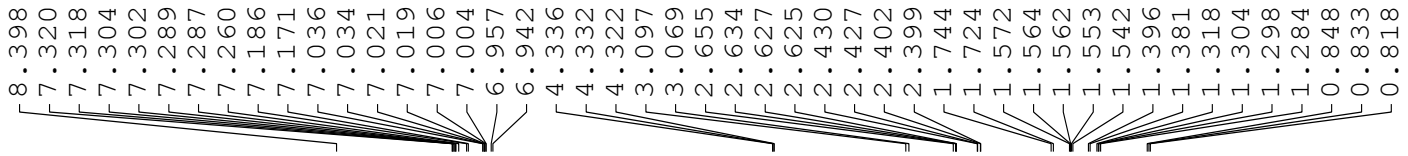
2b

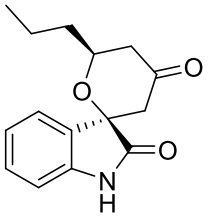
```
Current Data Parameters
NAME      MH-84-Major-Clean spectra-C13
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20190201
Time                19.13 h
INSTRUM            Avance
PROBHD   Z151574_0027 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    346
DS                      4
SWH             30120.482 Hz
FIDRES           0.919204 Hz
AQ              1.0878977 sec
RG                    101
DW                 16.600 usec
DE                  6.50 usec
TE                 298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1
SFO1          125.7703643 MHz
NUC1                  13C
P0                  3.33 usec
P1                 10.00 usec
PLW1          88.26000214 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz65
PCPD2              80.00 usec
PLW2          23.68499947 W
PLW12          0.23014790 W
PLW13          0.11535020 W

F2 - Processing parameters
SI                  32768
SF            125.7577972 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```





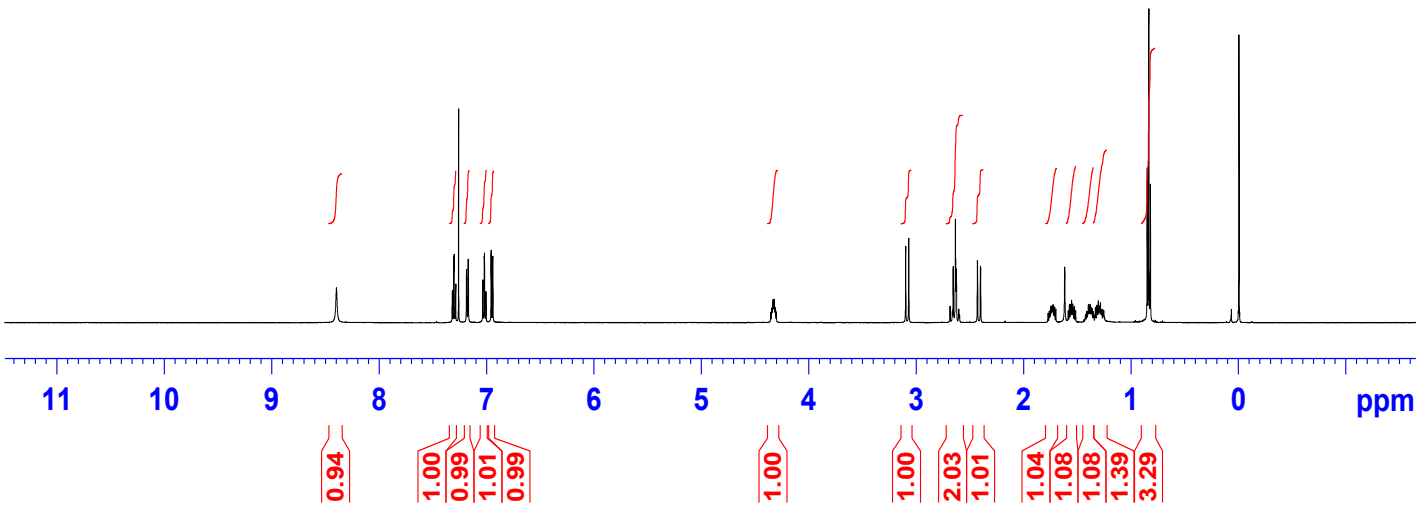


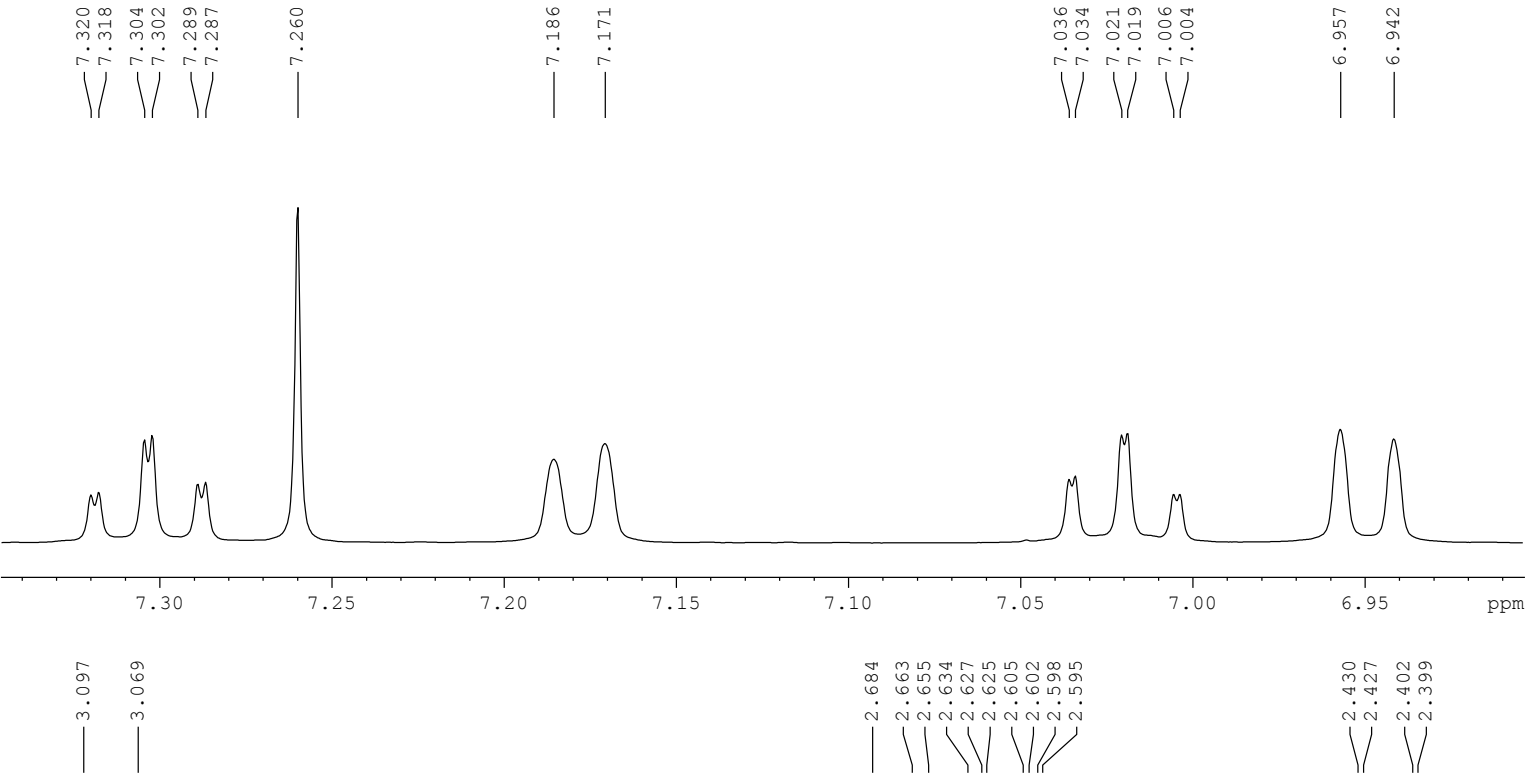
3b

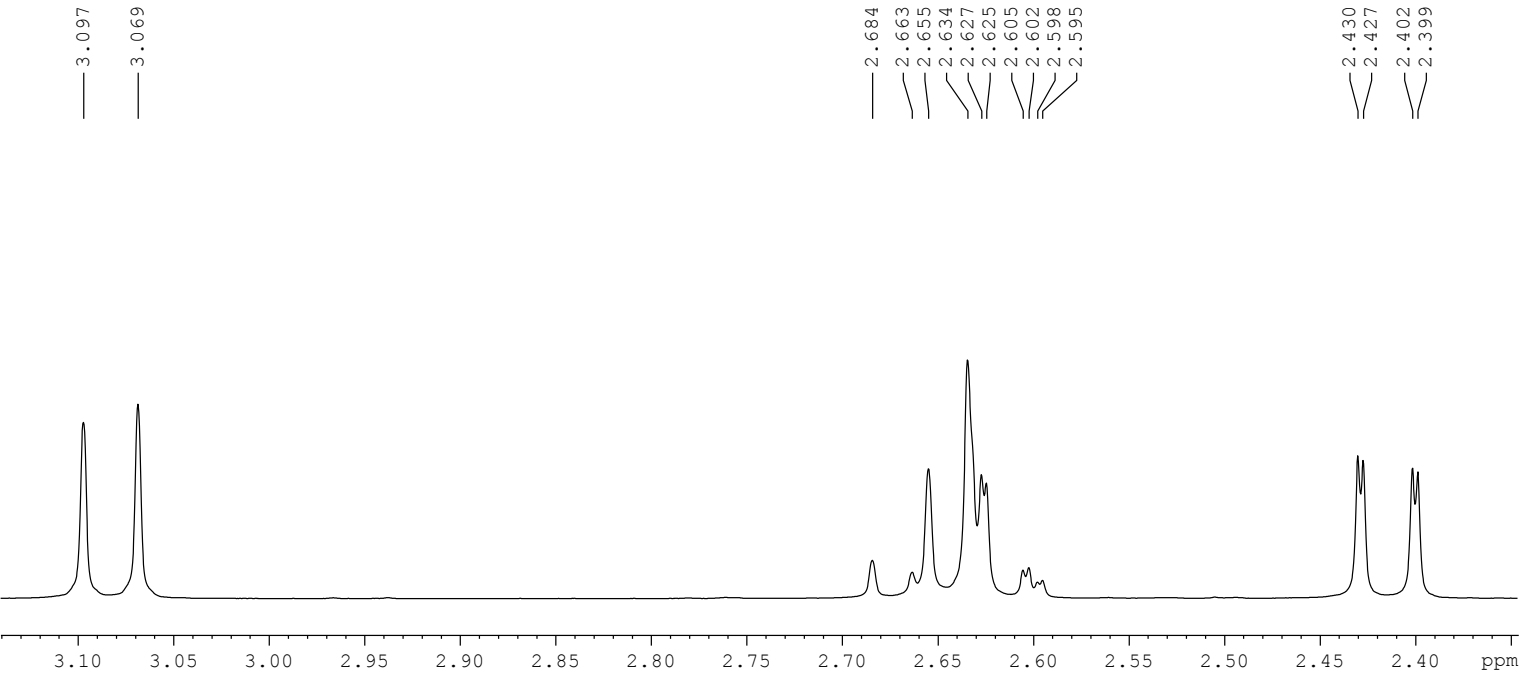
```
Current Data Parameters
NAME      MH-84-Minor-Clean spectra
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190201
Time              19.29 h
INSTRUM           Avance
PROBHD    Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300122 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



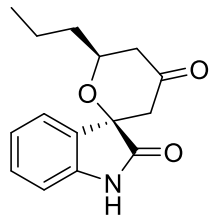




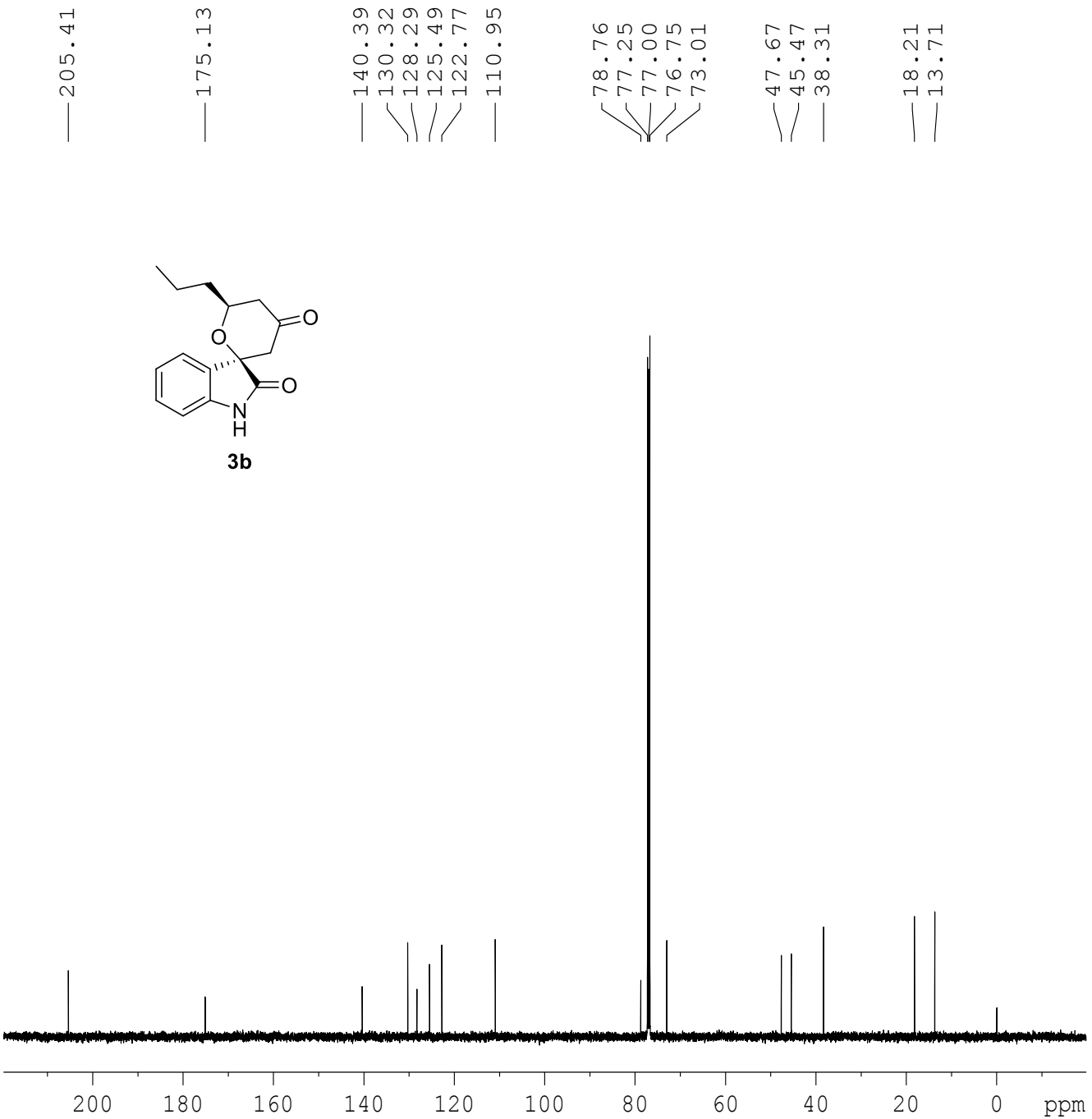

```
Current Data Parameters
NAME      MH-84-Minor-Clean spectra
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20190201
Time              19.29 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300122 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



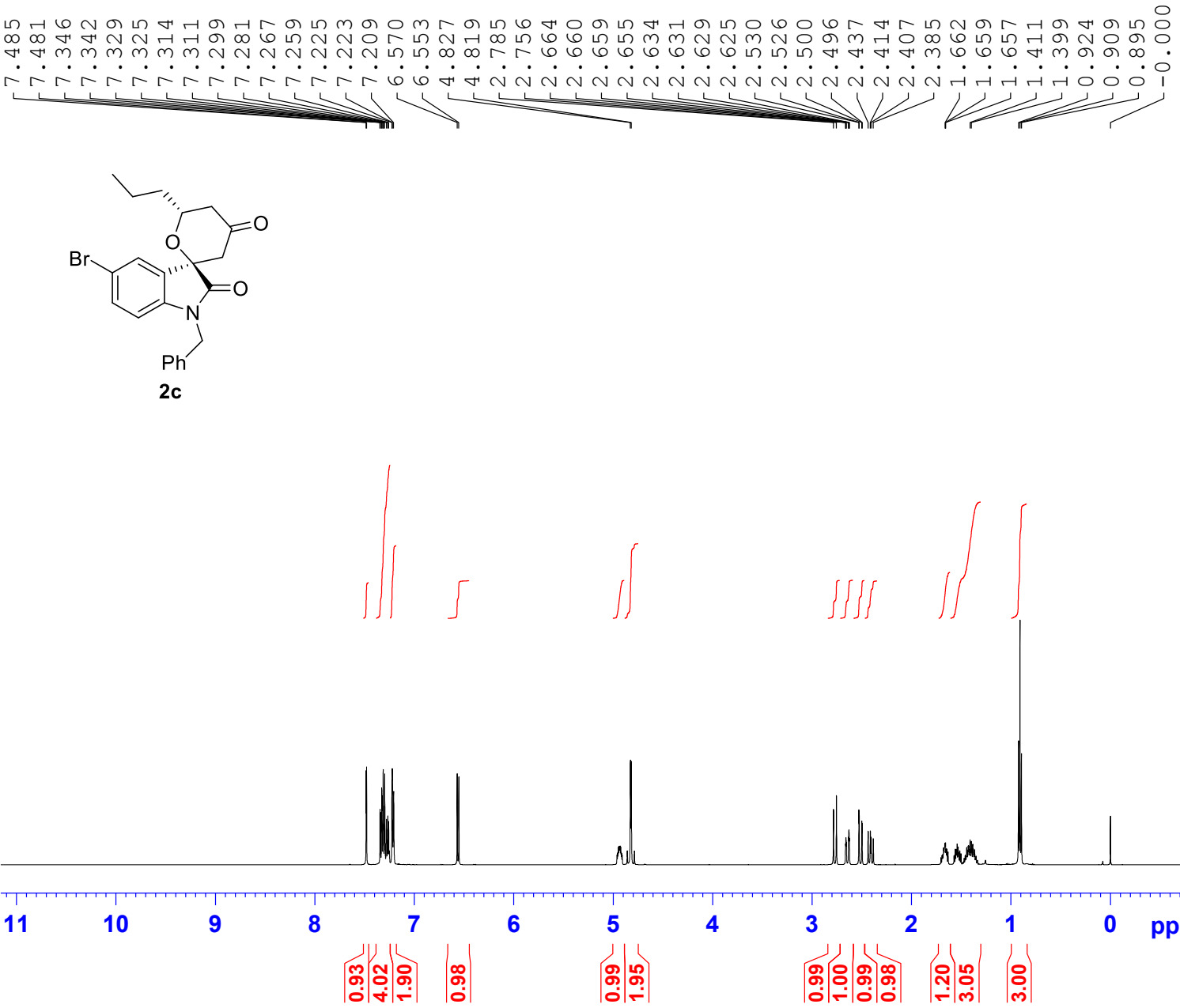
3b




```
Current Data Parameters
NAME      MH-84-Minor-Clean spectra-C13
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20190201
Time                19.51 h
INSTRUM            Avance
PROBHD    Z151574_0027 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    238
DS                      4
SWH             30120.482 Hz
FIDRES          0.919204 Hz
AQ              1.0878977 sec
RG                    101
DW                 16.600 usec
DE                  6.50 usec
TE                 298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1
SFO1         125.7703643 MHz
NUC1                 13C
P0                  3.33 usec
P1                 10.00 usec
PLW1         88.26000214 W
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2          waltz65
PCPD2               80.00 usec
PLW2         23.68499947 W
PLW12         0.23014790 W
PLW13         0.11535020 W

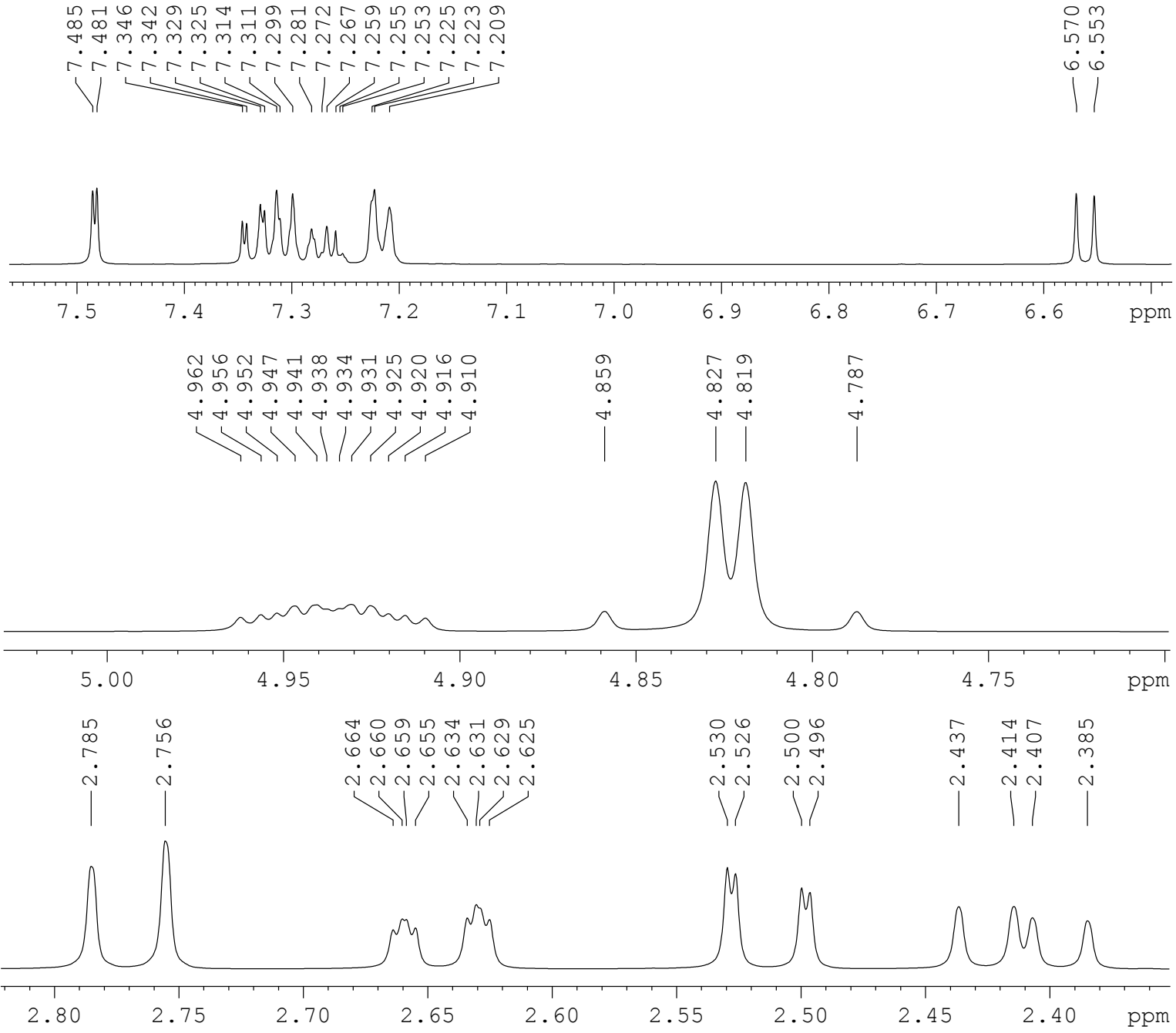
F2 - Processing parameters
SI                  32768
SF           125.7577920 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```




```
Current Data Parameters
NAME      MH-86-Major-Clean spectra
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20190131
Time              17.08 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          23.68499947 W

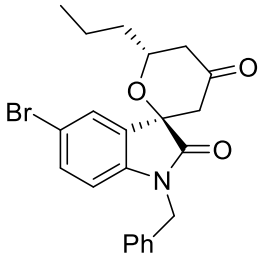
F2 - Processing parameters
SI                 65536
SF            500.1300125 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



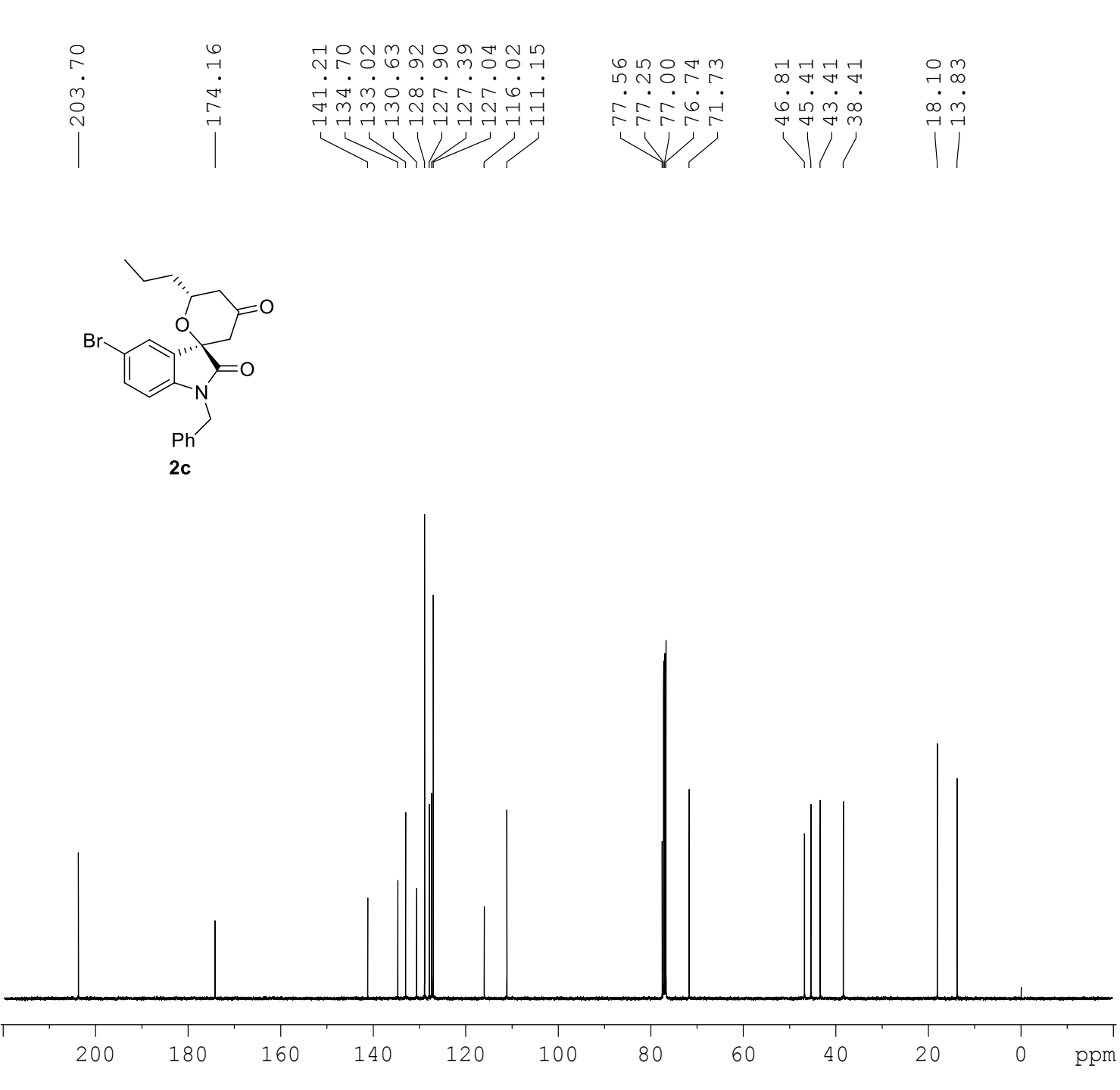

```
Current Data Parameters
NAME      MH-86-Major-Clean spectra
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190131
Time              17.08 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES        0.305176 Hz
AQ            3.2767999 sec
RG                    32
DW               50.000 usec
DE               11.14 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                2.67 usec
P1                8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300125 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



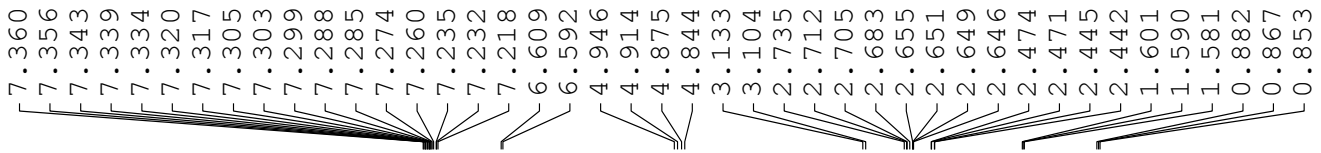
2c

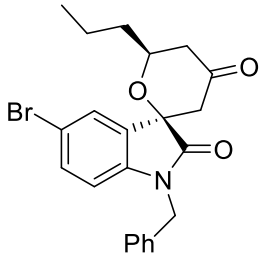



```
Current Data Parameters
NAME      MH-86-Major-Clean spectra-C13
EXPNO                 1
PROCNO                1

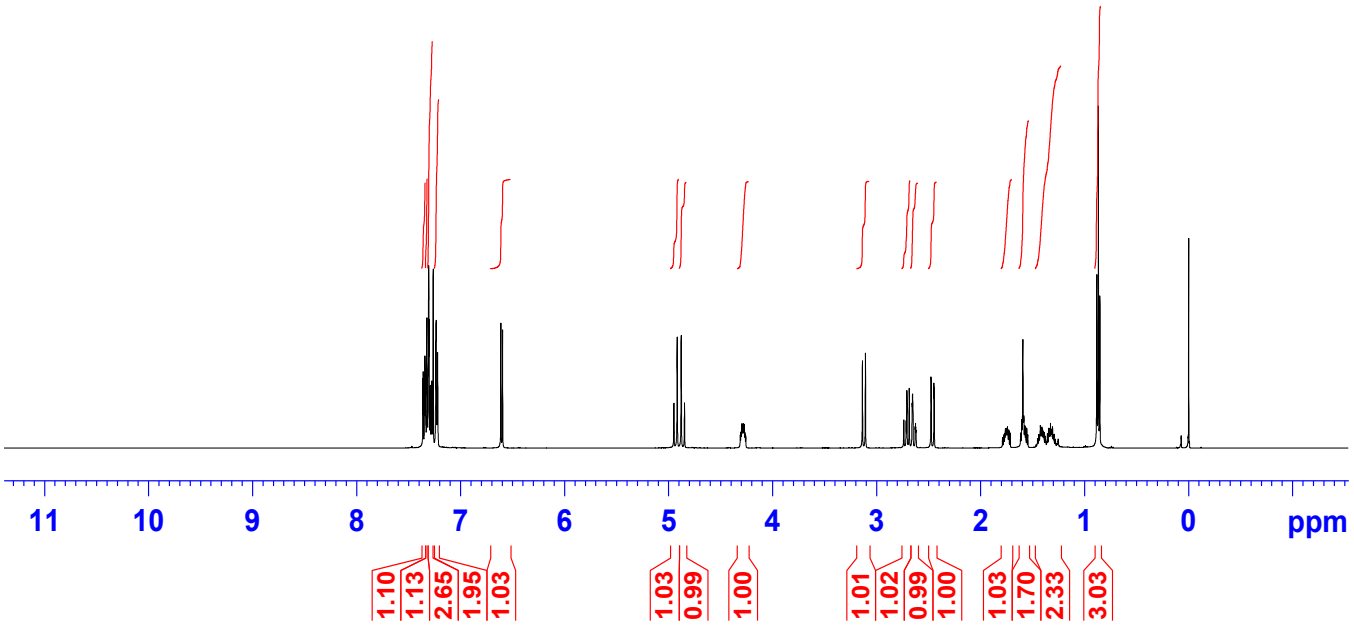
F2 - Acquisition Parameters
Date_          20190131
Time               17.46 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  611
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ             1.0878977 sec
RG                  101
DW                16.600 usec
DE                 6.50 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         125.7703643 MHz
NUC1                13C
P0                 3.33 usec
P1                10.00 usec
PLW1         88.26000214 W
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             80.00 usec
PLW2         23.68499947 W
PLW12         0.23014790 W
PLW13         0.11535020 W

F2 - Processing parameters
SI                 32768
SF           125.7577977 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```





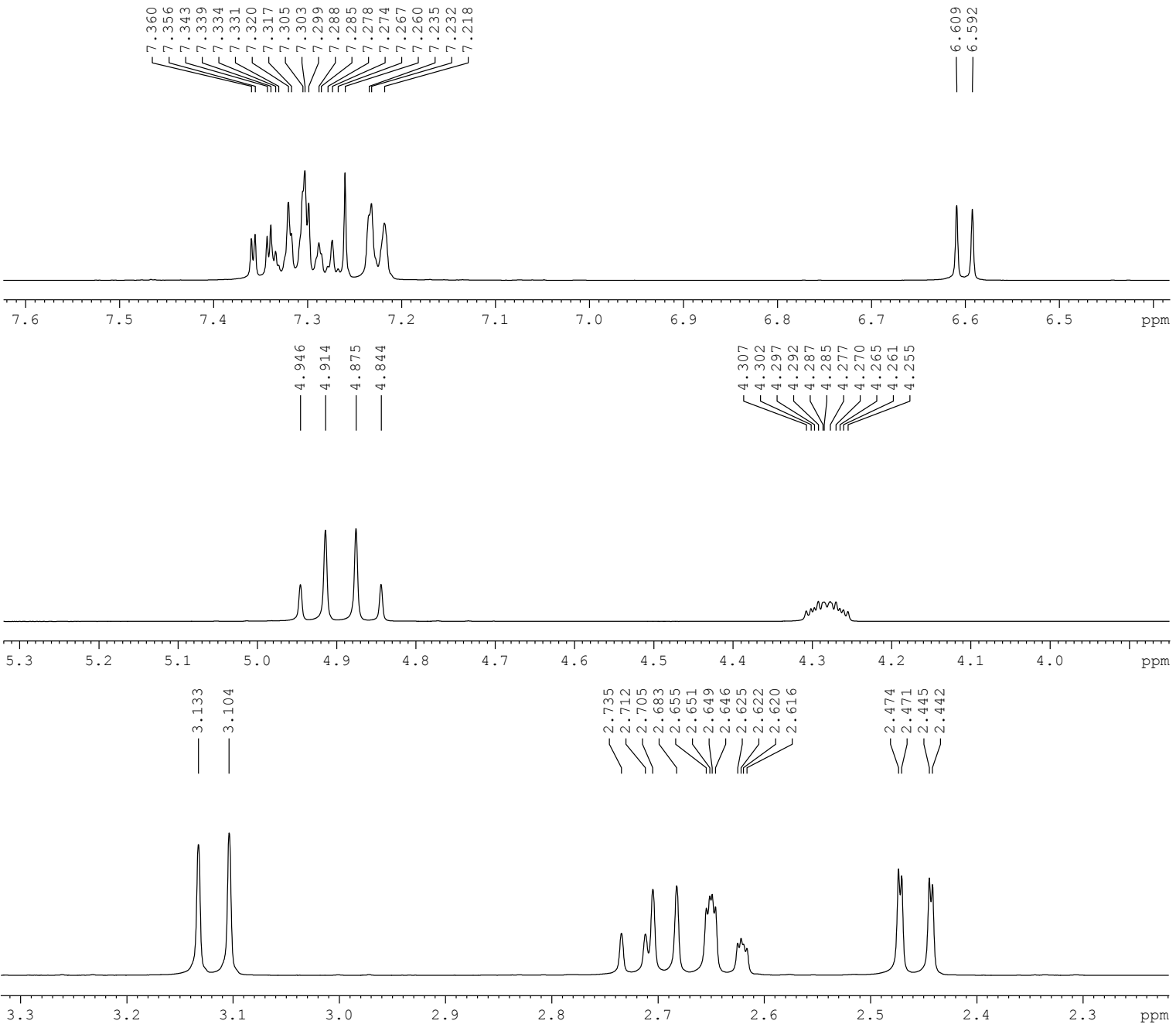
3c




```
Current Data Parameters
NAME      MH-86-MINOR-CLEAN SPECTRA
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20190131
Time              17.58 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ            3.2767999 sec
RG                    32
DW               50.000 usec
DE               11.14 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

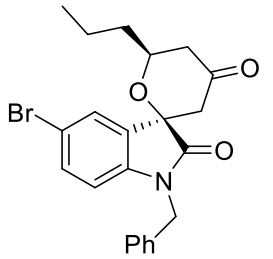
F2 - Processing parameters
SI                 65536
SF           500.1300120 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME      MH-86-MINOR-CLEAN SPECTRA
EXPNO                    1
PROCNO                   1

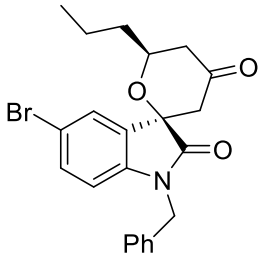
F2 - Acquisition Parameters
Date_           20190131
Time              17.58 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES        0.305176 Hz
AQ            3.2767999 sec
RG                    32
DW               50.000 usec
DE               11.14 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0               2.67 usec
P1               8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300120 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



3c

204.91
173.07
141.45
134.53
133.03
130.05
128.96
128.01
127.95
115.48
111.56
78.30
77.25
77.00
76.75
73.42
47.44
45.13
43.94
38.27
18.22
13.79



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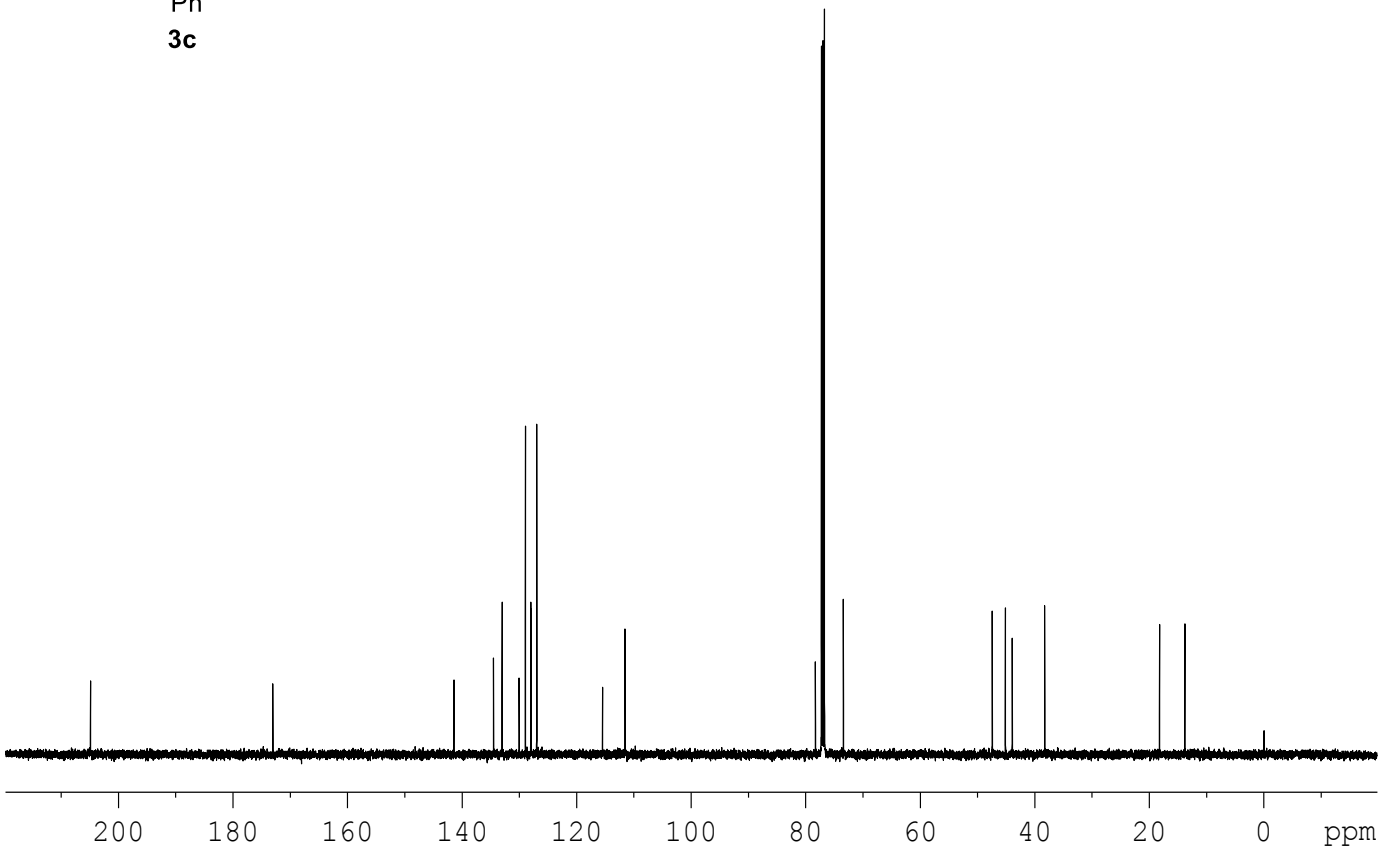
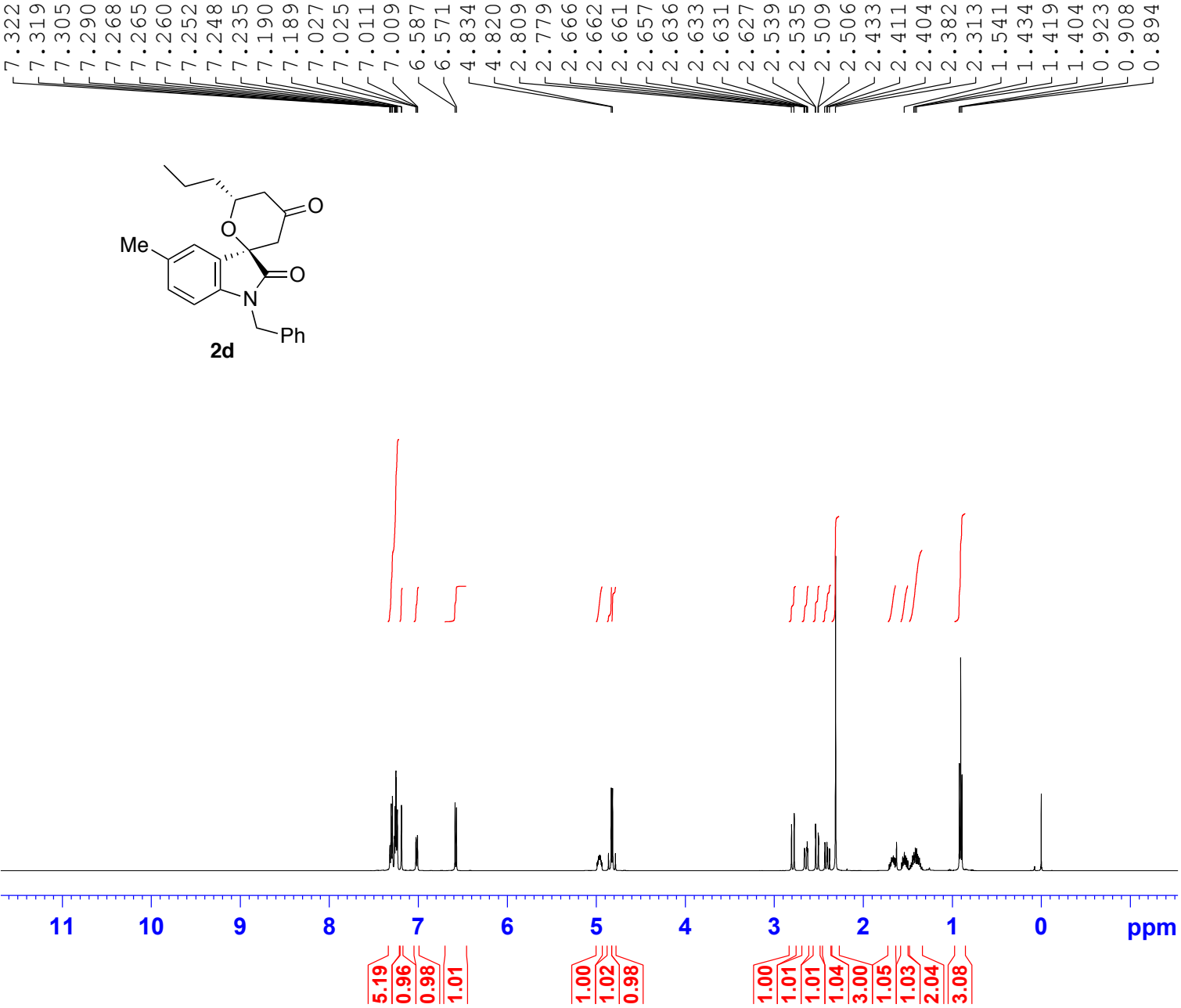



```
Current Data Parameters
NAME      MH-86-MINOR-CLEAN SPECTRA-C13
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20190131
Time              18.27 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  261
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ            1.0878977 sec
RG                  101
DW               16.600 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1                13C
P0                3.33 usec
P1               10.00 usec
PLW1        88.26000214 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2            80.00 usec
PLW2        23.68499947 W
PLW12        0.23014790 W
PLW13        0.11535020 W

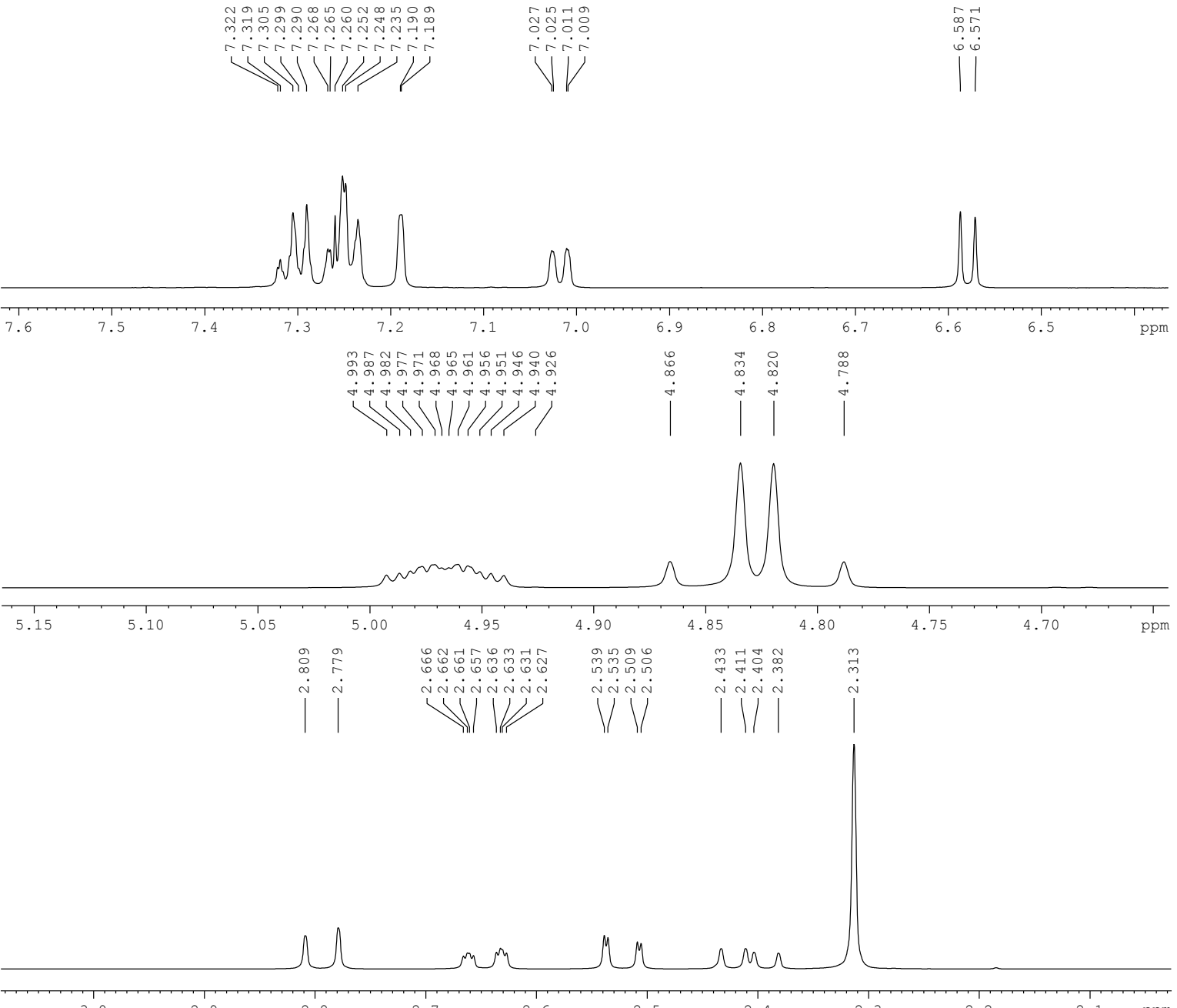
F2 - Processing parameters
SI                32768
SF          125.7577930 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```




```
Current Data Parameters
NAME      MH-87-MAJOR-CLEAN SPECTRA
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190131
Time              11.57 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

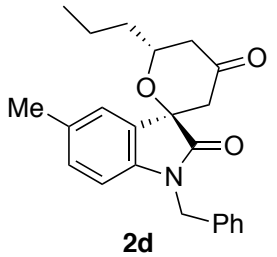
F2 - Processing parameters
SI                 65536
SF           500.1300120 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



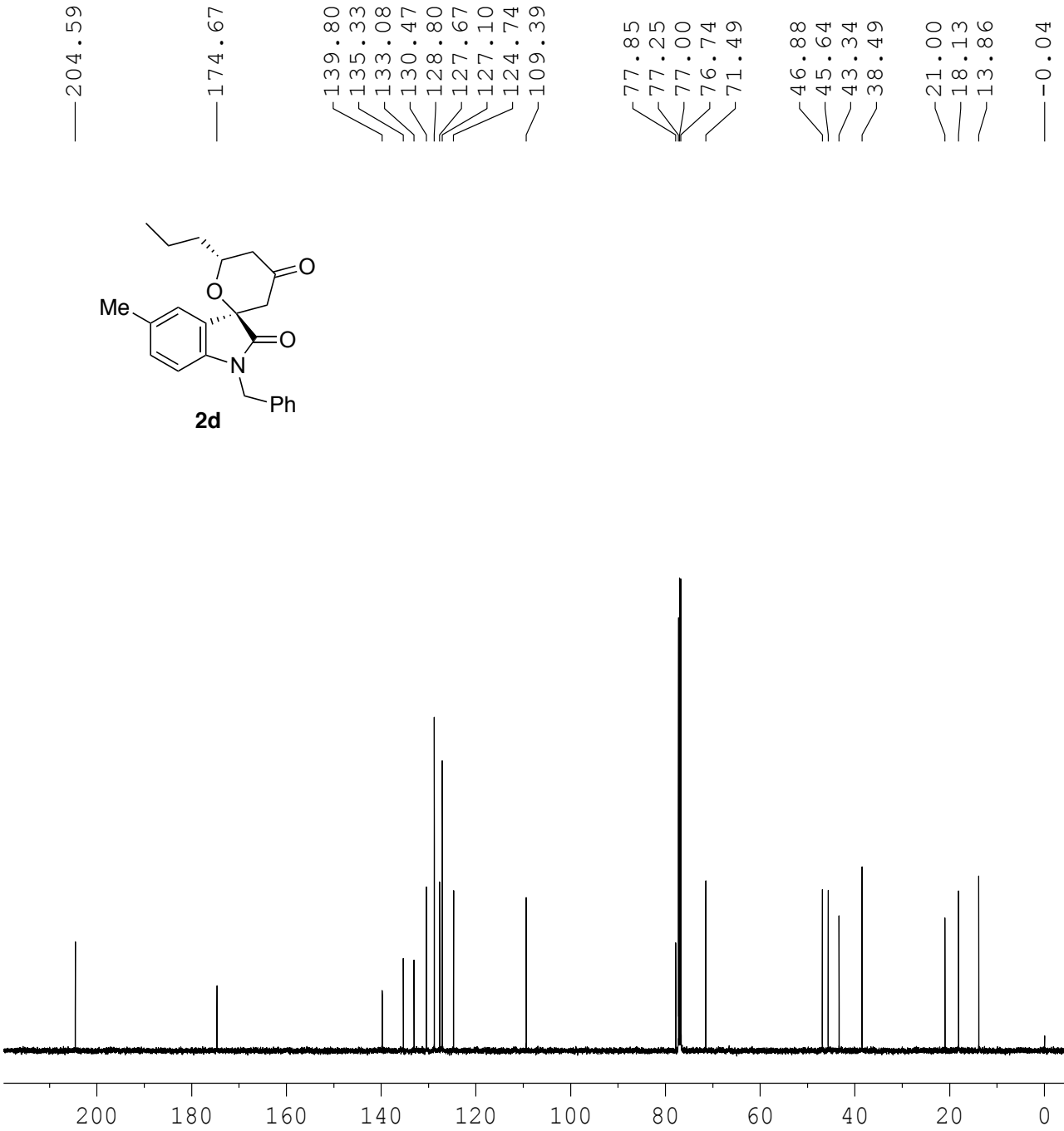

```
Current Data Parameters
NAME      MH-87-MAJOR-CLEAN SPECTRA
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190131
Time              11.57 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT             CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300120 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



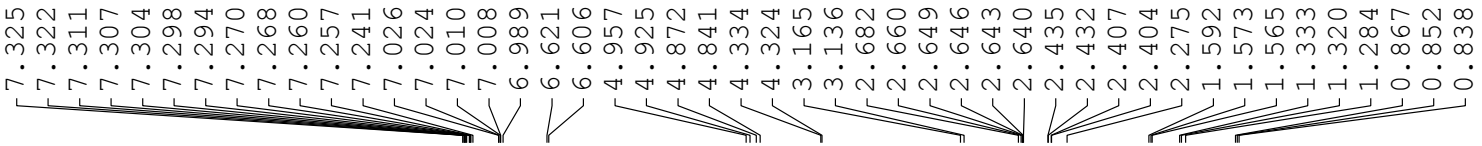
2d

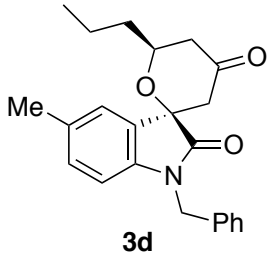



```
Current Data Parameters
NAME      MH-87-MAJOR-CLEAN SPECTRA-C13
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20190131
Time              12.27 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  241
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ            1.0878977 sec
RG                  101
DW               16.600 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1                13C
P0                3.33 usec
P1               10.00 usec
PLW1         88.26000214 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2            80.00 usec
PLW2         23.68499947 W
PLW12         0.23014790 W
PLW13         0.11535020 W

F2 - Processing parameters
SI                32768
SF          125.7577953 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



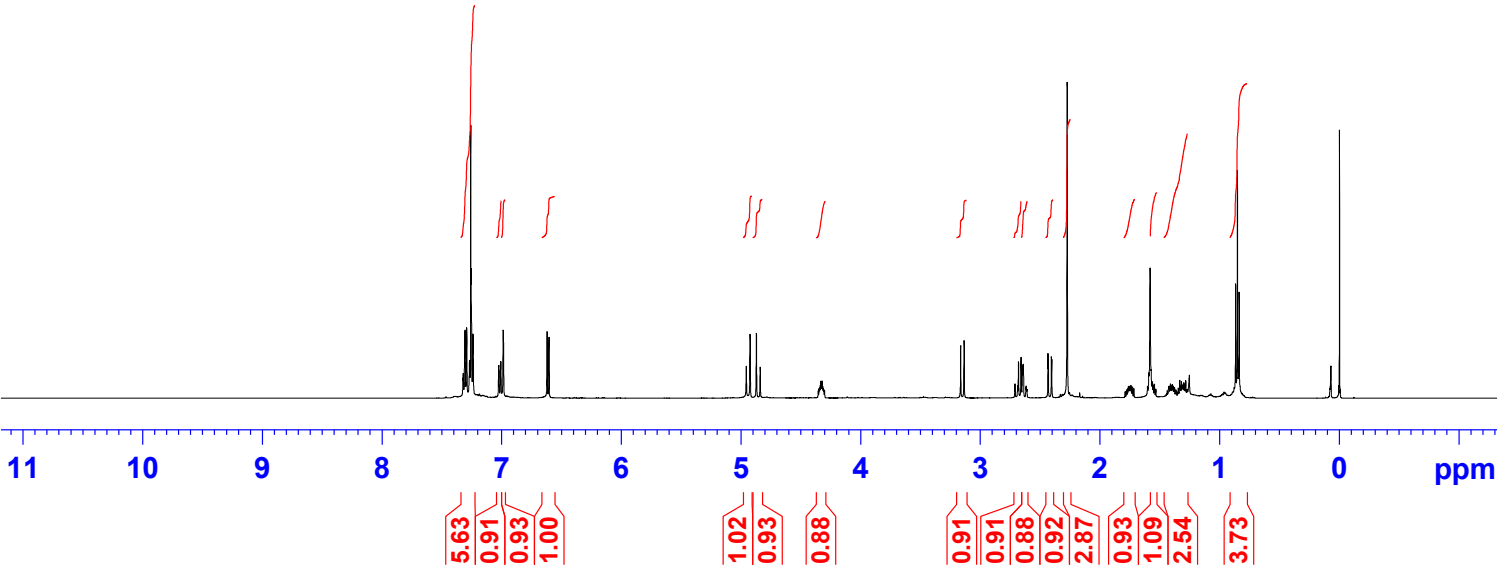


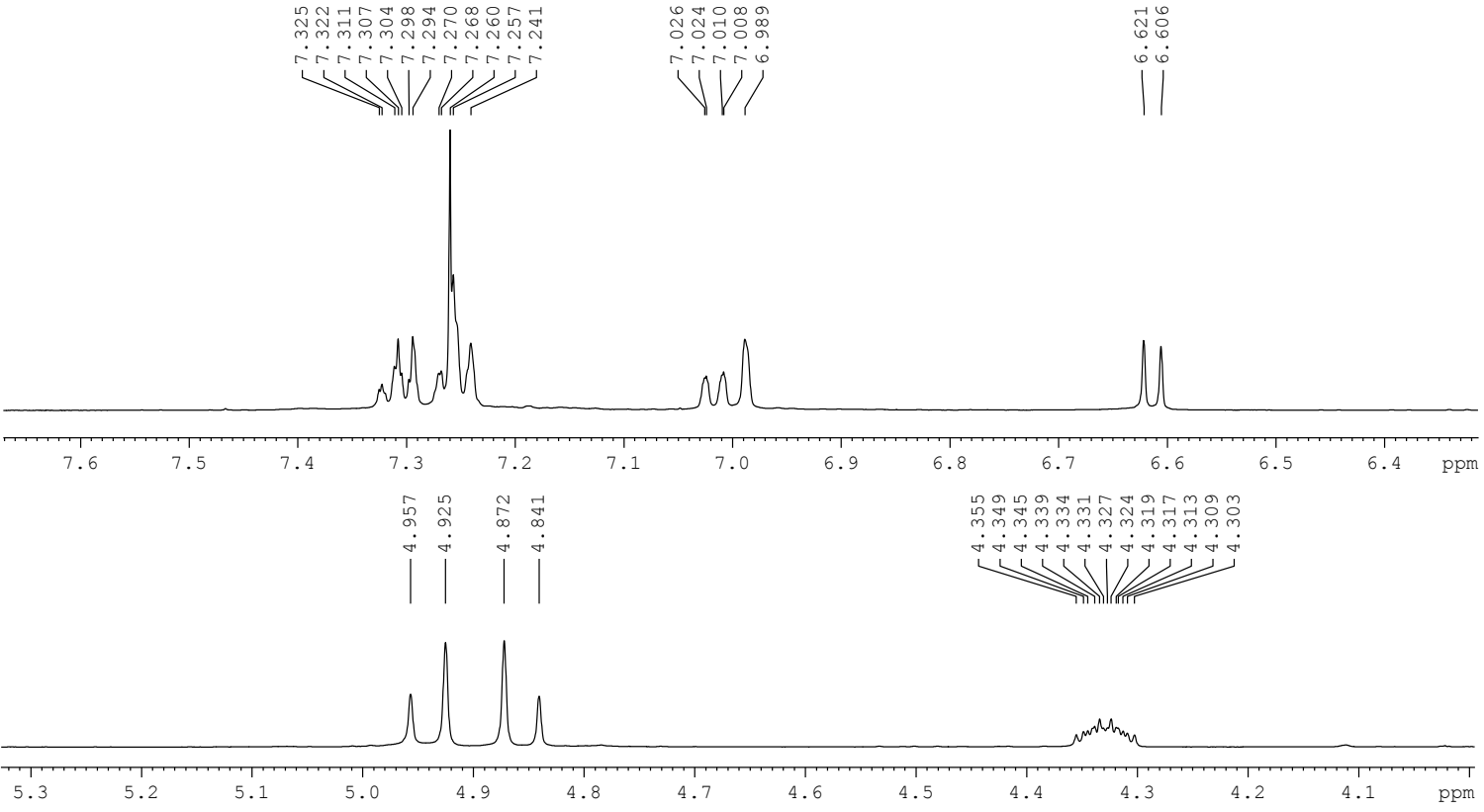
3d

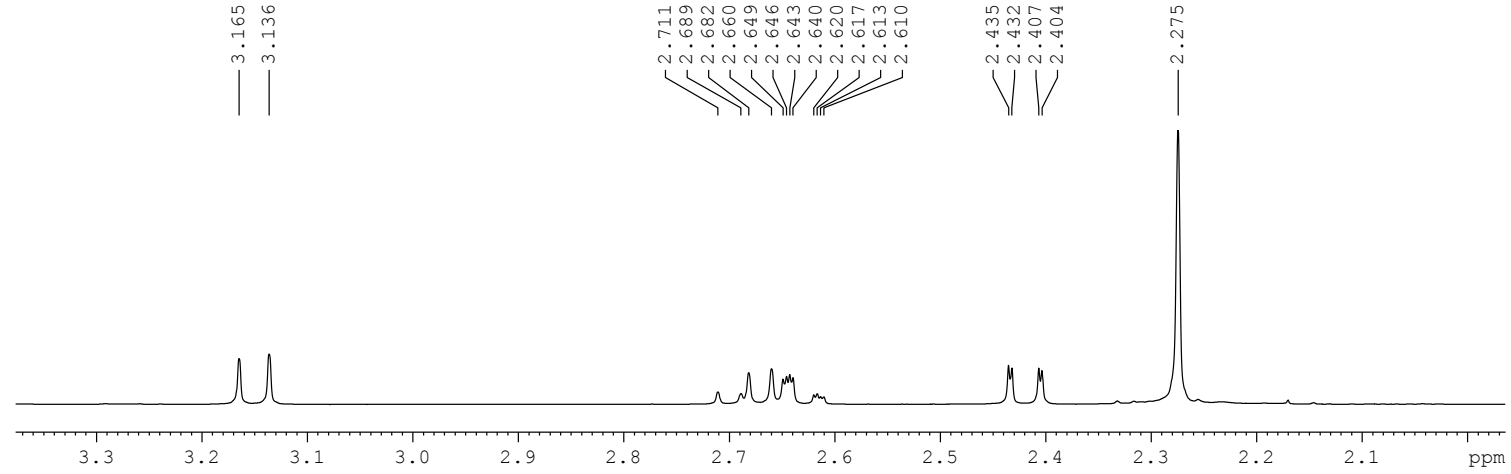
```
Current Data Parameters
NAME      MH-87-Minor- Clean spectra
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20190131
Time              12.33 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH          10000.000 Hz
FIDRES           0.305176 Hz
AQ            3.2767999 sec
RG                  32
DW               50.000 usec
DE               11.14 usec
TE               298.0 K
D1           1.00000000 sec
TD0                  1
SFO1        500.1330883 MHz
NUC1                1H
P0               2.67 usec
P1               8.00 usec
PLW1        23.68499947 W

F2 - Processing parameters
SI               65536
SF          500.1300120 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```



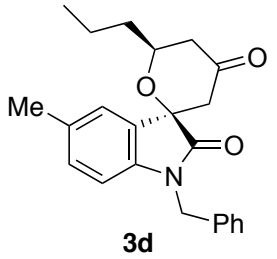




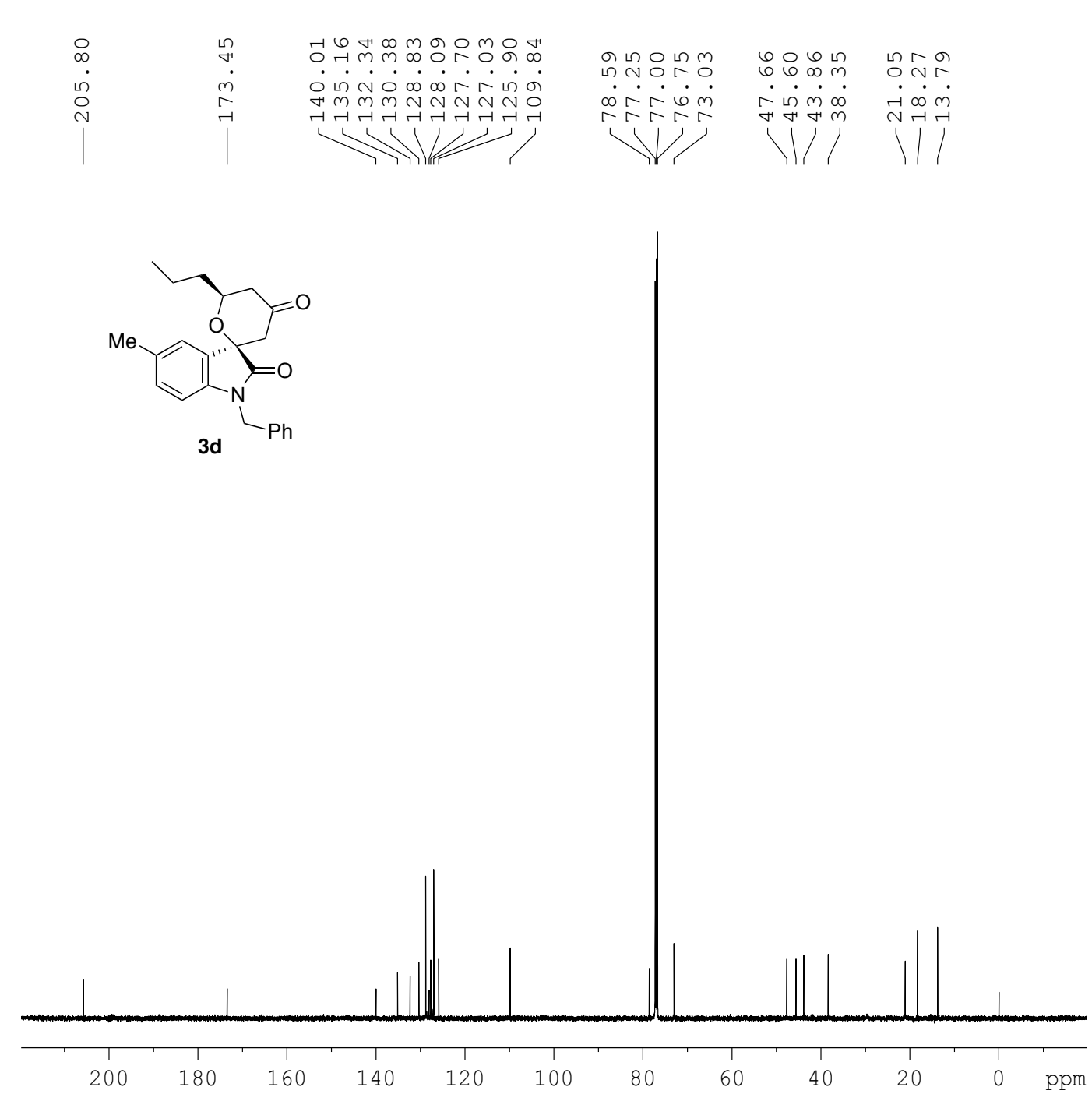

```
Current Data Parameters
NAME      MH-87-Minor- Clean spectra
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20190131
Time              12.33 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                   32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                   1
SFO1          500.1330883 MHz
NUC1                 1H
P0                2.67 usec
P1                8.00 usec
PLW1          23.68499947 W

F2 - Processing parameters
SI                65536
SF          500.1300120 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



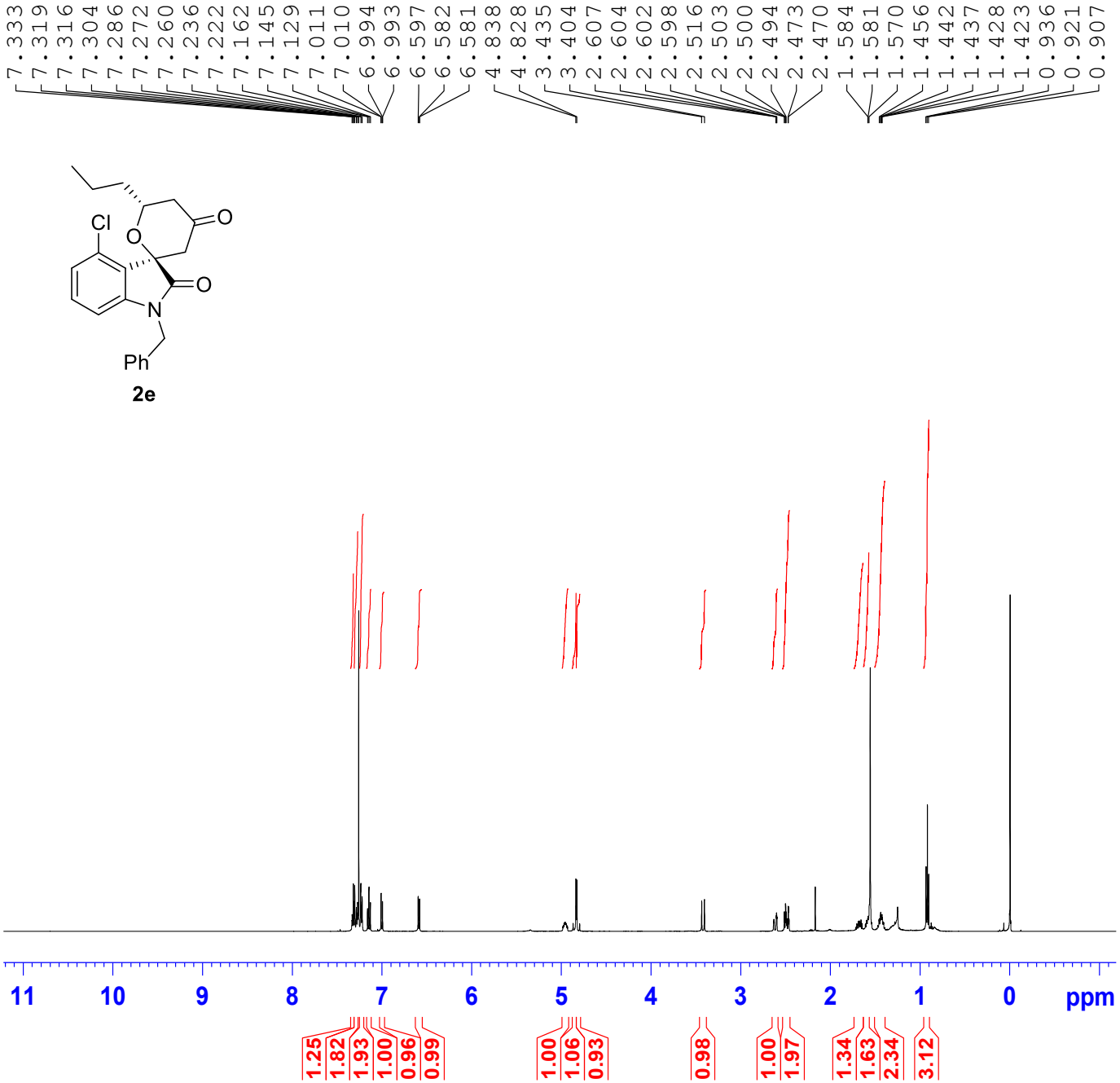
3d




```
Current Data Parameters
NAME      MH-87-Minor- Clean spectra-c13
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190131
Time              13.33 h
INSTRUM           Avance
PROBHD    Z151574_0027 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ            1.0878977 sec
RG                  101
DW               16.600 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1               13C
P0                3.33 usec
P1               10.00 usec
PLW1        88.26000214 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2         waltz65
PCPD2             80.00 usec
PLW2        23.68499947 W
PLW12        0.23014790 W
PLW13        0.11535020 W

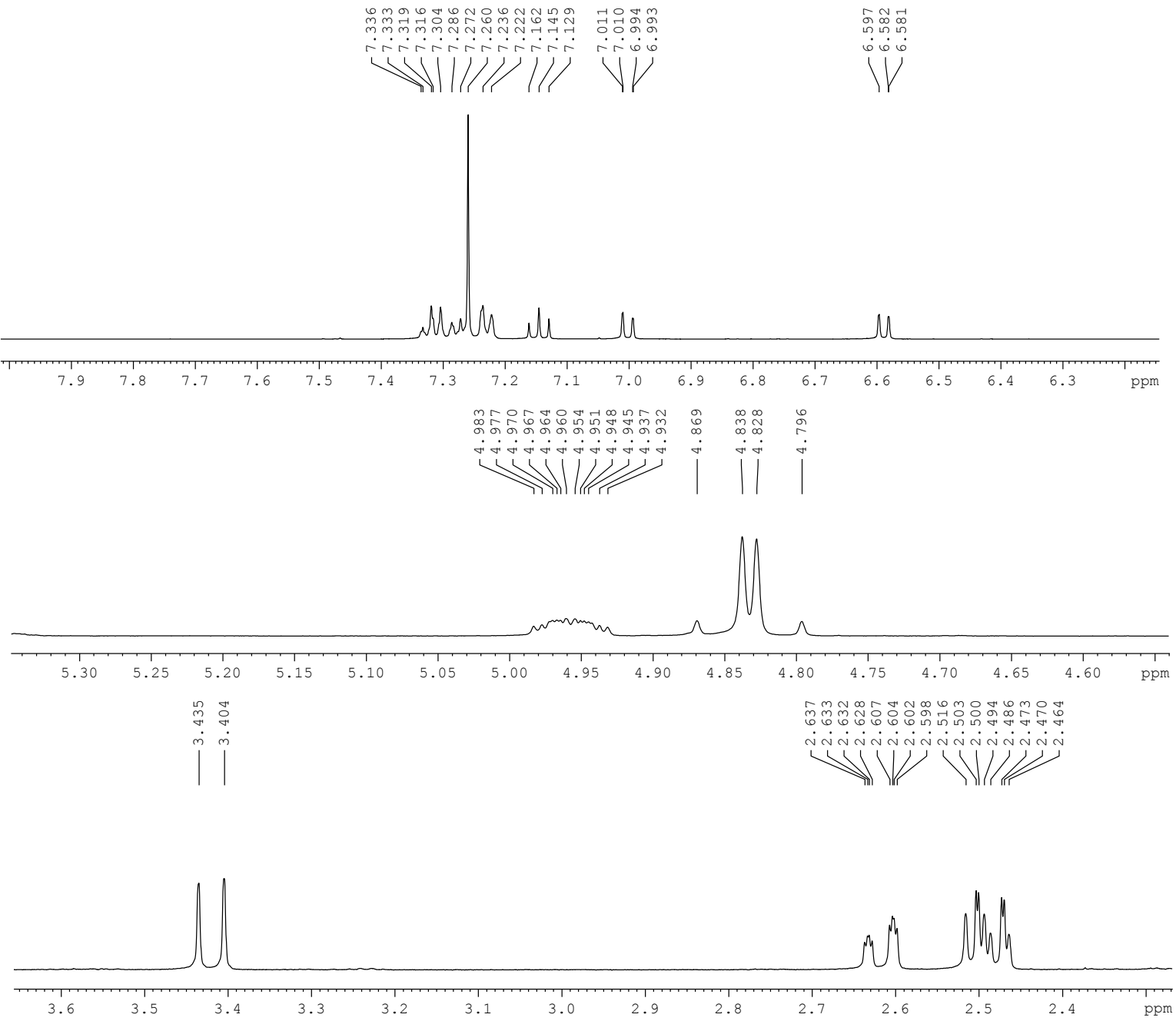
F2 - Processing parameters
SI                32768
SF          125.7577921 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```




```
Current Data Parameters
NAME      MH-89-Major-Clean Spectra
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20190204
Time              17.53 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES        0.305176 Hz
AQ            3.2767999 sec
RG                   32
DW               50.000 usec
DE               11.14 usec
TE               298.0 K
D1            1.00000000 sec
TD0                   1
SFO1         500.1330883 MHz
NUC1                 1H
P0                2.67 usec
P1                8.00 usec
PLW1         23.68499947 W

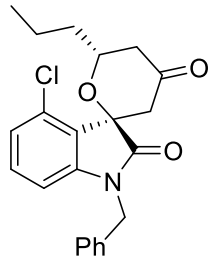
F2 - Processing parameters
SI                65536
SF           500.1300122 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```




```
Current Data Parameters
NAME      MH-89-Major-Clean Spectra
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20190204
Time                17.53 h
INSTRUM            Avance
PROBHD    Z151574_0027 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                     32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                     1
SFO1          500.1330883 MHz
NUC1                   1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          23.68499947 W

F2 - Processing parameters
SI                  65536
SF            500.1300122 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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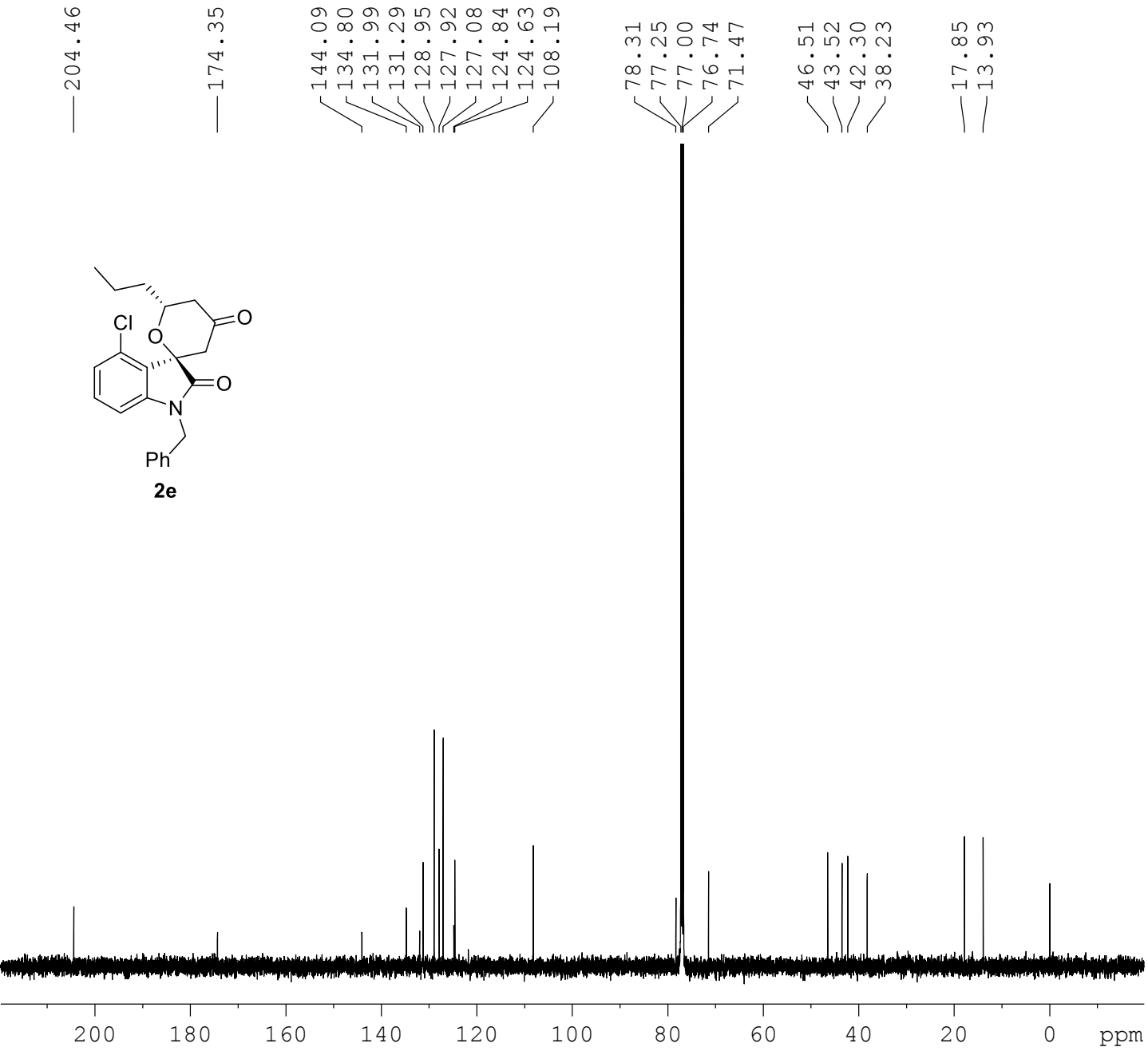
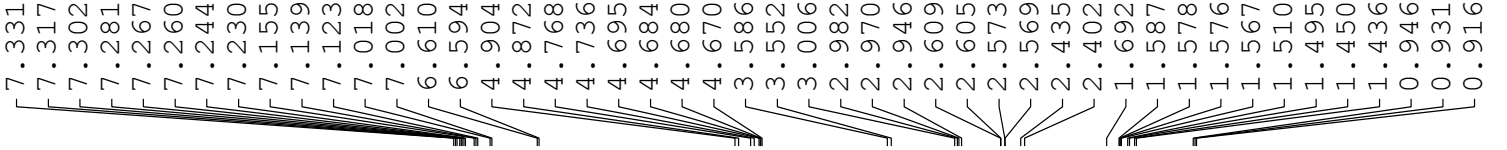
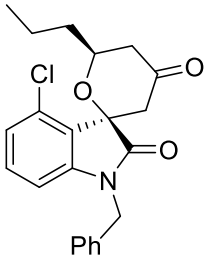



```
Current Data Parameters
NAME      MH-89-Major-Clean Spectra-C13
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20190204
Time              18.59 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ            1.0878977 sec
RG                  101
DW               16.600 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1         125.7703643 MHz
NUC1                13C
P0                 3.33 usec
P1                10.00 usec
PLW1         88.26000214 W
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             80.00 usec
PLW2         23.68499947 W
PLW12         0.23014790 W
PLW13         0.11535020 W

F2 - Processing parameters
SI                 32768
SF           125.7577915 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```





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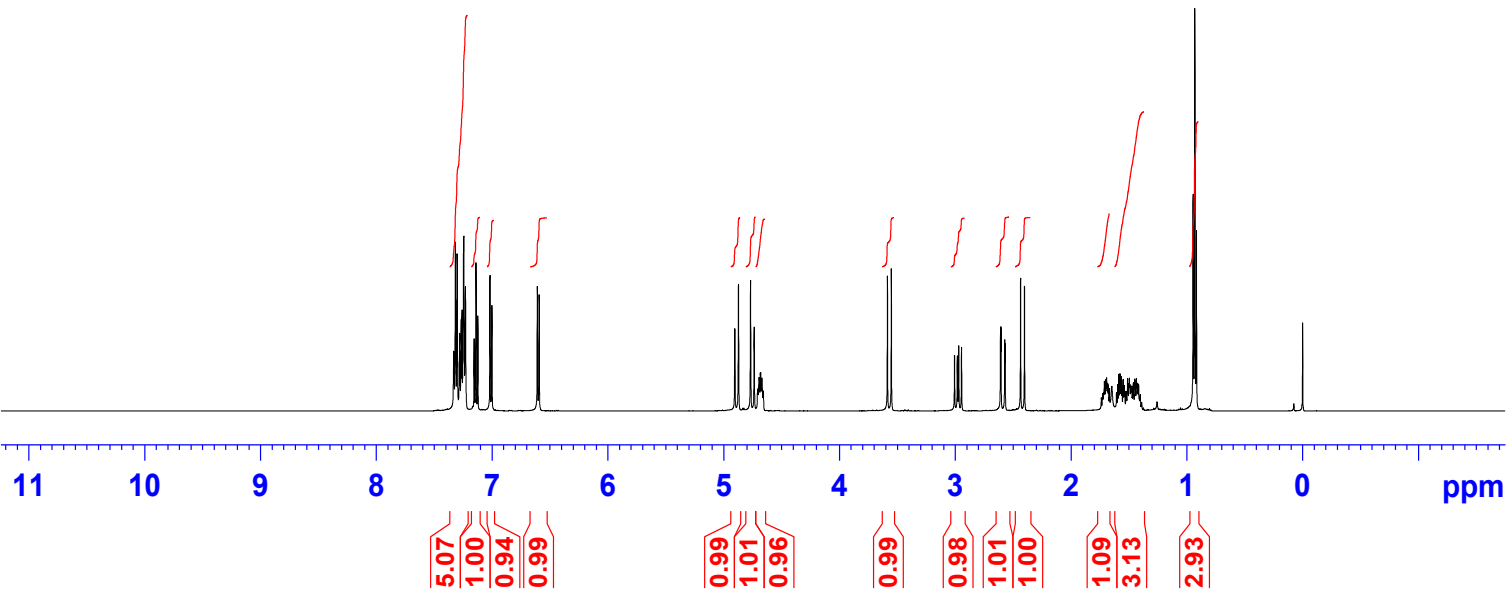
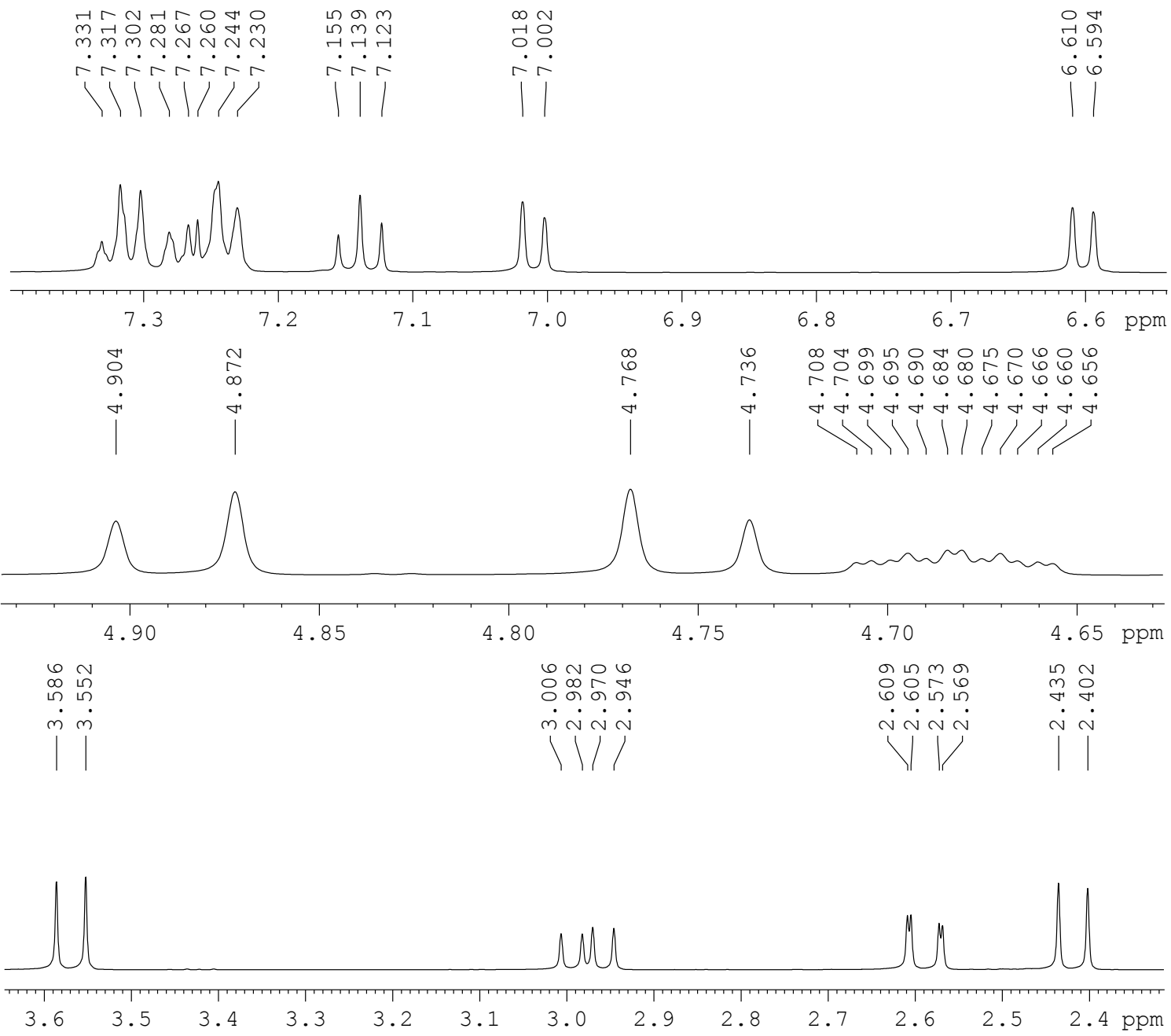
```
Current Data Parameters
NAME      MH-89-Minor-Clean spectra
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20190205
Time              13.35 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300123 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

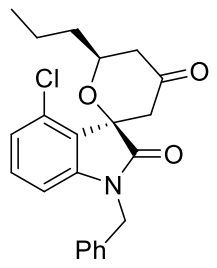





```
Current Data Parameters
NAME      MH-89-Minor-Clean spectra
EXPNO                  1
PROCNO                 1

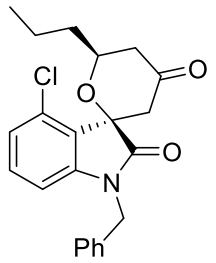
F2 - Acquisition Parameters
Date_           20190205
Time              13.35 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ              3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          23.68499947 W

F2 - Processing parameters
SI                 65536
SF            500.1300123 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



3e

205.70
175.63
144.08
134.80
131.17
128.92
127.89
127.14
124.21
108.32
78.10
77.25
77.00
76.74
72.54
45.89
43.95
40.90
37.68
18.19
13.91

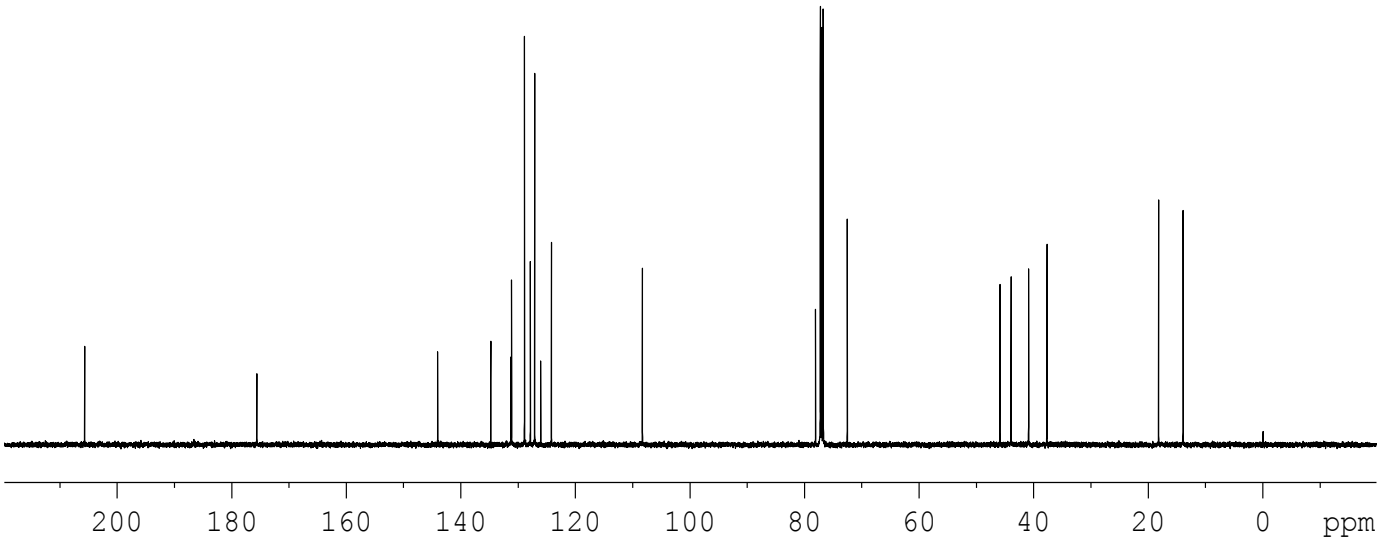


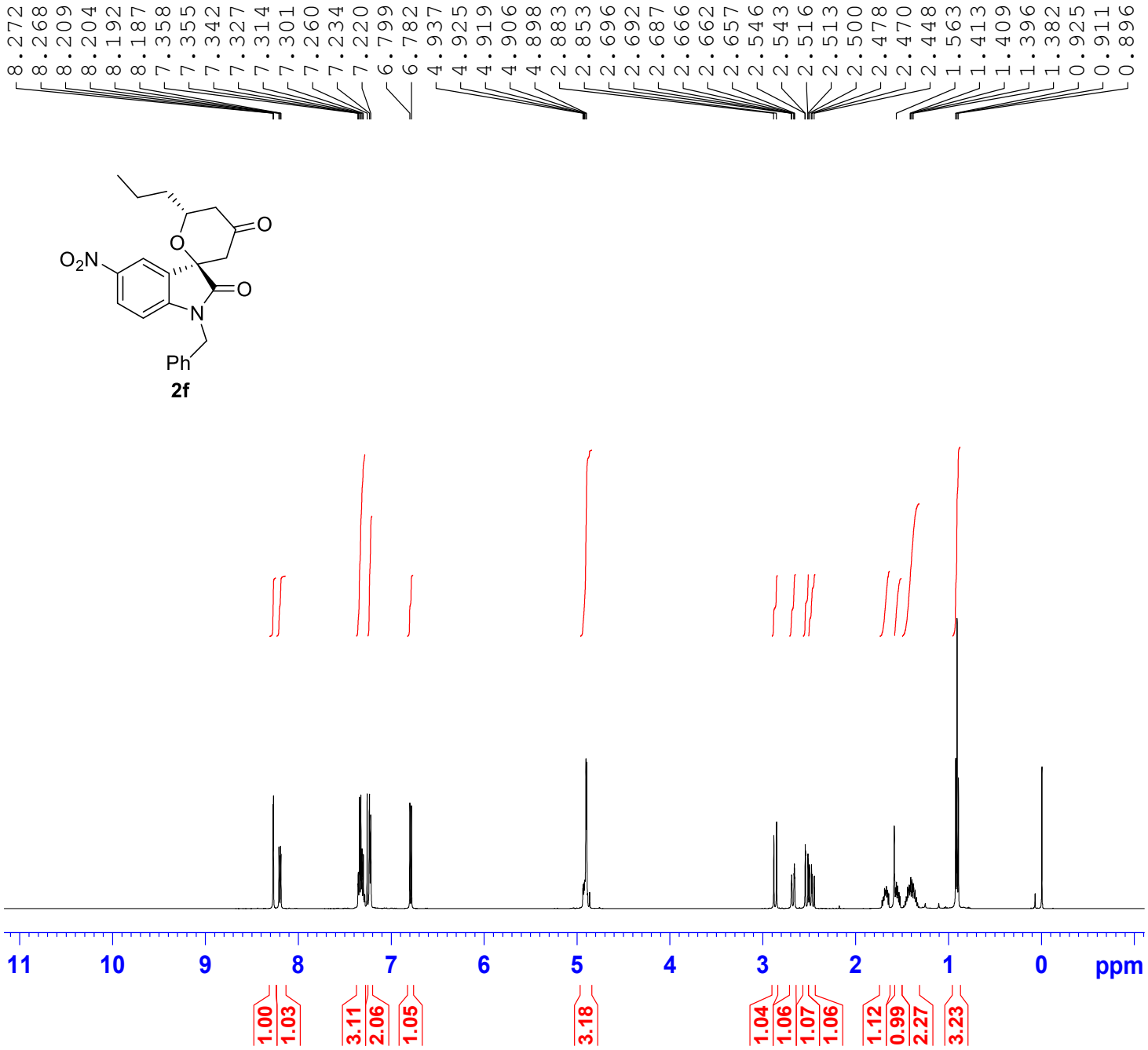
3e

```
Current Data Parameters
NAME      MH-89-Minor-Clean spectra-C13
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20190205
Time_            14.03 h
INSTRUM          Avance
PROBHD   Z151574_0027 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  311
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ             1.0878977 sec
RG                  101
DW               16.600 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1               13C
P0                3.33 usec
P1               10.00 usec
PLW1        88.26000214 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2        waltz65
PCPD2             80.00 usec
PLW2        23.68499947 W
PLW12        0.23014790 W
PLW13        0.11535020 W

F2 - Processing parameters
SI                32768
SF          125.7577959 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

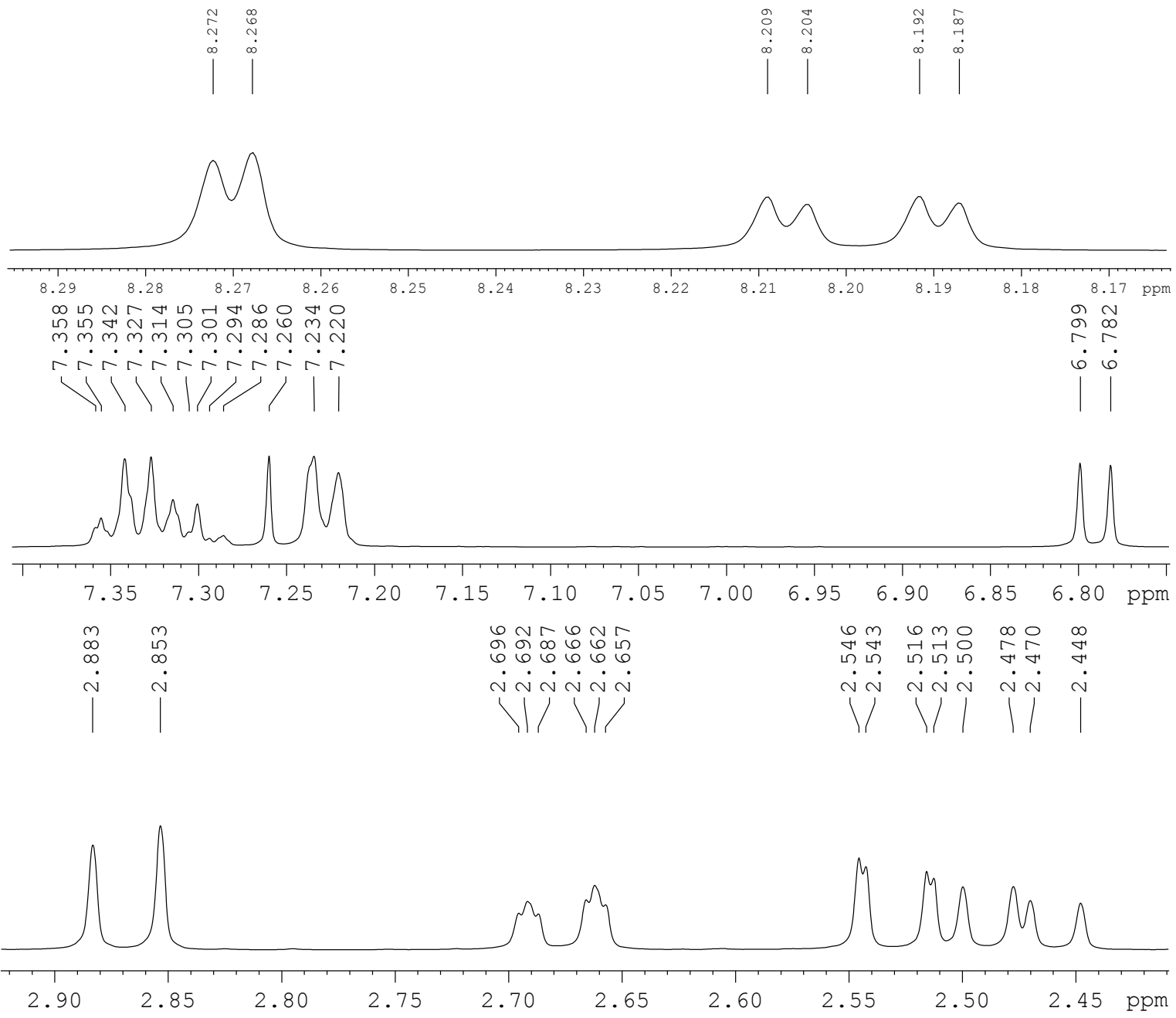





```
Current Data Parameters
NAME      MH-91-Major -clean spectra
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20190212
Time              14.11 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ            3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

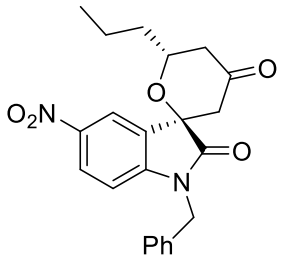
F2 - Processing parameters
SI                 65536
SF           500.1300121 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME      MH-91-Major -clean spectra
EXPNO                 1
PROCNO                1

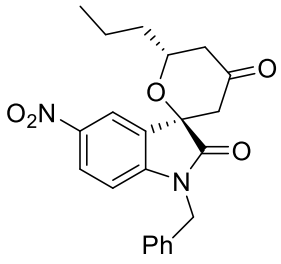
F2 - Acquisition Parameters
Date_           20190212
Time              14.11 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES        0.305176 Hz
AQ            3.2767999 sec
RG                    32
DW               50.000 usec
DE               11.14 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                2.67 usec
P1                8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300121 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



2f

202.93
174.75
147.80
143.98
134.07
129.59
129.16
128.28
127.19
127.07
120.22
109.44
77.25
77.15
77.00
76.74
72.12
46.83
45.12
43.79
38.38
18.11
13.80



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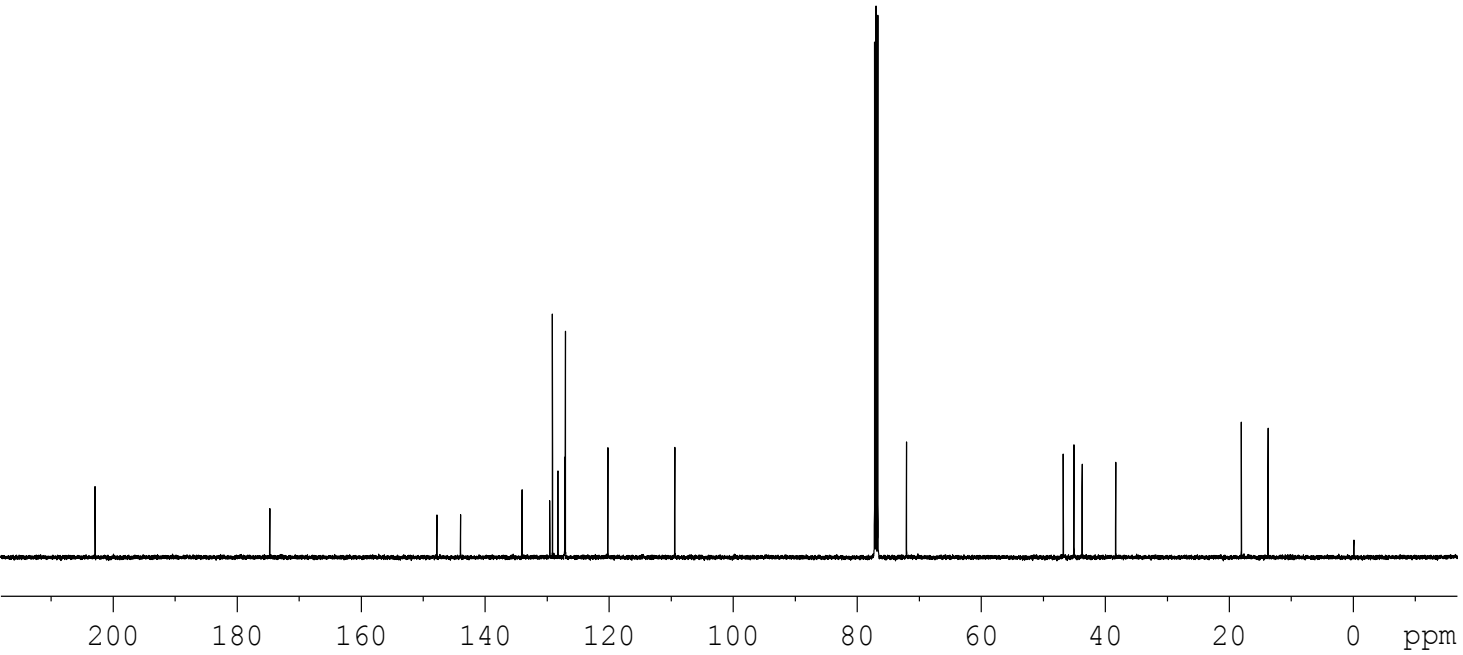
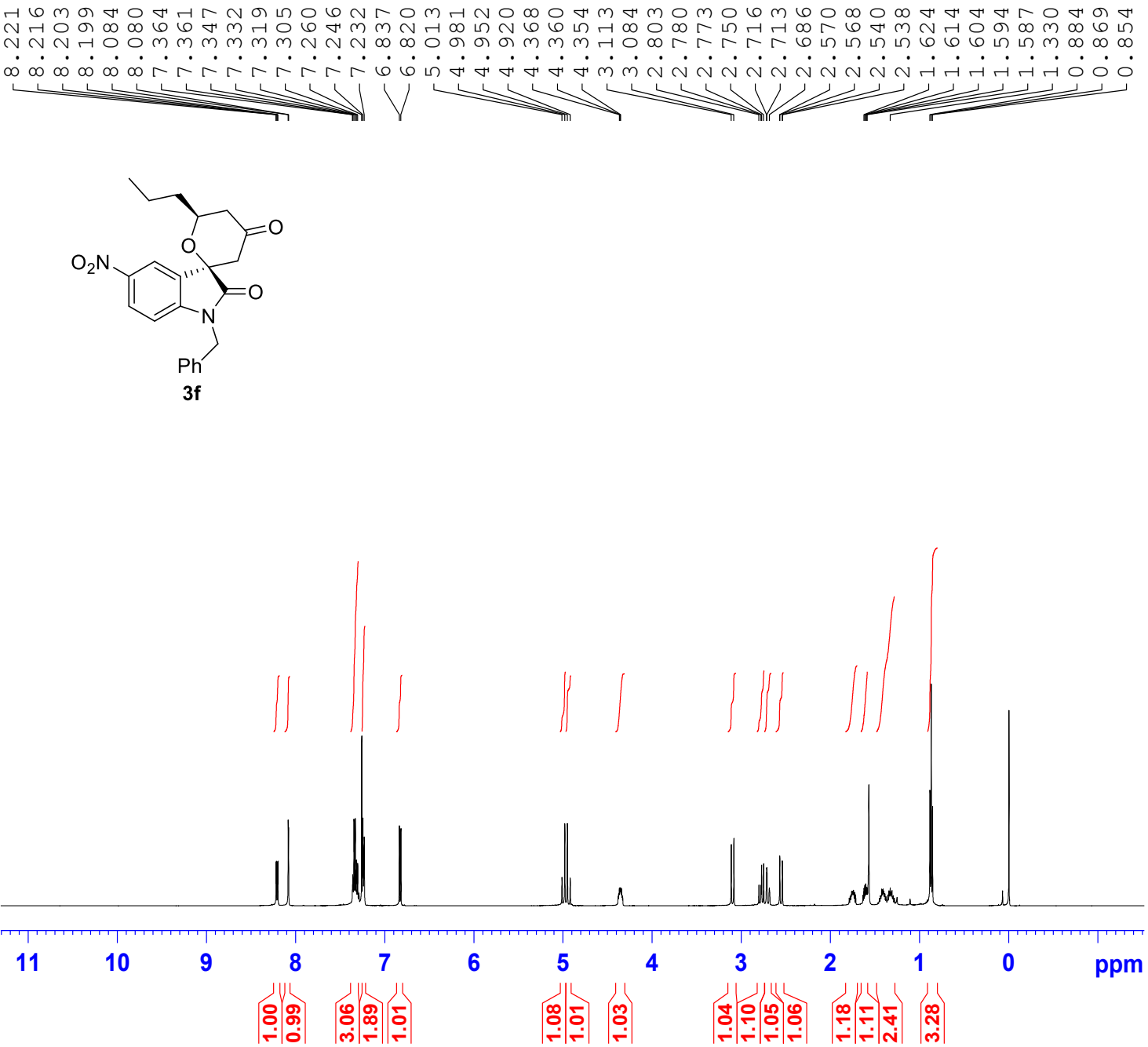



```
Current Data Parameters
NAME      MH-91-Major -clean spectra-C13
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190212
Time              14.55 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  707
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ            1.0878977 sec
RG                  101
DW               16.600 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1                13C
P0                3.33 usec
P1               10.00 usec
PLW1         88.26000214 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2            80.00 usec
PLW2         23.68499947 W
PLW12         0.23014790 W
PLW13         0.11535020 W

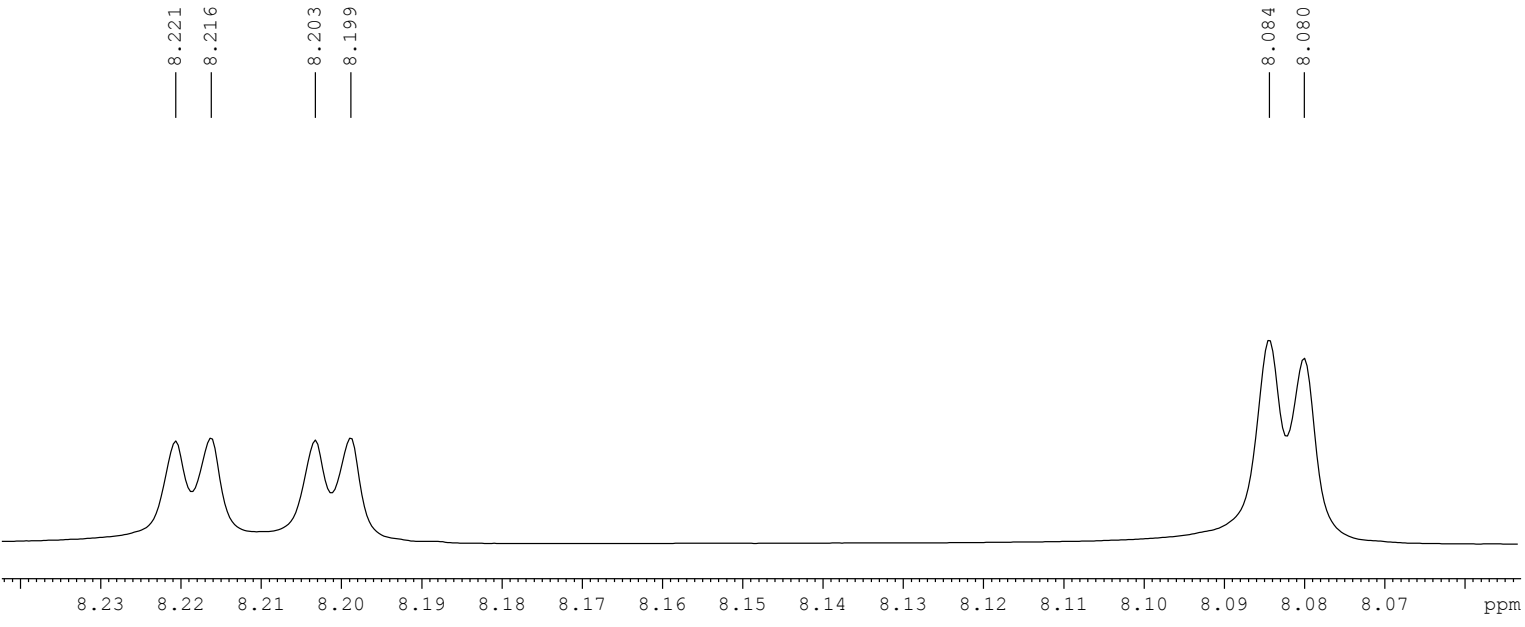
F2 - Processing parameters
SI                 32768
SF          125.7577934 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

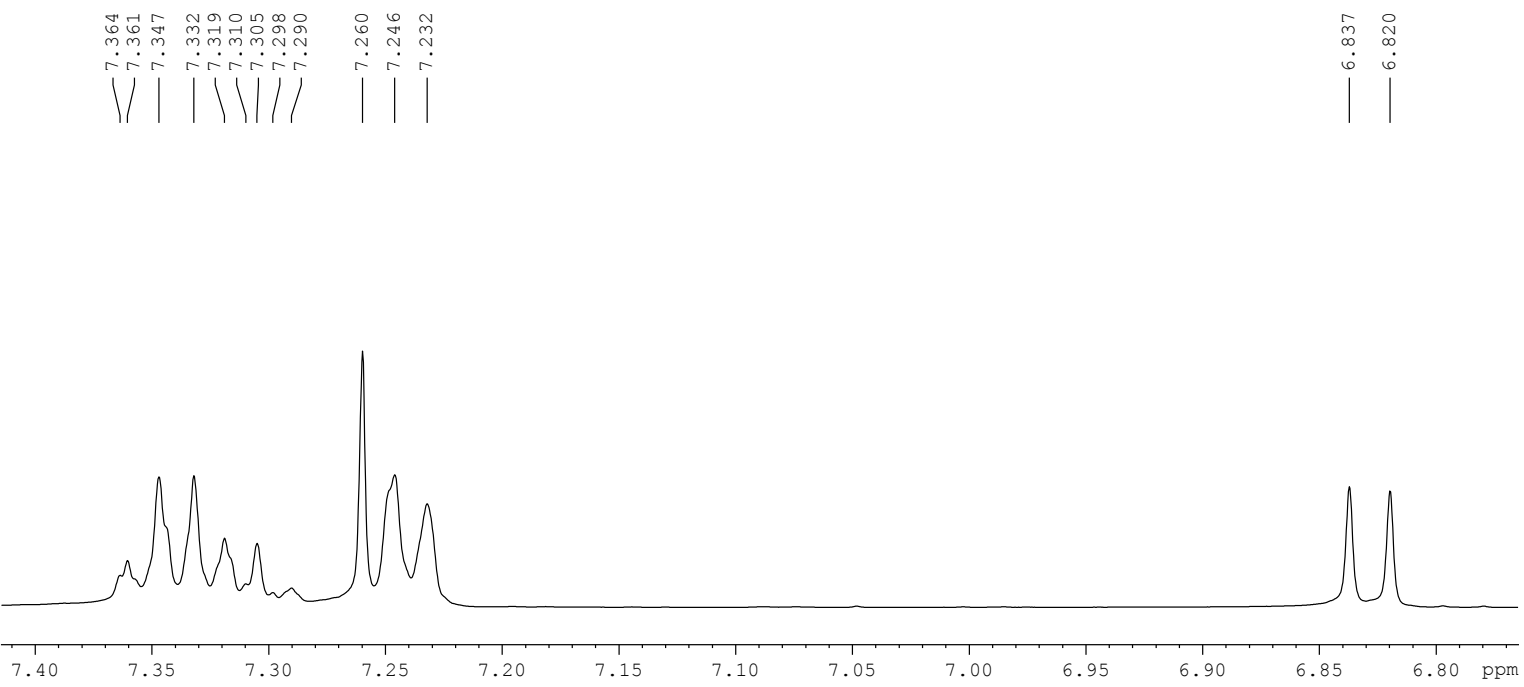



```
Current Data Parameters
NAME      MH-91-Minor -Clean spectra
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20190212
Time              15.01 h
INSTRUM           Avance
PROBHD    Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          23.68499947 W

F2 - Processing parameters
SI                 65536
SF            500.1300123 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

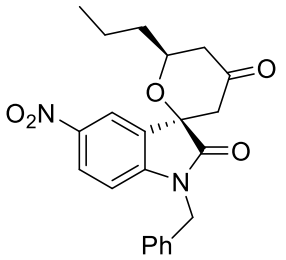





```
Current Data Parameters
NAME      MH-91-Minor -Clean spectra
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20190212
Time              15.01 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300123 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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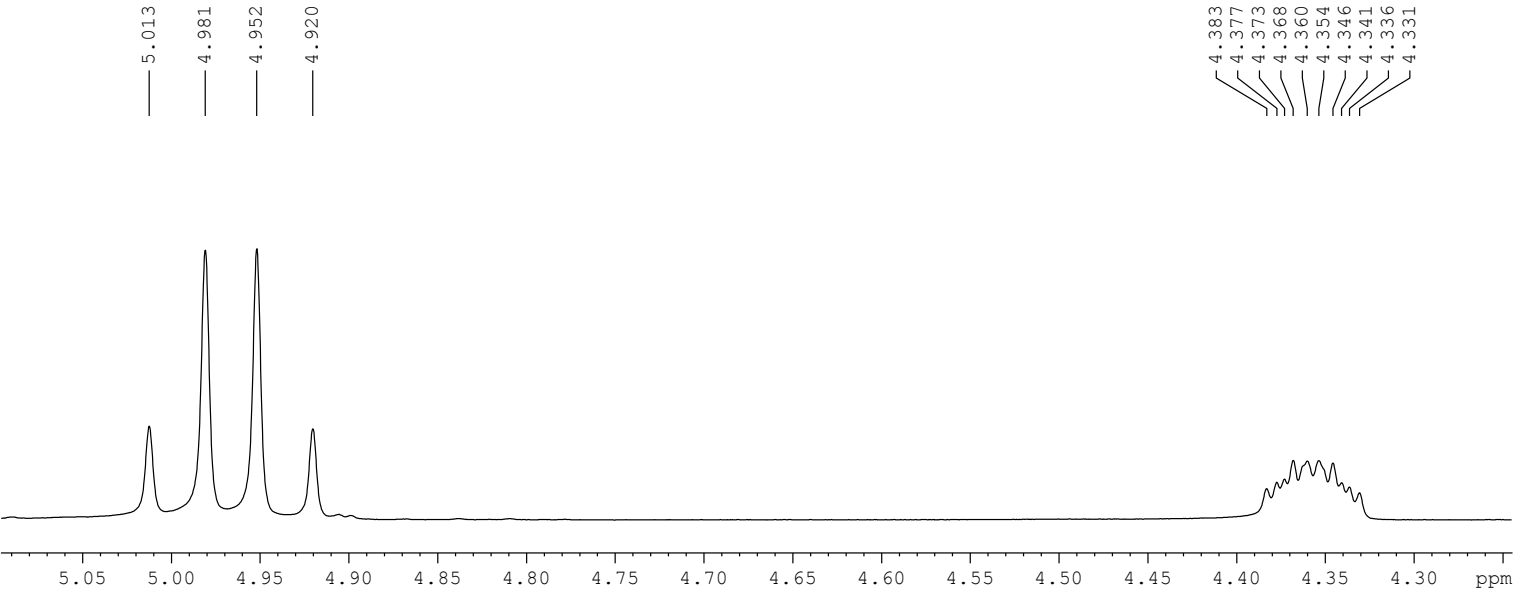
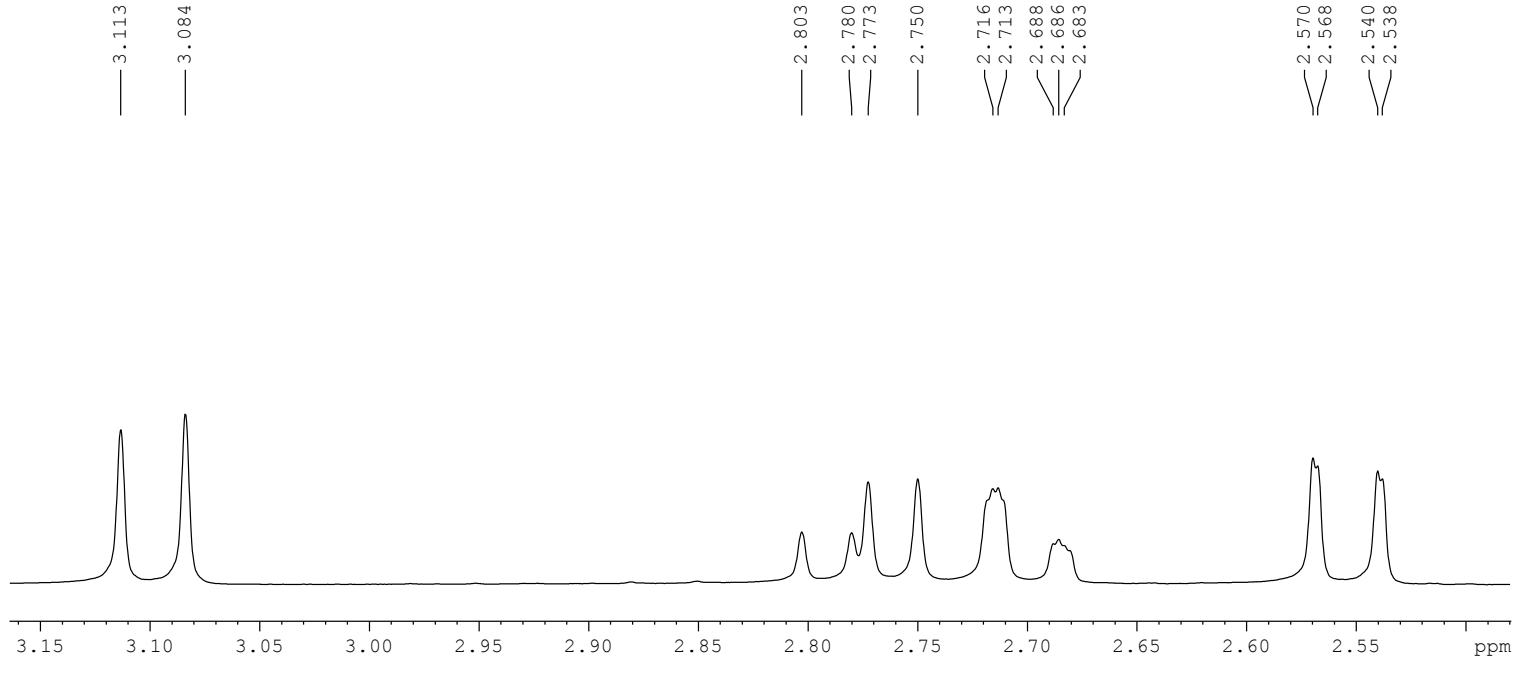
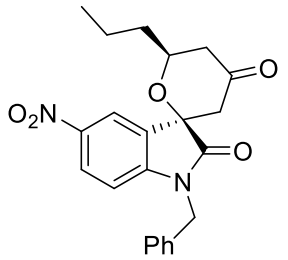




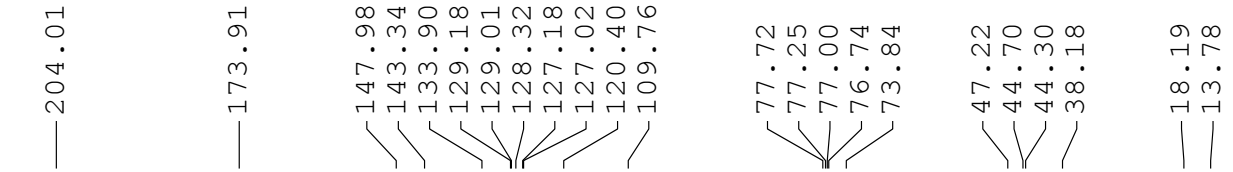

```
Current Data Parameters
NAME      MH-91-Minor -Clean spectra
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20190212
Time              15.01 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

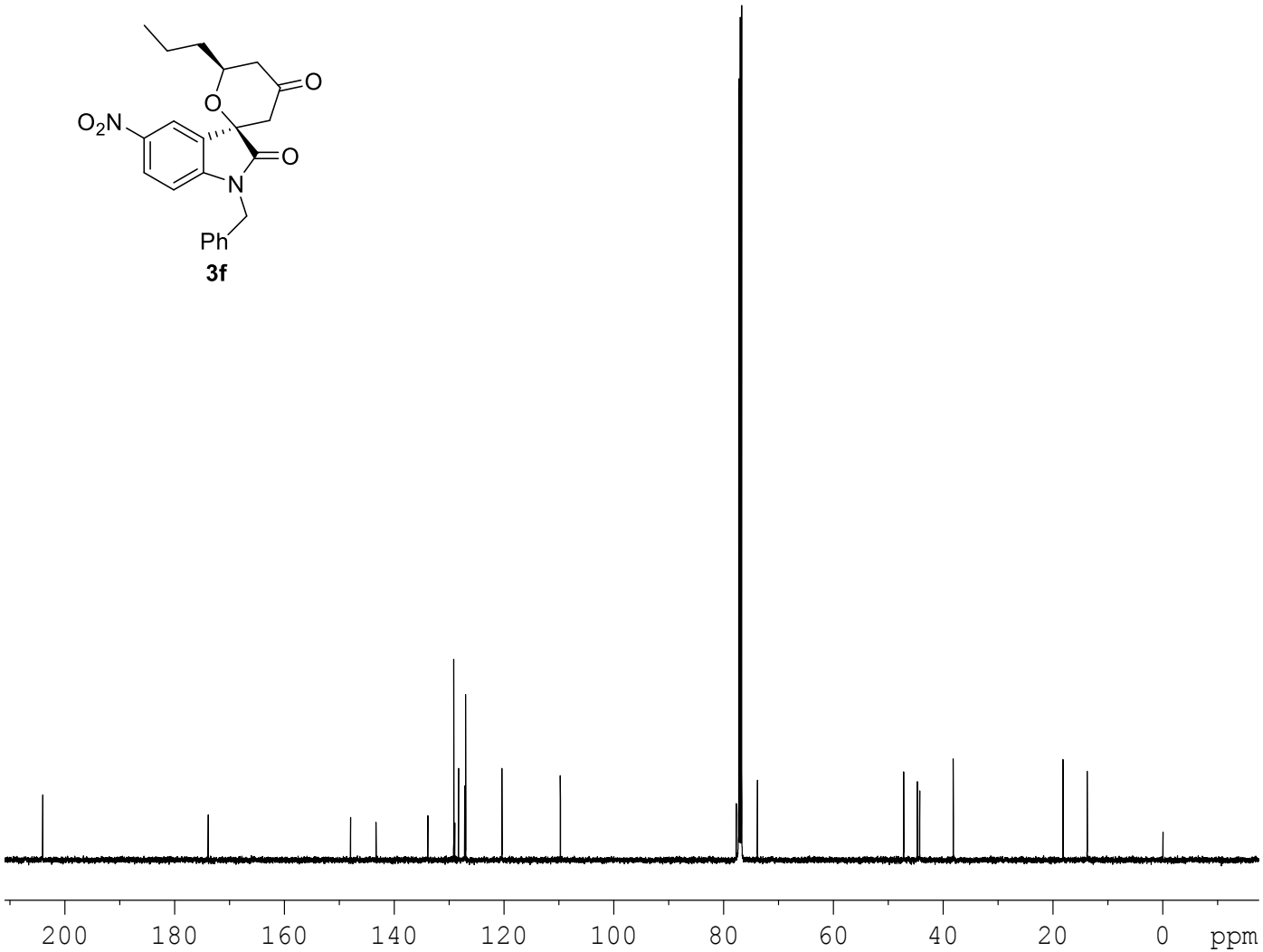
F2 - Processing parameters
SI                 65536
SF           500.1300123 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



3f



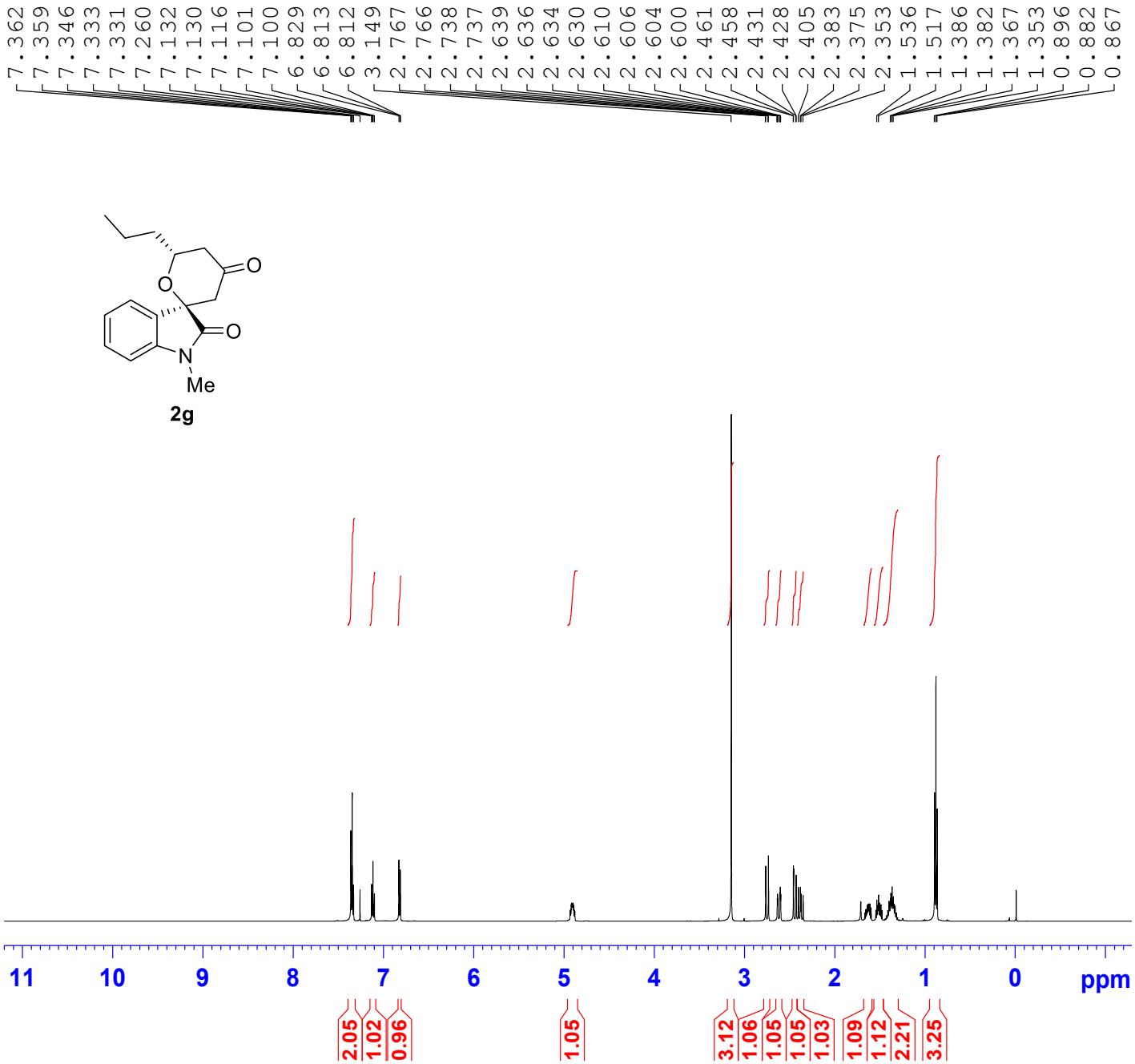
3f




```
Current Data Parameters
NAME      MH-91-Minor -Clean spectra-C13
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190212
Time              16.00 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ            1.0878977 sec
RG                  101
DW                16.600 usec
DE                 6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1               13C
P0                3.33 usec
P1               10.00 usec
PLW1         88.26000214 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2         waltz65
PCPD2             80.00 usec
PLW2         23.68499947 W
PLW12         0.23014790 W
PLW13         0.11535020 W

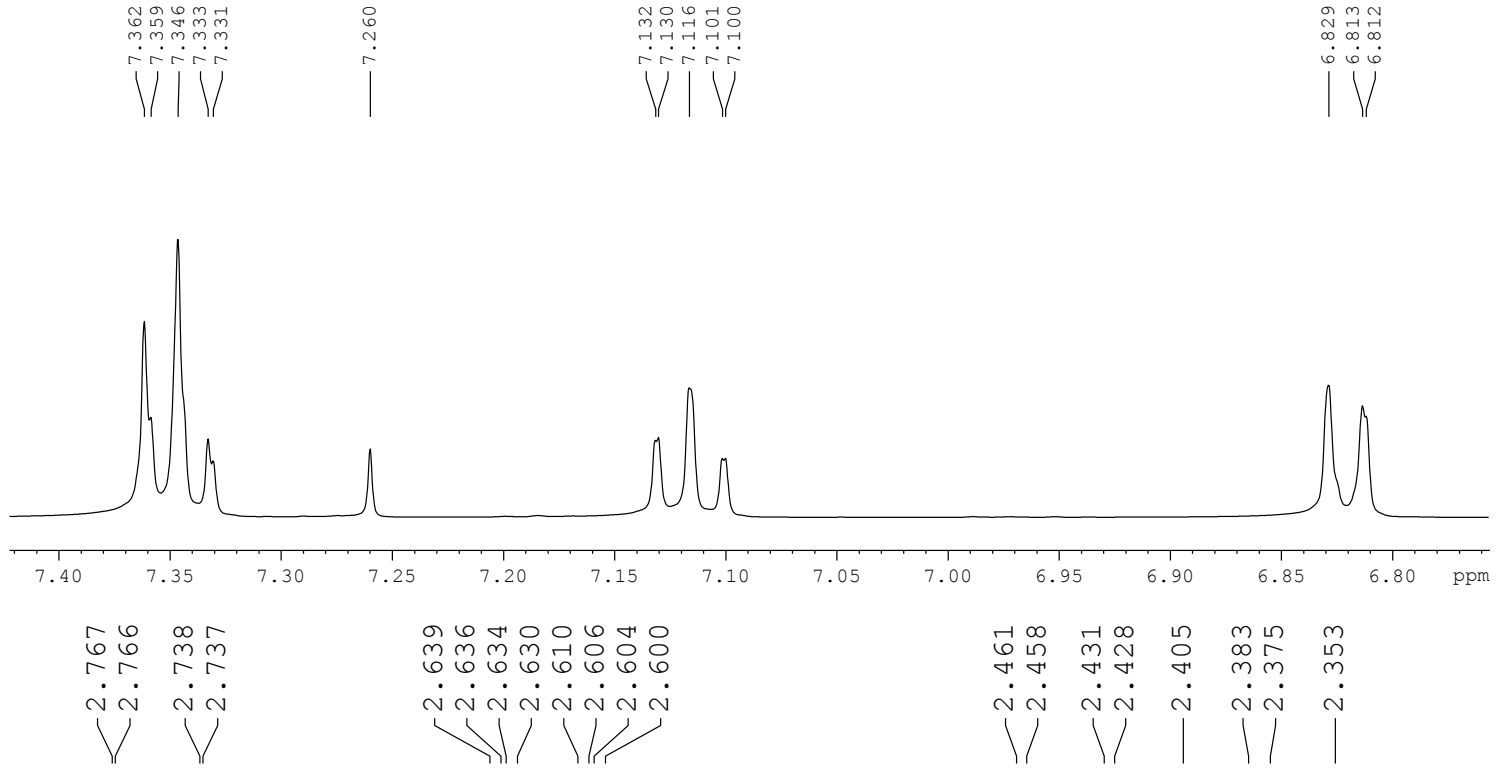
F2 - Processing parameters
SI                32768
SF          125.7577926 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

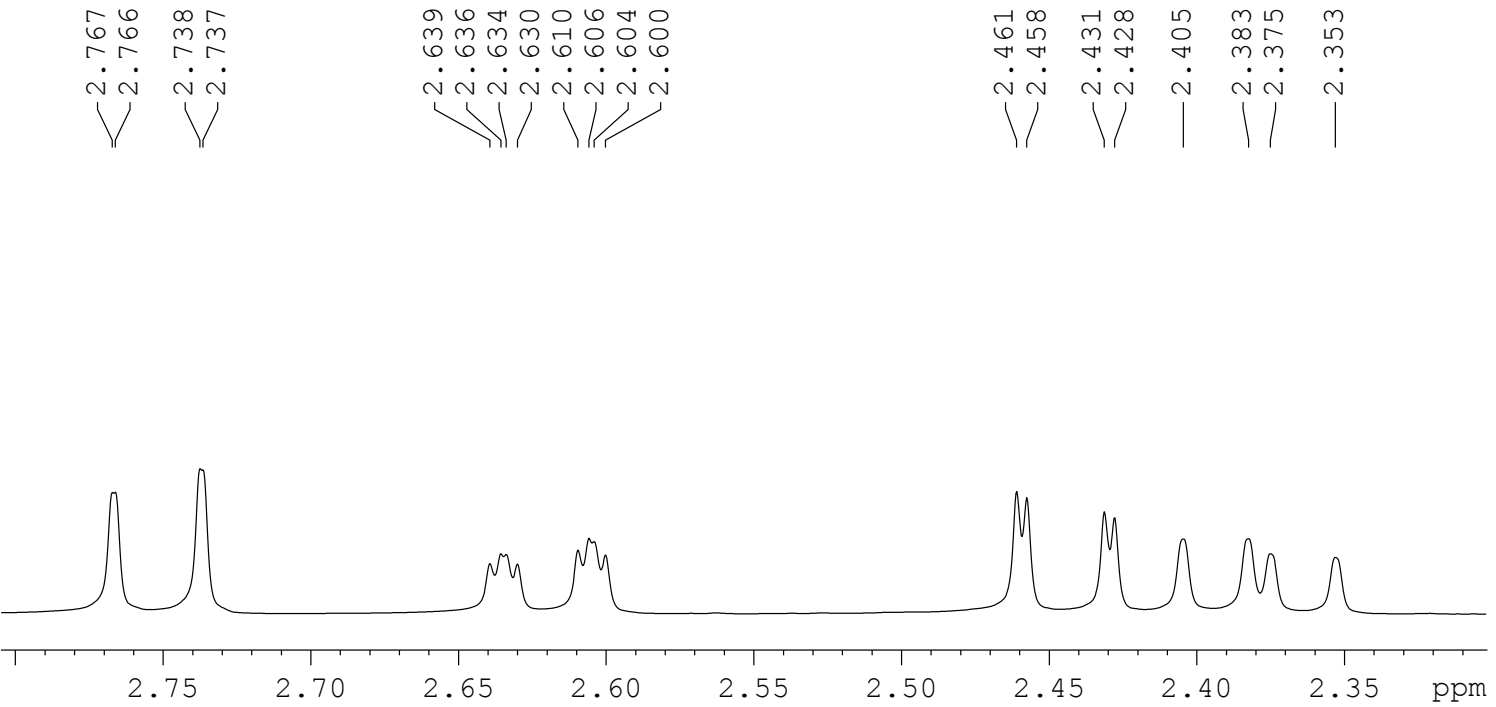



```
Current Data Parameters
NAME      MH-88-Major-Clean spectra
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190204
Time              11.12 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ            3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300122 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

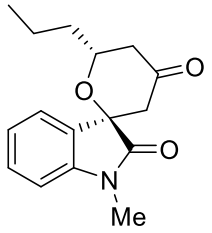





```
Current Data Parameters
NAME      MH-88-Major-Clean spectra
EXPNO                  1
PROCNO                 1

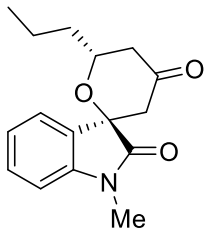
F2 - Acquisition Parameters
Date_           20190204
Time              11.12 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300122 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



2g

204.50
174.61
143.09
130.29
128.80
123.86
123.34
108.55
77.70
77.25
77.00
76.74
71.54
46.74
45.70
38.51
25.90
18.04
13.91

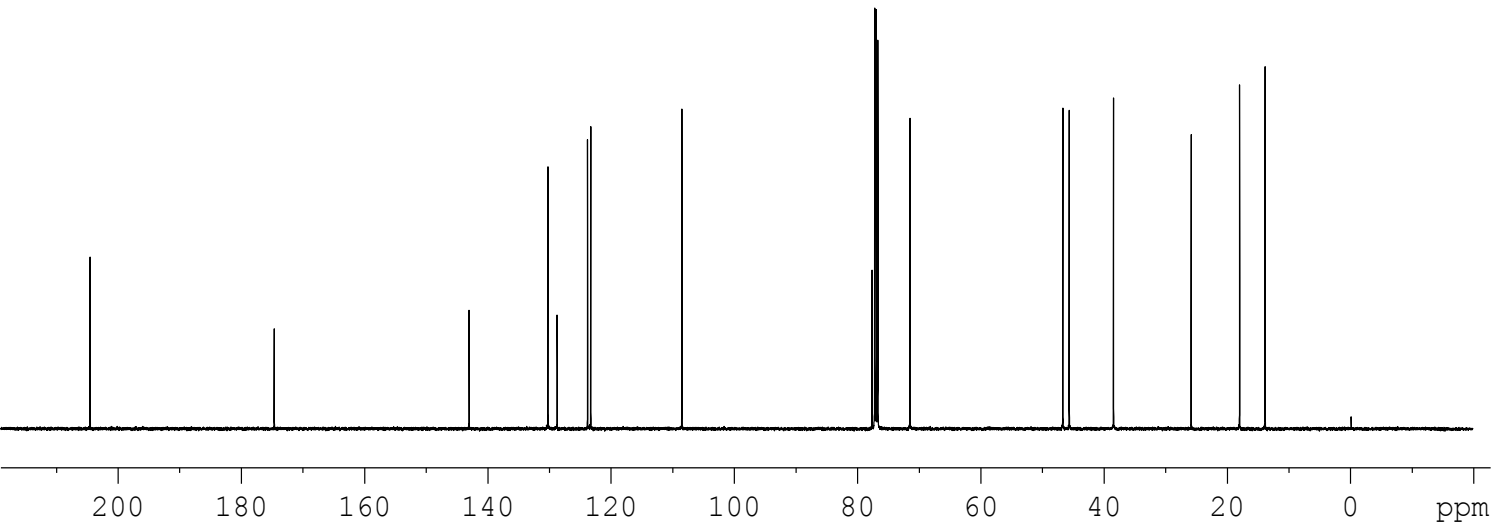


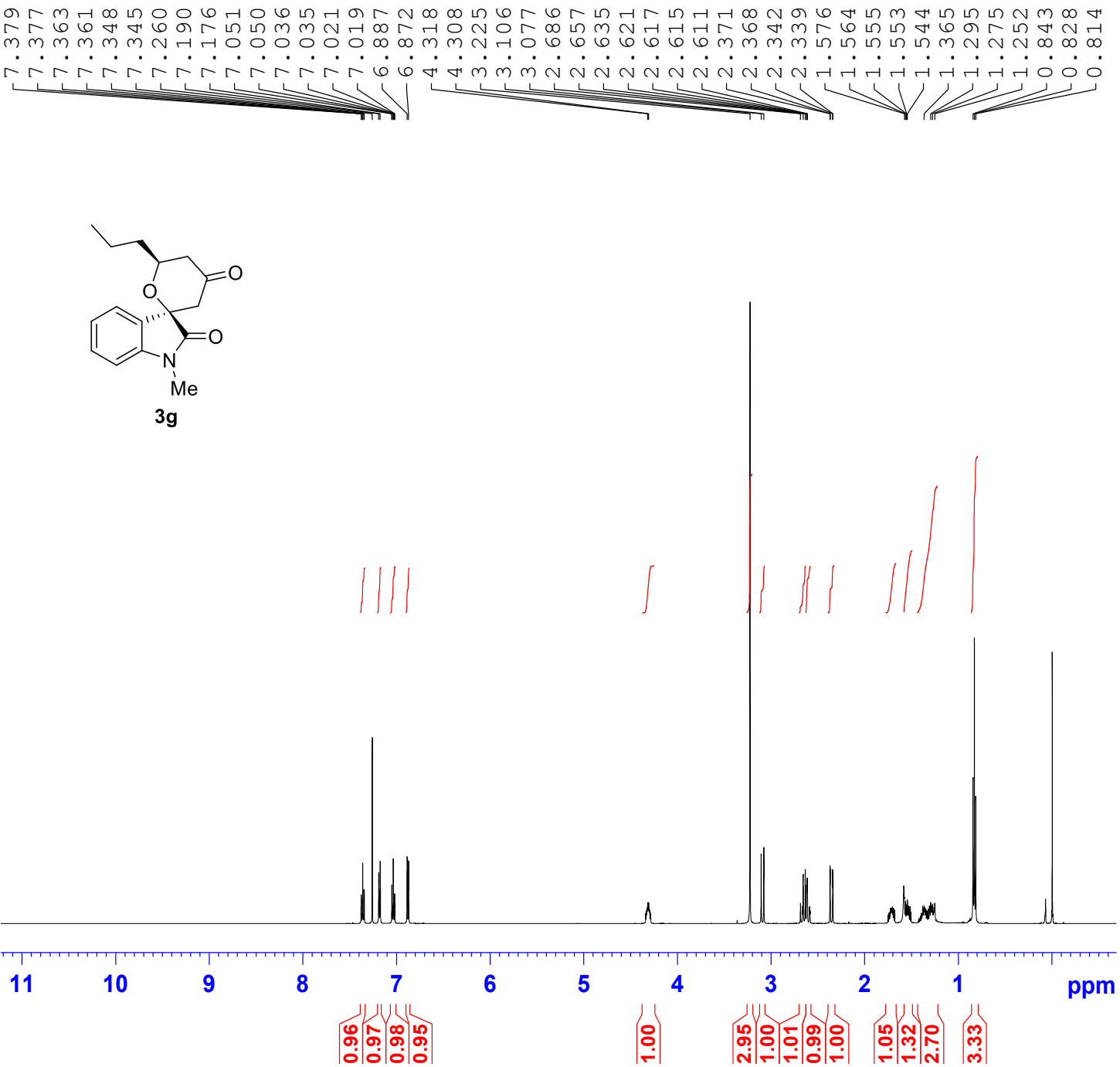
2g

```
Current Data Parameters
NAME      MH-88-Major-Clean spectra-C13
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20190204
Time              12.20 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ            1.0878977 sec
RG                  101
DW               16.600 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1         125.7703643 MHz
NUC1                13C
P0                3.33 usec
P1               10.00 usec
PLW1         88.26000214 W
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             80.00 usec
PLW2         23.68499947 W
PLW12         0.23014790 W
PLW13         0.11535020 W

F2 - Processing parameters
SI                32768
SF           125.7577975 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

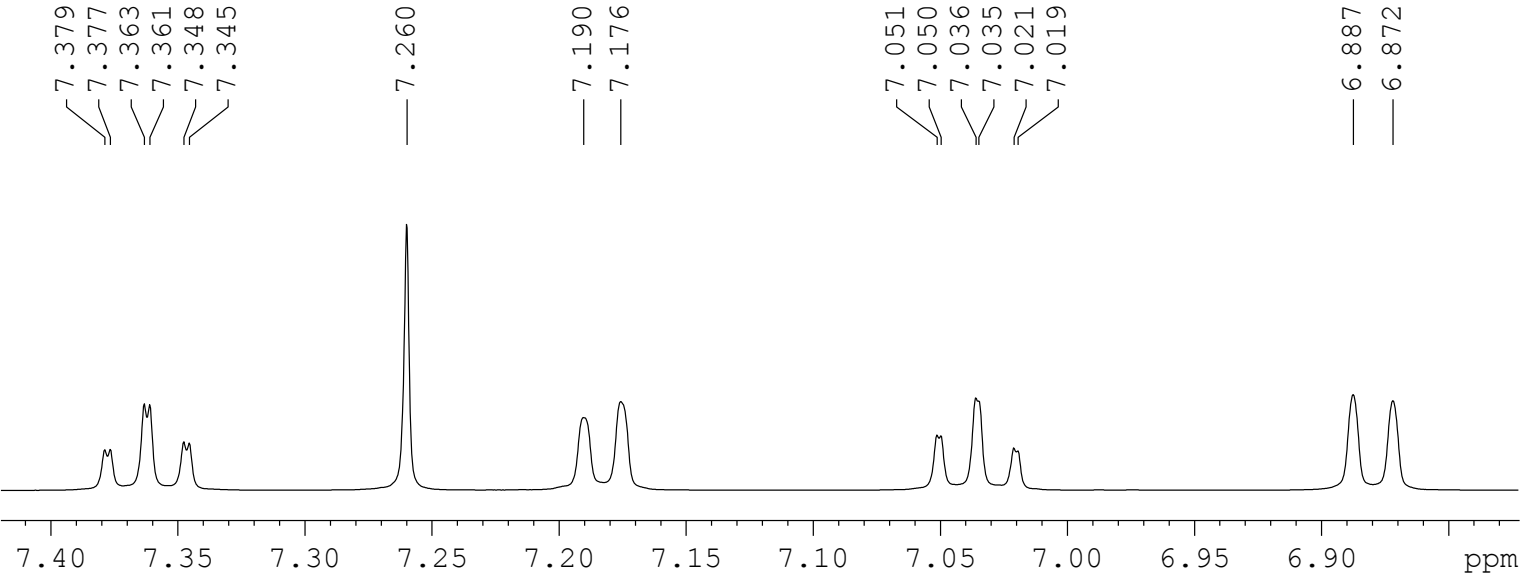


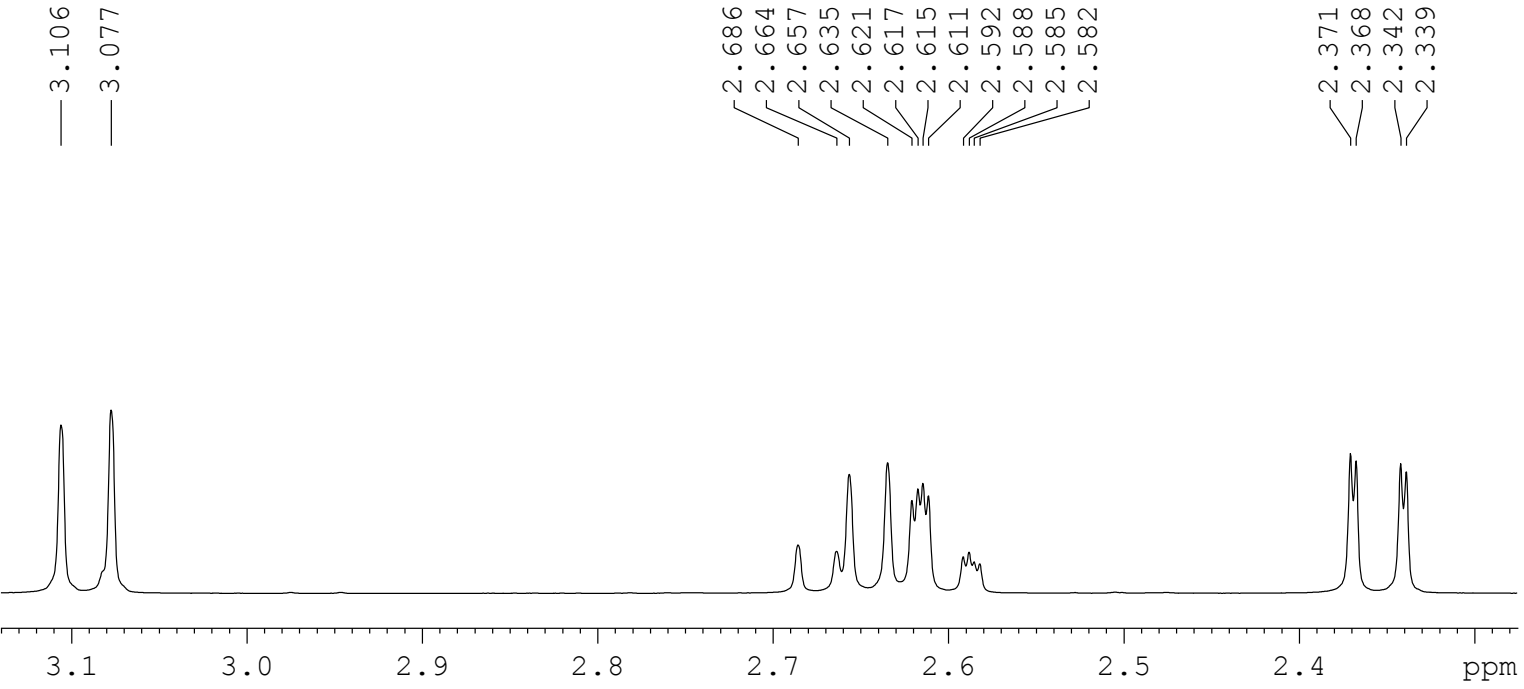



```
Current Data Parameters
NAME      MH-88-MINOR-Clean spectra
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190204
Time              12.27 h
INSTRUM           Avance
PROBHD    Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES        0.305176 Hz
AQ            3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300121 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

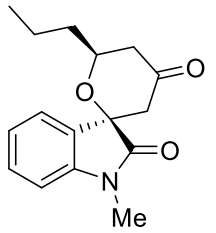




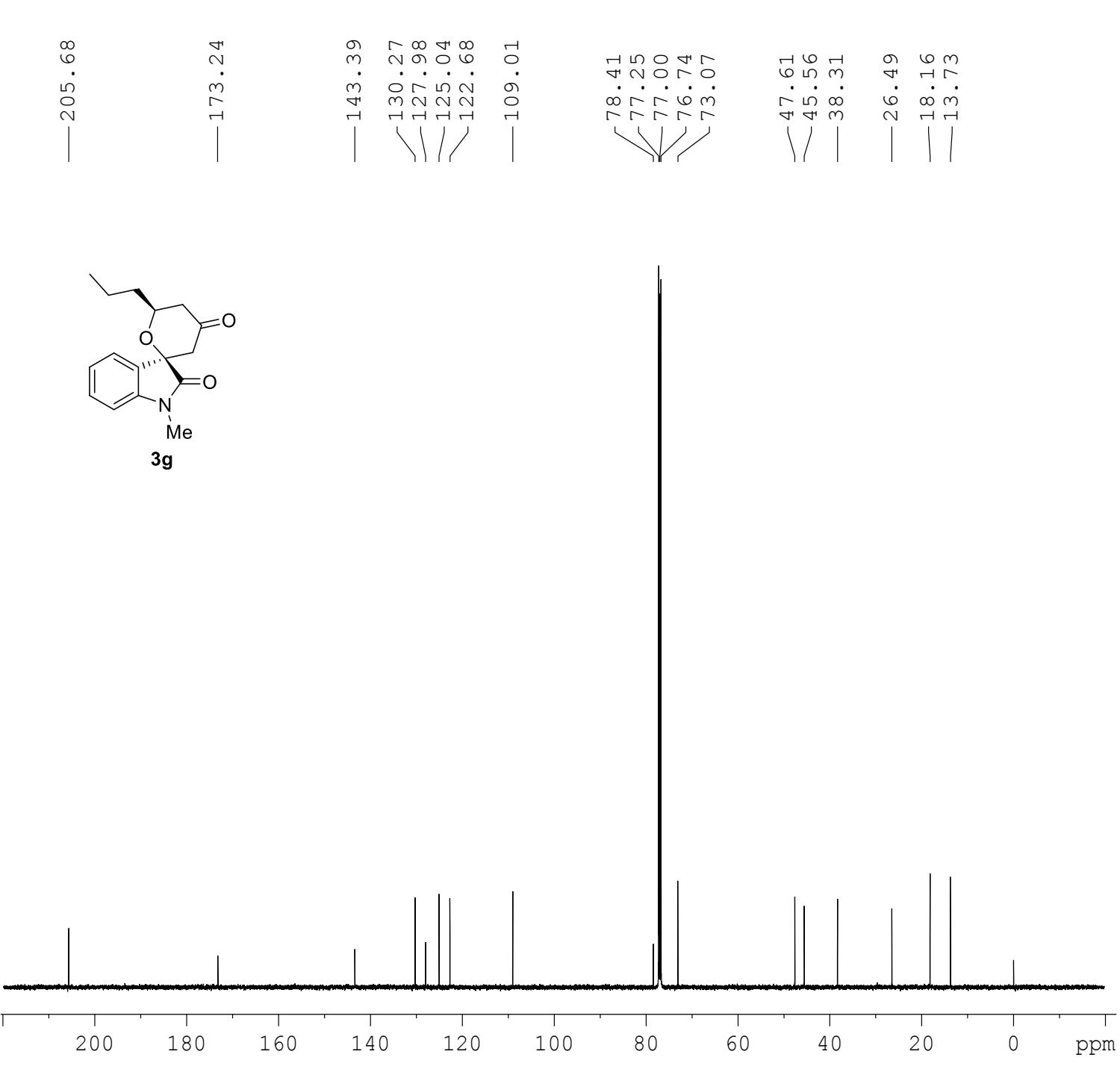

```
Current Data Parameters
NAME      MH-88-MINOR-Clean spectra
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20190204
Time              12.27 h
INSTRUM           Avance
PROBHD    Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          23.68499947 W

F2 - Processing parameters
SI                 65536
SF            500.1300121 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



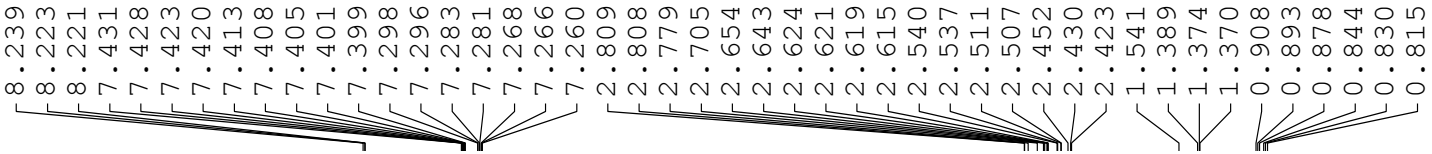
3g

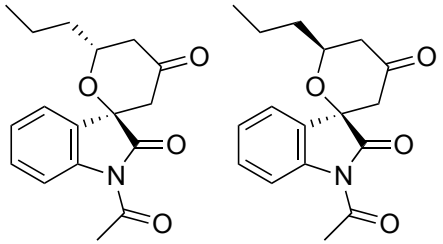



```
Current Data Parameters
NAME      MH-88-MINOR-Clean spectra-C13
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20190204
Time              13.33 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ            1.0878977 sec
RG                  101
DW               16.600 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1               13C
P0                3.33 usec
P1               10.00 usec
PLW1         88.26000214 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2         waltz65
PCPD2             80.00 usec
PLW2         23.68499947 W
PLW12         0.23014790 W
PLW13         0.11535020 W

F2 - Processing parameters
SI                32768
SF          125.7577921 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

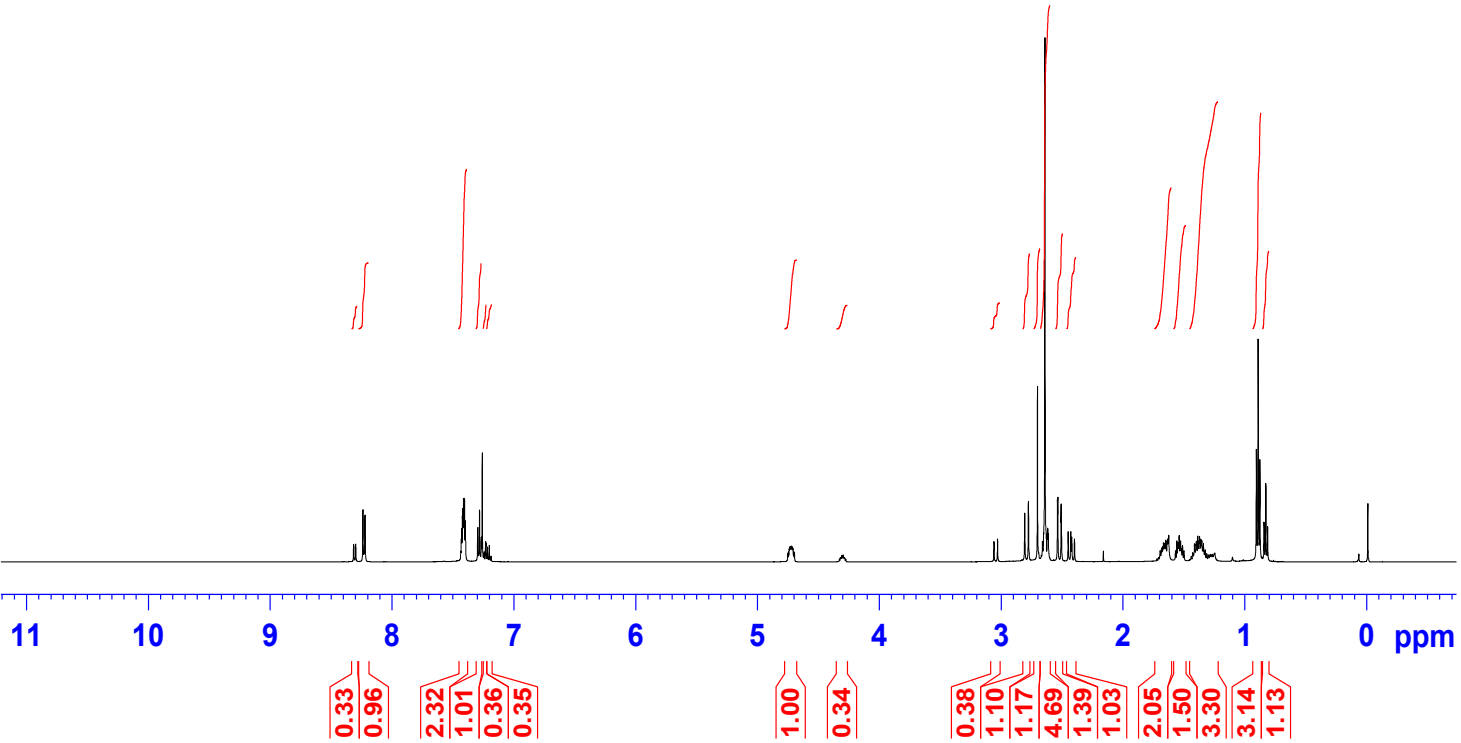




2h

3h

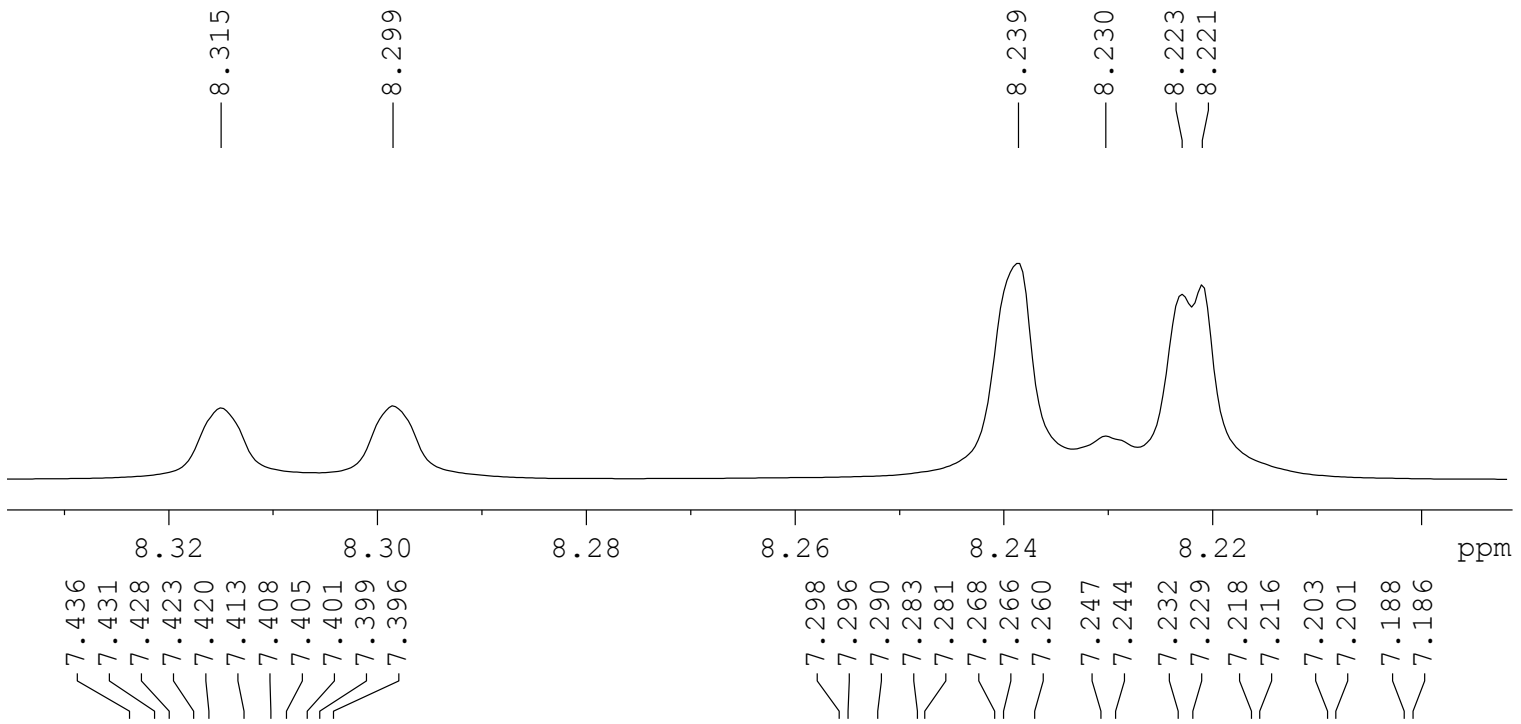
3:1

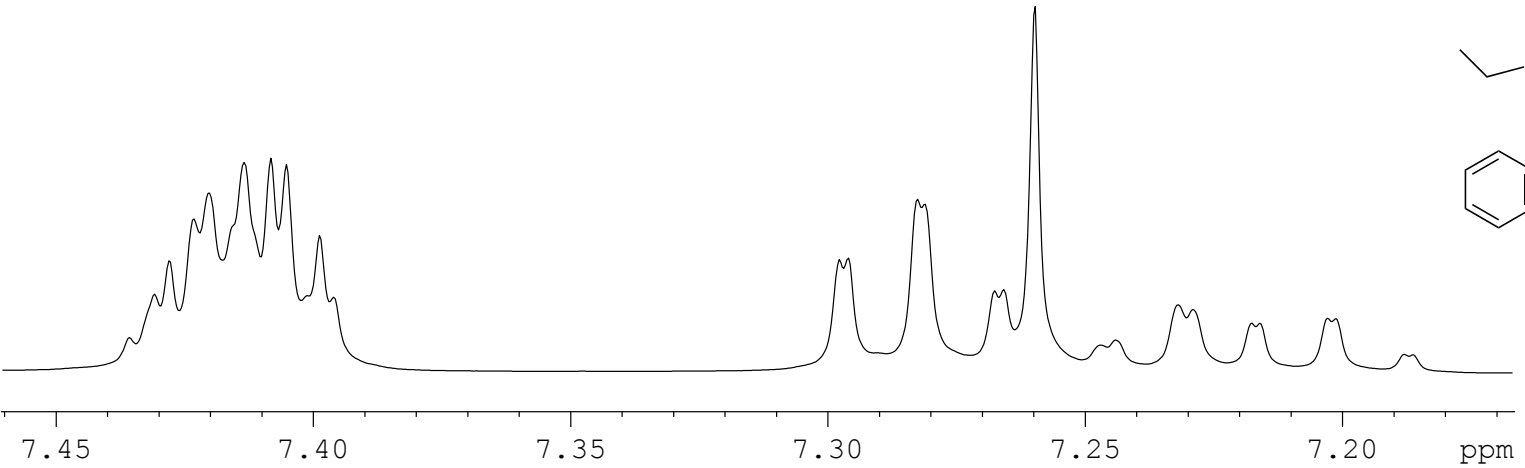



```
Current Data Parameters
NAME          MH-92-Pure
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190207
Time              15.31 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          23.68499947 W

F2 - Processing parameters
SI                 65536
SF          500.1300124 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

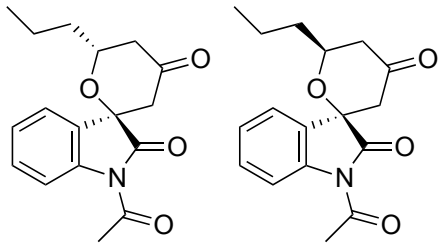




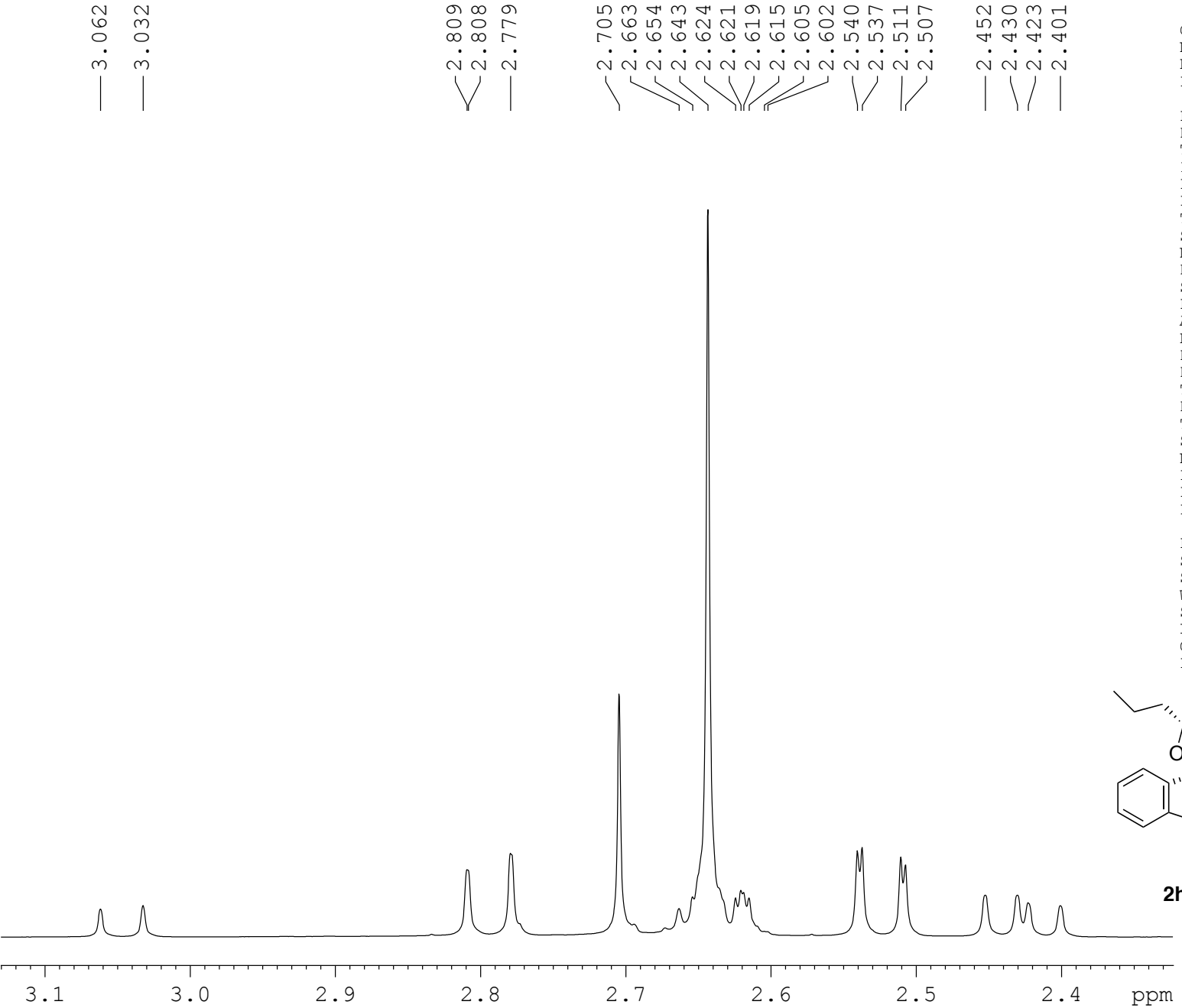

```
Current Data Parameters
NAME          MH-92-Pure
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190207
Time              15.31 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ              3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300124 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



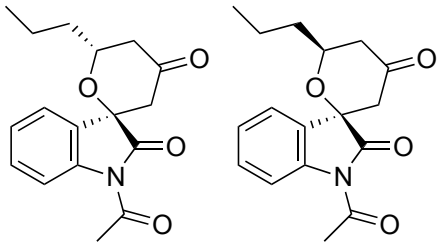
2h 3:1 **3h**



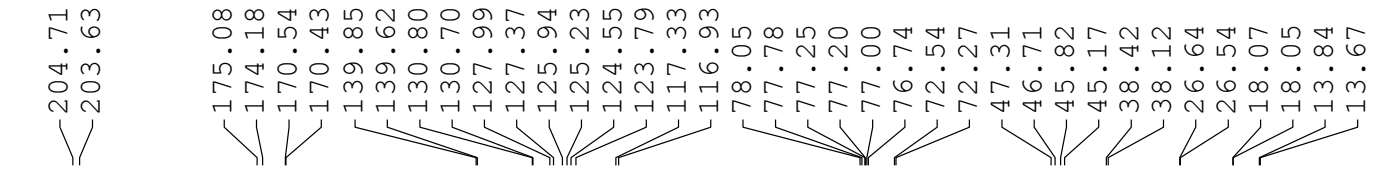

```
Current Data Parameters
NAME          MH-92-Pure
EXPNO                  1
PROCNO                 1

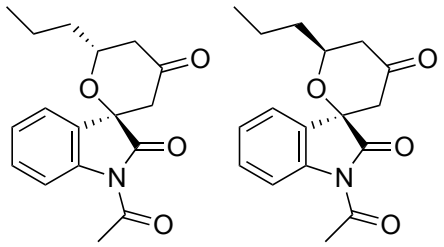
F2 - Acquisition Parameters
Date_           20190207
Time              15.31 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300124 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



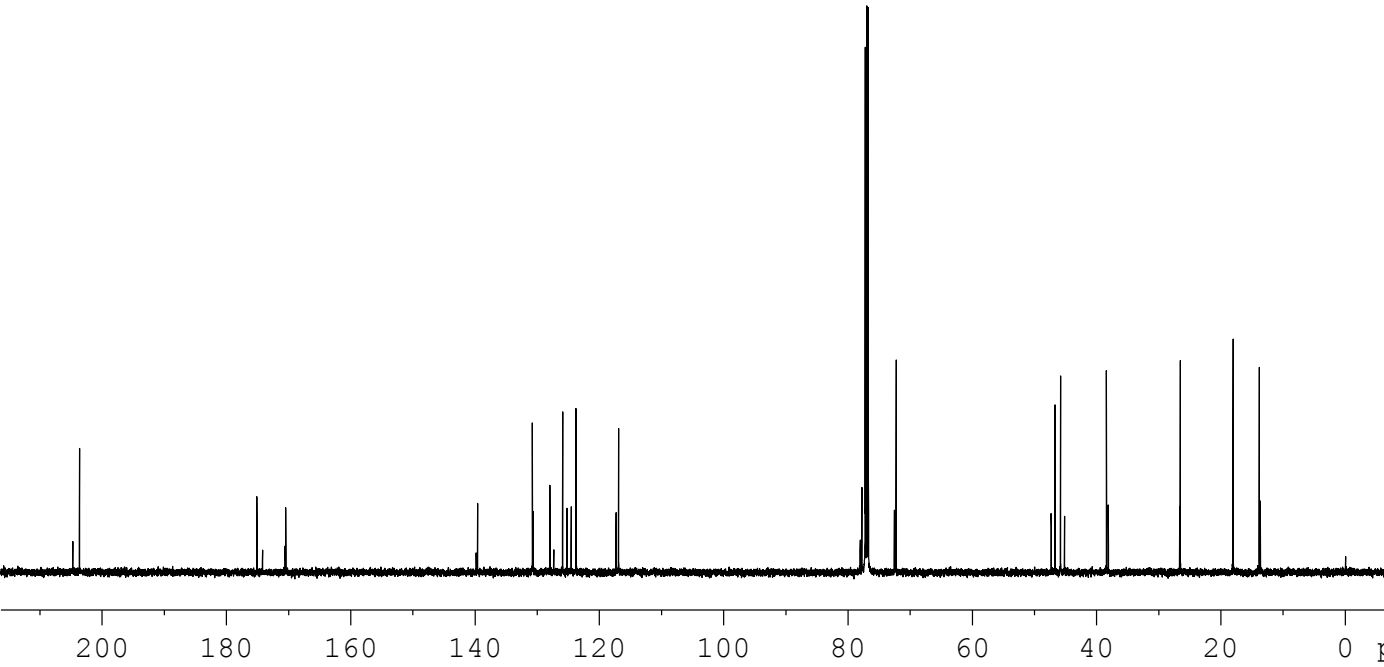
2h 3:1 **3h**





2h **3h**

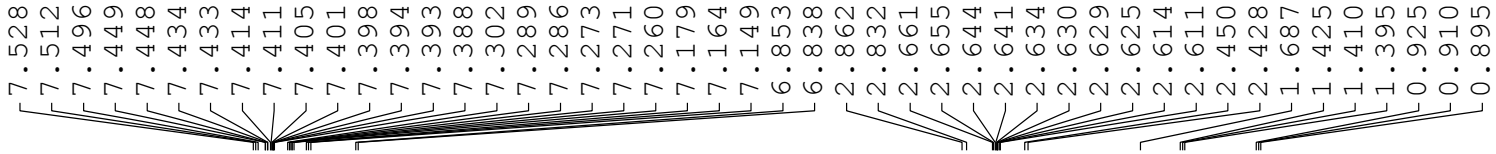
3:1

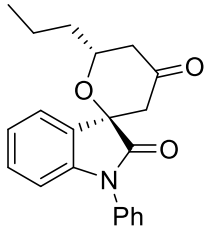



```
Current Data Parameters
NAME      MH-92-PURE-C13-again
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_              20190207
Time                 17.06 h
INSTRUM              Avance
PROBHD    Z151574_0027 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      286
DS                        4
SWH              30120.482 Hz
FIDRES            0.919204 Hz
AQ               1.0878977 sec
RG                      101
DW                  16.600 usec
DE                   6.50 usec
TE                   298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1
SFO1           125.7703643 MHz
NUC1                   13C
P0                   3.33 usec
P1                  10.00 usec
PLW1           88.26000214 W
SFO2           500.1320005 MHz
NUC2                   1H
CPDPRG[2            waltz65
PCPD2                80.00 usec
PLW2           23.68499947 W
PLW12           0.23014790 W
PLW13           0.11535020 W

F2 - Processing parameters
SI                    32768
SF             125.7577943 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```



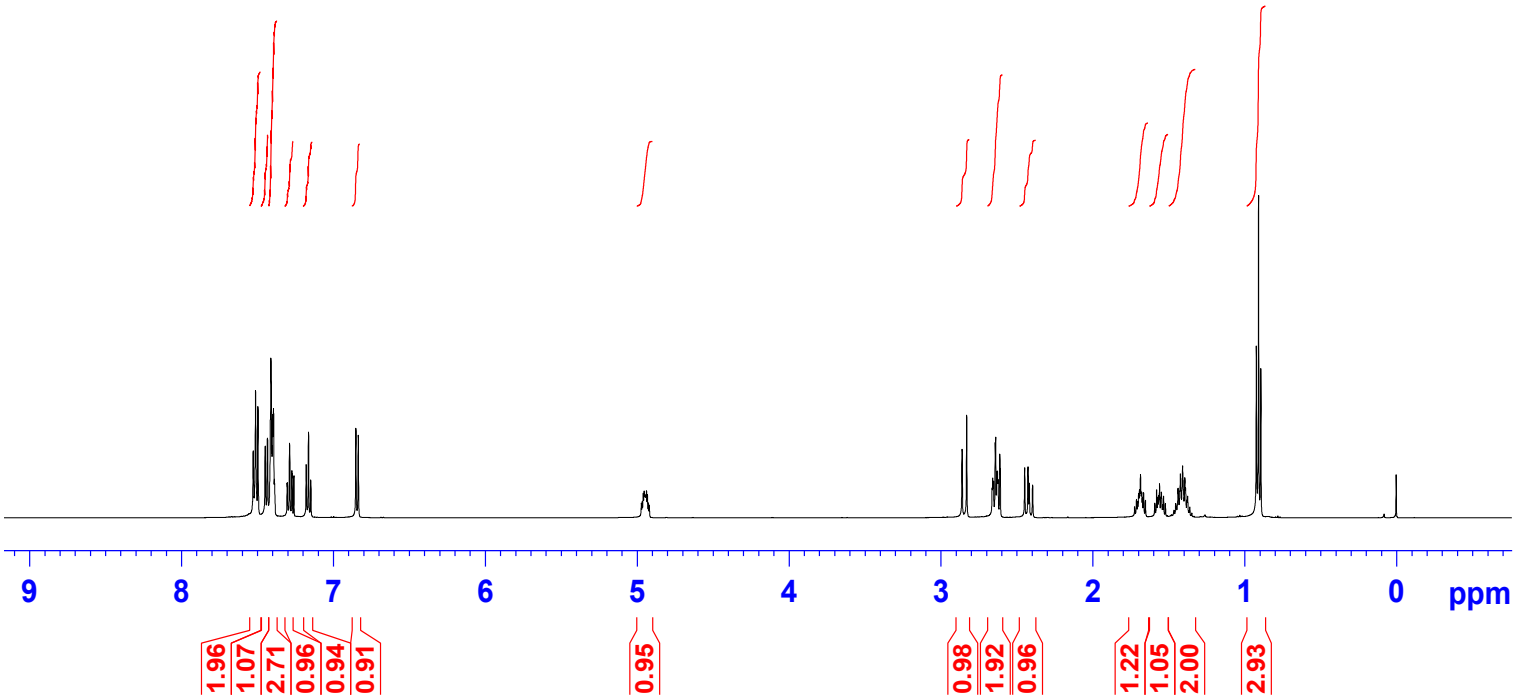


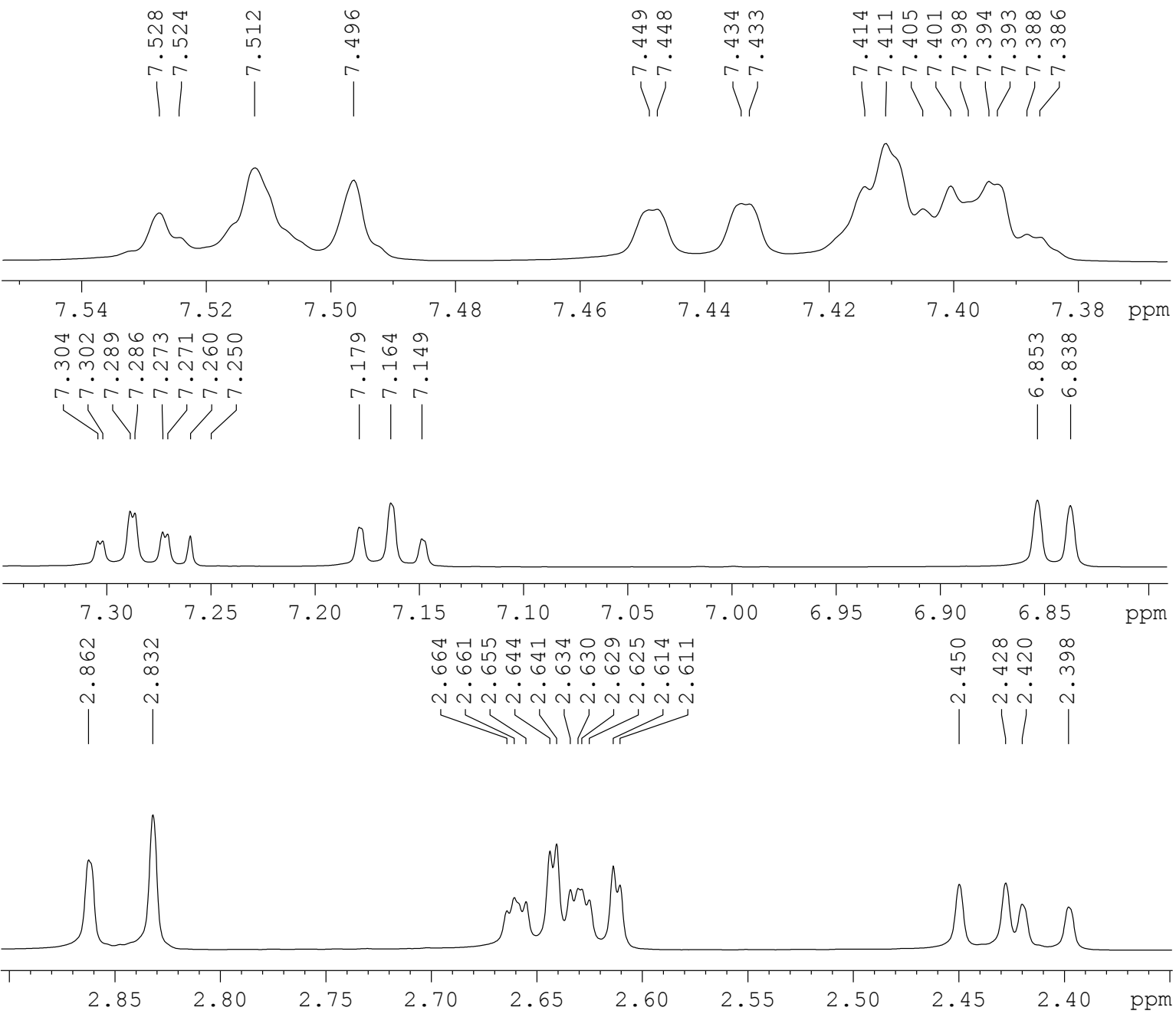
2i

```
Current Data Parameters
NAME      MH-93-Major-Clean spectra
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190205
Time              15.51 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ            3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300122 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

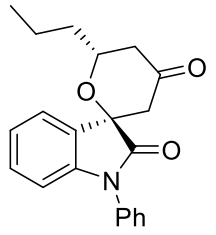




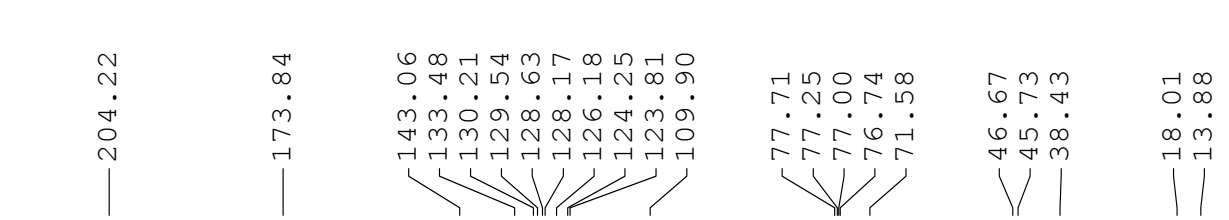

```
Current Data Parameters
NAME      MH-93-Major-Clean spectra
EXPNO                  1
PROCNO                 1

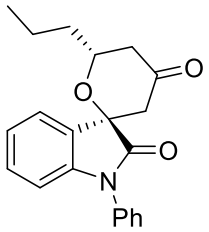
F2 - Acquisition Parameters
Date_           20190205
Time              15.51 h
INSTRUM           Avance
PROBHD    Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES        0.305176 Hz
AQ            3.2767999 sec
RG                    32
DW               50.000 usec
DE               11.14 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                2.67 usec
P1                8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300122 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

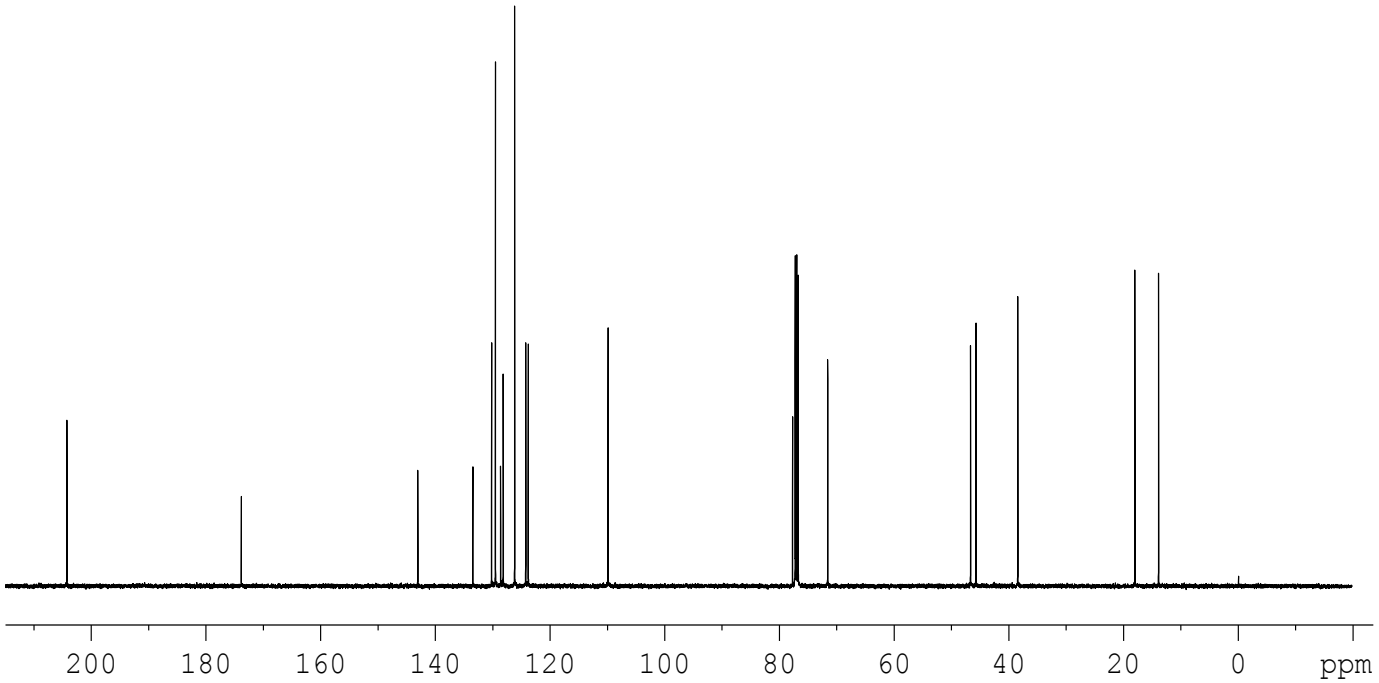


2i





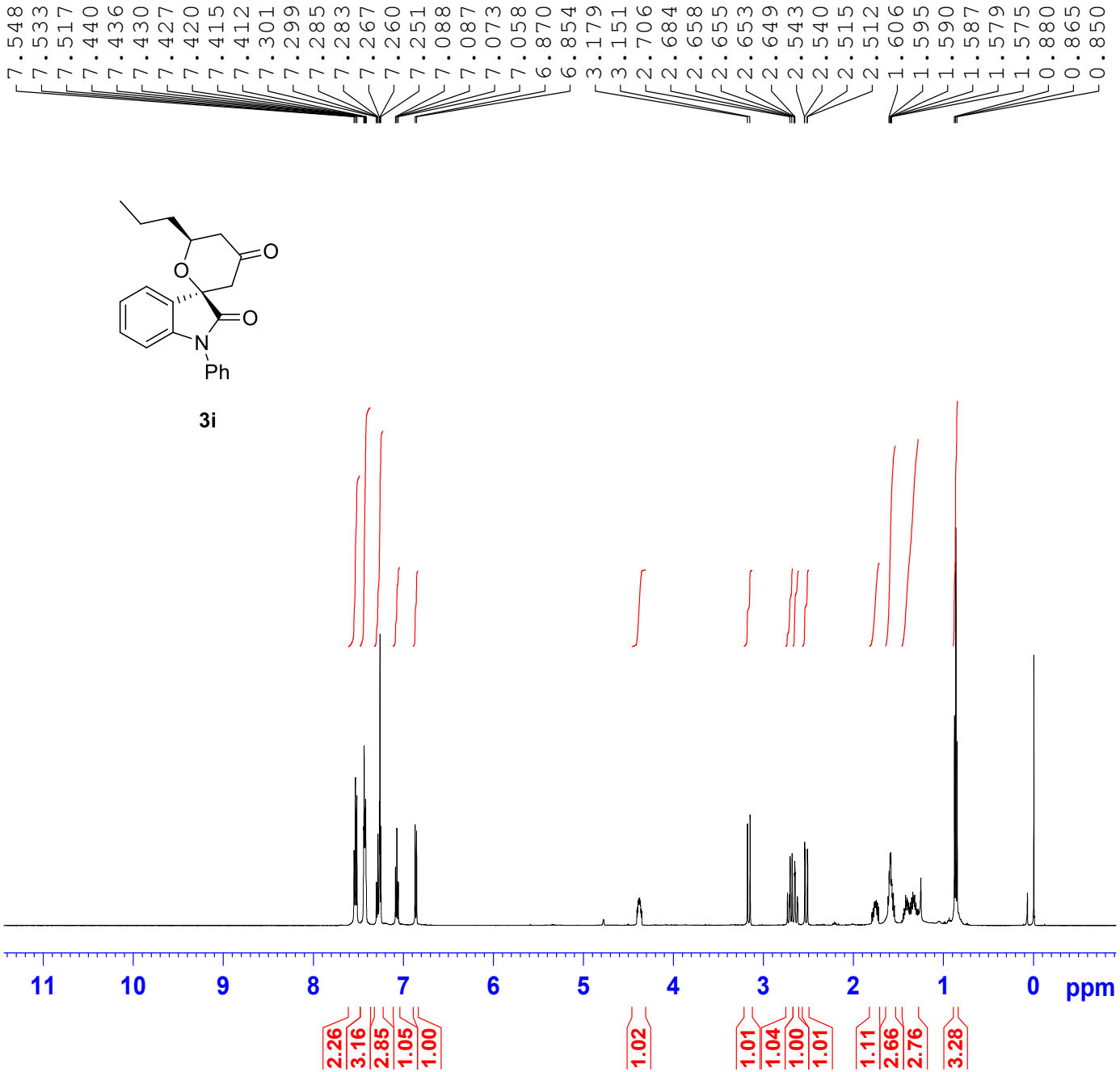
2i




```
Current Data Parameters
NAME      MH-93-Major-Clean spectra-C13
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20190205
Time              16.14 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  321
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ             1.0878977 sec
RG                  101
DW               16.600 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1         125.7703643 MHz
NUC1                13C
P0                 3.33 usec
P1                10.00 usec
PLW1         88.26000214 W
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             80.00 usec
PLW2         23.68499947 W
PLW12         0.23014790 W
PLW13         0.11535020 W

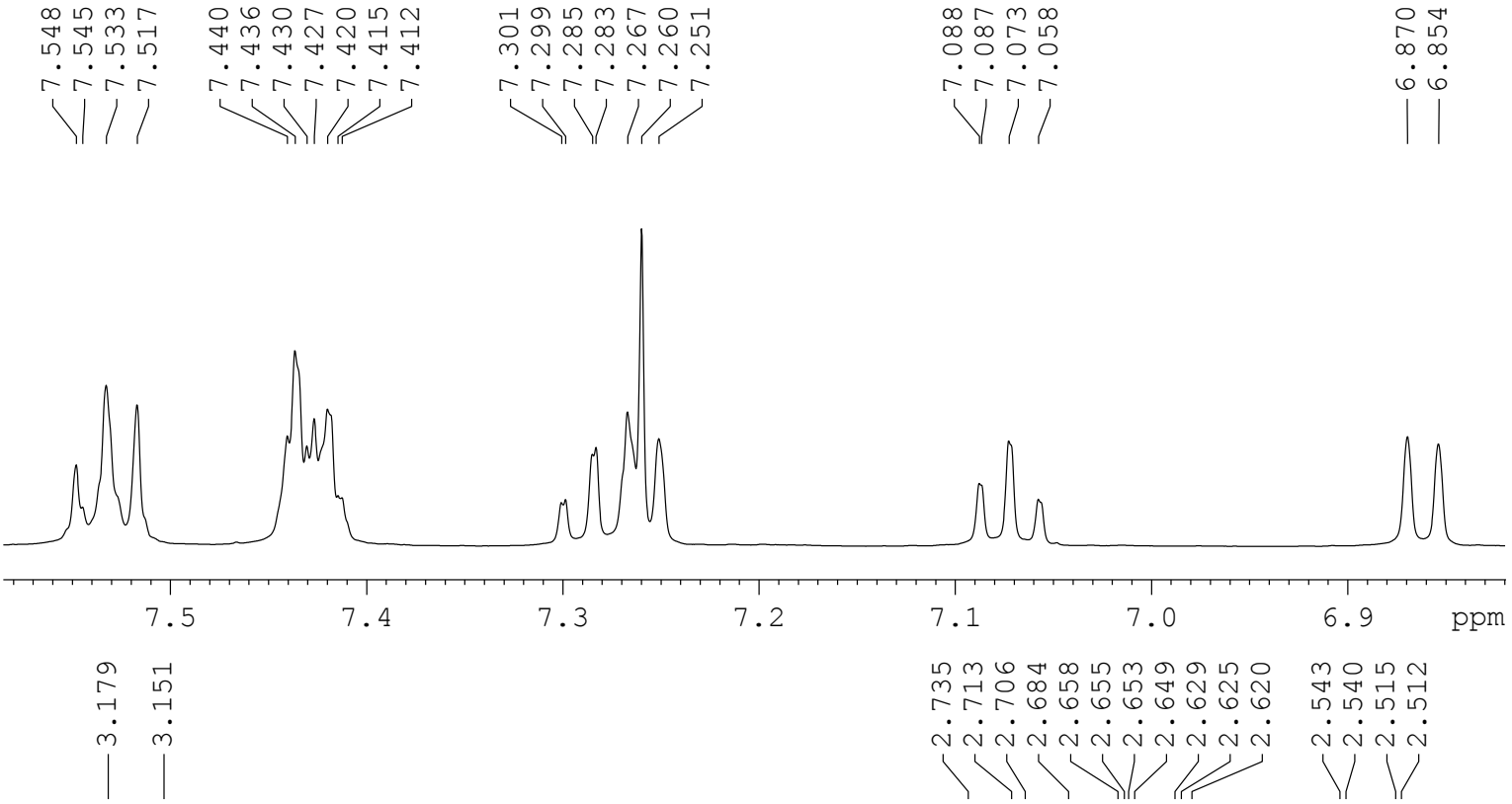
F2 - Processing parameters
SI                 32768
SF           125.7578002 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

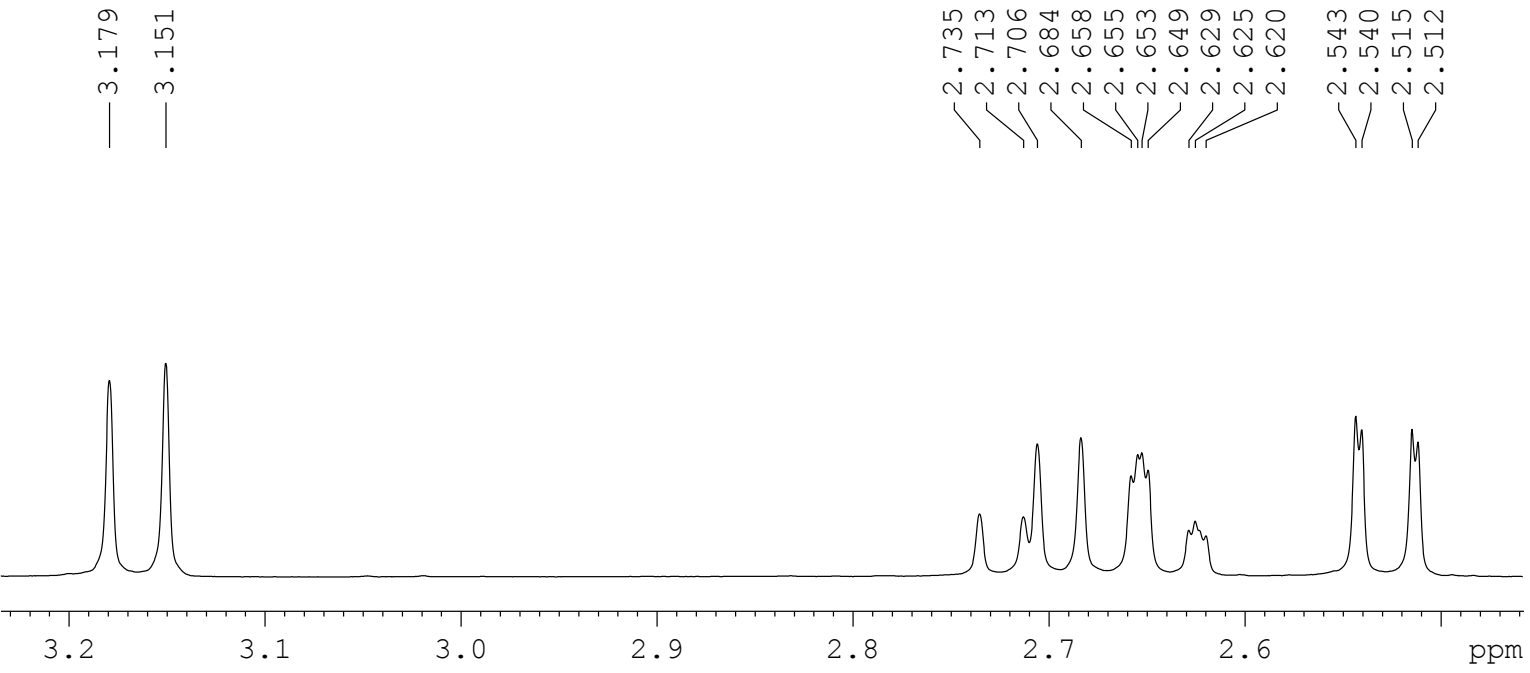



```
Current Data Parameters
NAME      MH-93-MINOR-Clean spectra
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190206
Time              12.56 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300122 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

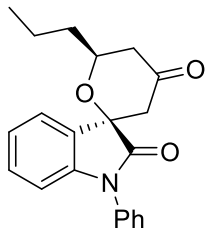




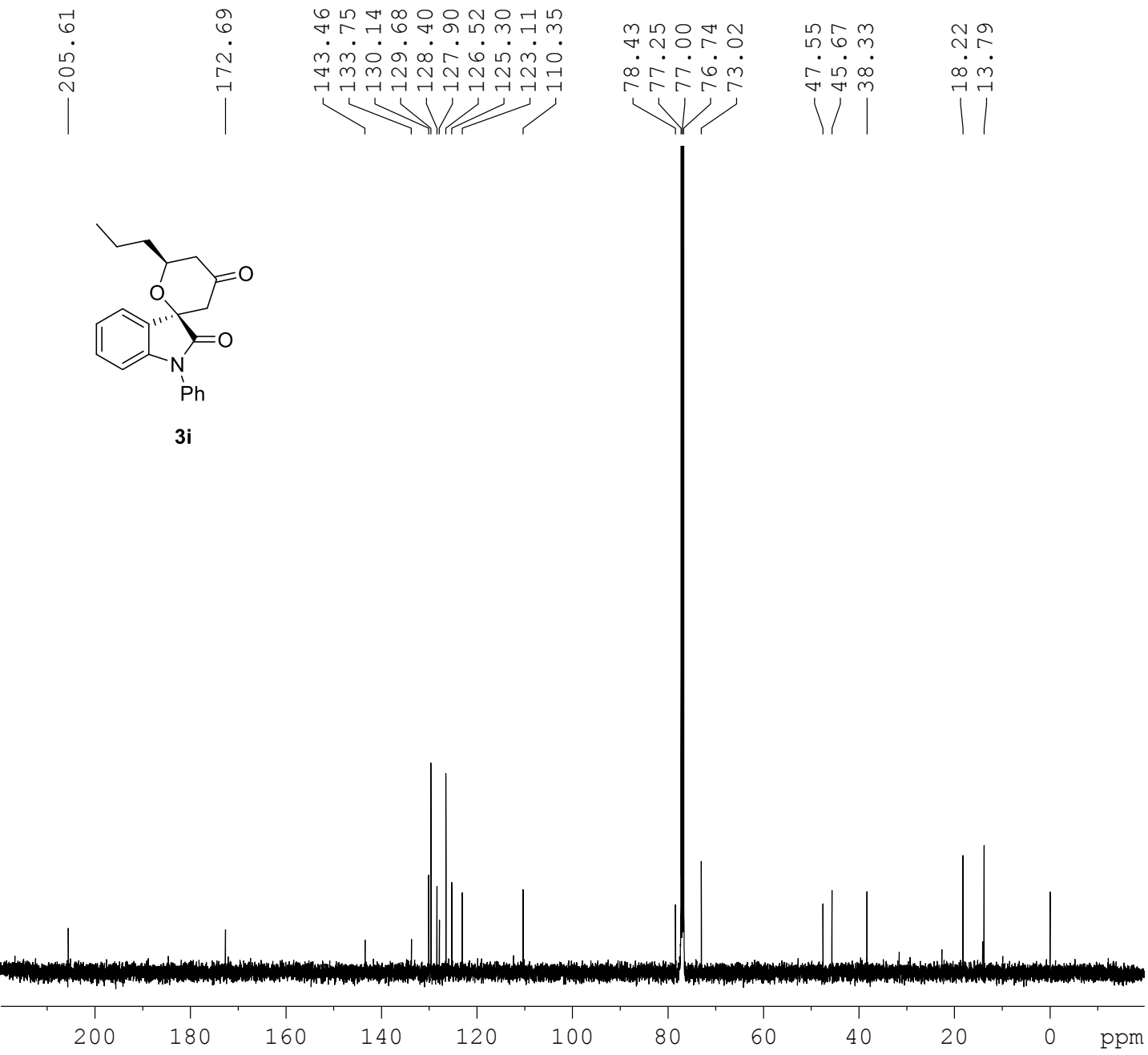

```
Current Data Parameters
NAME      MH-93-MINOR-Clean spectra
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20190206
Time              12.56 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300122 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



3i

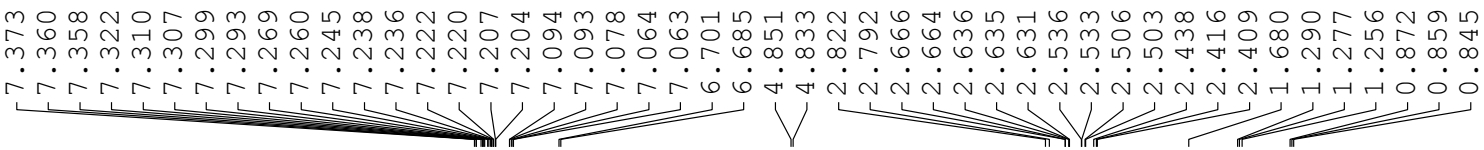


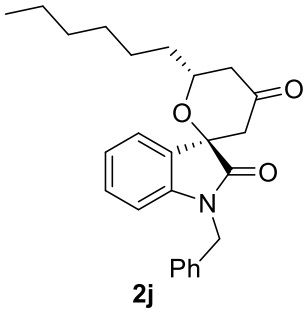
3i

```
Current Data Parameters
NAME      MH-93-Minor-C13
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190207
Time              15.23 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ             1.0878977 sec
RG                  101
DW                16.600 usec
DE                 6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1                13C
P0                 3.33 usec
P1                10.00 usec
PLW1        88.26000214 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             80.00 usec
PLW2        23.68499947 W
PLW12        0.23014790 W
PLW13        0.11535020 W

F2 - Processing parameters
SI                32768
SF          125.7577915 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



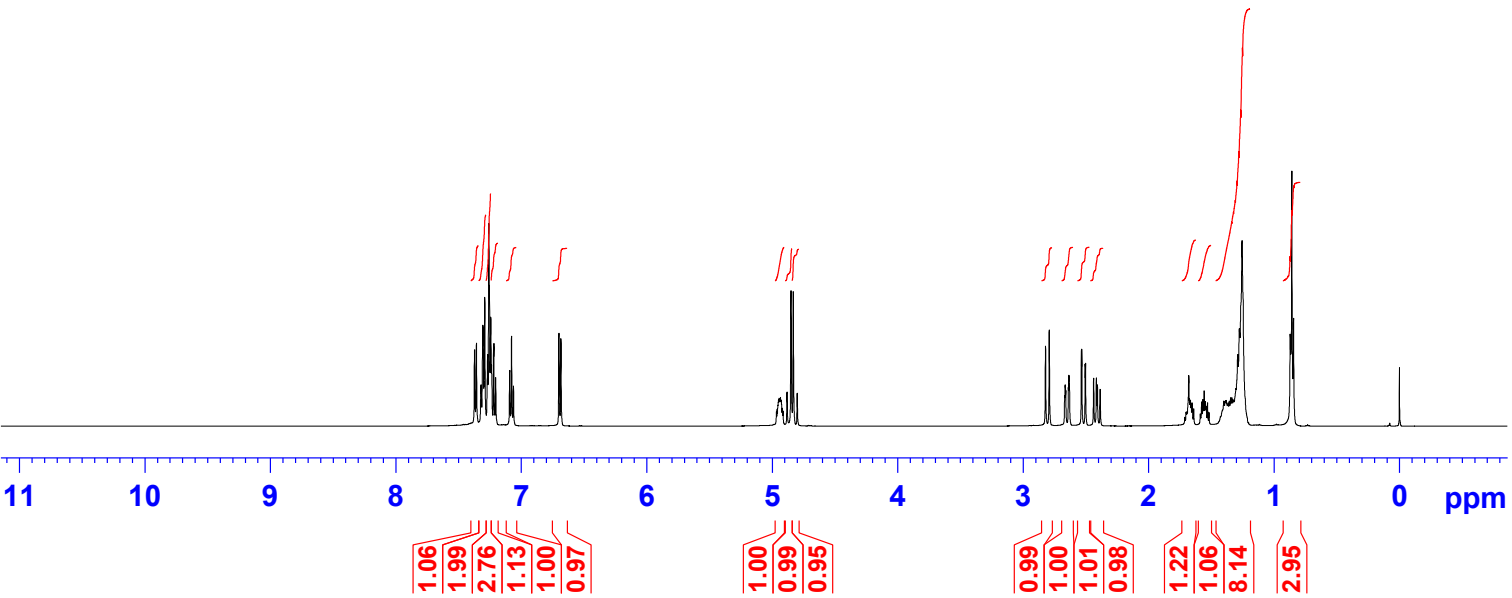


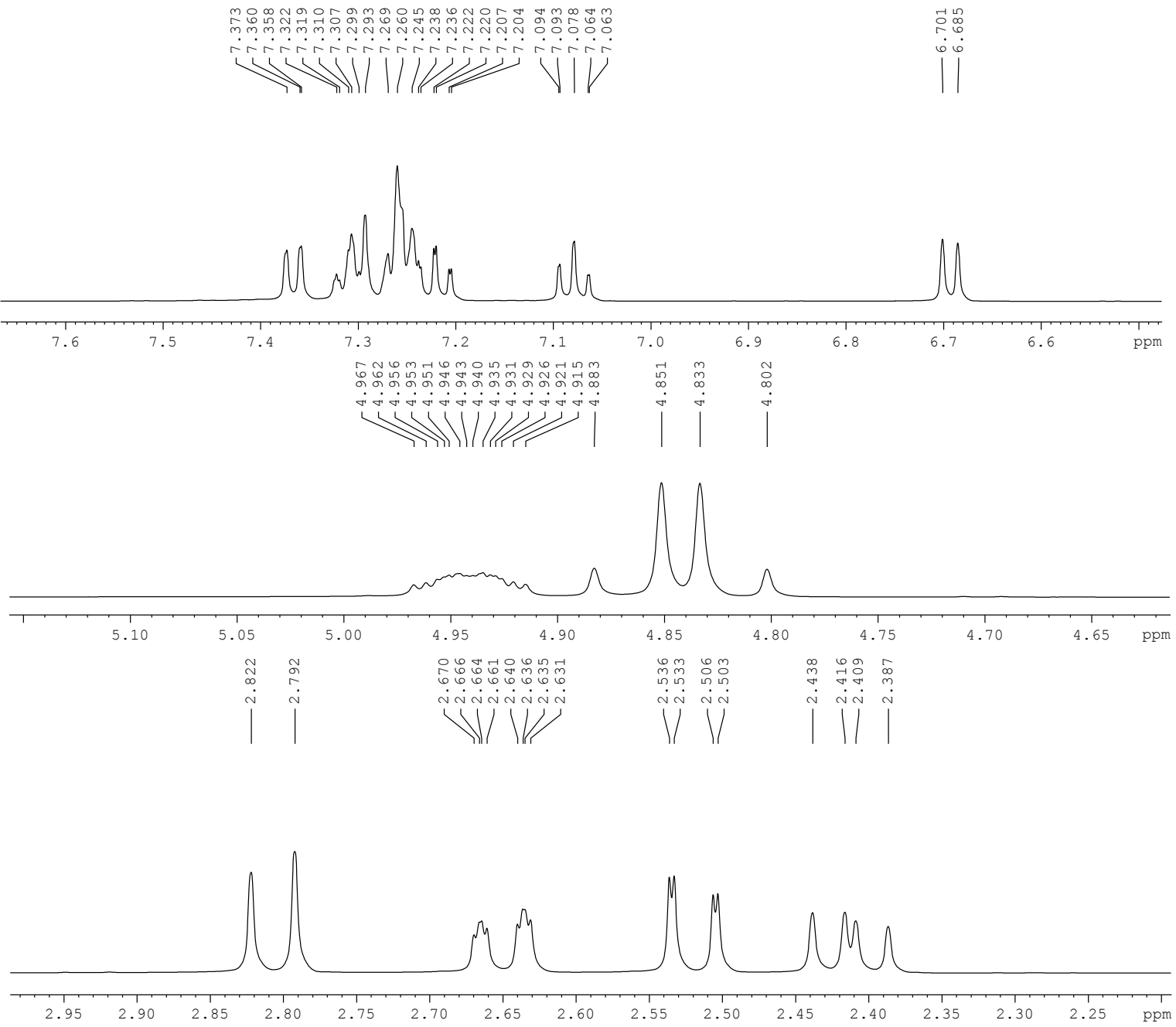
2j

```
Current Data Parameters
NAME      MH-94-Major-Clean spectra
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20190211
Time              11.34 h
INSTRUM           Avance
PROBHD    Z151574_0027 (
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                   32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                   1
SFO1          500.1330883 MHz
NUC1                 1H
P0                2.67 usec
P1                8.00 usec
PLW1          23.68499947 W

F2 - Processing parameters
SI                65536
SF           500.1300154 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

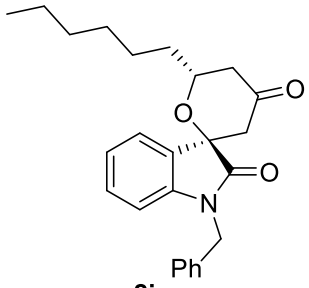





```
Current Data Parameters
NAME      MH-94-Major-Clean spectra
EXPNO                   1
PROCNO                  1

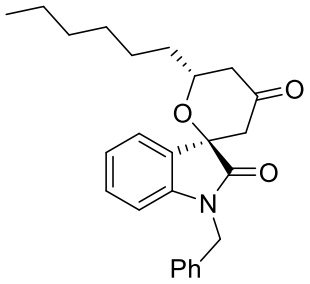
F2 - Acquisition Parameters
Date_            20190211
Time               11.34 h
INSTRUM            Avance
PROBHD   Z151574_0027 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH            10000.000 Hz
FIDRES          0.305176 Hz
AQ              3.2767999 sec
RG                     32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                     1
SFO1          500.1330883 MHz
NUC1                   1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          23.68499947 W

F2 - Processing parameters
SI                  65536
SF            500.1300154 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



2j

204.43
174.68
142.24
135.21
130.20
128.81
127.71
127.10
123.96
123.35
109.58
77.70
77.25
77.00
76.74
71.79
46.86
45.57
43.32
36.40
31.65
29.05
24.82
22.49
14.01

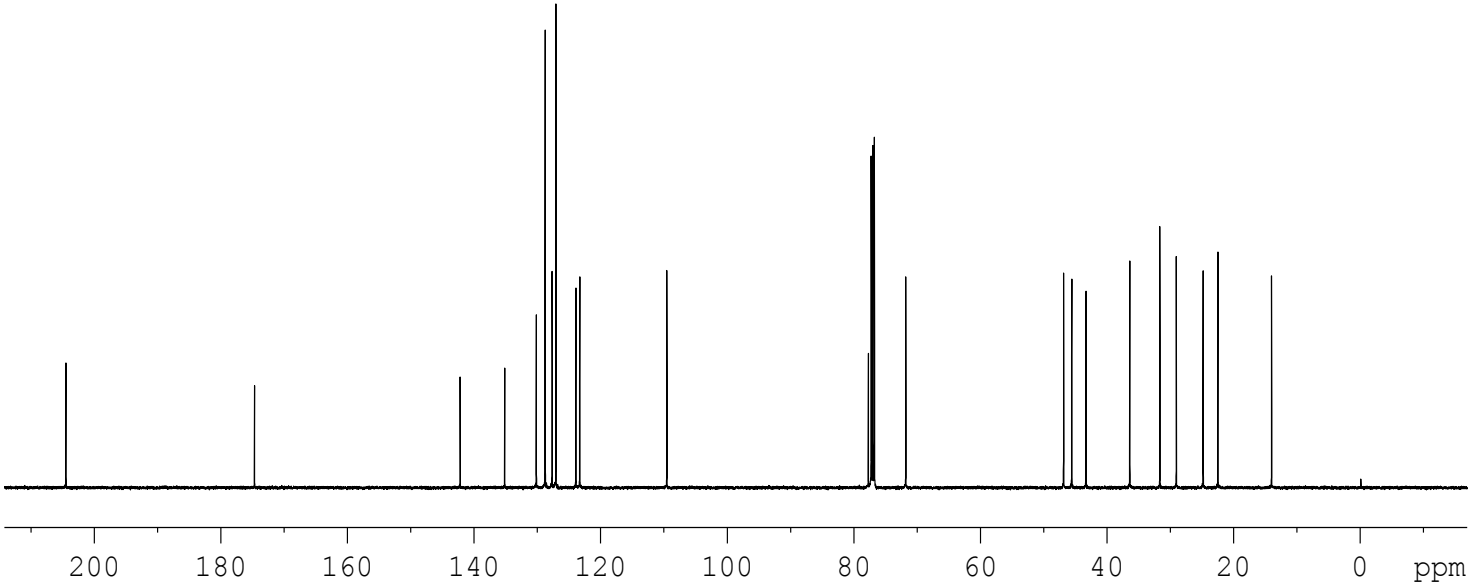


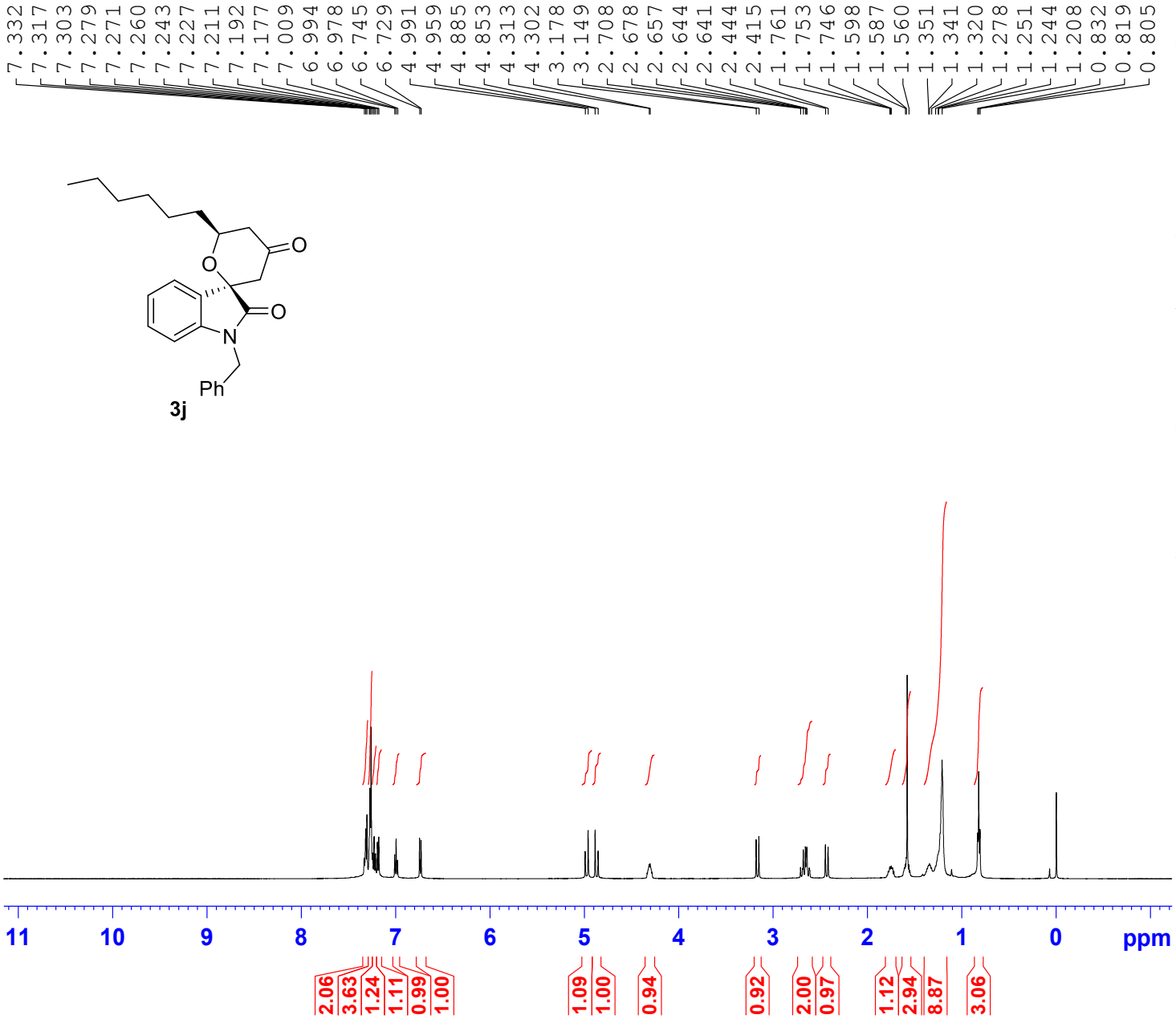
2j

```
Current Data Parameters
NAME      MH-94-Major-Clean spectra-C13
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20190211
Time              12.21 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  866
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ             1.0878977 sec
RG                  101
DW               16.600 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1                13C
P0                3.33 usec
P1               10.00 usec
PLW1         88.26000214 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2            80.00 usec
PLW2         23.68499947 W
PLW12         0.23014790 W
PLW13         0.11535020 W

F2 - Processing parameters
SI                 32768
SF          125.7577986 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

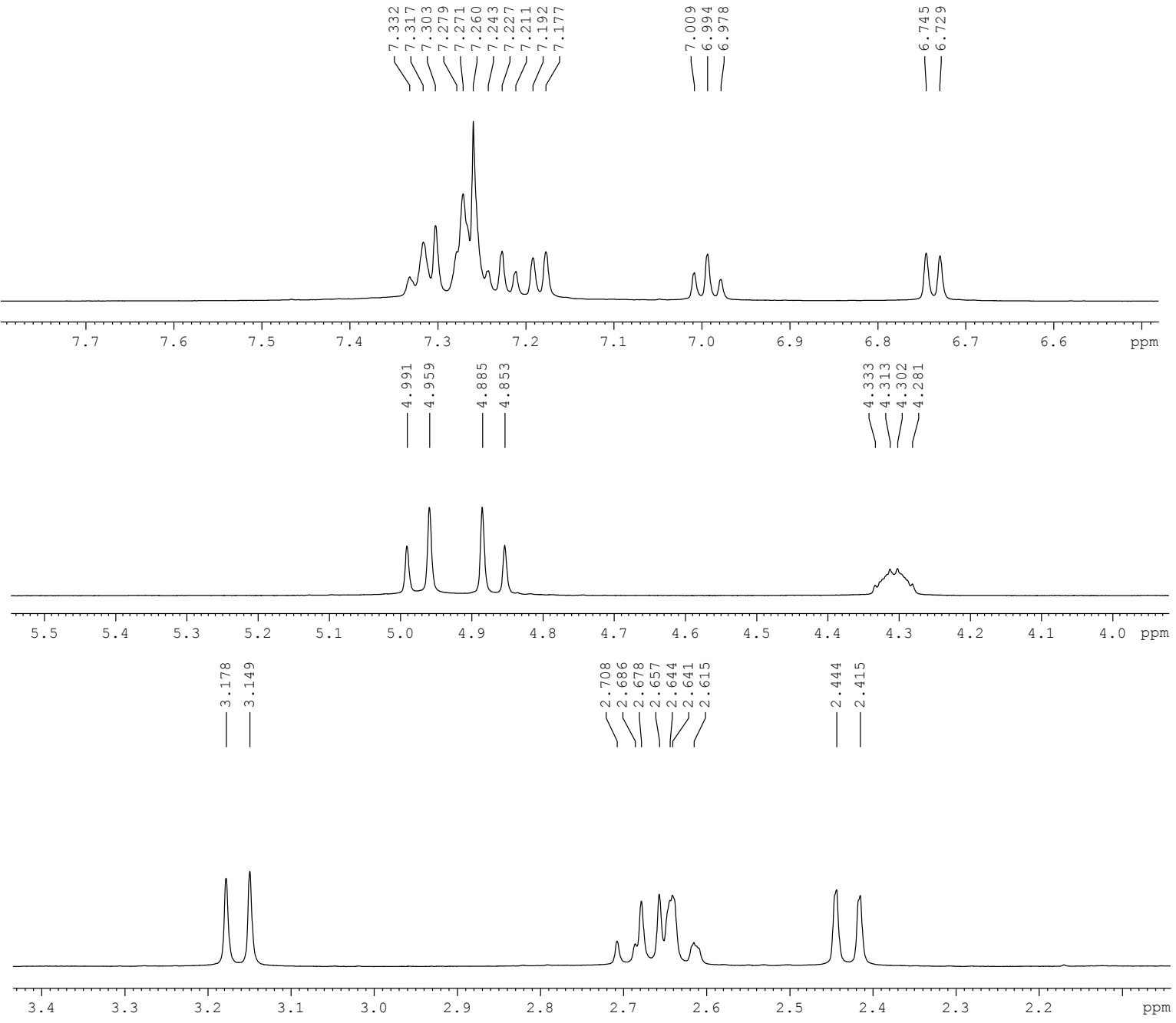





```
Current Data Parameters
NAME      MH-94-Minor-Clean spectra
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190208
Time              13.08 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          23.68499947 W

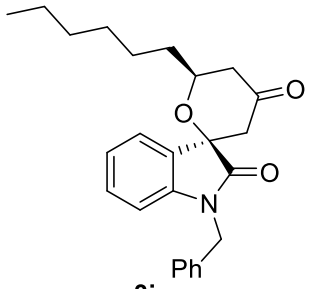
F2 - Processing parameters
SI                 65536
SF           500.1300126 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



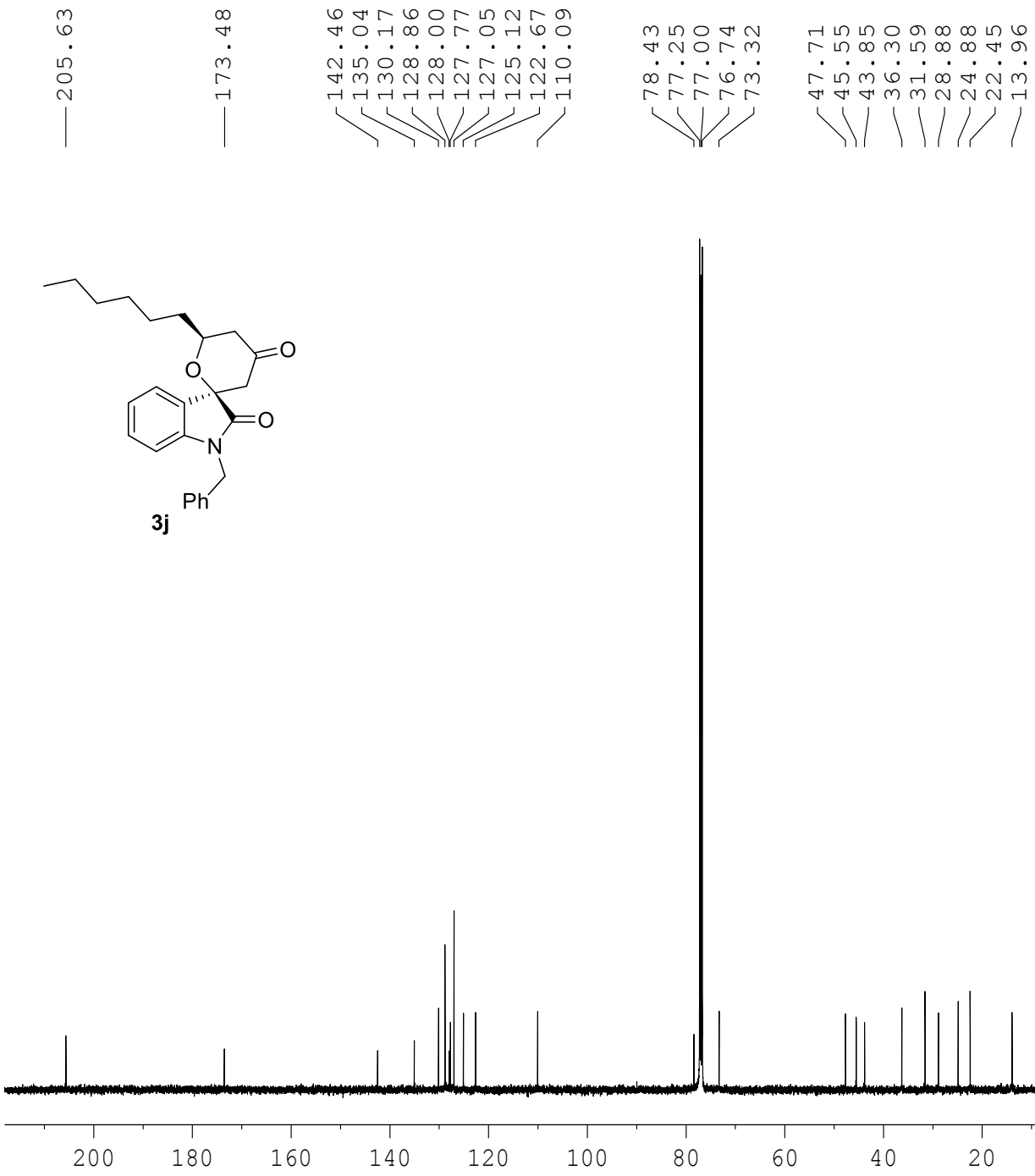

```
Current Data Parameters
NAME      MH-94-Minor-Clean spectra
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190208
Time              13.08 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT             CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300126 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



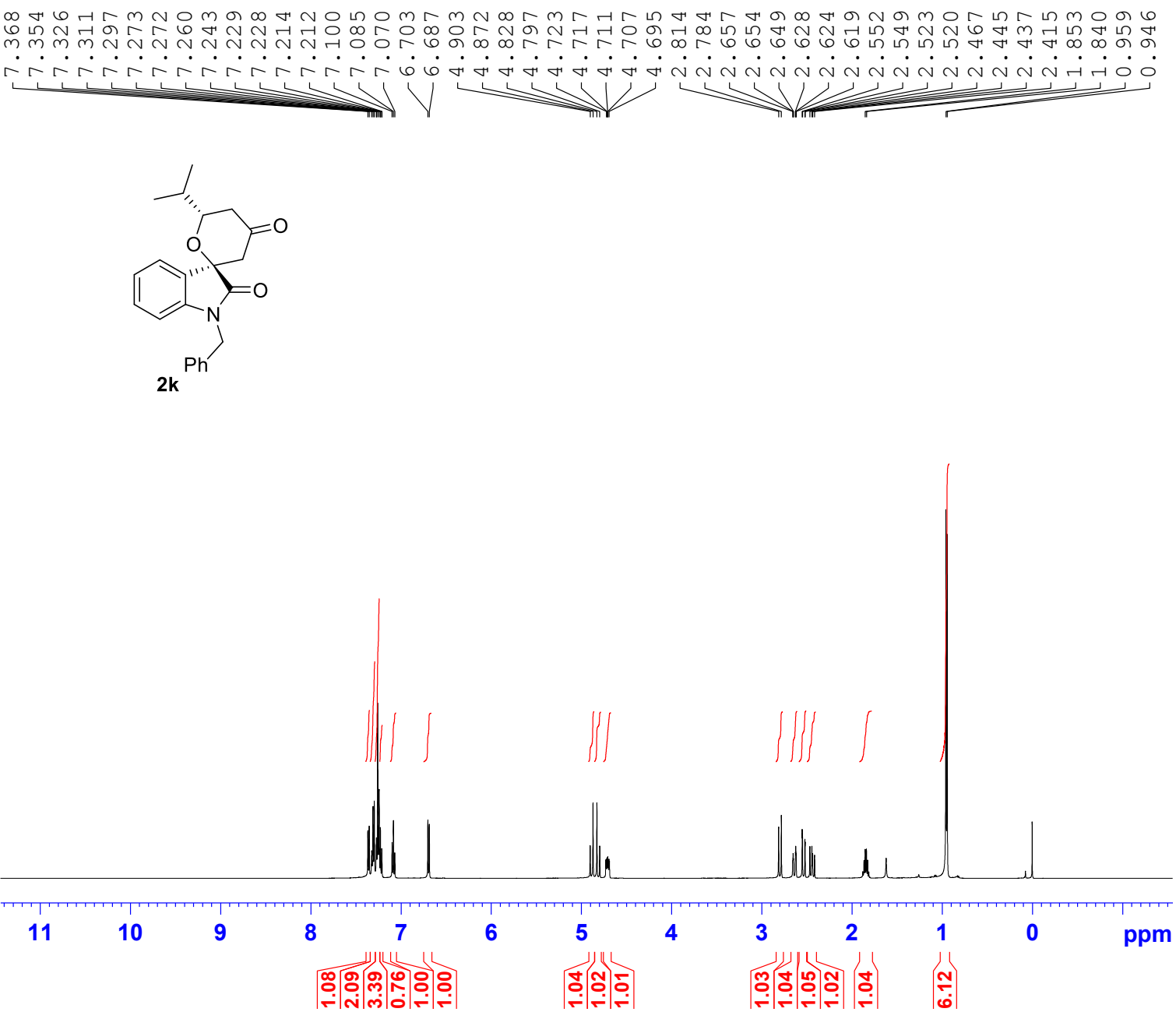
3j




```
Current Data Parameters
NAME      MH-94-Minor-Clean spectra-C13
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20190208
Time              14.10 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ            1.0878977 sec
RG                  101
DW               16.600 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1                13C
P0                3.33 usec
P1               10.00 usec
PLW1        88.26000214 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             80.00 usec
PLW2        23.68499947 W
PLW12        0.23014790 W
PLW13        0.11535020 W

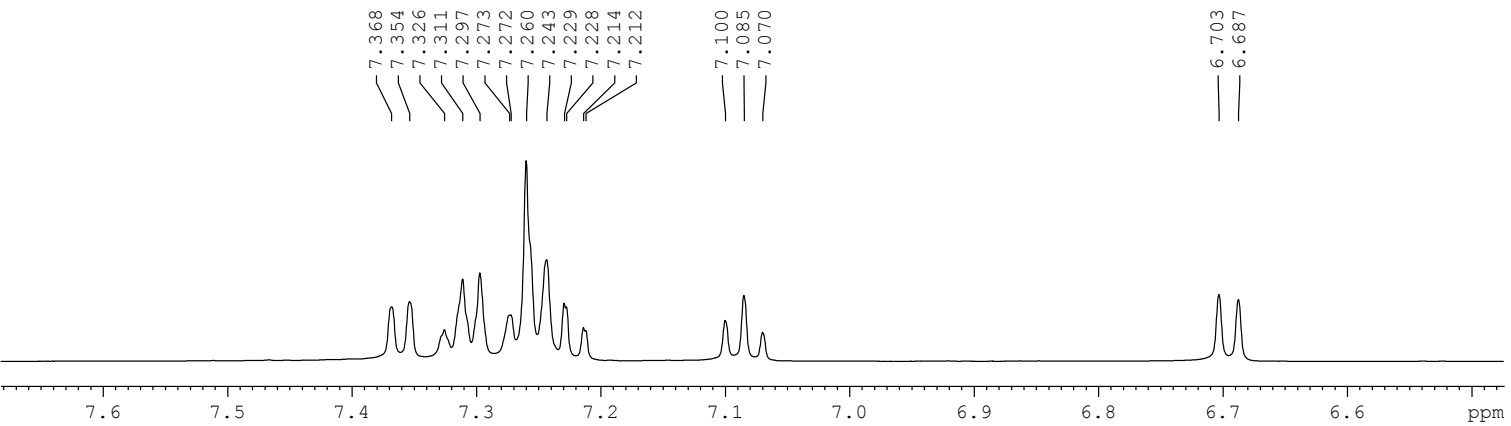
F2 - Processing parameters
SI                 32768
SF          125.7577924 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

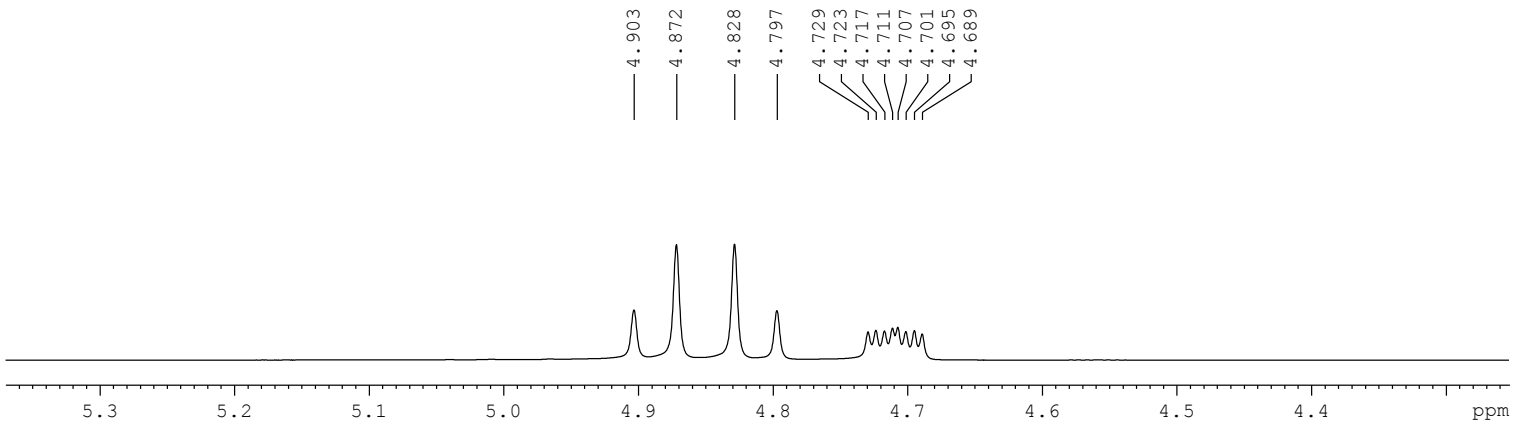


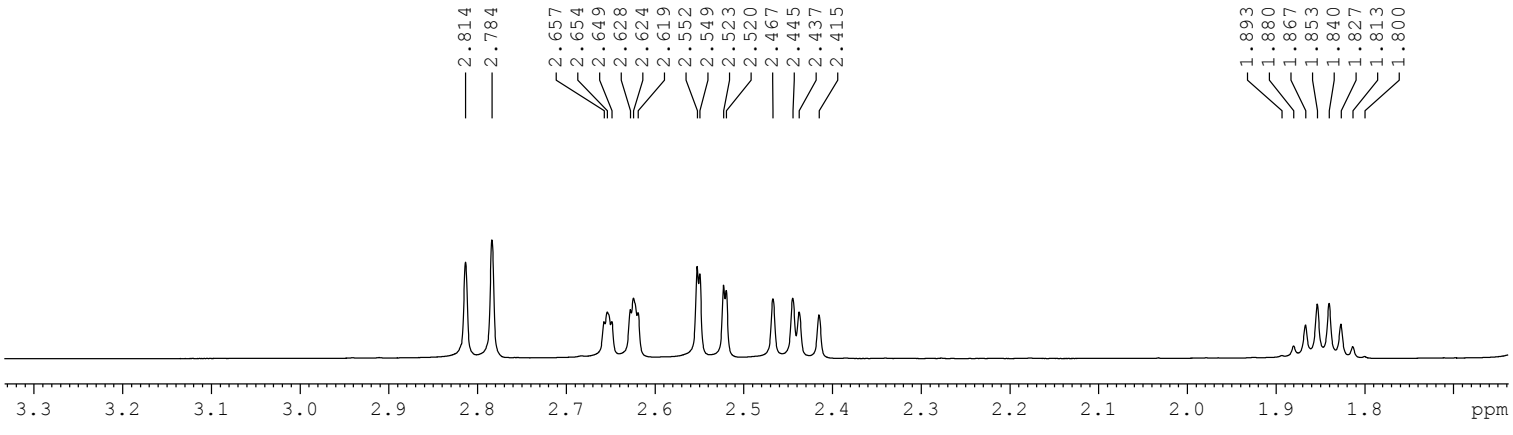

```
Current Data Parameters
NAME      MH-98-Major-Clean spectra
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20190212
Time              13.26 h
INSTRUM           Avance
PROBHD    Z151574_0027 (
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES            0.305176 Hz
AQ             3.2767999 sec
RG                   32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                   1
SFO1          500.1330883 MHz
NUC1                 1H
P0                2.67 usec
P1                8.00 usec
PLW1          23.68499947 W

F2 - Processing parameters
SI                65536
SF           500.1300123 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



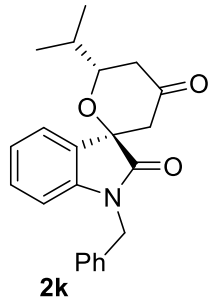





```
Current Data Parameters
NAME      MH-98-Major-Clean spectra
EXPNO                  1
PROCNO                 1

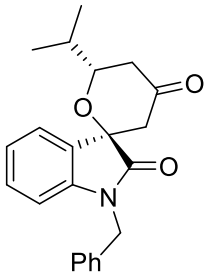
F2 - Acquisition Parameters
Date_           20190212
Time              13.26 h
INSTRUM           Avance
PROBHD    Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300123 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



2k

204.96
174.76
142.24
135.22
130.19
128.96
128.84
127.72
127.08
123.94
123.34
109.59
77.59
77.25
77.00
76.74
76.14
45.58
43.67
43.30
33.06
18.06
17.32

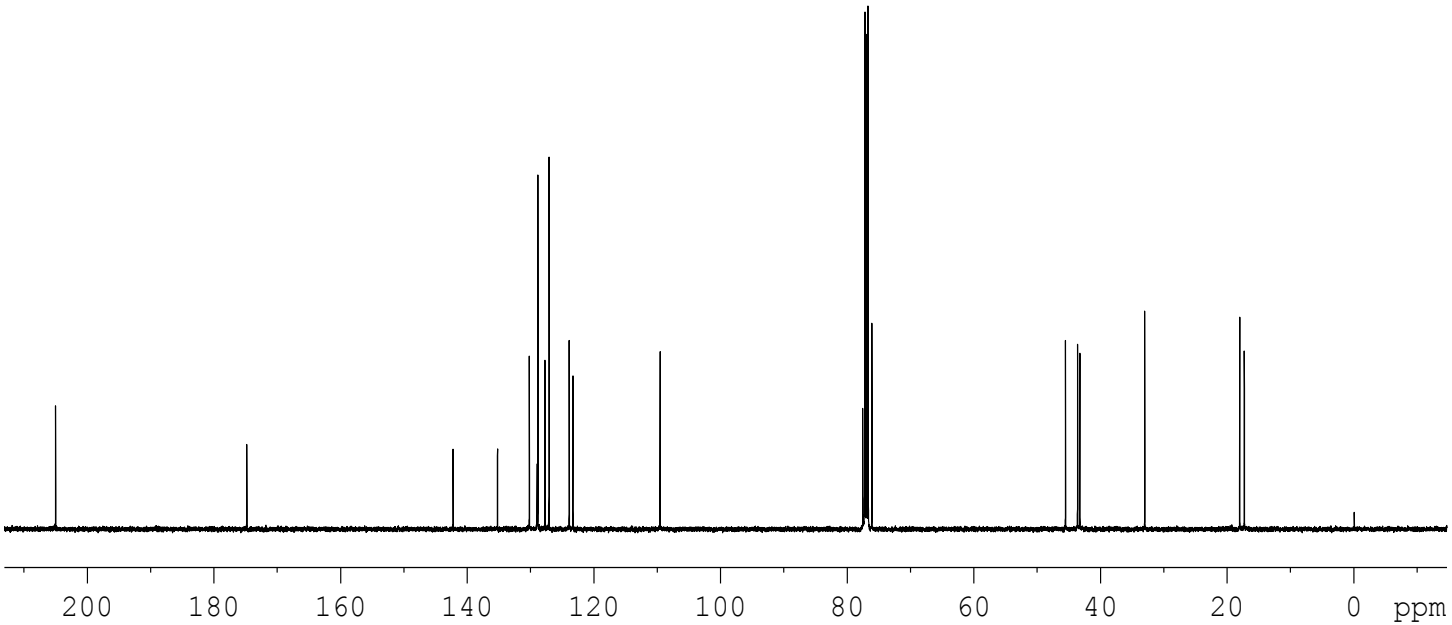


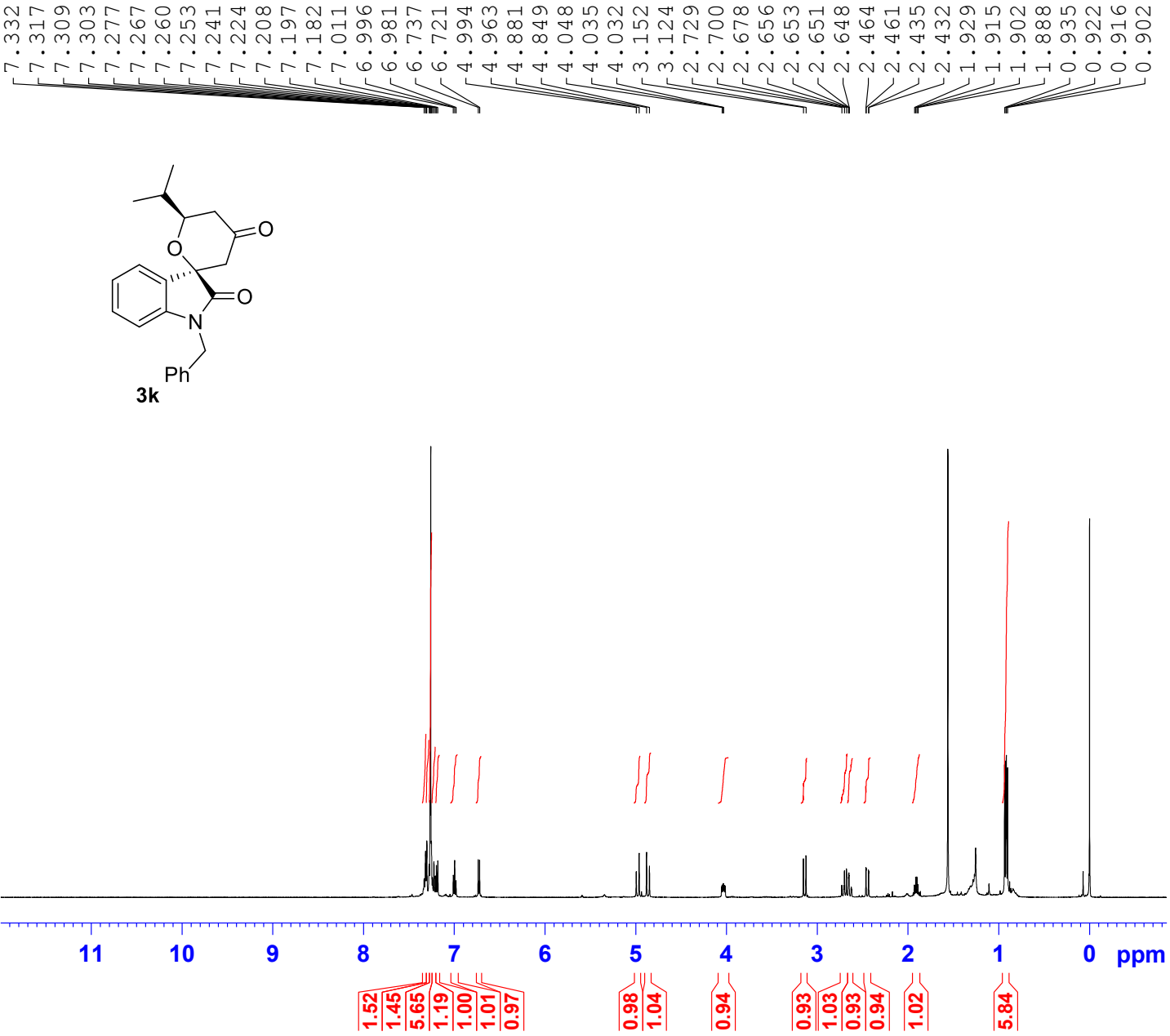
2k

```
Current Data Parameters
NAME      MH-98-Major-Clean spectra-C13
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20190212
Time              14.05 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  618
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ             1.0878977 sec
RG                  101
DW               16.600 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1                13C
P0                 3.33 usec
P1                10.00 usec
PLW1        88.26000214 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             80.00 usec
PLW2        23.68499947 W
PLW12        0.23014790 W
PLW13        0.11535020 W

F2 - Processing parameters
SI                32768
SF          125.7577952 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

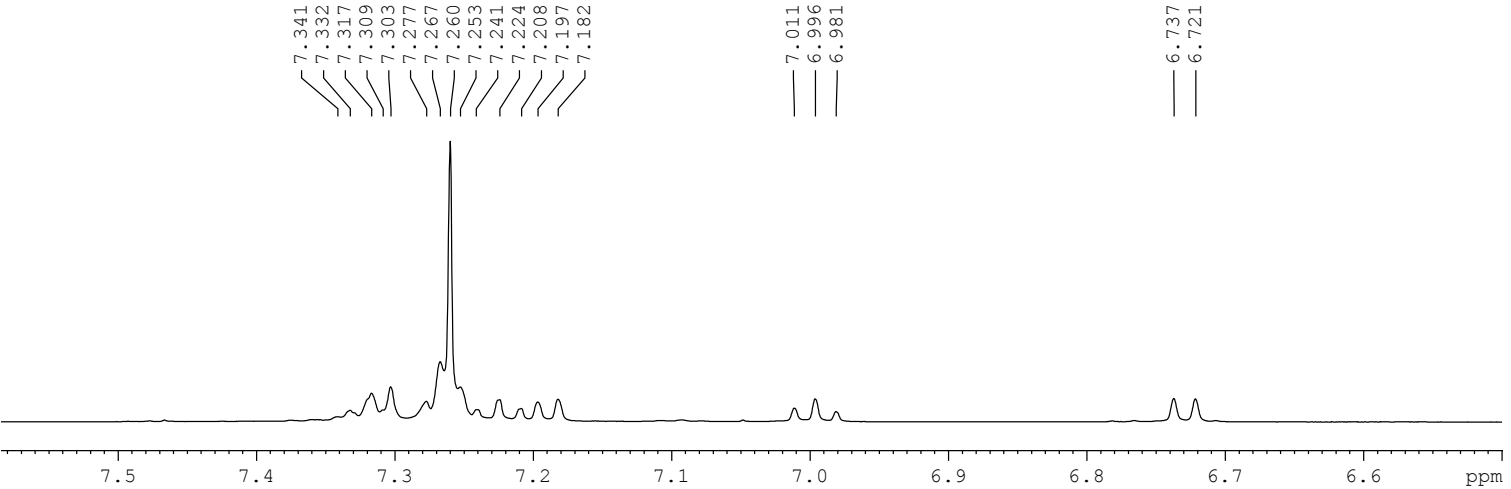


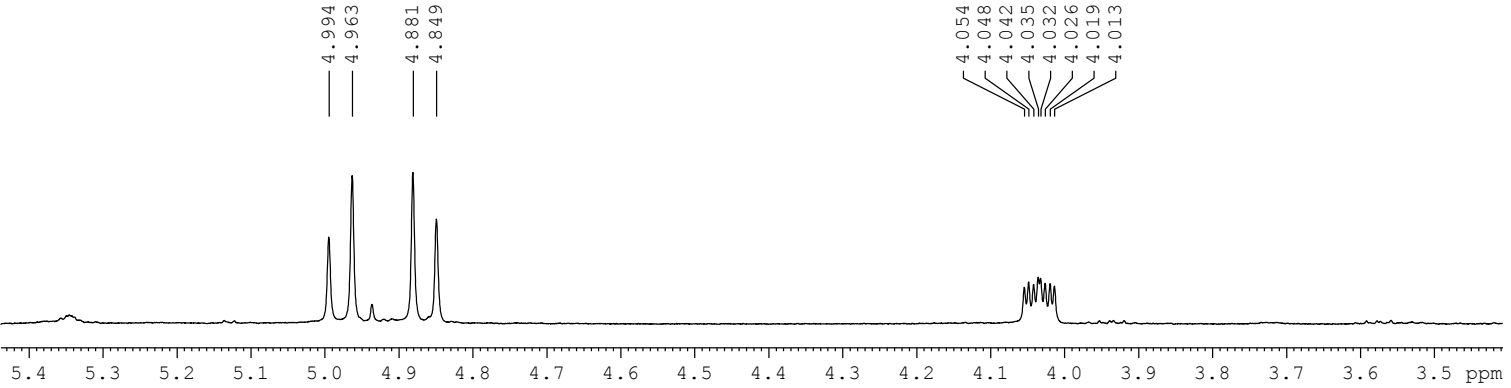


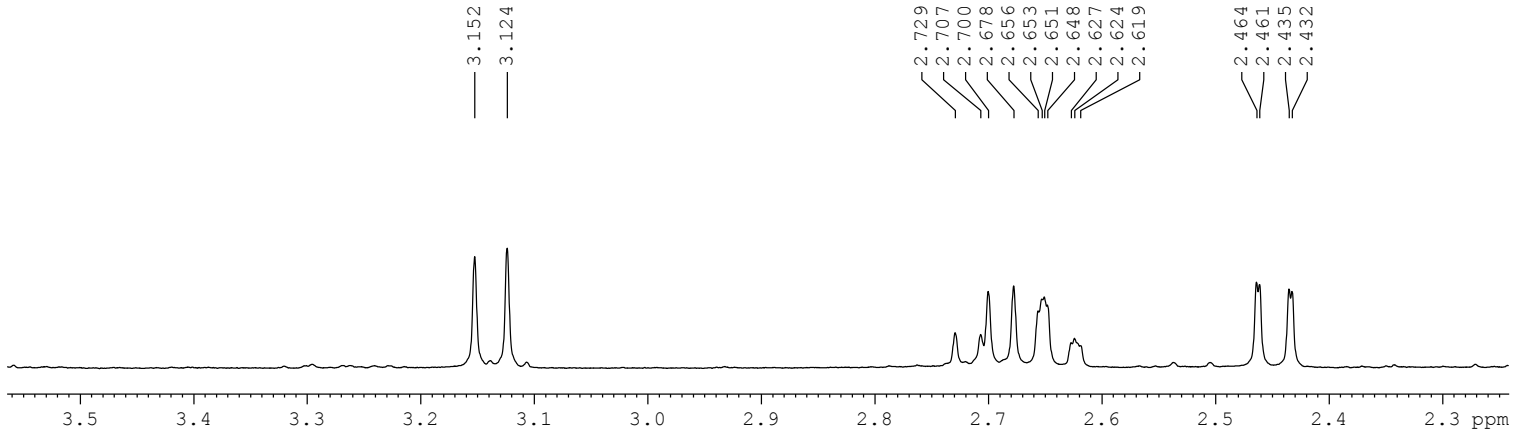

```
Current Data Parameters
NAME      MH-98-Minor -Clean spectra again
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20190213
Time              14.45 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES        0.305176 Hz
AQ            3.2767999 sec
RG                    32
DW               50.000 usec
DE               11.14 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1
SFO1        500.1330883 MHz
NUC1                1H
P0                2.67 usec
P1                8.00 usec
PLW1        23.68499947 W

F2 - Processing parameters
SI                 65536
SF          500.1300125 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



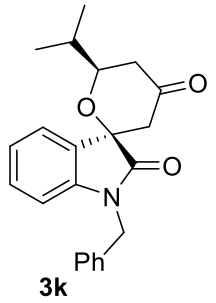




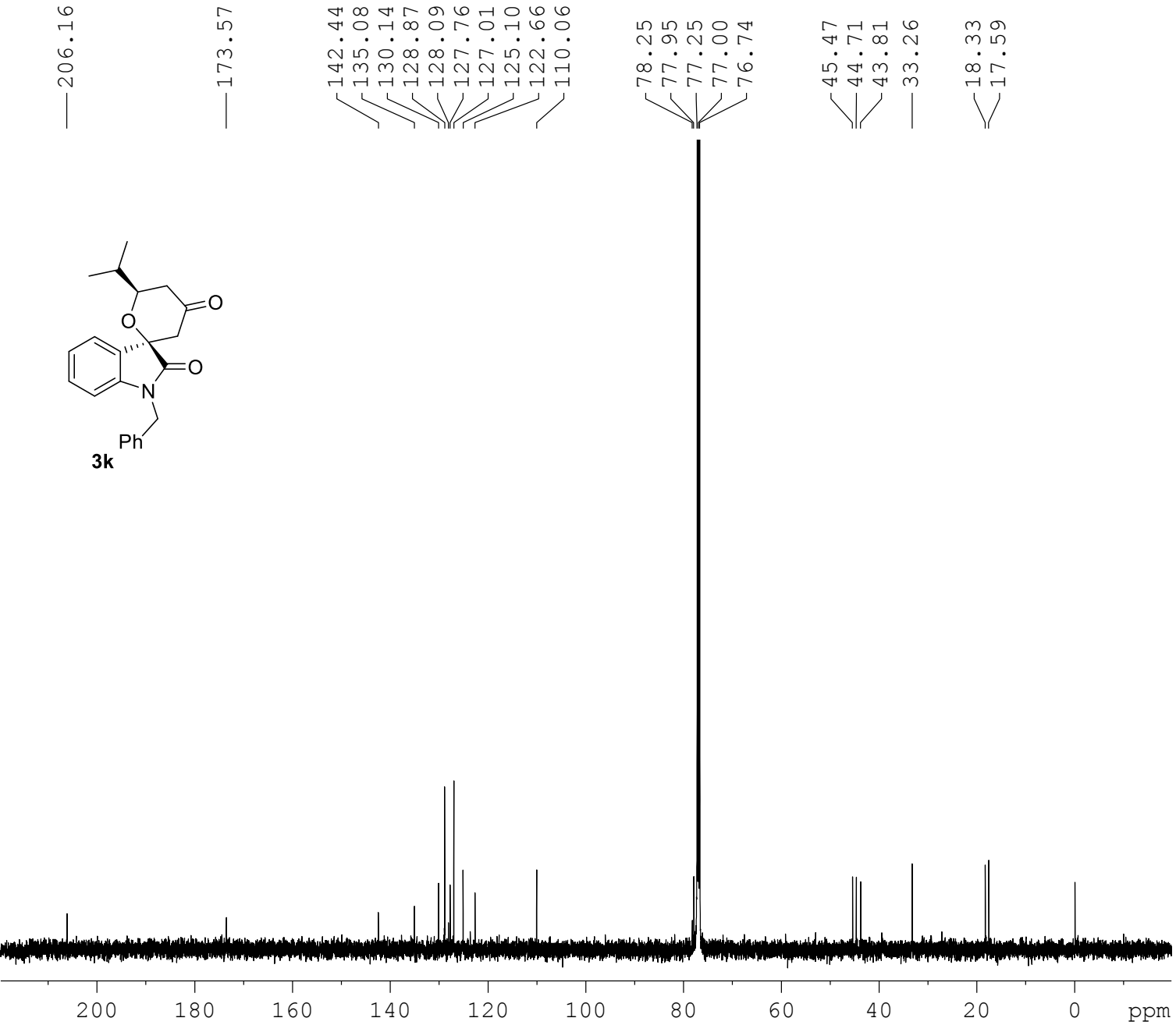

```
Current Data Parameters
NAME      MH-98-Minor -Clean spectra again
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20190213
Time              14.45 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1        23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300125 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



3k




```
Current Data Parameters
NAME      MH-98-Minor -Clean spectra again-C13
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_            20190213
Time               15.46 h
INSTRUM            Avance
PROBHD   Z151574_0027 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           30120.482 Hz
FIDRES         0.919204 Hz
AQ             1.0878977 sec
RG                  101
DW                16.600 usec
DE                 6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        125.7703643 MHz
NUC1               13C
P0                 3.33 usec
P1                10.00 usec
PLW1        88.26000214 W
SFO2        500.1320005 MHz
NUC2               1H
CPDPRG[2         waltz65
PCPD2             80.00 usec
PLW2        23.68499947 W
PLW12        0.23014790 W
PLW13        0.11535020 W

F2 - Processing parameters
SI                32768
SF          125.7577918 MHz
WDW                 EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
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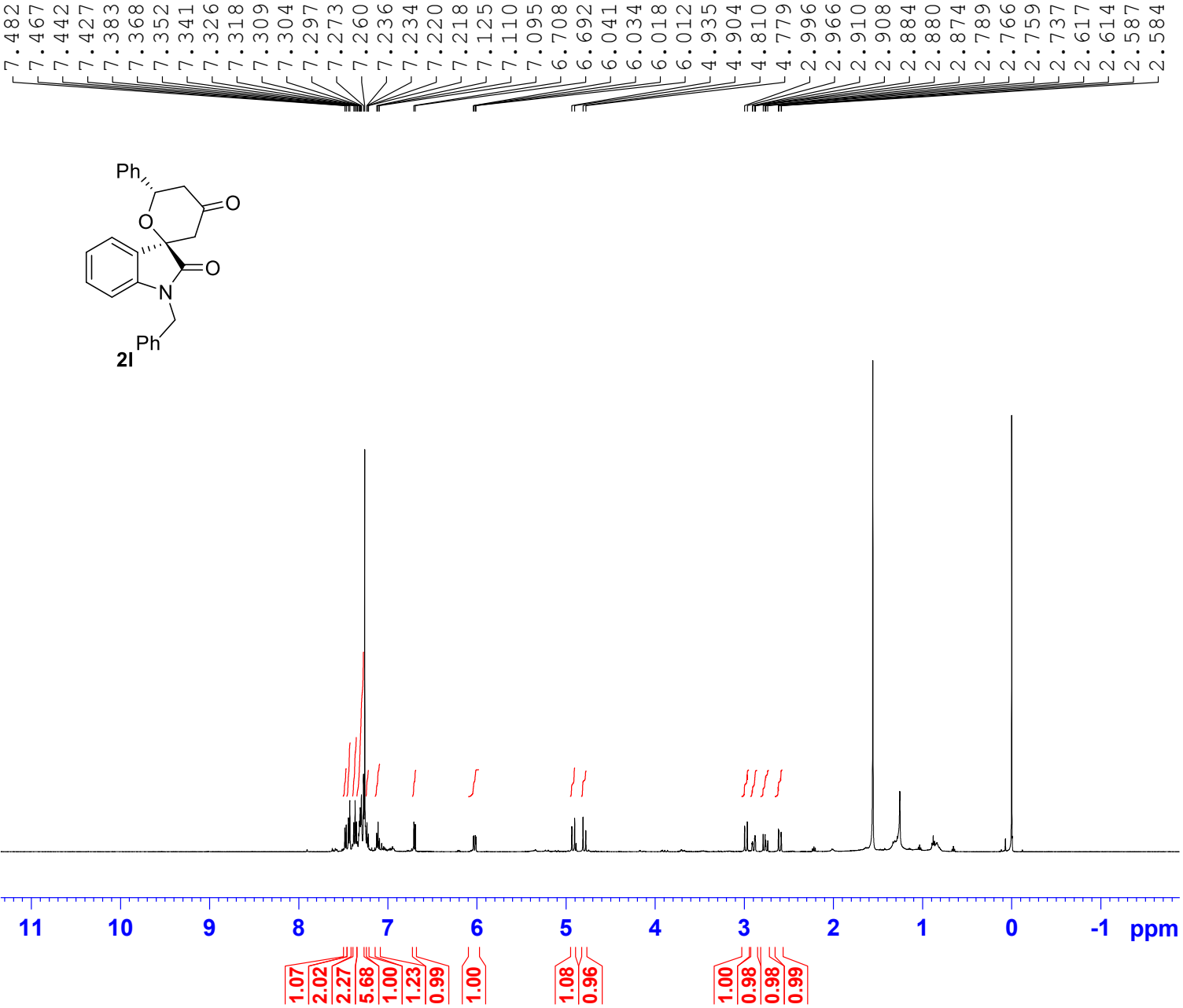
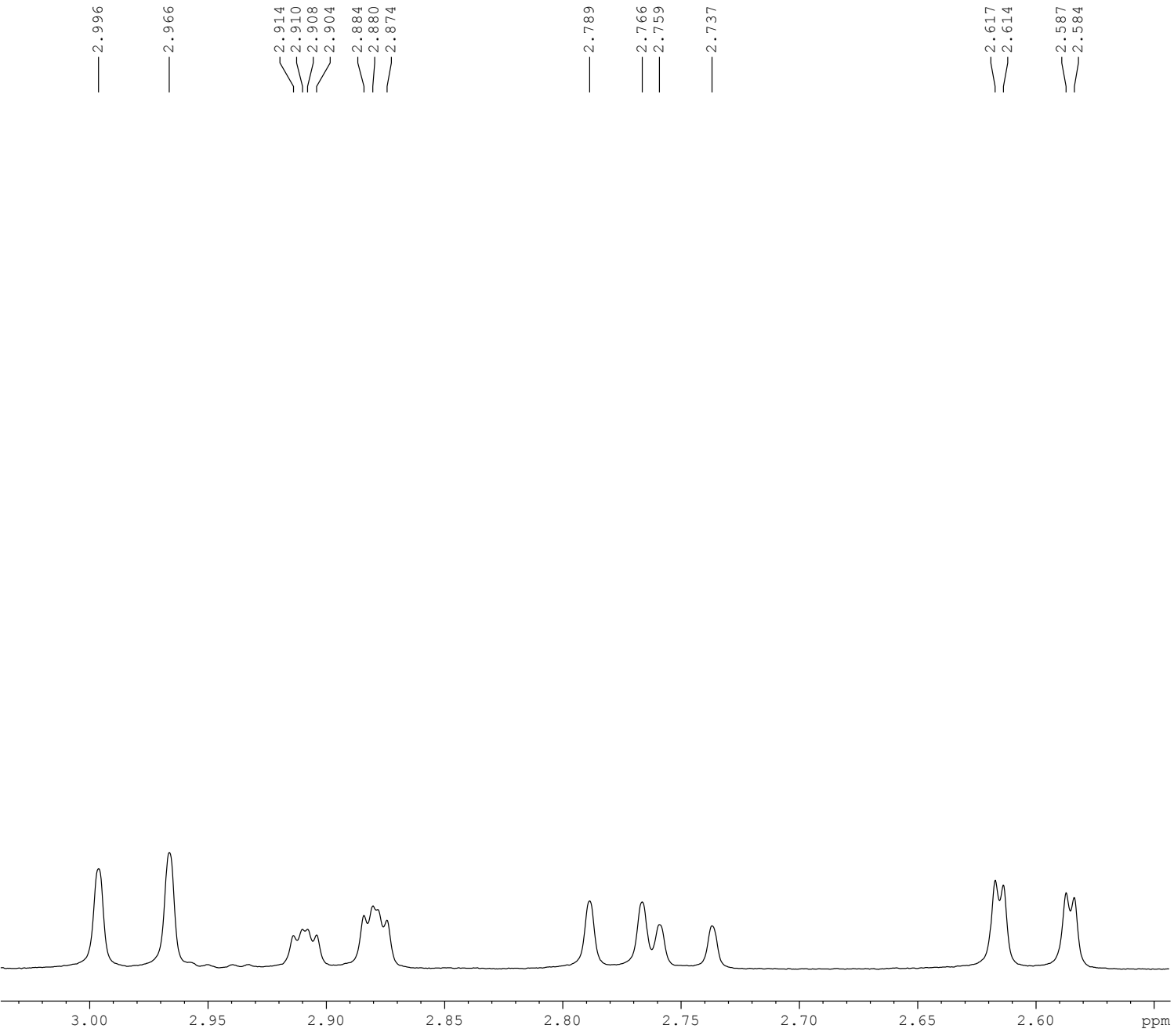



```
Current Data Parameters
NAME      MH-97-Major-Clean spectra
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20190215
Time                13.39 h
INSTRUM           Avance
PROBHD    Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10000.000 Hz
FIDRES          0.305176 Hz
AQ              3.2767999 sec
RG                    32
DW                50.000 usec
DE                11.14 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          23.68499947 W

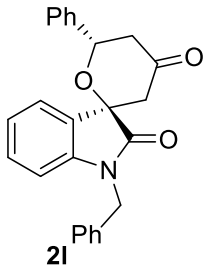
F2 - Processing parameters
SI                 65536
SF            500.1300123 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



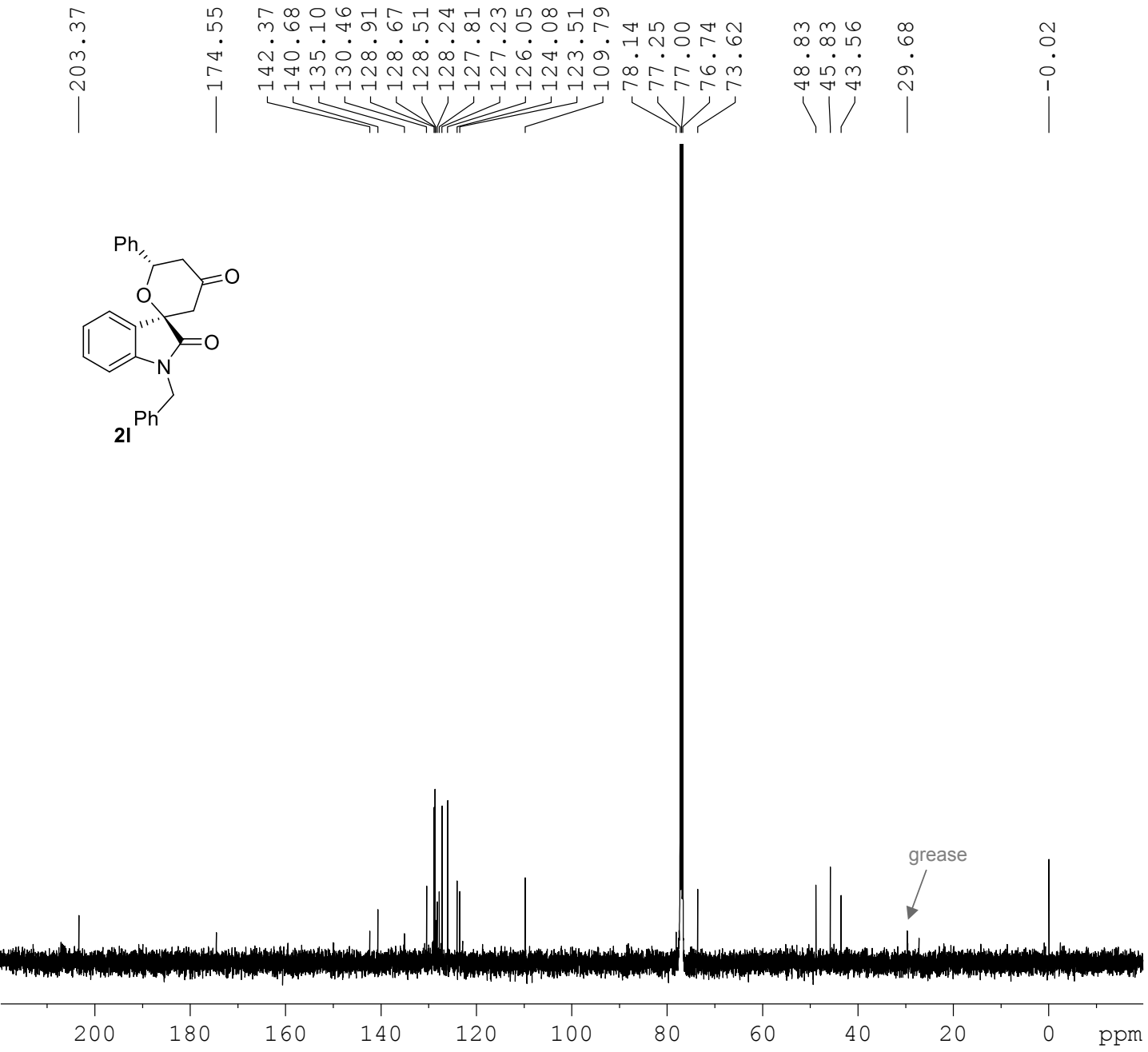

```
Current Data Parameters
NAME      MH-97-Major-Clean spectra
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190215
Time              13.39 h
INSTRUM           Avance
PROBHD   Z151574_0027 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                    32
DW               50.000 usec
DE               11.14 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1
SFO1         500.1330883 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1         23.68499947 W

F2 - Processing parameters
SI                 65536
SF           500.1300123 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



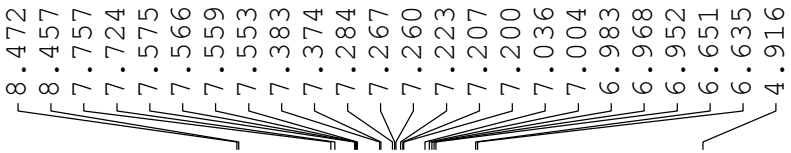
2l

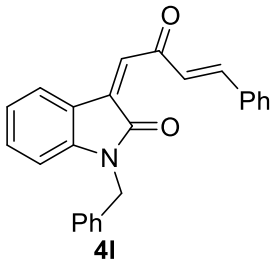



```
Current Data Parameters
NAME      MH-97-Major-C13AGAIN
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20190216
Time                11.43 h
INSTRUM            Avance
PROBHD    Z151574_0027 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH             30120.482 Hz
FIDRES           0.919204 Hz
AQ               1.0878977 sec
RG                    101
DW                 16.600 usec
DE                  6.50 usec
TE                 298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1
SFO1          125.7703643 MHz
NUC1                  13C
P0                  3.33 usec
P1                 10.00 usec
PLW1          88.26000214 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz65
PCPD2               80.00 usec
PLW2          23.68499947 W
PLW12          0.23014790 W
PLW13          0.11535020 W

F2 - Processing parameters
SI                   32768
SF            125.7577919 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



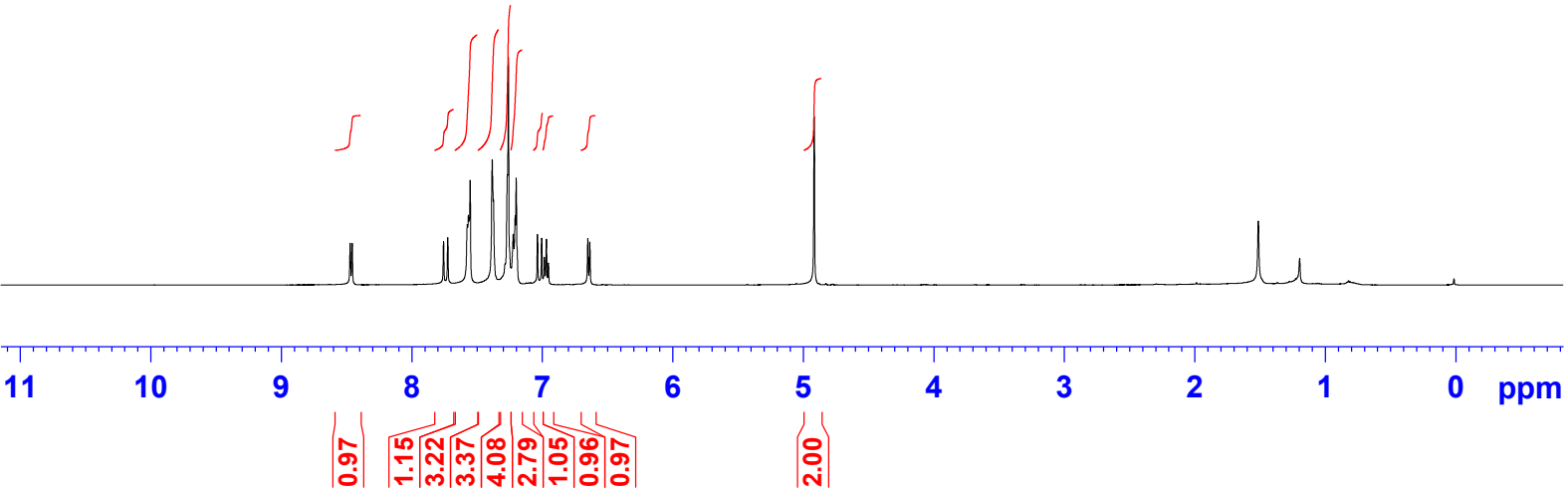


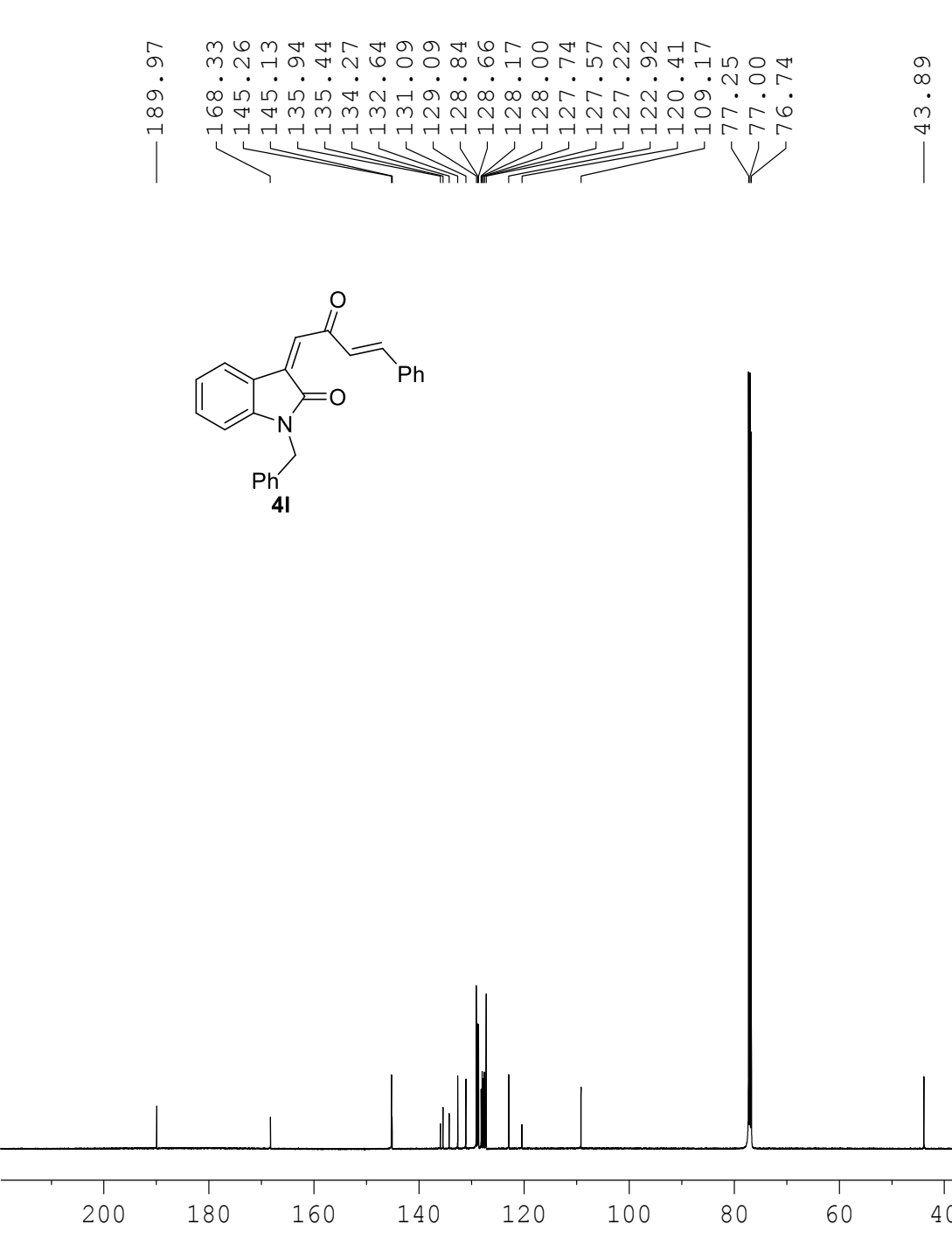
4l

```
Current Data Parameters
NAME      MH-97-Elimination
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20190409
Time                 13.32 h
INSTRUM              Avance
PROBHD    Z167889_0002 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH              10000.000 Hz
FIDRES               0.305176 Hz
AQ                3.2767999 sec
RG                   9.68812
DW                  50.000 usec
DE                  10.45 usec
TE                  298.2 K
D1               1.00000000 sec
TD0                       1
SFO1           500.1330883 MHz
NUC1                     1H
P0                   4.00 usec
P1                  12.00 usec
PLW1            7.84999990 W

F2 - Processing parameters
SI                    65536
SF             500.1300433 MHz
WDW                      EM
SSB          0
LB                   0.30 Hz
GB           0
PC                   1.00
```

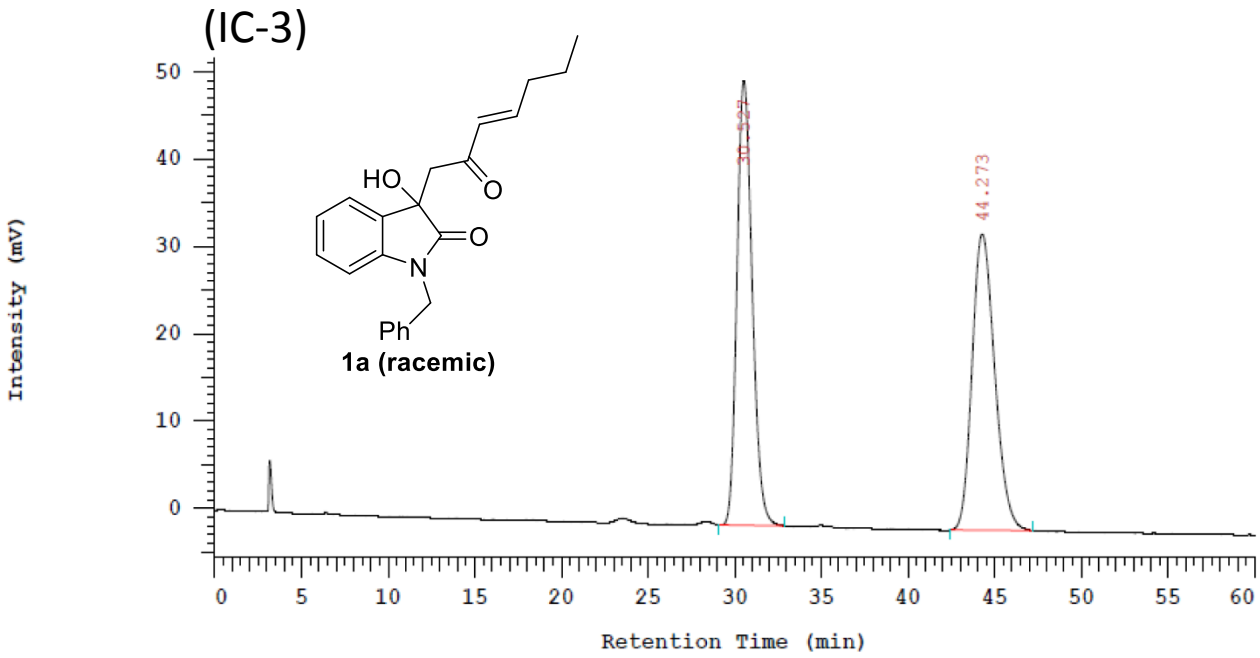




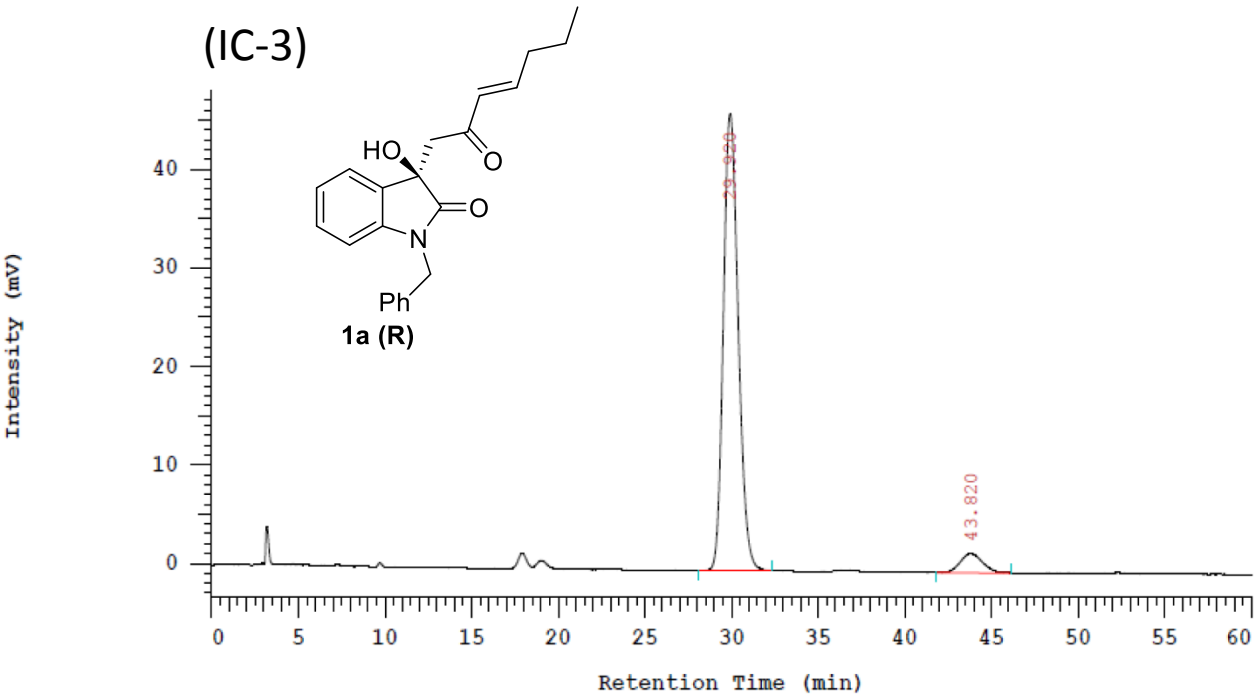

```
Current Data Parameters
NAME      MH-97-Elimination-C13
EXPNO                    12
PROCNO                    1

F2 - Acquisition Parameters
Date_            20190409
Time                14.37 h
INSTRUM            Avance
PROBHD    Z167889_0002 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH             30120.482 Hz
FIDRES          0.919204 Hz
AQ              1.0878977 sec
RG                    101
DW                 16.600 usec
DE                 30.00 usec
TE                 298.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1
SFO1          125.7703643 MHz
NUC1                  13C
P0                  3.33 usec
P1                 10.00 usec
PLW1          27.29999924 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz65
PCPD2              80.00 usec
PLW2           7.84999990 W
PLW12          0.17662001 W
PLW13          0.08870100 W

F2 - Processing parameters
SI                   32768
SF            125.7577928 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

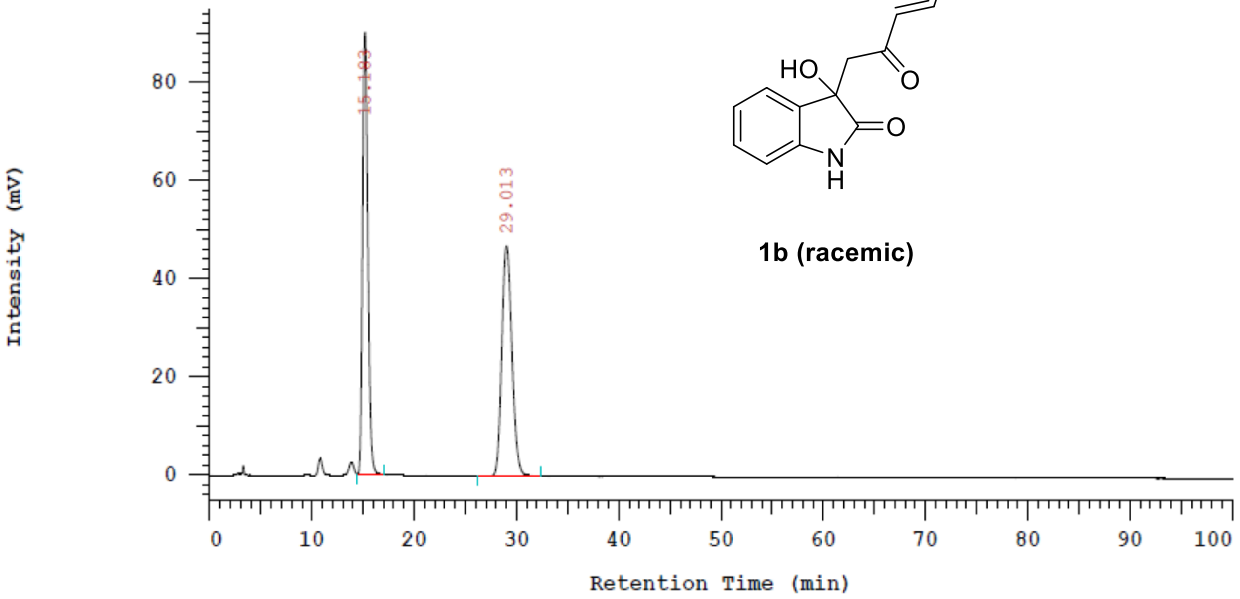


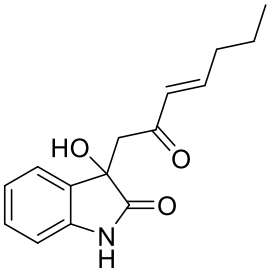
No.	RT	Area	Area %	BC
1	30.527	3060026	50.146	MC
2	44.273	3042247	49.854	MC
		6102273	100.000	



No.	RT	Area	Area %	BC
1	29.920	2712710	93.956	MC
2	43.820	174502	6.044	MC
		2887212	100.000	

(IC-3)

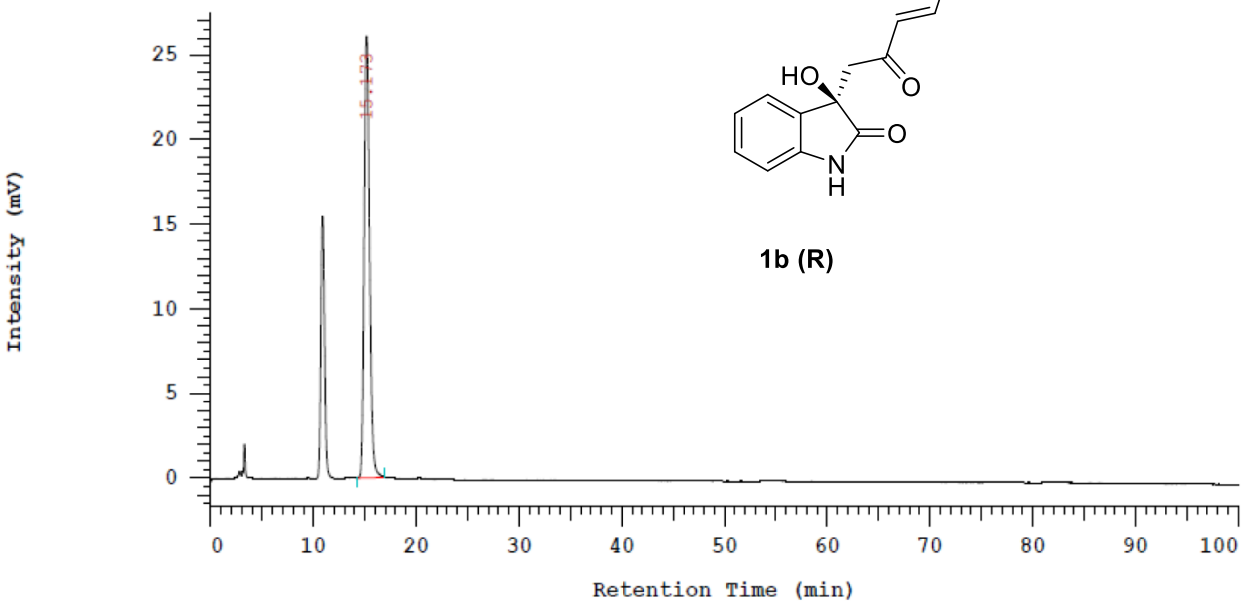


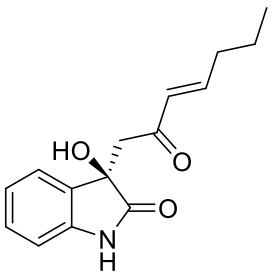


1b (racemic)

No.	RT	Area	Area %	BC
1	15.183	3088626	49.548	MC
2	29.013	3144945	50.452	MC
		6233571	100.000	

(IC-3)

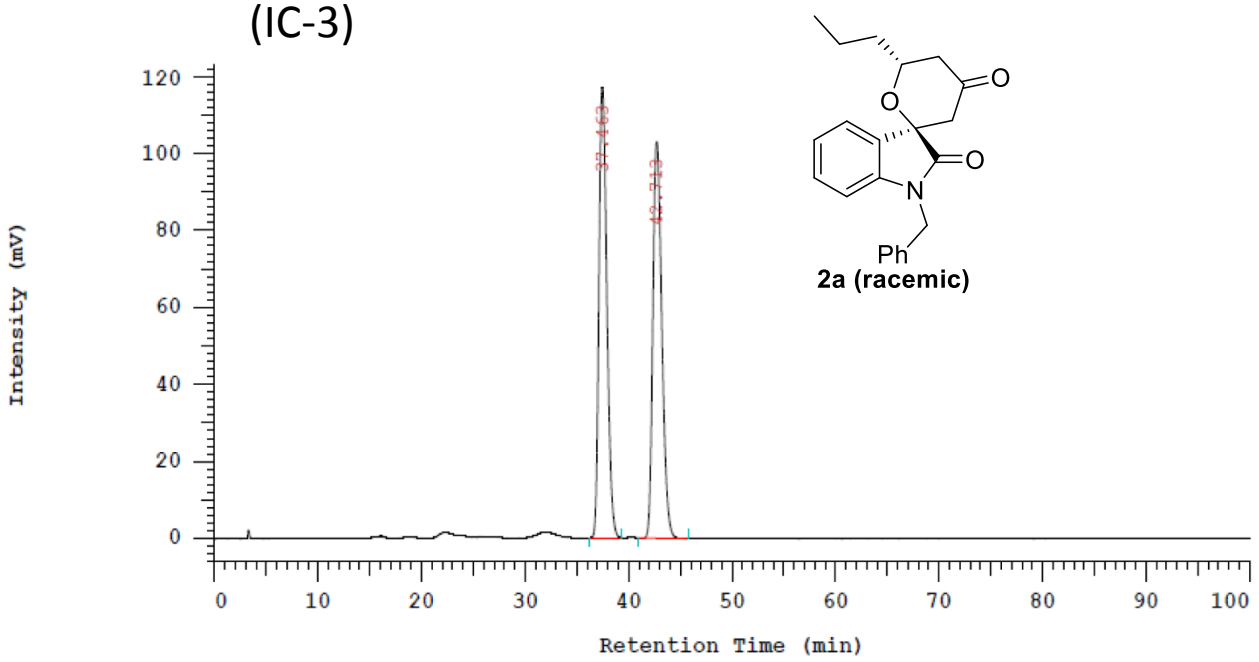




1b (R)

No.	RT	Area	Area %	BC
1	15.173	915181	100.000	MC
		915181	100.000	

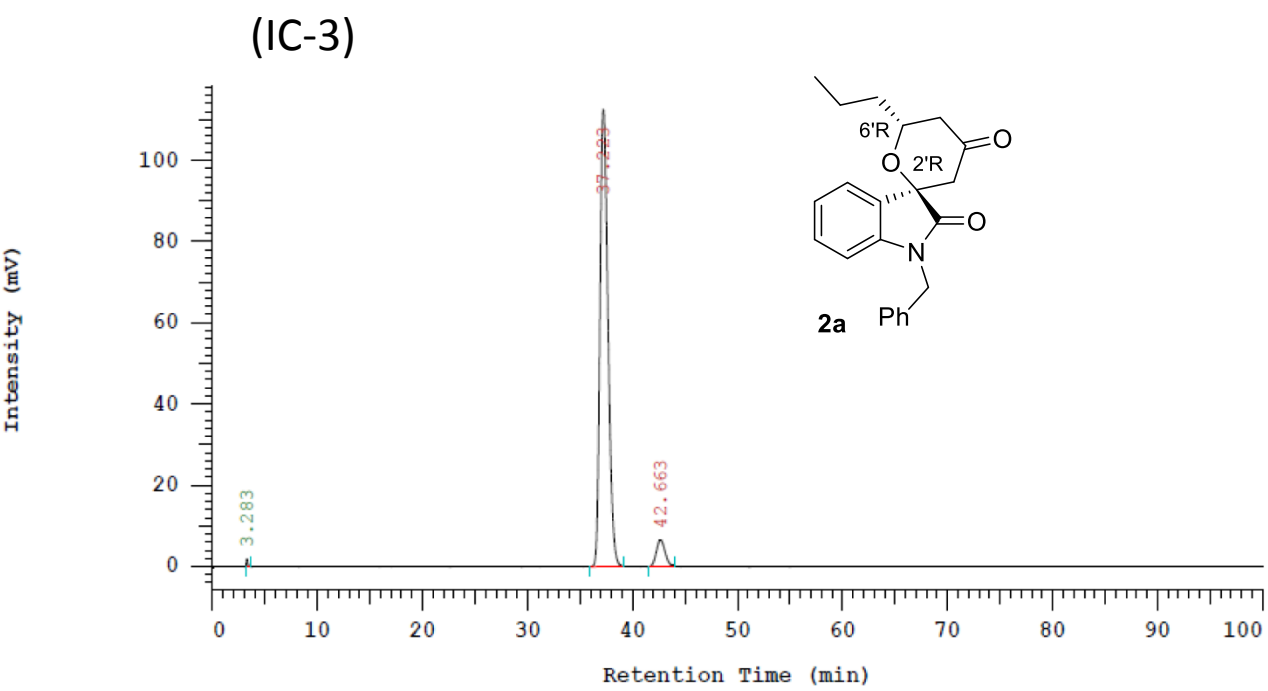
(IC-3)



2a (racemic)

No.	RT	Area	Area %	BC
1	37.463	6006670	49.948	MC
2	42.713	6019281	50.052	MC
		12025951	100.000	

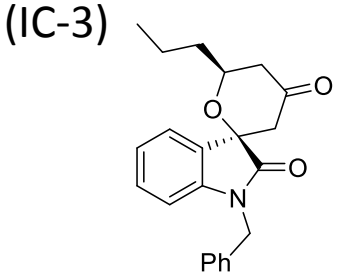
(IC-3)



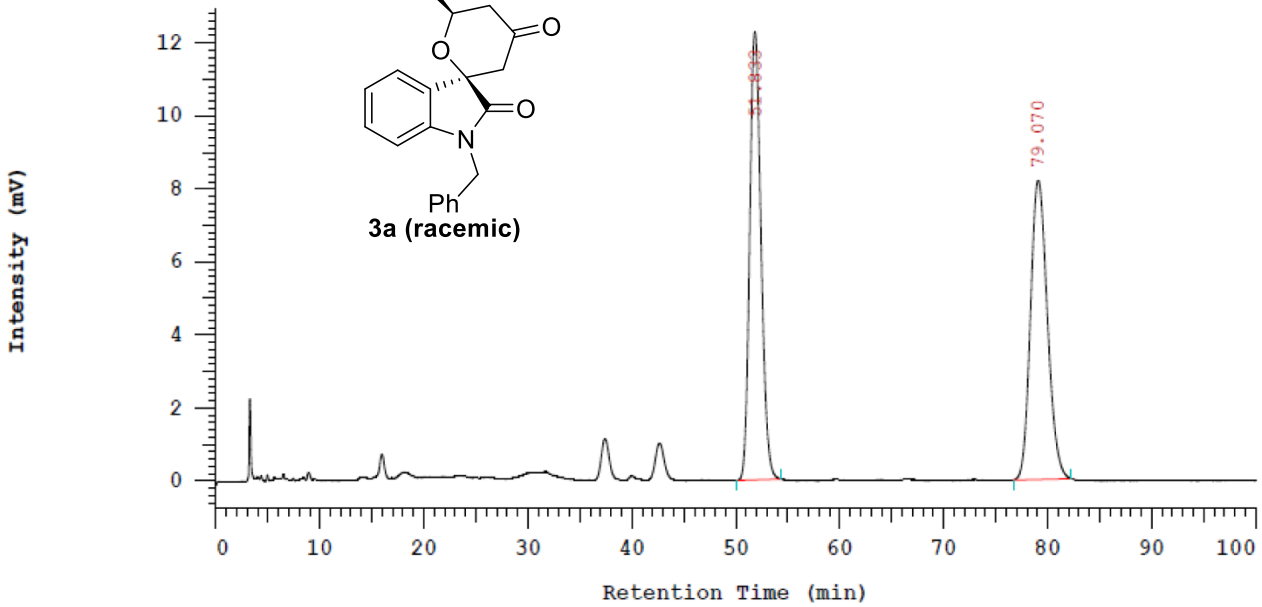
2a

No.	RT	Area	Area %	BC
1	3.283	15013	0.243	BB
2	37.223	5778143	93.697	MC
3	42.663	373683	6.060	MC
		6166839	100.000	

(IC-3)

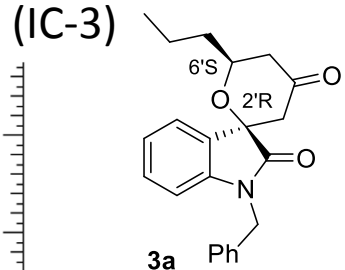


3a (racemic)

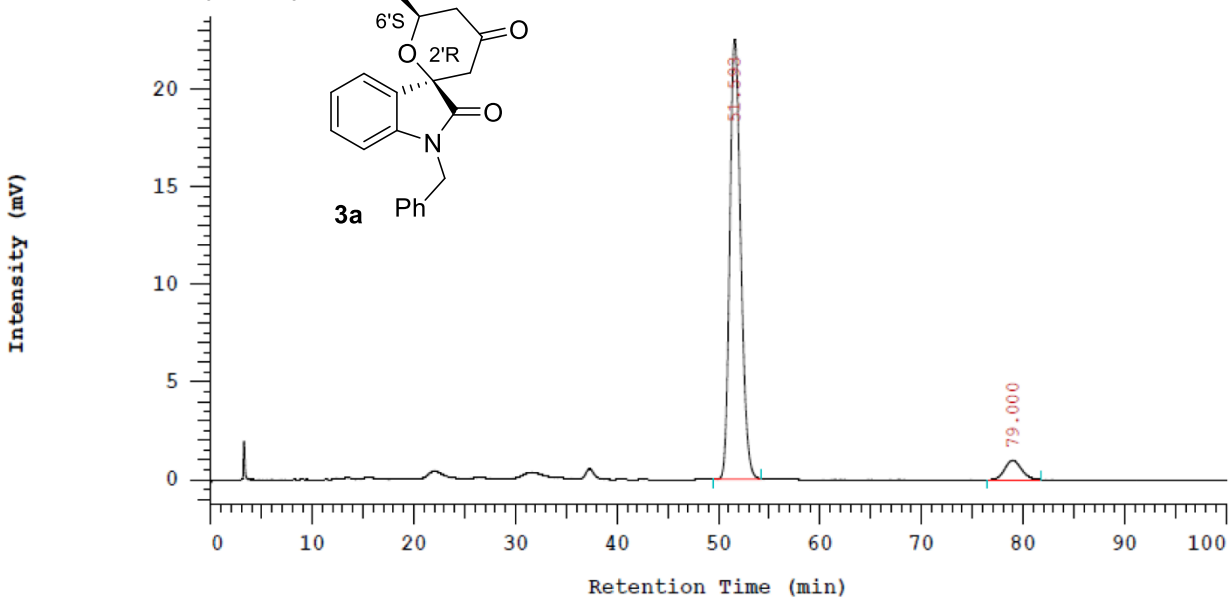


No.	RT	Area	Area %	BC
1	51.833	905582	49.806	MC
2	79.070	912644	50.194	MC
		1818226	100.000	

(IC-3)

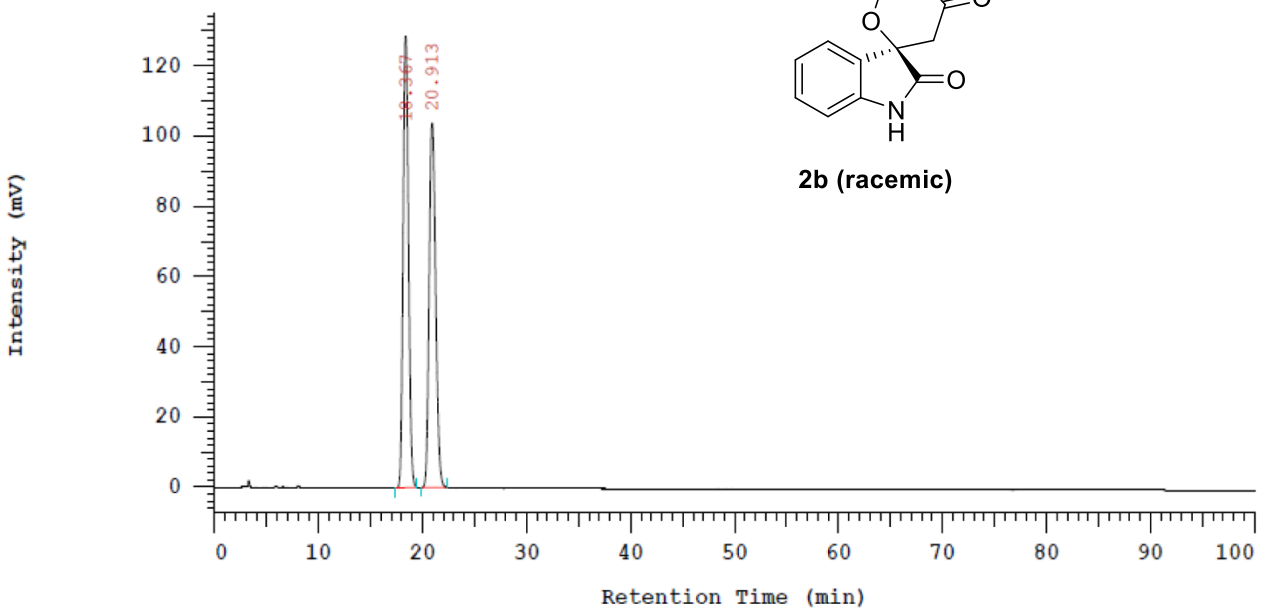


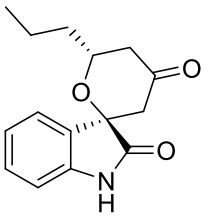
3a



No.	RT	Area	Area %	BC
1	51.593	1654757	93.741	MC
2	79.000	110489	6.259	MC
		1765246	100.000	

(IC-3)

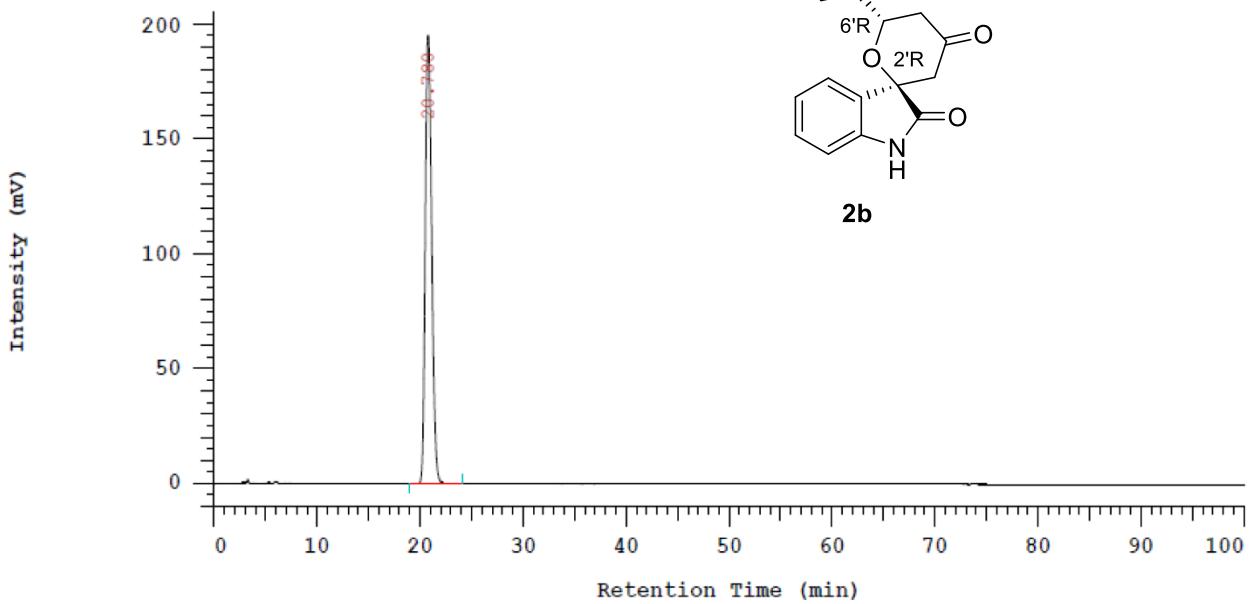


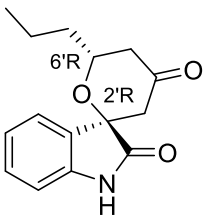


2b (racemic)

No.	RT	Area	Area %	BC
1	18.367	4410377	50.458	MC
2	20.913	4330327	49.542	MC
		8740704	100.000	

(IC-3)

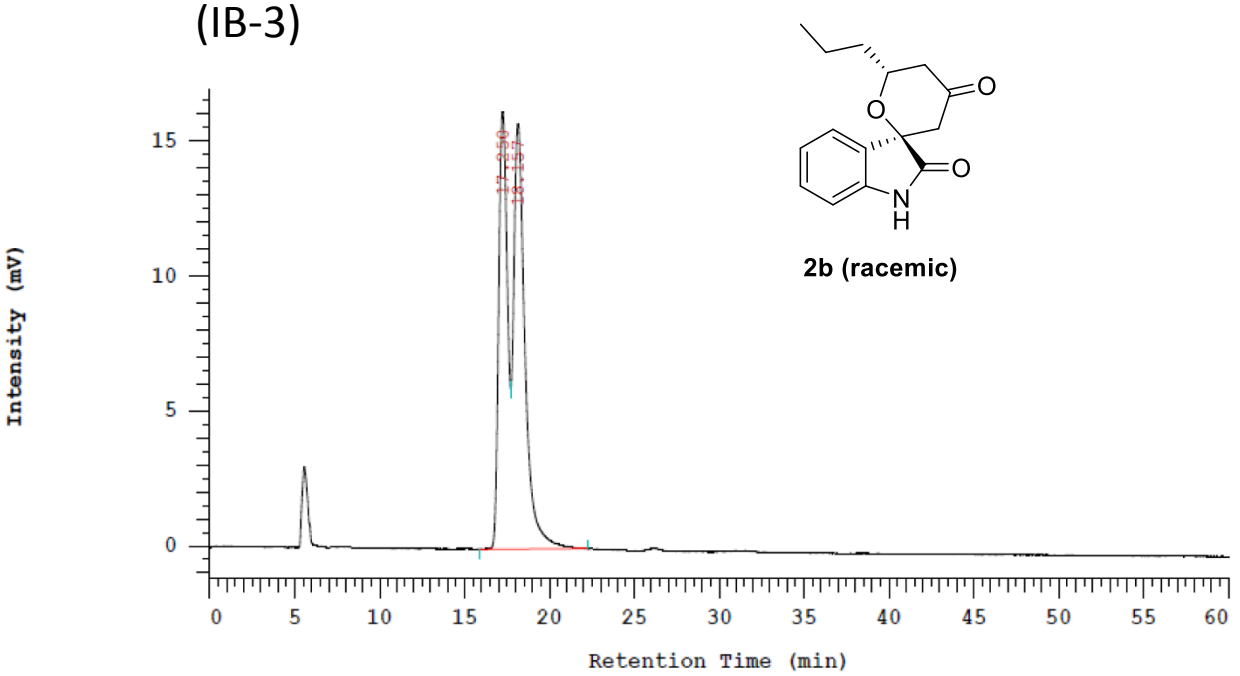




2b

No.	RT	Area	Area %	BC
1	20.780	8164230	100.000	MC
		8164230	100.000	

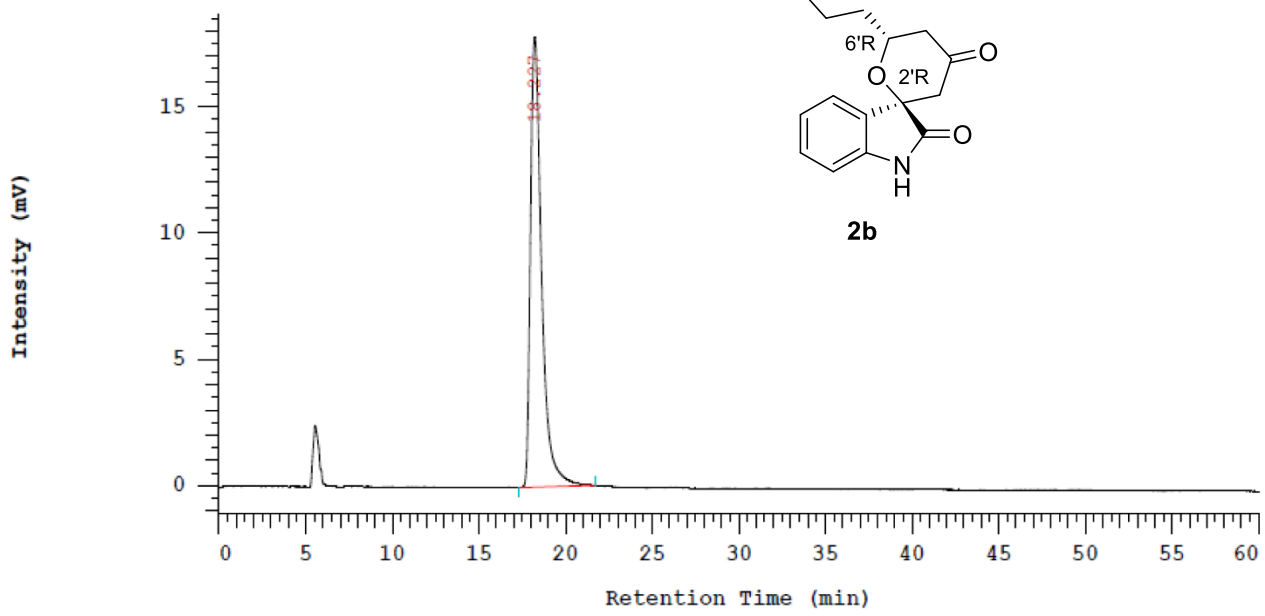
(IB-3)

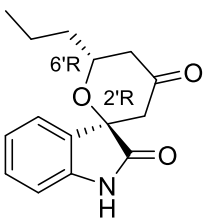


2b (racemic)

No.	RT	Area	Area %	BC
1	17.250	565280	43.372	MC
2	18.157	738047	56.628	MC
		1303327	100.000	

(IB-3)

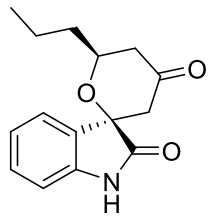




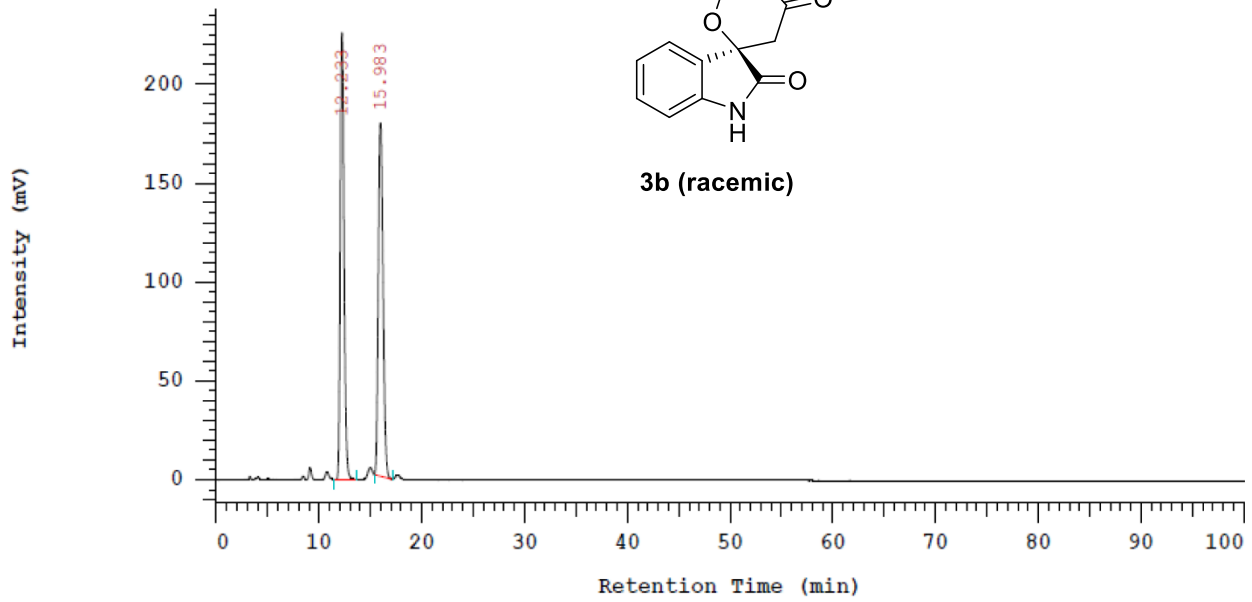
2b

No.	RT	Area	Area %	BC
1	18.227	780273	100.000	MC
		780273	100.000	

(IC-3)

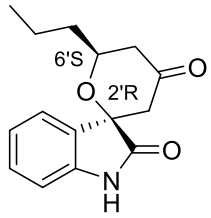


3b (racemic)

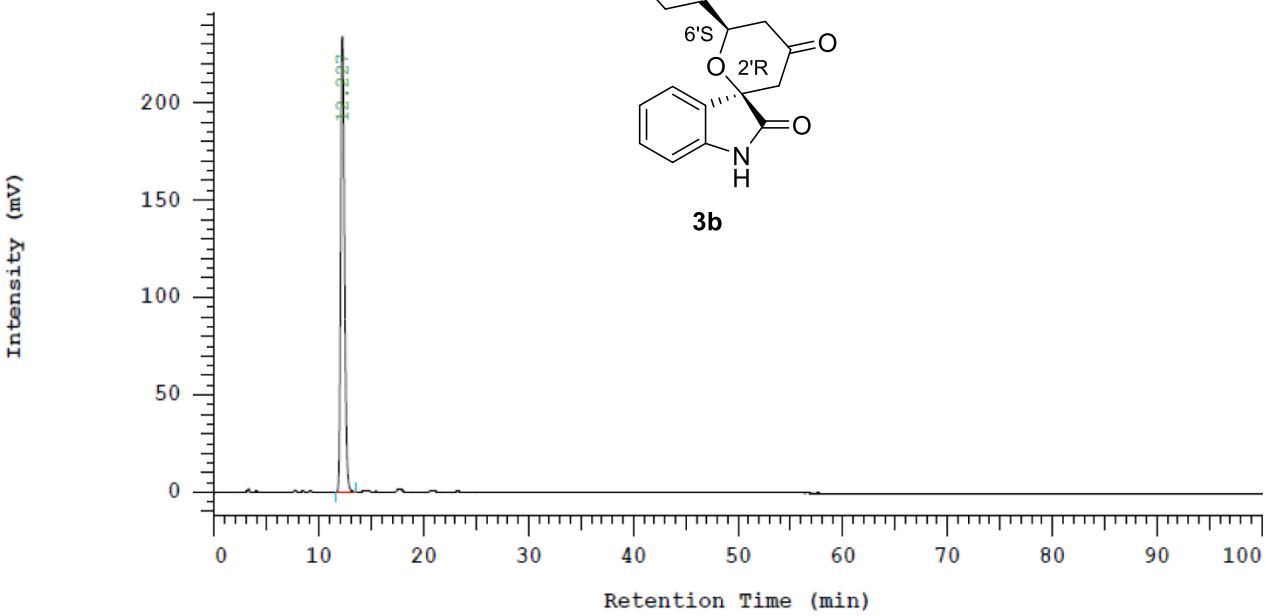


No.	RT	Area	Area %	BC
1	12.233	5470125	50.279	MC
2	15.983	5409514	49.721	MC
		10879639	100.000	

(IC-3)

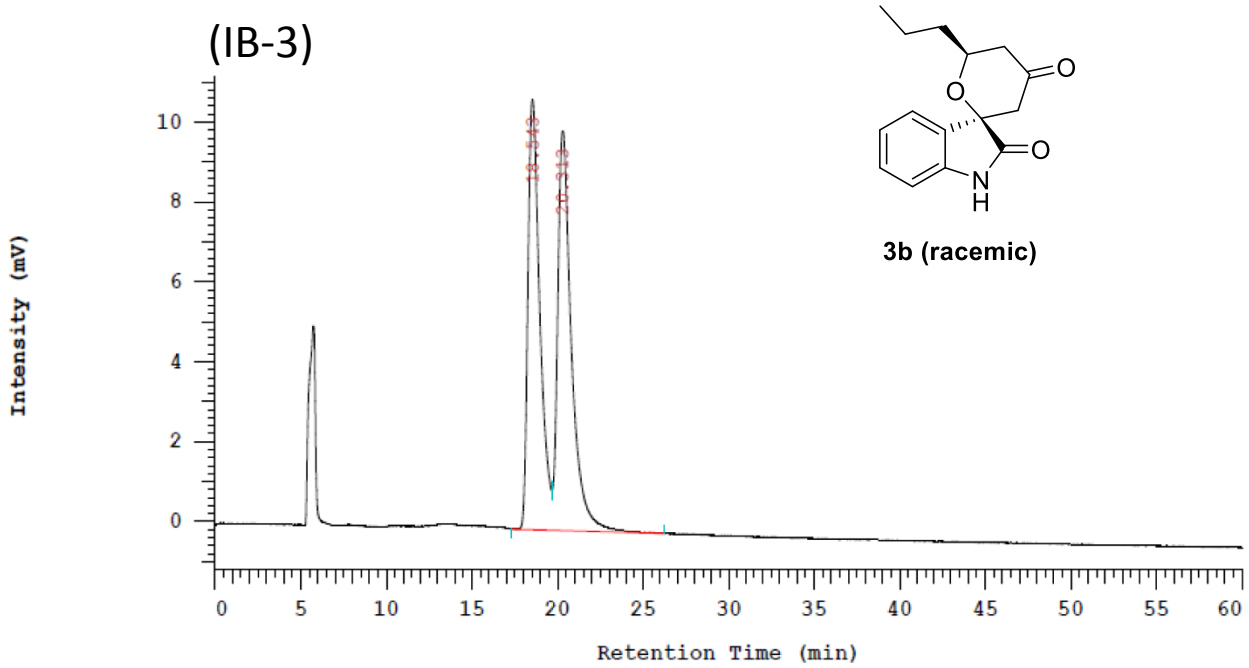


3b



No.	RT	Area	Area %	BC
1	12.227	5600163	100.000	BB
		5600163	100.000	

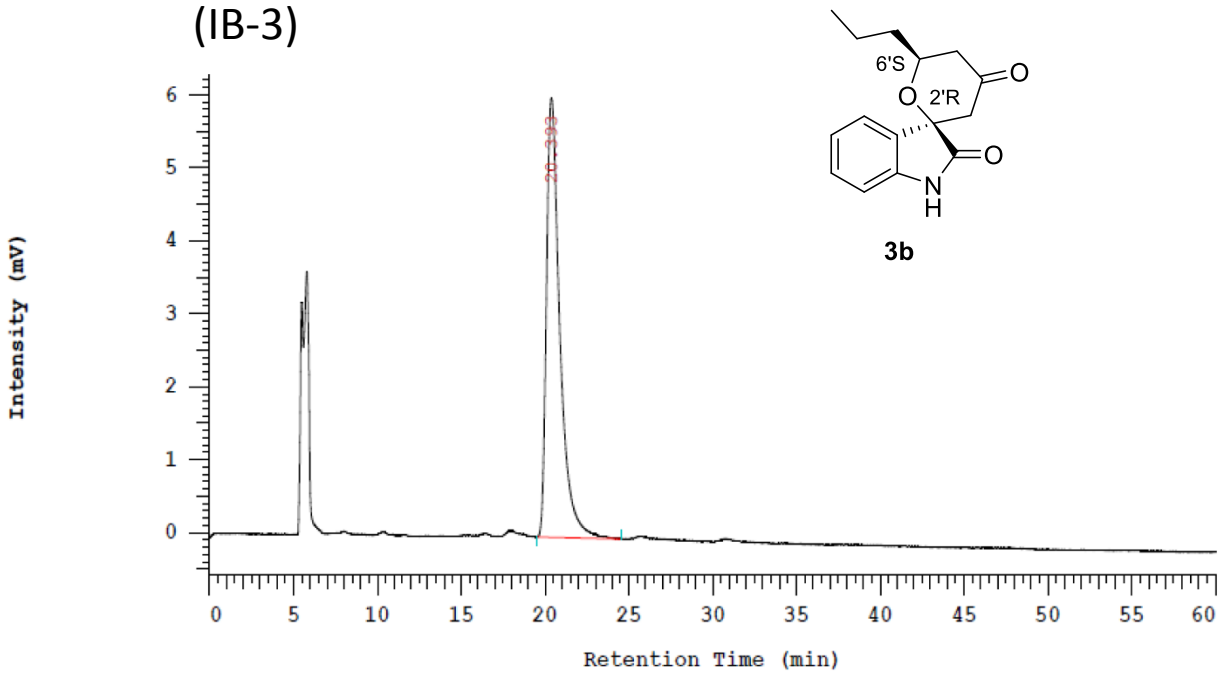
(IB-3)



3b (racemic)

No.	RT	Area	Area %	BC
1	18.543	509112	48.057	MC
2	20.313	550286	51.943	MC
		1059398	100.000	

(IB-3)



3b

No.	RT	Area	Area %	BC
1	20.393	328811	100.000	MC
		328811	100.000	