

Supporting Information

Reactions of Pyruvate-Derived Dihydropyrans with Formaldehyde: Synthesis of Functionalized Fuopyrans and Related Products

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1. General

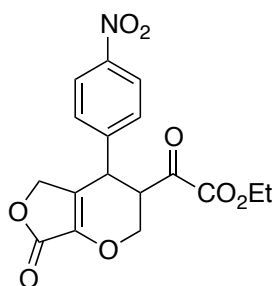
For thin layer chromatography (TLC), Merck Silica gel 60 F254 aluminum sheets were used. Flash column chromatography was performed using Merck silica gel 60 (230-400 mesh). ¹H NMR and ¹³C NMR were recorded on a Bruker Avance 400. Proton chemical shifts are reported in ppm downfield from tetramethylsilane or from the residual solvent as internal standard in CDCl₃ (δ 7.26 ppm). Carbon chemical shifts were internally referenced to the deuterated solvent signals in CDCl₃ (δ 77.0 ppm). High-resolution mass spectra were recorded on a Thermo Scientific LTQ Orbitrap ESI ion trap mass spectrometer.

2. Synthesis of Fuopyrans 2

Procedure for the Synthesis of Fuopyrans 2 (Table 2)

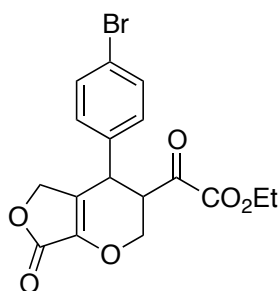
To a mixture of 4-substituted dihydropyran **1**¹ (0.1 mmol, 1equiv) and paraformaldehyde (1.0 mmol, 10 equiv) in CH₃CN (1.0 mL), pyrrolidine (0.05 mmol, 0.5 equiv, 50 mol %) and acetic acid (0.1 mmol, 1.0 equiv, 100 mol %) was added. The reaction mixture was stirred at room temperature (25 °C) until **1** was consumed (monitored by TLC). The mixture was purified by flash column chromatography (hexane/EtOAc) to afford fuopyran **2**.

Compound 2a



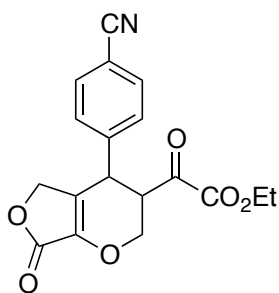
Flash column chromatography (hexane/EtOAc = 7:3), colorless solid. ¹H NMR (400 MHz, CDCl₃): δ 8.24 (d, *J* = 8.4 Hz, 2H), 7.42 (d, *J* = 8.4 Hz, 2H), 4.74 (dd, *J* = 16.2 Hz, 1.6 Hz, 1H), 4.58 (dd, *J* = 16.2 Hz, 0.8 Hz, 1H), 4.48 (dd, *J* = 11.6 Hz, 3.6 Hz, 1H), 4.43 (dd, *J* = 11.6 Hz, 6.0 Hz, 1H), 4.37-4.27 (m, 3H), 3.87-3.82 (m, 1H), 1.35 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 190.6, 165.8, 159.6, 147.8, 145.7, 142.1, 129.15, 129.09, 124.6, 67.8, 65.8, 63.5, 49.7, 38.0, 13.9. ESI-HRMS: calcd for C₁₇H₁₆O₈N ([M+H]⁺) 362.0870, found 362.0879.

Compound 2b



Flash column chromatography (hexane/EtOAc = 8:2); colorless solid. ¹H NMR (400 MHz, CDCl₃): δ 7.51 (d, *J* = 8.4 Hz, 2H), 7.07 (d, *J* = 8.4 Hz, 2H), 4.70 (dd, *J* = 16.4 Hz, 2.0 Hz, 1H), 4.58 (dd, *J* = 16.4 Hz, 1.2 Hz, 1H), 4.45 (dd, *J* = 11.6 Hz, 3.6 Hz, 1H), 4.40 (dd, *J* = 11.6 Hz, 6.2 Hz, 1H), 4.30 (q, *J* = 7.2 Hz, 2H), 4.17 (brd, *J* = 5.2 Hz, 1H), 3.79 (ddd, *J* = 6.2 Hz, 5.2 Hz, 3.6 Hz, 1H), 1.34 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 191.3, 166.1, 159.8, 141.7, 137.4, 132.6, 130.4, 129.6, 122.4, 68.0, 66.0, 63.3, 49.7, 38.0, 13.9. ESI-HRMS: calcd for C₁₇H₁₆O₄Br ([M+H]⁺) 395.0125, found 395.0134.

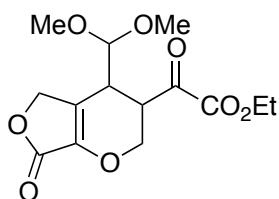
Compound 2c



Flash column chromatography (hexane/EtOAc = 7:3); pale yellow gum. ¹H NMR (400 MHz, CDCl₃): δ 7.69 (d, *J* = 8.4 Hz, 2H), 7.34 (d, *J* = 8.4 Hz, 2H), 4.72 (dd, *J* = 16.4 Hz, 1.6 Hz, 1H), 4.57 (dd, *J* = 16.4 Hz, 1.2 Hz, 1H), 4.46 (dd, *J* = 11.6 Hz, 3.6 Hz, 1H), 4.42 (dd, *J* = 11.6 Hz, 5.6 Hz, 1H), 4.37-4.26 (m, 3H), 3.83-3.79 (m, 1H); 1.35 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 190.7, 165.7, 159.6, 143.7, 142.1, 133.1, 129.1, 128.8, 117.9, 112.5, 67.8, 65.7, 63.4, 49.6, 38.2, 13.8. ESI-HRMS: calcd for C₁₈H₁₆NO₆ ([M+H]⁺) 342.0972, found 342.0971.

¹ P. V. Chouthaiwale, F. Tanaka, *Chem. Commun.* 2014, **50**, 14881. P. V. Chouthaiwale, S. Lapointe, F. Tanaka, *Heterocycles* 2017, **95**, 587.

Compound 2d



Flash column chromatography (hexane/EtOAc = 8:2); colorless gum, dr = 7:3. ^1H NMR (400 MHz, CDCl_3): δ 4.92-4.68 (m, 2H x 7/10), 4.52 (dd, J = 12.2 Hz, 3.4 Hz, 1H x 3/10), 4.43-4.16 (m, 5H + 1H x 3/10), 3.81 (td, J = 7.6 Hz, 3.6 Hz, 1H x 7/10), 3.71-3.64 (m, 1H x 3/10), 3.60-3.54 (m, 1H x 3/10), 3.39 (s, 3H x 3/10), 3.36 (s, 3H x 7/10), 3.28 (s, 3H x 7/10), 3.26 (s, 3H x 3/10), 1.40 (t, J = 7.2 Hz, 3H x 7/10), 1.38 (t, J = 7.2 Hz, 3H x 3/10). ^{13}C NMR (100 MHz, CDCl_3): δ 191.7, 190.1, 166.3, 160.2, 159.6, 142.0, 141.5, 128.9, 128.0, 105.6, 104.2, 69.4, 69.1, 67.3, 65.4, 63.0, 62.6, 56.4, 54.9, 54.1, 53.9, 42.0, 41.9, 40.2, 38.9, 14.0. ESI-HRMS: calcd for $\text{C}_{14}\text{H}_{19}\text{O}_8$ ($[\text{M}+\text{H}]^+$) 315.1074, found 315.1084.

3. Synthesis of Dimethylene Derivatives 3 and of Pyran Derivative 4a

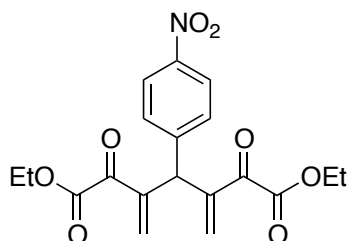
Procedure for the Synthesis of Dimethylene Derivatives 3 (Table 3)

Method A. To a mixture of 4-substituted dihydropyran **1** (0.1 mmol, 1equiv) and paraformaldehyde (1.0 mmol, 10 equiv) in CH_3CN (1.0 mL), diethylamine (0.025 mmol, 0.25 equiv, 25 mol %) and acetic acid (0.05 mmol, 0.5 equiv, 50 mol %) was added. The reaction mixture was stirred at room temperature (25 $^\circ\text{C}$) until **1** was consumed (monitored by TLC). The mixture was purified by flash column chromatography (hexane/EtOAc) to afford dimethylene derivative **3**.

Method B. To a mixture of 4-substituted dihydropyran **1** (0.2 mmol, 1equiv) and paraformaldehyde (2.0 mmol, 10 equiv) in CH_3CN (2.0 mL), diethylamine (0.14 mmol, 0.7 equiv, 70 mol %) and acetic acid (0.1 mmol, 0.5 equiv, 50 mol %) was added. The reaction mixture was stirred at room temperature (25 $^\circ\text{C}$) until **1** was consumed (monitored by TLC). The mixture was purified by flash column chromatography (hexane/EtOAc) to afford dimethylene derivative **3**.

A 1 g-scale reaction of **1a** to afford **3a** (Table 1, entry 10). To a mixture of 4-substituted dihydropyran **1a** (2.74 mmol, 1equiv) and paraformaldehyde (27.4 mmol, 10 equiv) in CH_3CN (27 mL), diethylamine (200 μL , 1.9 mmol, 0.7 equiv) and acetic acid (82 μL , 1.4 mmol, 0.5 equiv) was added. The reaction mixture was stirred at room temperature (25 $^\circ\text{C}$) for 24 h (until **1** was consumed, monitored by TLC). The mixture was concentrated under vacuum and was purified by flash column chromatography (hexane/EtOAc = 8:2 to 7:3) to afford dimethylene derivative **3a** (426 mg, 40%) and **4a** (205 mg, 20%). Compound **4a** was eluted first and then compound **3a** was eluted.

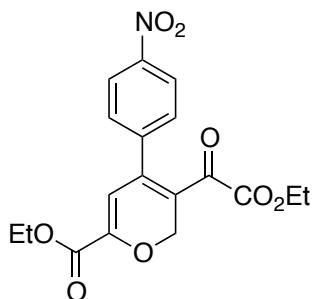
Compound 3a



Method A. Flash column chromatography (hexane/EtOAc = 8:2 to 7:3), colorless gum. ^1H NMR (400 MHz, CDCl_3): δ 8.19 (d, J = 8.8, Hz, 2H), 7.35 (d, J = 8.8, Hz, 2H), 6.48 (s, 2H), 5.96 (s, 2H), 5.49 (s, 1H), 4.36 (q, J = 7.2 Hz, 4H), 1.36

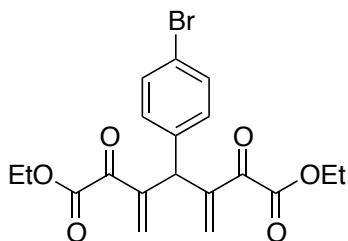
(t, $J = 7.2$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 186.1, 163.1, 147.4, 145.3, 144.1, 134.4, 129.8, 124.1, 62.6, 43.9, 14.0. ESI-HRMS: calcd for $\text{C}_{19}\text{H}_{20}\text{O}_8\text{N}$ ($[\text{M}+\text{H}]^+$) 390.1183, found 390.1175.

Compound 4a



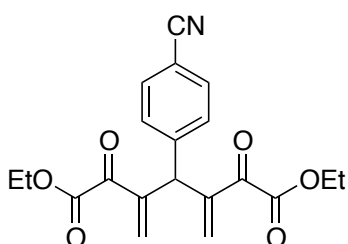
In the reaction to afford **3a** (Method A), compound **4a** was also obtained. Flash column chromatography (hexane/EtOAc = 8:2 to 7:3), colorless gum. ^1H NMR (400 MHz, CDCl_3): δ 8.25 (d, $J = 8.8$ Hz, 2H), 7.46 (dd, $J = 8.8, 2.0$ Hz, 2H), 6.55 (s, 1H), 5.16 (s, 2H), 4.34 (q, $J = 7.1$ Hz, 2H), 3.65 (q, $J = 7.1$ Hz, 2H), 1.36 (t, $J = 7.1$ Hz, 3H), 1.09 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 184.6, 162.6, 160.9, 150.2, 148.5, 146.3, 142.2, 129.5, 123.7, 119.5, 112.0, 66.3, 62.4, 62.3, 14.1, 13.5. ESI-HRMS: calcd for $\text{C}_{18}\text{H}_{18}\text{O}_8\text{N}$ ($[\text{M}+\text{H}]^+$) 376.1027, found 376.1030.

Compound 3b



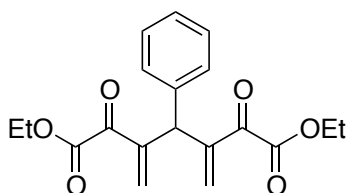
Method A. Flash column chromatography (hexane/EtOAc = 8:2), colorless gum. ^1H NMR (400 MHz, CDCl_3): δ 7.46 (d, $J = 8.4$ Hz, 2H), 7.04 (d, $J = 8.4$ Hz, 2H), 6.40 (s, 2H), 5.93 (s, 2H), 5.35 (s, 1H), 4.35 (q, $J = 7.2$ Hz, 4H), 1.36 (t, $J = 7.2$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 186.4, 163.3, 144.7, 136.6, 133.8, 132.0, 130.6, 121.5, 62.4, 43.6, 14.0. ESI-HRMS: calcd for $\text{C}_{19}\text{H}_{20}\text{O}_6\text{Br}$ ($[\text{M}+\text{H}]^+$) 423.0438, found 423.0429.

Compound 3c



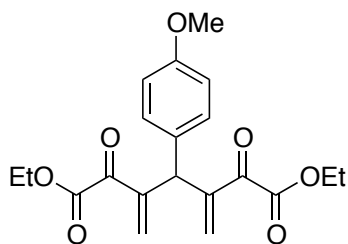
Method B. Flash column chromatography (hexane/EtOAc = 7:3), colorless gum. ^1H NMR (400 MHz, CDCl_3): δ 7.62 (d, $J = 8.2$ Hz, 2H), 7.27 (d, $J = 8.2$ Hz, 2H), 6.44 (s, 2H), 5.92 (s, 2H), 5.42 (s, 1H), 4.33 (q, $J = 7.1$ Hz, 4H), 1.34 (t, $J = 7.1$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 186.2, 163.1, 144.0, 143.3, 134.3, 132.6, 129.6, 118.4, 111.5, 62.5, 44.0, 14.0. ESI-HRMS: calcd for $\text{C}_{20}\text{H}_{20}\text{O}_6\text{N}$ ($[\text{M}+\text{H}]^+$) 370.1285, found 370.1259.

Compound 3d



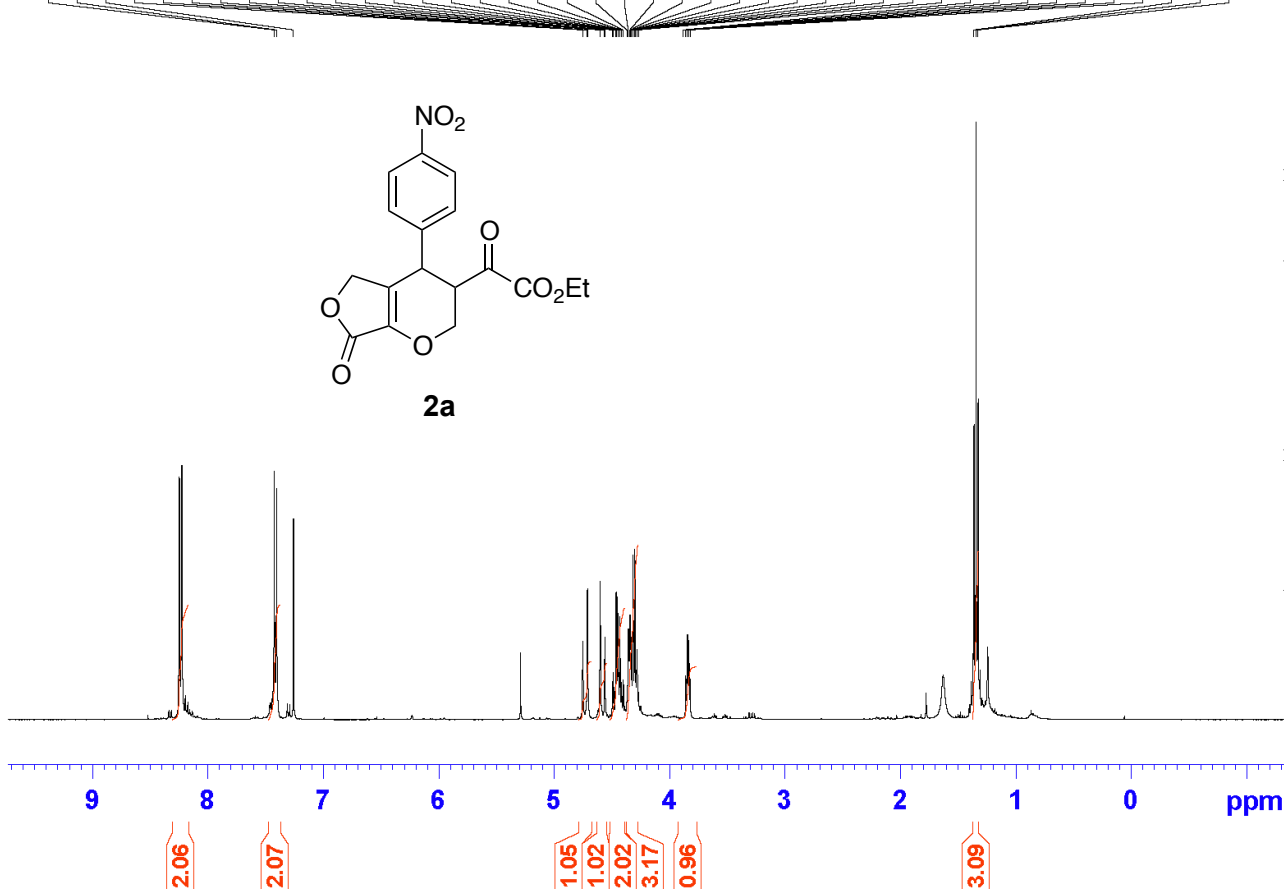
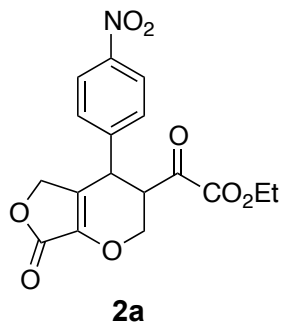
Method B. Flash column chromatography (hexane/EtOAc = 8:2), colorless gum. ^1H NMR (400 MHz, CDCl_3): δ 7.34-7.14 (m, 5H), 6.38 (s, 2H), 5.93 (s, 2H), 5.40 (s, 1H), 4.34 (q, $J = 7.1$ Hz, 4H), 1.35 (t, $J = 7.1$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 186.5, 163.5, 145.1, 137.4, 133.7, 128.9, 128.8, 127.4, 62.3, 44.0, 14.0. ESI-HRMS: calcd for $\text{C}_{19}\text{H}_{21}\text{O}_6$ ($[\text{M}+\text{H}]^+$) 345.1333, found 345.1327.

Compound 3e



Method B. Flash column chromatography (hexane/EtOAc = 8:2), colorless gum. ¹H NMR (400 MHz, CDCl₃): δ 7.06 (d, *J* = 8.5 Hz, 2H), 6.85 (d, *J* = 8.8, Hz, 2H), 6.35 (s, 2H), 5.92 (s, 2H), 5.34 (s, 1H), 4.34 (q, *J* = 7.1 Hz, 4H), 3.78 (s, 3H), 1.35 (t, *J* = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃): δ 186.6, 163.5, 158.9, 145.4, 133.4, 129.9, 129.2, 114.2, 62.3, 55.2, 43.2, 14.0. ESI-HRMS: calcd for C₂₀H₂₃O₇ ([M+H]⁺) 375.1438, found 375.1437.

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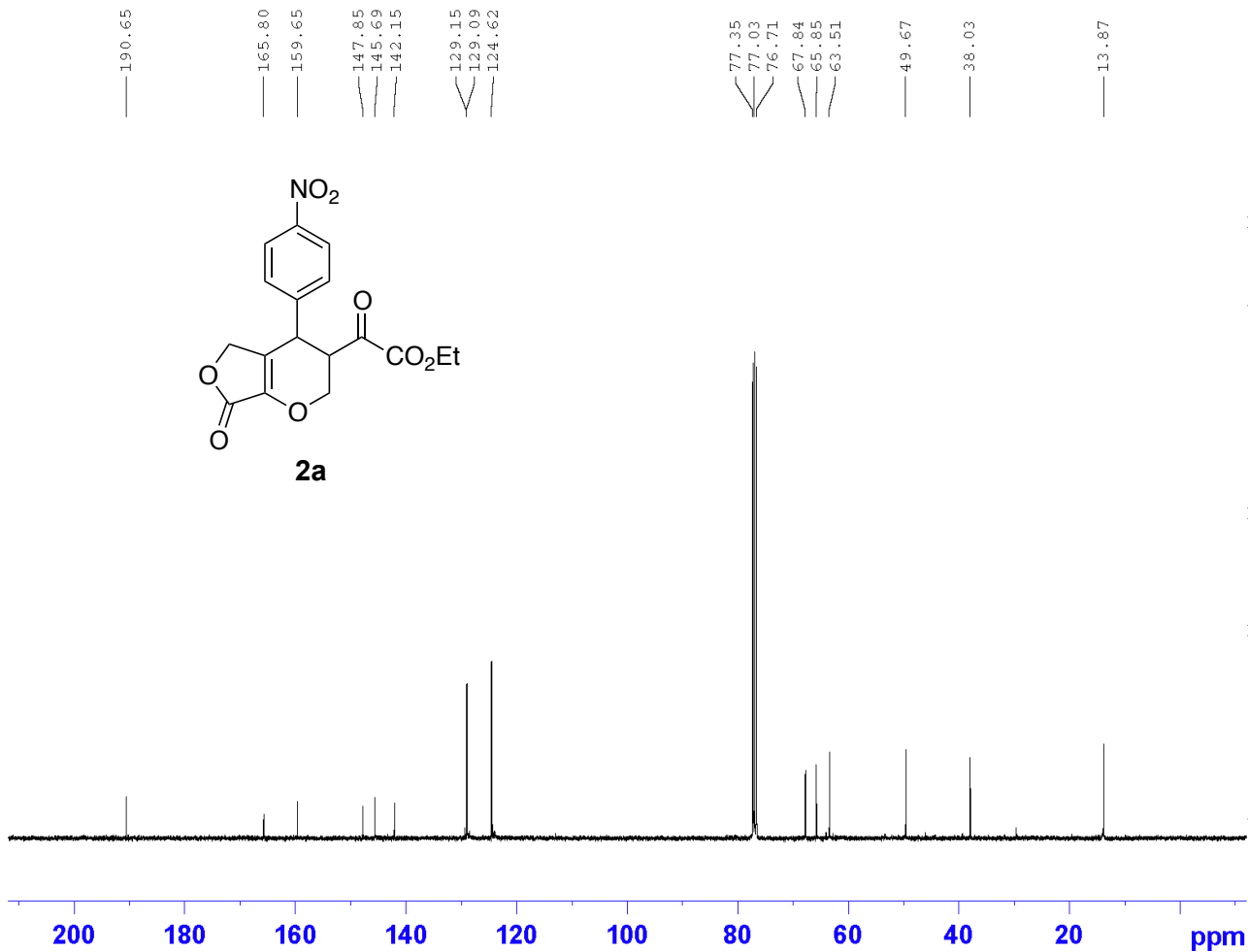


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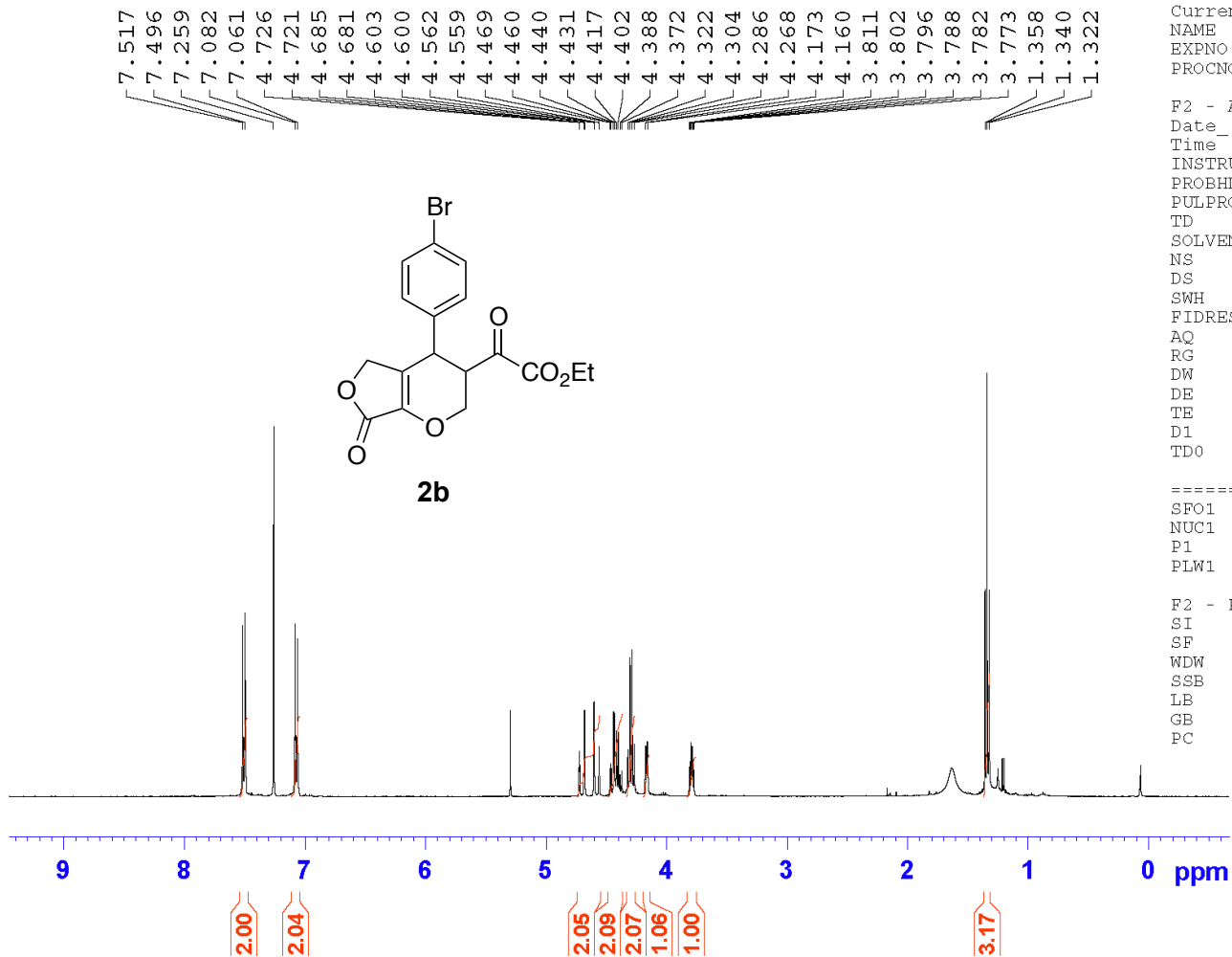
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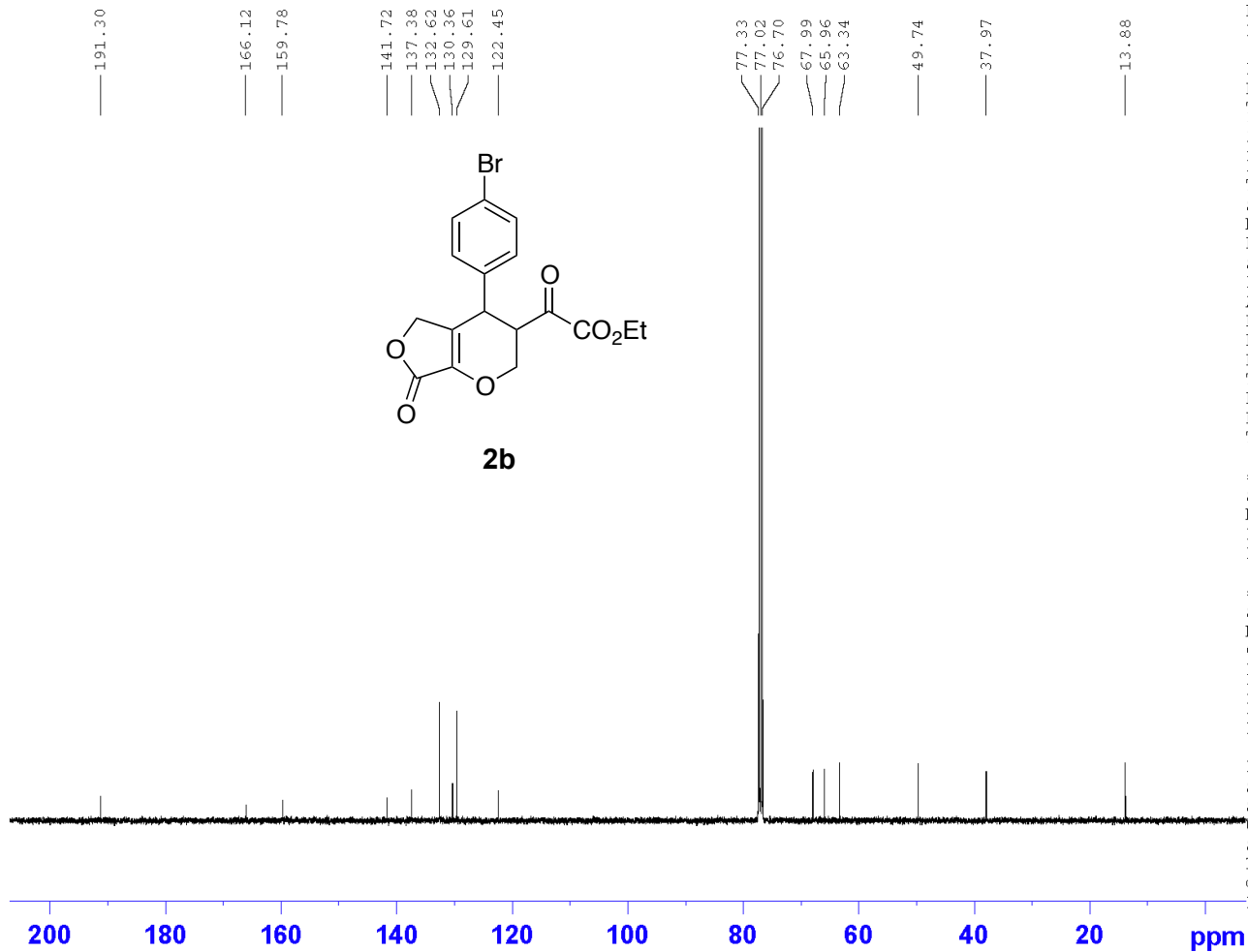
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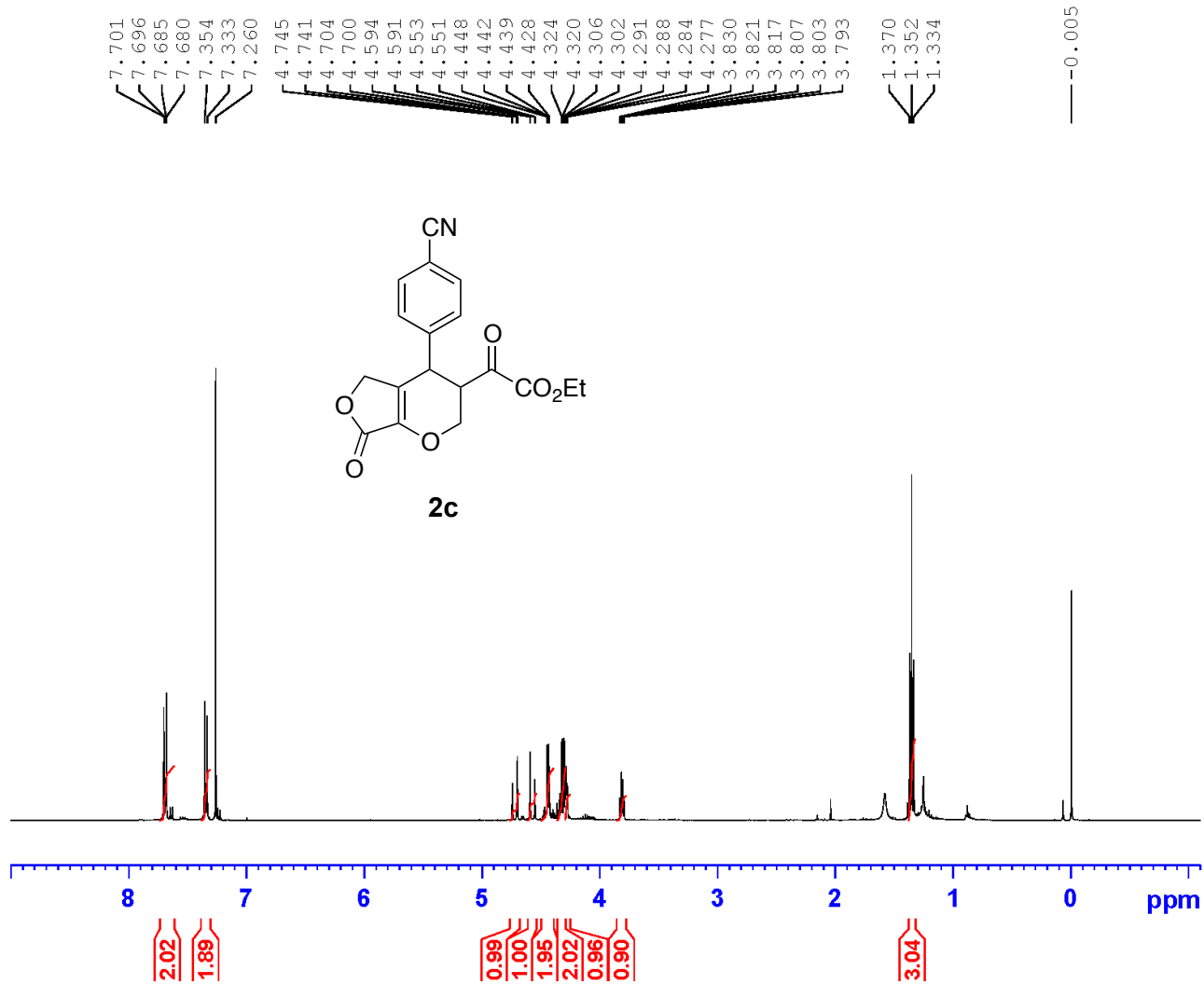
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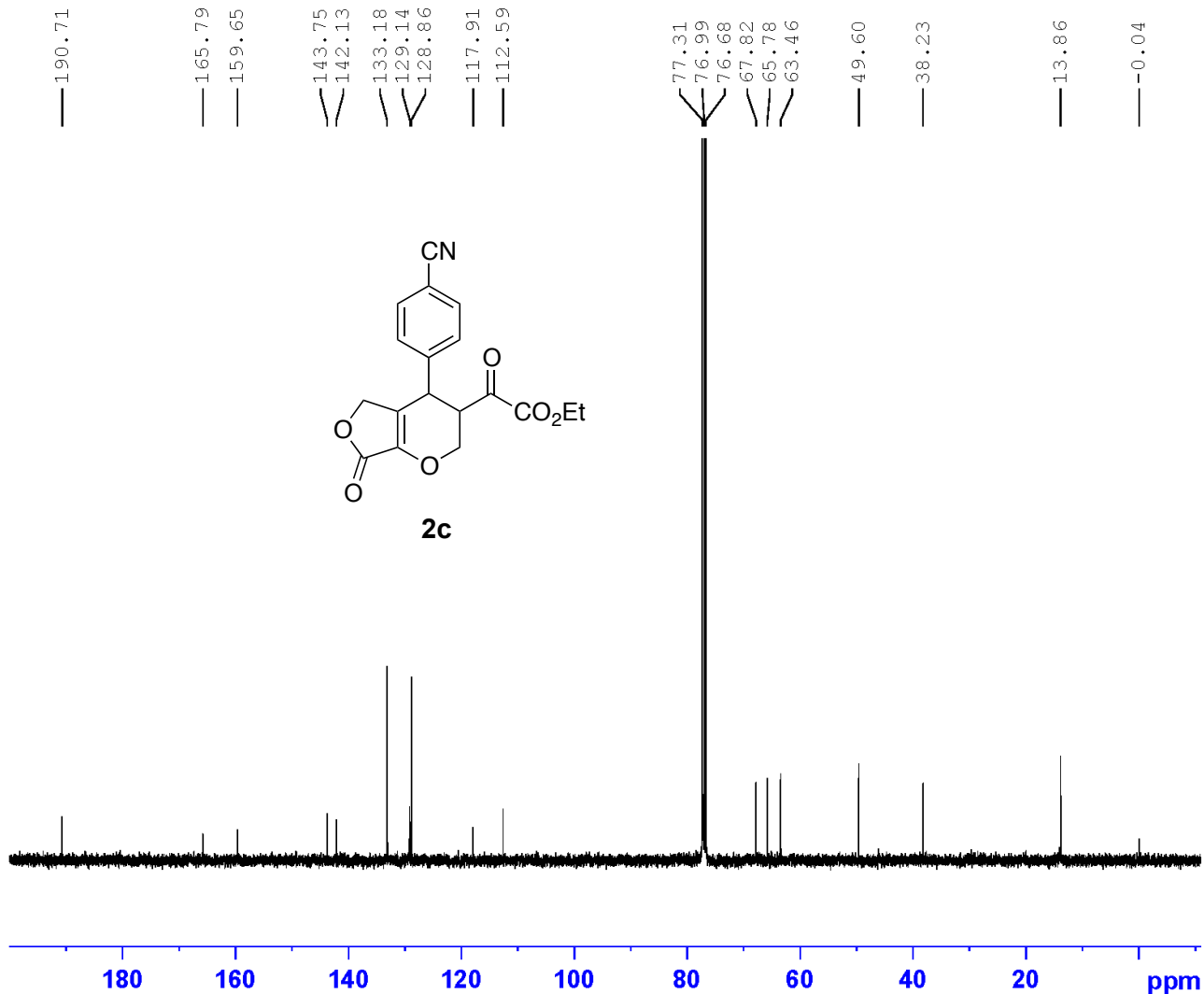


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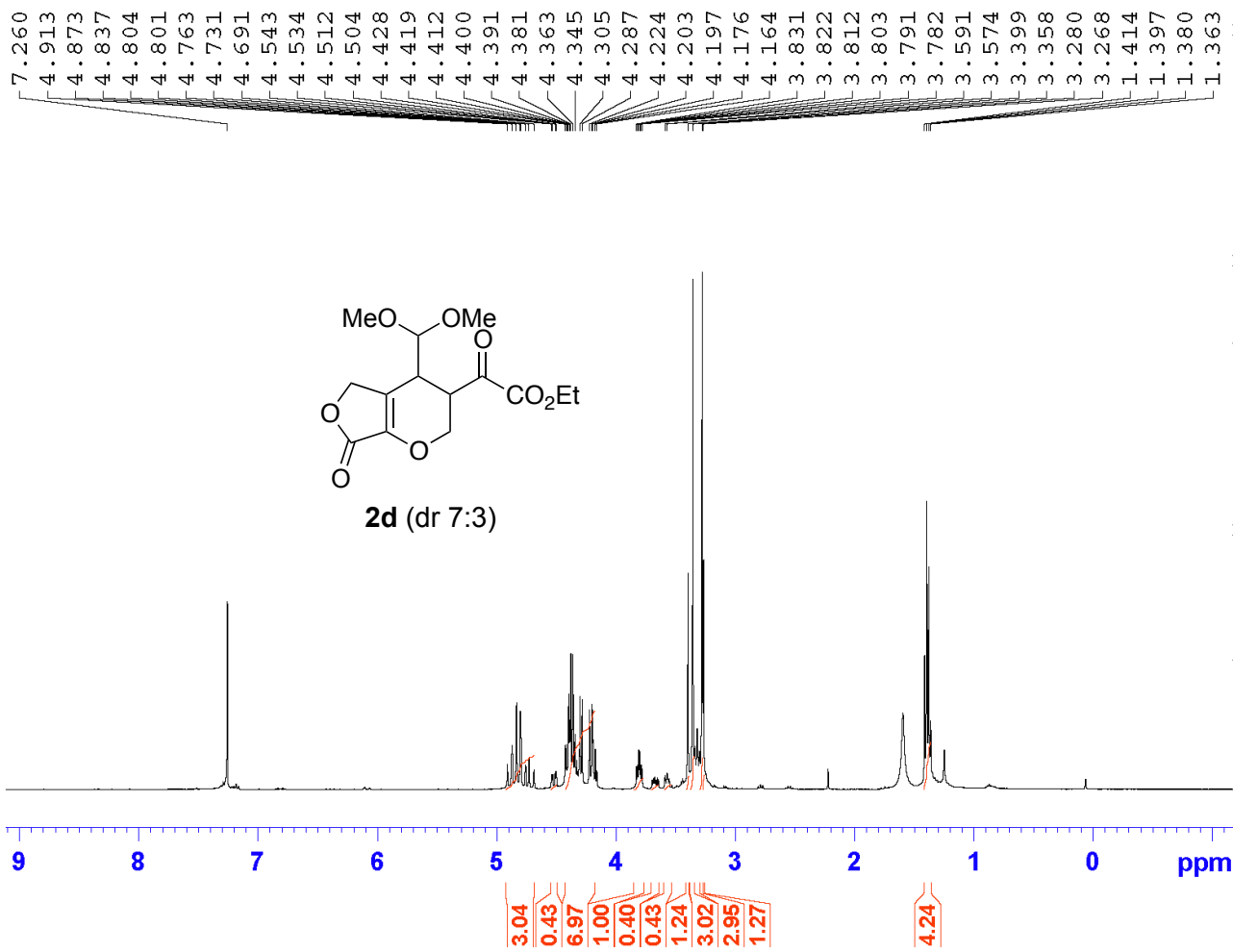
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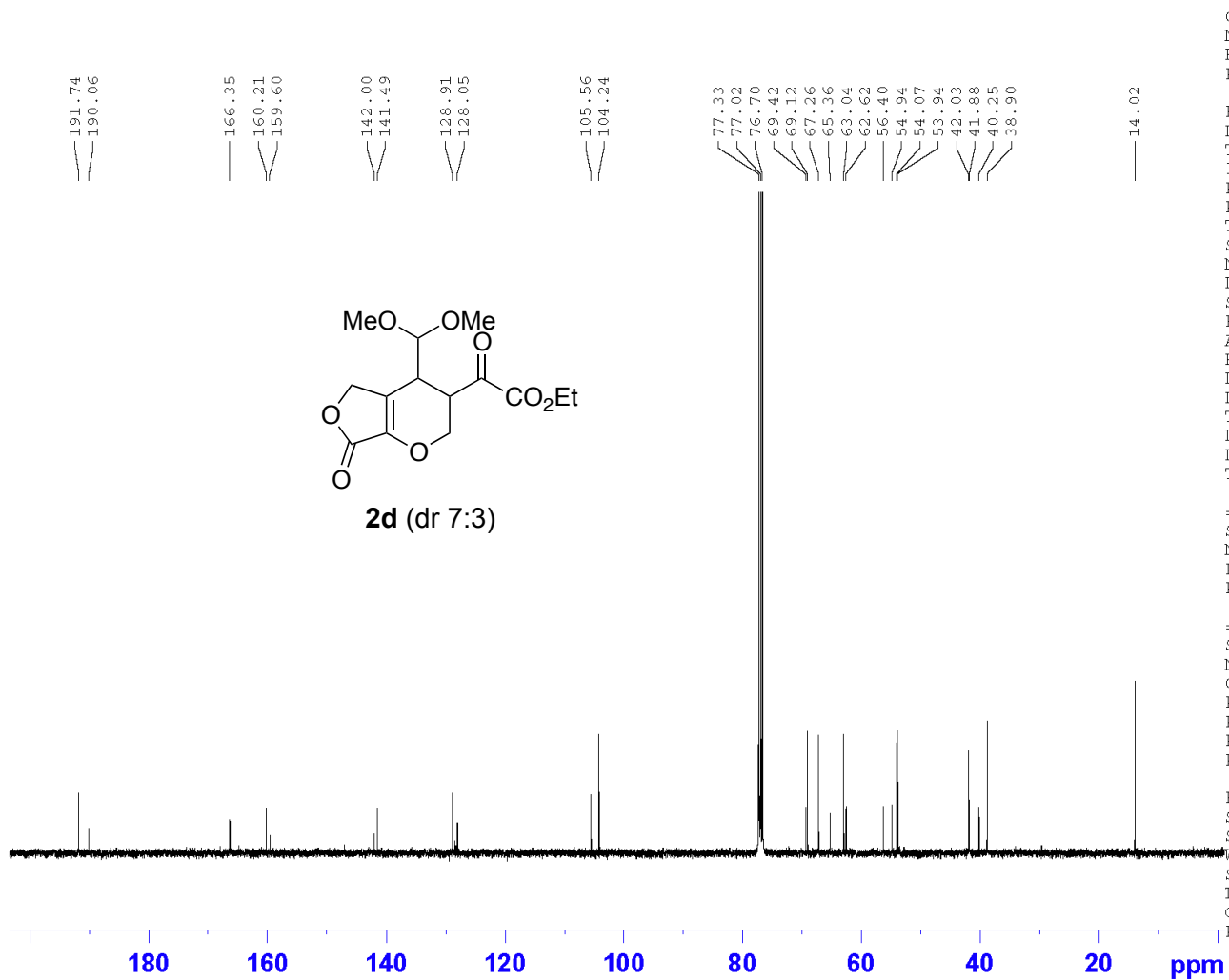
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SFO1        400.1324710 MHz
NUC1         1H
P1          15.00 usec
PLW1        8.00000000 W

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

```



Current Data Parameters
 NAME Aug18-2014
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140818
 Time_ 16.02
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1600
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.7 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

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

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

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

8.201
8.179

7.361
7.339
7.260

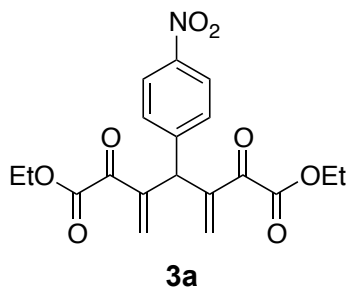
6.478

5.955

5.489

4.385
4.368
4.350
4.332

1.379
1.361
1.343

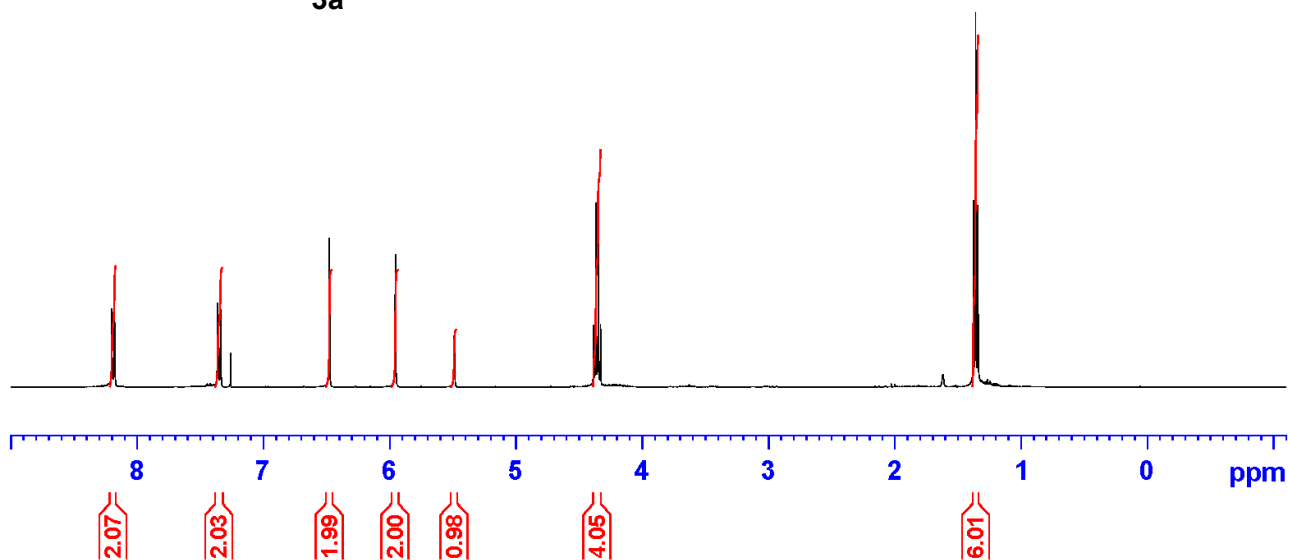


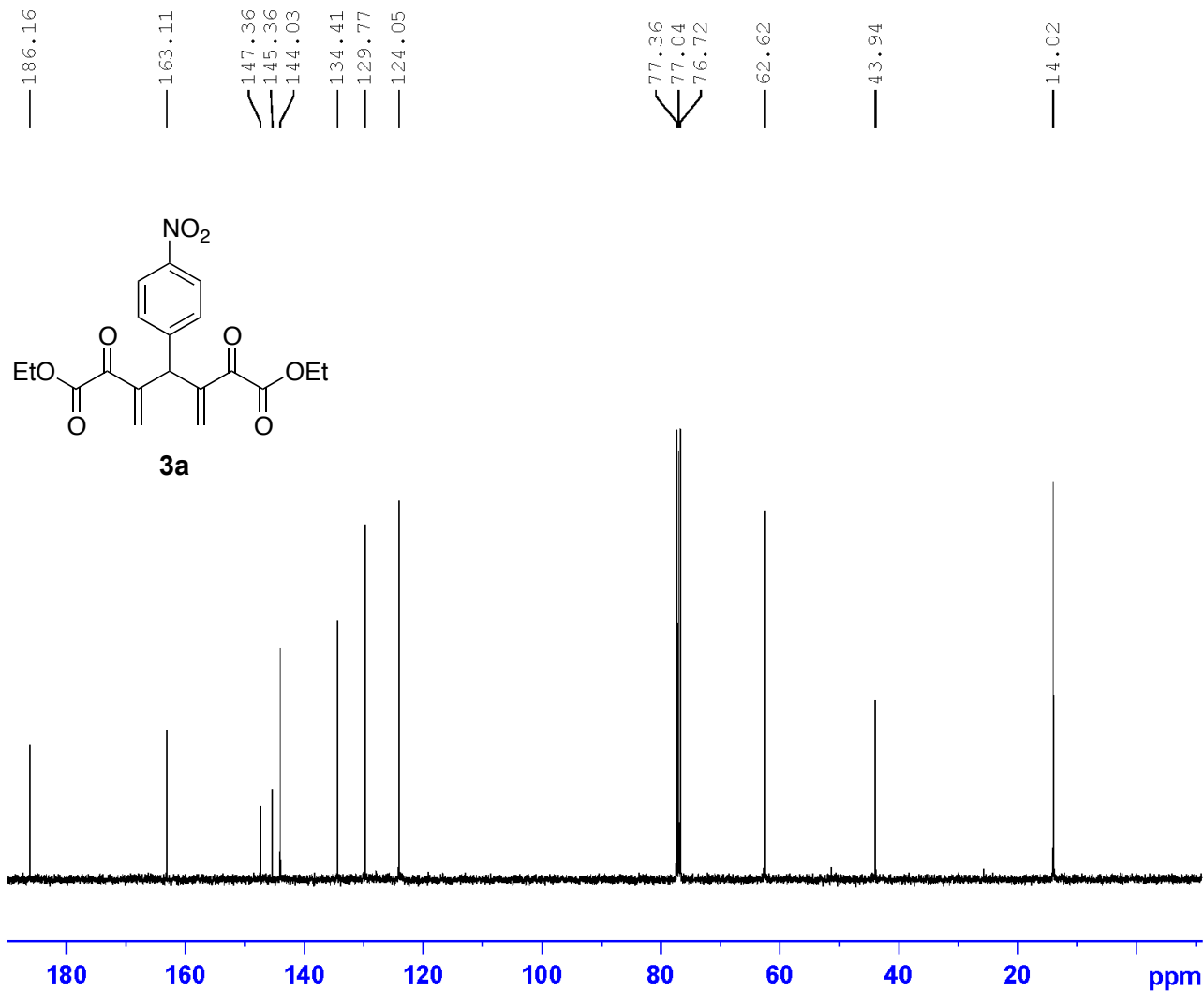
Current Data Parameters
NAME FT-RD-96B
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170303
Time_ 16.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 31.13
DW 62.400 usec
DE 6.50 usec
TE 298.9 K
D1 1.00000000 sec
TD0 1

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

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





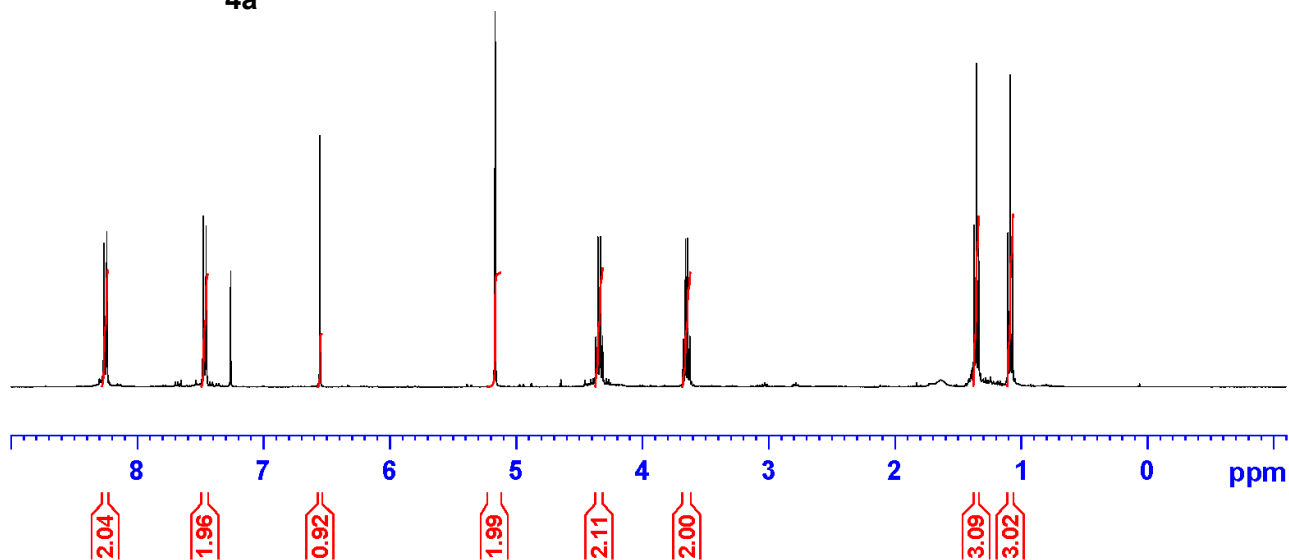
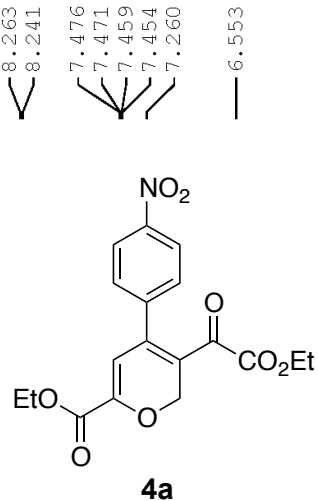
Current Data Parameters
 NAME FT-RD-96B
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170303
 Time_ 17.08
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 2
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.4 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 72.00000000 W

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

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

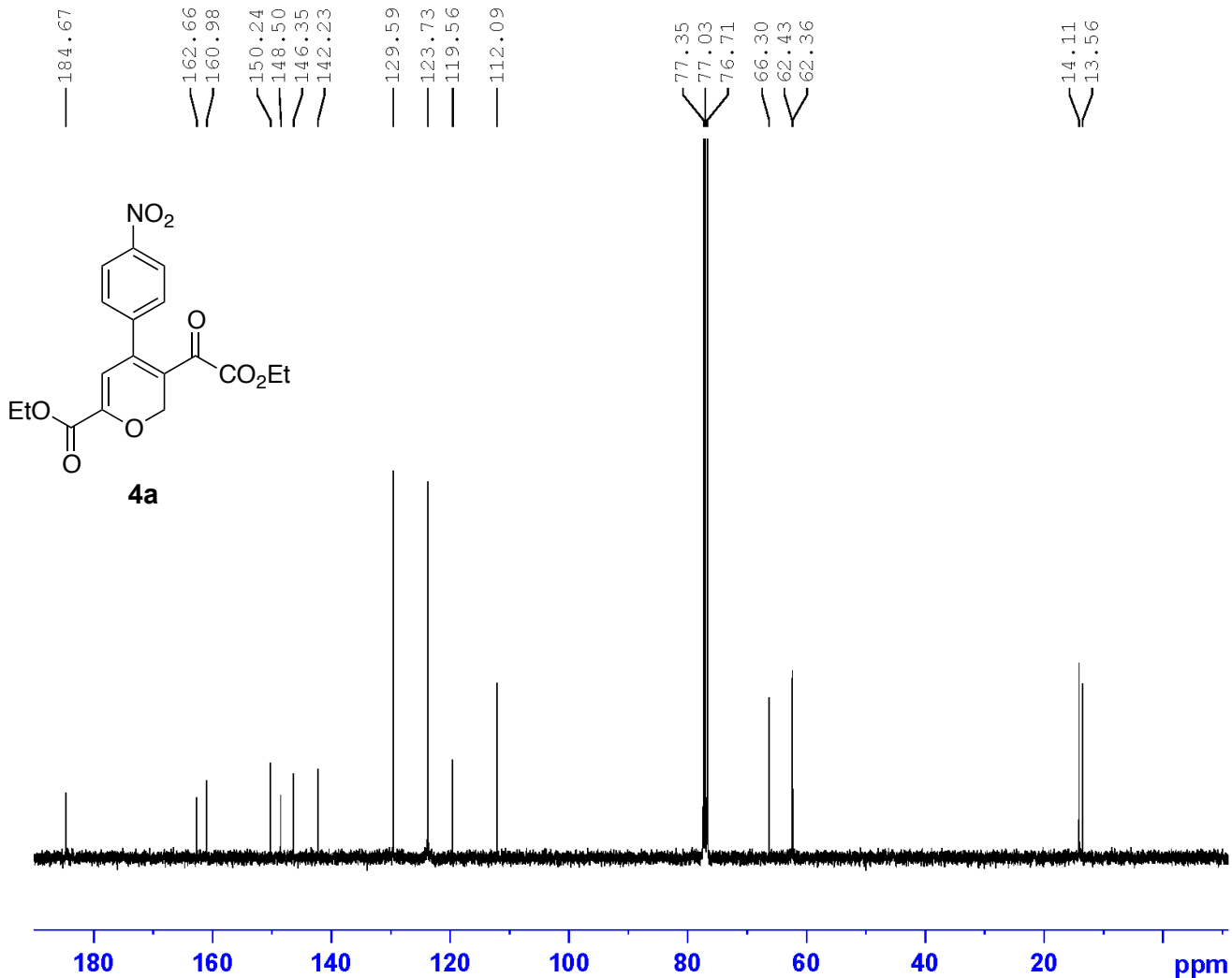


Current Data Parameters
 NAME FT-RD-96A
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date 20170303
 Time 16.16
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 54.59
 DW 62.400 usec
 DE 6.50 usec
 TE 298.8 K
 D1 1.00000000 sec
 TD0 1

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

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



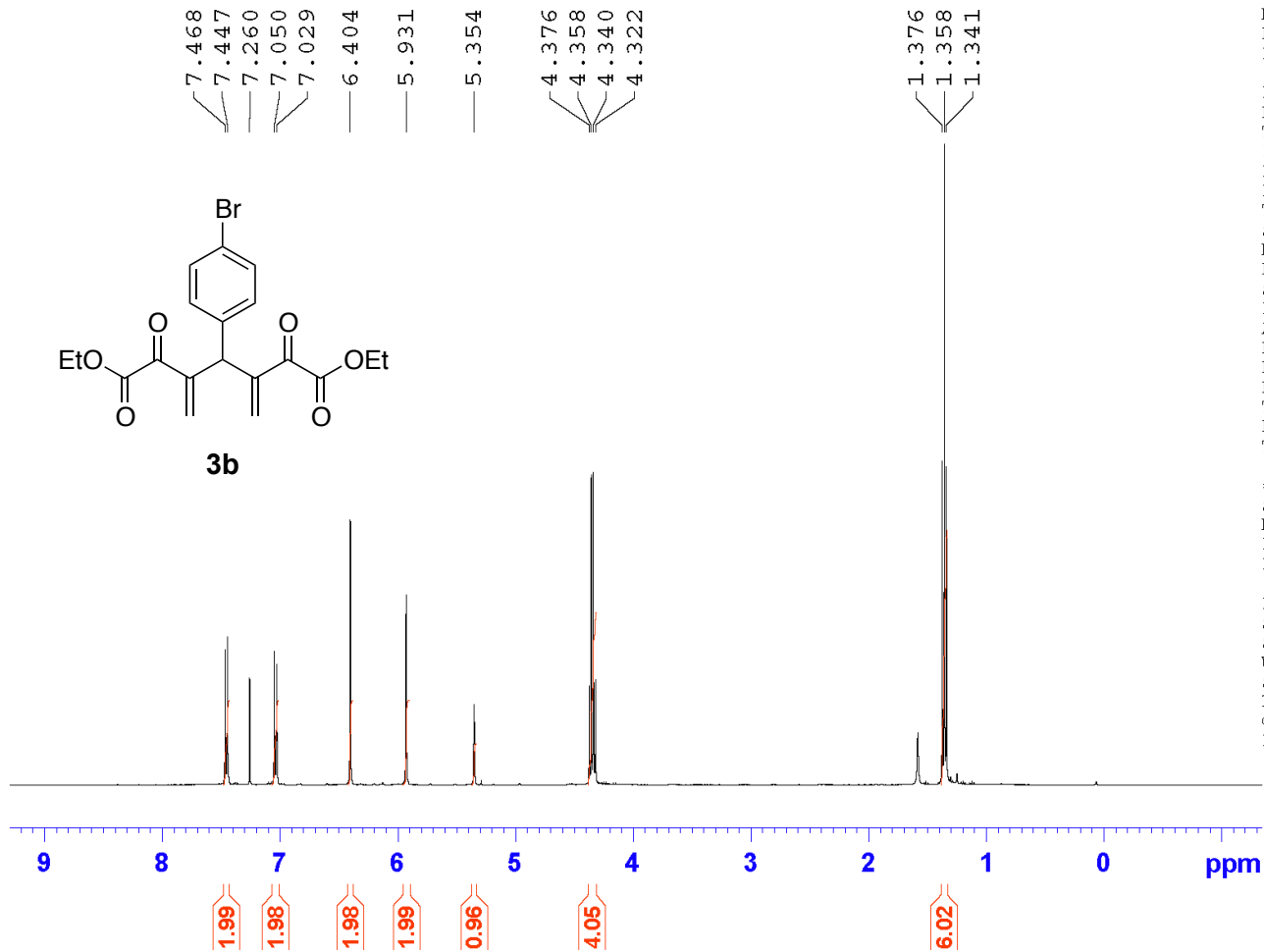
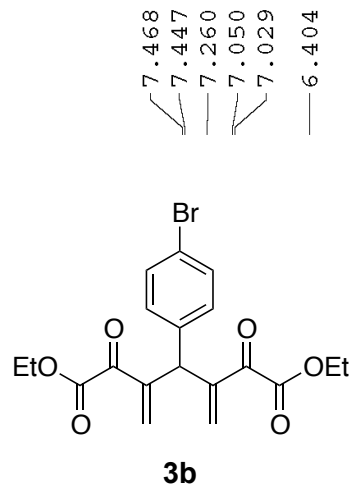
Current Data Parameters
 NAME FT-RD-96A
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date 20170303
 Time 16.31
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 2
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.4 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 72.00000000 W

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

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

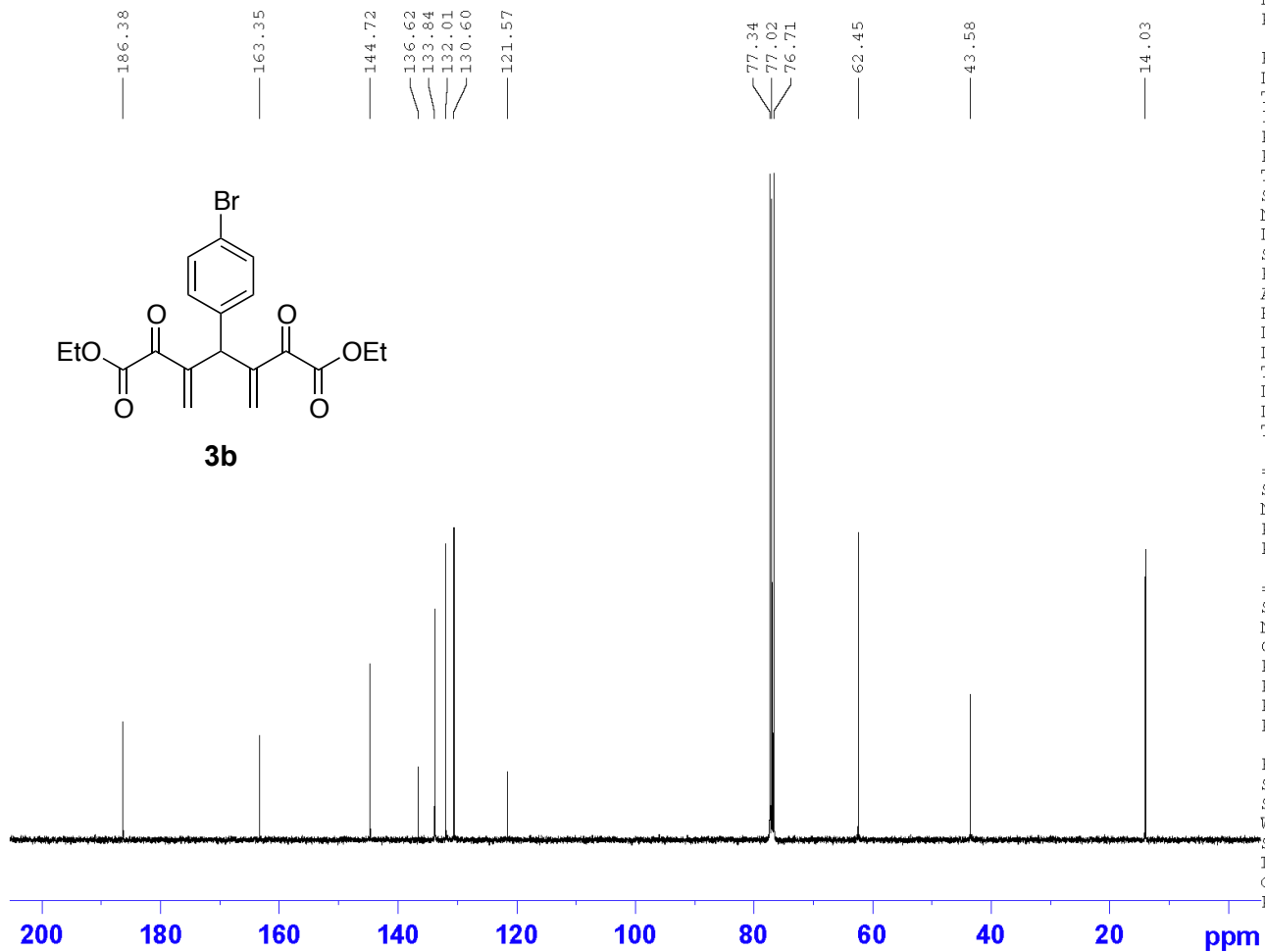


Current Data Parameters
 NAME Dec08-2014
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20141208
 Time_ 11.39
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 54.59
 DW 62.400 usec
 DE 6.50 usec
 TE 298.5 K
 D1 1.00000000 sec
 TD0 1

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

F2 - Processing parameters
 SI 65536
 SF 400.1300097 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



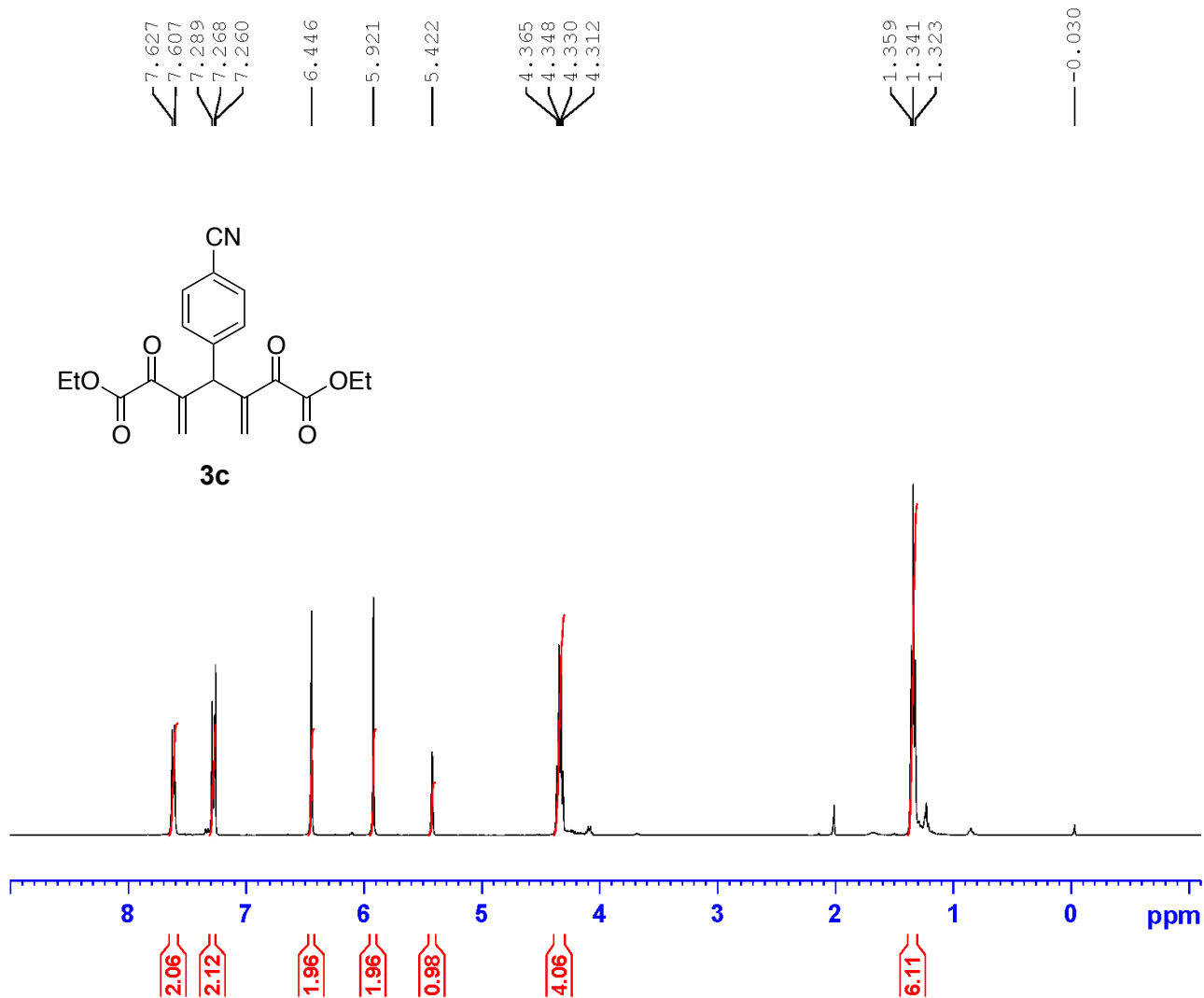
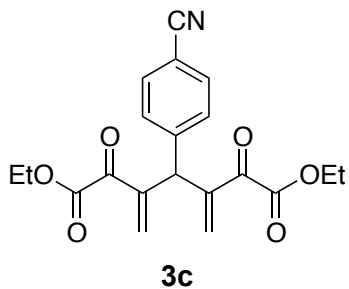
Current Data Parameters
 NAME Dec08-2014
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20141208
 Time_ 12.41
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.5 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

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

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

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

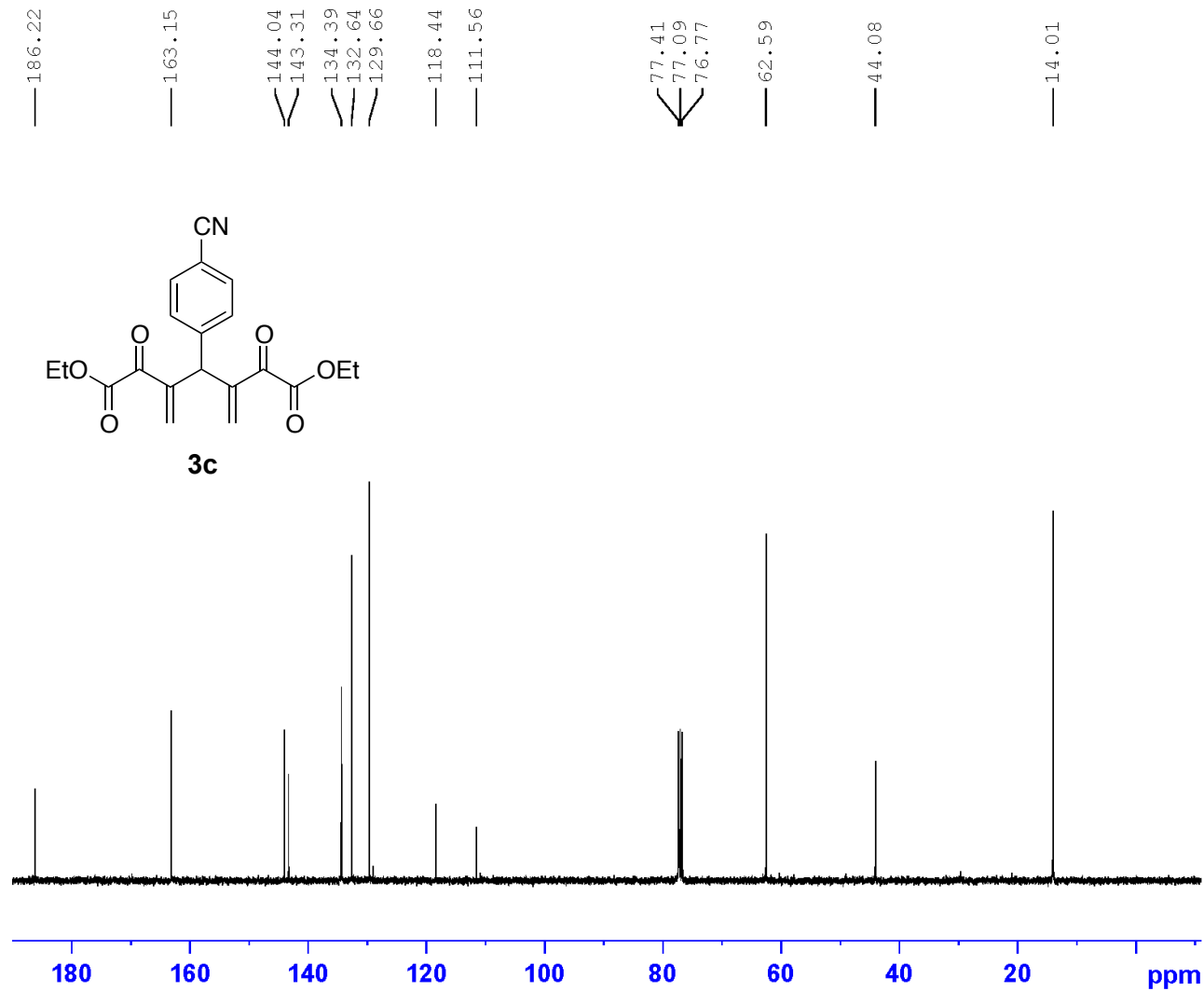


Current Data Parameters
 NAME FT-RD-257-A
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20171115
 Time_ 17.42
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 31.13
 DW 62.400 usec
 DE 6.50 usec
 TE 297.8 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 7.5000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300097 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



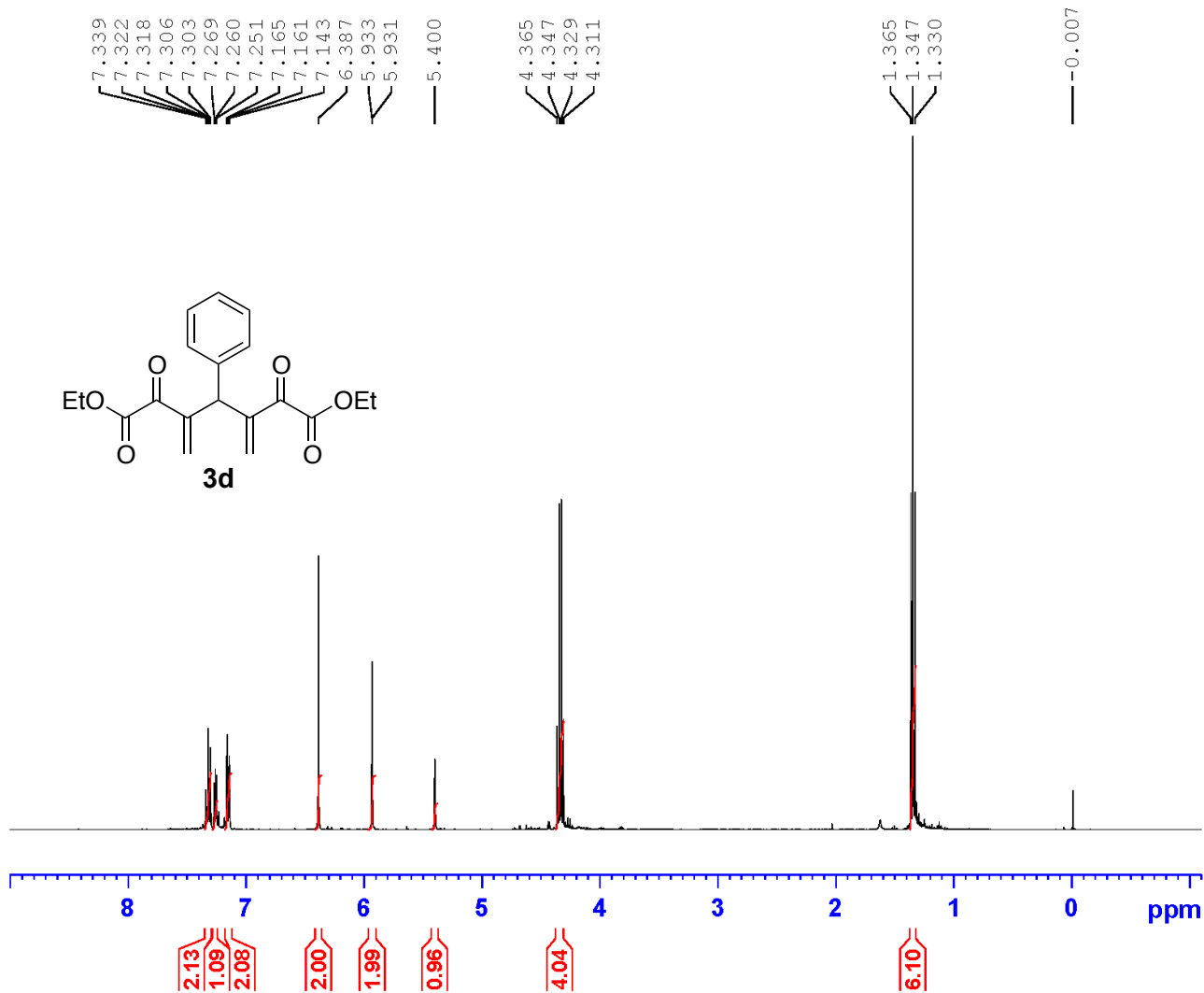
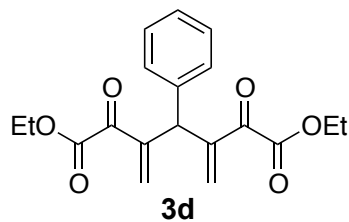
Current Data Parameters
 NAME FT-RD-257-A
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date 20171115
 Time 17.50
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 128
 DS 2
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 298.3 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 72.00000000 W

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

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

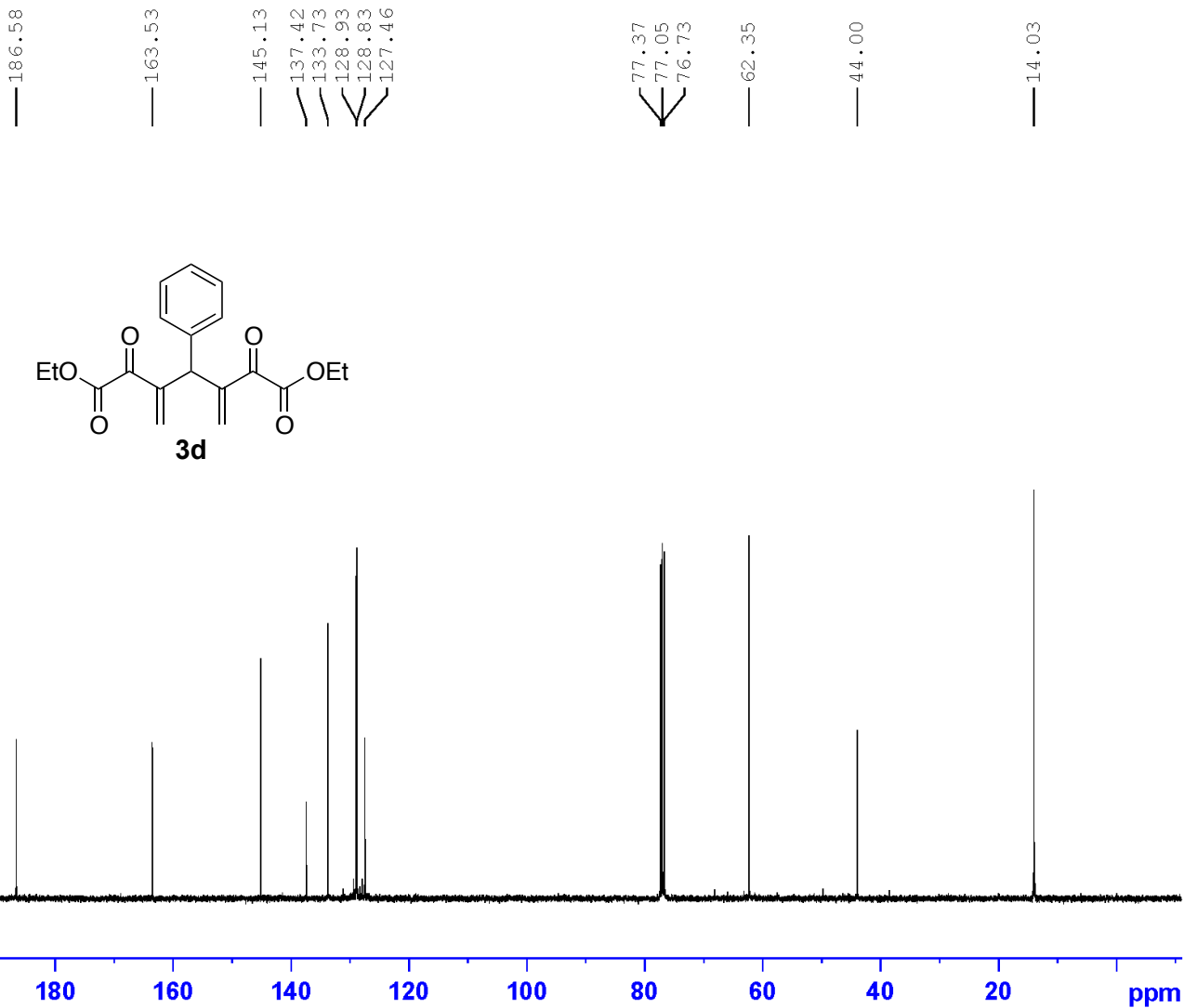


Current Data Parameters
 NAME FT-RD-256-A
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20171115
 Time_ 9.51
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 31.13
 DW 62.400 usec
 DE 6.50 usec
 TE 297.7 K
 D1 1.00000000 sec
 TD0 1

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

F2 - Processing parameters
 SI 65536
 SF 400.1300097 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



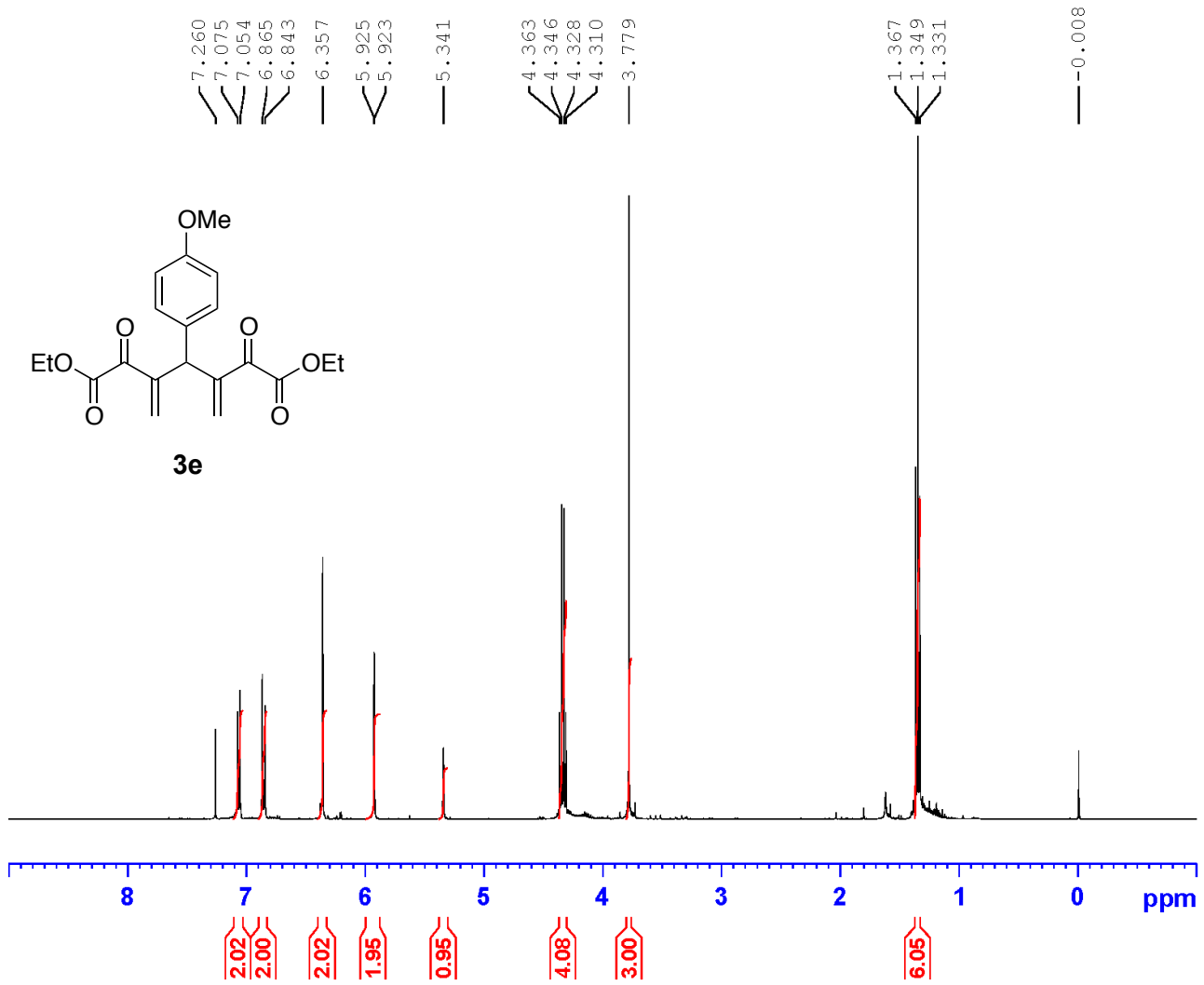
Current Data Parameters
 NAME FT-RD-256-A
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20171115
 Time_ 10.07
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 2
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 298.3 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 72.00000000 W

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

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

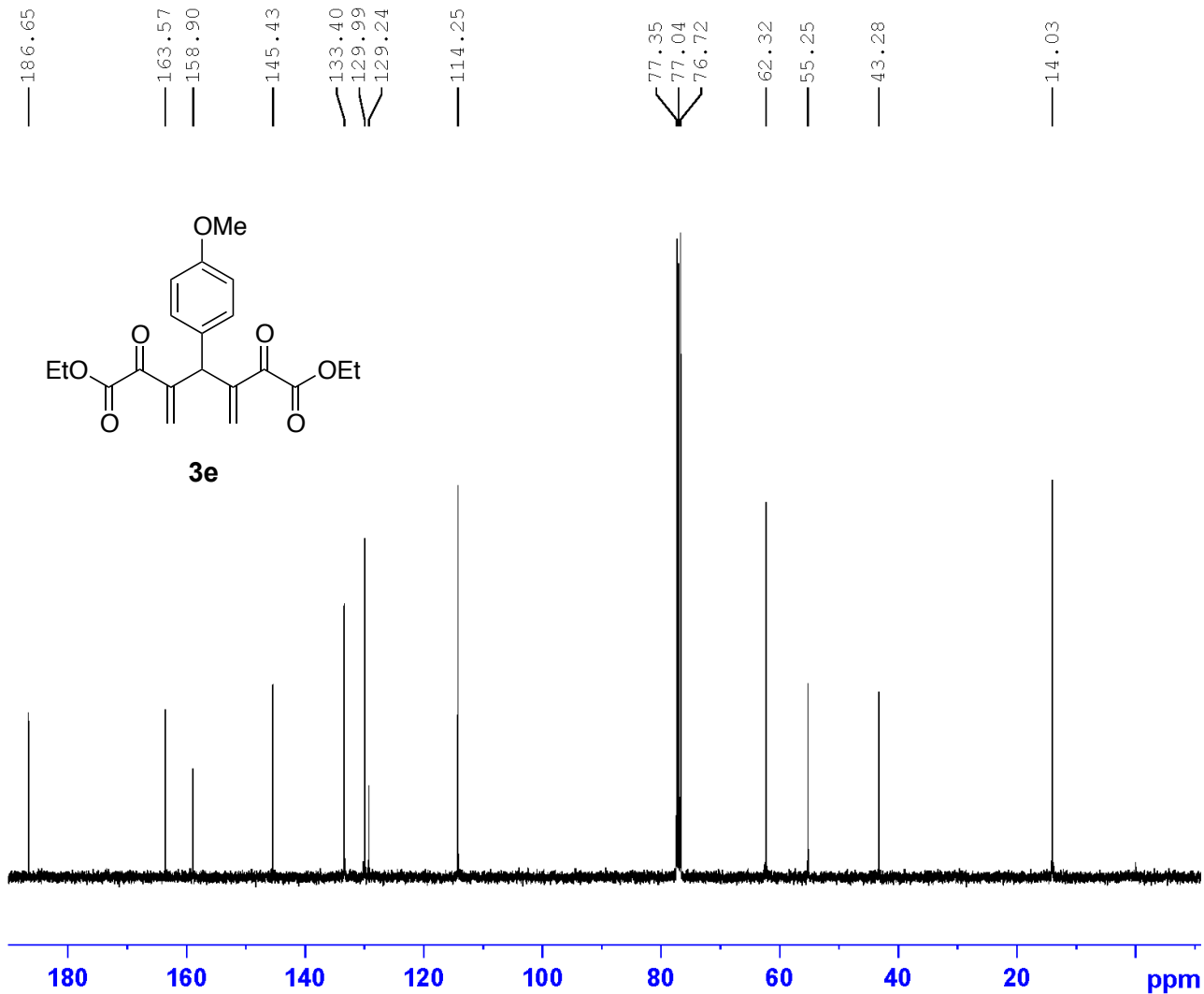


Current Data Parameters
 NAME FT-RD-255-A
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20171115
 Time_ 10.29
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 31.13
 DW 62.400 usec
 DE 6.50 usec
 TE 297.8 K
 D1 1.00000000 sec
 TD0 1

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

F2 - Processing parameters
 SI 65536
 SF 400.1300097 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME FT-RD-255-A
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20171115
 Time_ 10.44
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl₃
 NS 256
 DS 2
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 298.4 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 72.00000000 W

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

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40