

Supporting Information

Reactions of Pyruvates: Organocatalytic Synthesis of functionalized dihydropyrans in one pot and further transformations to functionalized carbocycles and heterocycles

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1. General -----	S1
2. Synthesis of Dihydropyrans 1 in One Pot from Pyruvates and Aldehydes -----	S2
3. Synthesis of Dihydropyrans from Pyruvates and β,γ -Unsaturated α -Ketoesters -----	S10
4. Synthesis of Cyclohexane Derivatives 7 from 1 -----	S11
5. Synthesis of 8 and 9 from 7 -----	S16
6. Synthesis of Fluorodihydropyrans 10 from 1 -----	S17
7. Synthesis of 11-15 from 1 -----	S19
8. Synthesis of Pyridines 16 from 1 -----	S21
9. Analysis of Reversibility between Cyclic and Acyclic, Linear Forms of 1aa -----	S25
10. Plausible Mechanism for the Formation of 1 from Pyruvates and Aldehydes -----	S25
11. X-Ray Crystal Structures of 1ab and 7aa -----	S26
12. References -----	S28
13. NMR Spectra -----	S29

1. General

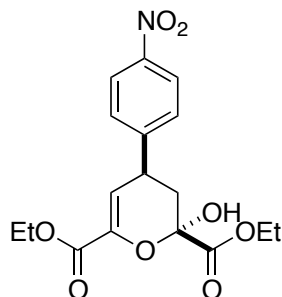
For thin layer chromatography (TLC), Merck Silica gel 60 F254 aluminum sheets were used. Flash column chromatography was performed using Merck silica gel 60 (230-400 mesh). ^1H NMR and ^{13}C NMR were recorded on a Bruker Avance 400. Proton chemical shifts are reported in ppm downfield from tetramethylsilane or from the residual solvent as internal standard in CDCl_3 (δ 7.26 ppm), in CD_3OD (δ 3.31 ppm), and in $(\text{CD}_3)_2\text{SO}$ (δ 2.50 ppm). Carbon chemical shifts were internally referenced to the deuterated solvent signals in CDCl_3 (δ 77.0 ppm), in CD_3OD (δ 49.0 ppm), and in $(\text{CD}_3)_2\text{SO}$ (δ 39.5 ppm). High-resolution mass spectra were recorded on a Thermo Scientific LTQ Orbitrap ESI ion trap mass spectrometer. IR spectra were recorded on a Jasco FT IR-4100 spectrometer.

2. Synthesis of Dihydropyrans **1** in One Pot from Pyruvates and Aldehydes

General Procedure for the Synthesis of Dihydropyran Derivatives **1** (Table 2)

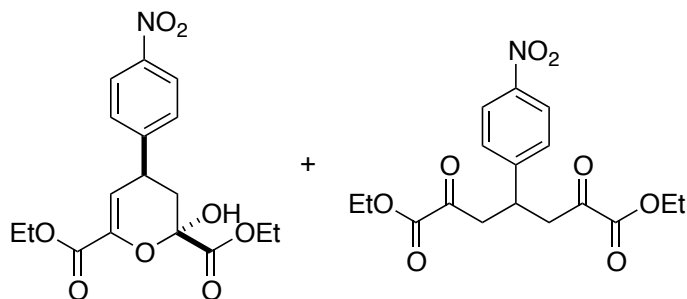
To a solution of aldehyde (1.0 mmol), and ethyl pyruvate (or methyl or benzyl pyruvate) (3.0 mmol) in CH₃CN (1.0 mL), (*S*)- β -proline (23 mg, 0.2 mmol) was added at the room temperature (25 °C) and the mixture was stirred at the same temperature for 24 h. The mixture was poured into saturated aqueous NH₄Cl solution (5 mL) and extracted with EtOAc. Organic layers were combined, dried over Na₂SO₄, filtered, concentrated, and purified by flash column chromatography (hexane/EtOAc) to afford **1**. (*S*)- β -Proline was used as the catalyst for the reaction because homochiral β -proline is more easily accessed than the racemic version.

(2*S**,4*S**)-Diethyl 2-hydroxy-4-(4-nitrophenyl)-3,4-dihydro-2*H*-pyran-2,6-dicarboxylate (**1aa**)



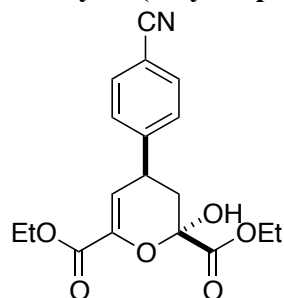
Flash column chromatography (hexane/EtOAc = 7:3); colorless solid. ¹H NMR (400 MHz, CDCl₃): δ 8.21 (d, *J* = 8.8 Hz, 2H), 7.45 (d, *J* = 8.8 Hz, 2H), 6.24 (dd, *J* = 2.4 Hz, 1.6 Hz, 1H), 4.60 (d, *J* = 2.0 Hz, 1H), 4.39-4.23 (m, 4H), 3.98 (ddd, *J* = 12.4 Hz, 6.0 Hz, 2.4 Hz, 1H), 2.22 (ddd, *J* = 13.2 Hz, 6.0 Hz, 1.6 Hz, 1H), 2.11 (ddd, *J* = 13.2 Hz, 12.4 Hz, 1.6 Hz, 1H), 1.33 (t, *J* = 7.2 Hz, 3H), 1.31 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 168.7, 161.9, 149.9, 147.2, 141.9, 128.5, 124.1, 113.0, 94.3, 63.6, 61.6, 35.3, 34.7, 14.1, 13.9. ESI-HRMS: calcd for C₁₇H₂₀O₈N ([M+H]⁺) 366.1183, found 366.1190.

Compound **1aa**; cyclic form/linear form = 4:1



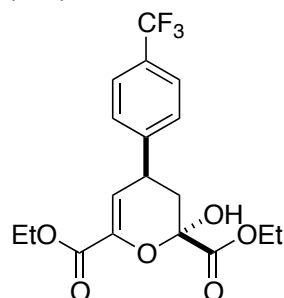
Colorless solid. *donates the linear form. ¹H NMR (400 MHz, CDCl₃): δ 8.21 (d, *J* = 8.8 Hz, 2H x 4/5), 8.15* (d, *J* = 8.8 Hz, 2H x 1/5), 7.45 (d, *J* = 8.8 Hz, 2H), 6.24 (dd, *J* = 2.4 Hz, 1.6 Hz, 1H x 4/5), 4.64 (d, *J* = 1.7 Hz, 1H x 4/5), 4.39-4.23 (m, 4H), 4.01-3.88 (m, 1H), 3.37-3.22* (m, 4H x 1/5), 2.22 (ddd, *J* = 13.2 Hz, 6.0 Hz, 1.6 Hz, 1H x 4/5), 2.11 (ddd, *J* = 13.2 Hz, 12.4 Hz, 1.6 Hz, 1H x 4/5), 1.36-1.30 (m, 6H). ¹³C NMR (100 MHz, CDCl₃): δ 191.6*, 168.7, 161.9, 160.3*, 150.0, 149.7*, 147.2, 141.9, 128.7*, 128.5, 124.1, 124.0*, 113.0, 94.3, 63.6, 62.8*, 61.5, 44.6*, 35.3, 34.79, 34.73*, 14.1, 13.95, 13.92*.

Diethyl 4-(4-cyanophenyl)-2-hydroxy-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1ba)



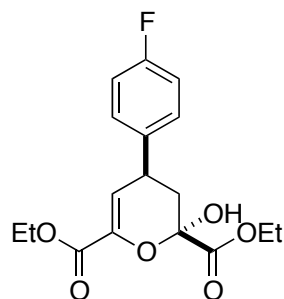
Flash column chromatography (hexane/EtOAc = 3:1); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 7.65 (d, J = 8.3 Hz, 2H), 7.39 (d, J = 8.3 Hz, 2H), 6.22 (dd, J = 2.0 Hz, 1.6 Hz, 1H), 4.64 (d, J = 2.0 Hz, 1H), 4.37-4.23 (m, 4H), 3.92 (ddd, J = 12.4 Hz, 6.0 Hz, 2.0 Hz 1H), 2.20 (ddd, J = 13.3 Hz, 6.0 Hz, 1.6 Hz, 1H), 2.09 (ddd, J = 13.3 Hz, 12.4 Hz, 1.6 Hz, 1H) 1.33 (t, J = 7.2 Hz, 3H), 1.31 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 168.7, 162.0, 147.9, 141.8, 132.7, 128.5, 118.6, 113.2, 111.1, 94.4, 63.2, 61.6, 35.3, 34.9, 14.1, 13.9. ESI-HRMS: calcd for $\text{C}_{18}\text{H}_{20}\text{O}_6\text{N}$ ($[\text{M}+\text{H}]^+$) 446.1285, found 446.1289.

Diethyl 2-hydroxy-4-(4-(trifluoromethyl)phenyl)-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1ca)



Flash column chromatography (hexane/EtOAc = 4:1); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 7.61 (d, J = 8.0 Hz, 2H), 7.39 (d, J = 8.0 Hz, 2H), 6.26 (dd, J = 2.0 Hz, 1.6 Hz, 1H), 4.55 (brs, 1H), 4.39-4.22 (m, 4H), 3.92 (ddd J = 12.4 Hz, 6.4 Hz, 2.0 Hz, 1H), 2.20 (ddd J = 13.3 Hz, 6.4 Hz, 1.6 Hz, 1H), 2.16-2.08 (m, 1H), 1.33 (t, J = 7.3 Hz, 3H), 1.31 (t, J = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 168.8, 162.0, 146.5, 141.7, 129.5 (q, $J_{\text{C,F}}$ = 33 Hz), 128.0, 125.8 (q, $J_{\text{C,F}}$ = 3.6 Hz), 124.0 (q, $J_{\text{C,F}}$ = 270 Hz), 113.9, 94.4, 63.2, 61.4, 35.5, 34.6, 14.1, 13.9. ESI-HRMS: calcd for $\text{C}_{18}\text{H}_{19}\text{F}_3\text{O}_6\text{Na}$ ($[\text{M}+\text{Na}]^+$) 411.1026, found 411.1028.

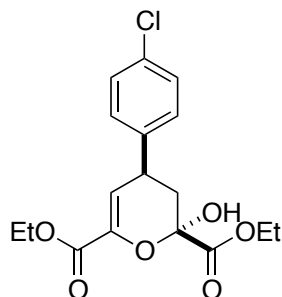
Diethyl 4-(4-fluorophenyl)-2-hydroxy-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1da)



Flash column chromatography (hexane/EtOAc = 3:1); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 7.25-7.21 (m, 2H), 7.06-7.01 (m, 2H), 6.26-6.24 (m, 1H), 4.62 (d, J = 2.0 Hz, 1H),

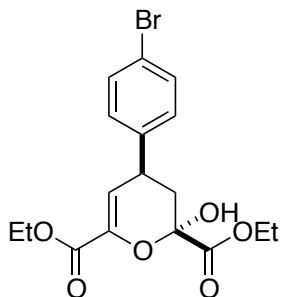
4.39-4.22 (m, 4H), 3.83 (ddd $J = 12.4$ Hz, 6.2 Hz, 2.1 Hz, 1H), 2.18 (ddd $J = 13.3$ Hz, 6.2 Hz, 1.6 Hz, 1H) 2.13-2.06 (m, 1H) 1.33 (t, $J = 7.1$ Hz, 3H), 1.31 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 169.0, 162.2, 161.9 (d, $J = 243$ Hz), 141.3, 138.1 (d, $J_{\text{C,F}} = 3$ Hz), 129.1 (d, $J_{\text{C,F}} = 8$ Hz), 115.6 (d, $J_{\text{C,F}} = 22$ Hz), 115.0, 94.6, 63.1, 61.4, 35.8, 34.0, 14.1, 13.9. ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{19}\text{FO}_6\text{Na}$ ($[\text{M}+\text{Na}]^+$) 361.1058, found 361.1059.

Diethyl 4-(4-chlorophenyl)-2-hydroxy-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1ea)



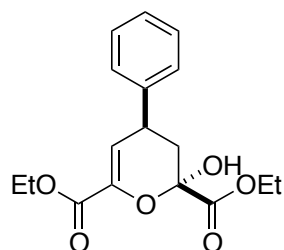
Flash column chromatography (hexane/EtOAc = 3:1); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 7.31 (d, $J = 8.4$ Hz, 2H), 7.20 (d, $J = 8.4$ Hz, 2H), 6.23 (dd, $J = 2.2$ Hz, 1.6 Hz, 1H), 4.54 (d, $J = 1.6$ Hz, 1H), 4.38-4.21 (m, 4H), 3.82 (ddd $J = 12.4$ Hz, 6.2 Hz, 2.2 Hz, 1H), 2.17 (ddd $J = 13.2$ Hz, 6.2 Hz, 1.6 Hz, 1H), 2.09 (ddd, $J = 13.2$ Hz, 12.4 Hz, 1.6 Hz, 1H), 1.32 (t, $J = 7.2$ Hz, 3H), 1.30 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 168.9, 162.1, 141.4, 140.9, 132.9, 128.99, 128.97, 114.5, 94.5, 63.1, 61.4, 35.6, 34.1, 14.1, 13.9. IR (CHCl_3): 3442, 2982, 1731, 1650, 1492, 1150, 830, 763 cm^{-1} . ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{19}\text{ClO}_6\text{Na}$ ($[\text{M}+\text{Na}]^+$) 377.0762, found 377.0764.

Diethyl 4-(4-bromophenyl)-2-hydroxy-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1fa)



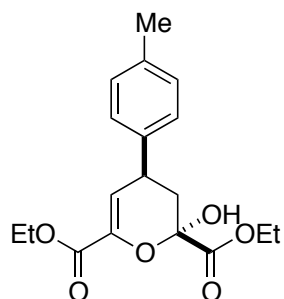
Flash column chromatography (hexane/EtOAc = 4:1); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 7.47 (d, $J = 8.4$ Hz, 2H), 7.15 (d, $J = 8.4$ Hz, 2H), 6.23 (dd, $J = 1.6$ Hz, 2.0 Hz, 1H), 4.56 (d, $J = 2.0$ Hz, 1H), 4.39-4.22 (m, 4H), 3.81 (ddd $J = 12.4$ Hz, 6.4 Hz, 2.0 Hz, 1H), 2.17 (ddd $J = 13.6$ Hz, 6.4 Hz, 1.6 Hz, 1H), 2.09 (ddd, $J = 13.6$ Hz, 12.4 Hz, 2.0 Hz, 1H), 1.33 (t, $J = 7.2$ Hz, 3H), 1.31 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 168.9, 162.1, 141.4, 141.4, 131.9, 129.3, 120.9, 114.4, 94.5, 63.1, 61.4, 35.5, 34.2, 14.1, 13.9. ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{19}\text{BrO}_6\text{Na}$ ($[\text{M}+\text{Na}]^+$) 421.0257, found 421.0254.

Diethyl 2-hydroxy-4-phenyl-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1ga)



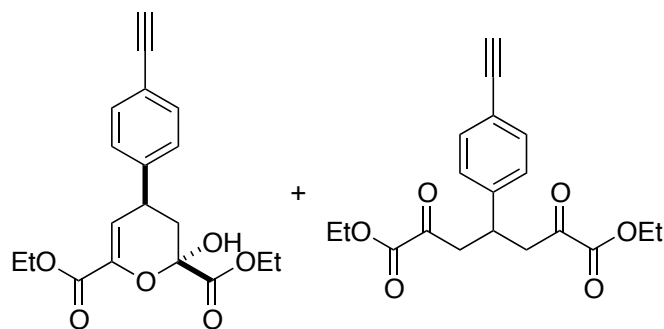
Flash column chromatography (hexane/EtOAc = 4:1); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 7.38-7.24 (m, 5H), 6.33-6.31 (m, 1H), 4.69 (brs, 1H), 4.39-4.23 (m, 4H), 3.86 (ddd J = 12.2 Hz, 6.2 Hz, 2.1 Hz, 1H), 2.50-2.11 (m, 2H), 1.33 (t, J = 7.0 Hz, 3H), 1.31 (t, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 169.1, 162.3, 142.4, 141.2, 128.8, 127.6, 127.1, 115.3, 94.6, 63.0, 61.3, 35.7, 34.6, 14.1, 13.9. ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{20}\text{O}_6\text{Na}$ ($[\text{M}+\text{Na}]^+$) 343.1152, found 343.1154.

Diethyl 2-hydroxy-4-(p-tolyl)-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1ha)



Flash column chromatography (hexane/EtOAc = 4:1); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 7.16 (s, 4H), 6.29 (dd, J = 2.0 Hz, 1.6 Hz, 1H), 4.50 (d, J = 1.6 Hz, 1H), 4.19-4.38 (m, 4H), 3.81 (ddd J = 12.0 Hz, 6.8 Hz, 2.0 Hz, 1H), 2.34 (s, 3H), 2.18 (ddd, 13.2 Hz, 6.8 Hz, 1.6 Hz, 1H), 2.16-2.09 (m, 1H), 1.32 (t, J = 7.2 Hz, 3H), 1.30 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 169.2, 162.3, 141.1, 139.3, 136.7, 129.5, 127.4, 115.6, 94.6, 63.0, 61.3, 35.7, 34.2, 21.0, 14.1, 13.9. IR (CHCl_3): 3436, 2983, 1730, 1649, 1151, 758, 701 cm^{-1} . ESI-HRMS: calcd for $\text{C}_{18}\text{H}_{23}\text{O}_6$ ($[\text{M}+\text{H}]^+$) 335.1489, found 335.1499.

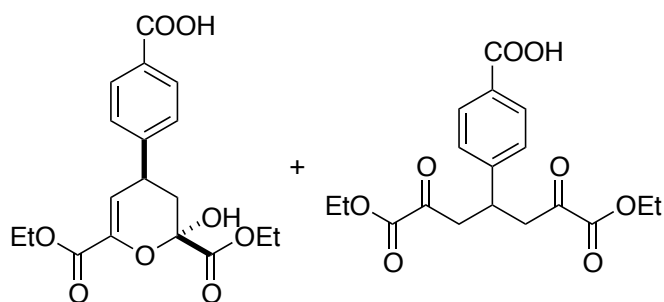
Diethyl 4-(4-ethynylphenyl)-2-hydroxy-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1ia)



Flash column chromatography (hexane/EtOAc = 1:3); cyclic form/linear form = 6:1; pale yellow gum. ^1H NMR (400 MHz, CDCl_3): δ cyclic form: 7.43 (d, J = 8.0 Hz, 2H), 7.20 (d, J = 8.0 Hz, 2H), 6.22 (s, Hz, 1H), 4.78 (d, J = 1.1 Hz, 1H), 4.43-4.17 (m, 4H), 3.88-3.79 (m, 1H), 3.06 (s, 1H),

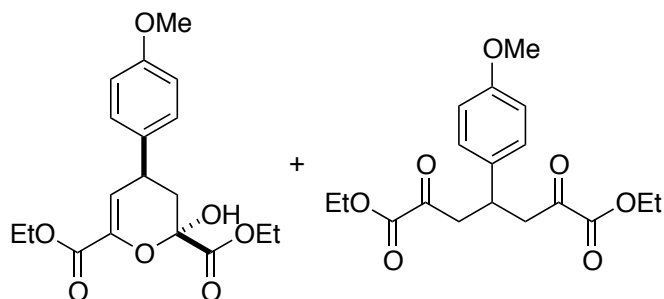
2.00-2.19 (m, 2H), 1.23-1.34 (m, 6H); linear form 3.30-3.10 (m, 4H, 2 x CH₂). ¹³C NMR (100 MHz, CDCl₃): δ cyclic form: 168.9, 162.1, 143.2, 141.4, 132.5, 127.6, 120.9, 114.4, 94.6, 83.2, 77.4, 63.0, 61.3, 35.5, 34.6, 14.0, 13.9; linear form (peaks distinguished from those of the cyclic form): 191.7, 175.3, 72.1, 62.6, 62.1, 48.1, 26.6, 14.1. IR (CHCl₃): 3456, 3277, 2983, 1729, 1260, 1649, 1150, 1055, 838, 767 cm⁻¹. ESI-HRMS: calcd for C₁₉H₂₀O₆Na ([M+Na]⁺) 367.1152, found 367.1154.

4-(2,6-Bis(ethoxycarbonyl)-2-hydroxy-3,4-dihydro-2H-pyran-4-yl)benzoic acid (1ja)



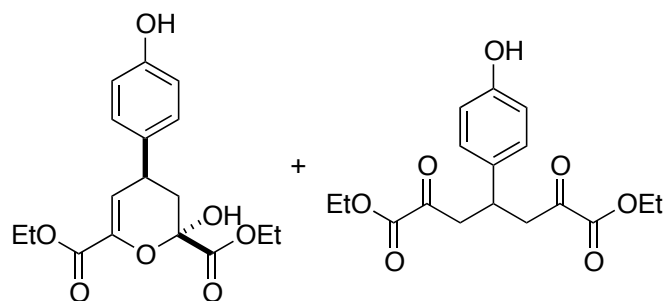
Flash column chromatography (EtOAc); cyclic form/linear form = 2:1; pale yellow solid. ¹H NMR (400 MHz, CDCl₃) *donates the linear form: δ 8.07 (d, *J* = 8.3 Hz, 2H x 2/3), 8.01* (d, *J* = 8.3 Hz, 2H x 1/3), 7.36 (d, *J* = 8.3 Hz, 2H), 6.27-6.26 (m, 1H x 2/3), 4.33-4.14 (m, 4H), 3.96-3.84 (m, 1H), 3.36-3.13* (m, 4H x 1/3), 2.27-2.06 (m, 2H x 2/3), 1.32-1.23 (m, 6H). ¹³C NMR (100 MHz, CDCl₃): δ cyclic form: 171.3, 169.0, 162.4, 160.4, 148.6, 148.4, 141.4, 130.7, 127.8, 127.7, 114.1, 94.7, 62.7, 61.6, 35.4, 34.8, 14.0, 13.9; linear form: 191.9 (CH₂C(=O)COOEt), 44.59 (CH₂C(=O)COOEt). ESI-HRMS: calcd for C₁₈H₂₀O₈ Na ([M+Na]⁺) 387.1050, found 387.1050.

Diethyl 2-hydroxy-4-(4-methoxyphenyl)-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1ka)



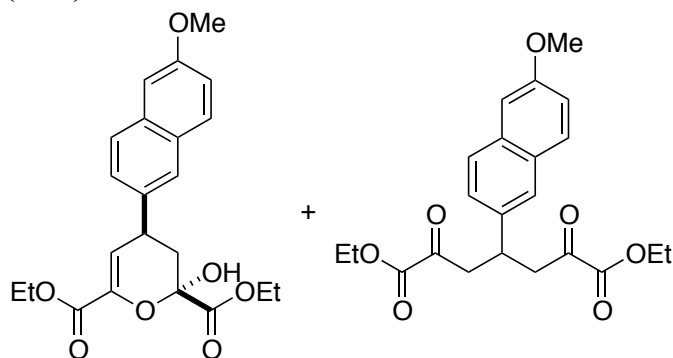
Flash column chromatography (hexane/EtOAc = 3:1); cyclic form/linear form = 5:1; yellow gum. ¹H NMR (400 MHz, CDCl₃) *donates the linear form: δ 7.18 (d, *J* = 8.6 Hz, 2H x 5/6), 7.15* (d, *J* = 8.6 Hz, 2H x 1/6), 6.88 (d, *J* = 8.6 Hz, 2H x 5/6), 6.81* (d, *J* = 8.6 Hz, 2H x 1/6), 6.28-6.26 (m, 1H x 5/6), 4.62 (brs, 1H x 5/6), 4.38-4.16 (m, 4H), 3.80 (s, 3H x 5/6), 3.76* (s, 3H x 1/6), 3.85-3.77 (m, 1H), 3.26* (dd, *J* = 17.6 Hz, 7.2 Hz, 2H x 1/6), 3.16* (dd, *J* = 17.6 Hz, 7.2 Hz, 2H x 1/6), 2.20-2.06 (m, 2H x 5/6), 1.40-1.25 (m, 6H). ¹³C NMR (100 MHz, CDCl₃): δ cyclic form: 169.2, 162.3, 158.6, 141.0, 134.4, 128.5, 115.7, 114.2, 94.6, 63.0, 61.3, 55.3, 35.8, 33.8, 30.9, 14.1, 13.9; linear form: 192.5 (CH₂C(=O)COOEt), 160.6 (CH₂C(=O)COOEt), 45.2 (CH₂C(=O)COOEt). ESI-HRMS: calcd for C₁₈H₂₂O₇Na ([M+Na]⁺) 373.1258, found 373.1268.

Diethyl 2-hydroxy-4-(4-hydroxyphenyl)-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1la)



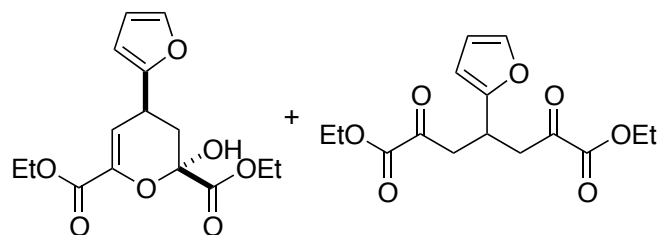
Flash column chromatography (hexane/EtOAc = 7:3); cyclic form/linear form = 4:1; yellow-brown gum. ^1H NMR (400 MHz, CDCl_3) *donates the linear form: δ 7.08 (d, J = 8.3 Hz, 2H), 6.82 (d, J = 8.5 Hz, 2H x 4/5), 6.76* (d, J = 8.6 Hz, 2H x 1/5), 6.27-6.26 (m, 1H x 4/5), 4.90 (brs, 1H x 4/5), 4.32-4.19 (m, 4H), 3.80-3.70 (m, 1H x 4/5), 3.24* (dd, J = 17.8 Hz, 7.2 Hz, 2H x 1/5), 3.14* (dd, J = 17.8 Hz, 7.2 Hz, 2H x 1/5), 2.20-2.03 (m, 2H x 4/5), 1.35-1.24 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ cyclic form: 169.2, 162.7, 154.9, 142.6, 140.8, 134.1, 128.7, 116.1, 115.7, 94.7, 63.0, 61.5, 35.8, 33.8, 14.1, 13.9; linear form: 192.6 ($\text{CH}_2\text{C}(=\text{O})\text{COOEt}$), 160.6 ($\text{CH}_2\text{C}(=\text{O})\text{COOEt}$), 45.2 ($\text{CH}_2\text{C}(=\text{O})\text{COOEt}$). ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{20}\text{O}_7\text{Na}$ ($[\text{M}+\text{Na}]^+$) 359.1101, found 359.1102.

Diethyl 2-hydroxy-4-(6-methoxynaphthalen-2-yl)-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1ma)



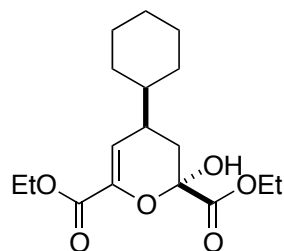
Flash column chromatography (hexane/EtOAc = 3:1); cyclic form/linear form = 4:1; yellow gum. ^1H NMR (400 MHz, CDCl_3) *donates the linear form: δ 7.75-7.11 (m, 6H), 6.39-6.35 (m, 1H x 4/5), 4.56 (brs, 1H x 4/5), 4.37-4.23 (m, 4H), 4.01-3.90 (m, 1H x 4/5), 3.92 (s, 3H x 4/5), 3.90* (s, 3H x 1/5), 3.38* (dd, J = 17.8 Hz, 7.0 Hz, 2H x 1/5), 3.26* (dd, J = 17.8 Hz, 7.0 Hz, 2H x 1/5), 2.30-2.13 (m, 2H x 4/5), 1.37-1.20 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ cyclic form: 169.2, 162.3, 157.6, 141.2, 137.4, 133.6, 129.1, 127.4, 126.3, 126.0, 125.9, 119.1, 115.5, 105.6, 94.6, 63.1, 61.3, 55.3, 35.7, 34.6, 14.1, 13.9; linear form: 192.4 ($\text{CH}_2\text{C}(=\text{O})\text{COOEt}$), 160.6 ($\text{CH}_2\text{C}(=\text{O})\text{COOEt}$), 45.0 ($\text{CH}_2\text{C}(=\text{O})\text{COOEt}$). ESI-HRMS: calcd for $\text{C}_{22}\text{H}_{25}\text{O}_7$ ($[\text{M}+\text{H}]^+$) 401.1594, found 401.1590.

Diethyl 4-(furan-2-yl)-2-hydroxy-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1na)



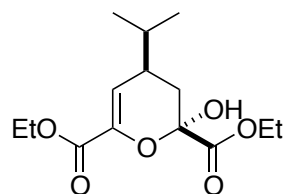
Flash column chromatography (hexane/EtOAc = 4:1); cyclic form/linear form = 3:1; brown gum. ^1H NMR (400 MHz, CDCl_3) *donates the linear form: δ 7.37-7.27 (m, 1H), 6.33-6.06 (m, 2H + 1H x 3/4), 4.57 (brs, 1H), 4.39-4.20 (m, 4H), 4.05-3.90 (m, 1H x 4/4), 3.32* (dd, J = 18.0 Hz, 6.8 Hz, 2H x 1/4), 3.18* (dd, J = 18.0 Hz, 6.8 Hz, 2H x 1/4), 2.27-2.24 (m, 2H x 3/4), 1.36-1.28 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ cyclic form: 168.9, 162.1, 154.6, 141.8, 141.5, 112.2, 110.2, 105.2, 94.4, 63.0, 61.3, 31.9, 28.4, 14.1, 13.9; linear form: 192.0 ($\text{CH}_2\text{C}(=\text{O})\text{COOEt}$), 160.4 ($\text{CH}_2\text{C}(=\text{O})\text{COOEt}$), 141.0, 105.9, 62.6, 42.3 ($\text{CH}_2\text{C}(=\text{O})\text{COOEt}$), 28.9. ESI-HRMS: calcd for $\text{C}_{15}\text{H}_{18}\text{O}_7\text{Na}$ ($[\text{M}+\text{Na}]^+$) 333.0945, found 333.0944.

Diethyl 4-cyclohexyl-2-hydroxy-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1oa)



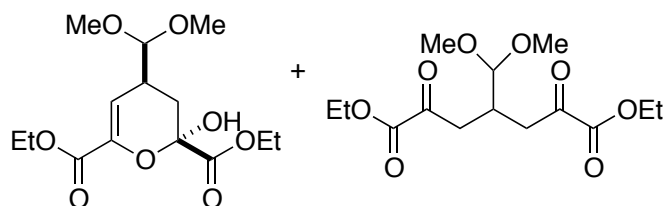
Flash column chromatography (hexane/EtOAc = 4:1); pale yellow gum: ^1H NMR (400 MHz, CDCl_3): δ 6.20 (m, 1H), 4.37-4.19 (m, 5H), 2.48-2.41 (m, 1H), 1.91-1.01 (m, 13H), 1.35 (t, J = 7.1 Hz, 3H), 1.30 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 169.5, 162.4, 140.7, 115.3, 94.8, 62.8, 61.0, 40.7, 33.4, 29.9, 29.84, 29.82, 26.3 14.1, 13.9. ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{26}\text{O}_6\text{Na}$ ($[\text{M}+\text{Na}]^+$) 349.1622, found 349.1631.

Diethyl 2-hydroxy-4-isopropyl-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1pa)



Flash column chromatography (hexane/EtOAc = 4:1); colorless gum. ^1H NMR (400 MHz, CDCl_3): δ 6.17 (dd, J = 2.0 Hz, 1.6 Hz, 1H), 4.39-4.18 (m, 5H), 2.47-2.40 (m, 1H), 1.93-1.70 (m, 3H), 1.35 (t, J = 7.1 Hz, 3H), 1.30 (t, J = 7.1 Hz, 3H), 0.98 (d, J = 7.0 Hz, 3H), 0.96 (dd, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 169.5, 162.4, 140.9, 115.2, 94.9, 62.9, 61.1, 34.2, 30.7, 29.7, 19.4, 14.2, 13.9. ESI-HRMS: calcd for $\text{C}_{14}\text{H}_{23}\text{O}_6$ ($[\text{M}+\text{H}]^+$) 287.1489, found 287.1497.

Diethyl 4-(dimethoxymethyl)-2-hydroxy-3,4-dihydro-2H-pyran-2,6-dicarboxylate (**1qa**)

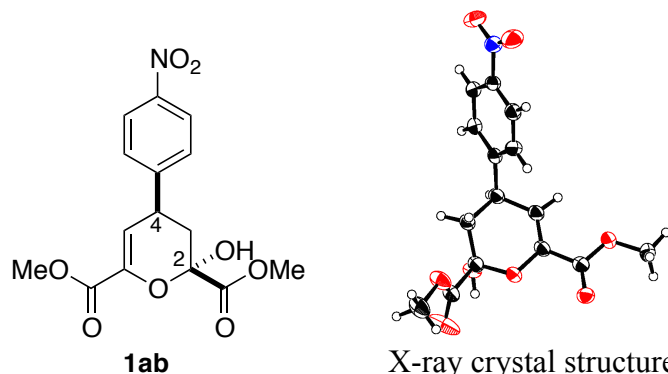


Flash column chromatography (hexane/EtOAc = 3:1); cyclic form (diastereomers)/linear form (partly present as enol form) = 2:1; colorless gum. ^1H NMR (400 MHz, CDCl_3) *donates the linear form: δ 6.19 (m, 1H x 2/3), 4.45-4.20 (m, 5H + 1H x 2/3), 3.48-3.30* (m, 4H x 1/3), 3.41 (s, 3H x 2/3), 3.37 (s, 3H x 2/3), 3.28* (s, 6H x 1/3), 3.04-2.98 (m, 1H x 2/3), 2.98-2.88* (m, 1H x 1/3), 2.04-1.88 (m, 2H x 2/3), 1.40-1.25 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ (as the mixture) 192.5, 169.2, 162.1, 160.6, 141.5, 111.2, 105.8, 105.4, 94.5, 62.9, 62.4, 61.2, 54.6, 53.8, 53.5, 38.8, 35.0, 31.6, 28.6, 14.1, 13.9. ESI-HRMS: calcd for $\text{C}_{14}\text{H}_{23}\text{O}_8$ ($[\text{M}+\text{H}]^+$) 319.1387, found 319.1389. Formation of **1qa** was confirmed by the transformation to **7qa**.

Compound **1ra**

Flash column chromatography (hexane/EtOAc = 7:3); colorless gum. Formation of **1ra** was confirmed by the transformation to **7ra**.

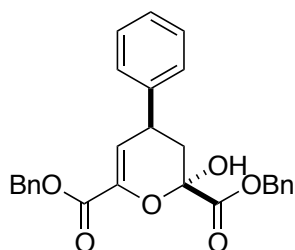
(2*S**,4*S**)-Dimethyl 2-hydroxy-4-(4-nitrophenyl)-3,4-dihydro-2H-pyran-2,6-dicarboxylate (**1ab**)



X-ray crystal structure of **1ab** (see also page S27)

Flash column chromatography (hexane/EtOAc = 1:1); colorless solid; crystallized ($\text{CHCl}_3/\text{MeOH}$) to give colorless crystals; mp 178-179 °C. Relative stereochemistry of was determined to be (2*S**, 4*S**) by X-ray structural analysis. ^1H NMR (400 MHz, CDCl_3): δ 8.21 (d, J = 8.7 Hz, 2H), 7.44 (d, J = 8.7 Hz, 2H), 6.26 (dd, J = 2.4 Hz, 1.6 Hz, 1H), 4.57 (d, J = 2.0 Hz, 1H), 3.99 (ddd, J = 12.4 Hz, 6.0 Hz, 2.4 Hz, 1H), 3.89 (s, 3H), 3.82 (s, 3H), 2.23 (ddd J = 13.2 Hz, 6.0 Hz, 1.6 Hz, 1H) 2.13 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3): δ 169.1, 162.3, 149.7, 147.2, 141.7, 128.5, 124.1, 113.4, 94.4, 53.8, 52.5, 35.3, 34.6. ESI-HRMS: calcd for $\text{C}_{15}\text{H}_{15}\text{O}_8\text{NNa}$ ($[\text{M}+\text{Na}]^+$) 360.0690, found 360.0695.

Dibenzyl 2-hydroxy-4-phenyl-3,4-dihydro-2H-pyran-2,6-dicarboxylate (1gc)



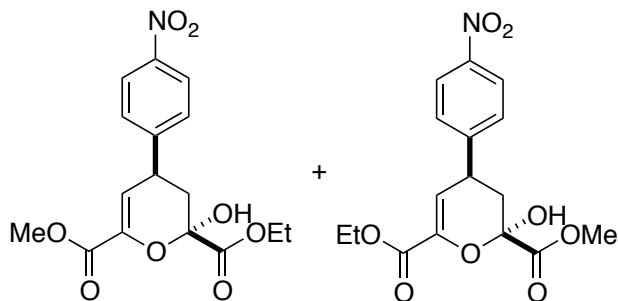
Flash column chromatography (hexane/EtOAc = 4:1); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 7.37-7.23 (m, 15H), 6.34 (m, 1H), 5.33 (d, J = 12.4 Hz, 1H), 5.28 (d, J = 12.4 Hz, 1H), 5.24 (d, J = 12.4 Hz, 1H), 5.20 (d, J = 12.4 Hz, 1H), 4.51 (brs, 1H), 3.84 (ddd, J = 12.4 Hz, 6.4 Hz, 2.0 Hz, 1H), 2.21 (ddd, J = 12.4 Hz, 6.4 Hz, 1.6 Hz, 1H), 2.18-2.09 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3): δ 168.9, 162.0, 142.2, 141.0, 135.5, 134.5, 128.8, 128.7, 128.5, 128.4, 128.36, 128.31, 127.6, 127.1, 115.9, 94.7, 68.4, 66.9, 35.5, 34.7. ESI-HRMS: calcd for $\text{C}_{27}\text{H}_{25}\text{O}_6$ ($[\text{M}+\text{H}]^+$) 445.1646, found 445.1645.

3. Synthesis of Dihydropyrans from Pyruvates and β,γ -Unsaturated α -Ketoesters

Procedure for the Synthesis of Dihydropyrans from Pyruvates and β,γ -Unsaturated α -Ketoesters (Scheme 2)

To a solution of (*E*)-methyl 4-(4-nitrophenyl)-2-oxobut-3-enoate¹ (235.1 mg, 1.0 mmol), and ethyl or benzyl pyruvate (2.0 mmol) in CH_3CN (1 mL), (*S*)- β -proline (17.3 mg, 0.15 mmol) was added at room temperature (25 $^\circ\text{C}$) and the mixture was stirred at the same temperature for 24 h. The mixture was poured into saturated aqueous NH_4Cl solution (5 mL) and extracted with EtOAc. Organic layers were combined, dried over Na_2SO_4 , filtered, concentrated, and purified by flash column chromatography (hexane/EtOAc = 7:3) to afford **6aba** (from ethyl pyruvate) or **6abc** (from benzyl pyruvate). Dihydropyran derivatives **6aba** (from ethyl pyruvate) and **6abc** (from benzyl pyruvate) had the methyl ester at both the sp^2 or sp^3 carbon of the dihydropyran ring.

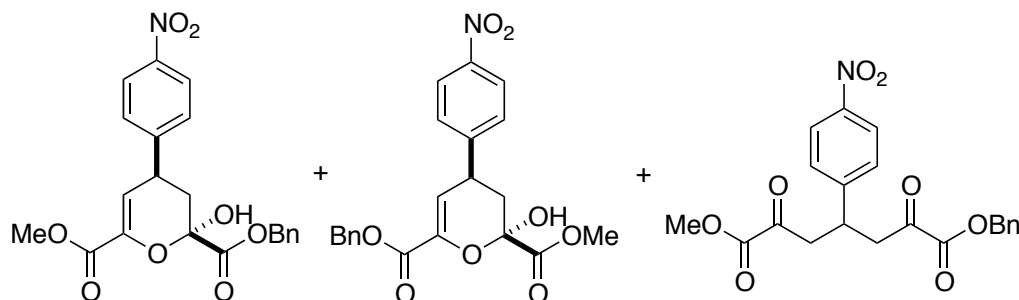
Compound 6aba (a mixture of dihydropyran derivatives in which the methyl ester group at either at the sp^2 or sp^3 carbon of the dihydropyran ring)



Colorless solid; ratio 2:1. *donates the minor isomer. ^1H NMR (400 MHz, CDCl_3): δ 8.22 (d, J = 8.7 Hz, 2H), 7.45 (d, J = 8.7 Hz, 2H), 6.27-6.24 (m, 1H), 4.56 (d, J = 2.0 Hz, 1H x 2/3), 4.51* (d, J = 2.0 Hz, 1H x 1/3), 4.40-4.26 (m, 2H), 4.01-3.95 (m, 1H), 3.89* (s, 3H x 1/3), 3.82 (s, 3H x 2/3), 2.28-2.08 (m, 2H), 1.34* (t, J = 7.2 Hz, 3H), 1.32 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 169.1, 168.6, 162.4, 161.9, 149.8, 147.1, 141.86, 141.81, 128.7, 128.5, 124.1, 123.8, 113.3, 113.0, 94.47, 94.41, 63.3, 61.6, 53.7, 52.4, 35.39, 35.37, 34.69, 34.65, 14.1, 13.9. ESI-

HRMS: calcd C₁₆H₂₁O₈N ([M+H]⁺) 352.1027, found 352.1030.

Compound 6abc (a mixture of dihydropyran derivatives in which the methyl ester group at either at the *sp*² or *sp*³ carbon of the dihydropyran ring) **with compound 5abc** (linear form of **6abc**)



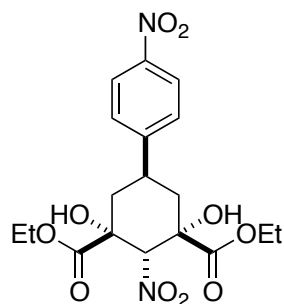
Colorless solid; cyclic form/cyclic form/linear form = 1:1:0.75. ¹H NMR (400 MHz, CDCl₃): δ 8.21-8.18 (m, 2H x 2/2.75), 8.13 (d, *J* = 8.8 Hz, 2H x 0.75/2.75), 7.45-7.33 (m, 7H), 6.28-6.25 (m, 1H x 2/2.75), 5.35-5.20 (m, 2H), 4.70-4.57 (brs, 1H x 2/2.75), 4.01-3.89 (m, 1H), 3.87 (s, 3H x 1/2.75), 3.83 (s, 3H x 0.75/2.75), 3.81 (s, 3H x 1/2.75), 3.37-3.21 (m, 4H x 0.75/2.75), 2.26-2.20 (m, 1H x 2/2.75), 2.16-2.07 (m, 1H x 2/2.75). ¹³C NMR (100 MHz, CDCl₃): δ 191.2, 191.1, 169.0, 168.5, 162.4, 161.7, 160.7, 160.0, 149.7, 149.4, 147.2, 147.0, 141.7, 141.6, 135.3, 134.3, 134.1, 129.0, 128.8, 128.76, 128.75, 128.68, 128.61, 128.56, 128.50, 128.3, 124.1, 124.0, 113.6, 113.4, 94.55, 94.51, 68.6, 68.3, 67.2, 53.7, 53.2, 52.4, 44.5, 44.4, 35.3, 35.2, 34.8, 34.7, 34.6. ESI-HRMS: calcd C₂₁H₂₀O₈N ([M+H]⁺) 414.1183, found 414.1187.

4. Synthesis of Cyclohexane Derivatives 7 from 1

General Procedure for the Synthesis of Cyclohexane Derivatives 7 from 1 (Table 3)

To a solution of dihydropyrane **1** (as a mixture with the corresponding linear form, 0.1 mmol), and nitromethane (61.0 mg, 1.0 mmol) in CH₂Cl₂ (0.5, mL), Et₃N (1.51mg, 15 mol %) was added at room temperature (25 °C). The mixture was stirred at the same temperature for 15 h. The mixture was poured into saturated aqueous NH₄Cl solution (4 mL) and extracted with CH₂Cl₂. Organic layers were combined, dried over Na₂SO₄, filtered, concentrated, and purified by flash column chromatography (hexane/EtOAc) to afford **7**.

Diethyl 1,3-dihydroxy-2-nitro-5-(4-nitrophenyl)cyclohexane-1,3-dicarboxylate (**7aa**)

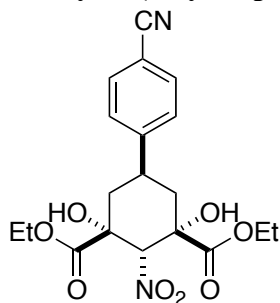


See page S28 for X-ray crystal structure of **7aa**.

Flash column chromatography (hexane/EtOAc = 7:3); colorless solid; crystallized (hexane/CHCl₃) to give colorless crystals; mp 136-138 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.18 (d, *J* = 8.7 Hz, 2H), 7.40 (d, *J* = 8.7 Hz, 2H), 5.56 (s, 1H), 4.37 (q, *J* = 7.2 Hz, 4H), 4.17 (s, 2H), 3.66 (tt, *J* = 12.8 Hz, 3.4 Hz, 1H), 2.22 (dd, *J* = 13.6 Hz, 3.4 Hz, 2H), 2.09 (dd, *J* = 13.6 Hz, 12.8 Hz, 2H), 1.36 (t, *J* =

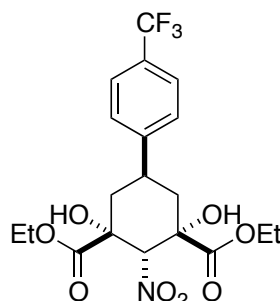
7.2 Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 172.8, 149.5, 147.1, 127.9, 124.1, 86.9, 75.5, 63.5, 42.7, 32.7, 14.0. ESI-HRMS: calcd for $\text{C}_{18}\text{H}_{22}\text{O}_{10}\text{N}_2\text{Na}$ ($[\text{M}+\text{Na}]^+$) 449.1167, found 449.1168.

Diethyl 5-(4-cyanophenyl)-1,3-dihydroxy-2-nitrocyclohexane-1,3-dicarboxylate (7ba)



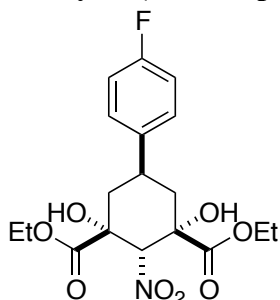
Flash column chromatography (hexane/EtOAc = 3:1); pale yellow solid. ^1H NMR (400 MHz, CDCl_3): δ 7.61 (d, J = 8.2 Hz, 2H), 7.34 (d, J = 8.2 Hz, 2H), 5.55 (s, 1H), 4.37 (q, J = 7.2 Hz, 4H), 4.13 (s, 2H), 3.59 (tt, J = 12.8 Hz, 3.2 Hz, 1H), 2.19 (dd, J = 13.6 Hz, 3.2 Hz, 2H), 2.06 (dd, J = 13.6 Hz, 12.8 Hz, 2H), 1.36 (t, J = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 172.8, 147.5, 132.6, 127.9, 118.5, 111.2, 86.9, 75.5, 63.5, 42.7, 32.8, 14.0. ESI-HRMS: calcd for $\text{C}_{19}\text{H}_{23}\text{O}_8\text{N}_2$ ($[\text{M}+\text{H}]^+$) 407.1449, found 407.1437.

Diethyl 1,3-dihydroxy-2-nitro-5-(4-(trifluoromethyl)phenyl)cyclohexane-1,3-dicarboxylate (7ca)



Flash column chromatography (hexane/EtOAc = 4:1); colorless amorphous solid. ^1H NMR (400 MHz, CDCl_3): δ 7.58 (d, J = 8.2 Hz, 2H), 7.34 (d, J = 8.2 Hz, 2H), 5.56 (s, 1H), 4.38 (q, J = 7.2 Hz, 4H), 4.12 (s, 2H), 3.60 (tt, J = 12.8 Hz, 3.4 Hz, 1H), 2.21 (dd, J = 13.6 Hz, 3.4 Hz, 2H), 2.06 (dd, J = 13.6 Hz, 12.8 Hz, 2H), 1.36 (t, J = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 172.9, 146.0, 129.5 (q, $J_{\text{C,F}}$ = 32 Hz), 125.8 (q, $J_{\text{C,F}}$ = 3.6 Hz), 123.9 (q, $J_{\text{C,F}}$ = 270 Hz), 87.0, 75.6, 63.4, 42.9, 32.5, 14.0. ESI-HRMS: calcd for $\text{C}_{19}\text{H}_{23}\text{F}_3\text{O}_8\text{N}$ ($[\text{M}+\text{H}]^+$) 450.1370, found 450.1360.

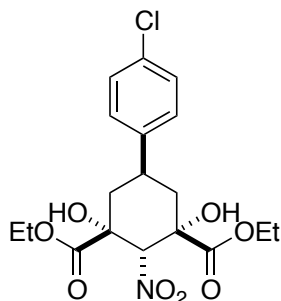
Diethyl 5-(4-fluorophenyl)-1,3-dihydroxy-2-nitrocyclohexane-1,3-dicarboxylate (7da)



Flash column chromatography (hexane/EtOAc = 3:1); pale yellow solid. ^1H NMR (400 MHz,

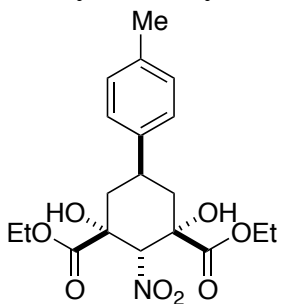
CDCl₃): δ 7.18 (dd, J = 8.6 Hz, 5.2 Hz, 2H), 7.00 (t, J = 8.6 Hz, 2H), 5.55 (s, 1H), 4.37 (q, J = 7.2 Hz, 4H), 4.12 (s, 2H), 3.50 (tt, J = 12.8 Hz, 3.4 Hz, 1H), 2.19 (dd, J = 13.6 Hz, 3.4 Hz, 2H), 2.04 (dd, J = 13.6 Hz, 12.8 Hz, 2H), 1.36 (t, J = 7.2 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃): δ 173.0, 161.8 (d, $J_{C,F}$ = 244 Hz), 137.7 (d, $J_{C,F}$ = 3 Hz), 128.4, (d, $J_{C,F}$ = 8 Hz), 115.6 (d, $J_{C,F}$ = 22 Hz), 87.0, 75.8, 63.3, 43.3, 31.9, 14.0. ESI-HRMS: calcd for C₁₈H₂₃FO₈N ([M+H]⁺) 400.1402, found 400.1397.

Diethyl 5-(4-chlorophenyl)-1,3-dihydroxy-2-nitrocyclohexane-1,3-dicarboxylate (7ea)



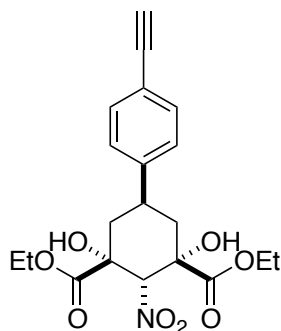
Flash column chromatography (hexane/EtOAc = 3:1); colorless solid. ¹H NMR (400 MHz, CDCl₃): δ 7.29 (d, J = 8.4 Hz, 2H), 7.15 (d, J = 8.4 Hz, 2H), 5.55 (s, 1H), 4.37 (q, J = 7.2 Hz, 4H), 4.11 (s, 2H), 3.50 (tt, J = 12.8 Hz, 3.4 Hz, 1H), 2.18 (dd, J = 13.6 Hz, 3.4 Hz, 2H), 2.04 (dd, J = 13.6 Hz, 12.8 Hz, 2H), 1.36 (t, J = 7.2 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃): δ 172.9, 140.4, 132.9, 128.9, 128.3, 87.0, 75.7, 63.4, 43.1, 32.0, 14.0. IR (CHCl₃): 3472, 2983, 2936, 1731, 1558, 1494, 1370, 1241, 1156, 1014, 760 cm⁻¹. ESI-HRMS: calcd for C₁₈H₂₃ClO₈N ([M+H]⁺) 416.1107, found 416.1095.

Diethyl 1,3-dihydroxy-2-nitro-5-(p-tolyl)cyclohexane-1,3-dicarboxylate (7ha)



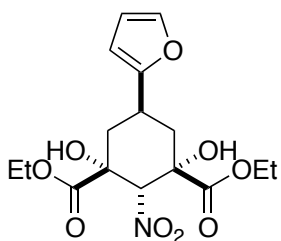
Flash column chromatography (hexane/EtOAc = 4:1); colorless solid. ¹H NMR (400 MHz, CDCl₃): δ 7.13 (d, J = 8.6 Hz, 2H), 7.10 (d, J = 8.6 Hz, 2H), 5.56 (s, 1H), 4.37 (q, J = 7.2 Hz, 4H), 4.12 (s, 2H), 3.47 (tt, J = 12.8 Hz, 3.2 Hz, 1H), 2.32 (s, 3H), 2.19 (dd, J = 13.6 Hz, 3.2 Hz, 2H), 2.04 (dd, J = 13.6 Hz, 12.8 Hz, 2H), 1.36 (t, J = 7.2 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃): δ 173.1, 139.9, 136.8, 129.5, 126.8, 87.1, 75.9, 63.2, 43.3, 32.0, 20.9, 14.0. ESI-HRMS: calcd for C₁₉H₂₆O₈N ([M+H]⁺) 396.1653, found 396.1644.

Diethyl 5-(4-ethynylphenyl)-1,3-dihydroxy-2-nitrocyclohexane-1,3-dicarboxylate (7ia)



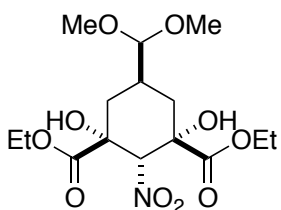
Flash column chromatography (hexane/EtOAc = 4:1); yellow solid. ^1H NMR (400 MHz, CDCl_3): δ 7.44 (d, J = 8.2 Hz, 2H), 7.18 (d, J = 8.2 Hz, 2H), 5.55 (s, 1H), 4.37 (q, J = 7.2 Hz, 4H), 4.12 (s, 2H), 3.52 (tt, J = 12.8 Hz, 3.4 Hz, 1H), 3.06 (s, 1H), 2.19 (dd, J = 13.6 Hz, 3.4 Hz, 2H), 2.06 (dd, J = 13.6 Hz, 12.8 Hz, 2H), 1.36 (t, J = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 172.9, 142.8, 132.5, 127.0, 121.0, 87.0, 83.1, 75.7, 63.3, 43.3, 32.4, 14.0. IR (CHCl_3): 3466, 3289, 2982, 1735, 1560, 1446, 1370, 1241, 1156, 1023, 850 cm^{-1} . ESI-HRMS: calcd for $\text{C}_{20}\text{H}_{24}\text{O}_8\text{N}$ ($[\text{M}+\text{H}]^+$) 406.1496, found 406.1495.

Diethyl 5-(furan-2-yl)-1,3-dihydroxy-2-nitrocyclohexane-1,3-dicarboxylate (7na)



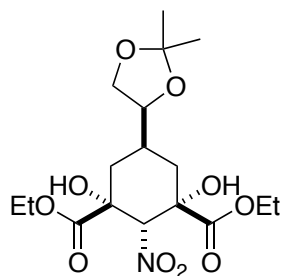
Flash column chromatography (hexane/EtOAc = 3:1); yellow gum. ^1H NMR (400 MHz, CDCl_3): δ 7.31 (dd, J = 1.6 Hz, 0.8 Hz, 1H), 6.28 (dd, J = 3.2 Hz, 1.6 Hz, 1H), 6.04 (d, J = 3.2 Hz, 1H), 5.50 (s, 1H), 4.36 (q, J = 7.2 Hz, 4H), 4.10 (s, 2H), 3.60 (tt, J = 12.8 Hz, 3.5 Hz, 1H), 2.30 (dd, J = 13.6 Hz, 3.5 Hz, 2H), 2.06 (dd, J = 13.6 Hz, 12.8 Hz, 2H), 1.35 (t, J = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 172.9, 155.3, 141.6, 110.2, 104.9, 87.0, 75.4, 63.3, 40.8, 26.8, 14.0. ESI-HRMS: calcd for $\text{C}_{16}\text{H}_{22}\text{O}_9\text{N}$ ($[\text{M}+\text{H}]^+$) 372.1295, found 372.1305.

Diethyl 5-(dimethoxymethyl)-1,3-dihydroxy-2-nitrocyclohexane-1,3-dicarboxylate (7qa)



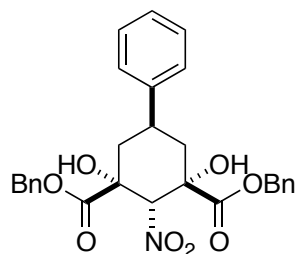
Flash column chromatography (hexane/EtOAc = 3:1); colorless gum. ^1H NMR (400 MHz, CDCl_3): δ 5.39 (s, 1H), 4.33 (q, J = 7.2 Hz, 4H), 4.11 (d, J = 5.4 Hz, 1H), 4.07 (s, 2H), 3.34 (s, 6H), 2.58-2.49 (m, 1H), 2.06 (dd, J = 13.6 Hz, 3.3 Hz, 2H), 1.70 (d, J = 13.6 Hz, 12.8 Hz, 2H), 1.36 (t, J = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 173.2, 106.4, 87.4, 75.3, 63.1, 54.5, 37.1, 29.7, 14.0. ESI-HRMS: calcd for $\text{C}_{15}\text{H}_{25}\text{O}_{10}\text{NNa}$ ($[\text{M}+\text{Na}]^+$) 402.1371, found 402.1370.

Diethyl 5-(2,2-dimethyl-1,3-dioxolan-4-yl)-1,3-dihydroxy-2-nitrocyclohexane-1,3-dicarboxylate (7ra)



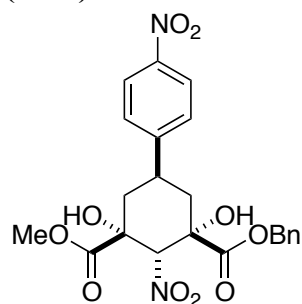
Flash column chromatography (hexane/EtOAc = 13:7); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 5.41 (s, 1H), 4.39- 4.29 (m, 4H), 4.10 (brs, 1H), 4.06 (brs, 1H), 4.01 (dd, J = 8.0 Hz, 6.4 Hz, 1H), 3.91 (dt, J = 12.4 Hz, 6.4 Hz, 1H), 3.66 (dd, J = 8.0 Hz, 6.4 Hz, 1H), 2.35-2.27 (m, 1H), 2.22-2.15 (m, 1H), 1.90-1.84 (m, 1H), 1.74 (t, J = 12.8 Hz, 1H), 1.72 (t, J = 12.8 Hz, 1H), 1.72 (t, J = 12.8 Hz, 1H), 1.37-1.30 (m, 12H). ^{13}C NMR (100 MHz, CDCl_3): δ 173.1, 173.0, 109.4, 87.3, 77.8, 75.3, 75.2, 67.2, 63.2, 39.2, 37.6, 30.5, 26.3, 25.1, 14.0. ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{28}\text{O}_{10}\text{N}$ ($[\text{M}+\text{H}]^+$) 406.1708, found 406.1707.

Dibenzyl 1,3-dihydroxy-2-nitro-5-phenylcyclohexane-1,3-dicarboxylate (7gc)



Flash column chromatography (hexane/EtOAc = 4:1); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 7.42-7.16 (m, 15H), 5.56 (s, 1H), 5.28 (s, 4H), 4.11 (brs, 2H), 3.49 (tt, J = 12.8 Hz, 3.2 Hz, 1H), 2.20 (dd, J = 13.6 Hz, 3.2 Hz, 2H), 2.04 (dd, J = 13.6 Hz, 12.8 Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3): δ 172.9, 141.8, 134.4, 128.87, 128.84, 128.7, 128.6, 127.2, 126.9, 87.1, 75.9, 68.9, 43.0, 32.4. ESI-HRMS: calcd for $\text{C}_{28}\text{H}_{28}\text{O}_8\text{N}$ ($[\text{M}+\text{H}]^+$) 506.1809, found 506.1794.

1-Benzyl 3-methyl 1,3-dihydroxy-2-nitro-5-(4-nitrophenyl)cyclohexane-1,3-dicarboxylate (7abc)

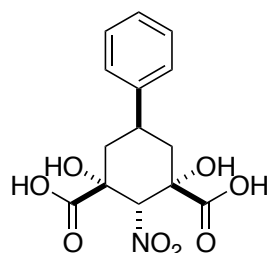


Synthesized from **6abc**. Flash column chromatography (hexane/EtOAc = 3:1); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 8.18 (d, J = 8.4 Hz, 2H), 7.45-7.30 (m, 7H), 5.56 (s, 1H), 5.34 (d, J = 12.0 Hz, 2H), 5.30 (d, J = 12.0 Hz, 1H), 4.12 (d, J = 3.8 Hz, 2H), 3.91 (s, 3H), 3.69-3.63 (m, 1H), 2.26-2.20 (m, 2H), 2.12-2.02 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3): δ 173.2, 172.7, 149.3, 147.1,

134.3, 129.0, 128.8, 128.7, 127.9, 124.1, 86.9, 75.6, 69.2, 54.0, 42.6, 32.6. IR (CHCl₃): 3465, 2983, 1740, 1559, 1509, 1347, 1245, 1155, 746, 698 cm⁻¹ ESI-HRMS: calcd for C₂₂H₂₃O₁₀N₂ ([M+H]⁺) 475.1347, found 475.1328.

5. Synthesis of 8 and 9 from 7

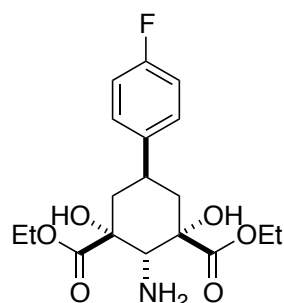
1,3-Dihydroxy-2-nitro-5-phenylcyclohexane-1,3-dicarboxylic acid (8gc)



To a solution of cyclohexane derivative **7gc** (50.5 mg, 0.1 mmol) in CH₃OH (1.0 mL), 10 wt% Pd-C (5.0 mg) was added. The mixture was stirred at room temperature (25 °C) under hydrogen (1 atm) for 6 h. The mixture was filtered through celite, concentrated in vacuo, and purified by flash column chromatographically (MeOH/EtOAc = 1:1) to afford **8gc** as a colorless solid (30.9 mg, 95%).

¹H NMR (400 MHz, CD₃OD): δ 7.33-7.18 (m, 5H), 5.69 (s, 1H), 3.45 (tt, *J* = 12.8 Hz, 3.6 Hz, 1H), 2.19 (dd, *J* = 13.6 Hz, 12.8 Hz, 2H), 2.10 (dd, *J* = 13.6 Hz, 3.6 Hz, 2H). ¹³C NMR (100 MHz, CD₃OD): δ 176.6, 144.3, 129.8, 128.1, 127.9, 88.7, 77.1, 44.7, 33.9. ESI-HRMS: calcd for C₁₄H₁₆O₈N ([M+H]⁺) 326.0870, found 326.0880.

Diethyl 2-amino-5-(4-fluorophenyl)-1,3-dihydroxycyclohexane-1,3-dicarboxylate (9da)



To a mixture of cyclohexane derivative **7da** (119.7 mg, 0.3 mmol) and 10 wt% Pd-C in MeOH (1.0 mL), Et₃SiH (478 μL, 3.0 mmol) was added dropwise from syringe at under nitrogen.² The mixture of stirred at room temperature (25 °C) for 24 h until complete consumption of **7da** (TLC). The mixture was filtered through celite, concentrated in vacuo, and purified by flash column chromatographically (hexane/EtOAc = 1:1) to afford **9da** as a colorless solid (33.2 mg, 30%).

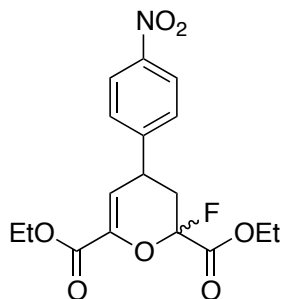
¹H NMR (400 MHz, CDCl₃): δ 7.22-7.19 (m, 2H), 7.01-6.97 (m, 2H), 4.41-4.22 (m, 4H), 4.14 (s, 1H), 3.48-3.36 (m, 1H), 2.08-2.00 (m, 4H), 1.36-1.32 (m, 6H). ¹³C NMR (100 MHz, CDCl₃): δ 174.3, 161.6 (d, *J*_{C,F} = 244 Hz), 138.8, 128.4, (d, *J*_{C,F} = 8 Hz), 115.5 (d, *J*_{C,F} = 21 Hz), 75.0, 63.9, 63.1, 42.9, 32.1, 14.0. ESI-HRMS: calcd for C₁₈H₂₅FO₆N ([M+H]⁺) 370.1660, found 370.1664.

6. Synthesis of Fluorodihydropyrans 10 from 1

General Procedure for the Synthesis Fluorodihydropyran Derivatives 10 from 1 (Table 4)

To a solution of dihydropyran **1** (as a mixture with the corresponding linear form, 0.05 mmol) in CH_2Cl_2 (0.3 mL), diethylaminosulfur trifluoride (DAST) (16.1 mg, 0.1 mmol) was added at 0 °C, and the mixture was stirred at the same temperature for 2 h.³ The mixture was concentrated and purified by flash column chromatography (hexane/EtOAc) to afford **10**.

Diethyl 2-fluoro-4-(4-nitrophenyl)-3,4-dihydro-2H-pyran-2,6-dicarboxylate (10aa)

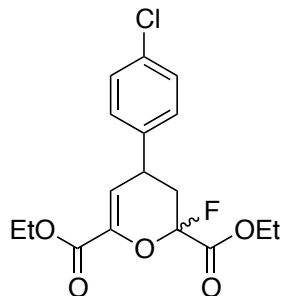


Flash column chromatography (hexane/EtOAc = 4:1).

Major isomer. R_f 0.37 (hexane/EtOAc = 7:3); pale yellow solid. ^1H NMR (400 MHz, CDCl_3): δ 8.19 (d, J = 8.6 Hz, 2H), 7.43 (d, J = 8.6 Hz, 2H), 6.47-6.46 (m, 1H), 4.39-4.29 (m, 4H), 3.94-3.90 (m, 1H), 2.66 (ddd, J = 30.0 Hz, 14.6 Hz, 8.2 Hz, 1H), 2.50-2.43 (m, 1H), 1.37 (t, J = 7.1 Hz, 3H), 1.34 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 164.8 (d, $J_{\text{C,F}}$ = 31 Hz), 161.0, 148.6, 147.1, 142.4, 128.75, 128.73, 123.9, 112.0, 105.8 (d, $J_{\text{C,F}}$ = 232 Hz), 63.0, 61.9, 34.1, 33.9 (d, $J_{\text{C,F}}$ = 21 Hz), 14.1, 13.9. ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{19}\text{FO}_7\text{N}$ ($[\text{M}+\text{H}]^+$) 368.1140, found 368.1141.

Minor isomer. R_f 0.50 (hexane/EtOAc = 7:3); pale yellow solid. ^1H NMR (400 MHz, CDCl_3): δ 8.23 (d, J = 8.7 Hz, 2H), 7.44 (d, J = 8.7 Hz, 2H), 6.34 (d, J = 1.0 Hz, 1H), 4.39-4.27 (m, 4H), 4.00-3.94 (m, 1H), 2.59-2.53 (m, 1H), 2.17-2.02 (m, 1H), 1.36 (t, J = 7.2 Hz, 3H), 1.34 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 164.6 (d, $J_{\text{C,F}}$ = 31 Hz), 160.9, 148.6, 147.4, 141.7 (d, $J_{\text{C,F}}$ = 3 Hz), 128.5, 124.3, 113.8, 105.4 (d, $J_{\text{C,F}}$ = 235 Hz), 63.1, 61.9, 34.5 (d, $J_{\text{C,F}}$ = 25 Hz), 34.0 (d, $J_{\text{C,F}}$ = 2 Hz), 14.1, 13.9. ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{19}\text{FNO}_7$ ($[\text{M}+\text{H}]^+$) 368.1140, found 368.1139.

Diethyl 4-(4-chlorophenyl)-2-fluoro-3,4-dihydro-2H-pyran-2,6-dicarboxylate (10ea)



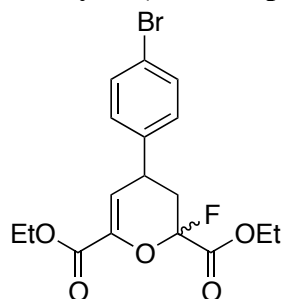
Flash column chromatography (hexane/EtOAc = 85:15).

Major isomer. R_f 0.45 (hexane/EtOAc = 4:1); yellow solid. ^1H NMR (400 MHz, CDCl_3): δ 7.29 (d, J = 8.4 Hz, 2H), 7.17 (d, J = 8.4 Hz, 2H), 6.43 (d, J = 4.4 Hz, 1H), 4.38-4.25 (m, 4H), 3.80-3.76 (m, 1H), 2.60 (ddd, J = 27.6 Hz, 14.4 Hz, 8.0 Hz, 1H), 2.41-2.35 (m, 1H), 1.35 (t, J = 7.1 Hz, 3H), 1.34 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 165.2 (d, $J_{\text{C,F}}$ = 32 Hz), 161.2, 141.8,

139.7, 133.0, 129.1, 129.0, 128.8, 113.5, 106.1 (d, $J_{C,F} = 232$ Hz), 62.9, 61.8, 34.4 (d, $J_{C,F} = 24$ Hz), 34.0 (d, $J_{C,F} = 2$ Hz), 14.1, 13.9. ESI-HRMS: calcd for $C_{17}H_{19}FCIO_5$ ($[M+H]^+$) 357.0900, found 357.0901.

Minor isomer. R_f 0.51 (hexane/EtOAc = 85:15); yellow solid. 1H NMR (400 MHz, $CDCl_3$): δ 7.33 (d, $J = 8.4$ Hz, 2H), 7.18 (d, $J = 8.4$ Hz, 2H), 6.33 (s, 1H), 4.38-4.26 (m, 4H), 3.84-3.78 (m, 1H), 2.54-2.48 (m, 1H), 2.13-1.98 (m, 1H), 1.35 (t, $J = 7.1$ Hz, 3H), 1.32 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.9 (d, $J_{C,F} = 31$ Hz), 161.2, 141.2 (d, $J_{C,F} = 2$ Hz), 139.6, 133.3, 129.1, 128.8, 115.3, 105.7 (d, $J_{C,F} = 232$ Hz), 62.9, 61.7, 34.7 (d, $J_{C,F} = 25$ Hz), 33.4 (d, $J_{C,F} = 3$ Hz), 14.1, 13.9. ESI-HRMS: calcd for $C_{17}H_{19}FCIO_5$ ($[M+H]^+$) 357.0900, found 357.0900.

Diethyl 4-(4-bromophenyl)-2-fluoro-3,4-dihydro-2H-pyran-2,6-dicarboxylate (10 fa)

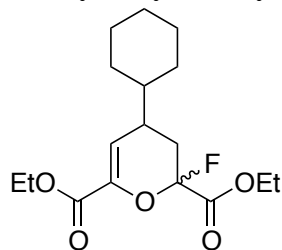


Flash column chromatography (hexane/EtOAc = 85:15).

Major isomer. R_f 0.45 (hexane/EtOAc = 4:1); yellow gum. 1H NMR (400 MHz, $CDCl_3$): δ 7.44 (d, $J = 8.3$ Hz, 2H), 7.12 (d, $J = 8.3$ Hz, 2H), 6.42 (dd $J = 0.8, 4.5$ Hz, 1H), 4.37-4.25 (m, 4H), 3.78-3.74 (m, 1H), 2.60 (ddd, $J = 27.6$ Hz, 14.4 Hz, 8.0 Hz, 1H), 2.41-2.35 (m, 1H), 1.35 (t, $J = 7.1$ Hz, 3H), 1.34 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.1 (d, $J_{C,F} = 32$ Hz), 161.2, 141.9, 140.3, 131.8, 129.48, 129.46, 121.1, 113.4, 106.0 (d, $J_{C,F} = 232$ Hz), 62.9, 61.8, 34.4 (d, $J_{C,F} = 24$ Hz), 34.0 (d, $J_{C,F} = 2$ Hz), 14.1, 13.9. ESI-HRMS: calcd for $C_{17}H_{19}FBrO_5$ ($[M+H]^+$) 401.0394, found 401.0404.

Minor isomer. R_f 0.51 (hexane/EtOAc = 4:1); yellow gum. 1H NMR (400 MHz, $CDCl_3$): δ 7.48 (d, $J = 8.4$ Hz, 2H), 7.12 (d, $J = 8.4$ Hz, 2H), 6.32 (s, 1H), 4.36-4.25 (m, 4H), 3.81-3.77 (m, 1H), 2.52-2.48 (m, 1H), 2.12-1.97 (m, 1H), 1.35 (t, $J = 7.1$ Hz, 3H), 1.32 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.8 (d, $J_{C,F} = 32$ Hz), 161.1, 141.2 (d, $J_{C,F} = 3$ Hz), 140.2, 132.1, 129.2, 121.3, 115.2, 105.6 (d, $J_{C,F} = 234$ Hz), 62.9, 61.7, 34.7 (d, $J_{C,F} = 24$ Hz), 33.5 (d, $J_{C,F} = 2$ Hz), 14.1, 13.9. ESI-HRMS: calcd for $C_{17}H_{19}FBrO_5$ ($[M+H]^+$) 401.0394, found 401.0395.

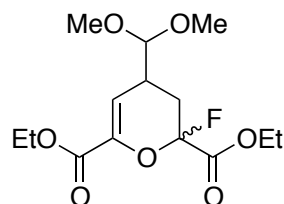
Diethyl 4-cyclohexyl-2-fluoro-3,4-dihydro-2H-pyran-2,6-dicarboxylate (10oa)



Flash column chromatography (hexane/EtOAc = 85:15); dr = 1:1, pale yellow gum. 1H NMR (400 MHz, $CDCl_3$): δ 6.33 (d, $J = 4.4$ Hz, 1H x 1/2), 6.26 (s, 1H x 1/2), 4.36-4.21 (m, 4H), 2.50-0.90 (m, 20H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.7 (d, $J_{C,F} = 33$ Hz), 165.5 (d, $J_{C,F} = 32$ Hz), 161.54, 161.50, 140.5 (d, $J_{C,F} = 3$ Hz), 140.3, 116.1, 115.2, 107.1 (d, $J_{C,F} = 228$ Hz), 106.4 (d, $J_{C,F} = 232$ Hz).

Hz), 62.7, 62.6, 61.5, 61.4, 40.6, 35.28, 35.25, 30.9, 30.5, 29.9, 29.8, 29.7, 29.3, 29.1, 29.0, 28.8, 26.3, 26.13, 26.11, 14.2, 14.1, 13.9. ESI-HRMS: calcd for $C_{17}H_{26}FO_5$ ($[M+H]^+$) 329.1759, found 329.1766.

Diethyl 4-(dimethoxymethyl)-2-fluoro-3,4-dihydro-2H-pyran-2,6-dicarboxylate (10qa)



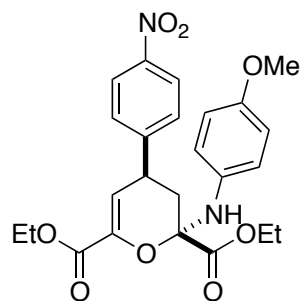
Flash column chromatography (hexane/EtOAc = 4:1); dr = 1:1, pale yellow gum. 1H NMR (400 MHz, $CDCl_3$): δ 6.31-6.28 (m, 1H), 4.39-4.22 (m, 5H), 3.41 (s, 3H), 3.388 (s, 3H x 1/2), 3.383 (s, 3H x 1/2), 2.93-2.85 (m, 1H x 1/2), 2.72-2.65 (m, 1H x 1/2), 2.44-2.29 (m, 1H), 2.18 (ddd, J = 34.4 Hz, 14.8 Hz, 8.0 Hz, 1H x 1/2), 1.88 (ddd, J = 34.4 Hz, 14.0 Hz, 12.8 Hz, 1H x 1/2), 1.35 (t J = 7.2 Hz, 3H x 1/2), 1.34 (t, J = 7.2 Hz, 3H x 1/2), 1.32 (t, J = 7.2 Hz, 3H x 1/2), 1.31 (t, J = 7.2 Hz, 3H x 1/2). ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.2 (d, $J_{C,F}$ = 32 Hz), 165.1 (d, $J_{C,F}$ = 32 Hz), 161.28, 161.20, 141.3 (d, $J_{C,F}$ = 3 Hz), 141.1, 112.1, 111.8, 106.1 (d, $J_{C,F}$ = 231 Hz), 106.0 (d, $J_{C,F}$ = 232 Hz), 105.2, 104.8, 104.7, 62.8, 61.6, 61.5, 54.5, 54.12, 54.09, 54.04, 32.8, 31.3, 28.0 (d, $J_{C,F}$ = 24 Hz), 27.2 (d, $J_{C,F}$ = 25 Hz), 14.1, 13.9. ESI-HRMS: calcd for $C_{14}H_{22}FO_7$ ($[M+H]^+$) 321.1344, found 321.1357.

7. Synthesis of 11-15 from 1

General procedure for the Synthesis of Aminodihydropyran Derivatives 11 and 12 from 1 (Scheme 4a)

To a solution of dihydropyran **1** (as a mixture with the corresponding linear form, 18.2 mg, 0.05 mmol) in CH_2Cl_2 (0.5 mL), amine (0.1 mmol) was added at room temperature (25 °C), and the mixture was stirred at the same temperature for 24 h. The mixture was concentrated and purified by flash column chromatography to afford aminodihydropyrane derivative **11** or **12**.

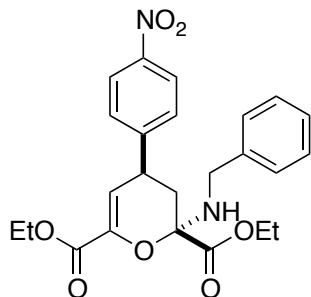
Diethyl 2-((4-methoxyphenyl)amino)-4-(4-nitrophenyl)-3,4-dihydro-2H-pyran-2,6-dicarboxylate (11)



Flash column chromatography (hexane/EtOAc = 7:3); pale yellow solid. 1H NMR (400 MHz, $CDCl_3$): δ 8.22 (d, J = 8.4 Hz, 2H), 7.51 (d, J = 8.4 Hz, 2H), 7.07 (d, J = 8.8 Hz, 2H), 6.81 (d, J = 8.8 Hz, 2H), 5.89 (m, 1H), 4.27-4.08 (m, 4H), 4.01-3.90 (m, 2H), 3.78 (s, 3H), 2.38 (t, J = 12.8 Hz, 1H), 2.18 (ddd, J = 12.8 Hz, 5.2 Hz, 1.8 Hz, 1H), 1.28 (t, J = 7.2 Hz, 3H), 0.96 (t, J = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3): δ 171.8, 164.4, 158.5, 151.5, 146.9, 138.0, 135.1, 129.7, 128.6, 124.0, 113.98, 113.94, 85.0, 63.0, 60.9, 55.3, 39.8, 35.7, 13.9, 13.7. IR (CHCl_3): 3347, 2935, 1729, 1582, 1509, 1346, 1246, 1180, 1029, 834, 749 cm^{-1} ESI-HRMS: calcd for $\text{C}_{24}\text{H}_{27}\text{O}_8\text{N}_2$ ($[\text{M}+\text{H}]^+$) 471.1762, found 471.1762.

Diethyl 2-(benzylamino)-4-(4-nitrophenyl)-3,4-dihydro-2H-pyran-2,6-dicarboxylate (12)

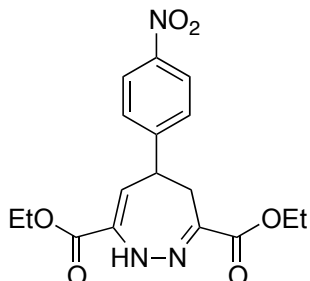


Flash column chromatography (hexane/EtOAc = 3:1); pale yellow gum: ^1H NMR (400 MHz, CDCl_3): δ 8.19 (d, J = 8.8 Hz, 2H), 7.46 (d, J = 8.8 Hz, 2H), 7.43-7.18 (m, 5H), 5.73 (m, 1H), 4.60 (s, 1H), 4.56 (d, J = 15.8 Hz, 1H), 4.19 (d, J = 15.8 Hz, 1H), 4.13-3.97 (m, 4H), 3.79-3.71 (m, 1H), 2.14 (t, J = 12.4 Hz, 1H), 2.07 (ddd, J = 12.4 Hz, 4.8 Hz, 1.6 Hz, 1H), 1.19 (t, J = 7.2 Hz, 3H), 1.11 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 173.4, 164.5, 151.5, 146.9, 138.1, 137.8, 128.8, 128.5, 127.9, 127.1, 123.9, 113.0, 83.9, 63.1, 61.1, 50.2, 41.4, 35.3, 13.9, 13.8. IR (CHCl_3): 3482, 2980, 1720, 1519, 1347, 1259, 1145, 1018, 856, 746, 697 cm^{-1} ESI-HRMS: calcd for $\text{C}_{24}\text{H}_{27}\text{O}_7\text{N}_2$ ($[\text{M}+\text{H}]^+$) 455.1813, found 455.1808.

General Procedure for the Synthesis Dihydro-1,2-diazepine Derivatives 13 and 14 (Scheme 4b)

To a solution of dihydropyran **1** (as a mixture with the corresponding linear form, 0.05 mmol) in CH_2Cl_2 (0.3, mL), hydrazine monohydrate (5.0 mg, 0.1 mmol) was added at room temperature (25 $^\circ\text{C}$), and the mixture was stirred at the same temperature for 2 h. The mixture was concentrated and purified by flash column chromatography. The purified compound was solved in CDCl_3 at 25 $^\circ\text{C}$ and kept for 3 h at the same temperature to give **13** or **14**.

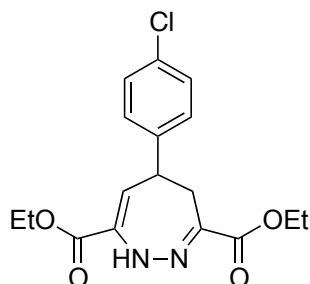
Diethyl 5-(4-nitrophenyl)-4,5-dihydro-1H-1,2-diazepine-3,7-dicarboxylate (13)



Flash column chromatography (hexane/EtOAc = 3:7); brown gum. ^1H NMR (400 MHz, CDCl_3): δ 8.82 (s, 1H), 8.19 (d, J = 8.7 Hz, 2H), 7.34 (d, J = 8.7 Hz, 2H), 6.21 (d, J = 4.5 Hz, 1H), 4.36-4.27 (m, 2H), 4.25-4.17 (m, 2H), 4.07-4.03 (m, 1H), 3.28 (dd, J = 13.6 Hz, 12.4 Hz, 1H), 2.98 (dd, J = 13.6 Hz, 7.2 Hz, 1H), 1.33 (t, J = 7.1 Hz, 3H), 1.28 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 164.5, 163.3, 151.0, 146.9, 142.5, 130.7, 128.4, 124.0, 116.5, 62.6, 61.9, 43.0, 36.3,

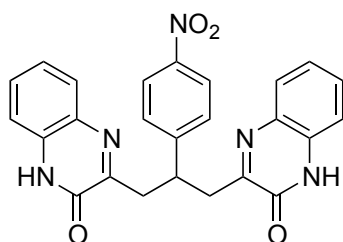
14.2, 14.1. ESI-HRMS: calcd for $C_{17}H_{20}O_6N_3$ ($[M+H]^+$) 362.1347, found 362.1347.

Diethyl 5-(4-chlorophenyl)-4,5-dihydro-1*H*-1,2-diazepine-3,7-dicarboxylate (14)



Flash column chromatography (hexane/EtOAc = 1:1); brown gum. 1H NMR (400 MHz, $CDCl_3$): δ 8.75 (s, 1H), 7.30 (d, J = 8.4 Hz, 2H), 7.10 (d, J = 8.4 Hz, 2H) 6.23 (m, 1H), 4.34-4.20 (m, 4H), 3.90-3.85 (m, 1H), 3.28 (dd, J = 13.6 Hz, 2.8 Hz, 1H), 2.88 (dd, J = 13.6 Hz, 7.4 Hz, 1H), 1.32 (t, J = 7.1 Hz, 3H), 1.29 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.6, 163.5, 142.9, 142.7, 132.8, 130.1, 128.8, 128.7, 118.3, 62.4, 61.7, 42.6, 36.6, 14.2, 14.1. ESI-HRMS: calcd for $C_{17}H_{20}ClO_4N_2$ ($[M+H]^+$) 351.1106, found 351.1079.

3,3'-(2-(4-Nitrophenyl)propane-1,3-diyl)bis(quinoxalin-2(1*H*)-one) (15)



To a solution of dihydropyran **1aa** (18.2 mg, 0.05 mmol) in CH_2Cl_2 (0.3 mL), 1,2-phenylenediamine (10.8 mg, 0.1 mmol) was added at room temperature (25 °C), and the mixture was stirred at the same temperature for 24 h. The mixture was concentrated and purified by flash column chromatography (EtOAc) to afford **15** as a pale yellow solid (18.1 mg, 80%).

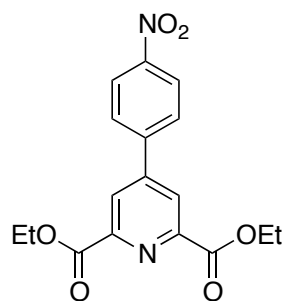
1H NMR (400 MHz, $(CD_3)_2SO$): δ 12.2 (brs 2H), 8.09 (d, J = 8.8 Hz, 2H), 7.67 (d, J = 8.8 Hz, 2H), 7.58 (dd, J = 8.0 Hz, 1.2 Hz, 2H), 7.39 (ddd, J = 8.0 Hz, 7.6 Hz, 1.2 Hz, 1H), 7.19 (ddd, J = 8.0 Hz, 7.6 Hz, 1.2 Hz, 1H), 7.15 (dd, J = 8.0 Hz, 1.2 Hz, 1H), 4.31 (quintet, J = 7.2 Hz, 1H), 3.33-3.23 (m, 4H). ^{13}C NMR (100 MHz, $(CD_3)_2SO$): δ 159.6, 154.6, 153.2, 145.9, 131.5, 131.3, 129.5, 129.1, 127.9, 123.2, 122.9, 115.1, 39.4, 38.7. ESI-HRMS: calcd for $C_{25}H_{20}O_4N_5$ ($[M+H]^+$) 454.1510, found 54.1515.

8. Synthesis of Pyridines 16 from 1

General Procedure for the Synthesis of Pyridines 16 from 1 (Table 5)

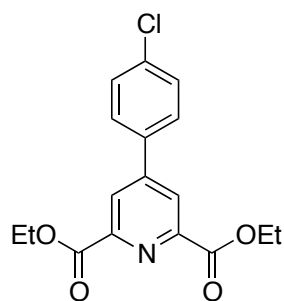
To a solution of dihydropyran **1** (as a mixture with the corresponding linear form, 0.1 mmol) in CH_3CN (0.4 mL), ammonium acetate (16.9 mg, 0.22 mmol) was added at room temperature (25 °C), and the mixture was stirred at the same temperature for 24 h. The mixture was concentrated and purified by flash column chromatography to afford **16**.

Diethyl 4-(4-nitrophenyl)pyridine-2,6-dicarboxylate (16aa)



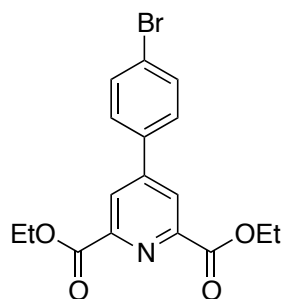
Flash column chromatography (hexane/EtOAc = 3:2); pale yellow solid. ^1H NMR (400 MHz, CDCl_3): δ 8.51 (s, 2H), 8.39 (d, $J = 8.8$ Hz, 2H), 7.91 (d, $J = 8.8$ Hz, 2H) 4.53 (q, $J = 7.1$ Hz, 4H), 1.48 (t, $J = 7.1$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 164.4, 149.7, 148.7, 148.5, 142.6, 128.3, 125.6, 124.6, 62.6, 14.2. ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{17}\text{O}_6\text{N}_2$ ($[\text{M}+\text{H}]^+$) 345.1081, found 345.1081.

Diethyl 4-(4-chlorophenyl)pyridine-2,6-dicarboxylate (16ea)



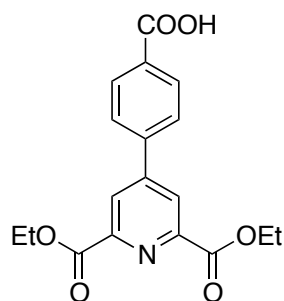
Flash column chromatography (hexane/EtOAc = 7:3); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 8.46 (s, 2H), 7.69 (d, $J = 8.4$ Hz, 2H), 7.51 (d, $J = 8.8$ Hz, 2H) 4.51 (q, $J = 7.1$ Hz, 4H), 1.47 (t, $J = 7.1$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 164.7, 149.7, 149.4, 136.4, 134.4, 129.6, 128.4, 125.2, 62.5, 14.2. ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{17}\text{ClO}_4\text{N}$ ($[\text{M}+\text{H}]^+$) 334.0841, found 334.0836.

Diethyl 4-(4-bromophenyl)pyridine-2,6-dicarboxylate (16fa)



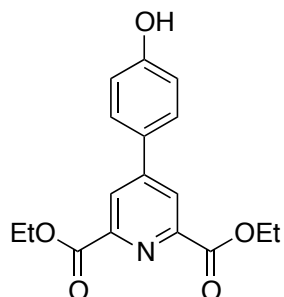
Flash column chromatography (hexane/EtOAc = 7:3); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 8.46 (s, 2H), 7.67 (d, $J = 8.8$ Hz, 2H), 7.62 (d, $J = 8.8$ Hz, 2H) 4.51 (q, $J = 7.1$ Hz, 4H), 1.47 (t, $J = 7.1$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 164.7, 149.9, 149.4, 135.2, 132.2, 128.6, 125.2, 124.7, 62.5, 14.2. ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{17}\text{BrO}_4\text{N}$ ($[\text{M}+\text{H}]^+$) 378.0335, found 378.0335.

4-(2,6-Bis(ethoxycarbonyl)pyridin-4-yl)benzoic acid (16ja)



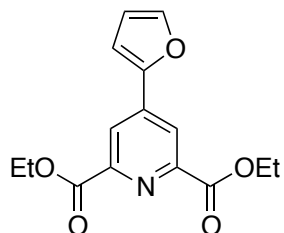
Flash column chromatography (EtOAc/MeOH = 10:1); colorless solid. ^1H NMR (400 MHz, CD_3OD): δ 8.58 (s, 2H), 8.19 (d, J = 8.4 Hz, 2H), 7.93 (d, J = 8.4 Hz, 2H) 4.50 (q, J = 7.2 Hz, 4H), 1.46 (t, J = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, CD_3OD): δ 169.5, 165.8, 151.7, 150.7, 141.5, 131.7, 131.2, 128.4, 126.6, 63.4, 14.5. ESI-HRMS: calcd for $\text{C}_{18}\text{H}_{18}\text{O}_6\text{N}$ ($[\text{M}+\text{H}]^+$) 344.1129, found 344.1127.

Diethyl 4-(4-hydroxyphenyl)pyridine-2,6-dicarboxylate (16la)

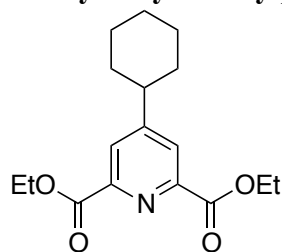


Flash column chromatography (hexane/EtOAc = 7:3); colorless solid. ^1H NMR (400 MHz, CD_3OD): δ 8.46 (s, 2H), 7.67 (d, J = 8.4 Hz, 2H), 7.00 (d, J = 8.4 Hz, 2H), 4.50 (q, J = 7.2 Hz, 4H), 1.46 (t, J = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, CD_3OD): δ 165.0, 157.9, 150.5, 149.1, 128.6, 128.4, 124.8, 116.4, 62.4, 14.2. ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{18}\text{O}_5\text{N}$ ($[\text{M}+\text{H}]^+$) 316.1179, found 316.1179.

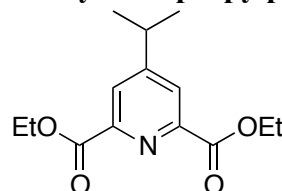
Diethyl 4-(furan-2-yl)pyridine-2,6-dicarboxylate (6na)



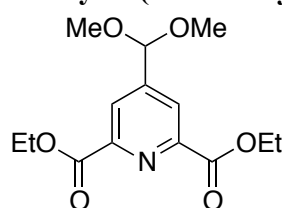
Flash column chromatography (hexane/EtOAc = 7:3); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 8.45 (s, 2H), 7.61 (d, J = 1.6 Hz, 1H), 7.06 (dd, J = 3.6 Hz, 0.4 Hz, 1H), 6.57 (dd, J = 3.6 Hz, 1.6 Hz, 1H), 4.50 (q, J = 7.2 Hz, 4H), 1.47 (t, J = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 164.7, 149.9, 149.3, 144.9, 139.9, 121.5, 112.5, 110.8, 62.4, 14.2. ESI-HRMS: calcd for $\text{C}_{15}\text{H}_{16}\text{O}_5\text{N}$ ($[\text{M}+\text{H}]^+$) 290.1023, found 290.1027.

Diethyl 4-cyclohexylpyridine-2,6-dicarboxylate (16oa)

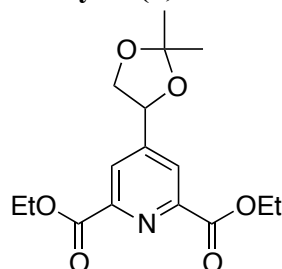
Flash column chromatography (hexane/EtOAc = 7:3); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 8.11 (s, 2H), 4.48 (q, $J = 7.2$ Hz, 4H), 2.70-2.63 (m, 1H), 1.96-1.86 (m, 4H), 1.82-1.76 (m, 1H), 1.45 (t, $J = 7.2$ Hz, 6H), 1.53-1.25 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3): δ 165.0, 159.5, 148.6, 126.5, 62.2, 43.9, 33.3, 26.3, 25.7, 14.2. ESI-HRMS: calcd for $\text{C}_{17}\text{H}_{24}\text{O}_4\text{N}$ ($[\text{M}+\text{H}]^+$) 306.1700, found 306.1702.

Diethyl 4-isopropylpyridine-2,6-dicarboxylate (16pa)

Flash column chromatography (hexane/EtOAc = 7:3); colorless gum. ^1H NMR (400 MHz, CDCl_3): δ 8.12 (d, $J = 0.8$ Hz, 2H), 4.47 (dq, $J = 7.2$ Hz, 1.6 Hz, 4H), 3.10- 3.08 (m, 1H), 1.45 (dt, $J = 7.2$ Hz, 1.6 Hz, 6H), 1.31 (dd, $J = 7.0$ Hz, 1.6 Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 165.0, 160.4, 148.7, 126.1, 62.2, 33.7, 22.9, 14.2. ESI-HRMS: calcd for $\text{C}_{14}\text{H}_{20}\text{O}_4\text{N}$ ($[\text{M}+\text{H}]^+$) 266.1387, found 266.1388.

Diethyl 4-(dimethoxymethyl)pyridine-2,6-dicarboxylate (16qa)

Flash column chromatography (hexane/EtOAc = 7:3); colorless solid. ^1H NMR (400 MHz, CDCl_3): δ 8.33 (s, 2H), 5.48 (s, 1H), 4.48 (q, $J = 7.2$ Hz, 4H), 3.34 (s, 6H), 1.44 (t, $J = 7.2$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 164.5, 149.8, 149.0, 125.9, 100.5, 62.3, 52.8, 14.2; ESI-HRMS: calcd for $\text{C}_{14}\text{H}_{20}\text{O}_6\text{N}$ ($[\text{M}+\text{H}]^+$) 298.1285, found 298.1287.

Diethyl 4-(2,2-dimethyl-1,3-dioxolan-4-yl)pyridine-2,6-dicarboxylate (16ra)

Flash column chromatography (hexane/EtOAc = 1:1); colorless gum. ^1H NMR (400 MHz, CDCl_3): δ 8.23 (s, 2H), 5.18 (t, J = 6.8 Hz, 1H), 4.48 (q, J = 7.2 Hz, 4H), 4.43 (dd, J = 8.4 Hz, 6.8 Hz, 1H), 3.72 (dd, J = 8.4 Hz, 7.2 Hz, 1H), 1.56 (s, 3H), 1.49 (s, 3H), 1.44 (t, J = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 164.5, 152.1, 149.0, 124.8, 110.9, 75.9, 70.7, 62.4, 26.3, 25.6, 14.2. ESI-HRMS: calcd for $\text{C}_{16}\text{H}_{22}\text{O}_6\text{N}$ ($[\text{M}+\text{H}]^+$) 324.1442, found 324.1440

9. Analysis of Reversibility between Cyclic and Acyclic, Linear Forms of **1aa**

When a solution of the cyclic form of **1aa** (cyclic form/linear form >20:1) in CDCl_3 or in CD_3CN was stored at 25 °C, no increase of the linear form was detected at least for a week.

When a solution of the cyclic form of **1aa** was kept in the presence of β -proline (0.2 equiv) in CD_3CN at 25 °C under the same condition as that of the catalytic reaction to form **1aa**, however, the linear form increased gradually to reach the ratio of the cyclic form/linear form to be 1:1. The time required to become the 1:1 ratio ranged from several hours to 2 days depending on the lot of compound **1aa** and of the solvent, suggesting that the moisture and/or acid content in solvents affect the time required to reach the 1:1 ratio. But, further keeping of the solutions of the 1:1 ratio of the cyclic/linear forms of **1aa** at the same temperature did not alter the ratio further. Subsequently, when acetic acid (0.4 equiv) was added to the mixture, the 1:1 ratio of the cyclic/linear forms of **1aa** was unchanged.

When a solution of the cyclic form of **1aa** was kept with methyl pyruvates in the presence of β -proline (0.2 equiv) in CD_3CN , the ratio of the cyclic/linear forms of **1aa** also became 1:1, and formation of **5aba** and **6aba** was not detected. That is, although the cyclic form of **1aa** was converted to the linear form in equilibrium in the presence of β -proline, no retro-Michael reaction of **1aa** occurred under this condition.

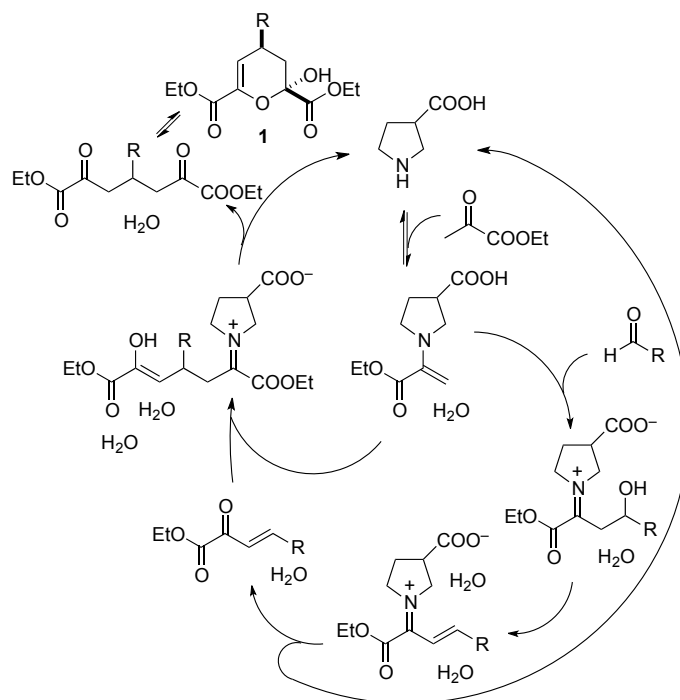
Whereas addition of β -proline to a solution of the cyclic form of **1aa** increased the linear form up to 1:1, addition of (*S*)-L-alanine instead of β -proline to the cyclic form of **1aa** did not increase the linear form; the cyclic/linear form of **1aa** was retained as >20:1. Addition of acetic acid to a solution of the cyclic form of **1aa** (cyclic form/linear form >20:1) in CD_3CN increased the ratio of the cyclic form/linear form to 10:1 at 25 °C after 1 day. Addition of Et_3N to a solution of the cyclic form of **1aa** (cyclic form/linear form >20:1) in CD_3CN increased the ratio of the cyclic form/linear form to 1:1 at 25 °C after 7 min. On the other hand, addition of pyrrolidine to a solution of **1aa** resulted in decomposition of **1aa** (formation of a complex mixture).

β -Proline catalyzed the formation of **1** and also enhanced the equilibrium between the cyclic and the linear forms of **1aa**; the ratio of the cyclic/linear forms was 1:1 under the equilibrium in the presence of β -proline. Depending on the conditions used, the ratios of the cyclic form/linear form reached to certain values.

10. Plausible Mechanism for the Formation of **1** from Pyruvates and Aldehydes

A plausible mechanism for the formation of products **1** ($\text{R}^1 = \text{R}$, $\text{R}^2 = \text{Et}$) is shown in Scheme S1. We suggest that pyruvate forms an enamine with β -proline and that this enamine reacts with

aldehydes to generate the intermediate β,γ -unsaturated α -ketoesters via an aldol condensation. The pyruvate enamine then further reacts with the β,γ -unsaturated α -ketoesters by Michael addition. Cyclization via enol formation of the diketoester intermediate gives the dihydropyran derivatives. The aldehyde may also form an iminium ion with β -proline before the reaction with the pyruvate enamine and the formation of the β,γ -unsaturated α -ketoesters may be accomplished via a Mannich reaction-elimination route.



Scheme S1. A plausible mechanism of the β -proline-catalyzed formation of **1** from pyruvate and aldehyde.

11. X-Ray Crystal Structures of **1ab** and **7aa**

Compound **1ab** was crystallized from $\text{CHCl}_3/\text{MeOH}$. The relative stereochemistry of **1ab** was determined to be ($2S^*,4S^*$) by the X-ray structural analysis (Figure S1).

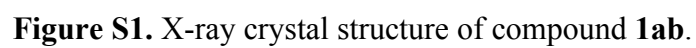
Compound **7aa** was crystallized from hexane/ CHCl_3 . The relative stereochemistry of **7aa** was determined by the X-ray structural analysis (Figure S2).

The X-ray crystal structure data of **1ab** and **7aa** have been deposited in the Cambridge Crystallographic Data Centre:

CCDC 1012430 for compound **1ab**.

CCDC 1012431 for compound **7aa**.

These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



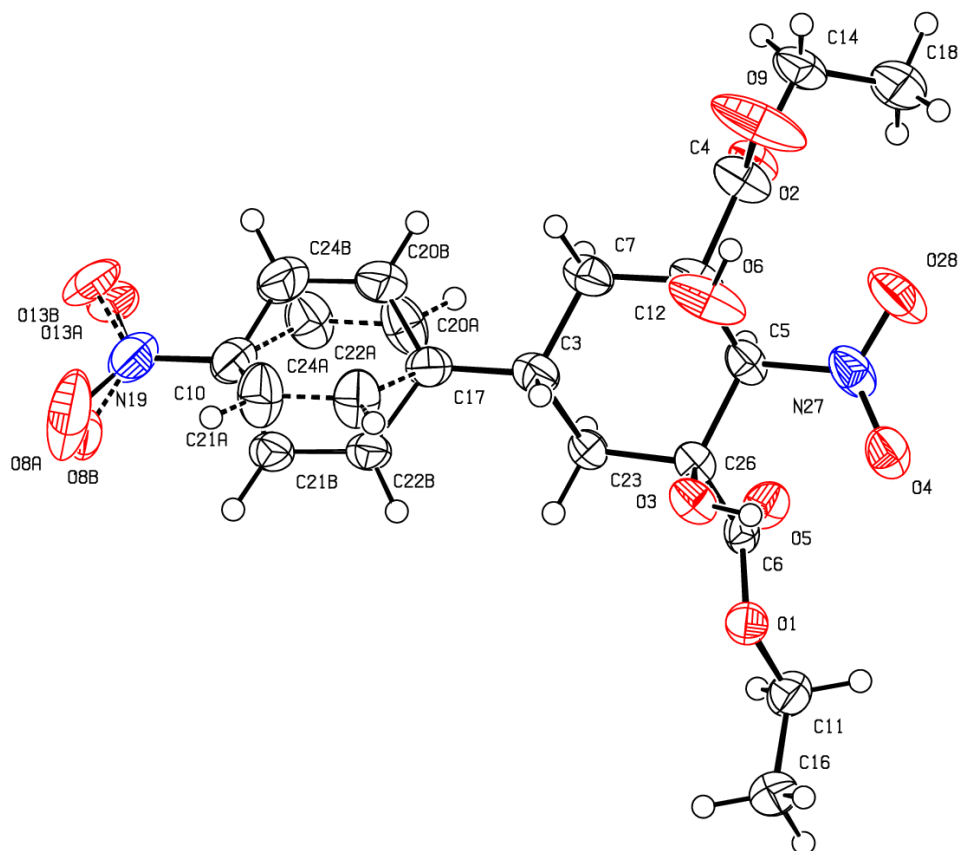
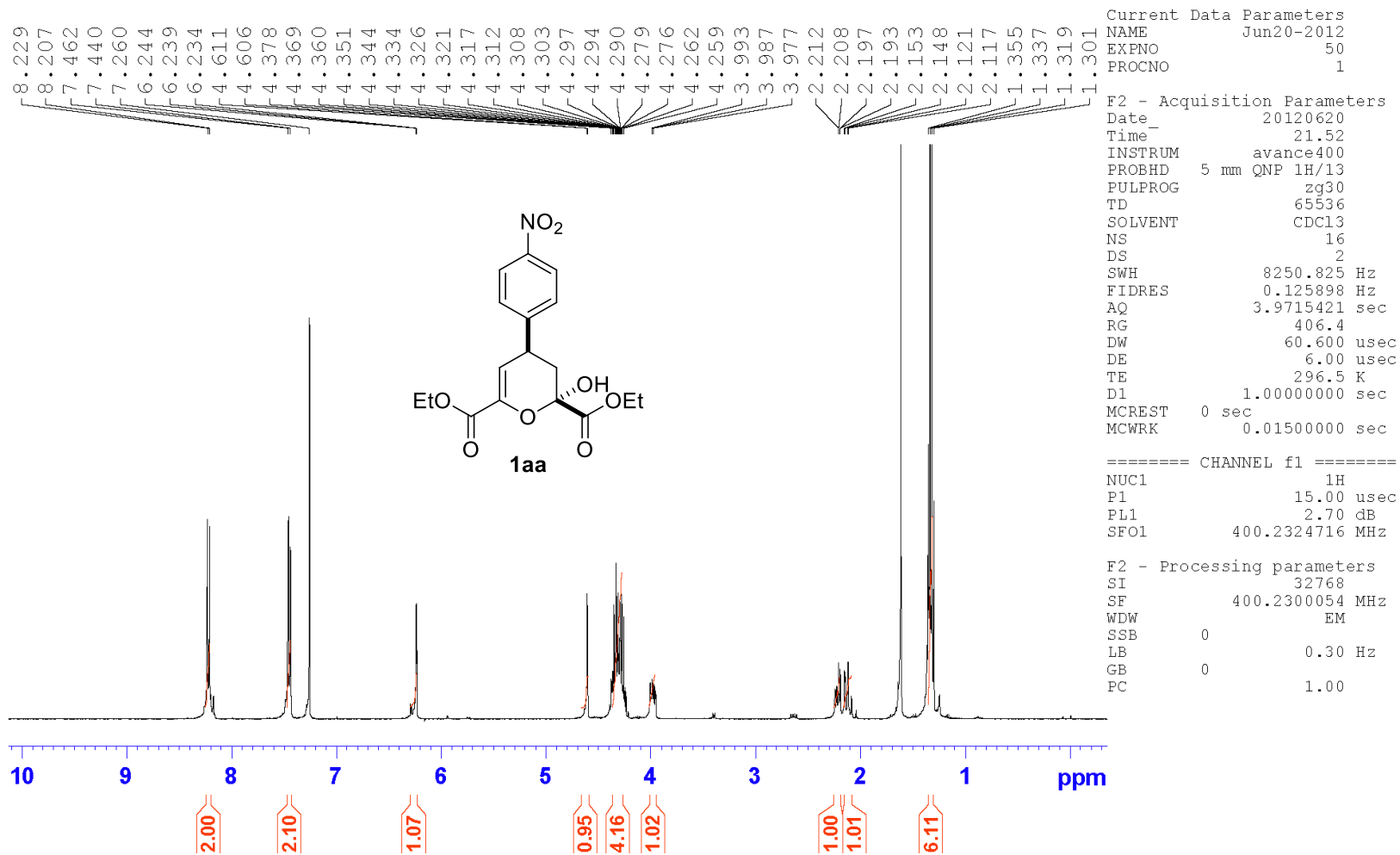
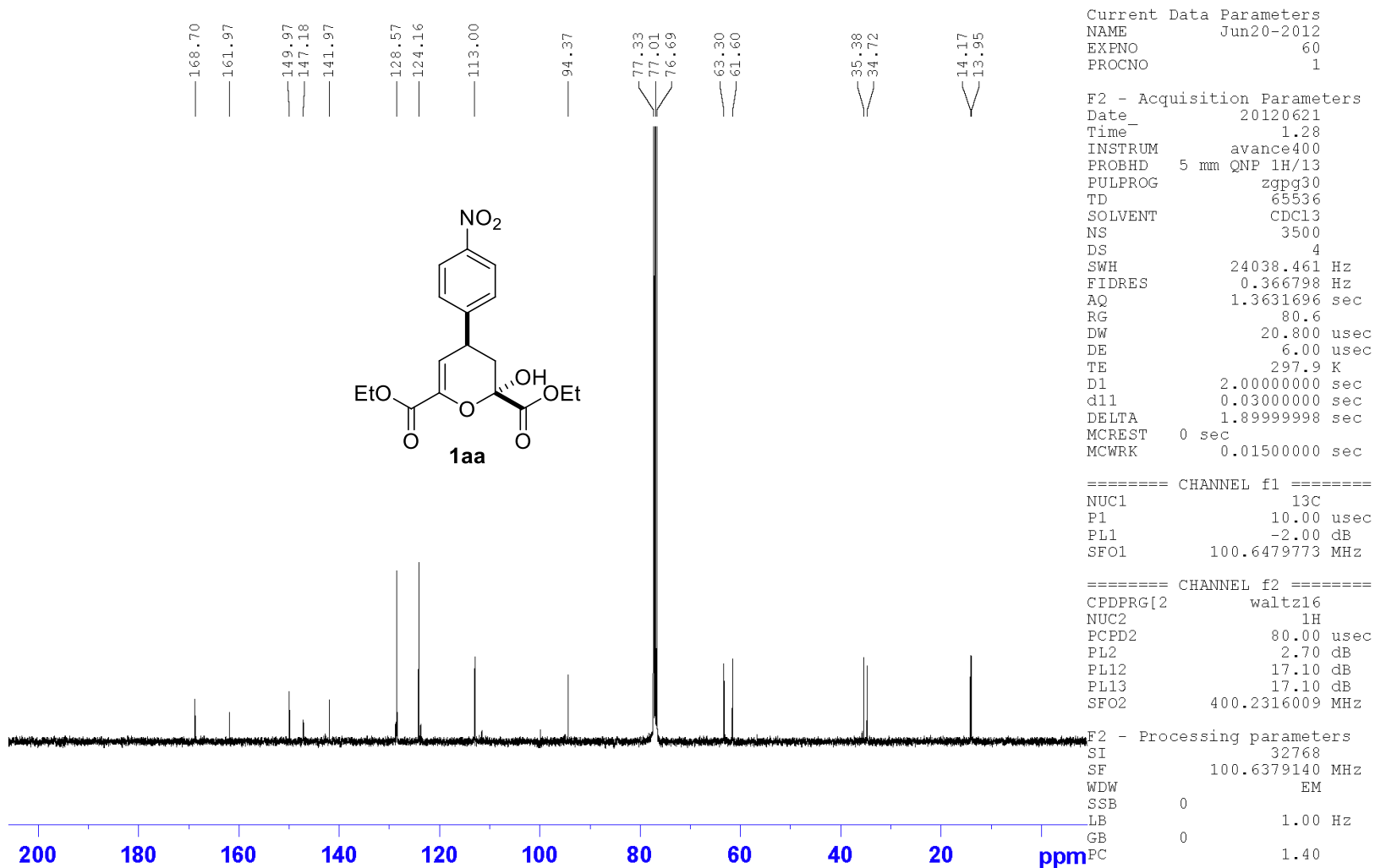


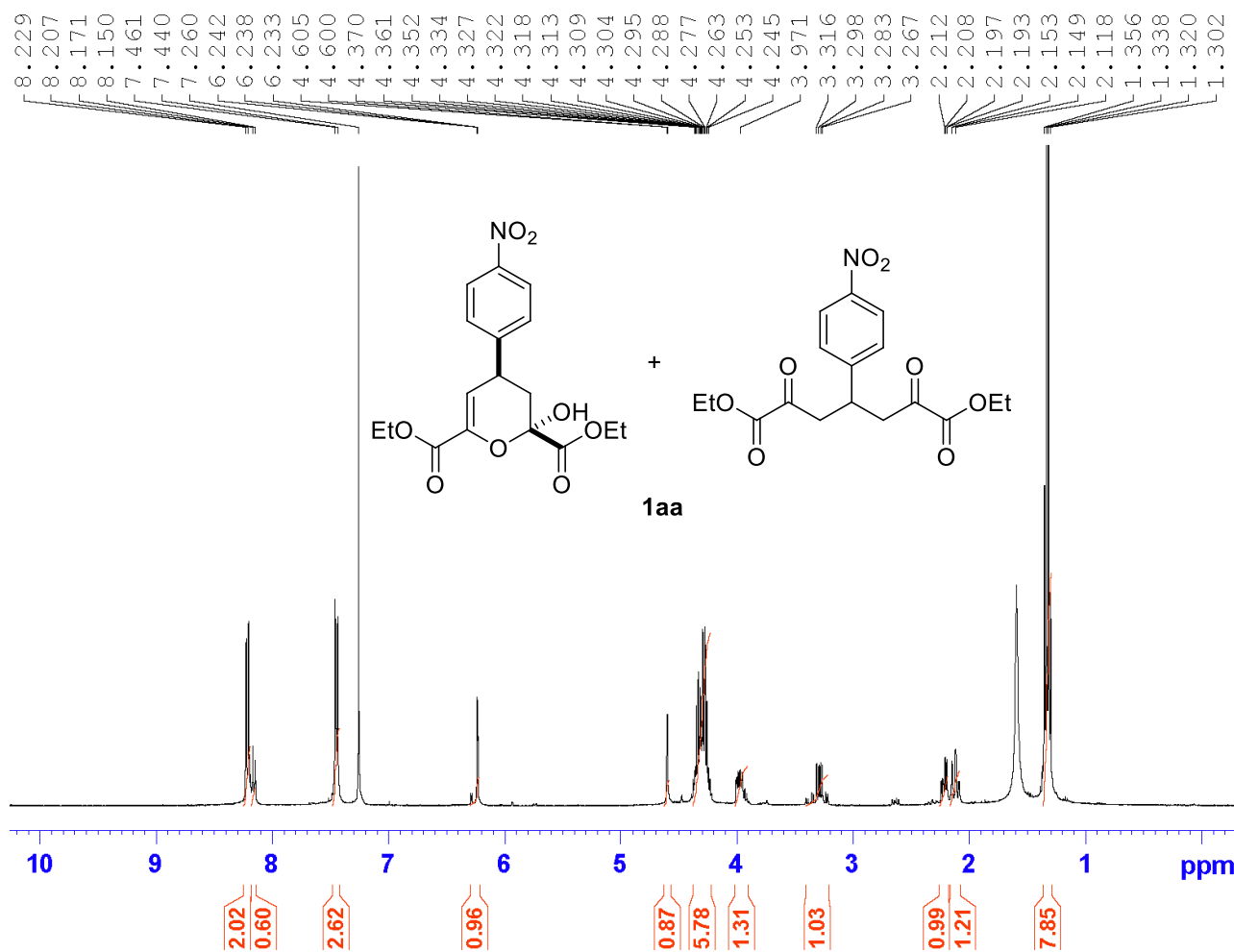
Figure S2. X-ray crystal structure of compound **7aa**.

12. References

1. G. Dujardin, S. Leconte, A. Bénard and E. Brown, *Synlett* 2001, 147.
2. P. K. Mandal and J. S. McMurray, *J. Org. Chem.* 2007, **72**, 6599.
3. J.-H. Kim, R. Resende, T. Wennekes, H.-M. Chen, N. Bance, S. Buchini, A. G. Watts, P. Pilling, V. A. Streltsov, M. Petric, R. Liggins, S. Barrett, McKimm- J. L. Breschkin, M. Niikura and S. G. Withers, *Science* 2013, **340**, 71.





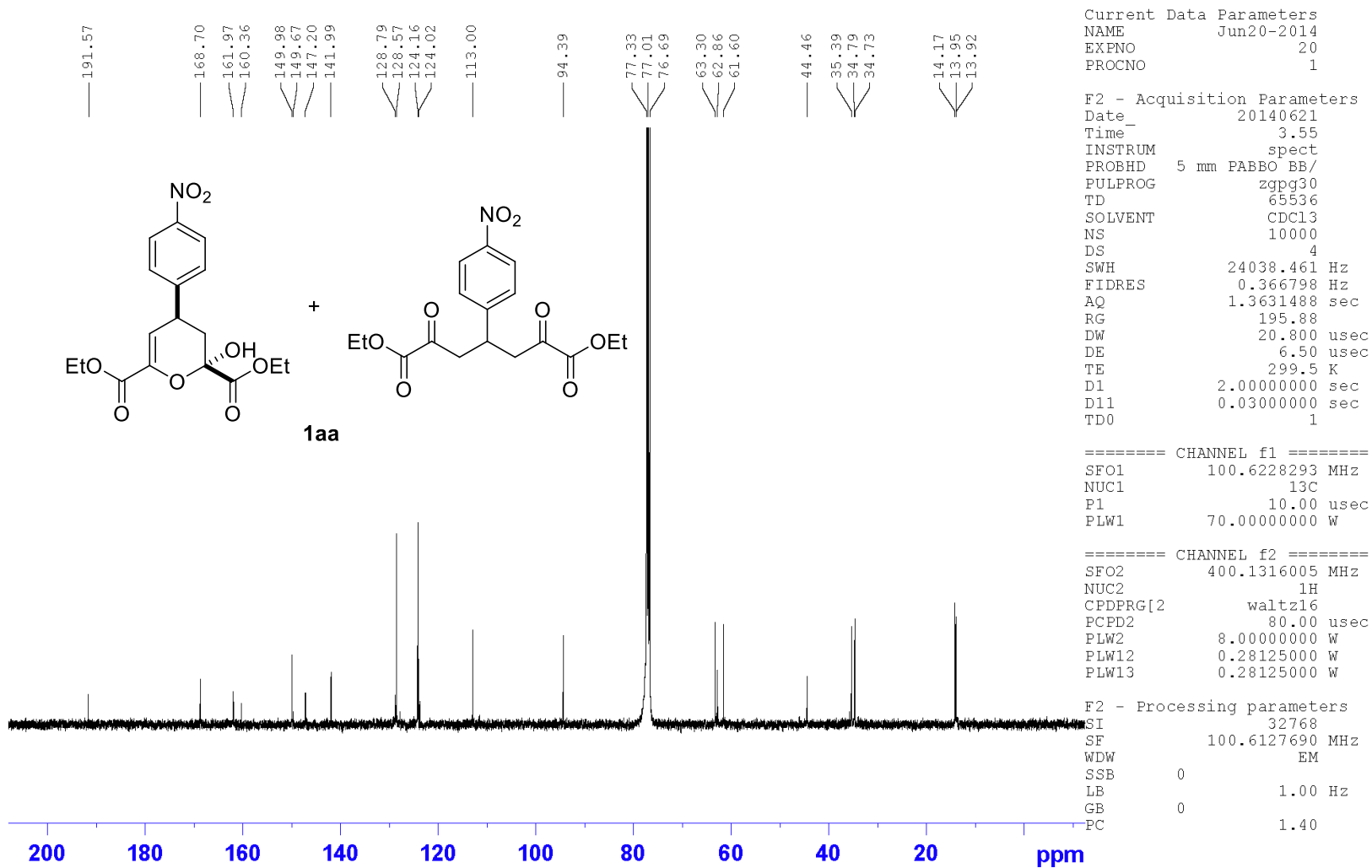


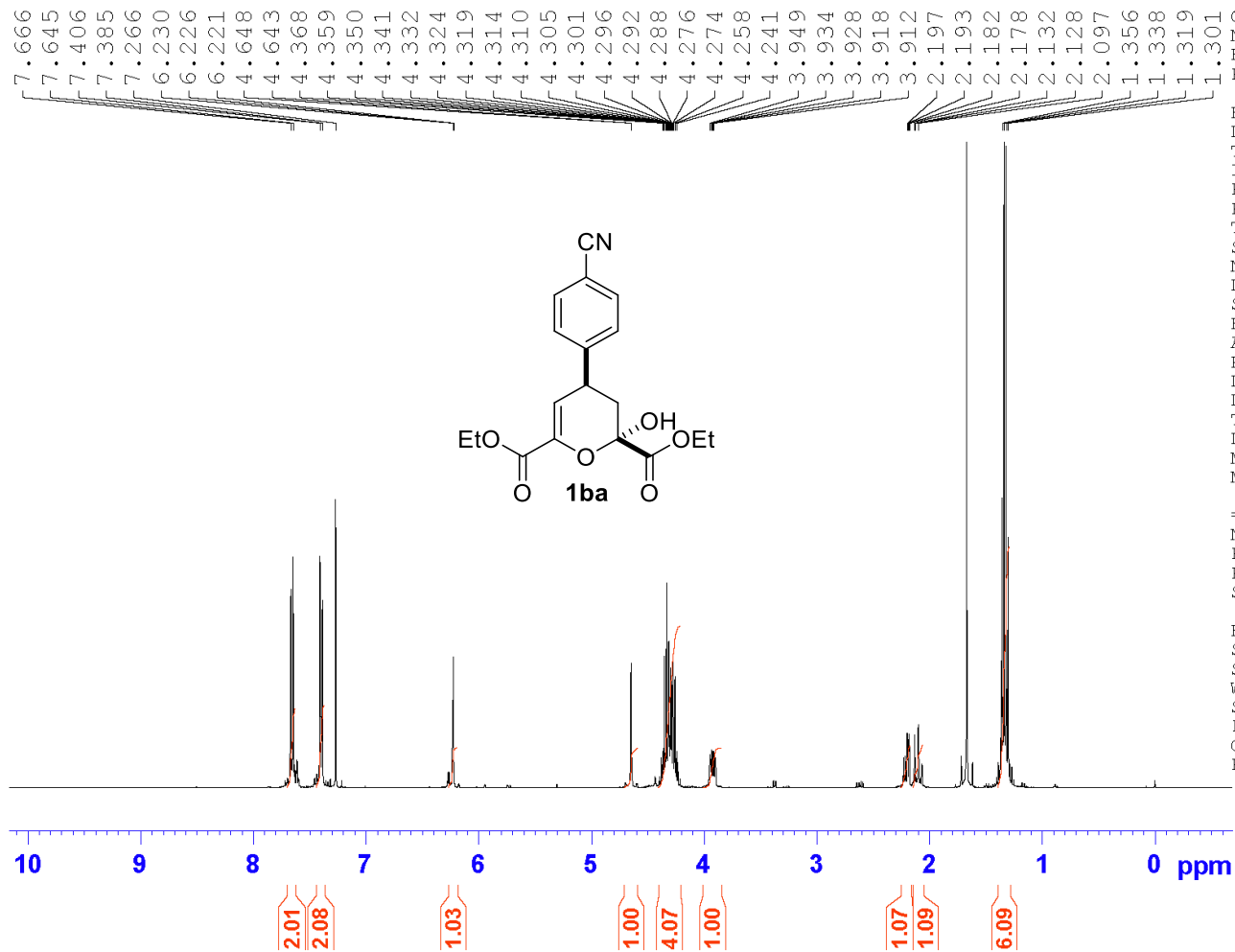
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F2 - Acquisition Parameters
Date_ 20140620
Time_ 12.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 77.81
DW 62.400 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 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 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



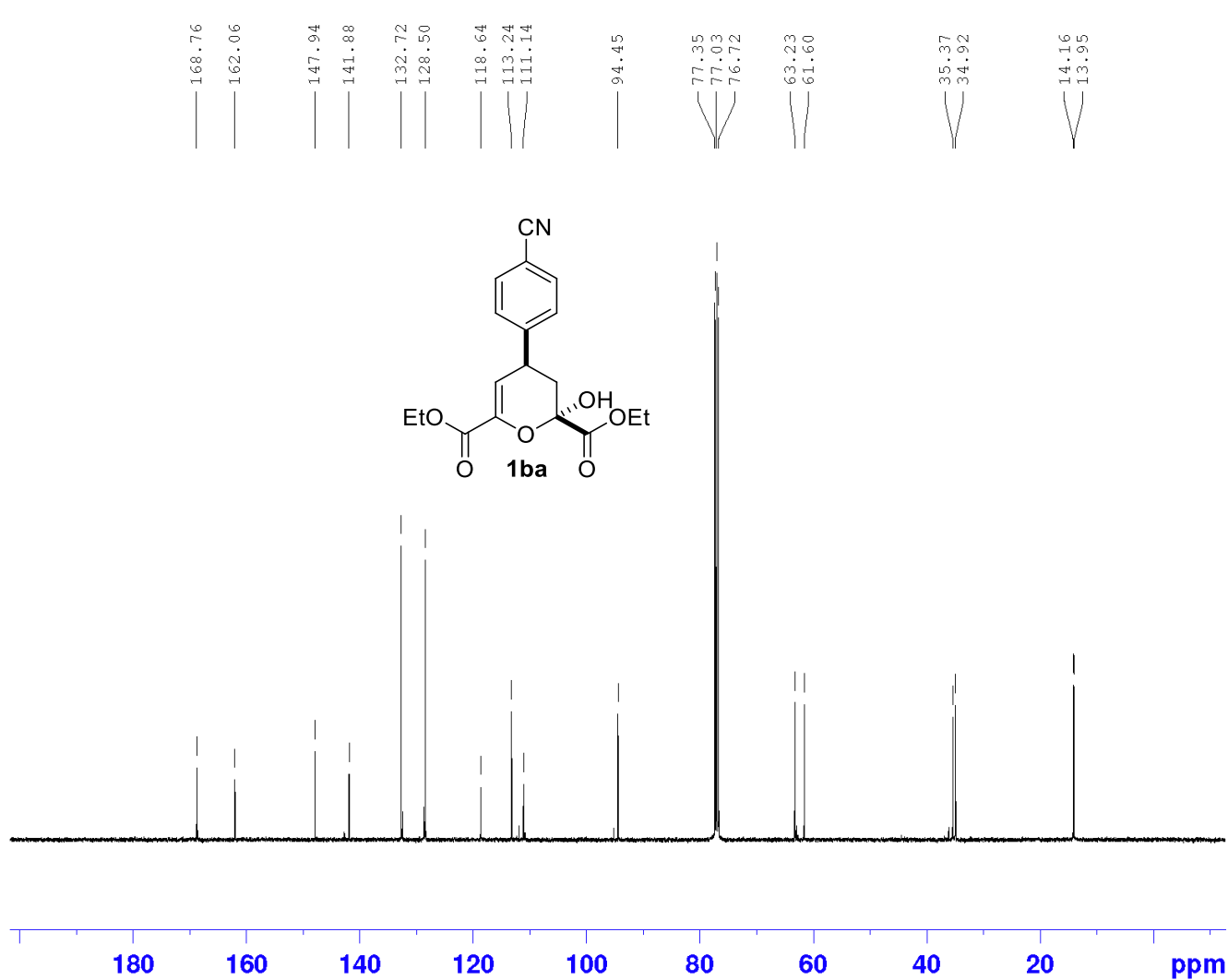


Current Data Parameters
NAME May30-2012
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120530
Time 22.03
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715421 sec
RG 322.5
DW 60.600 usec
DE 6.00 usec
TE 295.5 K
D1 1.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 2.70 dB
SFO1 400.2324716 MHz

F2 - Processing parameters
SI 32768
SF 400.2300029 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



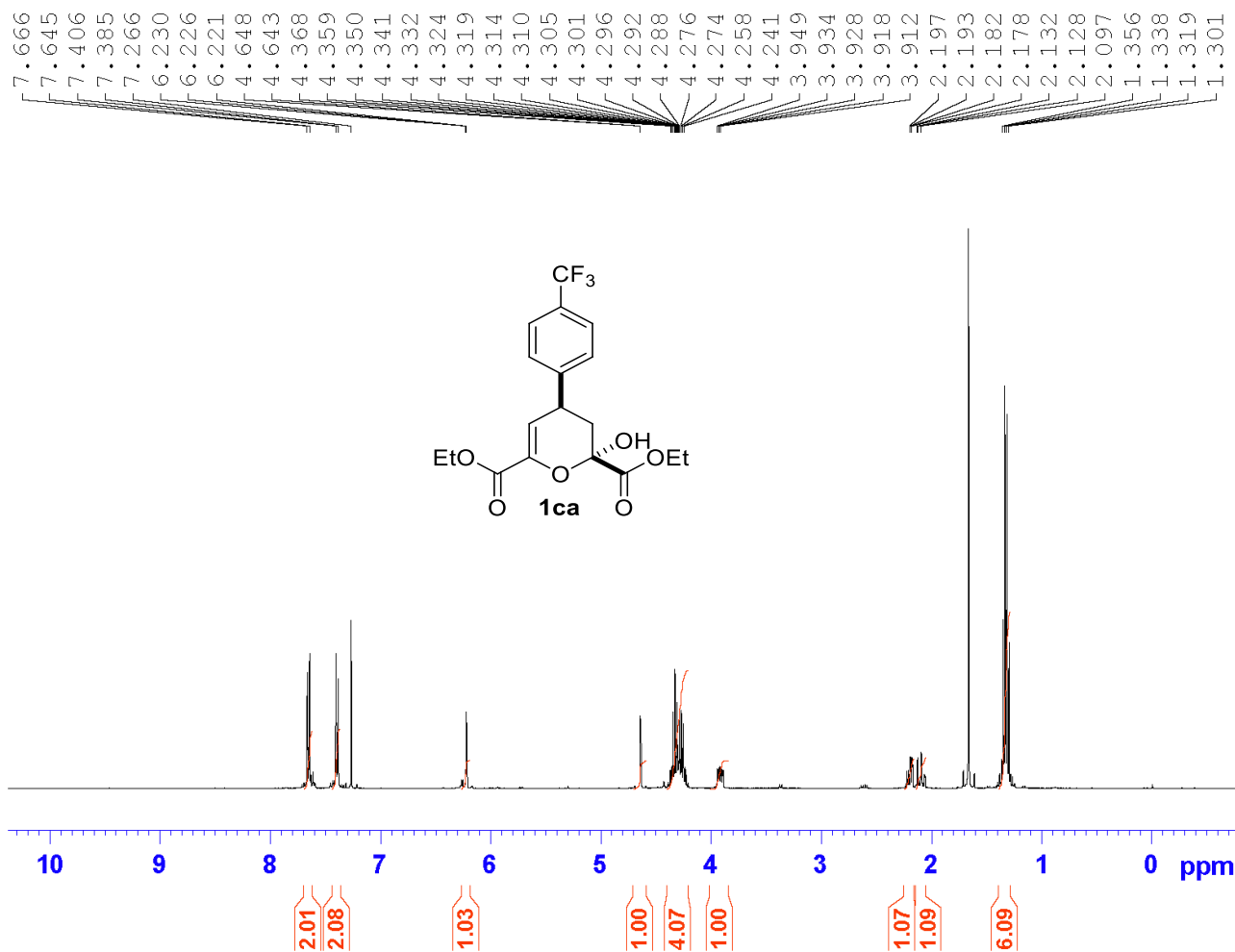
Current Data Parameters
 NAME May30-2012
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120531
 Time 1.04
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 3000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 161.3
 DW 20.800 usec
 DE 6.00 usec
 TE 296.9 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



Current Data Parameters
NAME May30-2012
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters

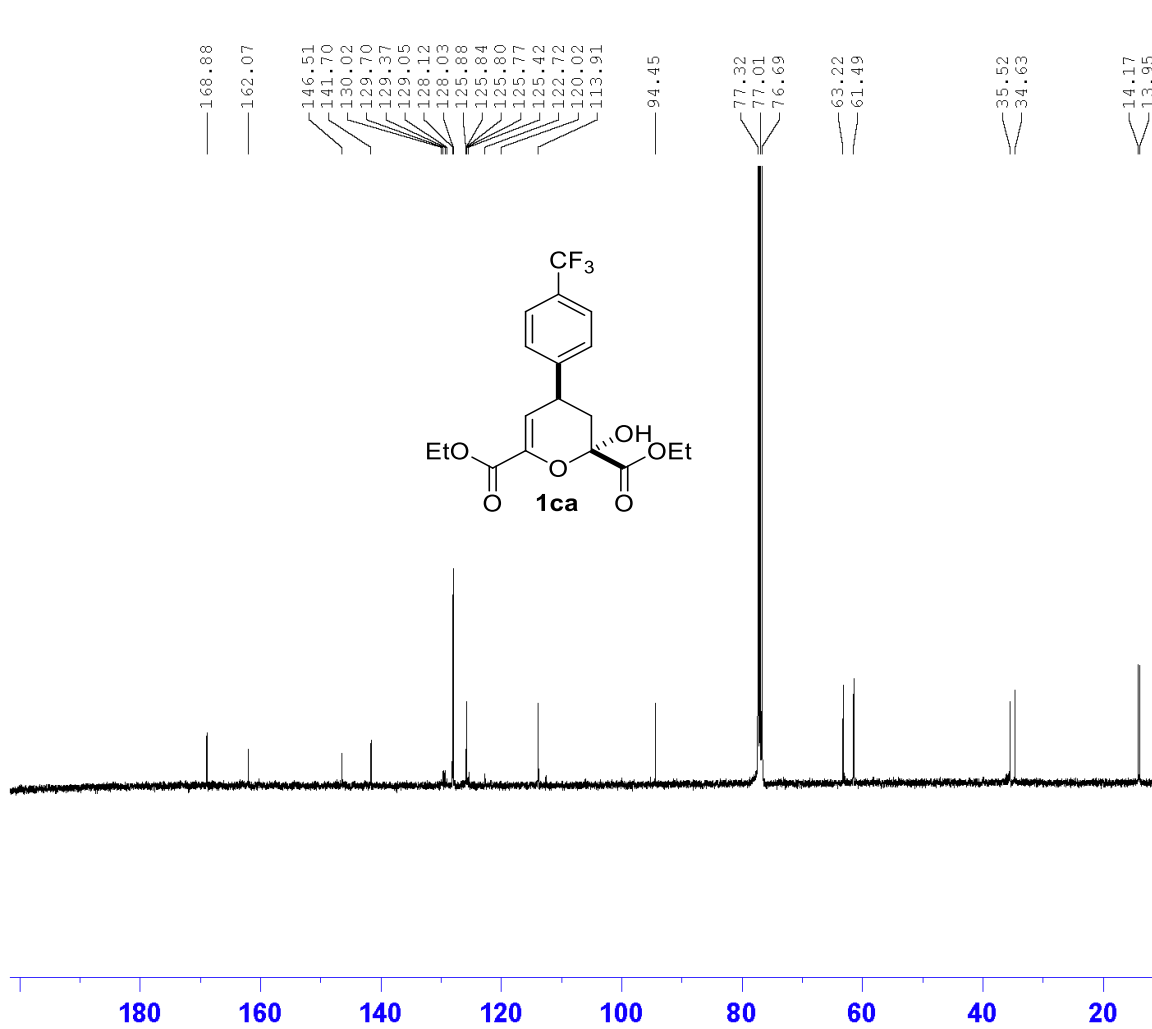
Date_ 20120530
Time 22.03
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715421 sec
RG 322.5
DW 60.600 usec
DE 6.00 usec
TE 295.5 K
D1 1.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 15.00 usec
PL1 2.70 dB
SFO1 400.2324716 MHz

F2 - Processing parameters

SI 32768
SF 400.2300029 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



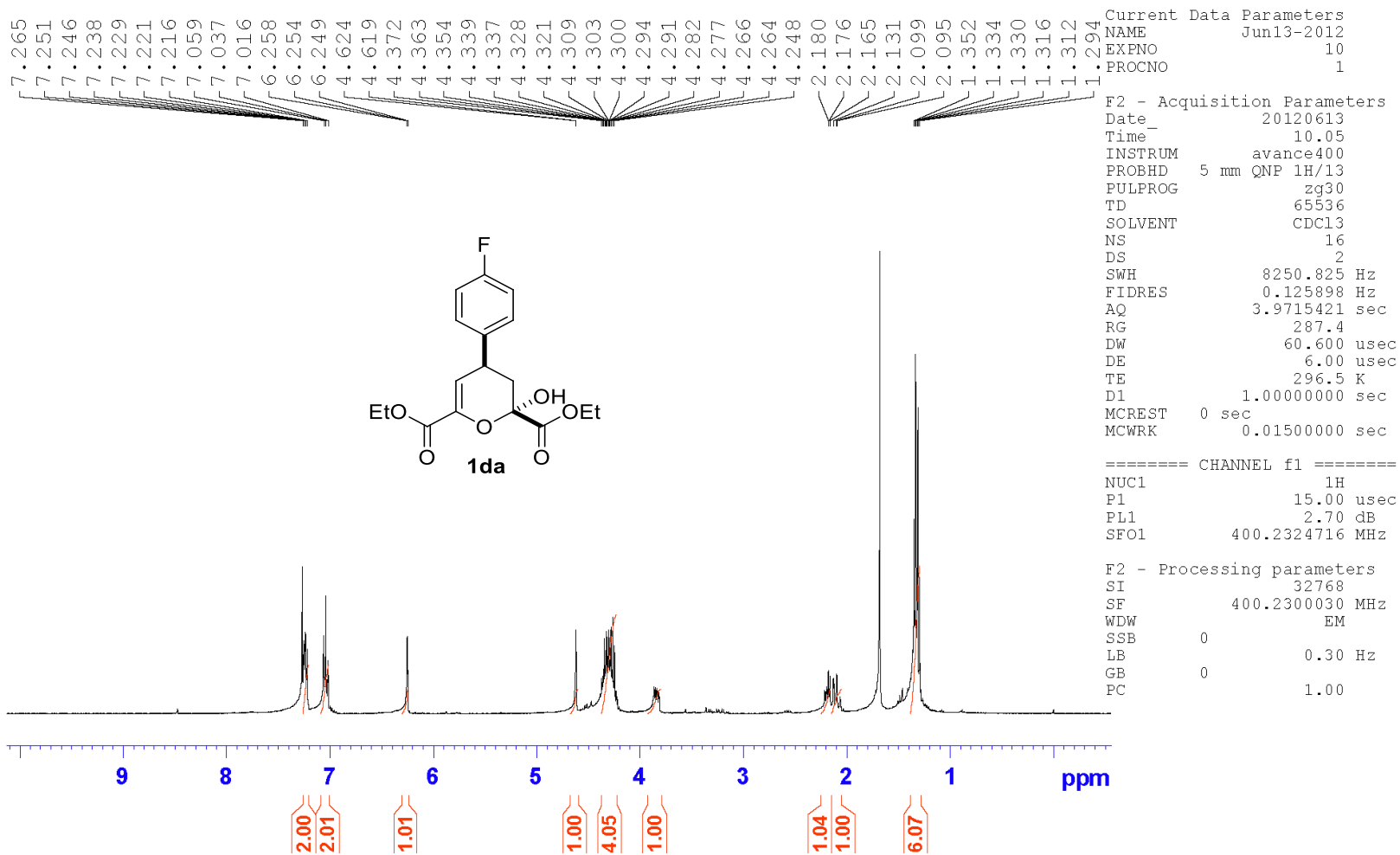
Current Data Parameters
 NAME Jun13-2012
 EXPNO 70
 PROCNO 1

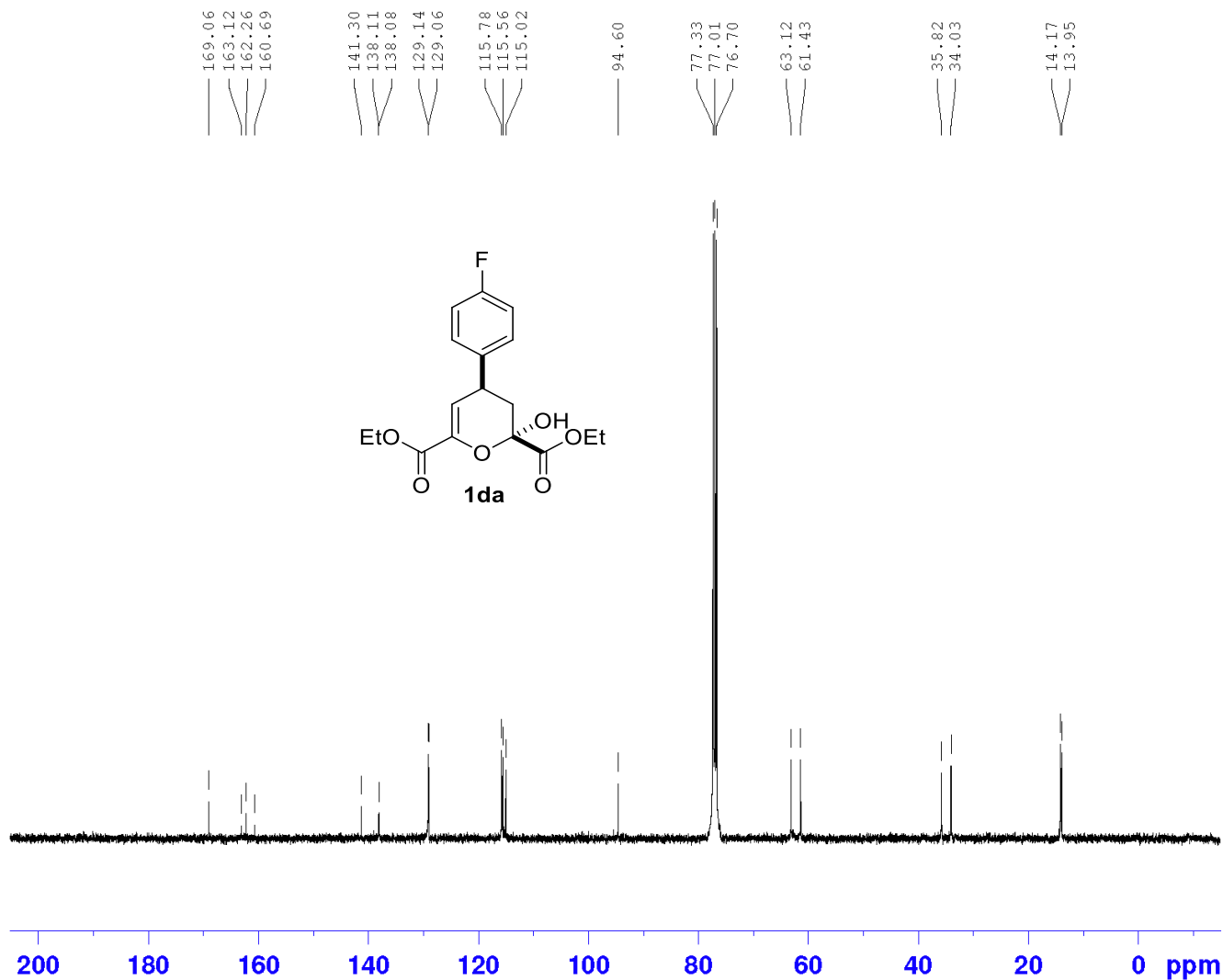
F2 - Acquisition Parameters
 Date_ 20120614
 Time_ 1.24
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 3500
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631696 sec
 RG 143.7
 DW 20.800 usec
 DE 6.00 usec
 TE 297.9 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





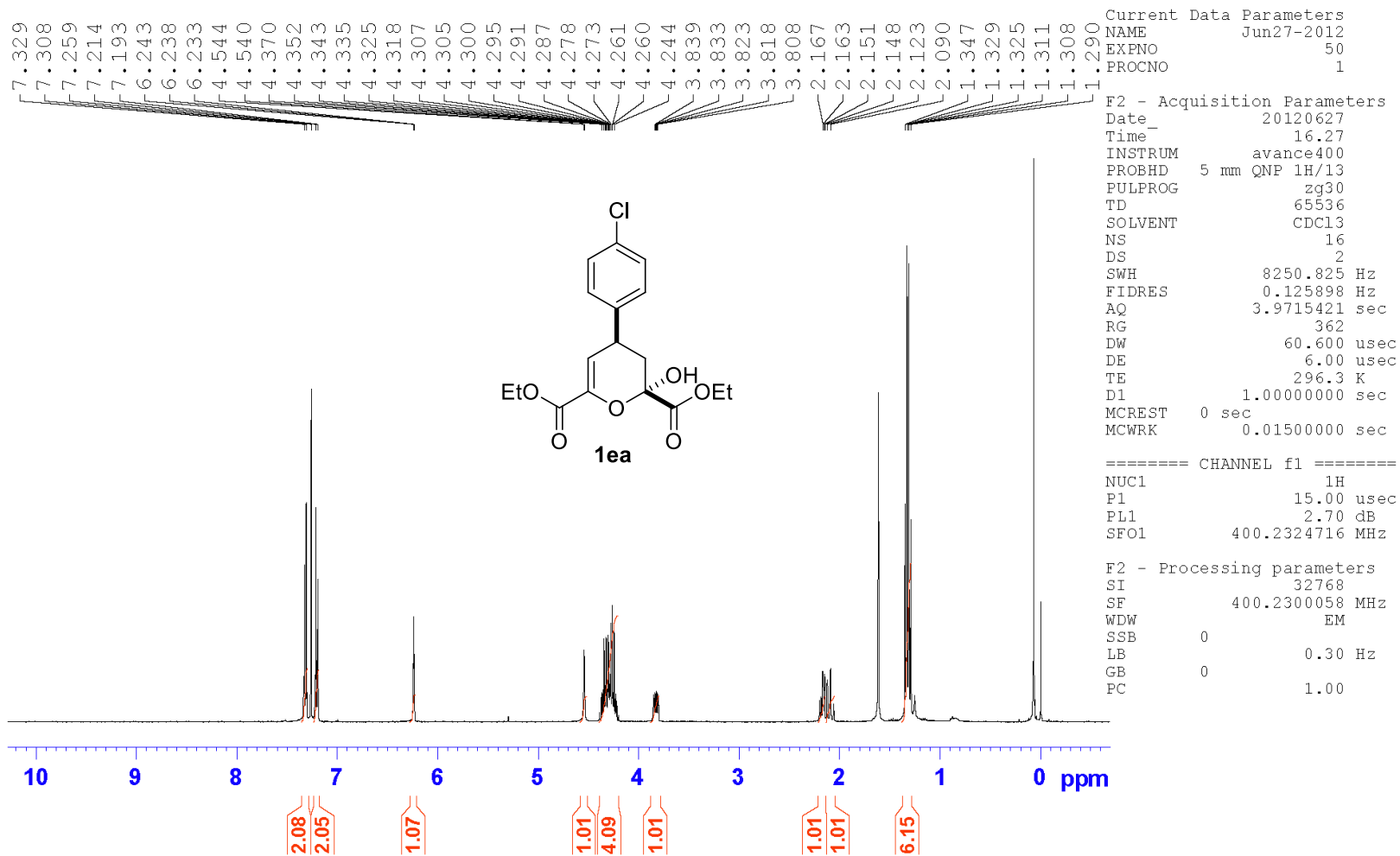
Current Data Parameters
NAME Jun14-2012
EXPNO 90
PROCNO 1

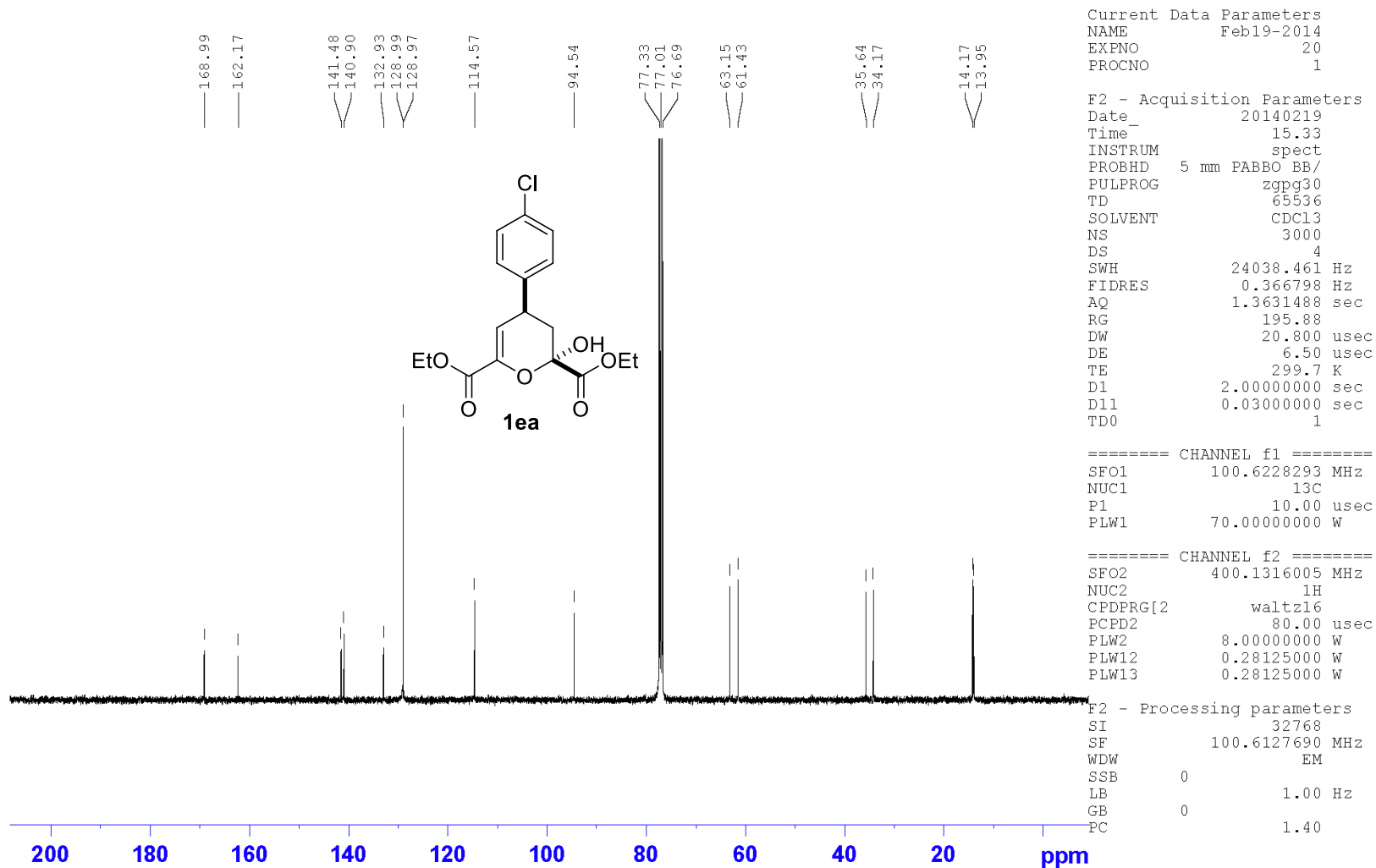
F2 - Acquisition Parameters
Date_ 20120615
Time 1.59
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 71.8
DW 20.800 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

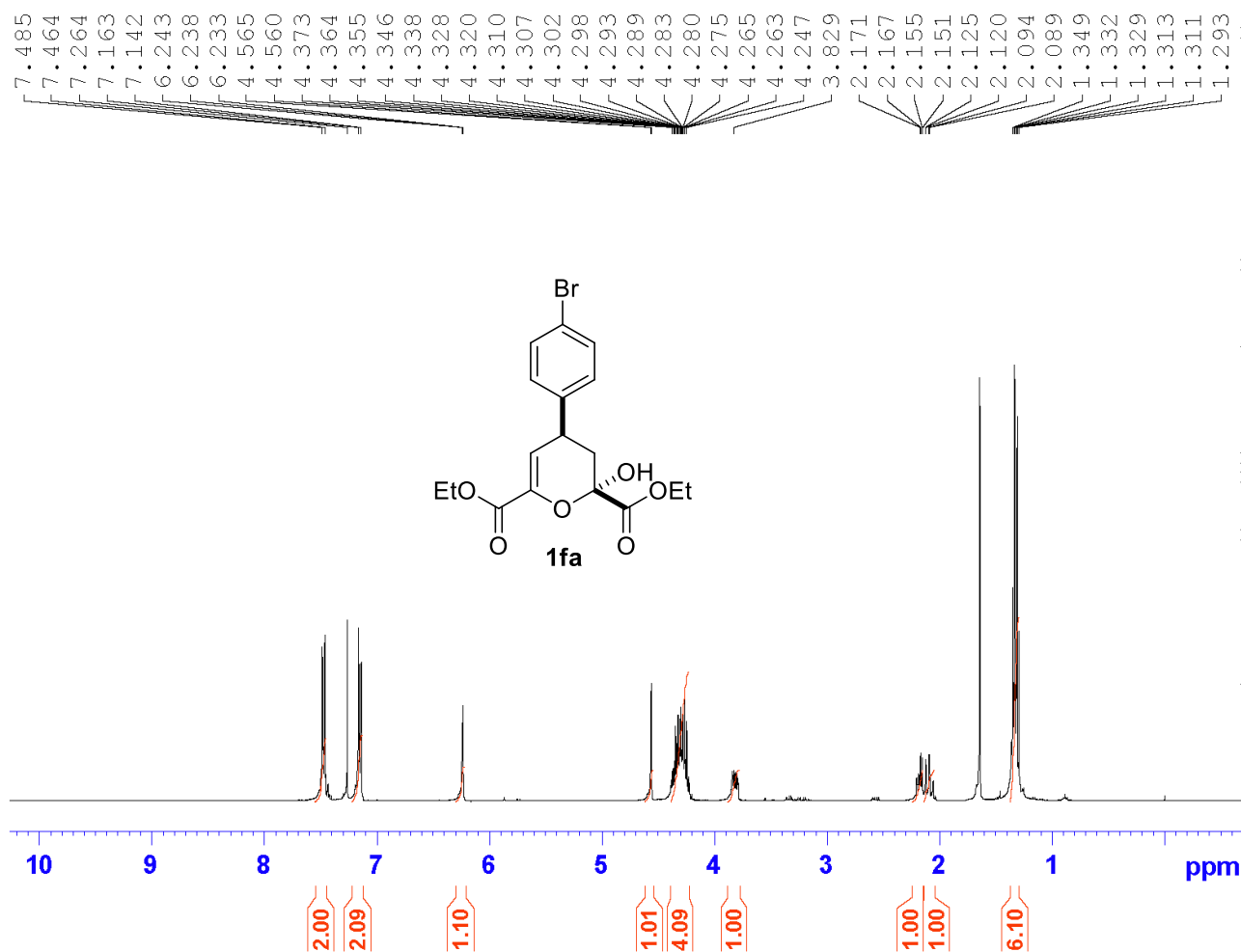
===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -2.00 dB
SFO1 100.6479773 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.70 dB
PL12 17.10 dB
PL13 17.10 dB
SFO2 400.2316009 MHz

F2 - Processing parameters
SI 32768
SF 100.6379140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





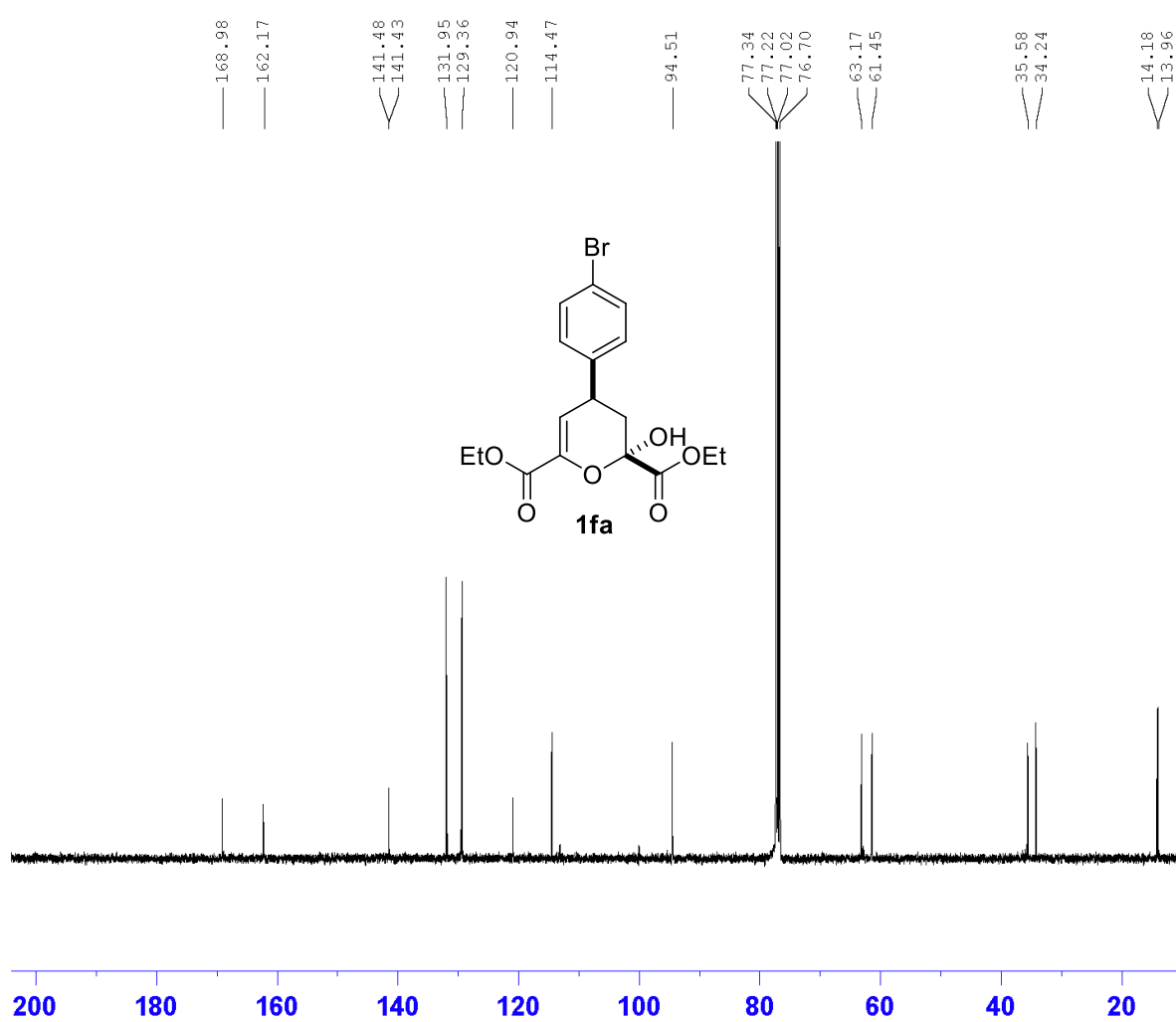


Current Data Parameters
 NAME May31-2012
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120531
 Time 21.12
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 322.5
 DW 60.600 usec
 DE 6.00 usec
 TE 295.5 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300037 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



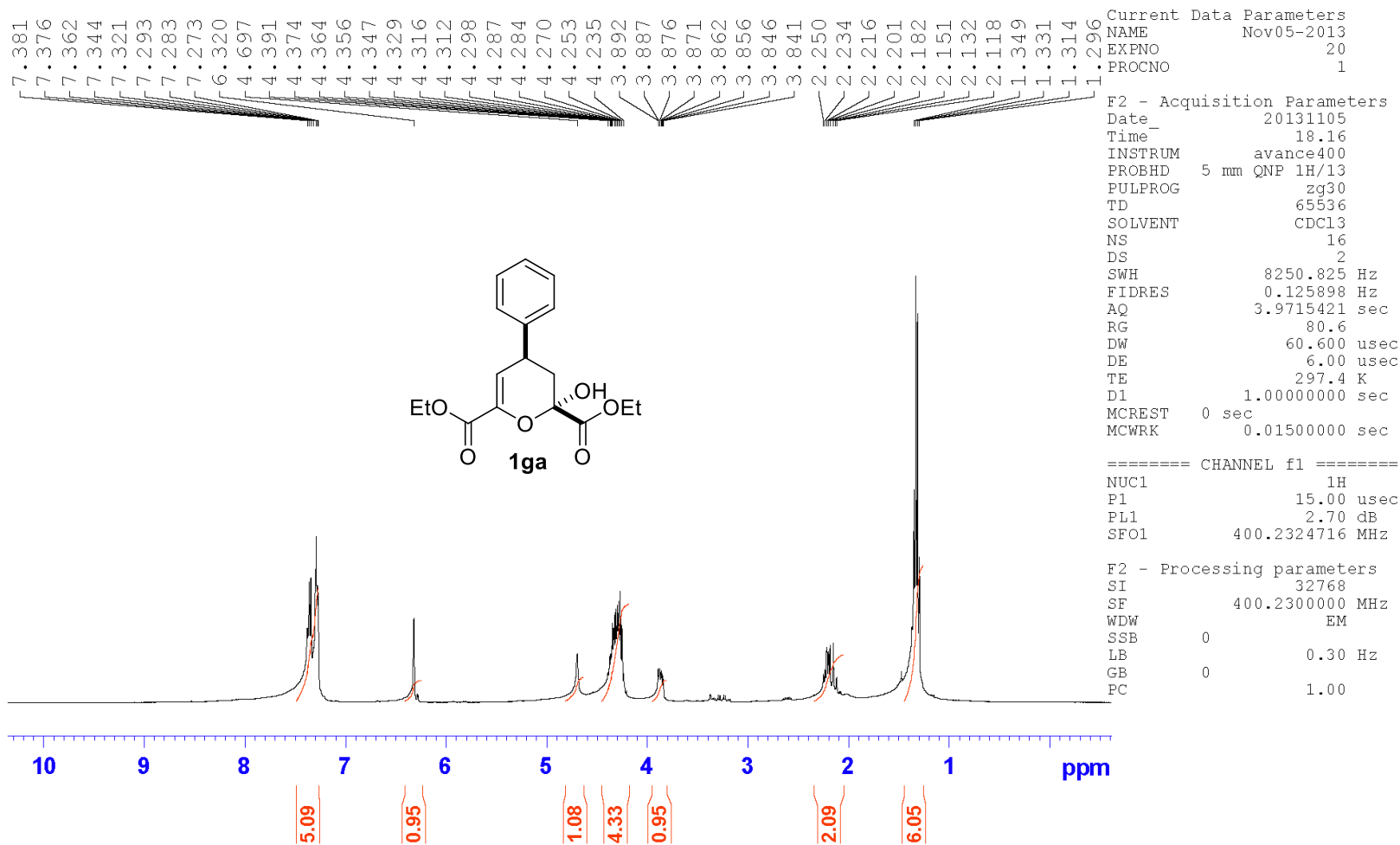
Current Data Parameters
 NAME May31-2012
 EXPNO 40
 PROCNO 1

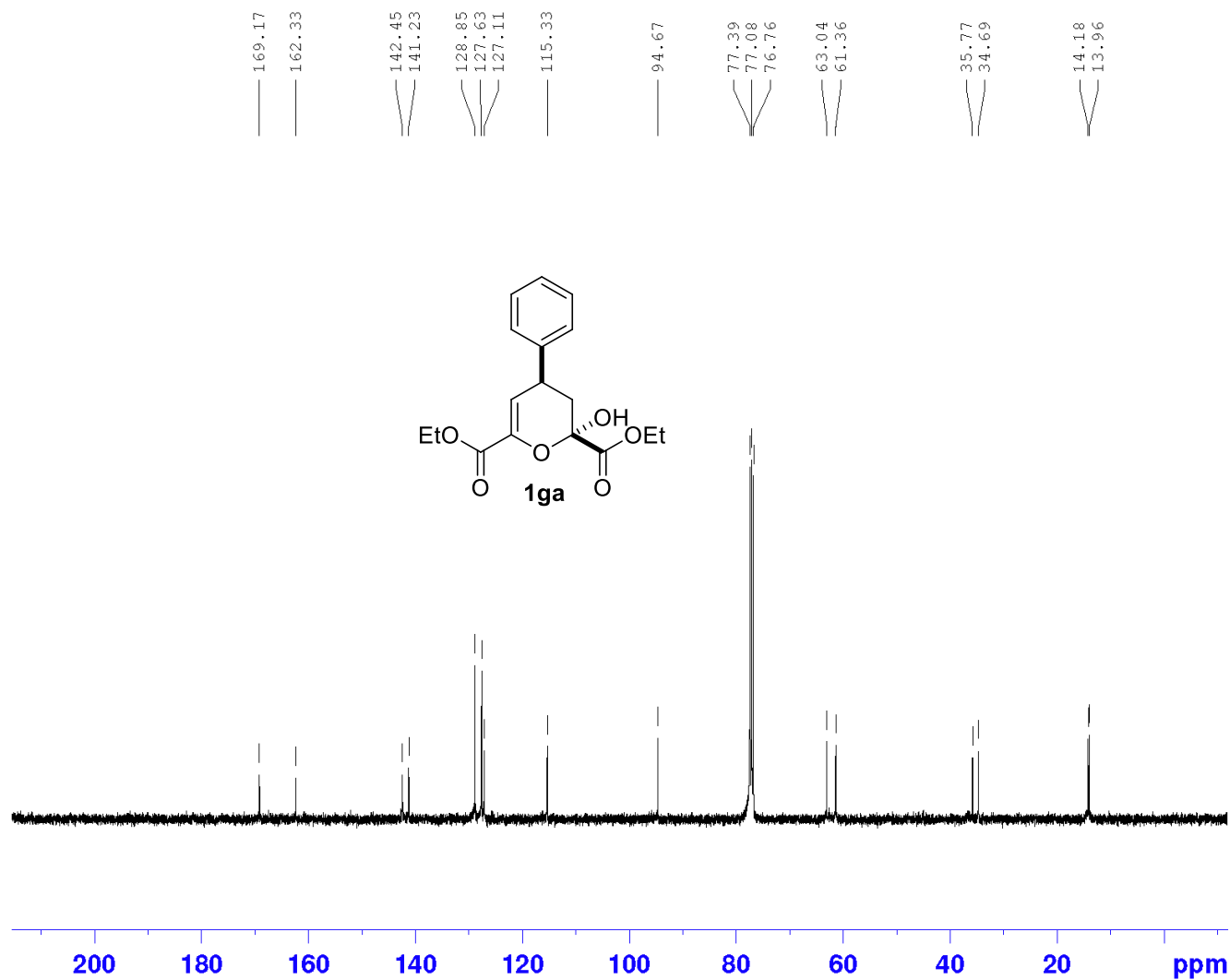
F2 - Acquisition Parameters
 Date_ 20120601
 Time_ 0.24
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 3000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631696 sec
 RG 101.6
 DW 20.800 usec
 DE 6.00 usec
 TE 297.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





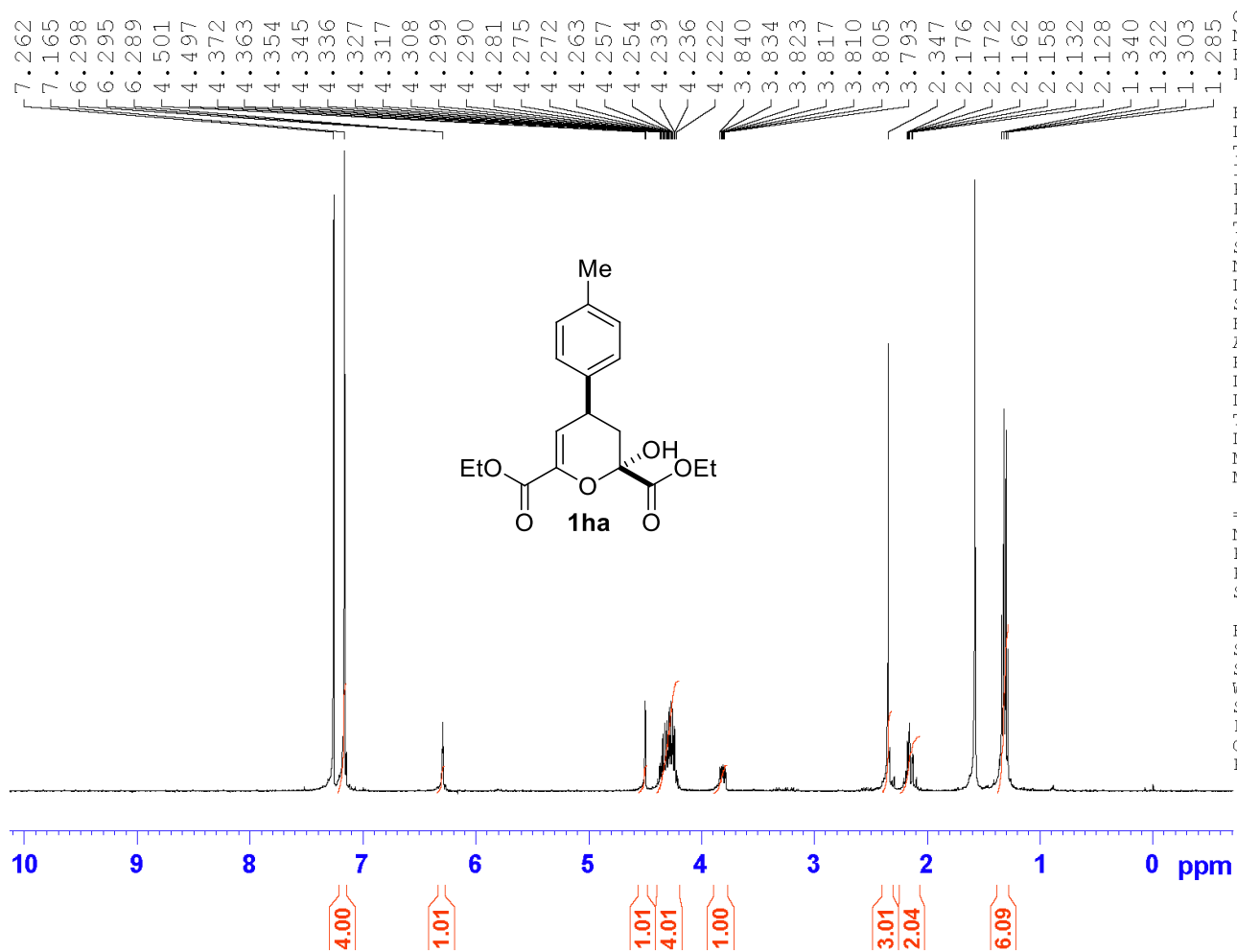
Current Data Parameters
 NAME Nov05-2013
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20131105
 Time 18.00
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 26008
 DW 20.800 usec
 DE 6.00 usec
 TE 298.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

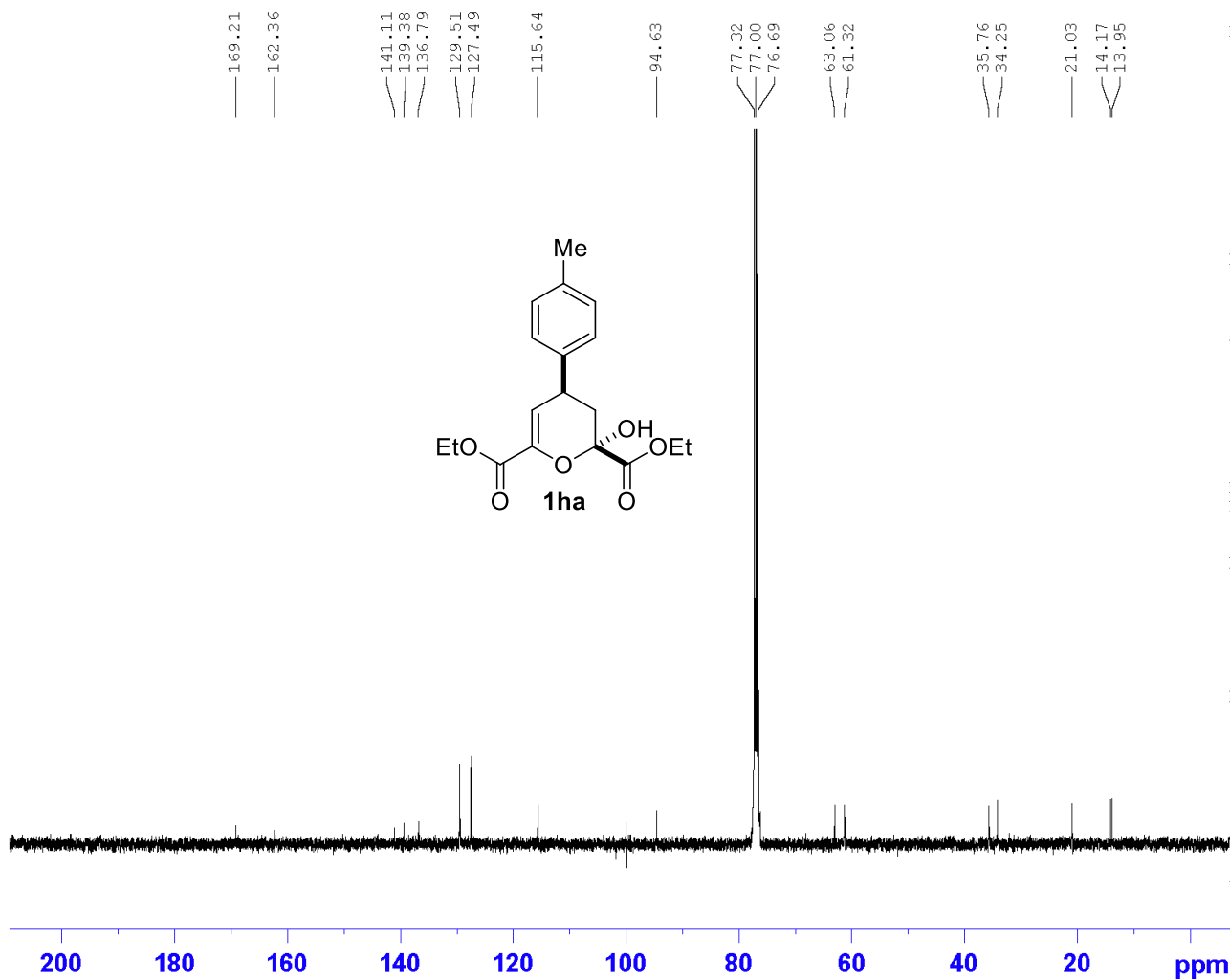


Current Data Parameters
 NAME Jun22-2012
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120622
 Time 11.41
 INSTRUM advance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 512
 DW 60.600 usec
 DE 6.00 usec
 TE 296.3 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300047 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



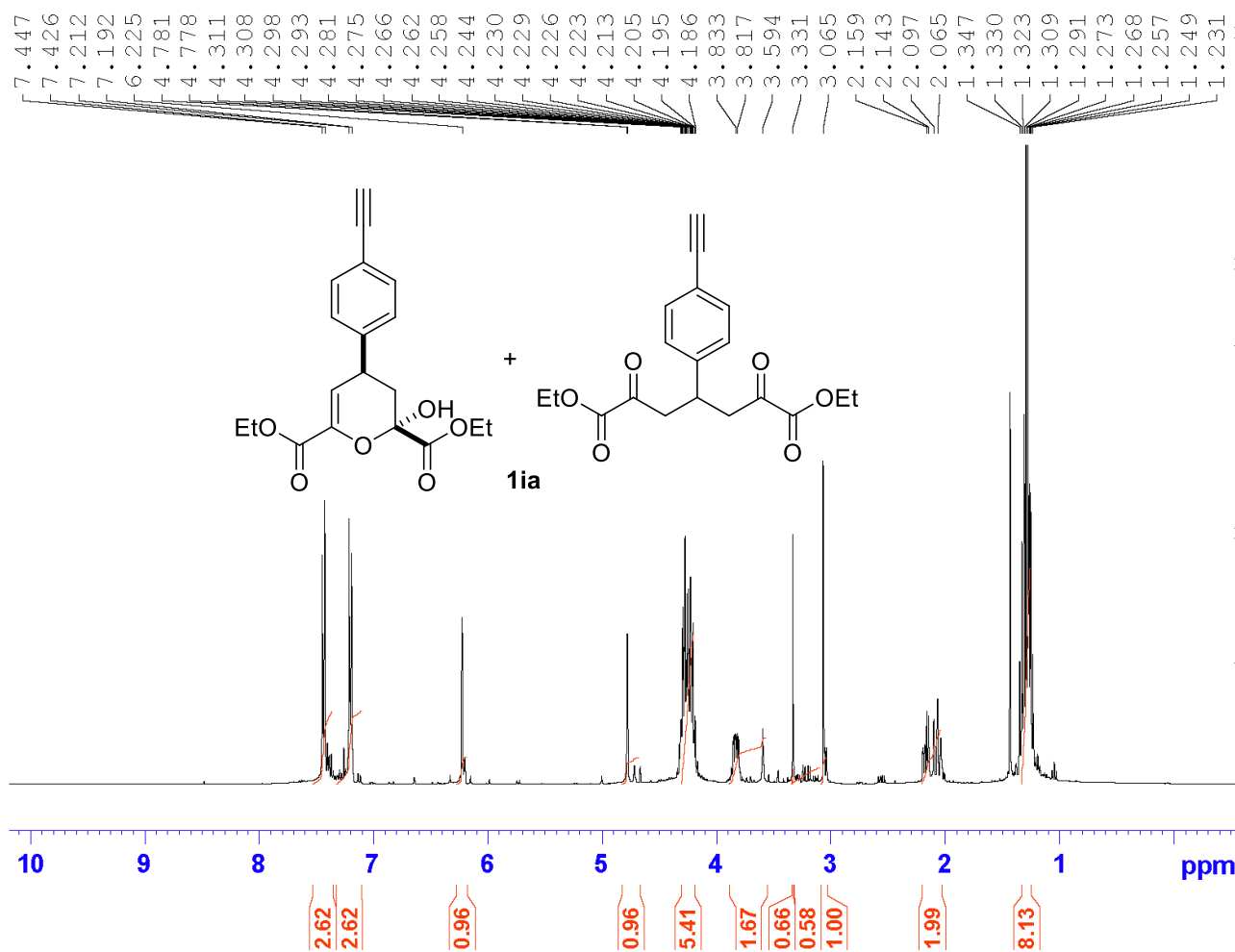
Current Data Parameters
 NAME Jun21-2012
 EXPNO 70
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120622
 Time_ 2.11
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 4000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631696 sec
 RG 80.6
 DW 20.800 usec
 DE 6.00 usec
 TE 297.9 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

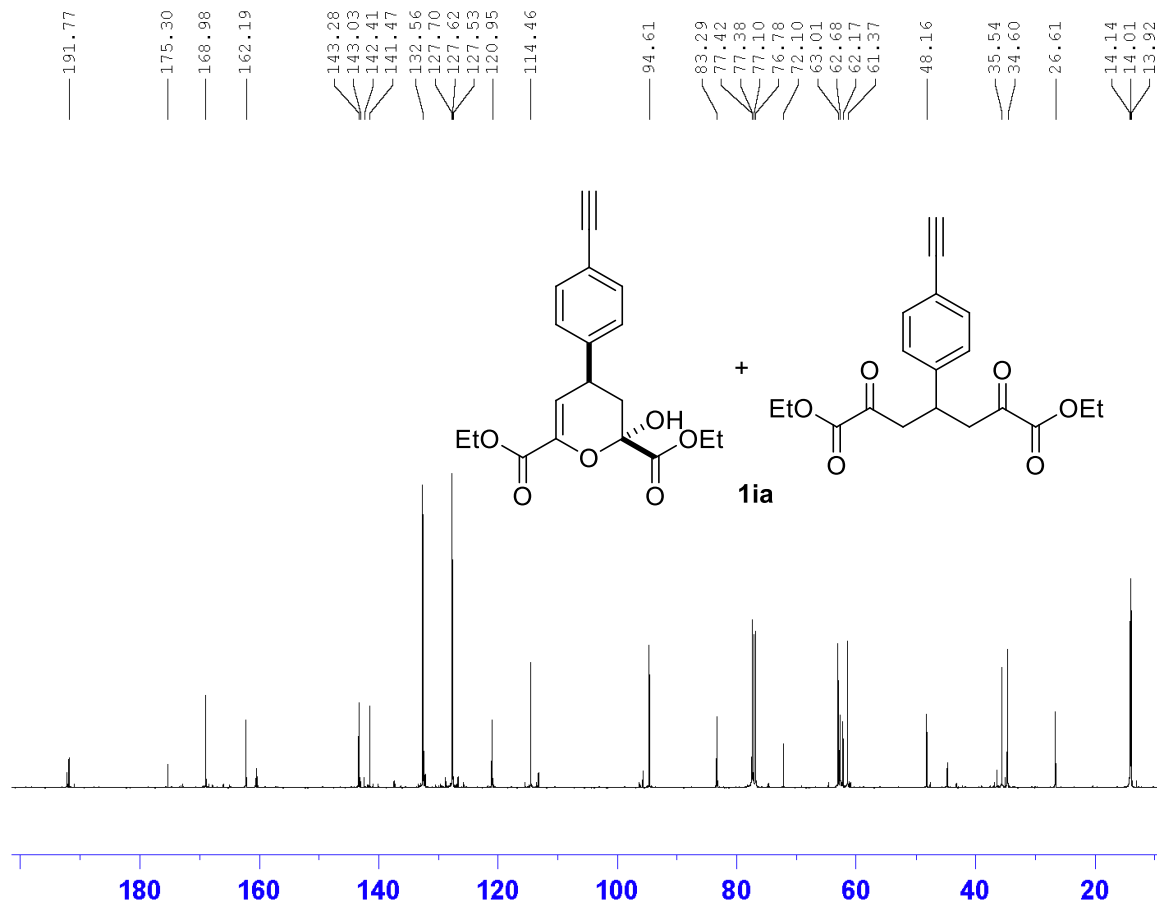


Current Data Parameters
 NAME Mar10-2014
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140310
 Time 16.21
 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 17.32
 DW 62.400 usec
 DE 6.50 usec
 TE 299.9 K
 D1 1.00000000 sec
 TD0 1

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

F2 - Processing parameters
 SI 65536
 SF 400.1300101 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



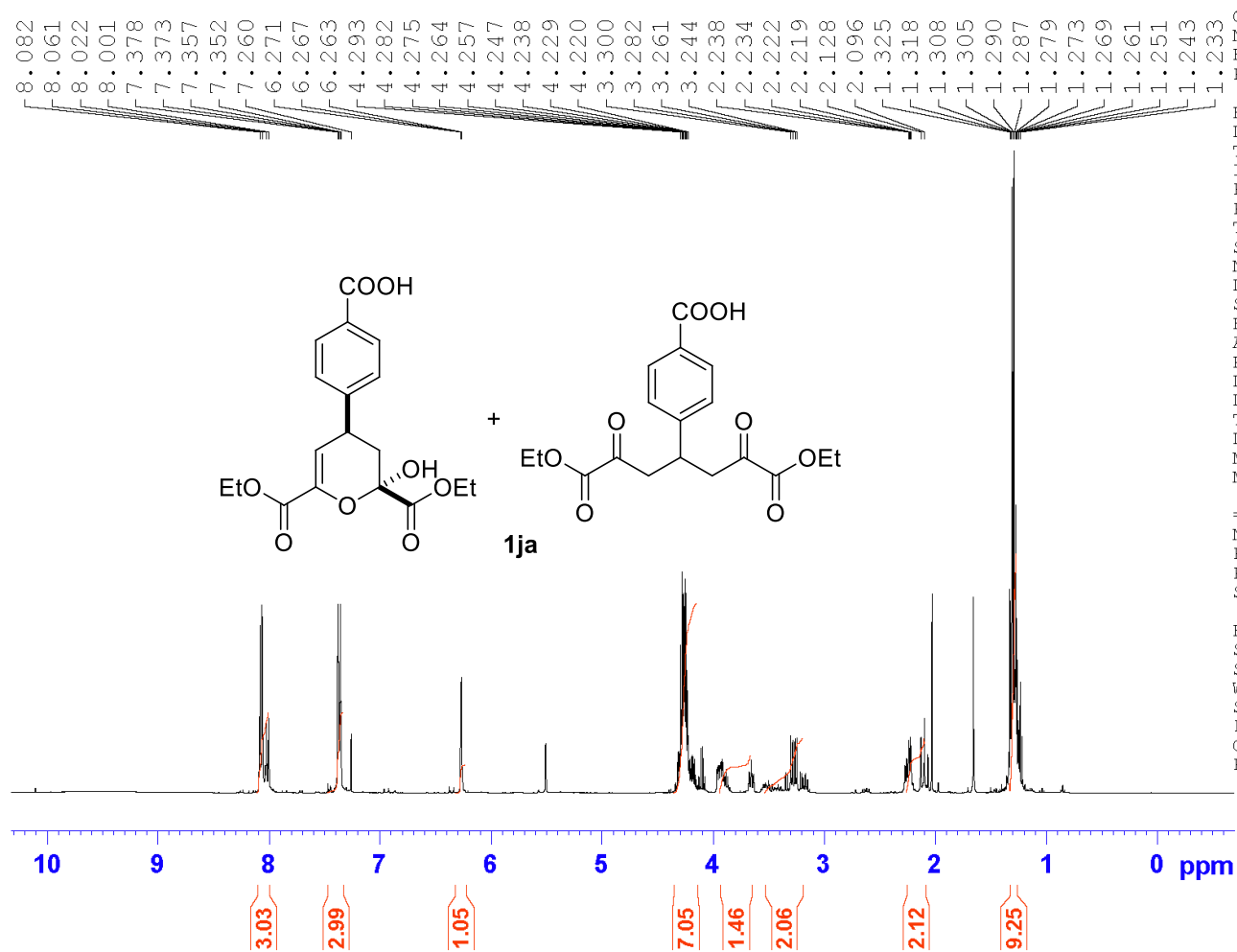
Current Data Parameters
NAME Mar10-2014
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140310
Time_ 20.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 195.88
DW 20.800 usec
DE 6.50 usec
TE 300.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

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

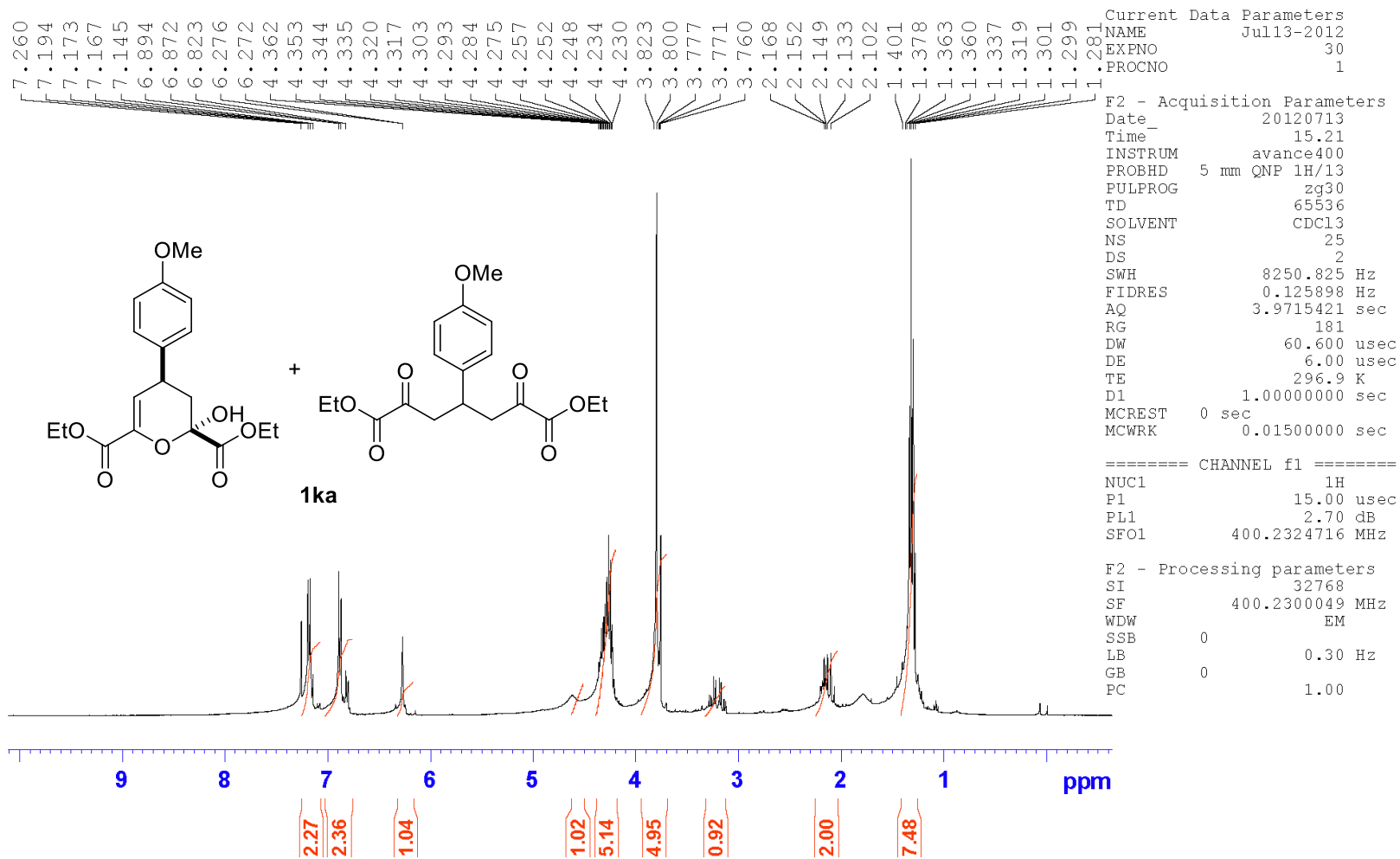


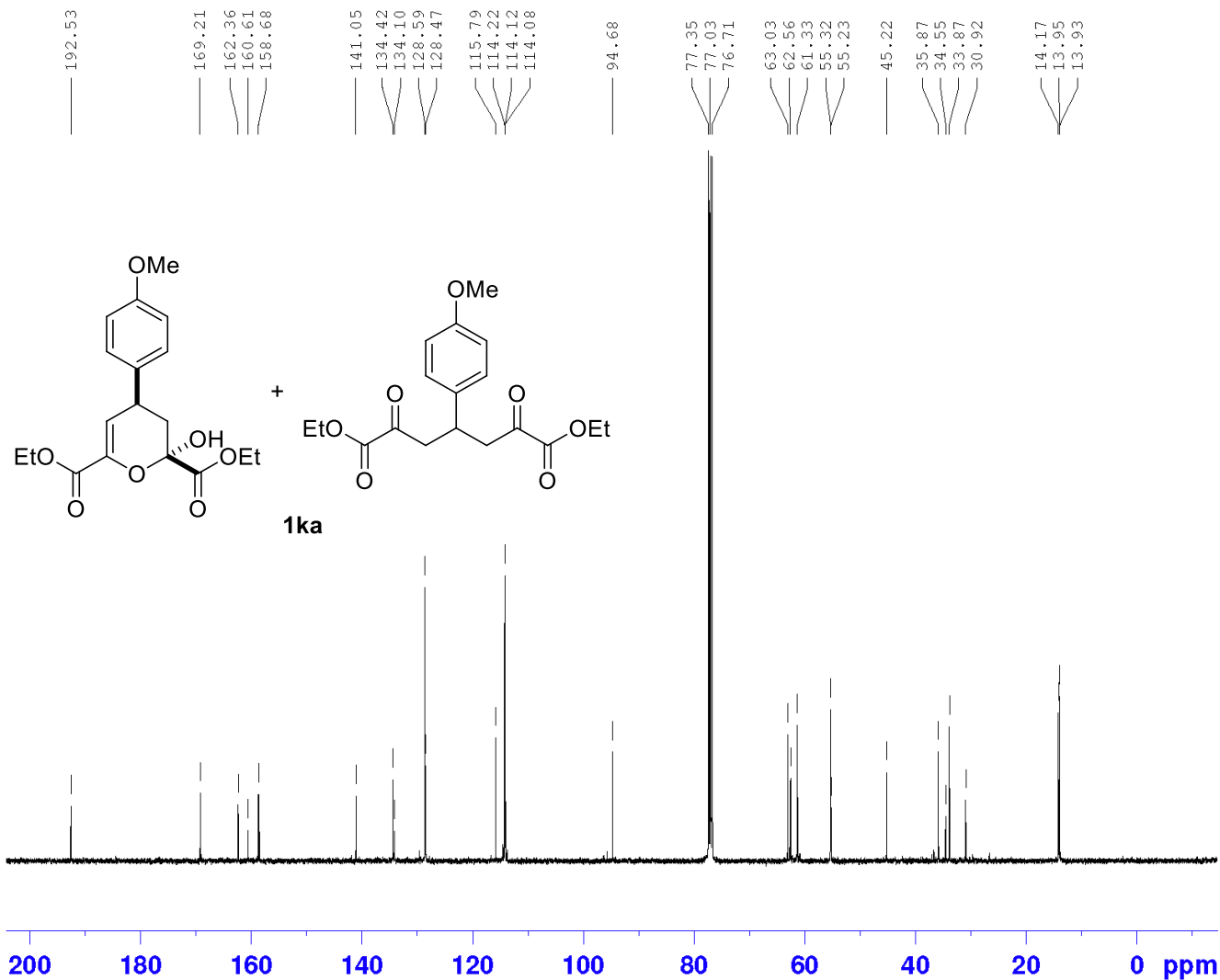
Current Data Parameters
NAME Nov06-2013
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131106
Time 12.22
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715421 sec
RG 57
DW 60.600 usec
DE 6.00 usec
TE 297.2 K
D1 1.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 2.70 dB
SFO1 400.2324716 MHz

F2 - Processing parameters
SI 32768
SF 400.2300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





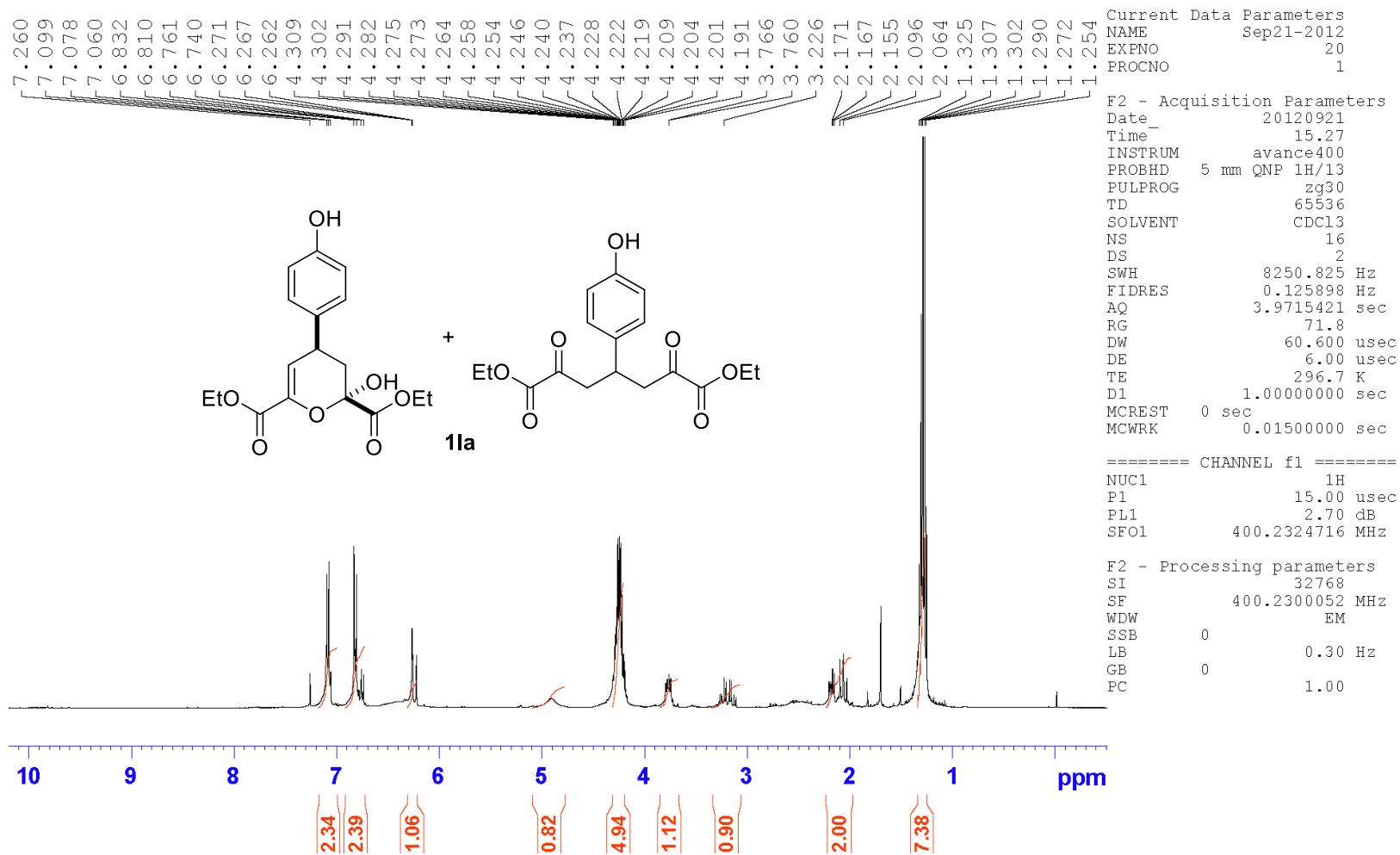
Current Data Parameters
NAME Jul16-2012
EXPNO 20
PROCNO 1

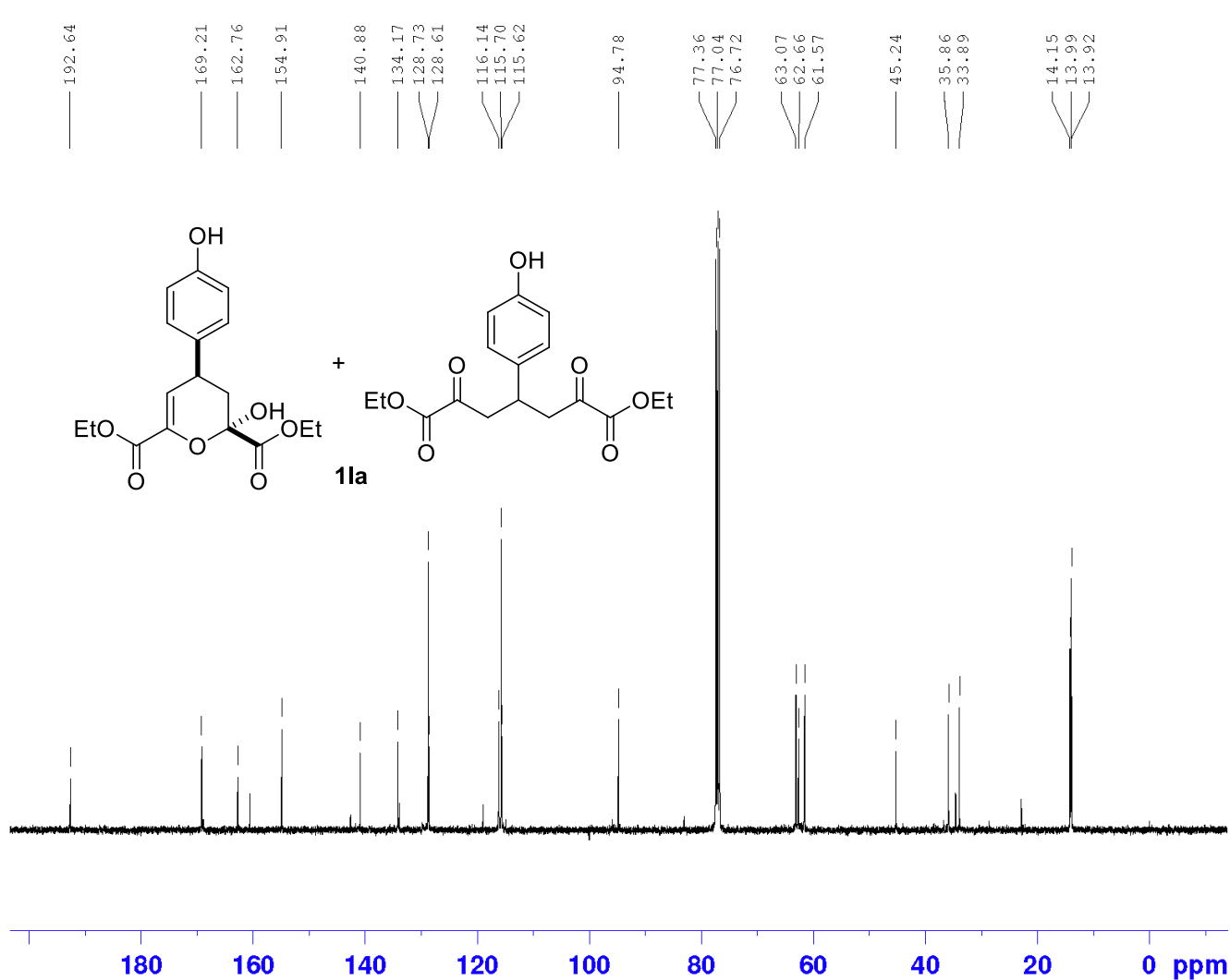
F2 - Acquisition Parameters
Date_ 20120717
Time 1.43
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 161.3
DW 20.800 usec
DE 6.00 usec
TE 298.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -2.00 dB
SFO1 100.6479773 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.70 dB
PL12 17.10 dB
PL13 17.10 dB
SFO2 400.2316009 MHz

F2 - Processing parameters
SI 32768
SF 100.6379140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





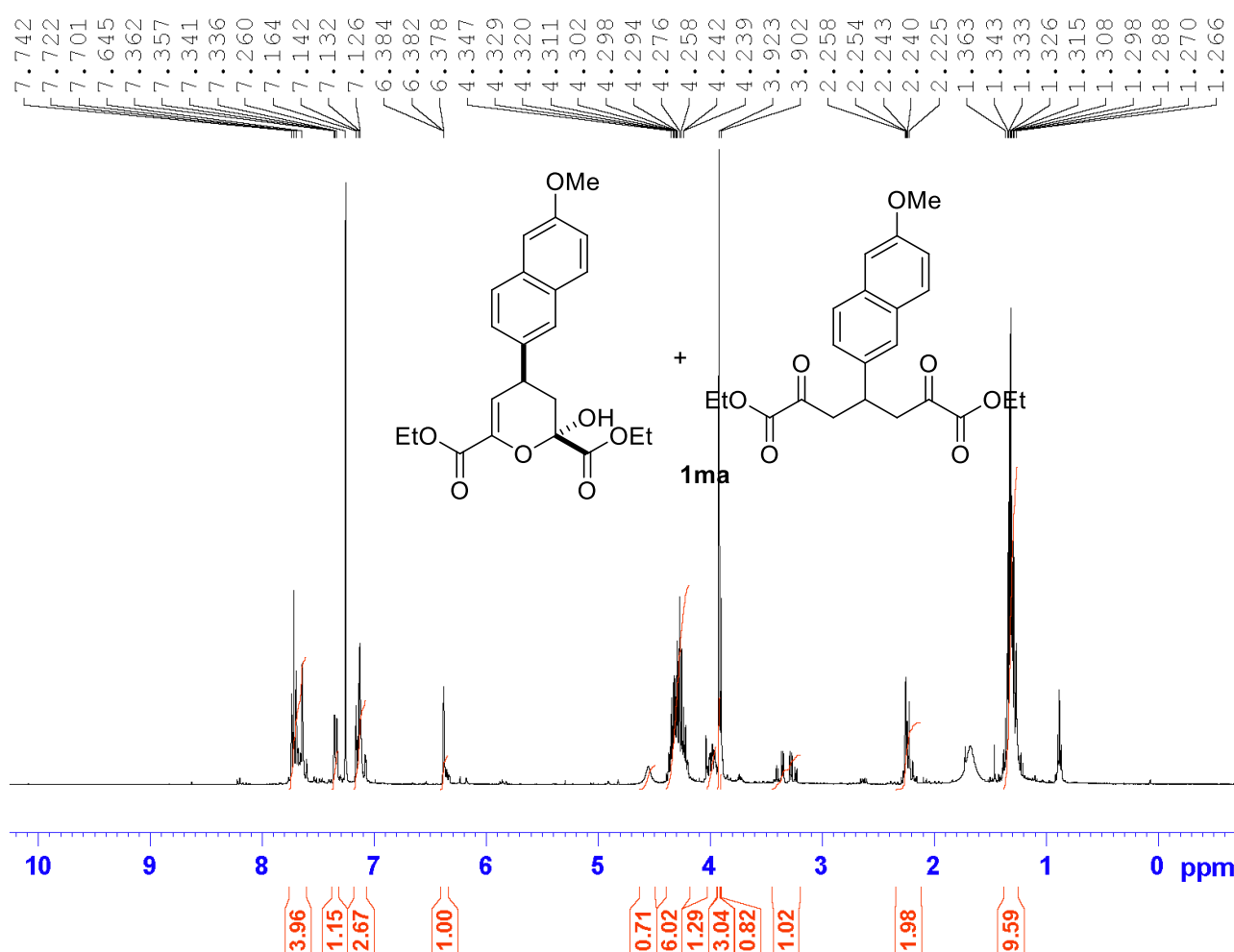
Current Data Parameters
 NAME Sep25-2012
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120925
 Time 19.27
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 71.8
 DW 20.800 usec
 DE 6.00 usec
 TE 297.7 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

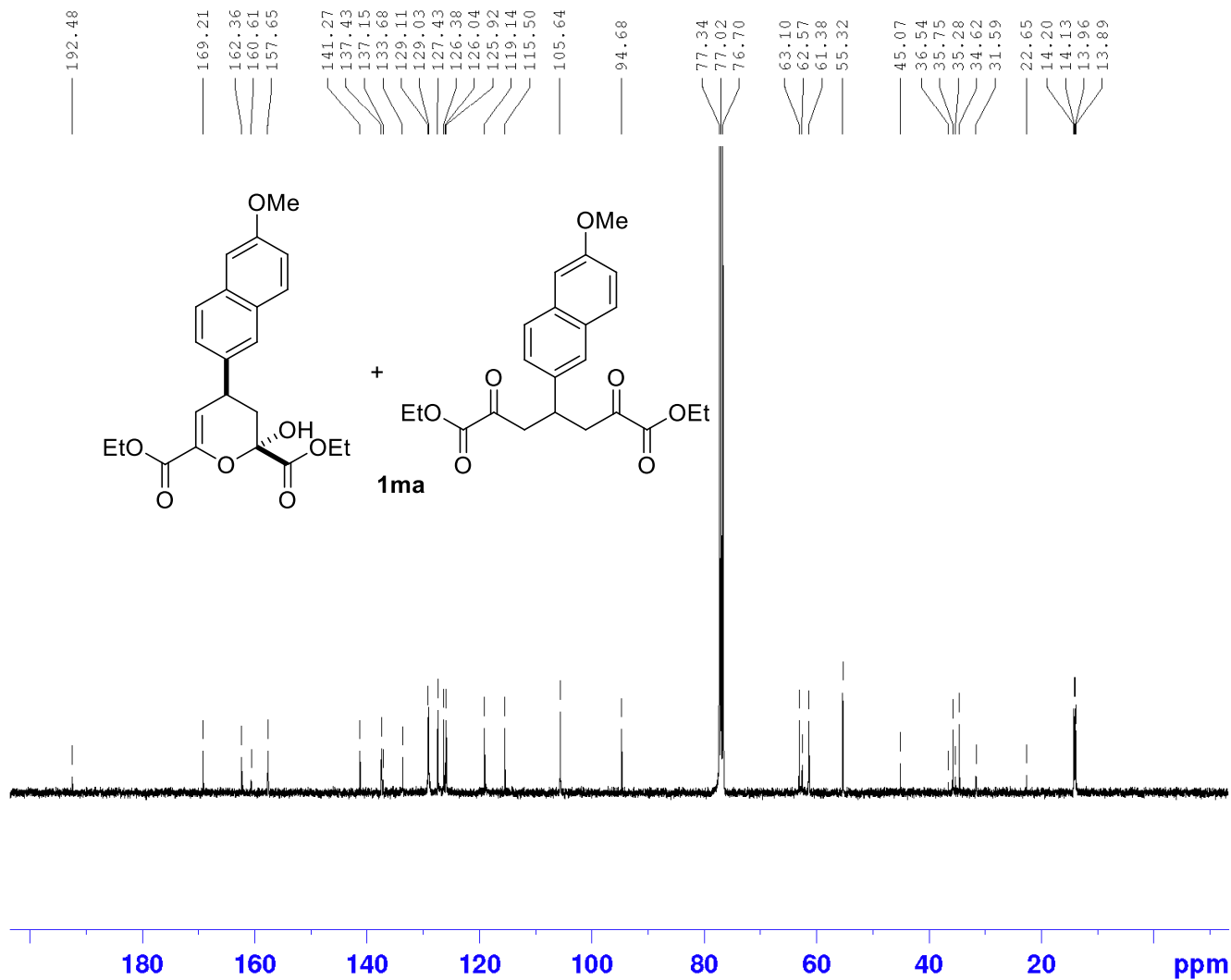


Current Data Parameters
NAME Jun19-2014
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140619
Time 12.22
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 71.01
DW 62.400 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1

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

F2 - Processing parameters
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



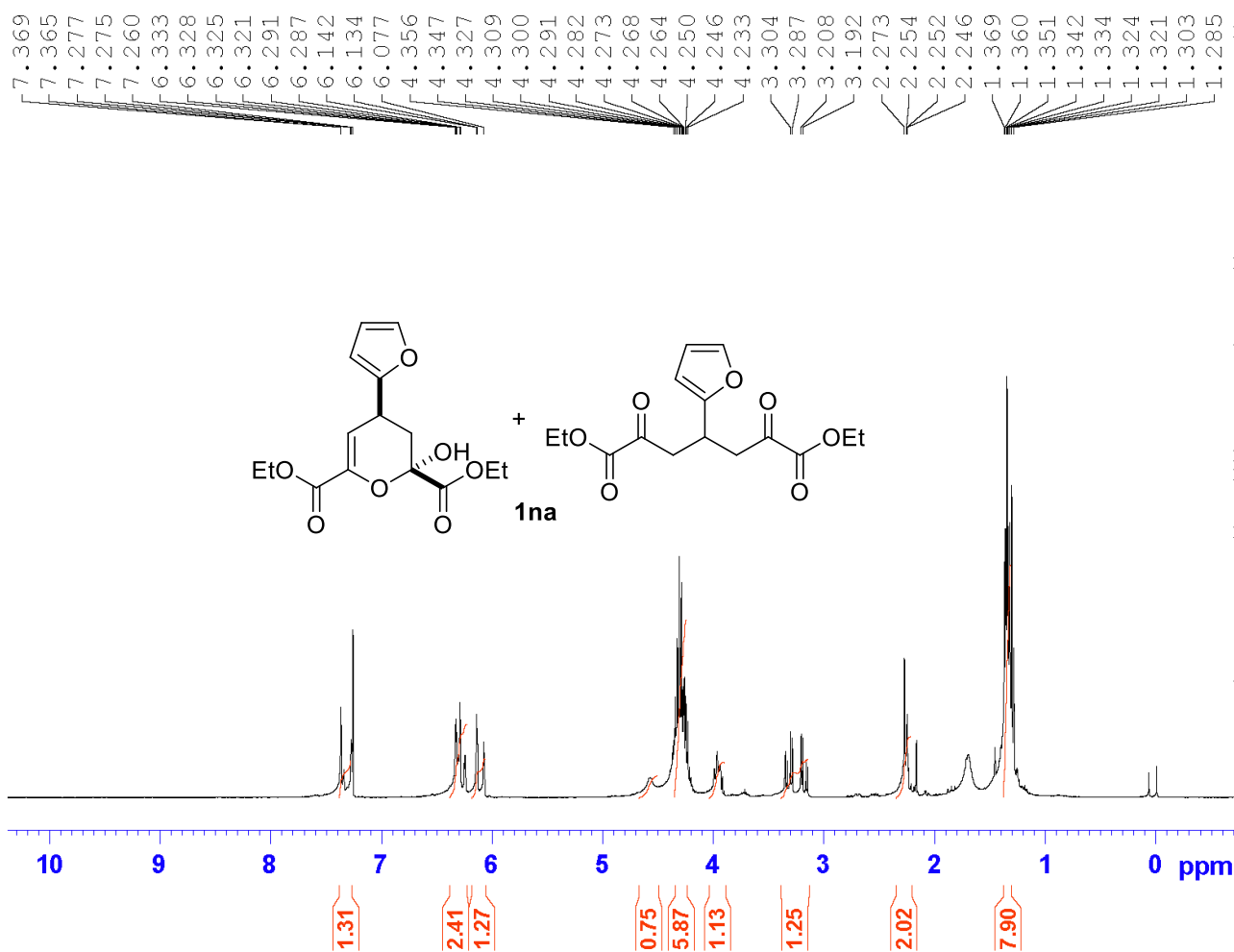
Current Data Parameters
 NAME Dec14-2012
 EXPNO 60
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121214
 Time 23.08
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 5000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 812.7
 DW 20.800 usec
 DE 6.00 usec
 TE 297.6 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

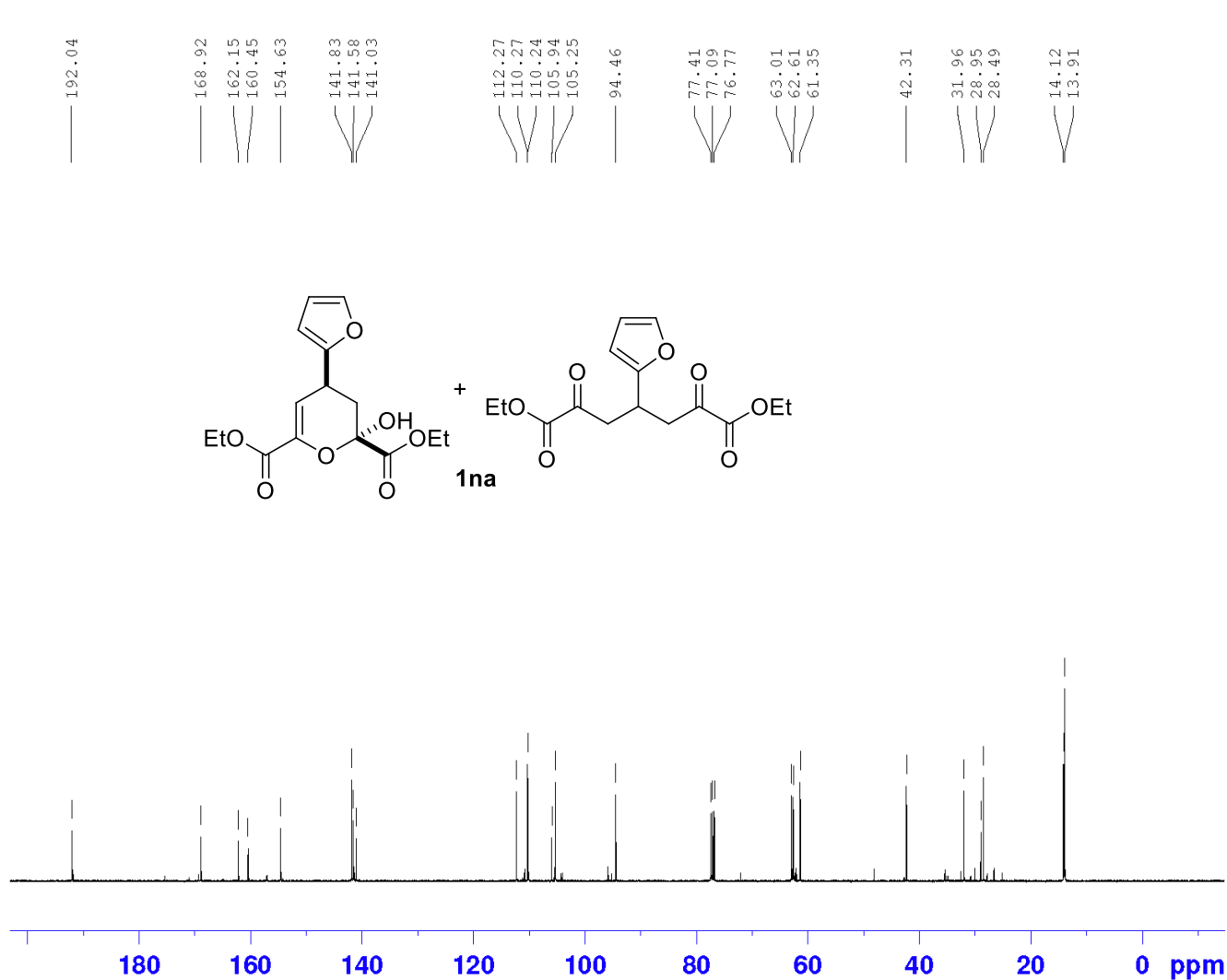


Current Data Parameters
NAME Jul17-2012
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120717
Time 22.17
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715421 sec
RG 228.1
DW 60.600 usec
DE 6.00 usec
TE 297.0 K
D1 1.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 2.70 dB
SFO1 400.2324716 MHz

F2 - Processing parameters
SI 32768
SF 400.2300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



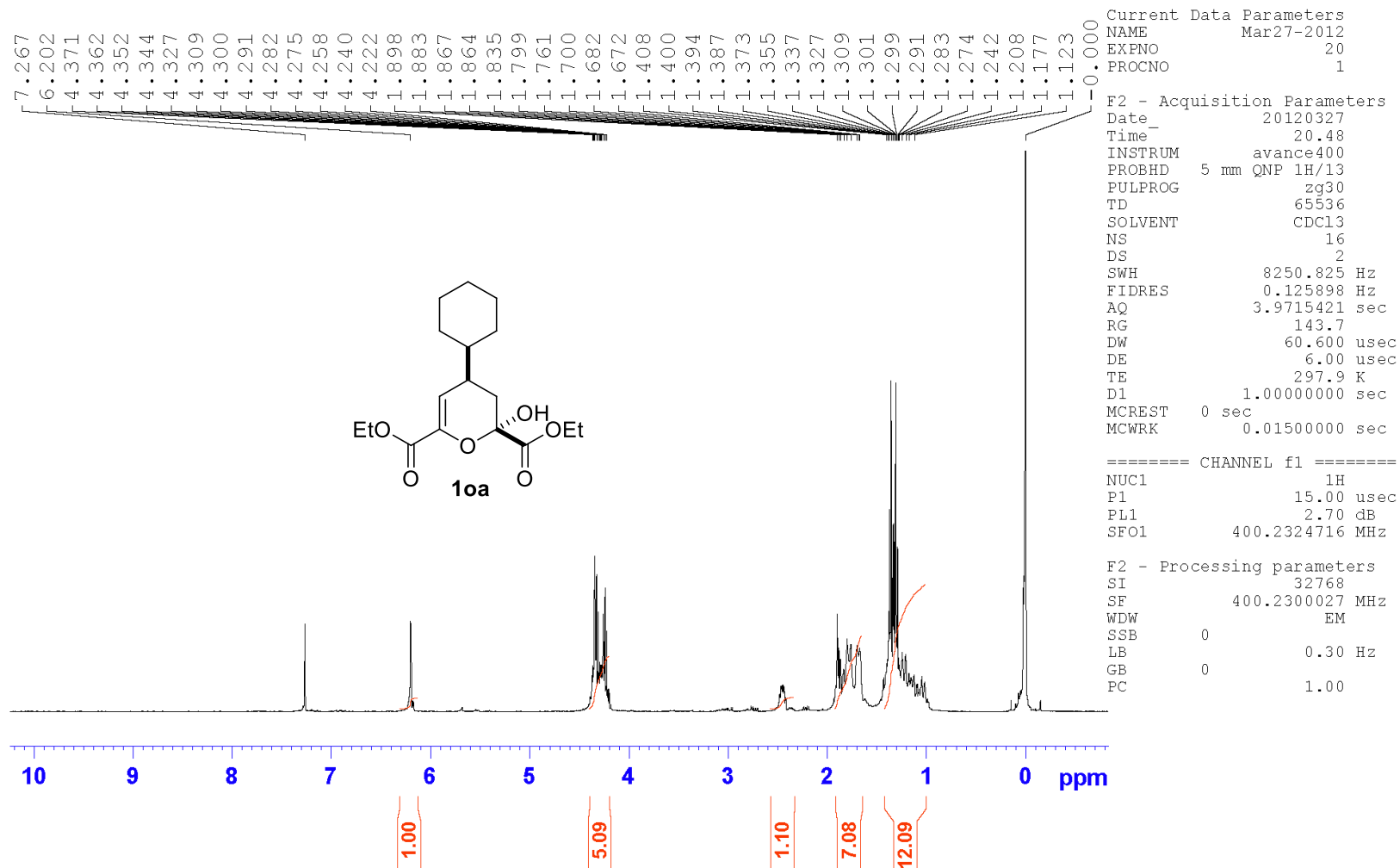
Current Data Parameters
NAME Mar15-2012
EXPNO 40
PROCNO 1

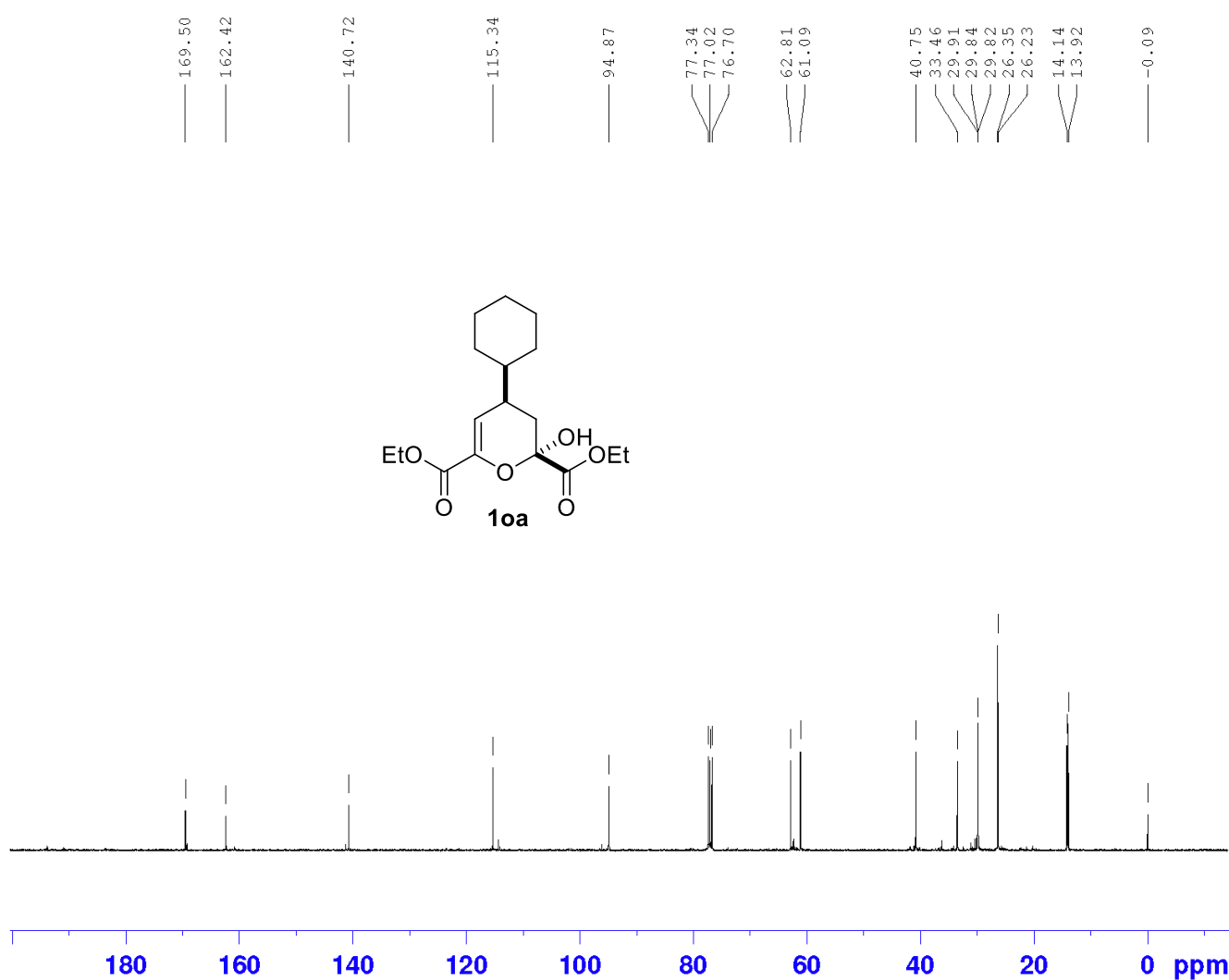
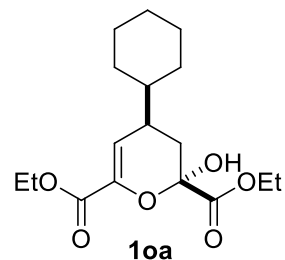
F2 - Acquisition Parameters
Date_ 20120315
Time 19.00
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 128
DW 20.800 usec
DE 6.00 usec
TE 298.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -2.00 dB
SFO1 100.6479773 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.70 dB
PL12 17.10 dB
PL13 17.10 dB
SFO2 400.2316009 MHz

F2 - Processing parameters
SI 32768
SF 100.6379140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





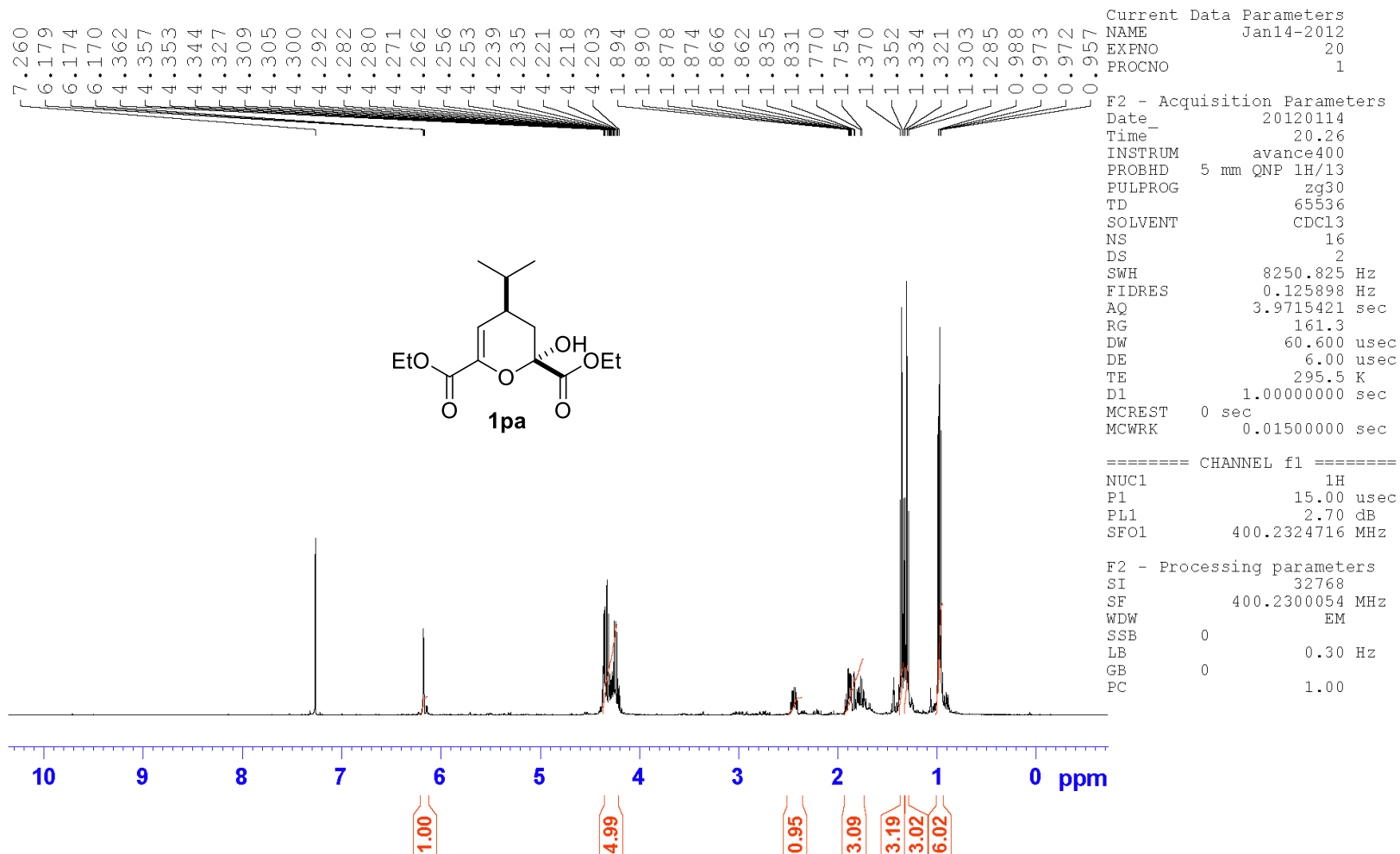
Current Data Parameters
 NAME Mar27-2012
 EXPNO 10
 PROCNO 1

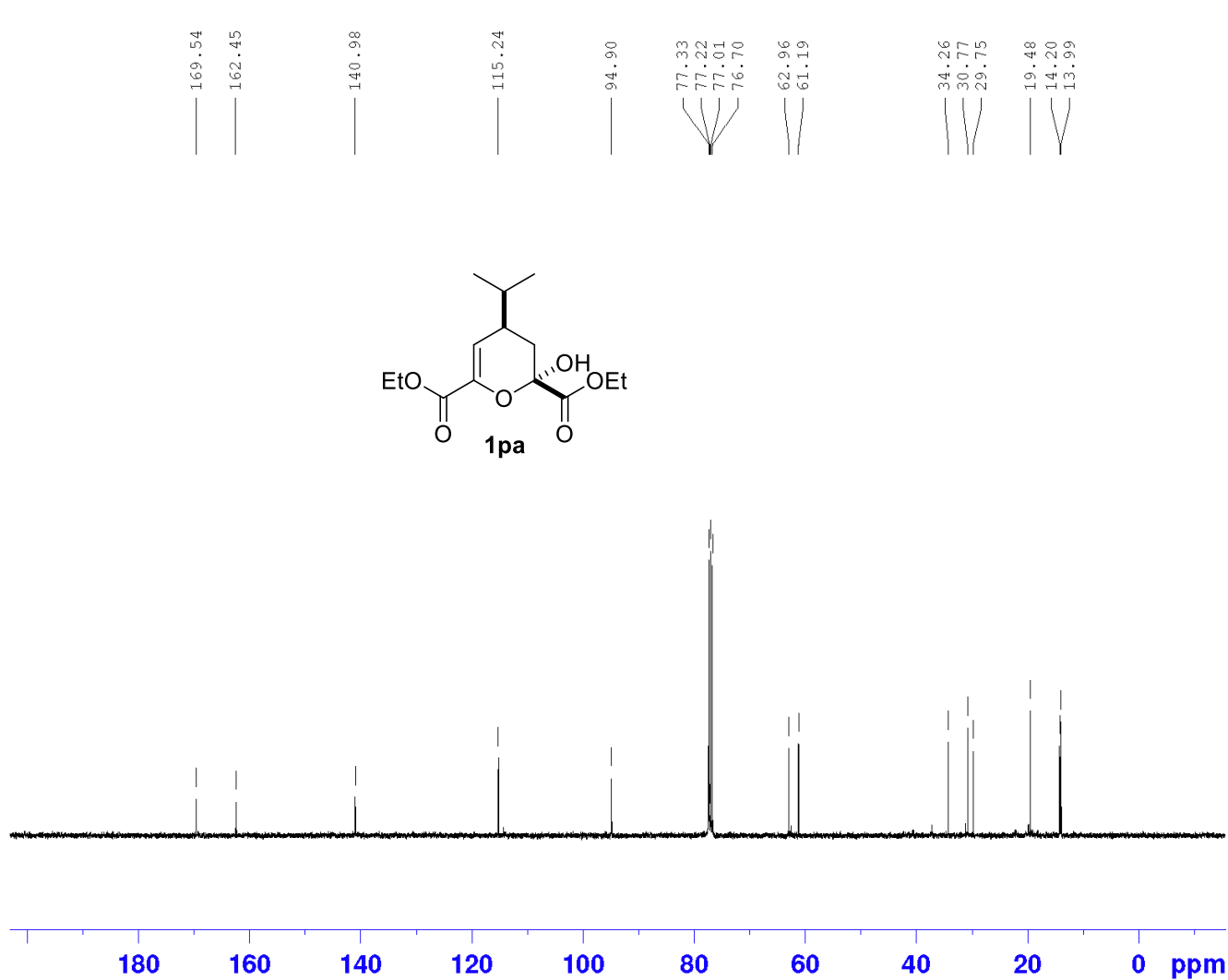
F2 - Acquisition Parameters
 Date_ 20120327
 Time 19.16
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 181
 DW 20.800 usec
 DE 6.00 usec
 TE 298.9 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379181 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





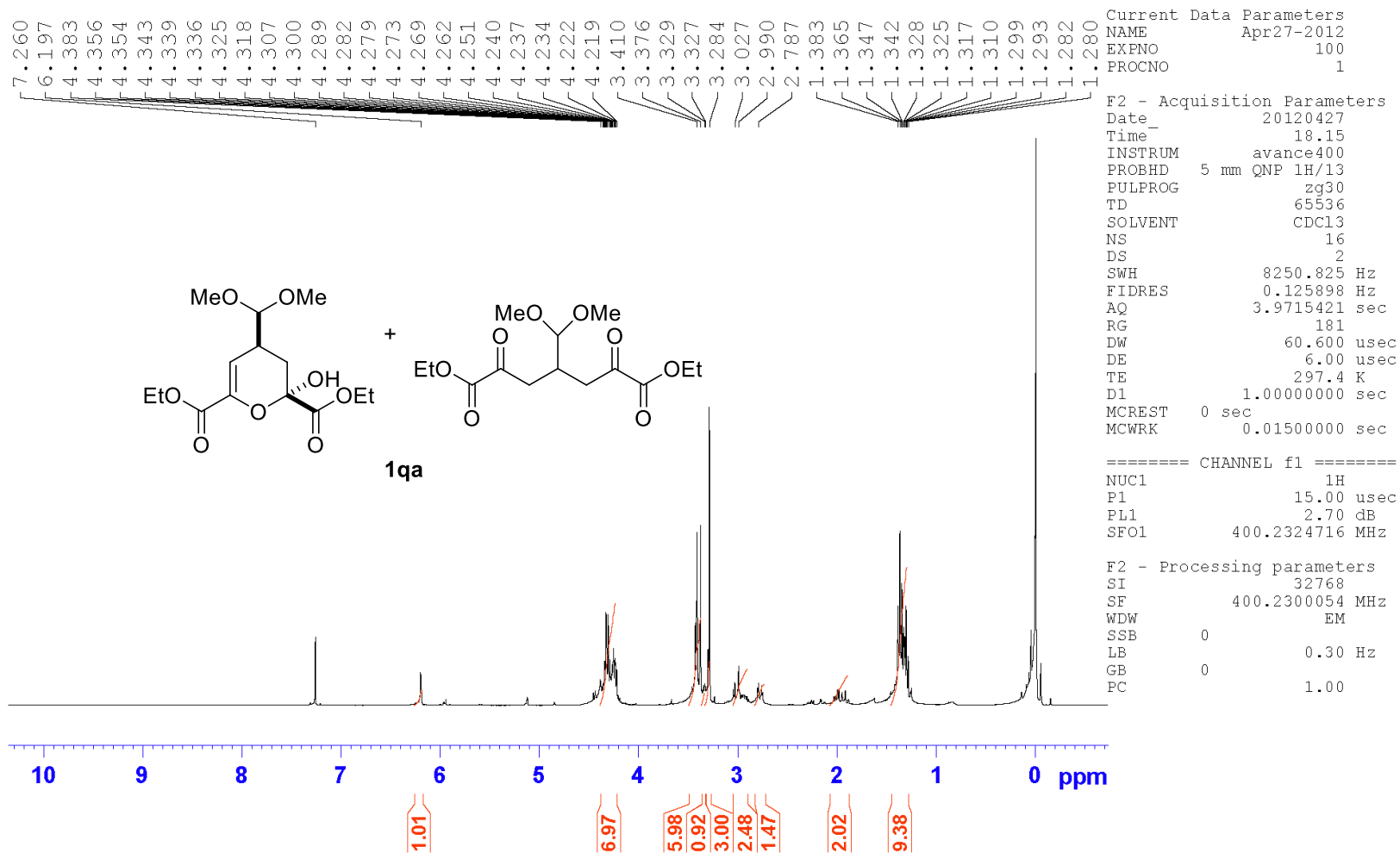
Current Data Parameters
 NAME Jan17-2012
 EXPNO 10
 PROCNO 1

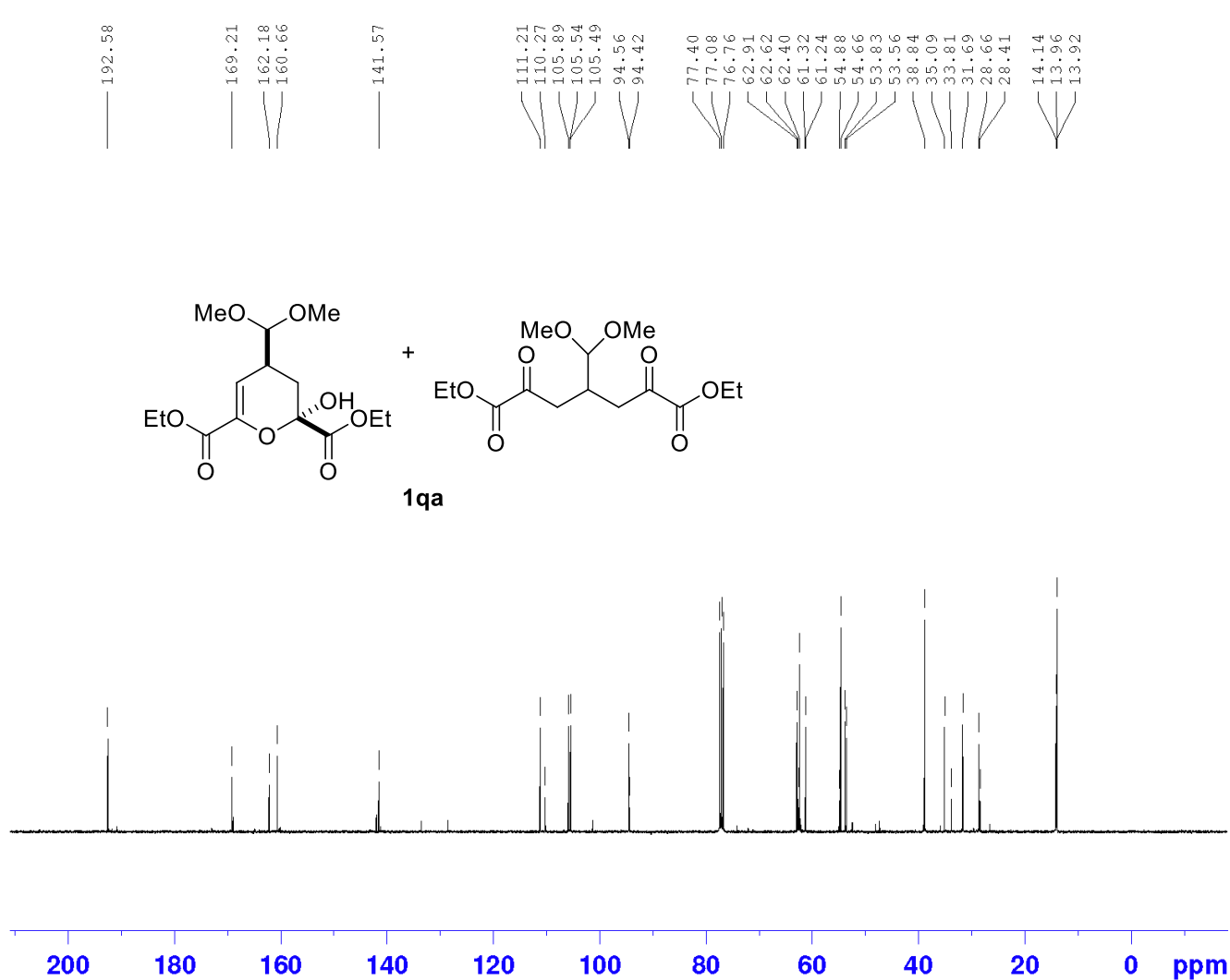
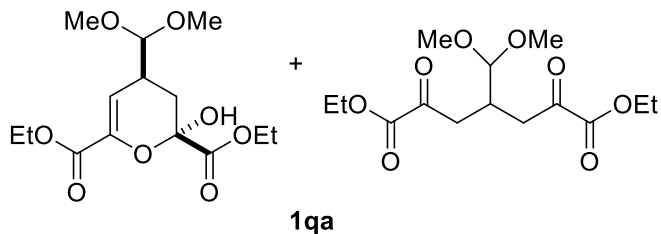
F2 - Acquisition Parameters
 Date_ 20120117
 Time 19.18
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 90.5
 DW 20.800 usec
 DE 6.00 usec
 TE 296.4 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





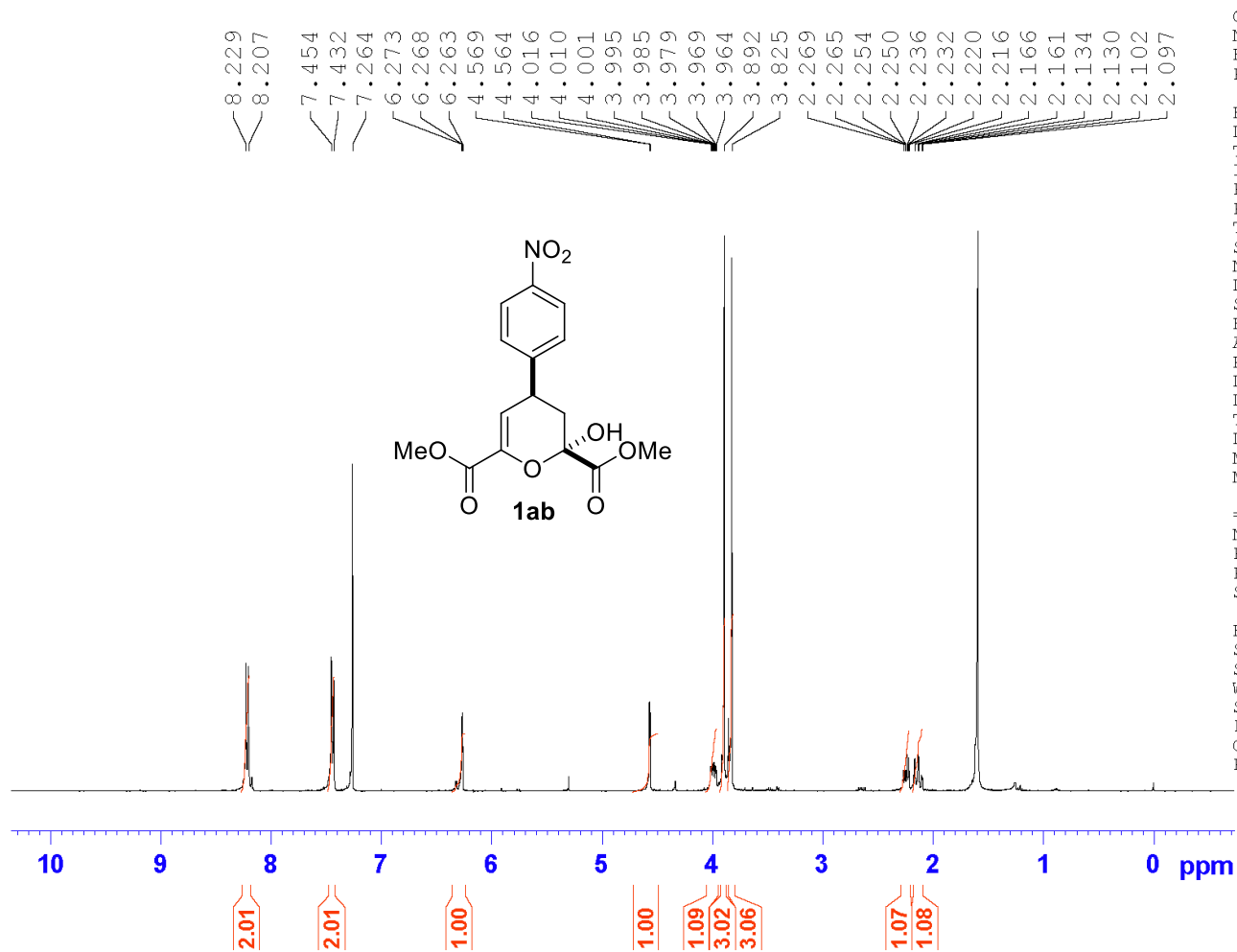
Current Data Parameters
 NAME May10-2012
 EXPNO 50
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120510
 Time 19.18
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 128
 DW 20.800 usec
 DE 6.00 usec
 TE 297.4 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

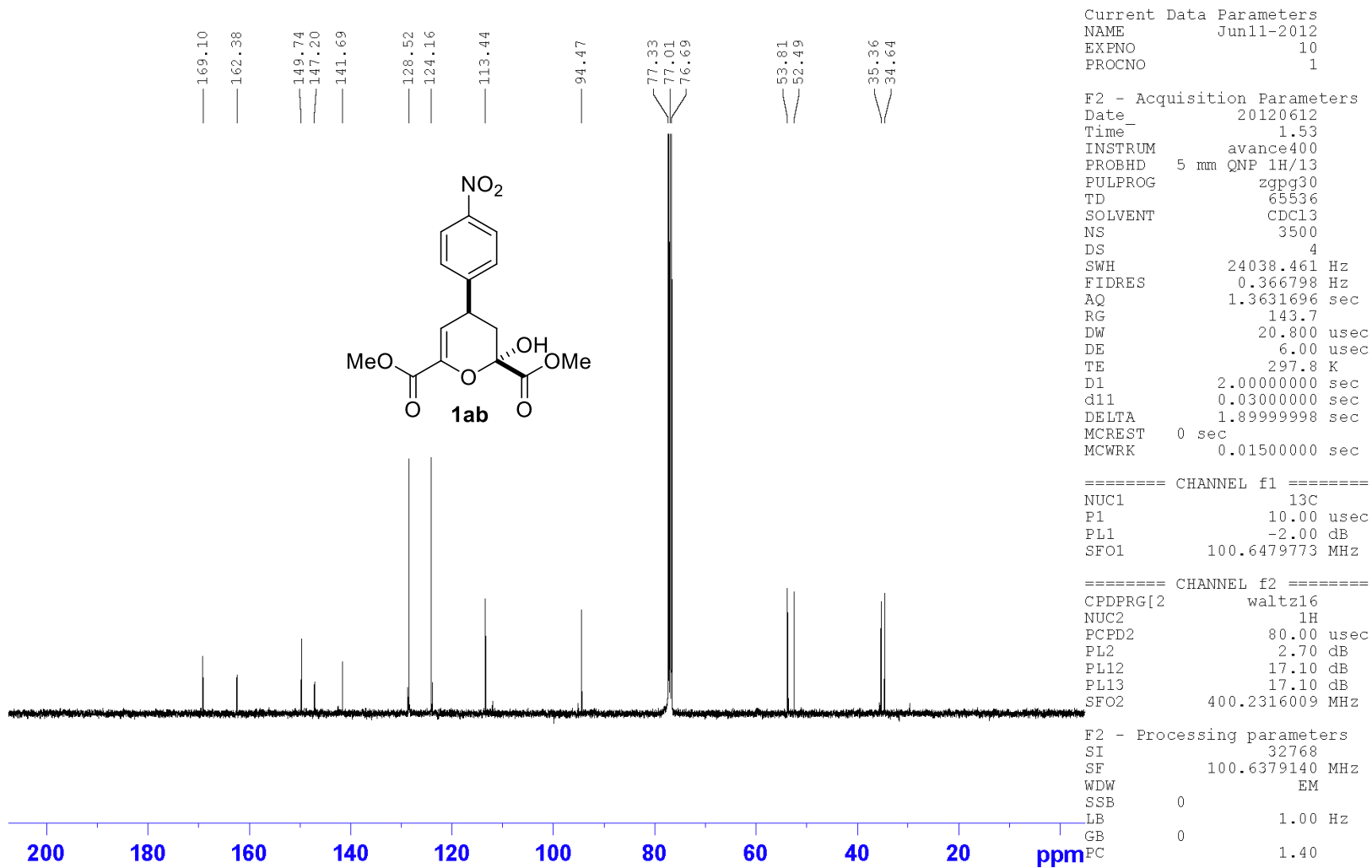


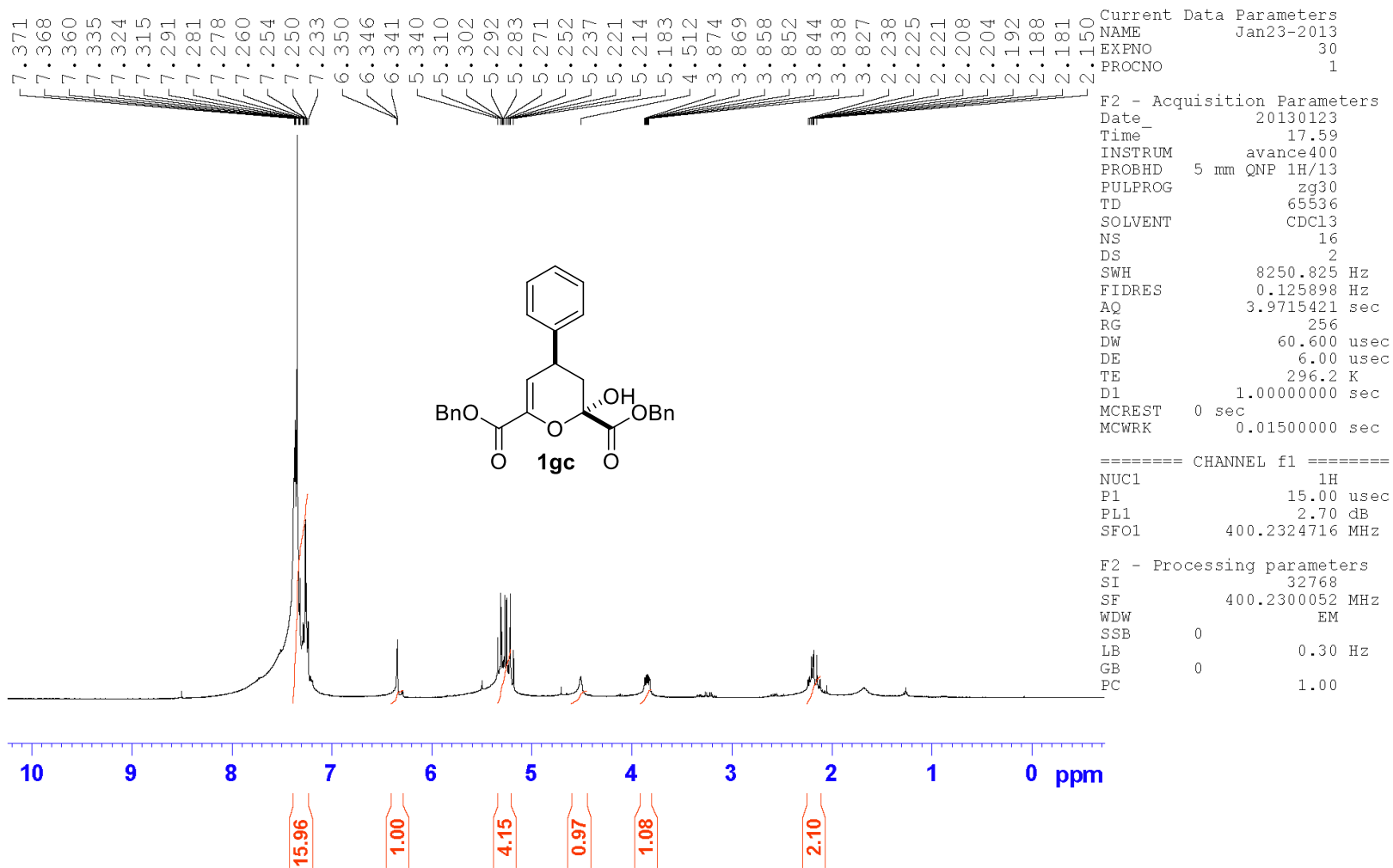
Current Data Parameters
 NAME Jun13-2012
 EXPNO 20
 PROCNO 1

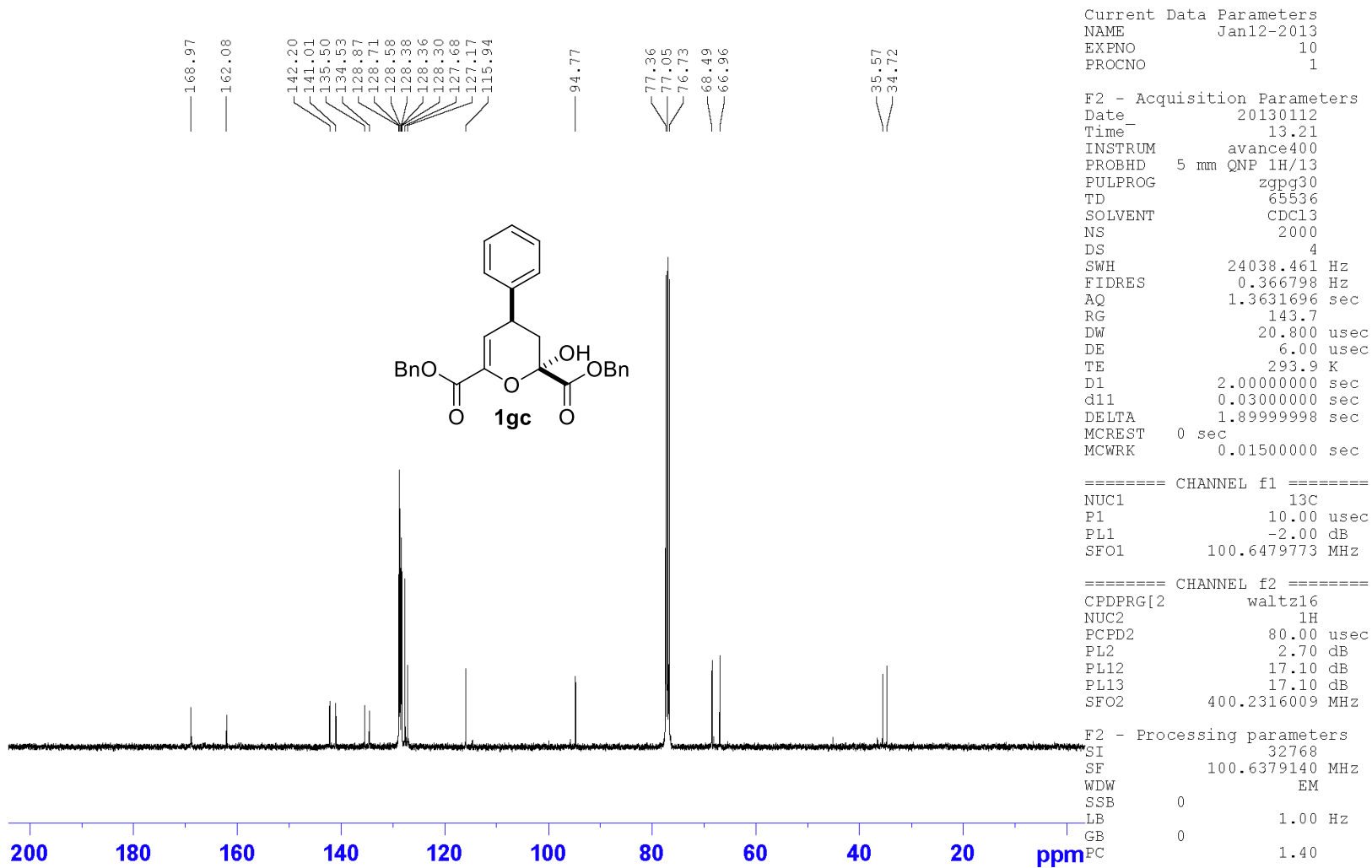
F2 - Acquisition Parameters
 Date_ 20120613
 Time 10.18
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 512
 DW 60.600 usec
 DE 6.00 usec
 TE 296.4 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

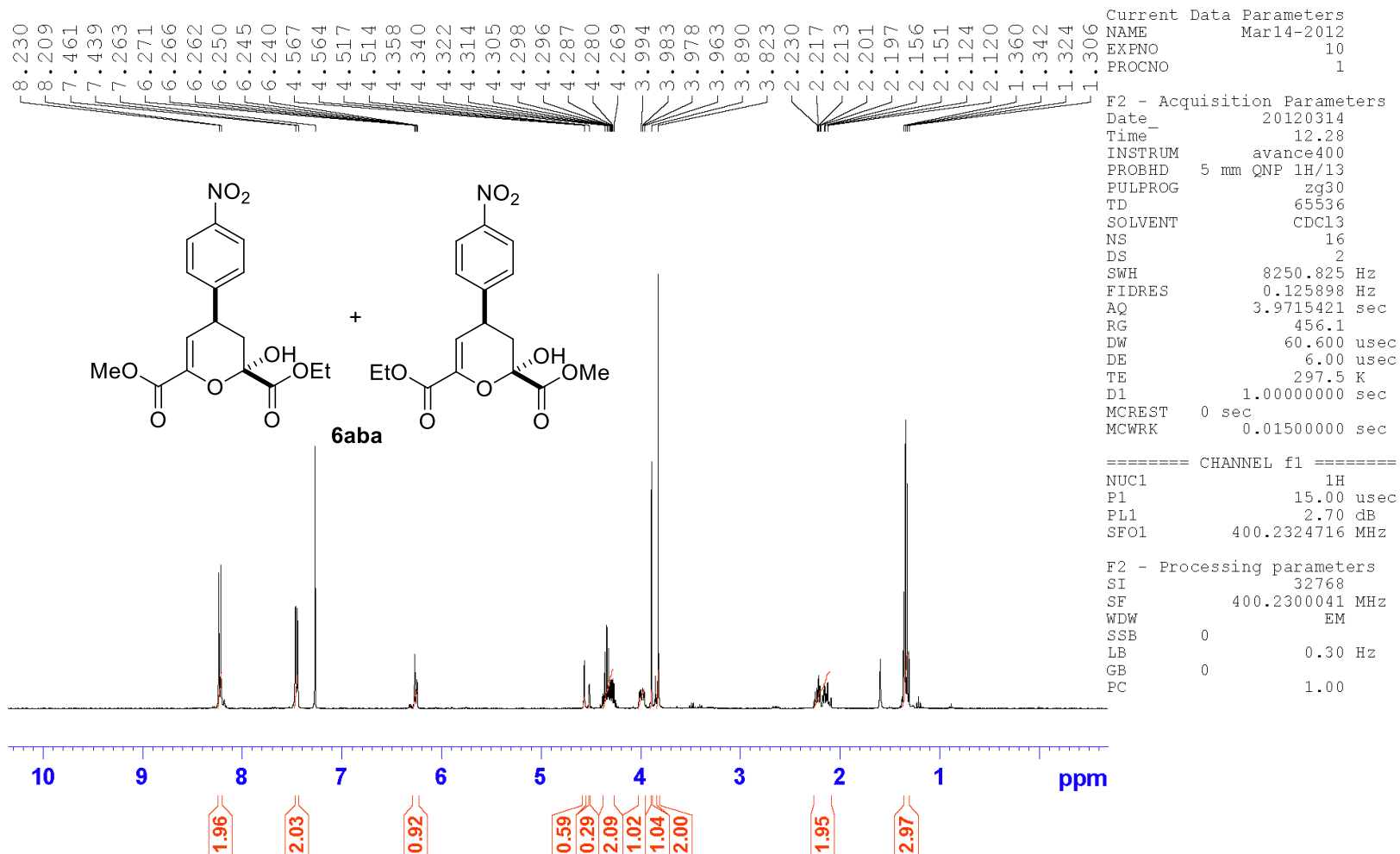
===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

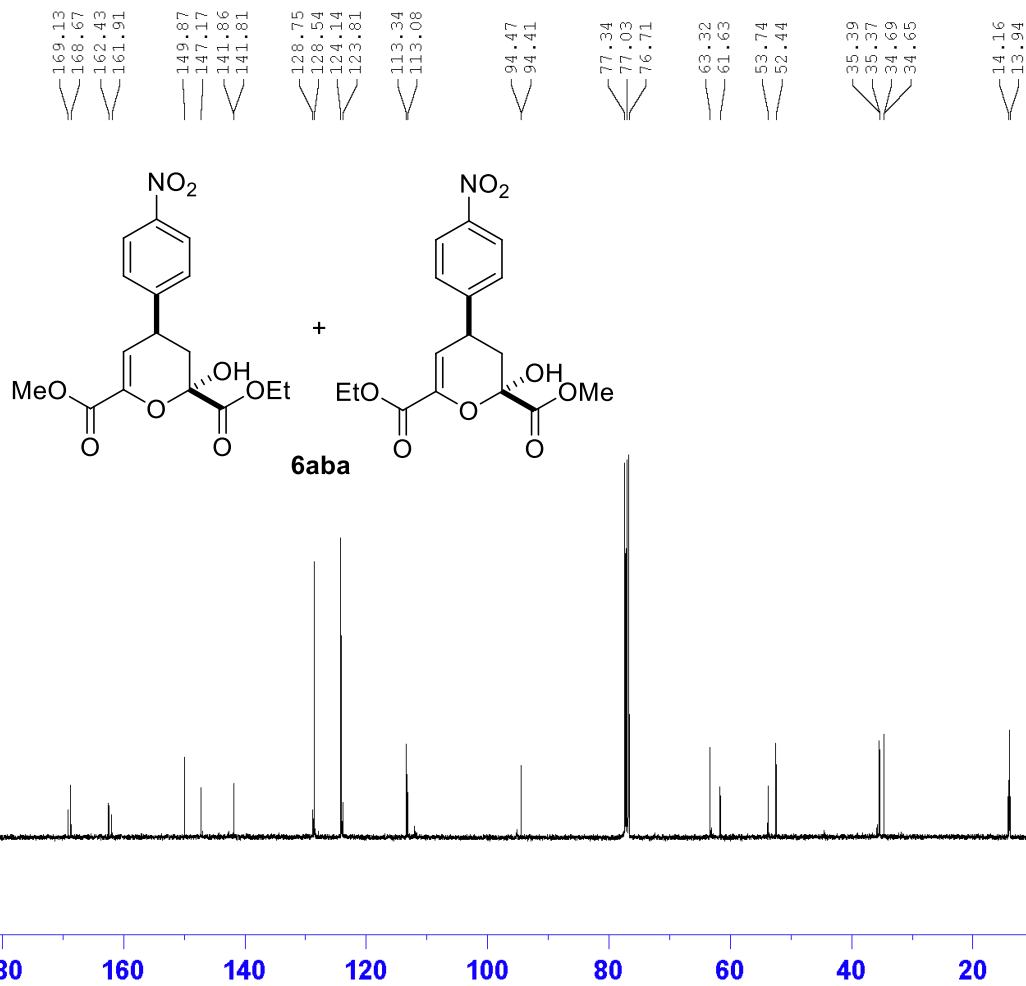
F2 - Processing parameters
 SI 32768
 SF 400.2300039 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00











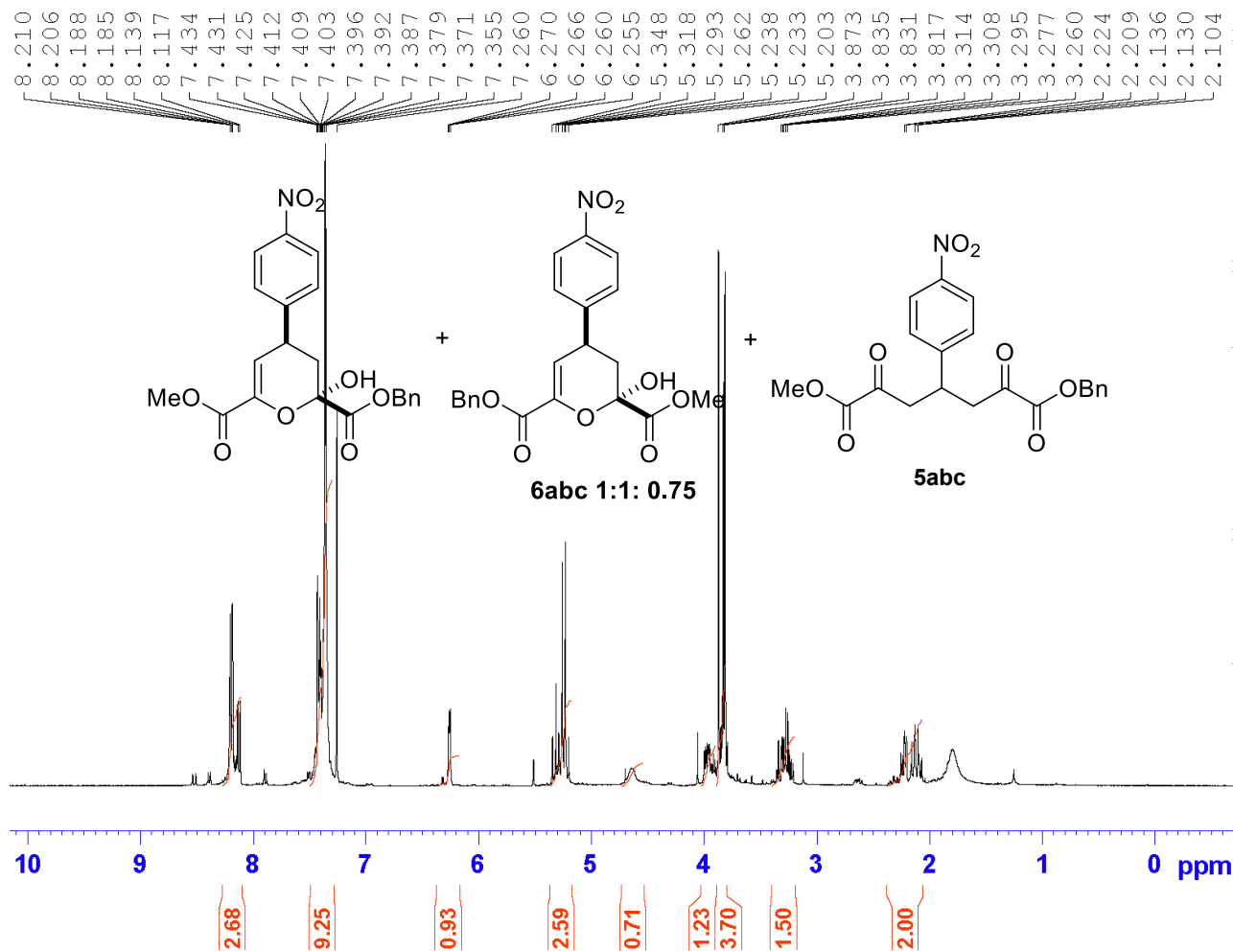
Current Data Parameters
 NAME Mar14-2012
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120314
 Time_ 13.40
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631696 sec
 RG 128
 DW 20.800 usec
 DE 6.00 usec
 TE 298.8 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

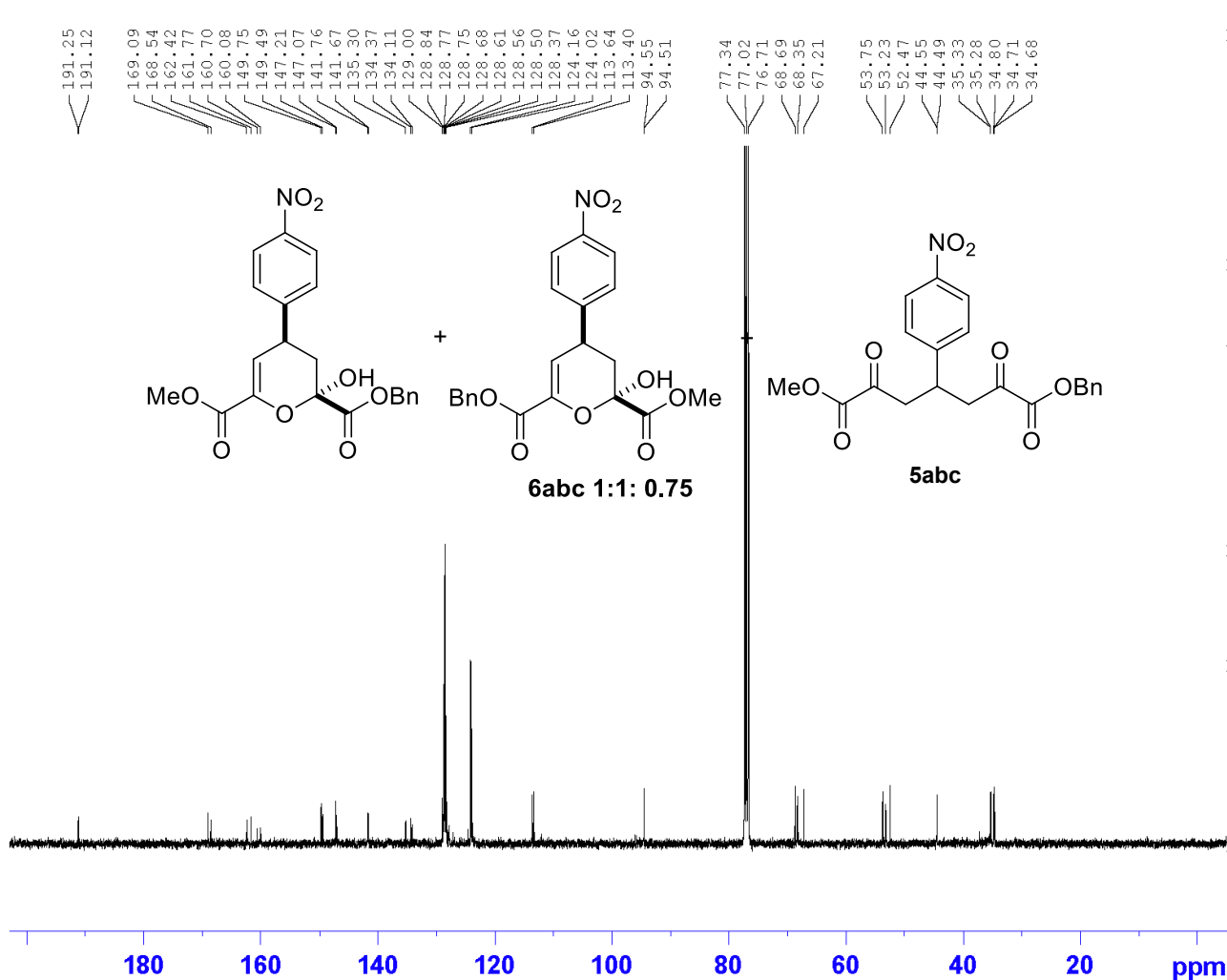


Current Data Parameters
NAME Jun12-2014
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140612
Time 14.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 62.88
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 8.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



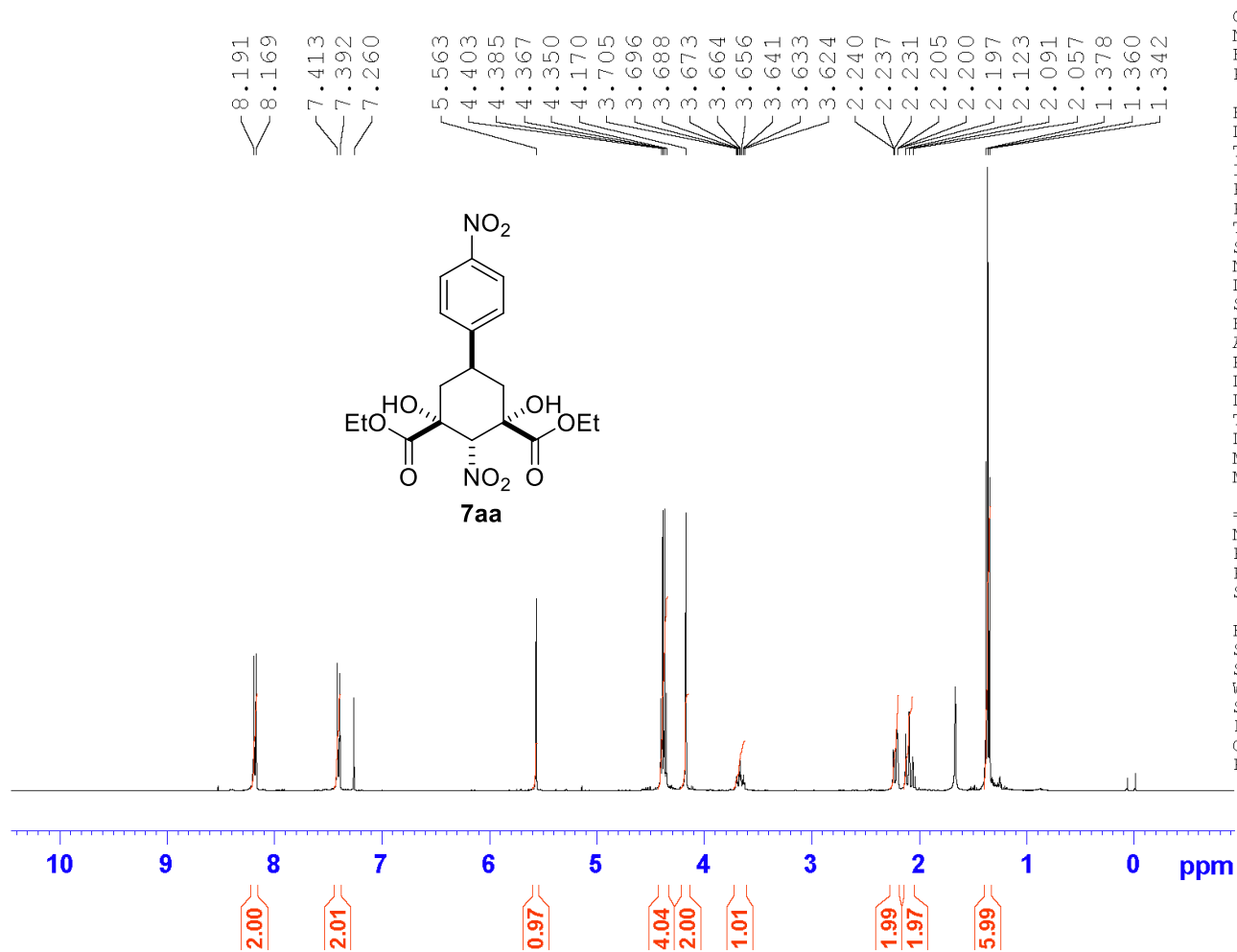
Current Data Parameters
 NAME Jun12-2014
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140612
 Time 16.59
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl₃
 NS 2000
 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 299.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

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

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

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

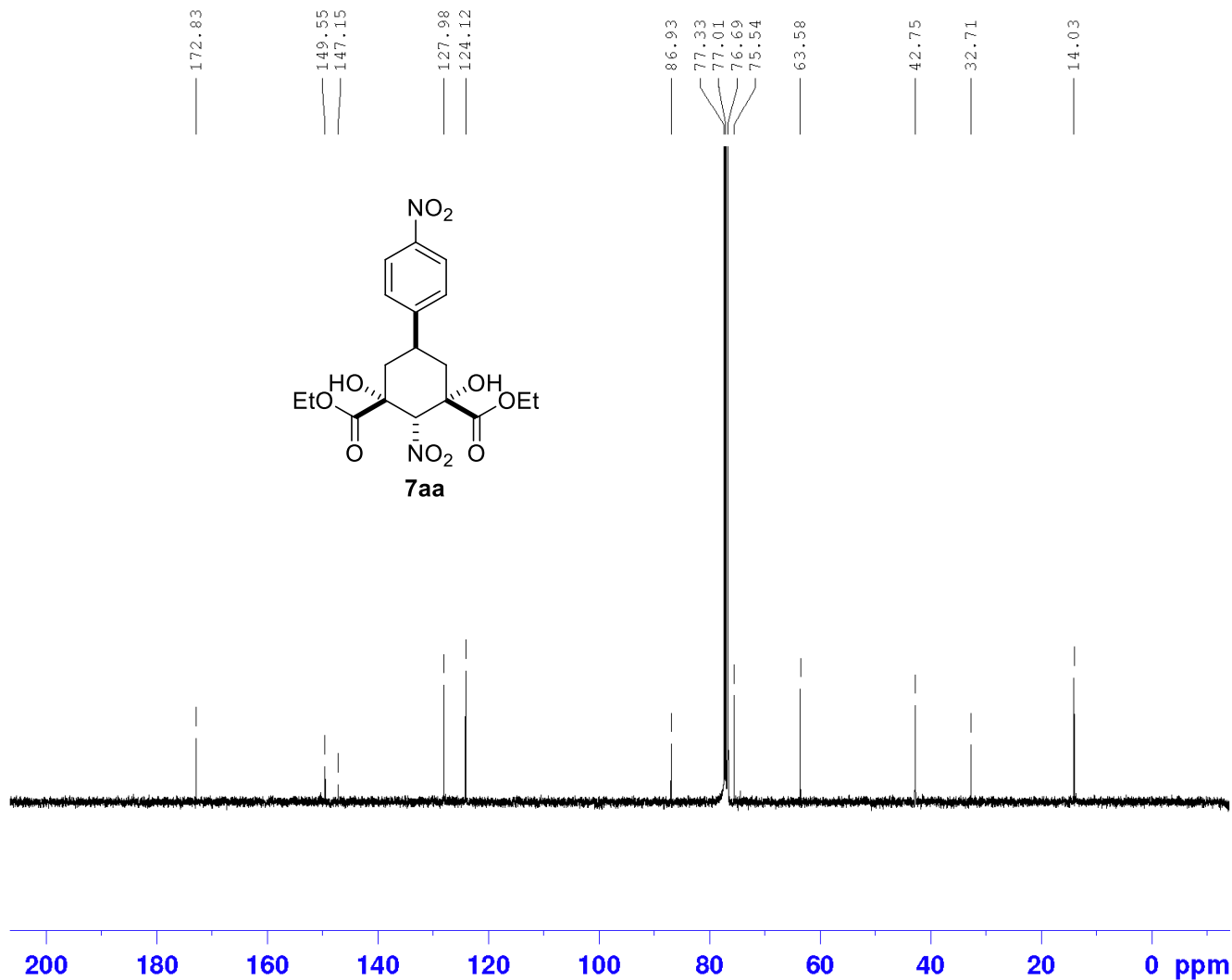
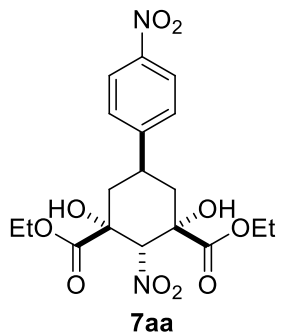


Current Data Parameters
 NAME Jul06-2012
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120706
 Time_ 20.48
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 161.3
 DW 60.600 usec
 DE 6.00 usec
 TE 296.7 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300054 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



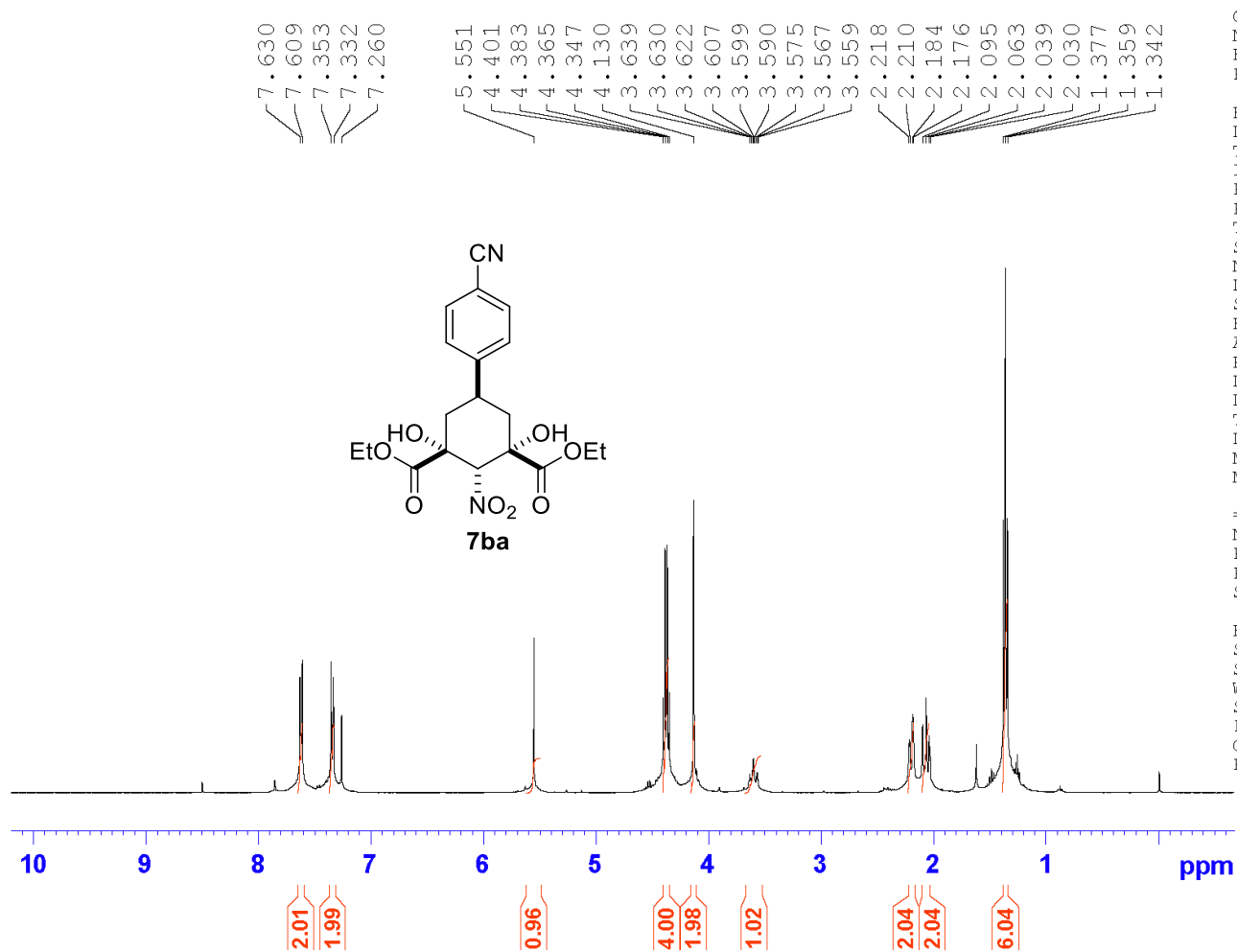
Current Data Parameters
NAME Jun29-2012
EXPNO 40
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120629
Time 20.41
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2500
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 114
DW 20.800 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -2.00 dB
SFO1 100.6479773 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.70 dB
PL12 17.10 dB
PL13 17.10 dB
SFO2 400.2316009 MHz

F2 - Processing parameters
SI 32768
SF 100.6379140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

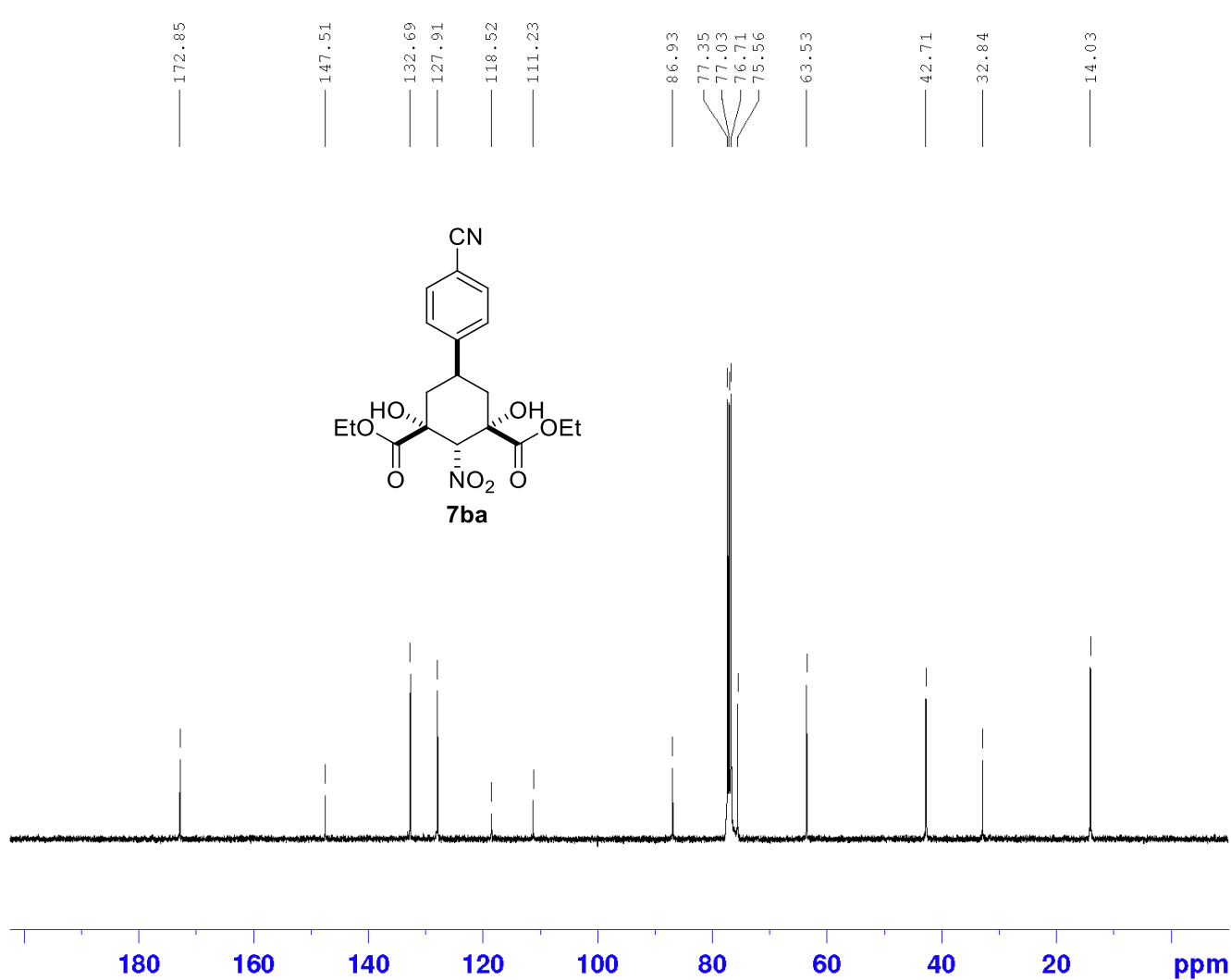


Current Data Parameters
 NAME Dec08-2012
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121208
 Time 17.53
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 256
 DW 60.600 usec
 DE 6.00 usec
 TE 294.9 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300055 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



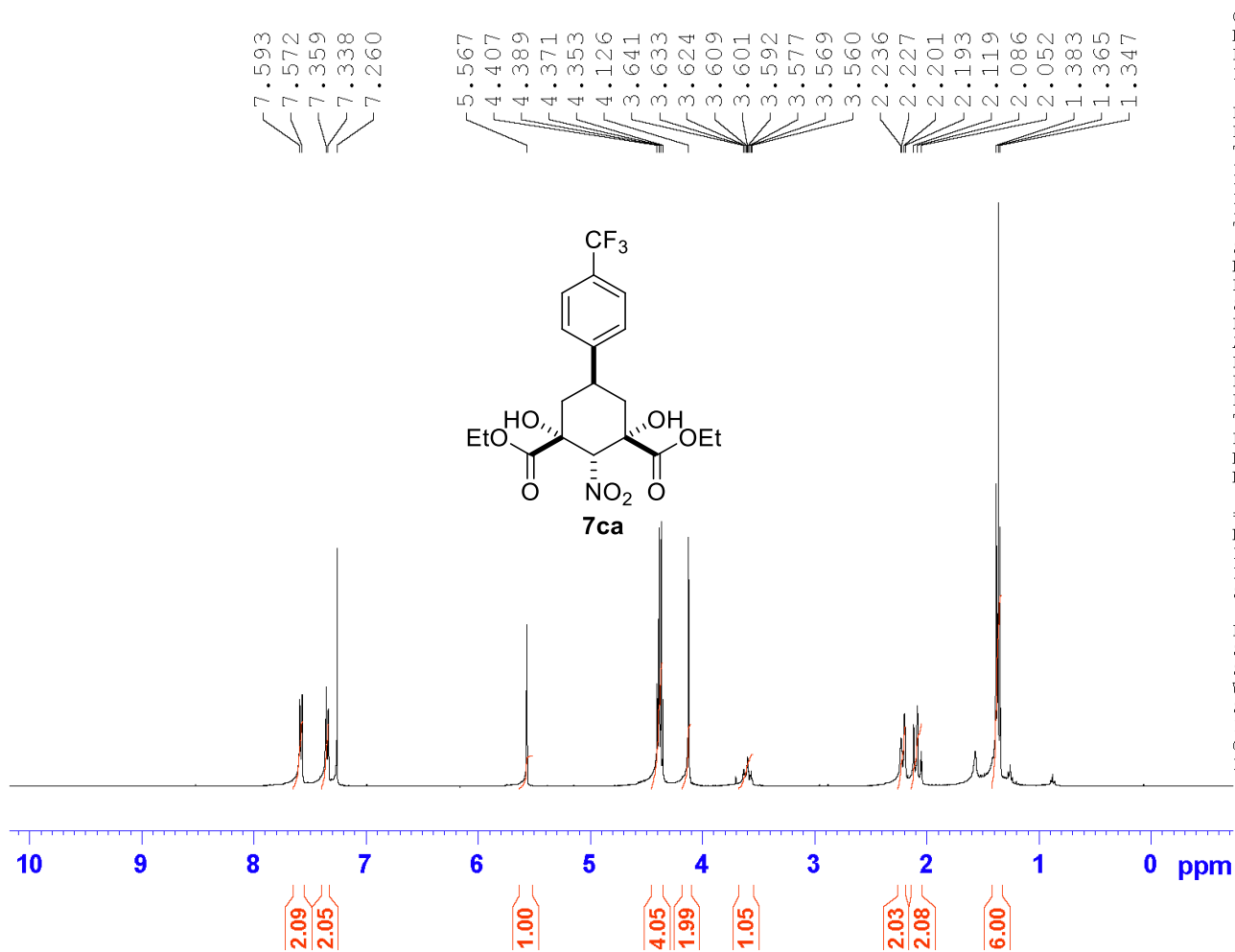
Current Data Parameters
 NAME Dec08-2012
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121208
 Time 21.02
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 3000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 101.6
 DW 20.800 usec
 DE 6.00 usec
 TE 296.9 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

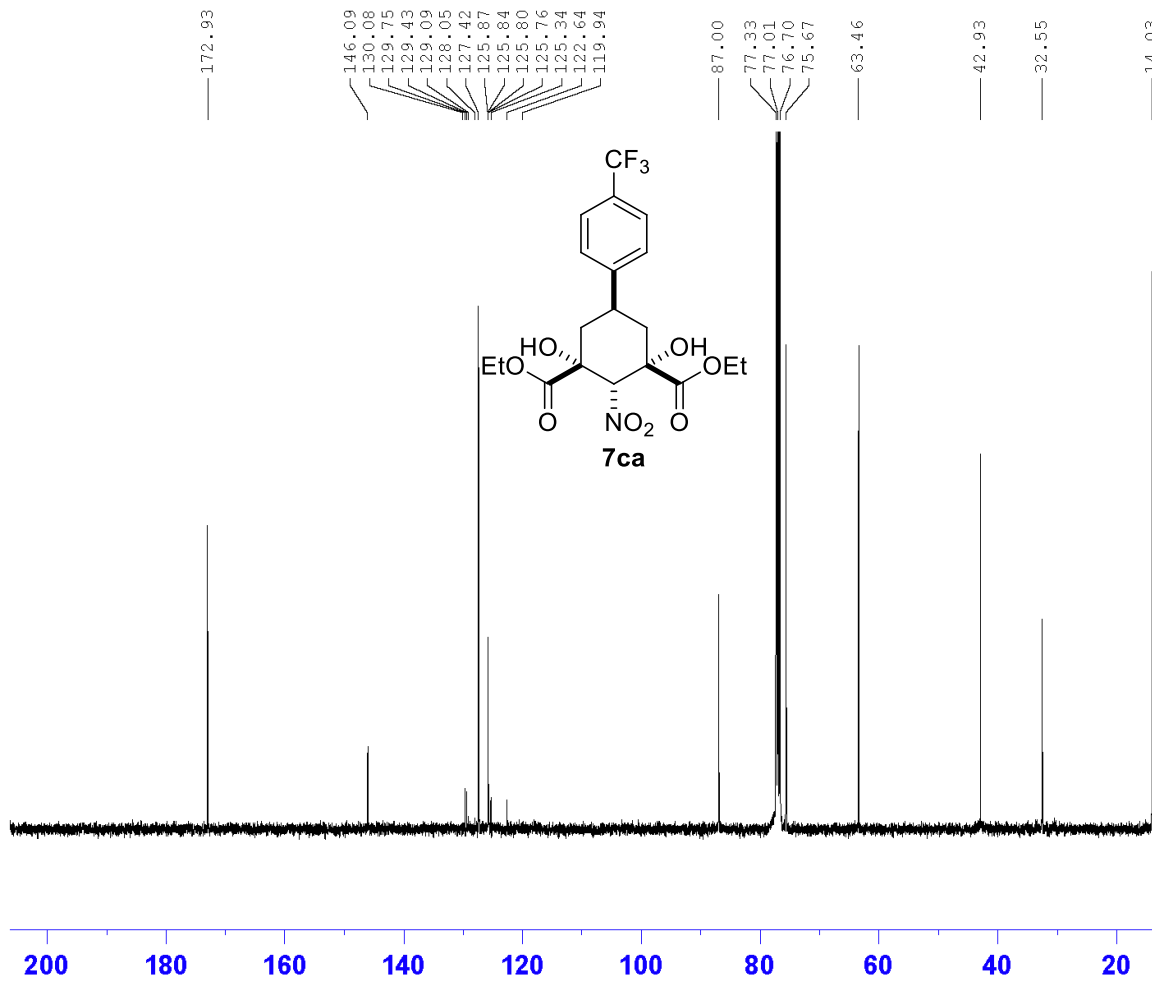


Current Data Parameters
 NAME Jan08-2013
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130108
 Time 15.40
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 50
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 406.4
 DW 60.600 usec
 DE 6.00 usec
 TE 673.2 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300052 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



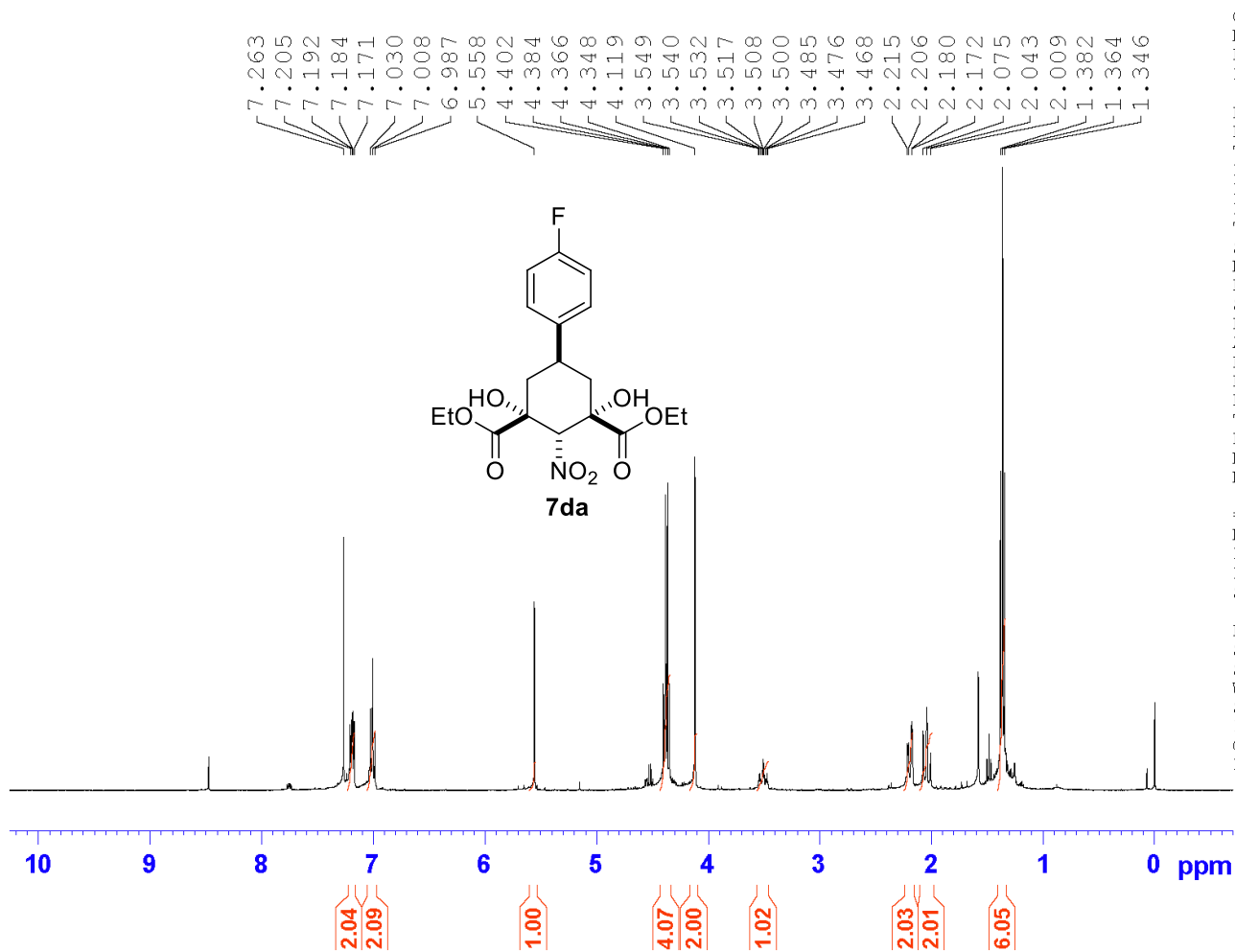
Current Data Parameters
NAME Jan08-2013
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130108
Time_ 23.20
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631696 sec
RG 912.3
DW 20.800 usec
DE 6.00 usec
TE 297.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -2.00 dB
SFO1 100.6479773 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.70 dB
PL12 17.10 dB
PL13 17.10 dB
SFO2 400.2316009 MHz

F2 - Processing parameters
SI 32768
SF 100.6379140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

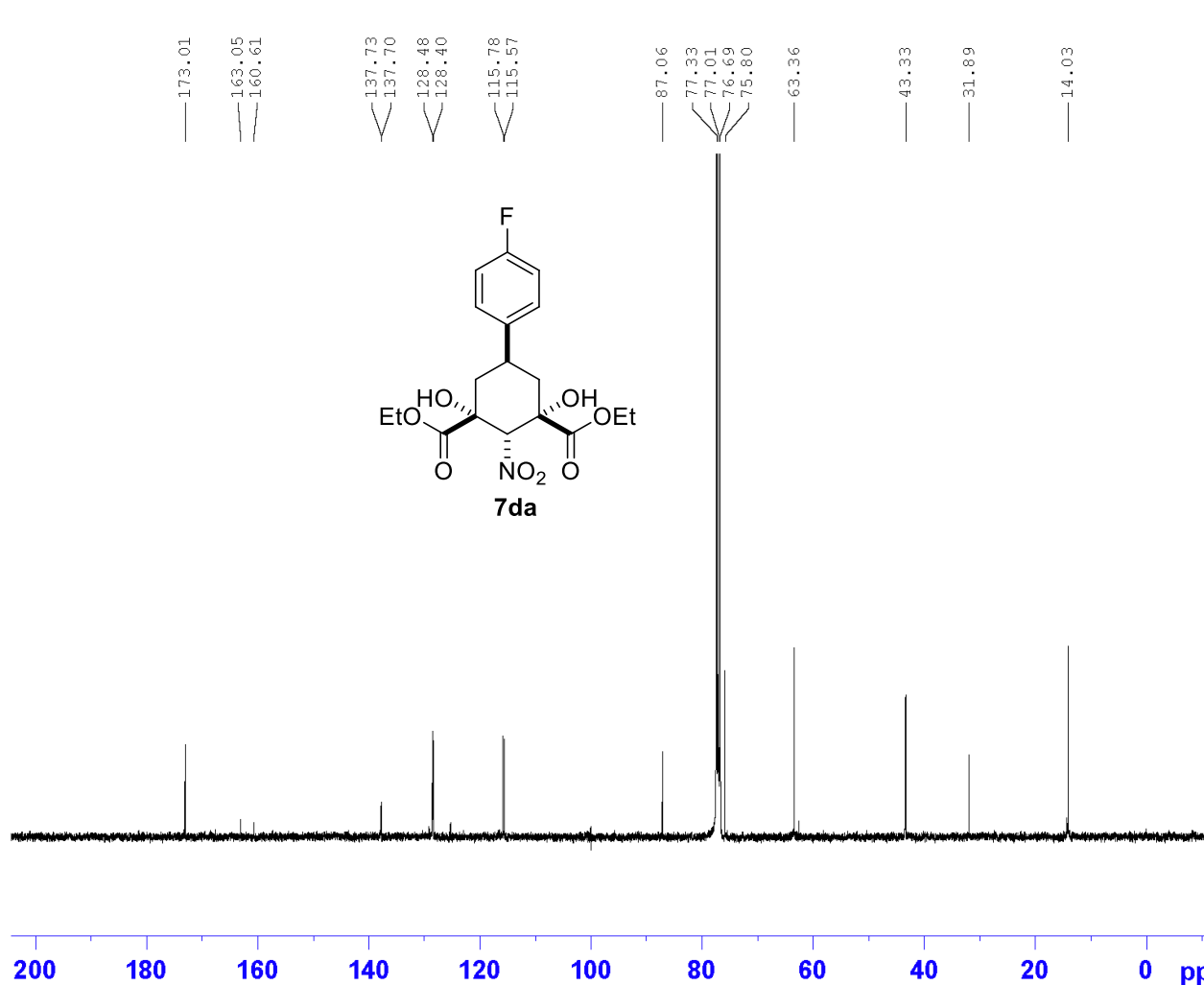


Current Data Parameters
 NAME Dec04-2012-userA
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121204
 Time 10.14
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 406.4
 DW 60.600 usec
 DE 6.00 usec
 TE 296.1 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300040 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



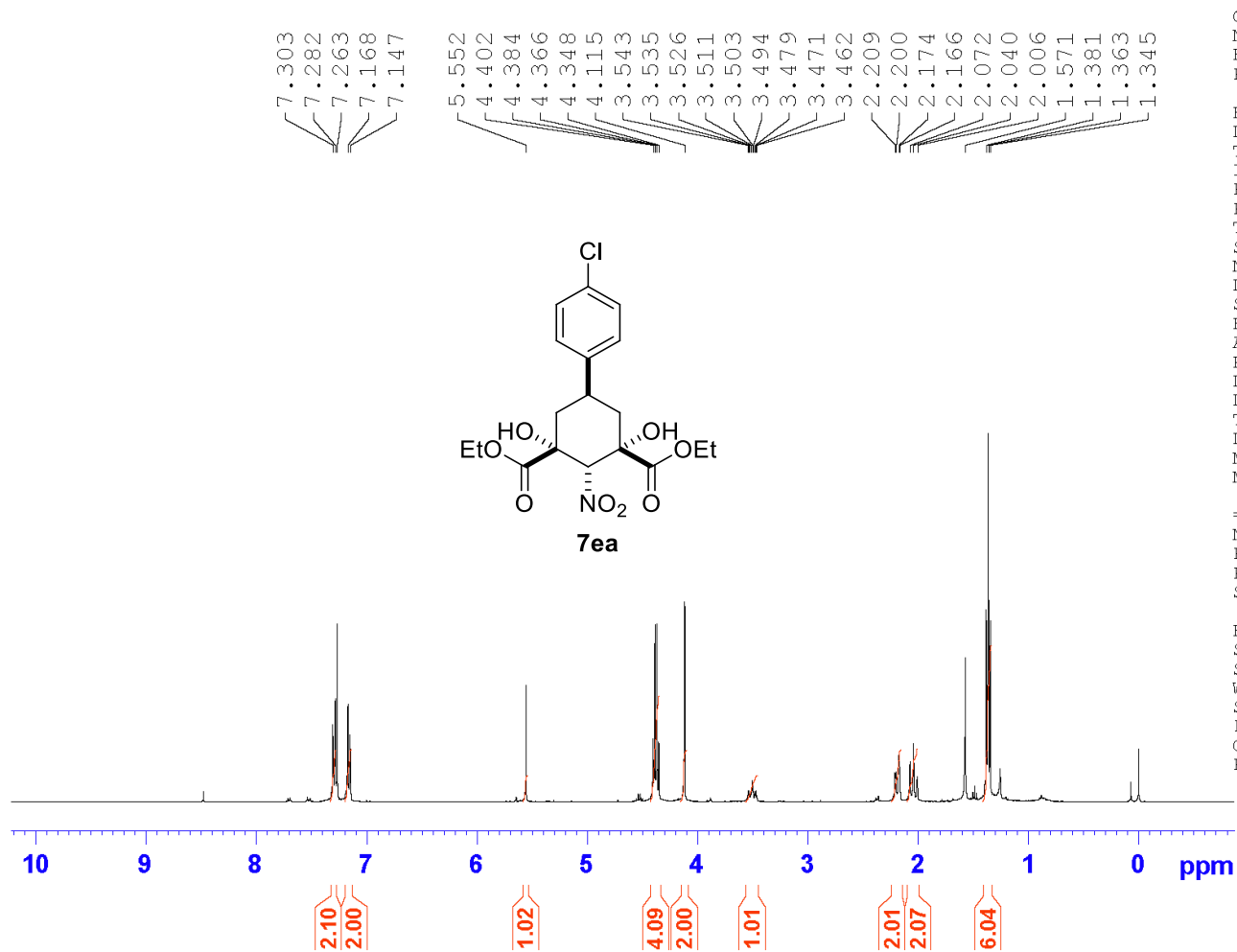
Current Data Parameters
 NAME Dec03-2012
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121204
 Time_ 0.44
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 6000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631696 sec
 RG 114
 DW 20.800 usec
 DE 6.00 usec
 TE 297.3 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

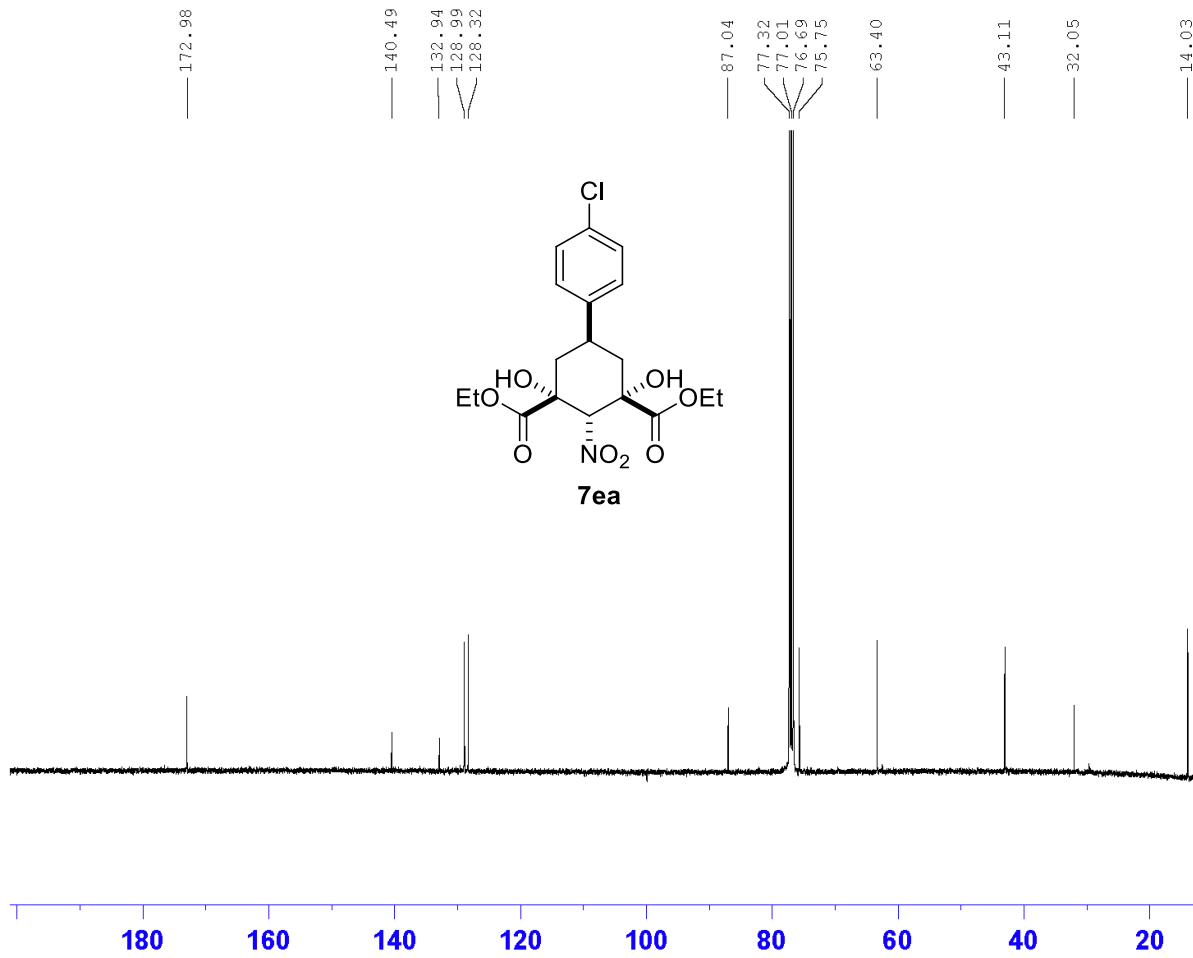


Current Data Parameters
 NAME Nov29-2012
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121129
 Time 17.13
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 40
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 456.1
 DW 60.600 usec
 DE 6.00 usec
 TE 296.0 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300044 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



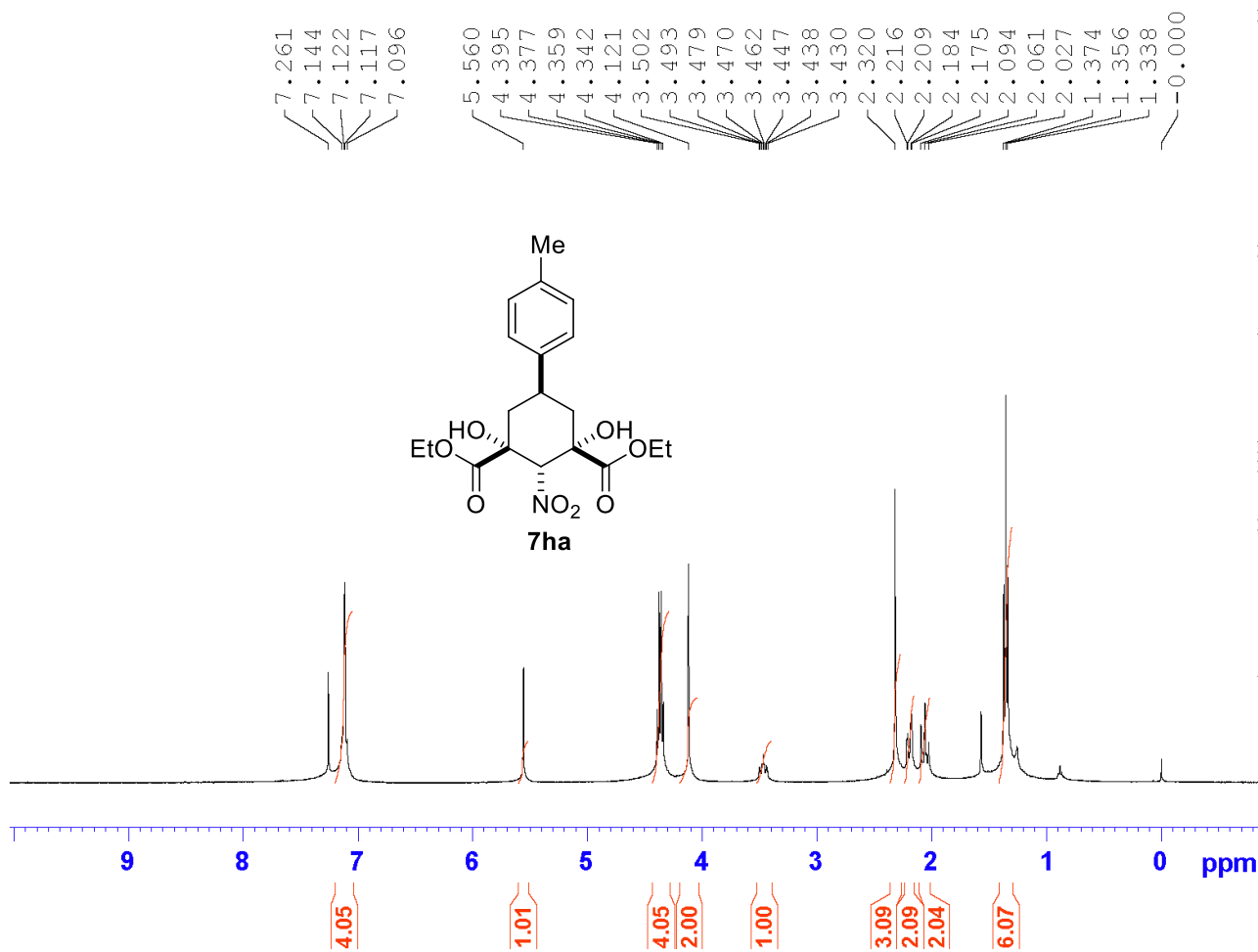
Current Data Parameters
 NAME Nov29-2012
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date 20121130
 Time 1.42
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 8000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631696 sec
 RG 114
 DW 20.800 usec
 DE 6.00 usec
 TE 297.5 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

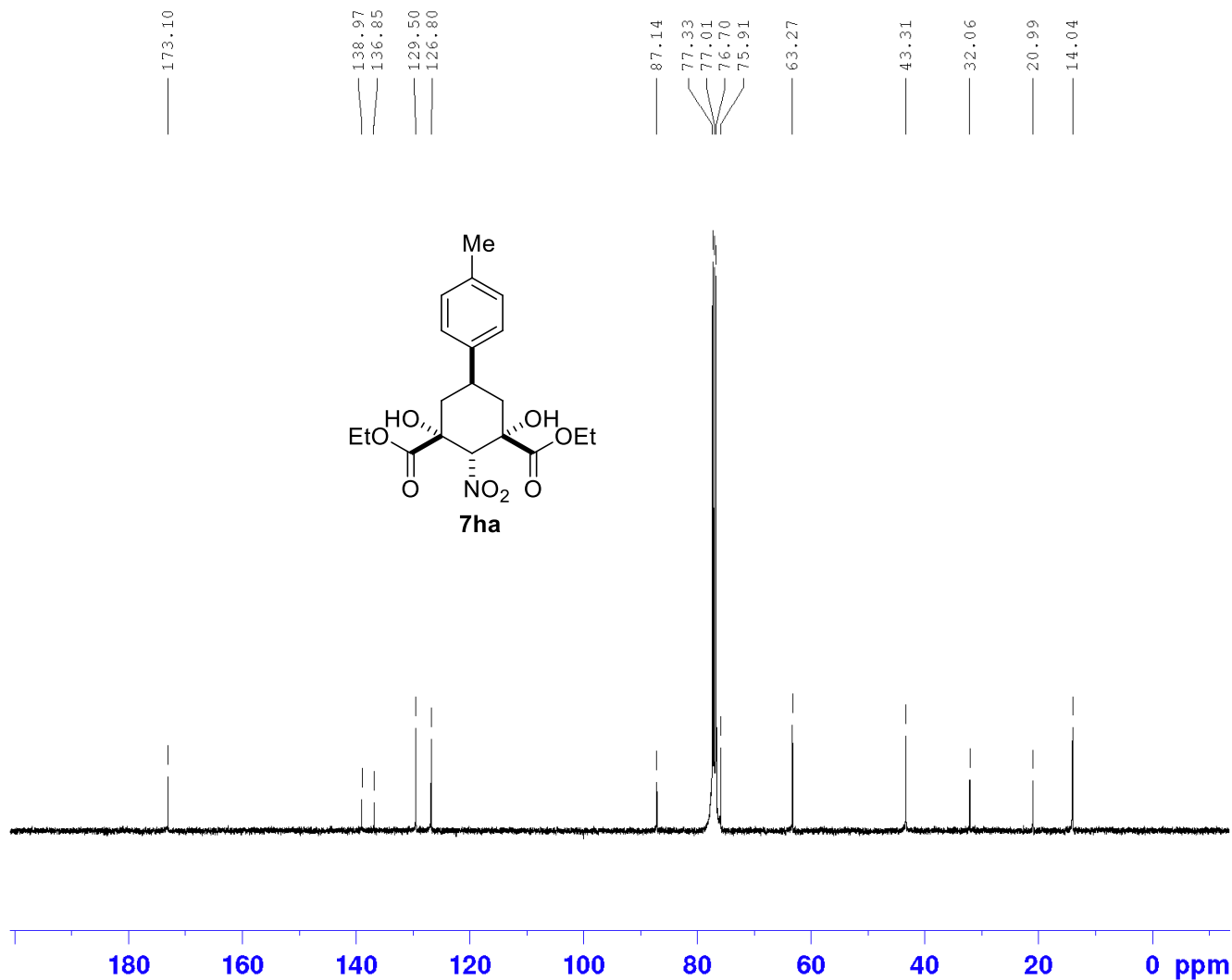


Current Data Parameters
 NAME Dec11-2012
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121211
 Time 10.07
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 406.4
 DW 60.600 usec
 DE 6.00 usec
 TE 295.9 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300049 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



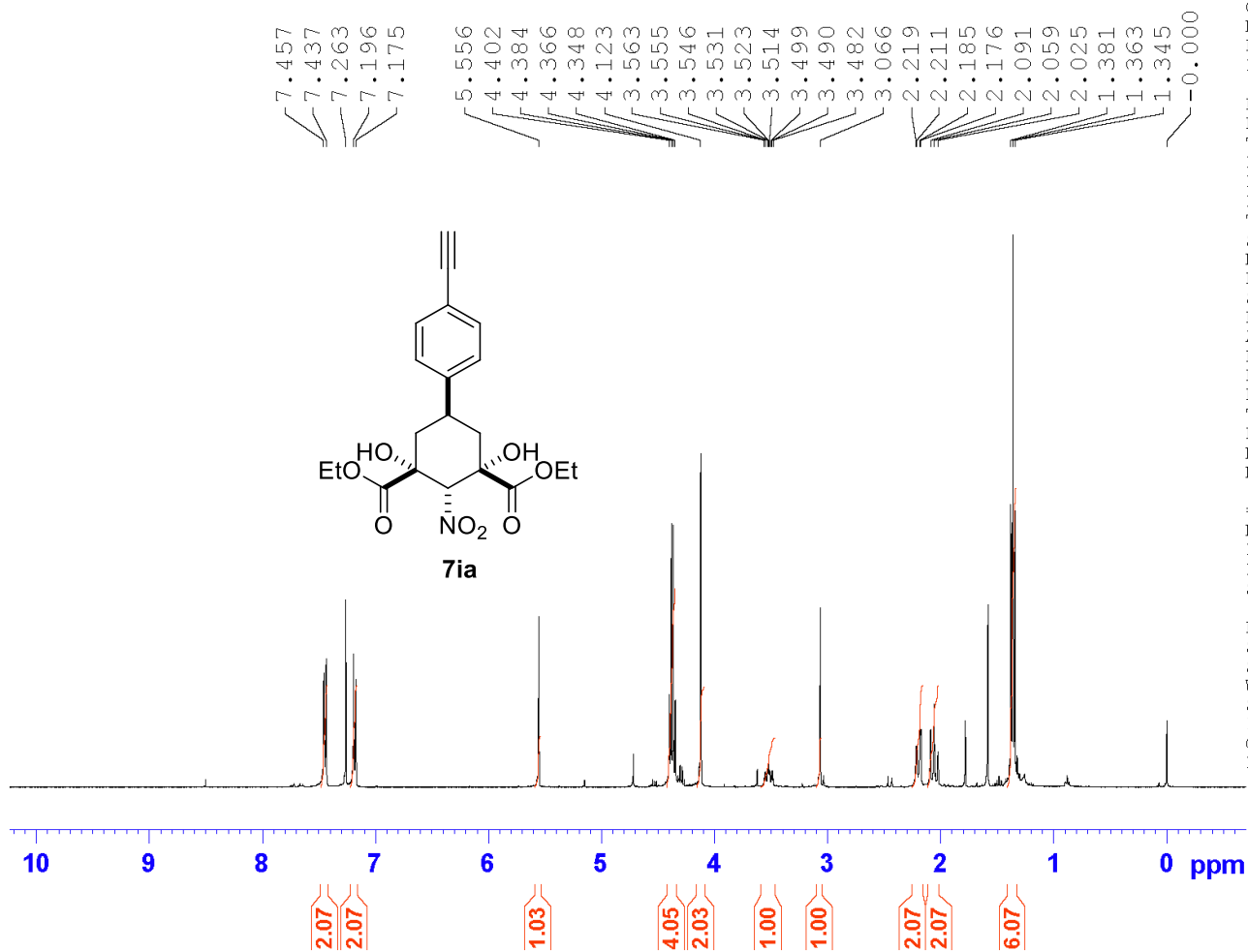
Current Data Parameters
 NAME Dec10-2012
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121211
 Time 0.54
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 6000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 101.6
 DW 20.800 usec
 DE 6.00 usec
 TE 297.3 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

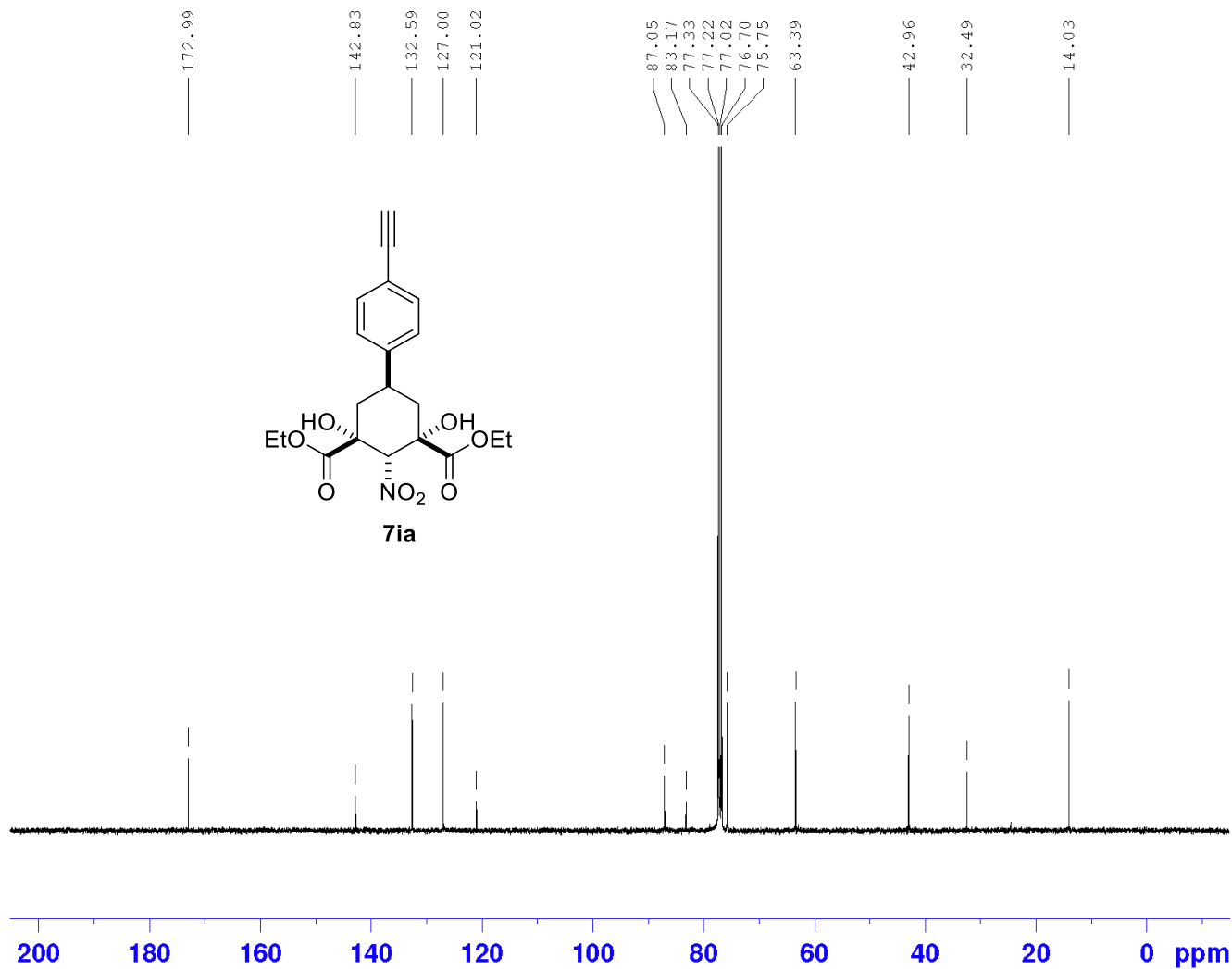
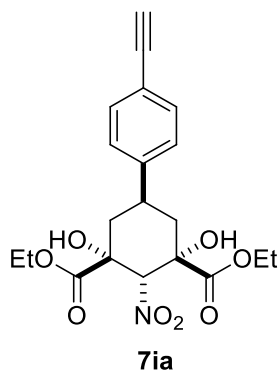


Current Data Parameters
 NAME Nov28-2012
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121128
 Time_ 17.45
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 406.4
 DW 60.600 usec
 DE 6.00 usec
 TE 295.9 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300041 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



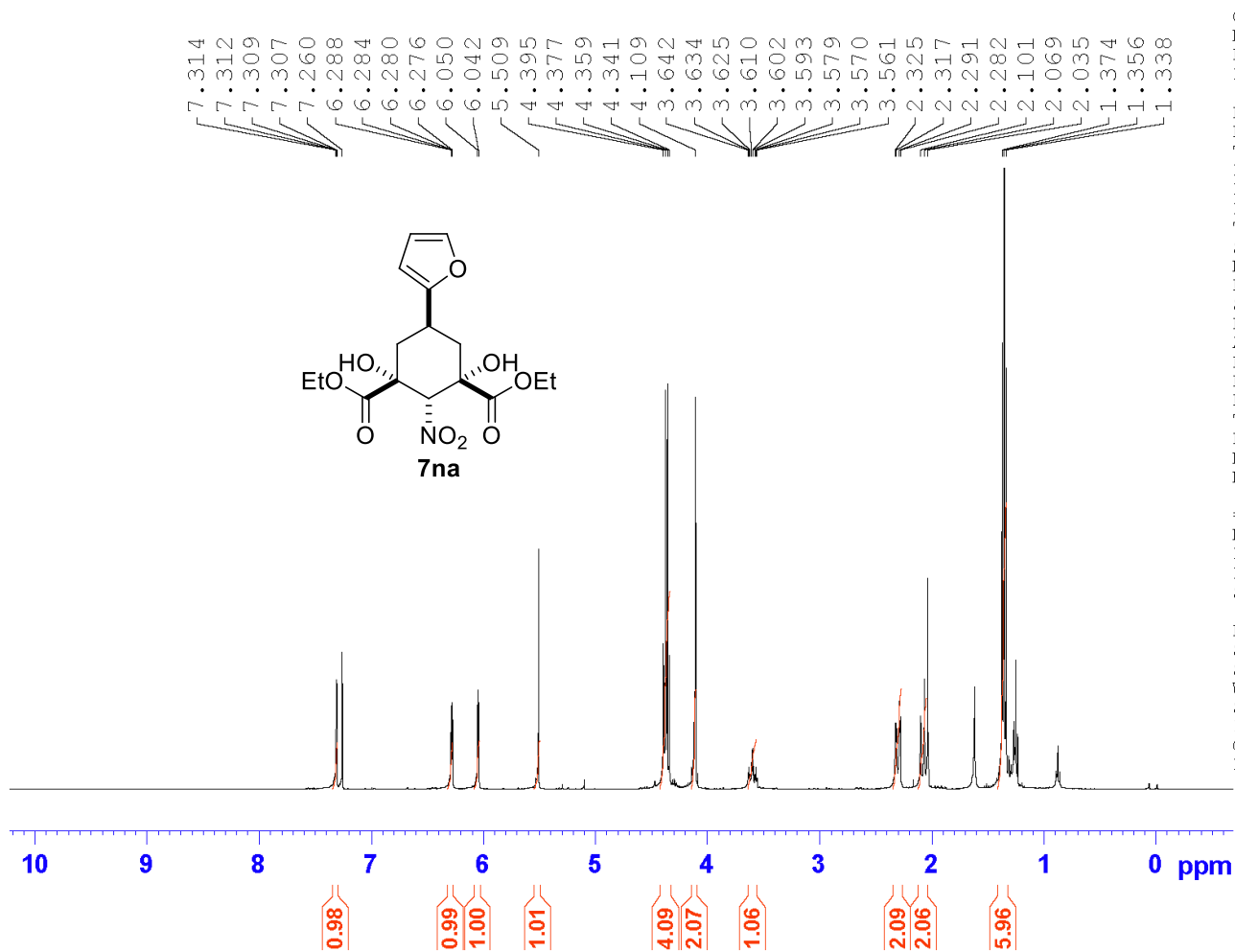
Current Data Parameters
 NAME Nov28-2012
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121128
 Time 23.00
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 5000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 181
 DW 20.800 usec
 DE 6.00 usec
 TE 298.1 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

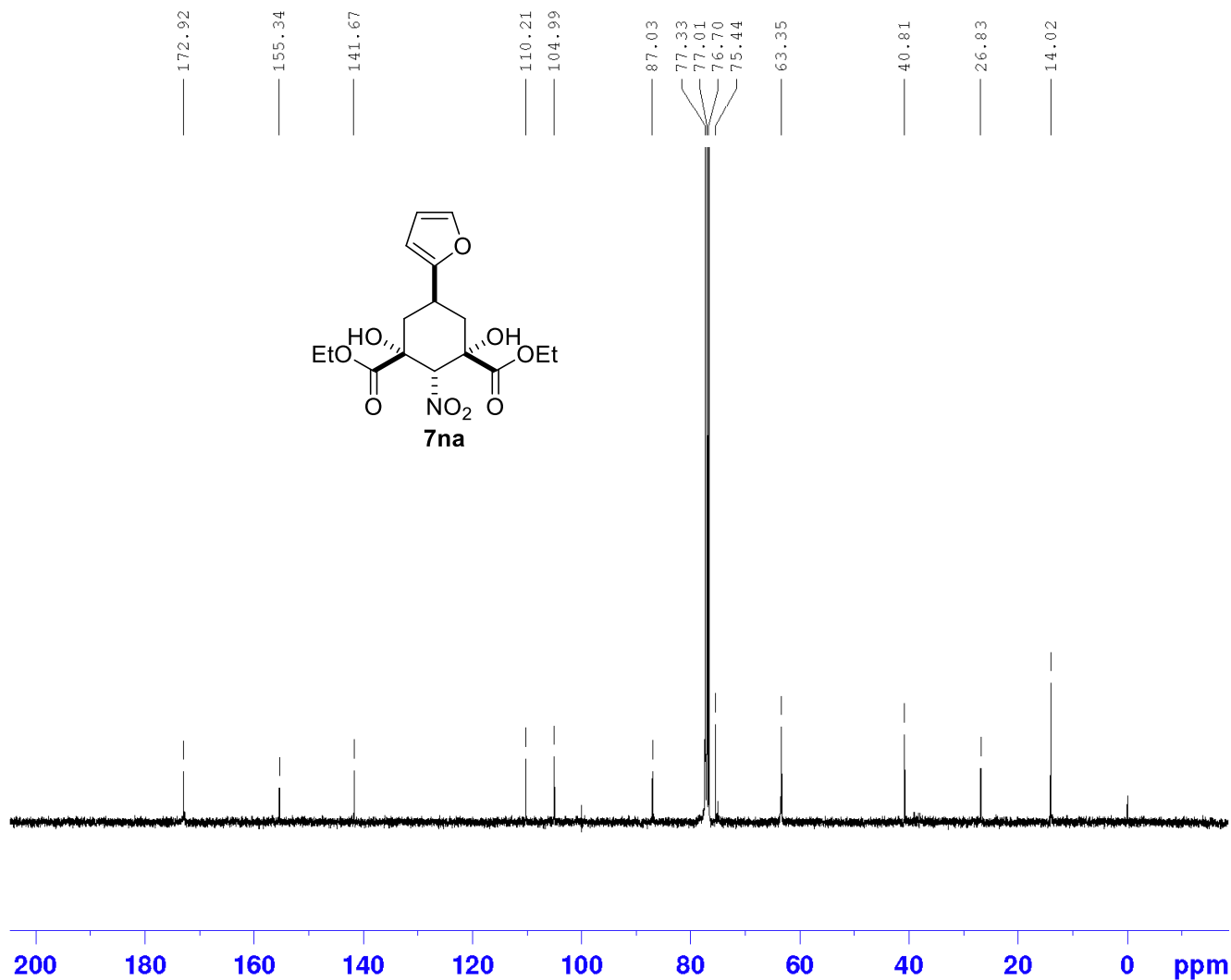
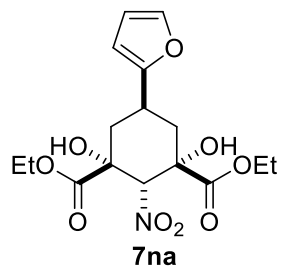


Current Data Parameters
 NAME Jul25-2012
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120725
 Time_ 12.02
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 181
 DW 60.600 usec
 DE 6.00 usec
 TE 297.0 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300054 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
NAME Sep28-2012
EXPNO 20
PROCNO 1

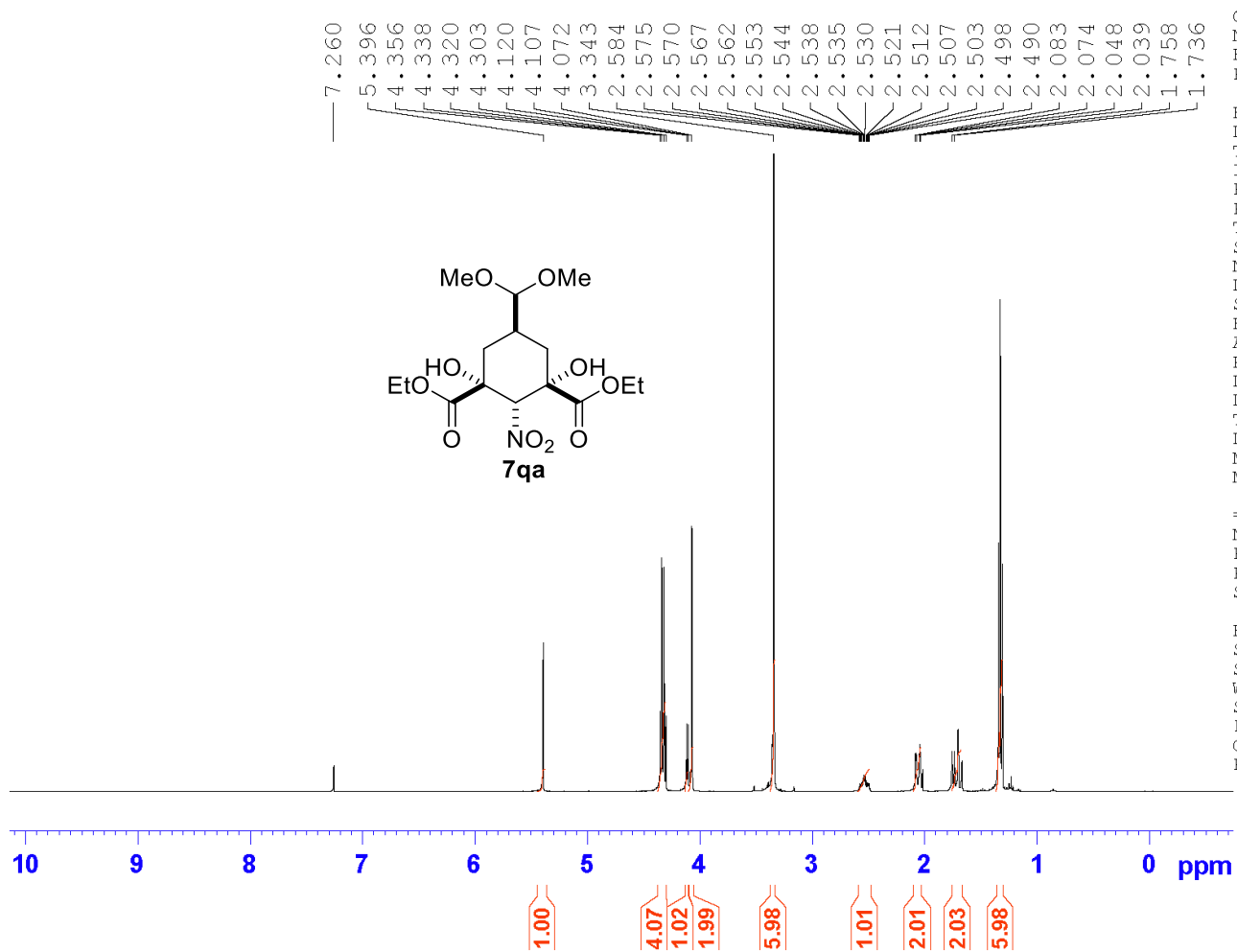
F2 - Acquisition Parameters

Date_ 20120928
Time 21.24
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 90.5
DW 20.800 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -2.00 dB
SFO1 100.6479773 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.70 dB
PL12 17.10 dB
PL13 17.10 dB
SFO2 400.2316009 MHz

F2 - Processing parameters
SI 32768
SF 100.6379140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

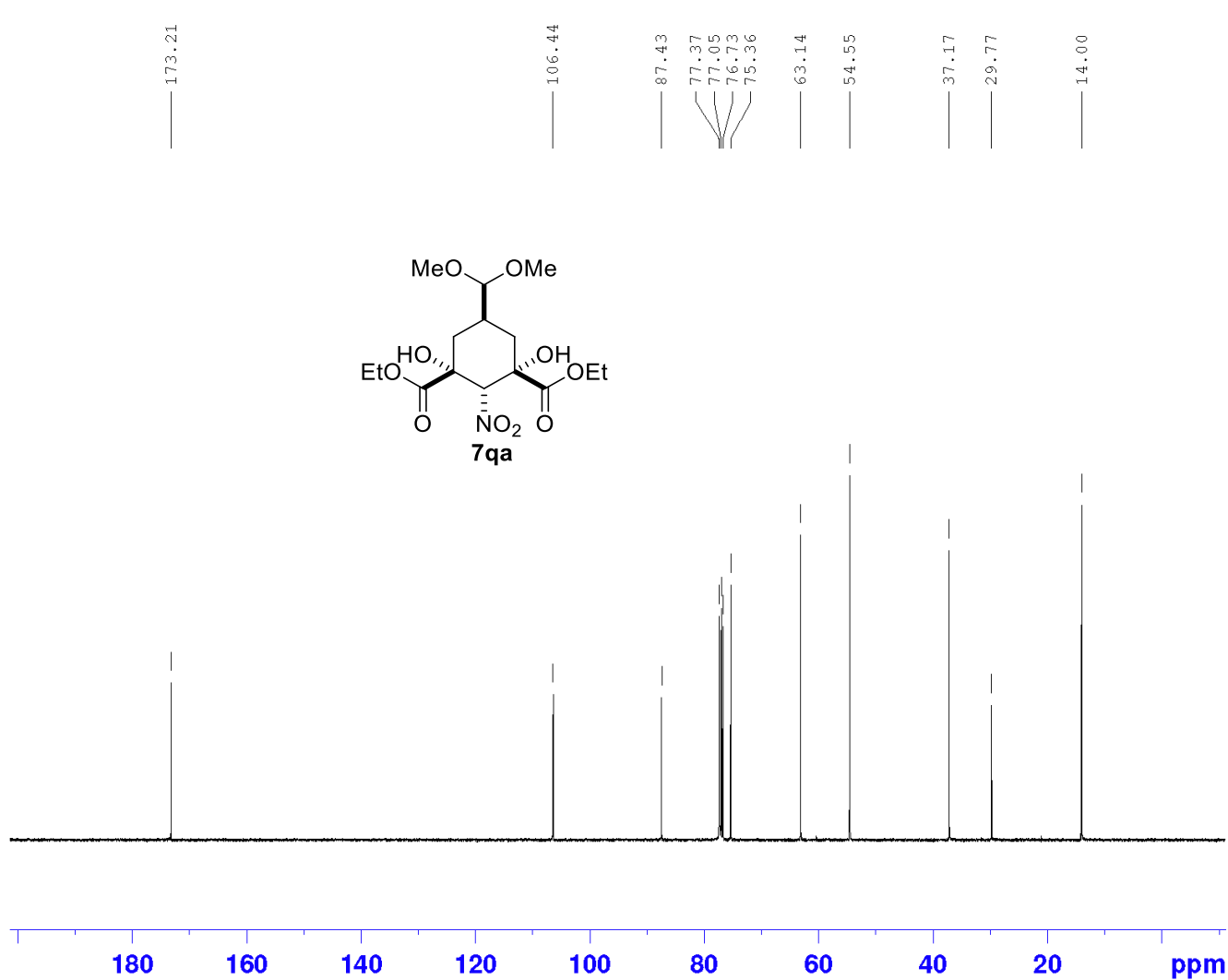
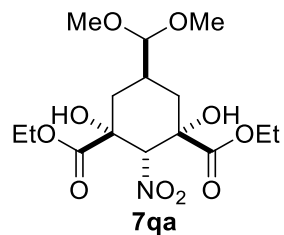


Current Data Parameters
 NAME Jul27-2012
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120727
 Time_ 18.14
 INSTRUM advance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 80.6
 DW 60.600 usec
 DE 6.00 usec
 TE 296.8 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300052 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



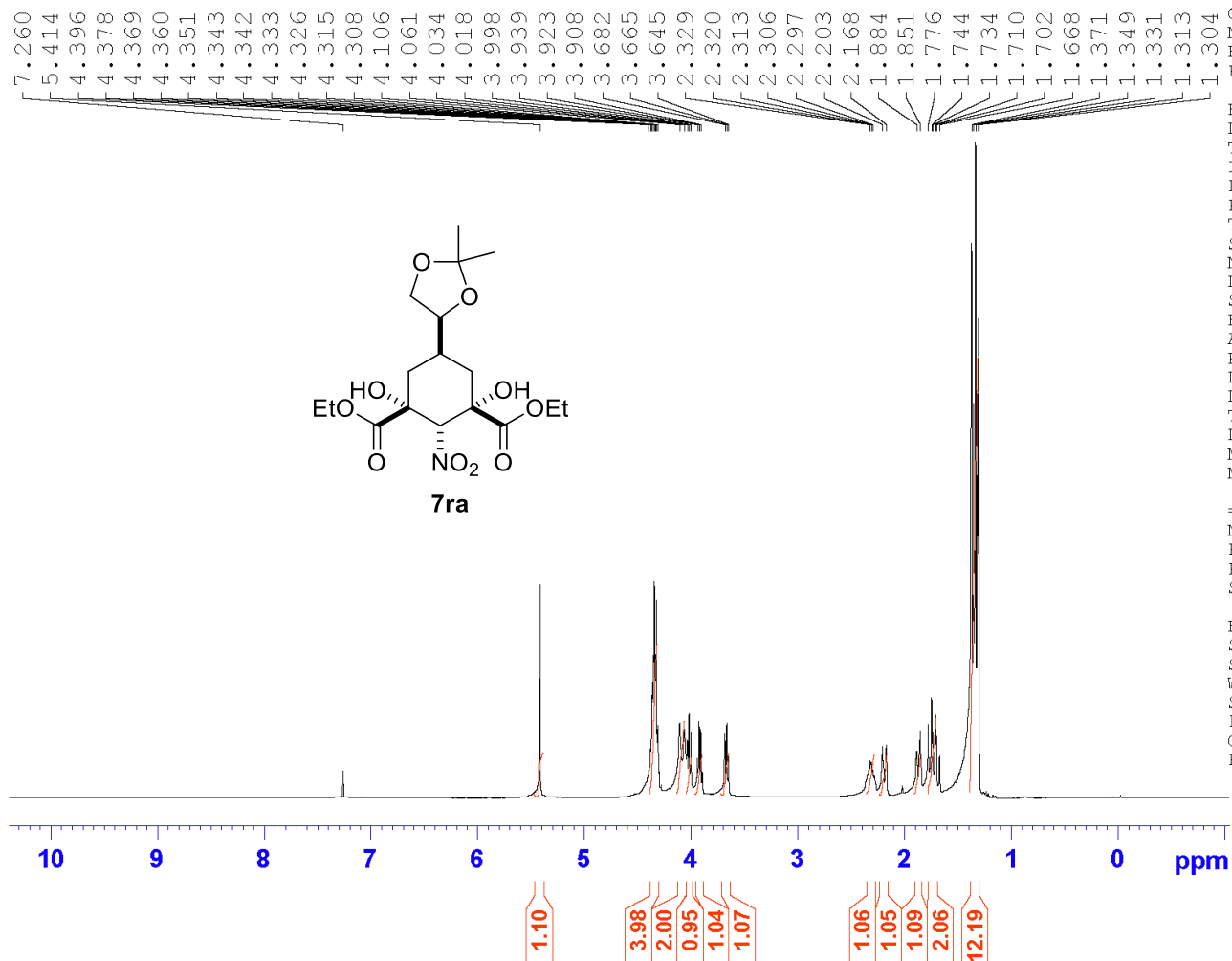
Current Data Parameters
 NAME Jul27-2012
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120727
 Time 19.47
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1500
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 1024
 DW 20.800 usec
 DE 6.00 usec
 TE 298.3 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

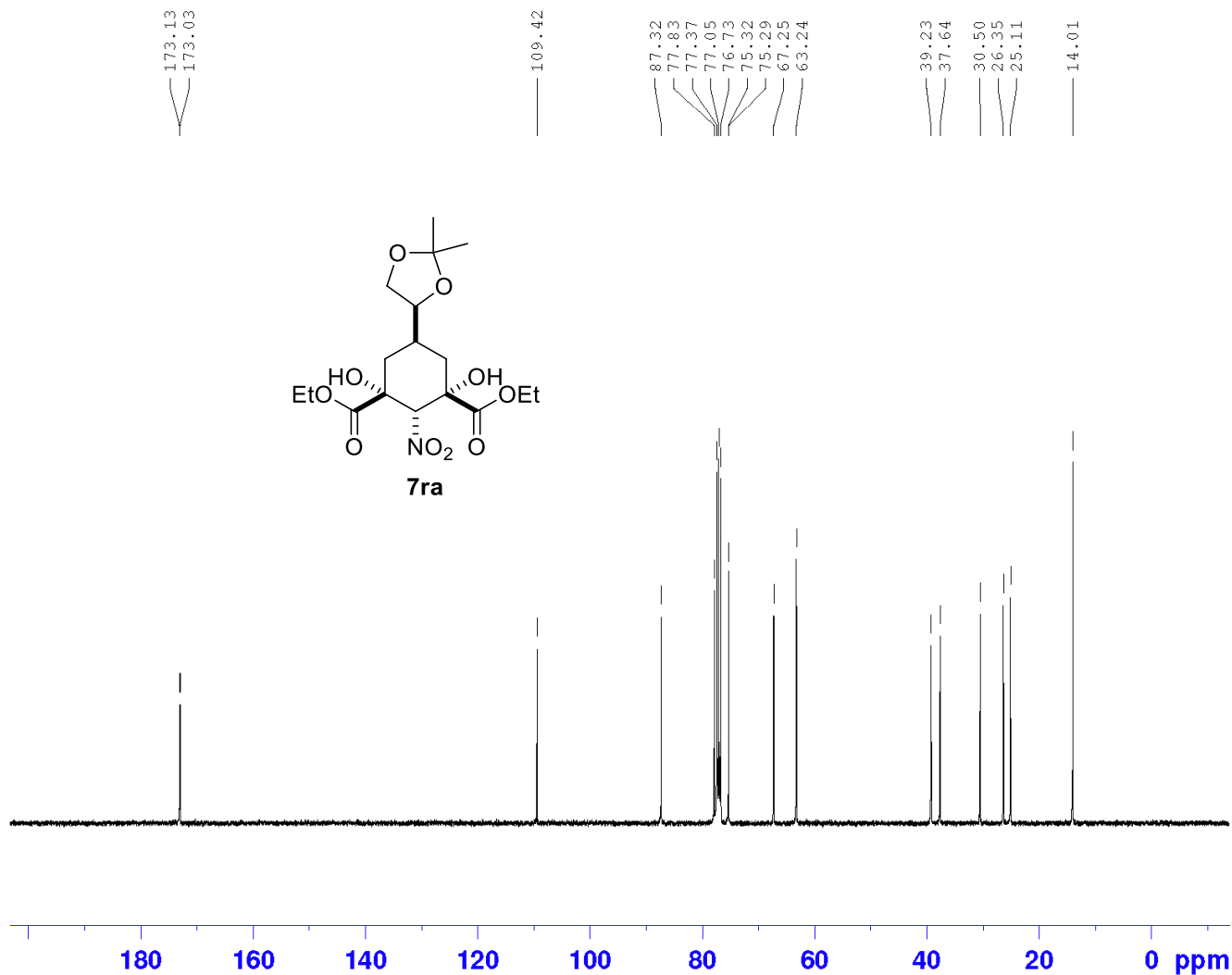
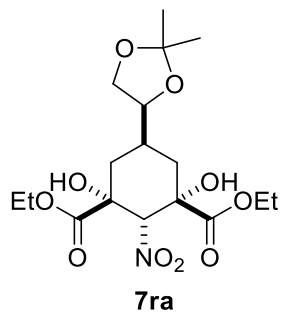


Current Data Parameters
 NAME Dec14-2012
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121214
 Time 12.18
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 71.8
 DW 60.600 usec
 DE 6.00 usec
 TE 295.6 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300052 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



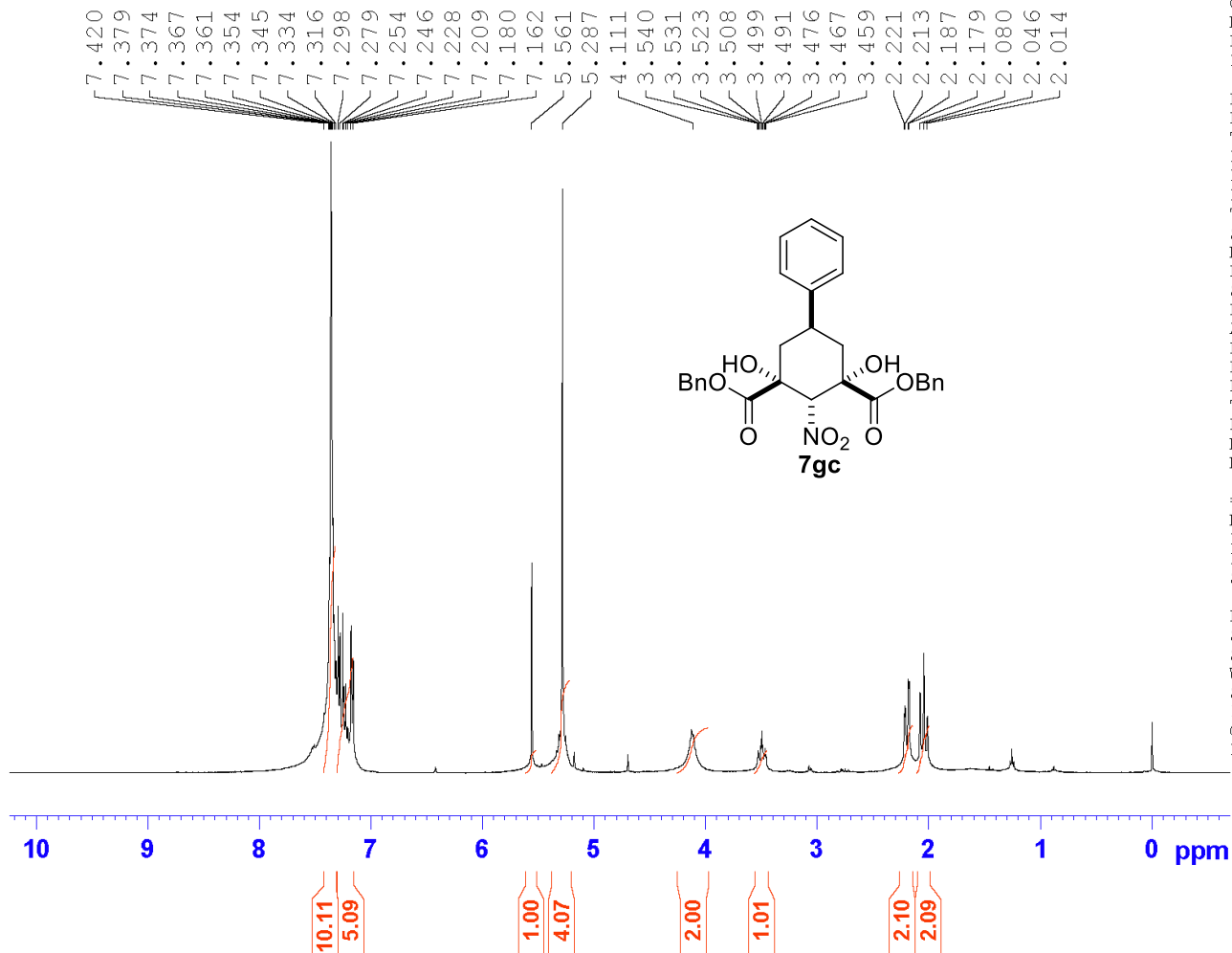
Current Data Parameters
 NAME Dec14-2012
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121214
 Time 14.40
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 1290.2
 DW 20.800 usec
 DE 6.00 usec
 TE 297.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

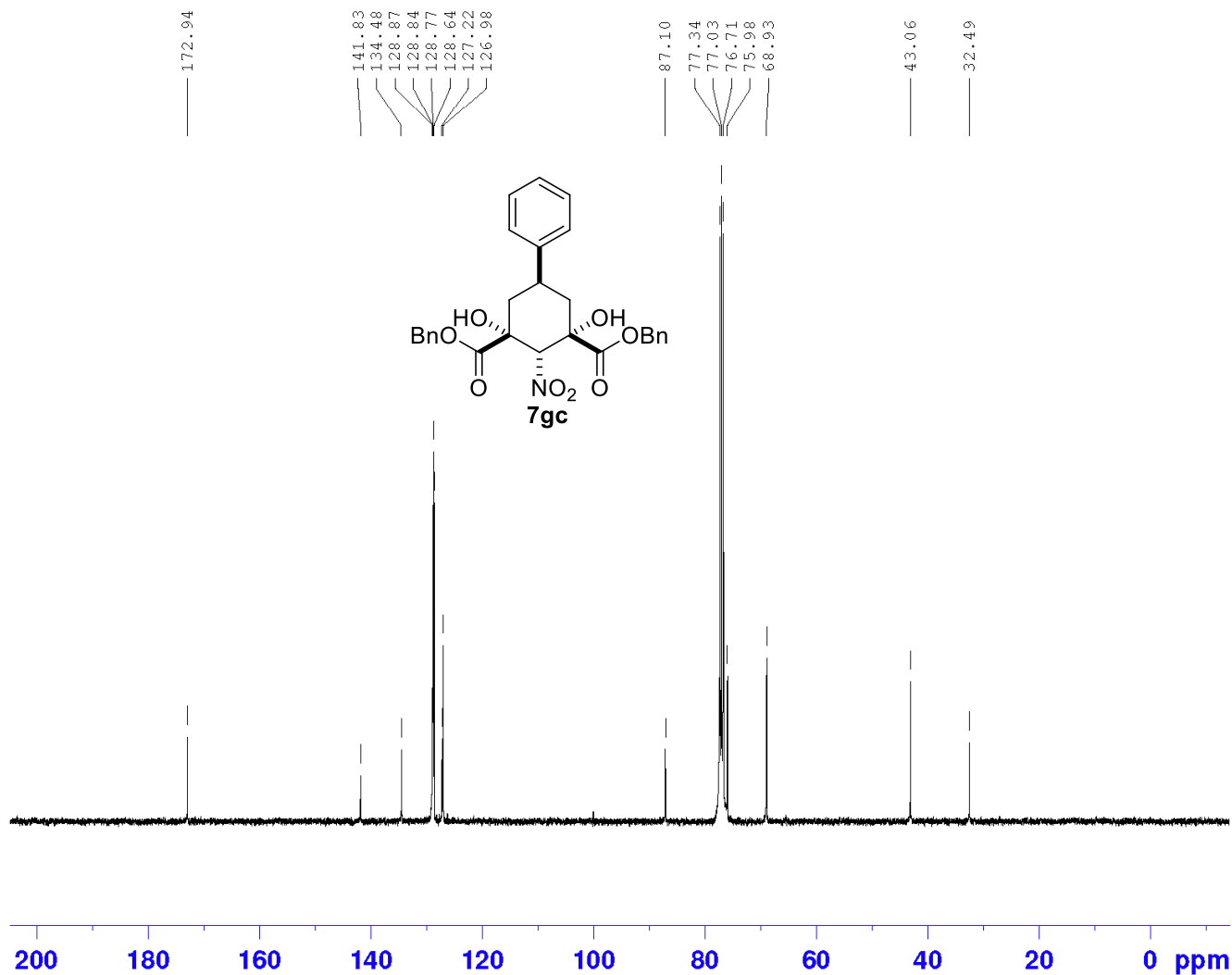


Current Data Parameters
 NAME Dec07-2012
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121207
 Time 18.34
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 287.4
 DW 60.600 usec
 DE 6.00 usec
 TE 296.3 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300078 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



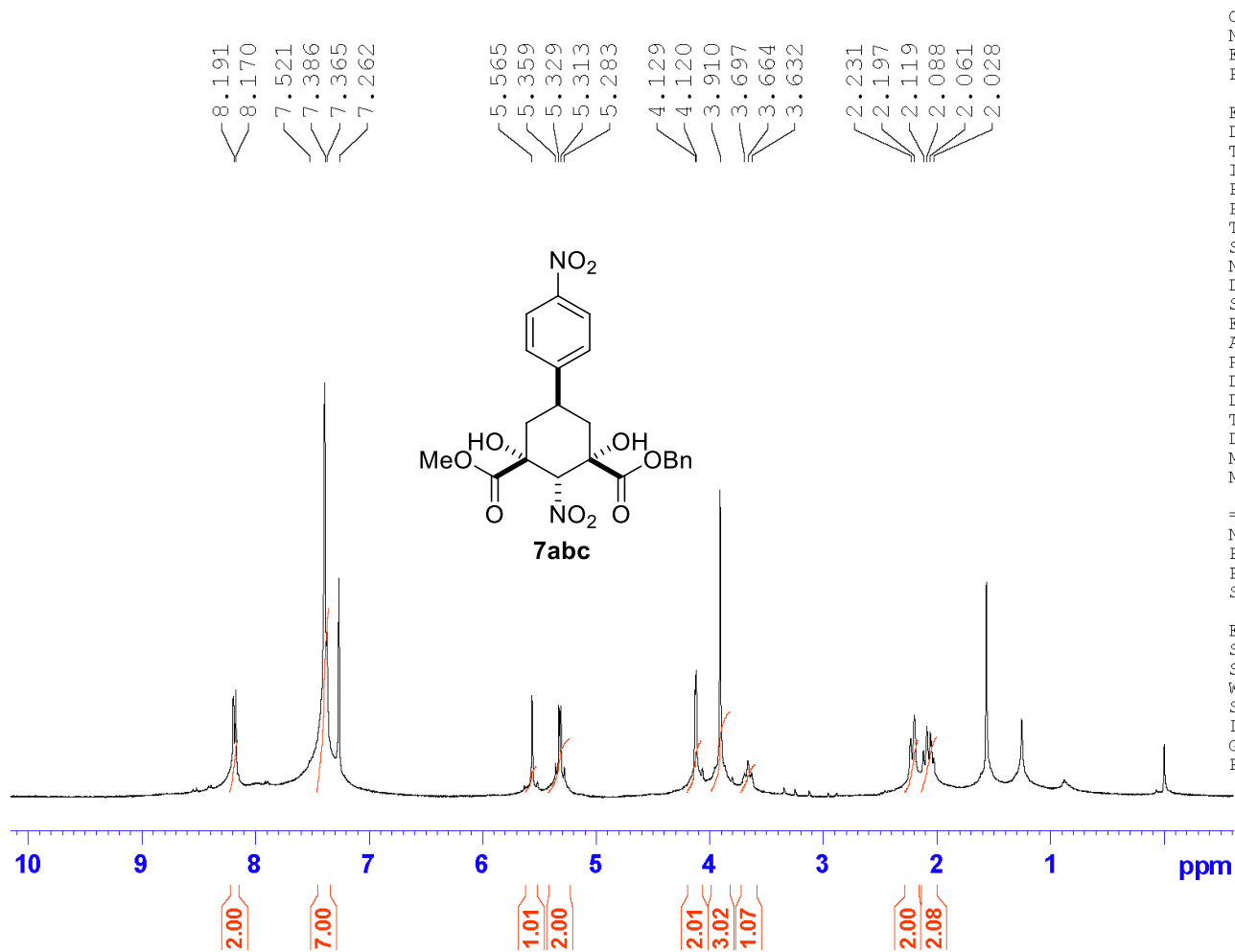
Current Data Parameters
 NAME Dec07-2012
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121207
 Time 23.31
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 5000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 101.6
 DW 20.800 usec
 DE 6.00 usec
 TE 297.4 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

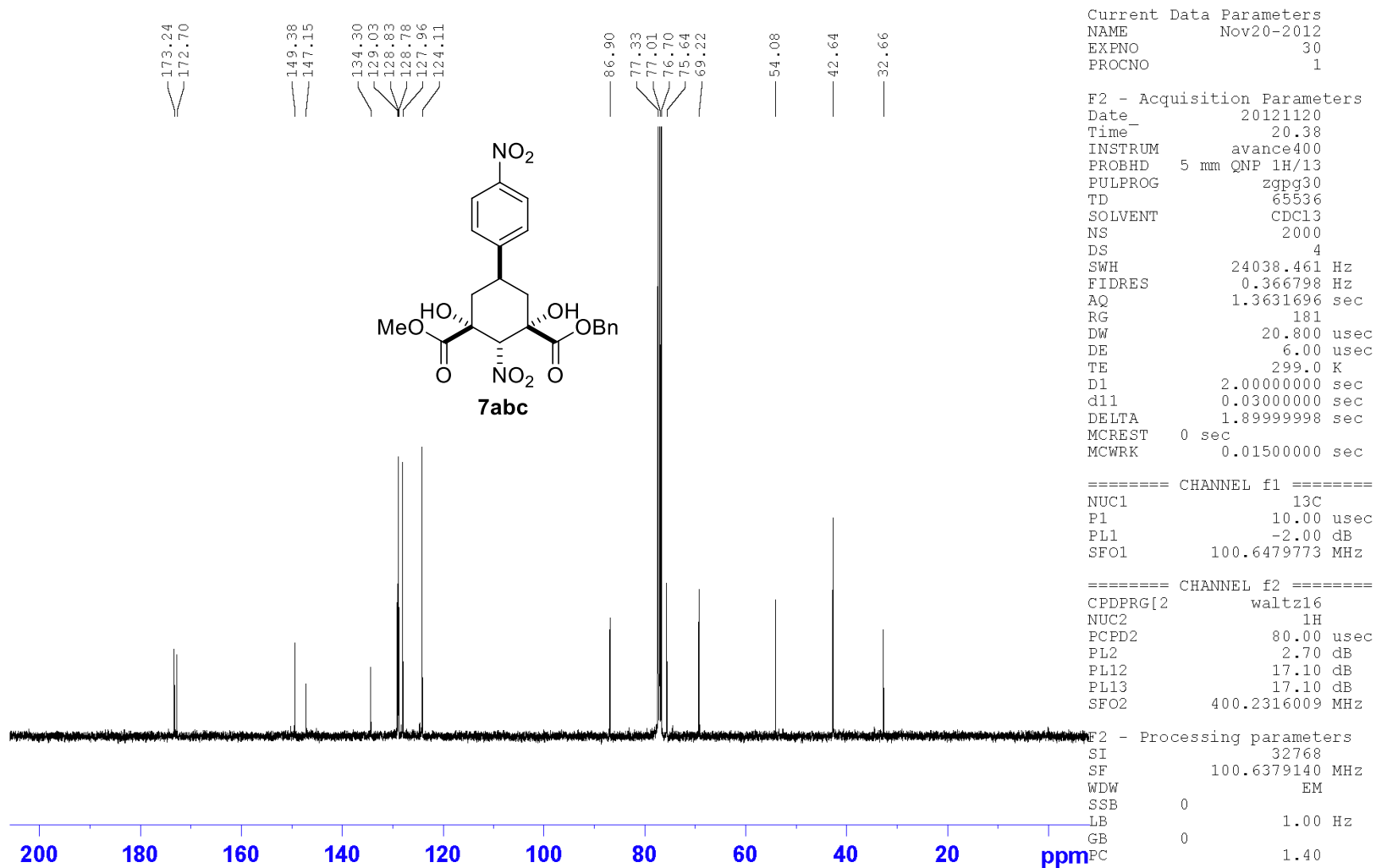


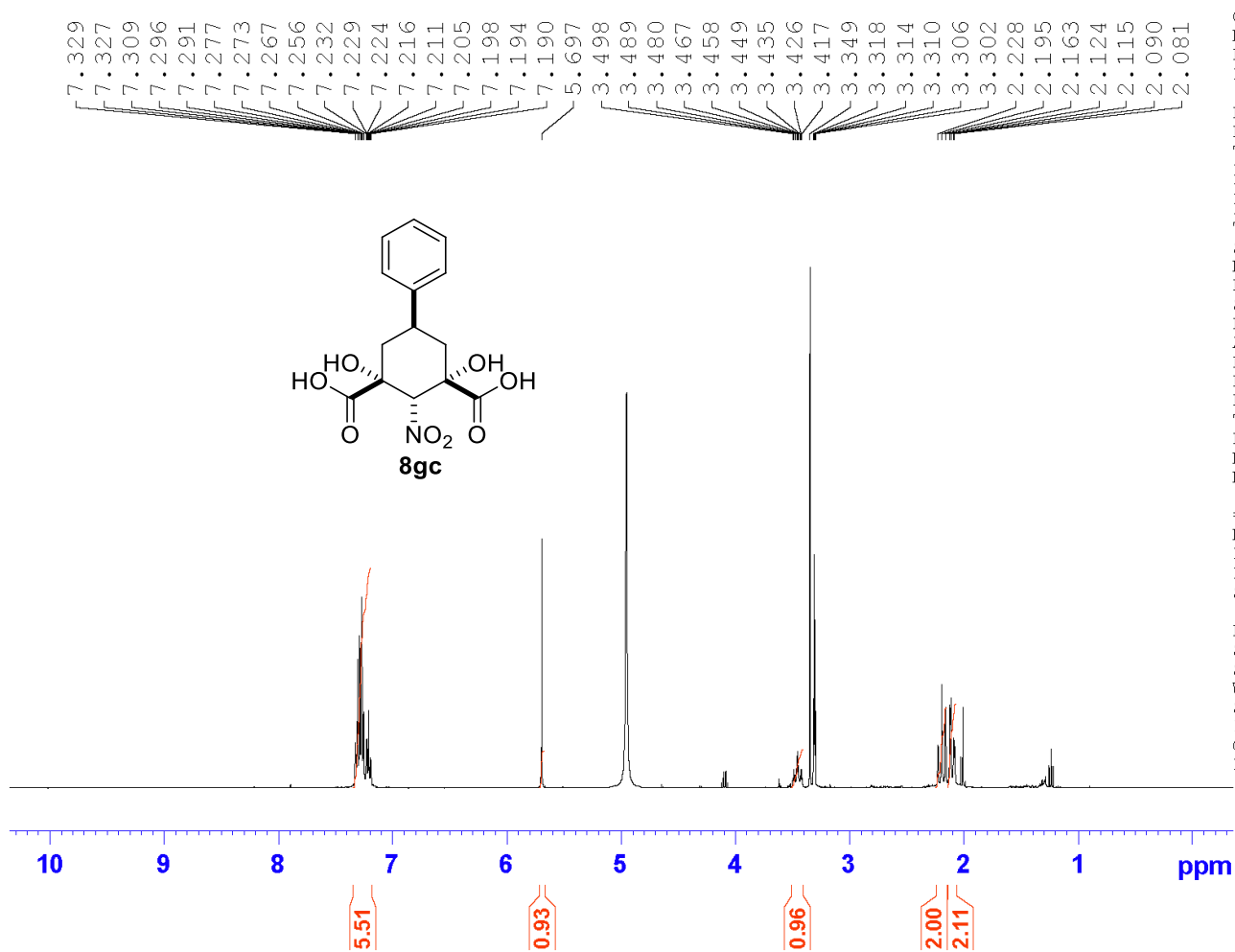
Current Data Parameters
 NAME Nov27-2012
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20121127
 Time 11.50
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 200
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 574.7
 DW 60.600 usec
 DE 6.00 usec
 TE 295.5 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300042 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



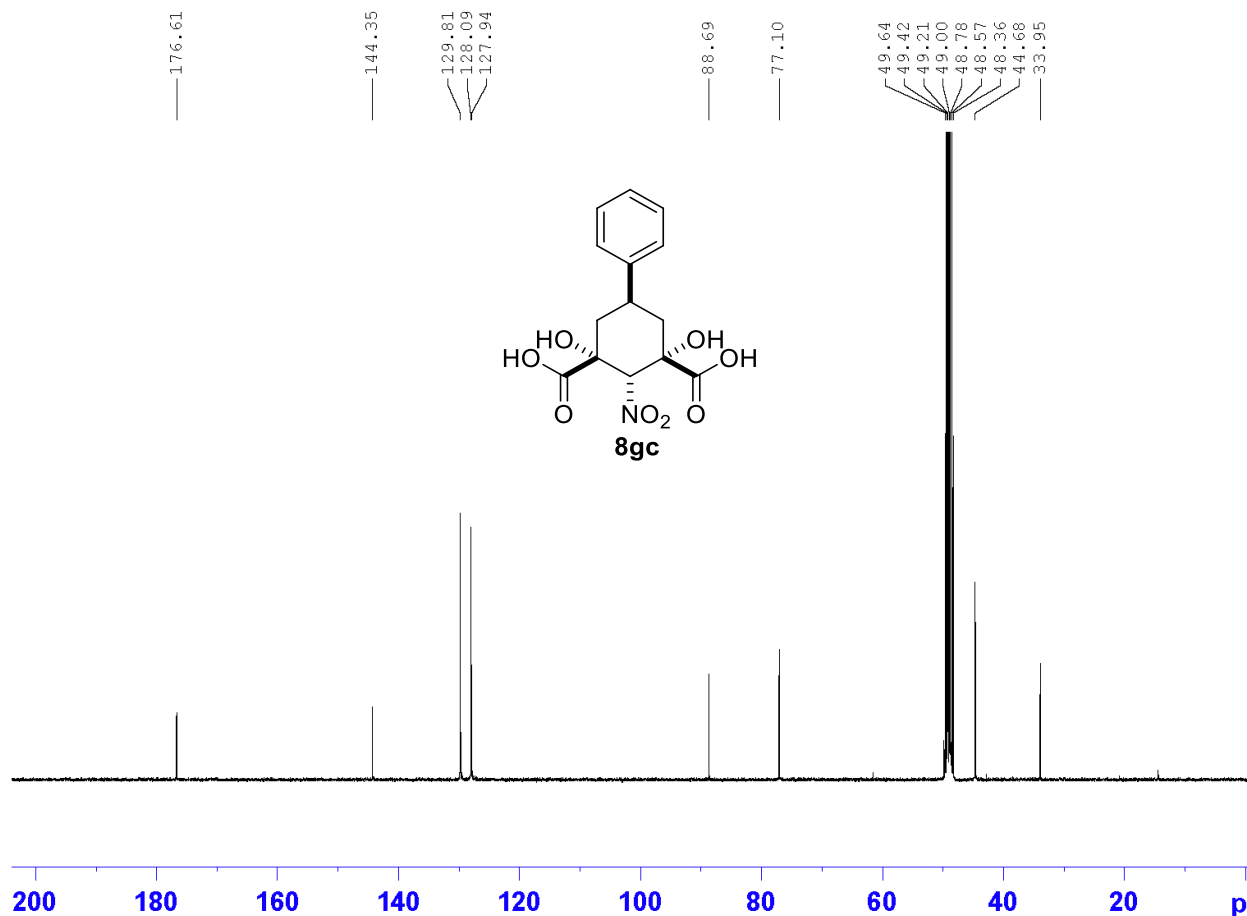


Current Data Parameters
 NAME Mar01-2013
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130301
 Time 10.19
 INSTRUM advance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT MeOD
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 203.2
 DW 60.600 usec
 DE 6.00 usec
 TE 294.8 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300035 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



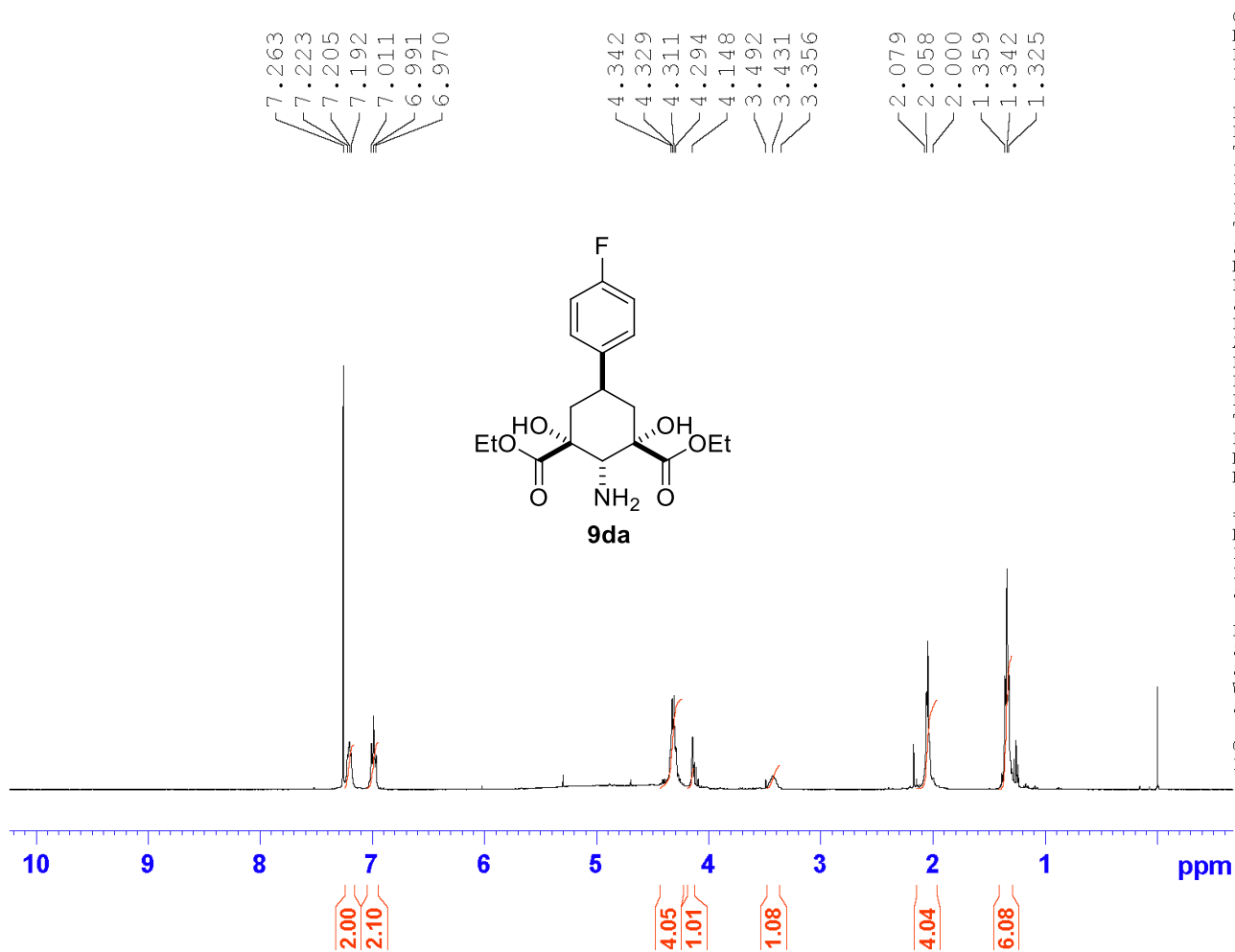
Current Data Parameters
 NAME Feb28-2013
 EXPNO 40
 PROCNO 1

F2 - Acquisition Parameters
 Date 20130301
 Time 2.23
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT MeOD
 NS 7000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631696 sec
 RG 4597.6
 DW 20.800 usec
 DE 6.00 usec
 TE 296.4 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6377732 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

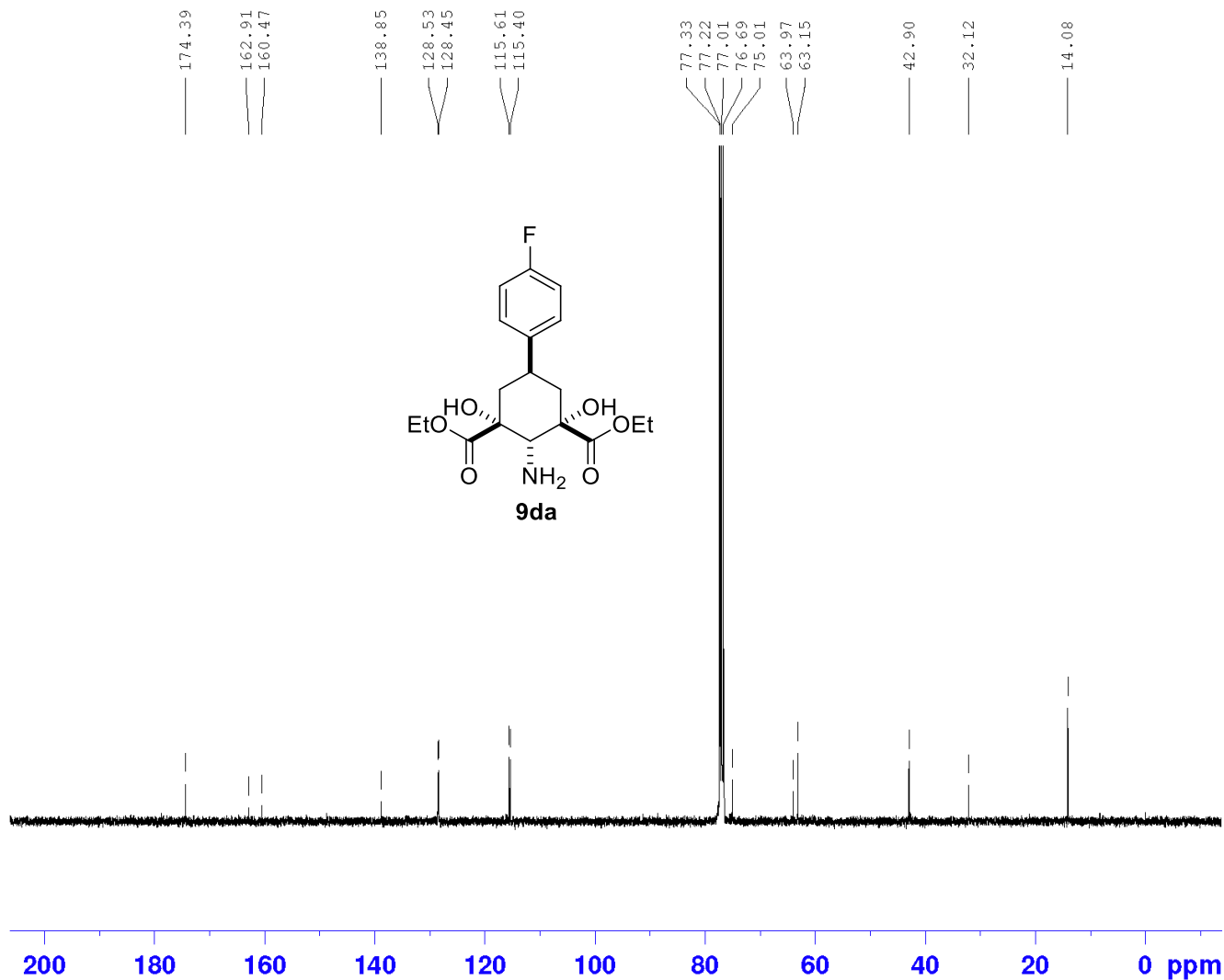


Current Data Parameters
 NAME Mar08-2013
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130308
 Time 10.06
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 50
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 406.4
 DW 60.600 usec
 DE 6.00 usec
 TE 296.3 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300045 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



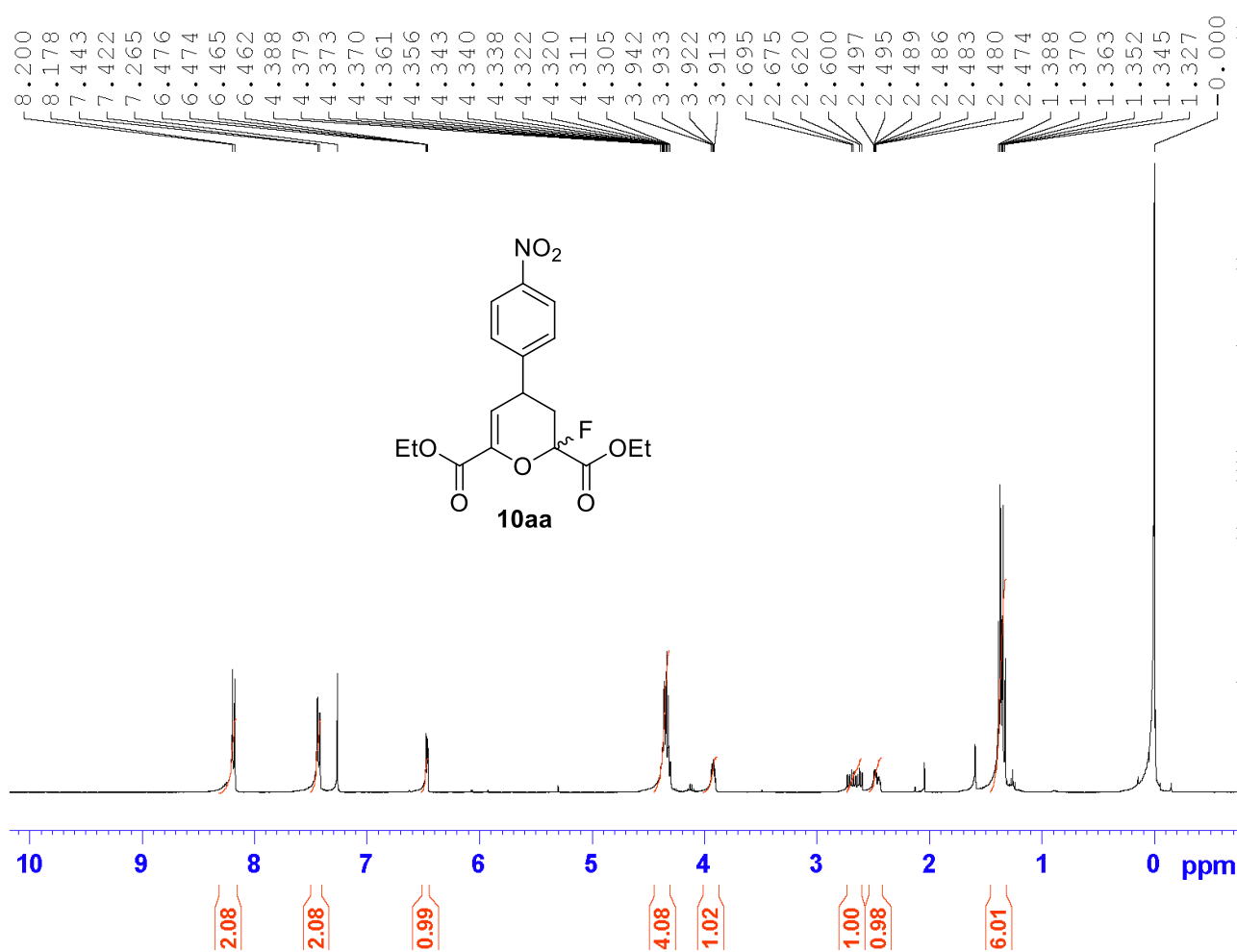
Current Data Parameters
 NAME Mar07-2013
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130308
 Time 0.42
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 6000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 1024
 DW 20.800 usec
 DE 6.00 usec
 TE 297.7 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379140 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

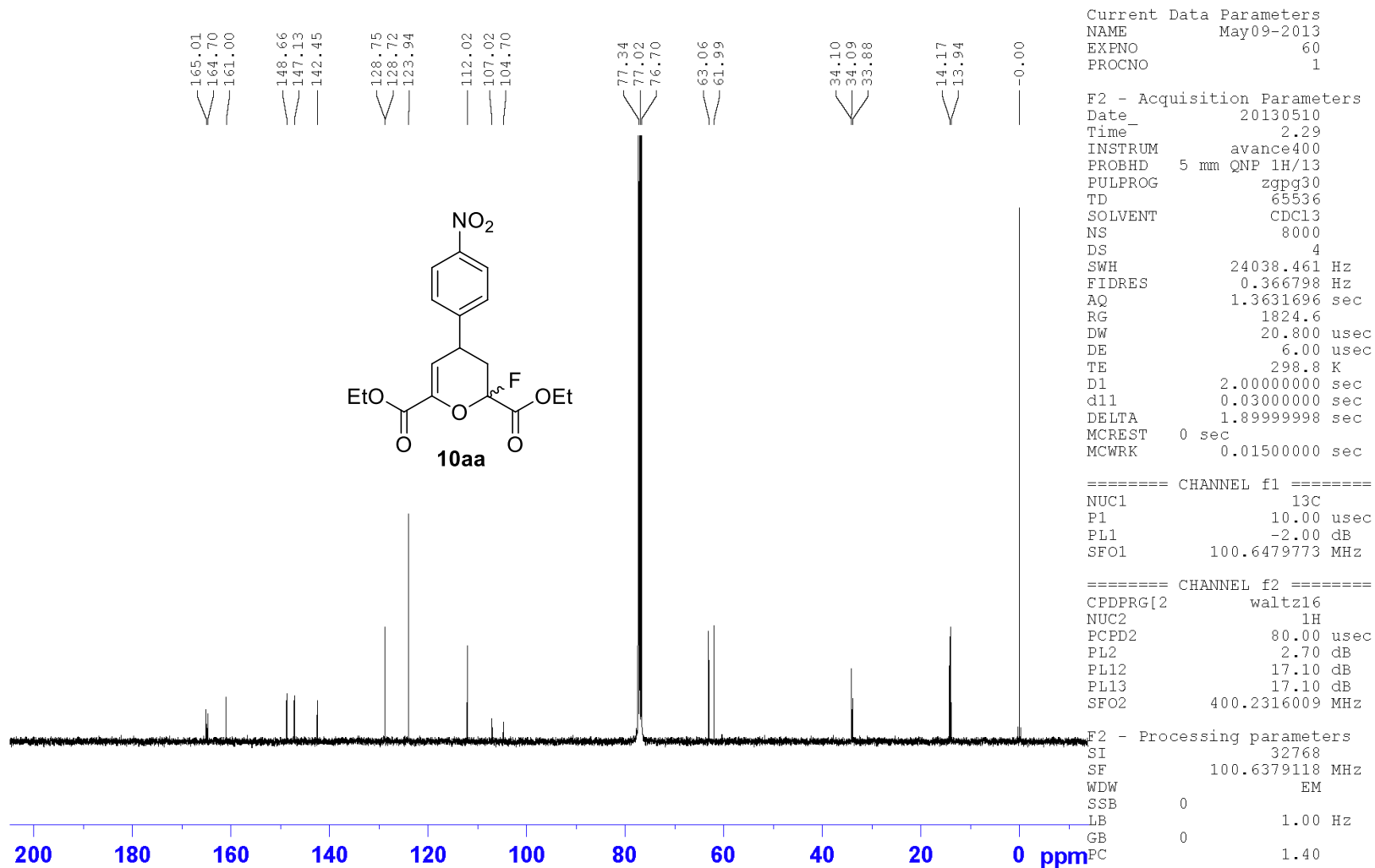


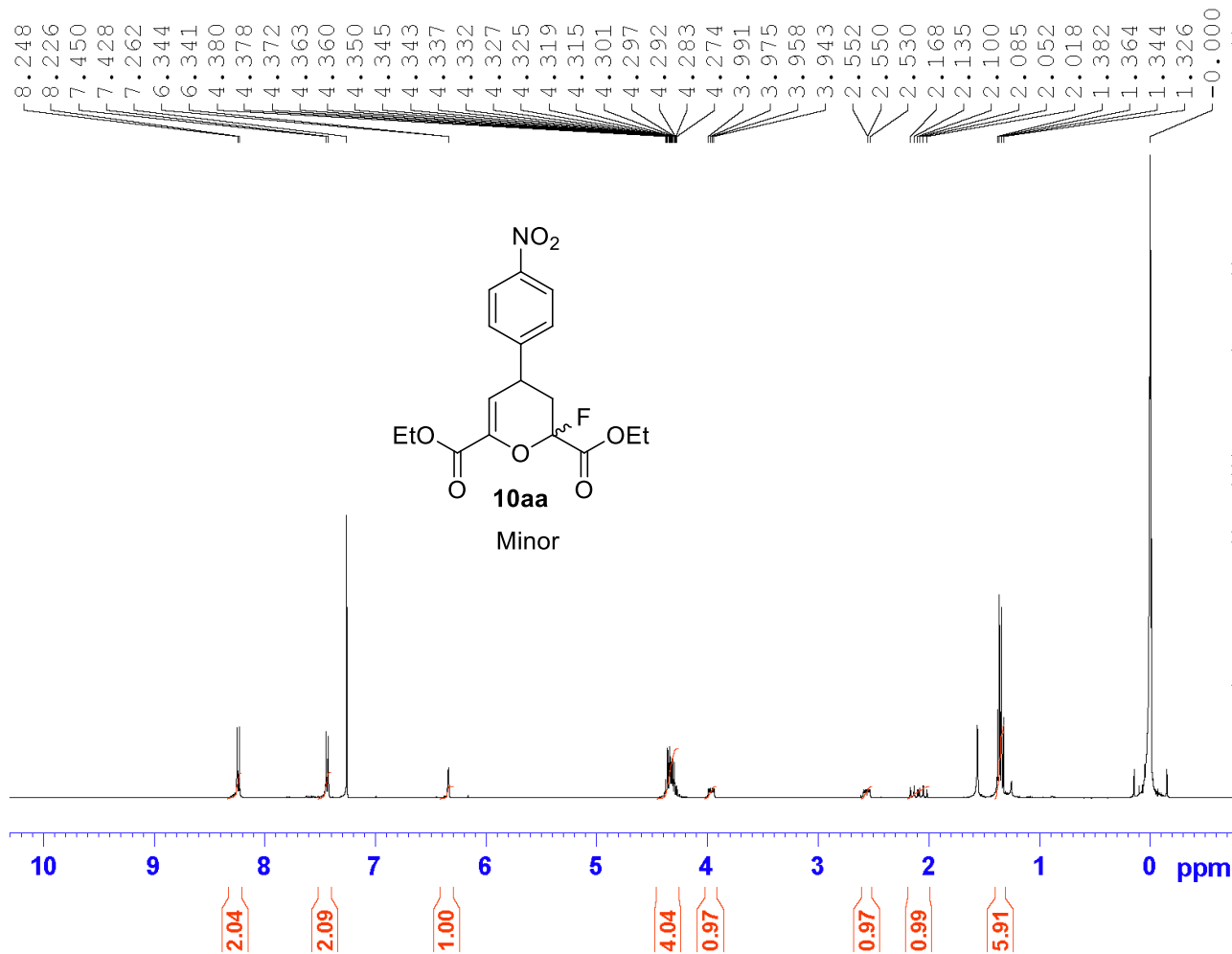
Current Data Parameters
 NAME Apr25-2013
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130425
 Time 12.06
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 203.2
 DW 60.600 usec
 DE 6.00 usec
 TE 298.1 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300030 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



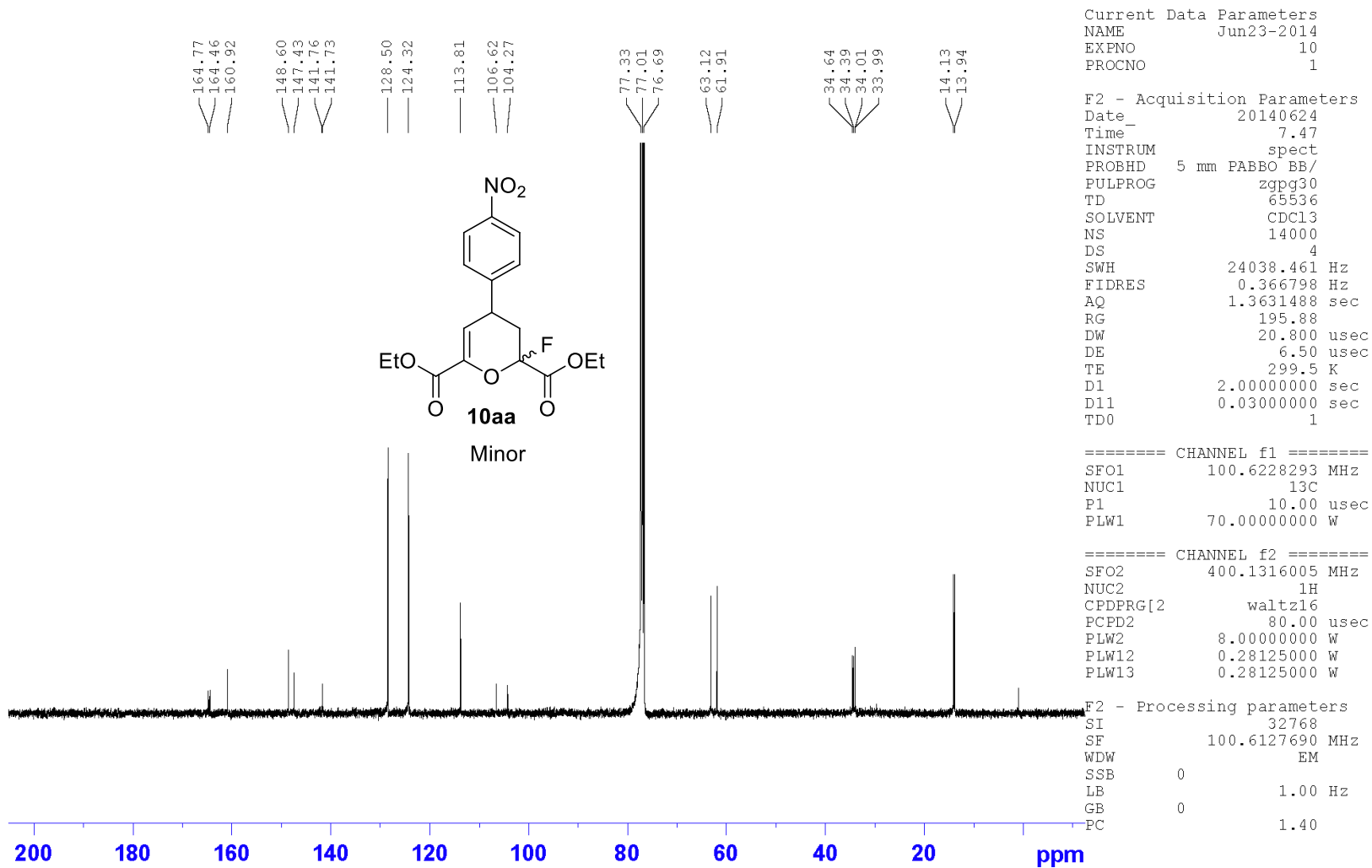


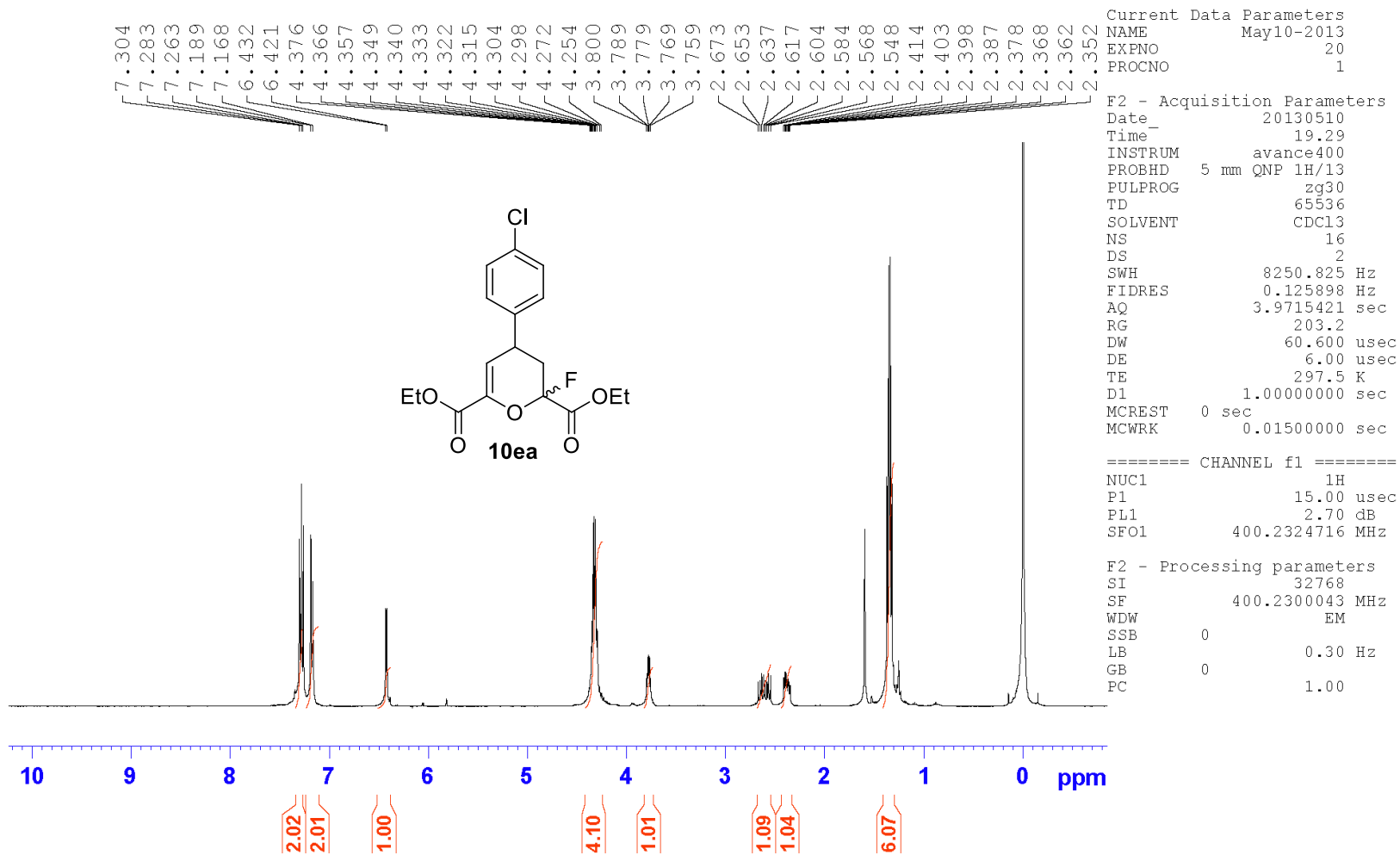
Current Data Parameters
NAME May09-2013
EXPNO 50
PROCNO 1

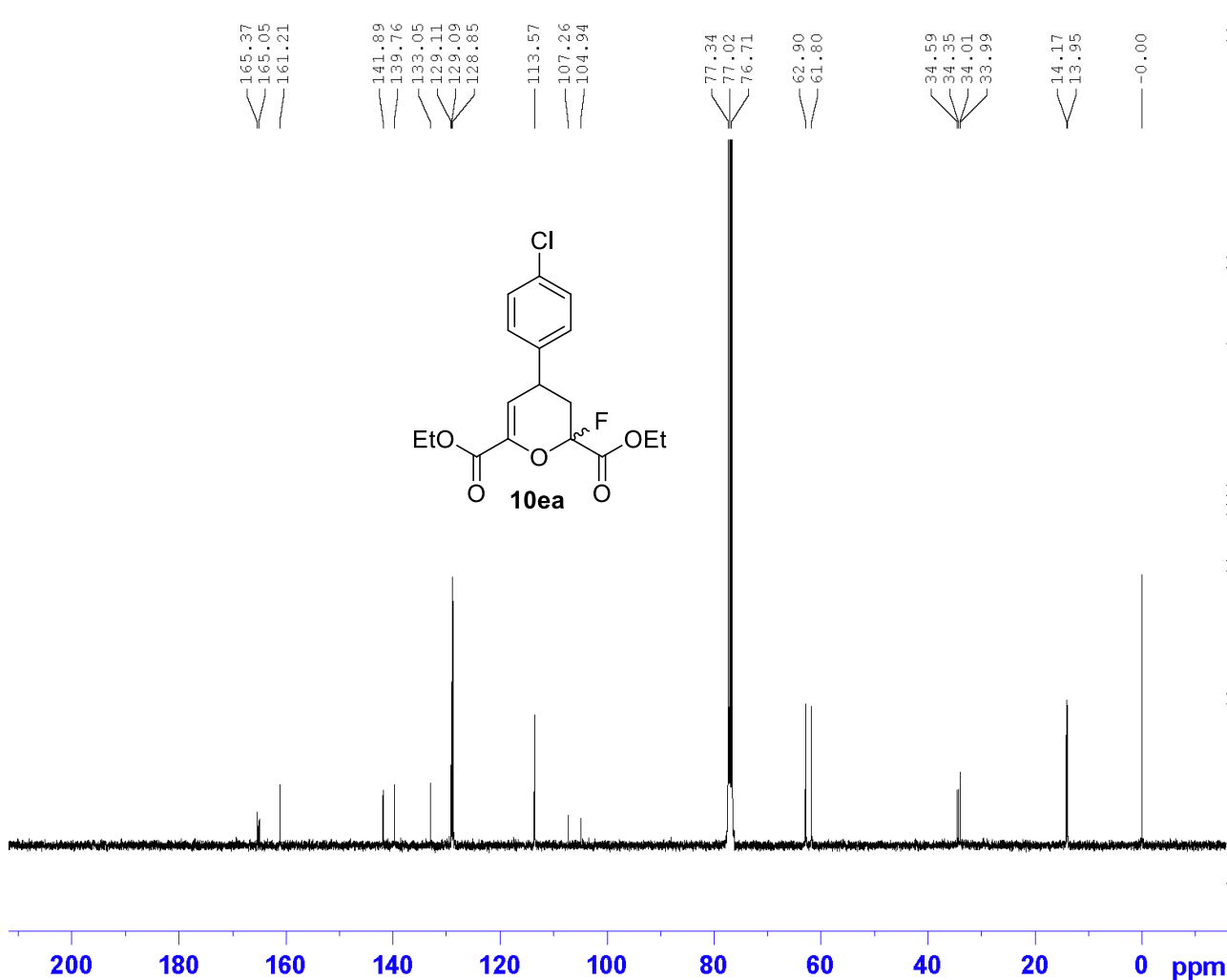
F2 - Acquisition Parameters
Date_ 20130509
Time_ 18.01
INSTRUM advance400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715421 sec
RG 256
DW 60.600 usec
DE 6.00 usec
TE 297.5 K
D1 1.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 2.70 dB
SFO1 400.2324716 MHz

F2 - Processing parameters
SI 32768
SF 400.2300047 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00







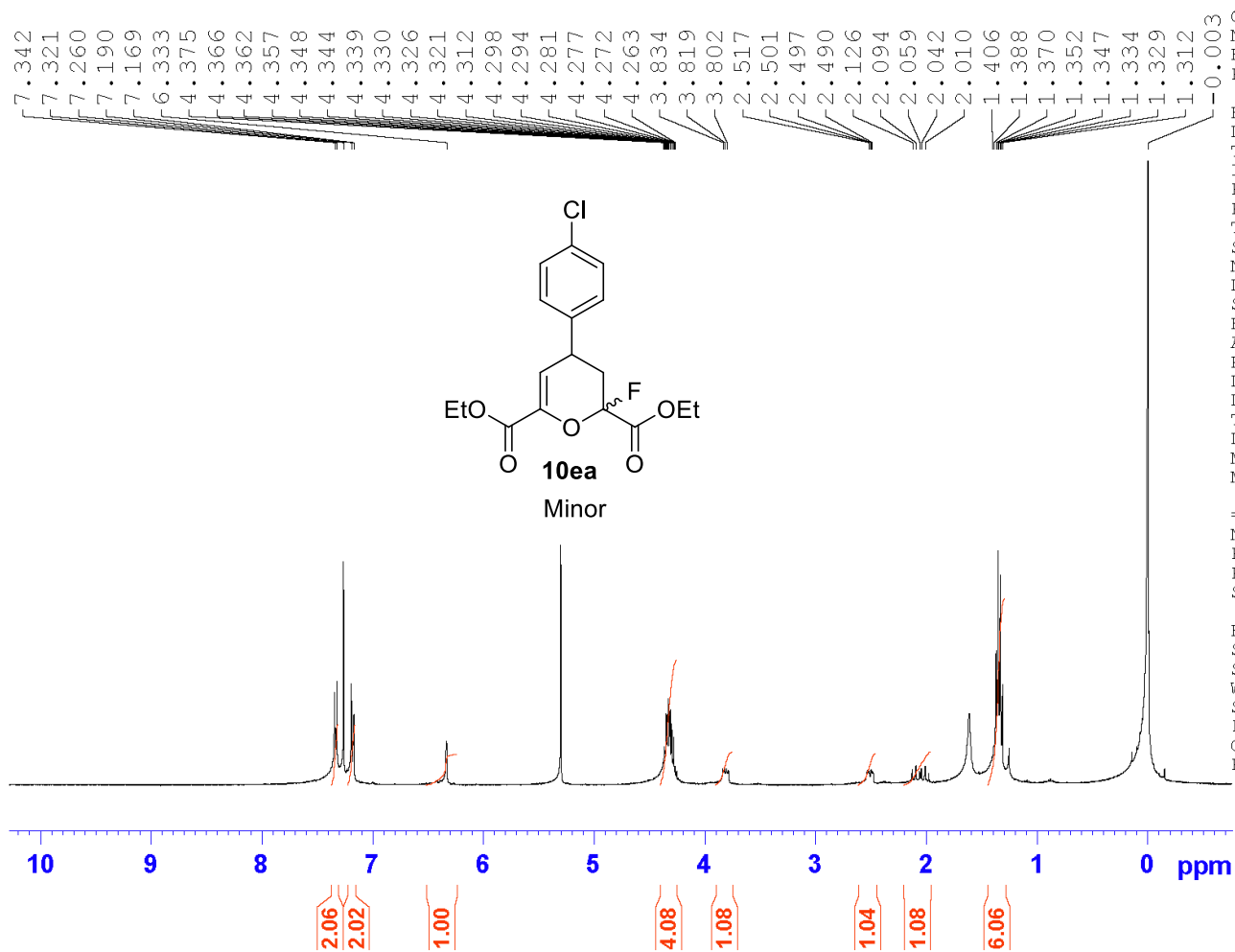
Current Data Parameters
 NAME May10-2013
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130511
 Time_ 3.19
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 8000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631696 sec
 RG 2048
 DW 20.800 usec
 DE 6.00 usec
 TE 298.7 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379121 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

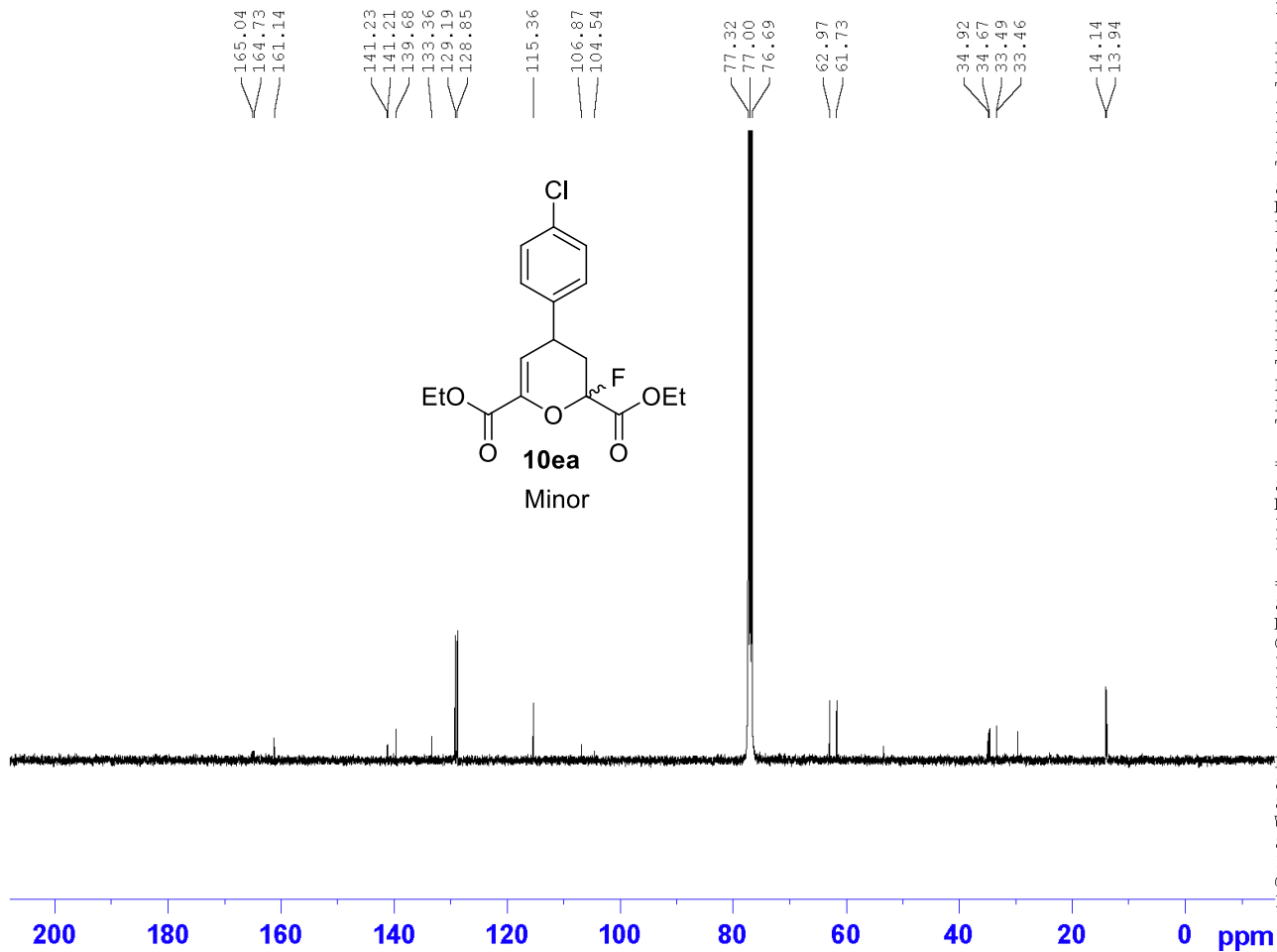


Current Data Parameters
NAME May23-2013
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130523
Time 12.40
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715421 sec
RG 287.4
DW 60.600 usec
DE 6.00 usec
TE 297.7 K
D1 1.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 2.70 dB
SFO1 400.2324716 MHz

F2 - Processing parameters
SI 32768
SF 400.2300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



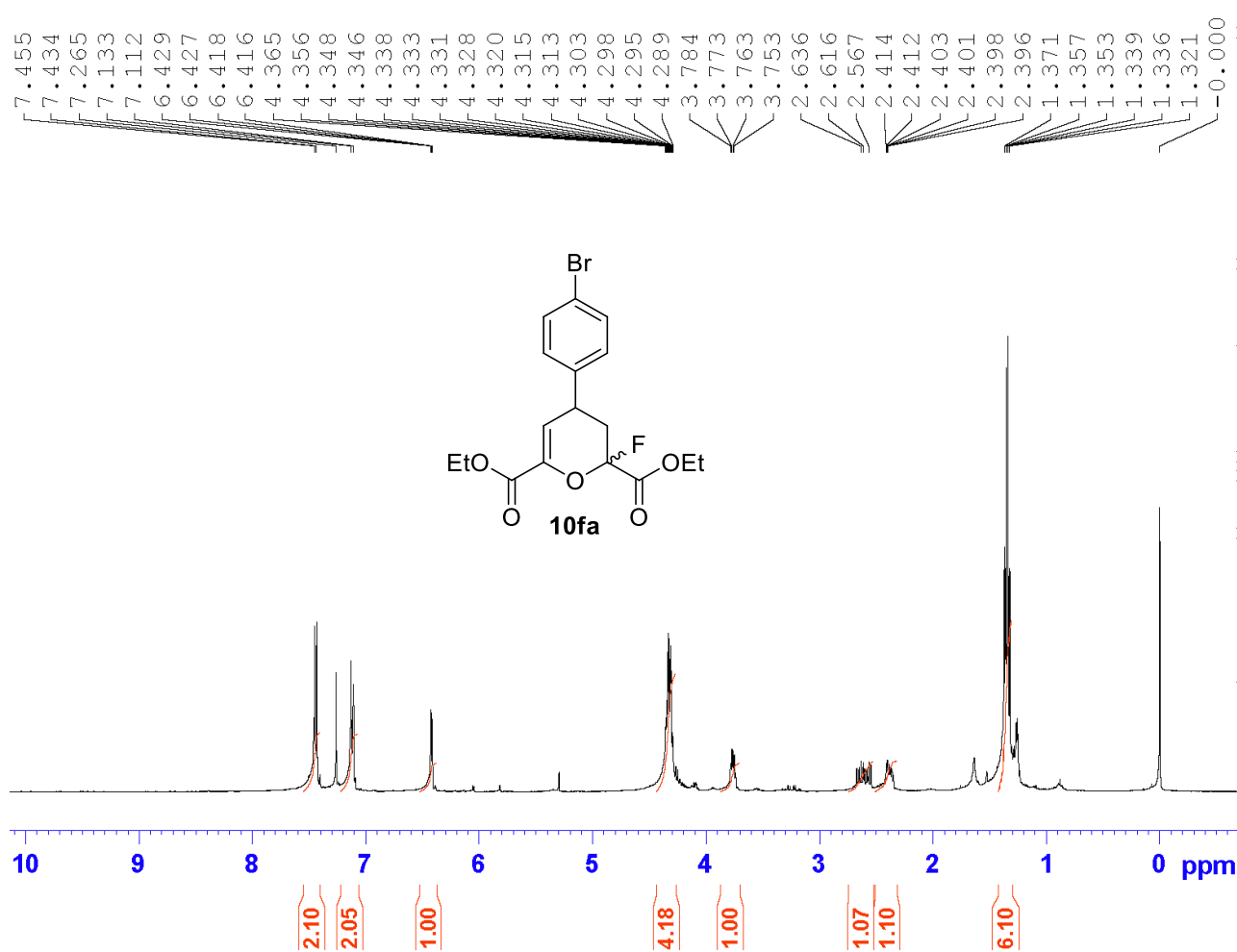
Current Data Parameters
 NAME Jun13-2014
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140614
 Time_ 8.14
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 15000
 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 299.6 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

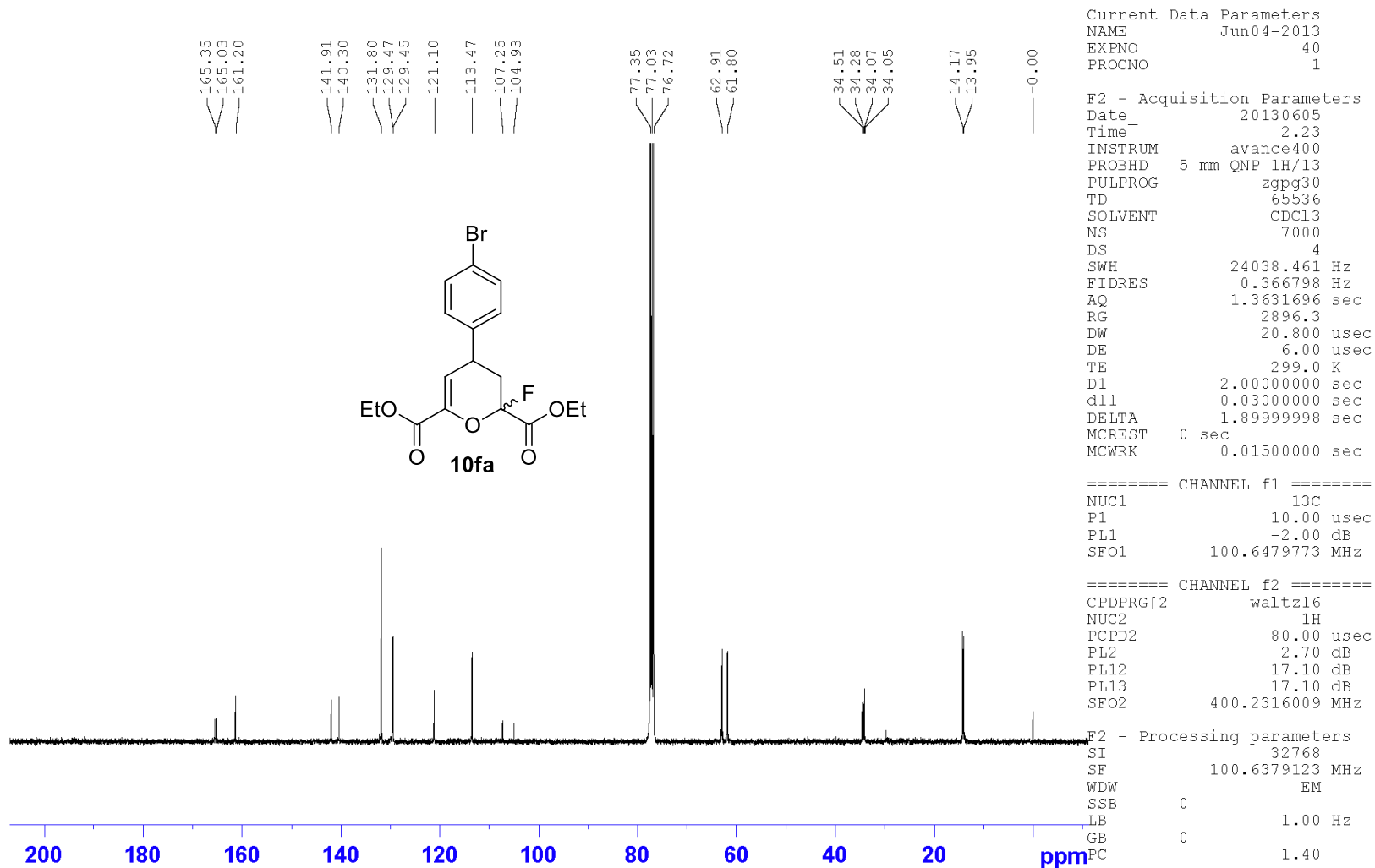


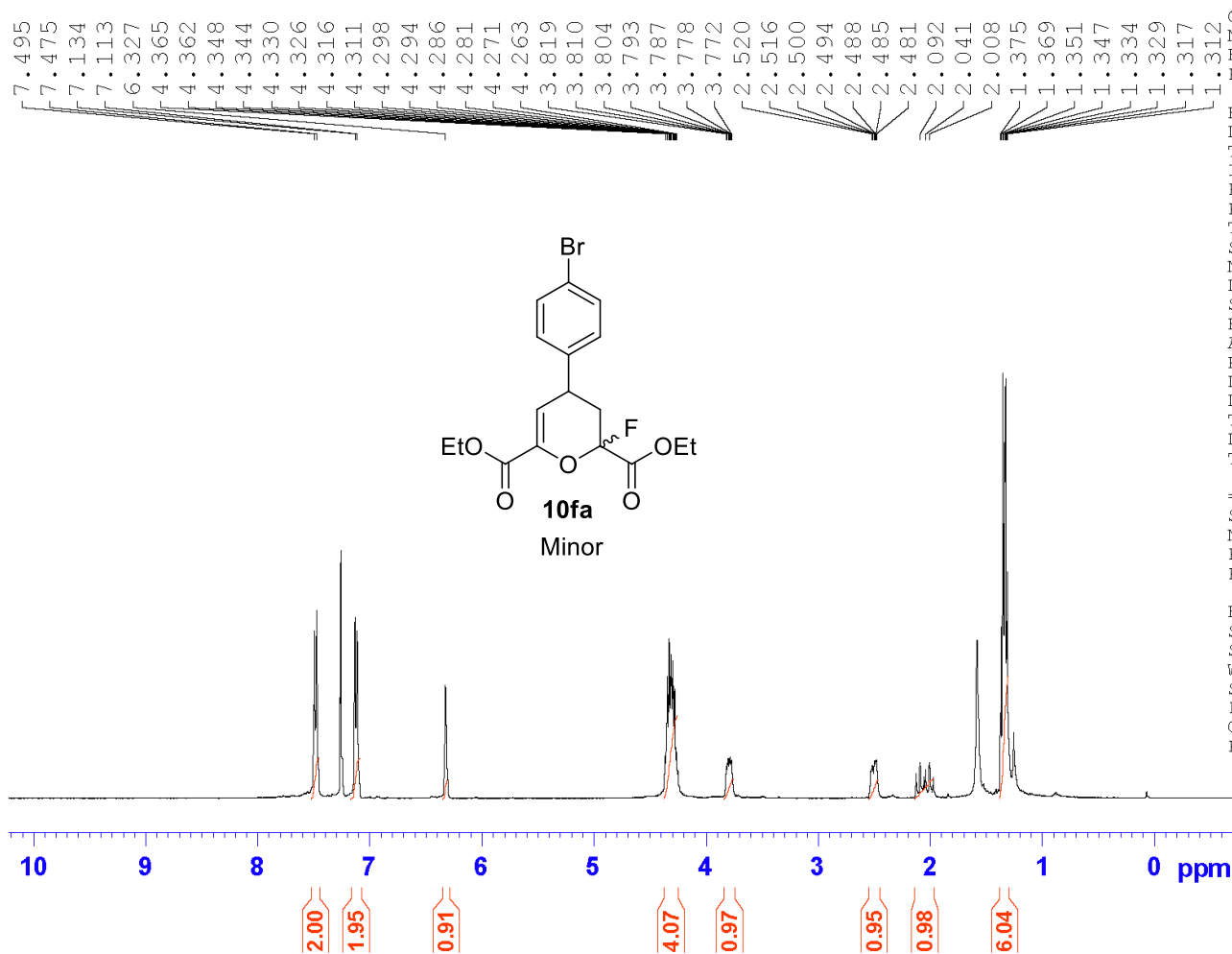
Current Data Parameters
 NAME Jun04-2013
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130604
 Time_ 19.36
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 181
 DW 60.600 usec
 DE 6.00 usec
 TE 297.7 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300032 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



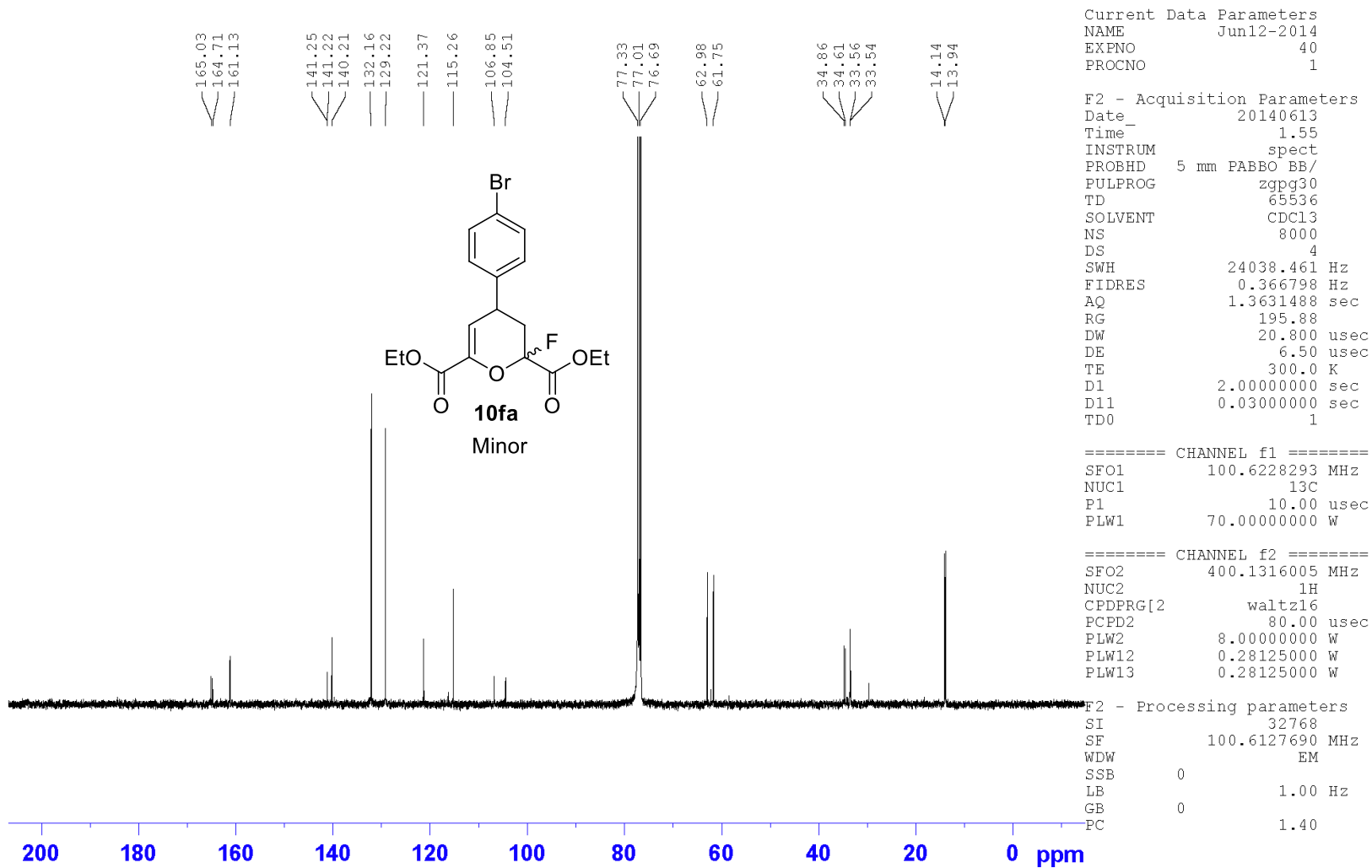


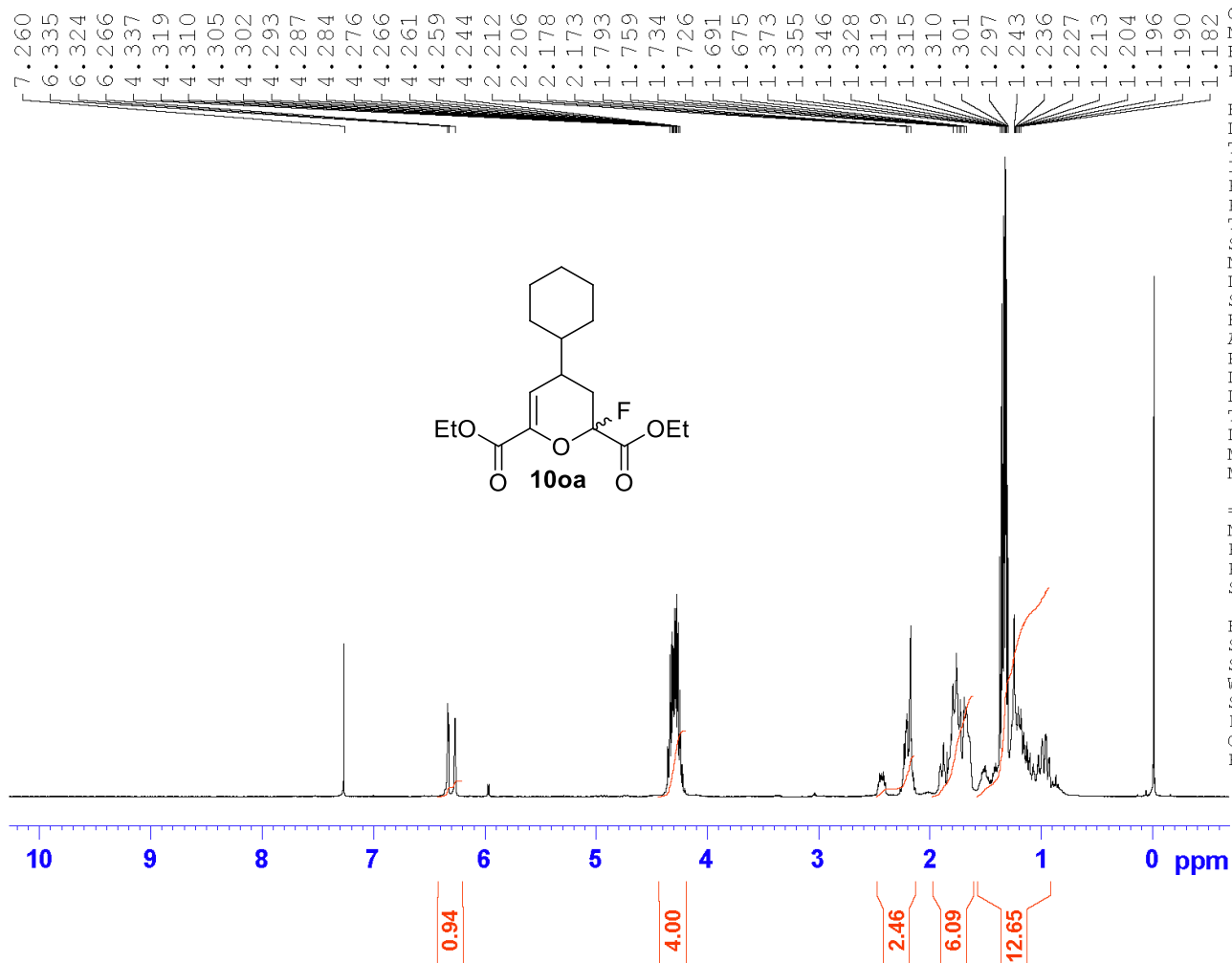
Current Data Parameters
NAME Mar28-2014
EXPNO 70
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140328
Time_ 12.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 77.81
DW 62.400 usec
DE 6.50 usec
TE 299.6 K
D1 1.00000000 sec
TD0 1

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

F2 - Processing parameters
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



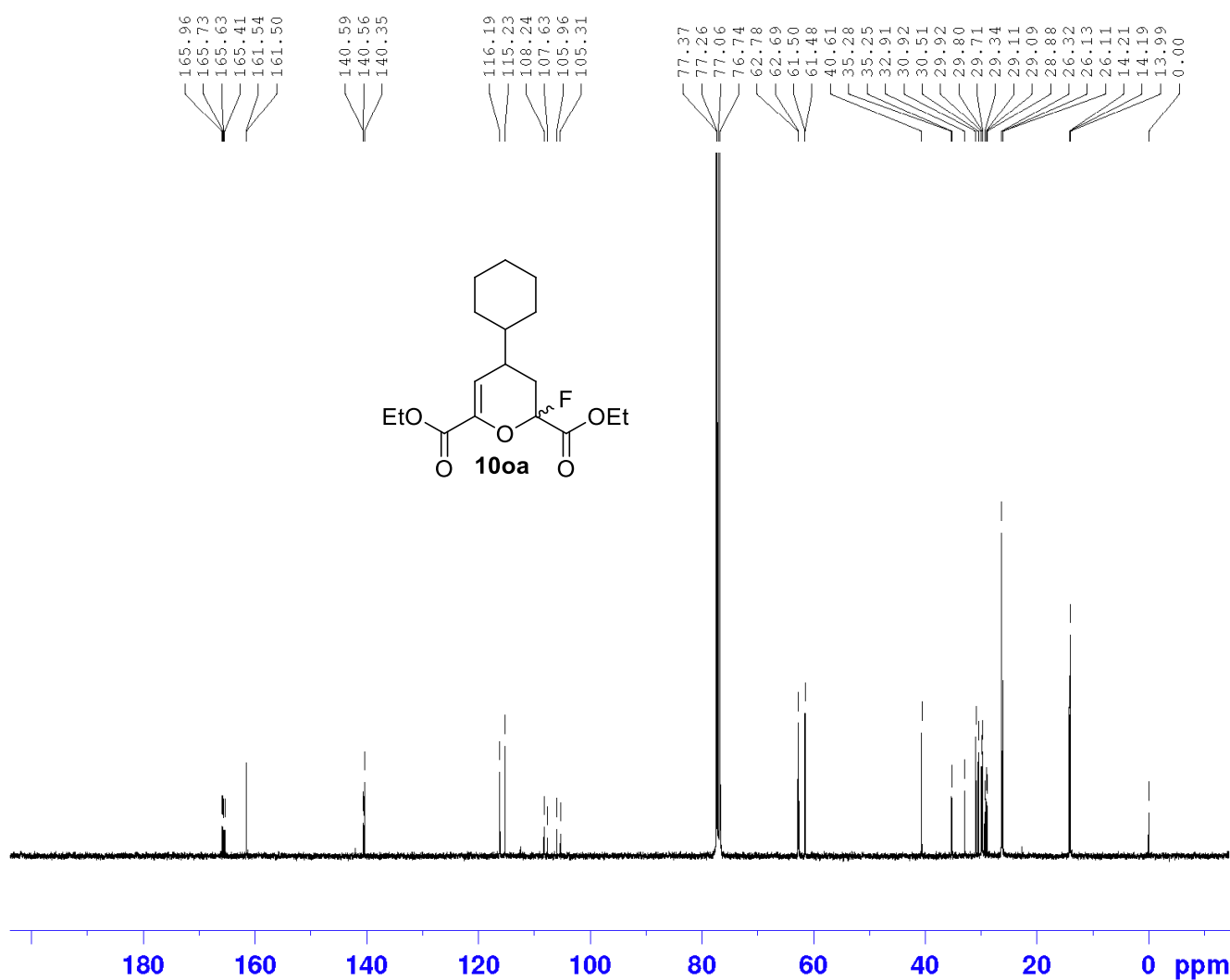


Current Data Parameters
 NAME Jun05-2013
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130605
 Time 18.24
 INSTRUM avance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8250.825 Hz
 FIDRES 0.125898 Hz
 AQ 3.9715421 sec
 RG 101.6
 DW 60.600 usec
 DE 6.00 usec
 TE 297.7 K
 D1 1.00000000 sec
 MCREST 0 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 2.70 dB
 SFO1 400.2324716 MHz

F2 - Processing parameters
 SI 32768
 SF 400.2300052 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



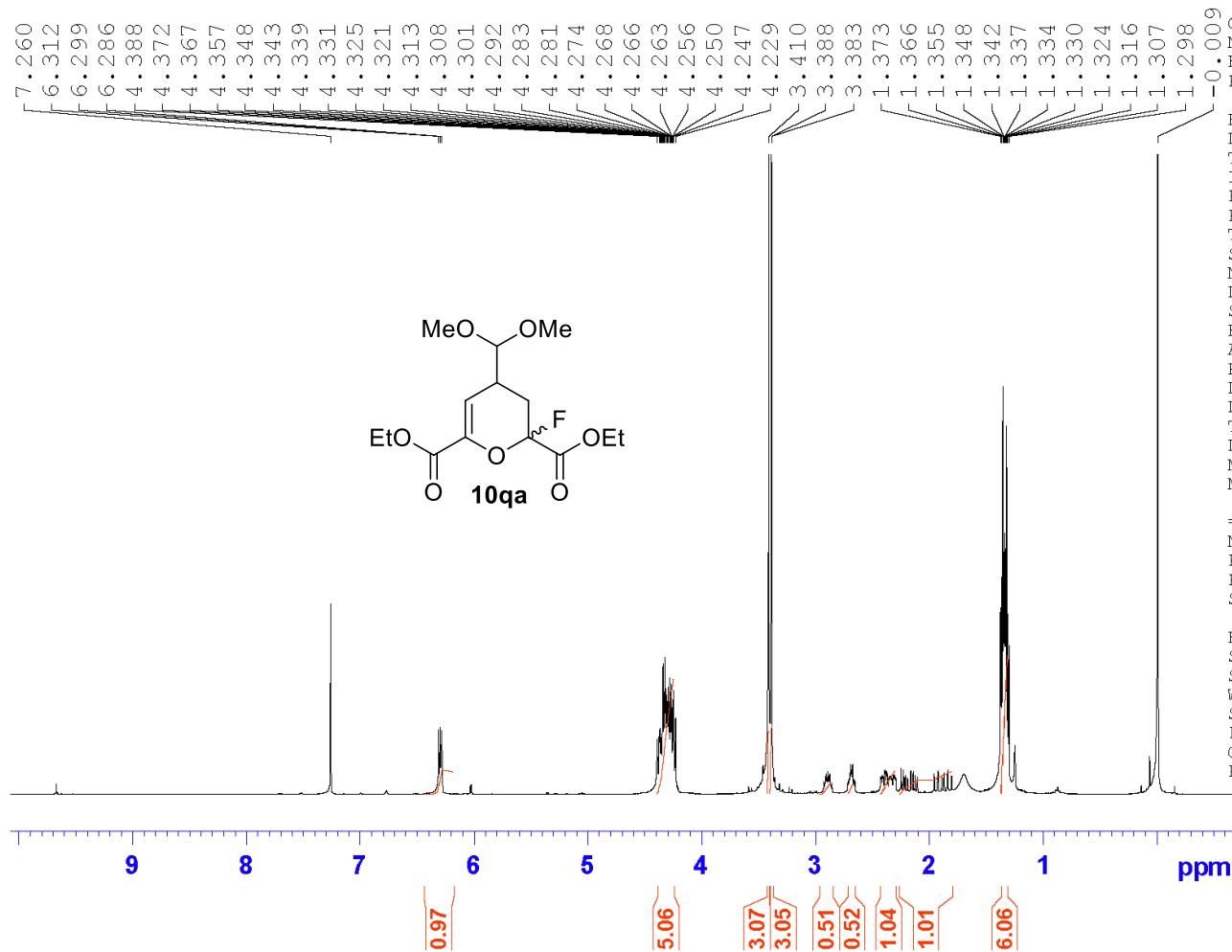
Current Data Parameters
 NAME Jun05-2013
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130605
 Time 23.24
 INSTRUM advance400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 5000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3632196 sec
 RG 4597.6
 DW 20.800 usec
 DE 6.00 usec
 TE 299.1 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -2.00 dB
 SFO1 100.6479773 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.70 dB
 PL12 17.10 dB
 PL13 17.10 dB
 SFO2 400.2316009 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6379107 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

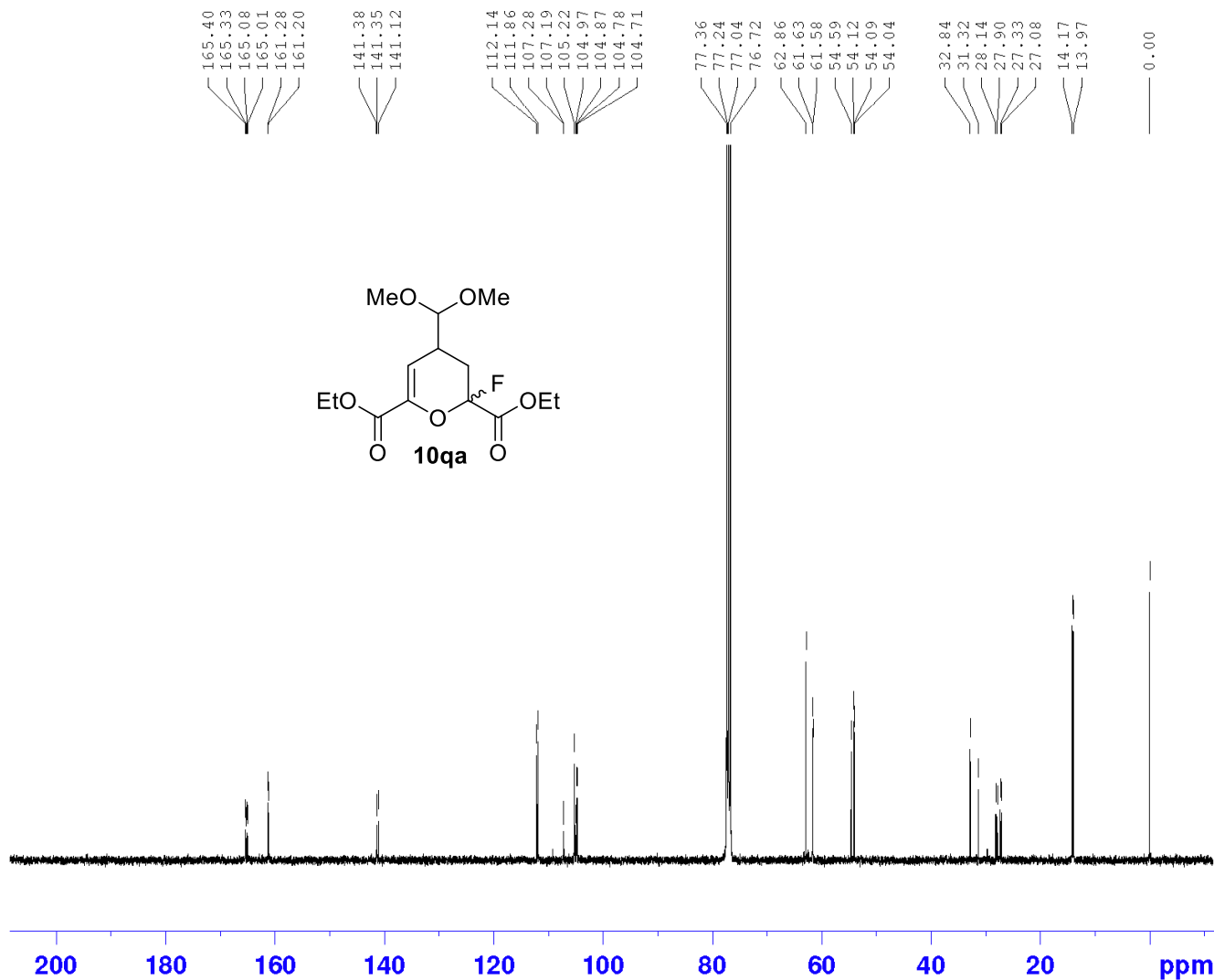


Current Data Parameters
NAME May21-2013
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130521
Time_ 18.21
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715421 sec
RG 181
DW 60.600 usec
DE 6.00 usec
TE 297.8 K
D1 1.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 2.70 dB
SFO1 400.2324716 MHz

F2 - Processing parameters
SI 32768
SF 400.2300052 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



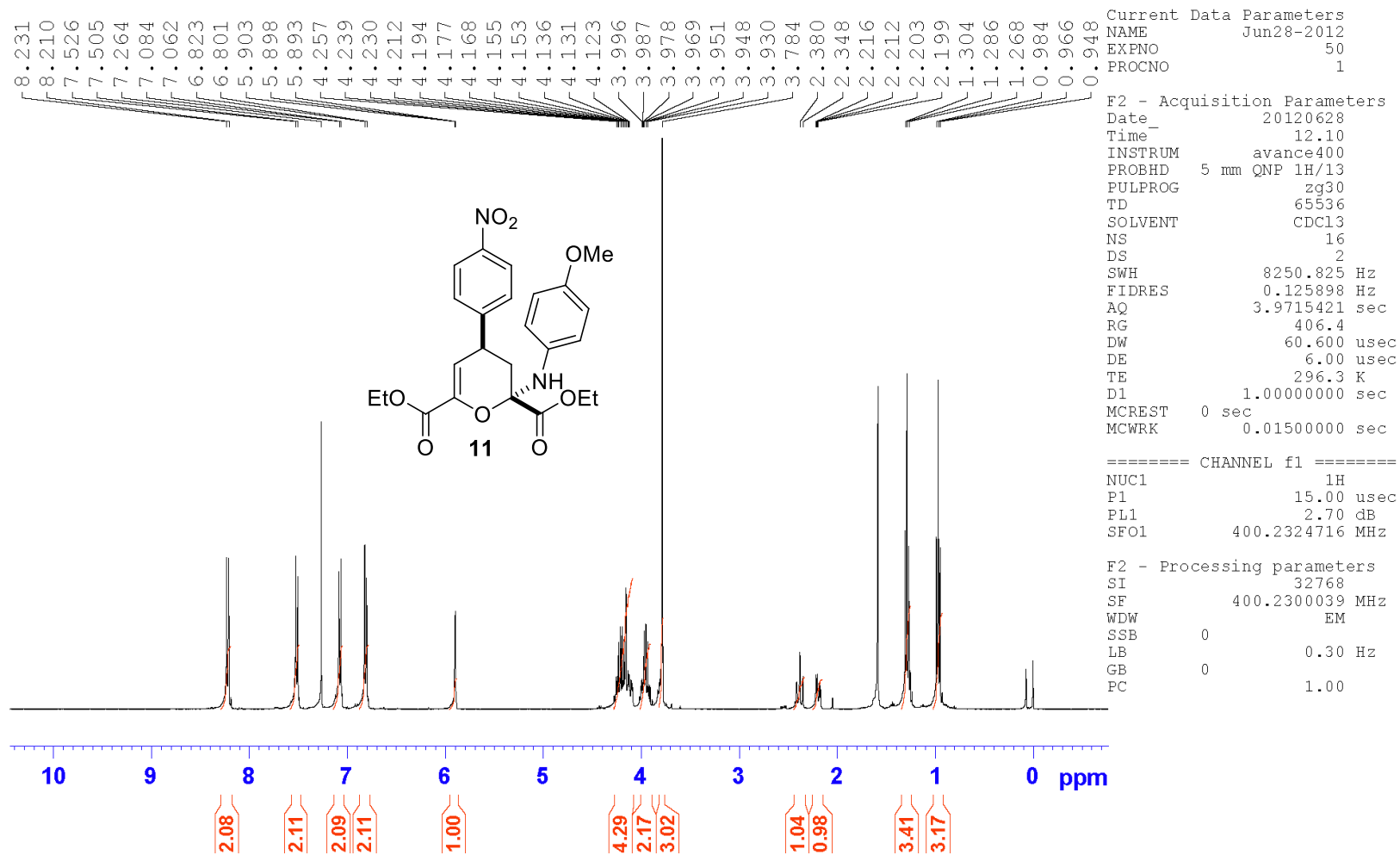
Current Data Parameters
NAME May21-2013
EXPNO 20
PROCNO 1

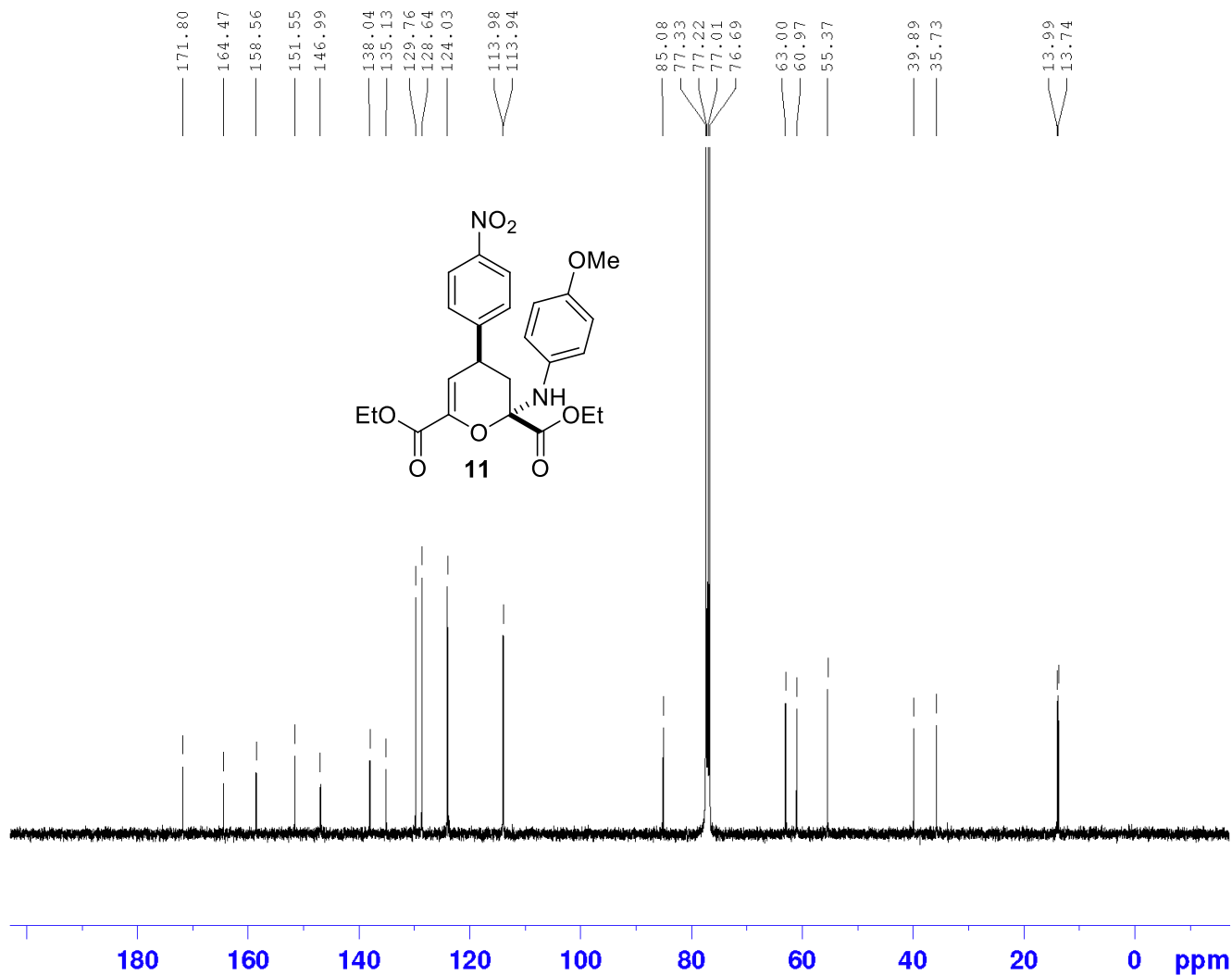
F2 - Acquisition Parameters
Date_ 20130522
Time 4.28
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 2896.3
DW 20.800 usec
DE 6.00 usec
TE 299.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -2.00 dB
SFO1 100.6479773 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.70 dB
PL12 17.10 dB
PL13 17.10 dB
SFO2 400.2316009 MHz

F2 - Processing parameters
SI 32768
SF 100.6379110 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





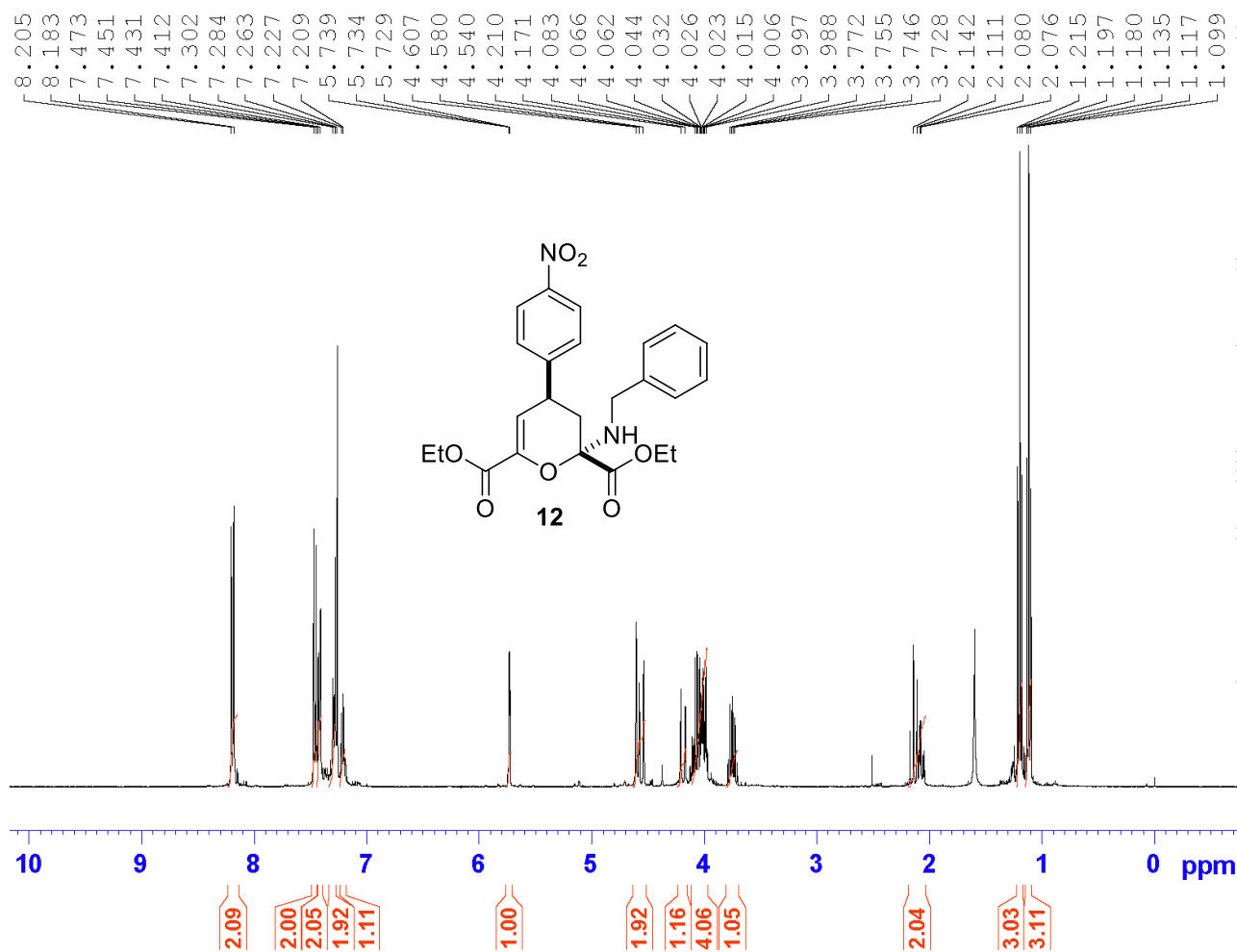
Current Data Parameters
NAME Jun28-2012
EXPNO 70
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120629
Time 1.49
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 1149.4
DW 20.800 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -2.00 dB
SFO1 100.6479773 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.70 dB
PL12 17.10 dB
PL13 17.10 dB
SFO2 400.2316009 MHz

F2 - Processing parameters
SI 32768
SF 100.6379140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

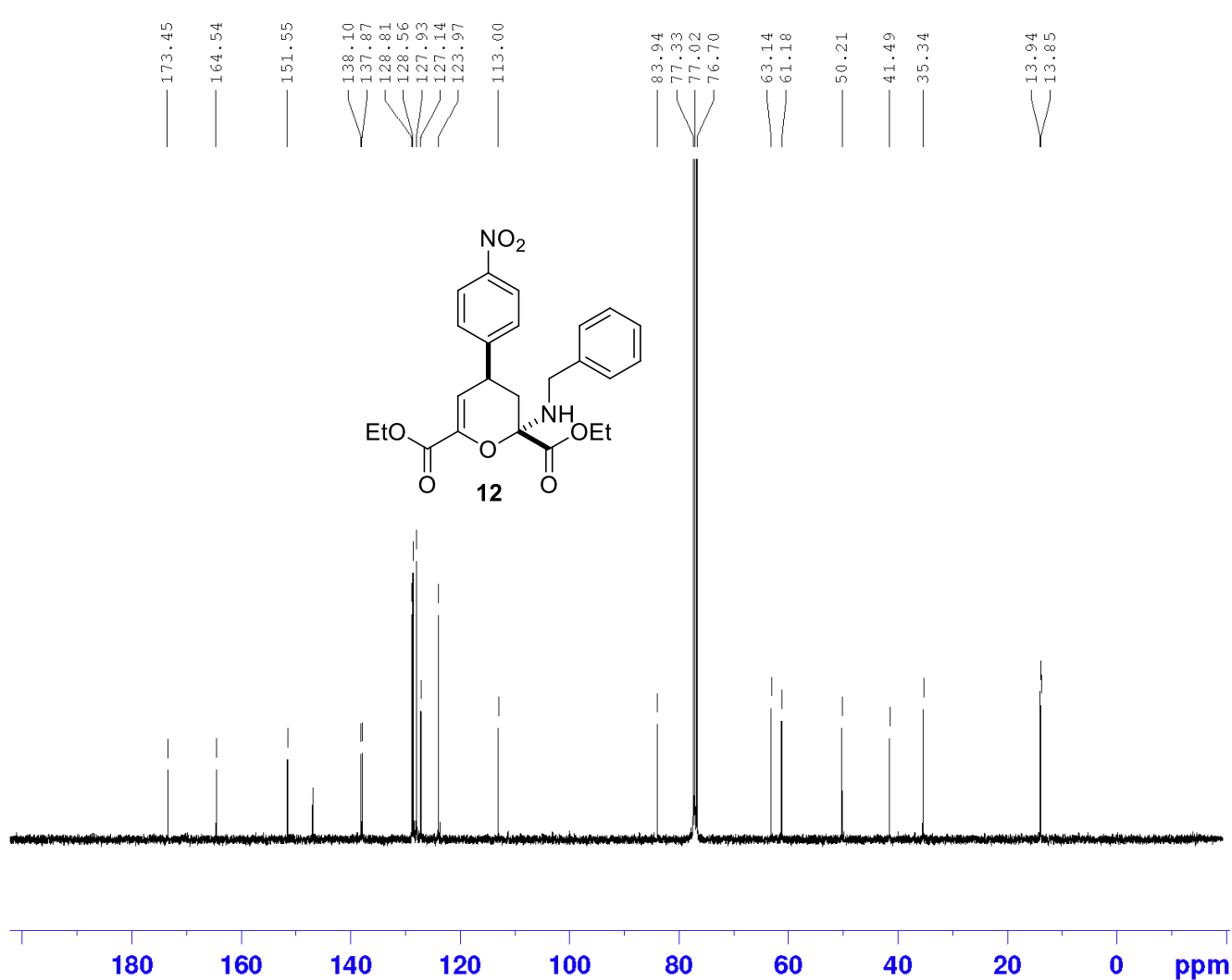


Current Data Parameters
NAME Jul26-2012
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120726
Time 12.15
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715421 sec
RG 287.4
DW 60.600 usec
DE 6.00 usec
TE 296.8 K
D1 1.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 2.70 dB
SFO1 400.2324716 MHz

F2 - Processing parameters
SI 32768
SF 400.2300044 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



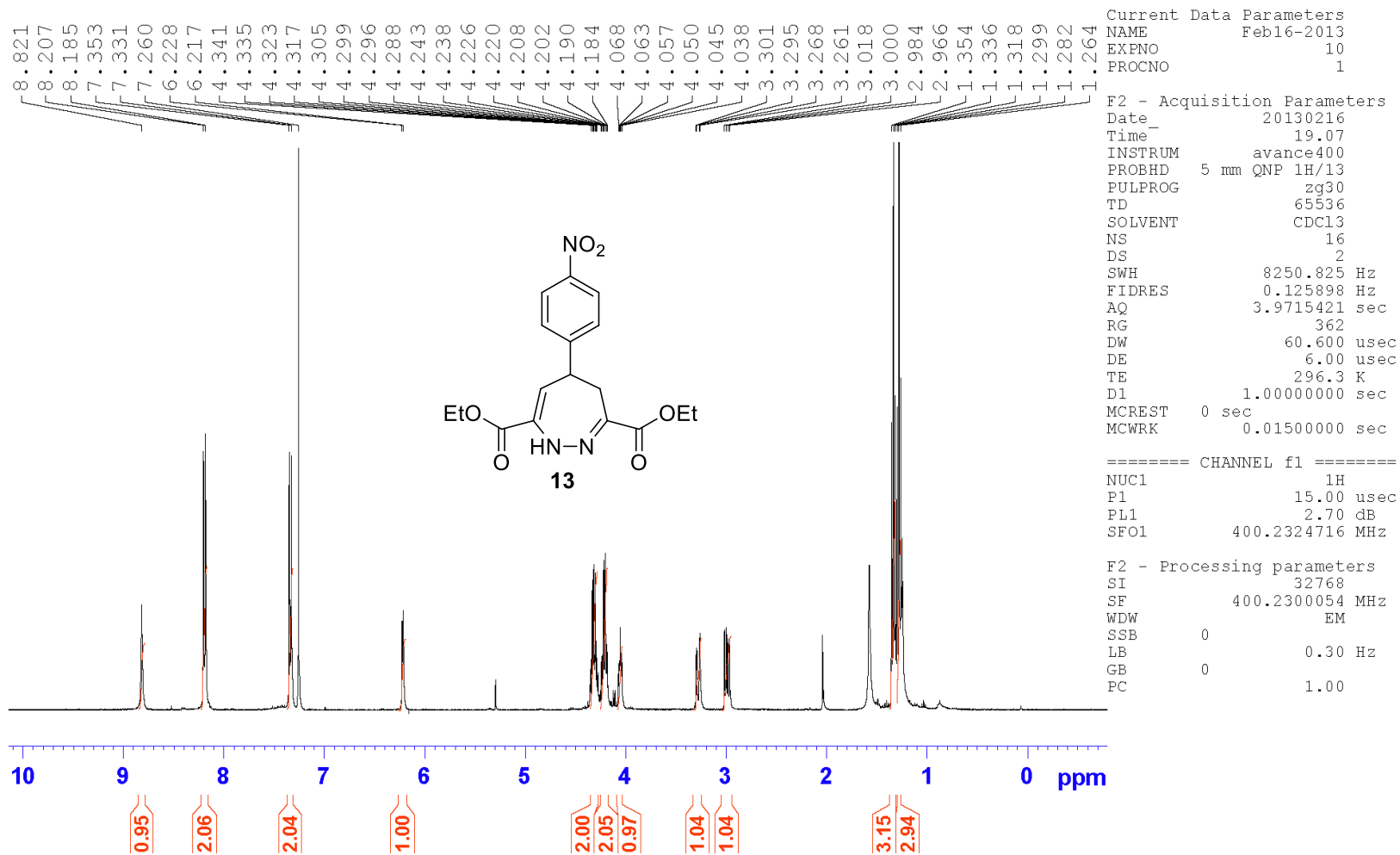
Current Data Parameters
NAME Jul26-2012
EXPNO 20
PROCNO 1

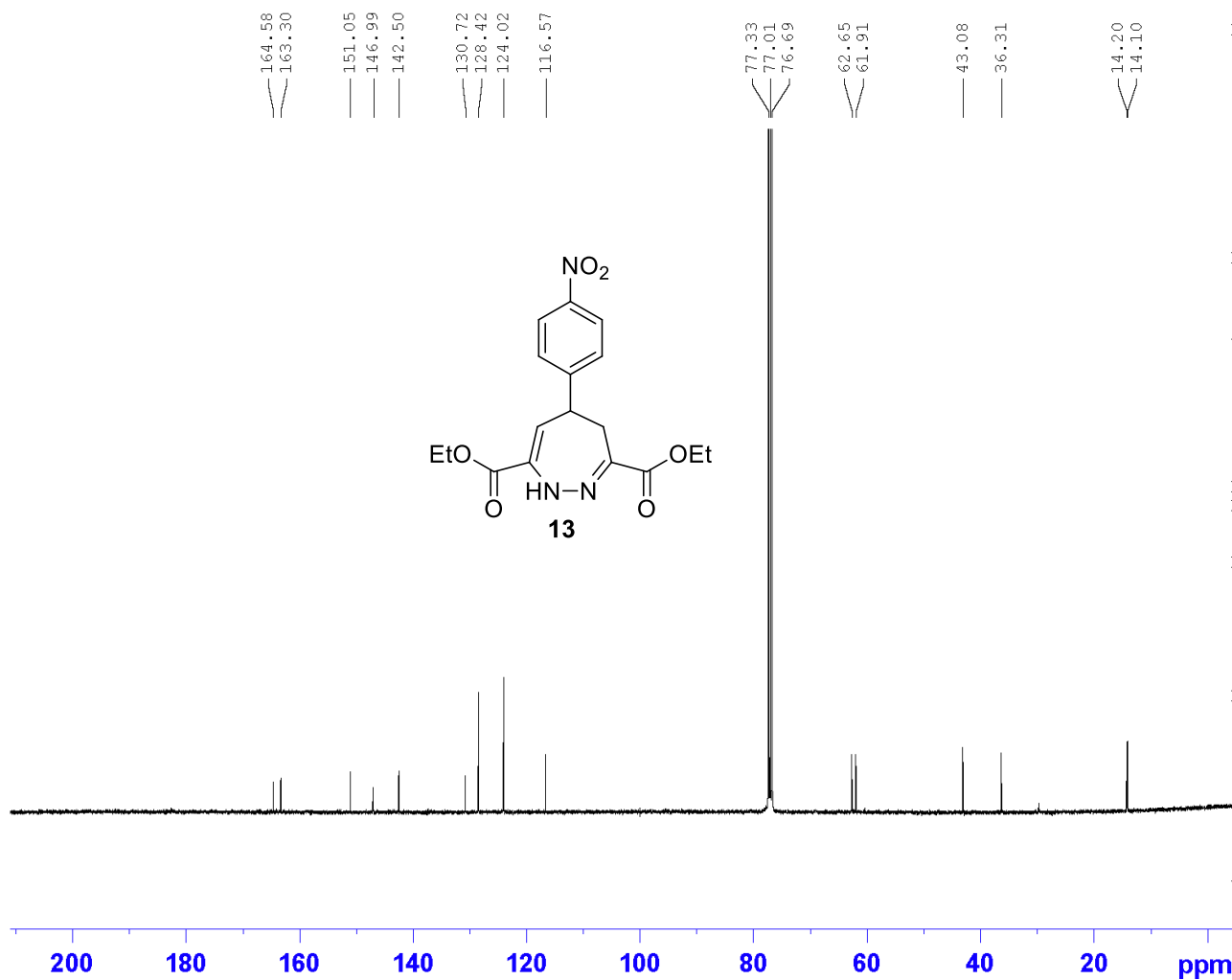
F2 - Acquisition Parameters
Date_ 20120726
Time 14.04
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1600
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 143.7
DW 20.800 usec
DE 6.00 usec
TE 298.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

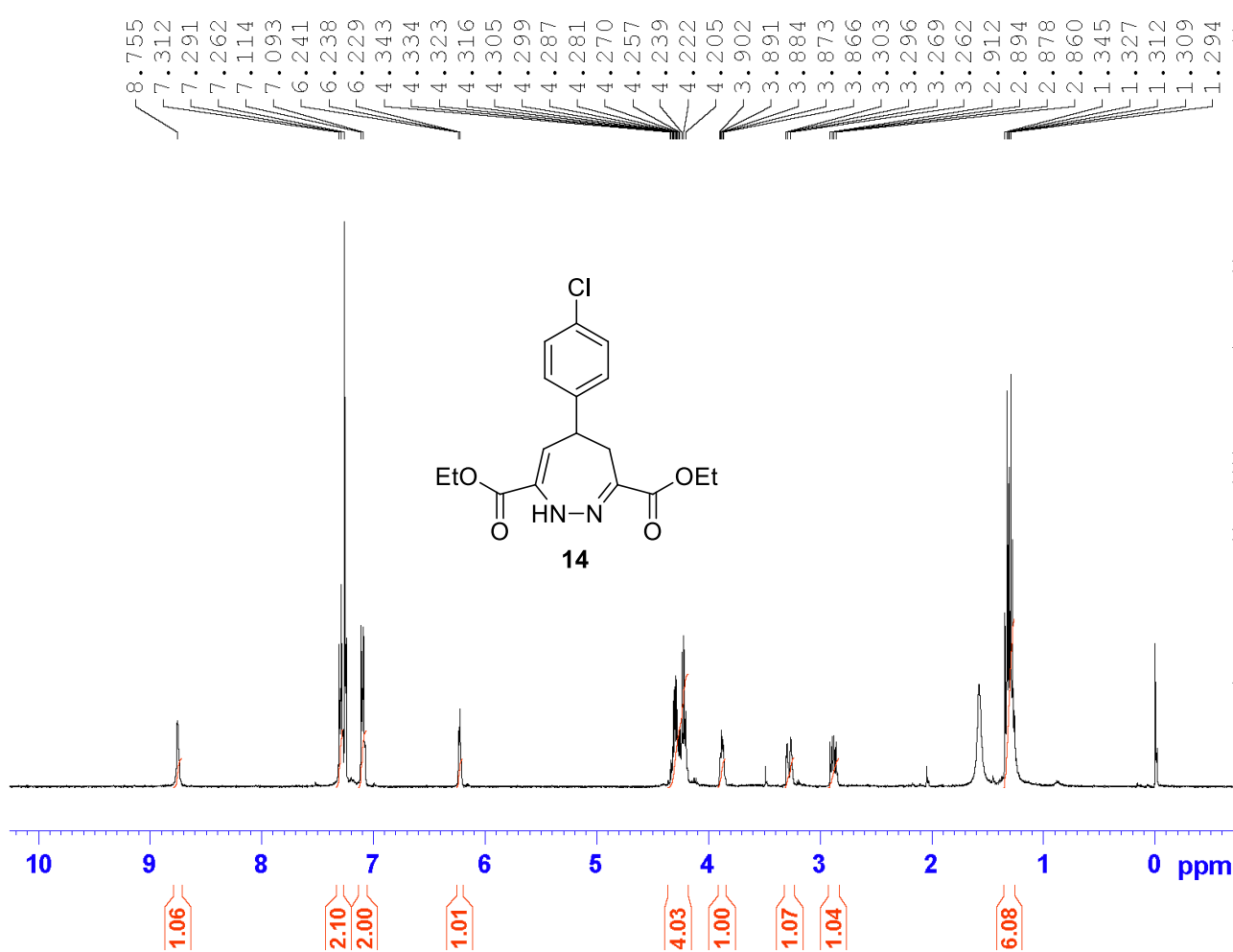
===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -2.00 dB
SFO1 100.6479773 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.70 dB
PL12 17.10 dB
PL13 17.10 dB
SFO2 400.2316009 MHz

F2 - Processing parameters
SI 32768
SF 100.6379140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





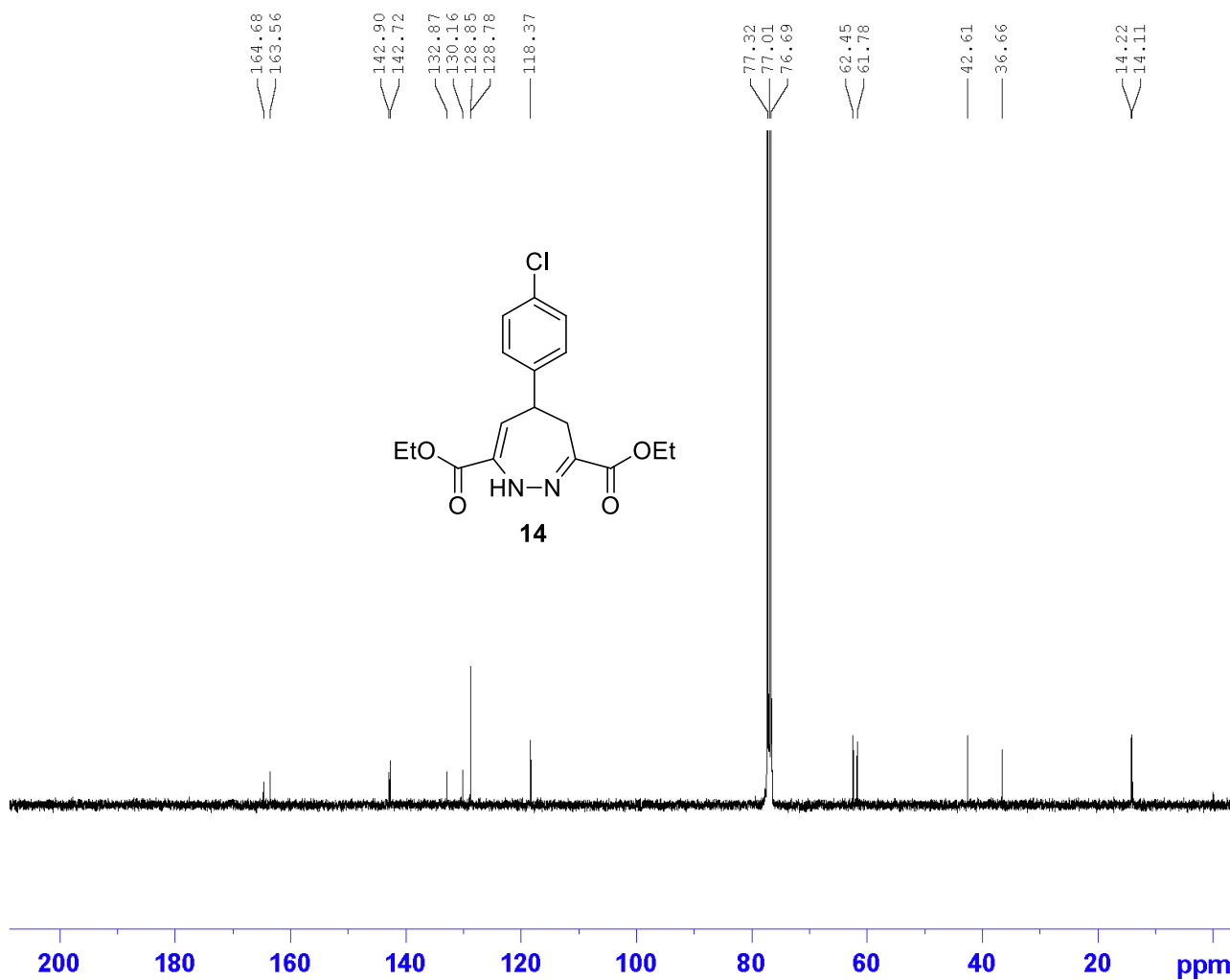


Current Data Parameters
NAME Mar14-2013
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130314
Time 18.03
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715421 sec
RG 512
DW 60.600 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 2.70 dB
SFO1 400.2324716 MHz

F2 - Processing parameters
SI 32768
SF 400.2300052 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



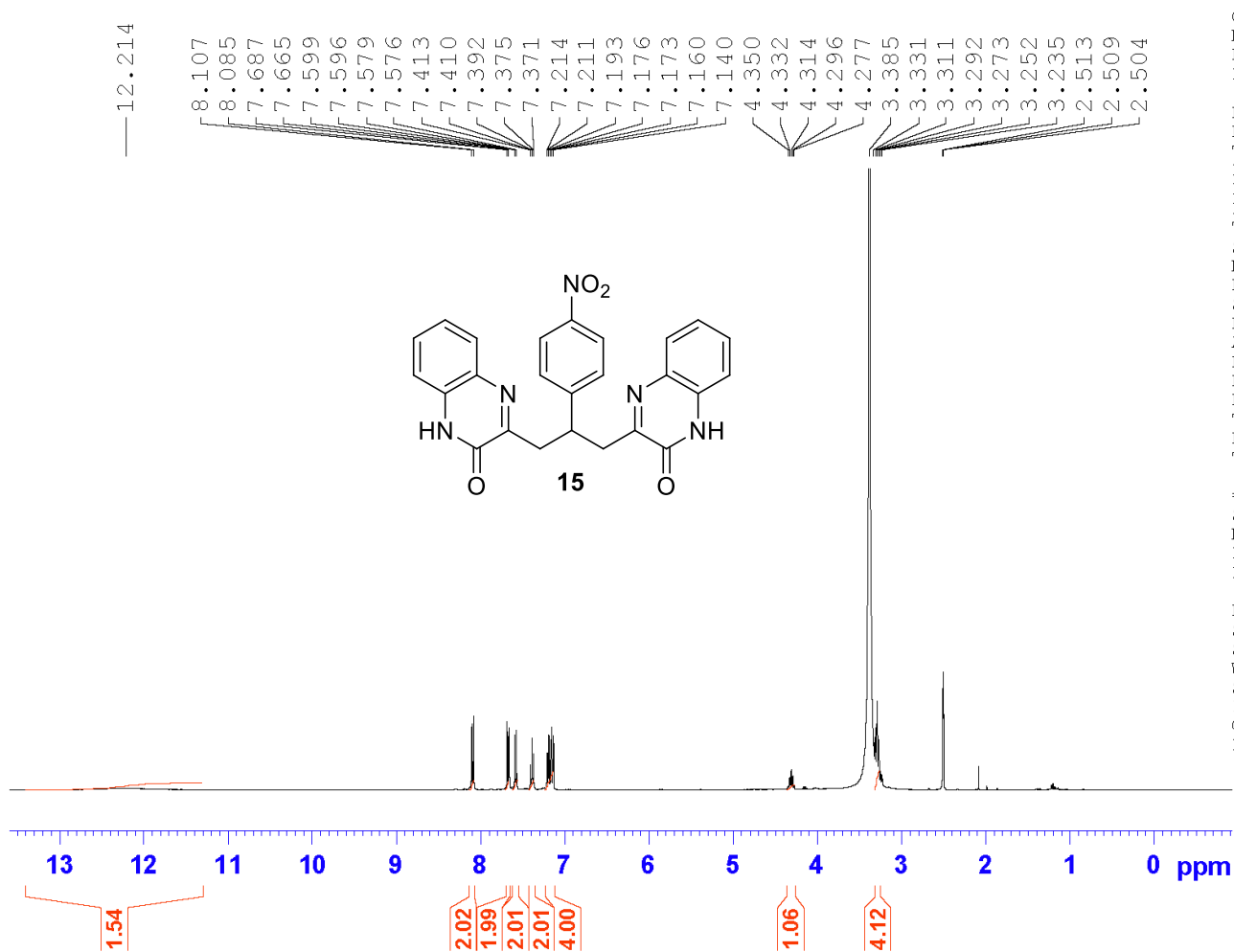
Current Data Parameters
NAME Mar14-2013
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130315
Time_ 1.39
INSTRUM avance400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 7000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631696 sec
RG 812.7
DW 20.800 usec
DE 6.00 usec
TE 296.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -2.00 dB
SFO1 100.6479773 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.70 dB
PL12 17.10 dB
PL13 17.10 dB
SFO2 400.2316009 MHz

F2 - Processing parameters
SI 32768
SF 100.6379140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

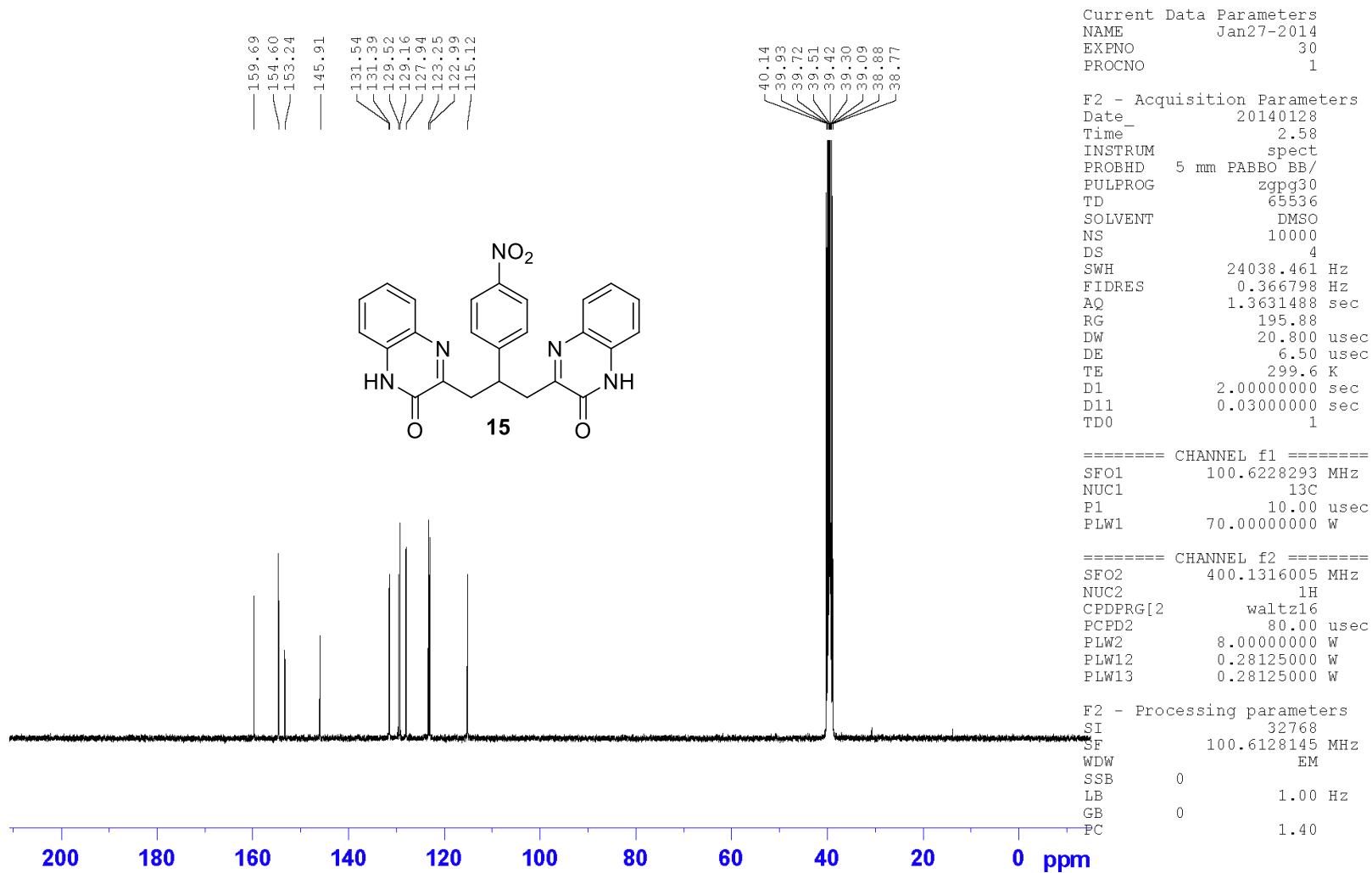


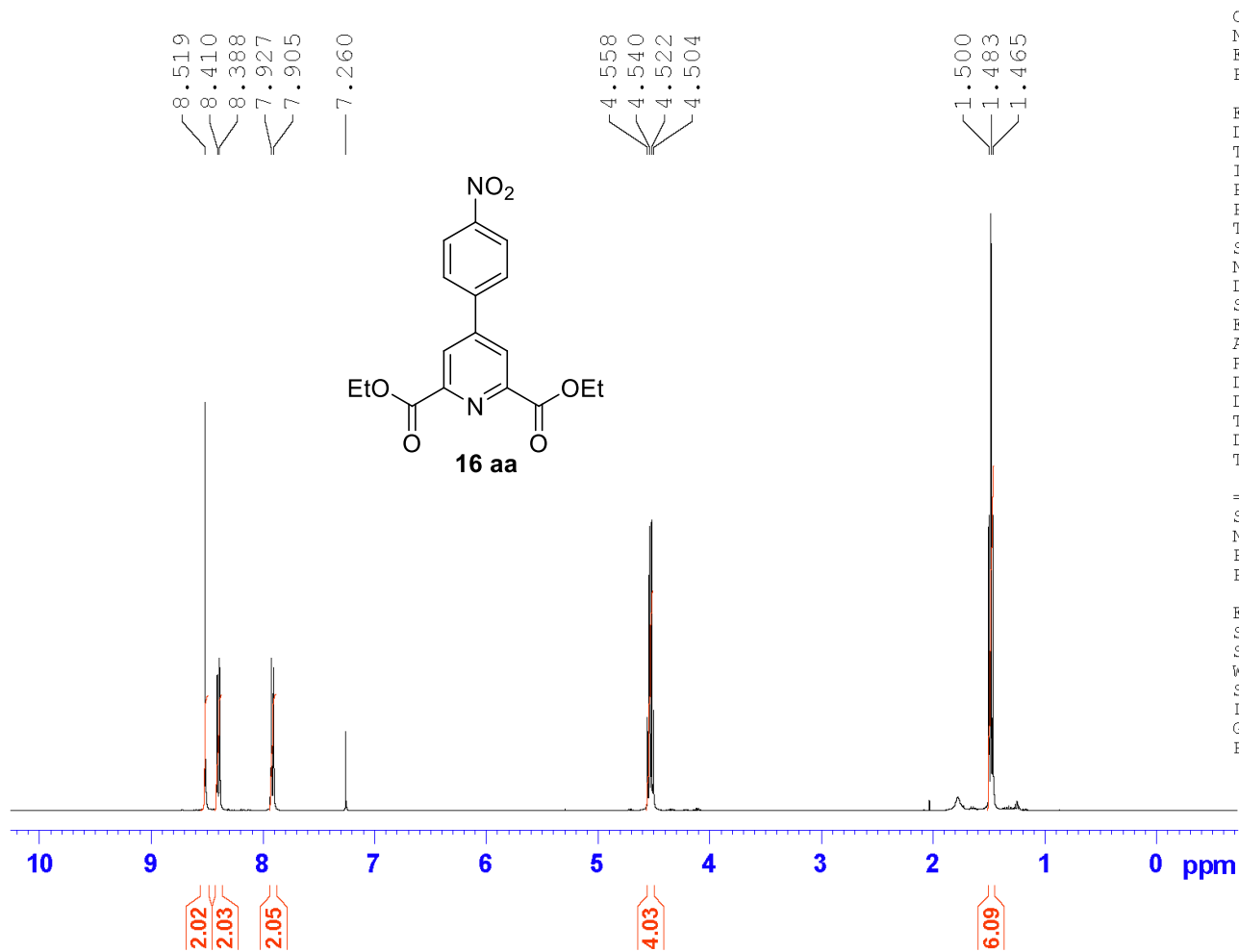
Current Data Parameters
NAME Jan27-2014
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140127
Time_ 17.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
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 298.2 K
D1 1.00000000 sec
TD0 1

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

F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



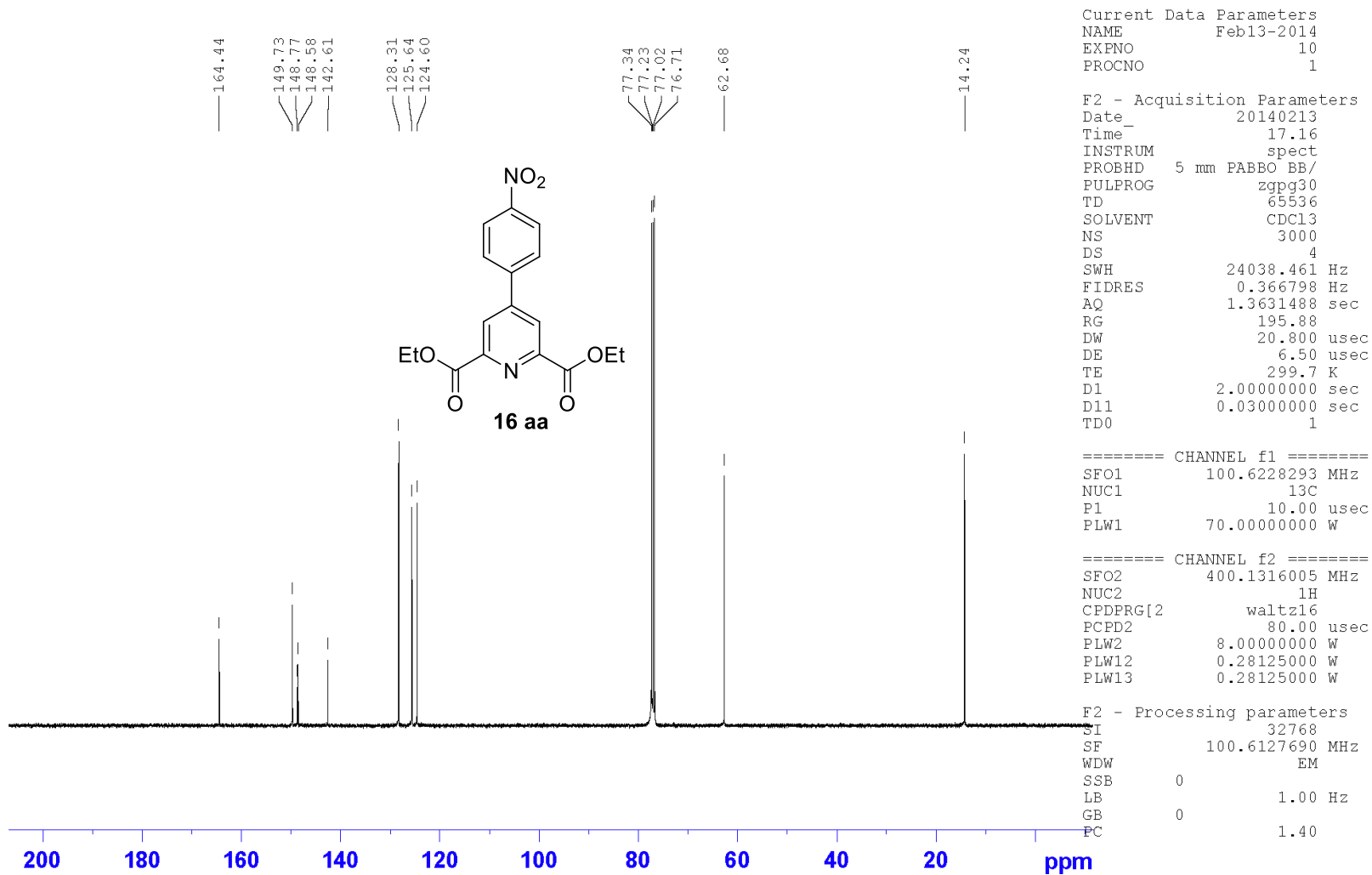


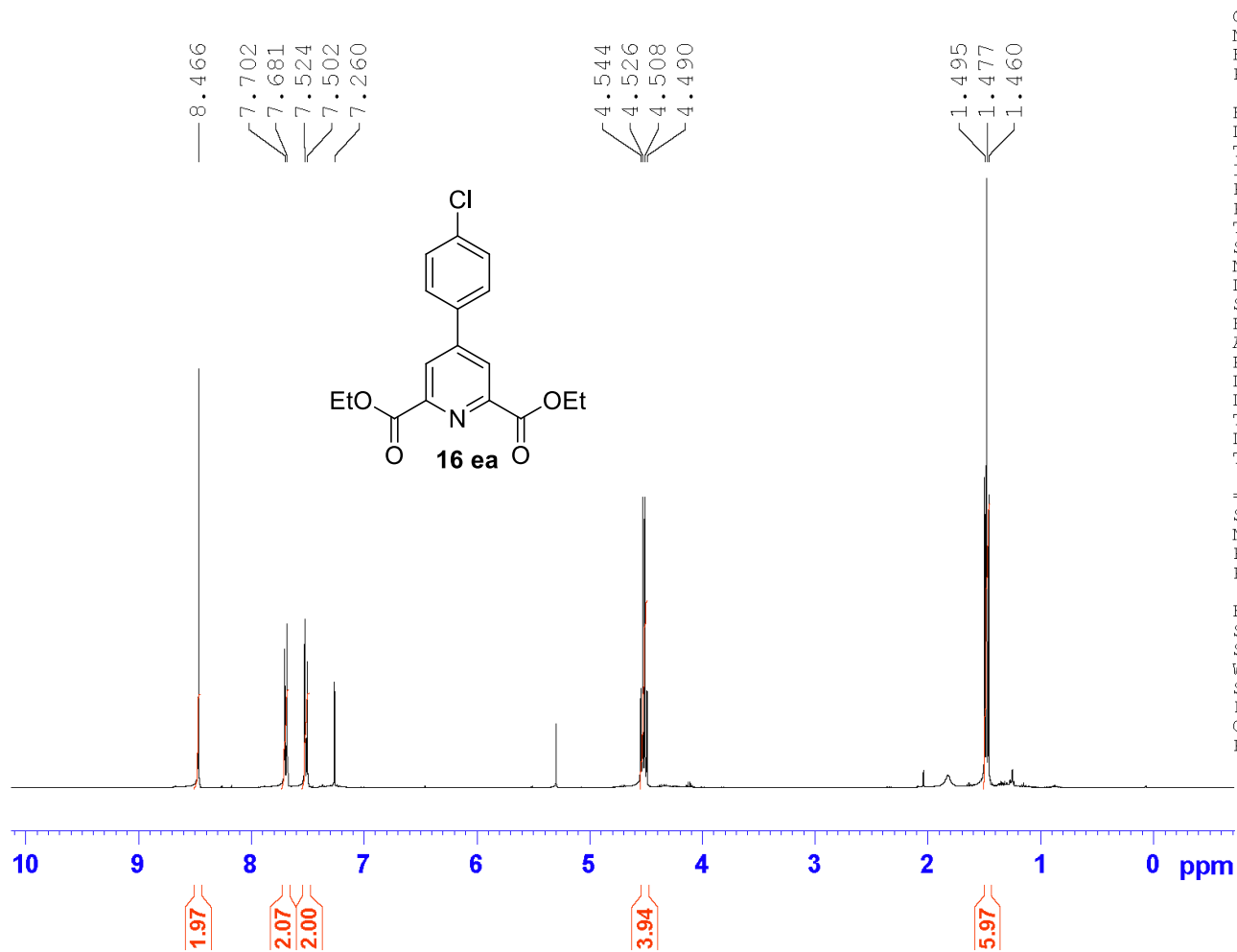
Current Data Parameters
 NAME Feb13-2014-PC
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140213
 Time_ 17.52
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 62.88
 DW 62.400 usec
 DE 6.50 usec
 TE 298.6 K
 D1 1.00000000 sec
 TD0 1

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

F2 - Processing parameters
 SI 65536
 SF 400.1300100 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



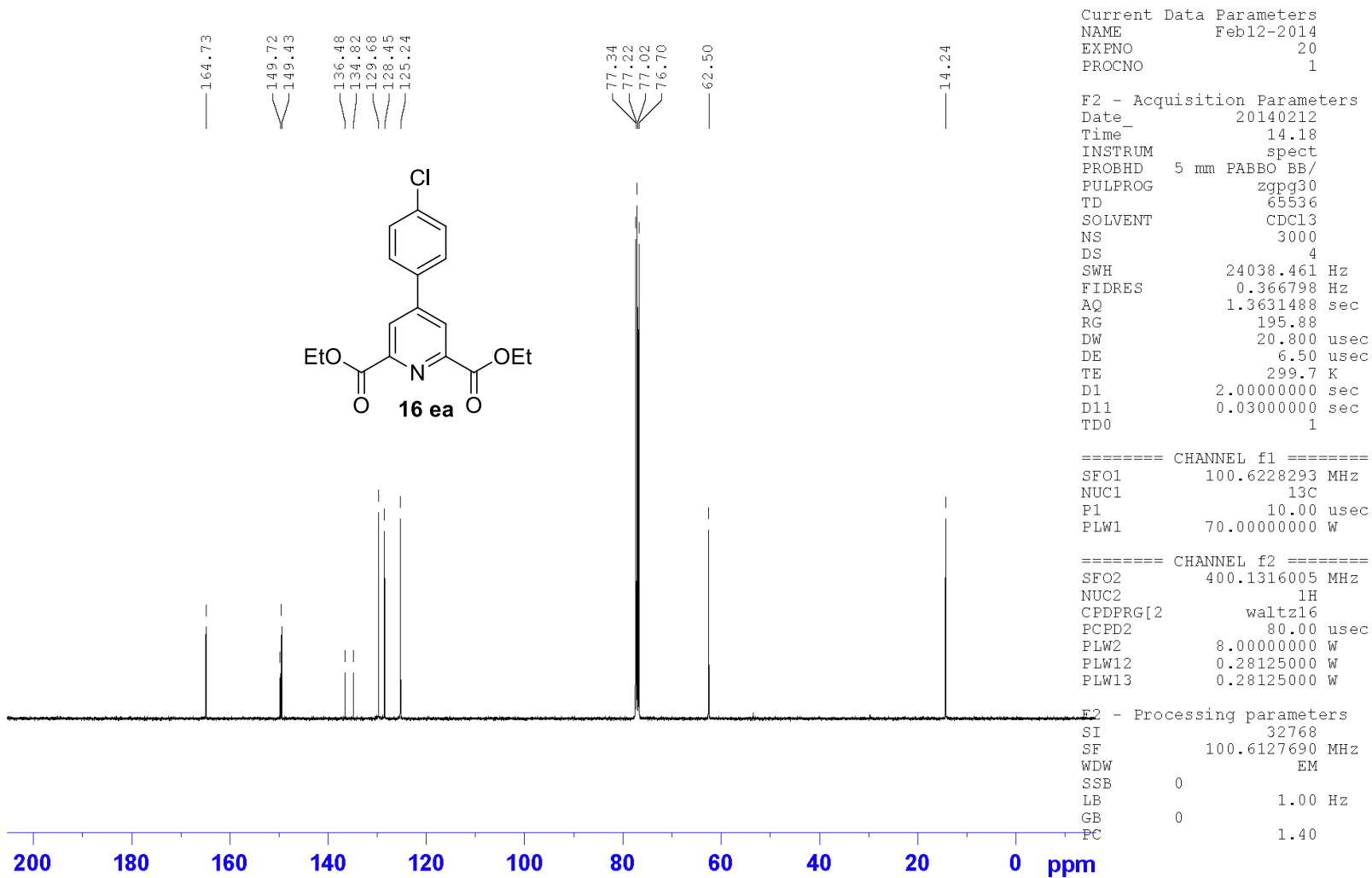


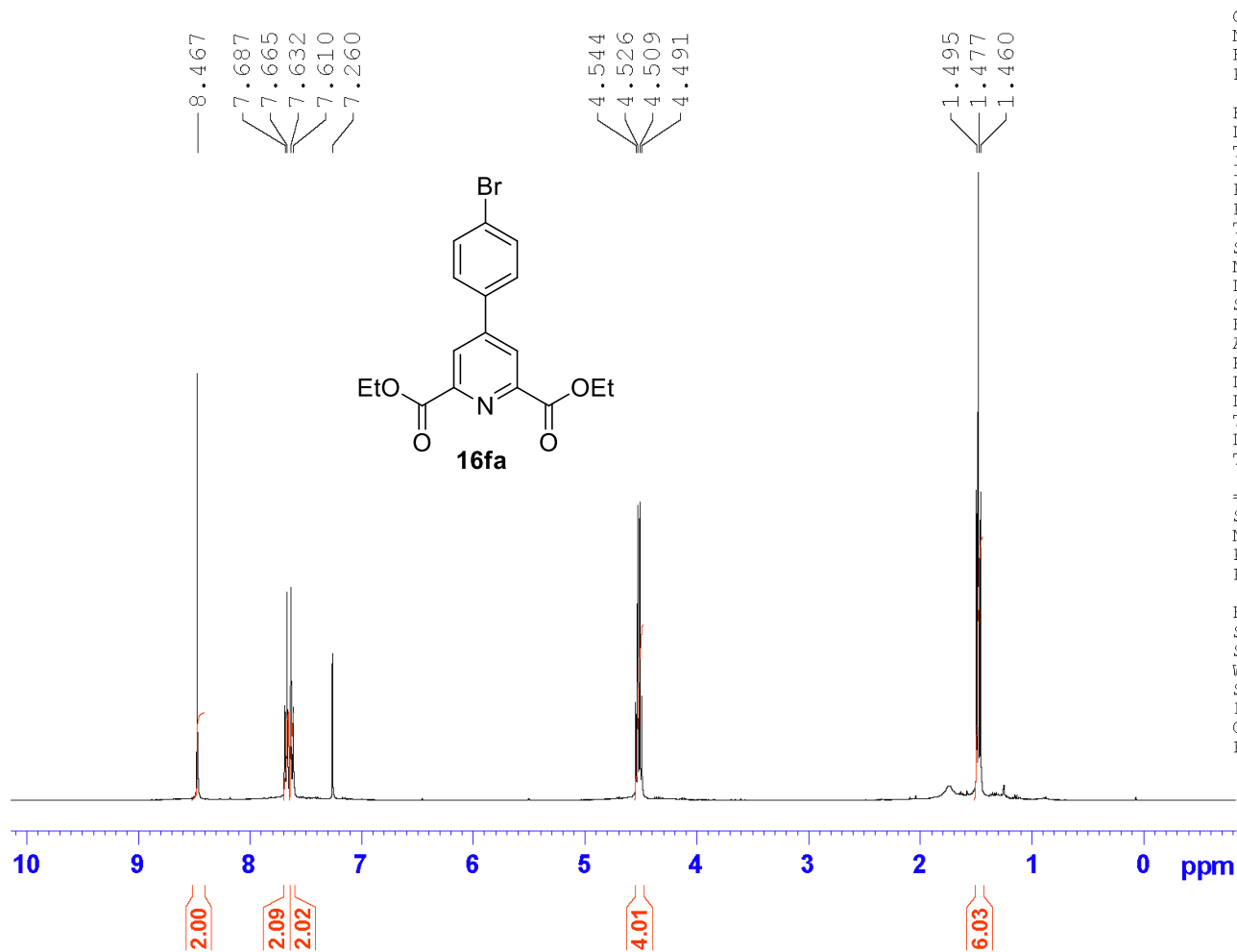
Current Data Parameters
 NAME Feb12-2014
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140212
 Time_ 11.22
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 62.88
 DW 62.400 usec
 DE 6.50 usec
 TE 298.4 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



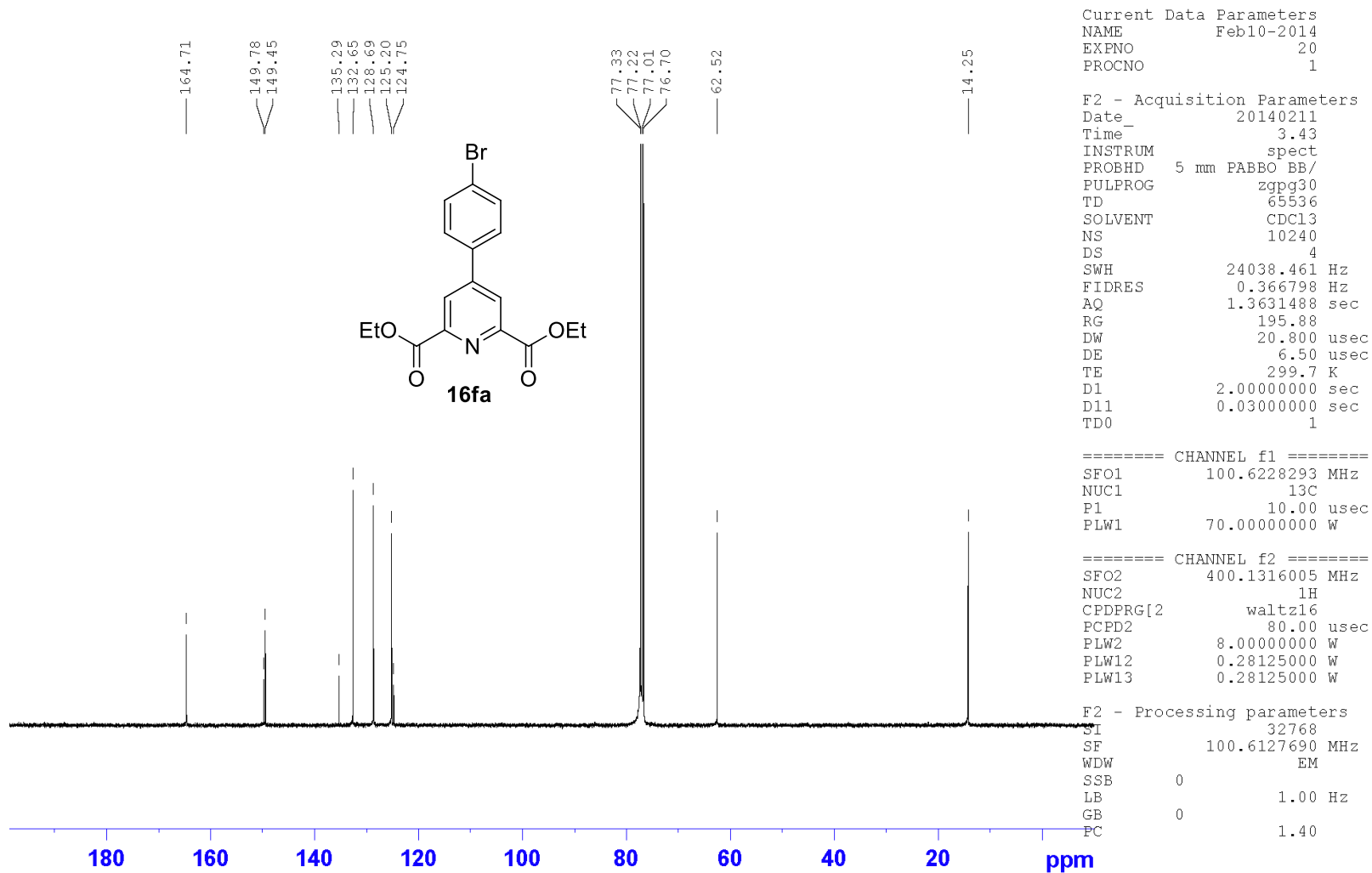


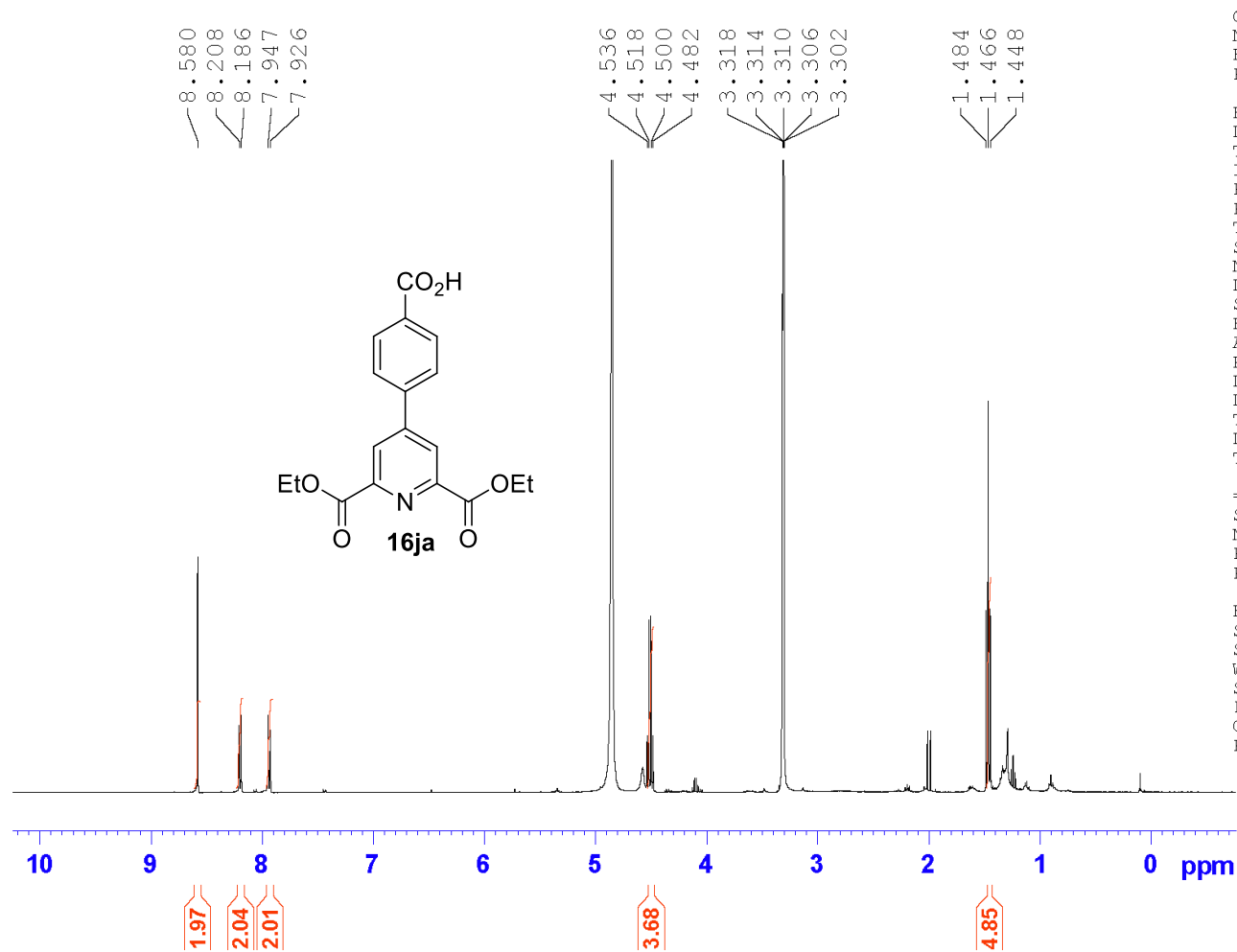
Current Data Parameters
 NAME Feb10-2014
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140210
 Time_ 17.55
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 71.01
 DW 62.400 usec
 DE 6.50 usec
 TE 298.4 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 400.1300098 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



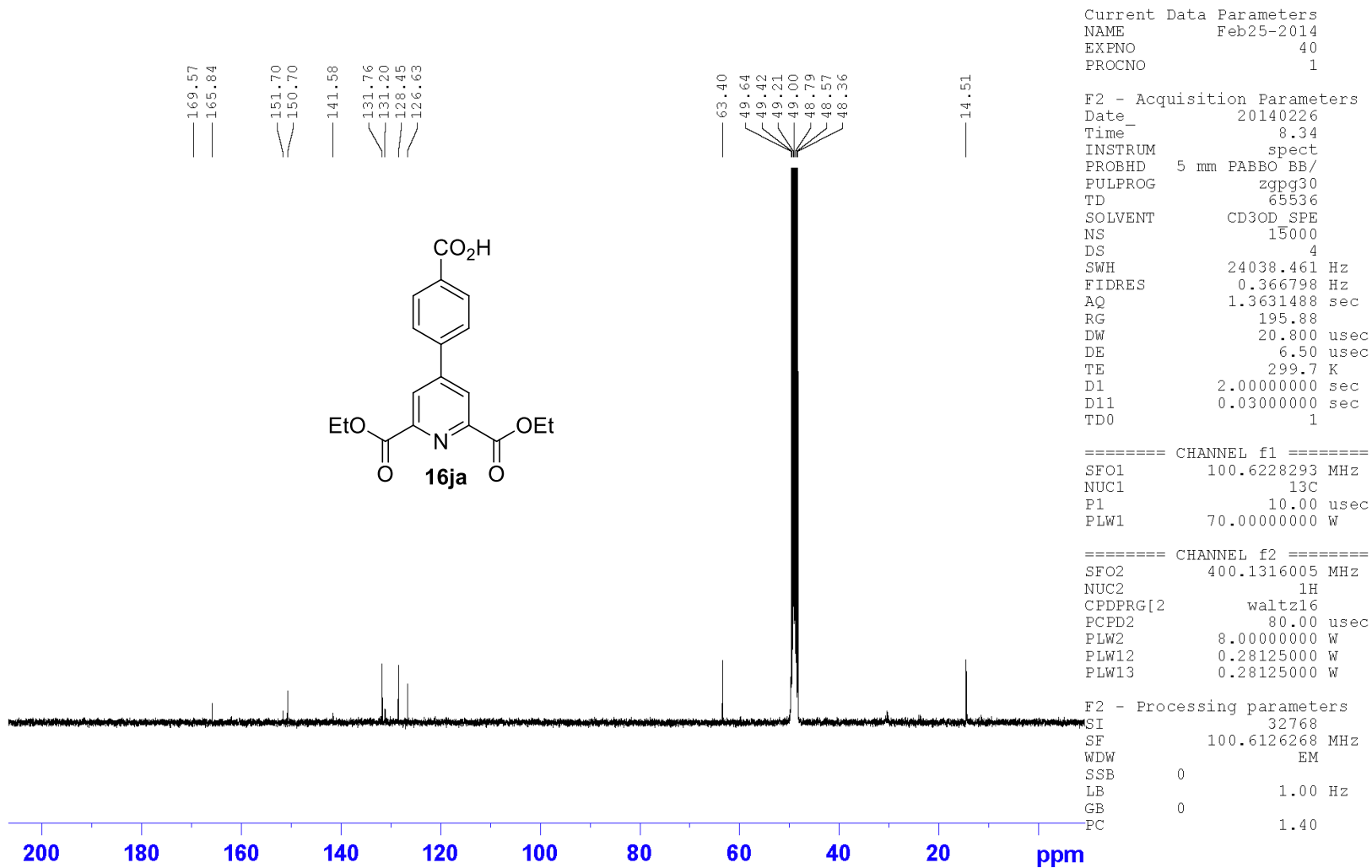


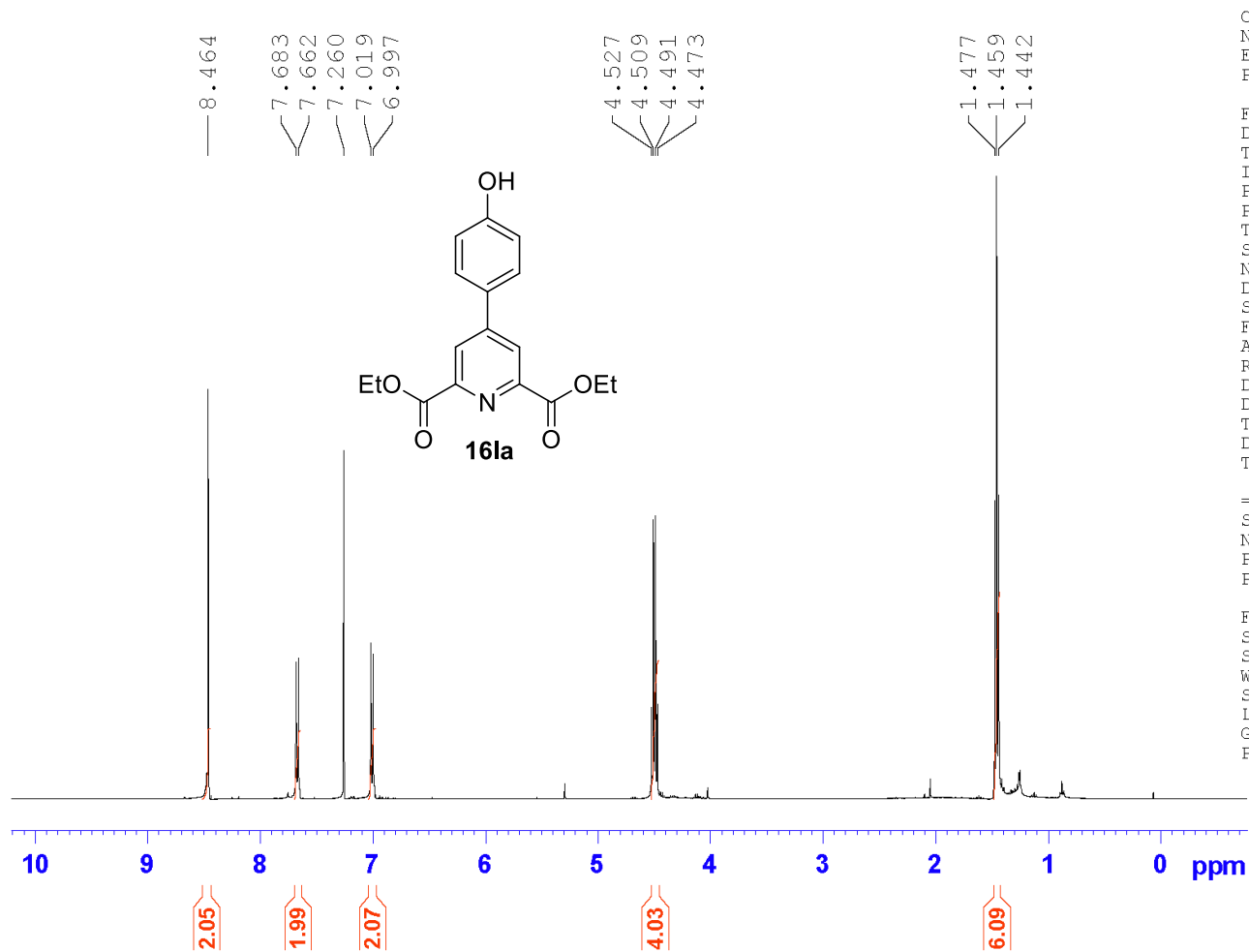
Current Data Parameters
 NAME Feb26-2014
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140226
 Time_ 11.07
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CD3OD_SPE
 NS 100
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 77.81
 DW 62.400 usec
 DE 6.50 usec
 TE 298.9 K
 D1 1.00000000 sec
 TD0 1

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

F2 - Processing parameters
 SI 65536
 SF 400.1300077 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



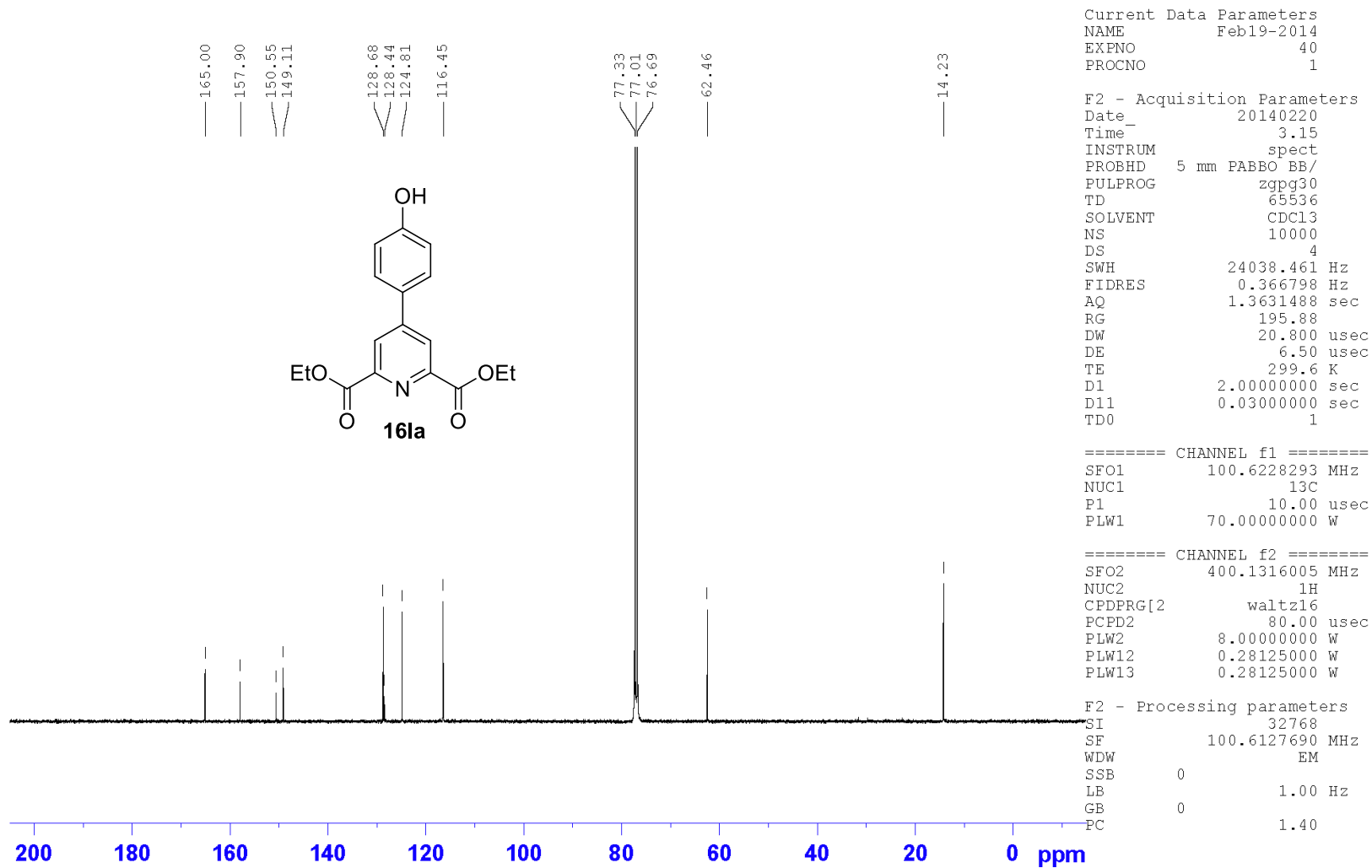


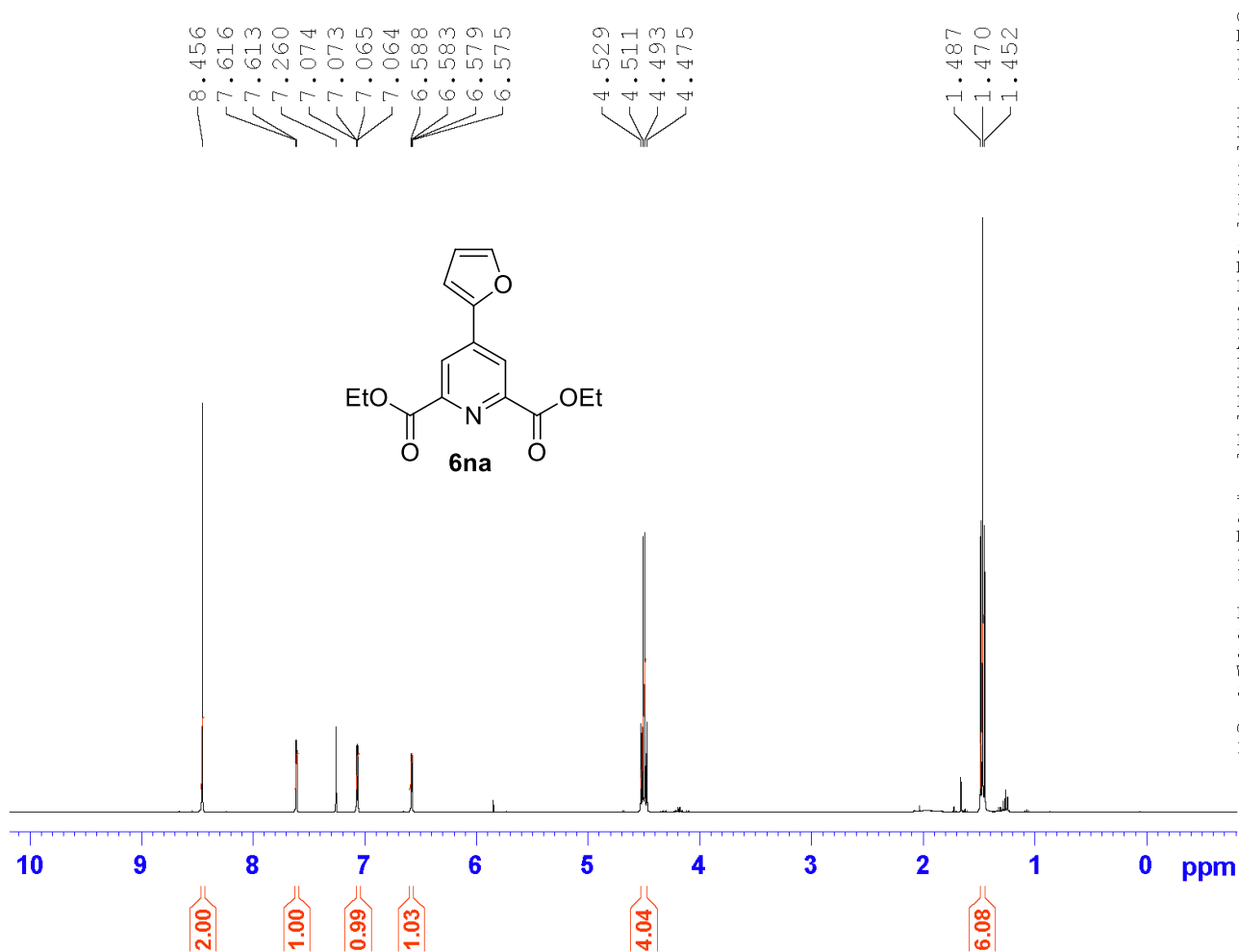
Current Data Parameters
 NAME Feb19-2014
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140219
 Time_ 17.41
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 77.81
 DW 62.400 usec
 DE 6.50 usec
 TE 298.3 K
 D1 1.00000000 sec
 TD0 1

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

F2 - Processing parameters
 SI 65536
 SF 400.1300099 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



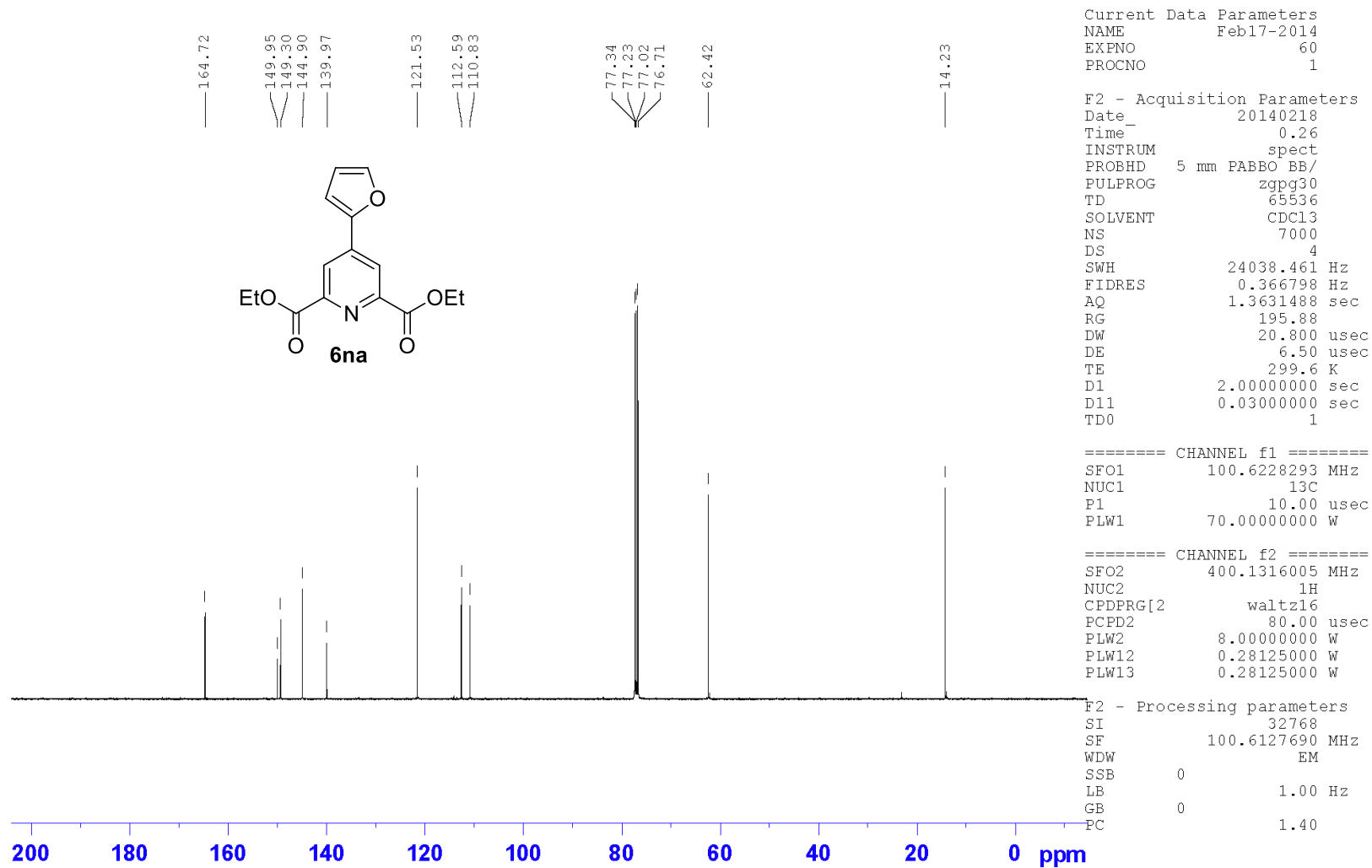


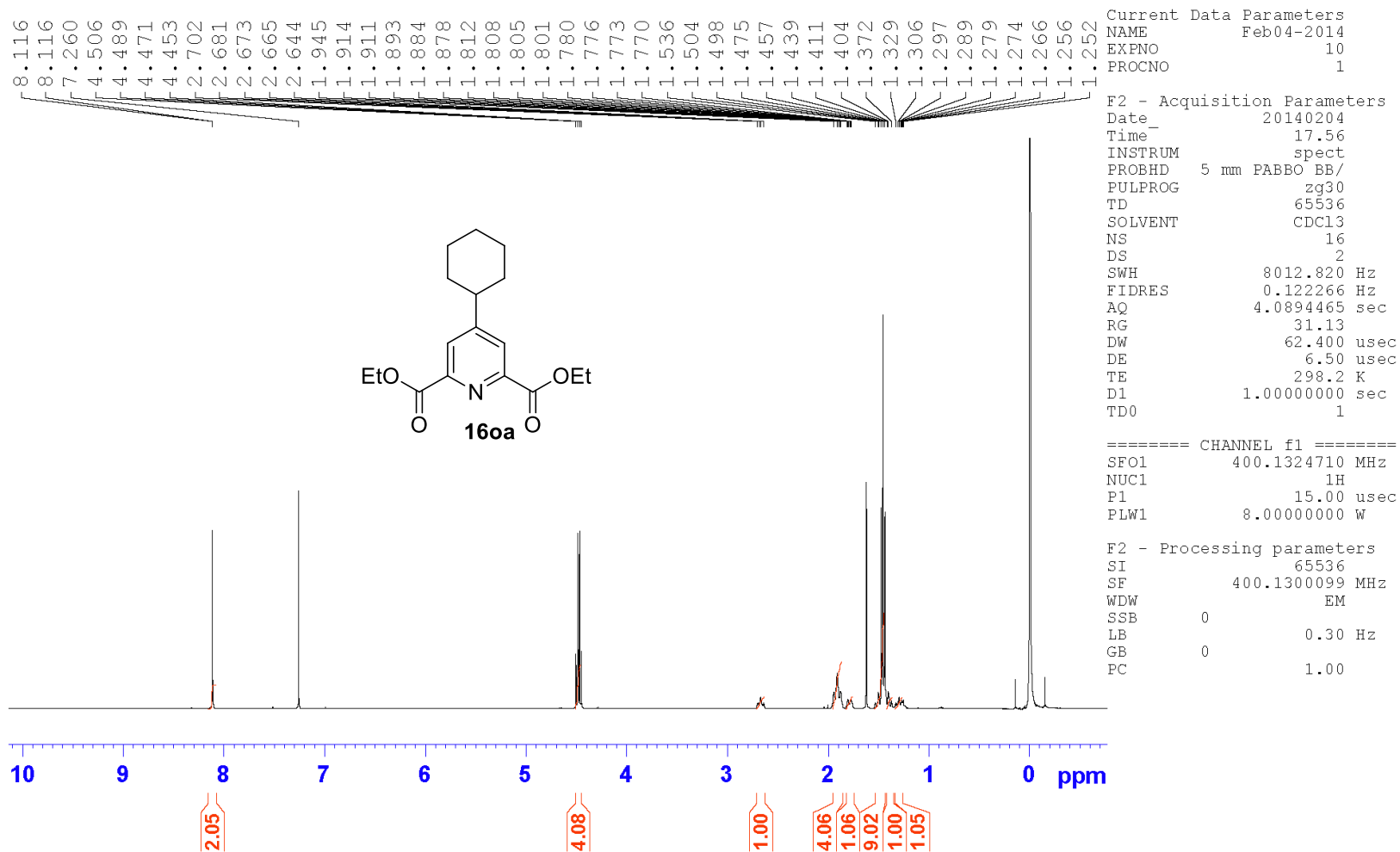
Current Data Parameters
 NAME Feb17-2014
 EXPNO 50
 PROCNO 1

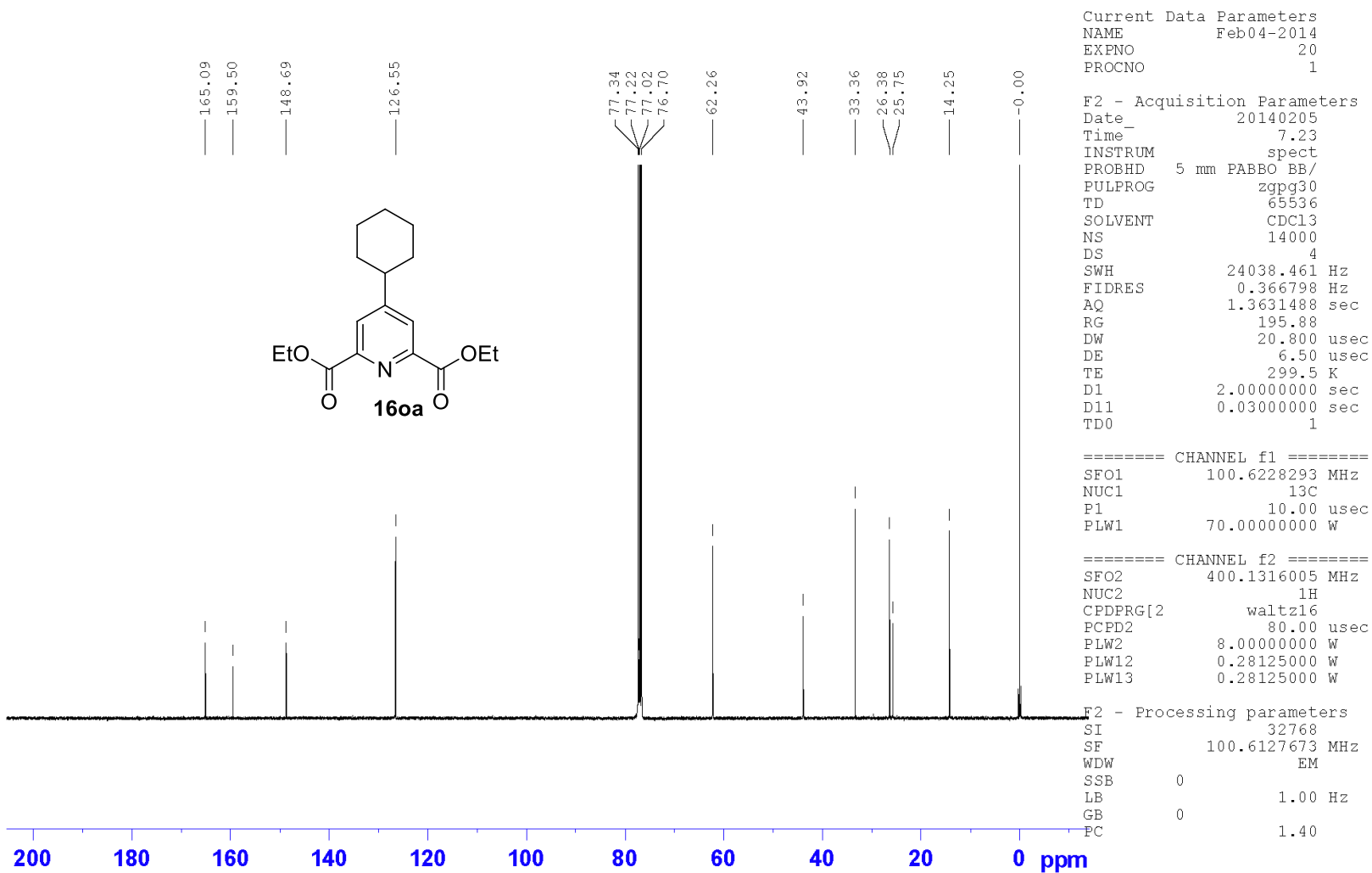
F2 - Acquisition Parameters
 Date_ 20140217
 Time 17.39
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 54.59
 DW 62.400 usec
 DE 6.50 usec
 TE 298.3 K
 D1 1.00000000 sec
 TD0 1

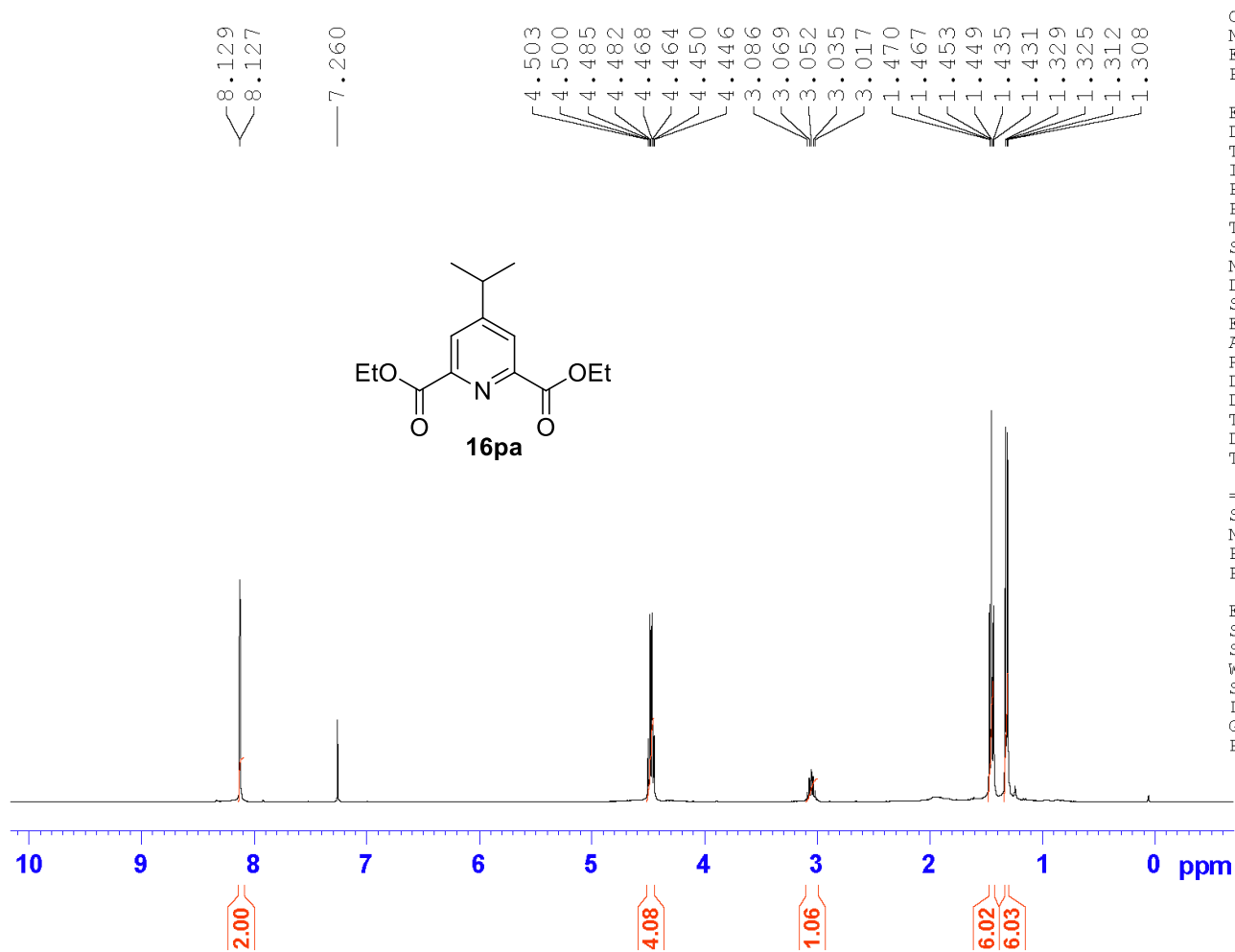
===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300099 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00







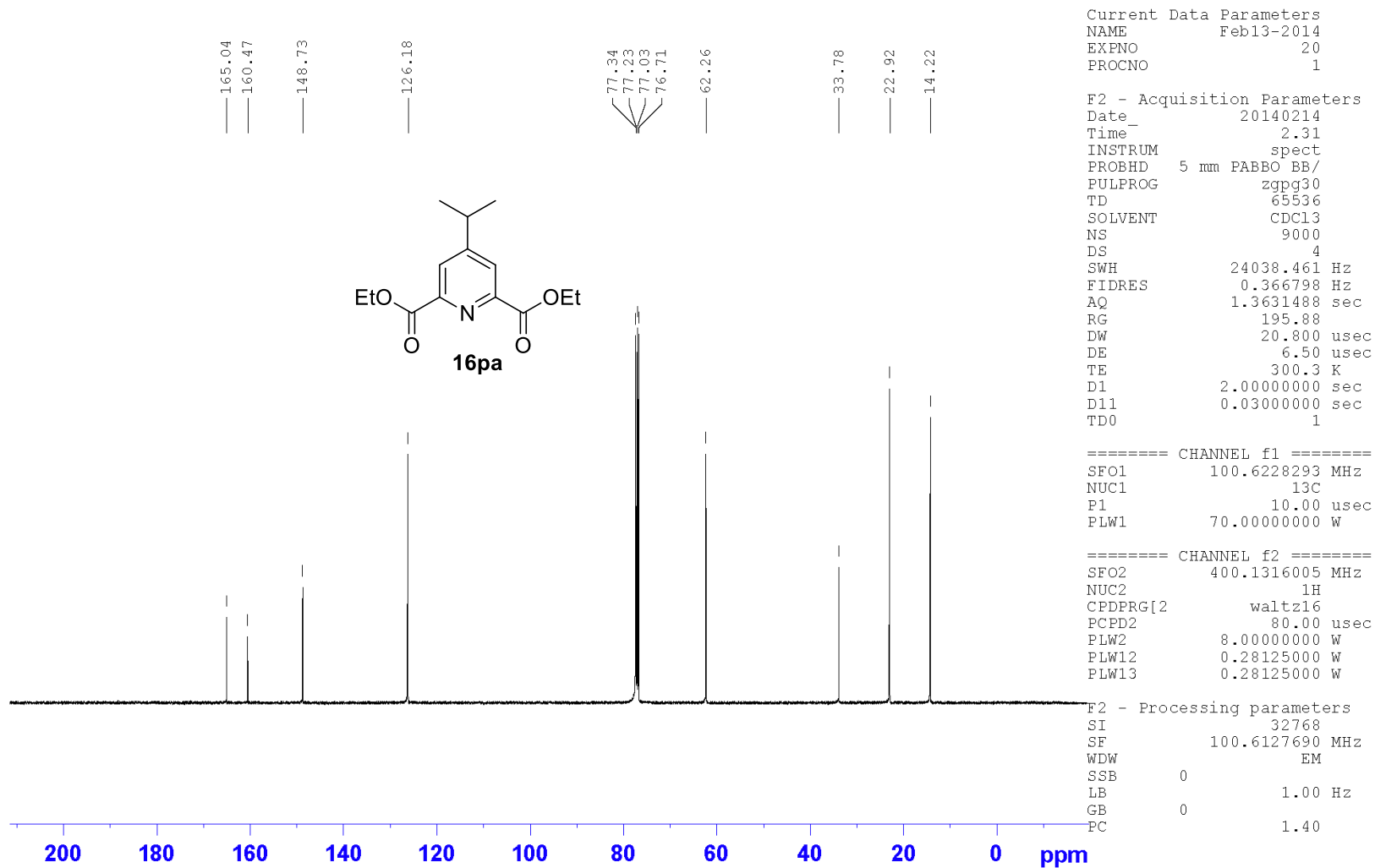


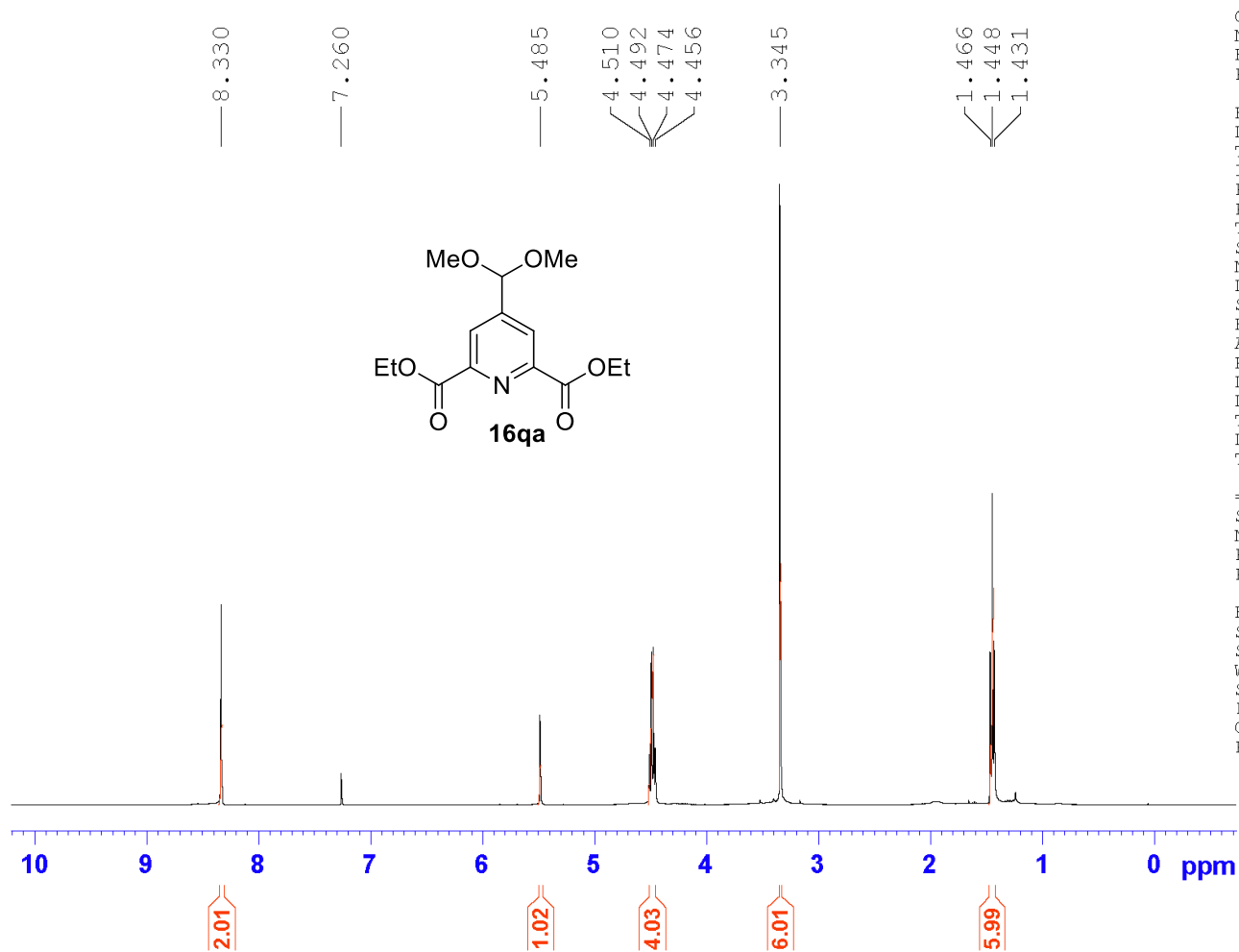
Current Data Parameters
 NAME Feb17-2014
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140217
 Time 12.45
 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 298.3 K
 D1 1.00000000 sec
 TD0 1

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

F2 - Processing parameters
 SI 65536
 SF 400.1300101 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



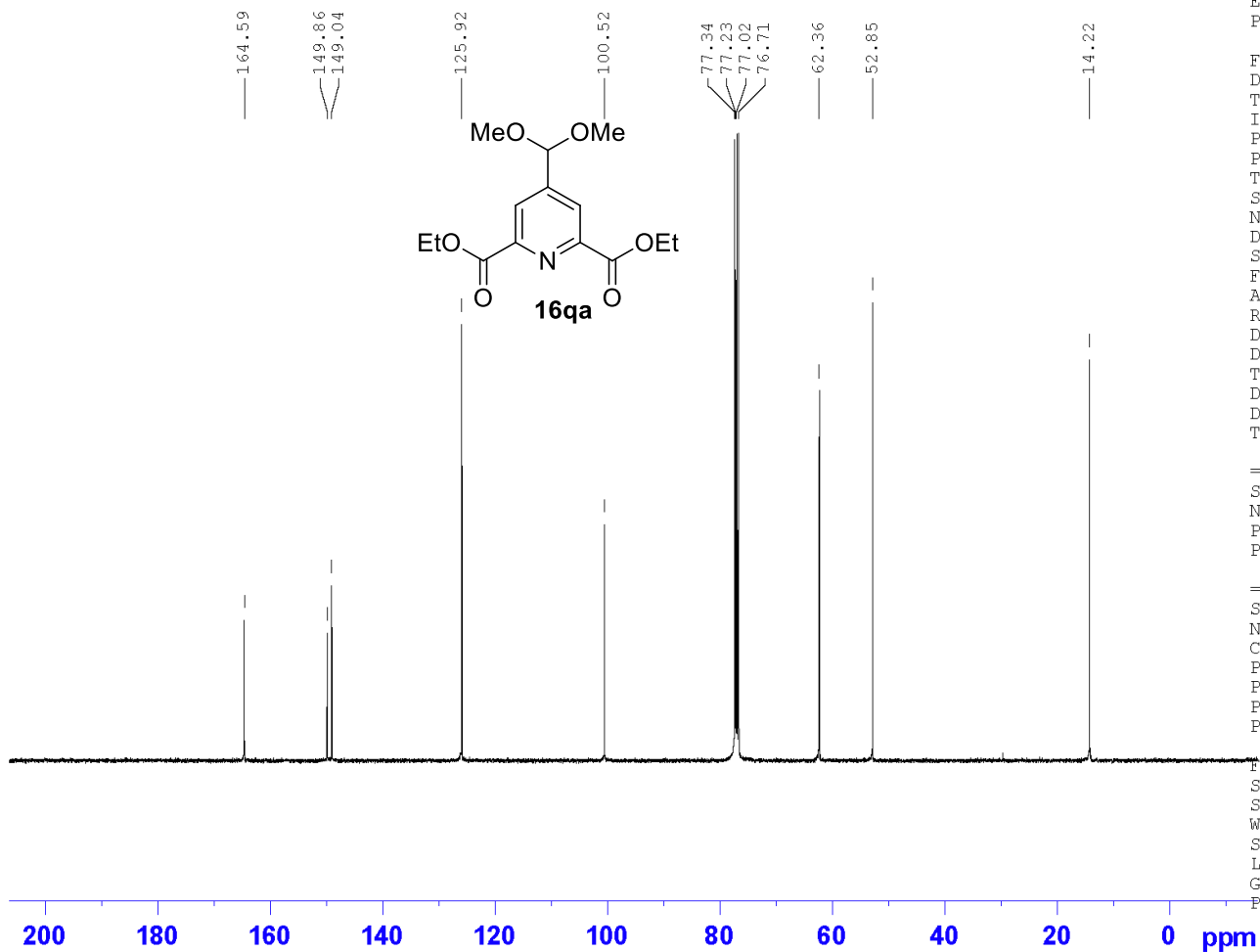


Current Data Parameters
 NAME Feb12-2014
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140212
 Time 17.46
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 54.59
 DW 62.400 usec
 DE 6.50 usec
 TE 298.4 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 400.1300106 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



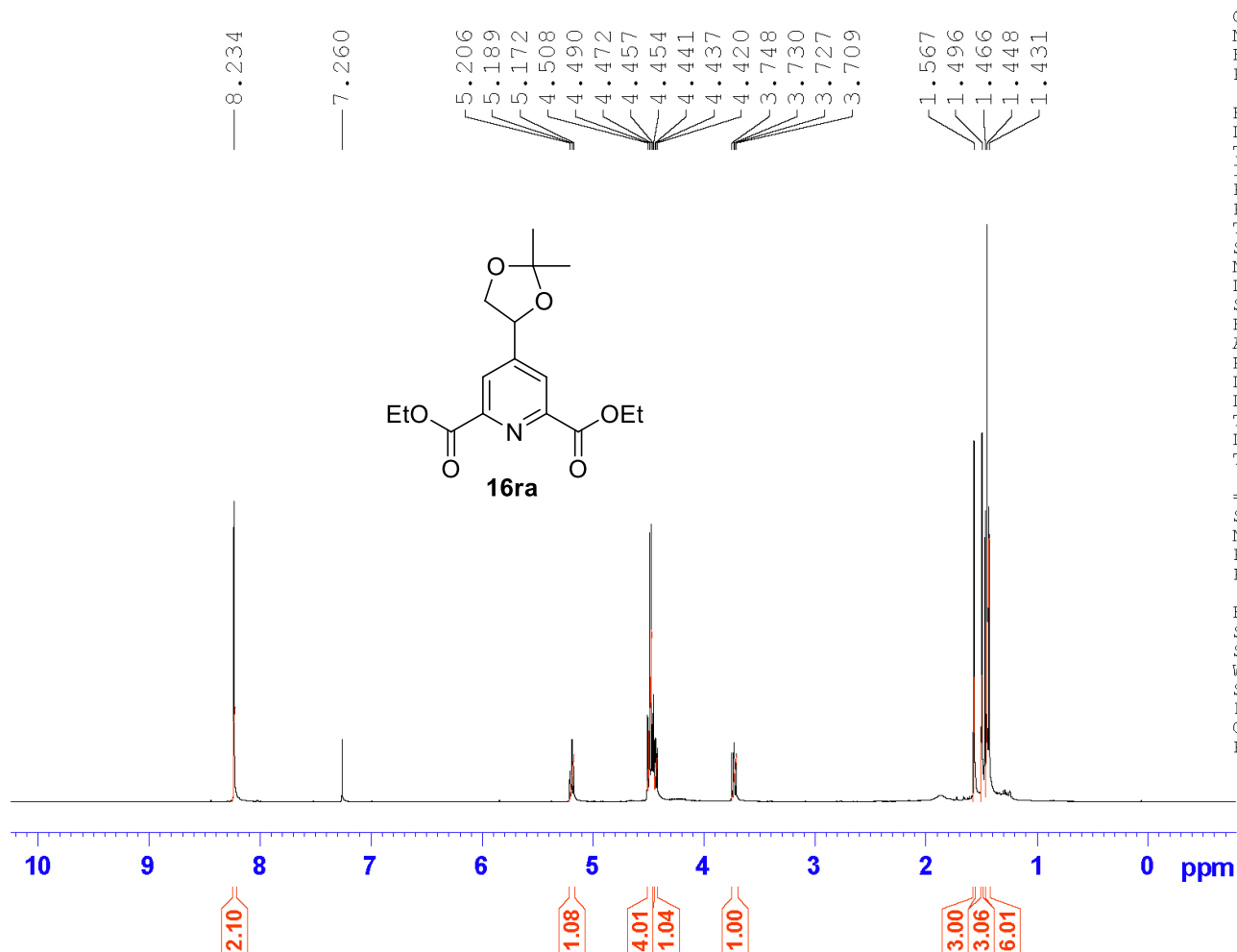
Current Data Parameters
 NAME Feb12-2014
 EXPNO 40
 PROCNO 1

F2 - Acquisition Parameters
 Date 20140213
 Time 2.31
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 9000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 299.9 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

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

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

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

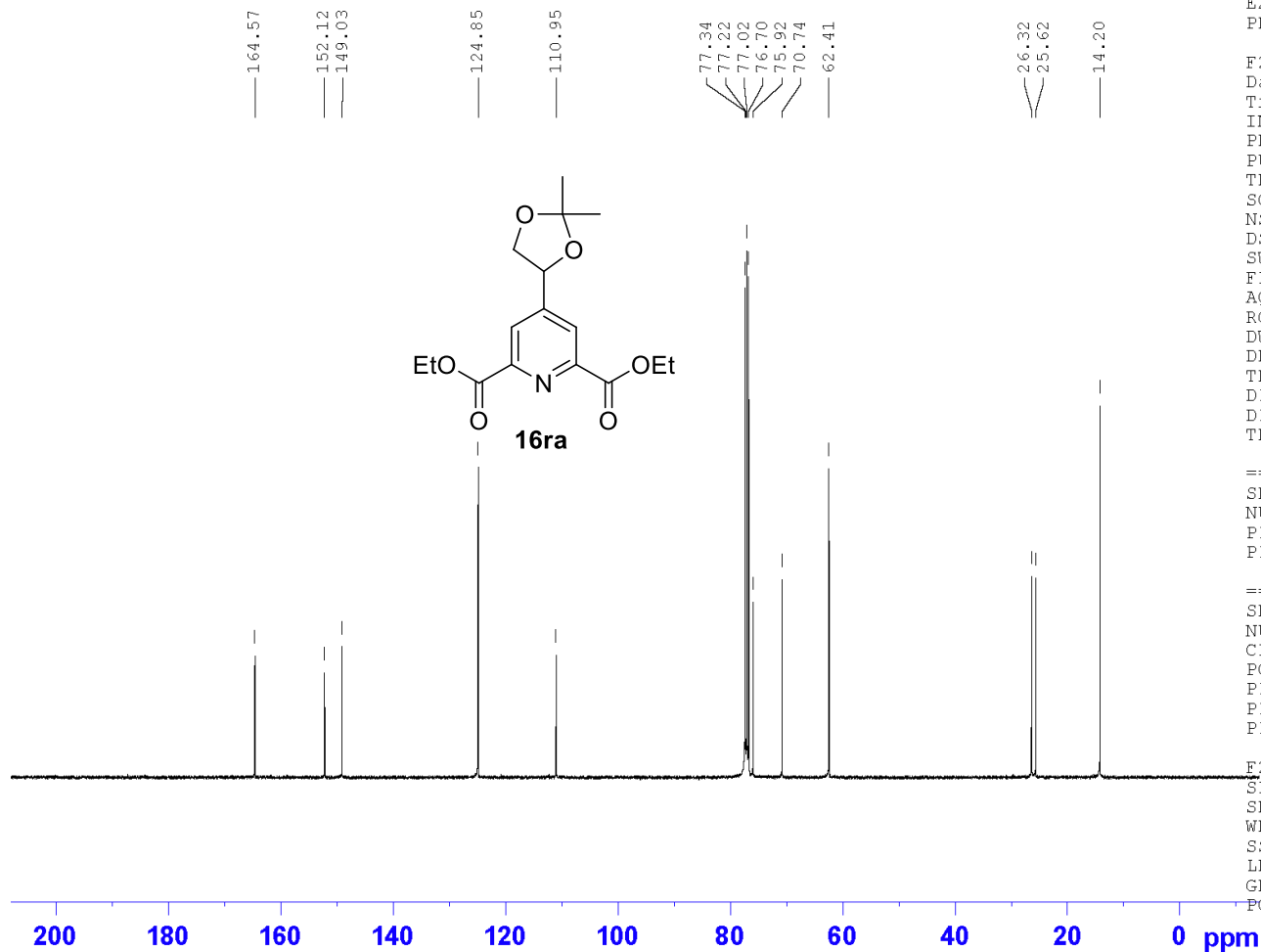


Current Data Parameters
 NAME Feb18-2014
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140218
 Time 14.28
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 49.09
 DW 62.400 usec
 DE 6.50 usec
 TE 299.3 K
 D1 1.00000000 sec
 TD0 1

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

F2 - Processing parameters
 SI 65536
 SF 400.1300099 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME Feb18-2014
 EXPNO 60
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140219
 Time 3.42
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 10000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.88
 DW 20.800 usec
 DE 6.50 usec
 TE 301.4 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

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