Vigorously Stirred La_2O_3 Suspensions for Michael Additions in Water

Tomoya Hisada, Rina Osada, Taku Kitanosono, Yasuhiro Yamashita, Shū Kobayashi**

Department of Chemistry, School of Sciences, The University of Tokyo, Hongo, Bunkyo-ku, Tokyo, Japan

Supporting Information

Table of Contents

1. General experimental

Nuclear magnetic resonance (NMR) spectra were recorded on a JEOL ECZ-500R spectrometer, operating at 500 MHz for ^{1}H, 126 MHz for ^{13}C NMR, and 471 MHz for ^{19}F NMR, or JEOL ECS-400 spectrometer, operating at 376 MHz for ^{19}F NMR in $CDCl_3$. Tetramethylsilane (TMS) served as the internal standard ($\delta = 0$) for ^{1}H NMR, $CDCl_3$ ($\delta = 77.0$) was used as the internal standard for ^{13}C NMR, and benzotrifluoride ($\delta = -63.72$) was used as the internal standard for ^{19}F NMR. Infrared (IR) spectra were obtained using a SHIMADZU IRSpirit spectrometer. Data are represented as frequency of absorption (cm^{-1}). Preparative thin-layer chromatography (PTLC) was carried out using Wakogel B-5F from Wako Pure Chemical Industries, Ltd. X-ray diffraction (XRD) was recorded using MiniFlex 300/600 spectrometer in the Bragg angle range of 3-60°. Thermogravimetric analyses (TGA) were recorded using TG-DTA8122 (corundum pans, heating rate: 5 K^{-1}; atmosphere). High Resolution Mass Spectra (HRMS) were recorded using a JEOL JMS-T100TD (DART) spectrometer or Bruker Compact System (ESI). Melting points were obtained using AS ONE Melting Point Meter (ATM-02). Deionized water from a MILLIPORE MilliQ machine (Gradient A 10) was used as solvent without further treatment. All commercially available reagents, unless otherwise noted, were purchased from Tokyo Chemical Industry Co. Ltd. Deuterated water (99.8 atom% D) was purchased from Kanto Chemical Co., Inc. Lanthanum hydroxide (99.9%) was purchased from Soekawa Chemicals Co., Ltd. Metal oxides except La_2O_3 were purchased from Wako Pure Chemical Industries, Ltd., Sigma-Aldrich Co. LLC., Koujundo Chemical Laboratory Co., Ltd. and Strem Chemicals, Inc. All organic solvents used were commercially available dry solvents, which were distilled appropriately under an argon atmosphere or were stored over molecular sieves prior to use. All reagents used as additives were either distilled or recrystallized before use.

2. Catalyst Characterization

Synthesis of La_2O_3

Calcination of commercially available $La(OH)_3$ (1.5 g) in a crucible at 600 ℃ for 4 hours under air led to the formation of white powder (1.3 g). Structural characterization was carried out by XRD pattern and TGA, proving to be a pure La_2O_3 phase.[1] The formed La_2O_3 was stored in a glove box because they react readily with water and CO_2 in the air.[2].

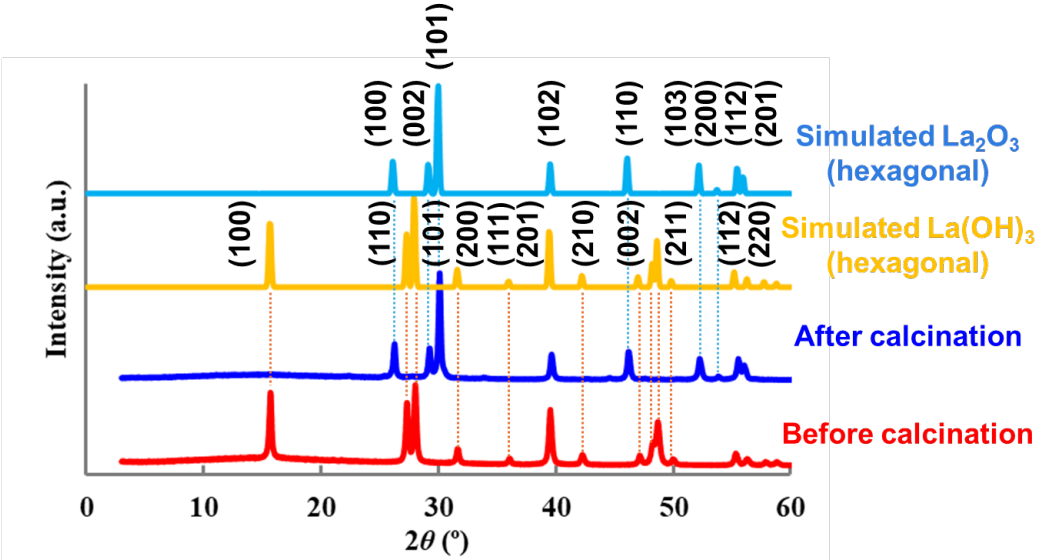


Figure S1. XRD patterns of catalysts.

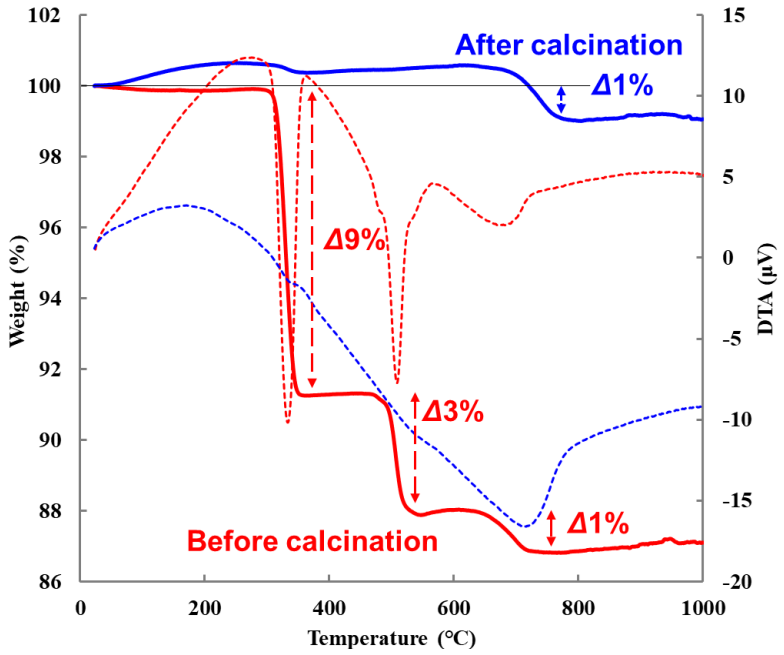


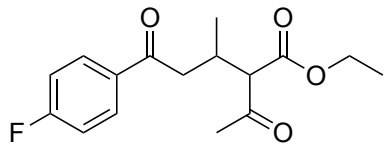
Figure S2. TG and DTA curves of catalysts. La_2O_3: Blue; $La(OH)_3$: Red

In thermogravimetry of commercially available $La(OH)_3$, a two-step dehydration mechanism was detected (<550 °C).[3] Considering that a mass loss in the temperature range from 600 to 800 °C corresponds to the release of CO_2,[4] commercially available $La(OH)_3$ includes oxide and carbonate impurities (dehydration of 9.5% with carbonate content of 1 wt%). According to the literature,[4] the carbonate impurities are derived from the reaction with the CO_2 content in the air. On the other hand, there is no observing the step of the water release after calcination, proving the powder to be pure La_2O_3. The calculation suggested a residual carbonate content of 1 wt%.

3. Typical procedure for 1,4-addition reaction

The anaerobically stored La_2O_3 (2.0 mg, 0.006 mmol) and ethyl acetoacetate (78.1 mg, 0.60 mmol) were added to a 2 mL vial in a glove box. After the addition of (*E*)-1-(4-fluorophenyl)but-2-en-1-one (118.2 mg, 0.72 mmol), degassed pure water (1.0 mL) was poured. The vial was purged with argon and the mixture was stirred vigorously for 1 hour. The reaction was quenched with a 1M HCl solution (1.0 mL), and the mixture was extracted with CH_2Cl_2 (20 × 3 mL). The combined organic layers were dried over anhydrous Na_2SO_4. After being concentrated under reduced pressure, the residue was purified by preparative TLC (^{n}hexane:EtOAc = 4:1) to afford the desired product.

Ethyl 2-acetyl-5-(4'-fluorophenyl)-3-methyl-5-oxopentanoate (3aa)
(mixture of diastereomers)



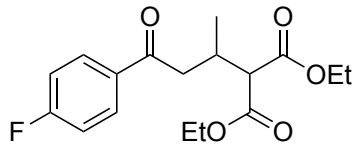
Colorless oil
IR (dichloromethane solution) ν = 3075, 2976, 1710, 1596 cm^{-1}.
^{1}H NMR (500 MHz): δ 8.05-8.00 (m, 2H), 7.14 (m, 2H), 4.24-4.19 (m, 2H), 3.62+3.57 (d, J = 7.4 Hz, 1H), 3.24-3.15 (m, 1H), 3.00-2.95 (m, 1H), 2.88-2.81 (m, 1H), 2.27 (s, 3H), 1.28+1.27 (t, J = 7.2 Hz, 3H), 1.06+1.04 (d, J = 6.7 Hz, 3H).
^{13}C NMR (126 MHz): δ 202.9, 197.3, 197.3, 169.0, 168.9, 165.7 (d, $J_{C\text{-}F}$ = 254.7 Hz), 165.7(d, $J_{C\text{-}F}$ = 254.7 Hz), 133.3 (d, $J_{C\text{-}F}$ = 3.0 Hz), 133.2 (d, $J_{C\text{-}F}$ = 3.0 Hz), 130.8 (d, $J_{C\text{-}F}$ = 7.5 Hz), 130.7 (d, $J_{C\text{-}F}$ = 7.8 Hz), 115.7 (d, $J_{C\text{-}F}$ = 22.0 Hz), 115.6 (d, $J_{C\text{-}F}$ = 21.7 Hz), 64.4, 64.0, 61.4, 61.3, 42.7, 42.4, 29.7, 29.6, 29.3, 29.0, 18.0, 17.5, 14.0.
^{19}F NMR (376 MHz): δ –106.2, –106.3.
HRMS (DART) calcd for $C_{15}H_{18}FO_3$ $[M+H]^+$: 295.1346; found 295.1303.

Modified procedure for some pronucleophiles

The anaerobically stored La_2O_3 (2.5 mg, 0.008 mmol) and diethyl malonate (48.1 mg, 0.30 mmol) were added to a 2 mL vial in a glove box. After the addition of (*E*)-1-(4-fluorophenyl)but-2-en-1-one (59.1 mg, 0.36 mmol), degassed pure water (1.0 mL) was poured. The vial was purged with argon and the reaction solution was stirred vigorously for 24 hours. The reaction was quenched with a 1M HCl solution (1.0 mL), and the mixture was extracted with CH_2Cl_2 (20 × 3 mL). The combined organic layers were dried

over anhydrous Na_2SO_4. After being concentrated under reduced pressure, the residue was purified by preparative TLC (nhexane:EtOAc = 4:1) to afford the desired product.

Ethyl 2-acetyl-5-(4'-fluorophenyl)-3-methyl-5-oxopentanoate (**3ea**)



Colorless oil

IR (methanol solution) v = 2596, 2520, 2228, 2042, 1748, 1729 cm^{-1}.

^1H NMR (500 MHz); δ 8.05-8.02 (m, 2H), 7.13 (t, J = 8.6 Hz, 2H), 4.25-4.18 (m, 4H), 3.47 (d, J = 6.6 Hz, 1H), 3.30+3.27 (d, J = 4.2 Hz, 1H), 2.96-2.95 (m, 1H), 2.90-2.85 (m, 1H), 1.29-1.25 (m, 6H), 1.09 (d, J = 6.7 Hz, 3H).

^{13}C NMR (126 MHz); δ 197.1, 168.6, 168.5, 165.7 (d, J_{C-F} = 254.7 Hz), 133.3 (d, J_{C-F} = 3.0 Hz), 130.7 (d, J_{C-F} = 9.4 Hz), 115.6 (d, J_{C-F} = 21.7 Hz), 61.2, 56.4, 42.5, 29.4, 17.6, 14.0, 14.0.

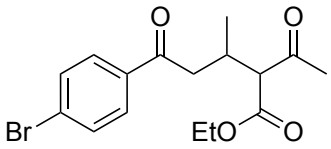
^{19}F NMR (376 MHz); δ –106.3.

HRMS (DART) calcd for $C_{17}H_{21}FO_5$ $[M+H]^+$: 325.1451; found 325.1450.

4. Analytical data

When the product is a diastereomeric mixture, two diastereomers cannot be distinguished further in NMR spectra and some of the peaks overlap.

Ethyl 2-acetyl-5-(4'-bromophenyl)-3-methyl-5-oxopentanoate (3ab)
(mixture of diastereomers)



Colorless oil

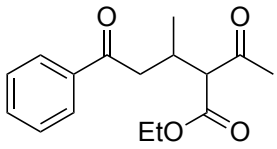
IR (dichloromethane solution) ν = 3055, 2980, 1711, 1682 cm^{-1}.

^1H NMR (500 MHz): δ 7.87-7.83 (m, 2H), 7.61-7.58 (m, 2H), 4.23-4.18 (m, 2H), 3.61+3.56 (d, J = 7.4 Hz, 1H), 3.23-3.13 (m, 1H), 2.97-2.95 (m, 1H), 2.87-2.80 (m, 1H), 2.27 (s, 3H), 1.28+1.27 (t, J = 7.2 Hz, 3H), 1.05+1.03 (d, J = 6.9 Hz, 3H).

^{13}C NMR (126 MHz): δ 202.6, 197.7, 197.6, 168.8, 168.7, 135.4, 135.3, 131.7, 131.7, 129.5, 129.5, 128.1, 128.0, 64.1, 63.8, 61.2, 61.1, 42.6, 42.3, 29.6, 29.5, 29.1, 28.8, 17.8, 17.3, 13.9.

HRMS (DART) calcd for $C_{16}H_{20}BrO_4$ $[M+H]^+$: 355.0545; found 355.0539.

Ethyl 2-acetyl-3-methyl-5-oxo-5-phenylpentanoate (3ac)
(mixture of diastereomers)



Colorless oil

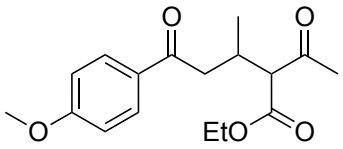
IR (dichloromethane solution) ν = 3060, 2935, 1732, 1711, 1681 cm^{-1}.

^1H NMR (500 MHz): δ 7.98 (td, J = 8.7, 1.2 Hz, 2H), 7.57-7.54 (m, 1H), 7.46 (m, 2H), 4.23-4.18 (m, 2H), 3.63+3.56 (d, J = 7.3 Hz, 1H), 3.23-3.15 (m, 1H), 3.01-2.98 (m, 1H), 2.92-2.85 (m, 1H), 2.27+2.26 (s, J = 2.0 Hz, 3H), 1.27+1.26 (t, J = 7.2 Hz, 3H), 1.06+1.04 (d, J = 6.9 Hz, 3H).

^{13}C NMR (126 MHz): δ 202.8, 202.8, 198.7, 198.6, 168.9, 168.8, 136.7, 136.7, 133.0, 132.9, 128.4, 128.4, 127.9, 127.9, 64.3, 63.8, 61.1, 61.1, 42.6, 42.3, 29.5, 29.5, 29.2, 28.8, 17.8, 17.3, 13.9.

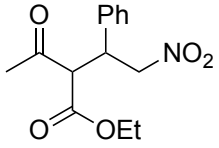
HRMS (DART) calcd for $C_{16}H_{21}O_4$ [M+H]$^+$: 277.1440; found 277.1451.

Ethyl 2-acetyl-5-(4'-methoxyphenyl)-3-methyl-5-oxopentanoate (3ad)
(mixture of diastereomers)



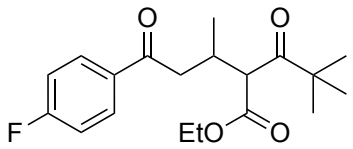
Colorless oil
IR (dichloromethane solution) ν = 2967, 2935, 1711, 1598 cm^{-1}.
^{1}H NMR (500 MHz): δ 7.98-7.94 (m, 2H), 6.94-6.93 (m, 2H), 4.22-4.18 (m, 2H), 3.86 (s, 3H), 3.63+3.56 (d, J = 7.4 Hz, 1H), 3.18-3.09 (m, 1H), 2.98-2.96 (m, 1H), 2.84-2.77 (m, 1H), 2.27+2.26 (s, 3H), 1.27+1.26 (t, J = 7.2 Hz, 3H), 1.05+1.02 (d, J = 6.9 Hz, 3H).
^{13}C NMR (126 MHz): δ 202.8, 202.8, 197.2, 197.1, 168.9, 168.8, 163.3, 163.3, 130.2, 130.2, 129.8, 129.7, 113.5, 64.4, 63.9, 61.1, 61.0, 55.2, 42.2, 42.0, 29.4, 29.4, 29.0, 17.7, 17.2, 13.9.
HRMS (DART) calcd for $C_{17}H_{23}O_5$ [M+H]$^+$: 307.1546; found 307.1546.

Ethyl 2-acetyl-4-nitro-3-phenylbutanoate (3ae)[5]
(mixture of diastereomers)



Yellow solid : m.p. 42-49 ℃.
^{1}H NMR (500 MHz); δ 7.32-7.26 (m, 3H), 7.21-7.20 (m, 2H), 4.85-4.74 (m, 2H), 4.24-4.20 (m, 2H), 4.11+4.03 (d, J = 10.0 Hz, 1H), 3.95 (q, J = 7.1 Hz, 1H), 2.29+2.04 (s, 3H), 1.27+0.99 (t, J = 7.2 Hz, 3H).
^{13}C NMR (126 MHz); δ 201.1, 200.3, 167.4, 166.8, 136.3, 136.3, 129.0, 128.8, 128.2, 128.2, 127.9, 127.8, 77.8, 77.7, 62.1, 61.9, 61.8, 61.5, 42.5, 42.2, 30.2, 30.0, 13.9, 13.6.

Ethyl 5-(4'-fluorophenyl)-3-methyl-5-oxo-2-pivaloylpentanoate (3ba)
(mixture of diastereomers)



Colorless oil

IR (dichloromethane solution) ν = 2970, 2874, 1733, 1596 cm^{-1}.

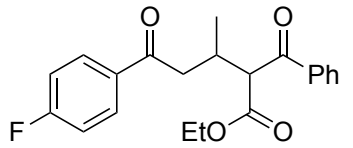
^1H NMR (500 MHz): δ 8.04-8.00 (m, 2H), 7.16-7.11 (m, 2H), 4.19-4.13 (m, 2H), 4.06+4.04 (d, J = 7.7 Hz, 1H), 3.31+3.16 (dd, J = 16.5, 3.9 Hz, 1H), 2.97-2.93 (m, 1H), 2.84+2.73 (m, 1H), 1.27-1.22 (m, 3H), 1.19+1.18 (s, 9H), 1.03 (t, J = 7.4 Hz, 3H).

^{13}C NMR (126 MHz): δ 209.7, 209.3, 197.3, 197.2, 168.7, 168.6, 165.6 (d, J_{C-F} = 254.7 Hz), 165.5(d, J_{C-F} = 254.7 Hz), 133.3 (d, J_{C-F} = 3.0 Hz), 133.1 (d, J_{C-F} = 3.0 Hz), 130.7 (d, J_{C-F} = 9.4 Hz), 130.6 (d, J_{C-F} = 9.4 Hz), 115.5 (d, J_{C-F} = 21.7 Hz), 115.4 (d, J_{C-F} = 21.7 Hz), 61.1, 61.1, 57.3, 57.0, 45.4, 43.2, 42.0, 30.6, 30.5, 26.0, 26.0, 18.6, 17.0, 13.9, 13.9.

^{19}F NMR (471 MHz): δ –106.3, –106.5.

HRMS (DART) calcd for $C_{19}H_{26}FO_4$ $[M+H]^+$: 337.1815; found 337.1814.

Ethyl 2-benzoyl-5-(4-fluorophenyl)-3-methyl-5-oxopentanoate (3ca)
(mixture of diastereomers)



Colorless oil

IR (dichloromethane solution) ν = 2976, 2936, 1730, 1596 cm^{-1}.

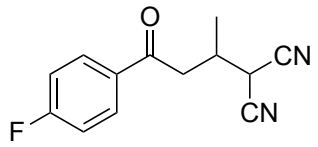
^1H NMR (500 MHz): δ 8.09-8.00 (m, 4H), 7.61-7.57 (m, 1H), 7.51-7.46 (m, 2H), 7.14-7.10 (m, 2H), 4.57+4.54 (d, J = 7.3 Hz, 1H), 4.18-4.11 (m, 2H), 3.36-3.31 (m, 1H), 3.15-3.12 (m, 1H), 2.93+2.83 (dd, J = 16.3, 9.0 Hz, 1H), 1.18-1.08 (m, 6H).

^{13}C NMR (126 MHz): δ 197.5, 197.3, 194.9, 194.7, 169.0, 168.8, 165.6 (d, J_{C-F} = 254.7 Hz), 165.6 (d, J_{C-F} = 254.7 Hz), 136.6, 136.3, 133.5, 133.5, 133.2 (d, J_{C-F} = 3.0 Hz), 133.2 (d, J_{C-F} = 3.0 Hz), 130.7 (d, J_{C-F} = 9.4 Hz), 130.7 (d, J_{C-F} = 9.4 Hz), 128.7, 128.5, 128.4, 115.5 (d, J_{C-F} = 22.0 Hz), 115.5 (d, J_{C-F} = 22.0 Hz), 59.7 (d, J_{C-F} = 364.3 Hz), 59.7 (d, J_{C-F} = 364.3 Hz), 43.1, 42.4, 30.1, 29.7, 18.1, 17.1, 13.8, 13.8.

^{19}F NMR (471 MHz): δ –106.2, –106.3.

HRMS (ESI) calcd for $C_{21}H_{21}FNaO_4$ $[M+Na]^+$: 379.1316; found 379.1335.

2-(4-(4'-Fluorophenyl)-4-oxobutan-2-yl)malononitrile (3da)



Colorless oil

IR (methanol solution) ν = 2361, 2342, 2255, 1684, 1227, 841 cm^{-1}.

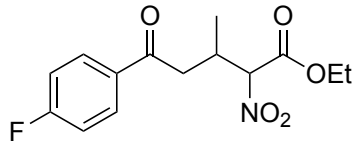
^{1}H NMR (500 MHz); δ 8.00-7.98 (m, 2H), 7.17 (t, J = 8.6 Hz, 2H), 4.38 (d, J = 4.7 Hz, 1H), 3.19-3.18 (m, 2H), 2.90-2.88 (m, 1H), 1.39 (d, J = 6.9 Hz, 3H).

^{13}C NMR (126 MHz); δ 195.4, 166.2 (d, $J_{C\text{-}F}$ = 256.5 Hz), 132.3 (d, $J_{C\text{-}F}$ = 3.0 Hz), 130.7 (d, $J_{C\text{-}F}$ = 9.4 Hz), 116.0 (d, $J_{C\text{-}F}$ = 22.0 Hz), 112.2, 111.4, 41.0, 31.3, 27.9, 17.1.

^{19}F NMR (471 MHz); δ –104.3.

HRMS (DART) calcd for $C_{13}H_{12}FN_2O$ $[M+H]^+$: 231.0934; found 231.0940.

Ethyl 5-(4'-fluorophenyl)-3-methyl-2-nitro-5-oxopentanoate (3fa)
(mixture of diastereomers)



Colorless oil

IR (dichloromethane solution) ν = 2959, 2927, 1745, 1682 cm^{-1}.

^{1}H NMR (500 MHz); δ 8.01-7.97 (m, 2H), 7.15 (t, J = 8.7 Hz, 2H), 5.39+5.32 (d, J = 5.6 Hz, 1H), 4.32-4.27 (m, 2H), 3.29-3.23 (m, 2H), 3.10-3.03 (m, 1H), 1.30 (td, J = 7.2, 3.0 Hz, 3H), 1.20 (t, J = 7.3 Hz, 3H).

^{13}C NMR (126 MHz); δ 195.8, 195.7, 165.9 (d, $J_{C\text{-}F}$ = 255.6 Hz), 165.9 (d, $J_{C\text{-}F}$ = 255.3 Hz), 163.7, 163.7, 132.9 (d, $J_{C\text{-}F}$ = 2.7 Hz), 132.9 (d, $J_{C\text{-}F}$ = 3.0 Hz), 130.7 (d, $J_{C\text{-}F}$ = 2.4 Hz), 130.7 (d, $J_{C\text{-}F}$ = 2.4 Hz), 115.9 (d, $J_{C\text{-}F}$ = 2.4 Hz), 115.7 (d, $J_{C\text{-}F}$ = 2.4 Hz), 91.2, 90.8, 62.9, 62.9, 40.8, 40.7, 30.5, 30.5, 16.3, 16.1, 13.9, 13.8.

^{19}F NMR (376 MHz); δ –105.4, –105.5.

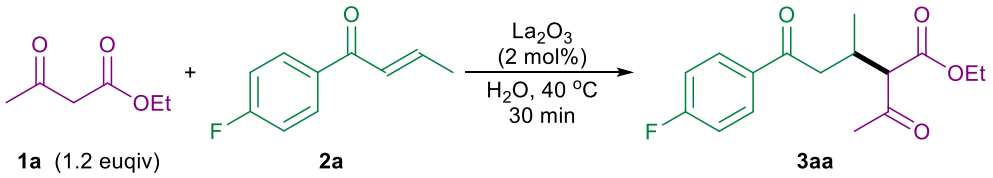
HRMS (ESI) calcd for $C_{14}H_{16}FNNaO_5$ $[M+Na]^+$: 320.0905; found 320.0913.

5. Gram-scale experiment

The anaerobically stored La_2O_3 (16.7 mg, 0.05 mmol), and ethyl acetoacetate (650.8 mg, 5.0 mmol) were added to a 6 mL vial in a glove box. After the addition of (*E*)-1-(4-fluorophenyl)but-2-en-1-one (984.8 mg, 6.0 mmol), degassed pure water (4.5 mL) was poured. The vial was purged with argon and the mixture was stirred vigorously for 1 hour. The reaction was quenched with a 1M HCl solution (8.4 mL), and the mixture was extracted with CH_2Cl_2 (20 × 3 mL). The combined organic layers were dried over anhydrous Na_2SO_4. After being concentrated under reduced pressure, the residue was purified by column chromatography (^{n}hexane:EtOAc = 4:1) to afford the desired product as a colorless oil (1.3674 g, 93%).

6. Control Experiments

Effect of rare earth oxides

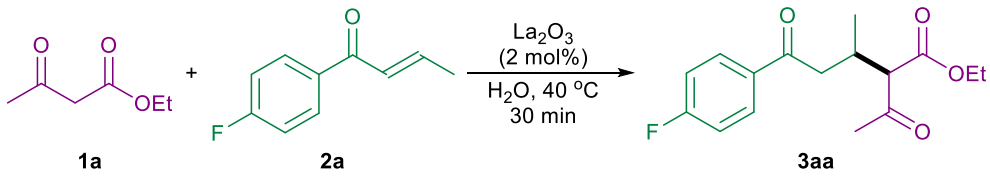


1a (1.2 euqiv) **2a** **3aa**

Rare earth oxides	Yield (%)[a]	Rare earth oxides	Yield (%)[a]
Sc_2O_3	0 (2 h)	Gd_2O_3	31 (62)[b]
Y_2O_3	11	Tb_4O_7	0 (2 h)
La_2O_3	90	Dy_2O_3	12
CeO_2	0 (2 h)	Ho_2O_3	11 (2 h)
Pr_6O_{11}	7 (2 h)	Er_2O_3	0 (2 h)
Nd_2O_3	38 (74)[b]	Tm_2O_3	7 (2 h)
Sm_2O_3	47 (80)[b]	Yb_2O_3	4
Eu_2O_3	38 (48)[c]	Lu_2O_3	1

[a] Determined by ^{1}H NMR analysis. [b] Synthesized from $M(OH)_3$ by calcination at 900 °C for 3 h.
[c] Prepared by calcination of commercially available Eu_2O_3 at 600 °C for 4 h.

Effect of stirring

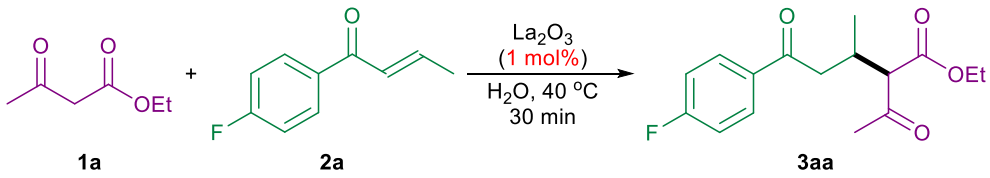


1a **2a** **3aa**

no stirring: 13% yield[a]
600 rpm: 77% yield[a]
1,200 rpm: 93% yield[a]

[a] Determined by ^{1}H NMR analysis.

Effect of La_2O_3 particle size

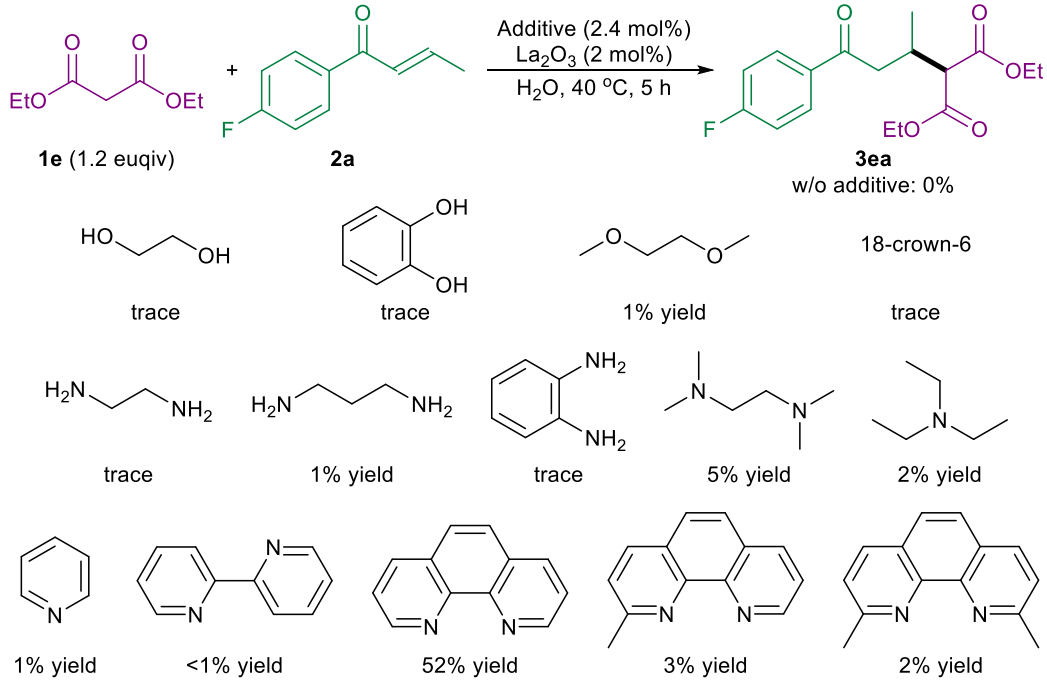


1a **2a** **3aa**

63% yield[a]
La_2O_3 from Sigma-Aldrich: 50% yield[a]

[a] Determined by ^{1}H NMR analysis.

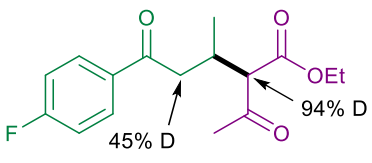
Effect of additives for reaction with diethyl malonate



Reaction run in deuterium oxide

(Table 1b, entry 1)

According to the general procedure, water was replaced with degassed pure D_2O. The mixture was quenched with 1M deuterium chloride solution in D_2O and extracted with CH_2Cl_2 (20×3 mL). The combined organic layers were dried over anhydrous Na_2SO_4. After being concentrated under reduced pressure, the rate of hydrogen and deuterium was determined by ^1H NMR analysis because deuterium was exchanged again with proton at the most acidic position upon purification by preparative TLC (^{n}hexane:EtOAc = 4:1). About 90% of C−H bond newly formed at C4 position was proved to be deuterium.



7. Hydrolytic Behavior of La_2O_3

It is known that La_2O_3-based materials can react relatively quickly with water/carbon dioxide in the atmosphere to form new bulk hydroxyl, carbonate, and mixed phases.[6] Because $La(OH)_3$ has no activity in the reaction developed in this study, the hydrolysis is associated with the catalytic activity. Therefore, the hydrolytic behavior of La_2O_3 was roughly studied. As-calcined La_2O_3 (>100 mg) was placed in a vial and stirred in water (3 mL) at 40 °C for an hour. The solid was collected by centrifugation, the removal of supernatant, and filtration. The full recovery was technically difficult due to the ease of forming colloidal dispersions. After drying in a vacuum overnight, the solid was subjected to XRD analysis (Figure S3). The peaks corresponding to La_2O_3 nearly disappeared and peaks corresponding to $La(OH)_3$ appeared instead, whether in the presence or absence of 1,10-phenanthroline. In addition, the observed decay of intensity suggests the transition of the crystalline state to an amorphous state of La_2O_3 as well as its conversion into $La(OH)_3$.

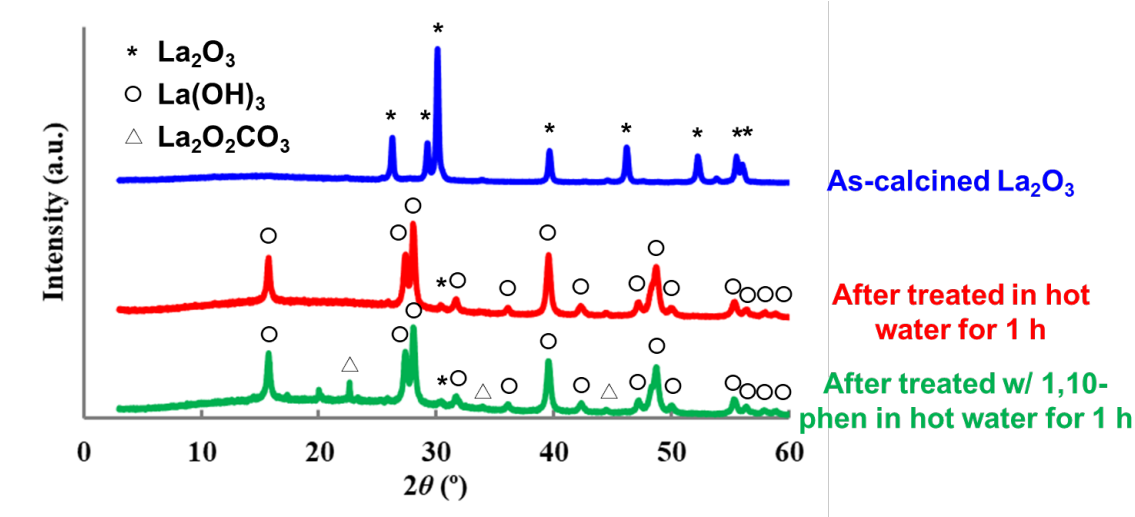
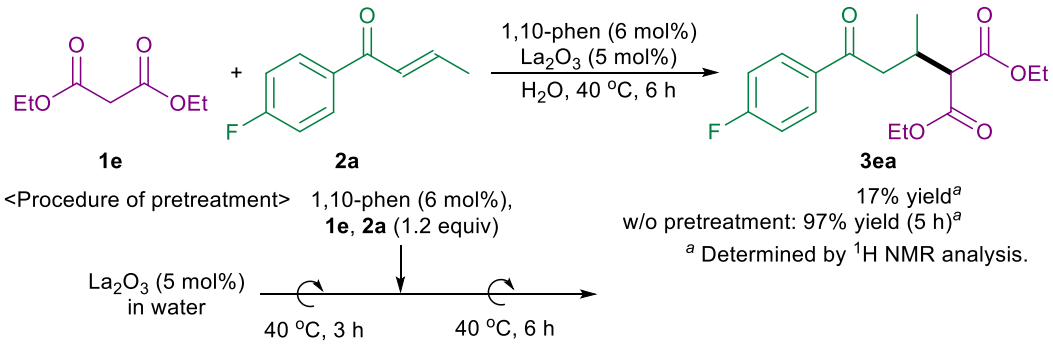


Figure S3. XRD patterns of La_2O_3 and effect of treatment in water.

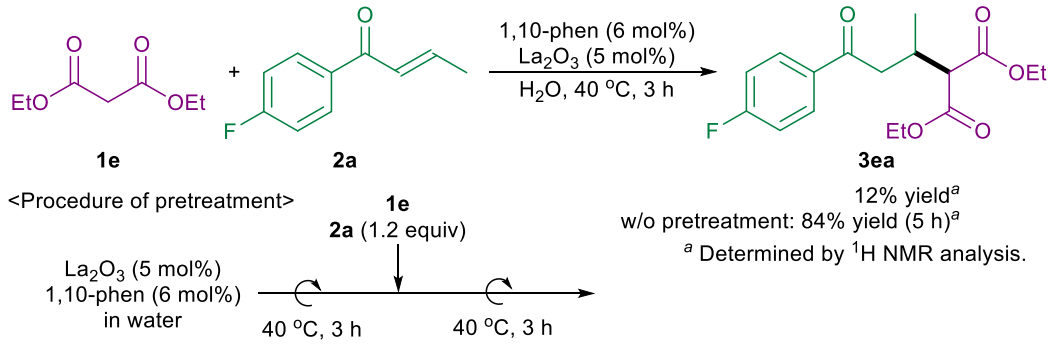
Similarly, the effect of the pretreatment in water on the activity of La_2O_3 was examined. In contrast to the optimized conditions that provide almost quantitative conversion of **1e** to the desired product **3ea**, the pre-stirring of La_2O_3 in water significantly reduced the activity of La_2O_3. This result suggests that amorphous La_2O_3 exhibits significantly poorer catalytic performance. The presence or absence of 1,10-phen in the pretreatment had no effect. Overall, the reaction outcome is consistent with the result of XRD analysis.

<pretreatment w/o 1,10-phen>



17% yield[a]
w/o pretreatment: 97% yield (5 h)[a]
[a] Determined by ^{1}H NMR analysis.

<pretreatment w/ 1,10-phen>



12% yield[a]
w/o pretreatment: 84% yield (5 h)[a]
[a] Determined by ^{1}H NMR analysis.

8. Reference

[1] W. C. Koehler, E. O. Wollan, *Acta Cryst.* **1953**. *6*, 741–742.

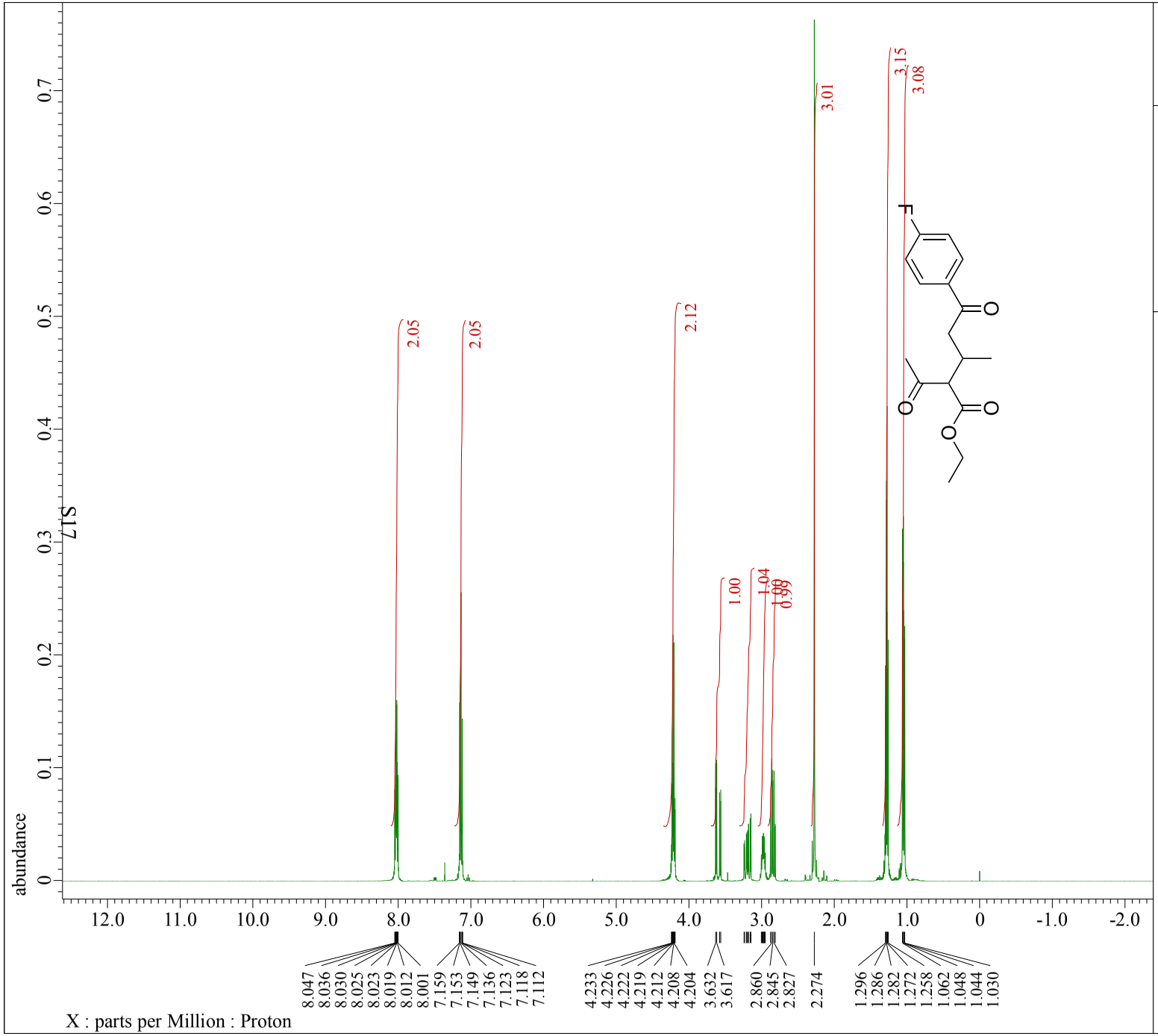
[2] S. Bernal, F. J. Botana, R. Garcia, J. M. Rodriquez-Izquierdo, *React. Solids* **1987**, *4*, 23–40.

[3] D. Walter, *Z. Anorg. Allg. Chem.* **2006**, 632, 2165.

[4] B. P. Gangwar, Y. Pakakollu, A. Singh, S. Kanvah, S. Sharma, *RSC Adv.* 2014, **4**, 55407–55416.

[5] D. A. Evans, D. Seidel, *J. Am. Chem. Soc.* **2005**, *127*, 9958–9959.

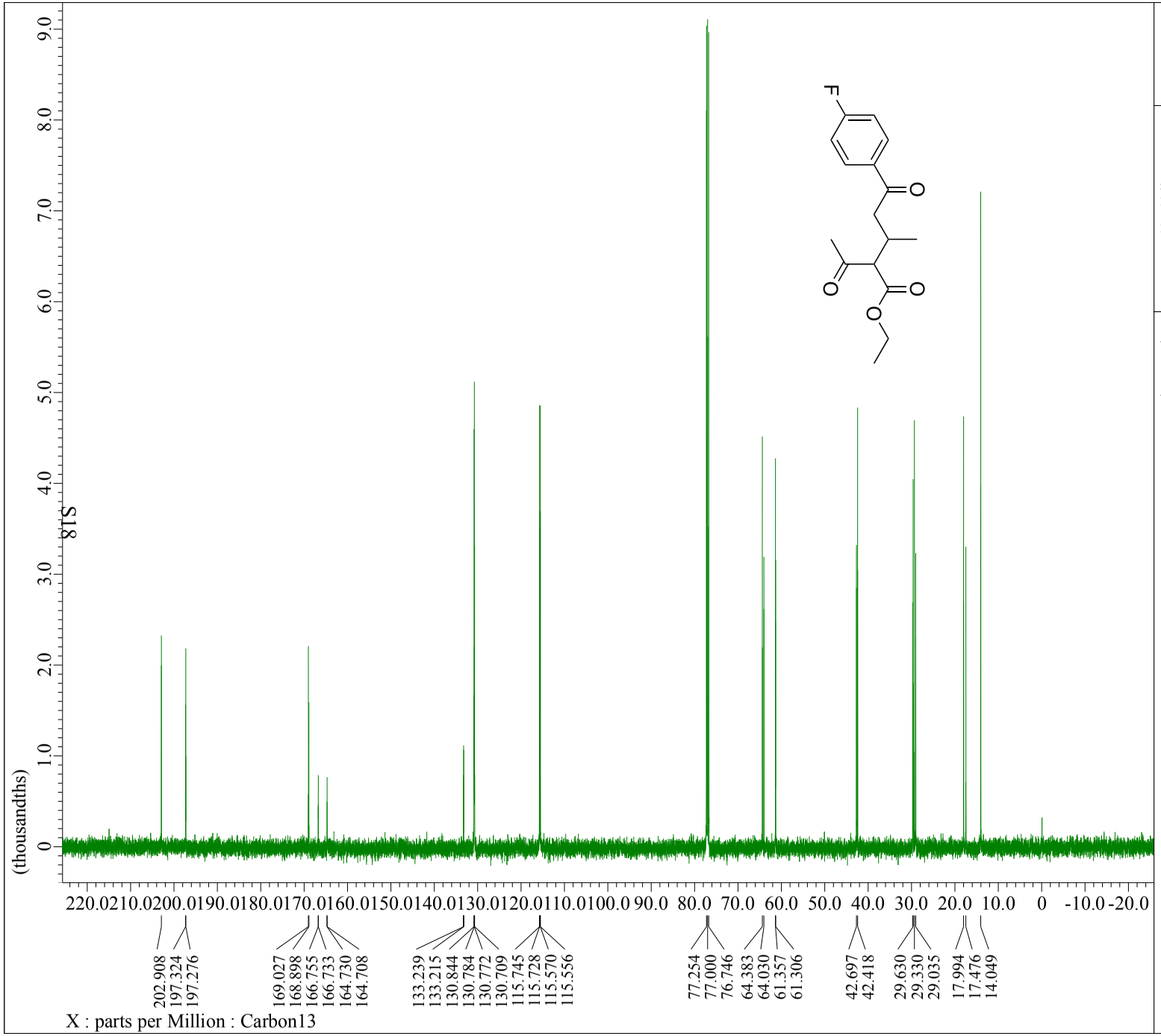
[6] P. Fleming, R. A. Farrell, J. D. Holmes, M. A. Morris, *J. Am. Ceram. Soc.* **2010**, *93*, 1187–1194.



^{1}H, ^{13}C & ^{19}F NMR spectra

```
---- PROCESSING PARAMETERS ----
sexp( 0.25016[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -0.10469, -1.78517, 8.93645[%] )
```

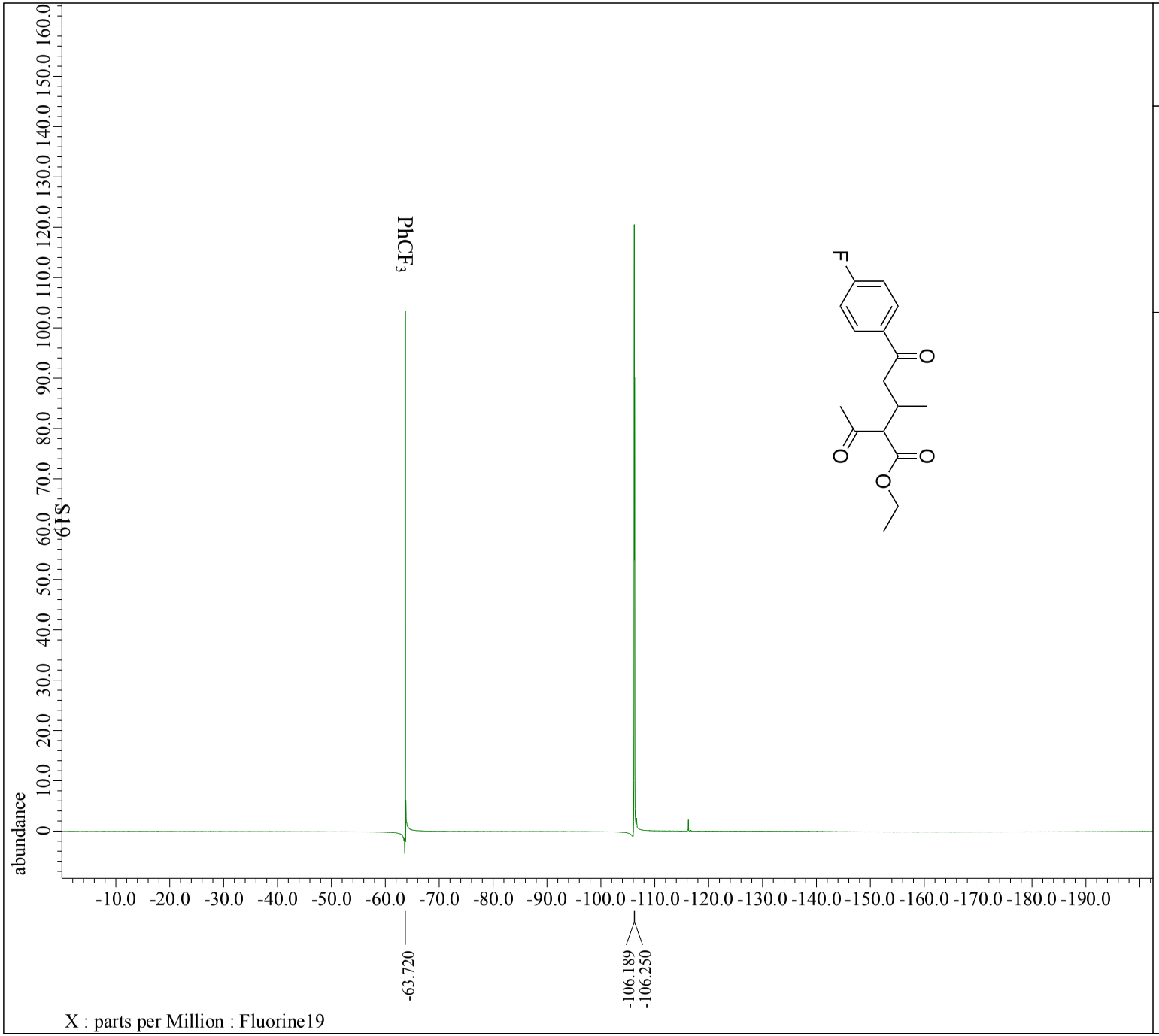
Filename	= RO0450-dry_PRO
Author	= console
Experiment	= single_pulse.j
Sample_Id	= RO0450-dry
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 9-FEB-2024 18
Revision_Time	= 2-MAY-2024 08
Data_Format	= 1D COMPLEX
Dim_Size	= 52429
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 1.99874592[s]
X_Domain	= Proton
X_Freq	= 500.15991521[M
X_Offset	= 5.0[ppm]
X_Points	= 18757
X_Points_Input	= 15005
X_Prescans	= 0
X_Resolution	= 0.50031372[Hz]
X_Sweep	= 9.38438438[kHz
X_Sweep_Clipped	= 7.50750751[kHz
X_Sweep_Input	= 15[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[M
Tri_Offset	= 5.0[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 4[s]
Recvr_Gain	= 32
Temp_Get	= 23.2[dC]
X_90_Width	= 7.67[us]
X_Acq_Time	= 1.99874592[s]
X_Angle	= 45[deg]
X_Atn	= 7.4[dB]
X_Data_Points	= 32768
X_Points_Default	= 31282
X_Pulse	= 3.835[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 400



JEOL

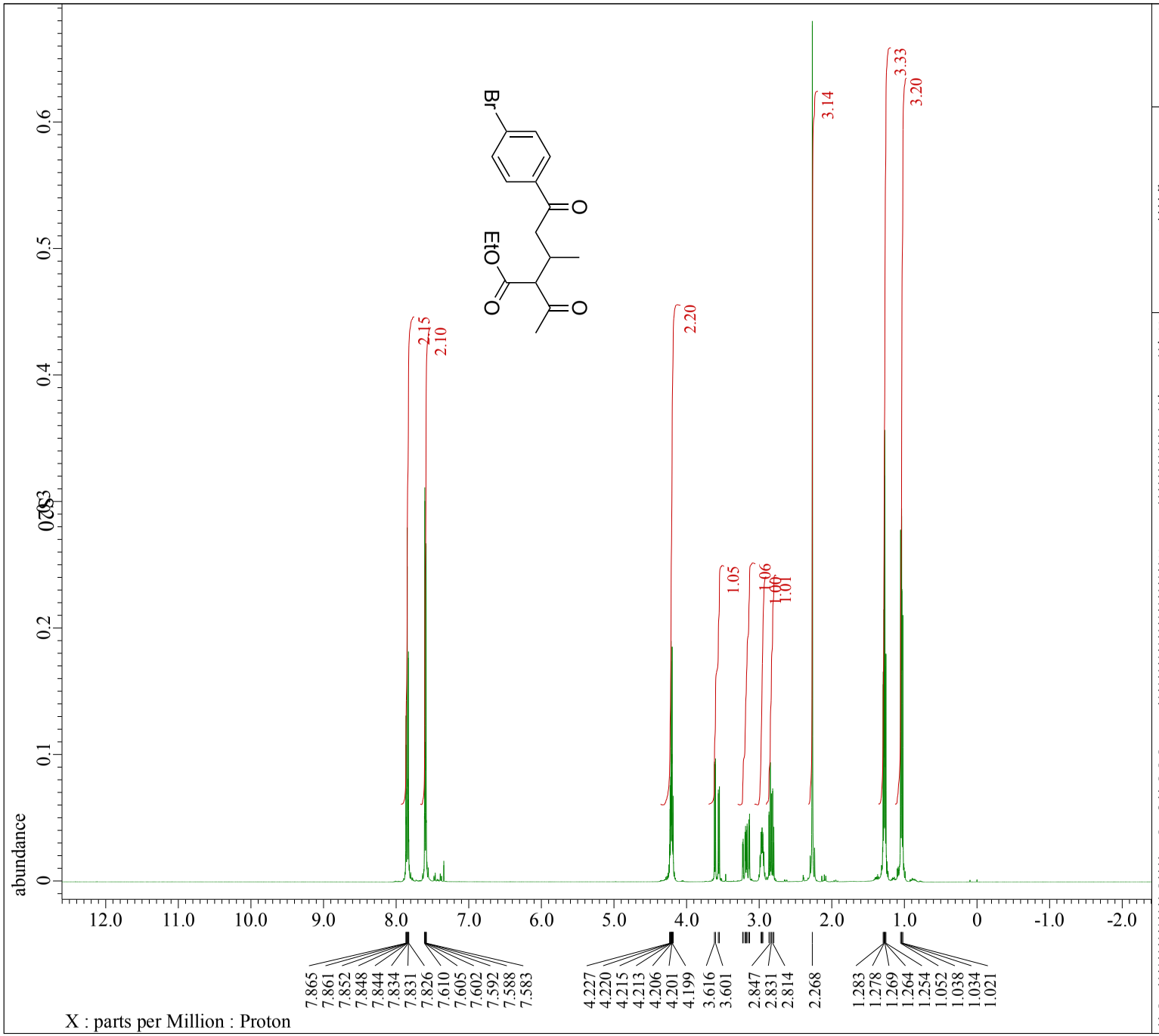
```
---- PROCESSING PARAMETERS ----
sexp( 1.00646[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 5[%], 1 )
peak_pick( 0[Hz], 0.1[ppm], Peaks, 0[Hz] )
reference( 77.12391[ppm], 77[ppm] )
```

Filename	= RO0515-purifie
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= RO0515-purifie
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 1-MAY-2024 19
Revision_Time	= 1-MAY-2024 20
Data_Format	= 1D COMPLEX
Dim_Size	= 104858
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 0.99357984[s]
X_Domain	= Carbon13
X_Freq	= 125.76529768[M
X_Offset	= 100[ppm]
X_Points	= 39303
X_Points_Input	= 31442
X_Prescans	= 4
X_Resolution	= 1.00646164[Hz]
X_Sweep	= 39.55696203[kH
X_Sweep_Clipped	= 31.64556962[kH
X_Sweep_Input	= 251.0[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Blanking	= 5[us]
Clipped	= FALSE
Scans	= 281
Total_Scans	= 281
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 23.4[dC]
X_90_Width	= 14.23[us]
X_Acq_Time	= 0.99357984[s]
X_Angle	= 30[deg]
X_Atn	= 11.5[dB]
X_Data_Points	= 32768
X_Points_Default	= 39557
X_Pulse	= 4.74333333[us]
Irr_Atn_Dec	= 23.9[dB]
Irr_Atn_Dec_Calc	= 23.9[dB]
Irr_Atn_Noe	= 23.9[dB]
Irr_Bandwidth	= 5.97826087[kHz
Irr_Bandwidth_Ppm	= 11.9526989[ppm
Irr_Corresp_Pw90	= 92[us]




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 12.5, 93, 65.55013[%] )
reference( -62.65821[ppm], -63.72[ppm] )
thresh( 64.39011[%], 1 )
reference( -63.72[ppm], -63.72[ppm] )

以下に由来：: RO0477b-purified_fluorine-1-1.jdf
```

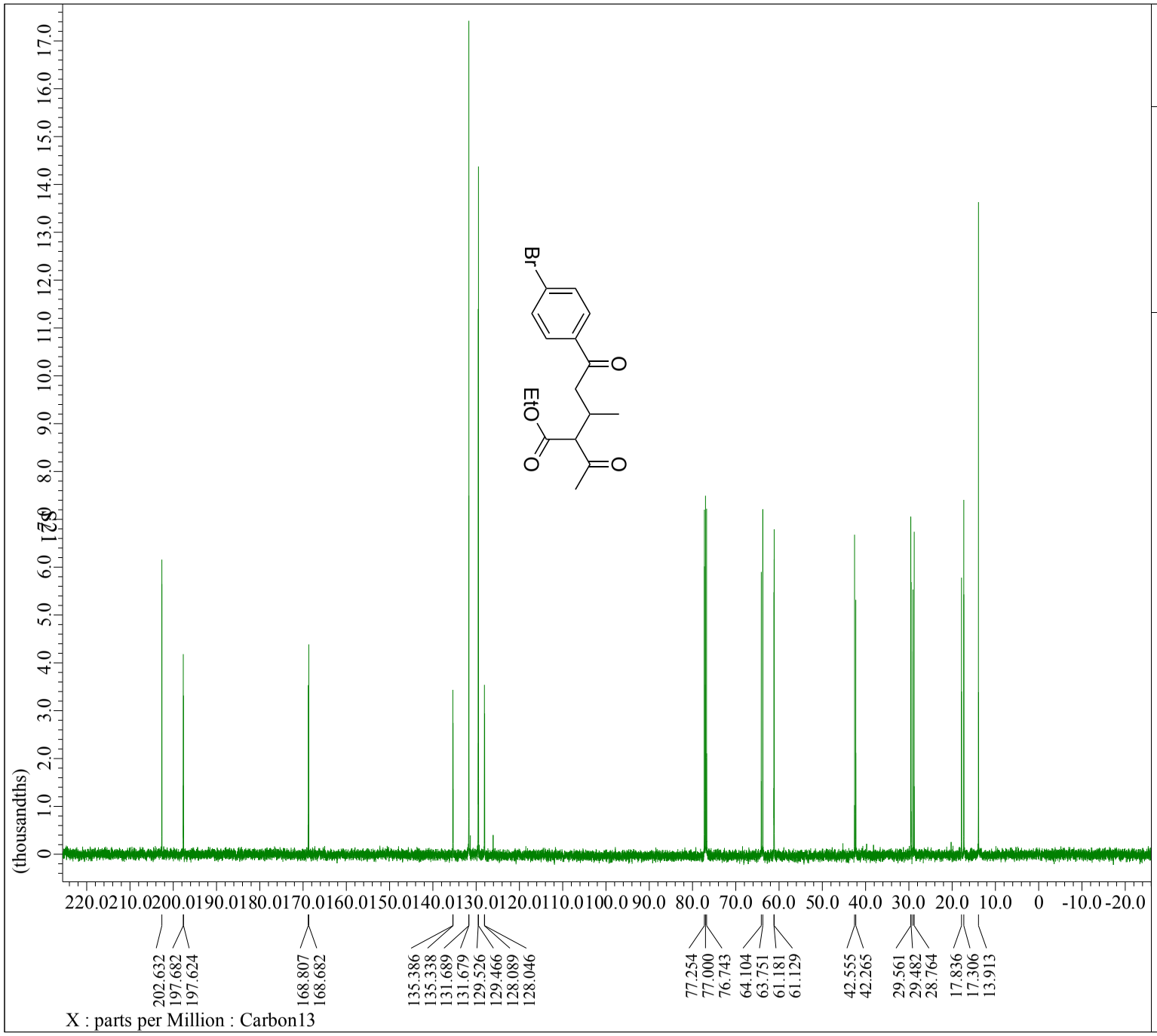
Filename	= RO0477b-purified_fluorine
Author	= delta
Experiment	= proton.jxp
Sample_Id	= RO0477b-purified
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 10-APR-2024 10:23:05
Revision_Time	= 12-APR-2024 10:51:48
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Fluori
Dim_Title	= Fluorine19
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 86.50752[ms]
X_Domain	= 19F
X_Freq	= 376.17105393[MHz]
X_Offset	= 0[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 11.55968868[Hz]
X_Sweep	= 189.39393939[kHz]
X_Sweep_Clipped	= 151.51515152[kHz]
Irr_Domain	= Fluorine19
Irr_Freq	= 376.17105393[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Fluorine19
Tri_Freq	= 376.17105393[MHz]
Tri_Offset	= 5[ppm]
Clipped	= TRUE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 50
Temp_Get	= 25[dC]
X_90_Width	= 7.95[us]
X_Acq_Time	= 86.50752[ms]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 3.975[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 5.08650752[s]



JEOL

```
---- PROCESSING PARAMETERS ----
sexp( 0.25016[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -0.76415, 8.49971, 68.86206[%] )
reference( -0.09723[ppm], 0[ppm] )
```

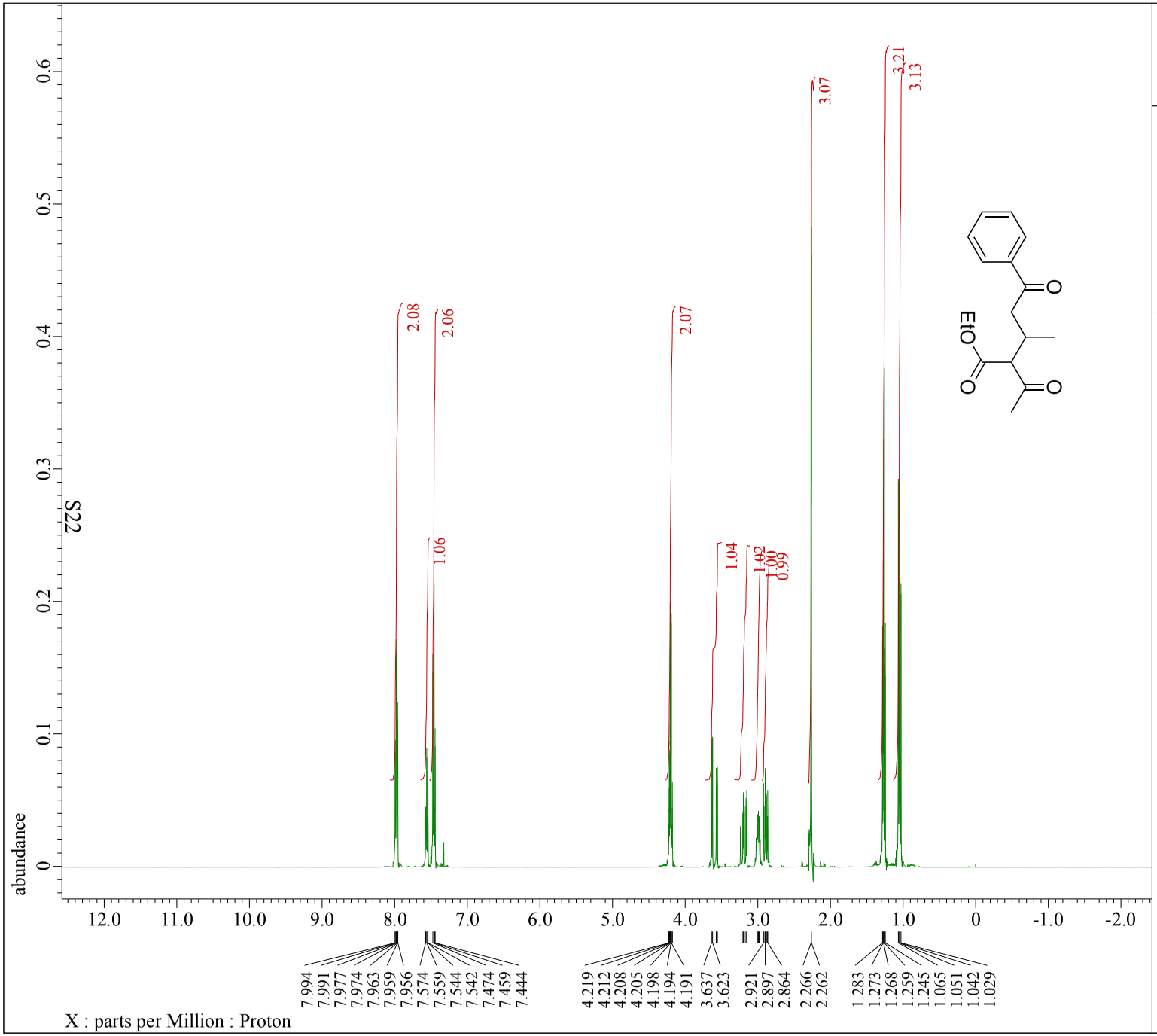
Filename	= RO0475-dry_PRO
Author	= delta
Experiment	= single_pulse.j
Sample_Id	= RO0475-dry
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 2-MAR-2024 12
Revision_Time	= 22-APR-2024 08
Data_Format	= 1D COMPLEX
Dim_Size	= 52429
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 1.99874592[s]
X_Domain	= Proton
X_Freq	= 500.15991521[M
X_Offset	= 5.0[ppm]
X_Points	= 18757
X_Points_Input	= 15005
X_Prescans	= 0
X_Resolution	= 0.50031372[Hz]
X_Sweep	= 9.38438438[kHz
X_Sweep_Clipped	= 7.50750751[kHz
X_Sweep_Input	= 15[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[M
Tri_Offset	= 5.0[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 4[s]
Recvr_Gain	= 32
Temp_Get	= 21.8[dC]
X_90_Width	= 7.67[us]
X_Acq_Time	= 1.99874592[s]
X_Angle	= 45[deg]
X_Atn	= 7.4[dB]
X_Data_Points	= 32768
X_Points_Default	= 31282
X_Pulse	= 3.835[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 400



JEOL

```
---- PROCESSING PARAMETERS ----
sexp( 1.00646[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 5[%], 1 )
peak_pick( 0[Hz], 0.1[ppm], Peaks, 0[Hz] )
reference( 77.24869[ppm], 77[ppm] )
```

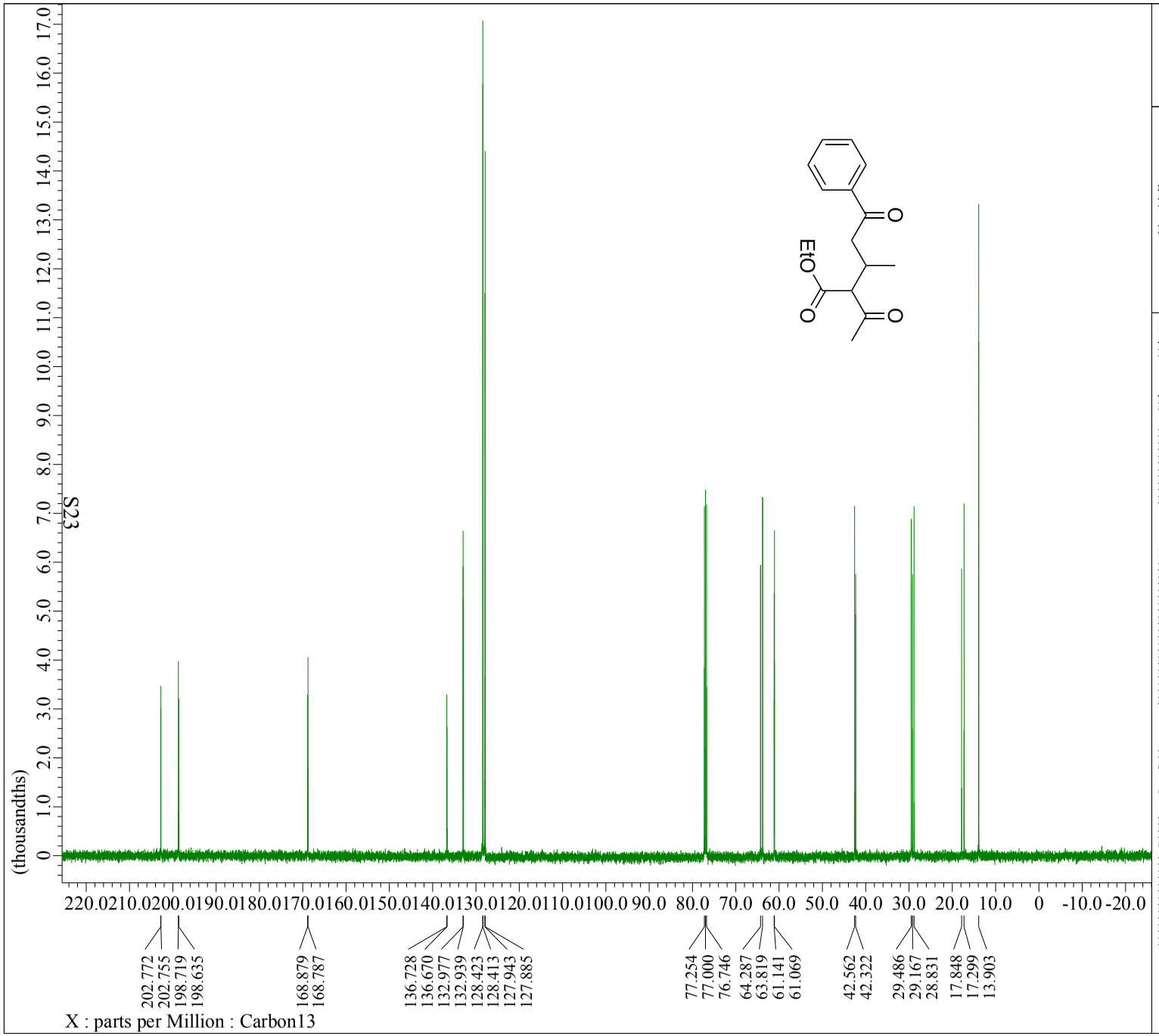
Filename	= RO0475-dry_CAR
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= RO0475-dry
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 2-MAR-2024 12
Revision_Time	= 12-APR-2024 15
Data_Format	= 1D COMPLEX
Dim_Size	= 104858
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 0.99357984[s]
X_Domain	= Carbon13
X_Freq	= 125.76529768[M
X_Offset	= 100[ppm]
X_Points	= 39303
X_Points_Input	= 31442
X_Prescans	= 4
X_Resolution	= 1.00646164[Hz]
X_Sweep	= 39.55696203[kH
X_Sweep_Clipped	= 31.64556962[kH
X_Sweep_Input	= 251.0[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Blanking	= 5.0[us]
Clipped	= FALSE
Scans	= 103
Total_Scans	= 103
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 21.8[dC]
X_90_Width	= 12.19[us]
X_Acq_Time	= 0.99357984[s]
X_Angle	= 30[deg]
X_Atn	= 11.5[dB]
X_Data_Points	= 32768
X_Points_Default	= 39557
X_Pulse	= 4.06333333[us]
Irr_Atn_Dec	= 29[dB]
Irr_Atn_Dec_Calc	= 29[dB]
Irr_Atn_Noe	= 29[dB]
Irr_Bandwidth	= 5.97826087[kHz
Irr_Bandwidth_Ppm	= 11.9526989[ppm
Irr_Corresp_Pw90	= 92[us]



JEOL

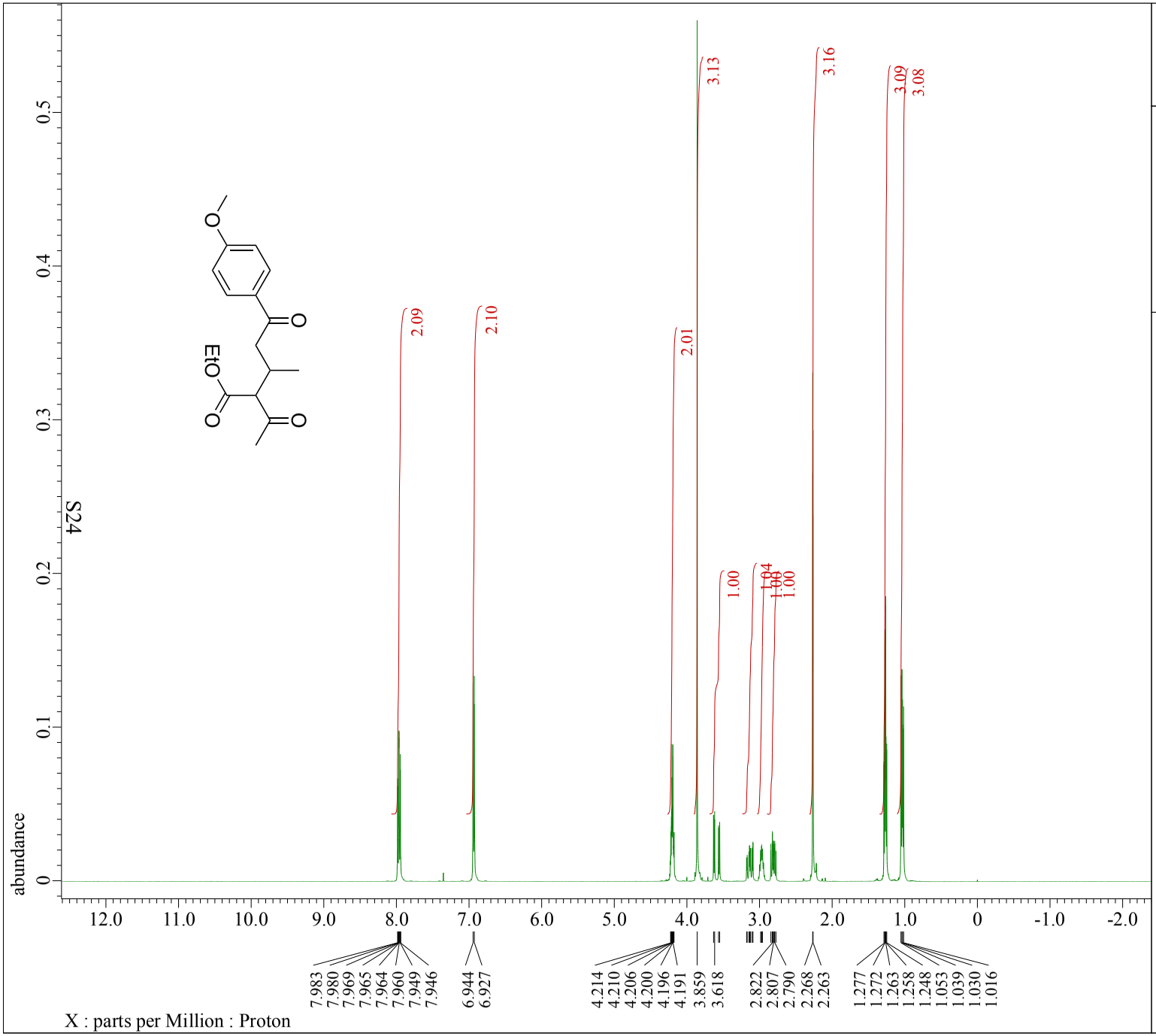
```
---- PROCESSING PARAMETERS ----
sexp( 0.25016[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -0.39695, 3.38065, 68.73045[%] )
reference( -0.07748[ppm], 0[ppm] )
```

Filename	= RO0471-dry_PRO
Author	= delta
Experiment	= single_pulse.j
Sample_Id	= RO0471-dry
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 2-MAR-2024 12
Revision_Time	= 22-APR-2024 07
Data_Format	= 1D COMPLEX
Dim_Size	= 52429
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 1.99874592[s]
X_Domain	= Proton
X_Freq	= 500.15991521[M
X_Offset	= 5.0[ppm]
X_Points	= 18757
X_Points_Input	= 15005
X_Prescans	= 0
X_Resolution	= 0.50031372[Hz]
X_Sweep	= 9.38438438[kHz
X_Sweep_Clipped	= 7.50750751[kHz
X_Sweep_Input	= 15[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[M
Tri_Offset	= 5.0[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 4[s]
Recvr_Gain	= 32
Temp_Get	= 21.8[dC]
X_90_Width	= 7.67[us]
X_Acq_Time	= 1.99874592[s]
X_Angle	= 45[deg]
X_Atn	= 7.4[dB]
X_Data_Points	= 32768
X_Points_Default	= 31282
X_Pulse	= 3.835[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 400



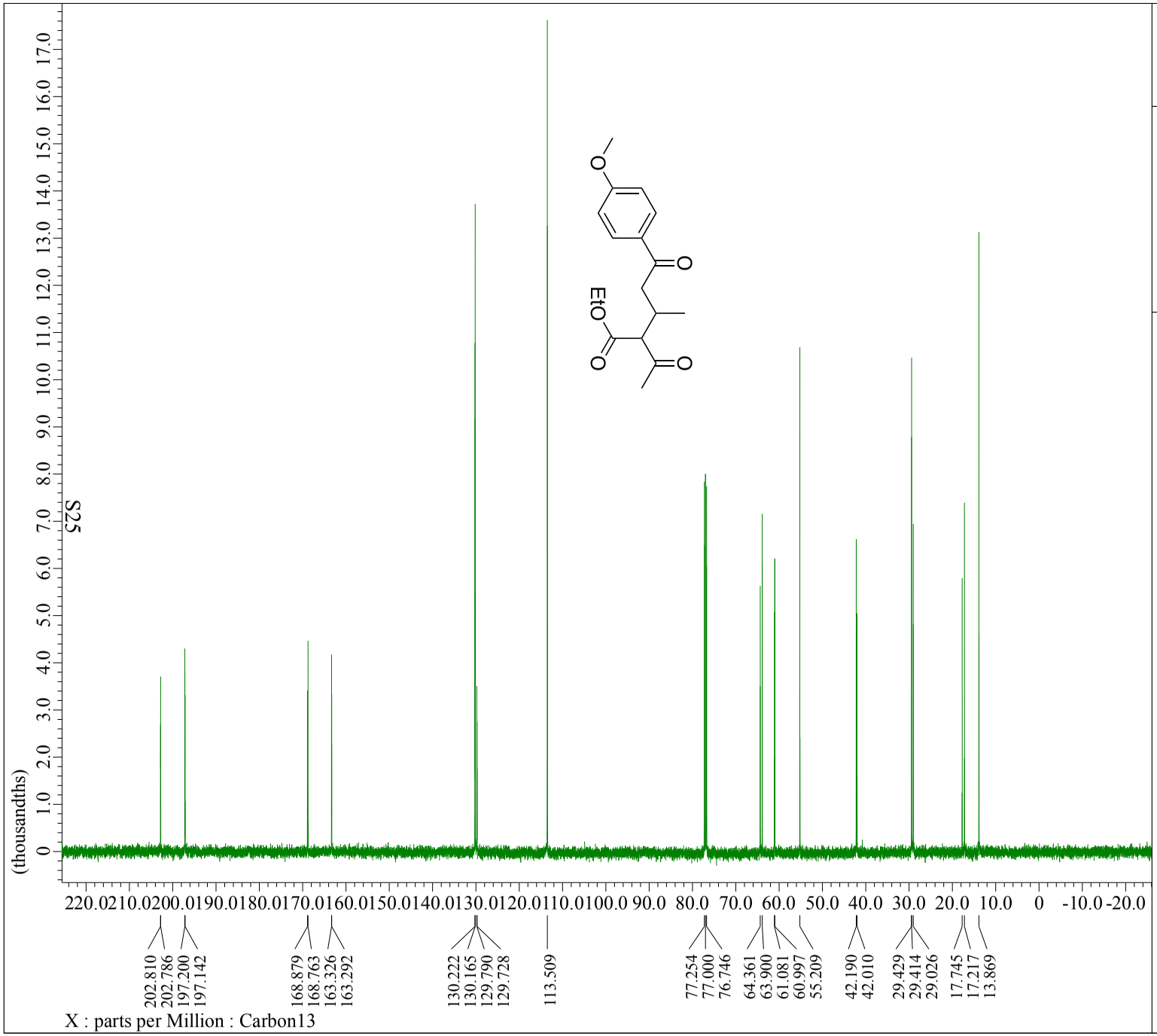

```
---- PROCESSING PARAMETERS ----
sexp( 1.00646[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 5[%], 1 )
peak_pick( 0[Hz], 0.1[ppm], Peaks, 0[Hz] )
reference( 77.24389[ppm], 77[ppm] )
```

Filename	= RO0471-dry_CAR
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= RO0471-dry
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 2-MAR-2024 12
Revision_Time	= 2-MAR-2024 22
Data_Format	= 1D COMPLEX
Dim_Size	= 104858
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 0.99357984[s]
X_Domain	= Carbon13
X_Freq	= 125.76529768[M
X_Offset	= 100[ppm]
X_Points	= 39303
X_Points_Input	= 31442
X_Prescans	= 4
X_Resolution	= 1.00646164[Hz]
X_Sweep	= 39.55696203[kH
X_Sweep_Clipped	= 31.64556962[kH
X_Sweep_Input	= 251.0[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Blanking	= 5.0[us]
Clipped	= FALSE
Scans	= 123
Total_Scans	= 123
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 21.8[dC]
X_90_Width	= 12.19[us]
X_Acq_Time	= 0.99357984[s]
X_Angle	= 30[deg]
X_Atn	= 11.5[dB]
X_Data_Points	= 32768
X_Points_Default	= 39557
X_Pulse	= 4.06333333[us]
Irr_Atn_Dec	= 29[dB]
Irr_Atn_Dec_Calc	= 29[dB]
Irr_Atn_Noe	= 29[dB]
Irr_Bandwidth	= 5.97826087[kHz
Irr_Bandwidth_Ppm	= 11.9526989[ppm
Irr_Corresp_Pw90	= 92[us]




```
---- PROCESSING PARAMETERS ----
sexp( 0.25016[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 0.12506, 6.75547, 58.32189[%] )
reference( -0.10668[ppm], 0[ppm] )
```

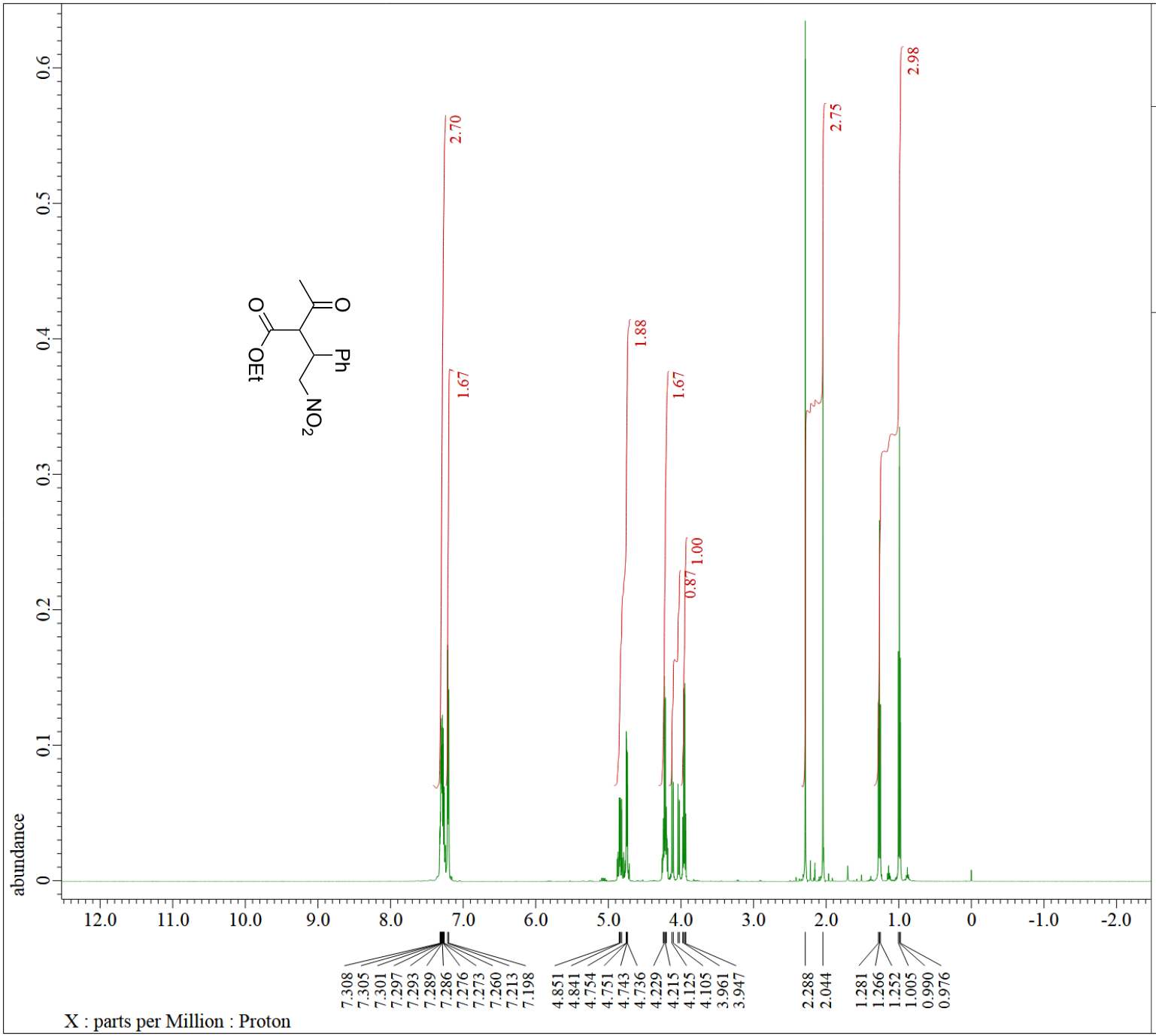
Filename	= RO0502-purifie
Author	= delta
Experiment	= single_pulse.j
Sample_Id	= RO0502-purifie
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 17-APR-2024 19
Revision_Time	= 22-APR-2024 17
Data_Format	= 1D COMPLEX
Dim_Size	= 52429
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 1.99874592[s]
X_Domain	= Proton
X_Freq	= 500.15991521[M
X_Offset	= 5.0[ppm]
X_Points	= 18757
X_Points_Input	= 15005
X_Prescans	= 0
X_Resolution	= 0.50031372[Hz]
X_Sweep	= 9.38438438[kHz
X_Sweep_Clipped	= 7.50750751[kHz
X_Sweep_Input	= 15[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[M
Tri_Offset	= 5.0[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 4[s]
Recvr_Gain	= 32
Temp_Get	= 22.9[dC]
X_90_Width	= 14[us]
X_Acq_Time	= 1.99874592[s]
X_Angle	= 45[deg]
X_Atn	= 8[dB]
X_Data_Points	= 32768
X_Points_Default	= 31282
X_Pulse	= 7[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 400



JEOL

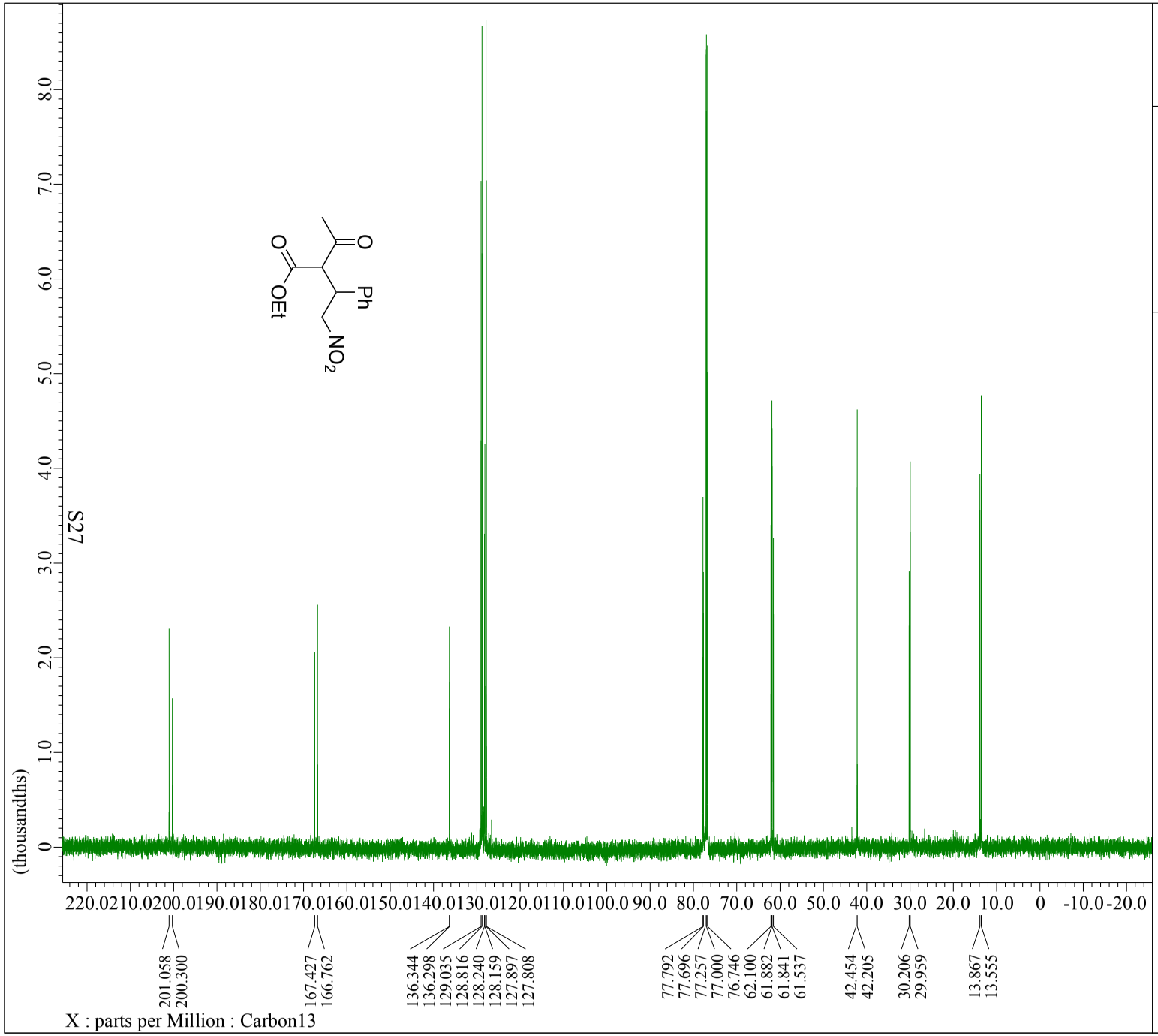
```
---- PROCESSING PARAMETERS ----
sexp( 1.00646[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 5[%], 1 )
peak_pick( 0[Hz], 0.1[ppm], Peaks, 0[Hz] )
reference( 77.25829[ppm], 0[ppm] )
reference( 0.0[ppm], 77[ppm] )

以下に由来：: RO0474-dry_CARBON-1-1.jdf
```

Filename	= RO0474-dry_CAR
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= RO0474-dry
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 1-MAR-2024 17
Revision_Time	= 13-APR-2024 18
Data_Format	= 1D COMPLEX
Dim_Size	= 104858
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 0.99357984[s]
X_Domain	= Carbon13
X_Freq	= 125.76529768[M
X_Offset	= 100[ppm]
X_Points	= 39303
X_Points_Input	= 31442
X_Prescans	= 4
X_Resolution	= 1.00646164[Hz]
X_Sweep	= 39.55696203[kH
X_Sweep_Clipped	= 31.64556962[kH
X_Sweep_Input	= 251.0[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Blanking	= 5.0[us]
Clipped	= FALSE
Scans	= 109
Total_Scans	= 109
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 22.1[dC]
X_90_Width	= 12.19[us]
X_Acq_Time	= 0.99357984[s]
X_Angle	= 30[deg]
X_Atn	= 11.5[dB]
X_Data_Points	= 32768
X_Points_Default	= 39557
X_Pulse	= 4.06333333[us]
Irr_Atn_Dec	= 29[dB]
Irr_Atn_Dec_Calc	= 29[dB]
Irr_Atn_Noe	= 29[dB]
Irr_Bandwidth	= 5.97826087[kHz
Irr_Bandwidth_Ppm	= 11.9526989[ppm
Irr_Corresp_Pw90	= 92[us]




```
---- PROCESSING PARAMETERS ----
sexp( 0.25016[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( -0.02595[ppm], 0[ppm] )
thresh( 5.92287[%], 1 )

以下に由来：: RO0491-purified-3_PROTON-1-1.jdf
```

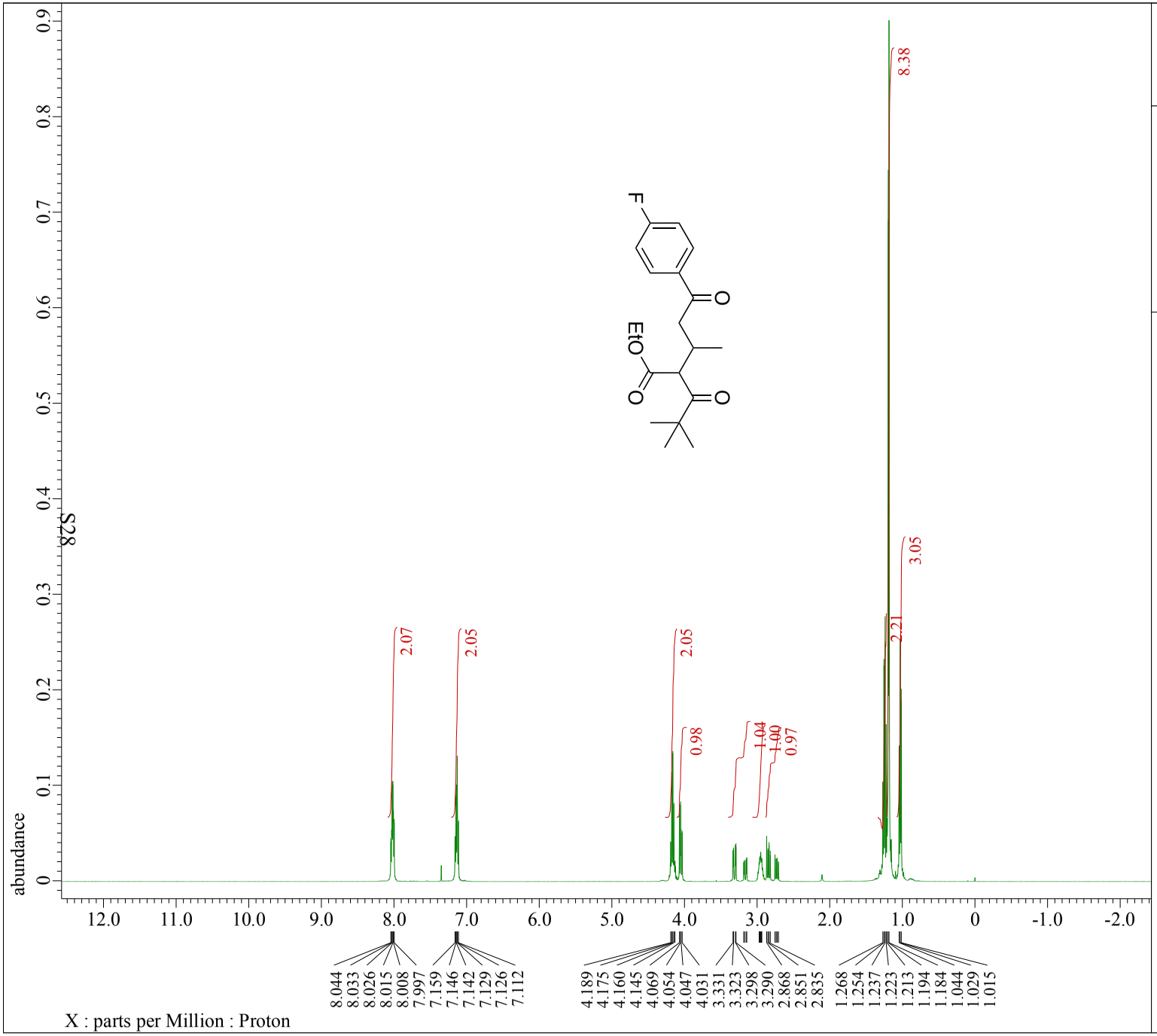
Filename	= RO0491-purifie
Author	= delta
Experiment	= single_pulse.j
Sample_Id	= RO0491-purifie
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 10-MAY-2024 15
Revision_Time	= 11-MAY-2024 18
Data_Format	= 1D COMPLEX
Dim_Size	= 52429
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 1.99874592[s]
X_Domain	= Proton
X_Freq	= 500.15991521[M
X_Offset	= 5.0[ppm]
X_Points	= 18757
X_Points_Input	= 15005
X_Prescans	= 0
X_Resolution	= 0.50031372[Hz]
X_Sweep	= 9.38438438[kHz
X_Sweep_Clipped	= 7.50750751[kHz
X_Sweep_Input	= 15[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[M
Tri_Offset	= 5.0[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 4[s]
Recvr_Gain	= 42
Temp_Get	= 22.8[dC]
X_90_Width	= 14.8[us]
X_Acq_Time	= 1.99874592[s]
X_Angle	= 45[deg]
X_Atn	= 8[dB]
X_Data_Points	= 32768
X_Points_Default	= 31282
X_Pulse	= 7.4[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 400



JEOL

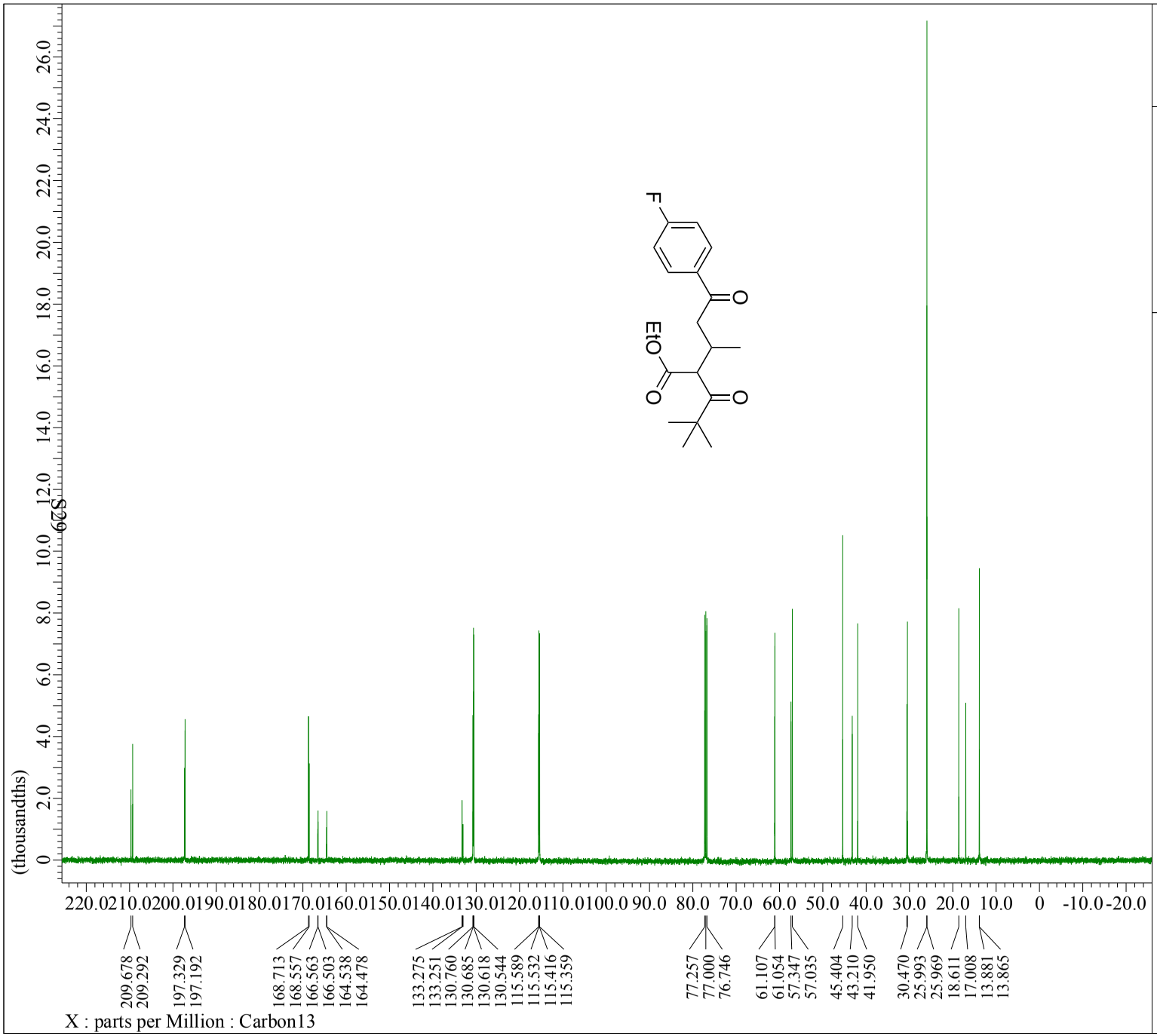
```
---- PROCESSING PARAMETERS ----
sexp( 1.00646[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 5[%], 1 )
peak_pick( 0[Hz], 0.1[ppm], Peaks, 0[Hz] )
reference( 77.1911[ppm], 77[ppm] )
thresh( 7.72221[%], 6.33611[%] )

以下に由来：: RO0484-dry2_CARBON-1-1.jdf
```

Filename	= RO0484-dry2_CA
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= RO0484-dry2
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 13-MAR-2024 09
Revision_Time	= 12-APR-2024 16
Data_Format	= 1D COMPLEX
Dim_Size	= 104858
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 0.99357984[s]
X_Domain	= Carbon13
X_Freq	= 125.76529768[M
X_Offset	= 100[ppm]
X_Points	= 39303
X_Points_Input	= 31442
X_Prescans	= 4
X_Resolution	= 1.00646164[Hz]
X_Sweep	= 39.55696203[kH
X_Sweep_Clipped	= 31.64556962[kH
X_Sweep_Input	= 251.0[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Blanking	= 5.0[us]
Clipped	= FALSE
Scans	= 256
Total_Scans	= 256
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 22[dC]
X_90_Width	= 12.19[us]
X_Acq_Time	= 0.99357984[s]
X_Angle	= 30[deg]
X_Atn	= 11.5[dB]
X_Data_Points	= 32768
X_Points_Default	= 39557
X_Pulse	= 4.06333333[us]
Irr_Atn_Dec	= 29[dB]
Irr_Atn_Dec_Calc	= 29[dB]
Irr_Atn_Noe	= 29[dB]
Irr_Bandwidth	= 5.97826087[kHz
Irr_Bandwidth_Ppm	= 11.9526989[ppm
Irr_Corresp_Pw90	= 92[us]



---- PROCESSING PARAMETERS ----
sexp(0.25016[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(2, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
reference(-0.07261[ppm], 0[ppm])
thresh(2.2627[%], 1)

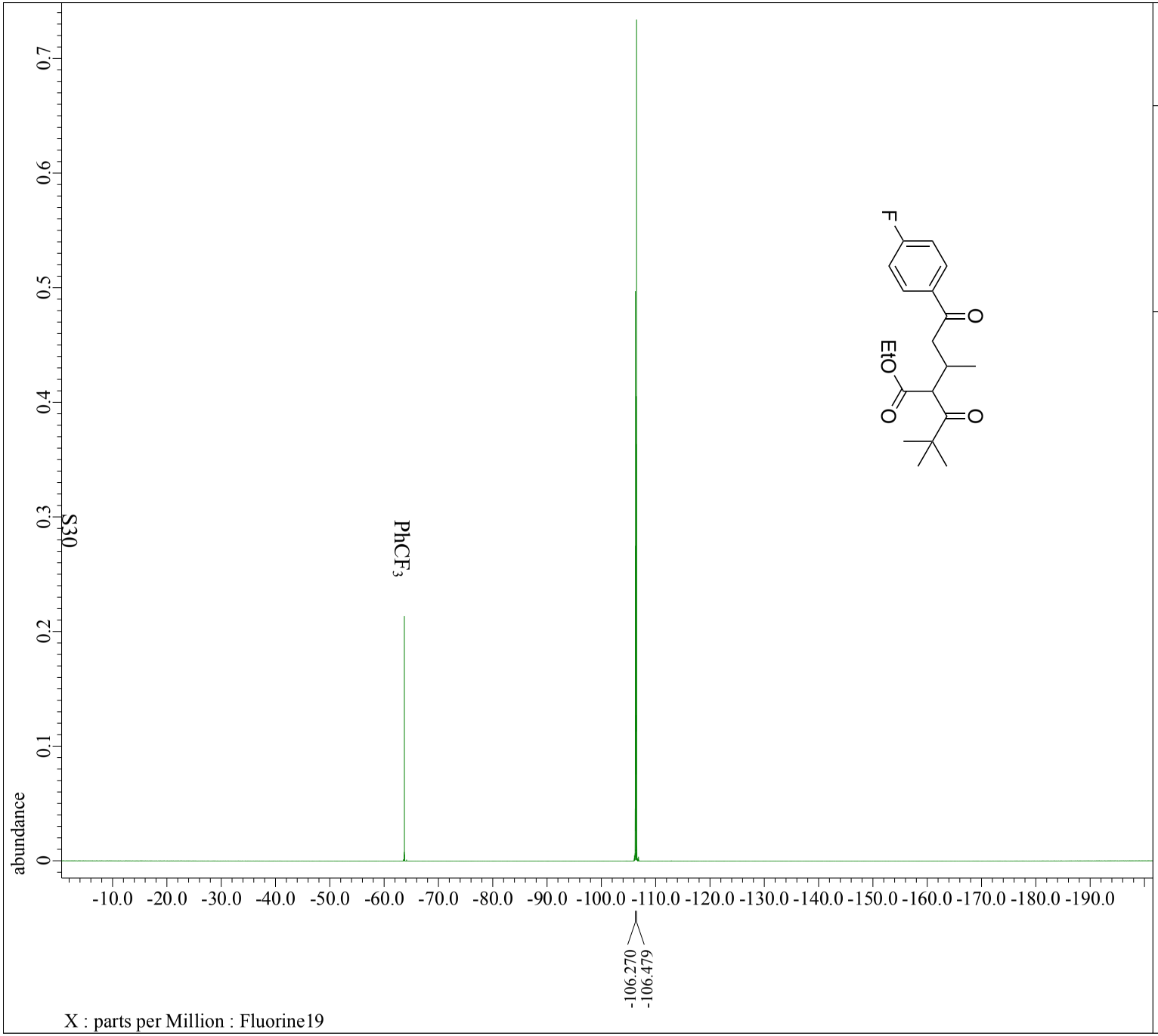
Filename	= RO0480-dry_PRO
Author	= delta
Experiment	= single_pulse.j
Sample_Id	= RO0480-dry
Solvent	= NONE
Actual_Start_Time	= 1-MAR-2024 10
Revision_Time	= 23-APR-2024 20
Data_Format	= 1D COMPLEX
Dim_Size	= 52429
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 1.99874592[s]
X_Domain	= Proton
X_Freq	= 500.15991521[M
X_Offset	= 5.0[ppm]
X_Points	= 18757
X_Points_Input	= 15005
X_Prescans	= 0
X_Resolution	= 0.50031372[Hz]
X_Sweep	= 9.38438438[kHz
X_Sweep_Clipped	= 7.50750751[kHz
X_Sweep_Input	= 15[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[M
Tri_Offset	= 5.0[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 4[s]
Recvr_Gain	= 32
Temp_Get	= 22[dC]
X_90_Width	= 7.67[us]
X_Acq_Time	= 1.99874592[s]
X_Angle	= 45[deg]
X_Atn	= 7.4[dB]
X_Data_Points	= 32768
X_Points_Default	= 31282
X_Pulse	= 3.835[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 400



JEOL

```
---- PROCESSING PARAMETERS ----
sexp( 1.00646[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 5[%], 1 )
peak_pick( 0[Hz], 0.1[ppm], Peaks, 0[Hz] )
reference( 77.1983[ppm], 77[ppm] )
reference( 221.35714[ppm], 77[ppm] )
reference( -67.3363[ppm], 77[ppm] )
thresh( 3.05154[%], 1 )
```

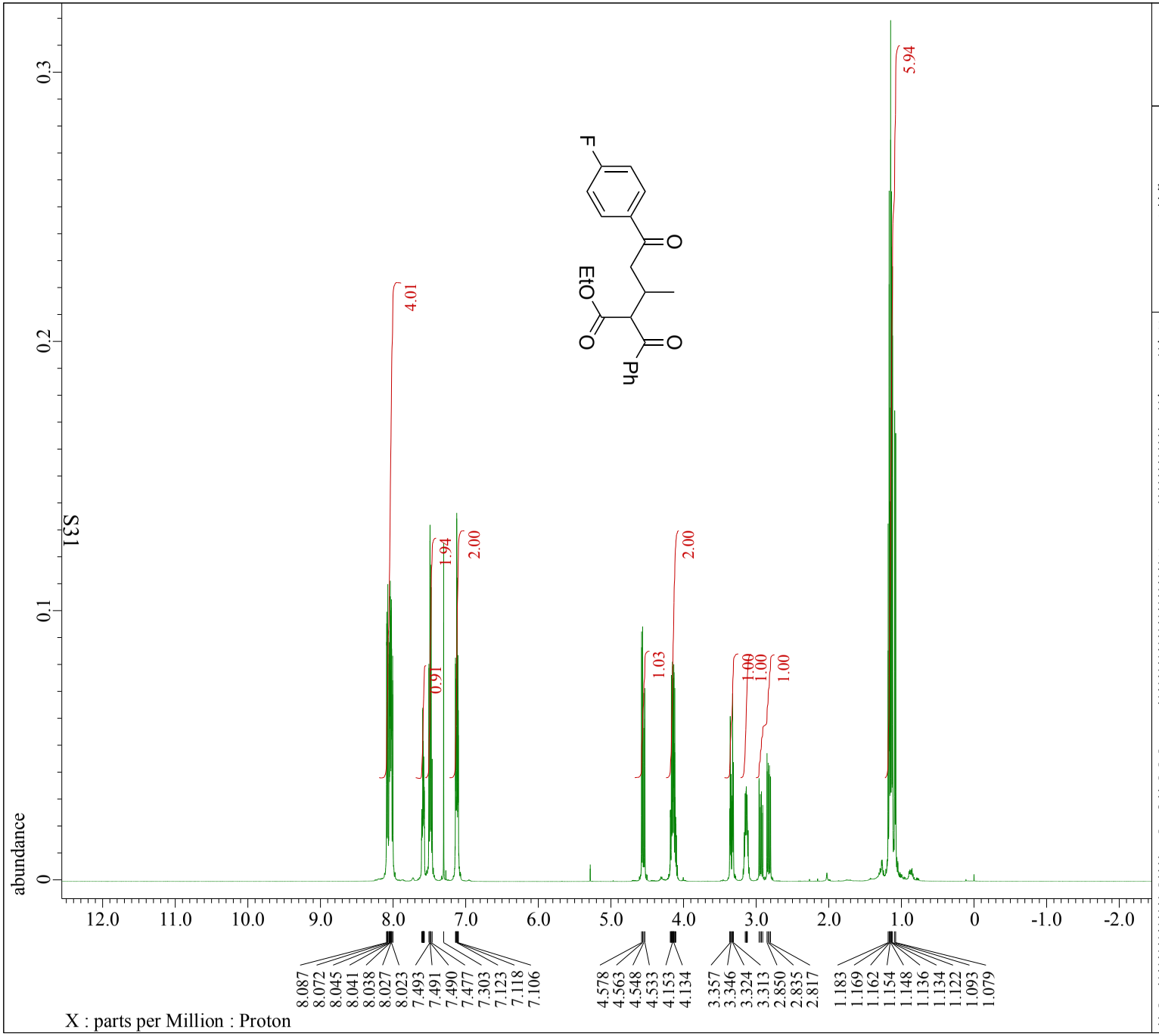
Filename	= RO0480-dry_CAR
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= RO0480-dry
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 1-MAR-2024 19
Revision_Time	= 12-APR-2024 15
Data_Format	= 1D COMPLEX
Dim_Size	= 104858
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 0.99357984[s]
X_Domain	= Carbon13
X_Freq	= 125.76529768[M
X_Offset	= 100[ppm]
X_Points	= 39303
X_Points_Input	= 31442
X_Prescans	= 4
X_Resolution	= 1.00646164[Hz]
X_Sweep	= 39.55696203[kH
X_Sweep_Clipped	= 31.64556962[kH
X_Sweep_Input	= 251.0[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Blanking	= 5.0[us]
Clipped	= FALSE
Scans	= 201
Total_Scans	= 201
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 22.1[dC]
X_90_Width	= 12.19[us]
X_Acq_Time	= 0.99357984[s]
X_Angle	= 30[deg]
X_Atn	= 11.5[dB]
X_Data_Points	= 32768
X_Points_Default	= 39557
X_Pulse	= 4.06333333[us]
Irr_Atn_Dec	= 29[dB]
Irr_Atn_Dec_Calc	= 29[dB]
Irr_Atn_Noe	= 29[dB]
Irr_Bandwidth	= 5.97826087[kHz
Irr_Bandwidth_Ppm	= 11.9526989[ppm
Irr_Corresp_Pw90	= 92[us]



---- PROCESSING PARAMETERS ----
sexp(0.33488[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(2, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
phase(0.16268, 9.3979, 52.68674[%])
reference(-62.63939[ppm], -63.72[ppm])
thresh(64.14036[%], 1)

以下に由来：: RO0458-purified-2_Fluorine-1-1.jdf

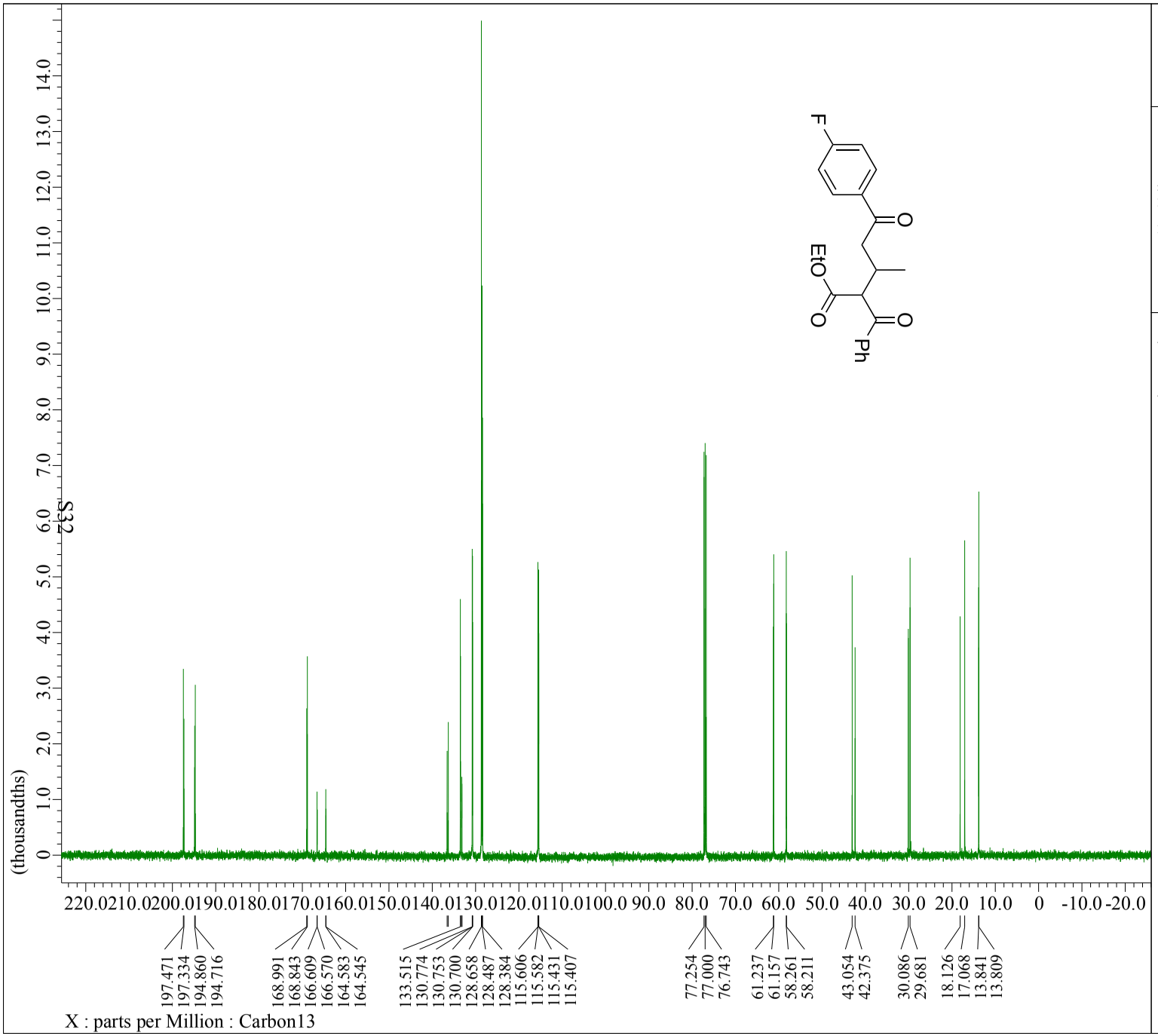
Filename	= RO0458-purifie
Author	= delta
Experiment	= single_pulse.j
Sample_Id	= RO0458-purifie
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 22-FEB-2024 10
Revision_Time	= 23-APR-2024 20
Data_Format	= 1D COMPLEX
Dim_Size	= 838861
X_Domain	= Fluorine19
Dim_Title	= Fluorine19
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 2.98610082[s]
X_Domain	= Fluorine19
X_Freq	= 470.62046084[M
X_Offset	= -100[ppm]
X_Points	= 352967
X_Points_Input	= 282373
X_Prescans	= 0
X_Resolution	= 0.33488488[Hz]
X_Sweep	= 118.20330969[k
X_Sweep_Clipped	= 94.56264775[kH
X_Sweep_Input	= 200[ppm]
Irr_Domain	= Fluorine19
Irr_Freq	= 470.62046084[M
Irr_Offset	= 5[ppm]
Tri_Domain	= Fluorine19
Tri_Freq	= 470.62046084[M
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 4[s]
Recvr_Gain	= 62
Temp_Get	= 23.2[dC]
X_90_Width	= 7.45[us]
X_Acq_Time	= 2.98610082[s]
X_Angle	= 45[deg]
X_Atn	= 7[dB]
X_Data_Points	= 65536
X_Points_Default	= 65536
X_Pulse	= 3.725[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 400



JEOL

```
---- PROCESSING PARAMETERS ----
sexp( 0.25016[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( -0.05715[ppm], 0[ppm] )
thresh( 6.82713[%], 1 )
```

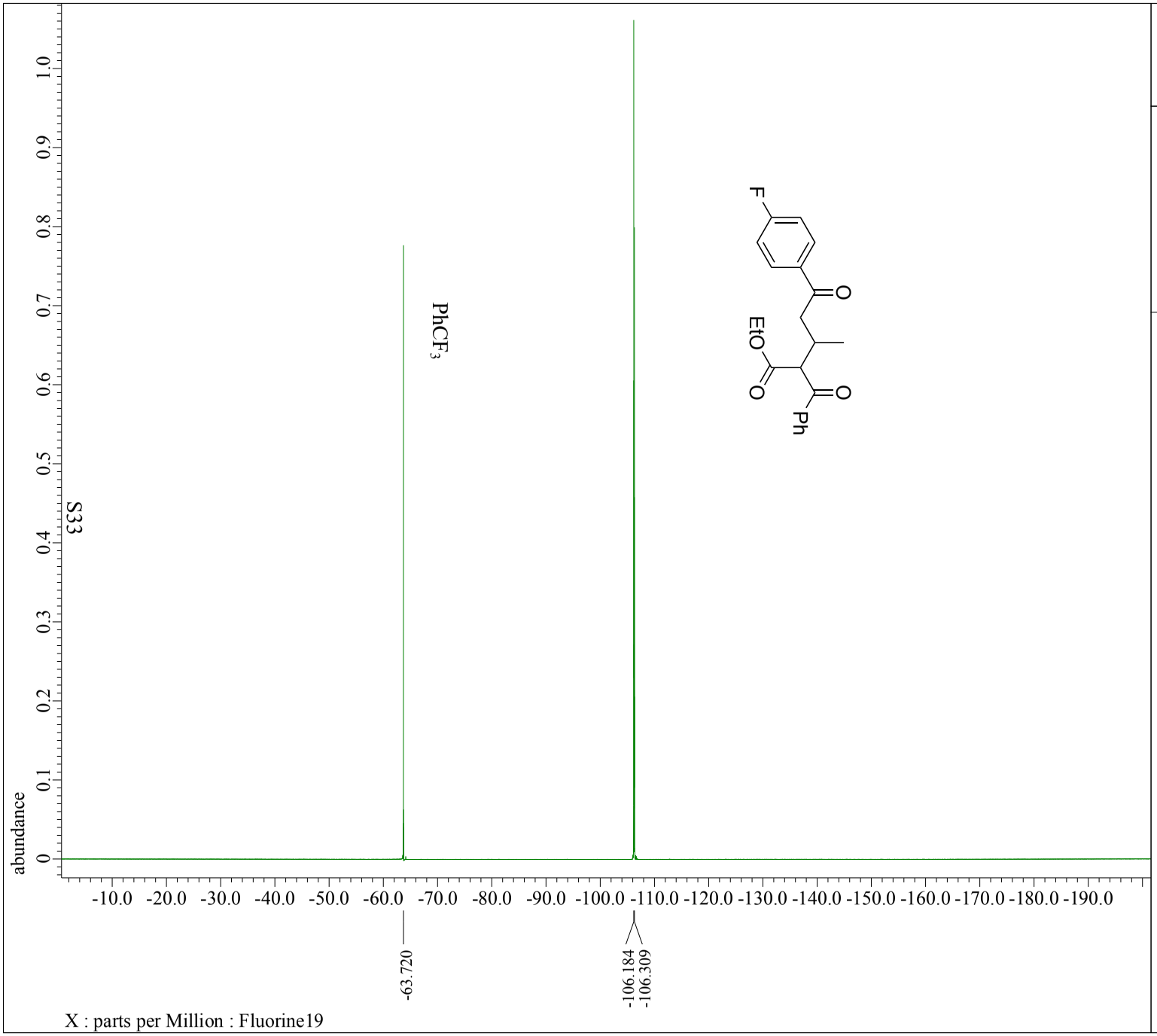
Filename	= RO0457-purifie
Author	= delta
Experiment	= single_pulse.j
Sample_Id	= RO0457-purifie
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 7-MAR-2024 14
Revision_Time	= 22-APR-2024 07
Data_Format	= 1D COMPLEX
Dim_Size	= 52429
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 1.99874592[s]
X_Domain	= Proton
X_Freq	= 500.15991521[M
X_Offset	= 5.0[ppm]
X_Points	= 18757
X_Points_Input	= 15005
X_Prescans	= 0
X_Resolution	= 0.50031372[Hz]
X_Sweep	= 9.38438438[kHz
X_Sweep_Clipped	= 7.50750751[kHz
X_Sweep_Input	= 15[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[M
Tri_Offset	= 5.0[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 4[s]
Recvr_Gain	= 32
Temp_Get	= 22.2[dC]
X_90_Width	= 7.67[us]
X_Acq_Time	= 1.99874592[s]
X_Angle	= 45[deg]
X_Atn	= 7.4[dB]
X_Data_Points	= 32768
X_Points_Default	= 31282
X_Pulse	= 3.835[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 400



---- PROCESSING PARAMETERS ----
sexp(1.00646[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(2, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
thresh(5[%], 1)
peak_pick(0[Hz], 0.1[ppm], Peaks, 0[Hz])
reference(77.2199[ppm], 77[ppm])
thresh(4.97668[%], 3.59869[%])

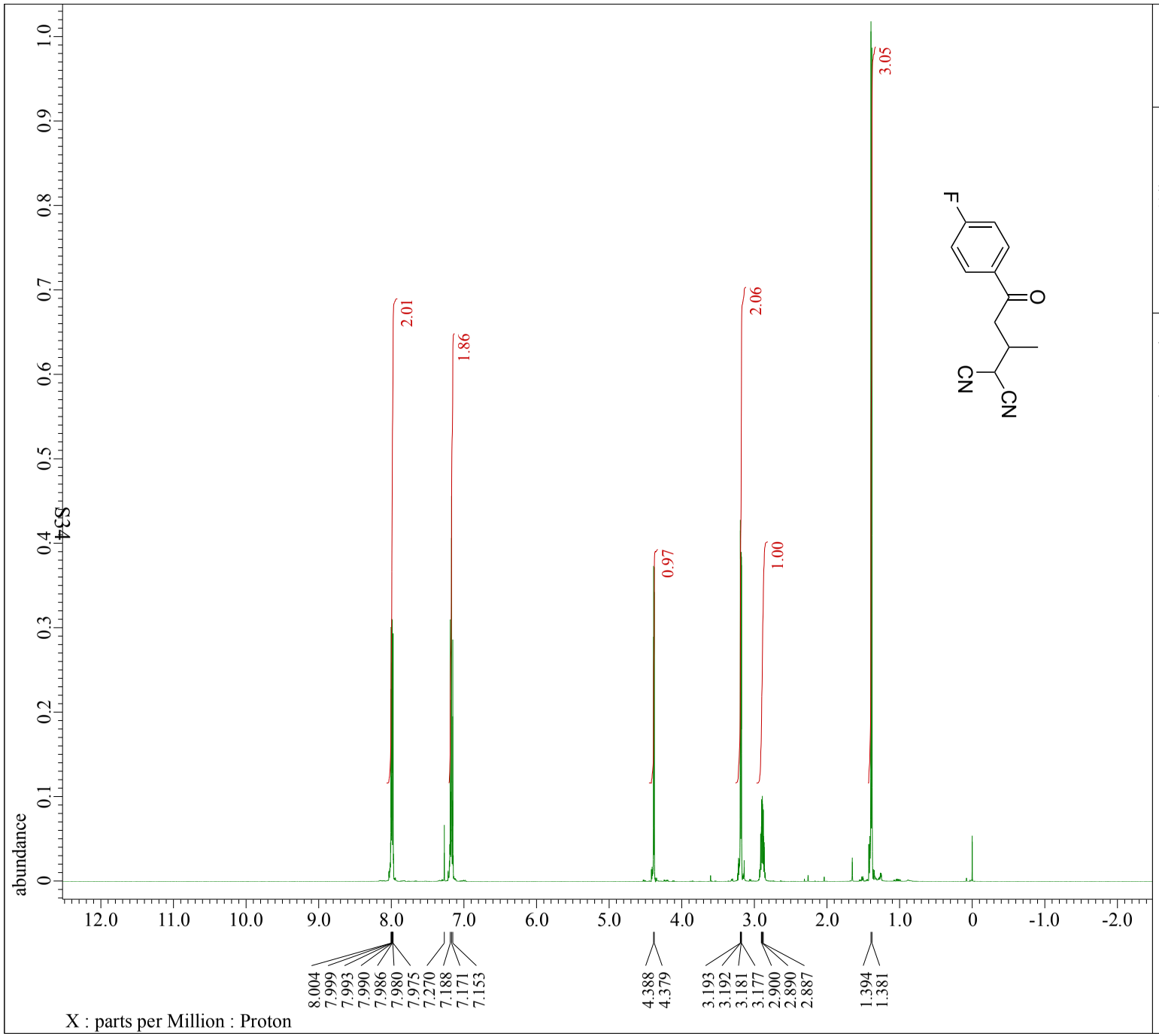
以下に由来：: RO0457-dry-new_CARBON-1-1.jdf

Filename	= RO0457-dry-new
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= RO0457-dry-new
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 8-MAR-2024 19
Revision_Time	= 12-APR-2024 14
Data_Format	= 1D COMPLEX
Dim_Size	= 104858
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 0.99357984[s]
X_Domain	= Carbon13
X_Freq	= 125.76529768[M
X_Offset	= 100[ppm]
X_Points	= 39303
X_Points_Input	= 31442
X_Prescans	= 4
X_Resolution	= 1.00646164[Hz]
X_Sweep	= 39.55696203[kH
X_Sweep_Clipped	= 31.64556962[kH
X_Sweep_Input	= 251.0[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Blanking	= 5.0[us]
Clipped	= FALSE
Scans	= 256
Total_Scans	= 256
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 22.1[dC]
X_90_Width	= 12.19[us]
X_Acq_Time	= 0.99357984[s]
X_Angle	= 30[deg]
X_Atn	= 11.5[dB]
X_Data_Points	= 32768
X_Points_Default	= 39557
X_Pulse	= 4.06333333[us]
Irr_Atn_Dec	= 29[dB]
Irr_Atn_Dec_Calc	= 29[dB]
Irr_Atn_Noe	= 29[dB]
Irr_Bandwidth	= 5.97826087[kHz
Irr_Bandwidth_Ppm	= 11.9526989[ppm
Irr_Corresp_Pw90	= 92[us]



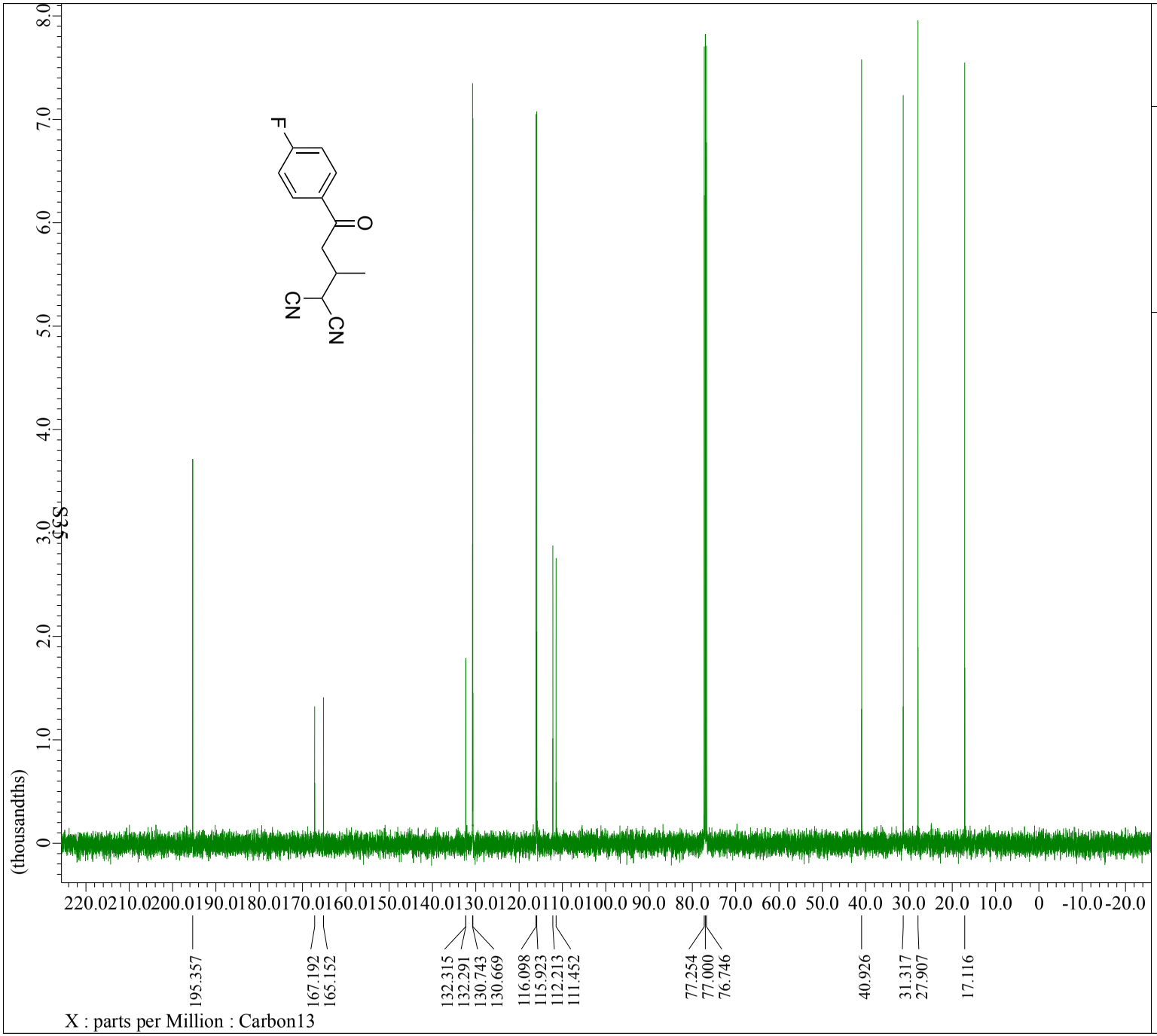

```
---- PROCESSING PARAMETERS ----
sexp( 0.33488[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( -62.59676[ppm], -63.72[ppm] )
thresh( 62.17139[%], 1 )

以下に由来：: RO0457-purified-2_Fluorine-1-1.jdf
```

Filename	= RO0457-purifie
Author	= delta
Experiment	= single_pulse.j
Sample_Id	= RO0457-purifie
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 22-FEB-2024 10
Revision_Time	= 26-MAR-2024 12
Data_Format	= 1D COMPLEX
Dim_Size	= 838861
X_Domain	= Fluorine19
Dim_Title	= Fluorine19
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 2.98610082[s]
X_Domain	= Fluorine19
X_Freq	= 470.62046084[M
X_Offset	= -100[ppm]
X_Points	= 352967
X_Points_Input	= 282373
X_Prescans	= 0
X_Resolution	= 0.33488488[Hz]
X_Sweep	= 118.20330969[k
X_Sweep_Clipped	= 94.56264775[kH
X_Sweep_Input	= 200[ppm]
Irr_Domain	= Fluorine19
Irr_Freq	= 470.62046084[M
Irr_Offset	= 5[ppm]
Tri_Domain	= Fluorine19
Tri_Freq	= 470.62046084[M
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= TRUE
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 4[s]
Recvr_Gain	= 72
Temp_Get	= 23.1[dC]
X_90_Width	= 7.45[us]
X_Acq_Time	= 2.98610082[s]
X_Angle	= 45[deg]
X_Atn	= 7[dB]
X_Data_Points	= 65536
X_Points_Default	= 65536
X_Pulse	= 3.725[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 400




```
---- PROCESSING PARAMETERS ----
sexp( 0.25016[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( -0.02251[ppm], 0[ppm] )
```

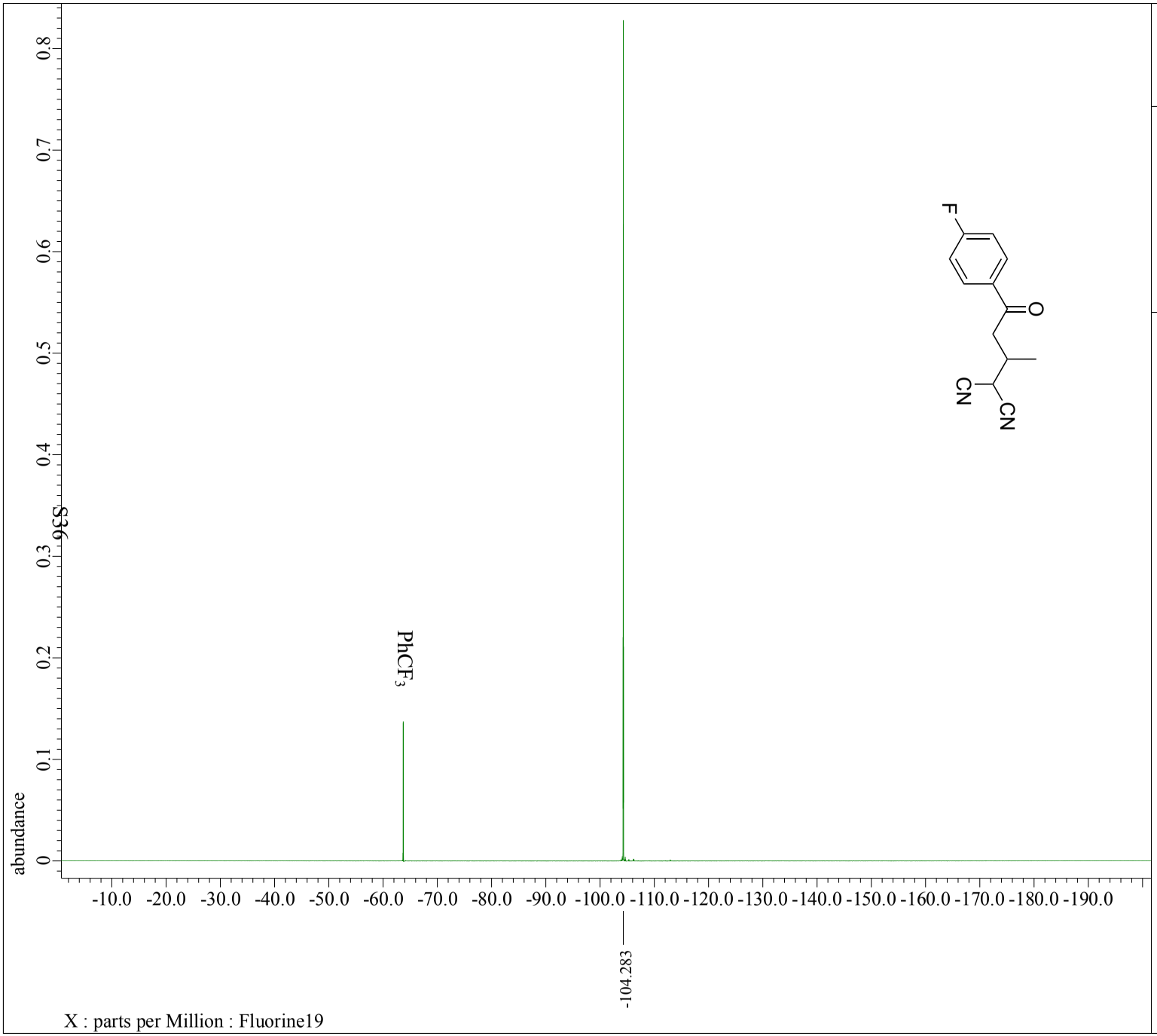
Filename	= RO0461-dry_PRO
Author	= console
Experiment	= single_pulse.j
Sample_Id	= RO0461-purifie
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 14-FEB-2024 17
Revision_Time	= 12-APR-2024 14
Data_Format	= 1D COMPLEX
Dim_Size	= 52429
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 1.99874592[s]
X_Domain	= Proton
X_Freq	= 500.15991521[M
X_Offset	= 5.0[ppm]
X_Points	= 18757
X_Points_Input	= 15005
X_Prescans	= 0
X_Resolution	= 0.50031372[Hz]
X_Sweep	= 9.38438438[kHz
X_Sweep_Clipped	= 7.50750751[kHz
X_Sweep_Input	= 15[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[M
Tri_Offset	= 5.0[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 4[s]
Recvr_Gain	= 42
Temp_Get	= 23.2[dC]
X_90_Width	= 7.67[us]
X_Acq_Time	= 1.99874592[s]
X_Angle	= 45[deg]
X_Atn	= 7.4[dB]
X_Data_Points	= 32768
X_Points_Default	= 31282
X_Pulse	= 3.835[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 400



JEOL

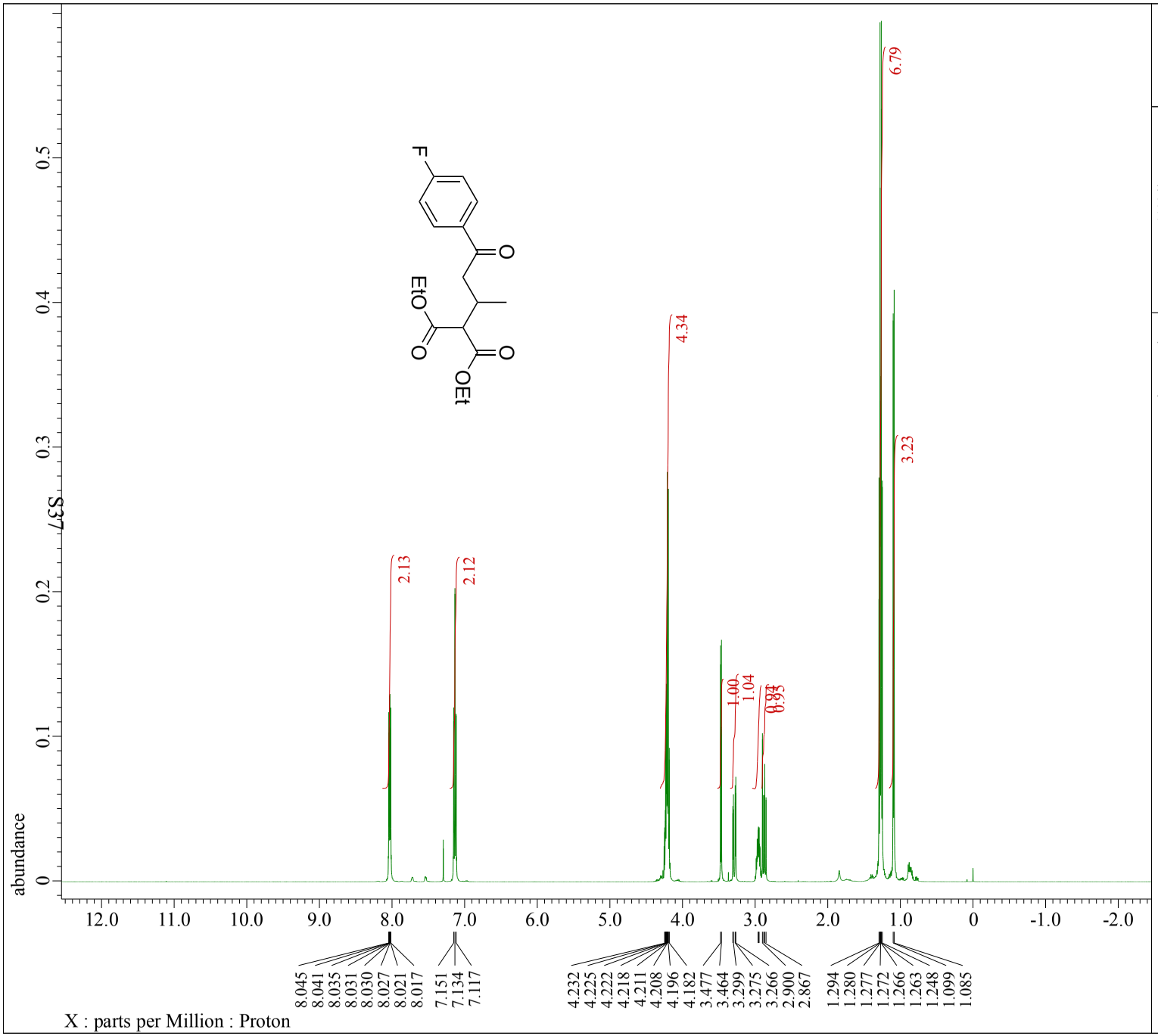
```
---- PROCESSING PARAMETERS ----
sexp( 1.00646[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 5[%], 1 )
peak_pick( 0[Hz], 0.1[ppm], Peaks, 0[Hz] )
reference( 77.16471[ppm], 77[ppm] )

以下に由来： RO0461-dry_CARBON-1-1.jdf
```

Filename	= RO0461-dry_CAR
Author	= console
Experiment	= carbon.jxp
Sample_Id	= RO0461-purifie
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 14-FEB-2024 17
Revision_Time	= 12-APR-2024 14
Data_Format	= 1D COMPLEX
Dim_Size	= 104858
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 0.99357984[s]
X_Domain	= Carbon13
X_Freq	= 125.76529768[M
X_Offset	= 100[ppm]
X_Points	= 39303
X_Points_Input	= 31442
X_Prescans	= 4
X_Resolution	= 1.00646164[Hz]
X_Sweep	= 39.55696203[kH
X_Sweep_Clipped	= 31.64556962[kH
X_Sweep_Input	= 251.0[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Blanking	= 5.0[us]
Clipped	= TRUE
Scans	= 129
Total_Scans	= 129
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 23[dC]
X_90_Width	= 12.19[us]
X_Acq_Time	= 0.99357984[s]
X_Angle	= 30[deg]
X_Atn	= 11.5[dB]
X_Data_Points	= 32768
X_Points_Default	= 39557
X_Pulse	= 4.06333333[us]
Irr_Atn_Dec	= 29[dB]
Irr_Atn_Dec_Calc	= 29[dB]
Irr_Atn_Noe	= 29[dB]
Irr_Bandwidth	= 5.97826087[kHz
Irr_Bandwidth_Ppm	= 11.9526989[ppm
Irr_Corresp_Pw90	= 92[us]



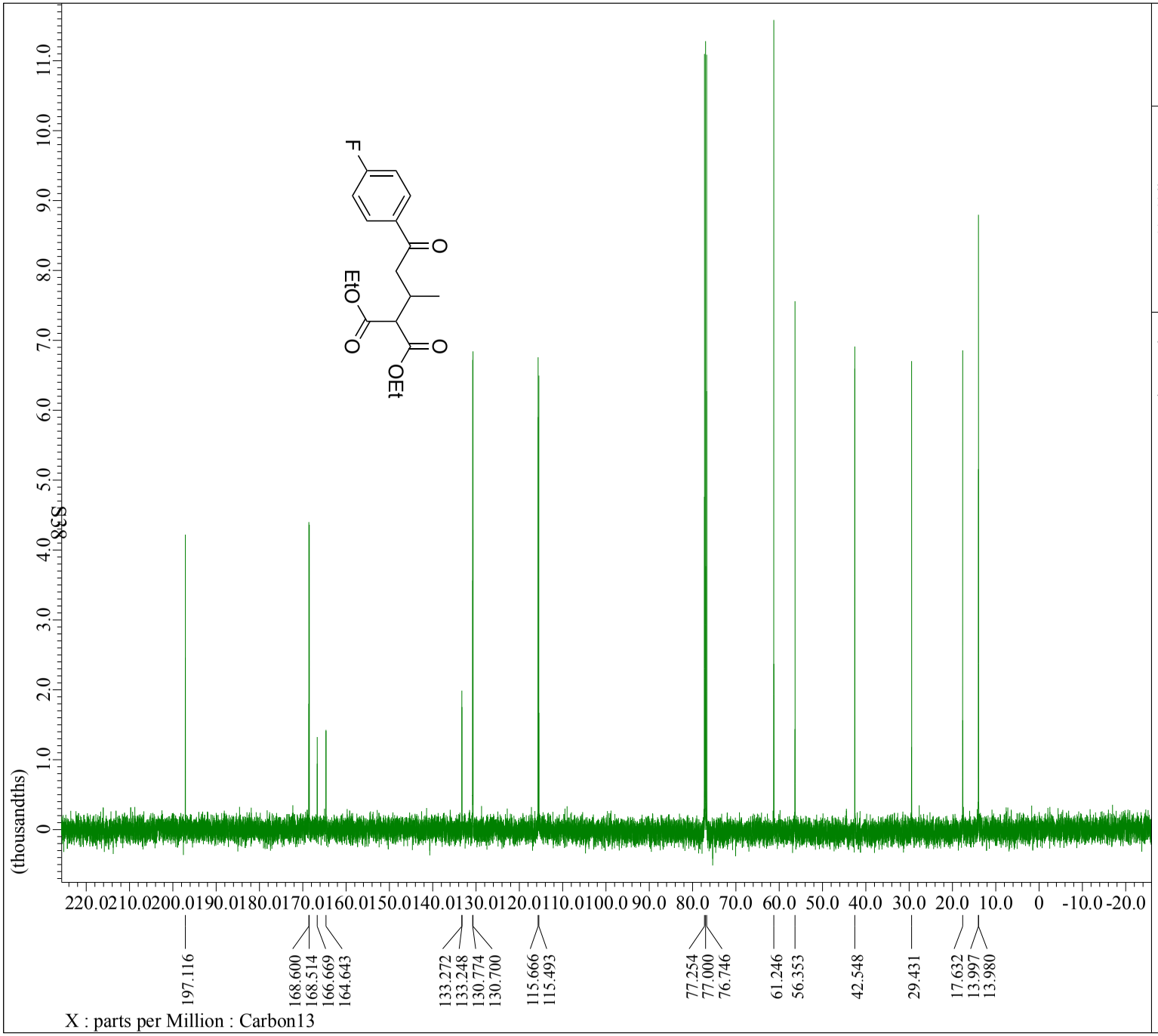

```
---- PROCESSING PARAMETERS ----
sexp( 0.33488[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( -62.55101[ppm], -63.72[ppm] )
thresh( 93.30321[%], 1 )

以下に由来：: RO0461-dry_Fluorine-1-1.jdf
```

Filename	= RO0461-dry_Flu
Author	= console
Experiment	= single_pulse.j
Sample_Id	= RO0461-dry
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 14-FEB-2024 17
Revision_Time	= 12-APR-2024 14
Data_Format	= 1D COMPLEX
Dim_Size	= 838861
X_Domain	= Fluorine19
Dim_Title	= Fluorine19
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 2.98610082[s]
X_Domain	= Fluorine19
X_Freq	= 470.62046084[M
X_Offset	= -100[ppm]
X_Points	= 352967
X_Points_Input	= 282373
X_Prescans	= 0
X_Resolution	= 0.33488488[Hz]
X_Sweep	= 118.20330969[k
X_Sweep_Clipped	= 94.56264775[kH
X_Sweep_Input	= 200[ppm]
Irr_Domain	= Fluorine19
Irr_Freq	= 470.62046084[M
Irr_Offset	= 5[ppm]
Tri_Domain	= Fluorine19
Tri_Freq	= 470.62046084[M
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 4[s]
Recvr_Gain	= 62
Temp_Get	= 23.3[dC]
X_90_Width	= 7.45[us]
X_Acq_Time	= 2.98610082[s]
X_Angle	= 45[deg]
X_Atn	= 7[dB]
X_Data_Points	= 65536
X_Points_Default	= 65536
X_Pulse	= 3.725[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 400




```
---- PROCESSING PARAMETERS ----
sexp( 0.25016[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -0.18196, 3.4973, 75.103[%] )
reference( -0.04656[ppm], 0[ppm] )
thresh( 5.65292[%], 1 )
```

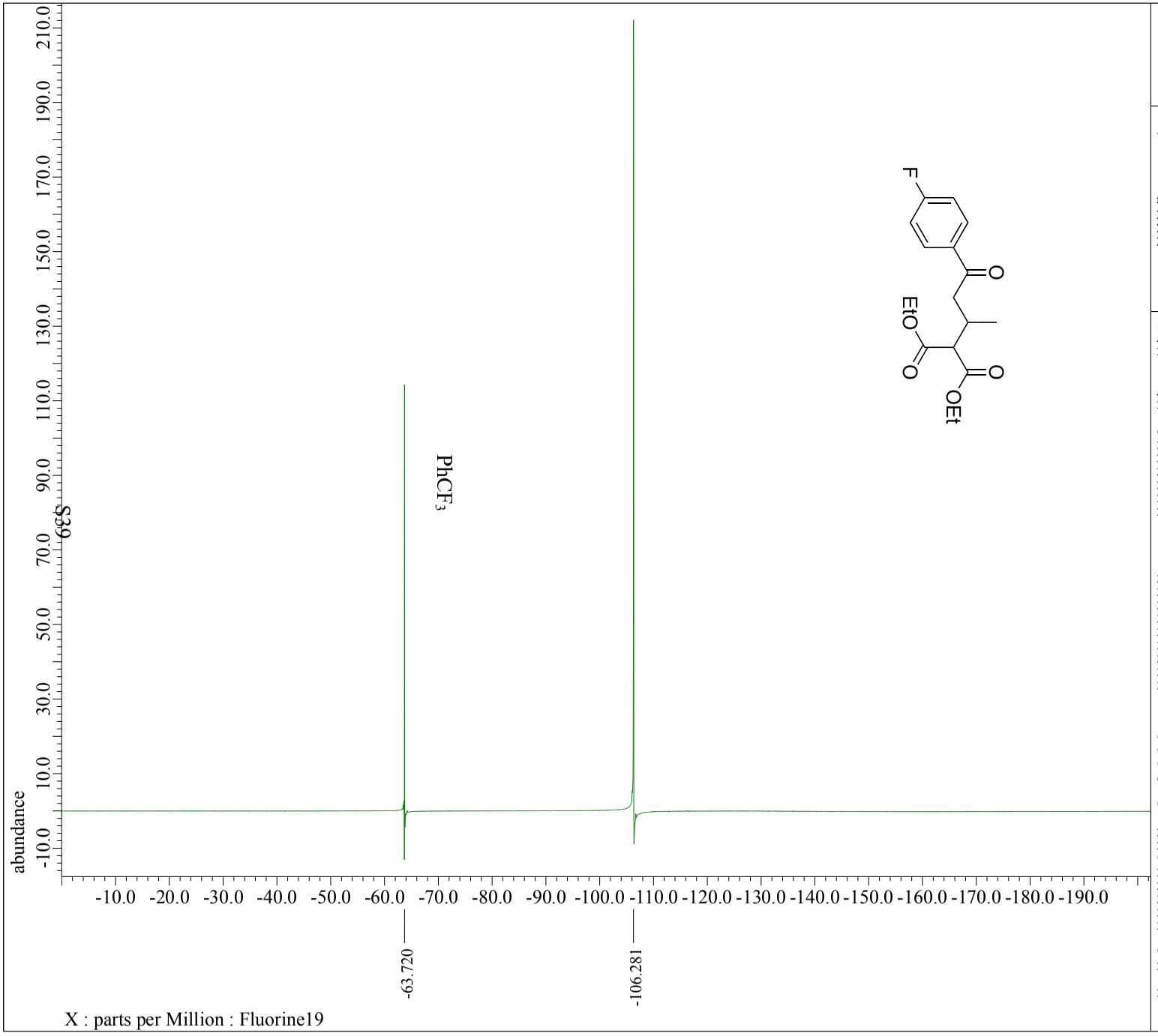
Filename	= RO0493-dry_PRO
Author	= console
Experiment	= single_pulse.j
Sample_Id	= RO0493-dry
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 12-APR-2024 19
Revision_Time	= 23-APR-2024 21
Data_Format	= 1D COMPLEX
Dim_Size	= 52429
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 1.99874592[s]
X_Domain	= Proton
X_Freq	= 500.15991521[M
X_Offset	= 5.0[ppm]
X_Points	= 18757
X_Points_Input	= 15005
X_Prescans	= 0
X_Resolution	= 0.50031372[Hz]
X_Sweep	= 9.38438438[kHz
X_Sweep_Clipped	= 7.50750751[kHz
X_Sweep_Input	= 15[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[M
Tri_Offset	= 5.0[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 4[s]
Recvr_Gain	= 42
Temp_Get	= 22.8[dC]
X_90_Width	= 14[us]
X_Acq_Time	= 1.99874592[s]
X_Angle	= 45[deg]
X_Atn	= 8[dB]
X_Data_Points	= 32768
X_Points_Default	= 31282
X_Pulse	= 7[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 400



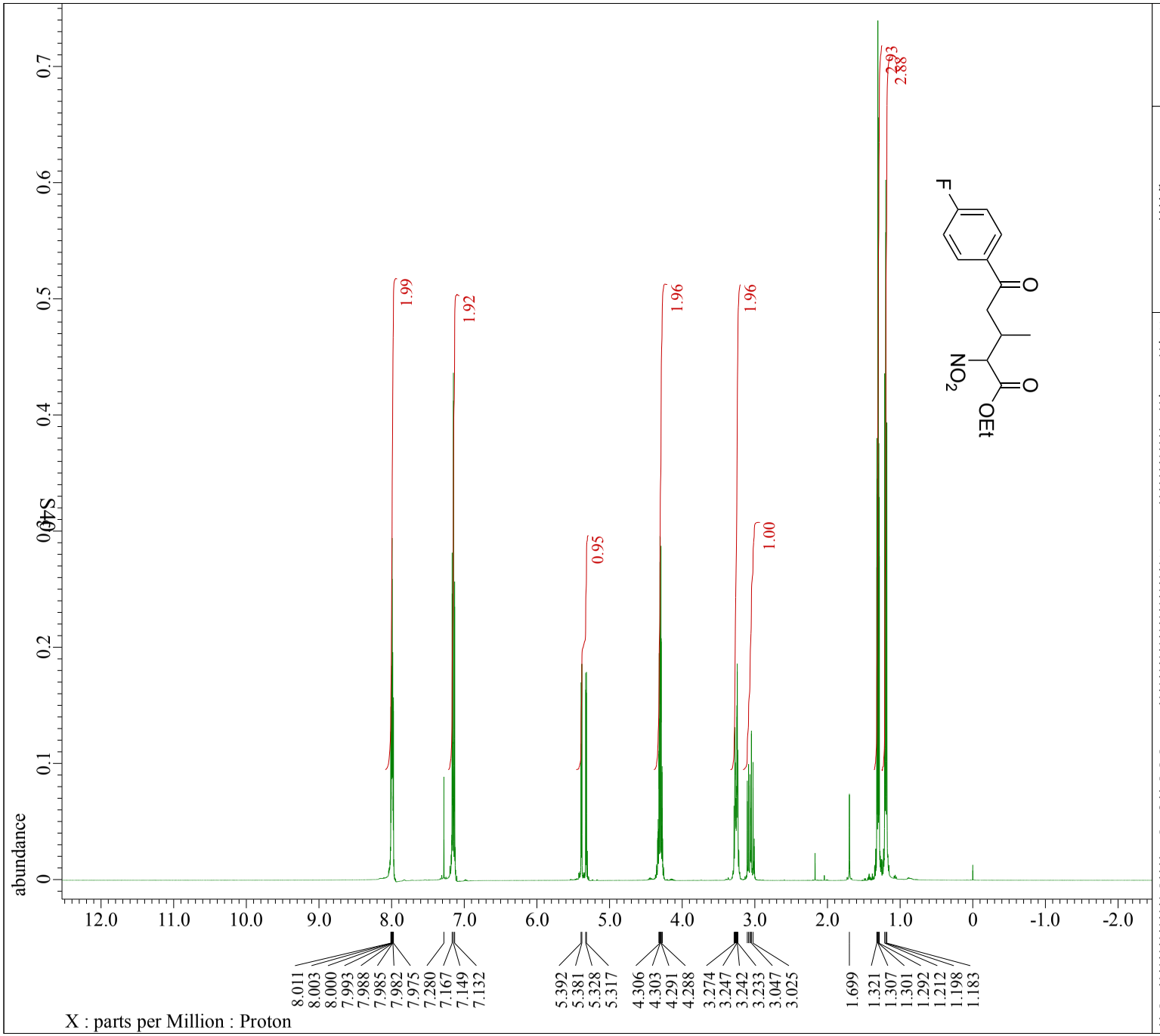
JEOL

```
---- PROCESSING PARAMETERS ----
sexp( 1.00646[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 5[%], 1 )
peak_pick( 0[Hz], 0.1[ppm], Peaks, 0[Hz] )
reference( 77.14791[ppm], 77[ppm] )

以下に由来：: RO0493-dry_CARBON-1-1.jdf
```

Filename	= RO0493-dry_CAR
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= RO0493-dry
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 30-MAR-2024 11
Revision_Time	= 12-APR-2024 16
Data_Format	= 1D COMPLEX
Dim_Size	= 104858
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 0.99357984[s]
X_Domain	= Carbon13
X_Freq	= 125.76529768[M
X_Offset	= 100[ppm]
X_Points	= 39303
X_Points_Input	= 31442
X_Prescans	= 4
X_Resolution	= 1.00646164[Hz]
X_Sweep	= 39.55696203[kH
X_Sweep_Clipped	= 31.64556962[kH
X_Sweep_Input	= 251.0[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Blanking	= 5.0[us]
Clipped	= FALSE
Scans	= 105
Total_Scans	= 105
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 22[dC]
X_90_Width	= 12.19[us]
X_Acq_Time	= 0.99357984[s]
X_Angle	= 30[deg]
X_Atn	= 11.5[dB]
X_Data_Points	= 32768
X_Points_Default	= 39557
X_Pulse	= 4.06333333[us]
Irr_Atn_Dec	= 29[dB]
Irr_Atn_Dec_Calc	= 29[dB]
Irr_Atn_Noe	= 29[dB]
Irr_Bandwidth	= 5.97826087[kHz
Irr_Bandwidth_Ppm	= 11.9526989[ppm
Irr_Corresp_Pw90	= 92[us]



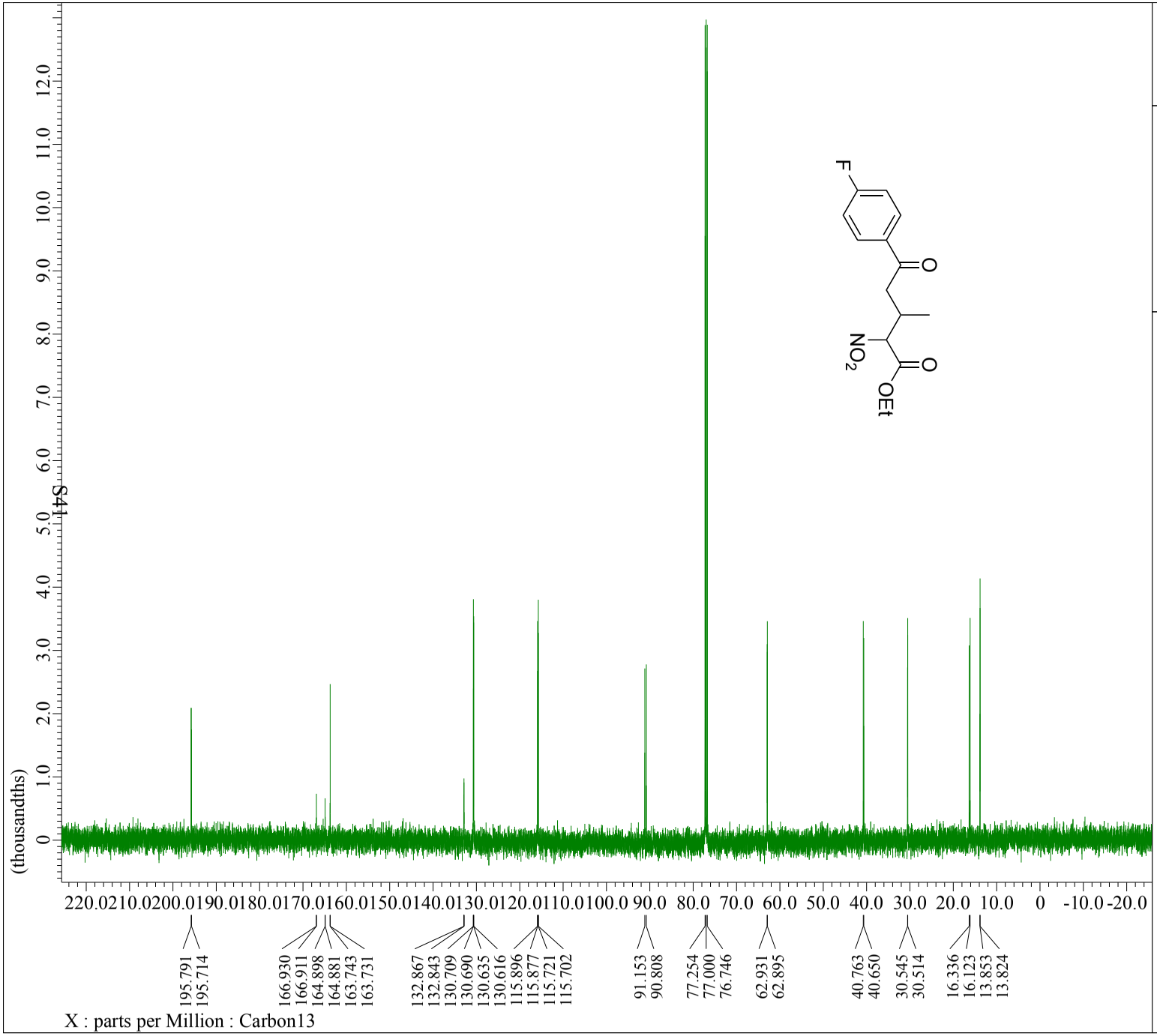

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -0.92513, 0, 76.1407[%] )
phase( -24, -50, 50[%] )
reference( -62.65821[ppm], -63.72[ppm] )
thresh( 41.14266[%], 1 )

以下に由来：: RO0493-dry_single_pulse(fluorine)-1

Filename           = RO0493-dry_single_pulse(f
Author             = delta
Experiment         = single_pulse.jxp
Sample_Id          = RO0493-dry
Solvent            = CHLOROFORM-D
Actual_Start_Time  = 13-APR-2024 10:43:00
Revision_Time      = 13-APR-2024 17:55:12

Comment            = single_pulse
Data_Format        = 1D COMPLEX
Dim_Size           = 13107
X_Domain           = Fluori
Dim_Title          = Fluorine19
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 86.50752[ms]
X_Domain           = 19F
X_Freq             = 376.17105393[MHz]
X_Offset           = 0[ppm]
X_Points           = 16384
X_Prescans         = 1
X_Resolution       = 11.55968868[Hz]
X_Sweep            = 189.39393939[kHz]
X_Sweep_Clipped    = 151.51515152[kHz]
Irr_Domain         = Fluorine19
Irr_Freq           = 376.17105393[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Fluorine19
Tri_Freq           = 376.17105393[MHz]
Tri_Offset         = 5[ppm]
Clipped            = TRUE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 50
Temp_Get           = 25[dC]
X_90_Width         = 7.95[us]
X_Acq_Time         = 86.50752[ms]
X_Angle            = 45[deg]
X_Atn              = 3[dB]
X_Pulse            = 3.975[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
Initial_Wait       = 1[s]
Repetition_Time    = 5.08650752[s]
```



JEOL

```
---- PROCESSING PARAMETERS ----
sexp( 0.25016[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 1.35846, -11.50809, 74.99237[%] )
reference( -0.03253[ppm], 0[ppm] )
```

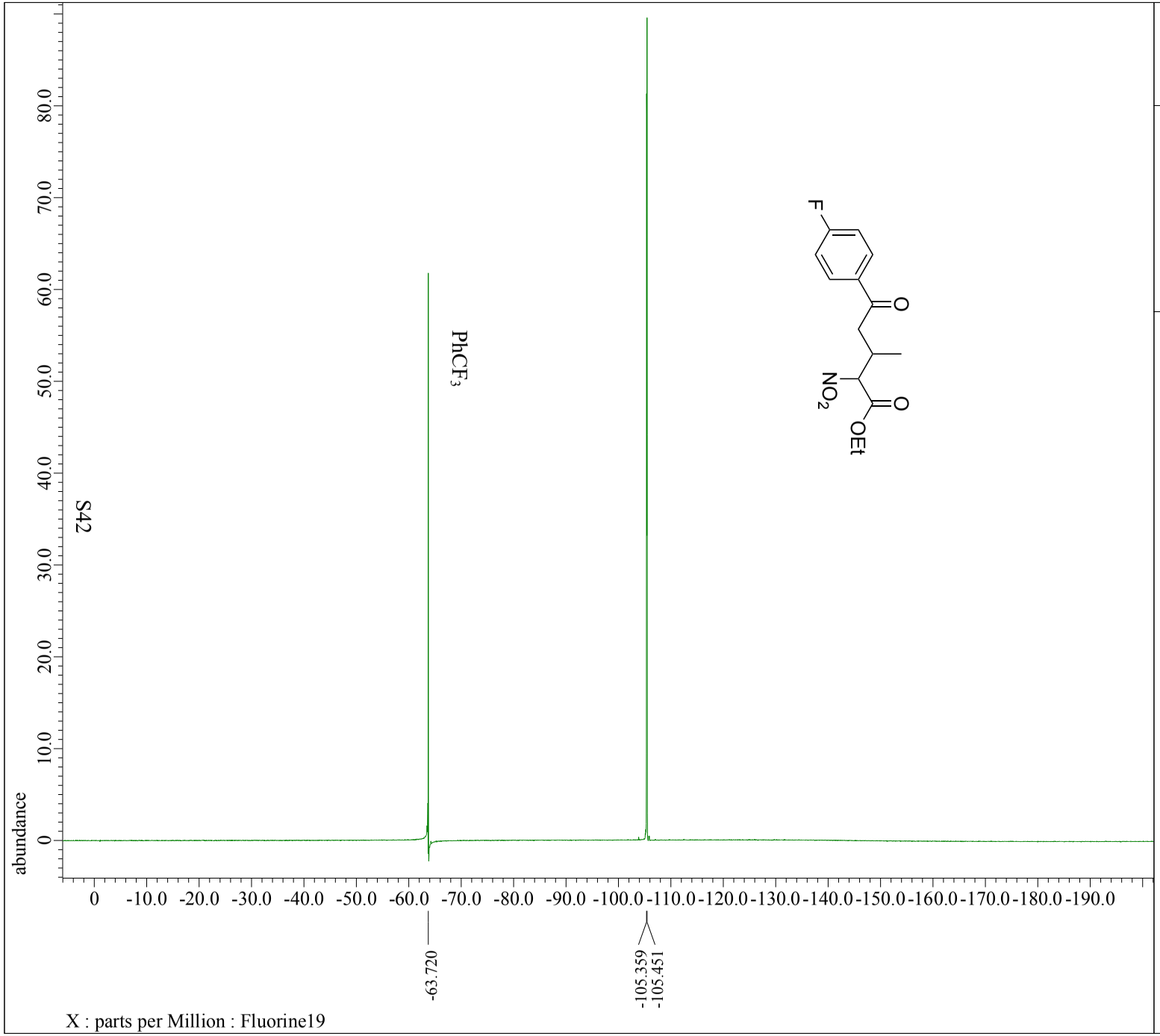
Filename	= RO0494-purifie
Author	= delta
Experiment	= single_pulse.j
Sample_Id	= RO0494-purifie
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 25-MAR-2024 15
Revision_Time	= 23-APR-2024 21
Data_Format	= 1D COMPLEX
Dim_Size	= 52429
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 1.99874592[s]
X_Domain	= Proton
X_Freq	= 500.15991521[M
X_Offset	= 5.0[ppm]
X_Points	= 18757
X_Points_Input	= 15005
X_Prescans	= 0
X_Resolution	= 0.50031372[Hz]
X_Sweep	= 9.38438438[kHz
X_Sweep_Clipped	= 7.50750751[kHz
X_Sweep_Input	= 15[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[M
Tri_Offset	= 5.0[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 4[s]
Recvr_Gain	= 42
Temp_Get	= 21.9[dC]
X_90_Width	= 7.67[us]
X_Acq_Time	= 1.99874592[s]
X_Angle	= 45[deg]
X_Atn	= 7.4[dB]
X_Data_Points	= 32768
X_Points_Default	= 31282
X_Pulse	= 3.835[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 400



---- PROCESSING PARAMETERS ----
sexp(1.00646[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(2, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
thresh(5[%], 1)
peak_pick(0[Hz], 0.1[ppm], Peaks, 0[Hz])
thresh(3.70024[%], 3.07836[%])
reference(77.13591[ppm], 77[ppm])

以下に由来：: RO0494-purified_CARBON-1-1.jdf

Filename	= RO0494-purifie
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= RO0494-purifie
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 25-MAR-2024 16
Revision_Time	= 15-APR-2024 16
Data_Format	= 1D COMPLEX
Dim_Size	= 104858
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ500R/M3
Spectrometer	= JNM-ECZ500R/M3
Field_Strength	= 11.7473579[T]
X_Acq_Duration	= 0.99357984[s]
X_Domain	= Carbon13
X_Freq	= 125.76529768[M
X_Offset	= 100[ppm]
X_Points	= 39303
X_Points_Input	= 31442
X_Prescans	= 4
X_Resolution	= 1.00646164[Hz]
X_Sweep	= 39.55696203[kH
X_Sweep_Clipped	= 31.64556962[kH
X_Sweep_Input	= 251.0[ppm]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[M
Irr_Offset	= 5.0[ppm]
Blanking	= 5.0[us]
Clipped	= FALSE
Scans	= 148
Total_Scans	= 148
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 22[dC]
X_90_Width	= 12.19[us]
X_Acq_Time	= 0.99357984[s]
X_Angle	= 30[deg]
X_Atn	= 11.5[dB]
X_Data_Points	= 32768
X_Points_Default	= 39557
X_Pulse	= 4.06333333[us]
Irr_Atn_Dec	= 29[dB]
Irr_Atn_Dec_Calc	= 29[dB]
Irr_Atn_Noe	= 29[dB]
Irr_Bandwidth	= 5.97826087[kHz
Irr_Bandwidth_Ppm	= 11.9526989[ppm
Irr_Corresp_Pw90	= 92[us]




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 1.25, -185, 75.89654[%] )
reference( -62.62748[ppm], -63.72[ppm] )
reference( -63.72[ppm], -63.72[ppm] )
thresh( 40.93535[%], 1 )
```

Filename	= RO0494-purified_fluorine-
Author	= delta
Experiment	= proton.jxp
Sample_Id	= RO0494-purified
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 8-APR-2024 16:40:23
Revision_Time	= 11-APR-2024 17:28:16
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Fluori
Dim_Title	= Fluorine19
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 86.50752[ms]
X_Domain	= 19F
X_Freq	= 376.17105393[MHz]
X_Offset	= 0[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 11.55968868[Hz]
X_Sweep	= 189.39393939[kHz]
X_Sweep_Clipped	= 151.51515152[kHz]
Irr_Domain	= Fluorine19
Irr_Freq	= 376.17105393[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Fluorine19
Tri_Freq	= 376.17105393[MHz]
Tri_Offset	= 5[ppm]
Clipped	= TRUE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 50
Temp_Get	= 25[dC]
X_90_Width	= 7.95[us]
X_Acq_Time	= 86.50752[ms]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 3.975[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 5.08650752[s]