# Efficient Diastereoselective Synthesis of Trifarane-type Sesquiterpenes, Trifarienols A and B

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## Supporting Information

1) General Experimental Procedures S-2

(3*R*)-3-[(1*S*, 4*S*)-1,4-Dimethyl-2-oxocyclohexyl]butyl benzoate and (3*S*)-3-[(1*S*, 4*S*)-1,4-Dimethyl-2-oxocyclohexyl]butyl benzoate (**4**) S-3

(3*R*)-3-{[(1*S*, 4*S*)-1,4-Dimethyl-2-(trimethylsilyl)oxy]cyclohex-2-en-1-yl}butyl benzoate and (3*S*)-3-{[(1*S*, 4*R*)-1,4-Dimethyl-2-(trimethylsilyl)oxy]cyclohex-2-en-1-yl}butyl benzoate **(5).** S-3

(3*R*)-3-{(1*S*)-1,4-Dimethyl-2-oxocyclohex-3-en-1-yl}butyl benzoate **(6)** S-4

(3*R*)-3-{(1*S*, 4*S*)-1,4-Dimethyl-2- oxo-4-vinylcyclohexyl}butyl benzoate and (3*R*)-3-{(1*S*, 4*R*)-1,4-Dimethyl-2-oxo-4-vinylcyclohexyl}butyl benzoate **(7)** S-4

(3*R*)-3-((1*S*,4*R*)1,4-dimethyl-2-{[(trifluoromethyl)sulfonyl]oxy}-4-vinylcyclohex-2-en-1-yl)butyl benzoate and (3*R*)-3-((1*S*,4*S*)-1,4-Dimethyl-2-{[(trifluoromethyl)sulfonyl]oxy}-4-vinylcyclohex-2-en-1-yl)butyl benzoate **(8)** S-5

2) NMR spectra

$^{1}$H NMR Spectrum of **4** S-7

$^{13}$C NMR Spectrum of **4** S-8

$^{1}$H NMR Spectrum of **5** S-9
$^{13}$C NMR Spectrum of **5** S-10
$^{1}$H NMR Spectrum of **6** S-11
$^{13}$C NMR Spectrum of **6** S-12
$^{1}$H NMR Spectrum of **7** S-13
$^{13}$C NMR Spectrum of **7** S-14
$^{1}$H NMR Spectrum of **8** S-15
$^{13}$C NMR Spectrum of **8** S-16
$^{1}$H NMR Spectrum of **9** S-17
$^{13}$C NMR Spectrum of **9** S-18
$^{1}$H NMR Spectrum of **10** S-19
$^{13}$C NMR Spectrum of **10** S-20
$^{1}$H NMR Spectrum of **11** S-21
$^{13}$C NMR Spectrum of **11** S-22
$^{1}$H NMR Spectrum of **12** S-23
$^{13}$C NMR Spectrum of **12** S-24
$^{1}$H NMR Spectrum of **13** S-25
$^{13}$C NMR Spectrum of **13** S-26
$^{1}$H NMR Spectrum of **14** S-27
$^{13}$C NMR Spectrum of **14** S-28
$^{1}$H NMR Spectrum of **15** S-29
$^{13}$C NMR Spectrum of **15** S-30
$^{1}$H NMR Spectrum of **16** S-31
$^{13}$C NMR Spectrum of **16** S-32
$^{1}$H NMR Spectrum of **17** S-33
$^{13}$C NMR Spectrum of **17** S-34
NMR Spectrum of **1** S-35
$^{13}$C NMR Spectrum of **1** S-36
$^{1}$H NMR Spectrum of **2** S-37
$^{13}$C NMR Spectrum of **2** S-38

**General Experimental Procedures.** Melting points were measured with a melting point

apparatus and are uncorrected. IR spectra were recorded as thin films on sodium chloride plates. Otherwise noted, $^{1}H$ and $^{13}C$ NMR spectra were obtained for solutions in $CDCl_3$, and chemical shifts are reported on the δ scale using TMS as an internal standard of δ 0.00 for $^{1}H$-NMR spectra, and $CDCl_3$ as an internal standard or δ 77.00 for $^{13}C$ NMR spectra, respectively ($^{1}H$-NMR: 400 MHz, $^{13}C$ NMR: 100 MHz). Reagents were purchased from commercial sources.

**(3*R*)-3-[(1*S*, 4*S*)-1,4-Dimethyl-2-oxocyclohexyl]butyl benzoate and (3*S*)-3-[(1*S*, 4*S*)-1,4-Dimethyl-2-oxocyclohexyl]butyl benzoate (4).**

To a stirred suspension of CuI (1.20 g, 6.51 mmol) in dry $Et_2O$ (6 mL), was added MeLi (1.09 M $Et_2O$ solution) (12 mL) (13 mmol) at -40 °C under Ar, and the resulting mixture was stirred for 20 min. To this mixture was added a solution of enone **3** (620 mg, 2.17 mmol) in $Et_2O$ (4 mL) at -78 °C, and the whole was stirred at -40 °C for further 1 h. After addition of saturated aq. ammonium chloride, the mixture was extracted with AcOEt. The extract was washed with brine and dried over $Na_2SO_4$. Evaporation of the solvent gave a residue, which was subjected to column chromatography on silica gel. Elution with *n*-hexane-AcOEt (7:1, v/v) afforded a diastereoisomeric mixture of ketone **4** (611 mg, 93%) as a colorless oil.

**(3*R*)-3-[(1*S*, 4*S*)-1,4-Dimethyl-2-oxocyclohexyl]butyl benzoate (major)**: IR νmax 2954, 1720, 1704 $cm^{-1}$; $^{1}H$-NMR ($CDCl_3$; 400 MHz) δ 8.01-8.05 (2H, m), 7.54-7.58 (1H, m), 7.42-7.46 (2H, m), 4.28-4.36 (2H, m), 2.25-2.32 (2H, m), 2.13-2.20 (1H, m), 2.02-2.07 (1H, m), 1.71-1.81 (1H, m), 1.29-1.65 (5H, m), 0.97 (3H, d, *J*=6.6 Hz), 0.95 (3H, d, *J*=6.4 Hz), 0.89 (3H, s); $^{13}C$-NMR ($CDCl_3$; 100 MHz) δ 216.0, 166.4, 132.9, 130.1, 129.4 (2), 128.3 (2), 63.3, 51.3, 46.8, 36.4, 35.5, 31.5, 30.8, 28.8, 22.2, 16.21, 12.3; MS (EI): 302 ($M^+$); HRMS (EI): Calcd for $C_{19}H_{26}O_3$: 302.1882. Found: 302.1904.

**(3*R*)-3-[(1*S*, 4*S*)-1,4-Dimethyl-2-oxocyclohexyl]butyl benzoate (minor)**: $^{1}H$-NMR ($CDCl_3$; 400 MHz) δ 8.01-8.05 (2H, m), 7.54-7.58 (1H, m), 7.42-7.76 (2H, m), 4.38-4.43 (2H, m), 2.25-2.32 (2H, m), 2.13-2.20 (1H, m), 2.02-2.07 (1H, m), 1.71-1.81 (1H, m), 1.57-1.65 (1H, m), 1.29 -1.50 (4H, m), 0.99 (3H, d, *J*=5.9 Hz), 0.94 (3H, d, *J*=6.7 Hz), 0.89 (3H, s); $^{13}C$- NMR ($CDCl_3$, 100 MHz) δ 215.5, 166.4, 132.8, 130.3, 129.5 (2), 128.3 (2), 64.0, 50.9, 46.4, 33.8, 33.3, 32.7, 30.7, 28.5, 21.2, 19.7, 14.3.

**(3*R*)-3-{[(1*S*, 4*S*)-1,4-Dimethyl-2-(trimethylsilyl)oxy]-cyclohex-2-en-1-yl}butyl benzoate and (3*R*)-3-{[(1*S*, 4*R*)-1,4-Dimethyl-2-(trimethylsilyl)oxy]-cyclohex-2-en-1-yl}butyl benzoate (5).**

To a stirred solution of (3*R*)-ketone **4** (6.8 g, 22.5 mmol) in $CH_2Cl_2$ (140 mL) were added $Et_3N$ (53 ml, 225 mmol) and TMSOTf (16.3 mL, 90 mmol) at 0 °C under Ar, and the resulting mixture was stirred for further 30 min at rt. After treatment with saturated aq. $NaHCO_3$ at 0 °C, the mixture was extracted with $Et_2O$-pentane (1:1, v/v). The extract was washed with brine and dried over $MgSO_4$.

Evaporation of the solvent gave a residue, which was purified by column chromatography on silica gel. Elution with *n*-hexane-AcOEt (20:1, v/v) afforded silyl enol ether **5** (7.9 g, 94%) as a colorless oil.

**(3*R*)-3-{[(1*S*, 4*S*)-1,4-Dimethyl-2-(trimethylsilyl)oxy]-cyclohex-2-en-1-yl}butyl benzoate (major):** IR νmax 2959, 1724, 1653 $cm^{-1}$; $^1$H-NMR ($CDCl_3$; 400 MHz) δ 8.01-8.06 (2H, m), 7.53-7.57 (1H, m), 7.41-7.46 (2H, m), 4.66 (1H, d, *J*=4.8 Hz), 4.40-4.46 (1H, m), 4.25-4.35 (1H, m), 2.21-2.27 (1H, m), 1.90-2.07 (1H, m), 1.77-1.89 (1H, m), 1.63-1.75 (1H, m), 1.20-1.47 (3H, m), 1.11-1.16 (1H, m), 1.05 (3H, s), 0.96 (3H, d, *J*=7.0 Hz), 0.91 (3H, d, *J*=6.7 Hz), 0.17 (9H, s); $^{13}$C-NMR ($CDCl_3$; 100 MHz) δ 166.7, 155.2, 132.7, 130.6, 129.5 (2), 128.3 (2), 108.6, 64.6, 40.9, 35.3, 29.9, 28.73, 26.7, 26.3, 23.8, 21.7, 15.2, 0.3 (3); MS (EI): 374 ($M^+$); HRMS (EI): Calcd for $C_{22}H_{34}O_3Si$: 374.2277. Found: 374.2298.

**(3*R*)-3-{[(1*S*, 4*R*)-1,4-Dimethyl-2-(trimethylsilyl)oxy]cyclohex-2-en-1-yl}butyl benzoate (minor):** $^1$H-NMR ($CDCl_3$; 400 MHz) δ 8.01-8.06 (2H, m), 7.53-7.57 (1H, m), 7.41-7.46 (2H, m), 4.52 (1H, s), 4.40-4.46 (1H, m), 4.24-4.35 (1H, m), 2.21-2.27 (1H, m), 1.90-2.07 (1H, m), 1.77-1.89 (1H, m), 1.63-1.75 (1H, m), 1.20-1.47 (3H, m), 1.11-1.16 (1H, m), 1.07 (3H, s), 0.93 (3H, d, *J*=6.9 Hz), 0.91 (3H, d, *J*=6.7 Hz), 0.17 (9H, m); $^{13}$C-NMR ($CDCl_3$, 100 MHz) δ 166.7, 155.2, 132.7, 130.6, 129.5 (2), 128.3 (2), 109.7, 64.5, 40.9, 35.5, 30.4, 29.7, 26.7, 26.3, 24.1, 22.9, 14.7, 0.3 (3).

**(3*R*)-3-{(1*S*)-1,4-Dimethyl-2-oxocyclohex-3-en-1-yl}butyl benzoate (6).**

To a stirred solution of silyl enol ether **5** (124 mg, 0.332 mmol) in dry DMSO (2 mL) was added $Pd(OAc)_2$ (30 mg, 0.133 mmol) at rt, and the mixture was stirred at 80 °C for 20 h under an atmospheric pressure of oxygen. The mixture was treated with $H_2O$ and extracted with AcOEt. The extract was washed with brine and dried over $MgSO_4$. Evaporation of the solvent gave a residue, which was purified by column chromatography on silica gel. Elution with *n*-hexane-AcOEt (5:1, v/v) afforded enone **6** (74.9 mg, 75%) as a pale yellowish oil: $[\alpha]_D^{22}$ = +18.3 (c 0.6, $CHCl_3$); mp 71-73 °C; IR νmax 2965, 1719, 1663, 1276 $cm^{-1}$; $^1$H-NMR ($CDCl_3$; 400 MHz) δ 7.99-8.02 (2H, m), 7.53-7.57 (1H, m), 7.41-7.44 (2H, m), 5.77 (1H, d, *J*=1.3 Hz), 4.23-4.38 (2H, m), 1.99-2.35 (4H, m), 1.88 (3H, s), 1.82-1.89 (1H, m), 1.66-1.73 (1H, m), 1.34-1.43 (1H, m), 1.01 (3H, s), 0.97 (3H, d, *J*=6.8 Hz); $^{13}$C-NMR ($CDCl_3$; 100 MHz) δ 204.1, 166.5, 159.9, 132.8, 130.3, 129.5 (2), 128.2 (2), 125.4, 63.7, 46.4, 32.3, 30.8, 30.1, 27.9, 23.7, 18.0, 14.6; MS (EI): 300 ($M^+$); HRMS (EI): Calcd for $C_{19}H_{24}O_3$: 300.1725. Found: 300.1748. Anal. Calcd for $C_{19}H_{24}O_3$: C, 75.97; H, 8.05. Found: C, 75.98, H, 8.17.

**(3*R*)-3-{(1*S*, 4*S*)-4-Ethenyl-1,4-dimethyl-2- oxocyclohexyl}butyl benzoate and (3*R*)-3-{(1*S*, 4*R*)-4-Ethenyl-1,4-dimethyl-2-oxocyclohexyl}butyl benzoate (7).** Vinylmagnesium bromide (1.0 M THF solution) (22 mL, 22 mmol) was added lithium-2-thienylcyanocuprate (0.25 M THF solution) (44 mL, 11.0 mmol) at -78 °C under Ar, and the whole was stirred at 0 °C for 30 min. The mixture was cooled to -78 °C, and a solution of enone **6** (1.1 g, 3.67 mmol) in THF (18 mL) was added to the mixture

at the same temperature, and stirred at -40 °C for 12 h. After treatment with saturated aq. ammonium chloride, the mixture was extracted with AcOEt. The organic layer was washed with brine and dried over $Na_2SO_4$. Evaporation of the solvent gave a residue, which was subjected to column chromatography on silica gel. Elution with *n*-hexane-AcOEt (9:1, v/v) afforded ketone **7** (1.1 g, 90%) as a colorless oil.

**(3*R*)-3-{(1*S*, 4*S*)-4-Ethenyl-1,4-dimethyl-2-oxocyclohexyl}butyl benzoate (major):** IR νmax 2966, 1719, 1704, 1274 $cm^{-1}$; $^1$H-NMR ($CDCl_3$; 400 MHz) δ 8.00-8.03 (2H, m), 7.52-7.58 (1H, m), 7.41-7.46 (2H, m), 5.72 (1H, dd, *J*=17.5 and 10.8 Hz), 4.92 (1H, dd, *J*=10.8 and 0.71 Hz), 4.88 (1H, dd, *J*=17.5 and 0.71 Hz), 4.28-4.39 (2H, m), 2.60 (1H, d, *J*=13.8 Hz), 2.19-2.29 (1H, m), 2.15 (1H, dd, *J*=12.2 and 1.5 Hz), 1.91-1.97 (1H, m), 1.78-1.85 (1H, m), 1.41-1.66 (4H, m), 1.00 (3H, s), 0.97 (3H, s), 0.96 (3H, d, *J*=6.6 Hz); $^{13}$C-NMR ($CDCl_3$; 100MHz) δ 215.4, 166.5, 146.9, 132.9, 130.2, 129.5 (2), 128.4 (2), 111.2, 63.4, 51.1, 48.6, 41.8, 32.3, 31.8, 31.5, 30.7, 25.3, 18.0, 13.2; MS (EI): 328 ($M^+$); HRMS (EI): Calcd for $C_{21}H_{28}O_3$: 328.2038. Found: 328.2044.

**(3*R*)-3-{(1*S*, 4*R*)-4-Ethenyl-1,4-dimethyl-2-oxocyclohexyl}butyl benzoate (minor):** $^1$H-NMR ($CDCl_3$; 400 MHz) δ 8.00-8.03 (2H, m), 7.52-7.58 (1H, m), 7.41-7.46 (2H, m), 5.63 (1H, dd, *J*=17.5 and 11.0 Hz), 5.02 (1H, dd, *J*=11.0 and 0.71 Hz), 4.99 (1H, dd, *J*=17.5 and 0.71 Hz), 4.28-4.39 (2H, m), 2.60 (1H, d, *J*=13.8 Hz), 2.19- 2.41 (1H, m), 2.15 (1H, dd, *J*=12.2 and 1.5 Hz), 1.91-1.97 (1H, m), 1.78-1.85 (1H, m), 1.58-1.66 (1H, m), 1.41-1.54 (3H, m), 0.96 (3H, d, *J*=6.6 Hz), 0.89 (3H, s), 0.89 (3H, s); $^{13}$C-NMR ($CDCl_3$; 100MHz) δ 215.4, 166.5, 146.9, 132.9, 130.2, 129.5 (2), 128.4 (2), 111.2, 63.4, 51.1, 48.6, 41.8, 32.3, 31.8, 31.5, 30.7, 25.3, 18.0, 13.2.

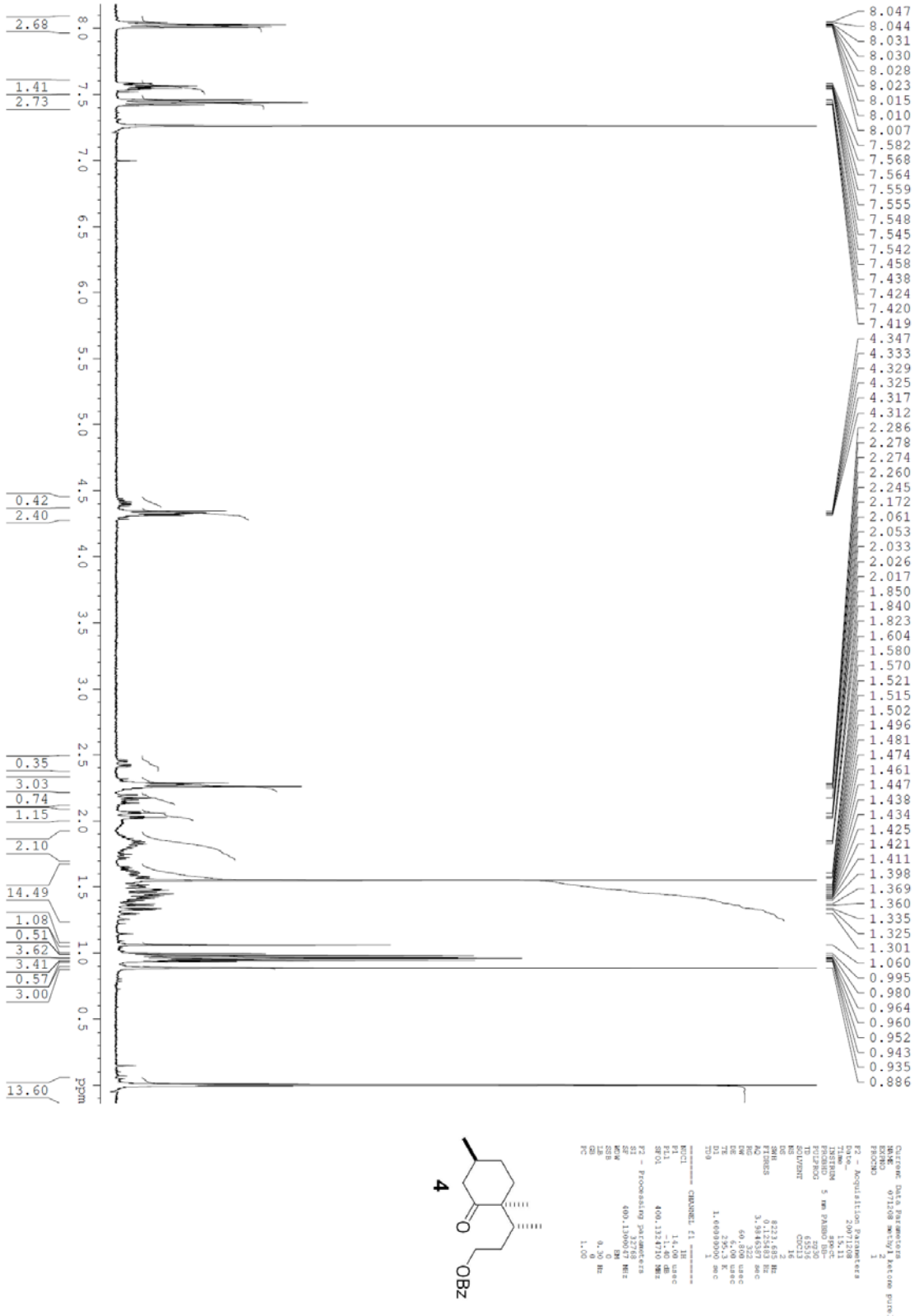
**(3*R*)-3-((1*S*,4*R*)-4-Ethenyl-1,4-dimethyl-2-{[(trifluoromethyl)sulfonyl]oxy}-cyclohex-2-en-1-yl)butyl benzoate and (3*R*)-3-((1*S*,4*S*)-4-Ethenyl-1,4-dimethyl-2-{[(trifluoromethyl)sulfonyl]oxy}-cyclohex-2-en-1-yl)butyl benzoate (8).**

To a stirred solution of ketone **7** (158 mg, 0.482 mmol) and Comins reagent (284 mg, 0.723 mmol) in THF (2.4 mL) was gradually added NaHMDS (1.9 M THF solution) (0.3 ml, 0.578 mmol) at -40 °C, and the resulting mixture was stirred at the same temperature for further 12 h. After treatment with saturated aq. ammonium chloride, the mixture was extracted with AcOEt. The organic layer was washed with brine and dried over $Na_2SO_4$. Evaporation of the solvent gave a residue, which was subjected to column chromatography on silica gel. Elution with *n*-hexane-AcOEt (95:5, v/v) afforded triflate **8** (153 mg, 69%) as a colorless oil.

**(3*R*)-3-((1*S*,4*R*)-4-Ethenyl-1,4-dimethyl-2-{[(trifluoromethyl)sulfonyl]oxy}-cyclohex-2-en-1-yl)butyl benzoate (major)**: IR νmax 2968, 1721, 1453 $cm^{-1}$; $^1$H-NMR ($CDCl_3$; 400 MHz) δ 8.01-8.03 (2H, m), 7.49-7.55 (1H, m), 7.40-7.44 (2H, m), 5.72 (1H, dd, *J*=17.4 and 10.6 Hz), 5.51 (1H, s), 5.05 (1H, dd, *J*=10.6 and 1.1 Hz), 4.95 (1H, dd, *J*=17.4 and 1.1 Hz), 4.41-4.47 (1H, m), 4.21-4.28 (1H, m), 1.83-2.01

(2H, m), 1.69-1.77 (1H, m), 1.52-1.55 (2H, m), 1.25-1.42 (2H, m), 1.23 (3H, s), 1.15 (3H, s), 0.95 (3H, d, *J*=6.7 Hz); $^{13}$C-NMR ($CDCl_3$; 100 MHz) δ 166.4, 154.8, 144.7, 132.8, 130.2, 129.4 (2), 128.3 (2), 123.1, 118.3 (q), 114.2, 63.5, 41.5, 40.3, 35.6, 31.4, 29.4, 28.7, 26.8, 23.1, 14.6; MS (CI): 461 $(M+1)^+$, HRMS (CI): Calcd for $C_{22}H_{27}O_5F_3S$+H: 461.1609. Found: 461.1628.

**(3*R*)-3-((1*S*,4*S*)-4-Ethenyl-1,4-dimethyl-2-{[(trifluoromethyl)sulfonyl]oxy}-cyclohex-2-en-1-yl)butyl benzoate (minor):** $^1$H-NMR ($CDCl_3$; 400 MHz) δ 8.01-8.03 (2H, m), 7.49-7.55 (1H, m), 7.40-7.44 (2H, m), 5.77 (1H, dd, *J*=17.4 and 10.6 Hz), 5.57 (1H, s), 5.05 (1H, dd, *J*=17.4 and 1.1 Hz), 4.95 (1H, dd, *J*=10.6 and 1.1 Hz), 4.41-4.47 (1H, m), 4.21-4.28 (1H, m), 1.83-2.01 (2H, m), 1.69-1.77 (1H, m), 1.52-1.55 (2H, m), 1.25-1.42 (2H, m), 1.20 (3H, s), 1.14 (3H, s), 0.99 (3H, d, *J*=6.8 Hz); $^{13}$C-NMR ($CDCl_3$; 100 MHz) δ 166.4, 154.2, 145.8, 132.0, 130.2, 129.9 (2), 128.7 (2), 124.1, 118.3 (q), 112.5, 68.0, 41.3, 39.6, 35.3, 31.6, 29.9, 29.6, 27.4, 25.8, 14.9.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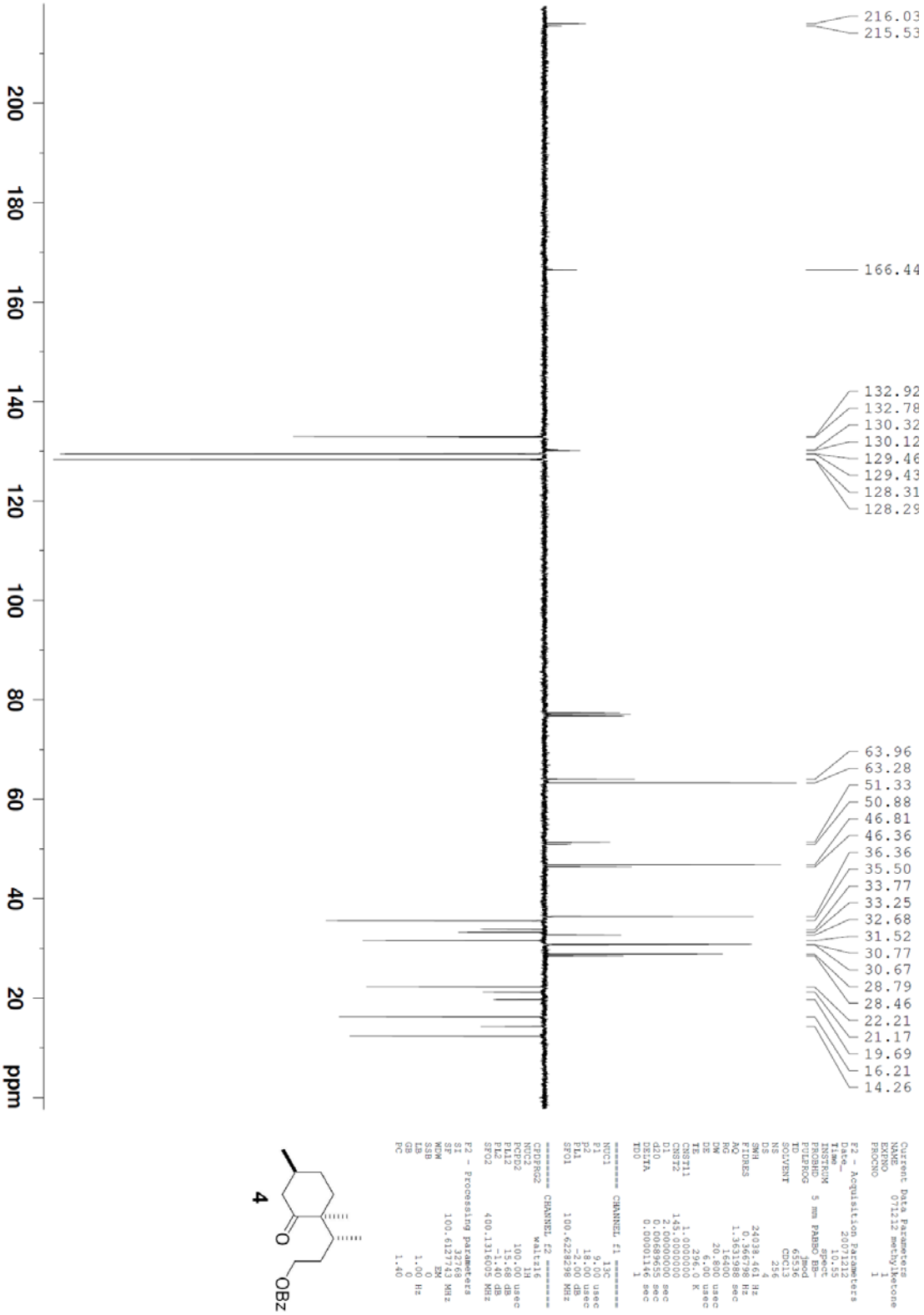
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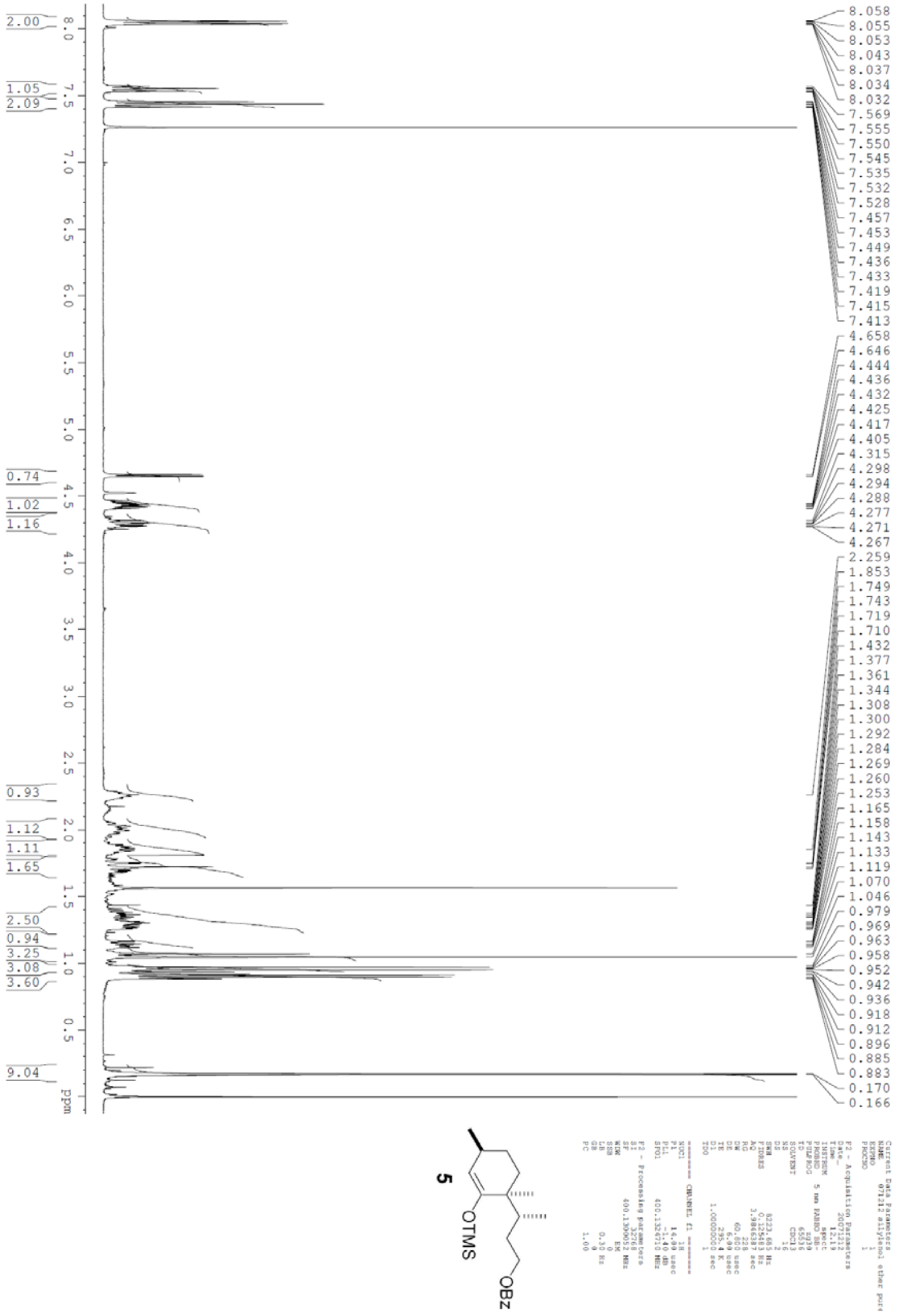
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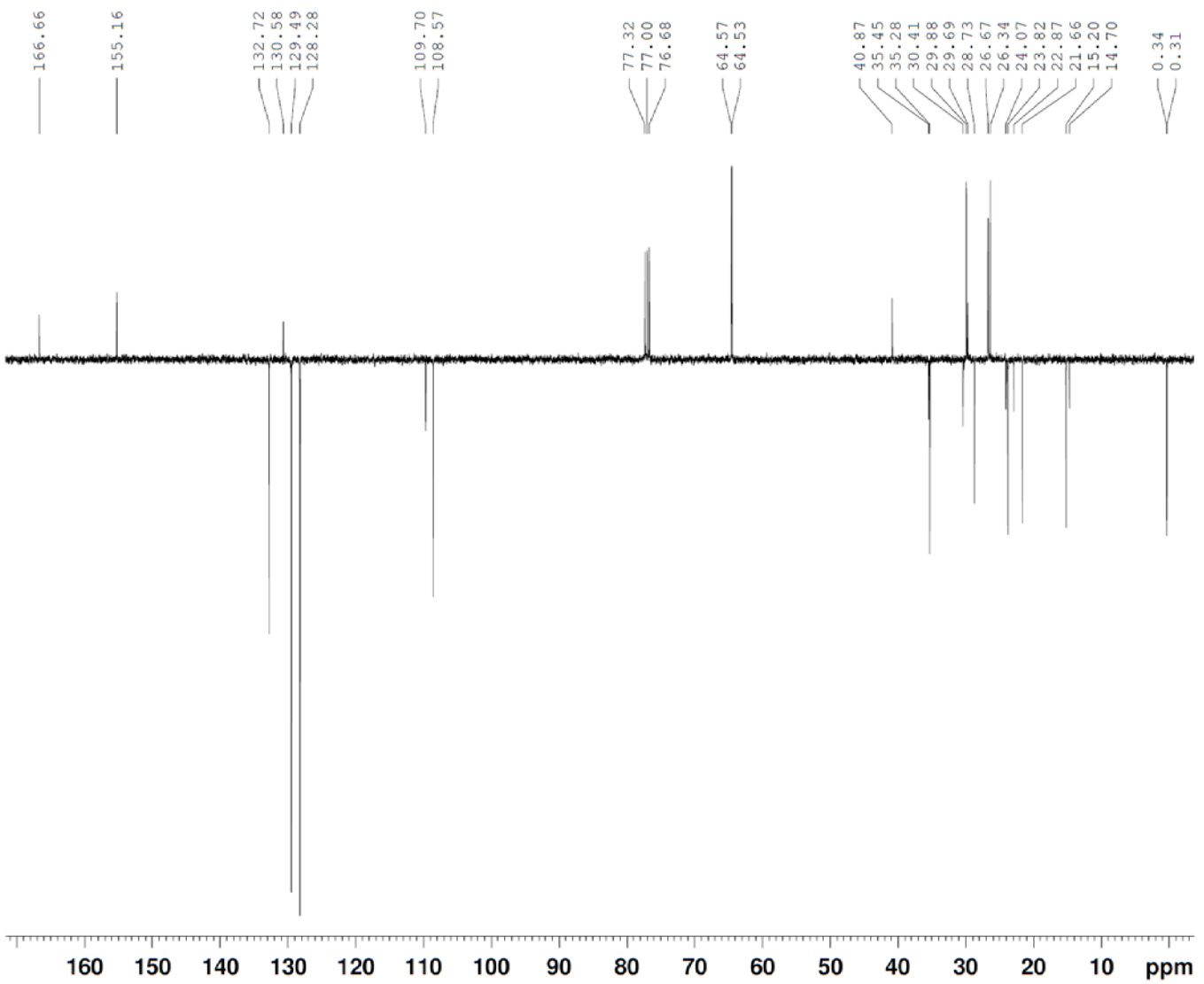


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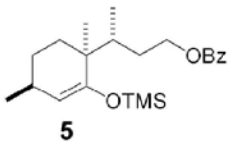
DW                  20.800 usec
DE                   6.00 usec
TE                   295.8 K
CNST11             1.0000000
CNST2            145.0000000
D1                 2.00000000 sec
d20                0.00689655 sec
DELTA              0.00001146 sec
TD0                        1

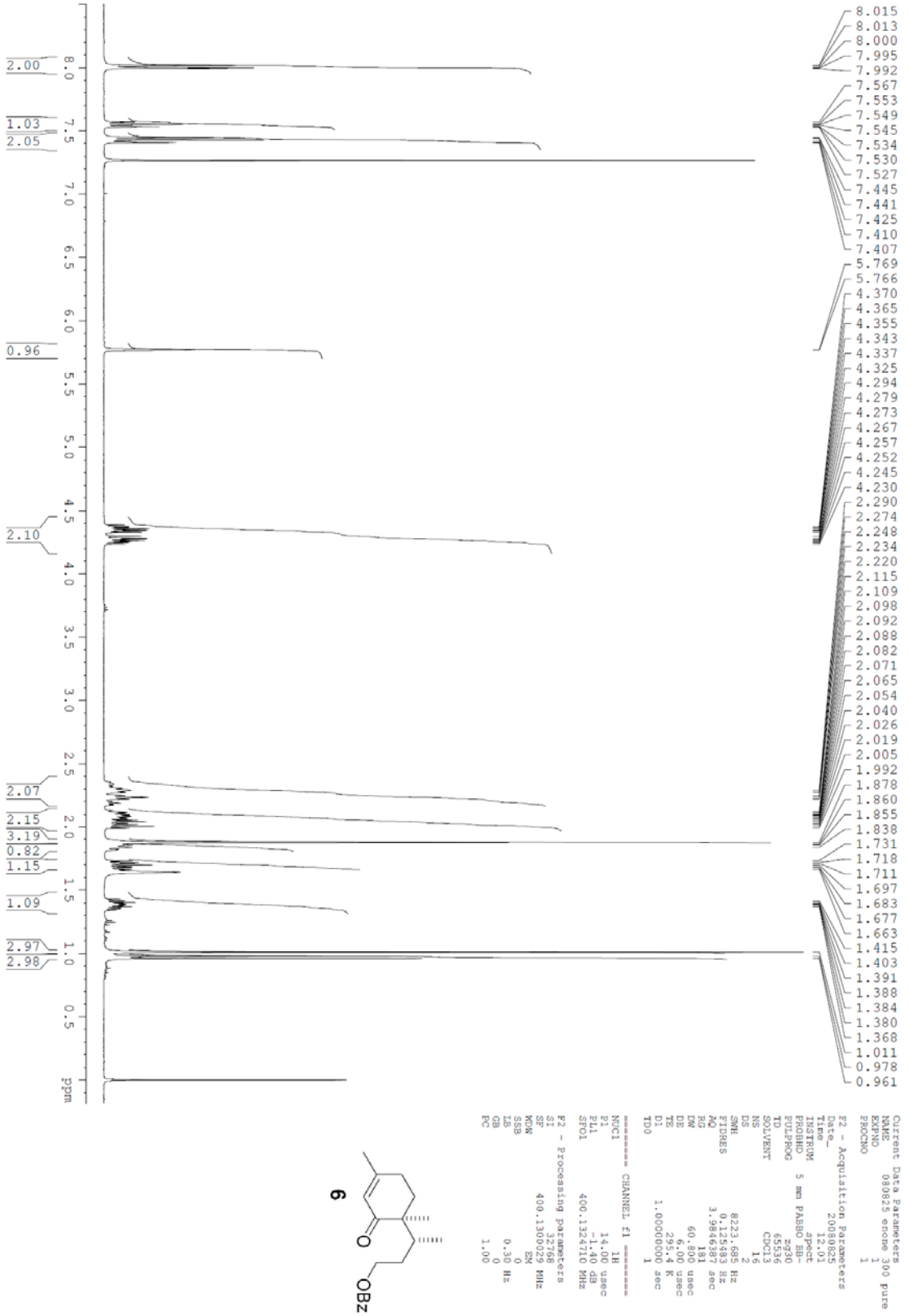
======== CHANNEL f1 ========
NUC1                      13C
P1                     9.00 usec
p2                    18.00 usec
PL1                   -2.00 dB
SFO1           100.6228298 MHz

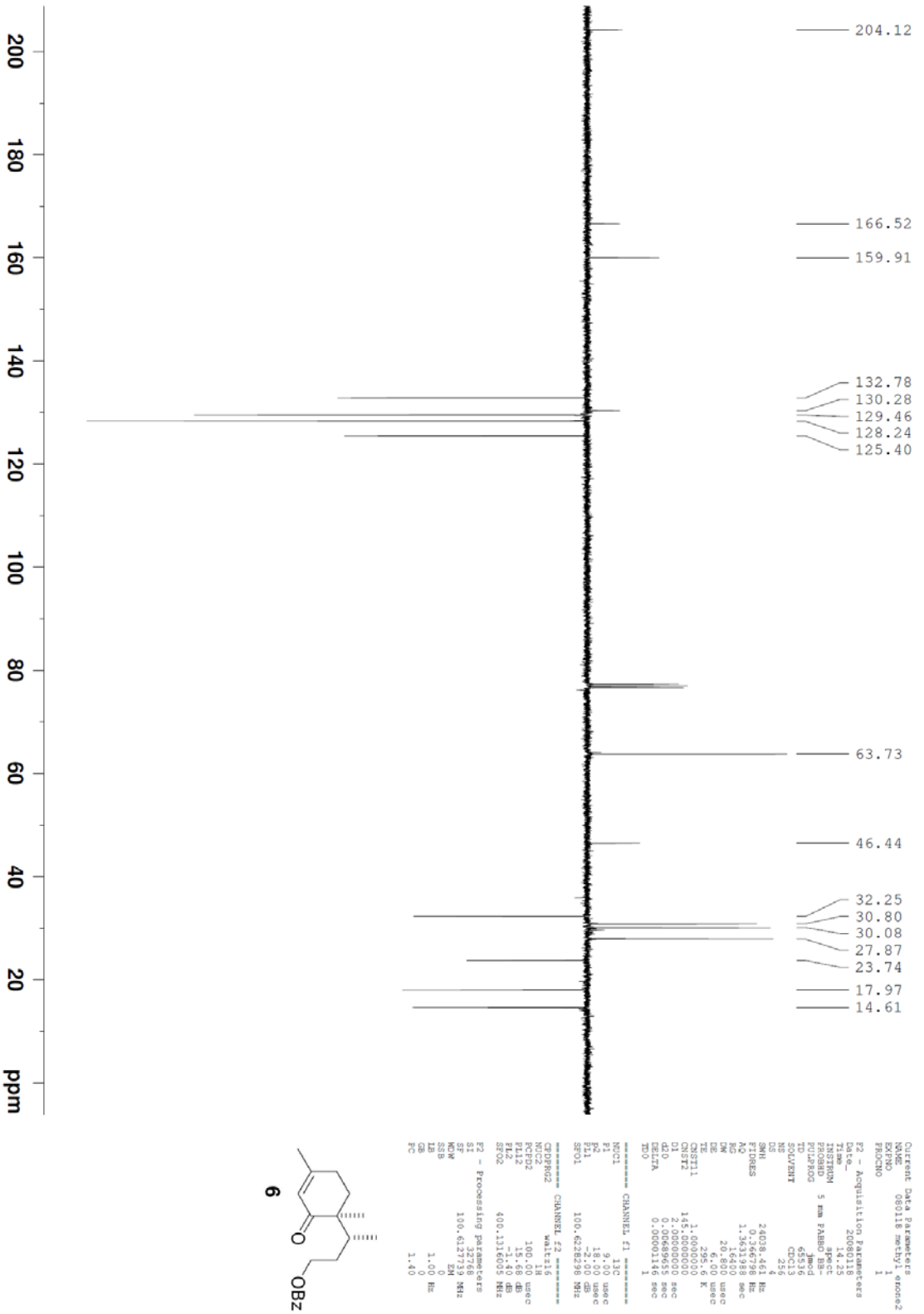
======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                       1H
PCPD2                100.00 usec
PL12                  15.68 dB
PL2                   -1.40 dB
SFO2           400.1316005 MHz

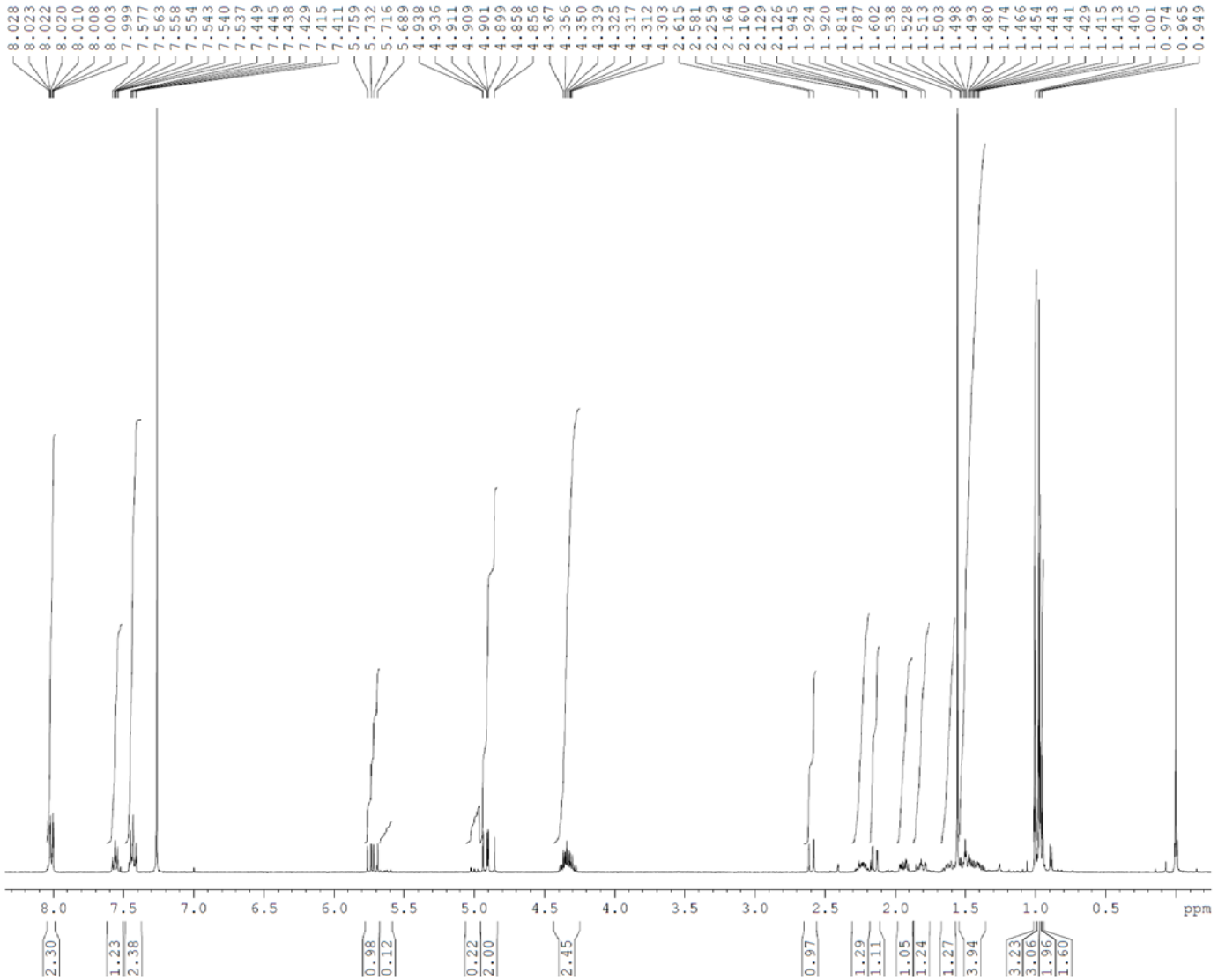
F2 - Processing parameters
SI                        32768
SF             100.6127711 MHz
WDW                          EM
SSB                           0
LB                     1.00 Hz
GB                            0
PC                     1.40
```




**5**


8.015
8.013
8.000
7.995
7.992
7.567
7.553
7.549
7.545
7.534
7.530
7.527
7.445
7.441
7.425
7.410
7.407
5.769
5.766
4.370
4.365
4.355
4.343
4.337
4.325
4.294
4.279
4.273
4.267
4.257
4.252
4.245
4.230
2.290
2.274
2.248
2.234
2.220
2.115
2.109
2.098
2.092
2.088
2.082
2.071
2.065
2.054
2.040
2.026
2.019
2.005
1.992
1.878
1.860
1.855
1.838
1.731
1.718
1.711
1.697
1.683
1.677
1.663
1.415
1.403
1.391
1.388
1.384
1.380
1.368
1.011
0.978
0.961
2.00
1.03
2.05
0.96
2.10
2.07
2.15
3.19
0.82
1.15
1.09
2.97
2.98
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
Current Data Parameters
NAME 080825
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080825
Time 12.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 11.8
DW 60.800 usec
DE 6.00 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.40 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300029 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
6
O
OBz


204.12
166.52
159.91
132.78
130.28
129.46
128.24
125.40
63.73
46.44
32.25
30.80
30.08
27.87
23.74
17.97
14.61
200
180
160
140
120
100
80
60
40
20
ppm
OBz
O
6

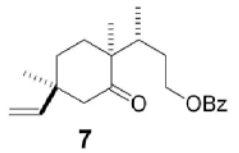



```
Current Data Parameters
NAME      071226 vinyl ketone
EXPNO                       1
PROCNO                      1

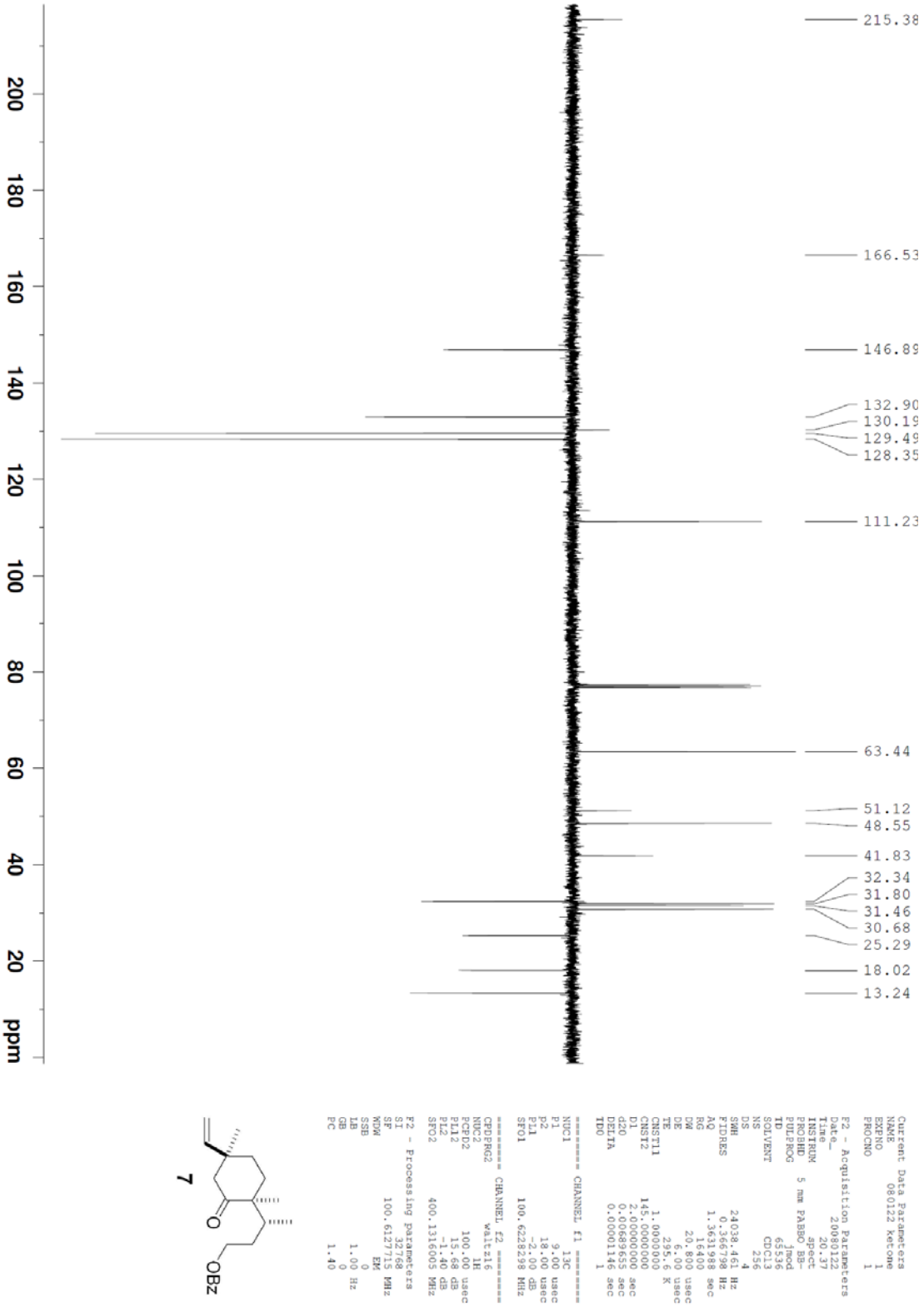
F2 - Acquisition Parameters
Date_               20071226
Time                   14.22
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      322
DW                   60.800 usec
DE                    6.00 usec
TE                   295.1 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                   14.00 usec
PL1                  -1.40 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                        32768
SF             400.1300045 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```




**7**





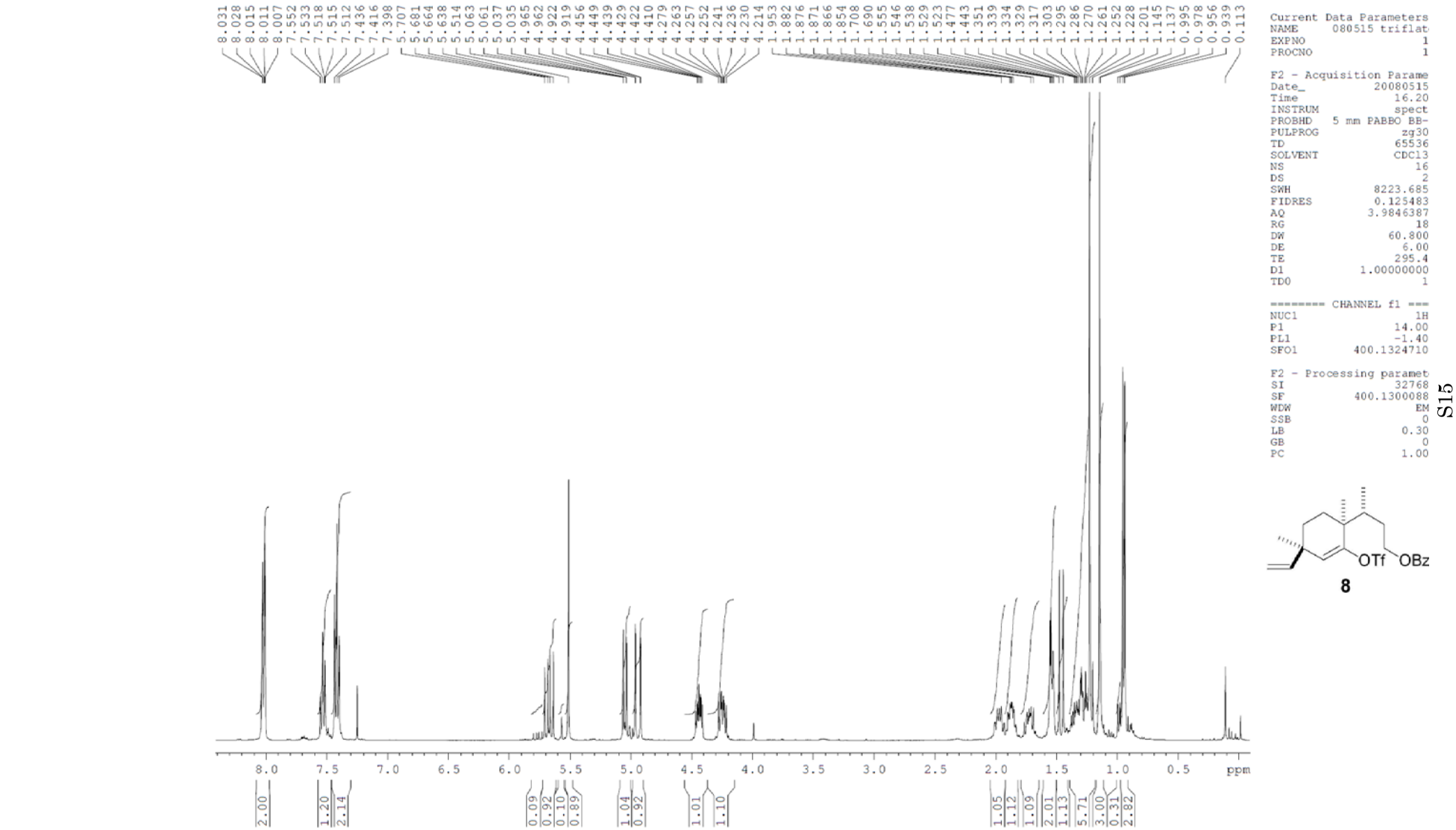
Current Data Parameters
NAME 080122 ketone
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080122
Time 20.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 16400
DW 20.800 usec
DE 6.00 usec
TE 295.6 K
CNST11 1.0000000
CNST12 145.0000000
D1 2.0000000 sec
d20 0.00689655 sec
DELTA 0.00001146 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
P2 18.00 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL12 15.68 dB
PL2 15.40 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127715 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




```
Current Data Parameters
NAME      080515 triflat
EXPNO                  1
PROCNO                 1

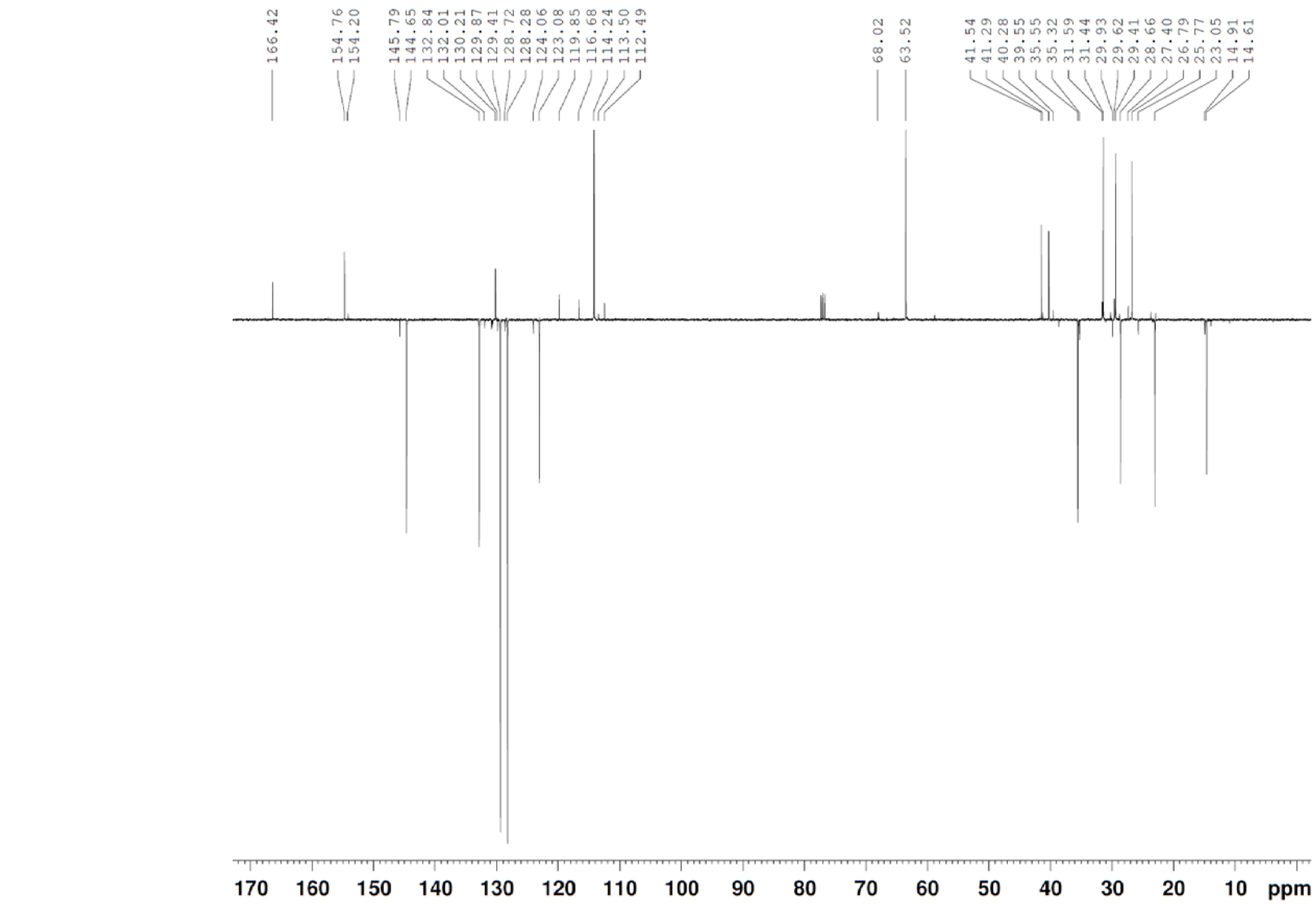
F2 - Acquisition Parame
Date_           20080515
Time               16.20
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685
FIDRES          0.125483
AQ              3.9846387
RG                    18
DW                60.800
DE                 6.00
TE                295.4
D1             1.00000000
TD0                    1

======== CHANNEL f1 ===
NUC1                  1H
P1                 14.00
PL1                -1.40
SFO1          400.1324710

F2 - Processing paramet
SI                 32768
SF            400.1300088
WDW                   EM
SSB                    0
LB                  0.30
GB                     0
PC                  1.00
```




**8**



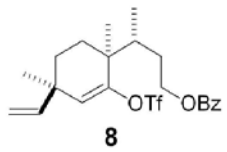

```
Current Data Parameters
NAME      080515 triflate c13
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_               20080515
Time                   16.37
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                jmod
TD                    65536
SOLVENT               CDCl3
NS                      256
DS                        4
SWH               24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                     16400
DW                  20.800 usec
DE                   6.00 usec
TE                   296.2 K
CNST11           1.0000000
CNST2          145.0000000
D1                2.00000000 sec
d20               0.00689655 sec
DELTA             0.00001146 sec
TD0                       1

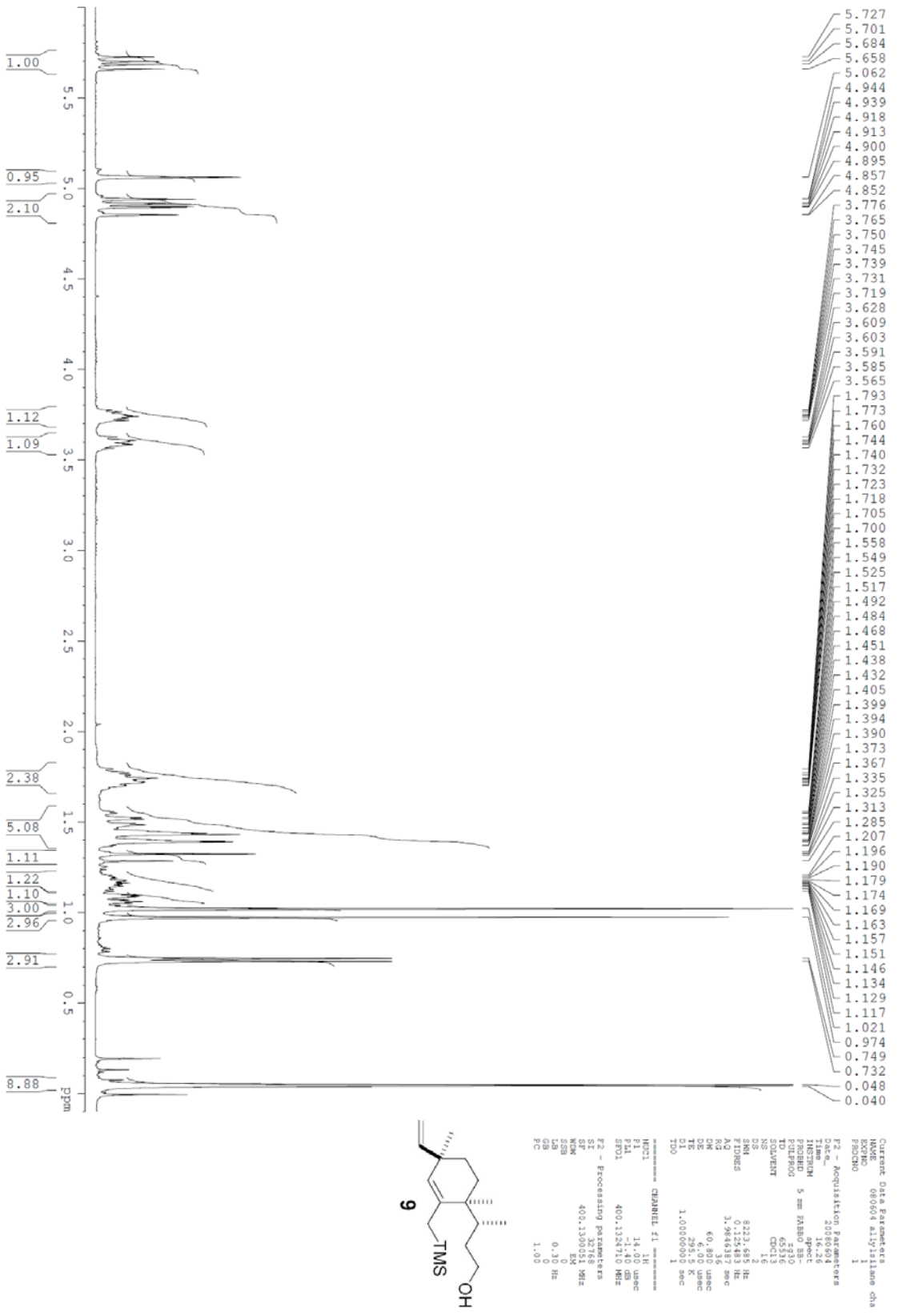
======== CHANNEL f1 ========
NUC1                    13C
P1                   9.00 usec
P2                  18.00 usec
PL1                 -2.00 dB
SFO1          100.6228298 MHz

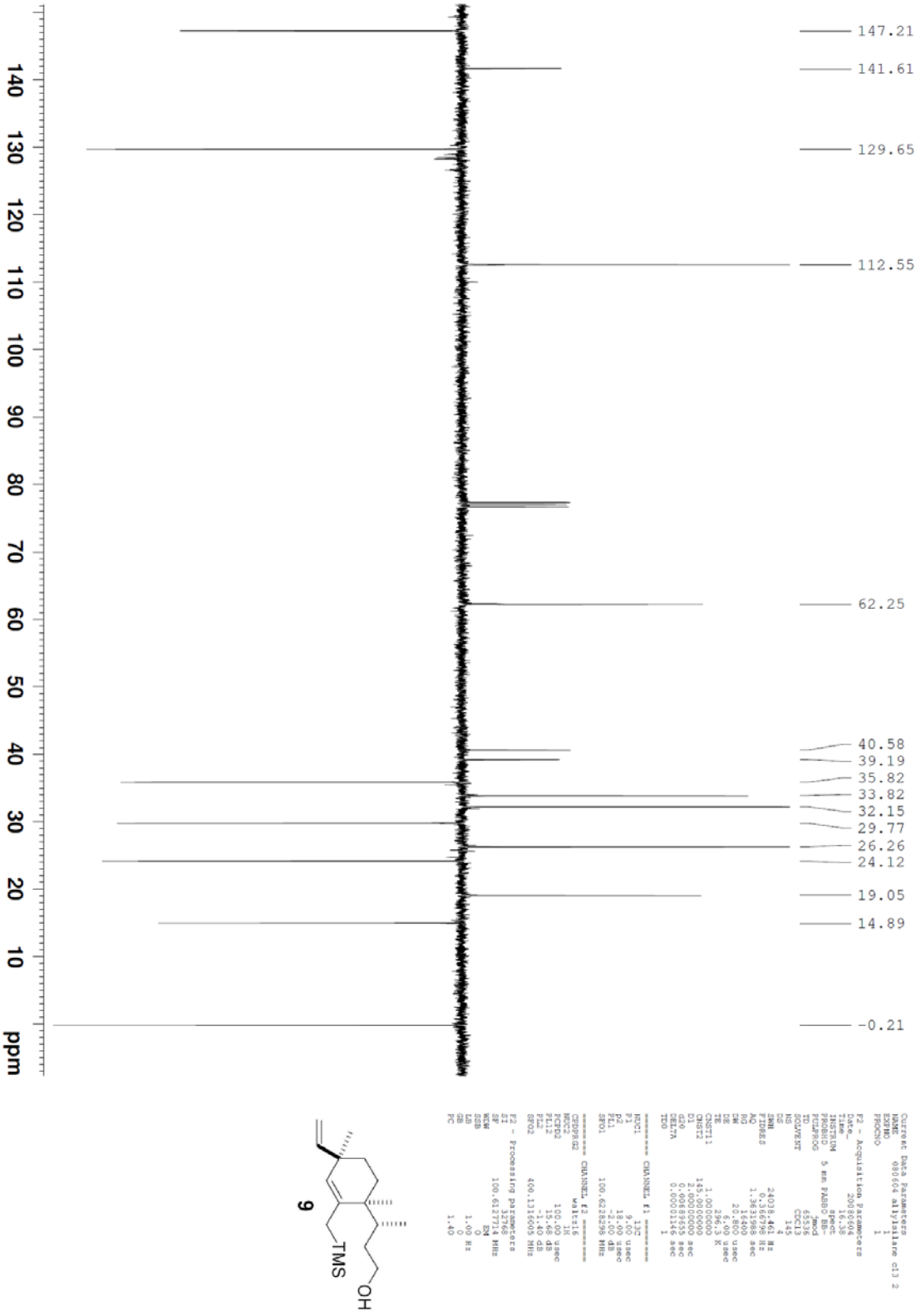
======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2              100.00 usec
PL12                15.68 dB
PL2                 -1.40 dB
SFO2          400.1316005 MHz

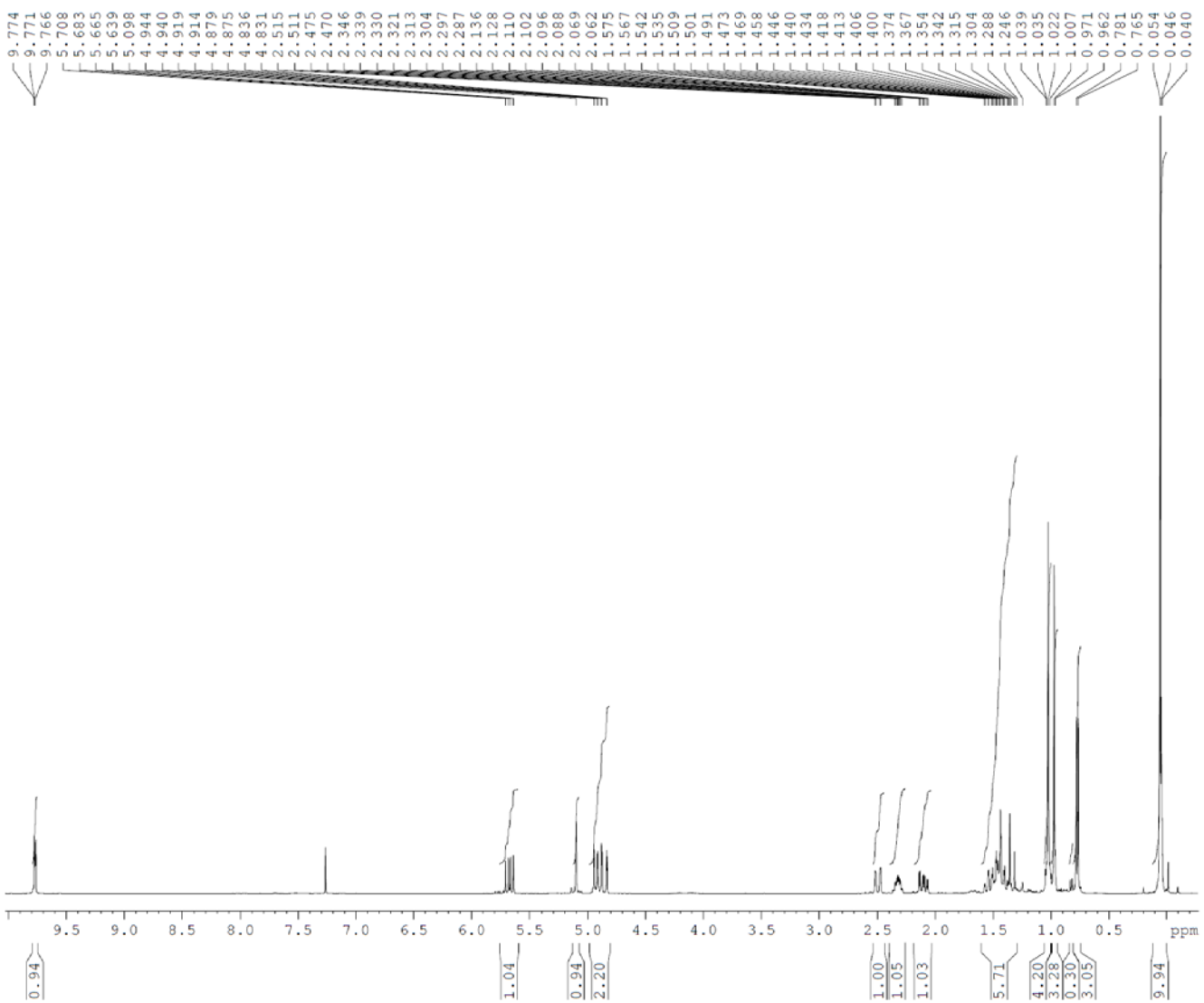
F2 - Processing parameters
SI                      32768
SF            100.6127763 MHz
WDW                        EM
SSB                         0
LB                   1.00 Hz
GB                          0
PC                       1.40
```




**8**


1.00
0.95
2.10
1.12
1.09
2.38
5.08
1.11
1.22
1.10
3.00
2.96
2.91
8.88
ppm
5.727
5.701
5.684
5.658
5.062
4.944
4.939
4.918
4.913
4.900
4.895
4.857
4.852
3.776
3.765
3.750
3.745
3.739
3.731
3.719
3.628
3.609
3.603
3.591
3.585
3.565
1.793
1.773
1.760
1.744
1.740
1.732
1.723
1.718
1.705
1.700
1.558
1.549
1.525
1.517
1.492
1.484
1.468
1.451
1.438
1.432
1.405
1.399
1.394
1.390
1.373
1.367
1.335
1.325
1.313
1.285
1.207
1.196
1.190
1.179
1.174
1.169
1.163
1.157
1.151
1.146
1.134
1.129
1.117
1.021
0.974
0.749
0.732
0.048
0.040
Current Data Parameters
NAME 080604 allylsilane cha
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180604
Time 16:26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125481 Hz
AQ 3.9845187 sec
RG 36
DW 60.800 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.40 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 131072
SF 400.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
TMS
OH


147.21
141.61
129.65
112.55
62.25
40.58
39.19
35.82
33.82
32.15
29.77
26.26
24.12
19.05
14.89
-0.21
ppm
140 130 120 110 100 90 80 70 60 50 40 30 20 10
9
TMS
OH

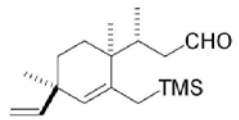



```
Current Data Parameters
NAME      080519 aldehyde
EXPNO                  1
PROCNO                 1

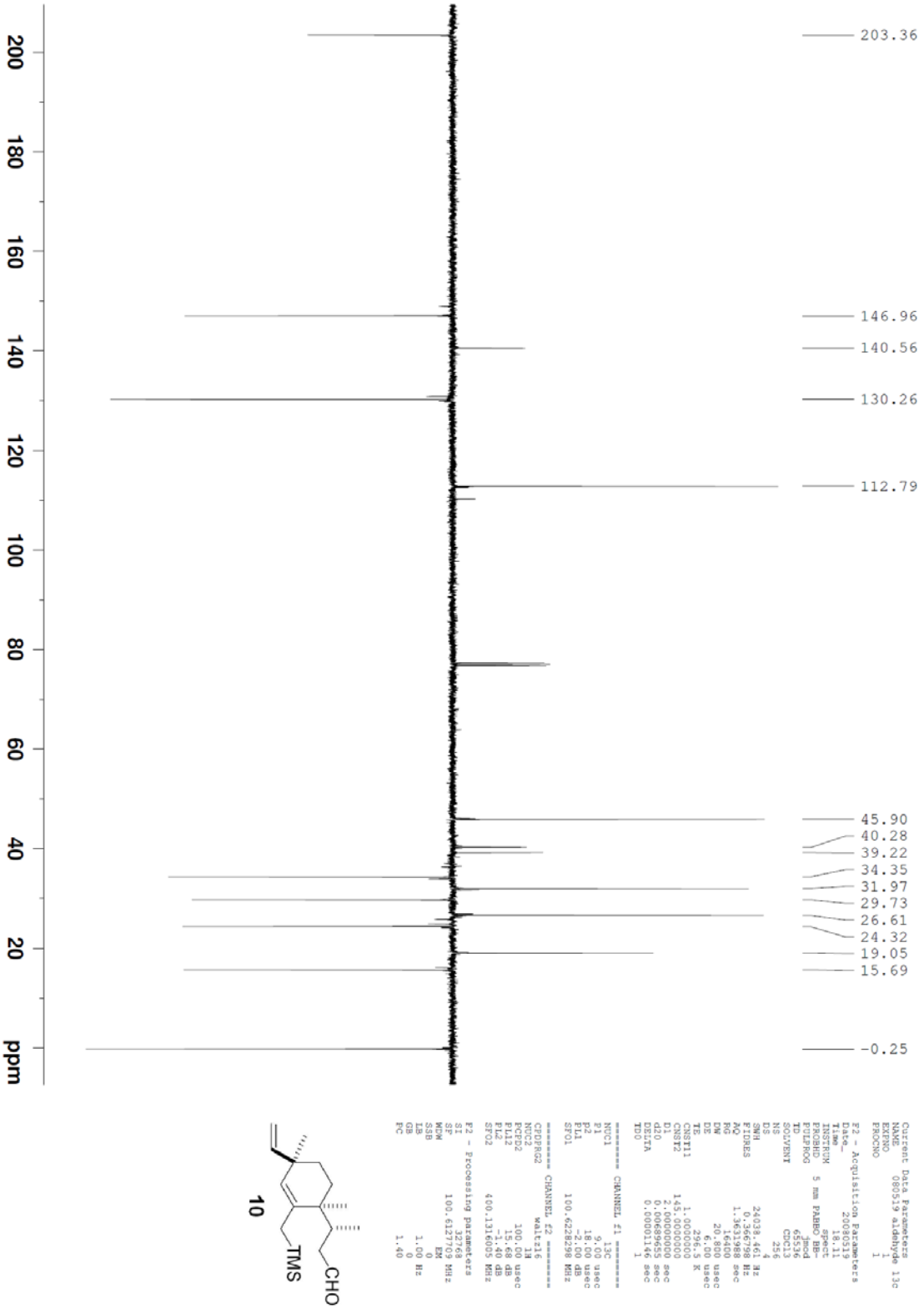
F2 - Acquisition Parameters
Date_           20080519
Time               17.53
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                    36
DW                60.800 usec
DE                 6.00 usec
TE                295.6 K
D1             1.00000000 sec
TD0                    1

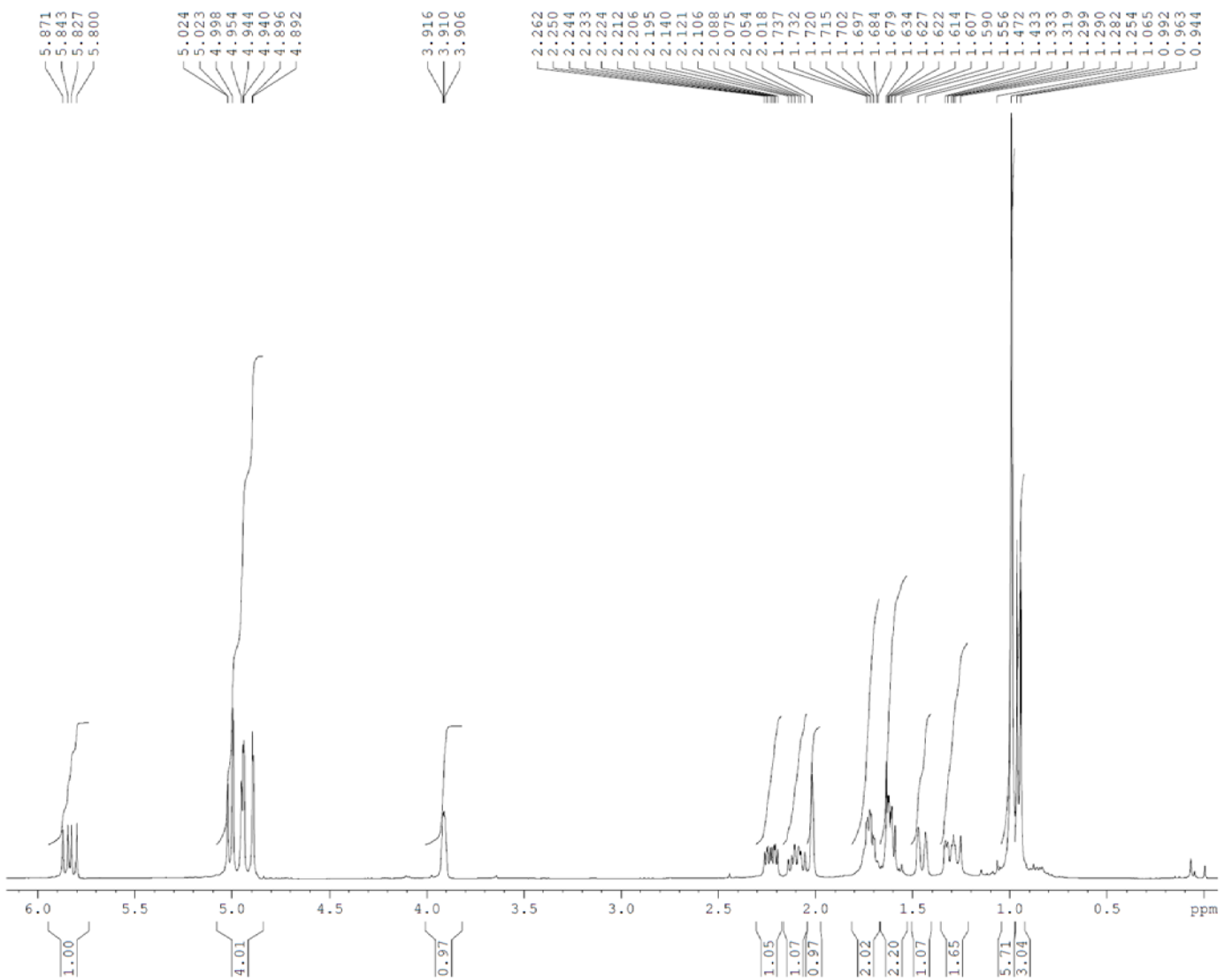
======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                -1.40 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF            400.1300051 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```




**10**


203.36
146.96
140.56
130.26
112.79
45.90
40.28
39.22
34.35
31.97
29.73
26.61
24.32
19.05
15.69
-0.25
ppm
200
180
160
140
120
100
80
60
40
20
10
TMS
CHO

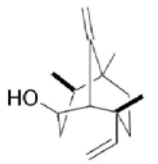



```
Current Data Parameters
NAME      080616 exoalchol 1H
EXPNO                   1
PROCNO                  1

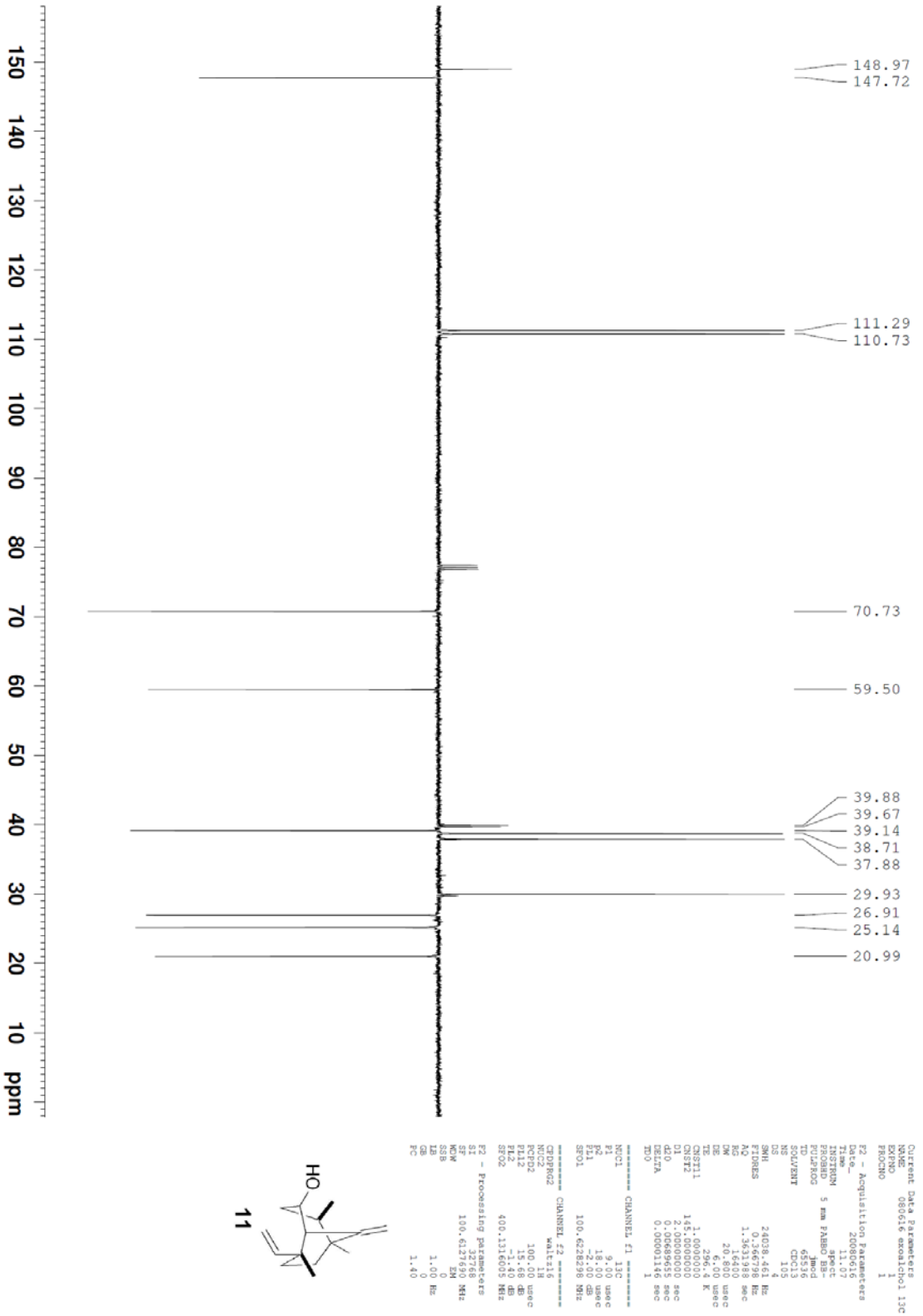
F2 - Acquisition Parameters
Date_            20080616
Time                10.57
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     18
DW                60.800 usec
DE                 6.00 usec
TE                295.6 K
D1             1.00000000 sec
TD0                     1

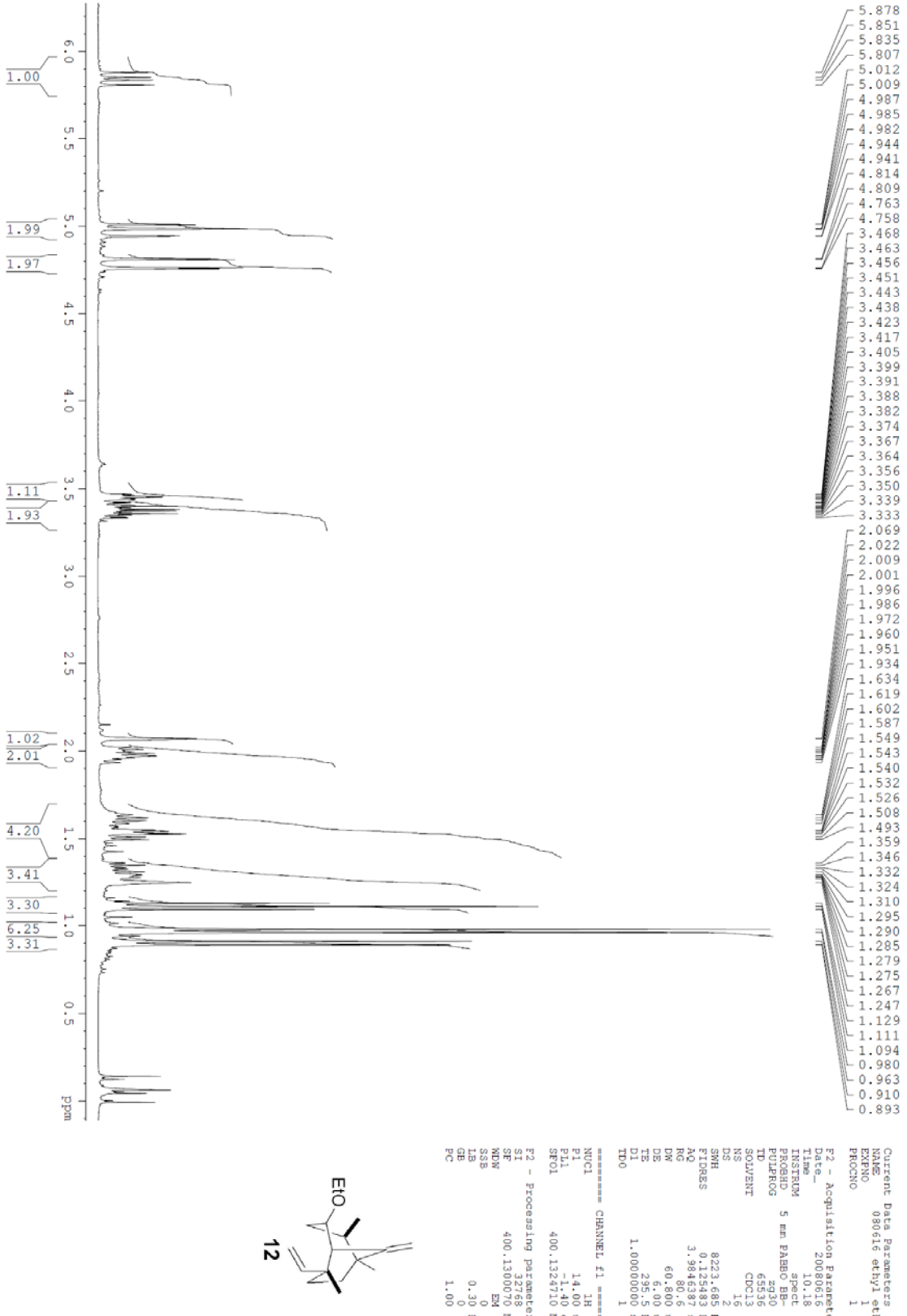
======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PL1                -1.40 dB
SFO1          400.1324710 MHz

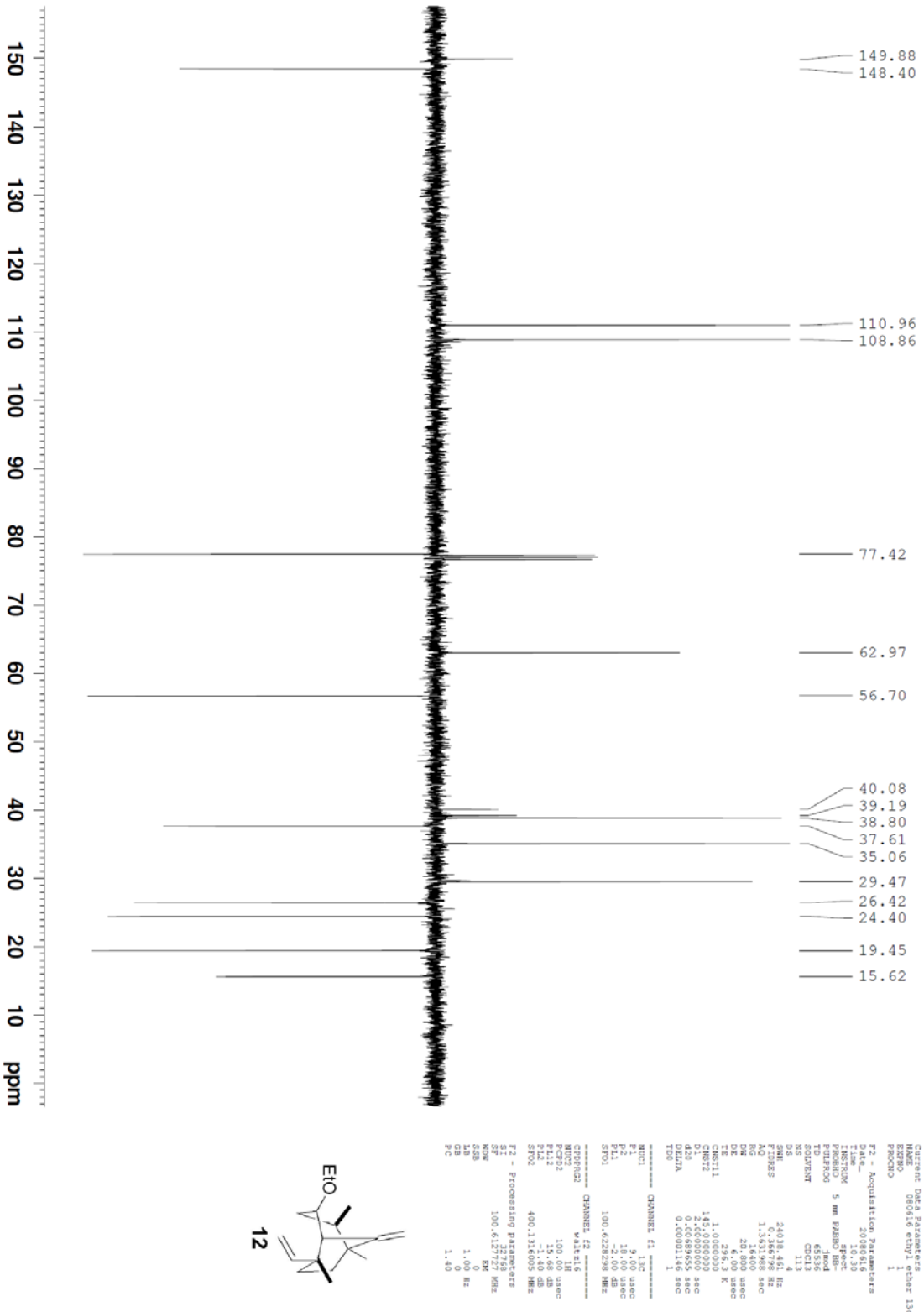
F2 - Processing parameters
SI                  32768
SF            400.1299952 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

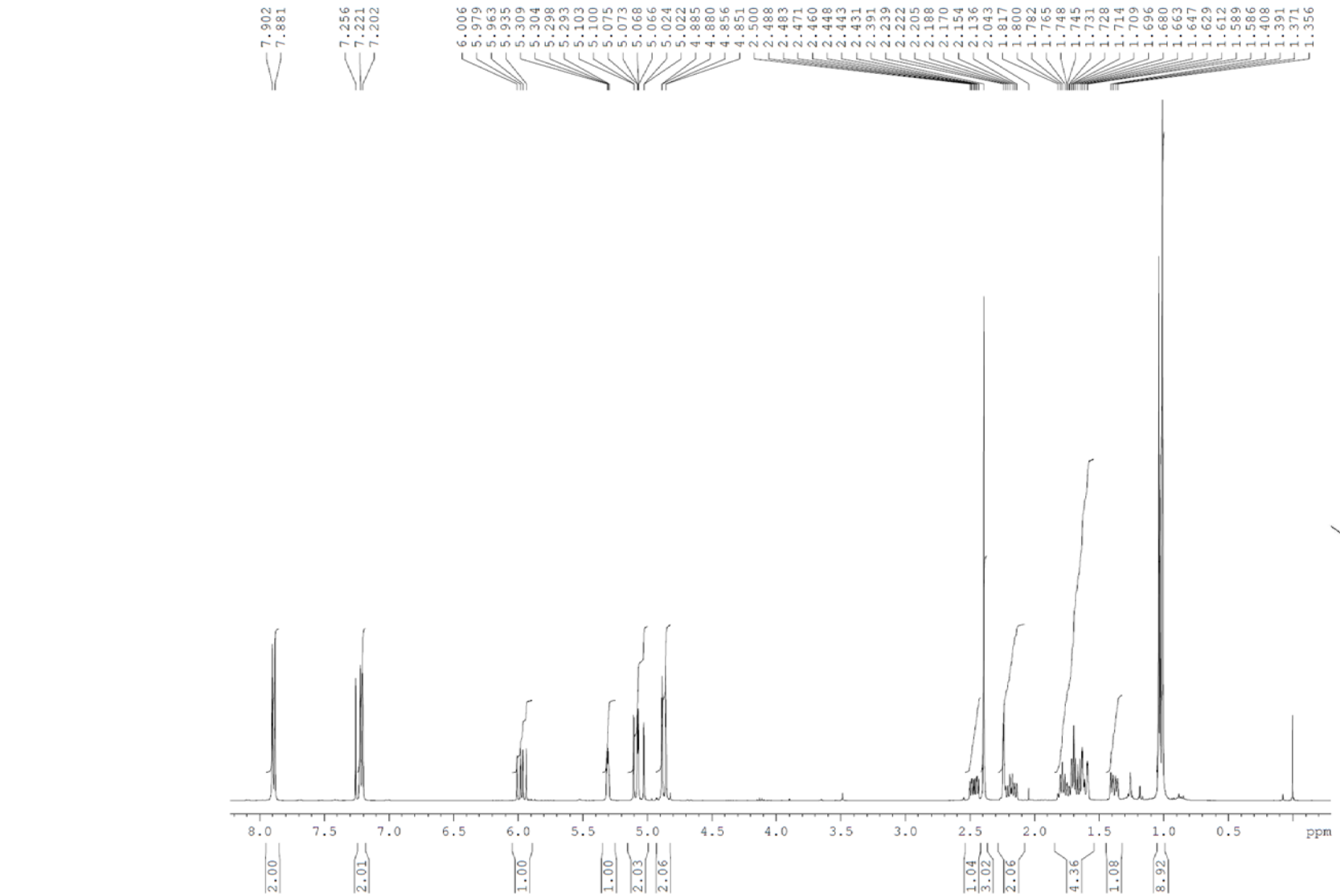



**11**


148.97
147.72
111.29
110.73
70.73
59.50
39.88
39.67
39.14
38.71
37.88
29.93
26.91
25.14
20.99
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
HO
11


1.00
1.99
1.97
1.11
1.93
1.02
2.01
4.20
3.41
3.30
6.25
3.31
ppm
5.878
5.851
5.835
5.807
5.012
5.009
4.987
4.985
4.982
4.944
4.941
4.814
4.809
4.763
4.758
3.468
3.463
3.456
3.451
3.443
3.438
3.423
3.417
3.405
3.399
3.391
3.388
3.382
3.374
3.367
3.364
3.356
3.350
3.339
3.333
2.069
2.022
2.009
2.001
1.996
1.986
1.972
1.960
1.951
1.934
1.634
1.619
1.602
1.587
1.549
1.543
1.540
1.532
1.526
1.508
1.493
1.359
1.346
1.332
1.324
1.310
1.295
1.290
1.285
1.279
1.275
1.267
1.247
1.129
1.111
1.094
0.980
0.963
0.910
0.893
Current Data Parameters
NAME 080616 ethyl eth
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20080616
Time 10.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 H
FIDRES 0.125483 H
AQ 3.9846387 s
RG 80.6
DW 60.800 u
DE 6.00 u
TE 295.5 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 u
PL1 -1.40 d
SFO1 400.1324710 M
F2 - Processing parameter
SI 32768
SF 400.1300070 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00
EtO
12


149.88
148.40
110.96
108.86
77.42
62.97
56.70
40.08
39.19
38.80
37.61
35.06
29.47
26.42
24.40
19.45
15.62
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
EtO
12

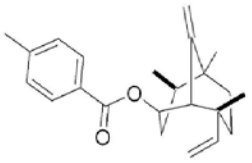



Current Data Parameters
NAME 081213 p-toluat
EXPNO 1
PROCNO 1

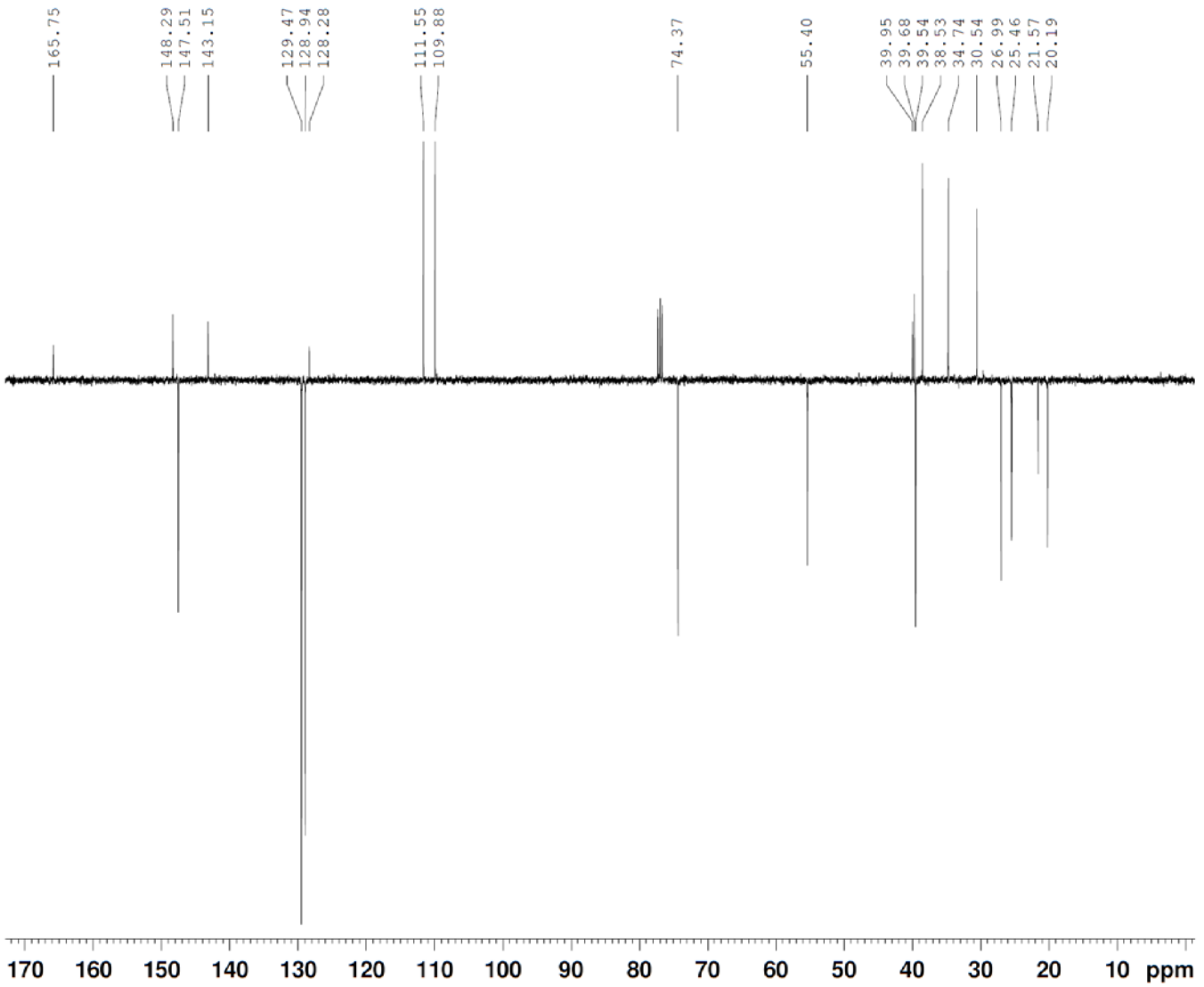
F2 - Acquisition Paramet
Date_ 20081213
Time 15.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685
FIDRES 0.125483
AQ 3.9846387
RG 71.8
DW 60.800
DE 6.00
TE 294.9
D1 1.00000000
TD0 1

======== CHANNEL f1 ====
NUC1 1H
P1 12.30
PL1 -1.00
SFO1 400.1324710

F2 - Processing paramete
SI 32768
SF 400.1300065
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00



**13**



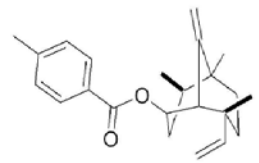

```
Current Data Parameters
NAME      081213 p-toluate 13c
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20081213
Time              15.45
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           jmod
TD               65536
SOLVENT          CDCl3
NS                 205
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                16400
DW               20.800 usec
DE                6.00 usec
TE               296.5 K
CNST11        1.0000000
CNST2       145.0000000
D1            2.00000000 sec
d20           0.00689655 sec
DELTA         0.00001019 sec
TD0                  1

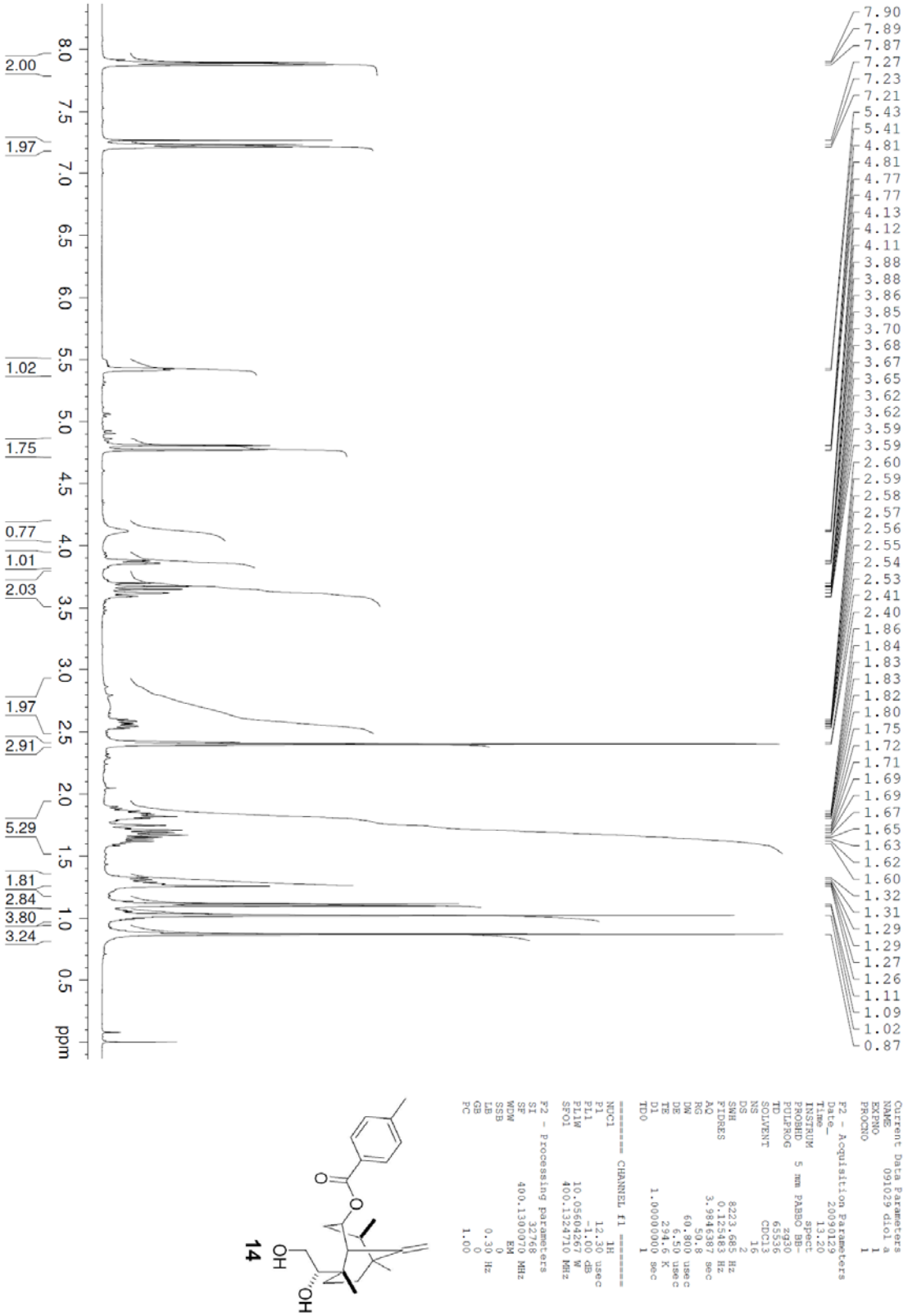
======== CHANNEL f1 ========
NUC1                13C
P1                 8.00 usec
p2                16.00 usec
PL1               -2.00 dB
SFO1        100.6228296 MHz

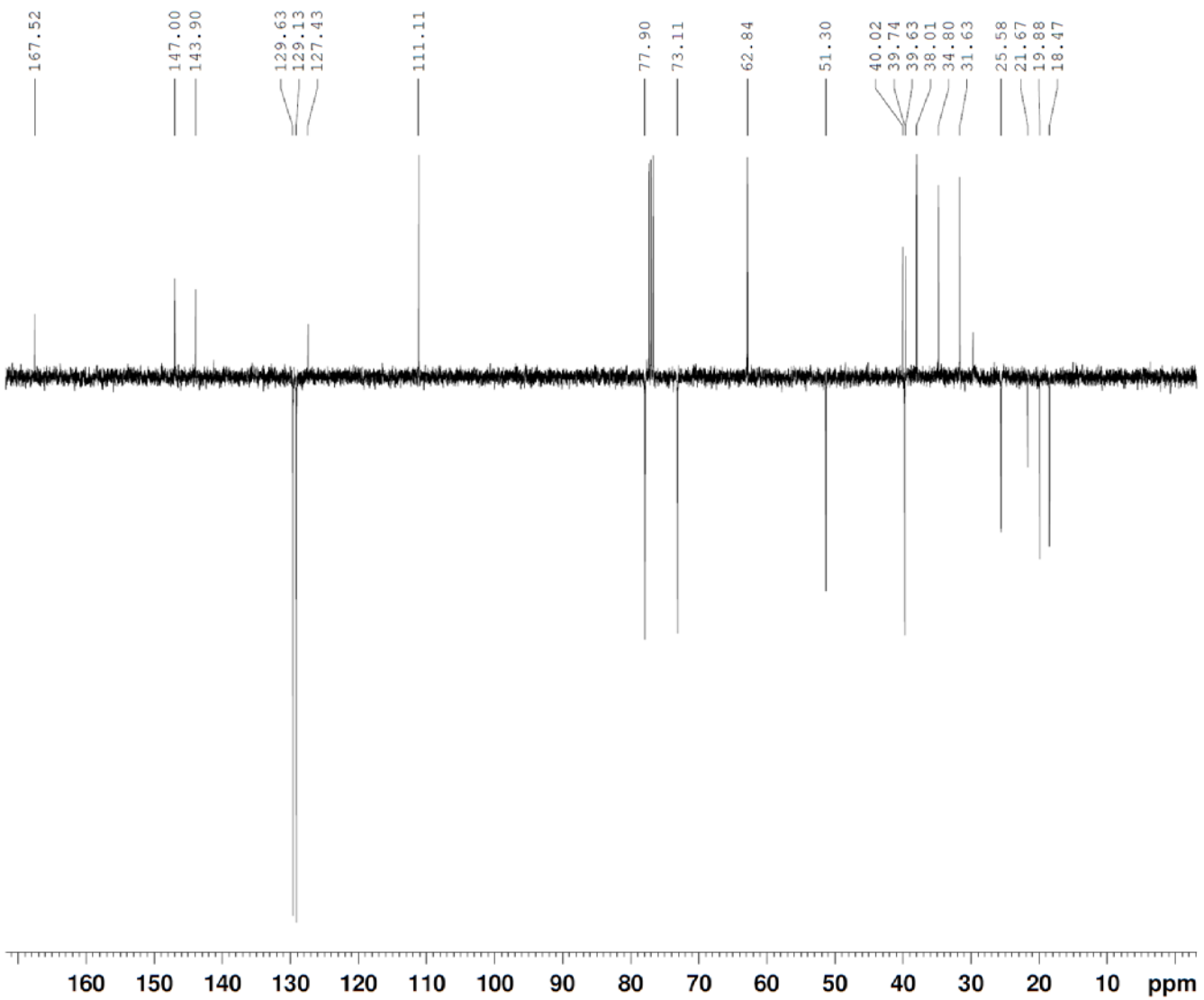
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             80.00 usec
PL12              15.26 dB
PL2               -1.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127753 MHz
WDW                  EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```




**13**


2.00
1.97
1.02
1.75
0.77
1.01
2.03
1.97
2.91
5.29
1.81
2.84
3.80
3.24
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
7.90
7.89
7.87
7.27
7.23
7.21
5.43
5.41
4.81
4.81
4.77
4.77
4.13
4.12
4.11
3.88
3.88
3.86
3.85
3.70
3.68
3.67
3.65
3.62
3.62
3.59
3.59
2.60
2.59
2.58
2.57
2.56
2.55
2.54
2.53
2.41
2.40
1.86
1.84
1.83
1.83
1.82
1.80
1.75
1.72
1.71
1.69
1.69
1.67
1.65
1.63
1.62
1.60
1.32
1.31
1.29
1.29
1.27
1.26
1.11
1.09
1.02
0.87
Current Data Parameters
NAME 091029 diol 1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090129
Time 13.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 50.8
DW 60.800 usec
DE 6.50 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.30 usec
PL1 -1.00 dB
PL1W 10.05604267 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
OH
OH
14



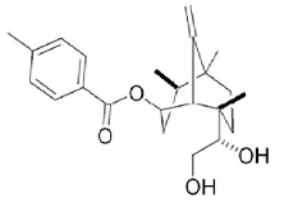

```
Current Data Parameters
NAME      081218 diol a 13c
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20081217
Time                  19.20
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                jmod
TD                    65536
SOLVENT               CDCl3
NS                      256
DS                        4
SWH               24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                    16400
DW                  20.800 usec
DE                   6.00 usec
TE                   295.6 K
CNST11           1.0000000
CNST2          145.0000000
D1               2.00000000 sec
d20              0.00689655 sec
DELTA            0.00001019 sec
TD0                       1

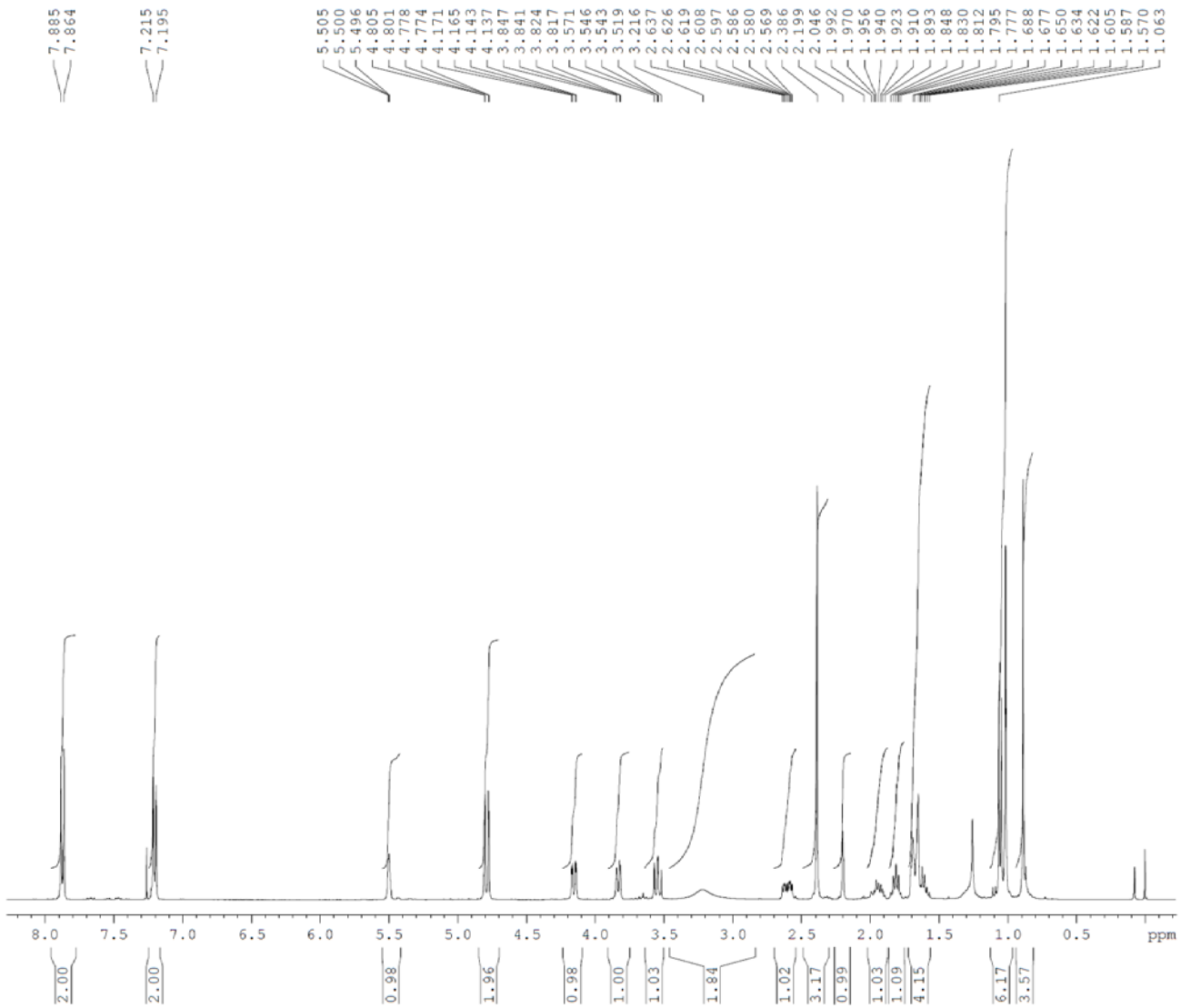
======== CHANNEL f1 ========
NUC1                    13C
P1                    8.00 usec
p2                   16.00 usec
PL1                  -2.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                80.00 usec
PL12                 15.26 dB
PL2                  -1.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                      32768
SF            100.6127714 MHz
WDW                        EM
SSB                         0
LB                    1.00 Hz
GB                          0
PC                       1.40
```




**14**

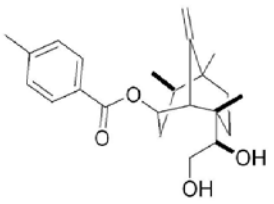



```
Current Data Parameters
NAME      081216  diol  blue
EXPNO                    1
PROCNO                   1

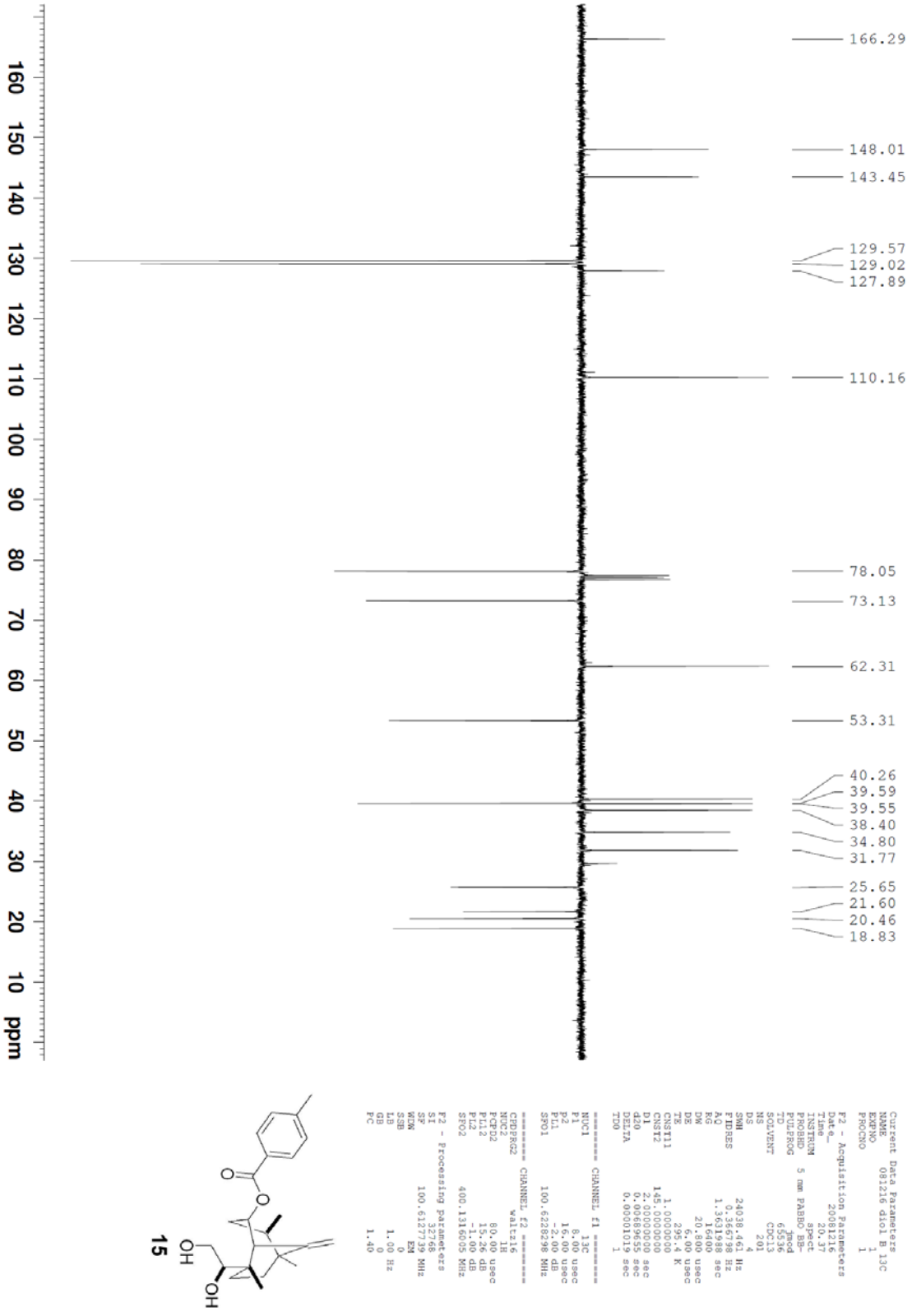
F2 - Acquisition Parameters
Date_              20081216
Time                  19.13
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                       64
DW                    60.800 use
DE                     6.00 use
TE                    294.4 K
D1              1.00000000 sec
TD0                      1

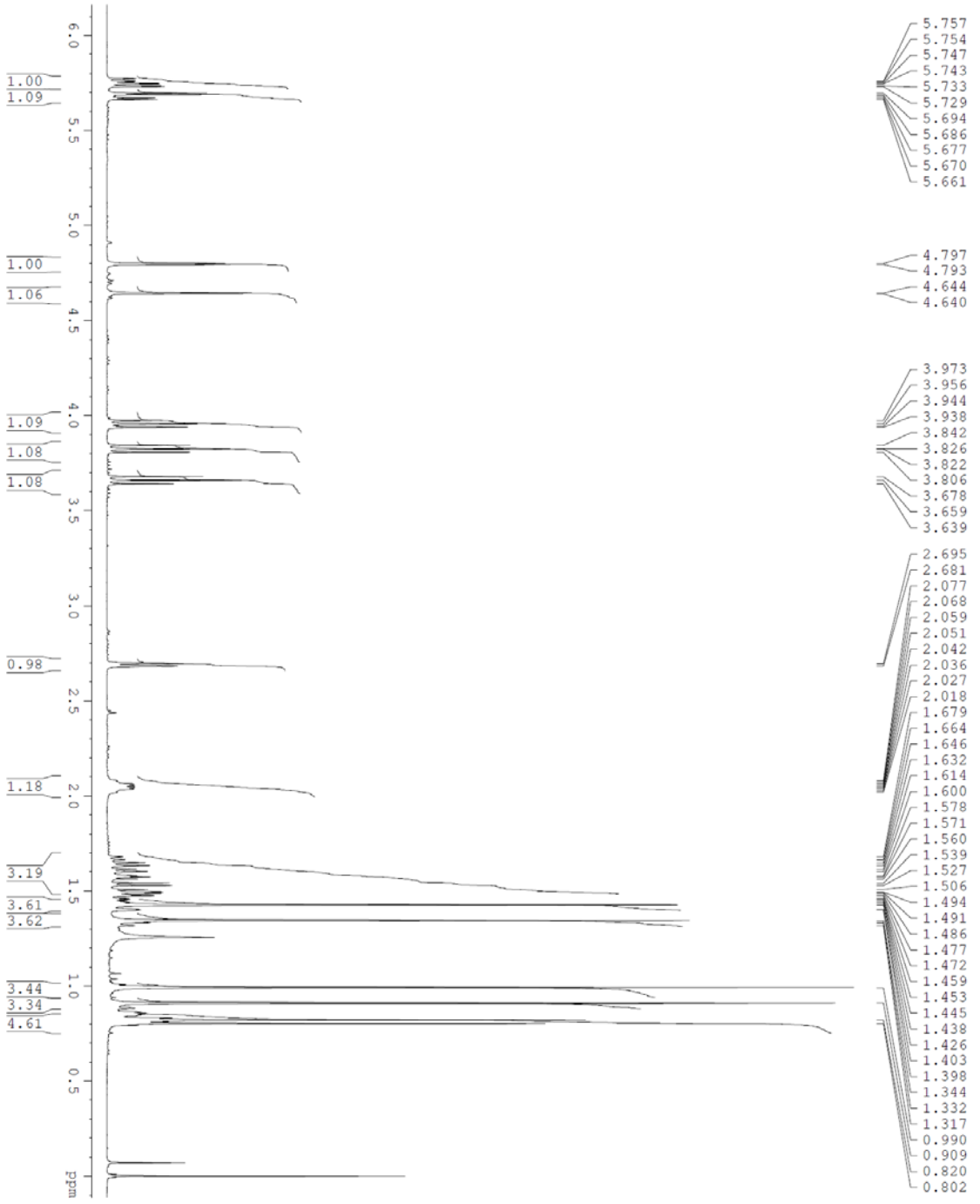
======== CHANNEL f1 =======
NUC1                     1H
P1                    12.30 use
PL1                   -1.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                       32768
SF             400.1300038 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

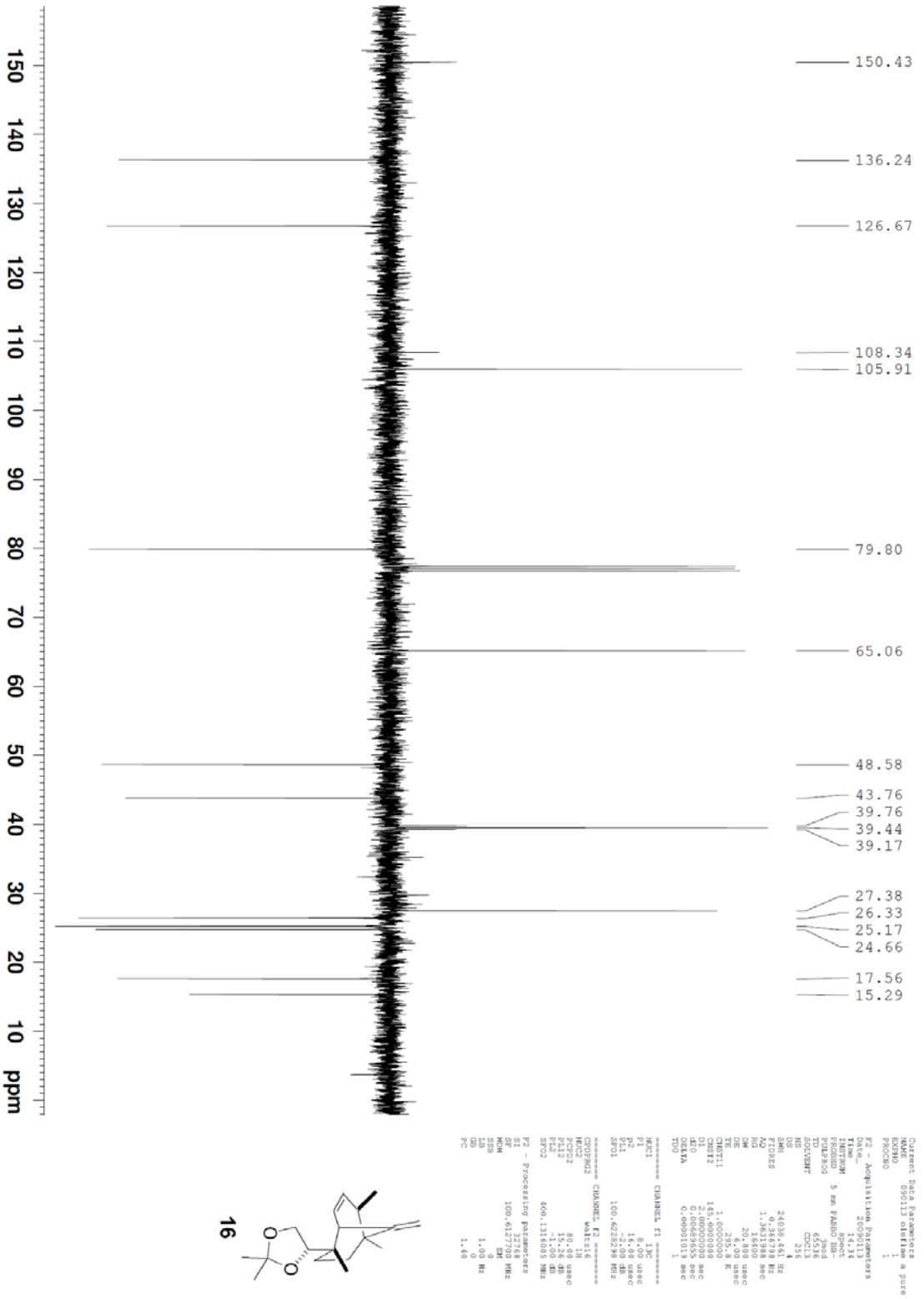


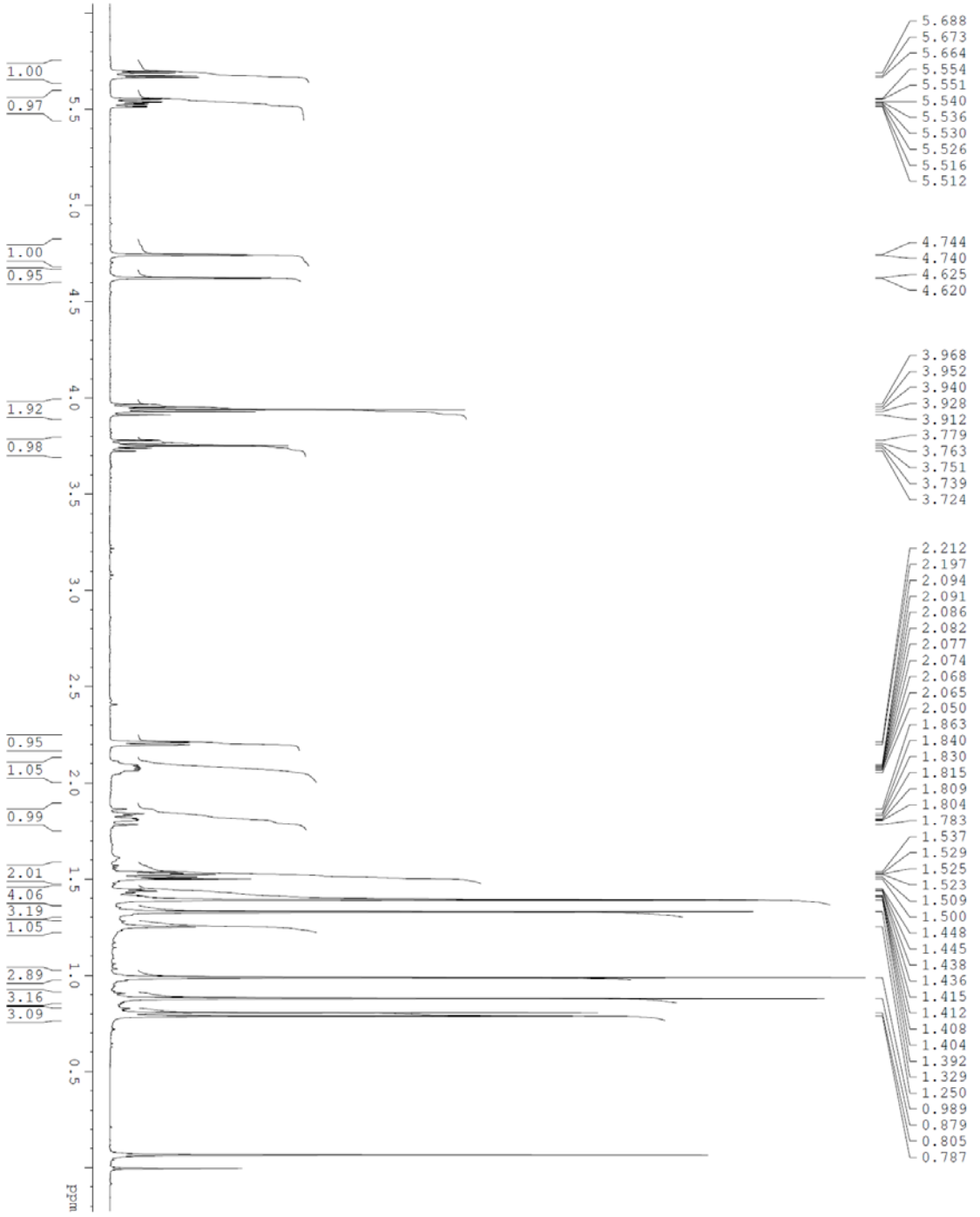

**15**


166.29
148.01
143.45
129.57
129.02
127.89
110.16
78.05
73.13
62.31
53.31
40.26
39.59
39.55
38.40
34.80
31.77
25.65
21.60
20.46
18.83
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
OH
OH
O
O
15


5.757
5.754
5.747
5.743
5.733
5.729
5.694
5.686
5.677
5.670
5.661
4.797
4.793
4.644
4.640
3.973
3.956
3.944
3.938
3.842
3.826
3.822
3.806
3.678
3.659
3.639
2.695
2.681
2.077
2.068
2.059
2.051
2.042
2.036
2.027
2.018
1.679
1.664
1.646
1.632
1.614
1.600
1.578
1.571
1.560
1.539
1.527
1.506
1.494
1.491
1.486
1.477
1.472
1.459
1.453
1.445
1.438
1.426
1.403
1.398
1.344
1.332
1.317
0.990
0.909
0.820
0.802
1.00
1.09
1.00
1.06
1.09
1.08
1.08
0.98
1.18
3.19
3.61
3.62
3.44
3.34
4.61
ppm

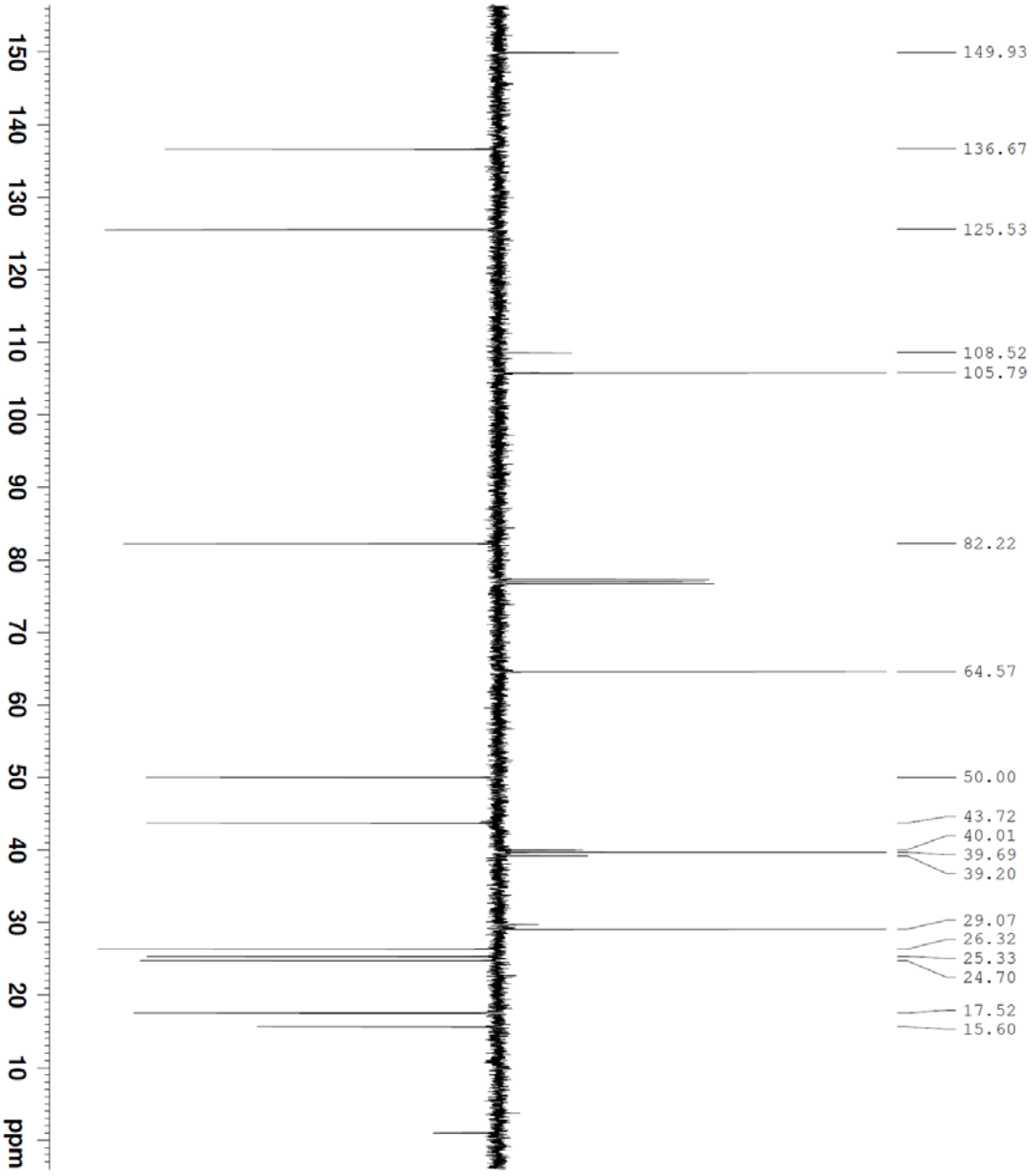
16

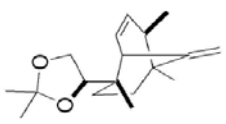

150.43
136.24
126.67
108.34
105.91
79.80
65.06
48.58
43.76
39.76
39.44
39.17
27.38
26.33
25.17
24.66
17.56
15.29
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
16


5.688
5.673
5.664
5.554
5.551
5.540
5.536
5.530
5.526
5.516
5.512
4.744
4.740
4.625
4.620
3.968
3.952
3.940
3.928
3.912
3.779
3.763
3.751
3.739
3.724
2.212
2.197
2.094
2.091
2.086
2.082
2.077
2.074
2.068
2.065
2.050
1.863
1.840
1.830
1.815
1.809
1.804
1.783
1.537
1.529
1.525
1.523
1.509
1.500
1.448
1.445
1.438
1.436
1.415
1.412
1.408
1.404
1.392
1.329
1.250
0.989
0.879
0.805
0.787
1.00
0.97
1.00
0.95
1.92
0.98
0.95
1.05
0.99
2.01
4.06
3.19
1.05
2.89
3.16
3.09
ppm

17

Current Data Parameters
NAME 090108 elimination prc
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090108
Time 12.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125468
AQ 3.9845187 sec
RG 101
DW 60.800 usec
DE 6.00 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.30 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300052 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



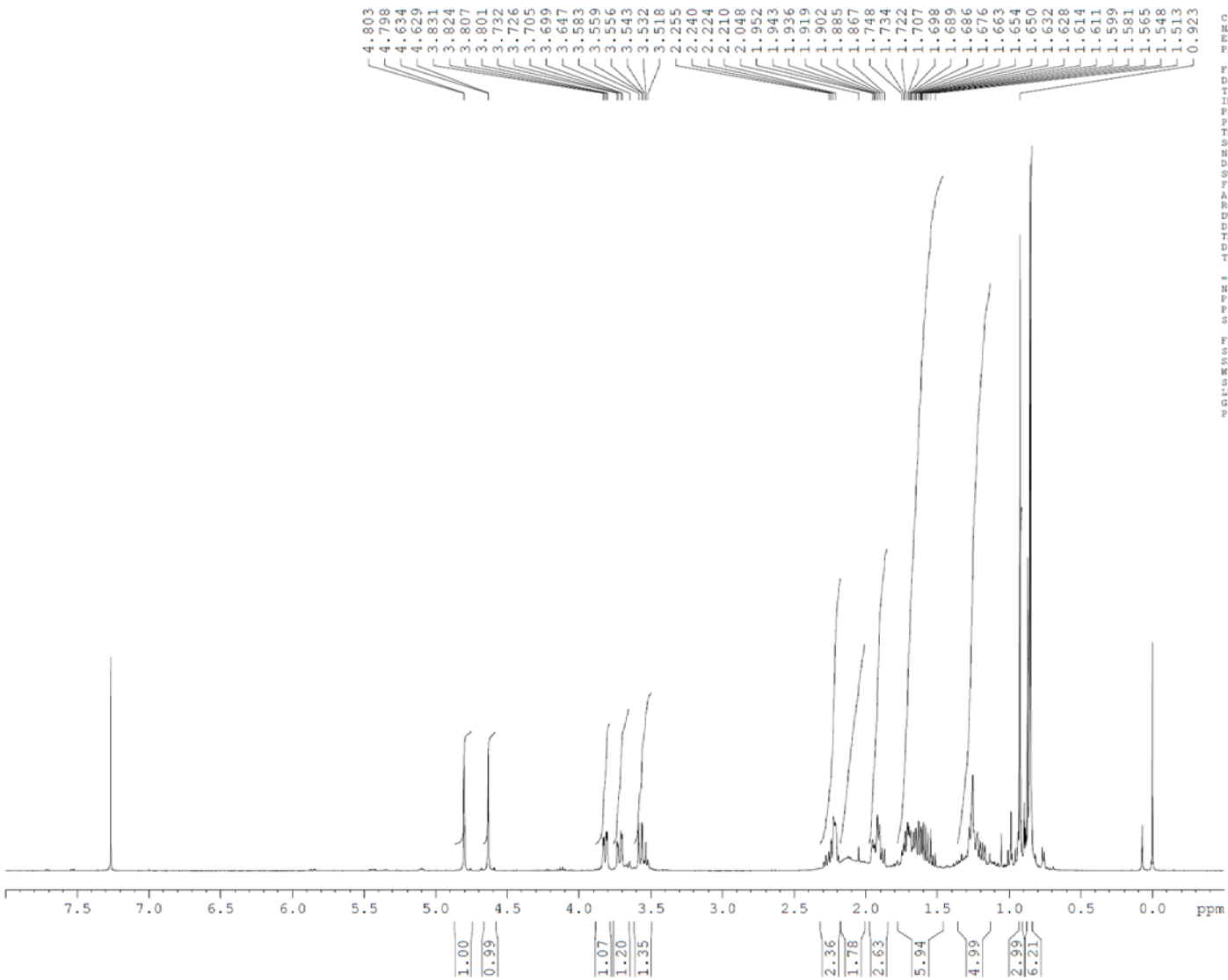

```
Current Data Parameters
NAME          090108 elimination product b 13c
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_         20090108
Time              13.06
INSTRUM           spect
PROBHD     5 mm PABBO BB-
PULPROG           jmod
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    0
SWH           24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                16400
DW               20.800 usec
DE                6.00 usec
TE               295.6 K
CNST11      1.0000000
CNST2     145.0000000
D1          2.0000000 sec
d20         0.0068963 sec
DELTA       0.0000101 sec
TD0               1

======== CHANNEL f1 ========
NUC1              13c
P1                8.00 usec
p2               16.00 usec
PL1              -2.00 dB
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2            80.00 usec
PL12             15.26 dB
PL2              -1.00 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF         100.6127704 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC                1.40
```

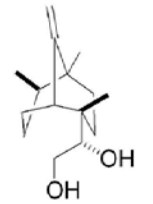



```
Current Data Parameters
NAME      090116 trifarienol a pure
EXPNO                1
PROCNO               1

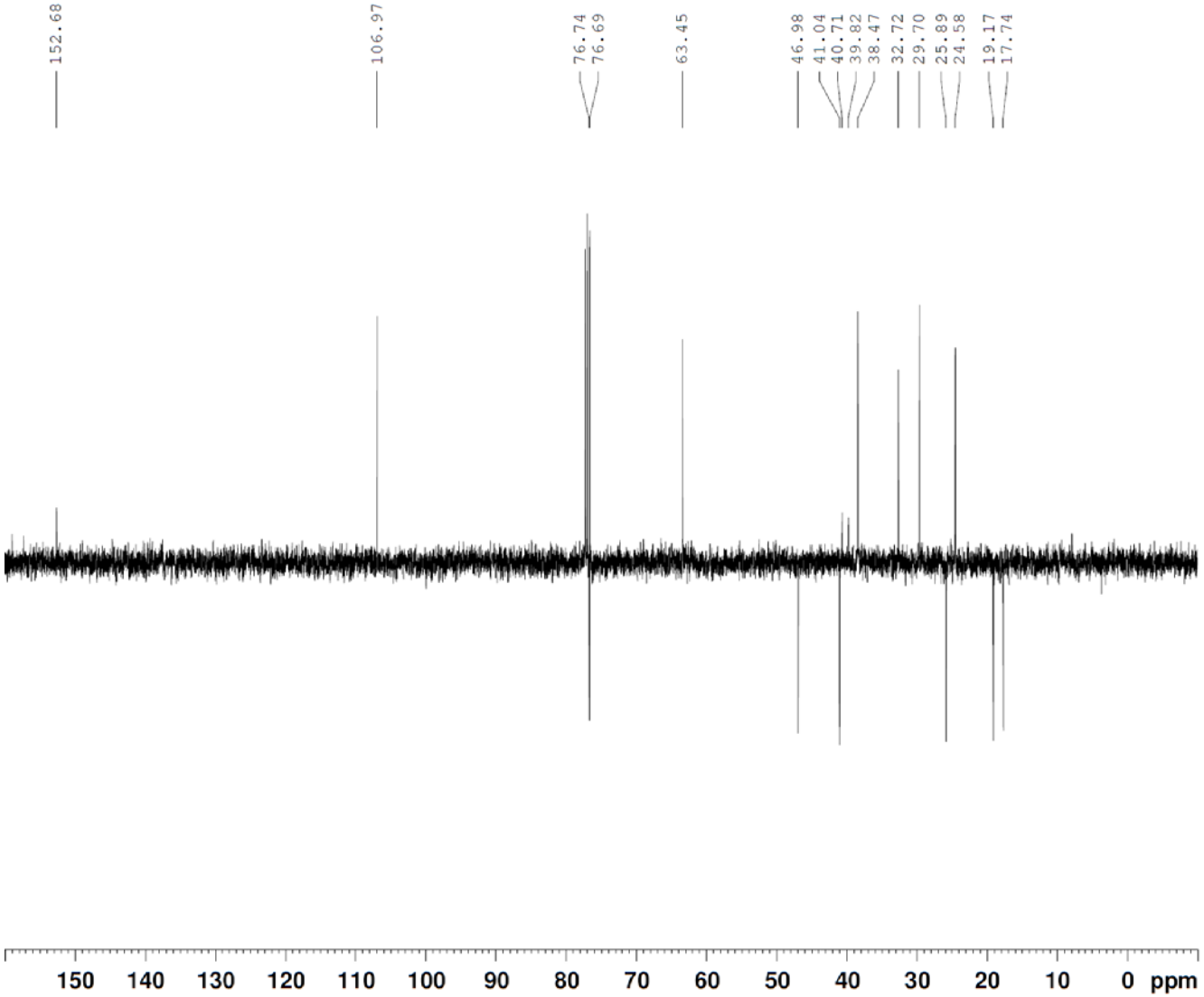
F2 - Acquisition Parameters
Date_             20090116
Time                21.10
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   181
DW                60.800 usec
DE                 6.00 usec
TE                294.8 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.30 usec
PL1               -1.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300034 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                  1.00
```




Trifarienol A (**1**)



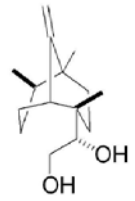

```
Current Data Parameters
NAME      090120 trifarienol a apt
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20090120
Time              13.48
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             jmod
TD                 65536
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                 16400
DW               20.800 usec
DE                6.00 usec
TE                296.0 K
CNST11         1.0000000
CNST2        145.0000600
D1             2.00000000 sec
d20            6.00689655 sec
DELTA          0.00001019 sec
TD0                    1

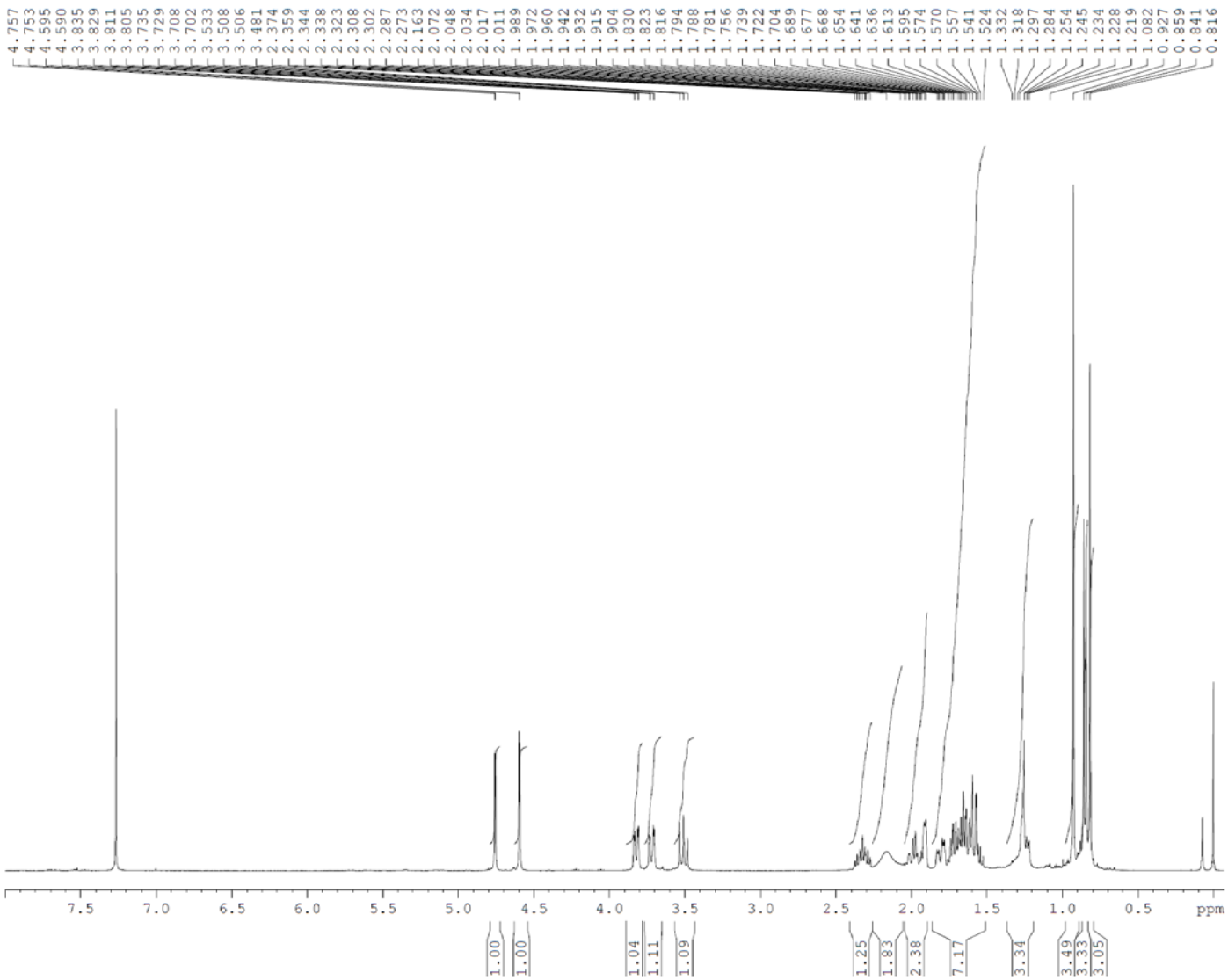
======== CHANNEL f1 ========
NUC1                  13C
P1                   8.00 usec
p2                  16.00 usec
PL1                 -2.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               80.00 usec
PL12                15.26 dB
PL2                 -1.00 dB
SFO2         400.1316005 MHz

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




Trifarienol A **(1)**

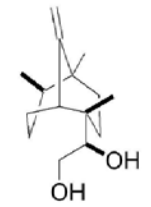



```
Current Data Parameters
NAME      090120 trifarienol b
EXPNO                1
PROCNO               1

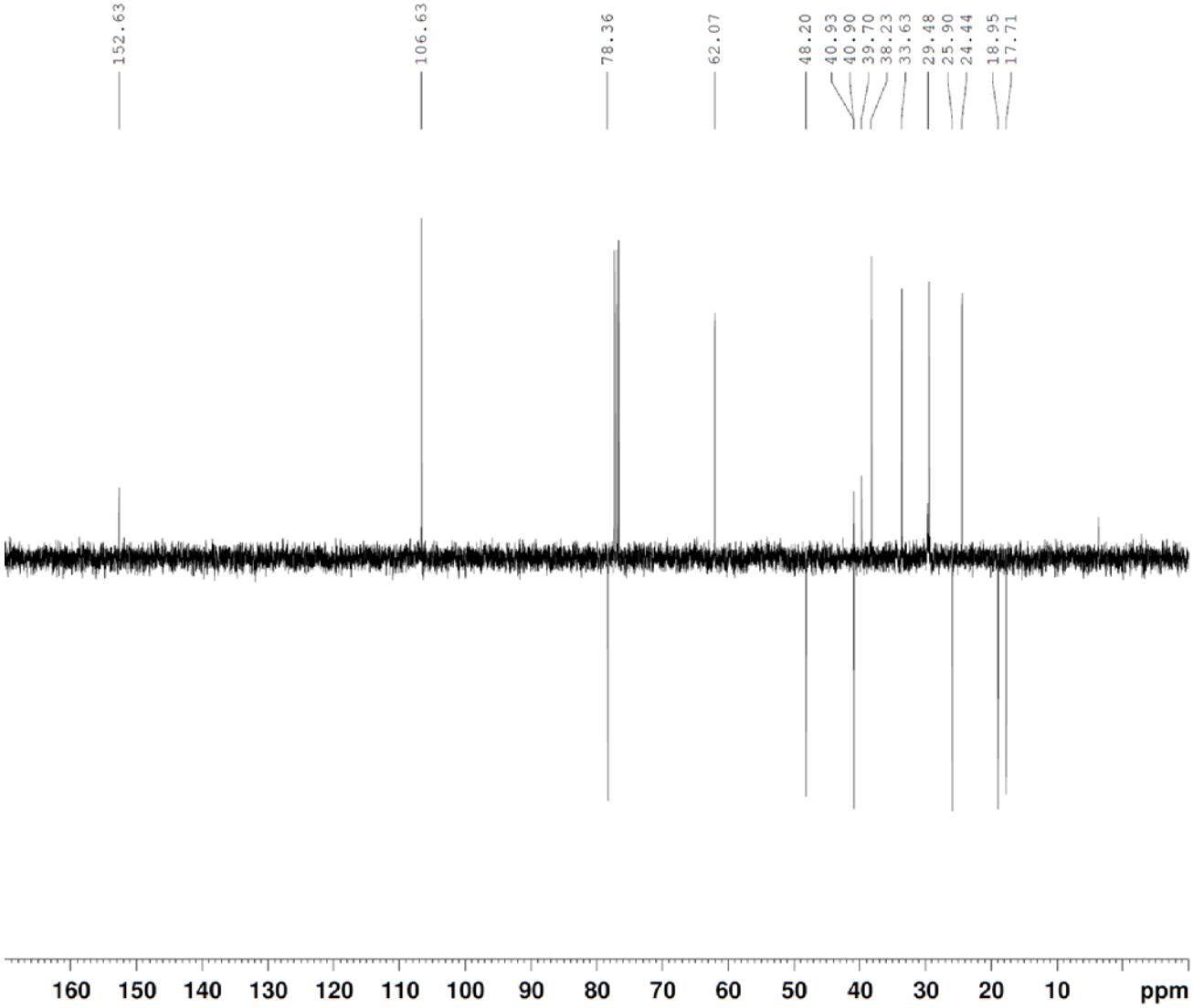
F2 - Acquisition Parameters
Date_          20090120
Time              10.29
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                  161
DW               60.800 usec
DE                6.00 usec
TE               294.7 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                12.30 usec
PL1               -1.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                32768
SF           400.1300033 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
```




Trifarienol B (**2**)



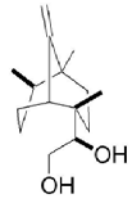

```
Current Data Parameters
NAME          090120 trifarienol b 13c
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_        20090120
Time              10.47
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           jmod
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                16400
DW               20.800 usec
DE                6.00 usec
TE               295.7 K
CNST11        1.0000000
CNST2       145.0000000
D1            2.00000000 sec
d20           0.00689655 sec
DELTA         0.00001019 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                8.00 usec
p2               16.00 usec
PL1              -2.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            80.00 usec
PL12             15.26 dB
PL2              -1.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127705 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```




Trifarienol B (**2**)