SUPPORTING INFORMATION

Rhodium-catalyzed redox allylation reactions of ketones

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Table of Contents:

A. General Information

All reactions were carried out using flame-dried glassware. Toluene was dried by passage through *activated alumina* using a GlassContour solvent purification system. Allyl acetate was purified by distillation and stored under N_2 over solid sodium sulfate. Acetophenone was distilled prior to use. All other ketones were used without distillation, but had been stored under a dry inert atmosphere for several months prior to use. $[Rh(cod)Cl]_2$, bis(pinacolato)diboron ($(Bpin)_2$), dimethylphosphinoethane (dmpe), and 1,2-bis[(2*S*,5*S*)-2,5-dimethylphospholano]benzene [(*S,S*)-MeDuphos] were stored in a N_2 atmosphere glovebox, with $(Bpin)_2$ being stored in a freezer within the glovebox. All reactions were run in sealed vials. Bis(pinacolato)diboron was donated by Frontier Chemicals and $[Rh(cod)Cl]_2$ was donated by Hareaus.

^1H NMR, and ^{13}C NMR, Bruker GN-500 (500 MHz ^1H, 125.7 MHz ^{13}C), Bruker CRYO-500 (500 MHz ^1H, 125.7 MHz ^{13}C), Bruker DRX-400 (400 MHz ^1H, 100 MHz ^{13}C), or Bruker Avance-600 (600 MHz ^1H, 150 MHz ^{13}C) NMR spectra are reported as δ values in ppm. Coupling constants are reported in Hz. The term br refers to "broad" and the term app refers to "apparent". The term dddd refers to a doublet of doublet of doublet of doublets. Infrared (IR) spectra were obtained using a Galaxy Series FTIR 5000 spectrometer. Low-resolution mass spectra (LRMS) and high resolution mass spectra (HRMS) were obtained using a Waters-MicroMass Analytical LCT (ESI) spectrometer. Melting points were obtained on a Electrothermal melting point apparatus and are uncorrected. Analytical thin-layer chromatography (TLC) was performed using Silica Gel 60 F254 precoated plates (0.25 mm thickness). Flash chromatography was performed using Silica Gel 60A (170-400 mesh) from Fisher Scientific. Silver impregnated silica gel was prepared according to literature procedure,[1,2] and was used to isolate compounds **1c**, **1d**, and **5**. PAA stands for *p*-anisaldehyde stain.

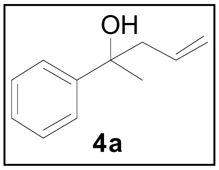
Spectral data of compound **6** can be found in the literature.[3]

B. Allylation Reactions

i. Experimental procedures for general allylation method.

In a glovebox $[Rh(cod)Cl]_2$ (5 mg, 0.01 mmol, 0.03 equiv), ligand (0.02 mmol, 0.05 equiv), and bisboron pinacol ester (306 mg, 1.20 mmol, 3.00 equiv) were added to a vial with stirbar. Outside of the glovebox the vial was briefly opened to air and allyl acetate (130 μL, 1.2 mmol, 3.0 equiv), ketone (0.4 mmol, 1 equiv), and toluene (1mL) were added to the vial after which it was sealed with a cap. The reaction was allowed to stir in a 50 °C oil bath for 18h, after which it was allowed to cool to room temperature. Unless otherwise indicated, the reaction material was loaded directly onto a column for chromatographic separation (5–15% EtOAc/Hex using Davisil® when indicated to avoid streaking), followed by a second column (15–30% Et_2O/pentane) when necessary.

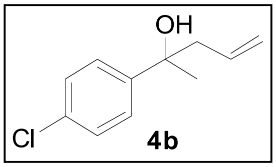
ii. Characterization data for 4a–4i[4]



4a

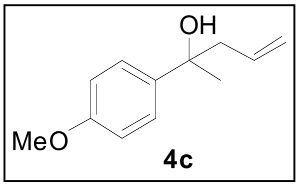
2-phenylpent-4-en-2-ol (4a): Davisil® was used for isolation. 46 mg isolated as a pale yellow oil (71%): R_f = 0.6 (25% EtOAc/Hex; stains purple with PAA); ^1H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 7.5, 2H), 7.34 (t, *J* = 7.3, 2H), 7.23 (app t, *J* = 7.2, 1H), 5.62

(dddd, *J* = 17.0, 10.2, 8.5, 6.5, 1H), 5.13 (d, *J* = 18.9, 1H), 5.12 (d, *J* = 7.0, 1H), 2.69 (dd, *J* = 13.5, 6.8, 1H) 2.50 (dd, *J* = 13.8, 8.5, 1H), 2.08 (br s, 1H), 1.55 (s, 3H).



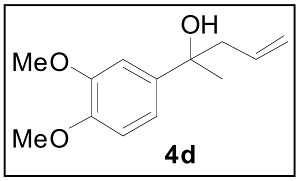
4b

2-(4-chlorophenyl)pent-4-en-2-ol (4b): Davisil® was used for isolation. 65 mg isolated as a colorless oil (83%): R_f = 0.6 (25% EtOAc/Hex; stains purple-blue with PAA); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.5, 2H), 7.29 (d, *J* = 8.6, 2H), 5.60 (dddd, *J* = 14.7, 9.4, 8.1, 6.7, 1H), 5.13 (br d, *J* = 12.7, 2H), 2.64 (dd, *J* = 13.6, 6.5, 1H), 2.48 (dd, *J* = 13.8, 8.3, 1H), 2.10 (br s, 1H), 1.52 (s, 3H).



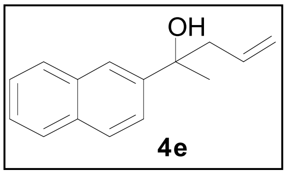
4c

2-(4-methoxyphenyl)pent-4-en-2-ol (4c): Silver impregnated silica was used for isolation (40% Et_2O/pentane then 25–40% EtOAc/Hex). 47 mg isolated as a colorless oil (61%): R_f = 0.5 (EtOAc/Hex; stains purple-blue with PAA); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (app d, *J* = 8.9, 2H), 6.88 (app d, *J* = 8.9, 1H), 5.63 (dddd, *J* = 17.0, 10.8, 8.5, 6.6, 1H), 5.13 (br d, J= 8.7, 1H), 5.12 (br d, *J* = 17.3, 1H), 3.81 (s, 3H), 2.66 (dd, *J* = 13.9, 6.6, 1H), 2.49 (dd, *J* = 13.6, 8.2, 1H), 2.01 (br s, 1H), 1.53 (s, 3H).



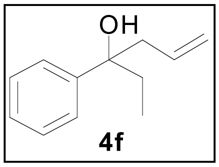
4d

2-(3,4-dimethoxyphenyl)pent-4-en-2-ol (4d): Silver impregnated silica was used for isolation (90% Et_2O/pentane then 80% EtOAc/Hex) followed by a silica plug with 20% Et_2O/pentane then 35% EtOAc/Hex). 48 mg isolated as a colorless solid (54%): R_f = 0.4 (25% EtOAc/Hex, stains blue with PAA); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 (d, *J* = 2.2, 1H), 6.93 (dd, *J* = 8.4, 2.0, 1H), 6.83 (d, *J* = 8.3, 1H), 5.64 (dddd, *J* = 16.7, 10.2, 8.2, 6.5, 1H), 5.15 (br d, *J* = 7.6, 1H), 5.14 (br d, *J* = 17.4, 1H), 3.90 (s, 3H), 3.88 (s, 3H), 2.67 (dd, *J* = 13.8, 6.6, 1H), 2.49 (dd, *J* = 13.8, 8.3, 1H), 2.01 (s, 3H).



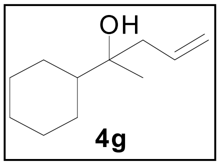
4e

2-(naphthalen-2-yl)pent-4-en-2-ol (4e): Davisil® was used for isolation. 55 mg isolated as a colorless oil (65%): R_f = 0.6 (25% EtOAc/Hex, stains purple with PAA); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (s, 1H), 7.84 (m, 3H), 7.55 (dd, *J* = 8.7, 1.9, 1H), 7.48 (m, 3H) 5.64 (dddd, *J* = 16.7, 10.1, 8.4, 6.6, 1H), 5.17 (d, *J* = 17.3, 1H), 5.13 (d, *J* = 10.3, 1H), 2.82 (dd, *J* = 13.9, 6.3, 1H), 2.60 (dd, *J* = 13.8, 8.5, 1H), 2.22 (br s, 1H), 1.65 (s, 3H).



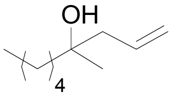
4f

3-phenylhex-5-en-3-ol (4f): Davisil® was used for isolation. 32 mg isolated as a colorless oil (46%): R_f = 0.6 (25% EtOAc/Hex), stains purple with PAA); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 7.1, 2H), 7.34 (t, *J* = 7.2, 2H), 7.23 (t, *J* = 7.0, 1H), 5.58 (dddd, *J* = 16.7, 10.0, 8.6, 6.1, 1H), 5.13 (d, *J* = 17.0, 1H), 5.10 (d, *J* = 10.0, 1H), 2.72 (dd, *J* = 13.6, 6.0, 1H), 2.50 (dd, *J* = 13.8, 8.7, 1H), 2.00 (s, 3H) 1.92–1.77 (m, 2H), 1.57 (s, 3H), 0.77 (t, *J* = 7.4, 3H).



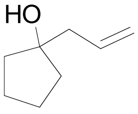
4g

2-cyclohexylpent-4-en-2-ol (4g): 55 mg isolated as a colorless oil (82%): R_f = 0.6 (25% EtOAc/Hex, stains purple with PAA); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.88 (ddt, *J* = 17.6, 10.2, 7.4, 1H), 5.14 (app dd, *J* = 10.4, 1.3, 1H), 5.11 (app ddt, *J* = 17.0, 2.1, 1.3, 1H), 2.25 (dd, *J* = 13.7, 7.5, 1H), 2.19 (ddt, *J* = 13.8, 7.5, 1.3, 1H), 1.89–1.62 (m, 6H), 1.31 (tt, *J* = 11.9, 2.9, 1H), 1.27–0.96 (m, 10H).



4h

4-methylnon-1-en-4-ol (4h): 63 mg isolated as a colorless oil (quantitative) - slightly volatile: R_f = 0.6 (25% EtOAc/Hex, stains purple with PAA); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.86 (ddt, *J* = 17.6, 10.4, 7.4, 1H), 5.13 (app dd, *J* = 8.0, 2.2, 1H), 5.10 (app ddt, *J* = 16.2, 2.2, 1.5, 1H), 2.24 (dd, *J* = 14.4, 7.5, 1H), 2.19 (dd, *J* = 14.5, 7.8, 1H), 1.47–1.40 (m, 1 H), 1.38–1.22 (m, 7H), 1.16 (s, 3H), 0.89 (t, *J* = 6.8, 3H).



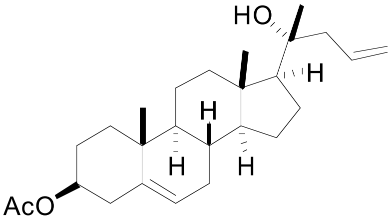
4i

1-allylcyclopentanol (4i): 21 mg isolated as a very volatile colorless liquid (42%): R_f = 0.6 (25% EtOAc/Hex, stains purple with PAA); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.90 (dddd, *J* = 15.5, 12.1, 8.5, 7.6, 1H), 5.15 (br d, *J* = 11.7, 1H), 5.14 (br d, *J* = 15.5, 1H), 2.34 (d, *J* = 7.5, 2H), 1.86–1.74 (m, 2H), 1.67–1.57 (m, 6H), 1.47 (s, 1H).

iii. Experimental procedures for pregnenolone acetate and compound 6.

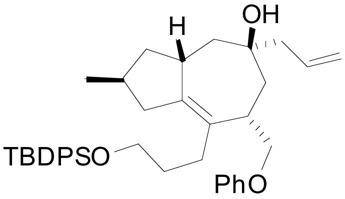
The general procedures in part **i** used with 12 equivalents of allyl acetate (4.8 mmol). A septa cap was used rather than a vial cap in order to monitor reaction progress by TLC. Upon starting material consumption as determined by TLC, the reaction was allowed to cool and was isolated by column chromatography (5–15% EtOAc/Hex).

iv. Characterization data for compounds 5 and 7.



5

Allylated pregnenolone acetate (5)[5]: Silver impregnated silica used for isolation (5–15% EtOAc/Hex). 133 mg isolated as a clear crystalline solid in 10:1 diastereomeric ratio epimeric at the tertiary alcohol (83%): R_f = 0.7 (25% EtOAc/Hex, stains blue-purple with PAA); ^{1}H NMR (400 MHz, $CDCl_3$, major reported) δ 5.80 (ddt, *J* = 17.5, 10.4, 7.5, 1H), 5.36 (d, *J* = 4.8, 1H), 5.08 (dd, *J* = 10.8, 2.0, 1H), 5.06 (dd, *J* = 16.8, 2.0, 1H), 4.63–4.54 (m, 1H), 2.33–2.27 (m, 2H), 2.16 (app dd, *J* = 7.7, 3.1, 2H), 2.15 (s, 1H), 2.07 (dt, *J* = 12.5, 3.4, 1H), 2.01 (s, 3H), 1.96 (dt, *J* = 17.7, 5.5, 1H), 1.84 (br d, *J* = 11.1, 2H), 1.77 (app tt, *J* = 11.4, 2.0, 1H), 1.73-1.51 (m, 5H), 1.51–1.40 (m, 5H), 1.27 (s, 3H), 1.25–1.07 (m, 4H), 1.02–0.98 (m, 4H), 0.97–0.89 (m, 1H), 0.85 (s, 3H).



7

Compound 7: 241 mg isolated as a colorless oil (97%): R_f = 0.6 (25% EtOAc/Hex, stains purple with PAA); ^{1}H NMR (500 MHz, $CDCl_3$, major reported) δ 7.65 (d, *J* = 7.6, 4H), 7.41 (t, *J* = 7.5, 2H), 7.36 (t, *J* = 7.1, 5H) 7.24 (app d, *J* = 7.8, 1H), 6.92 (t, *J* = 7.5, 1H), 6.88 (d, *J* = 8.1, 2H), 5.84 (ddt, *J* = 17.5, 10.2, 7.5, 1H), 5.14 (dd, *J* = 10.2, 1.7, 1H), 5.08 (dd, *J* = 17.2, 1.2, 1H), 4.05 (t, *J* = 8.6, 1H), 3.83 (dd, *J* = 8.8, 7.3, 1H), 3.61 (t, *J* = 6.4, 2H), 3.27 (br q, *J* = 10.0, 1H), 3.04 (br t, *J* = 10.0, 1H), 2.45 (dd, *J* = 15.9, 6.7, 1H), 2.13 (app d, *J* = 16.7, 1H), 2.10 (app d, *J* = 17.6, 1H), 2.16–2.02 (m, 4H), 1.85 (dd, *J* = 16.0, 8.8, 1H), 1.78–1.69 (m, 1H), 1.63 (d, *J* = 13.5, 1H), 1.48–1.43 (m, 3H), 1.34 (app t, *J* = 13.1, 2H), 1.26 (app d, *J* = 11.1, 1H), 1.26 (s, 3H), 1.03 (br s, 9H), 0.95 (d, *J* = 6.6, 3H); ^{13}C NMR (125 MHz, $CDCl_3$, major reported) δ 159.1, 145.7, 135.8 (4C), 133.7, 133.2, 129.8 (2C), 129.7 (2C), 127.8 (4C), 120.8, 119.3, 114.9 (2C), 73.8, 70.4, 64.2, 50.5, 44.8, 43.4, 41.8, 39.3, 36.4, 35.9, 33.2, 33.0, 27.0 (3C), 25.2, 20.2, 19.4; IR (thin film, cm^{-1}) 2931, 2860, 1242, 1111, 702; LRMS (ESI, m+Na) 631.2, HRMS (ESI, m+Na) calc. for 631.3583, found 631.3595.

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[1] Ruprah, P.; Cros, J.-P.; Pease, J. E.; Whittingham, W. G.; Williams, J. M. J. *Eur. J. Org. Chem.* **2002**, 3145–3152.

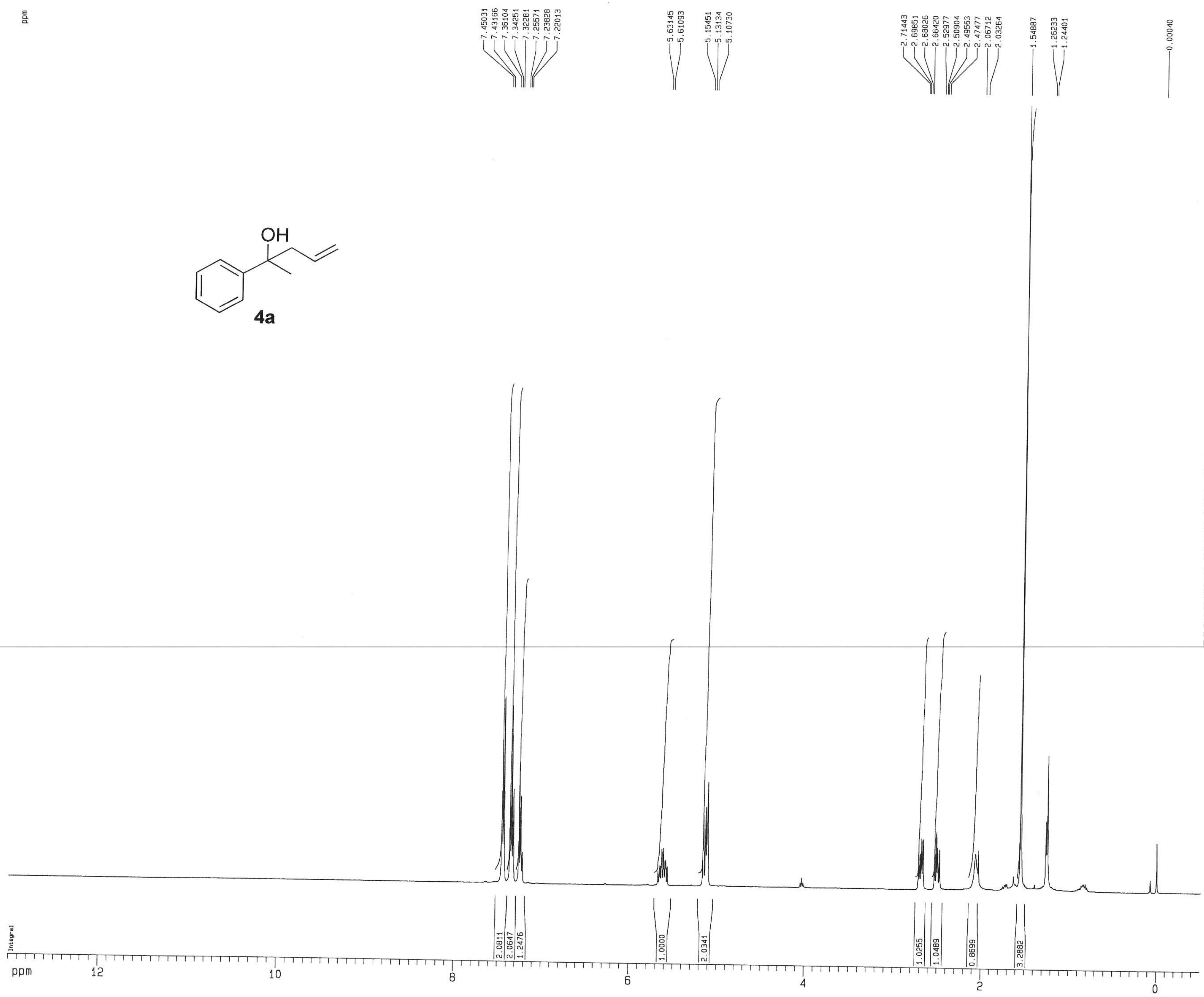
[2] Silica preparation was scaled up. For 200 g silica 30 g $AgNO_3$ was used and the mixture was stirred for 2 hours. The silica was dried in an oven overnight, protected from light at all times including during column chromatography.

[3] Canham, S. M.; France, D. J.; Overman, L. E. *J. Am. Chem. Soc.* **2010**, *132*, 7876–7877.

[4] All compounds **4a-4i** match spectral data from the literature. See Barker, T.; Jarvo, E. R. *Org. Lett*. **2009**, *11*, 1047–1049.

[5] Reetz, M. T.; Steinbach, R.; Westermann, J.; Peter, R.; Wenderoth, B. *Eur. J. Org. Chem.* **1985**, *118*, 1441–1454.

1H spectrum



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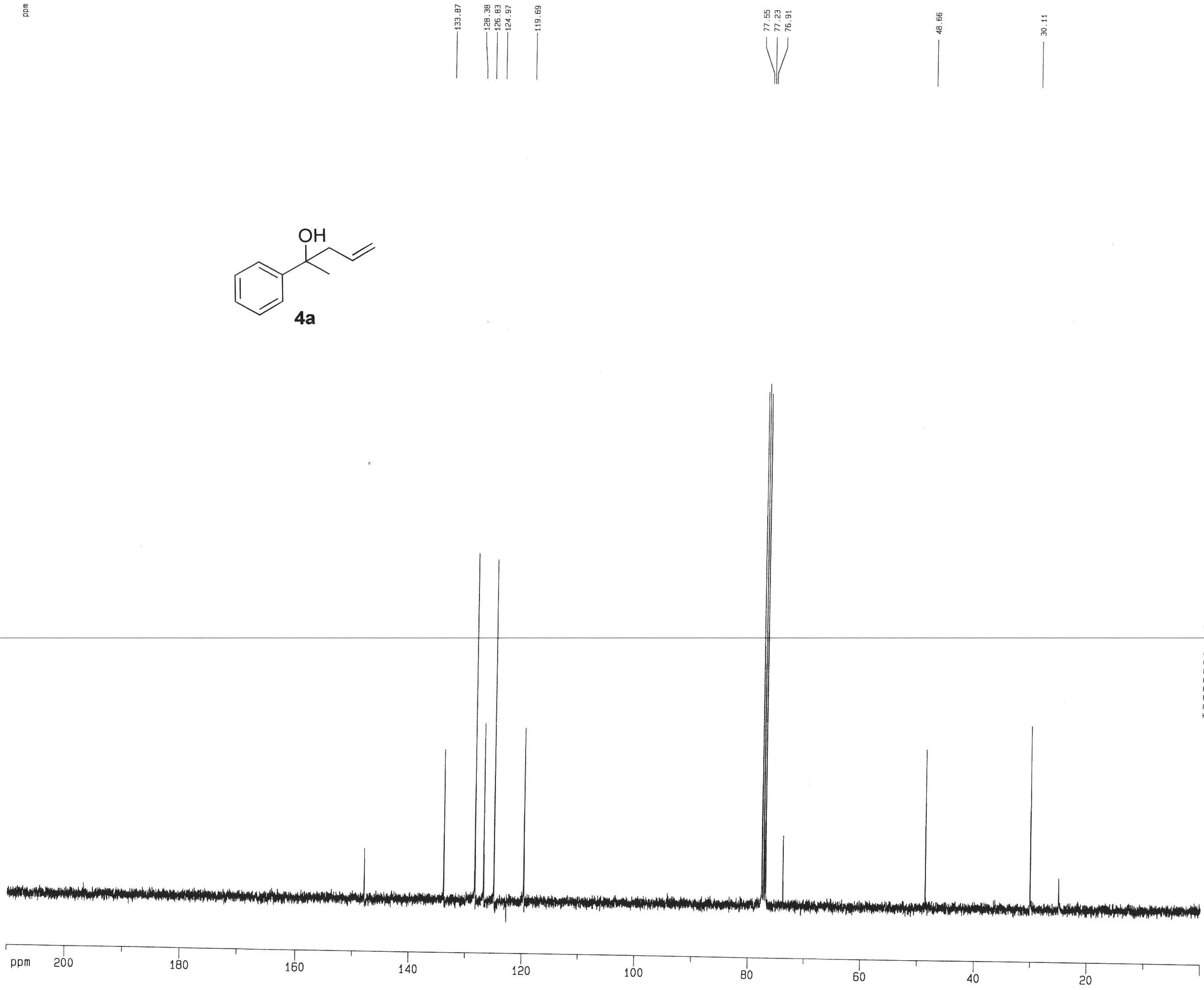
13C spectrum with 1H decoupling



4a

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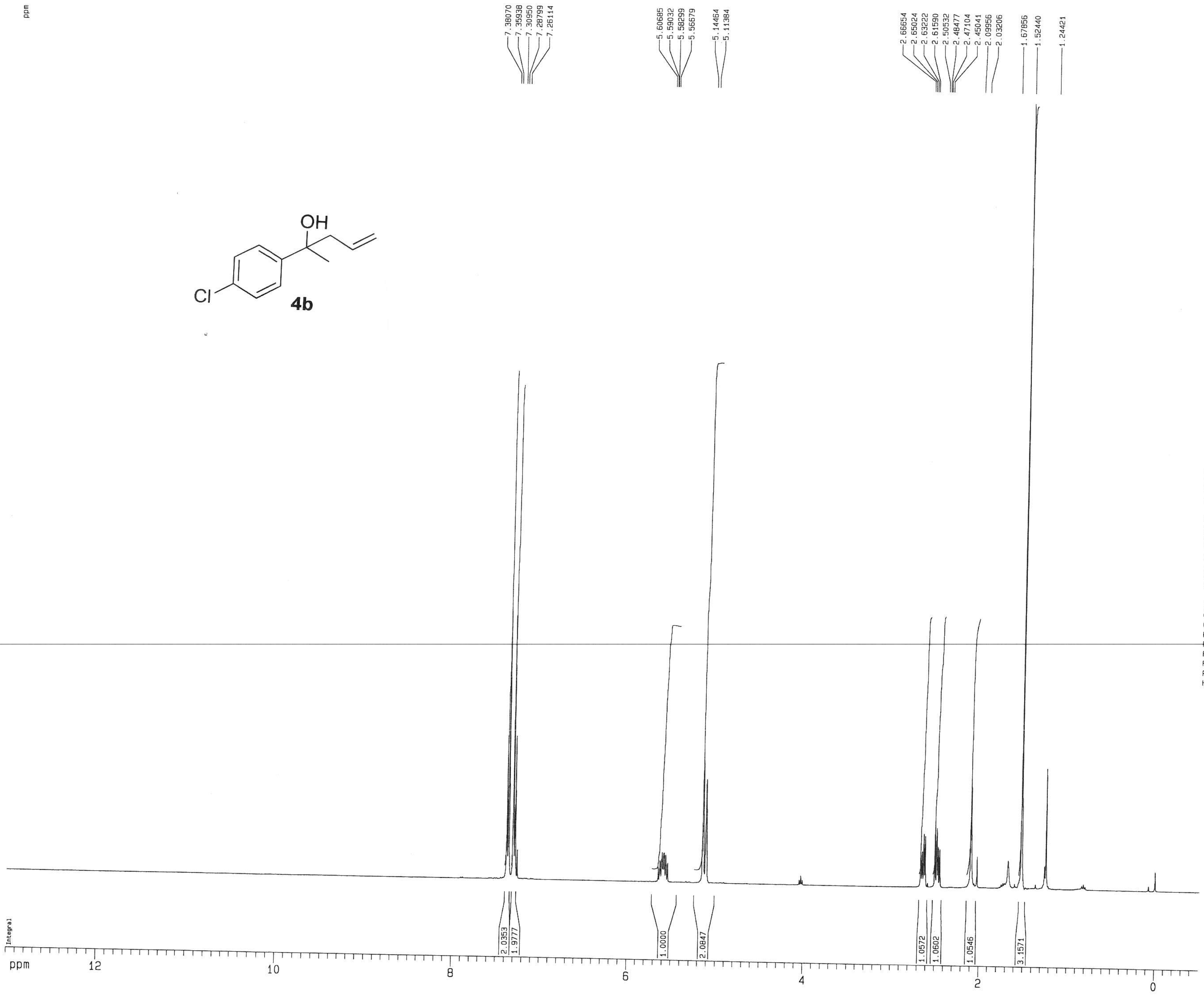
1H spectrum



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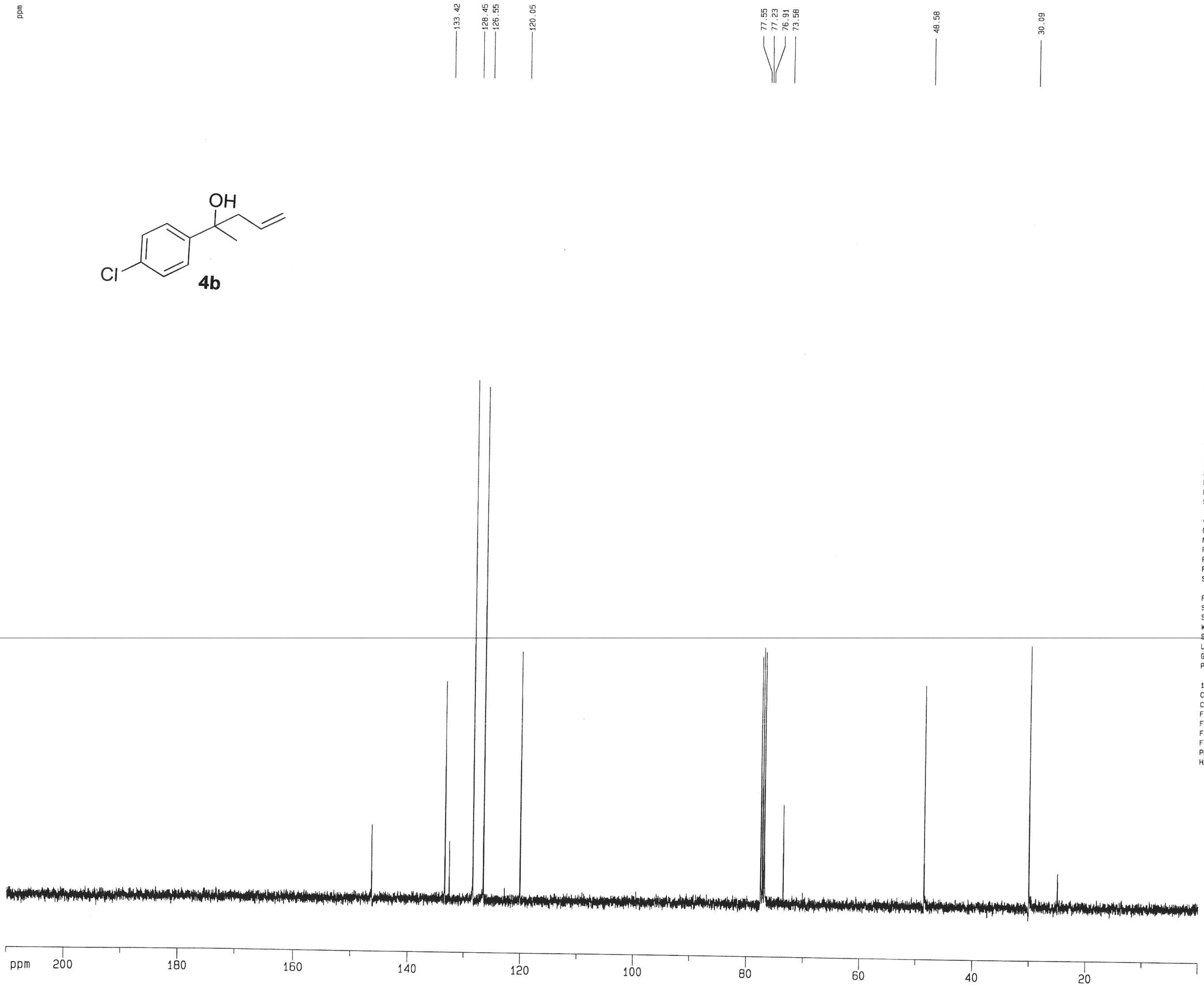
13C spectrum with 1H decoupling



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1D NMR plot parameters
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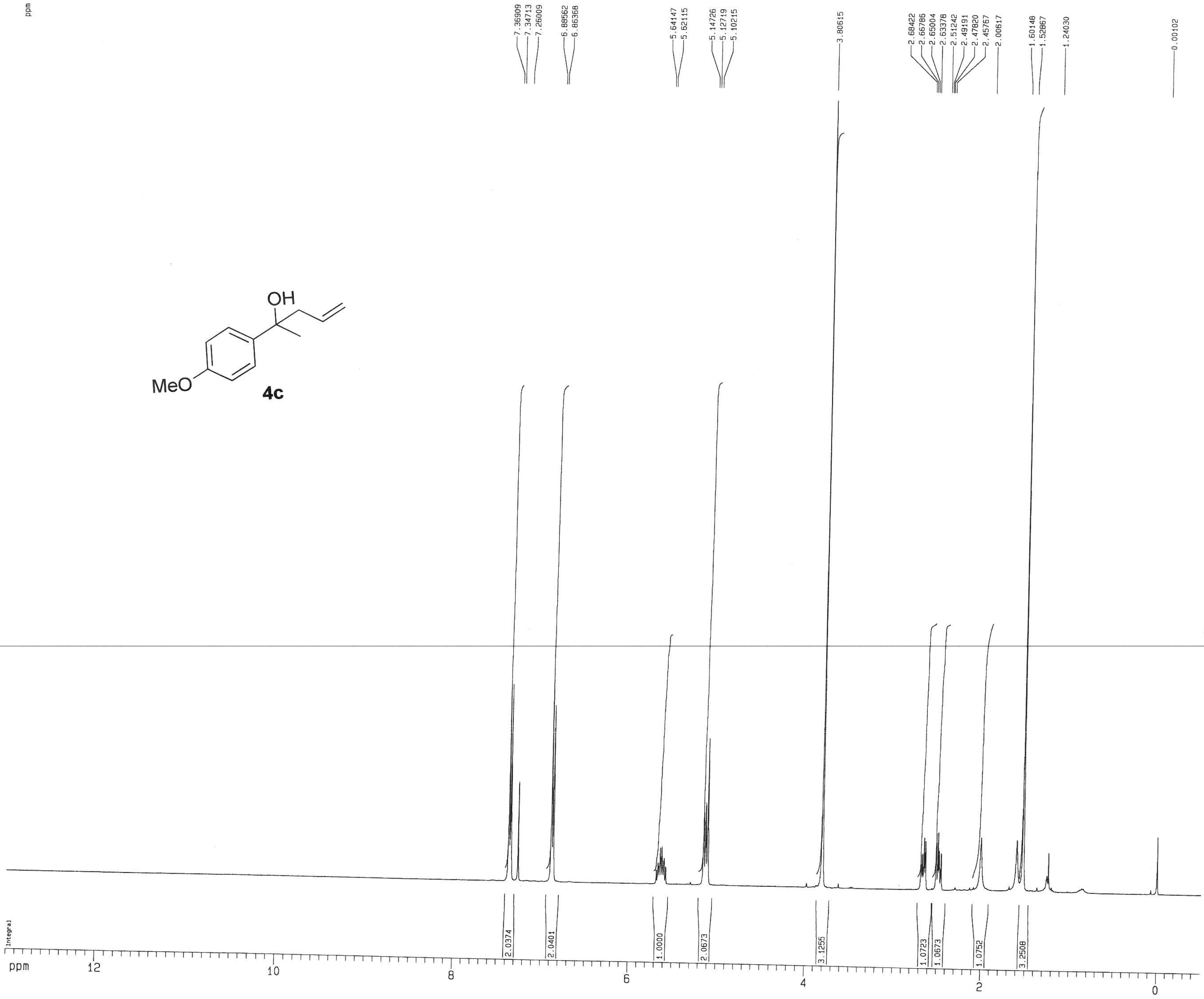
CY                  10.00 cm
F1P               210.000 ppm
F1               21128.68 Hz
F2P                 0.000 ppm
F2                   0.00 Hz
PPMCM             9.21053 ppm/cm
HZCM            926.69641 Hz/cm
```

1H spectrum



```
Current Data Parameters
USER                  willia
NAME     fjw-iv-112-MeO-Agcol-dry
EXPNO                      1
PROCNO                     1

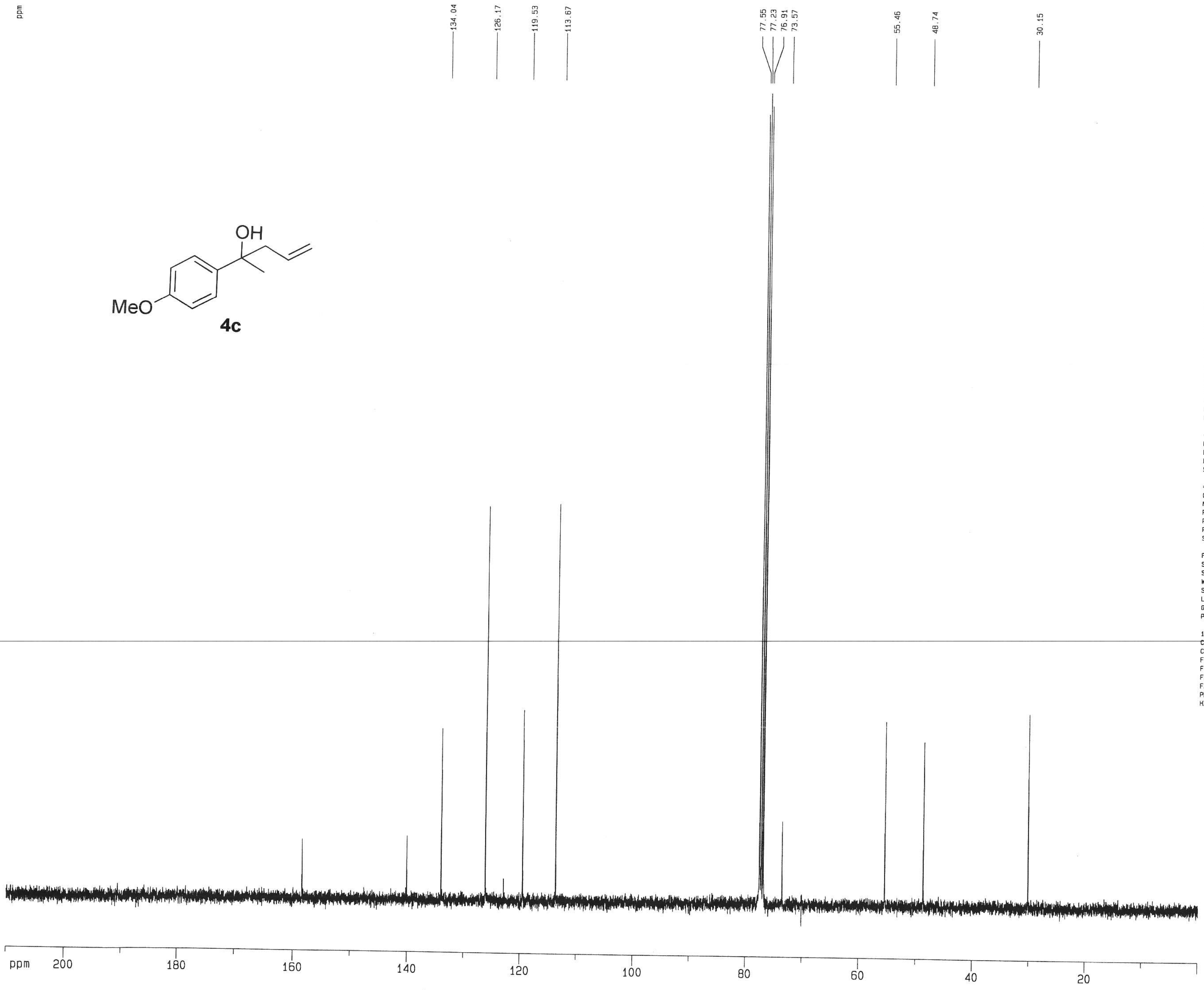
F2 - Acquisition Parameters
Date_              20110725
Time                  22.46
INSTRUM              drx400
PROBHD   5 mm QNP H/F/P
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        9
DS                        2
SWH              6410.256 Hz
FIDRES           0.097813 Hz
AQ              5.1118579 sec
RG                    287.4
DW                 78.000 usec
DE                   4.50 usec
TE                  298.0 K
D1             0.10000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                  12.00 usec
PL1                 -0.60 dB
SFO1          400.1328009 MHz

F2 - Processing parameters
SI                    65536
SF            400.1300214 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     2.00

1D NMR plot parameters
CX                    22.80 cm
CY                    15.00 cm
F1P                  13.000 ppm
F1                  5201.69 Hz
F2P                  -0.500 ppm
F2                  -200.07 Hz
PPMCM               0.59211 ppm/cm
HZCM              236.91911 Hz/cm
```

13C spectrum with 1H decoupling



4c

```
Current Data Parameters
USER                willia
NAME      fjw-iv-112-MeO-Agcol-dry
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20110725
Time                 22.48
INSTRUM             drx400
PROBHD    5 mm QNP H/F/P
PULPROG            zgdc30
TD                   65536
SOLVENT             CDCl3
NS                     628
DS                       4
SWH           24154.590 Hz
FIDRES         0.368570 Hz
AQ           1.3566452 sec
RG                  7298.2
DW              20.700 usec
DE               20.39 usec
TE                 298.0 K
D1          0.10000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

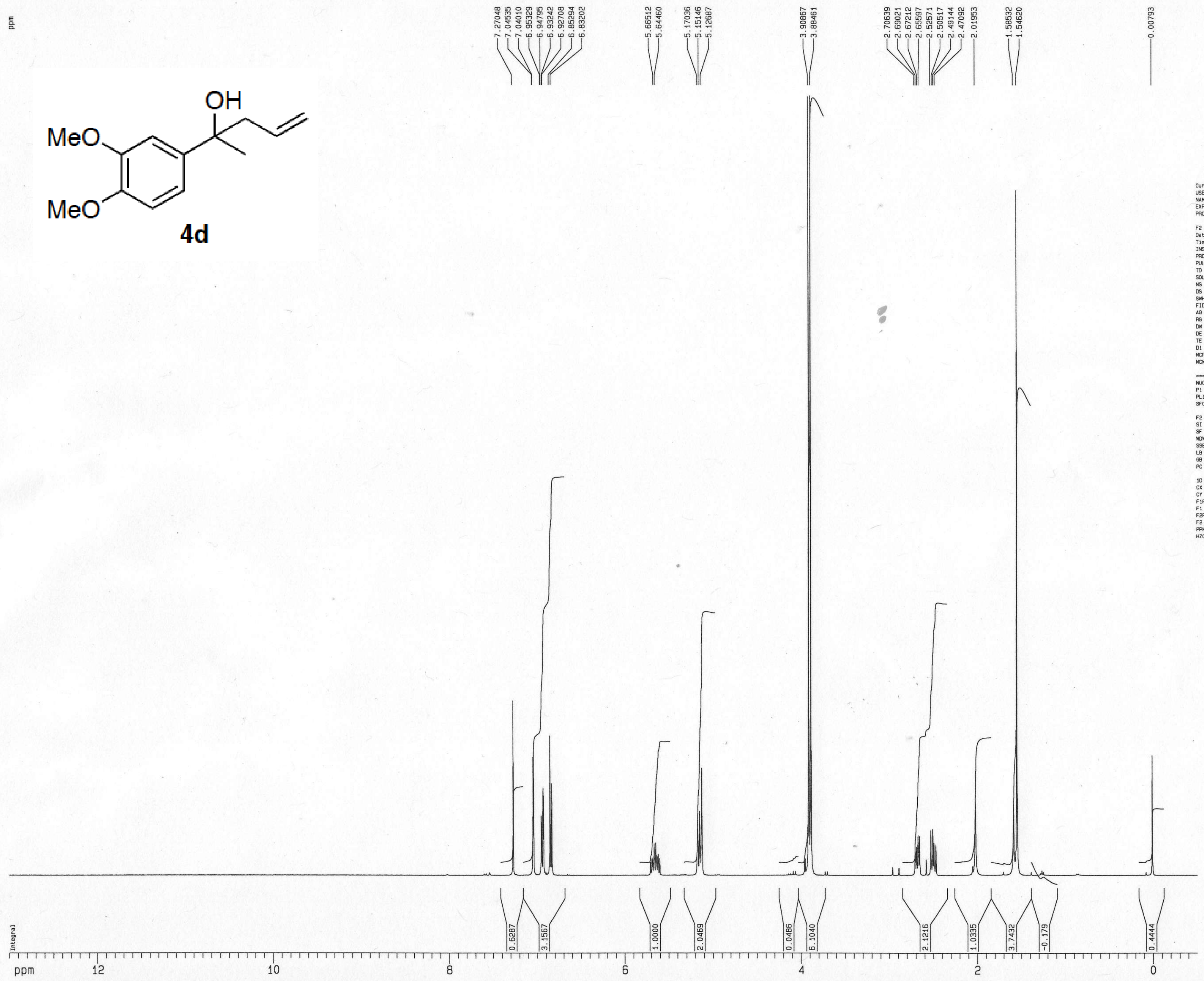
======== CHANNEL f1 ========
NUC1                   13C
P1               11.00 usec
PL1                0.00 dB
SFO1       100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2             mlev16
NUC2                    1H
PCPD2            80.00 usec
PL2                0.00 dB
PL12              16.20 dB
SFO2       400.1328009 MHz

F2 - Processing parameters
SI                   65536
SF         100.6127517 MHz
WDW                     EM
SSB                      0
LB                 1.00 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                22.80 cm
CY                15.50 cm
F1P            210.000 ppm
F1            21128.68 Hz
F2P              0.000 ppm
F2                0.00 Hz
PPMCM      9.21053 ppm/cm
HZCM      926.69641 Hz/cm
```

1H spectrum



```
Current Data Parameters
USER               willia
NAME     fjw-iv-152-diMeO-Rh-allyaltion-col2
EXPNO                   2
PROCNO                  1

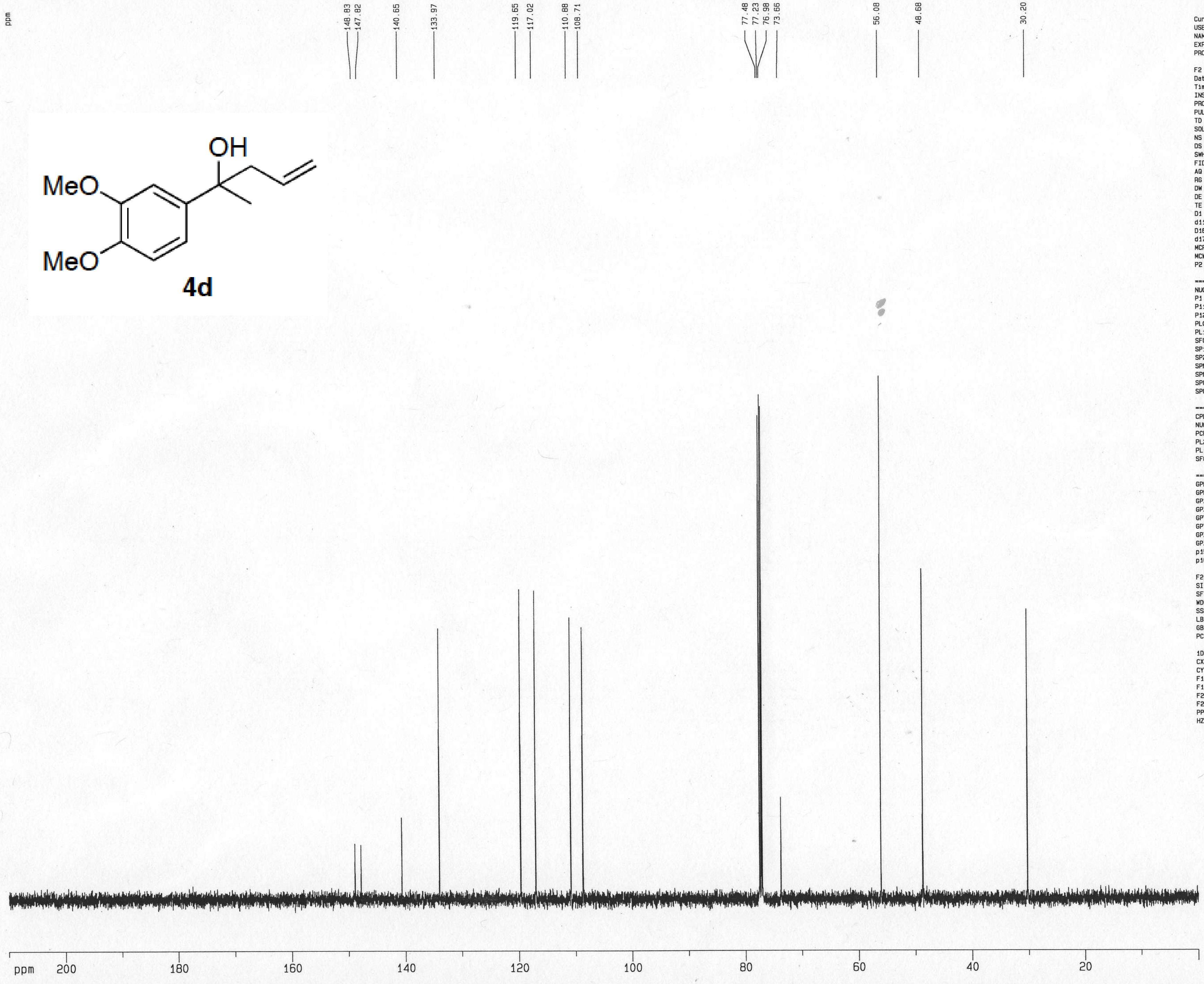
F2 - Acquisition Parameters
Date_            20110825
Time                15.12
INSTRUM            drx400
PROBHD   5 mm QNP H/F/P
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     10
DS                      2
SWH             6410.256 Hz
FIDRES          0.097813 Hz
AQ             5.1118579 sec
RG                  322.5
DW                 78.000 usec
DE                   4.50 usec
TE                  298.0 K
D1             0.10000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  12.00 usec
PL1                 -0.60 dB
SFO1          400.1328009 MHz

F2 - Processing parameters
SI                  65536
SF            400.1300175 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   2.00

1D NMR plot parameters
CX                  22.80 cm
CY                  15.00 cm
F1P                13.000 ppm
F1                5201.69 Hz
F2P                -0.500 ppm
F2                -200.07 Hz
PPMCM             0.59211 ppm/cm
HZCM            236.91908 Hz/cm
```

Z-restored spin-echo 13C spectrum with 1H decoupling



4d

```
Current Data Parameters
USER              willia
NAME      fjw-iv-155-MeO2-allyl-C13
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110831
Time               14.46
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   172
DS                    16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG                7298.2
DW                16.500 usec
DE                  6.00 usec
TE                 298.0 K
D1            0.25000000 sec
d11           0.03000000 sec
D16           0.00020000 sec
d17           0.00019600 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
P2                 31.00 usec

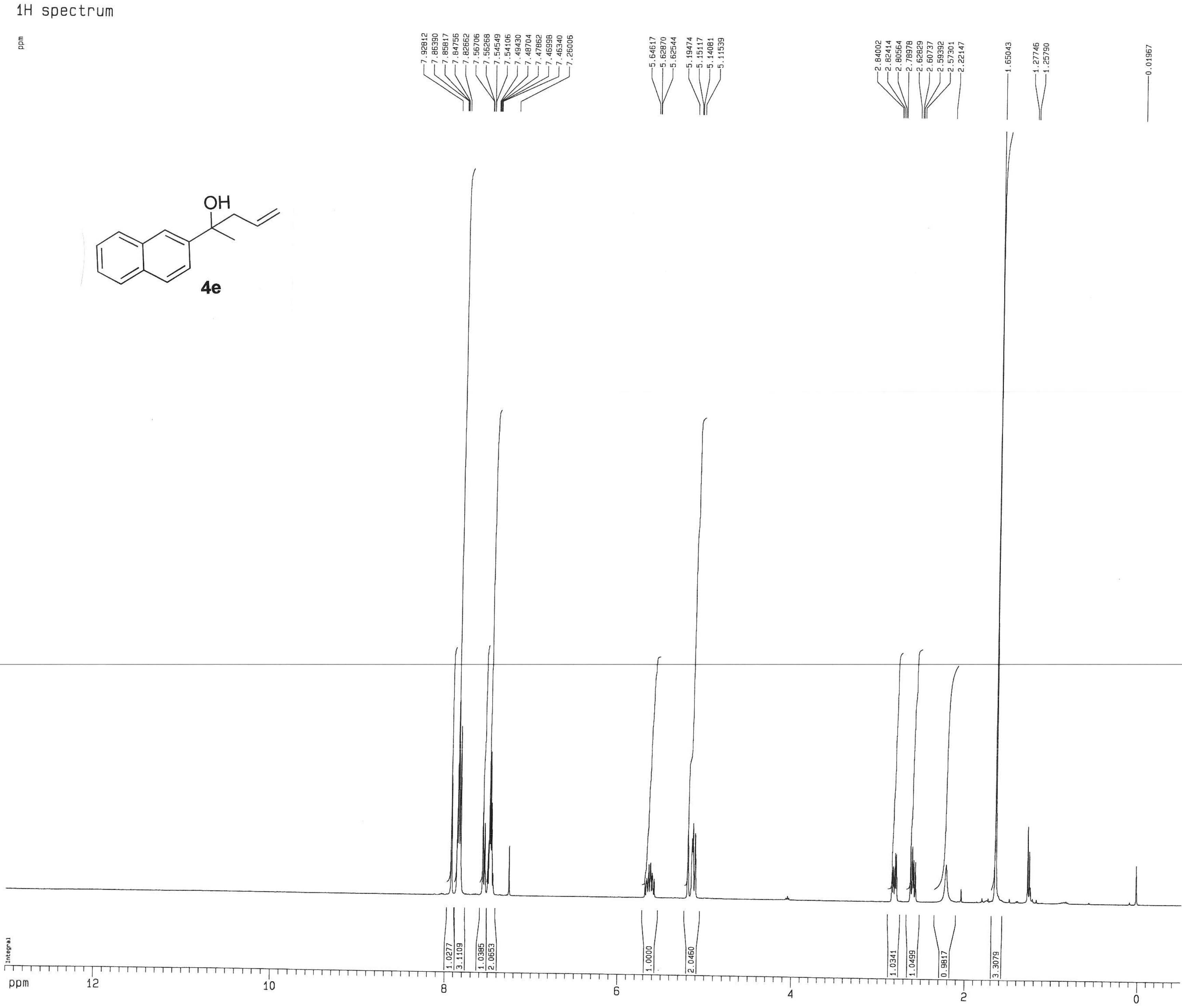
======== CHANNEL f1 ========
NUC1                 13C
P1                 15.50 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                 3.20 dB
SP2                 3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2       Crp60comp.4
SPOFF1              0.00 Hz
SPOFF2              0.00 Hz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 1.60 dB
PL12               24.60 dB
SFO2         500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPX1                0.00 %
GPX2                0.00 %
GPY1                0.00 %
GPY2                0.00 %
GPZ1               30.00 %
GPZ2               50.00 %
p15               500.00 usec
p16              1000.00 usec

F2 - Processing parameters
SI                 65536
SF           125.7804006 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 10.00 cm
F1P              210.000 ppm
F1              26413.88 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            9.21053 ppm/cm
HZCM          1158.50366 Hz/cm
```



```
Current Data Parameters
USER                willia
NAME     fjw-iv-07-25-2011-Np
EXPNO                    1
PROCNO                   1

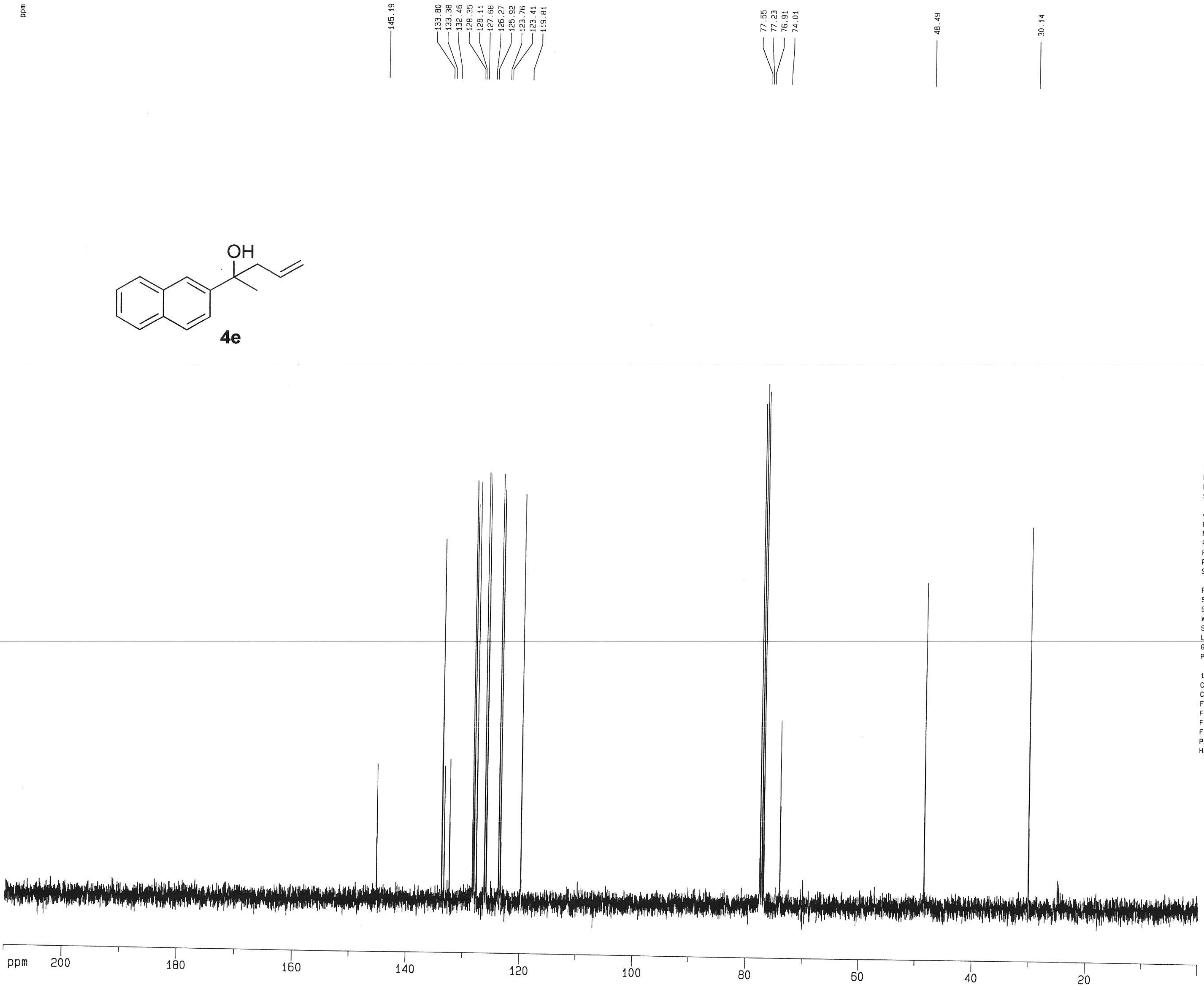
F2 - Acquisition Parameters
Date_             20110725
Time                 23.07
INSTRUM             drx400
PROBHD   5 mm QNP H/F/P
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       7
DS                       2
SWH             6410.256 Hz
FIDRES          0.097813 Hz
AQ             5.1118579 sec
RG                   143.7
DW                78.000 usec
DE                  4.50 usec
TE                 298.0 K
D1            0.10000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                 12.00 usec
PL1                -0.60 dB
SFO1         400.1328009 MHz

F2 - Processing parameters
SI                   65536
SF           400.1300214 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    2.00

1D NMR plot parameters
CX                   22.80 cm
CY                   15.00 cm
F1P                 13.000 ppm
F1                 5201.69 Hz
F2P                 -0.500 ppm
F2                 -200.07 Hz
PPMCM              0.59211 ppm/cm
HZCM             236.91911 Hz/cm
```

13C spectrum with 1H decoupling



4e

```
Current Data Parameters
USER                 willia
NAME     fjw-iv-07-25-2011-Np
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20110725
Time                  23.08
INSTRUM              drx400
PROBHD   5 mm QNP H/F/P
PULPROG             zgdc30
TD                    65536
SOLVENT               CDCl3
NS                      199
DS                        4
SWH            24154.590 Hz
FIDRES          0.368570 Hz
AQ            1.3566452 sec
RG                  14596.5
DW             20.700 usec
DE              20.39 usec
TE                298.0 K
D1           0.10000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

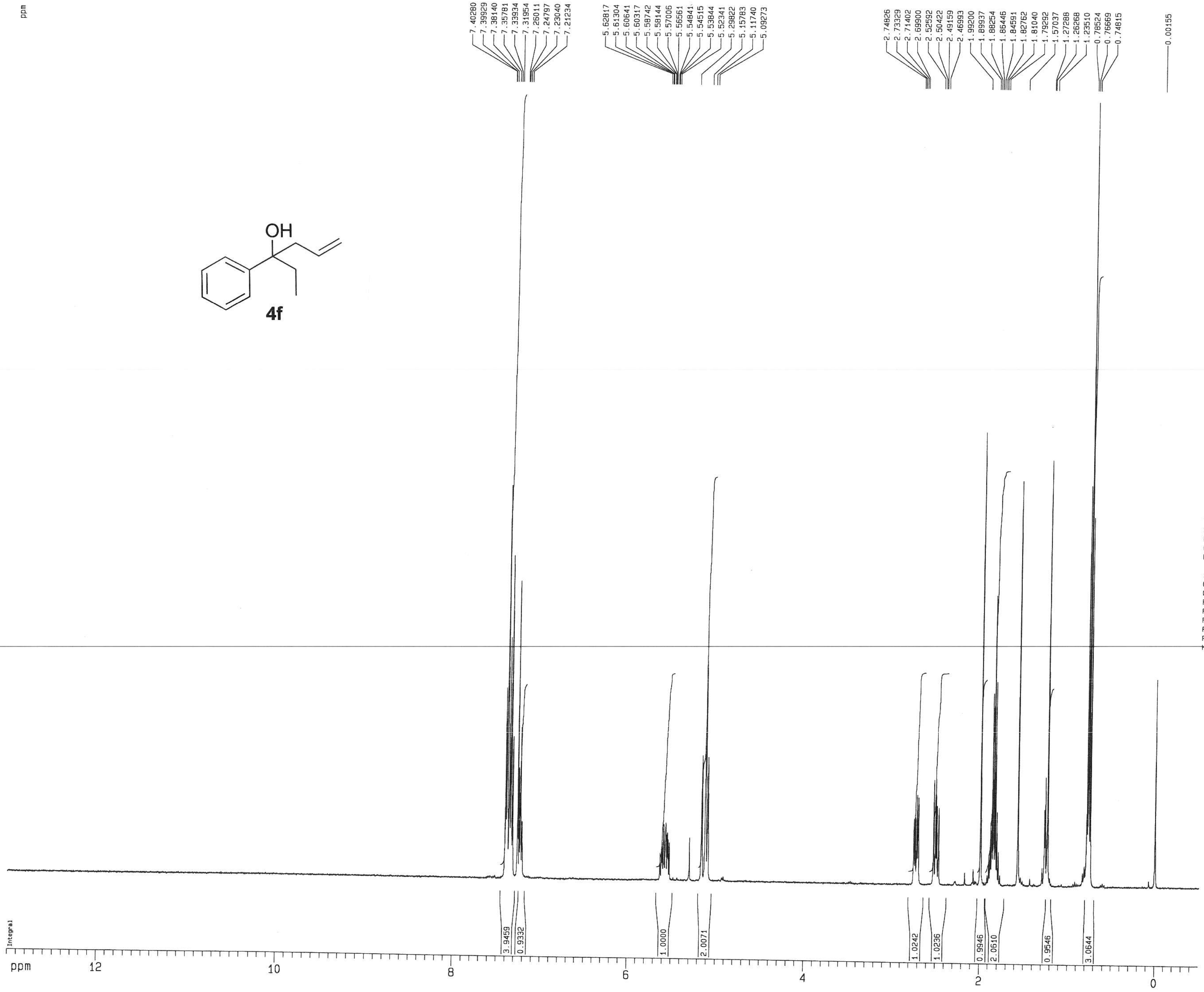
======== CHANNEL f1 ========
NUC1                    13C
P1              11.00 usec
PL1                0.00 dB
SFO1       100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2              mlev16
NUC2                     1H
PCPD2           80.00 usec
PL2                0.00 dB
PL12              16.20 dB
SFO2       400.1328009 MHz

F2 - Processing parameters
SI                    65536
SF         100.6127543 MHz
WDW                      EM
SSB                       0
LB                  1.00 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                 22.80 cm
CY                 10.00 cm
F1P             210.000 ppm
F1             21128.68 Hz
F2P               0.000 ppm
F2                  0.00 Hz
PPMCM        9.21053 ppm/cm
HZCM       926.69641 Hz/cm
```

1H spectrum



```
Current Data Parameters
USER              willia
NAME    fjw-iv-141-propio-col2
EXPNO                  1
PROCNO                 1

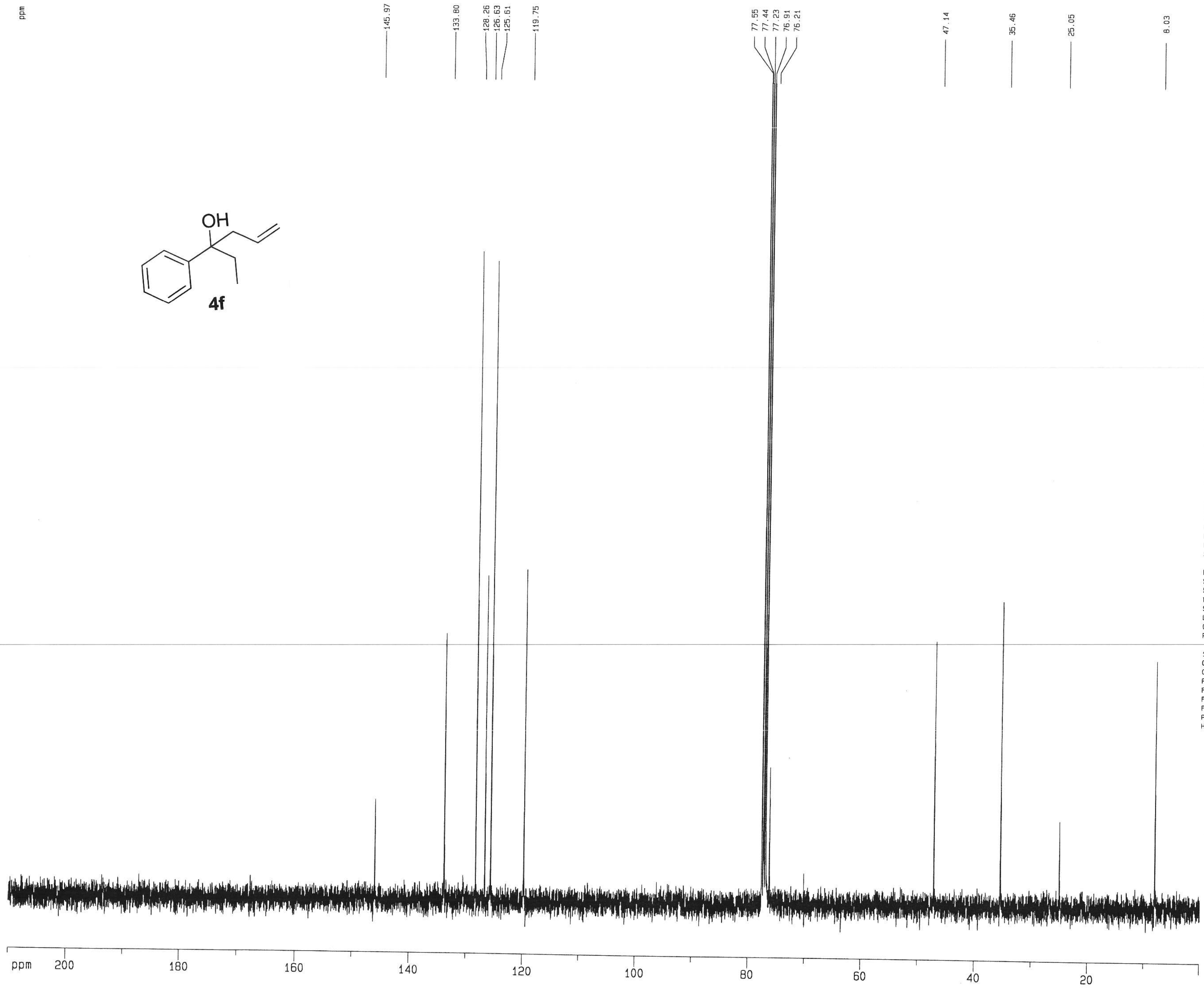
F2 - Acquisition Parameters
Date_           20110726
Time                0.41
INSTRUM           drx400
PROBHD   5 mm QNP H/F/P
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     9
DS                     2
SWH             6410.256 Hz
FIDRES          0.097813 Hz
AQ             5.1118579 sec
RG                 322.5
DW                78.000 usec
DE                  4.50 usec
TE                 298.0 K
D1            0.10000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                -0.60 dB
SFO1         400.1328009 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300214 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.00 cm
F1P               13.000 ppm
F1               5201.69 Hz
F2P               -0.500 ppm
F2               -200.07 Hz
PPMCM            0.59211 ppm/cm
HZCM           236.91911 Hz/cm
```

13C spectrum with 1H decoupling



```
Current Data Parameters
USER                willia
NAME      fjw-iv-141-propio-col2
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20110726
Time                  0.43
INSTRUM             drx400
PROBHD   5 mm QNP H/F/P
PULPROG            zgdc30
TD                   65536
SOLVENT              CDCl3
NS                     789
DS                       4
SWH          24154.590 Hz
FIDRES        0.368570 Hz
AQ           1.3566452 sec
RG                  9195.2
DW              20.700 usec
DE               20.39 usec
TE               298.0 K
D1          0.10000000 sec
d11         0.03000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1              11.00 usec
PL1               0.00 dB
SFO1     100.6237964 MHz

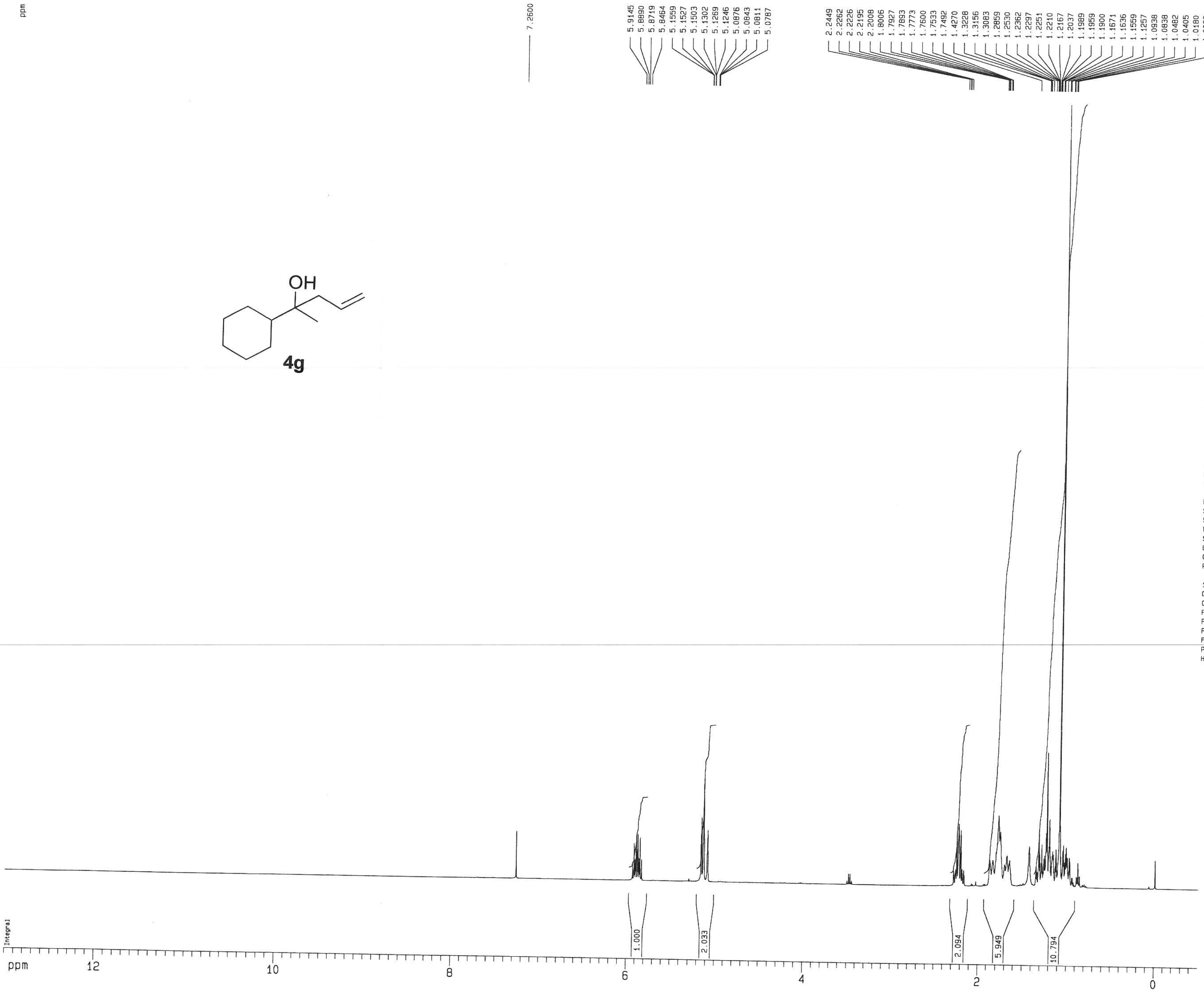
======== CHANNEL f2 ========
CPDPRG2            mlev16
NUC2                    1H
PCPD2           80.00 usec
PL2               0.00 dB
PL12             16.20 dB
SFO2     400.1328009 MHz

F2 - Processing parameters
SI                   65536
SF       100.6127510 MHz
WDW                     EM
SSB                      0
LB                 1.00 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                22.80 cm
CY                35.00 cm
F1P            210.000 ppm
F1            21128.68 Hz
F2P              0.000 ppm
F2                 0.00 Hz
PPMCM        9.21053 ppm/cm
HZCM       926.69629 Hz/cm
```

1H spectrum

4g



```
Current Data Parameters
USER              willia
NAME      fjw-iv-127-Cy-Me-col2
EXPNO                  1
PROCNO                 1

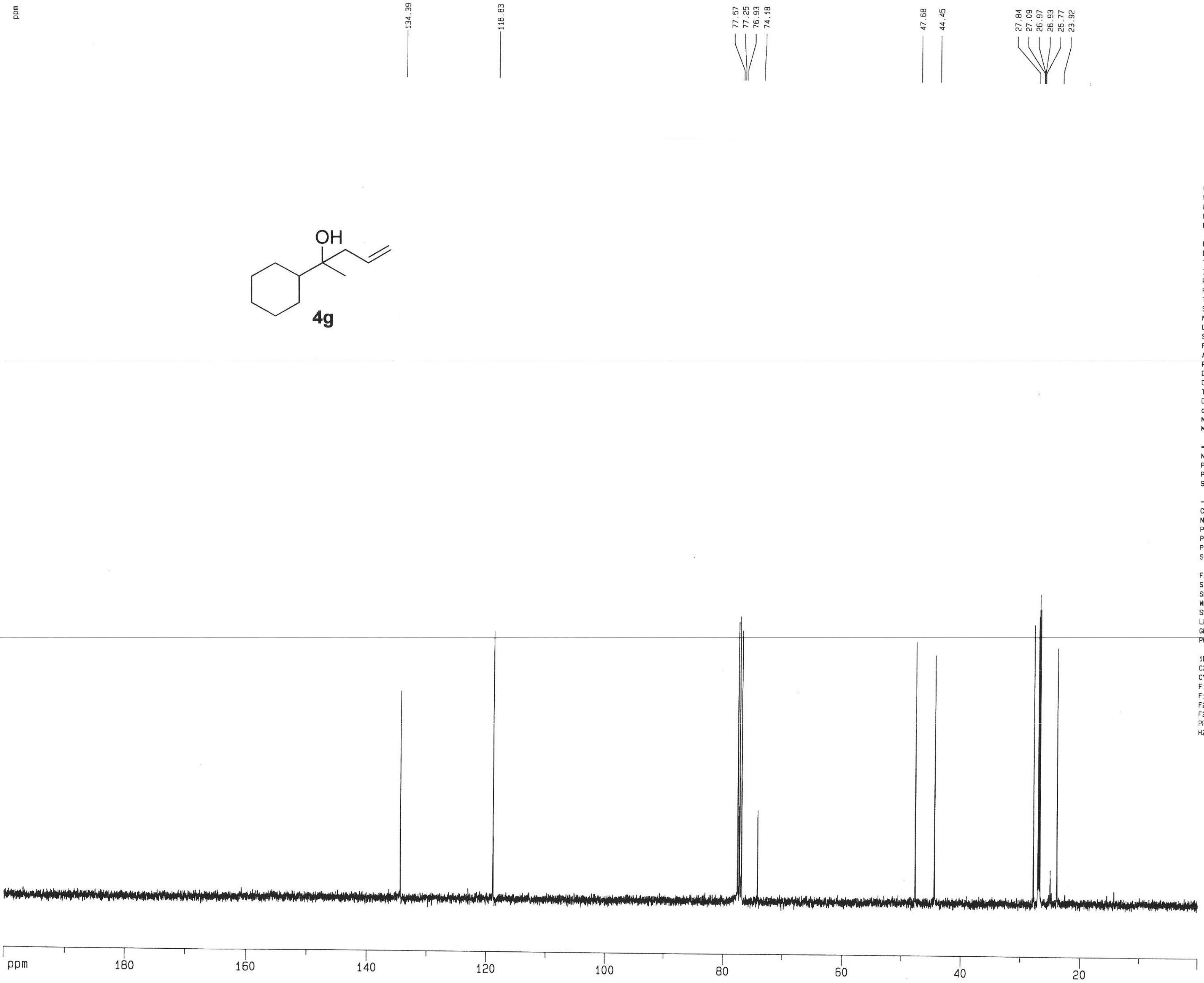
F2 - Acquisition Parameters
Date_           20110708
Time               14.55
INSTRUM           drx400
PROBHD   5 mm QNP H/F/P
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     9
DS                     2
SWH          6410.256 Hz
FIDRES       0.097813 Hz
AQ          5.1118579 sec
RG                  90.5
DW            78.000 usec
DE              4.50 usec
TE               298.1 K
D1        0.10000000 sec
MCREST    0.00000000 sec
MCWRK     0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1             12.00 usec
PL1              -0.60 dB
SFO1      400.1328009 MHz

F2 - Processing parameters
SI                 65536
SF        400.1300217 MHz
WDW                   EM
SSB                    0
LB               0.30 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX              22.80 cm
CY              15.00 cm
F1P           13.000 ppm
F1            5201.69 Hz
F2P           -0.500 ppm
F2            -200.07 Hz
PPMCM      0.59211 ppm/cm
HZCM      236.91911 Hz/cm
```

13C spectrum with 1H decoupling



```
Current Data Parameters
USER                  willia
NAME      fjw-iv-127-Cy-Me-col2
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_               20110708
Time                   15.00
INSTRUM               drx400
PROBHD   5 mm QNP H/F/P
PULPROG               zgdc30
TD                     65536
SOLVENT                CDCl3
NS                       324
DS                         4
SWH               24154.590 Hz
FIDRES             0.368570 Hz
AQ                1.3566452 sec
RG                    9195.2
DW                    20.700 usec
DE                     20.39 usec
TE                     298.1 K
D1                0.10000000 sec
d11               0.03000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

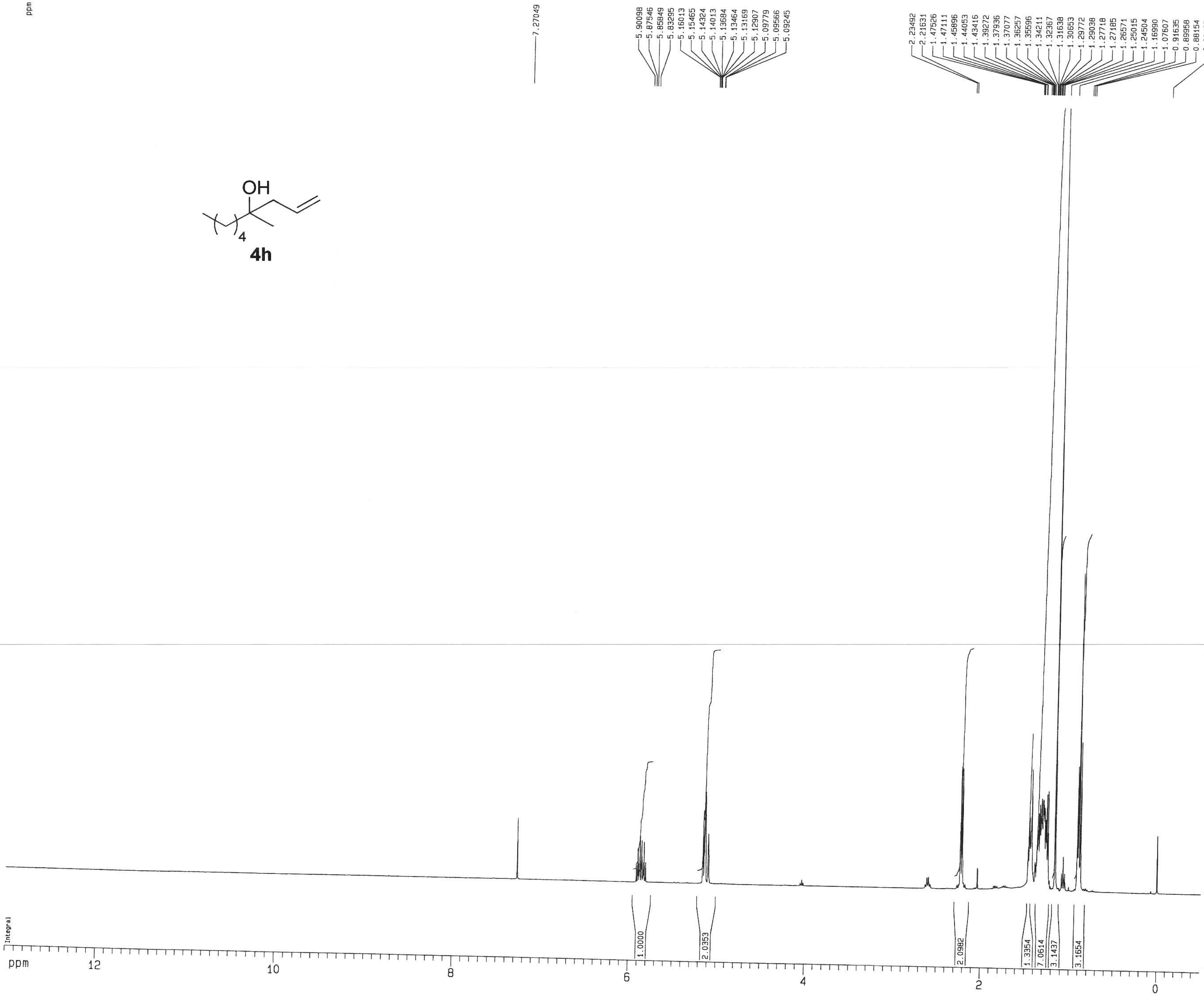
======== CHANNEL f1 ========
NUC1                     13C
P1                     11.00 usec
PL1                     0.00 dB
SFO1            100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2               mlev16
NUC2                      1H
PCPD2                  80.00 usec
PL2                     0.00 dB
PL12                   16.20 dB
SFO2            400.1328009 MHz

F2 - Processing parameters
SI                     65536
SF              100.6127500 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     22.80 cm
CY                      6.00 cm
F1P                  200.000 ppm
F1                  20122.55 Hz
F2P                    0.000 ppm
F2                      0.00 Hz
PPMCM                8.77193 ppm/cm
HZCM               882.56799 Hz/cm
```

1H spectrum



4h

Current Data Parameters
USER willia
NAME fjw-iv-124-heptane
EXPNO 1
PROCNO 1

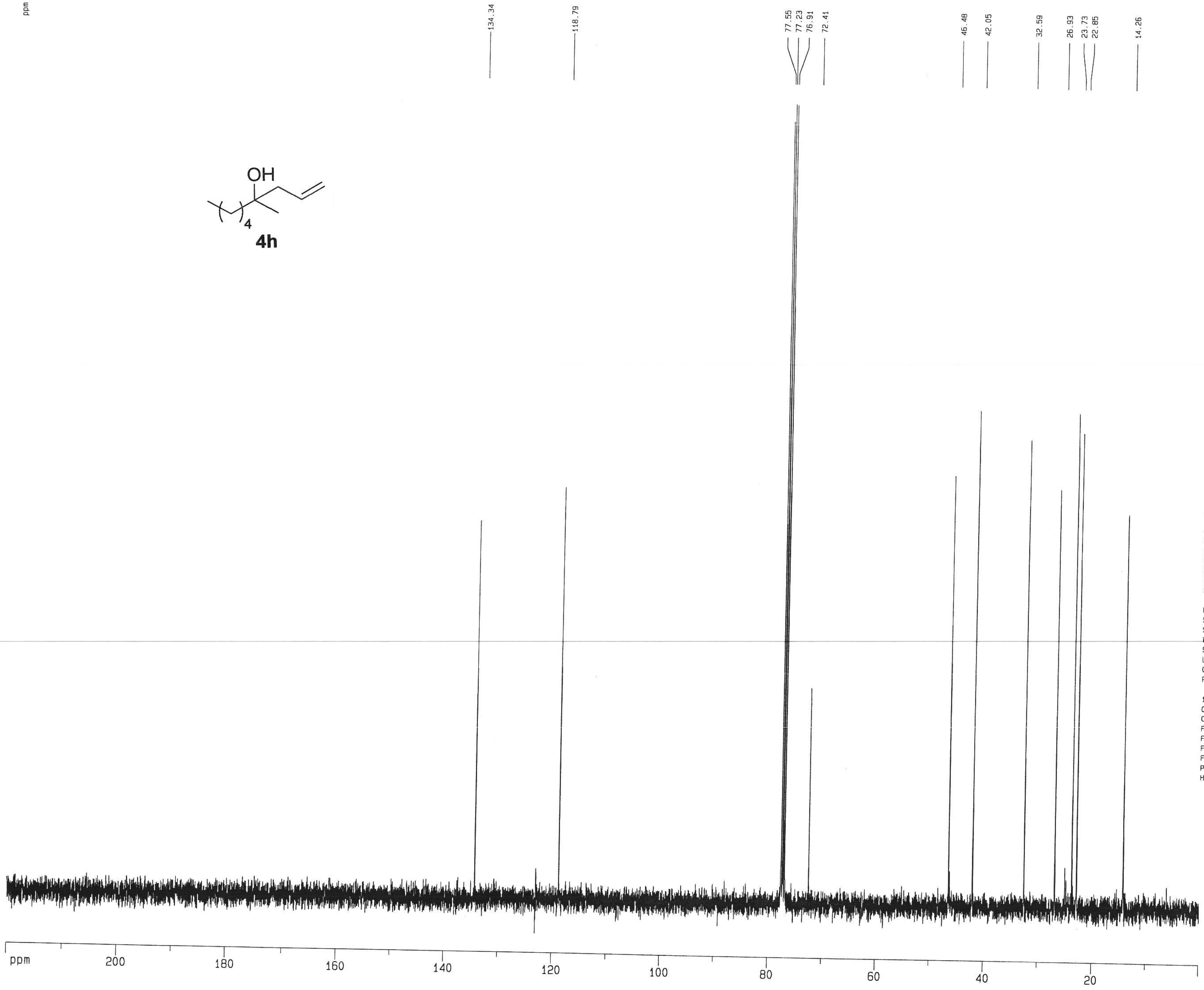
F2 - Acquisition Parameters
Date_ 20110708
Time 15.08
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 48
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 143.7
DW 78.000 usec
DE 4.50 usec
TE 298.1 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -0.60 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 13.000 ppm
F1 5201.69 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.59211 ppm/cm
HZCM 236.91908 Hz/cm

13C spectrum with 1H decoupling



4h

```
Current Data Parameters
USER                    willia
NAME       fjw-iv-124-heptane
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20110708
Time                     15.14
INSTRUM                 drx400
PROBHD      5 mm QNP H/F/P
PULPROG                zgdc30
TD                       65536
SOLVENT                  CDCl3
NS                         333
DS                           4
SWH               24154.590 Hz
FIDRES             0.368570 Hz
AQ                1.3566452 sec
RG                      9195.2
DW                  20.700 usec
DE                   20.39 usec
TE                    298.0 K
D1              0.10000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

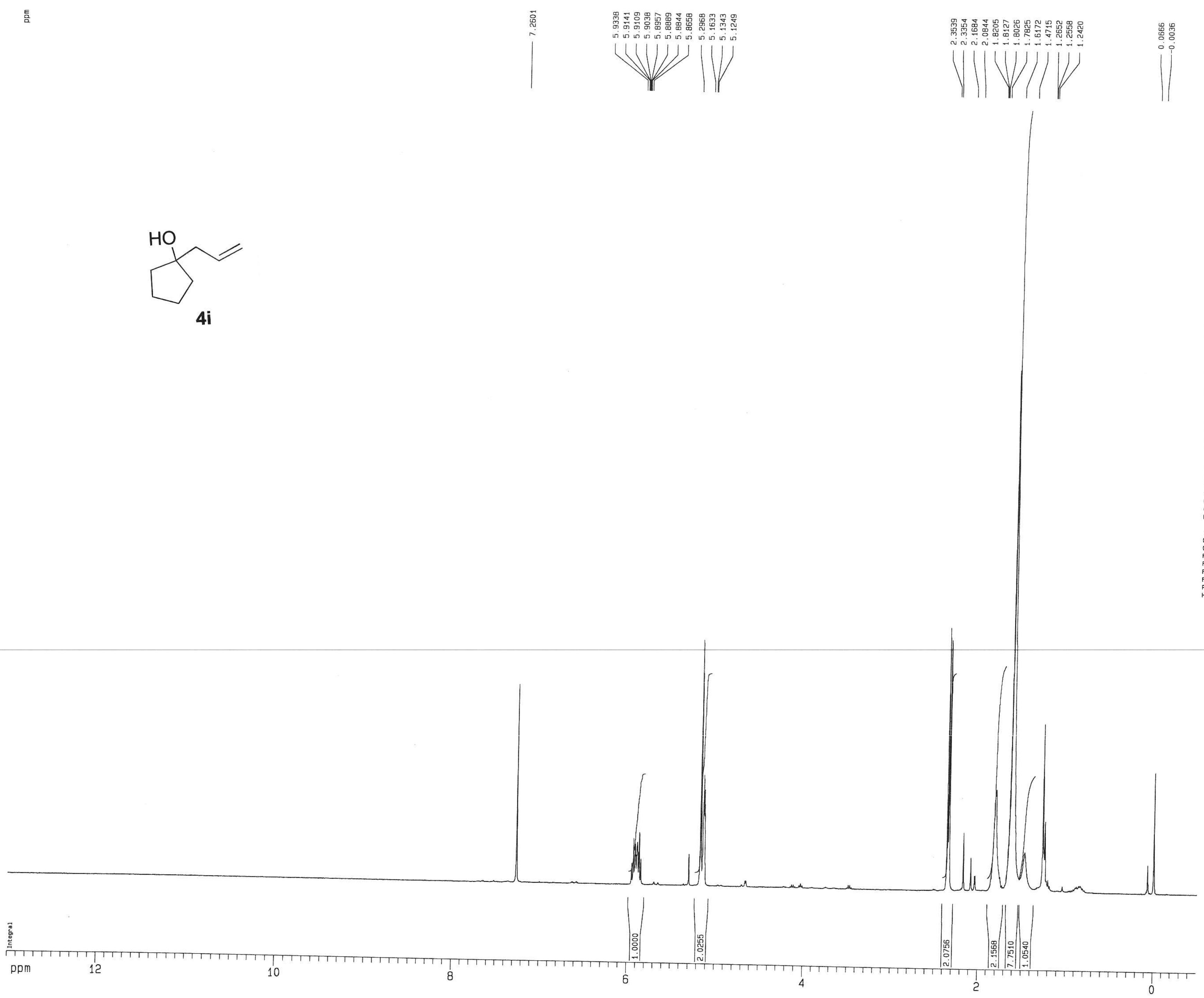
======== CHANNEL f1 ========
NUC1                       13C
P1                  11.00 usec
PL1                    0.00 dB
SFO1           100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2                mlev16
NUC2                        1H
PCPD2               80.00 usec
PL2                    0.00 dB
PL12                  16.20 dB
SFO2           400.1328009 MHz

F2 - Processing parameters
SI                       65536
SF             100.6127510 MHz
WDW                         EM
SSB                          0
LB                     1.00 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                    22.80 cm
CY                    15.50 cm
F1P                220.000 ppm
F1                22134.80 Hz
F2P                  0.000 ppm
F2                     0.00 Hz
PPMCM            9.64912 ppm/cm
HZCM            970.82477 Hz/cm
```

1H spectrum



4i

```
Current Data Parameters
USER                  willia
NAME      fjw-iv-07-25-2011-cyclopentyl
EXPNO                      1
PROCNO                     1

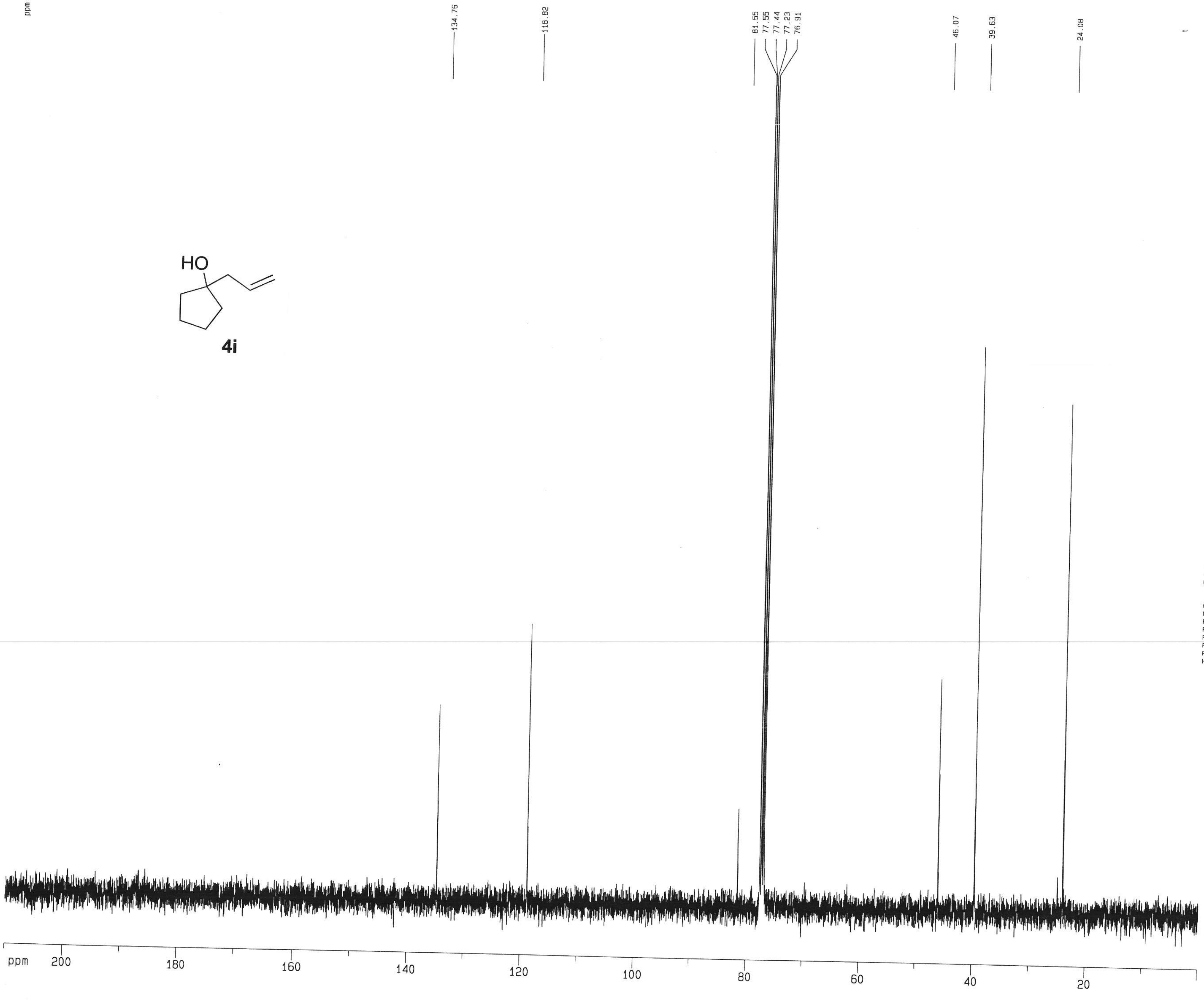
F2 - Acquisition Parameters
Date_               20110726
Time                    0.22
INSTRUM               drx400
PROBHD   5 mm QNP H/F/P
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        12
DS                         2
SWH                 6410.256 Hz
FIDRES              0.097813 Hz
AQ                 5.1118579 sec
RG                     322.5
DW                    78.000 usec
DE                      4.50 usec
TE                     298.0 K
D1                0.10000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     12.00 usec
PL1                    -0.60 dB
SFO1             400.1328009 MHz

F2 - Processing parameters
SI                     65536
SF               400.1300216 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      2.00

1D NMR plot parameters
CX                     22.80 cm
CY                     10.00 cm
F1P                   13.000 ppm
F1                   5201.69 Hz
F2P                   -0.500 ppm
F2                   -200.07 Hz
PPMCM                0.59211 ppm/cm
HZCM               236.91911 Hz/cm
```

13C spectrum with 1H decoupling



```
Current Data Parameters
USER                 willia
NAME      fjw-iv-07-25-2011-cyclopentyl
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20110726
Time                   0.25
INSTRUM              drx400
PROBHD     5 mm QNP H/F/P
PULPROG             zgdc30
TD                    65536
SOLVENT               CDCl3
NS                      590
DS                        4
SWH              24154.590 Hz
FIDRES            0.368570 Hz
AQ               1.3566452 sec
RG                   9195.2
DW                   20.700 usec
DE                    20.39 usec
TE                    298.0 K
D1              0.10000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

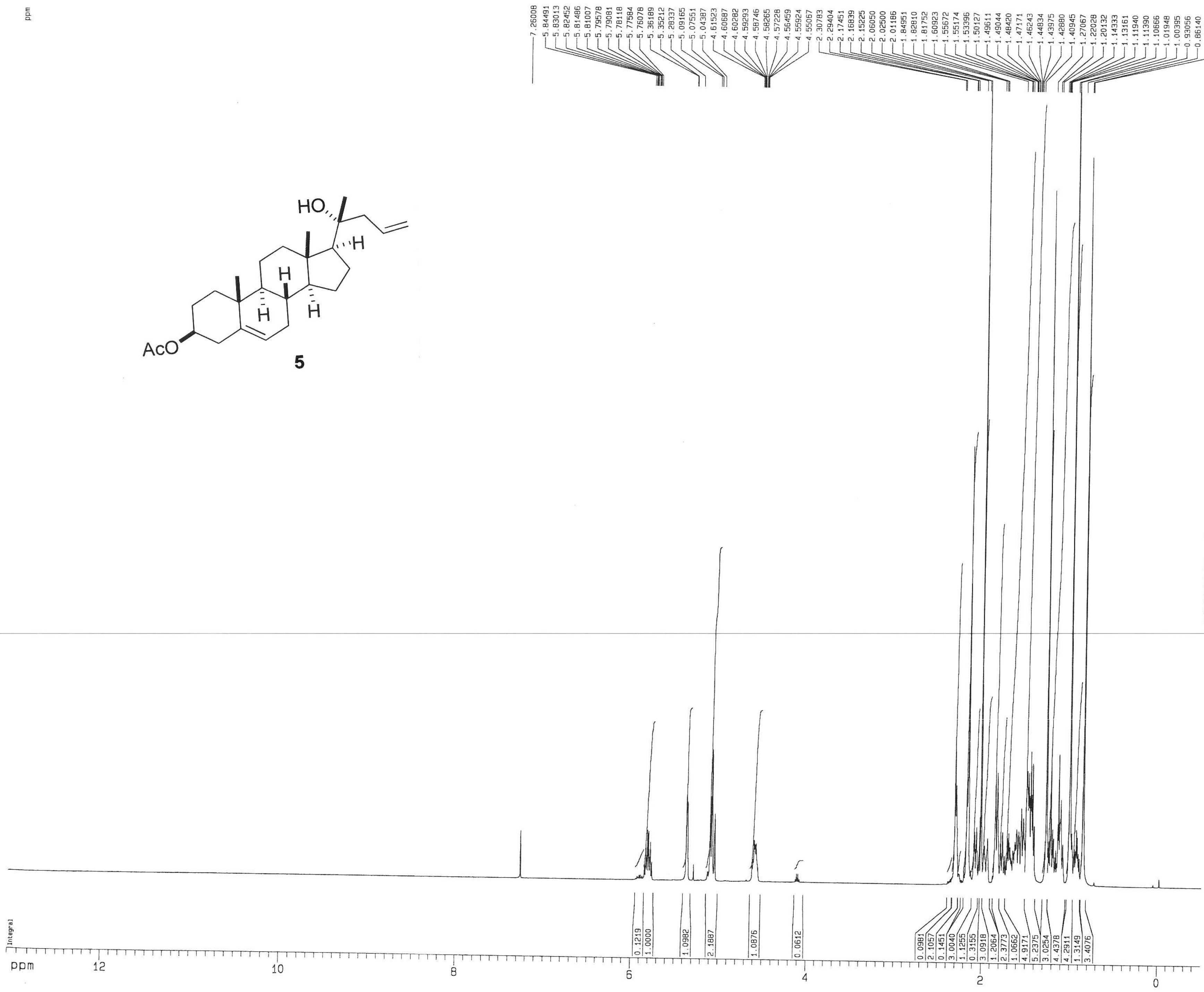
======== CHANNEL f1 ========
NUC1                    13C
P1                    11.00 usec
PL1                    0.00 dB
SFO1          100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2              mlev16
NUC2                     1H
PCPD2                 80.00 usec
PL2                    0.00 dB
PL12                  16.20 dB
SFO2          400.1328009 MHz

F2 - Processing parameters
SI                    65536
SF            100.6127510 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    22.80 cm
CY                    35.00 cm
F1P                 210.000 ppm
F1                 21128.68 Hz
F2P                   0.000 ppm
F2                     0.00 Hz
PPMCM               9.21053 ppm/cm
HZCM              926.69629 Hz/cm
```

1H spectrum



5

```
Current Data Parameters
USER                willia
NAME       fjw-iv-134-preg-Agcol-sp2
EXPNO                    1
PROCNO                   1

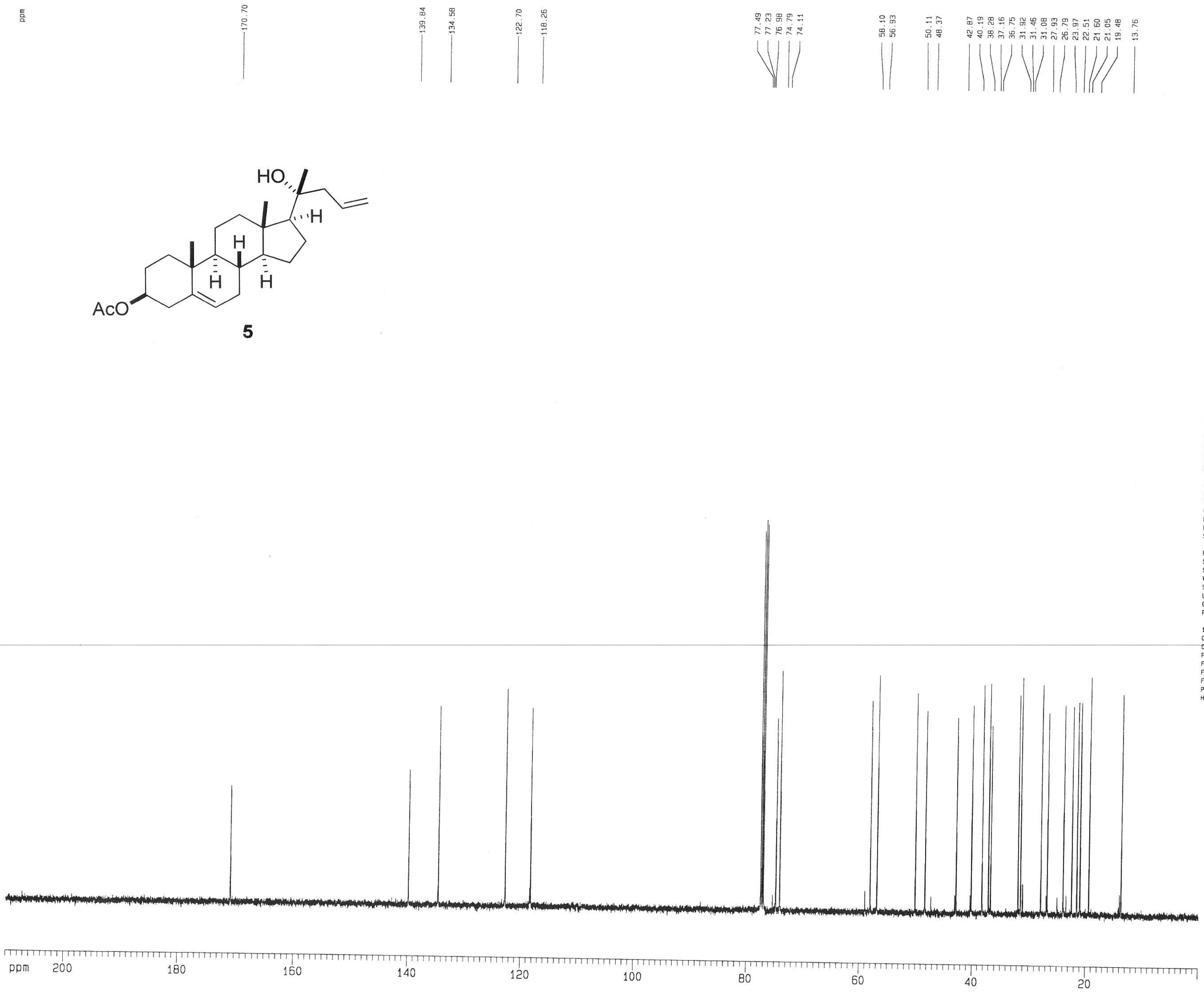
F2 - Acquisition Parameters
Date_             20110718
Time                 17.22
INSTRUM              gn500
PROBHD   5 mm broadband
PULPROG               zg30
TD                   81728
SOLVENT            CDCl3T
NS                       9
DS                       2
SWH               8012.820 Hz
FIDRES            0.098043 Hz
AQ               5.0999398 sec
RG                    40.3
DW                  62.400 usec
DE                    6.00 usec
TE                   298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   12.20 usec
PL1                  -5.00 dB
SFO1           499.5134966 MHz

F2 - Processing parameters
SI                   65536
SF             499.5100276 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   22.80 cm
CY                   20.00 cm
F1P                 13.000 ppm
F1                 6493.63 Hz
F2P                 -0.500 ppm
F2                 -249.76 Hz
PPMCM              0.59211 ppm/cm
HZCM             295.76254 Hz/cm
```

13C spectrum with 1H decoupling



```
Current Data Parameters
USER                willia
NAME      fjw-iv-134-preg-Agcol-sp2
EXPNO                    2
PROCNO                   1

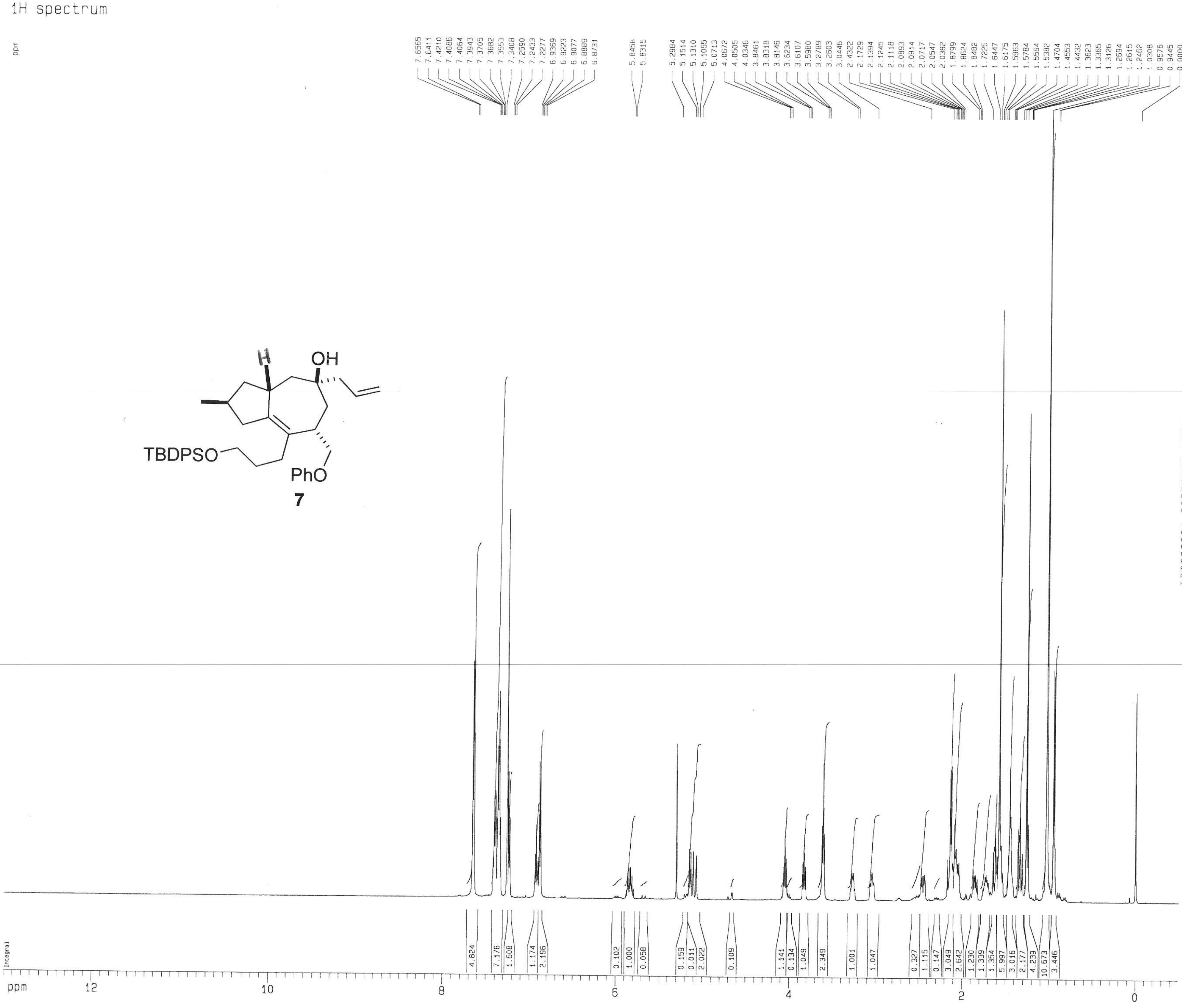
F2 - Acquisition Parameters
Date_             20110718
Time                 17.26
INSTRUM              gn500
PROBHD    5 mm broadband
PULPROG             zgdc30
TD                   65536
SOLVENT              CDCl3
NS                     256
DS                       4
SWH             30303.031 Hz
FIDRES           0.462388 Hz
AQ              1.0814105 sec
RG                  4597.6
DW                  16.500 usec
DE                    4.50 usec
TE                   298.0 K
D1             0.25000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    7.70 usec
PL1                   0.00 dB
SFO1           125.6157052 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -3.00 dB
PL12                 13.20 dB
SFO2           499.5124975 MHz

F2 - Processing parameters
SI                   65536
SF             125.6018712 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    2.00

1D NMR plot parameters
CX                   22.80 cm
CY                    7.50 cm
F1P                210.000 ppm
F1                26376.39 Hz
F2P                  0.000 ppm
F2                    0.00 Hz
PPMCM              9.21053 ppm/cm
HZCM            1156.85925 Hz/cm
```



7

```
Current Data Parameters
USER                willia
NAME       fjw-iv-130-smc-iv-1233-col2-char
EXPNO                    1
PROCNO                   1

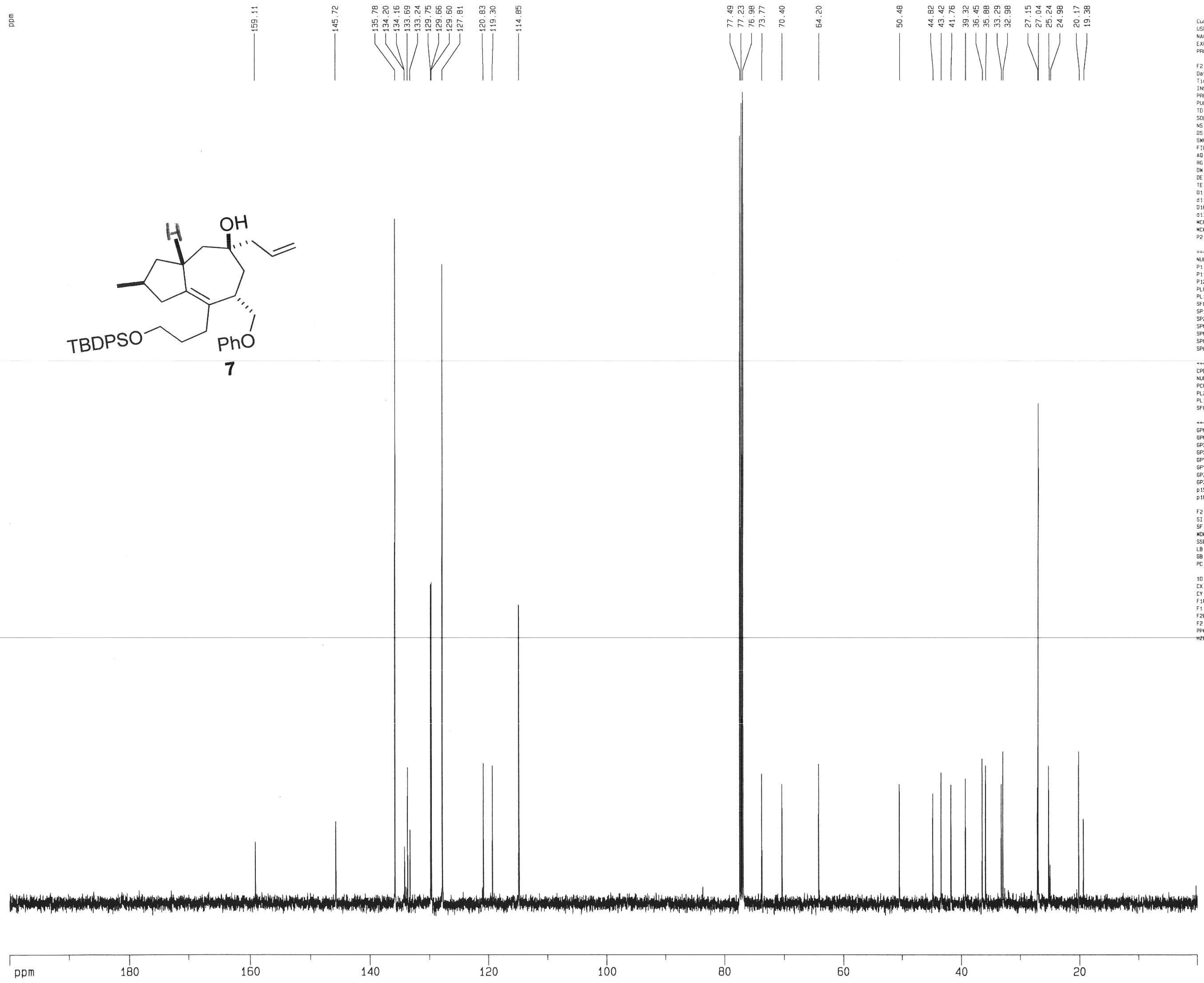
F2 - Acquisition Parameters
Date_             20110702
Time                 19.27
INSTRUM            cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG               zg30
TD                   81728
SOLVENT              CDCl3
NS                       8
DS                       2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0999398 sec
RG                       5
DW              62.400 usec
DE                6.00 usec
TE               298.0 K
D1          0.10000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                7.50 usec
PL1               1.60 dB
SFO1       500.2235015 MHz

F2 - Processing parameters
SI                   65536
SF         500.2200314 MHz
WDW                     EM
SSB                      0
LB                0.30 Hz
GB                       0
PC                    4.00

1D NMR plot parameters
CX               22.80 cm
CY               25.00 cm
F1P             13.000 ppm
F1             6502.86 Hz
F2P             -0.500 ppm
F2             -250.11 Hz
PPMCM          0.59211 ppm/cm
HZCM         296.18292 Hz/cm
```

Z-restored spin-echo 13C spectrum with 1H decoupling



7

```
Current Data Parameters
USER                willia
NAME      fjw-iv-130-smc-iv-1233-col2-char
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20110702
Time               19.34
INSTRUM          cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   581
DS                    16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG                  8192
DW                16.500 usec
DE                  6.00 usec
TE                 298.0 K
D1            0.25000000 sec
d11           0.03000000 sec
D16           0.00020000 sec
d17           0.00019600 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
P2                 31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                 15.50 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                 3.20 dB
SP2                 3.20 dB
SPNAM1    Crp60,0.5,20.1
SPNAM2       Crp60comp.4
SPOFF1              0.00 Hz
SPOFF2              0.00 Hz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 1.60 dB
PL12               24.60 dB
SFO2         500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPX1                0.00 %
GPX2                0.00 %
GPY1                0.00 %
GPY2                0.00 %
GPZ1               30.00 %
GPZ2               50.00 %
p15               500.00 usec
p16              1000.00 usec

F2 - Processing parameters
SI                 65536
SF           125.7803992 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P              200.000 ppm
F1              25156.08 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            8.77193 ppm/cm
HZCM          1103.33679 Hz/cm
```