Supporting Information (SI)

Syntheses of Arabinose Derived Pyrrolidine Catalysts and Their Applications in Intramolecular Diels-Alder Reactions

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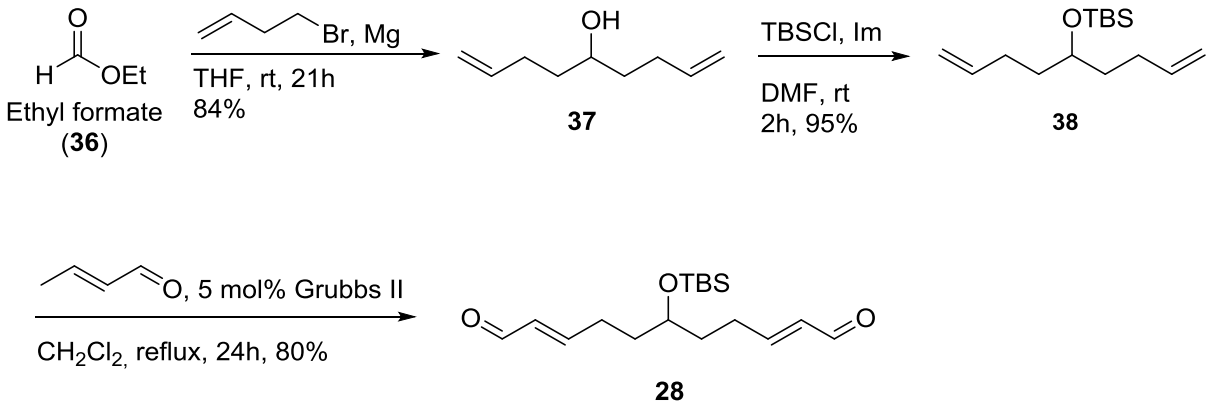
Preparation of Dienal 28



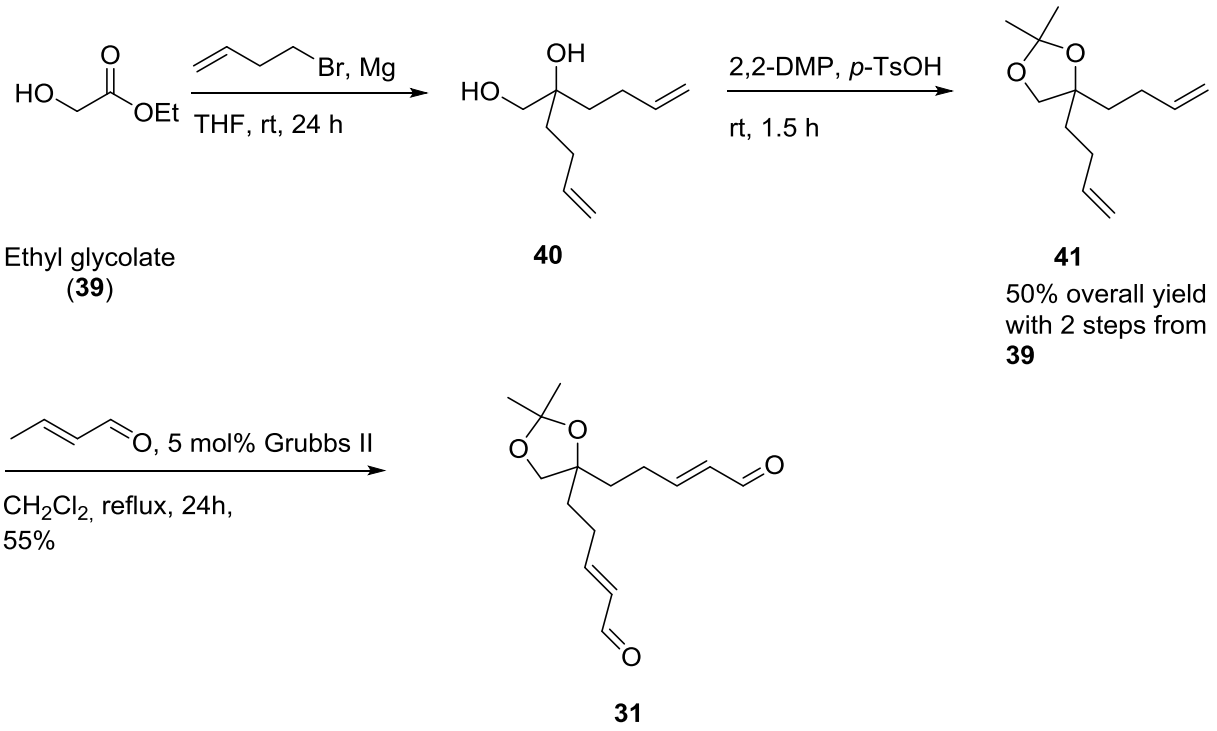
Homoallylic alcohol 37. To a suspension of magnesium powder (1.35 g, 55.7 mmol) in THF (15 mL) was added 1,2-dibromoethane (0.03 mL, 0.309 mmol) and the mixture was stirred at room temperature for 15 min. A solution of butenyl bromide (1.9 mL, 18.6 mmol) was added dropwise to the mixture at a rate to maintain a gentle reflux of the THF. After the addition of the butenyl bromide solution, the mixture was heated under reflux for 2 h. Then the reaction mixture was cooled down to 0 °C and a solution of ethyl formate **36** (0.25 mL, 3.09 mmol) was added and stirring was continued for 21 h. Saturated NH_4Cl solution (10 mL) was added to the reaction mixture at 0 °C. The reaction mixture was extracted with EtOAc (4 × 100 mL). The combined organic extracts were dried over $MgSO_4$ and filtered. The filtrate was concentrated under reduced pressure and the residue was purified by flash column chromatography (hexane:Et_2O, 3:1) to afford alcohol **37** (366 mg, 84%) as a colorless oil: R_f 0.33 (hexane:Et_2O, 1:1); IR (thin film) 3526, 3339, 3077, 2928, 2851, 1697, 1643, 1451, 910 cm^{-1}; ^1H NMR δ 1.46–1.55 (4H, m), 1.98 (1H, br s), 2.06–2.21 (4H, m), 3.59–3.62 (1H, m), 4.94 (2H, ddt, *J* = 1.3, 3.2, 10.2 Hz), 5.01 (2H, ddt, *J* = 1.6, 3.5, 17.1 Hz), 5.75–5.86 (2H, m); ^{13}C NMR δ 30.0 (CH_2), 36.4 (CH_2), 70.8 (CH), 114.7 (CH_2), 138.5 (CH); MS (ESI) *m/z* (relative intensity) 141 ($[M+H]^+$ 100); HRMS (ESI) calcd for $C_9H_{16}O$ $[M+H]^+$ 141.1274, found 141.1274.

Silyl ether 38. A solution of the alcohol **37** (53.0 mg, 0.378 mmol), imidazole (206 mg, 3.02 mmol) and *tert*-butyl dimethyl silyl chloride (TBDMSCl) (228 mg, 1.51 mmol) in dry DMF (0.5 mL) was stirred at room temperature for 2 h. The mixture was quenched with saturated $NaHCO_3$ (30 mL) solution and the aqueous phase was extracted with CH_2Cl_2 (3 × 100 mL). The combined organic extracts were washed with brine, dried over anhydrous $MgSO_4$, and filtered. The filtrate was concentrated under reduced

pressure and the residue was purified by flash chromatography (hexane) to afford silyl ether **38** (91.1 g, 95%) as a colorless oil: R_f 0.50 (hexane:CH_2Cl_2, 1:8); IR (thin film) 3338, 3113, 2930, 2707, 1697, 1517, 834 cm^{-1}; ^1H NMR δ 0.06 (6H, s), 0.90 (9H, s), 1.51–1.57 (4H, m), 2.04–2.17 (4H, m), 3.68–3.73 (1H, m), 4.93–4.96 (2H, m), 4.99–5.04 (2H, m), 5.77–5.87 (2H, m); ^{13}C NMR δ -4.33 (CH_3), 18.2 (C), 26.0 (CH_3), 29.6 (CH_2), 36.3 (CH_2), 71.3 (CH), 114.3 (CH_2), 139.0 (CH); MS (ESI) *m/z* (relative intensity) 255 ($[M+H]^+$, 100); HRMS (ESI) calcd for $C_{15}H_{30}OSi$ $[M+H]^+$ 255.2139, found 255.2134.

Dienal 28. To a solution of the sily ether **38** (92.3 mg, 0.363 mmol) in a dry CH_2Cl_2 (4 mL) was added crotonaldehyde (0.09 mL, 1.09 mmol) and 2nd generation Grubbs catalyst (15.4 mg, 0.0181 mmol). The resultant mixture was heated under reflux for 5 h. The reaction mixture was concentrated under reduced pressure and the crude residue was directly purified by flash chromatography (hexane :EtOAc, 3:1) to afford dienal **28** (90 mg, 80%) as a pale yellow oil: R_f 0.33 (hexane :EtOAc, 3:1); IR (thin film) 2934, 2857, 2816, 2734, 1691, 1638, 1255, 1134, 1094, 975, 838, 777 cm^{-1}; ^1H NMR δ 0.06 (6H, s), 0.89 (9H, s), 1.64–1.69 (4H, m), 2.31–2.45 (4H, m), 3.76–3.82 (1H, m), 6.12 (2H, ddt, *J* = 15.6, 7.84, 1.48 Hz), 6.86 (2H, dt, *J* = 15.6, 6.64 Hz), 9.51 (2H, d, *J* = 7.84 Hz);^{13}C NMR δ -4.41 (CH_3), 18.0 (C), 25.8 (CH_3), 28.3 (CH_2), 34.8 (CH_2), 70.5 (CH), 133.0 (CH), 158.2 (CH), 193.8 (CH); MS (ESI) *m/z* (relative intensity) 333 ($[M+Na]^+$, 100); HRMS (ESI) calcd for $C_{17}H_{30}O_3Si$ $[M+Na]^+$ 333.1856, found 333.1857.

Preparation of Dienal 31

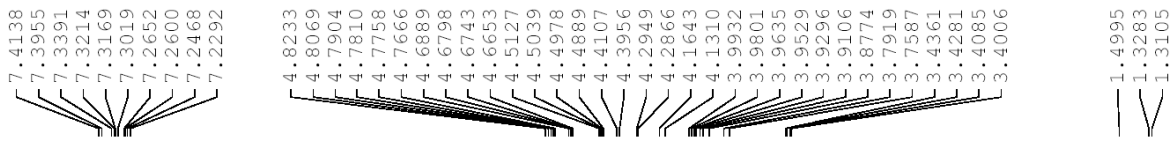


Diol 40. To a suspension of magnesium powder (3.85 g, 158 mmol) in THF (53 mL) was added 1,2-dibromoethane (0.05 mL, 0.528 mmol) and the mixture was stirred at room temperature for 15 min. A solution of butenyl bromide (5.40 mL, 52.8 mmol) was added dropwise to the mixture at a rate to maintain a gentle reflux of the THF. After the addition of the butenyl bromide solution, the mixture was heated under reflux for 2 h. Then the reaction mixture was cooled down to 0 °C and a solution of ethyl glycolate **39** (0.5 mL, 5.28 mmol) was added and stirring was continued for 24 h. Saturated NH_4Cl solution (10 mL) was added to the reaction mixture at 0 °C. The reaction mixture was extracted with EtOAc (4 × 100 mL). The combined organic extracts were dried over $MgSO_4$ and filtered. The filtrate was concentrated under reduced pressure and the residue was purified by flash column chromatography (hexane:EtOAc, 3:1) to afford diol **40** (629 mg, 70%) as a colorless oil: R_f 0.37 (hexane:EtOAc, 1:1); IR (thin film) 3381, 3076, 2937, 2865, 1642, 1448, 1060, 998, 911 cm^{-1}; ^1H NMR δ 1.55–1.60 (4H, m), 2.05–2.10 (4H, m), 2.33 (1H, br s), 2.54 (1H, br s), 3.46 (2H, s), 4.94–5.06 (4H, m), 5.77–5.87 (2H, m); ^{13}C NMR δ 27.8 (CH_2), 34.9 (CH_2), 67.8 (CH_2), 74.6 (C), 114.7 (CH_2), 138.5 (CH); MS (ESI) *m/z* (relative intensity) 193 ($[M+Na]^+$, 100); HRMS (ESI) calcd for $C_{10}H_{10}O_2$ $[M+Na]^+$ 193.1199, found 193.1198.

Diene 41. 2,2-dimethoxypropane (16 mL, 132 mmol) and TsOH (90.9 mg, 0.528 mmol) were added to diol **40** (629 mg, 3.70 mmol) at room temperature. The resultant solution was stirred at room temperature for another 1.5 h. The reaction was quenched by dropwise addition of Et_3N until pH~8 (test by pH paper). Concentration of the filtrate followed by flash chromatography (hexane:Et_2O, 10:1) yielded diene **41** (552 mg, 71%) as a colorless oil: R_f 0.33 (hexane:Et_2O, 10:1); IR (thin film) 3076, 2984, 2938, 2865, 1642, 1450, 1373, 1251, 1212, 1062, 991, 870 cm^{-1}; ^1H NMR δ 1.39 (6H, s), 1.59–1.74 (4H, m), 2.02–2.15 (4H, m), 3.76 (2H, s), 4.93–4.97 (2H, m), 5.00–5.06 (2H, m), 5.77–5.87 (2H, m); ^{13}C NMR δ 27.1 (CH_3), 28.5 (CH_2), 36.5 (CH_2), 72.8 (CH_2), 83.0 (C), 109.0 (C), 114.6 (CH_2), 138.4 (CH); MS (ESI) *m/z* (relative intensity) 211 ($[M+H]^+$, 100); HRMS (ESI) calcd for $C_{13}H_{22}O_2$ $[M+H]^+$ 211.1693, found 211.1694.

Dienal 31. To a solution of the diene **41** (100 mg, 0.475 mmol) in a dry CH_2Cl_2 (6 mL) was added crotonaldehyde (0.12 mL, 1.43 mmol) and 2nd generation Grubbs catalyst (20.2 mg, 0.00238 mmol). The resultant mixture was heated under reflux for 24 h. The reaction mixture was concentrated under reduced pressure and the crude residue was directly purified by flash chromatography (hexane:EtOAc, 1:1) to afford dienal **31** (69.4 mg, 55%) as a pale yellow oil: R_f 0.20 (hexane:EtOAc, 2:1); IR (thin film) 2986, 2936, 2867, 2818, 2734, 1688, 1639, 1374, 1057, 978, 871 cm^{-1}; ^1H NMR δ 1.40 (6H, s), 1.78 (4H, t, *J* = 8.3 Hz), 2.32–2.50 (4H, m), 3.82 (2H, s), 6.14 (2H, ddt, *J* = 15.6, 7.8, 1.4 Hz), 6.86 (2H, dt, *J* = 15.6, 6.6 Hz), 9.51 (2H, d, *J* = 7.8 Hz); ^{13}C NMR δ 27.0 (CH_3), 27.4 (CH_2), 35.4 (CH_2), 72.4 (CH_2), 82.0 (C), 109.8 (C), 133.1 (CH), 157.4 (CH), 193.8 (CH); MS (ESI) *m/z* (relative intensity) 289 ($[M+Na]^+$, 100); HRMS (ESI) calcd for $C_{15}H_{22}O_4$ $[M+Na]^+$ 289.1410, found 289.1407.

^{1}H NMR



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FIDRES	0.125483 Hz
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TE	294.3 K
D1	1.00000000 sec
TD0	1
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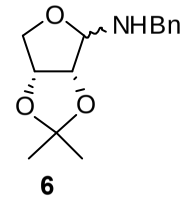
PC	1.00



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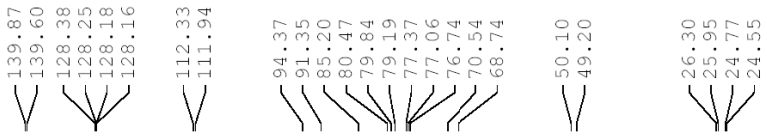
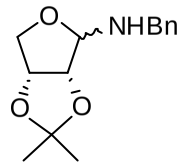
^{13}C NMR





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SFO1	100.6228298 MHz
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NUC2	1H
PCPD2	90.00 usec
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PL12	15.66 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.22585411 W
PL13W	0.21272963 W
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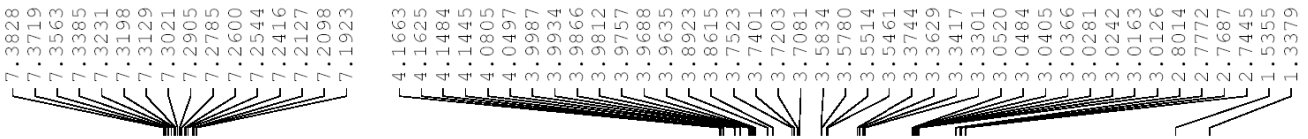
PC	1.40

^{1}H NMR



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SOLVENT CDCl3
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SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 32
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DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1

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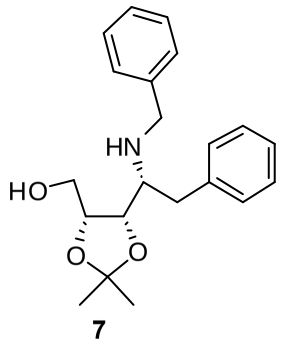

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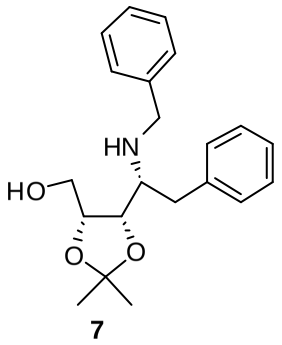
^{13}C NMR



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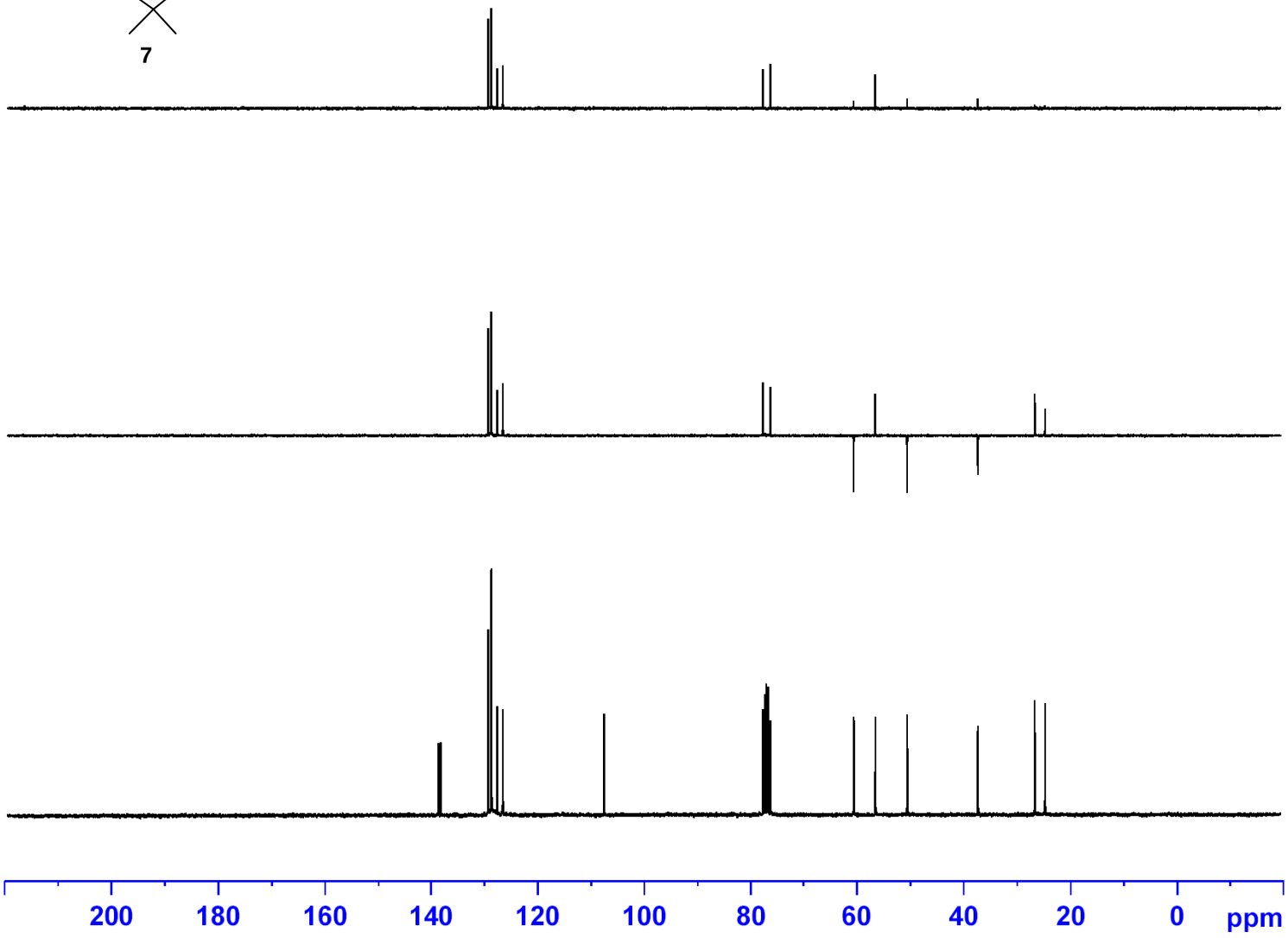

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TD0                    1

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======== CHANNEL f2 ========
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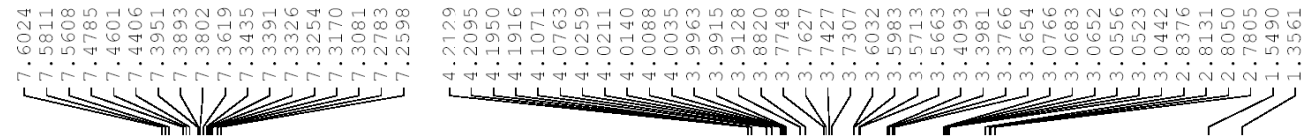
^{1}H NMR



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FIDRES	0.125483 Hz
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RG	57
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TE	294.8 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
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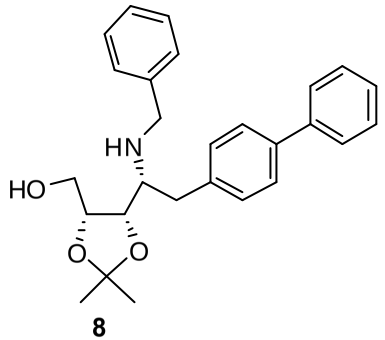

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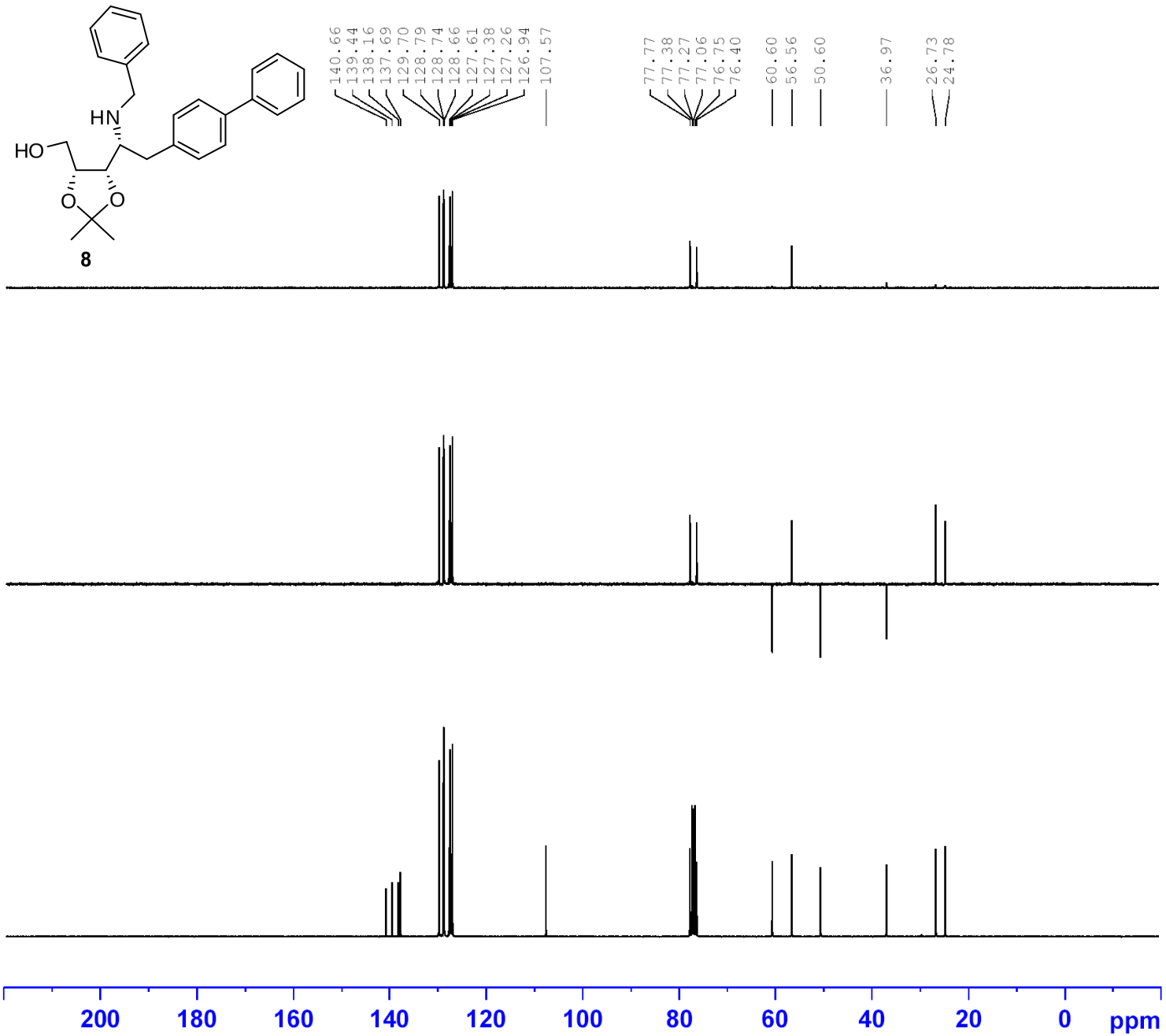

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======== CHANNEL f2 ========	
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NUC2	1H
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PL12	15.66 dB
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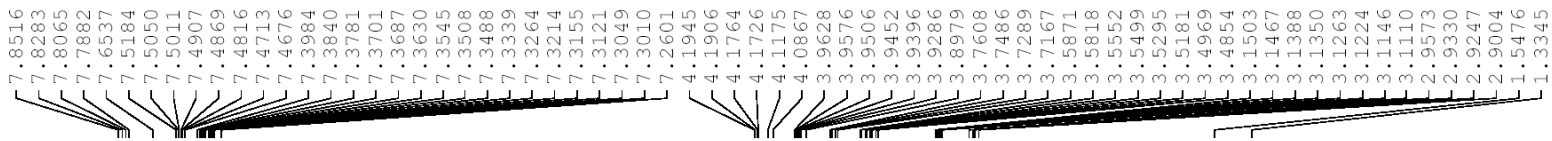
^{13}C NMR

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FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                      32
DW                  60.800 usec
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TE                  297.9 K
D1             1.00000000 sec
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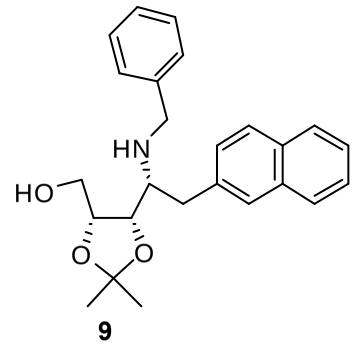

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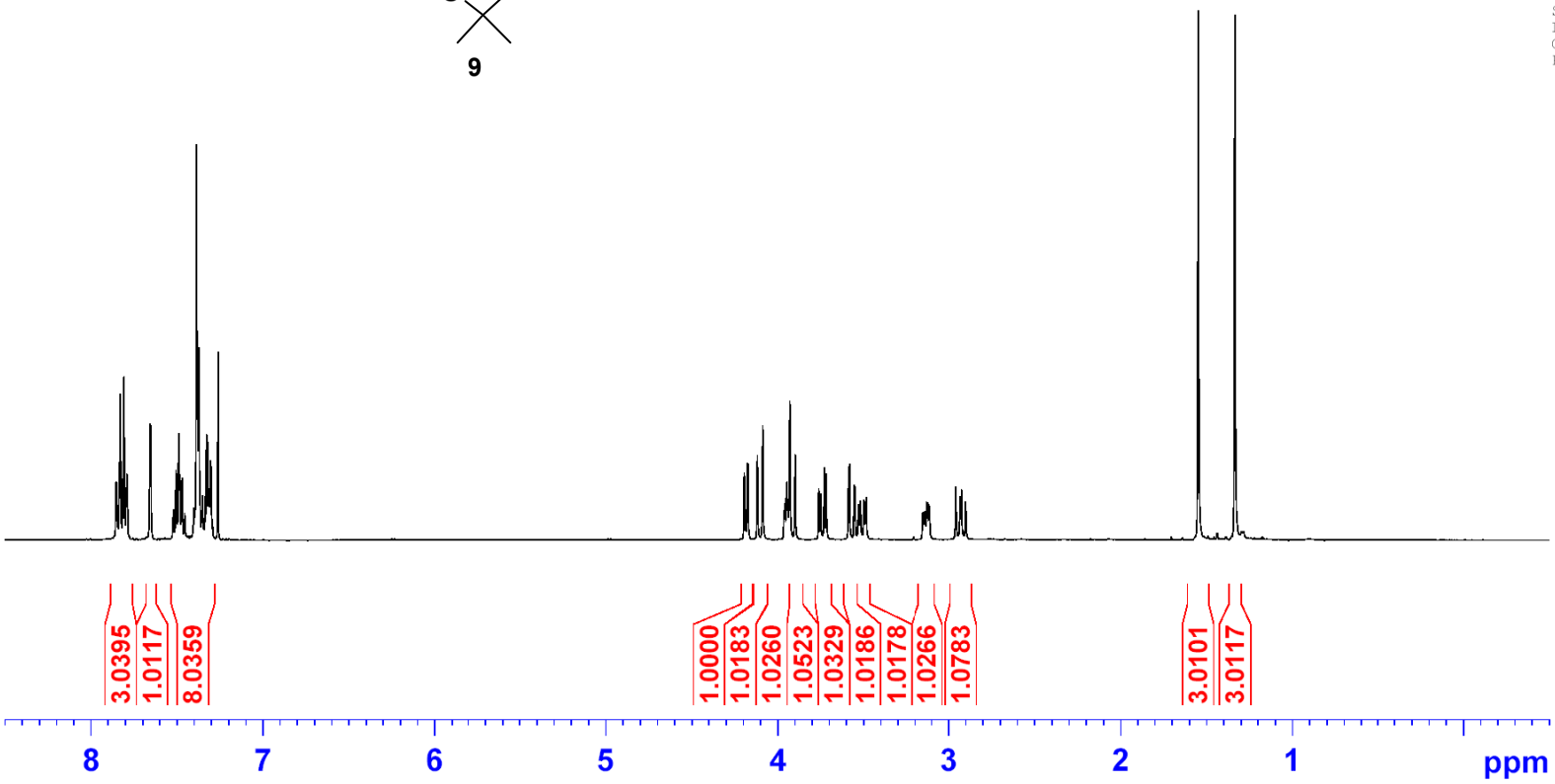
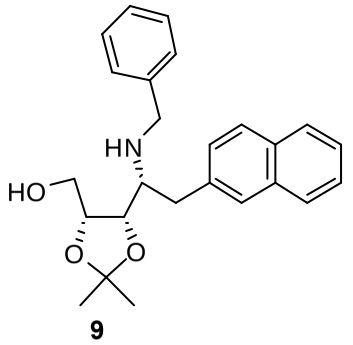


^{13}C NMR



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50.67

37.53

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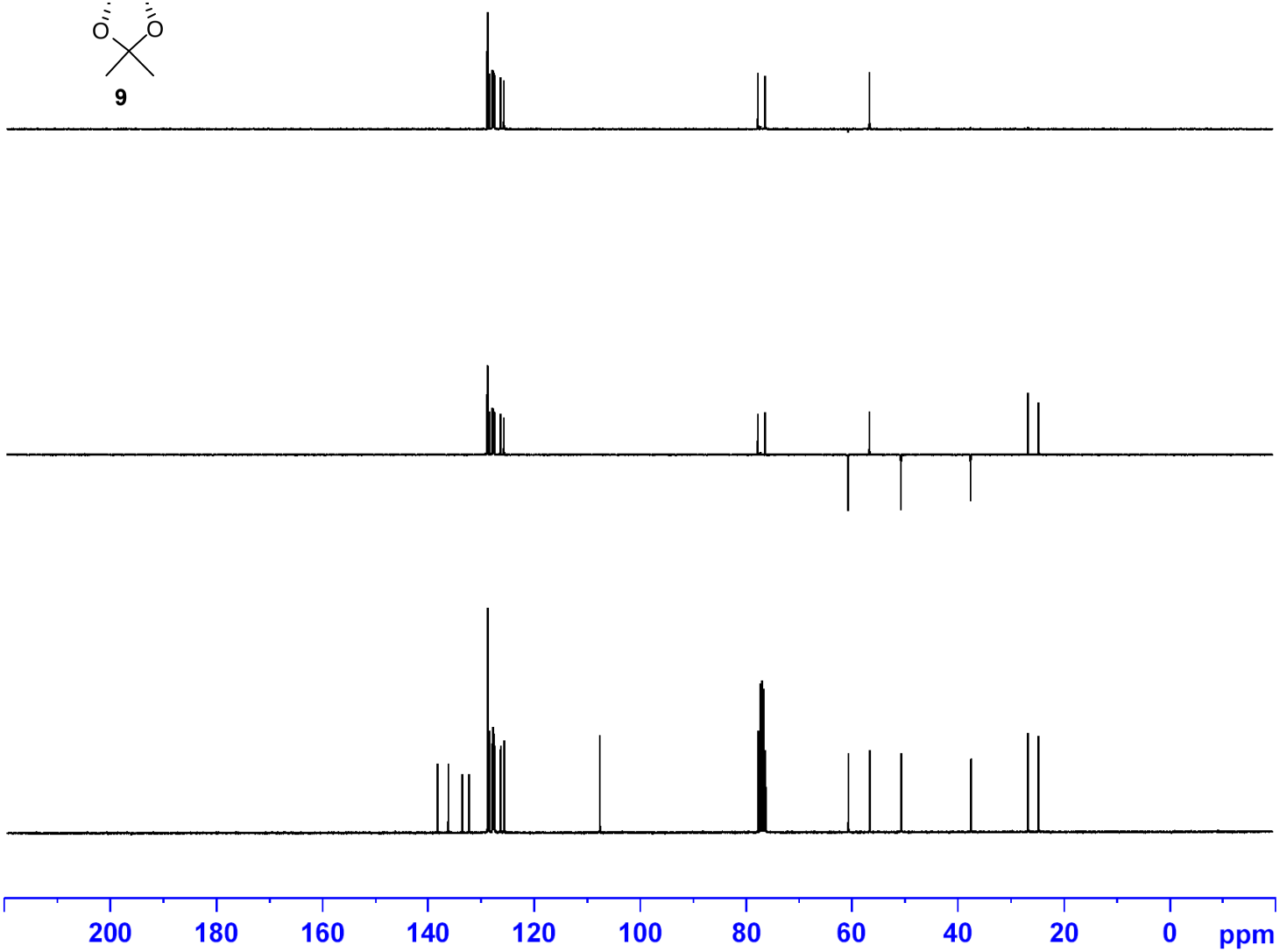

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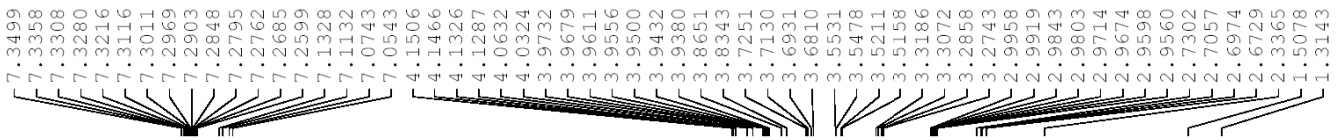
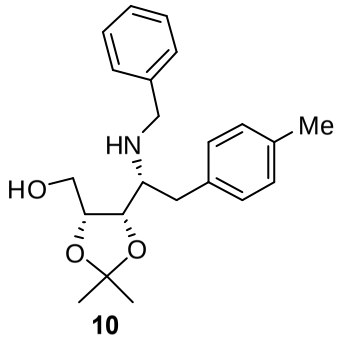
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	298.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.90 usec
PL1	-2.00 dB
PL1W	55.33689499 W
SFO1	100.6379183 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-1.00 dB
PL12	15.16 dB
PL13	18.62 dB
PL2W	13.56617069 W
PL12W	0.32844096 W
PL13W	0.14806664 W
SFO2	400.1916008 MHz
SI	32768
SF	100.6278608 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{1}H NMR

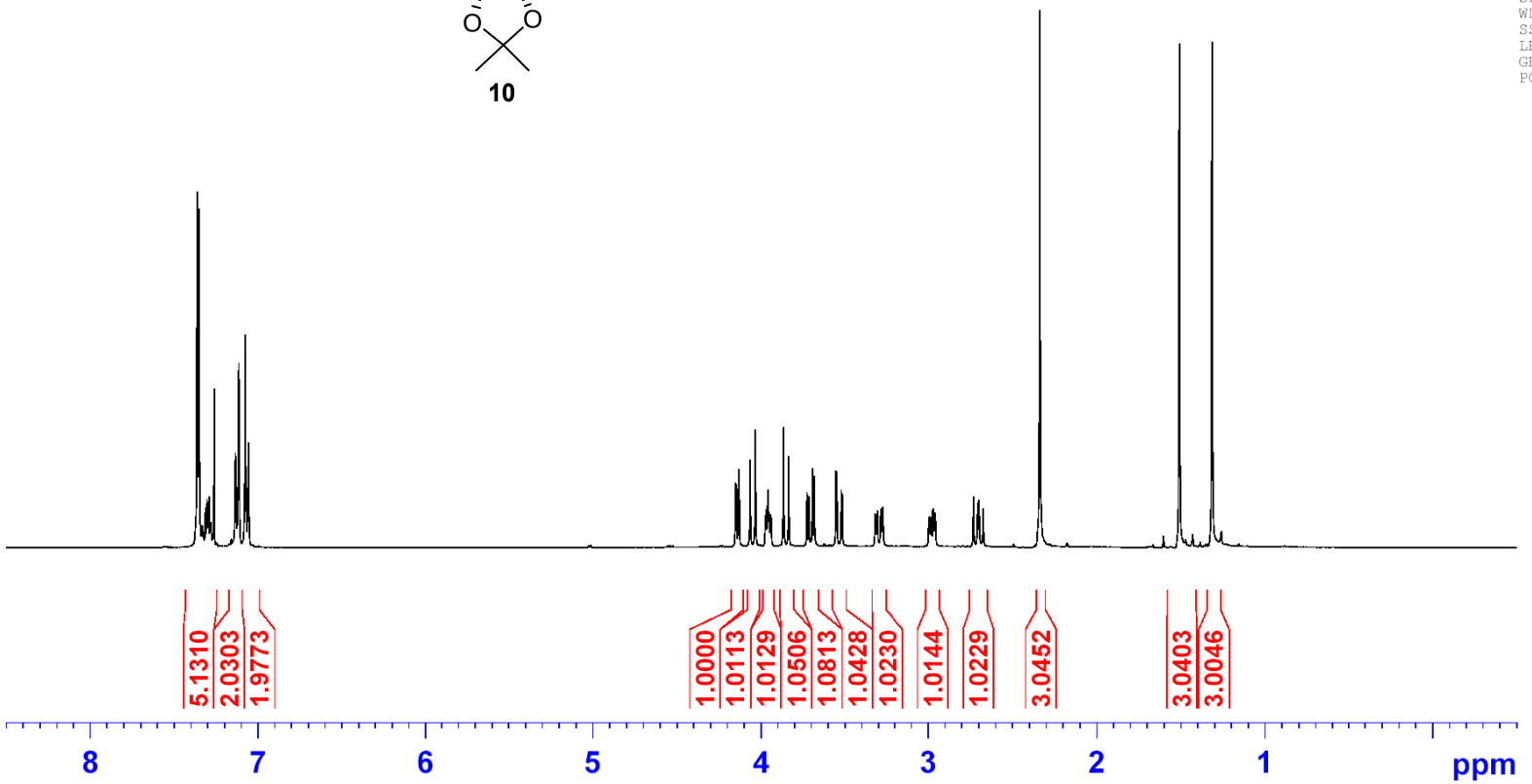




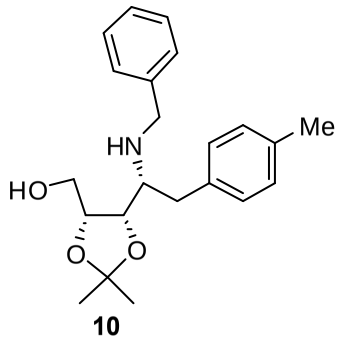
10

NAME WKWD32_2
EXPNO 1
PROCNO 1
Date_ 20110519
Time 12.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 64
DW 60.800 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 13.56617069 W
SFO1 400.1924713 MHz
SI 32768
SF 400.1900138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



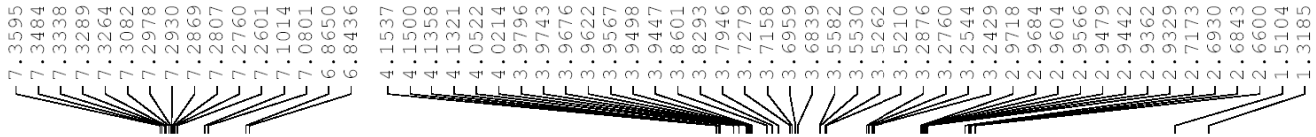
^{13}C NMR



10

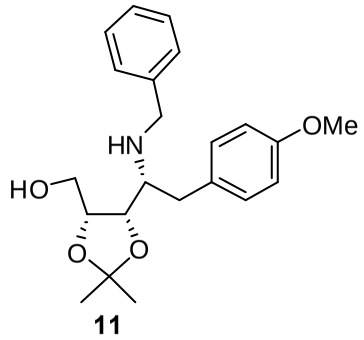
NAME	WKWD32_c13_1
EXPNO	1
PROCNO	1
Date_	20100828
Time	9.41
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	11637
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	14.50 usec
PL1	-4.00 dB
PL1W	90.22689819 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	18.80 dB
PL13	18.80 dB
PL2W	13.17734718 W
PL12W	0.10960442 W
PL13W	0.10960442 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127675 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{1}H NMR

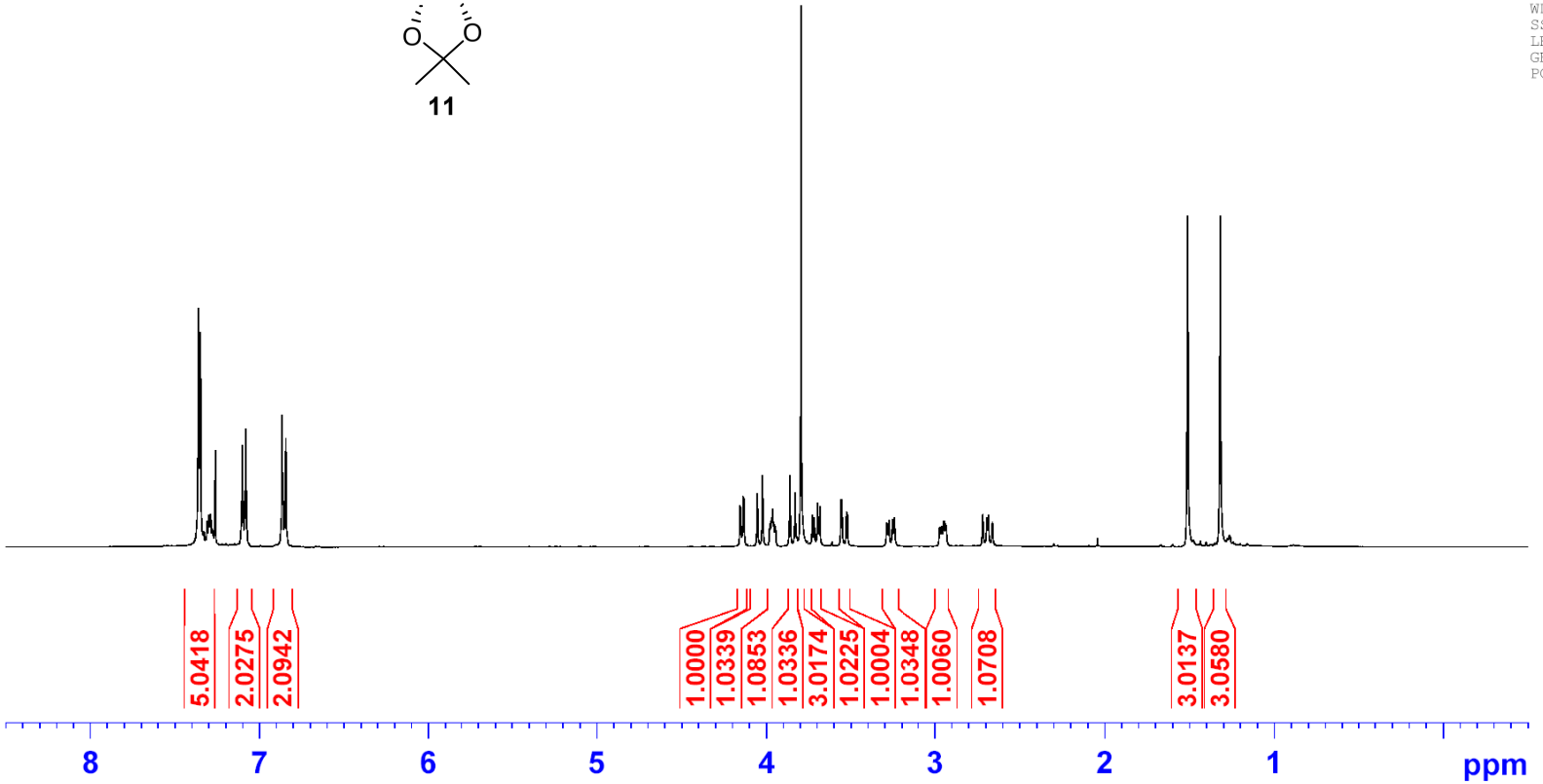


NAME WKWD34_1
EXPNO 1
PROCNO 1
Date_ 20110513
Time 18.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 25.4
DW 60.800 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1

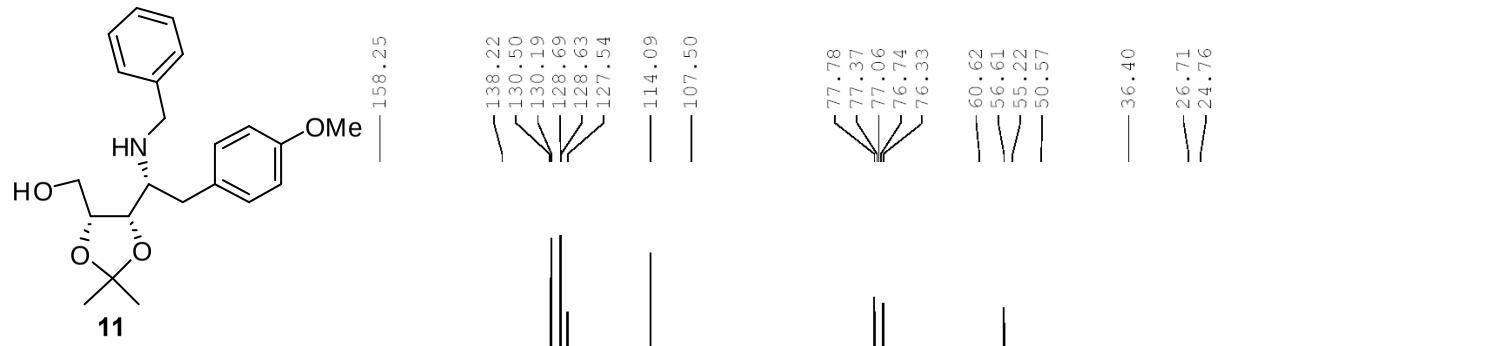
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 13.56617069 W
SFO1 400.1924713 MHz
SI 32768
SF 400.1900136 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



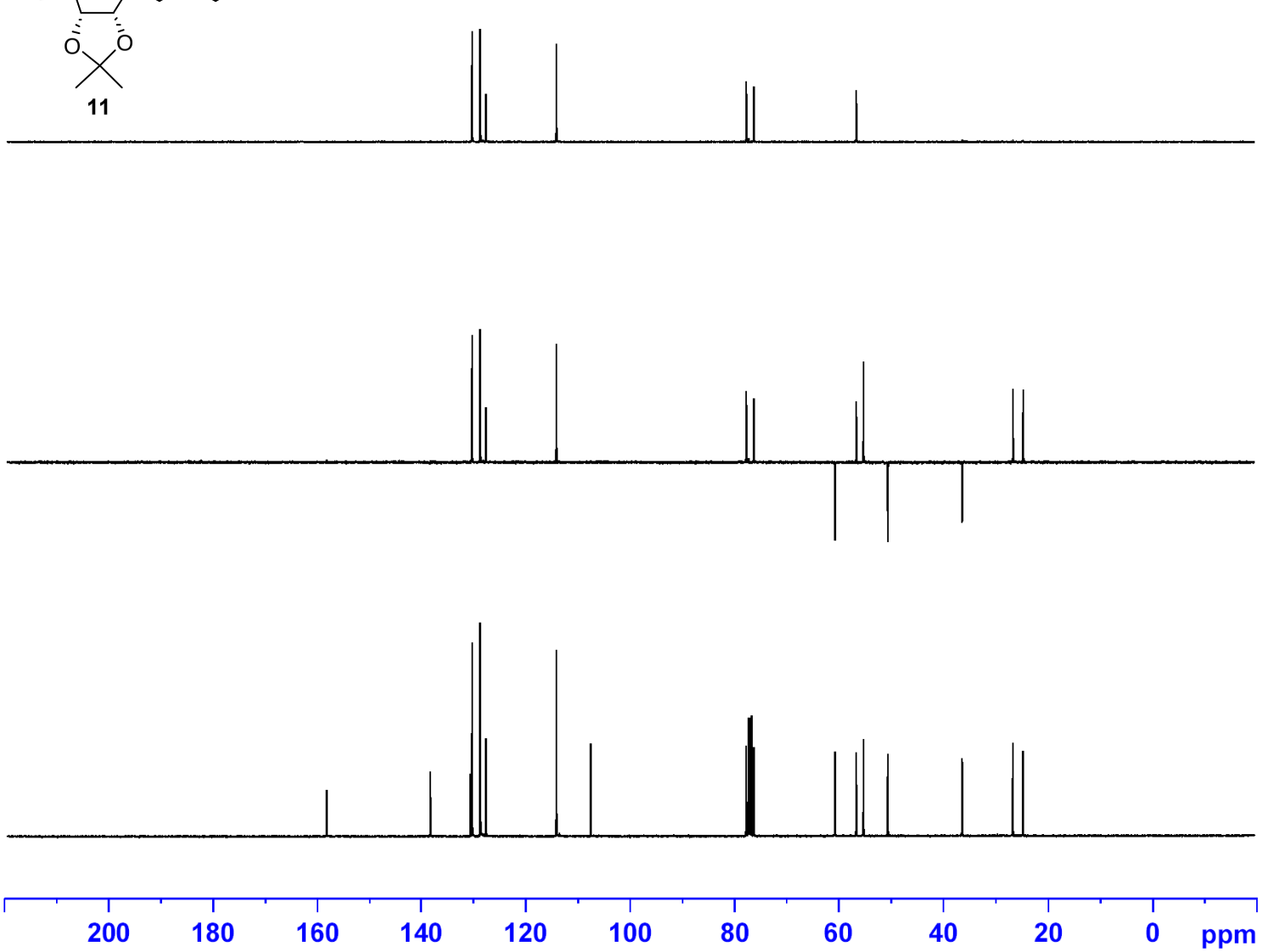
11



^{13}C NMR

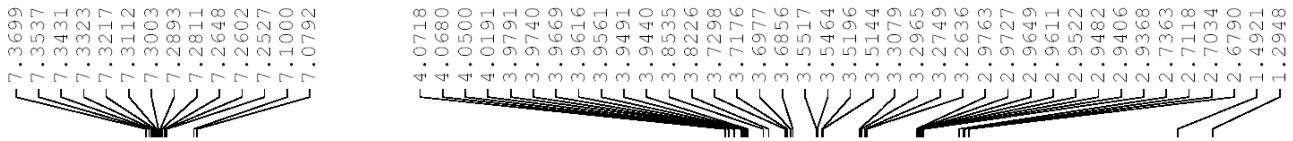


158.25
138.22
130.50
130.19
128.69
128.63
127.54
114.09
107.50
77.78
77.37
77.06
76.74
76.33
60.62
56.61
55.22
50.57
36.40
26.71
24.76

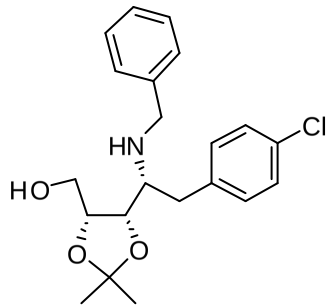


NAME	WKWD34_C13
EXPNO	1
PROCNO	1
Date_	20110513
Time	18.54
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	439
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	297.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.90 usec
PL1	-2.00 dB
PL1W	55.33689499 W
SFO1	100.6379183 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-1.00 dB
PL12	15.16 dB
PL13	18.62 dB
PL2W	13.56617069 W
PL12W	0.32844096 W
PL13W	0.14806664 W
SFO2	400.1916008 MHz
SI	32768
SF	100.6278623 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{1}H NMR



NAME	WKWD39
EXPNO	1
PROCNO	1
Date_	20100903
Time	22.58
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	40.3
DW	60.800 usec
DE	6.50 usec
TE	294.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300081 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



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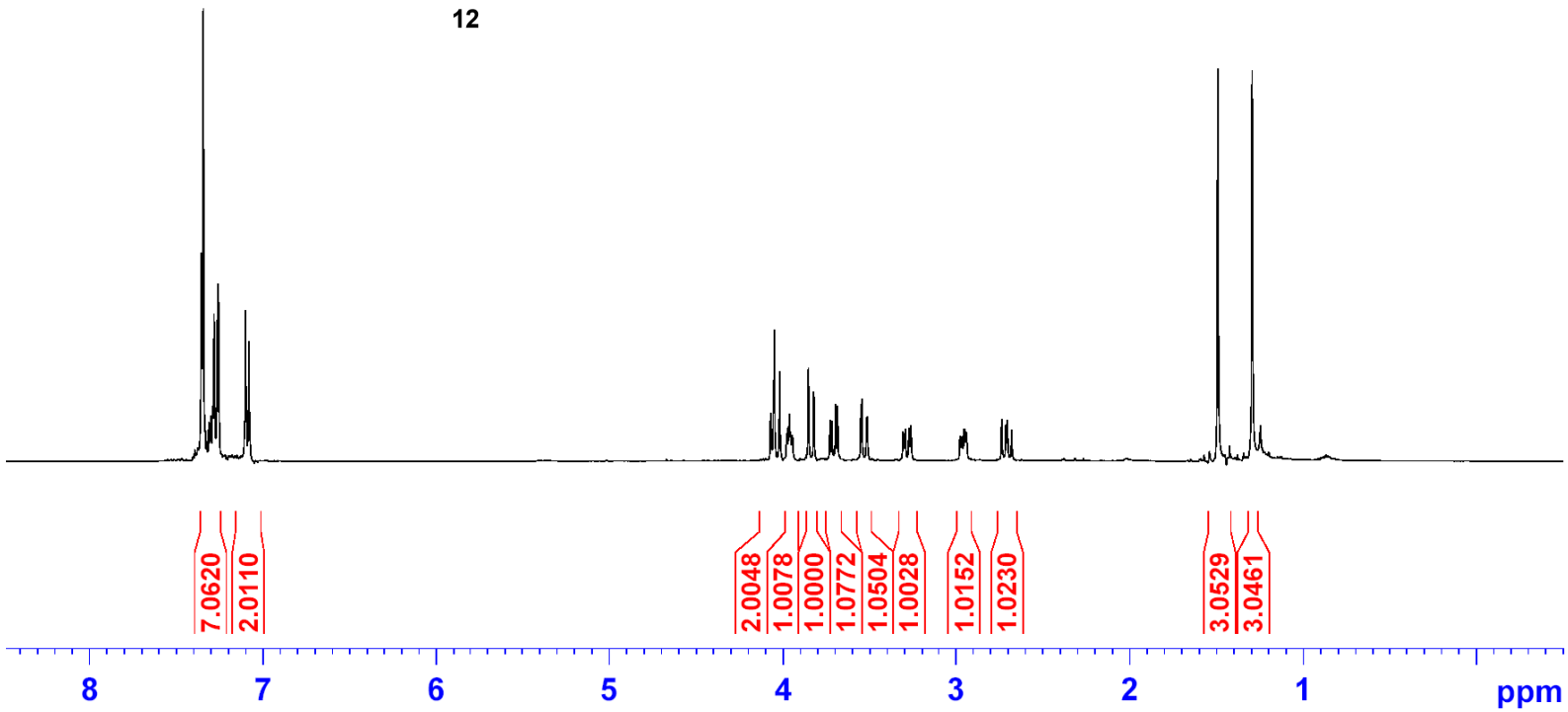
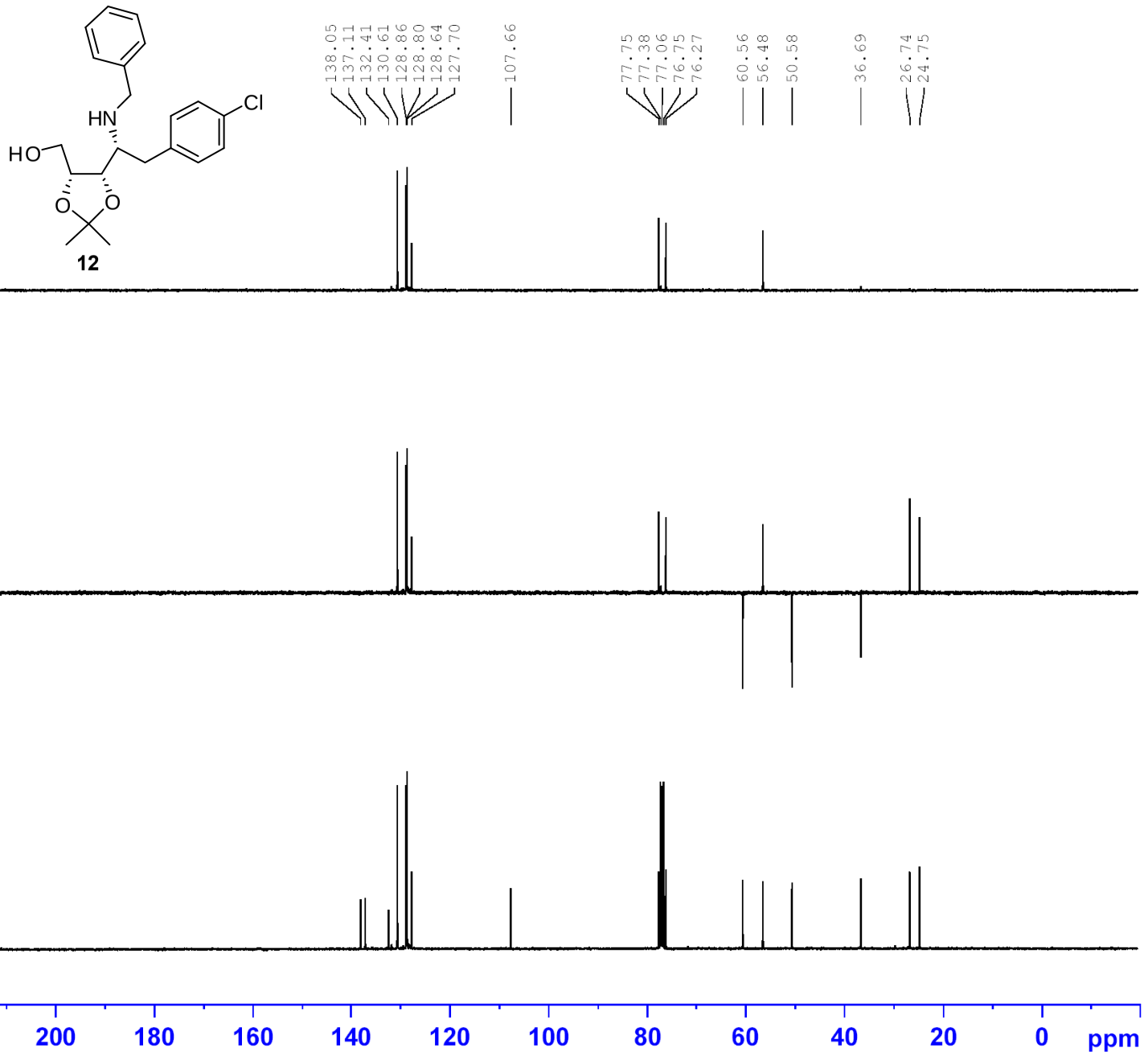
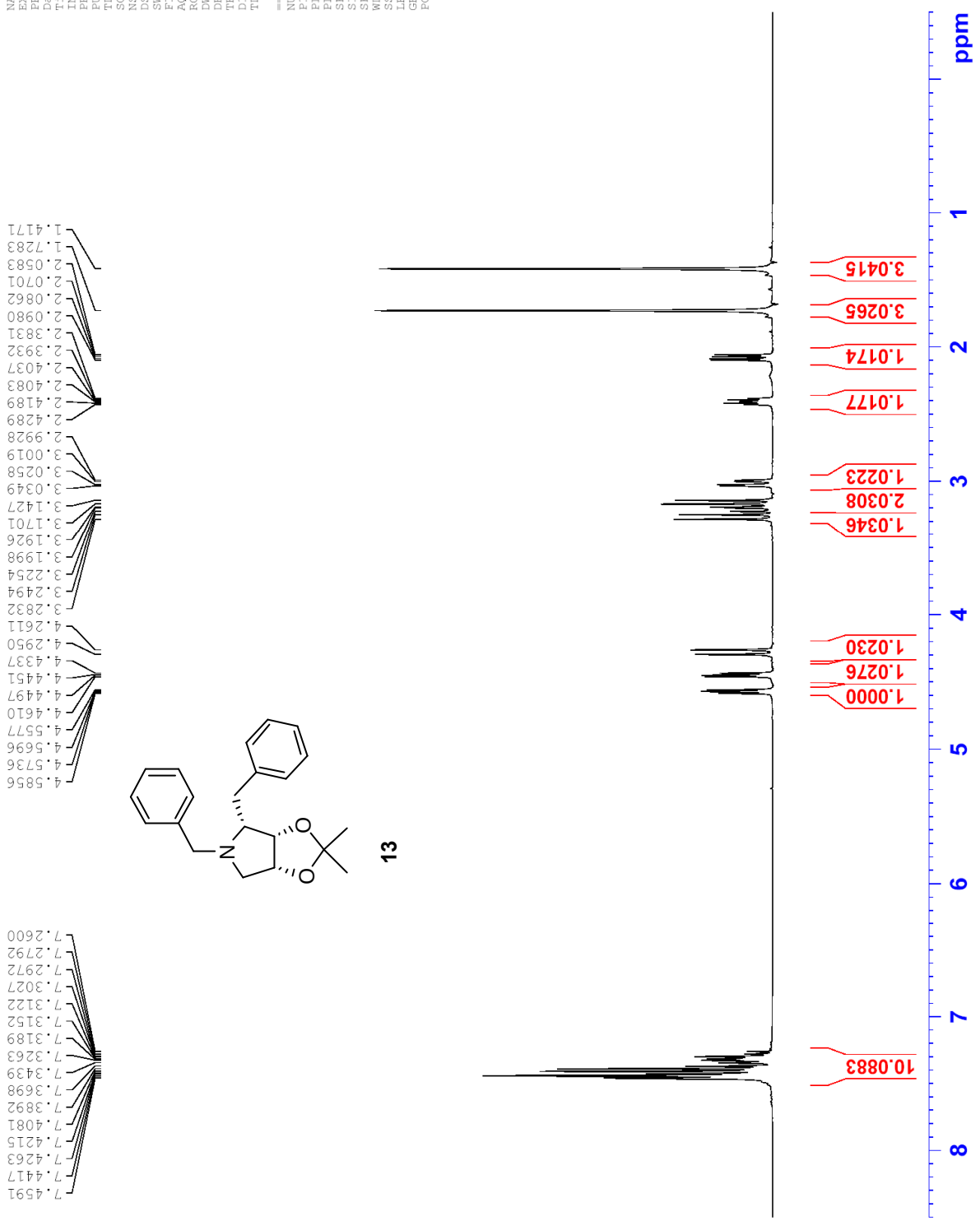


^{13}C NMR

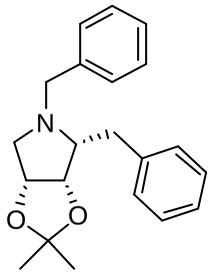



```
NAME          WKWD39_C13_1
EXPNO                    1
PROCNO                   1
Date_             20110523
Time                 11.24
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   617
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                   228
DW                20.800 usec
DE                 6.50 usec
TE                297.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.90 usec
PL1                -2.00 dB
PL1W        55.33689499 W
SFO1       100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -1.00 dB
PL12               15.16 dB
PL13               18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2       400.1916008 MHz
SI                32768
SF         100.6278564 MHz
WDW                  EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

^{1}H NMR


NAME WKWD07_1
EXPNO 1
PROCNO 1
Date_ 20100901
Time_ 19.53
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 4
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 12.7
DW 60.800 usec
DE 6.50 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.10 usec
PL1 -2.00 dB
PL1W 13.17734718 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300048 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.4591
7.4417
7.4263
7.4215
7.4081
7.3892
7.3698
7.3439
7.3263
7.3189
7.3152
7.3122
7.3027
7.2972
7.2792
7.2600
4.5856
4.5736
4.5696
4.5577
4.4610
4.4497
4.4451
4.4337
4.2950
4.2611
3.2832
3.2494
3.2254
3.1998
3.1926
3.1701
3.1427
3.0349
3.0258
3.0019
2.9928
2.4289
2.4189
2.4083
2.4037
2.3932
2.3831
2.0980
2.0862
2.0701
2.0583
1.7283
1.4171
13
10.0883
1.0000
1.0276
1.0230
1.0346
2.0308
1.0223
1.0177
1.0174
3.0265
3.0415
ppm

^{13}C NMR



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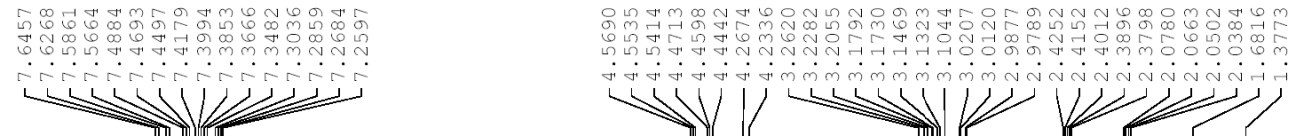
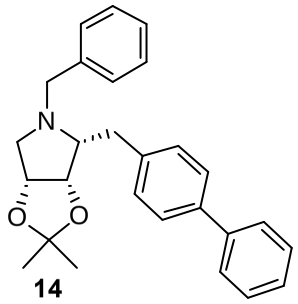
```
NAME          WKWD07_c13_1
EXPNO                    1
PROCNO                   2
Date_             20100901
Time                 20.25
INSTRUM              spect
PROBHD    5 mm PABBI 1H/
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    378
DS                      4
SWH             24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                    203
DW                20.800 usec
DE                 6.50 usec
TE                294.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 14.50 usec
PL1                -4.00 dB
PL1W         90.22689819 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               18.80 dB
PL13               18.80 dB
PL2W         13.17734718 W
PL12W         0.10960442 W
PL13W         0.10960442 W
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127852 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

^{1}H NMR

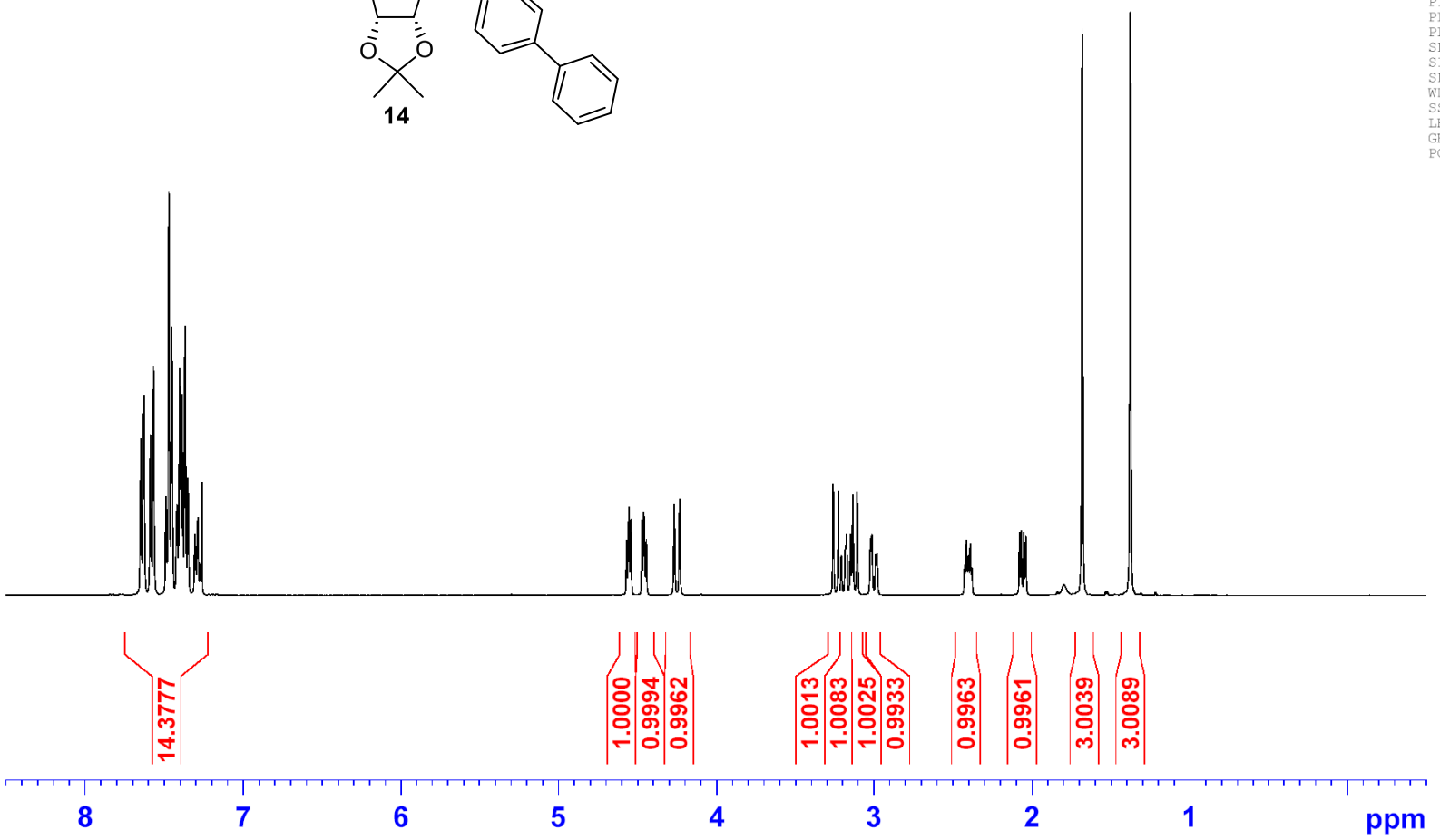




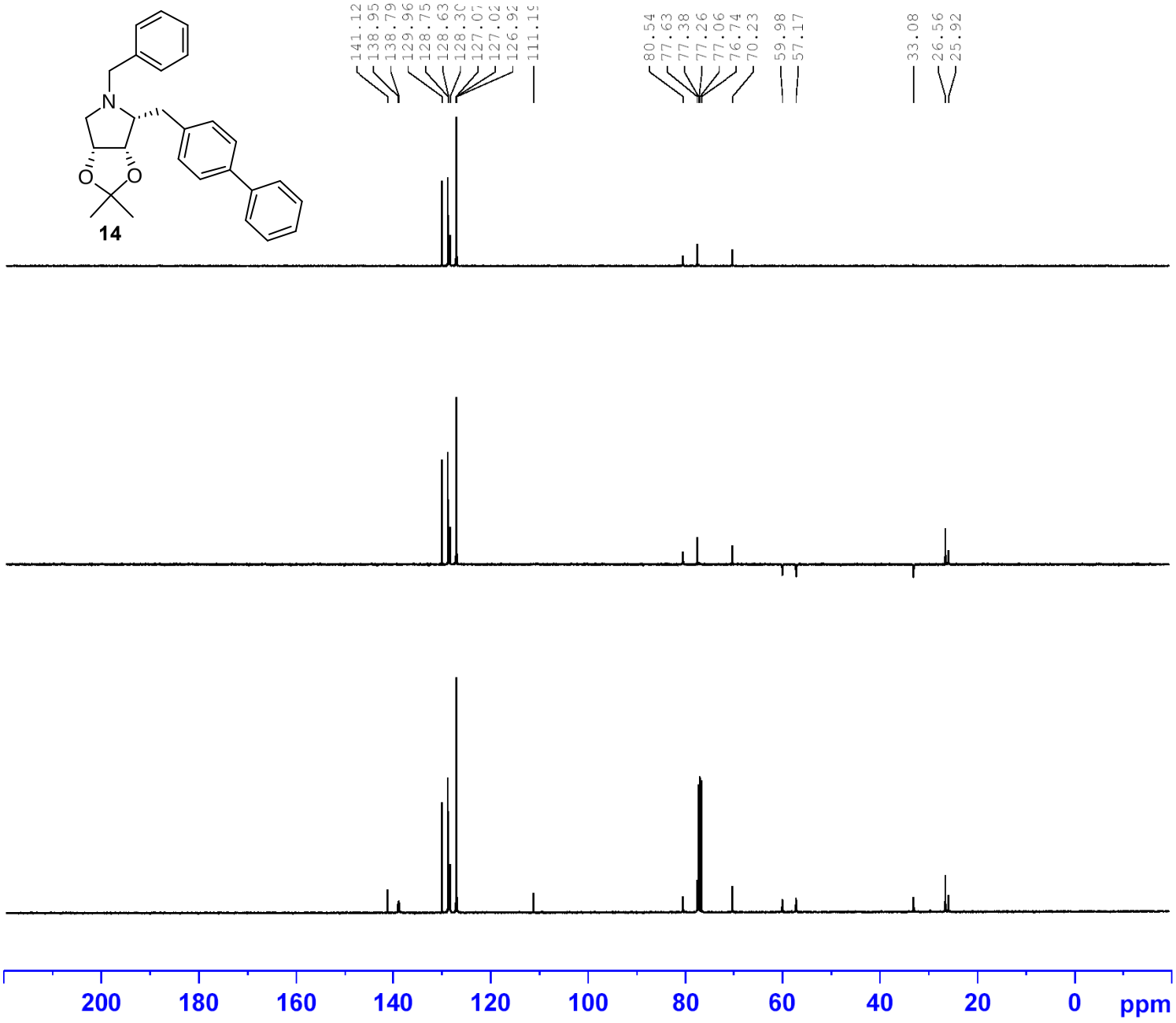
NAME	WKWD45_1
EXPNO	1
PROCNO	1
Date_	20110503
Time	20.35
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	4
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	32
DW	60.800 usec
DE	6.50 usec
TE	296.8 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
P1	14.00 usec
PL1	-1.00 dB
PL1W	13.56617069 W
SFO1	400.1924713 MHz
SI	32768
SF	400.1900138 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

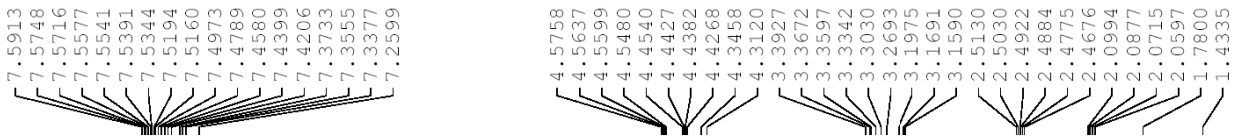


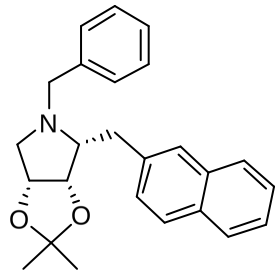
^{13}C NMR



NAME	WKWD45_c13
EXPNO	1
PROCNO	1
Date_	20110212
Time	11.56
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	627
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	181
DW	20.800 usec
DE	6.50 usec
TE	298.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.90 usec
PL1	-2.00 dB
PL1W	55.33689499 W
SFO1	100.6379183 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-1.00 dB
PL12	15.16 dB
PL13	18.62 dB
PL2W	13.56617069 W
PL12W	0.32844096 W
PL13W	0.14806664 W
SFO2	400.1916008 MHz
SI	32768
SF	100.6278571 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{1}H NMR





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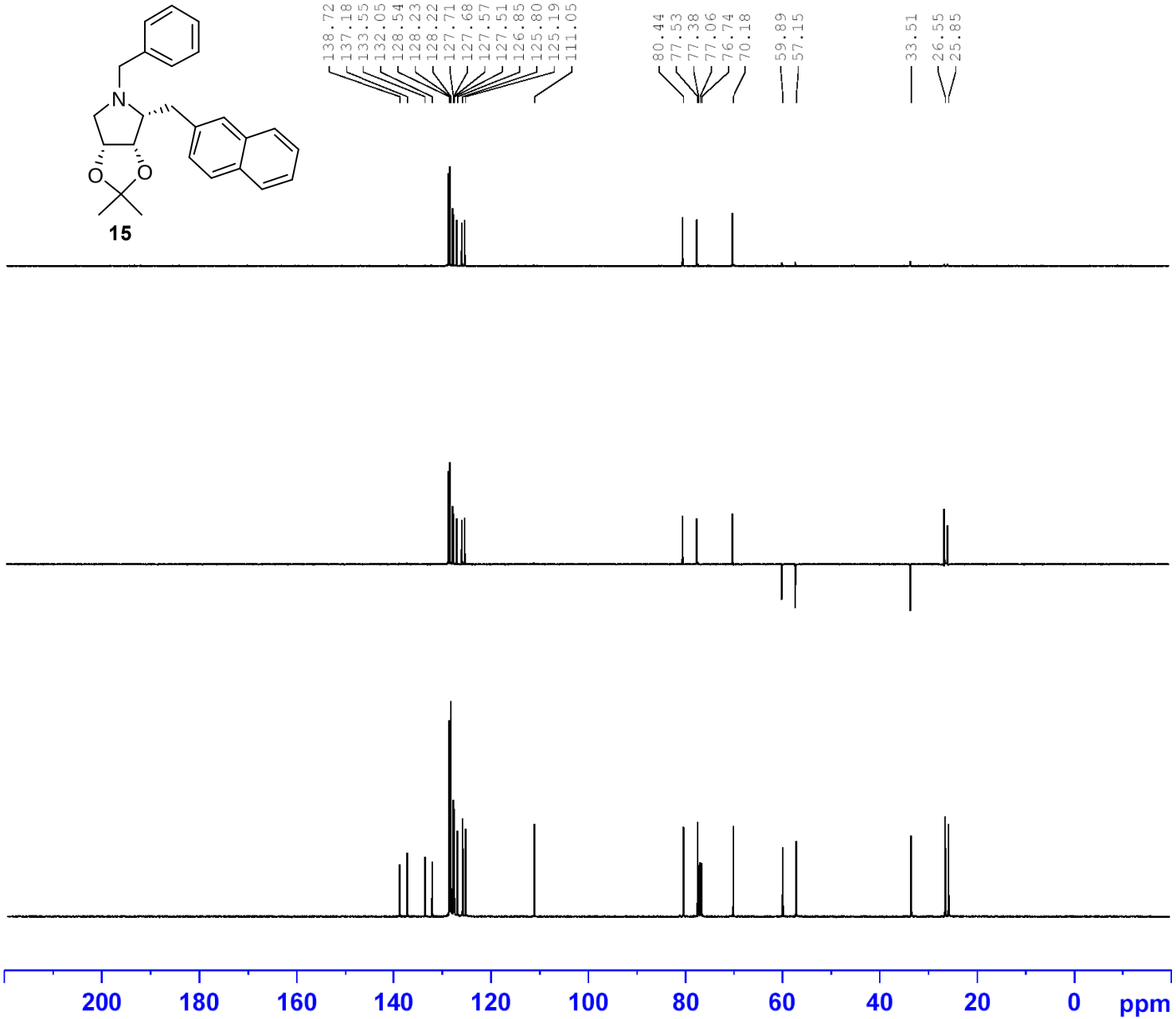
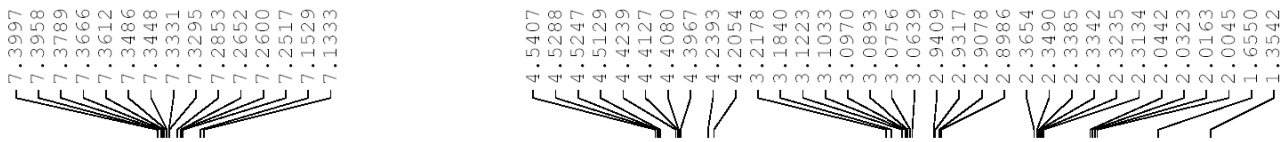
NAME	WKWD42
EXPNO	1
PROCNO	1
Date_	20100916
Time	13.43
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	4
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	14.2
DW	60.800 usec
DE	6.50 usec
TE	294.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300048 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

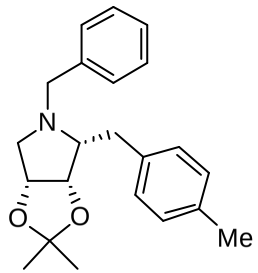
^{13}C NMR



NAME	WKWD42_c13
EXPNO	1
PROCNO	1
Date_	20100916
Time	14.22
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	CDC13
NS	442
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	294.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	14.50 usec
PL1	-4.00 dB
PL1W	90.22689819 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	18.80 dB
PL13	18.80 dB
PL2W	13.17734718 W
PL12W	0.10960442 W
PL13W	0.10960442 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127859 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{1}H NMR





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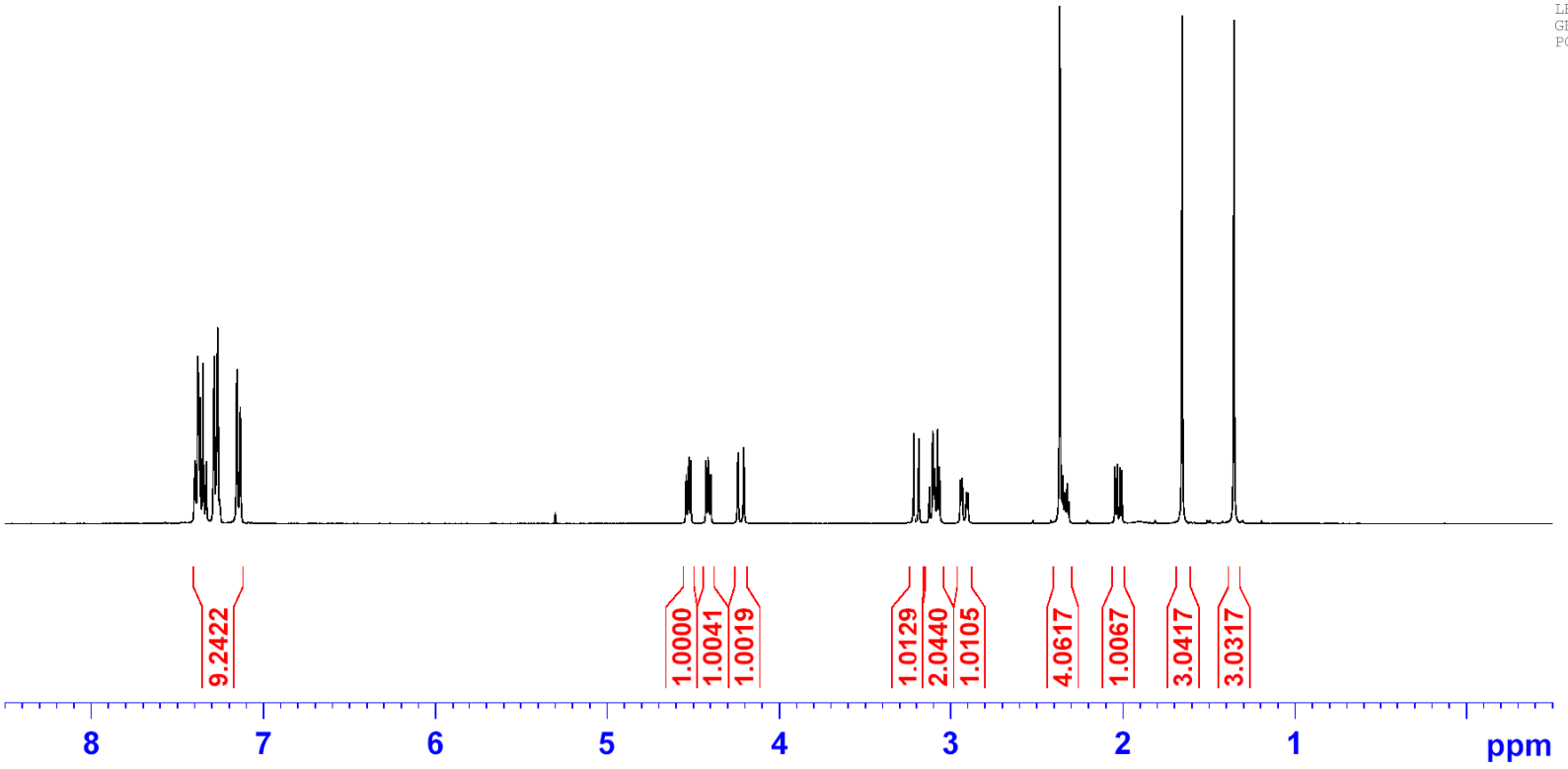
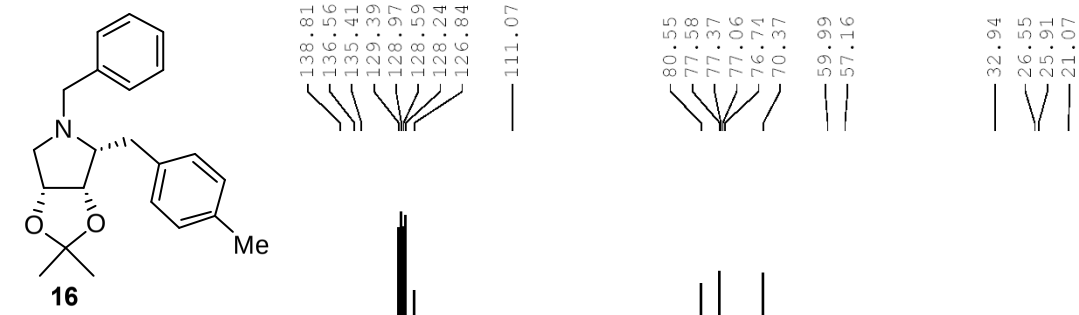
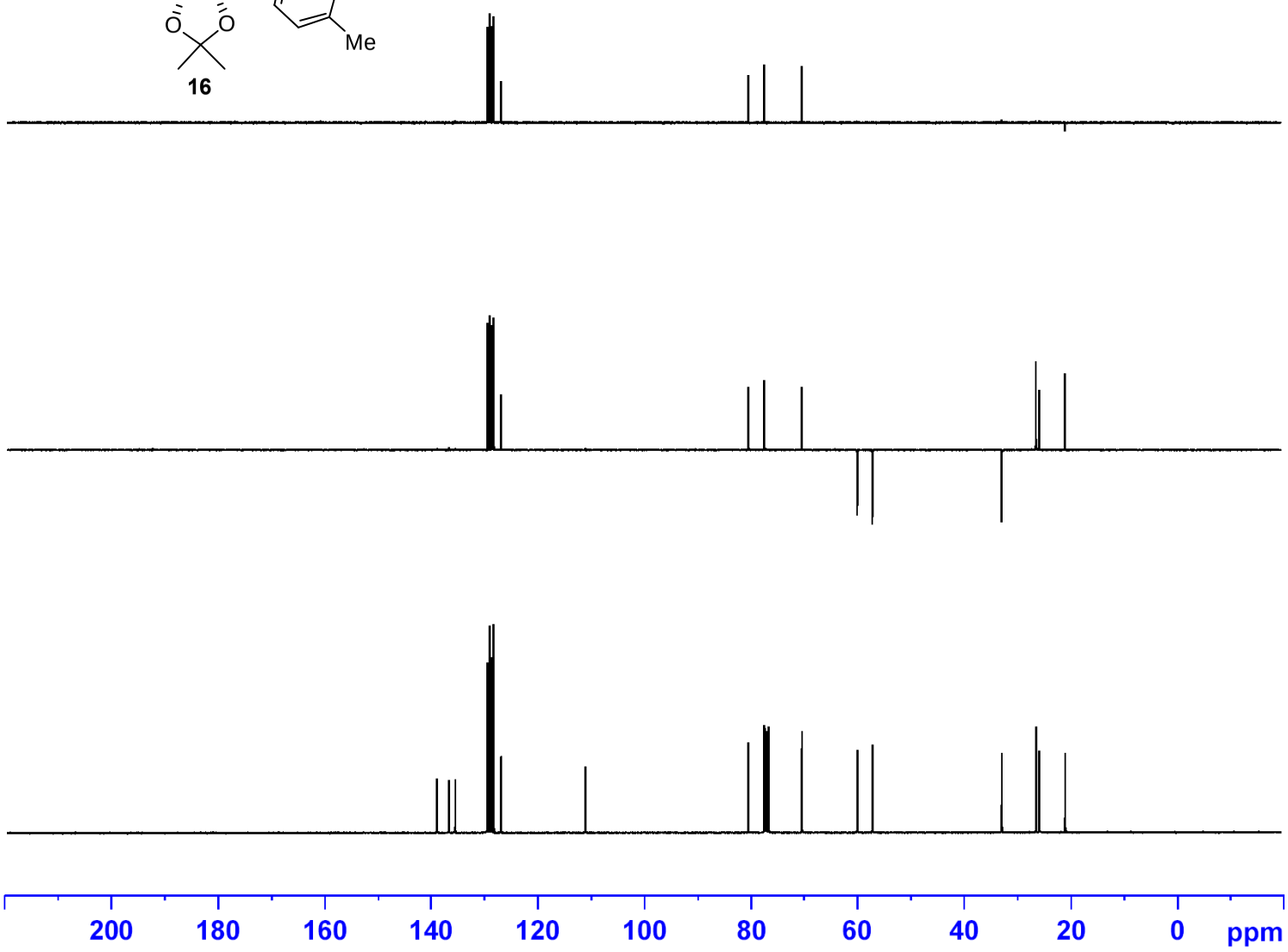
```
NAME              WKWD33
EXPNO                  1
PROCNO                 1
Date_           20100831
Time               10.44
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     4
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                  28.5
DW                60.800 usec
DE                 6.50 usec
TE                297.8 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                 32768
SF          400.1900138 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```



^{13}C NMR

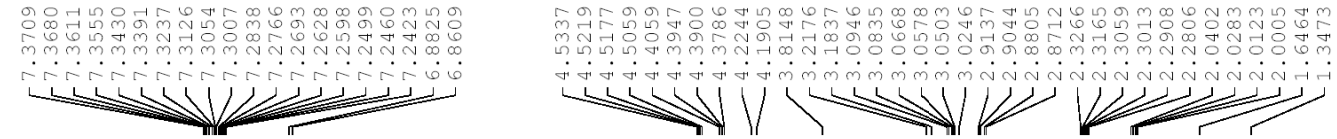


16

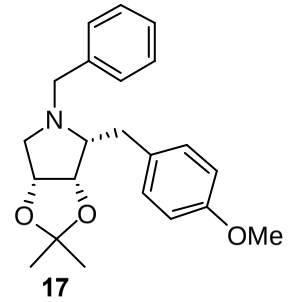



```
NAME          WKWD33_c13
EXPNO                  1
PROCNO                 1
Date_           20100831
Time               10.59
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  546
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE                298.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.90 usec
PL1               -2.00 dB
PL1W        55.33689499 W
SFO1       100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -1.00 dB
PL12              15.16 dB
PL13              18.62 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
PL13W        0.14806664 W
SFO2       400.1916008 MHz
SI                32768
SF         100.6278601 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

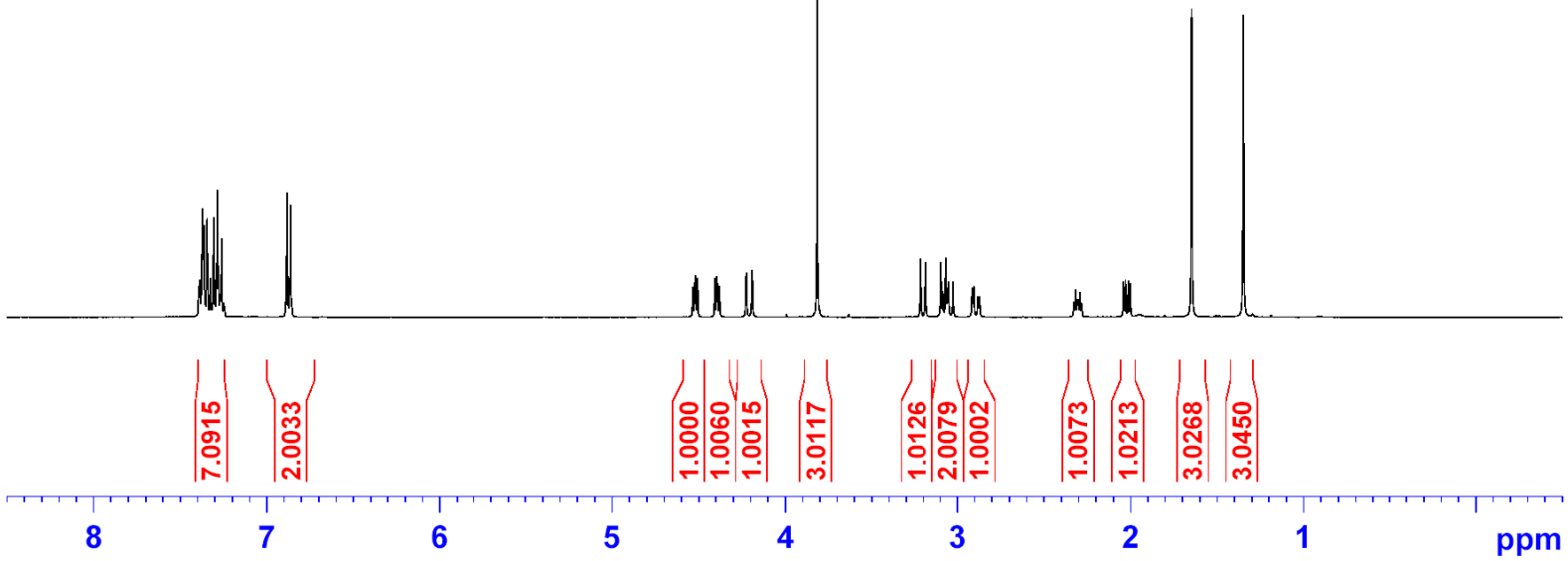
^{1}H NMR



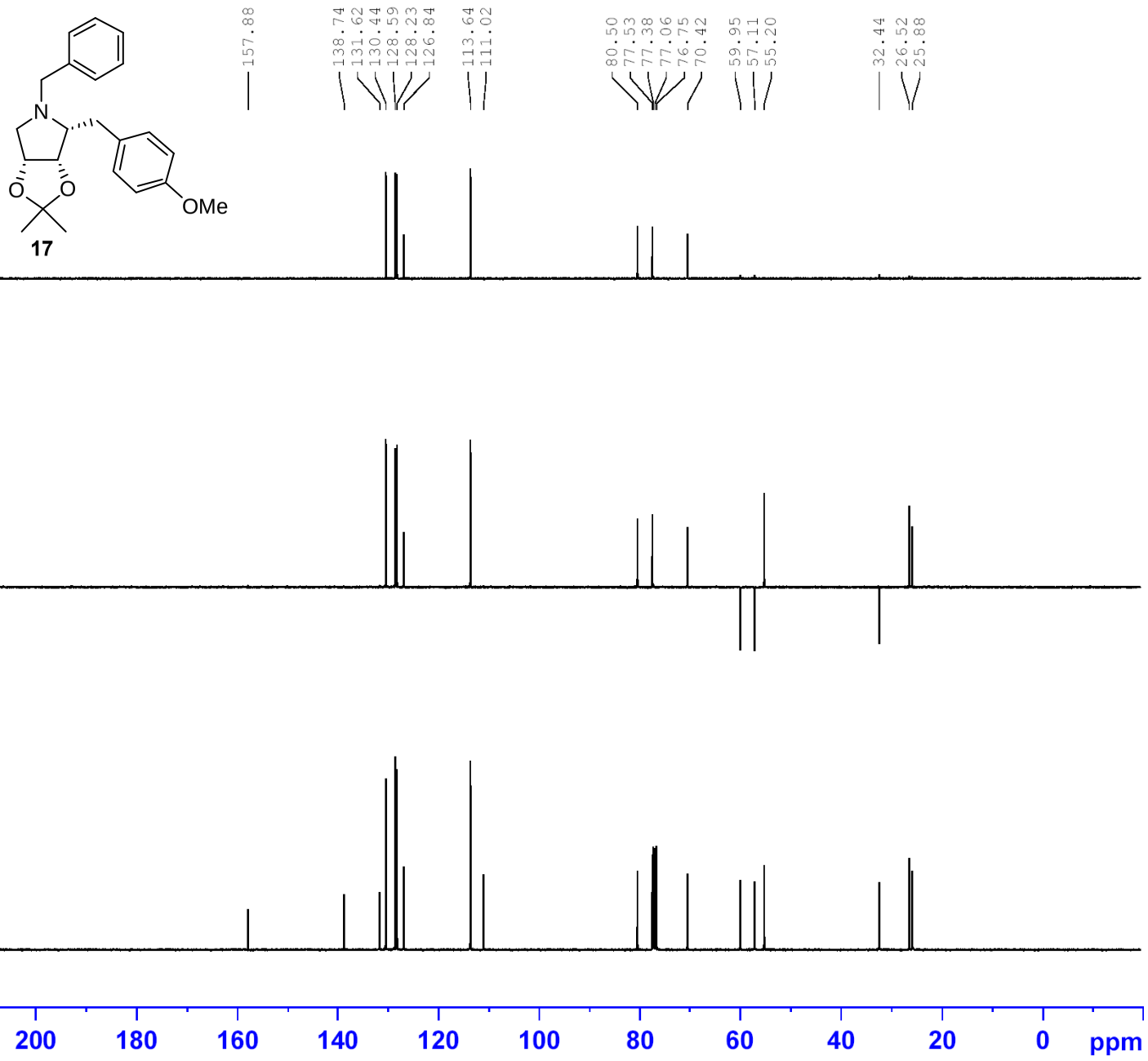

```
NAME            WKWD35_1
EXPNO                  1
PROCNO                 1
Date_           20110514
Time               17.27
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     4
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                    36
DW                60.800 usec
DE                 6.50 usec
TE                294.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.83 usec
PL1                0.00 dB
PL1W          8.31434441 W
SFO1        400.1324710 MHz
SI                 32768
SF          400.1300096 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```



17

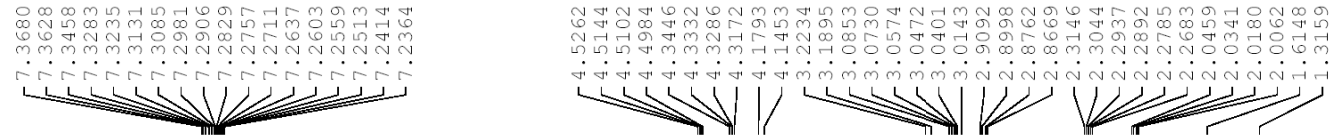


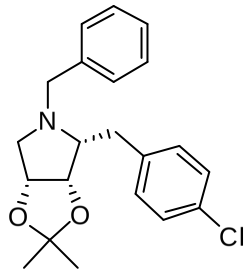
^{13}C NMR



NAME	WKWD35_c13
EXPNO	1
PROCNO	2
Date_	20110514
Time	17.42
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	415
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	294.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.66 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.22585411 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127735 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

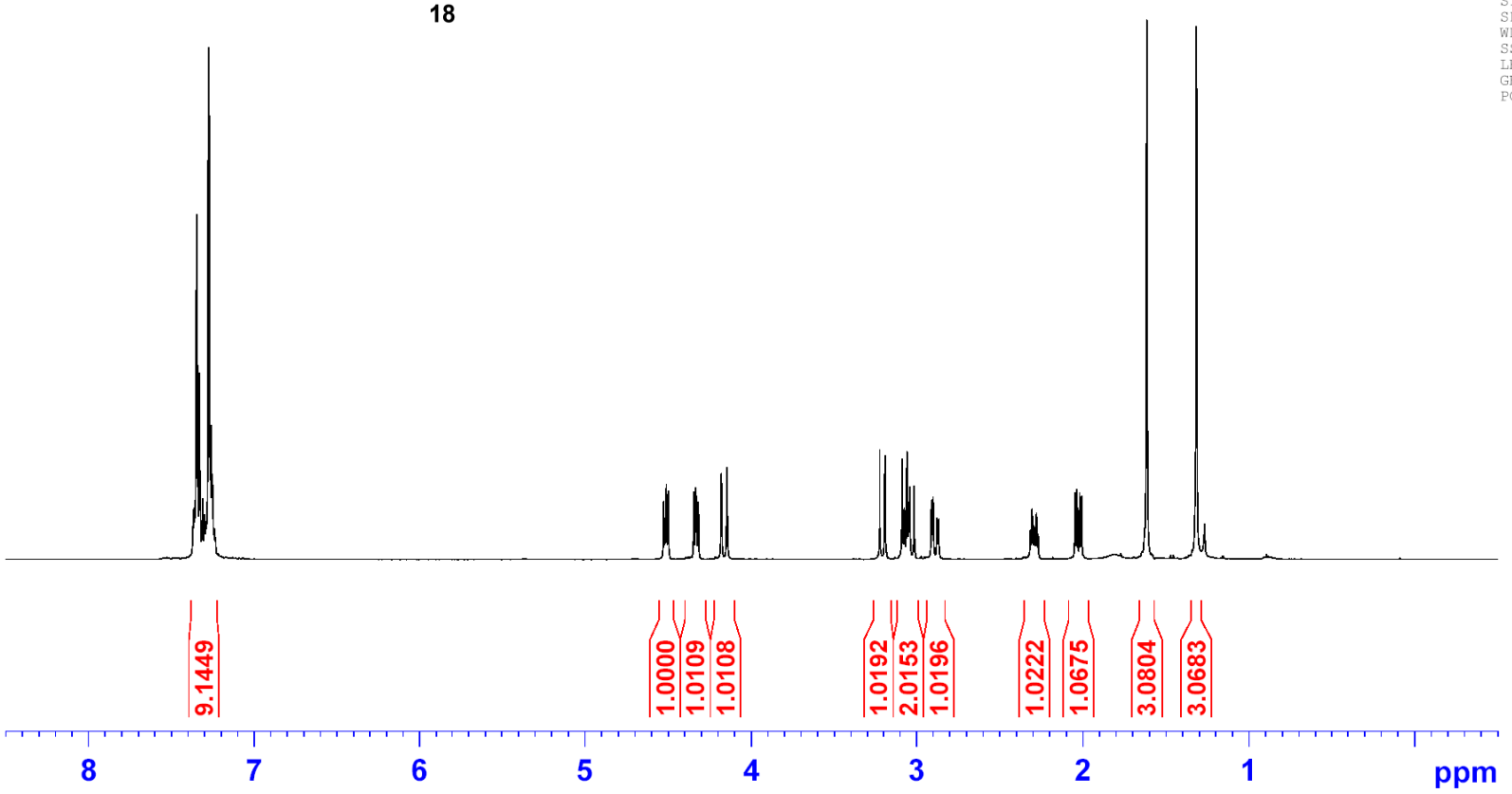
^{1}H NMR



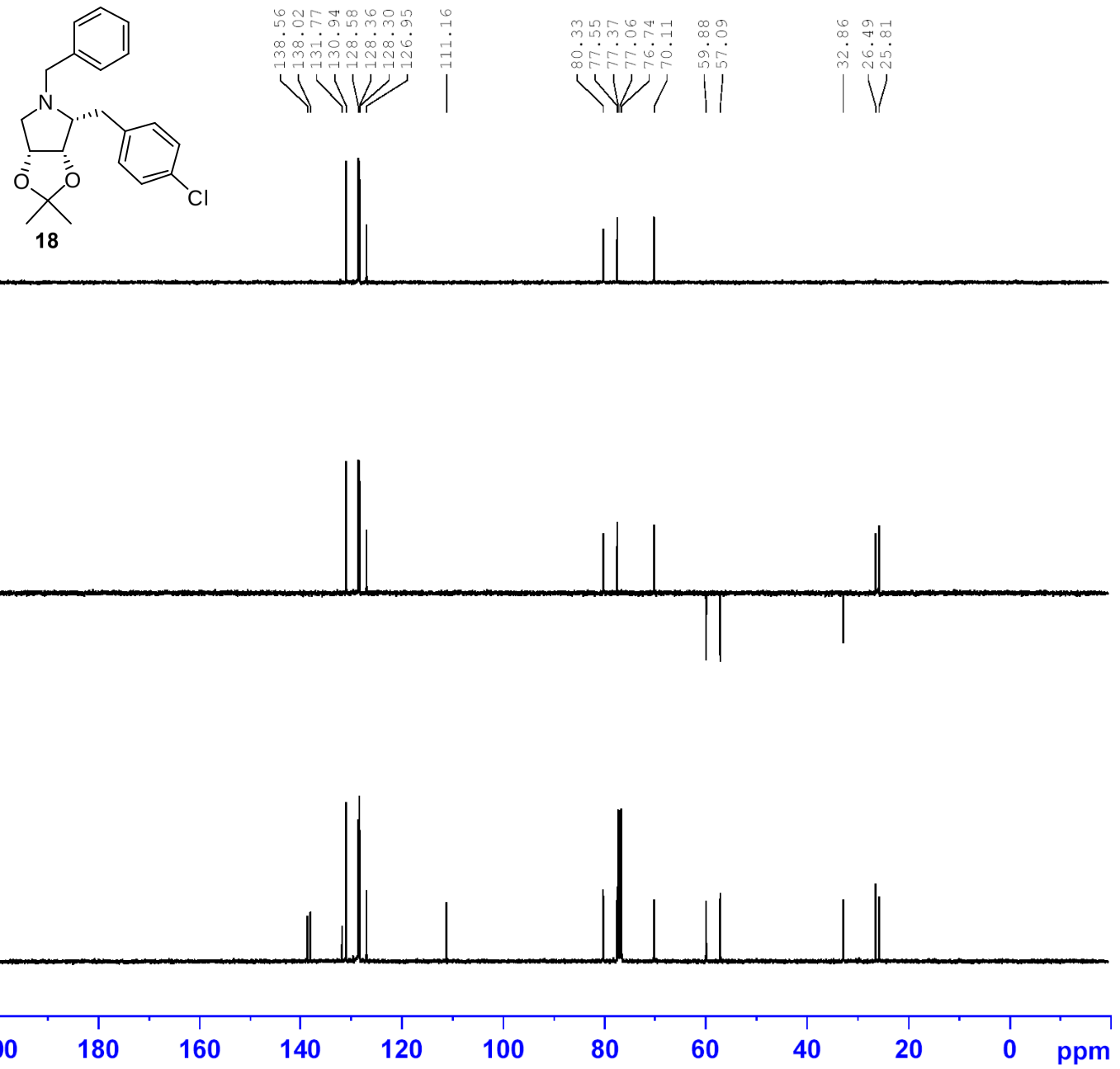


18

```
NAME                WKWD40
EXPNO                    1
PROCNO                   1
Date_             20100908
Time                 19.36
INSTRUM              spect
PROBHD   5 mm PABBI 1H/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                     40.3
DW                   60.800 usec
DE                    6.50 usec
TE                   294.3 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    7.10 usec
PL1                  -2.00 dB
PL1W          13.17734718 W
SFO1          400.1324710 MHz
SI                    32768
SF            400.1300051 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00
```

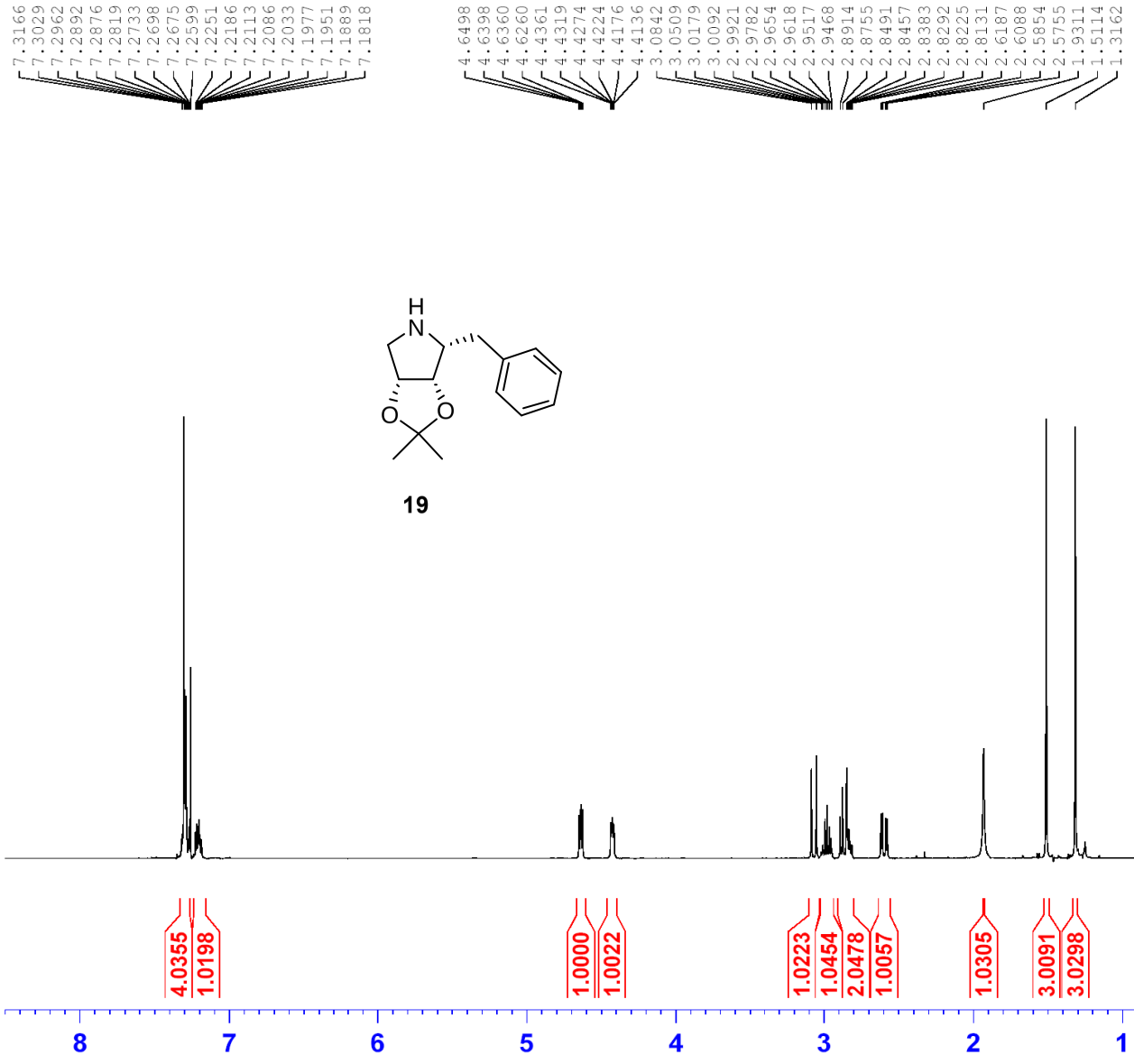


^{13}C NMR



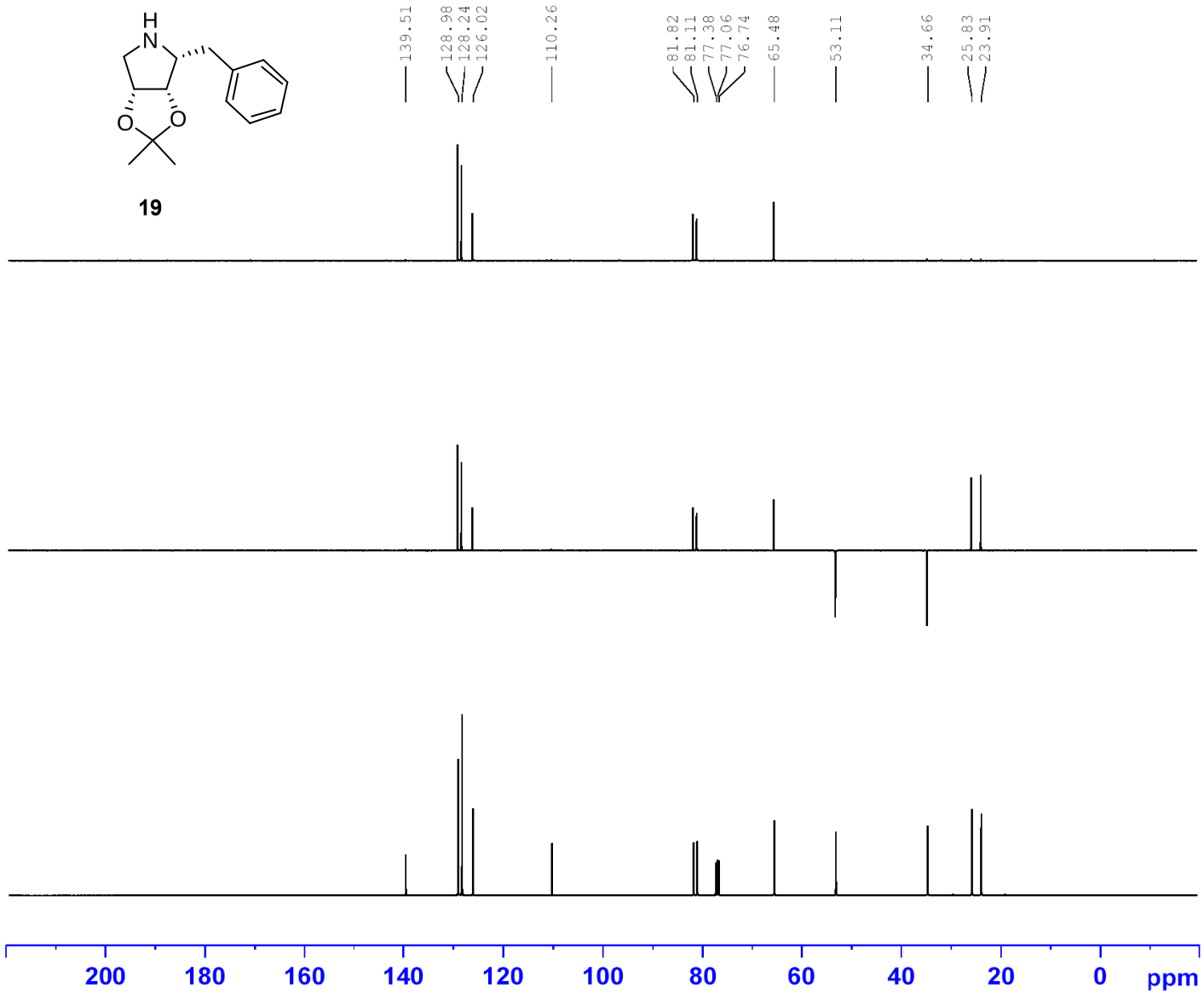

```
NAME          WKWD40_c13
EXPNO                  1
PROCNO                 1
Date_           20100908
Time               19.55
INSTRUM            spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  429
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE               294.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                14.50 usec
PL1               -4.00 dB
PL1W        90.22689819 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              18.80 dB
PL13              18.80 dB
PL2W        13.17734718 W
PL12W        0.10960442 W
PL13W        0.10960442 W
SFO2       400.1316005 MHz
SI                32768
SF         100.6127676 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

^{1}H NMR



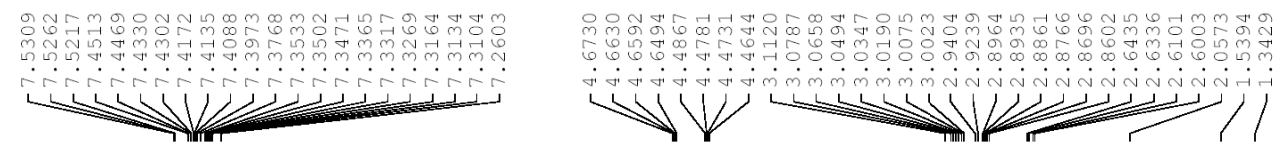
NAME	WKWD08
EXPNO	1
PROCNO	1
Date_	20100505
Time	12.03
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	128
DW	60.800 usec
DE	6.50 usec
TE	294.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300056 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

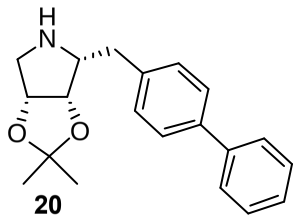
^{13}C NMR




```
NAME          WKWD07_c13_1
EXPNO                    1
PROCNO                   1
Date_             20100906
Time                 23.05
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    9892
DS                       4
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                    90.5
DW                  20.800 usec
DE                   6.50 usec
TE                  298.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.90 usec
PL1                  -2.00 dB
PL1W          55.33689499 W
SFO1         100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                  -1.00 dB
PL12                 15.16 dB
PL13                 18.62 dB
PL2W          13.56617069 W
PL12W          0.32844096 W
PL13W          0.14806664 W
SFO2         400.1916008 MHz
SI                   32768
SF           100.6278682 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

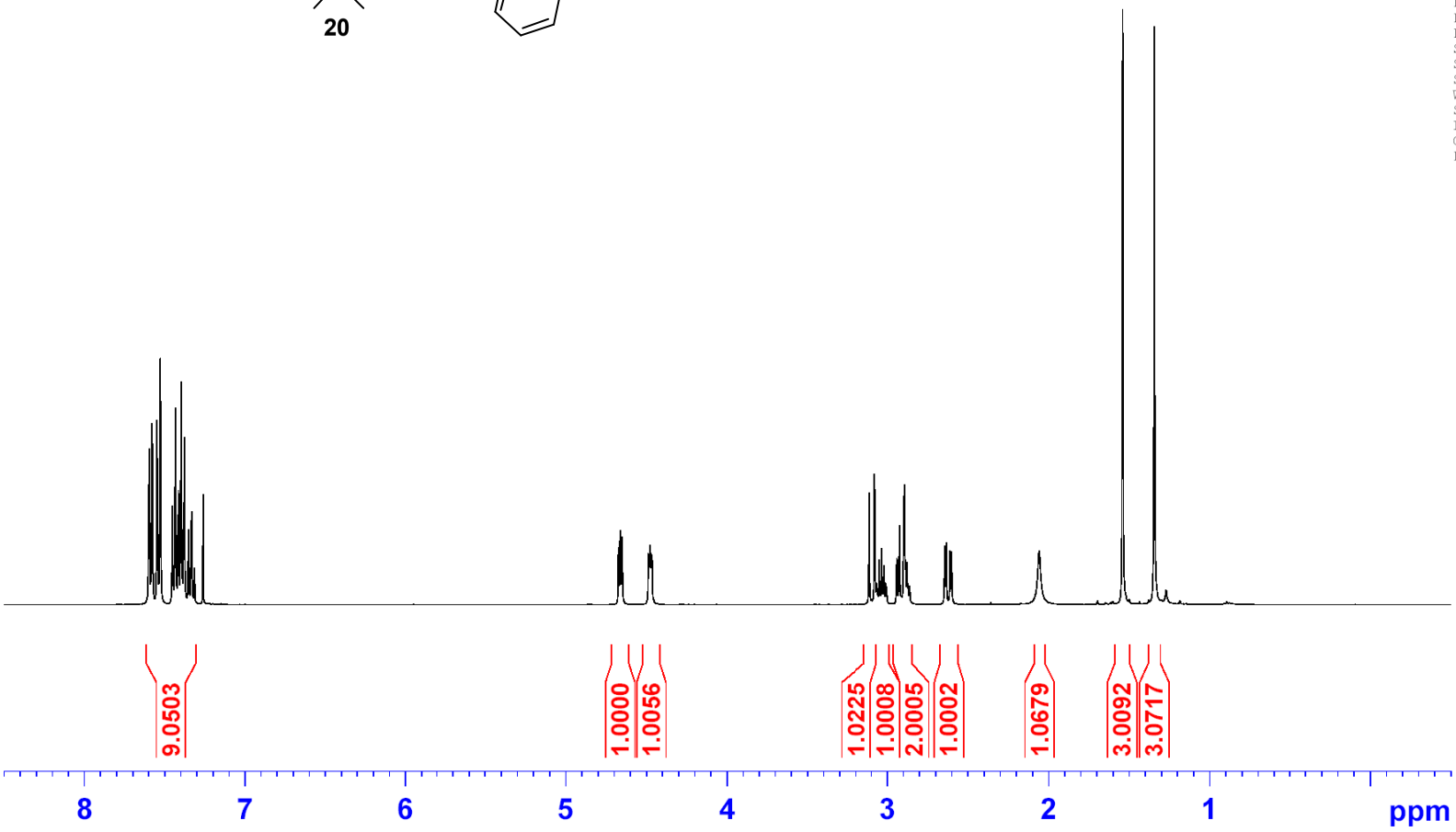
^{1}H NMR



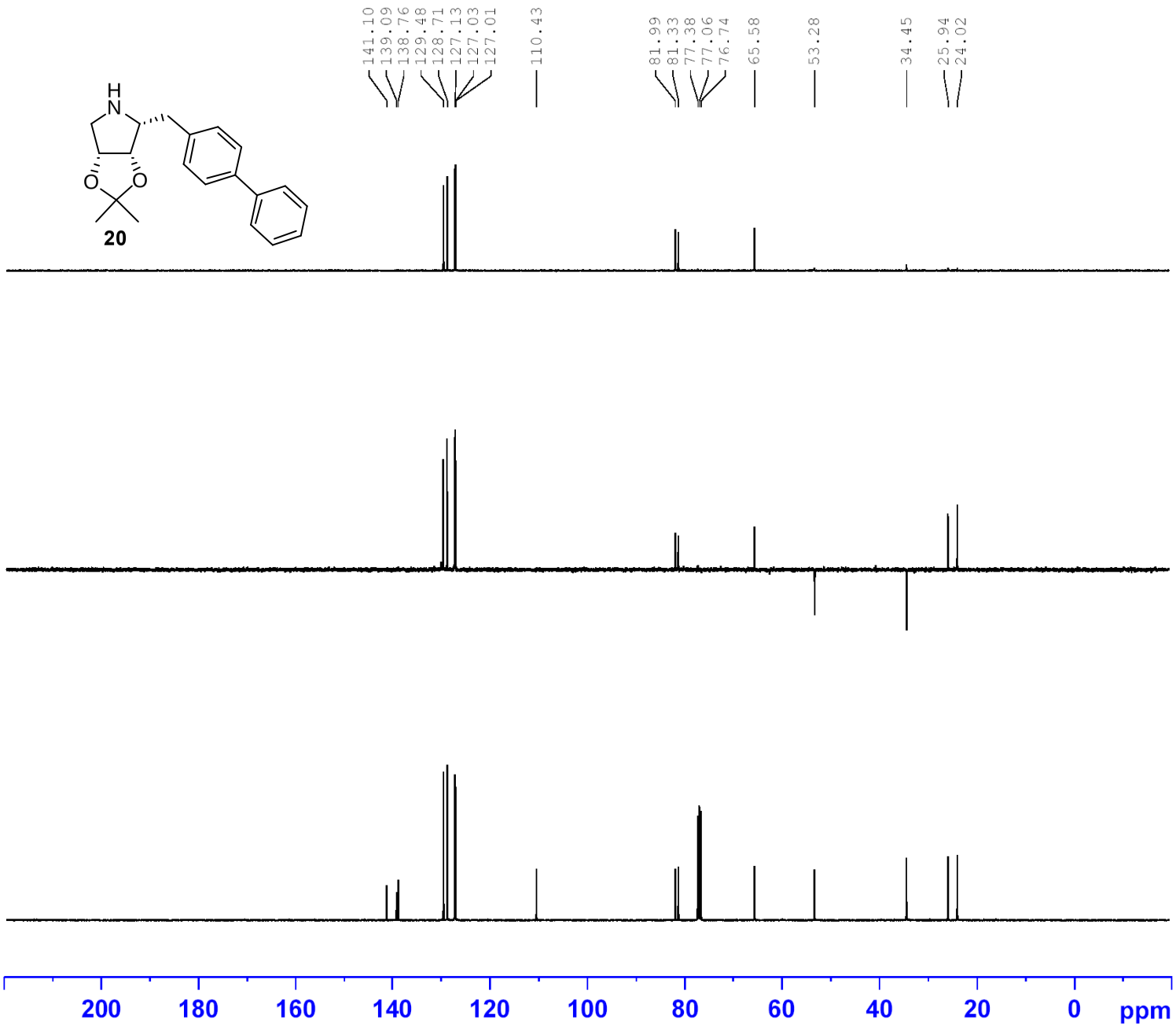


20

NAME	WKWD46_2
EXPNO	1
PROCNO	1
Date_	20110507
Time	17.12
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	4
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	80.6
DW	60.800 usec
DE	6.50 usec
TE	294.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300096 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

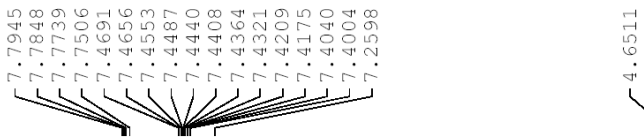


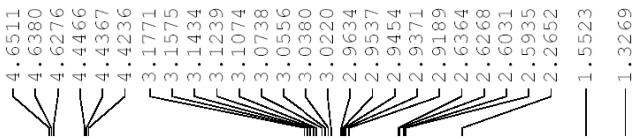
^{13}C NMR

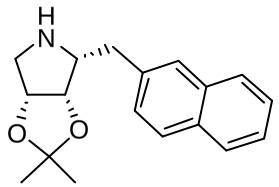


NAME	WKWD46_c13_2
EXPNO	1
PROCNO	1
Date_	20110507
Time	17.22
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	676
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	294.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.66 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.22585411 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127712 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{1}H NMR

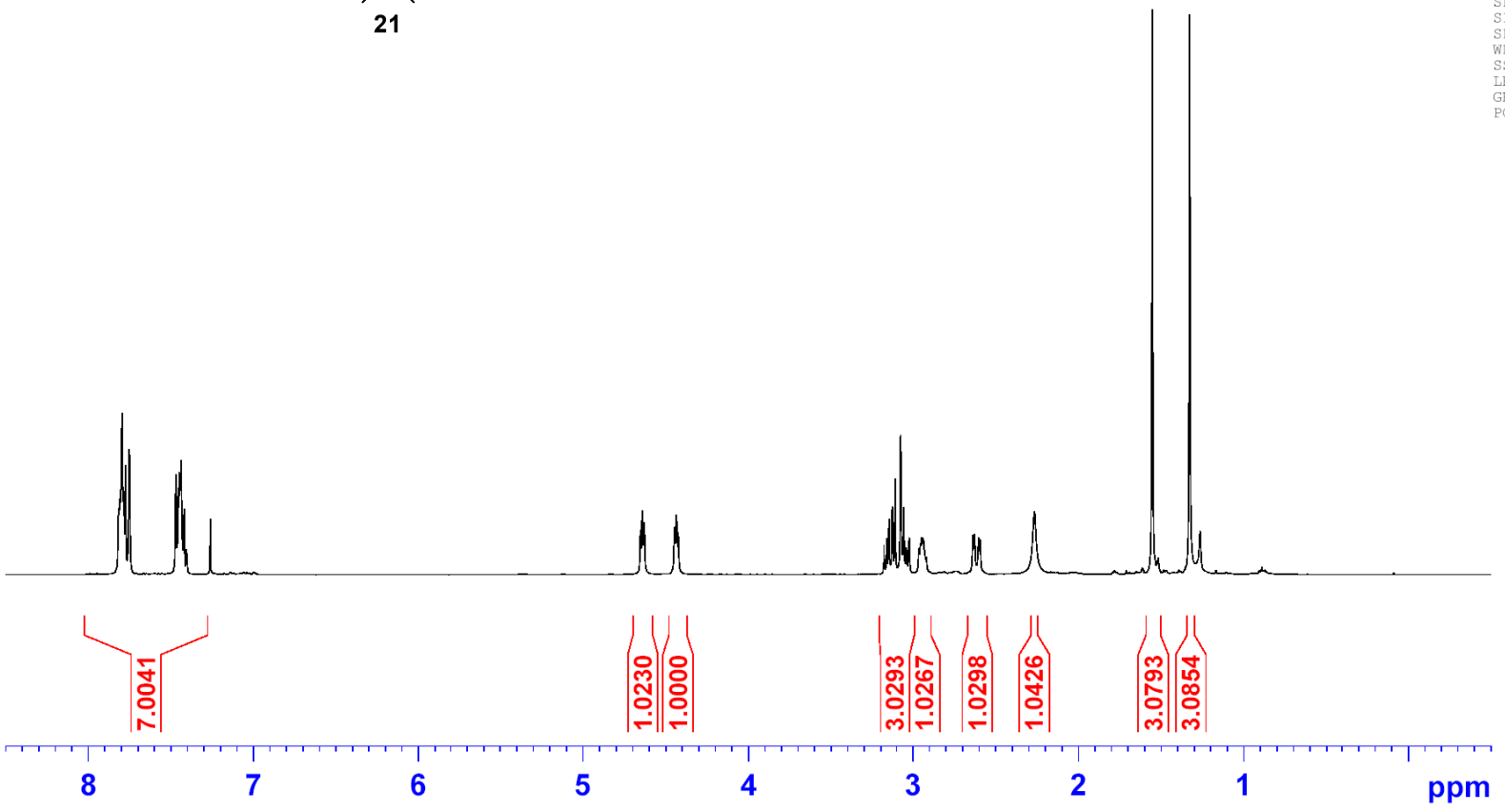




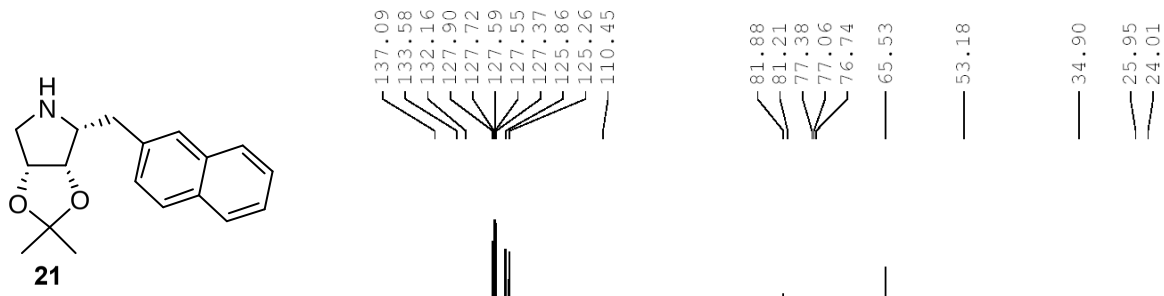


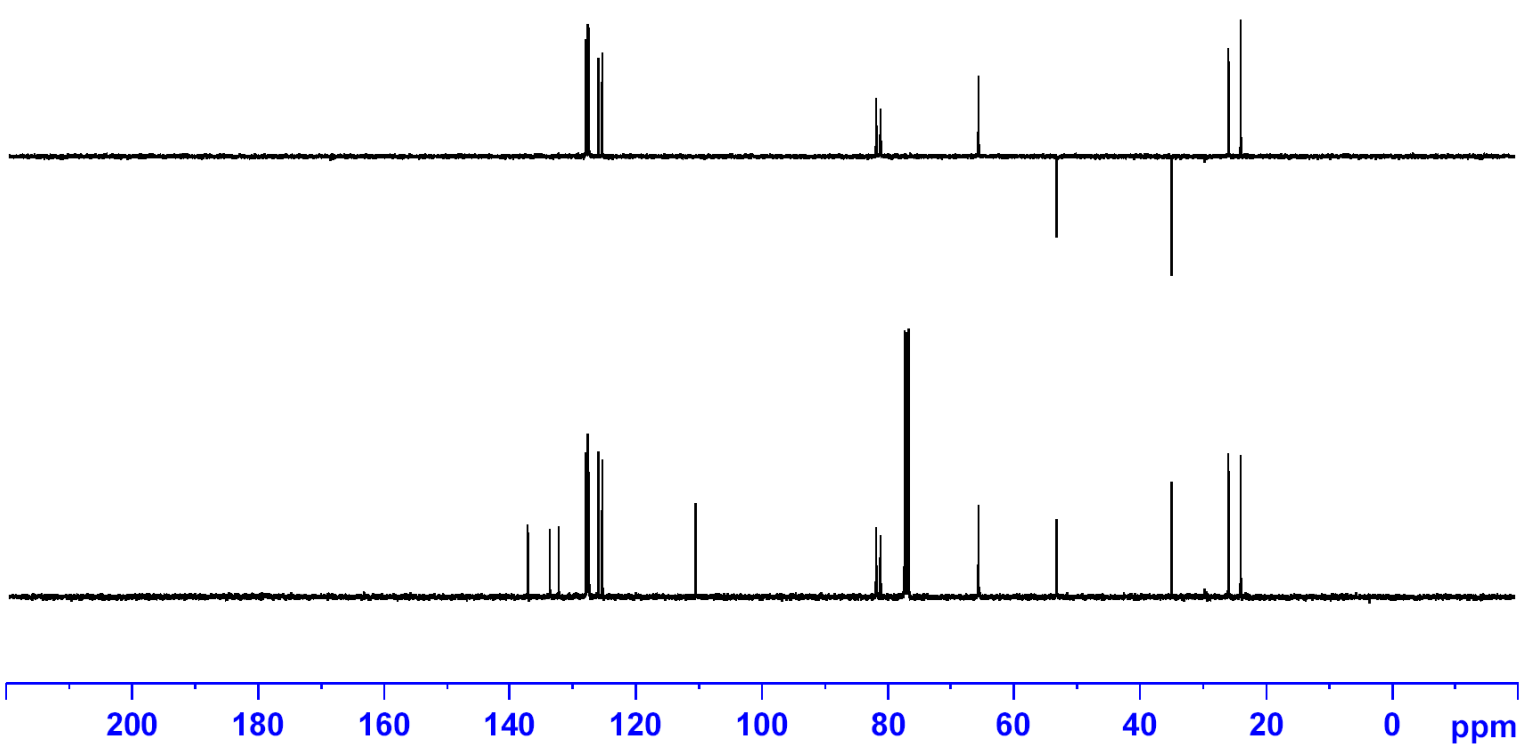
21

NAME	WKWD43
EXPNO	1
PROCNO	1
Date_	20110427
Time	21.47
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	4
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	80.6
DW	60.800 usec
DE	6.50 usec
TE	293.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300098 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



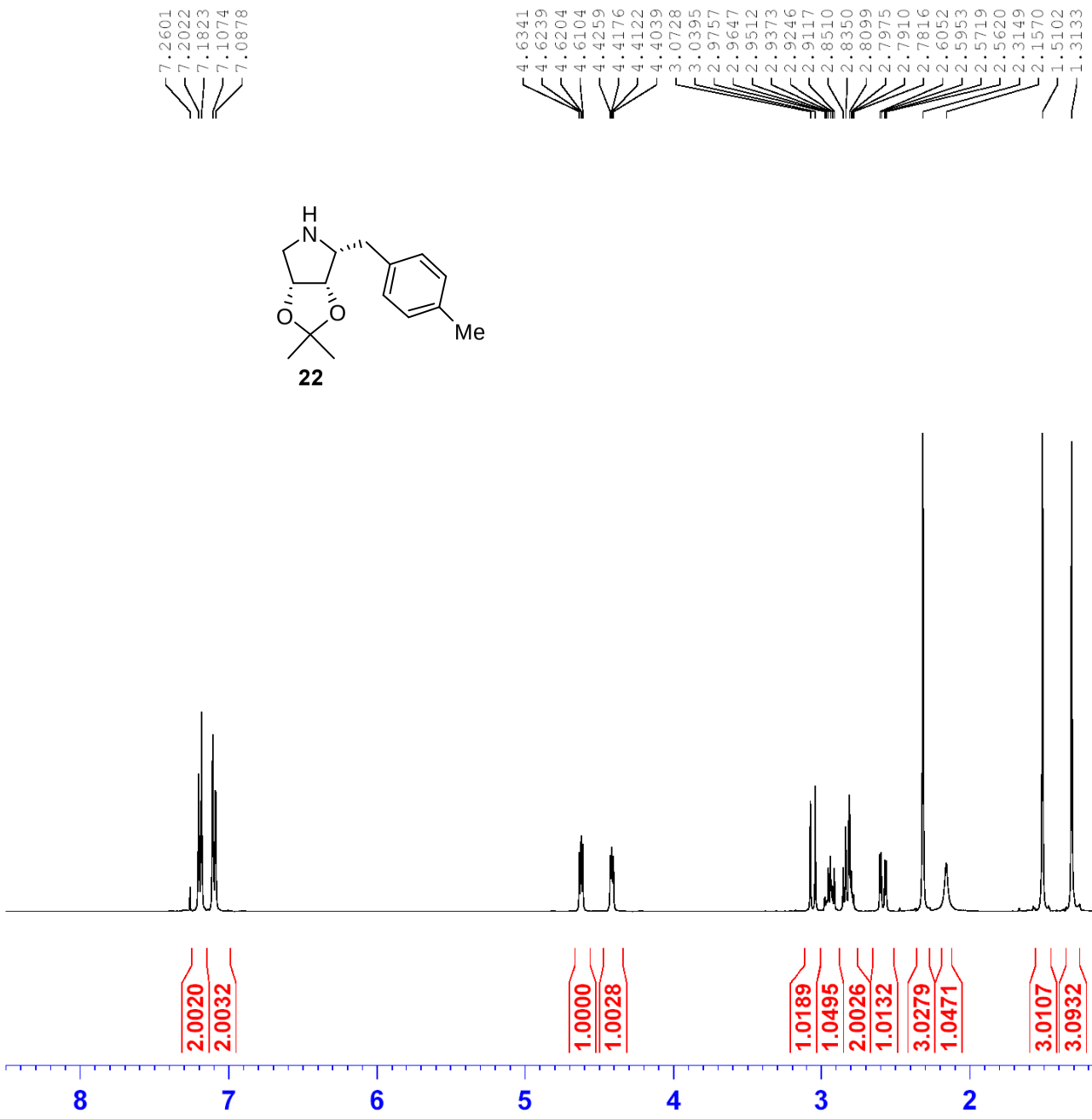
^{13}C NMR





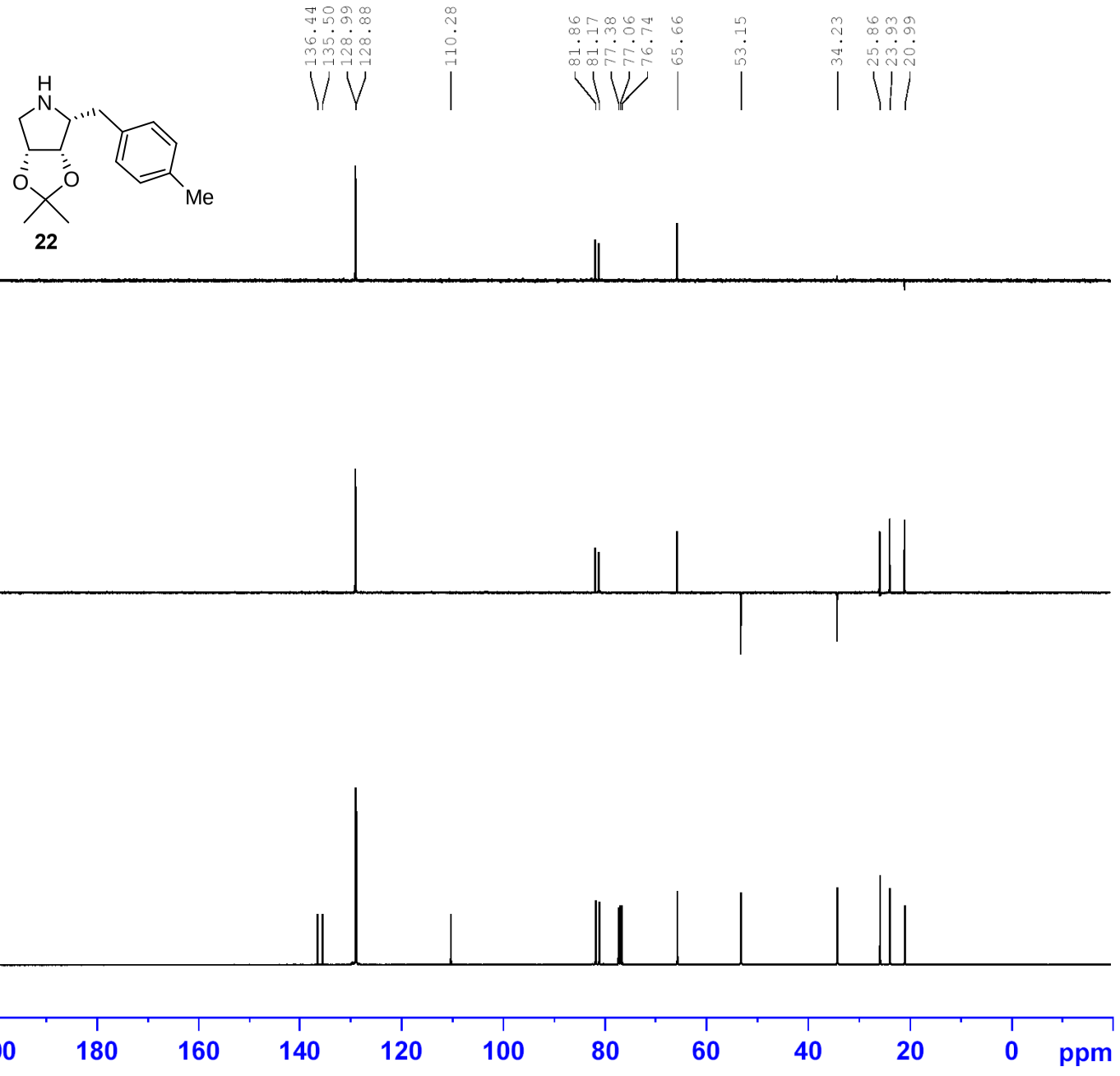
NAME	WKWD43_c13
EXPNO	1
PROCNO	1
Date_	20110427
Time	21.54
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	203
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	294.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.66 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.22585411 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127712 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{1}H NMR



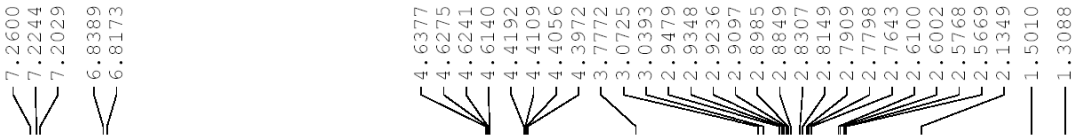
NAME	WKWD37_3
EXPNO	1
PROCNO	1
Date_	20100903
Time	23.18
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	4
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	20.2
DW	60.800 usec
DE	6.50 usec
TE	294.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300051 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

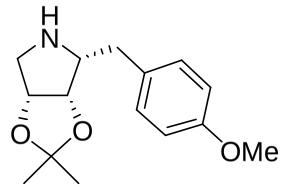
^{13}C NMR



NAME	WKWD37_c13_1
EXPNO	1
PROCNO	1
Date_	20100903
Time	23.32
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	10782
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	294.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	14.50 usec
PL1	-4.00 dB
PL1W	90.22689819 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	18.80 dB
PL13	18.80 dB
PL2W	13.17734718 W
PL12W	0.10960442 W
PL13W	0.10960442 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127756 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{1}H NMR





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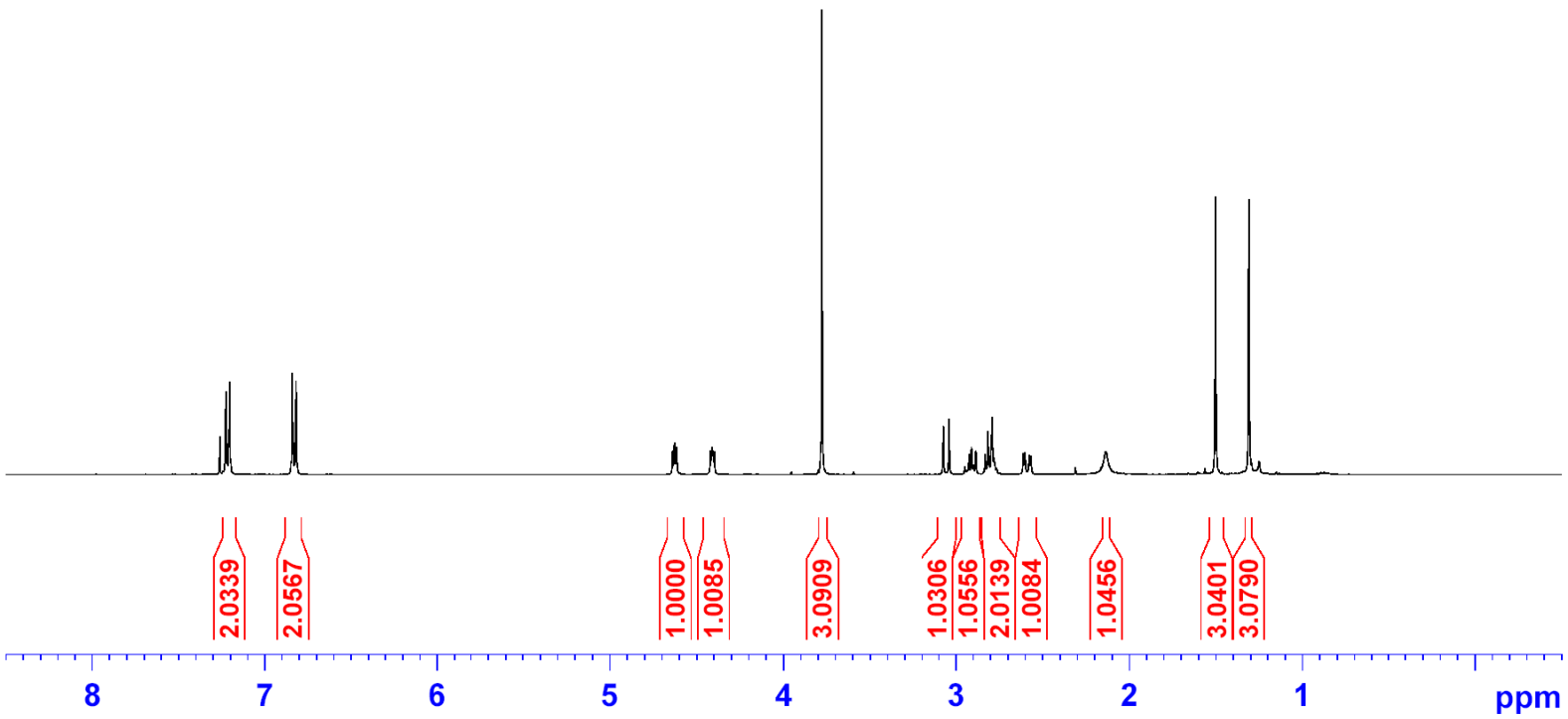
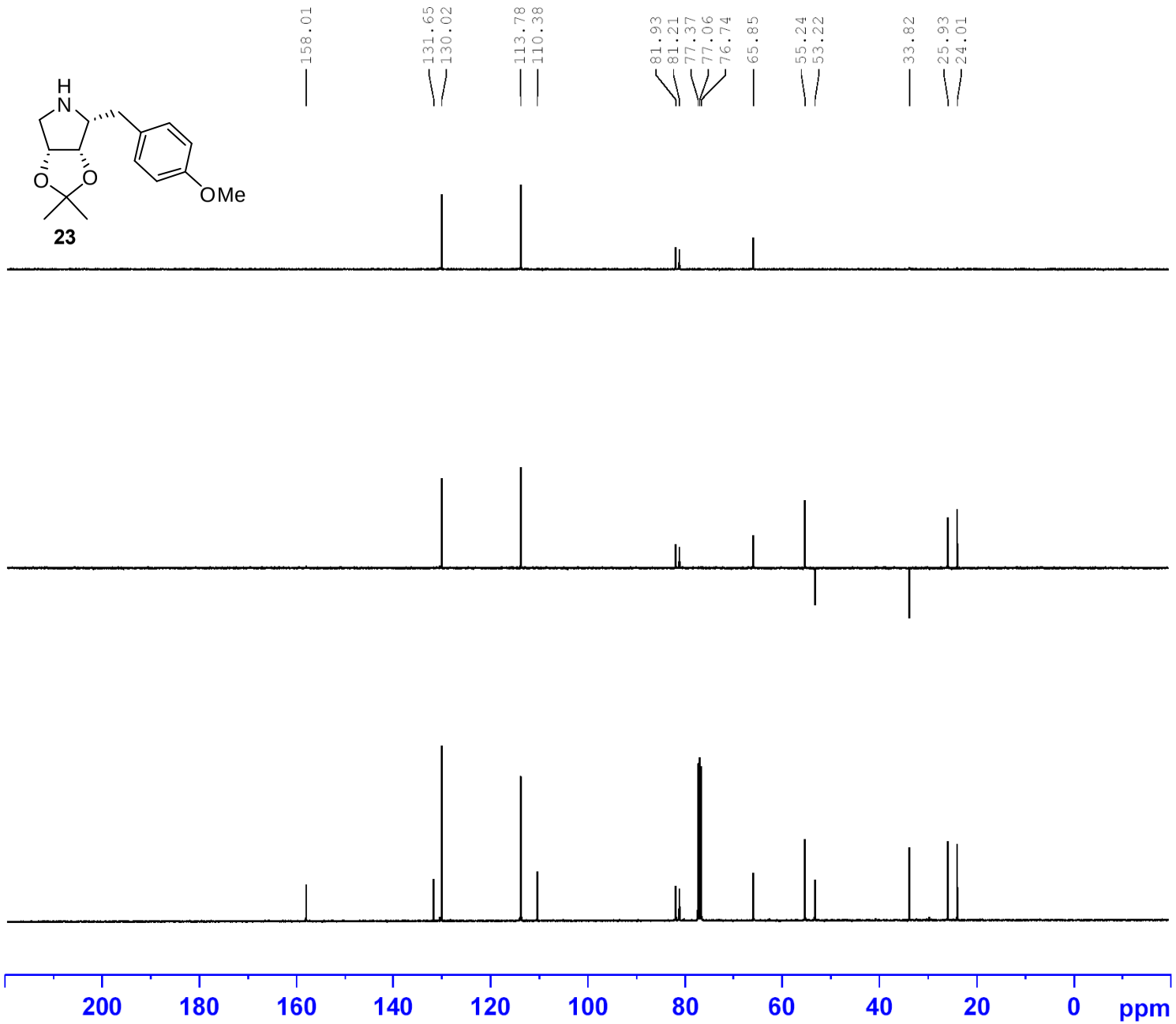
```
NAME            WKWD36_1
EXPNO                  1
PROCNO                 1
Date_           20110517
Time                19.27
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     4
DS                     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                297.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1               -1.00 dB
PL1W         13.56617069 W
SFO1         400.1924713 MHz
SI                 32768
SF           400.1900138 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```

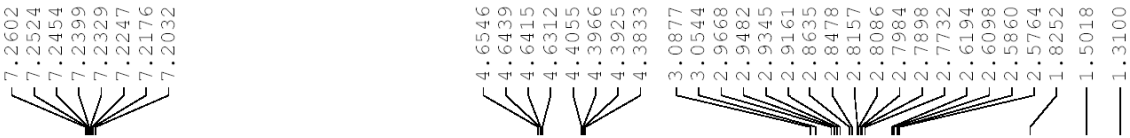



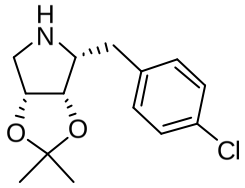
^{13}C NMR



NAME	WKWD36_c13
EXPNO	1
PROCNO	1
Date_	20110517
Time	19.43
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1146
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	181
DW	20.800 usec
DE	6.50 usec
TE	297.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.90 usec
PL1	-2.00 dB
PL1W	55.33689499 W
SFO1	100.6379183 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-1.00 dB
PL12	15.16 dB
PL13	18.62 dB
PL2W	13.56617069 W
PL12W	0.32844096 W
PL13W	0.14806664 W
SFO2	400.1916008 MHz
SI	32768
SF	100.6278564 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{1}H NMR



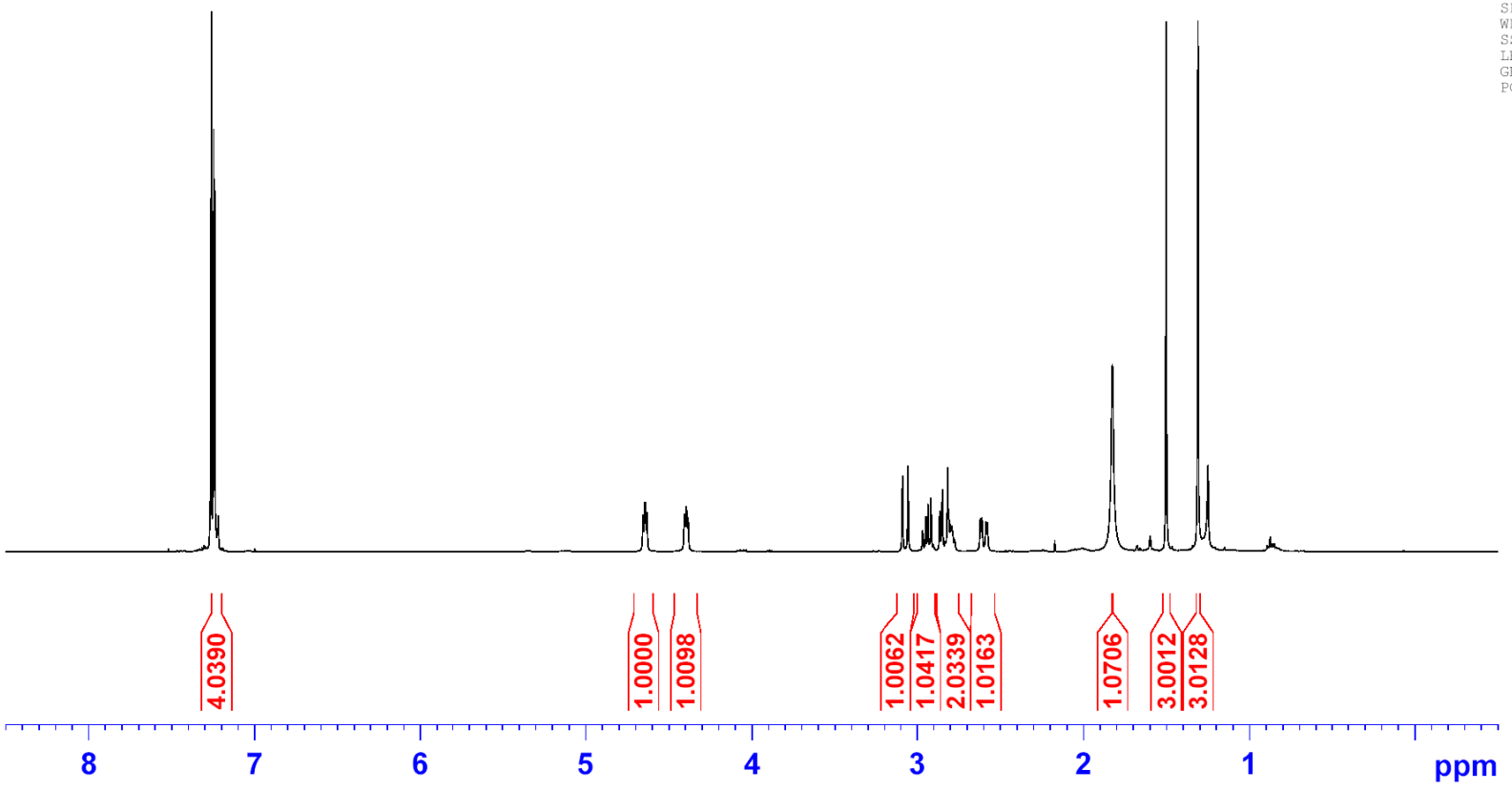


24

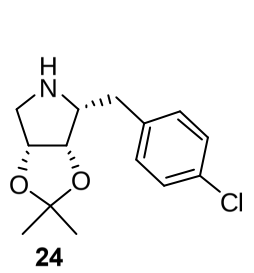
NAME	WKWD47_2
EXPNO	1
PROCNO	1
Date_	20110707
Time	13.28
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	203
DW	60.800 usec
DE	6.50 usec
TE	294.7 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========	
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300096 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

42



^{13}C NMR



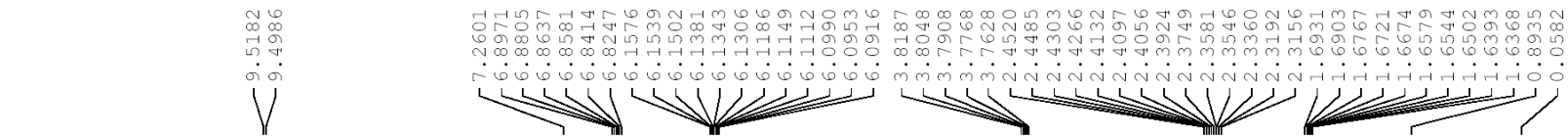
24

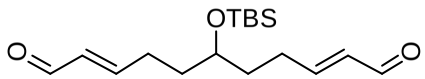

```
NAME          WKWD47_c13_1
EXPNO                    1
PROCNO                   1
Date_             20110708
Time                 22.44
INSTRUM              spect
PROBHD   5 mm PADUL 13C
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                  10805
DS                      4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                    203
DW                20.800 usec
DE                 6.50 usec
TE                295.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.68 usec
PL1                 -0.60 dB
PL1W         41.24164963 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                 0.00 dB
PL12               15.66 dB
PL13               15.92 dB
PL2W          8.31434441 W
PL12W         0.22585411 W
PL13W         0.21272963 W
SFO2        400.1316005 MHz
SI                 32768
SF          100.6127654 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

^{1}H NMR

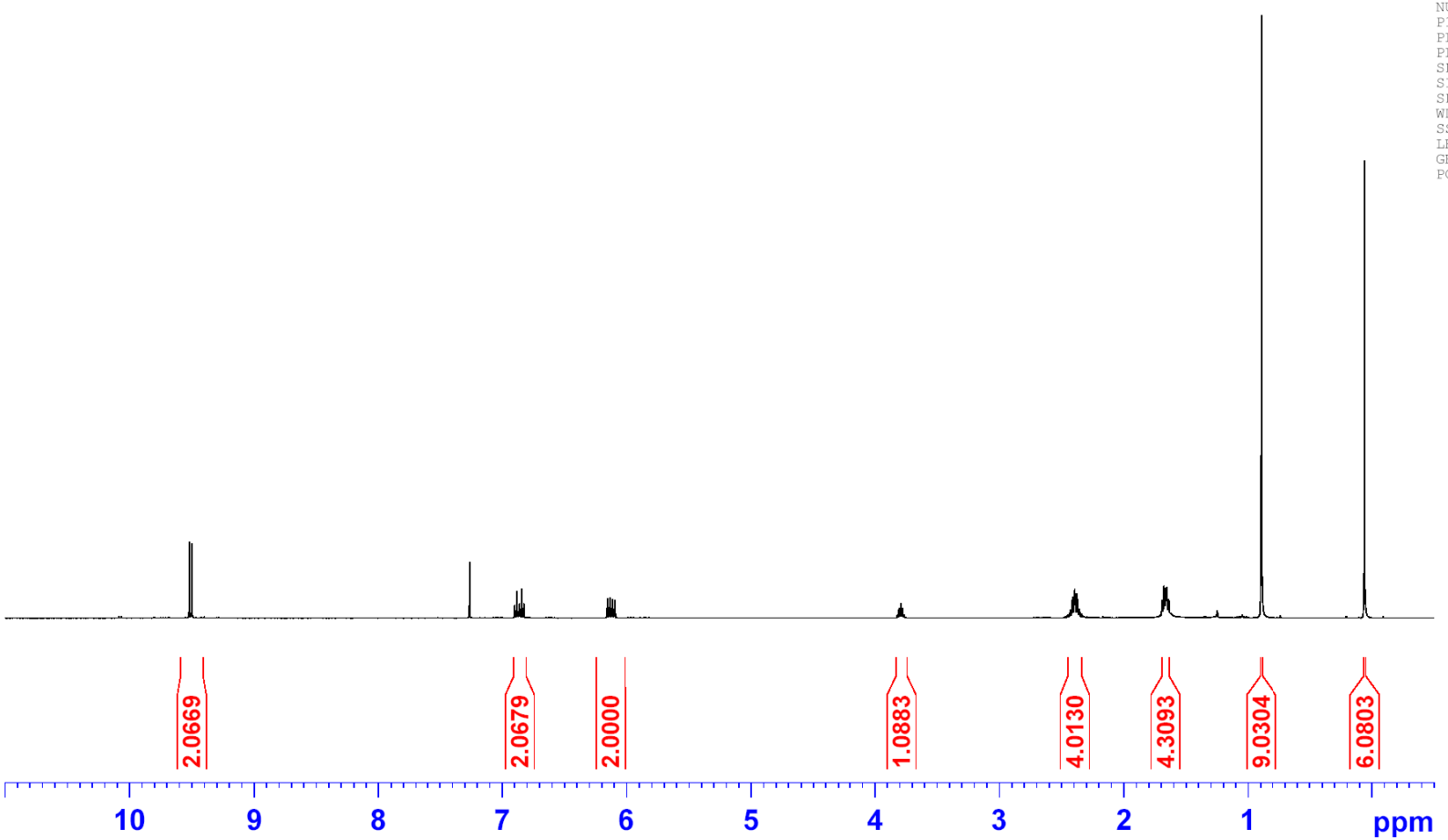




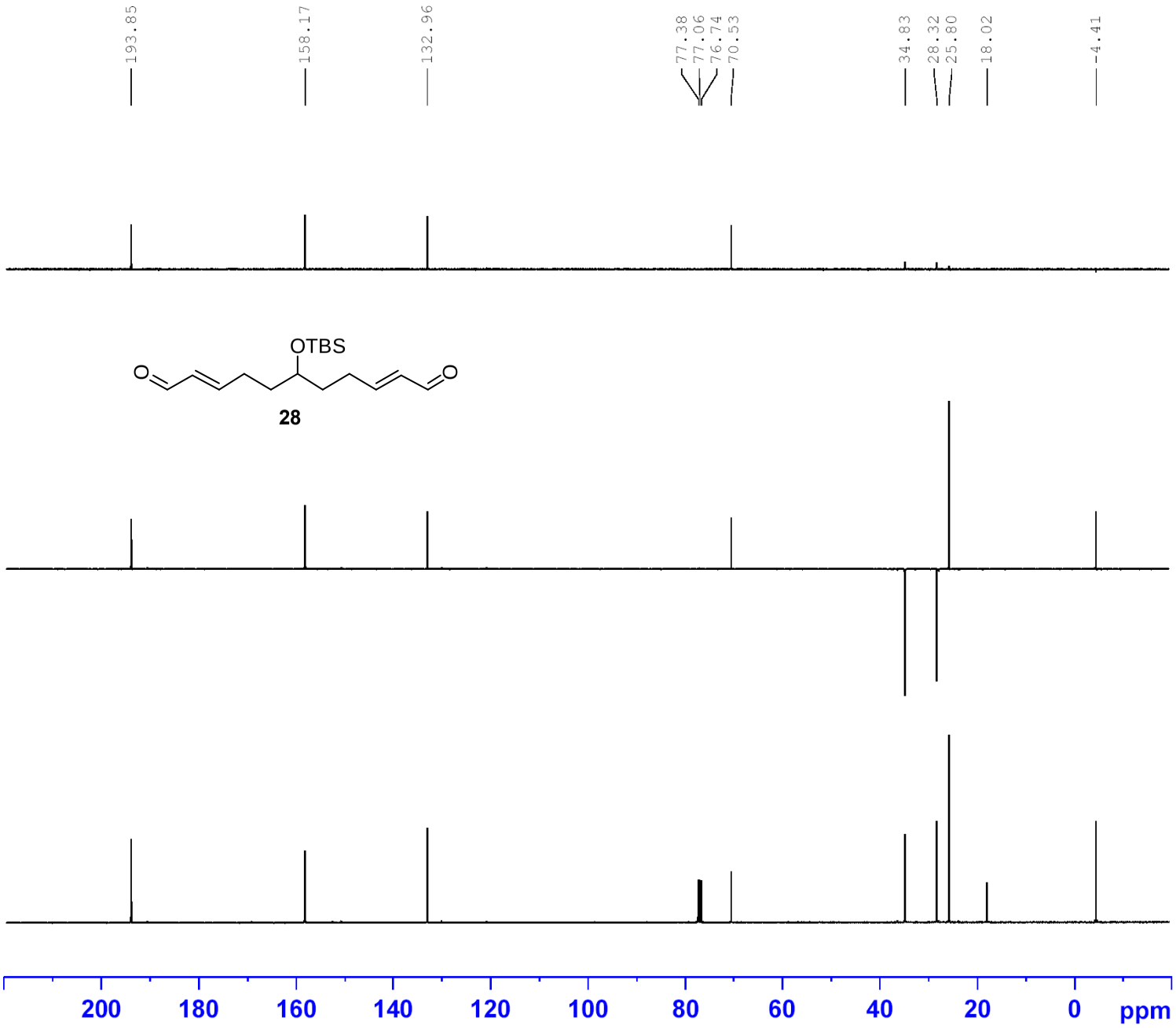
28

```
NAME                WKWG09
EXPNO                    1
PROCNO                   1
Date_             20101026
Time                 18.00
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH               8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    80.6
DW                60.800 usec
DE                 6.50 usec
TE                297.8 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
PL1                 -1.00 dB
PL1W          13.56617069 W
SFO1          400.1924713 MHz
SI                   32768
SF            400.1900138 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    1.00
```

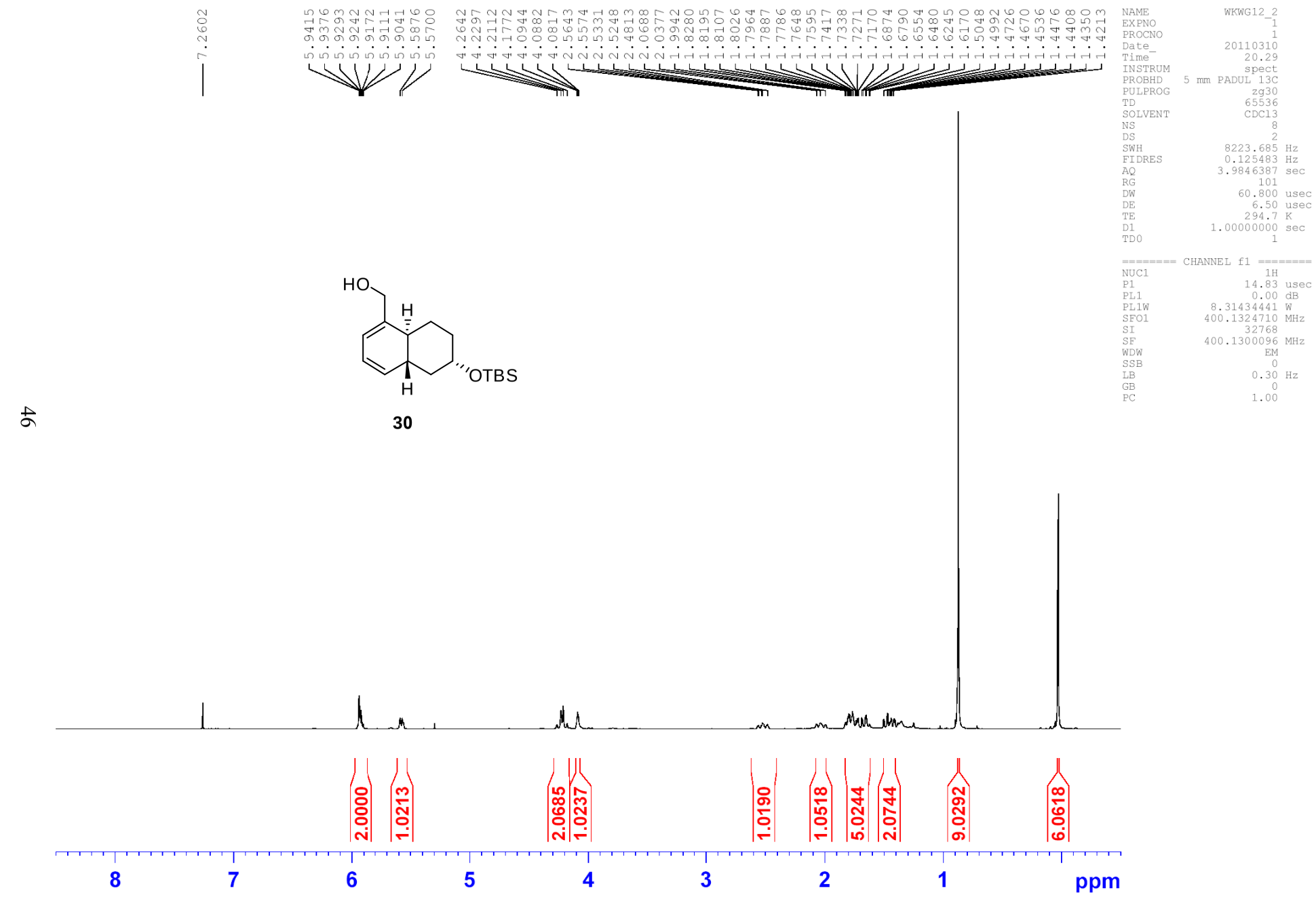


^{13}C NMR

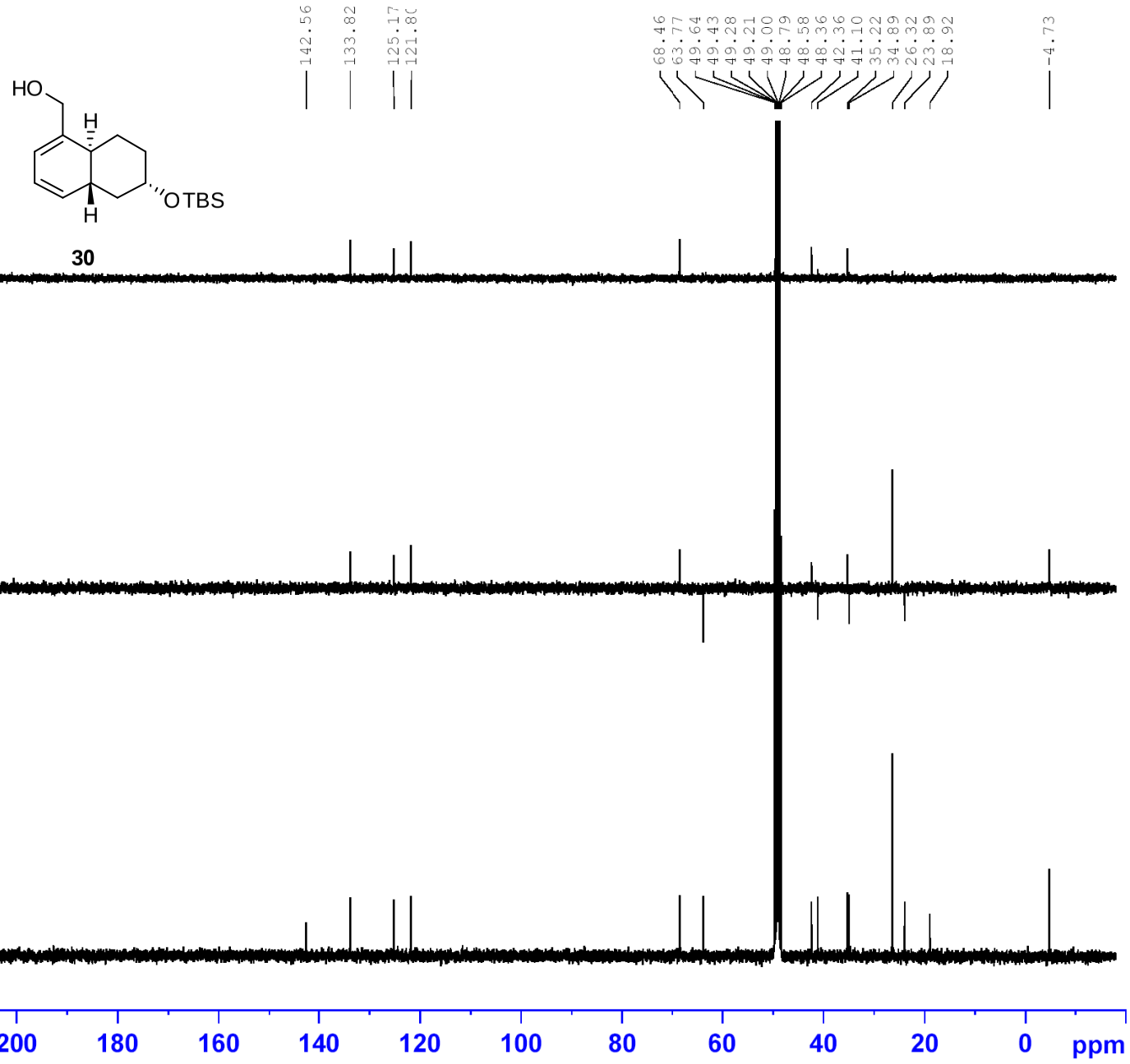


NAME	WKWG09_c13_1
EXPNO	1
PROCNO	1
Date_	20101216
Time	22.12
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	440
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	228
DW	20.800 usec
DE	6.50 usec
TE	299.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.90 usec
PL1	-2.00 dB
PL1W	55.33689499 W
SFO1	100.6379183 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-1.00 dB
PL12	15.16 dB
PL13	18.62 dB
PL2W	13.56617069 W
PL12W	0.32844096 W
PL13W	0.14806664 W
SFO2	400.1916008 MHz
SI	32768
SF	100.6278571 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{1}H NMR


7.2602
5.9415
5.9376
5.9293
5.9242
5.9172
5.9111
5.9041
5.5876
5.5700
4.2642
4.2297
4.2112
4.1772
4.0944
4.0882
4.0817
2.5643
2.5574
2.5331
2.5248
2.4813
2.0688
2.0377
1.9942
1.8280
1.8195
1.8107
1.8026
1.7964
1.7887
1.7786
1.7648
1.7595
1.7417
1.7338
1.7271
1.7170
1.6874
1.6790
1.6554
1.6480
1.6245
1.6170
1.5048
1.4992
1.4726
1.4670
1.4536
1.4476
1.4408
1.4350
1.4213
NAME WKWG12_2
EXPNO 1
PROCNO 1
Date_ 20110310
Time 20.29
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 101
DW 60.800 usec
DE 6.50 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.83 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HO
H
H
OTBS
30
2.0000
1.0213
2.0685
1.0237
1.0190
1.0518
5.0244
2.0744
9.0292
6.0618
8
7
6
5
4
3
2
1
ppm

^{13}C NMR (Solvent: MeOD)



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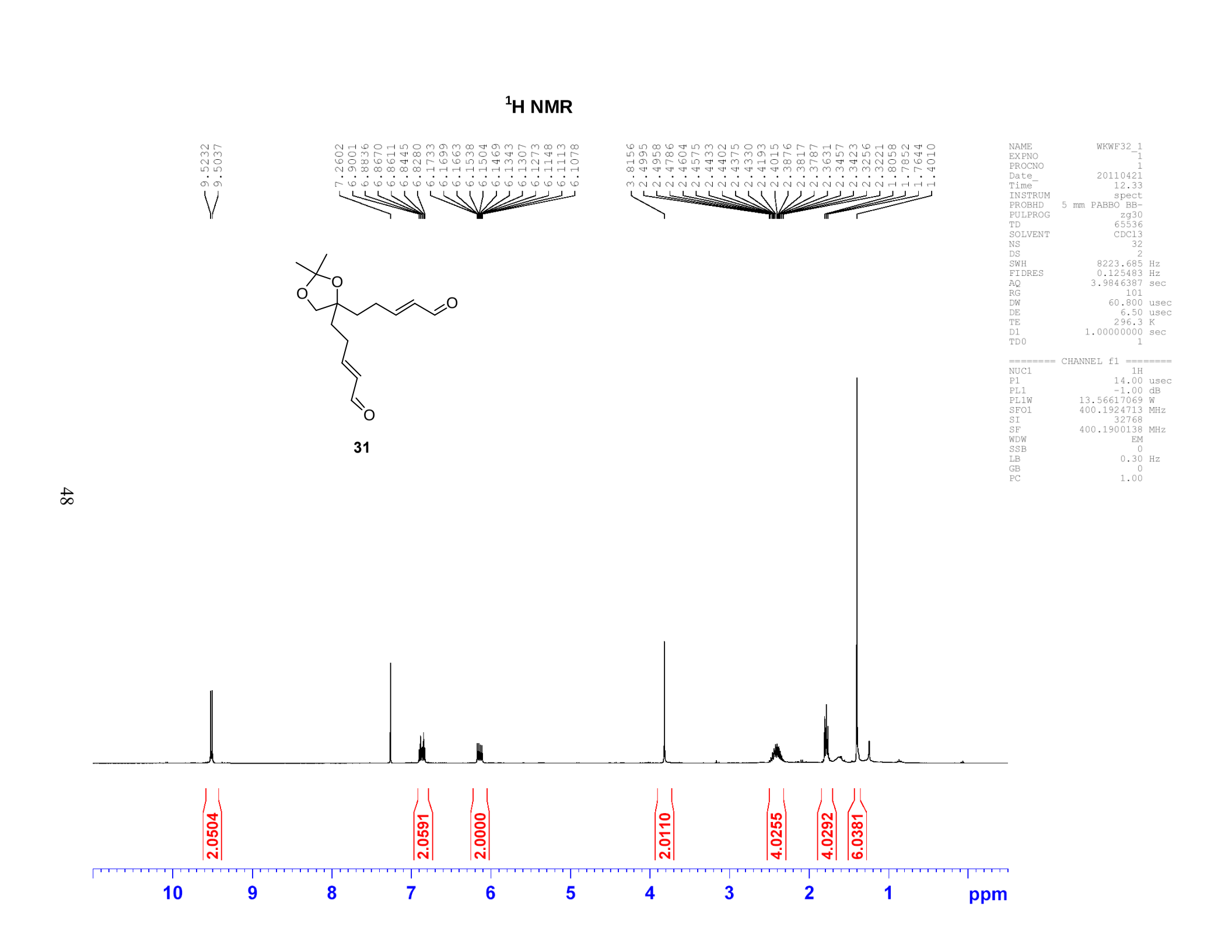
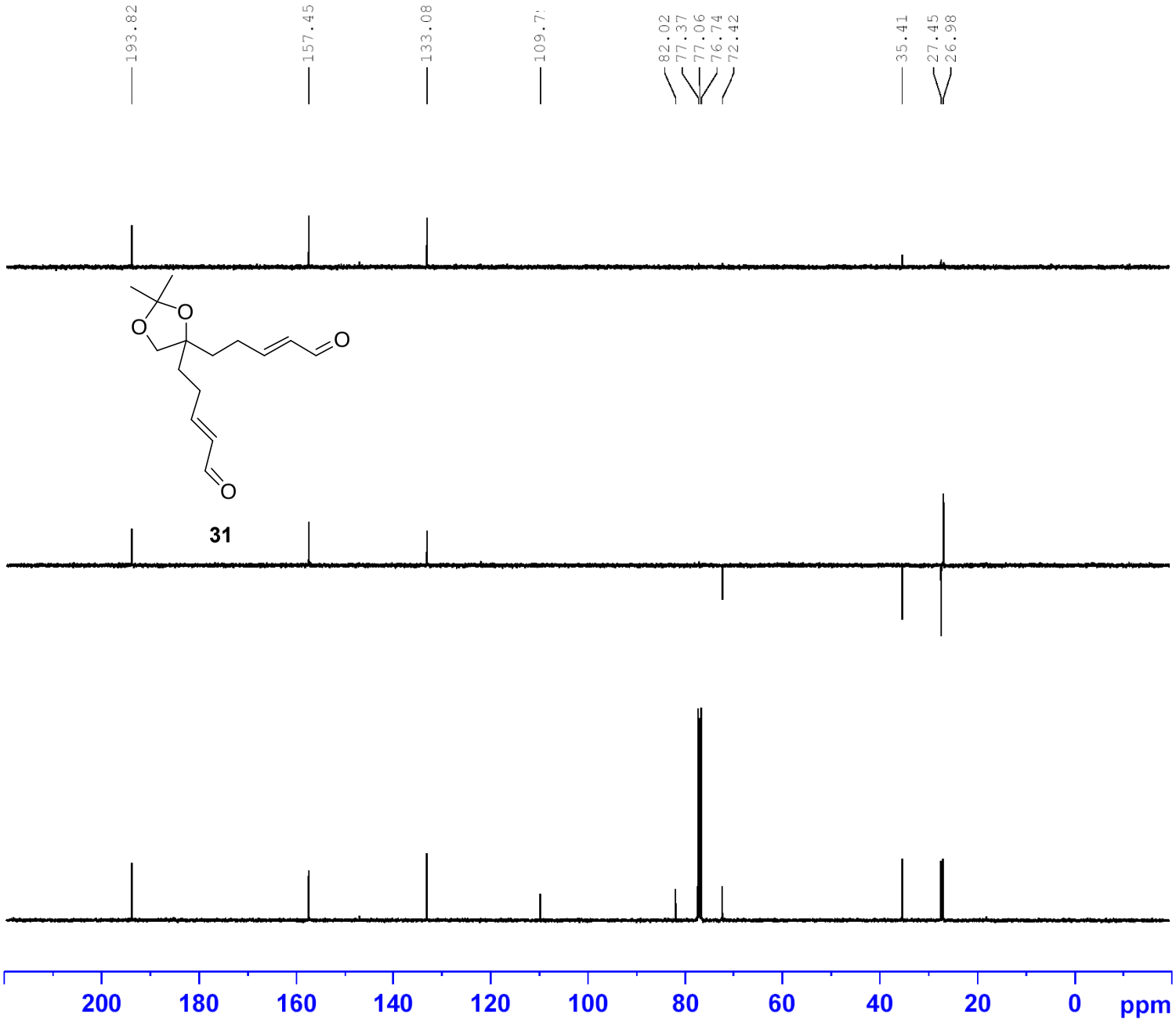
NAME	WKWG12_c13(MeOD)
EXPNO	1
PROCNO	1
Date_	20101222
Time	19.40
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	MeOD
NS	874
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	673.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
========	CHANNEL f2 ========
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.66 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.22585411 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6126271 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{1}H NMR

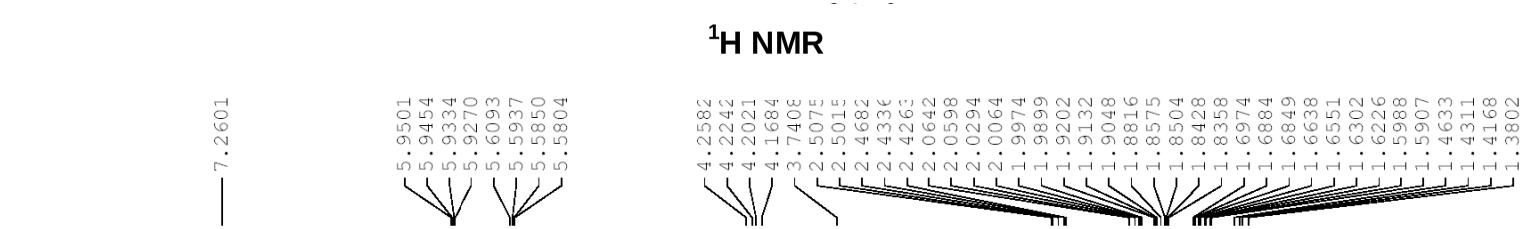

9.5232
9.5037
7.2602
6.9001
6.8836
6.8670
6.8611
6.8445
6.8280
6.1733
6.1699
6.1663
6.1538
6.1504
6.1469
6.1343
6.1307
6.1273
6.1148
6.1113
6.1078
3.8156
2.4995
2.4958
2.4786
2.4604
2.4575
2.4433
2.4402
2.4375
2.4330
2.4193
2.4015
2.3876
2.3817
2.3787
2.3631
2.3457
2.3423
2.3256
2.3221
1.8058
1.7852
1.7644
1.4010
O
O
O
O
31
NAME WKWF32_1
EXPNO 1
PROCNO 1
Date_ 20110421
Time 12.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 101
DW 60.800 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 13.56617069 W
SFO1 400.1924713 MHz
SI 32768
SF 400.1900138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.0504
2.0591
2.0000
2.0110
4.0255
4.0292
6.0381
10
9
8
7
6
5
4
3
2
1
ppm

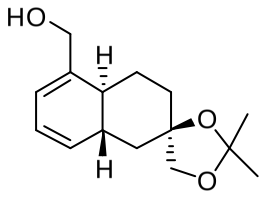
^{13}C NMR



NAME	WKWF32_c13
EXPNO	1
PROCNO	1
Date_	20110416
Time	11.00
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	503
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	294.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.66 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.22585411 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127669 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

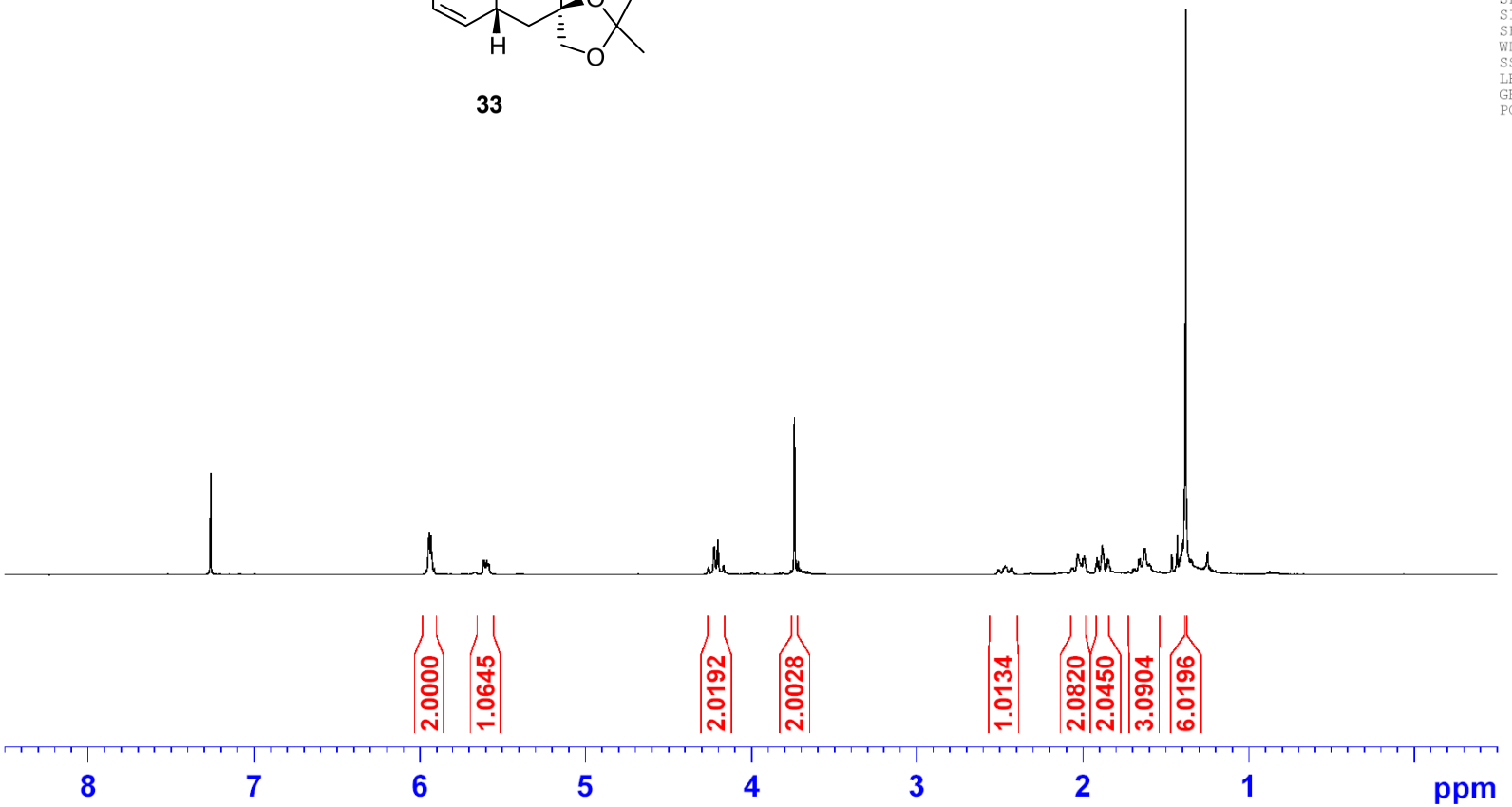
^{1}H NMR

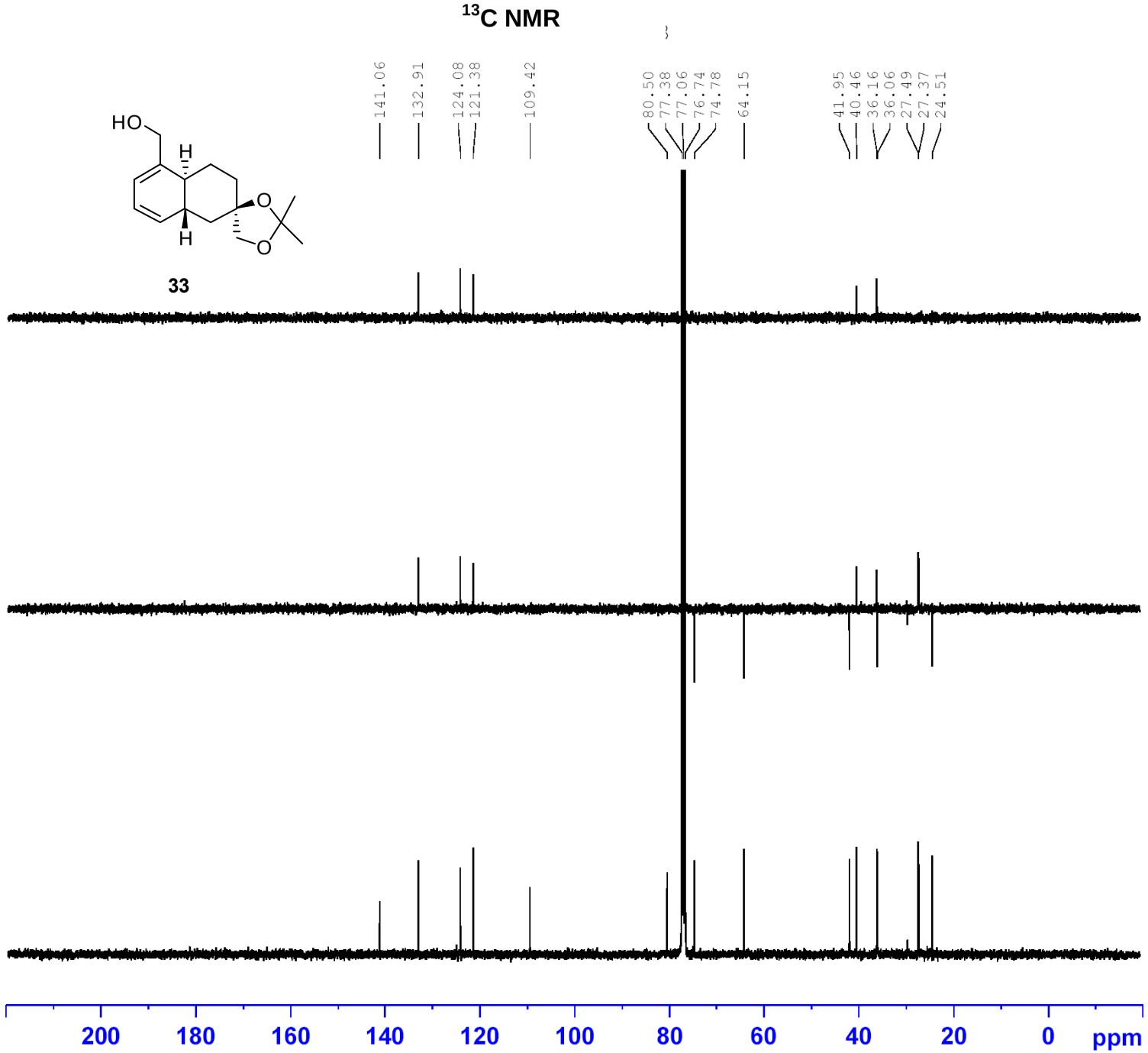




33

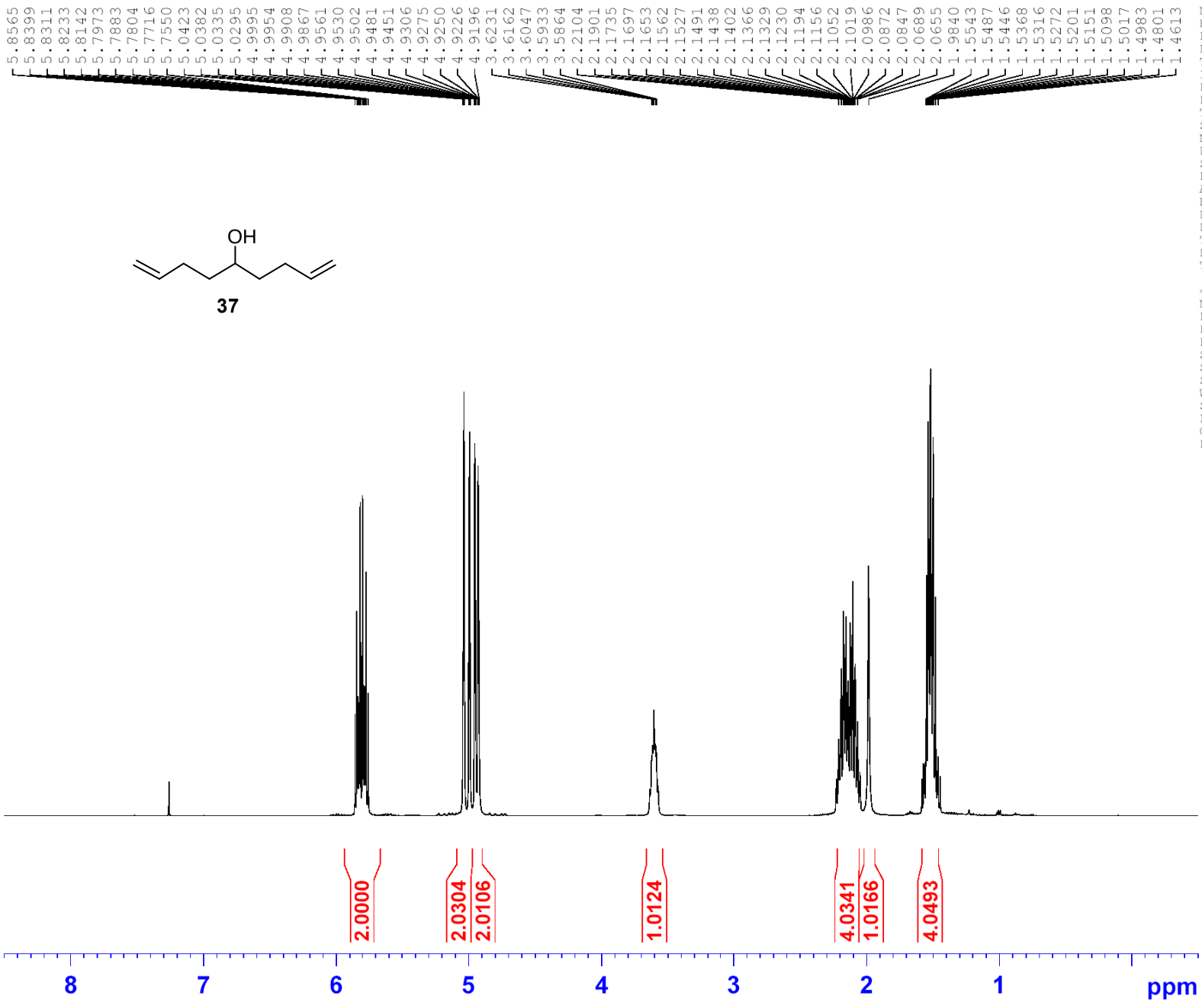
```
NAME            WKWF34_2
EXPNO                  1
PROCNO                 1
Date_           20110722
Time_              22.10
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   203
DW                60.800 usec
DE                 6.50 usec
TE                673.2 K
D1          1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.83 usec
PL1                0.00 dB
PL1W          8.31434441 W
SFO1        400.1324710 MHz
SI                 32768
SF          400.1300096 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```



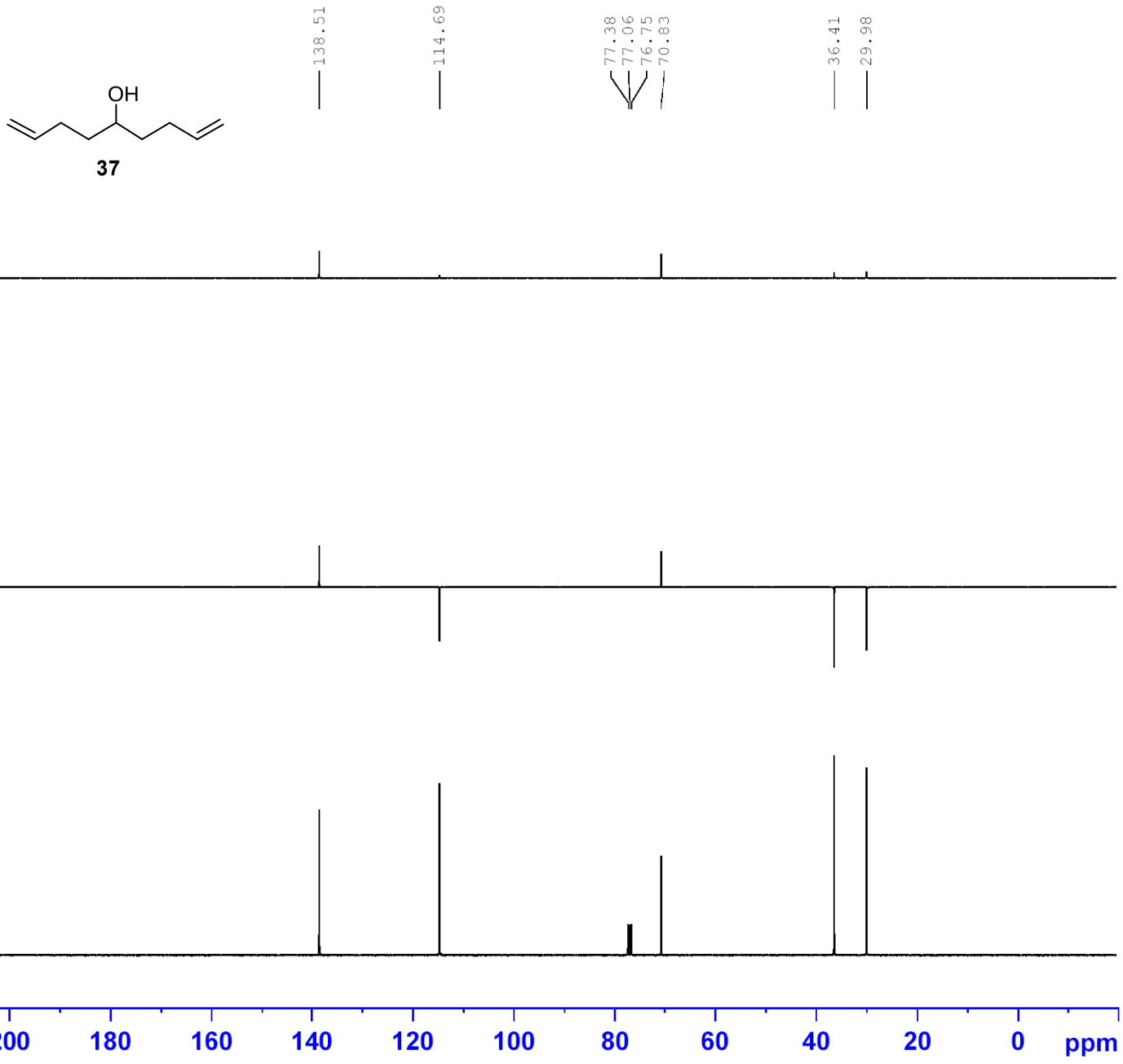


NAME	WKWF34_c13
EXPNO	1
PROCNO	1
Date_	20110427
Time	23.02
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	10980
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	294.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.66 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.22585411 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127646 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{1}H NMR

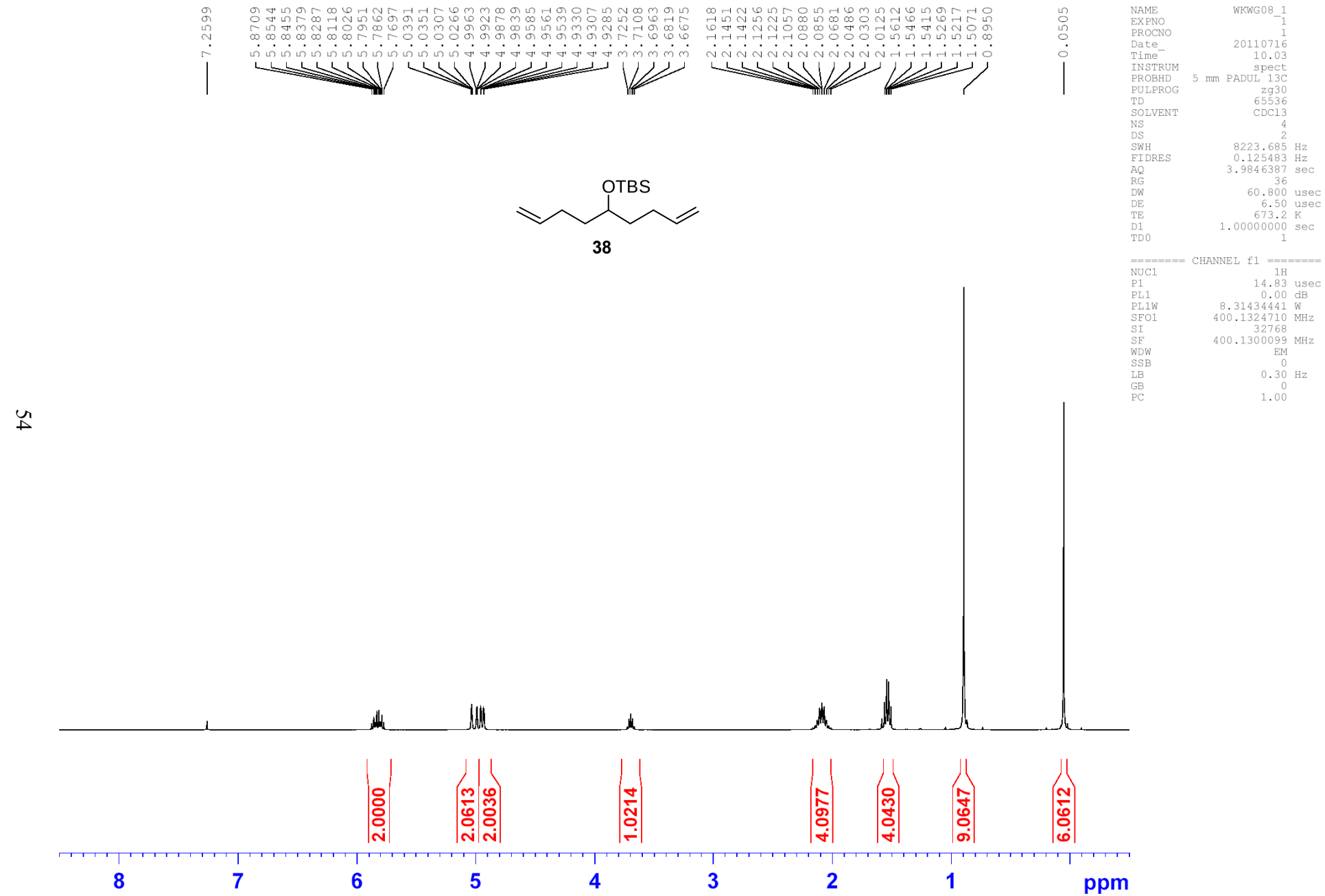

5.8565 5.8399 5.8311 5.8233 5.8142 5.7973 5.7883 5.7804 5.7716 5.7550 5.0423 5.0382 5.0335 5.0295 4.9995 4.9954 4.9908 4.9867 4.9561 4.9530 4.9502 4.9481 4.9451 4.9306 4.9275 4.9250 4.9226 4.9196 3.6231 3.6162 3.6047 3.5933 3.5864 2.2104 2.1901 2.1735 2.1697 2.1653 2.1562 2.1527 2.1491 2.1438 2.1402 2.1366 2.1329 2.1230 2.1194 2.1156 2.1052 2.1019 2.0986 2.0872 2.0847 2.0689 2.0655 1.9840 1.5543 1.5487 1.5446 1.5368 1.5316 1.5272 1.5201 1.5151 1.5098 1.5017 1.4983 1.4801 1.4613
OH
37
NAME WKWG05
EXPNO 1
PROCNO 1
Date_ 20101021
Time 14.12
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 22.6
DW 60.800 usec
DE 6.50 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.83 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.0000
2.0304
2.0106
1.0124
4.0341
1.0166
4.0493
8
7
6
5
4
3
2
1
ppm

^{13}C NMR




```
NAME          WKWG05_c13
EXPNO                  1
PROCNO                 1
Date_           20101021
Time              14.18
INSTRUM           spect
PROBHD   5 mm PADUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   89
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                  203
DW                20.800 usec
DE                 6.50 usec
TE                295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.68 usec
PL1                -0.60 dB
PL1W        41.24164963 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                 0.00 dB
PL12               15.66 dB
PL13               15.92 dB
PL2W         8.31434441 W
PL12W        0.22585411 W
PL13W        0.21272963 W
SFO2       400.1316005 MHz
SI               32768
SF         100.6127720 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

^{1}H NMR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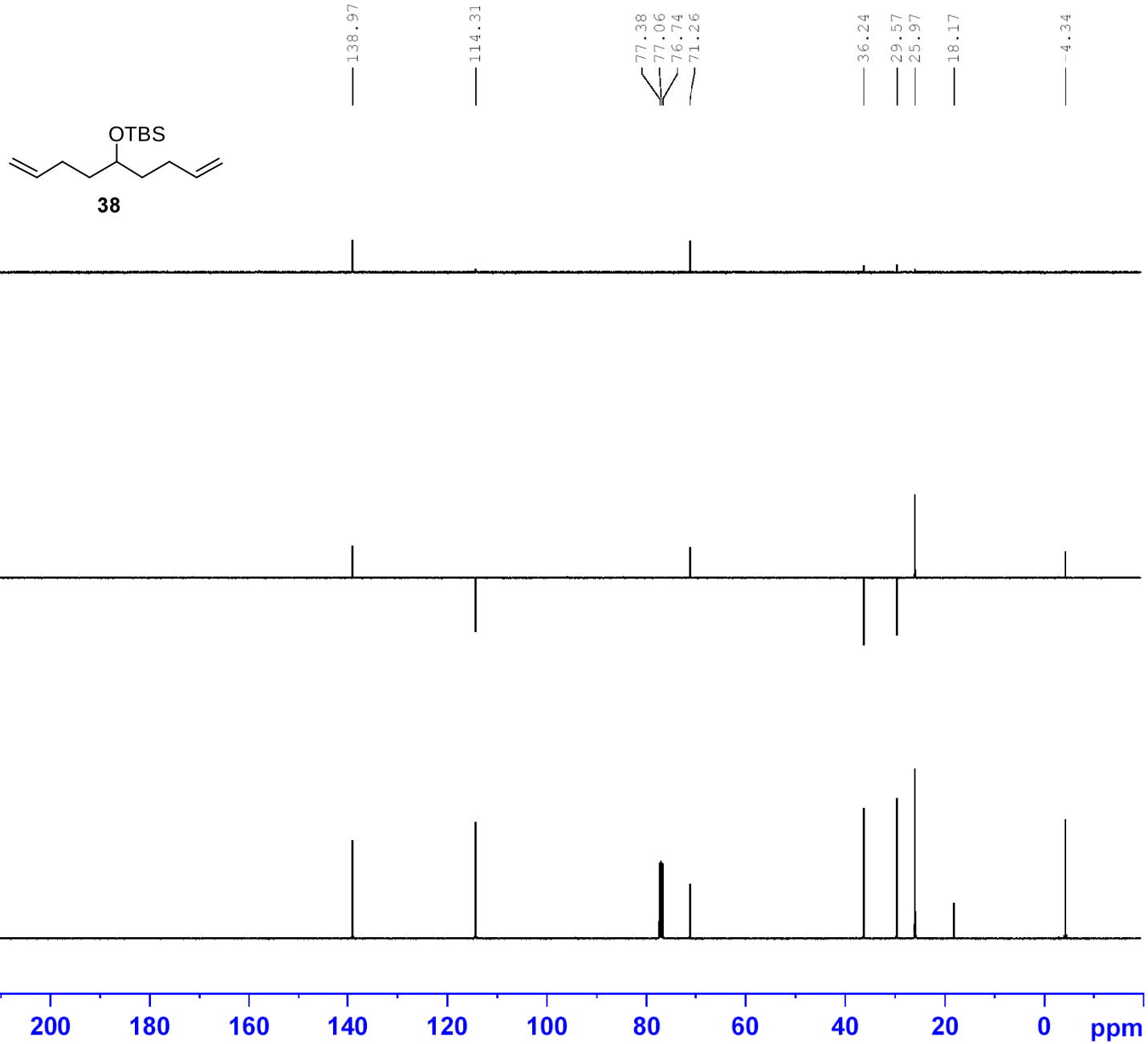
5.8709
5.8544
5.8455
5.8379
5.8287
5.8118
5.8026
5.7951
5.7862
5.7697
5.0391
5.0351
5.0307
5.0266
4.9963
4.9923
4.9878
4.9839
4.9585
4.9561
4.9539
4.9330
4.9307
4.9285
3.7252
3.7108
3.6963
3.6819
3.6675
2.1618
2.1451
2.1422
2.1256
2.1225
2.1057
2.0880
2.0855
2.0681
2.0486
2.0303
2.0125
1.5612
1.5466
1.5415
1.5269
1.5217
1.5071
0.8950
0.0505
OTBS
38
NAME WKWG08_1
EXPNO 1
PROCNO 1
Date_ 20110716
Time 10.03
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 36
DW 60.800 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.83 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.0000
2.0613
2.0036
1.0214
4.0977
4.0430
9.0647
6.0612
8
7
6
5
4
3
2
1
ppm

^{13}C NMR




```
NAME          WKWG08_c13_1
EXPNO                    1
PROCNO                   1
Date_             20110716
Time                 10.16
INSTRUM              spect
PROBHD   5 mm PADUL 13C
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  657
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                  203
DW                20.800 usec
DE                 6.50 usec
TE                673.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.68 usec
PL1                -0.60 dB
PL1W        41.24164963 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2                0.00 dB
PL12              15.66 dB
PL13              15.92 dB
PL2W         8.31434441 W
PL12W        0.22585411 W
PL13W        0.21272963 W
SFO2       400.1316005 MHz
SI                32768
SF         100.6127639 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

^{1}H NMR

7.2599

5.8724
5.8562
5.8476
5.8401
5.8306
5.8137
5.7976
5.7883
5.7720
5.0589
5.0160
4.9696
4.9445

3.4618

2.5451
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2.0874
2.0678
2.0491
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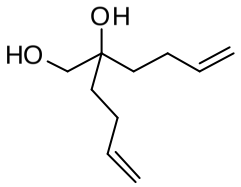
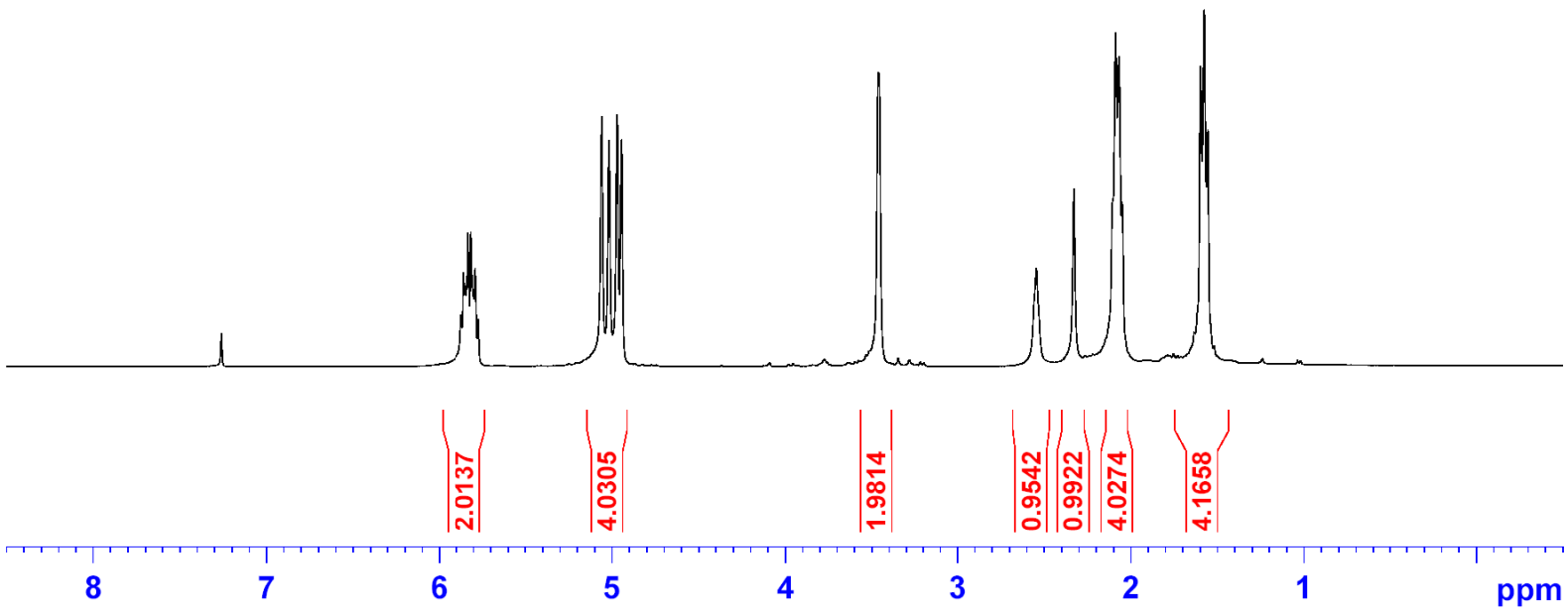
1.5873
1.5754
1.5589
1.5549

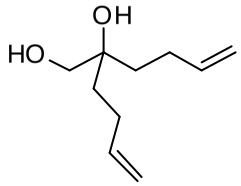


40

NAME	WKWF30
EXPNO	1
PROCNO	1
Date_	20110411
Time	19.31
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	4
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	71.8
DW	60.800 usec
DE	6.50 usec
TE	294.5 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300099 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



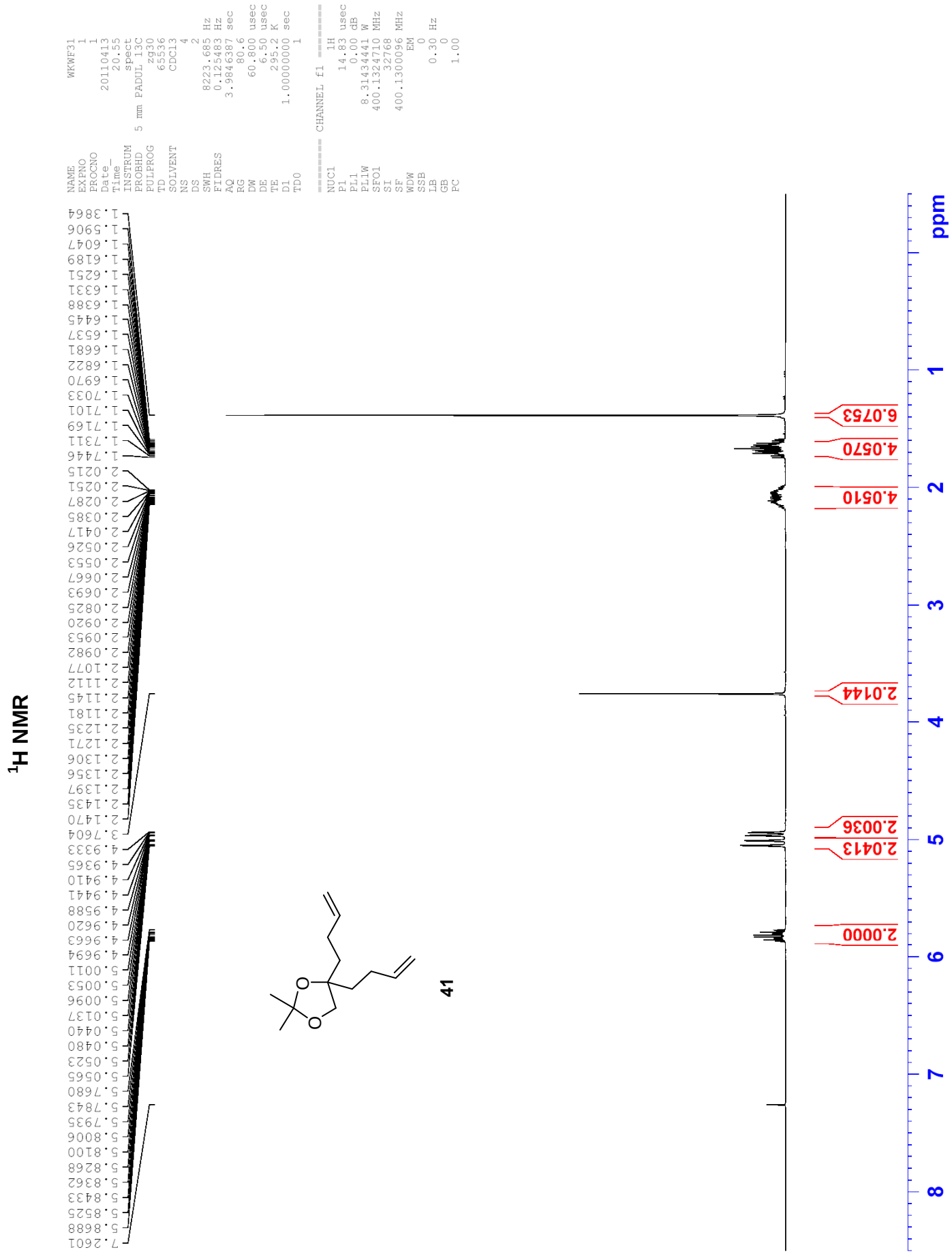
^{13}C NMR



40

NAME	WKWF30_c13
EXPNO	1
PROCNO	1
Date_	20110411
Time	19.45
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	76
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	294.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.66 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.22585411 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127684 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{1}H NMR


NAME WKWF31
EXPNO 1
PROCNO 1
Date_ 20110413
Time_ 20.55
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.83 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.3864
1.5906
1.6047
1.6189
1.6251
1.6331
1.6388
1.6445
1.6537
1.6681
1.6822
1.6970
1.7033
1.7101
1.7169
1.7311
1.7446
2.0215
2.0251
2.0287
2.0385
2.0417
2.0526
2.0553
2.0667
2.0693
2.0825
2.0920
2.0953
2.0982
2.1077
2.1112
2.1145
2.1181
2.1235
2.1271
2.1306
2.1356
2.1397
2.1435
2.1470
3.7604
4.9333
4.9365
4.9410
4.9441
4.9588
4.9620
4.9663
4.9694
5.0011
5.0053
5.0096
5.0137
5.0440
5.0480
5.0523
5.0565
5.7680
5.7843
5.7935
5.8006
5.8100
5.8268
5.8362
5.8433
5.8525
5.8688
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4.0570
4.0510
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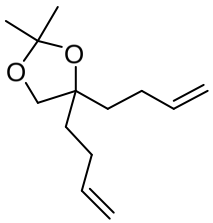
2.0413
2.0000
ppm

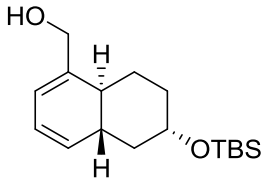
41

^{13}C NMR



41

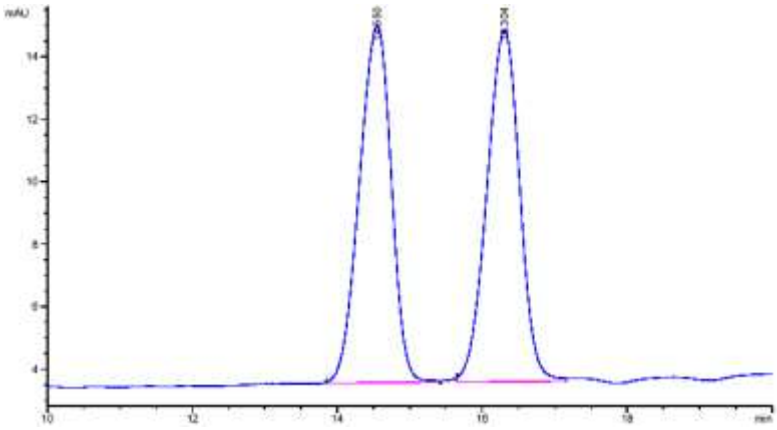
NAME	WKWF31_c13_1	
EXPNO	1	
PROCNO	1	
Date_	20110413	
Time	23.41	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	1373	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	203	
DW	20.800	usec
DE	6.50	usec
TE	295.2	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.68	usec
PL1	-0.60	dB
PL1W	41.24164963	W
SFO1	100.6228298	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	90.00	usec
PL2	0.00	dB
PL12	15.66	dB
PL13	15.92	dB
PL2W	8.31434441	W
PL12W	0.22585411	W
PL13W	0.21272963	W
SFO2	400.1316005	MHz
SI	32768	
SF	100.6127654	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	



30

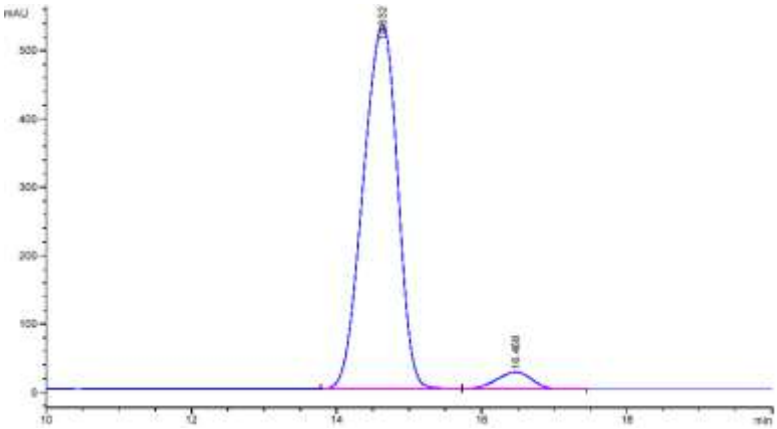
HPLC-Condition: Column: Chiralcel OD-H, Chiral Technologies, Inc.
Eluent: Hexane/2-propanol (99/1); **Flow rate:** 0.70 ml/min;
Detection: UV 254nm

Racemic cycloadduct

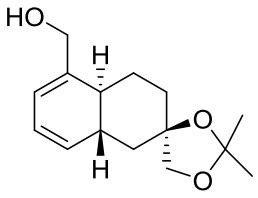


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.550	BB	0.4878	349.54221	11.42980	49.7626
2	16.304	BB	0.4942	352.87723	11.24769	50.2374

Chiral cycloadduct



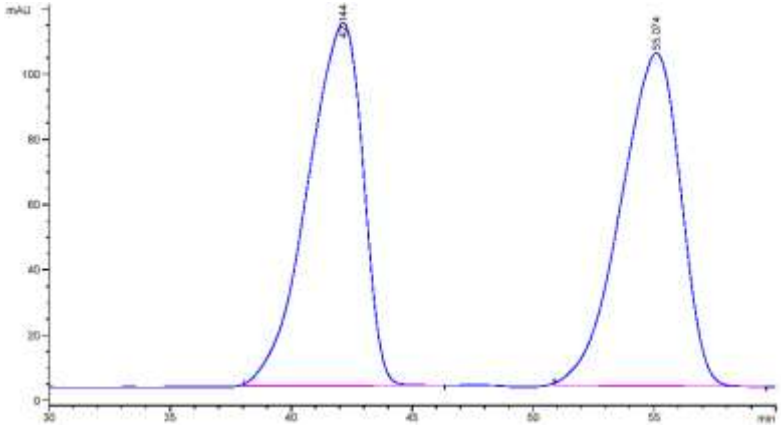
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.632	BB	0.5321	1.75390e4	533.44531	95.3607
2	16.468	BF	0.5418	853.27319	24.94400	4.6393



33

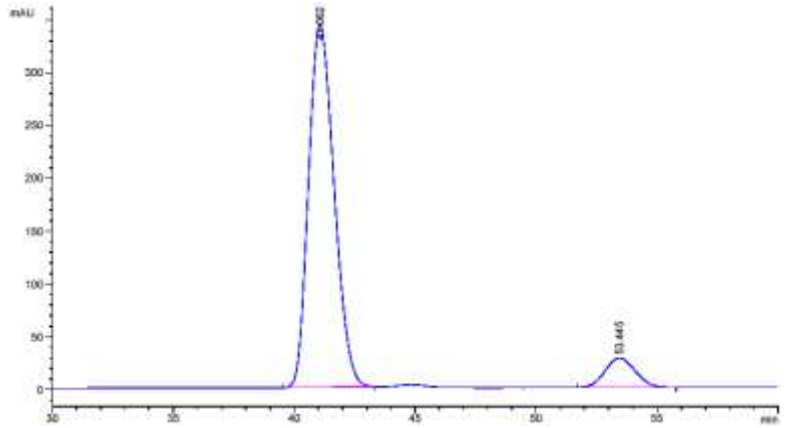
HPLC-Condition: Column: Chiralcel OD-H, Chiral Technologies, Inc.
Eluent: Hexane/2-propanol (99/1); **Flow rate:** 0.70 ml/min;
Detection: UV 254nm

Racemic cycloadduct



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	42.144	BP	2.4089	1.76916e4	111.22813	50.1379
2	55.074	BB	2.6503	1.75943e4	101.99398	49.8621

Chiral cycloadduct



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	41.062	PB	1.1650	2.52116e4	343.49353	91.2276
2	53.445	BB	1.3661	2424.32788	27.81921	8.7724