

Supporting information

Discovery of a phosphine mediated cycloisomerization of alkynyl hemiketals: access to spiroketals and dihydropyrazoles via tandem reactions

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General Experimental.

Unless otherwise noted, all reactions were conducted at room temperature under nitrogen atmosphere and all the solvents and reagents were purchased commercially and used as it is. Reactions were monitored by TLC (silica gel, 60 Å, F254, 250 nm). TLC was visualized either under UV light or by charring with 2.5% *p*-anisaldehyde in H₂SO₄, acetic acid, and ethanol solution or by ninhydrin solution for primary and secondary amine compounds. Flash column chromatography was conducted on silica gel (60 Å, 32-63 µm). Optical rotations were measured at 22 ± 2 °C. ¹H NMR spectra were recorded at 300, 400 and 500 MHz with chemical shifts referenced to (CH₃)₄Si (δ_{H} 0.00 ppm), the residual signal in CDCl₃ (δ_{H} 7.27 ppm) or CD₃OD (δ_{H} 3.31 ppm). ¹³C spectra were collected at 75, 100 and 125 MHz and referenced to the residual signal in CDCl₃ (δ_{C} 77.2) or CD₃OD (δ_{C} 49.0 ppm).

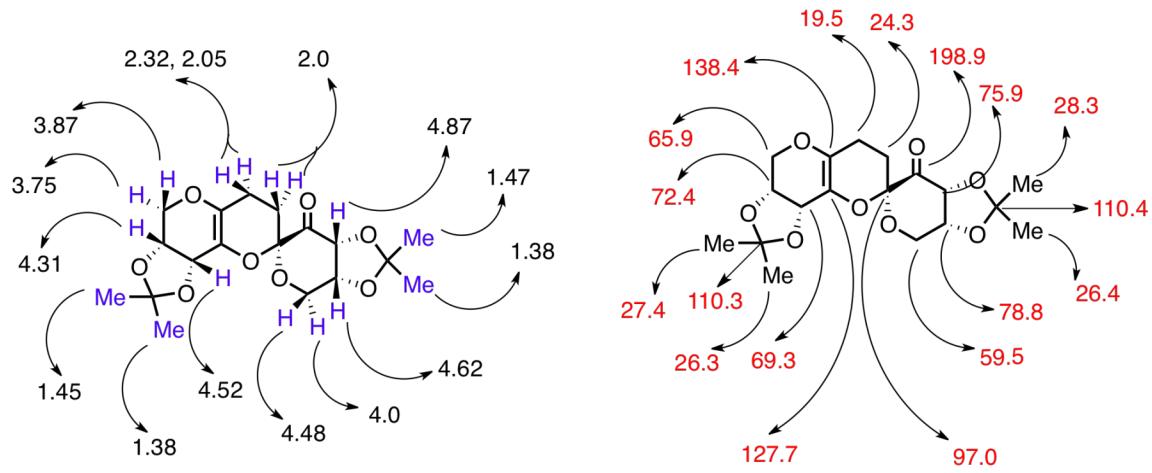
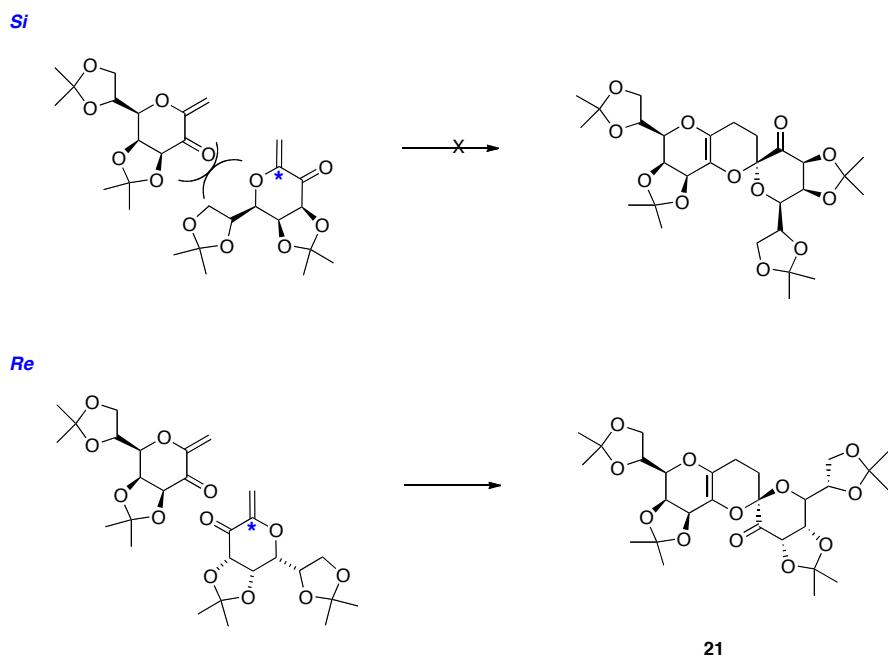
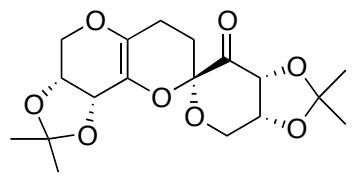


Figure S-1. ^1H and ^{13}C chemical shift assignment for compound **15**.

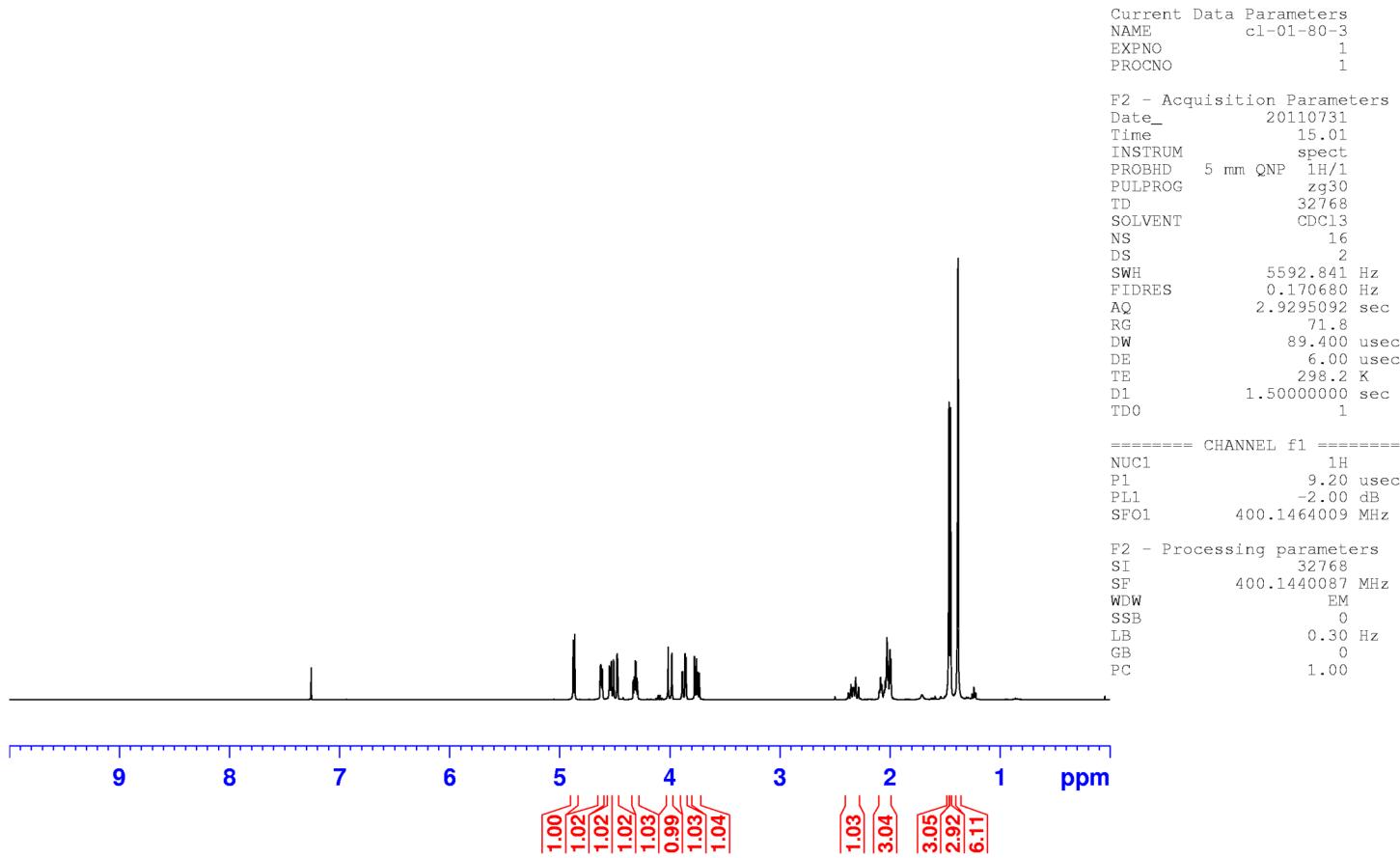
Scheme S1. Facial selectivity of the dienophile in the [4+2] dimerization reaction

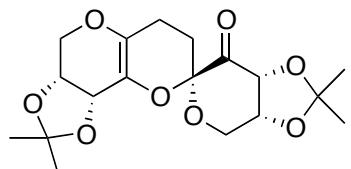


Scheme S1 shows proposed transition states (TS) for the [4+2] hetero Diels Alder reactions that give rise to spiroketal product **21**. The model should hold for products **21-23** in Figure 3 of the main text. The spiroketal stereochemistry of **15** has been confirmed by an X-ray crystal structure of the product; the reaction proceeds through the Si face of the dienophile (Si/Re nomenclature refers to the face of the enol ether carbon that ends up as the spiroketal carbon.). In Scheme S1, two transition states (Si and Re) are shown. We propose that the reaction for C5-substituted systems proceeds through the Re face of the dienophile based on the precedent in *Carbohydr. Res.* **1994**, 264, 141-146 and on the potential for steric congestion in reactions that go through the Si face based on substitutents at the C5 position of the keto-enol ether.

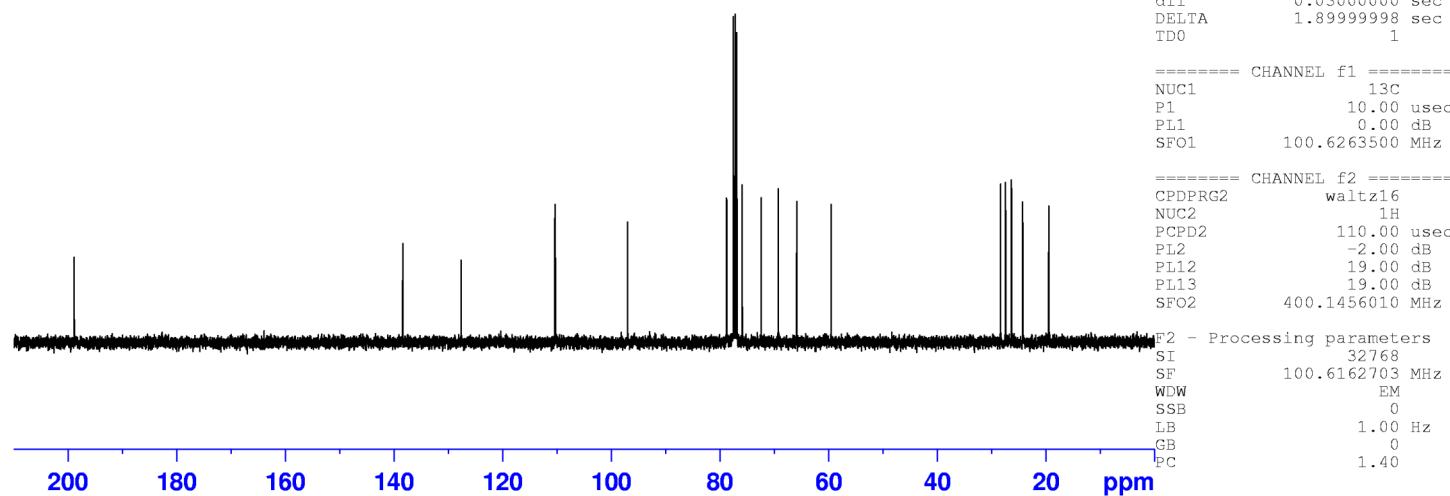


15





15



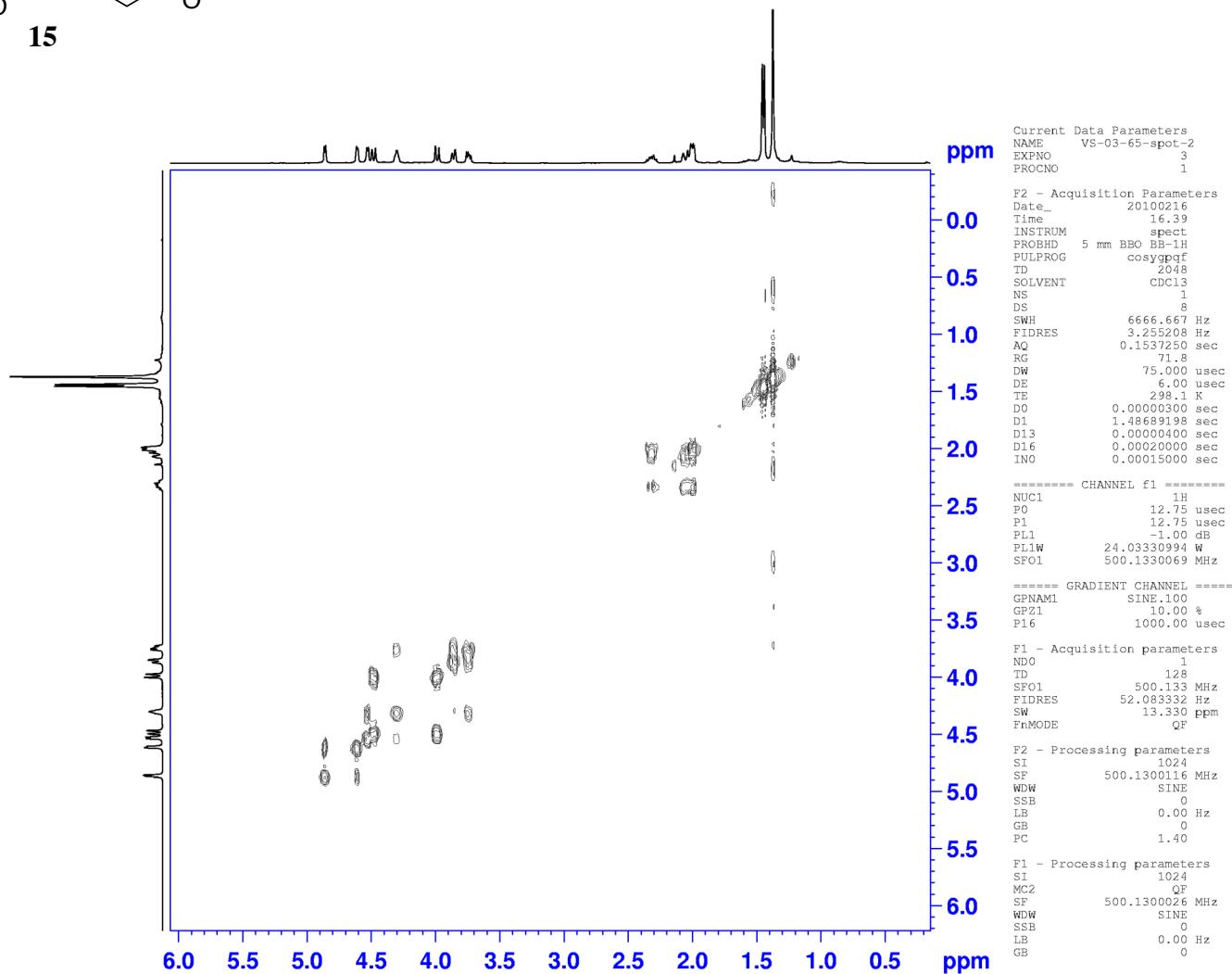
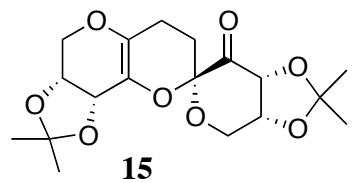
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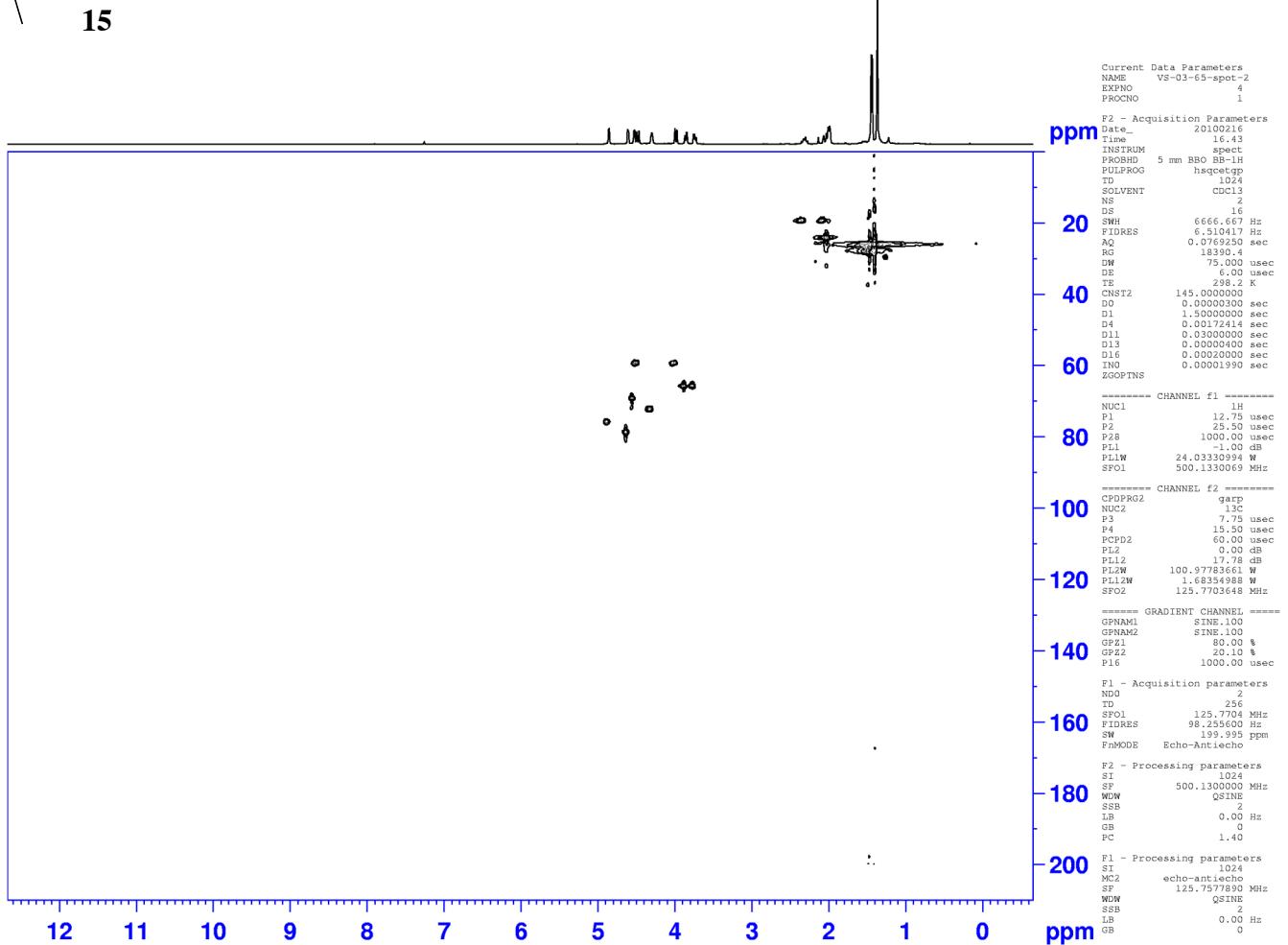
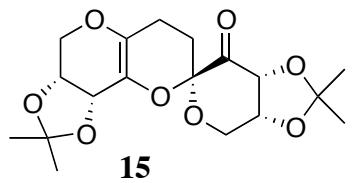
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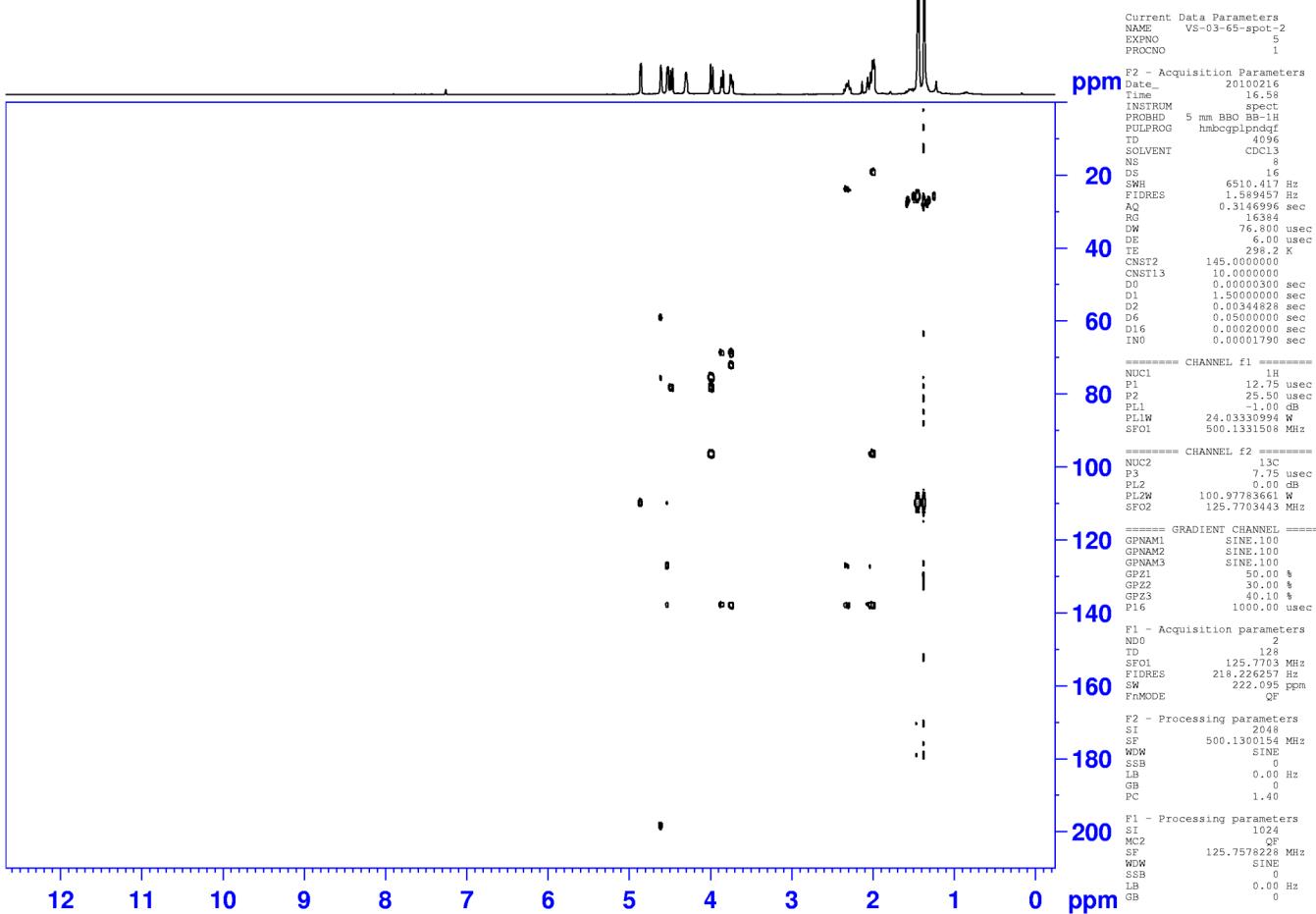
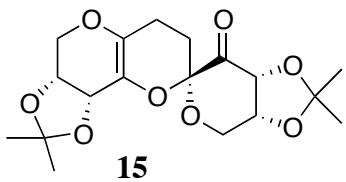
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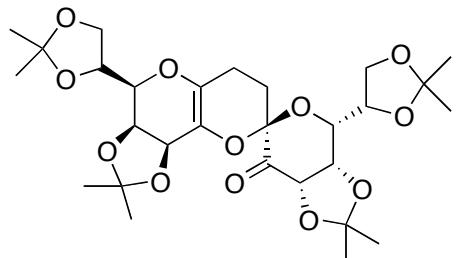
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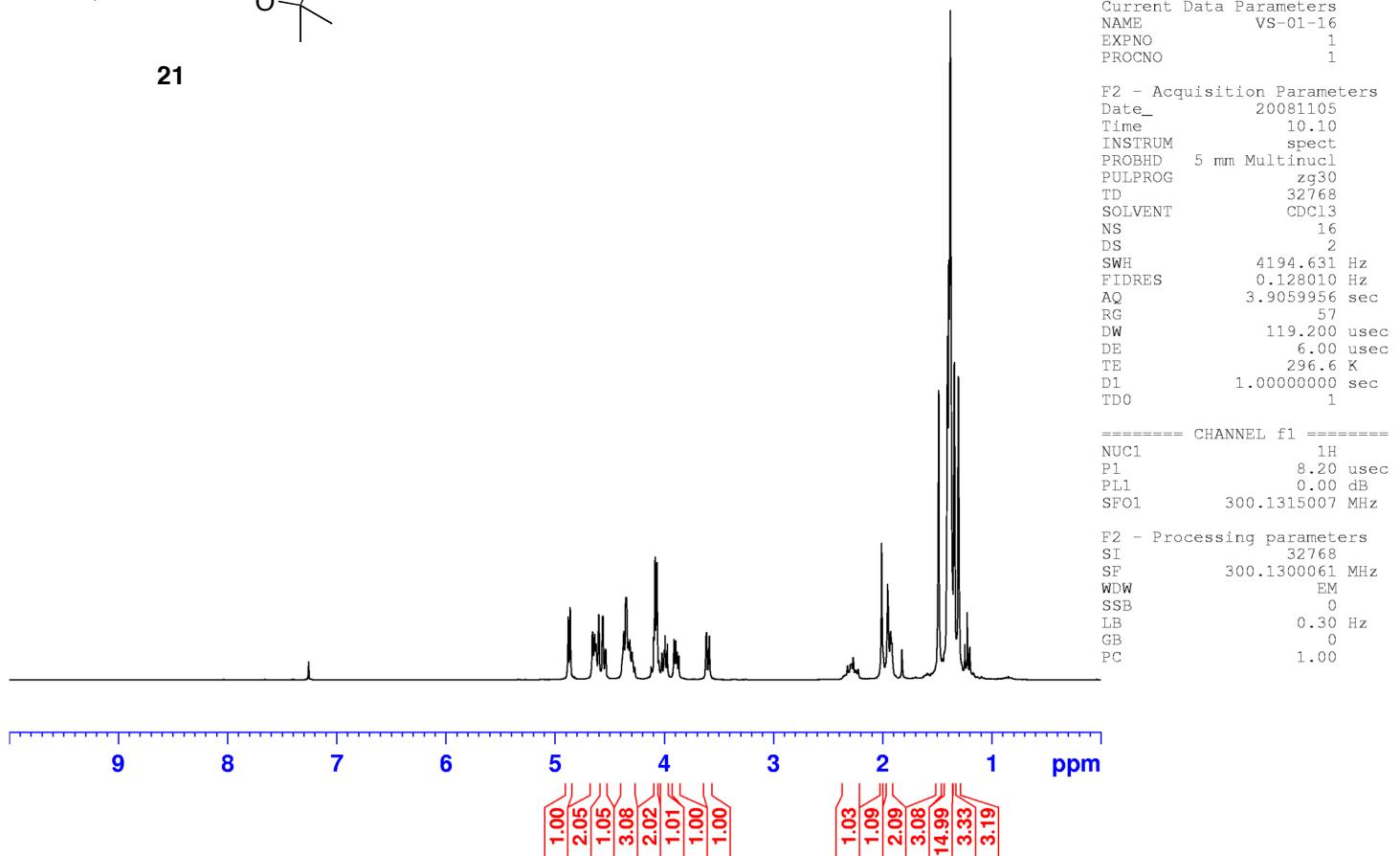


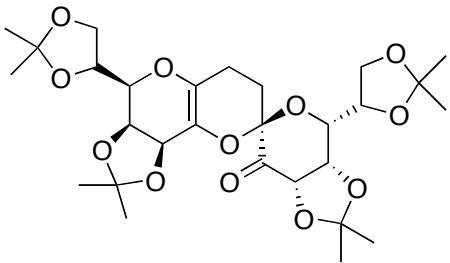




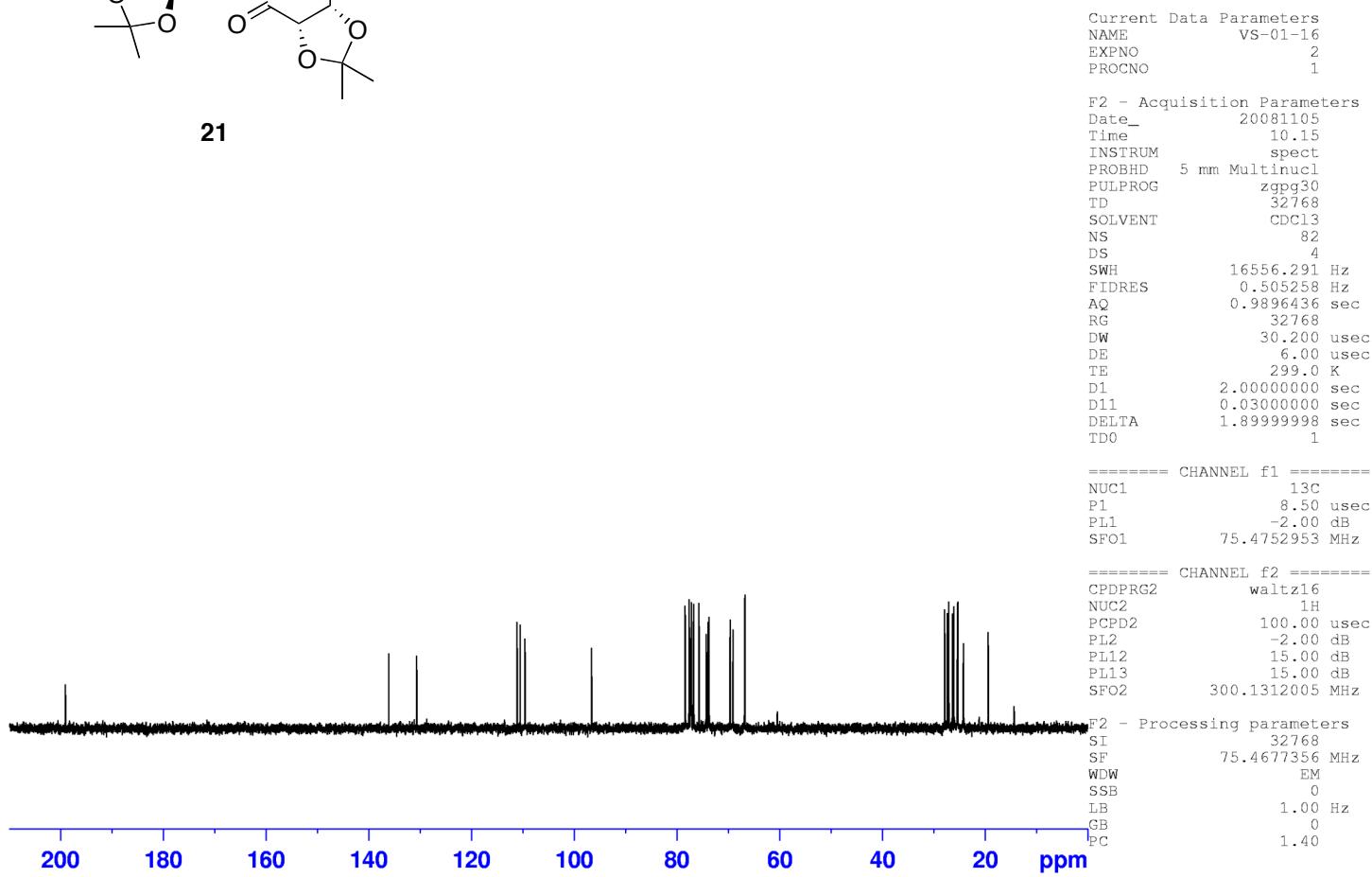


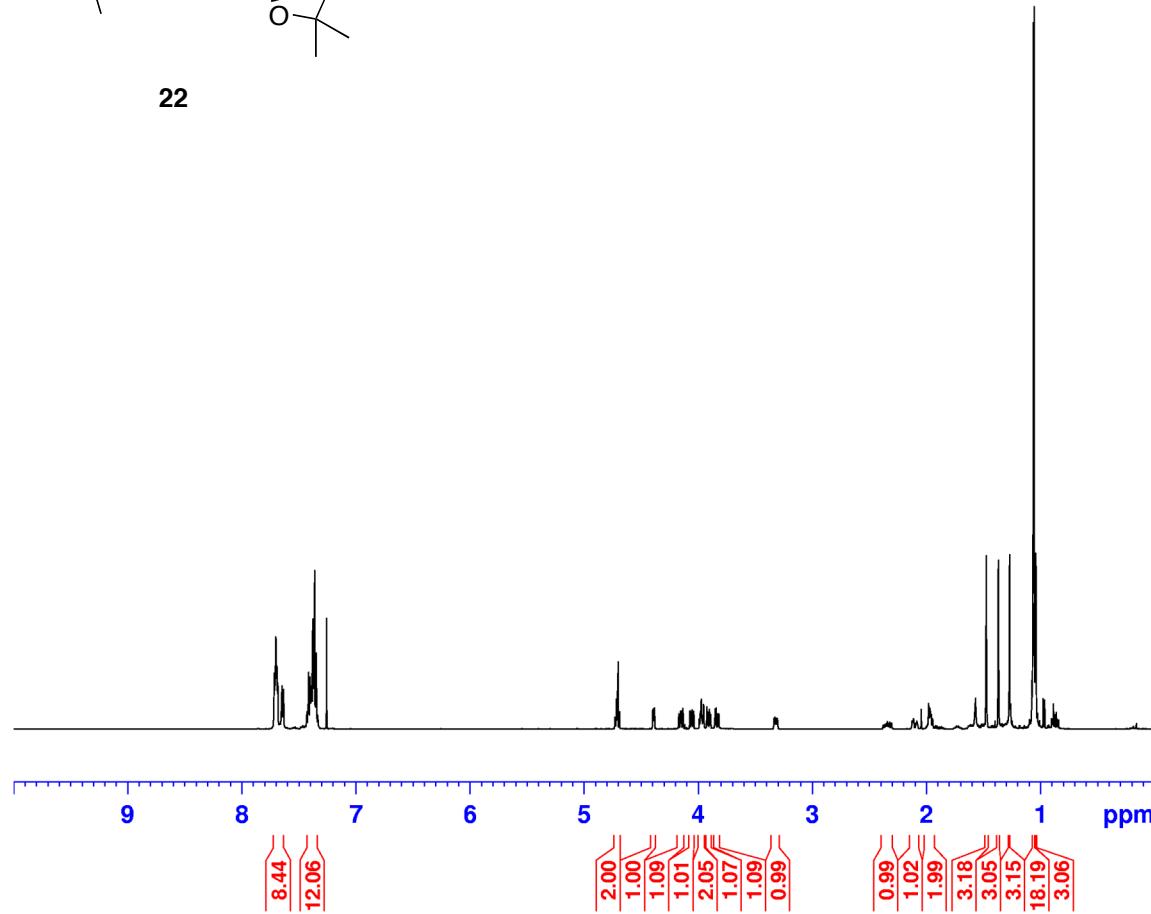
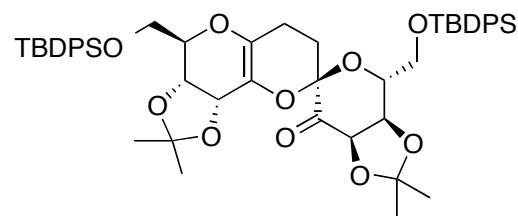
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21



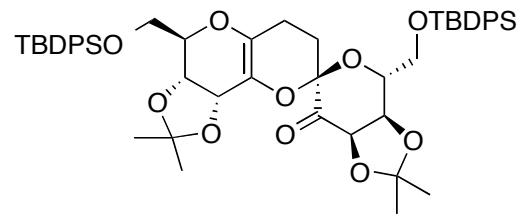


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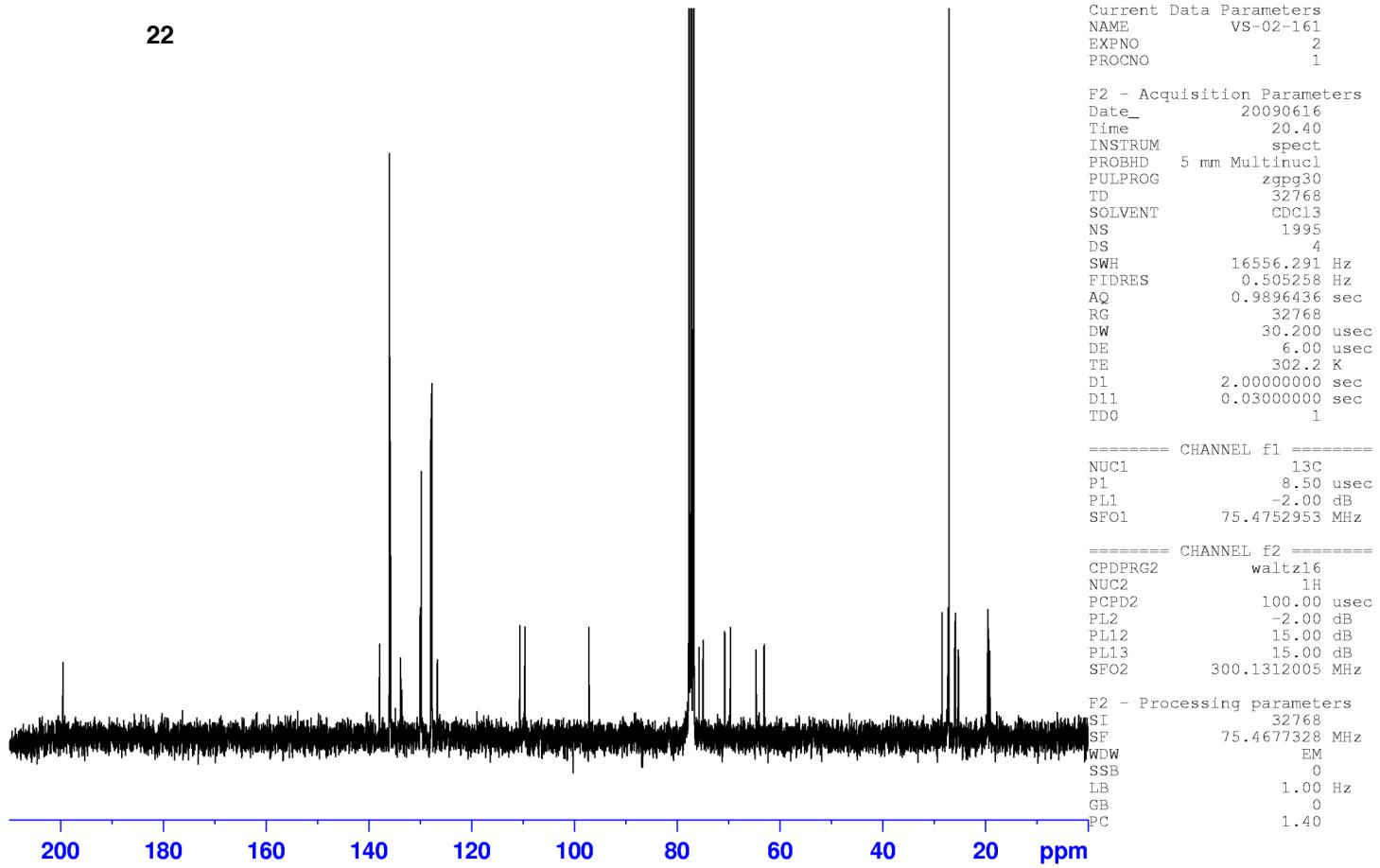
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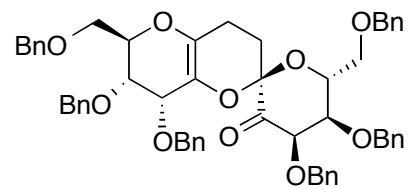
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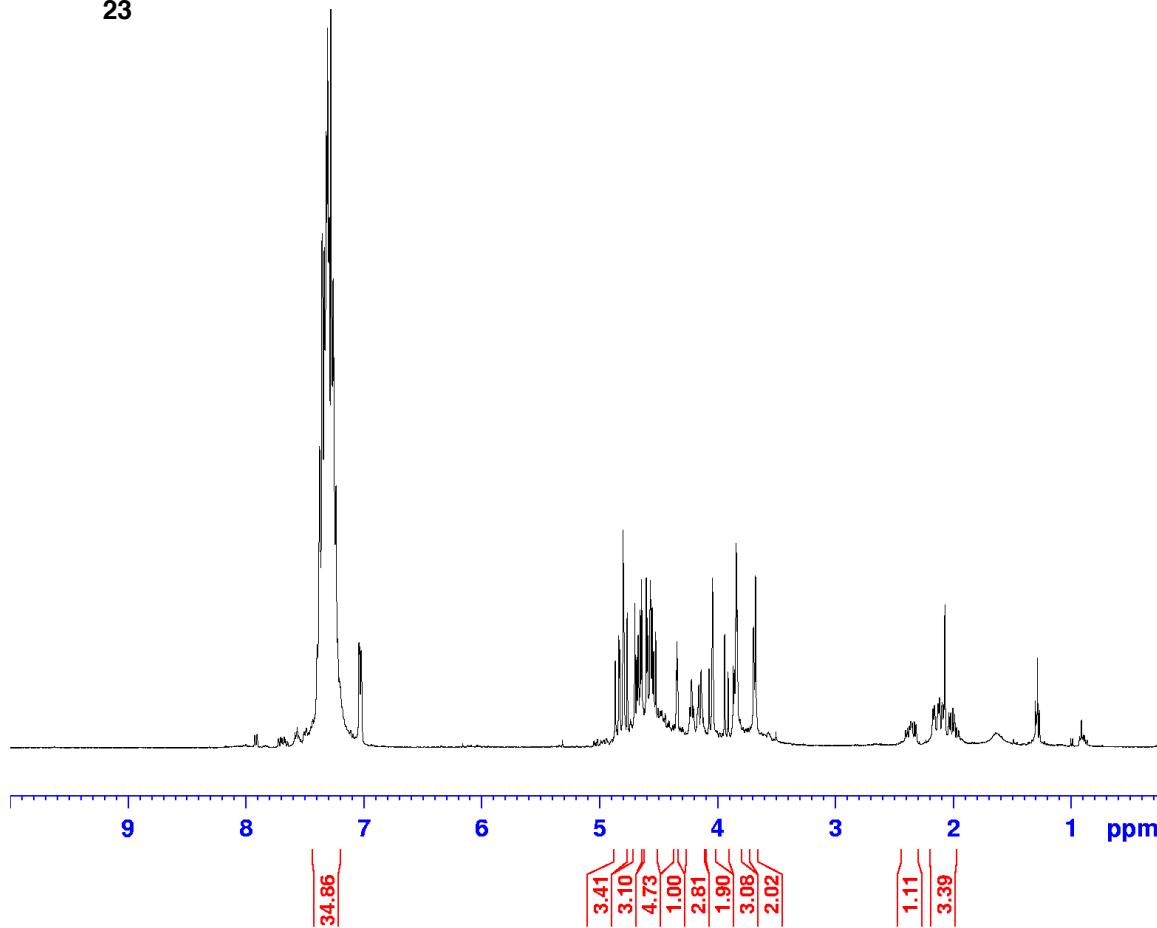


22





23

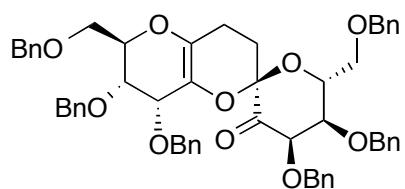


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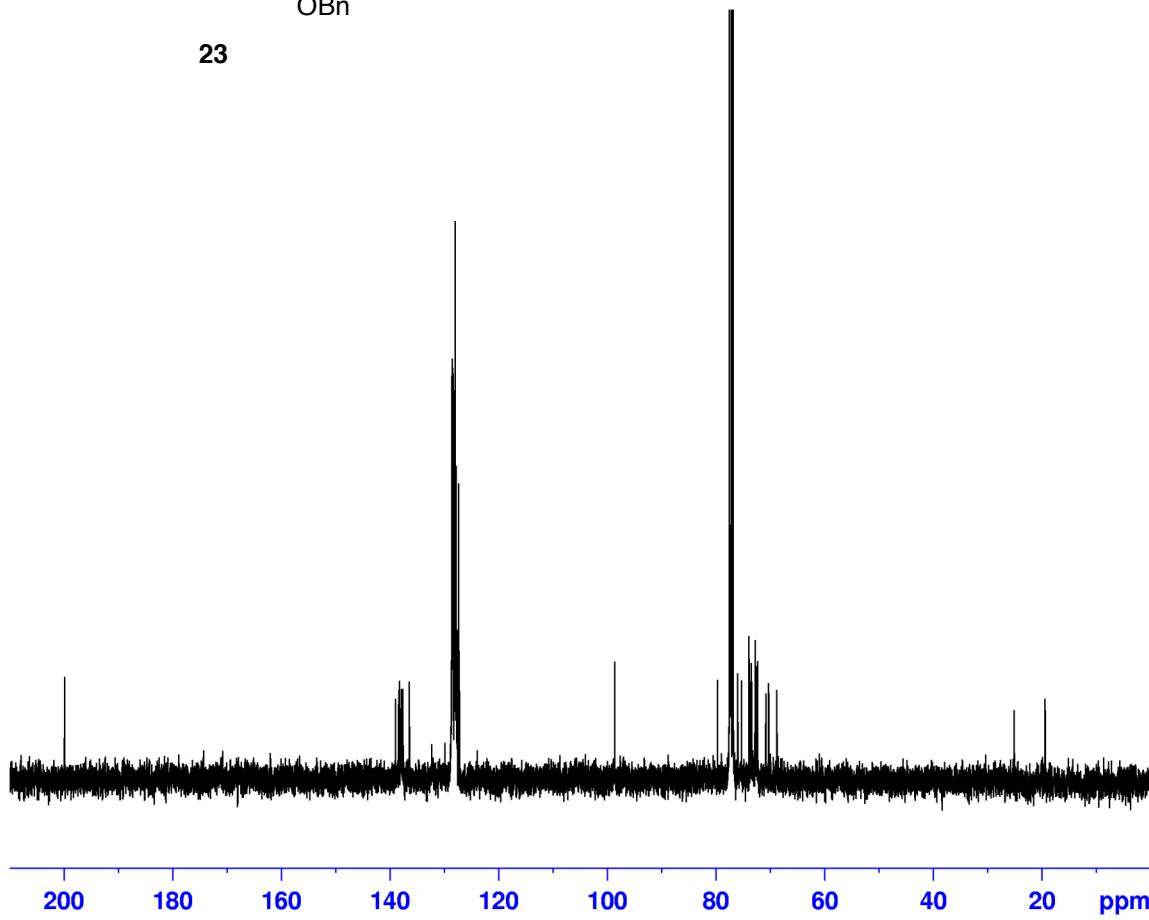
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23

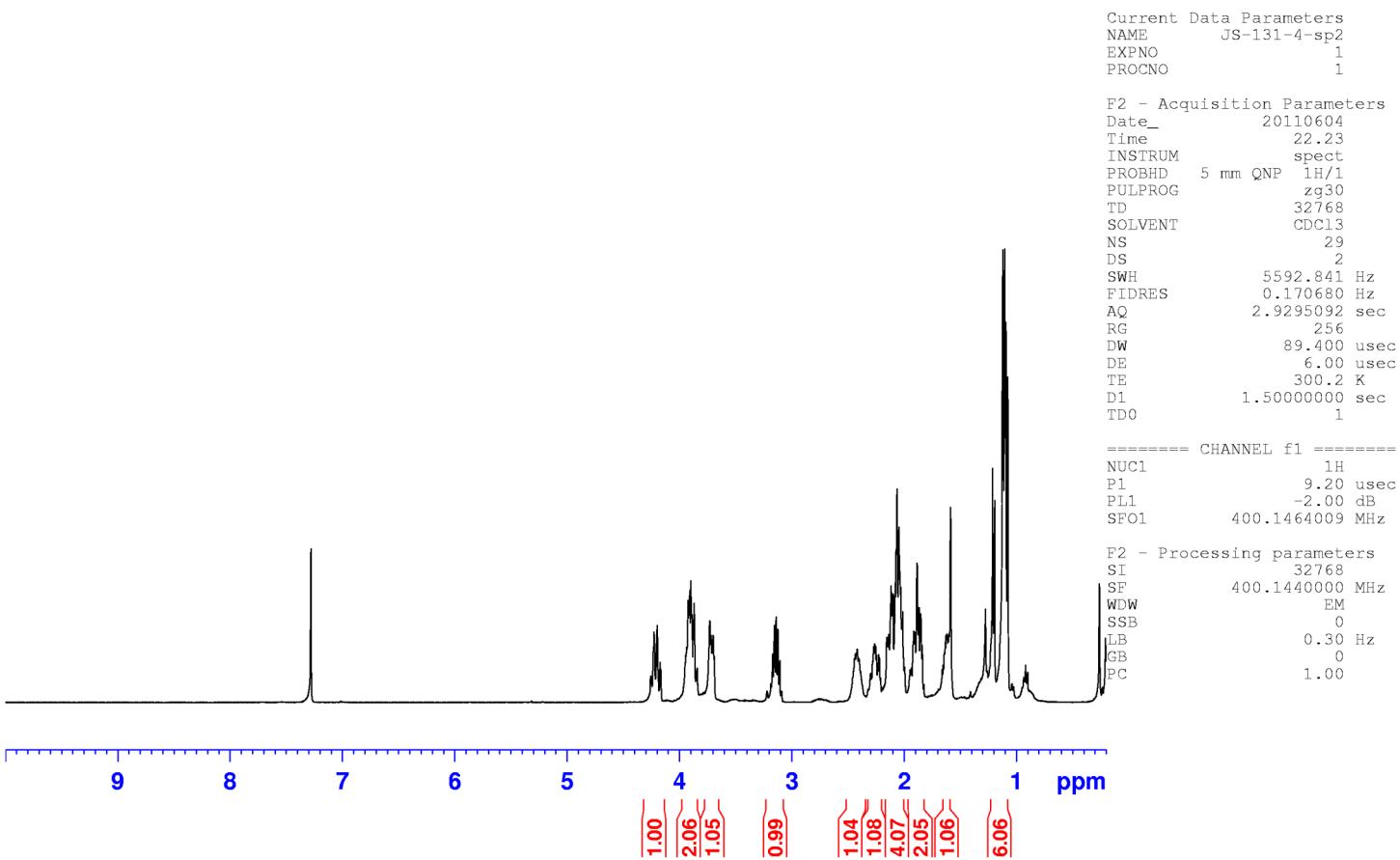
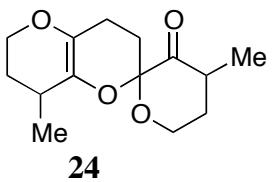


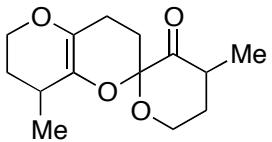
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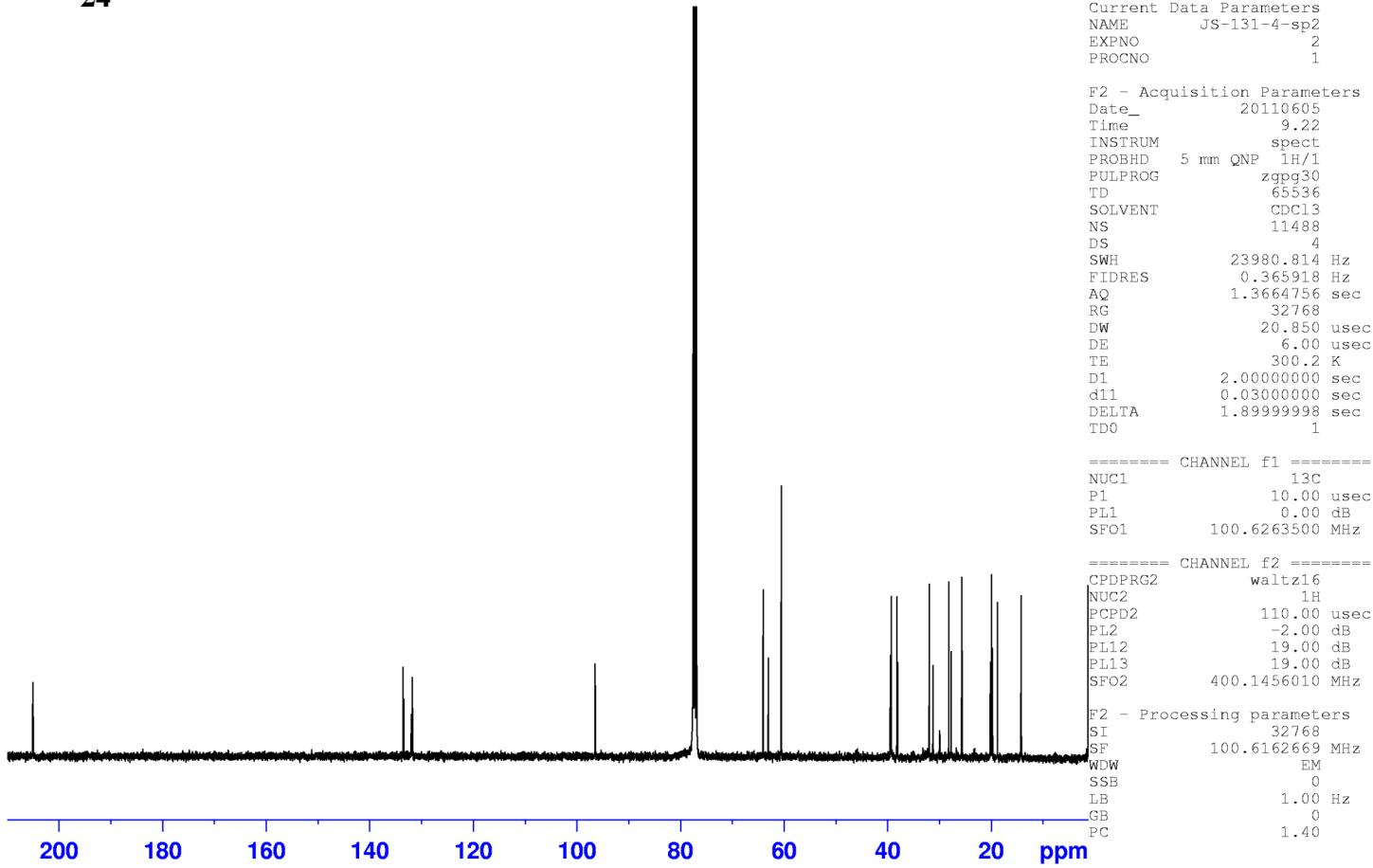
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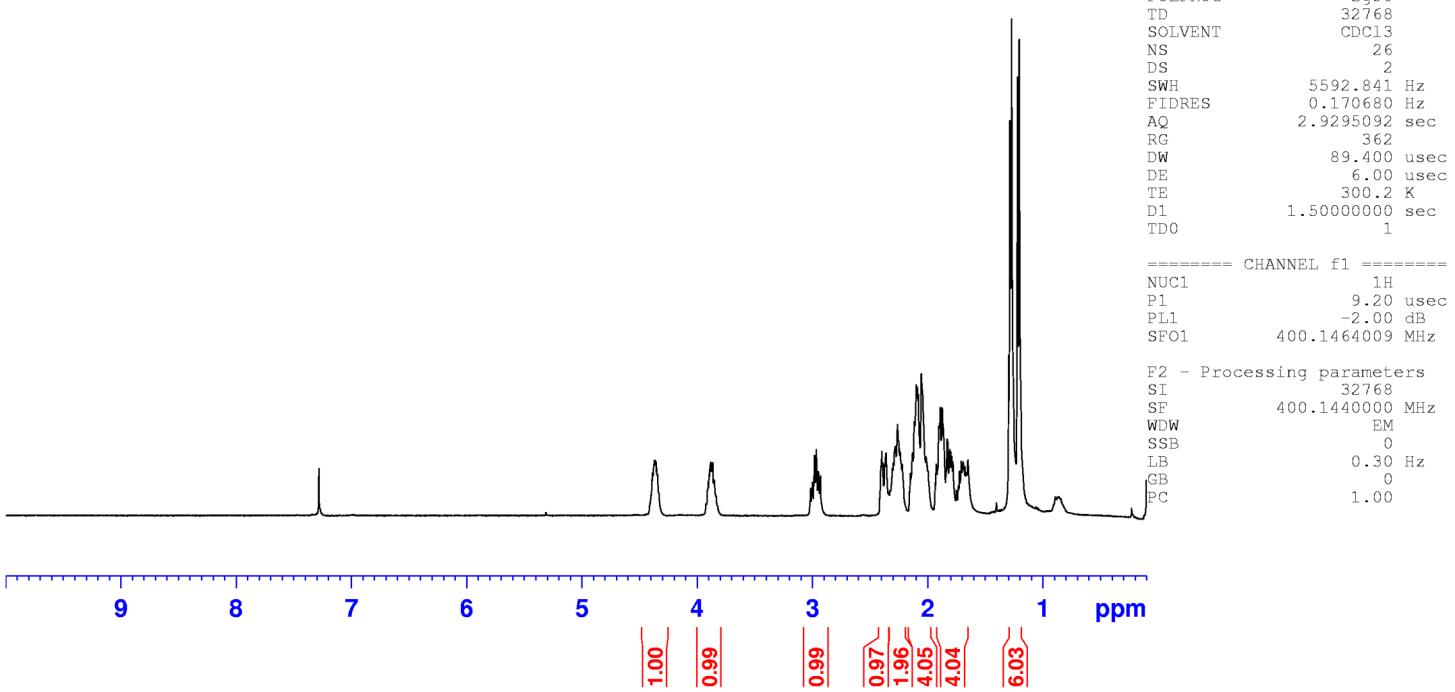
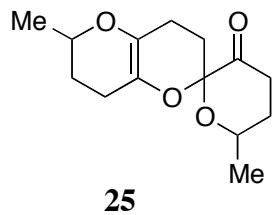
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24



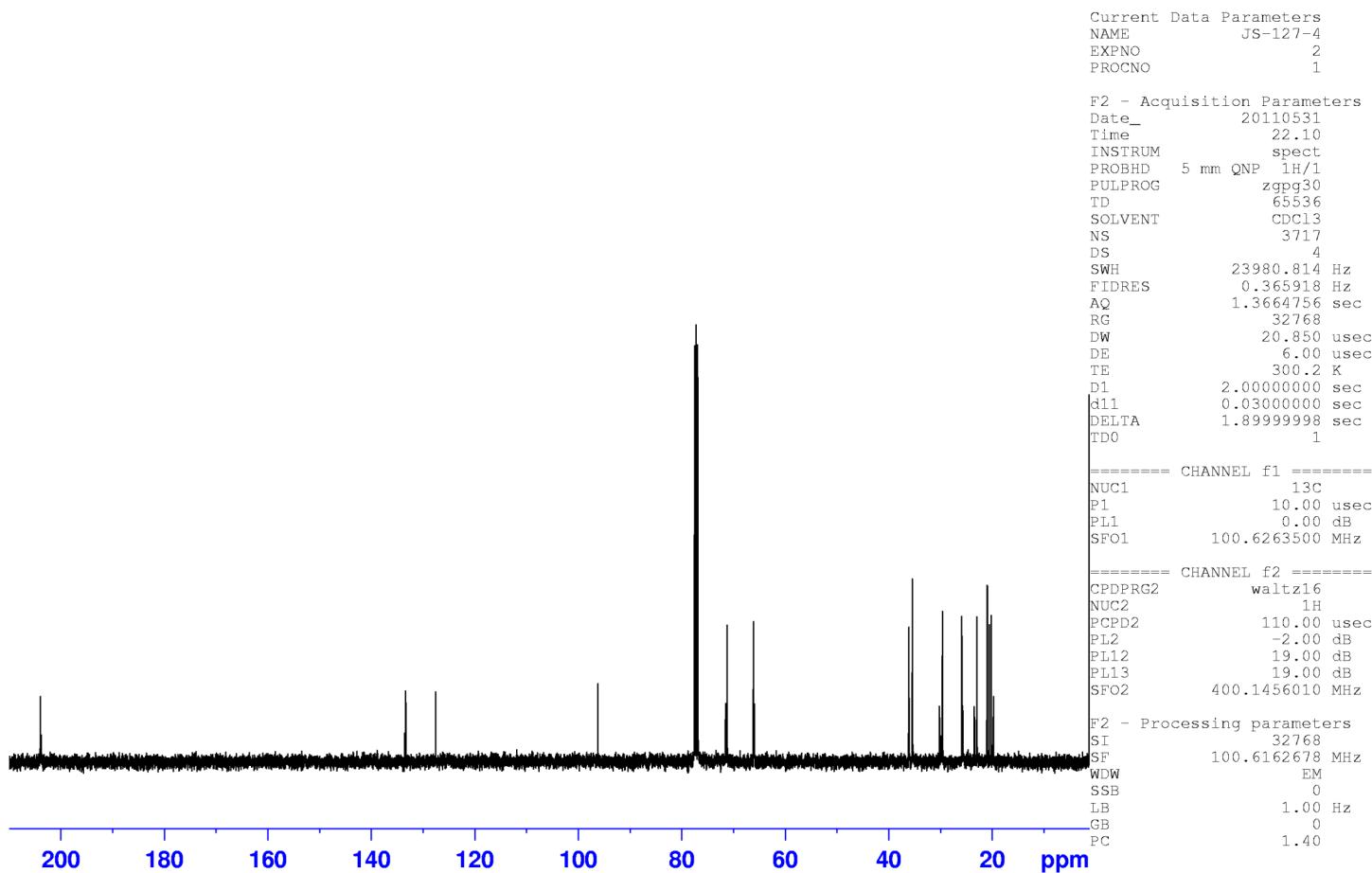
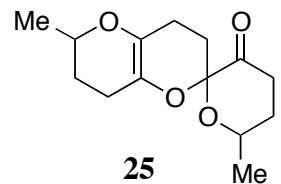


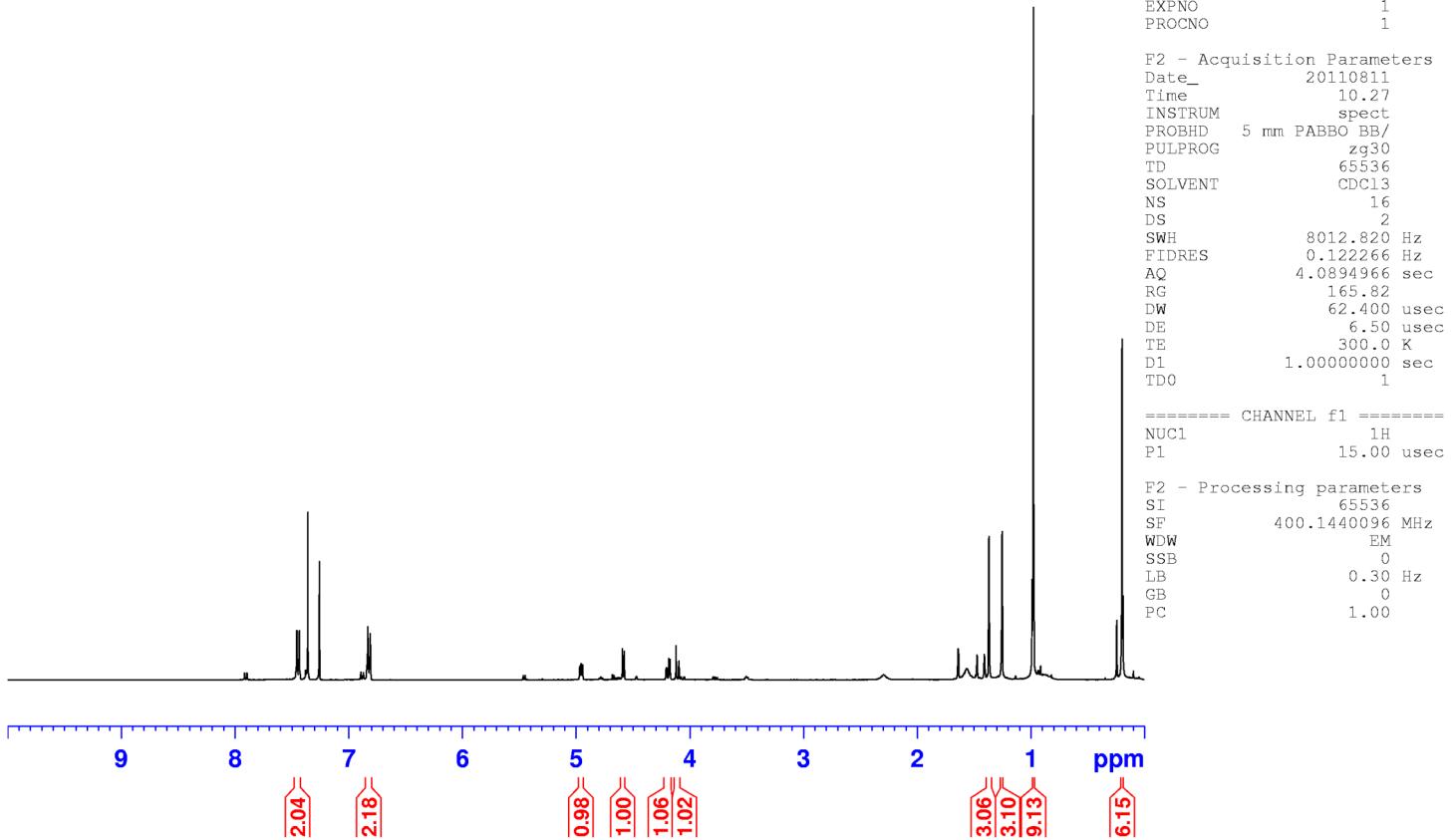
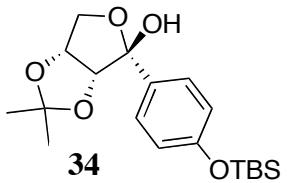
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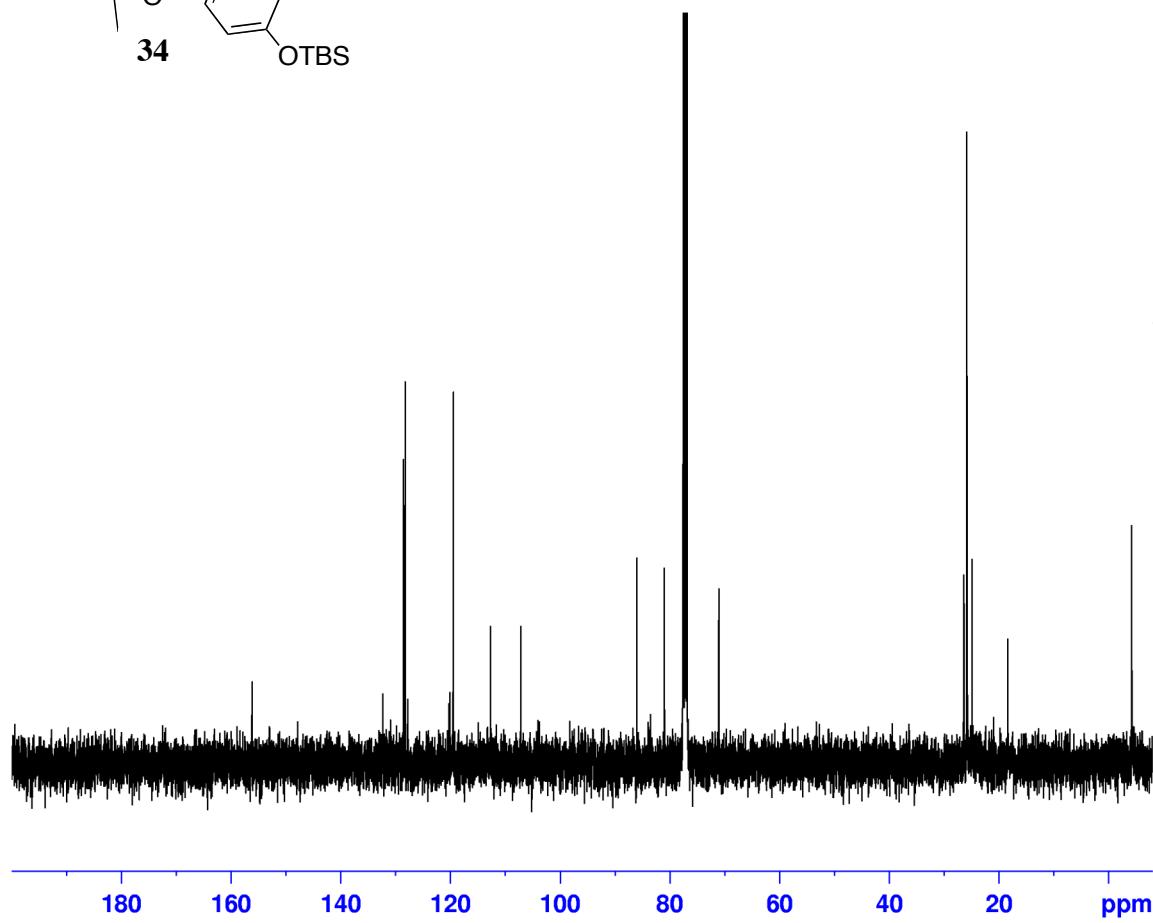
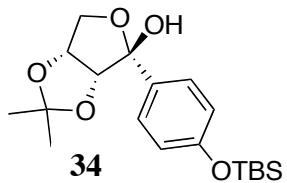
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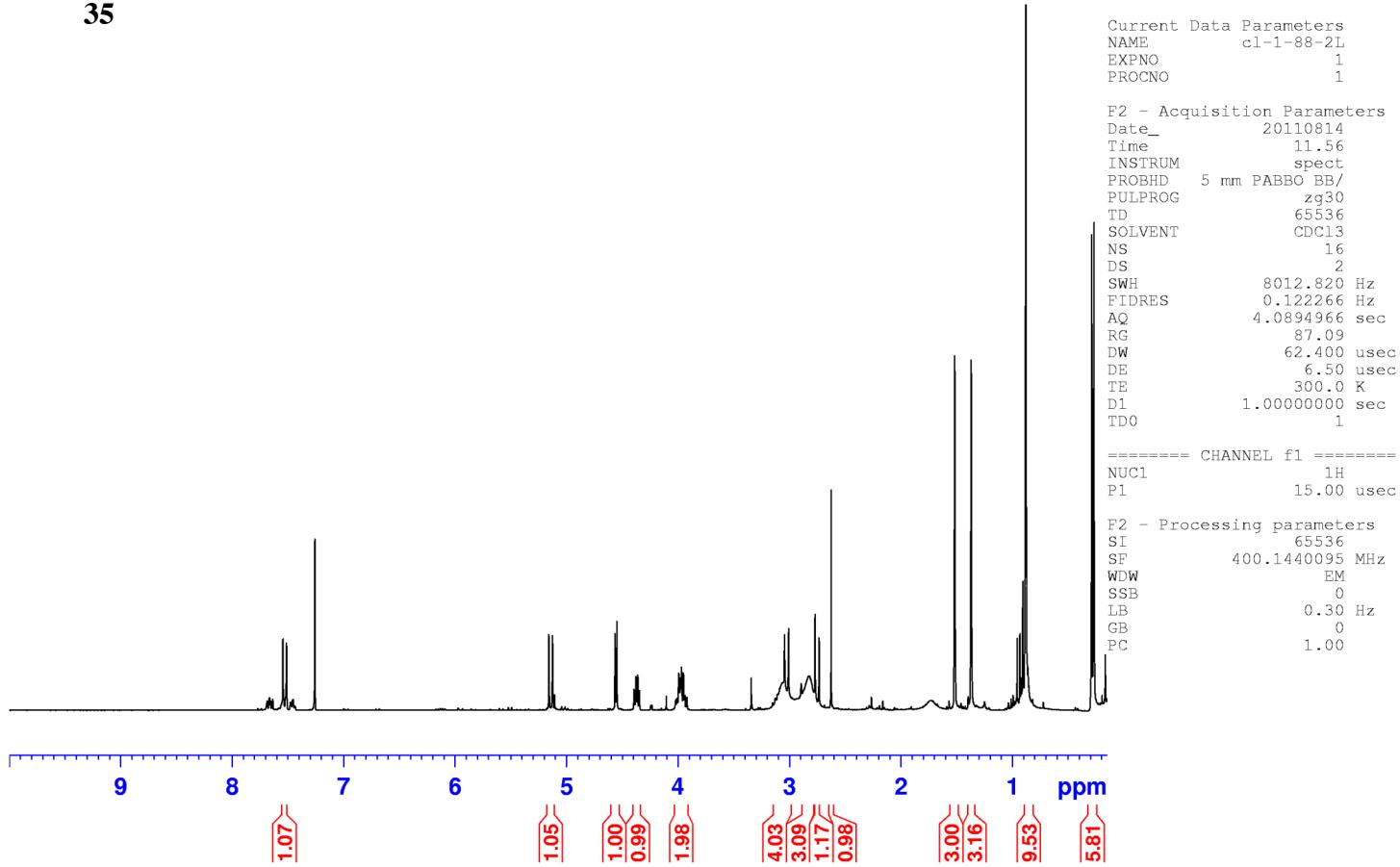
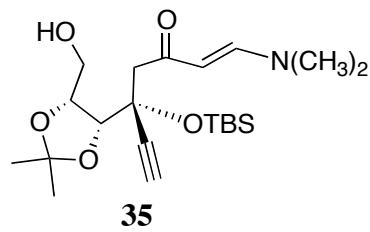


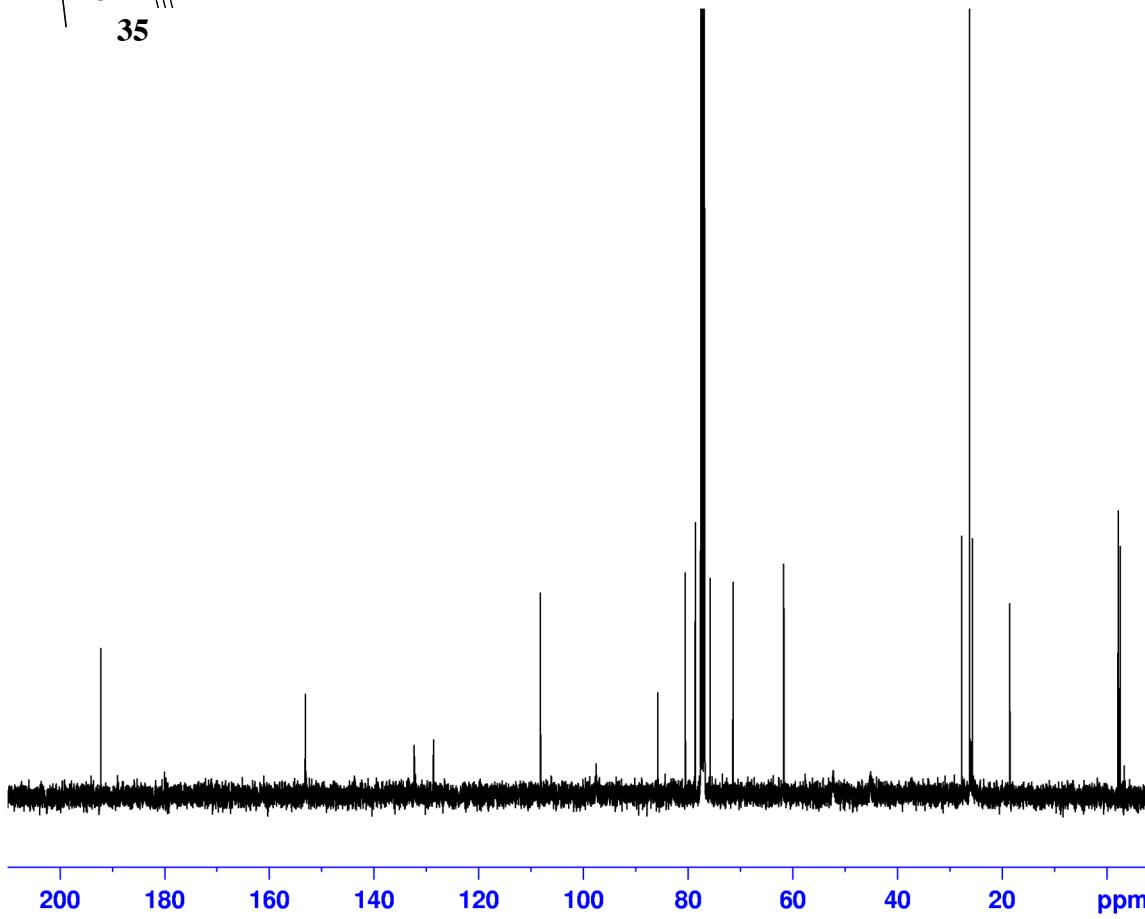
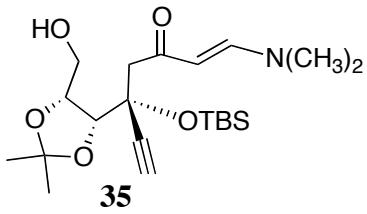
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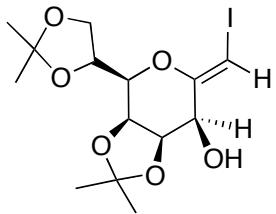


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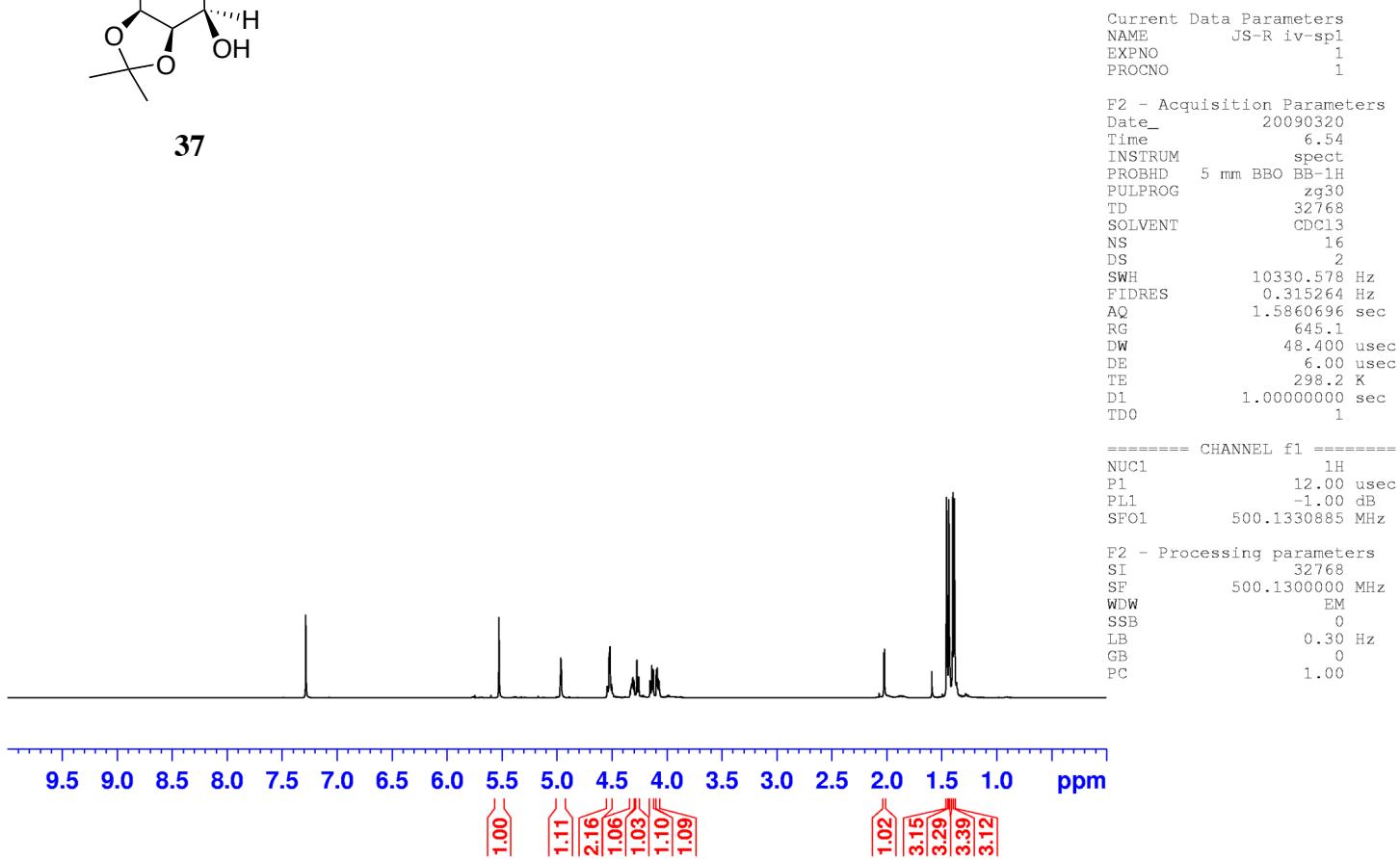
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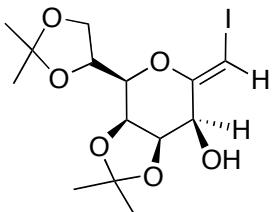
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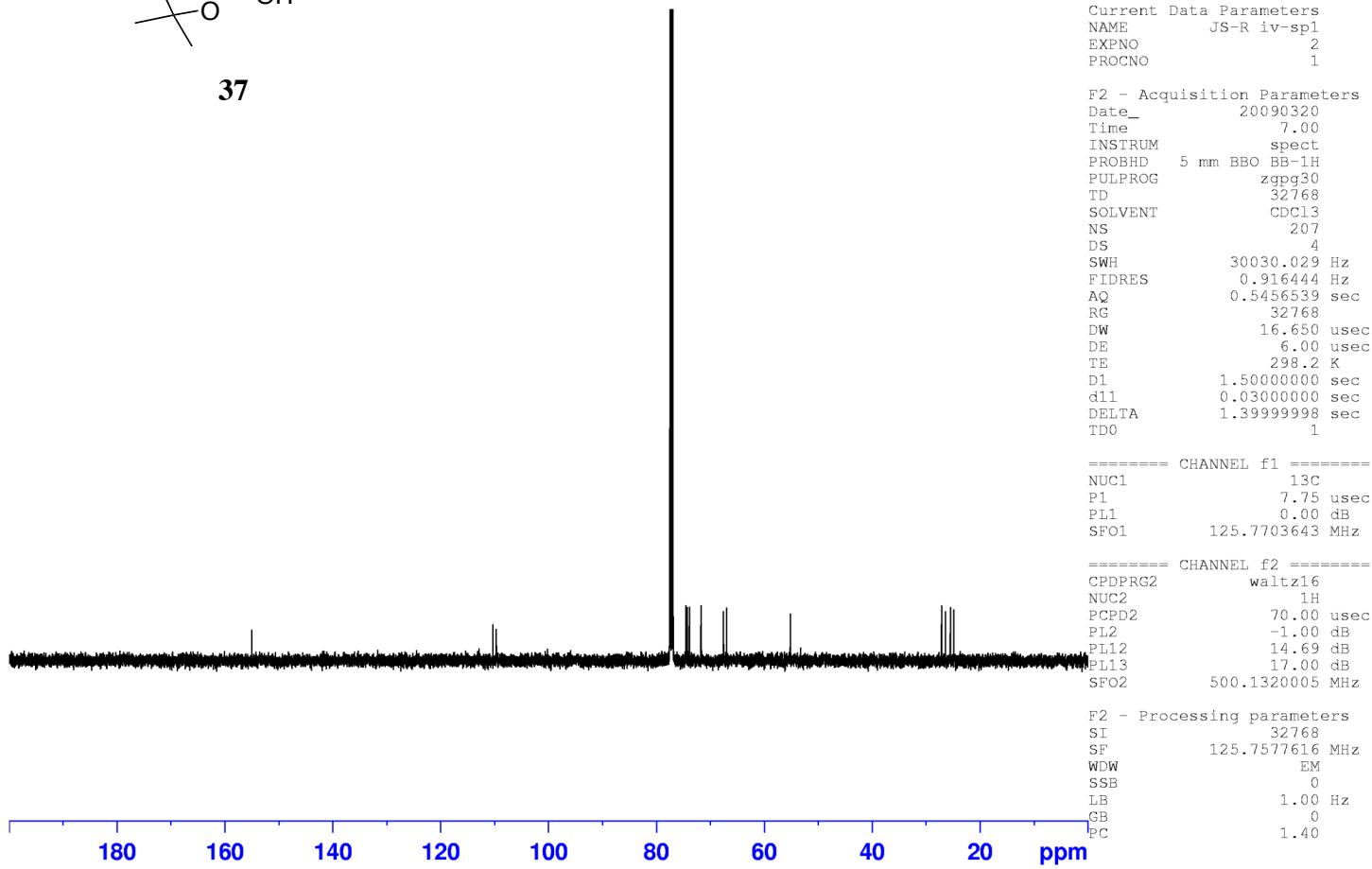


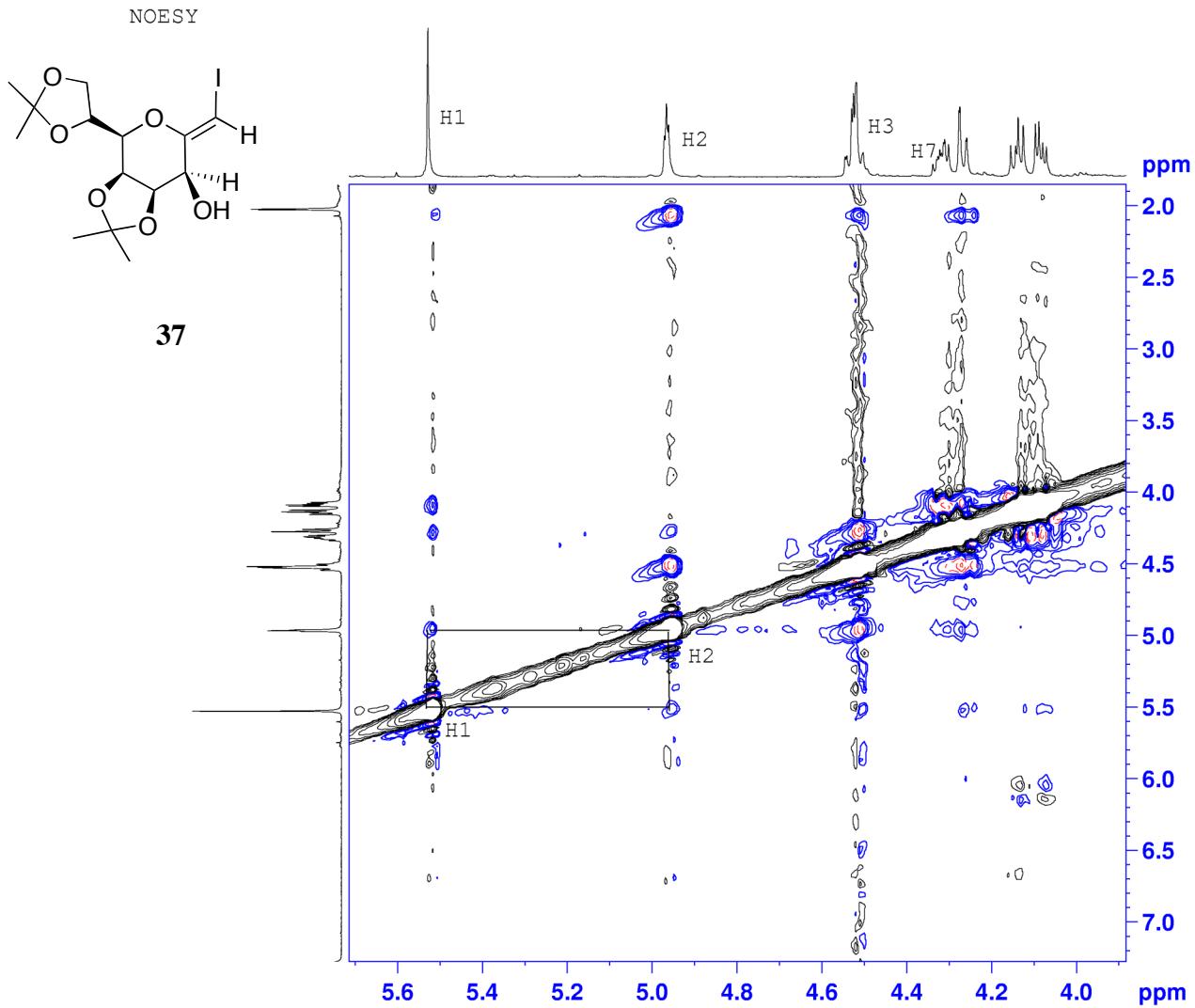
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37





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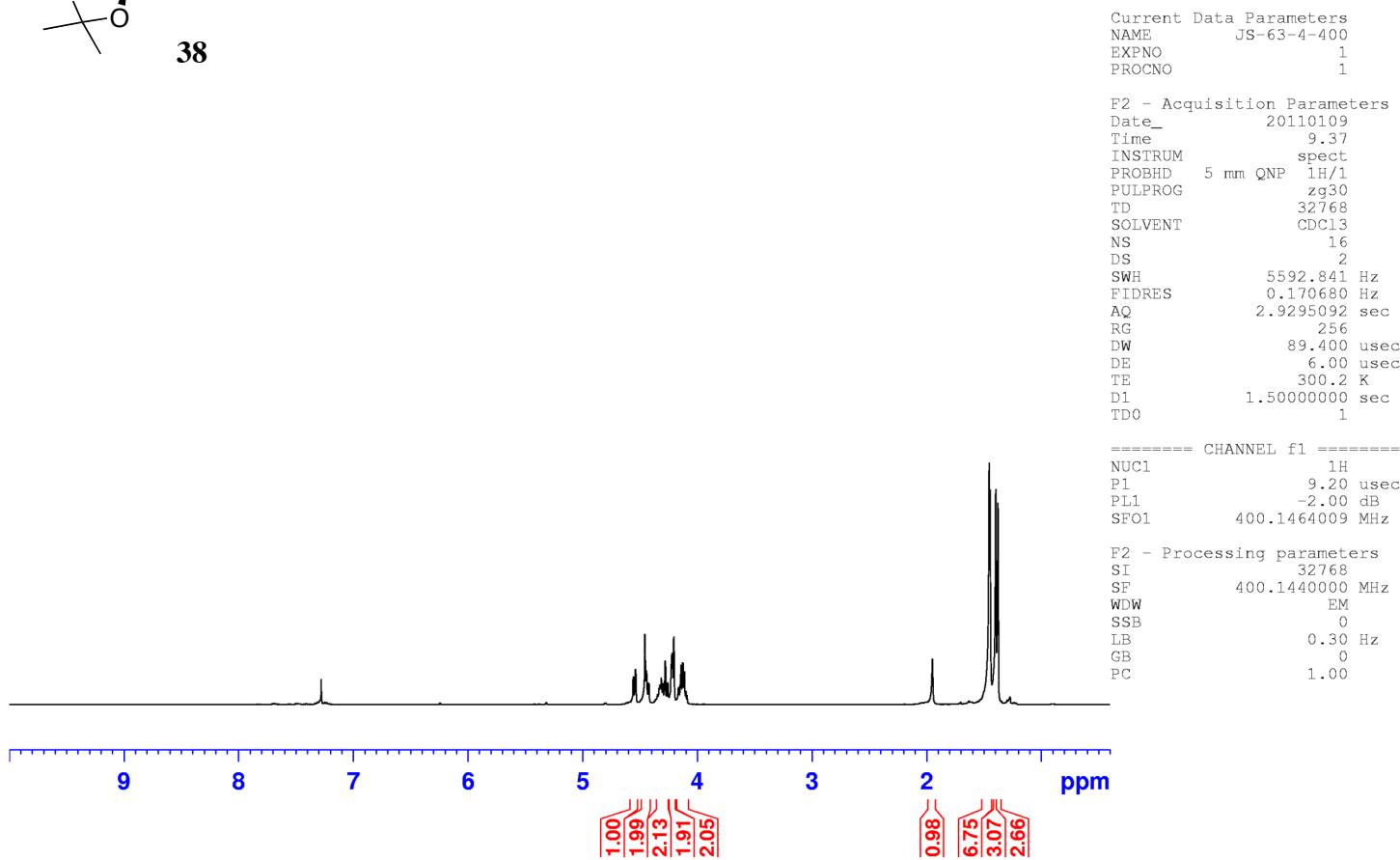
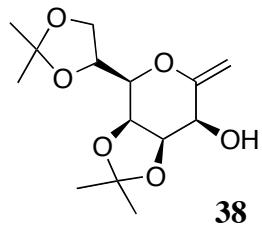
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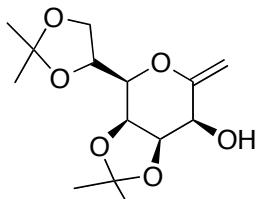
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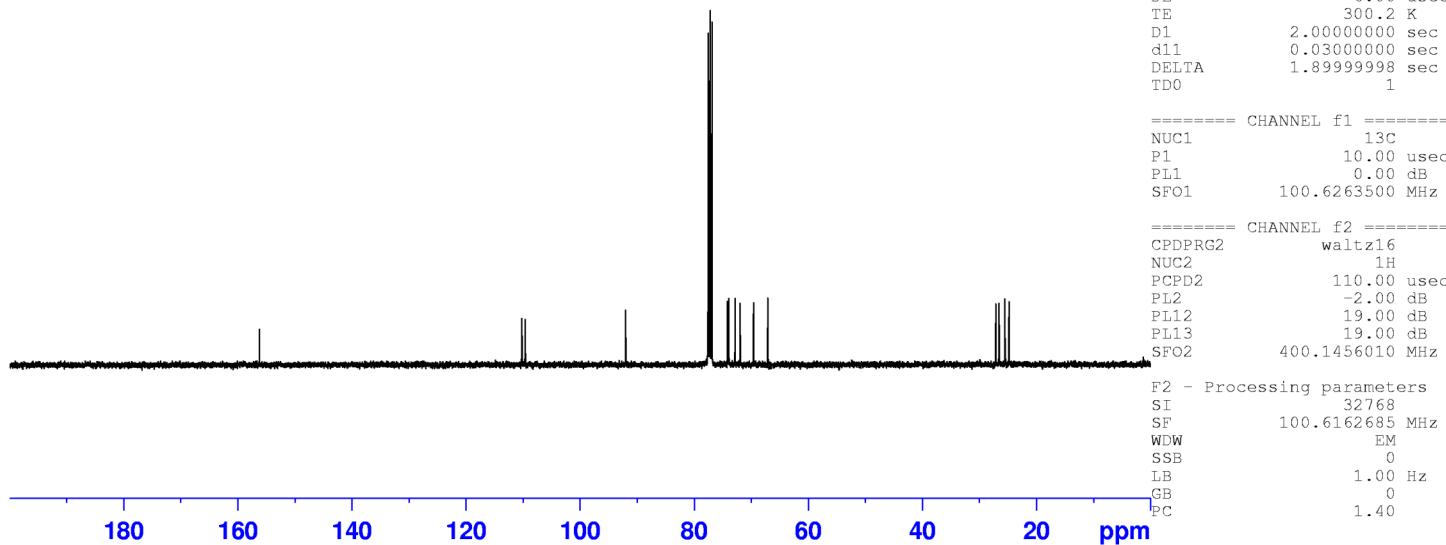
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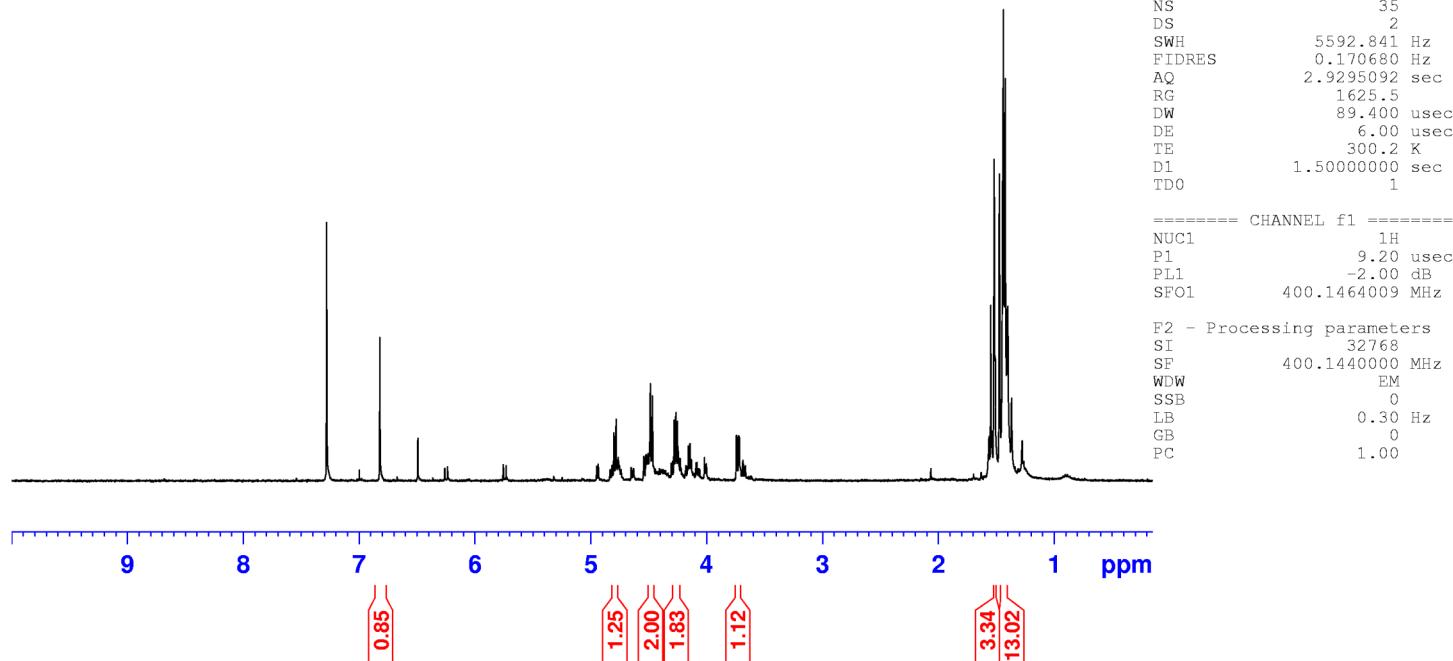
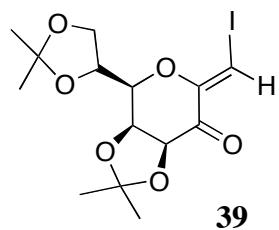
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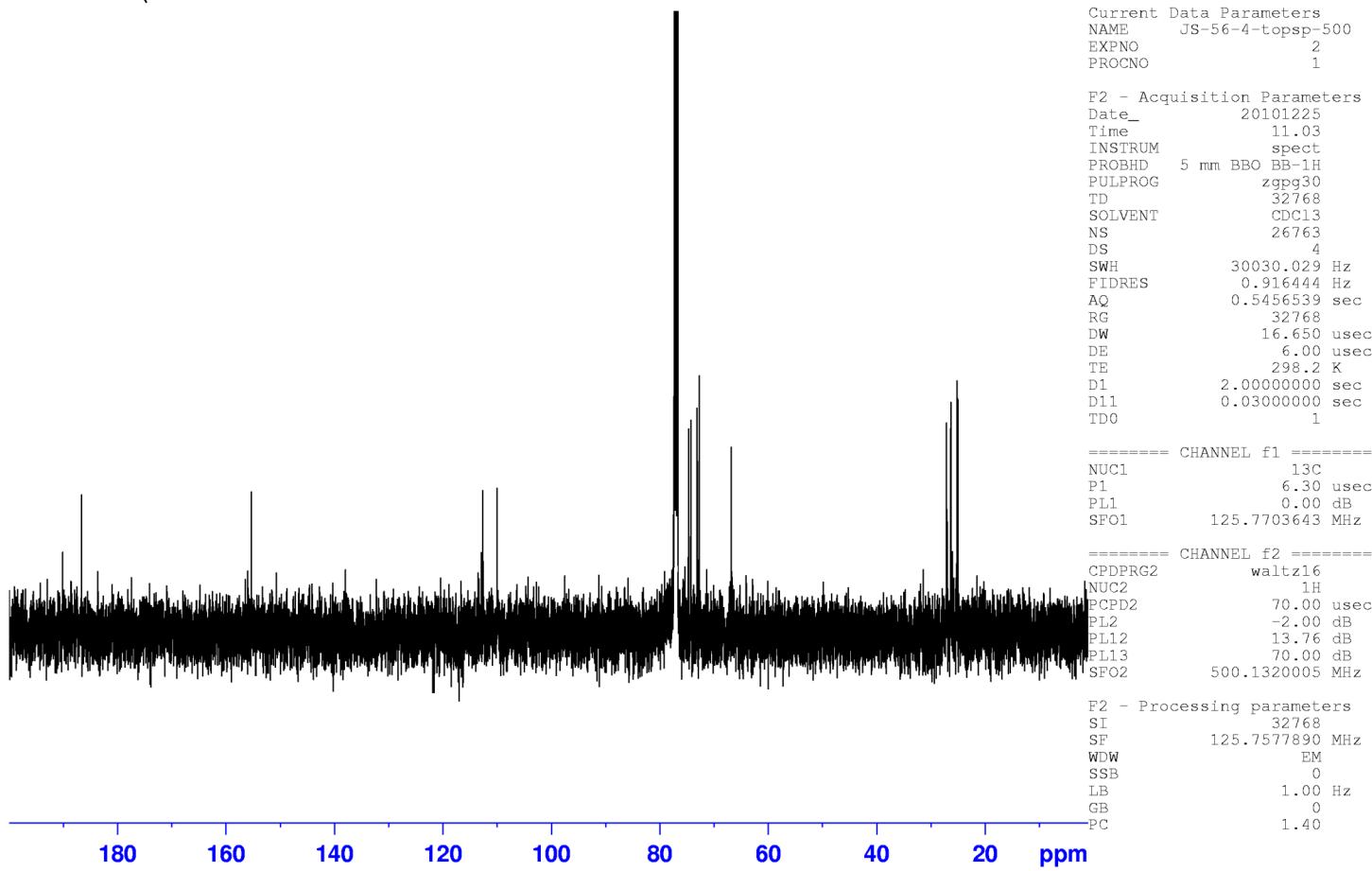
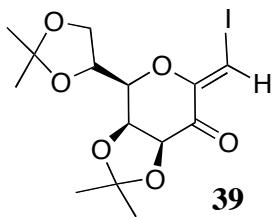


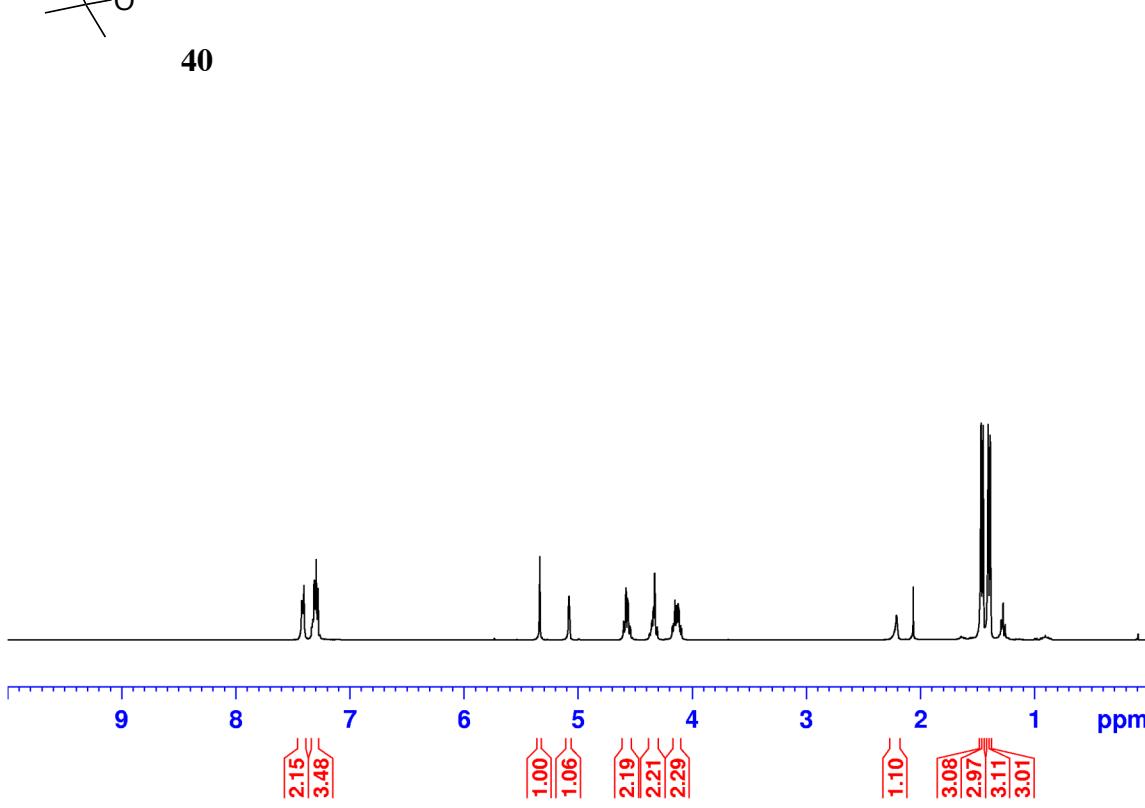
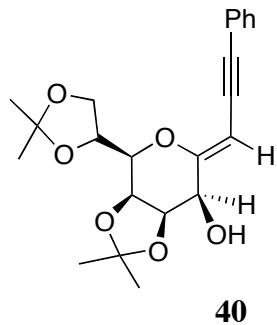


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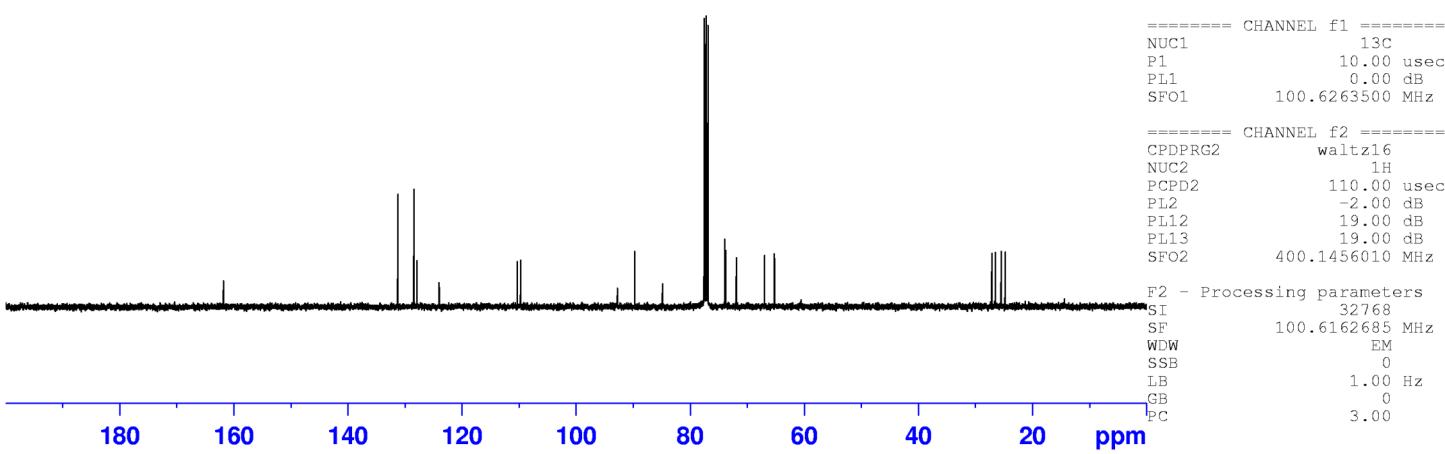
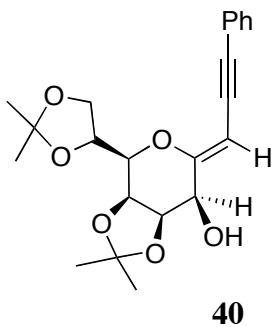


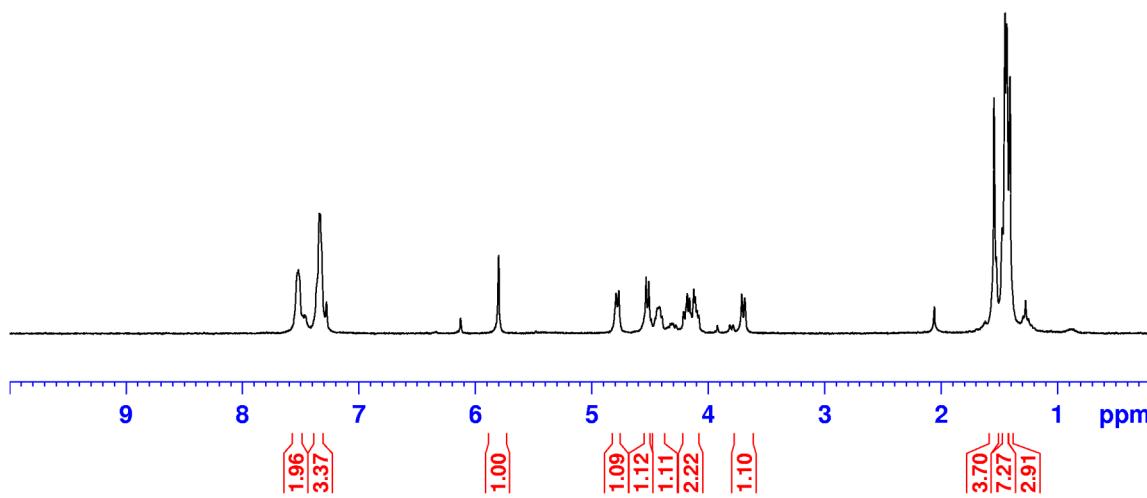
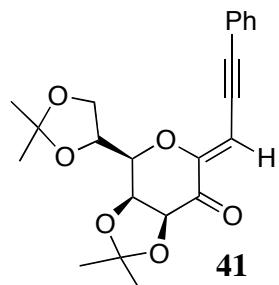
Current Data Parameters
NAME JS-54-4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101220
Time 22.20
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 23
DS 2
SWH 5592.841 Hz
FIDRES 0.170680 Hz
AQ 2.9295092 sec
RG 256
DW 89.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.5000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.20 usec
PL1 -2.00 dB
SFO1 400.1464009 MHz

F2 - Processing parameters
SI 32768
SF 400.1440000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 10.00



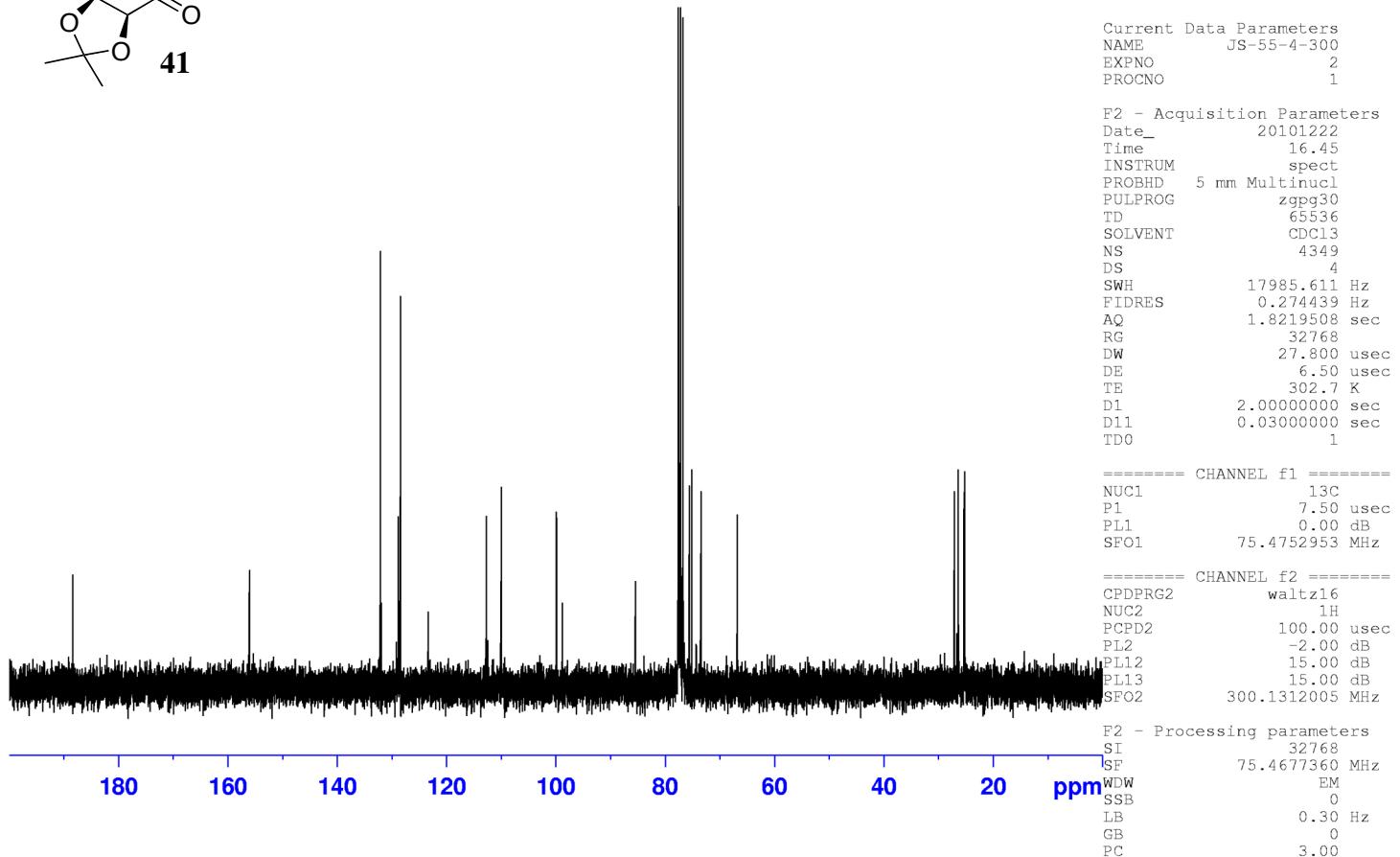
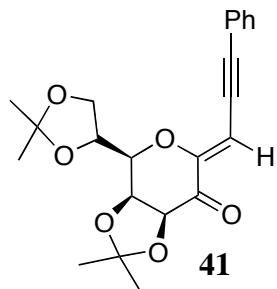


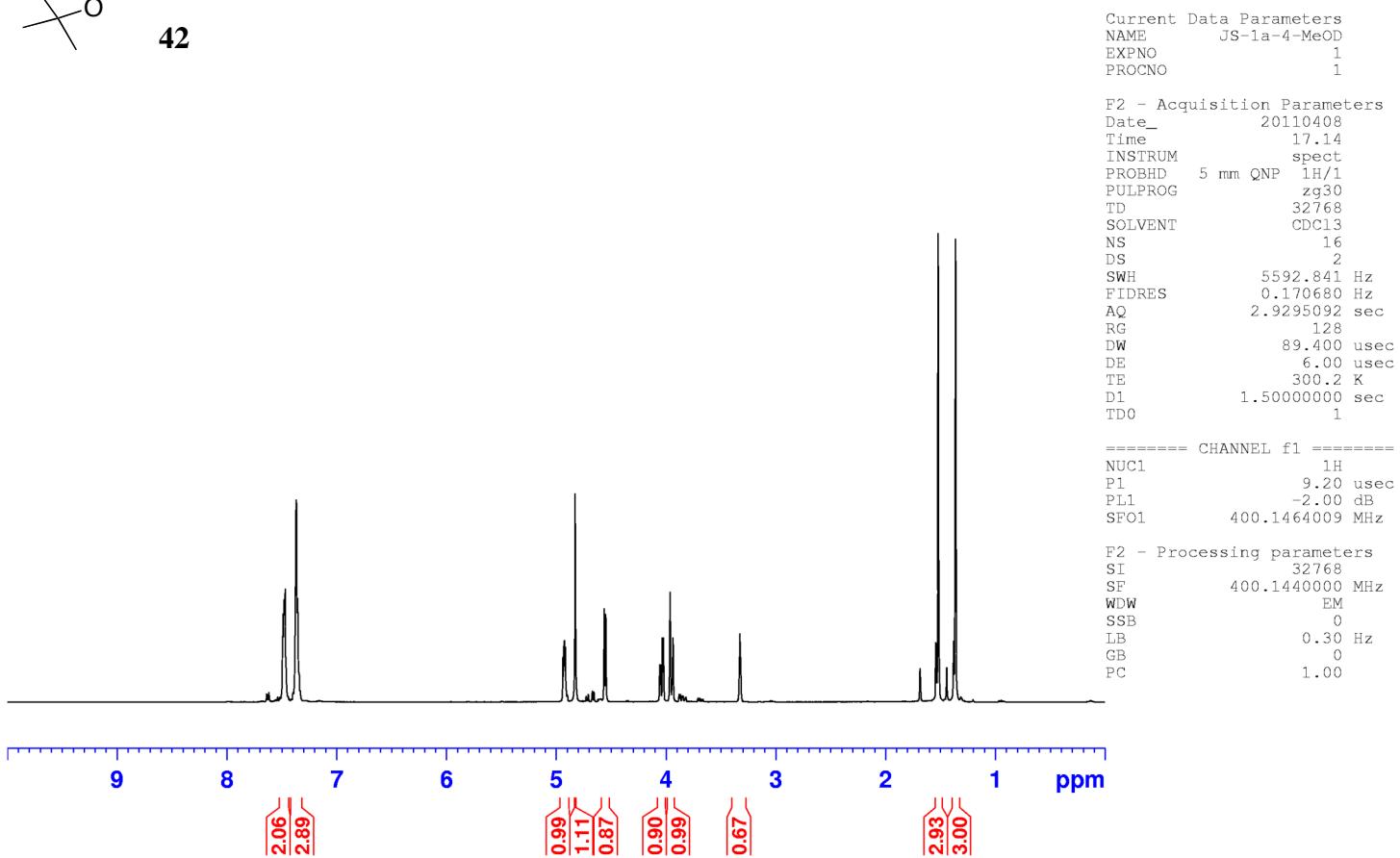
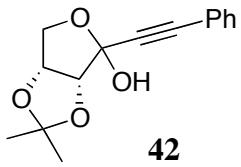
Current Data Parameters
NAME JS-55-4-300
Time 16.28
EXPNO 1
PROCNO 1

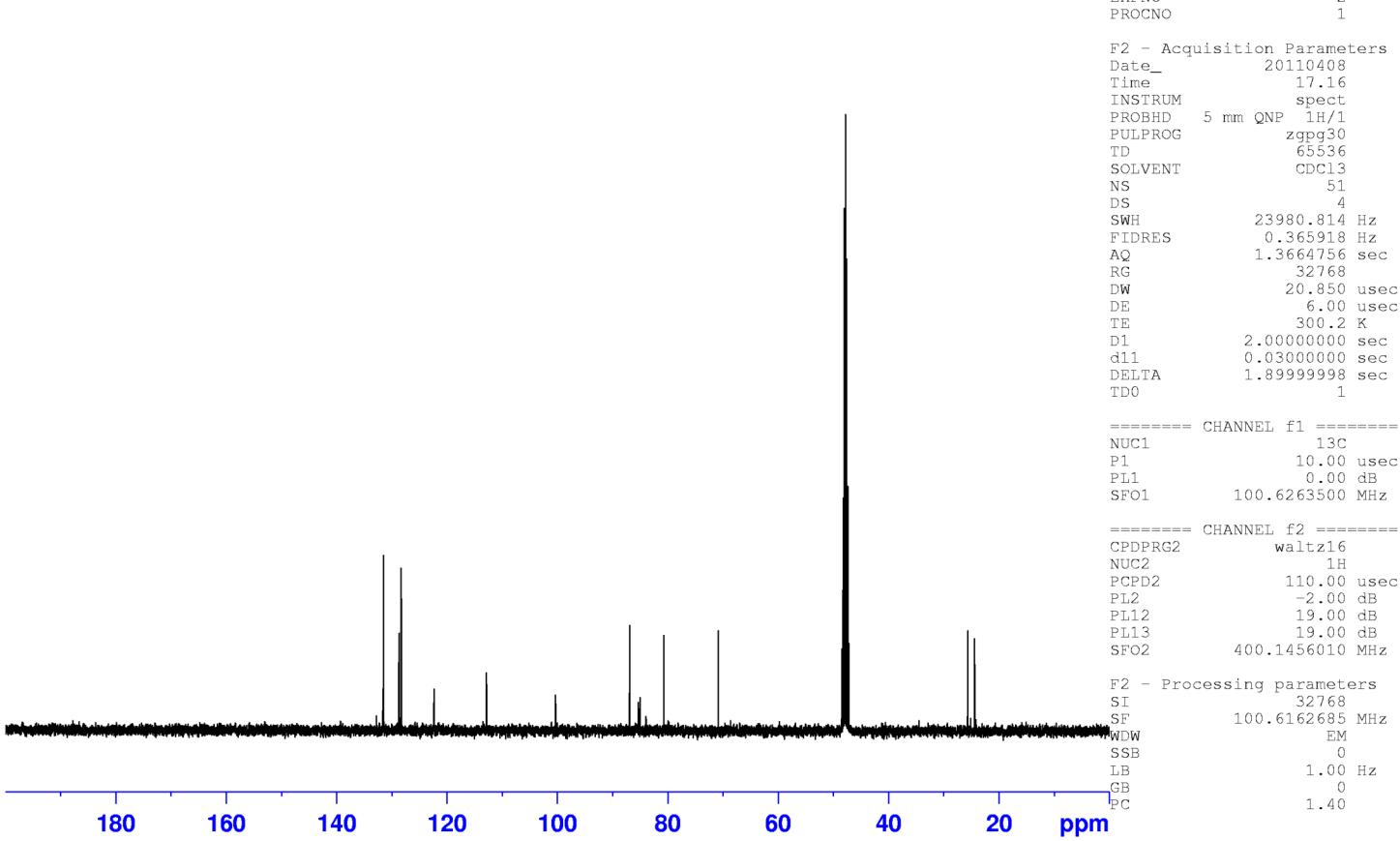
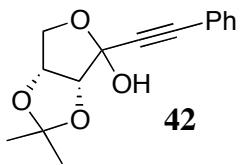
F2 - Acquisition Parameters
Date_ 20101222
Time 16.28
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 27
DS 2
SWH 4496.403 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 111.3
DW 111.200 usec
DE 6.00 usec
TE 300.2 K
D1 1.5000000 sec
TDO 1

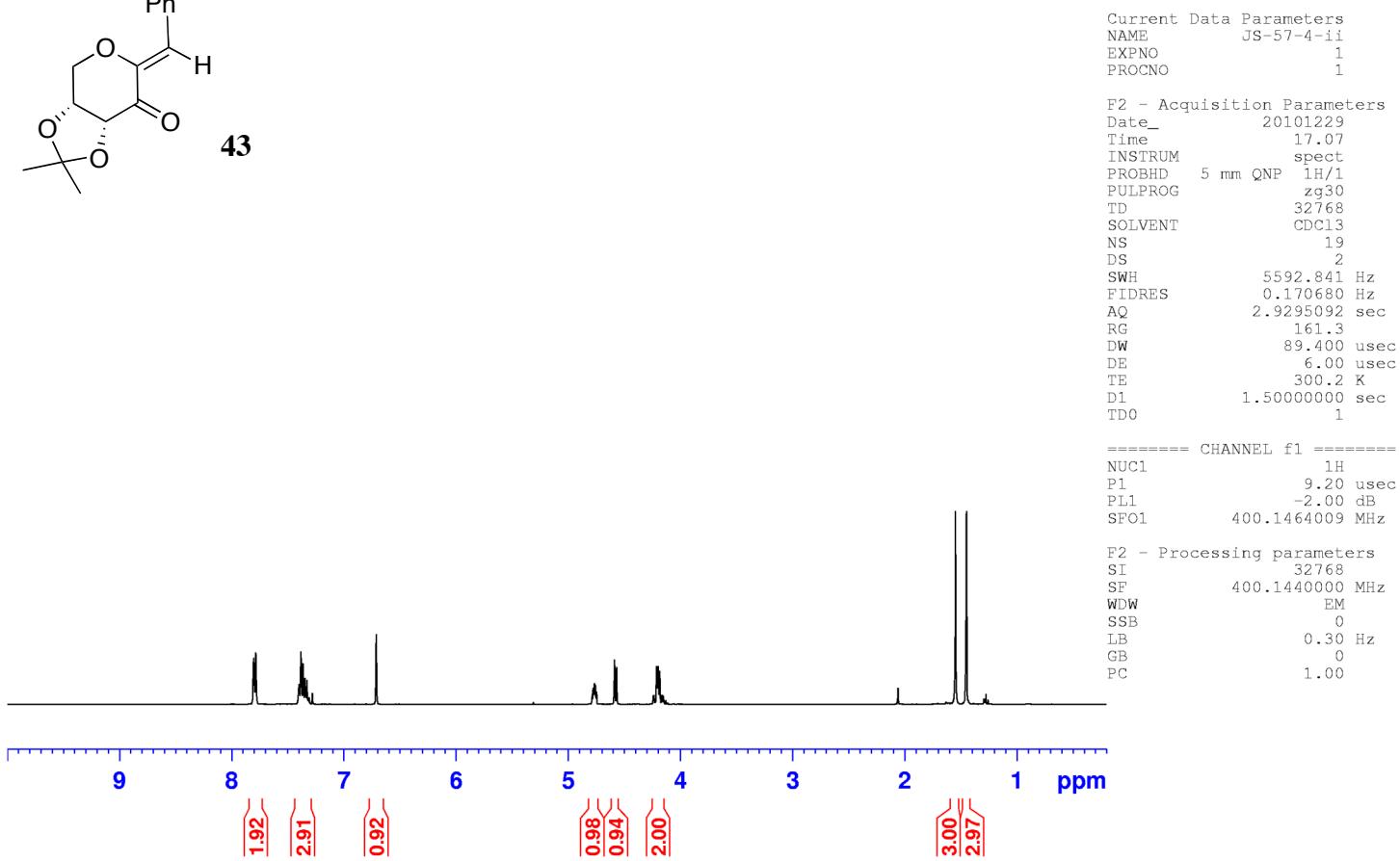
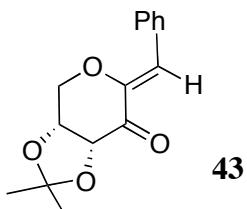
===== CHANNEL f1 =====
NUC1 1H
P1 8.20 usec
PL1 0.00 dB
SFO1 300.1318008 MHz

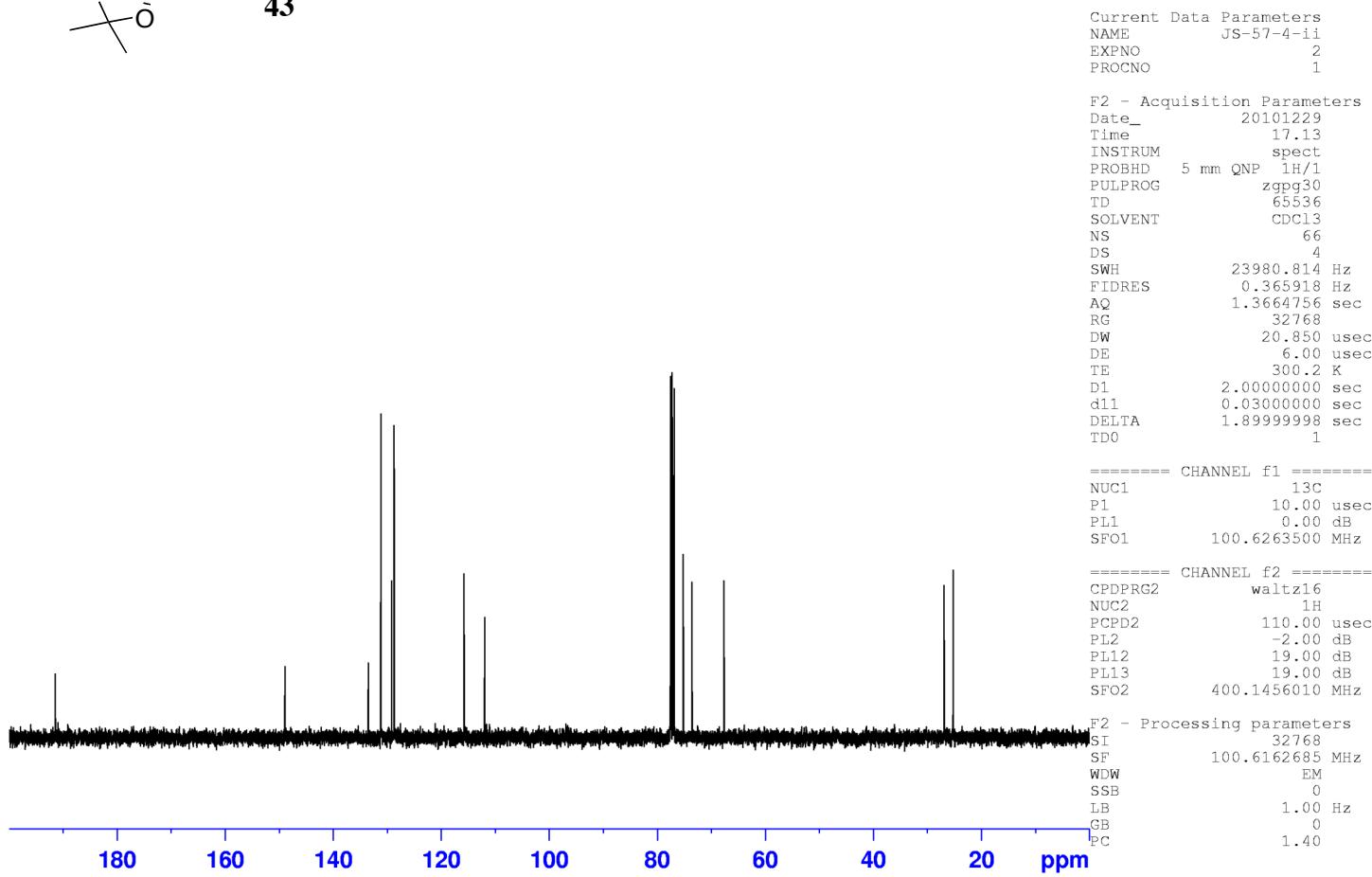
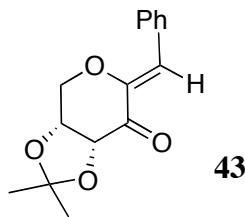
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

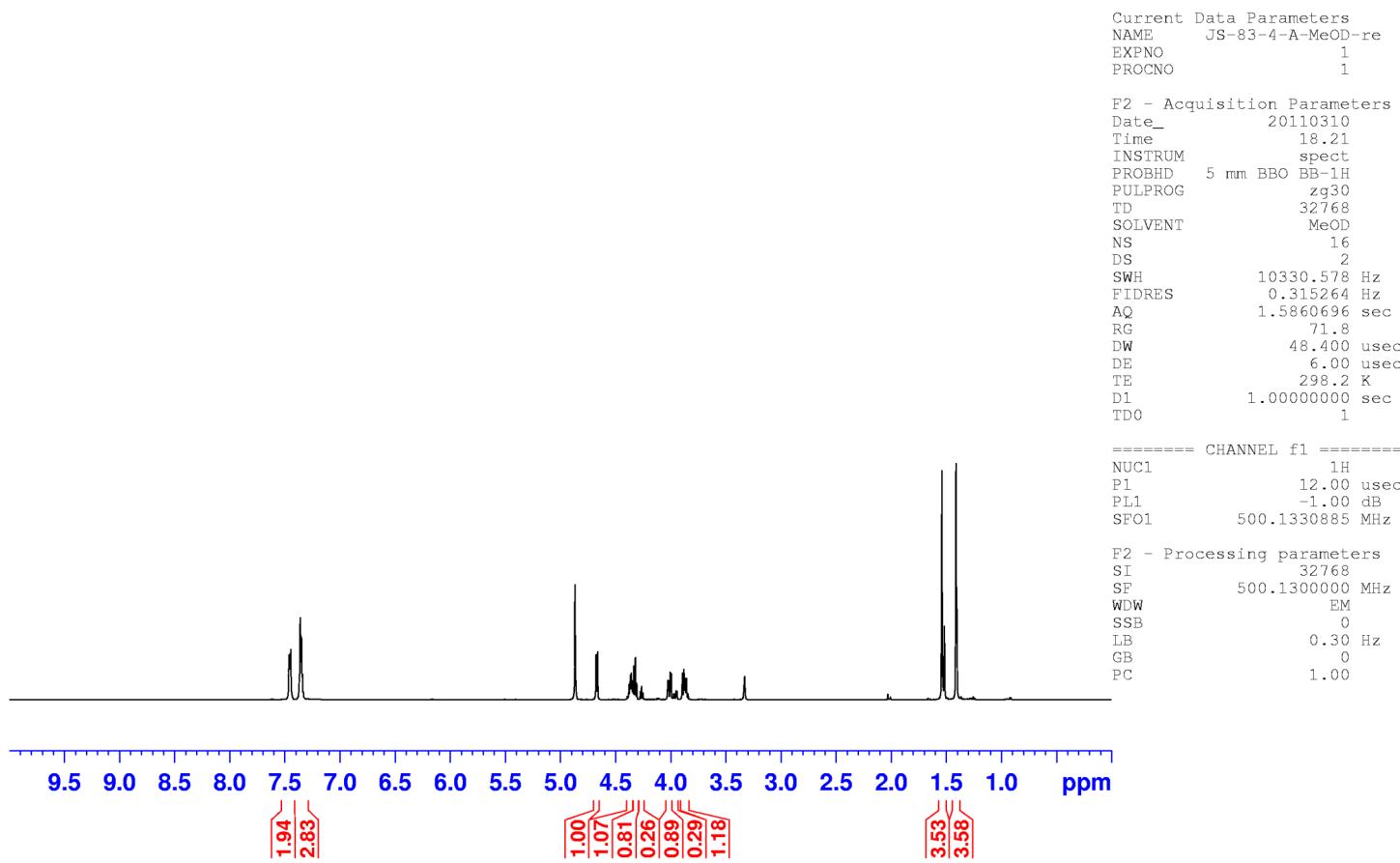
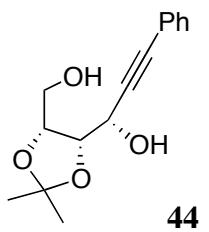


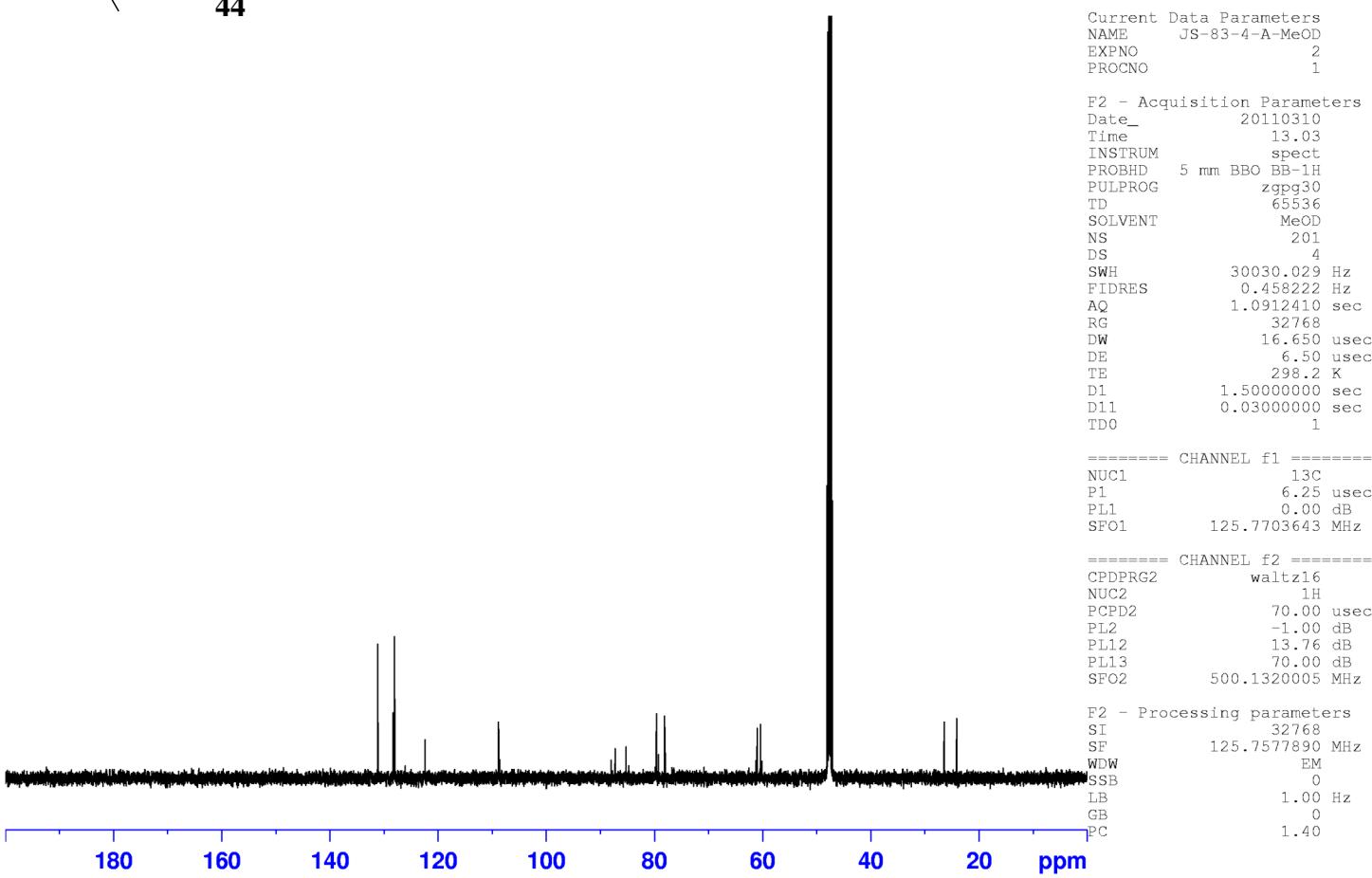
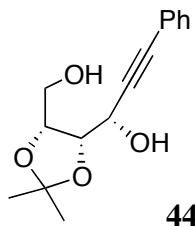


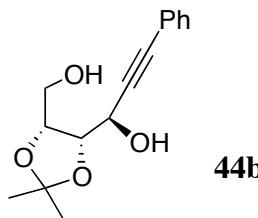










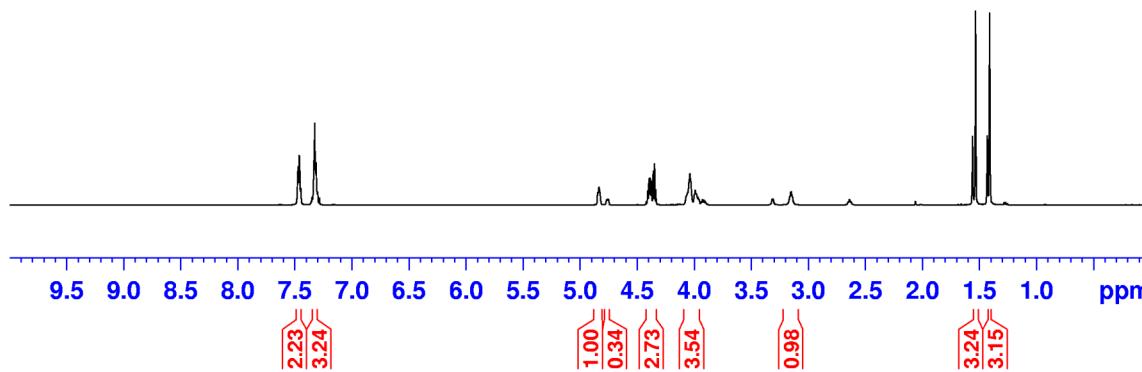


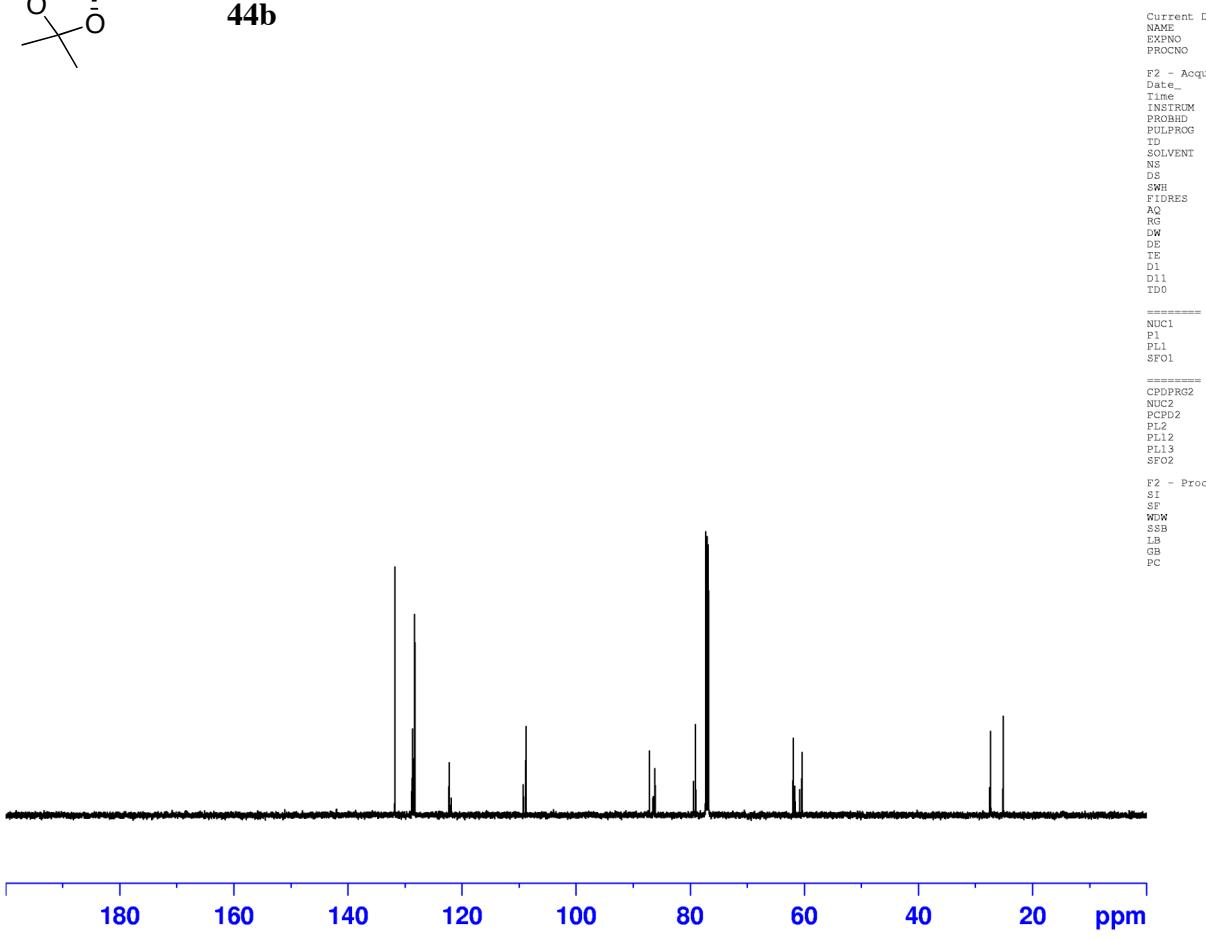
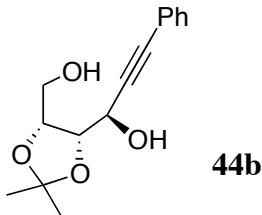
Current Data Parameters
NAME J3-Ph-acetylde add to erythrolactol
EXPNO 1
PROCNO 1

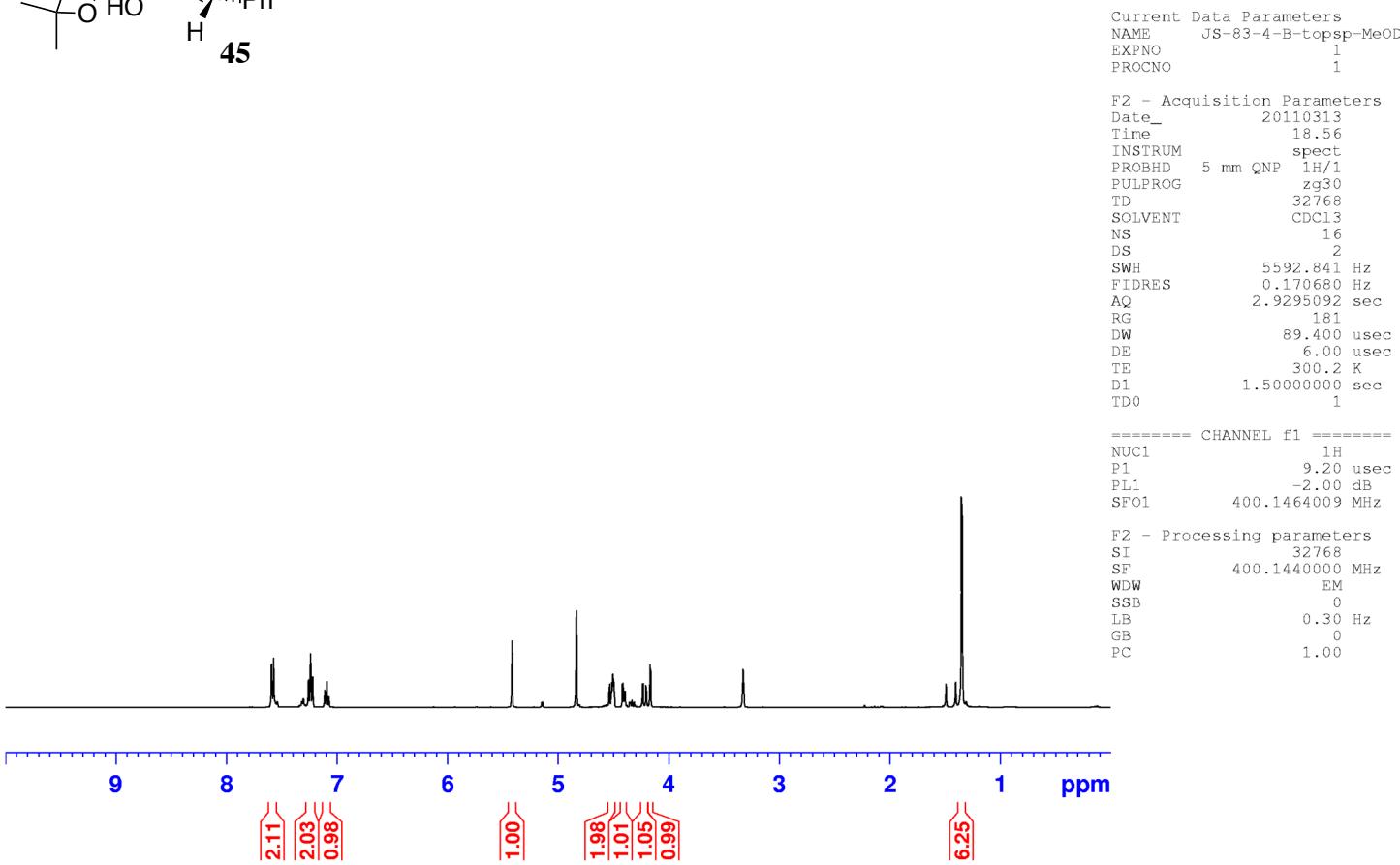
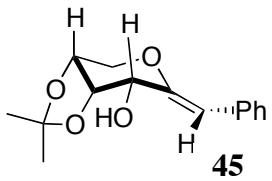
F2 - Acquisition Parameters
Date_ 20130308
Time 14:19:18
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.586064 sec
RG 64
DW 48,400 usec
DE 6.00 usec
TE 298.2 K
D1 1.0000000 sec
TD0 1

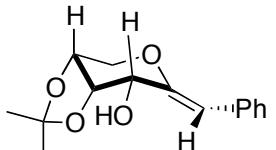
===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 1.00 dB
SF01 500.1330485 MHz

F2 - Processing parameters
SI 32768
SF 500.13300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

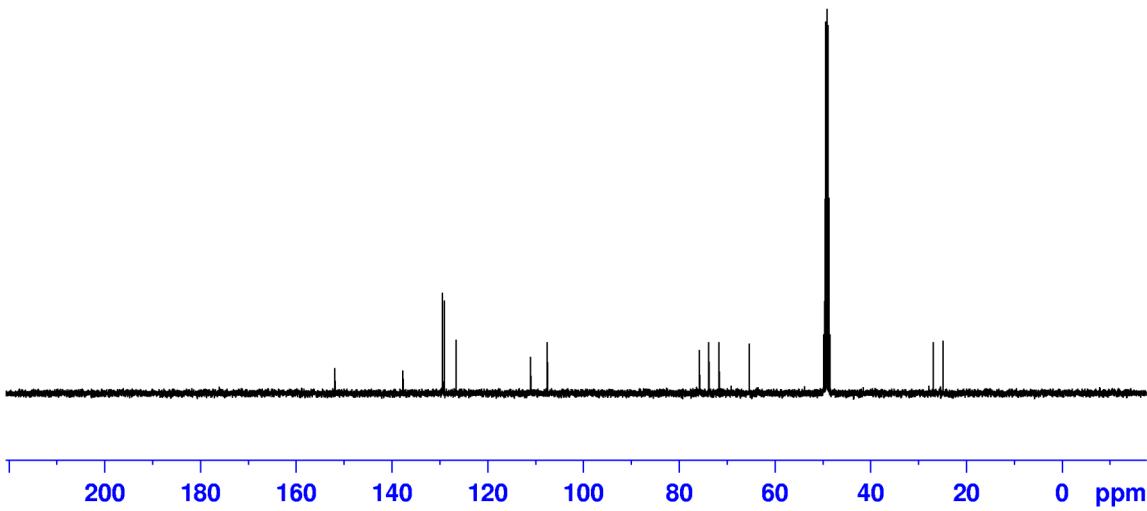








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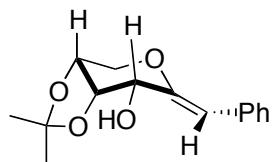
Current Data Parameters
NAME JS-83-4-B-topsp-MeOD
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110313
Time 19.05
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 81
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 32768
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.8999998 sec
TDO 1

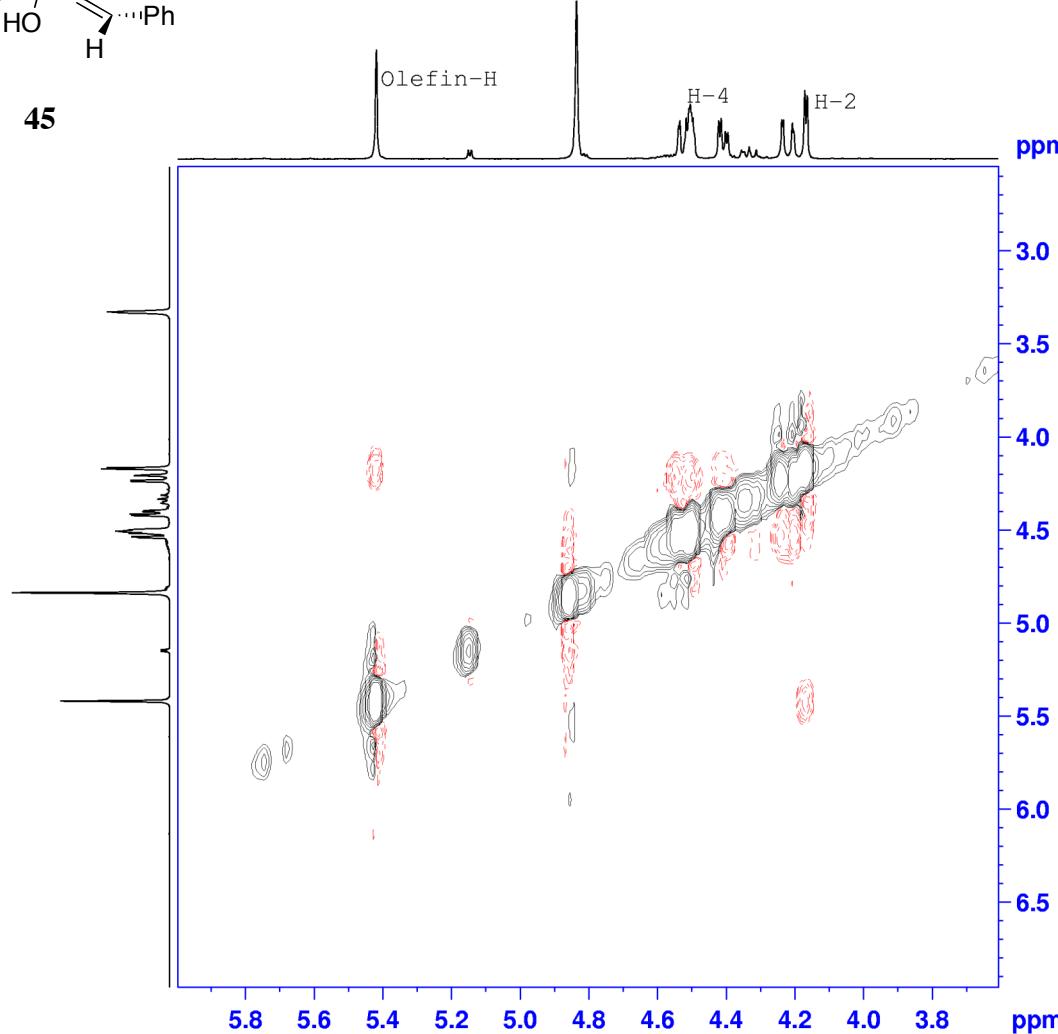
===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 100.6263500 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 110.00 usec
PL2 -2.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 400.1456010 MHz

F2 - Processing parameters
SI 32768
SF 100.6161341 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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ppm

Current Data Parameters
NAME JS-83-4-topsp-MeOD-300
EXPNO 6
PROCNO 1

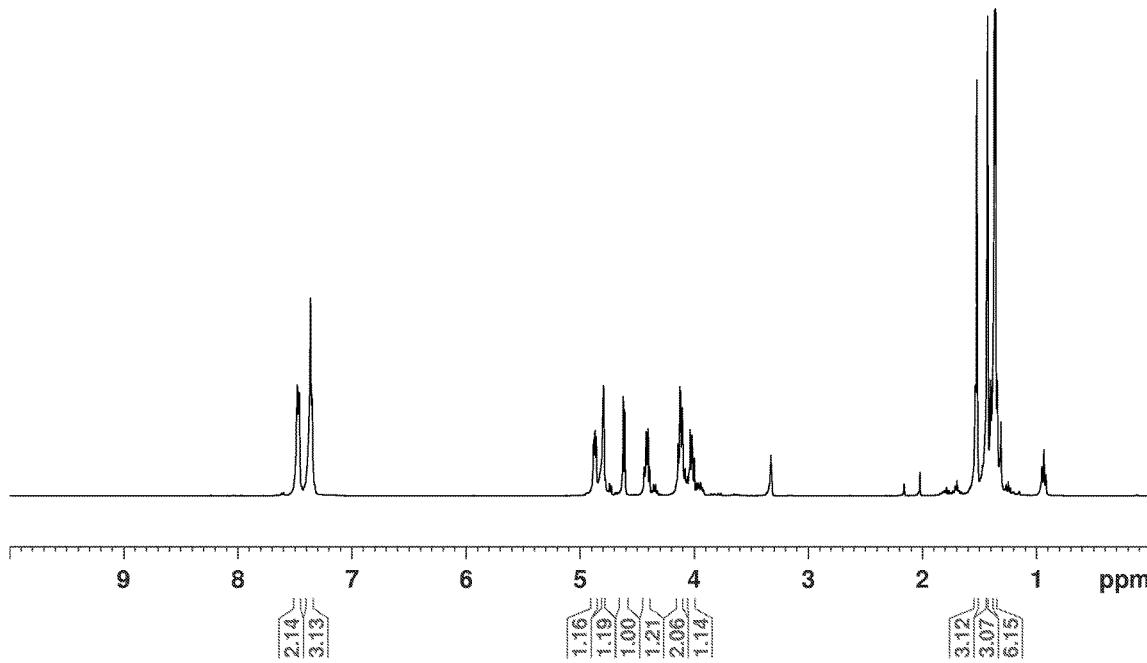
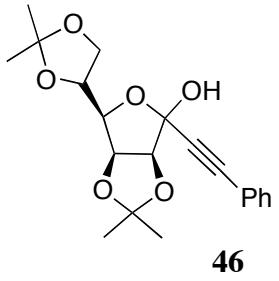
F2 - Acquisition Parameters
Date_ 20110313
Time 19.46
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG noesyph
TD 2048
SOLVENT MeOD
NS 80
DS 4
SWH 3906.250 Hz
FIDRES 1.907349 Hz
AQ 0.2621940 sec
RG 71.8
DW 128.000 usec
DE 6.50 usec
TE 300.2 K
D0 0.0001771 sec
D1 2.0000000 sec
D8 0.6999999 sec
INO 0.00025630 sec

===== CHANNEL f1 =====
NUC1 1H
P1 8.20 usec
PL1 0.00 dB
SFO1 300.1325007 MHz

F1 - Acquisition parameters
ND0 1
TD 217
SFO1 300.1325 MHz
FIDRES 17.980288 Hz
SW 13.000 ppm
FnMODE States-TPPI

F2 - Processing parameters
SI 1024
SF 300.1310000 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 300.1310000 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

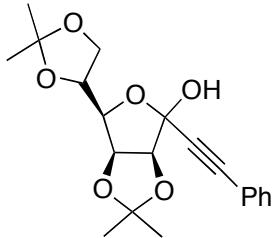


Current Data Parameters
 NAME JS-1b-4-MeOD
 EXPNO 1
 PROCN0 1

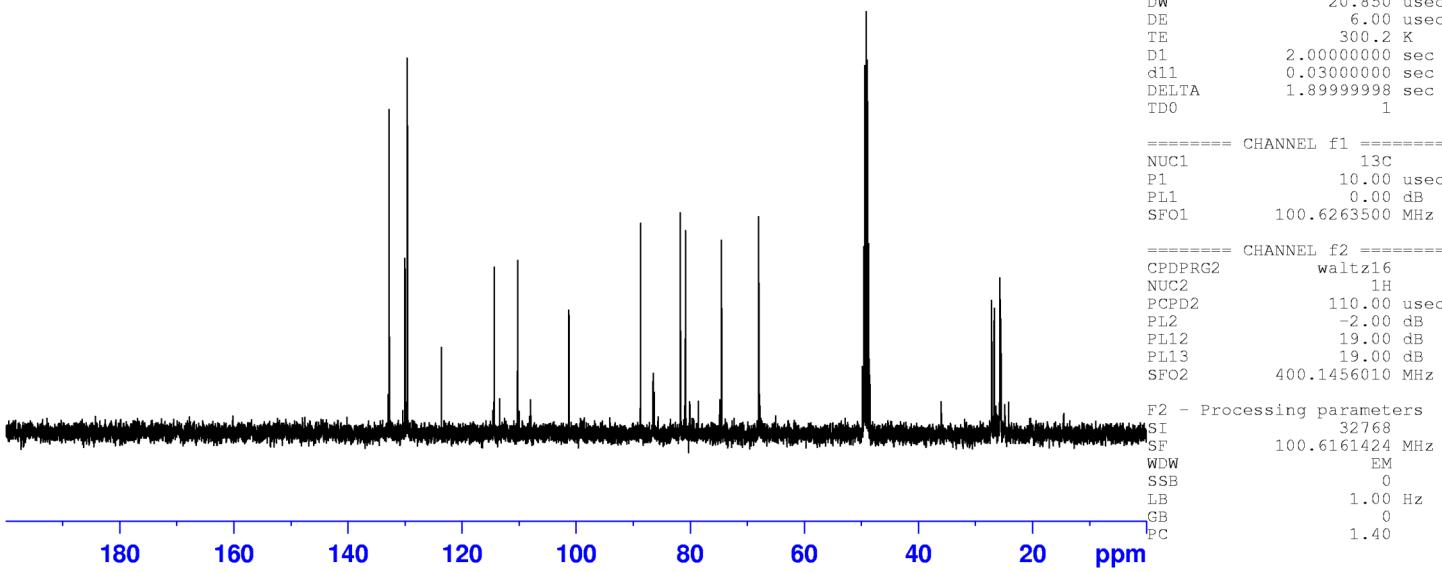
F2 - Acquisition Parameters
 Date_ 20110408
 Time 17.26
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 57
 DW 89.400 usec
 DE 6.00 usec
 TE 300.2 K
 D1 1.5000000 sec
 TDO 1

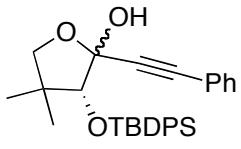
===== CHANNEL f1 =====
 NUC1 1H
 P1 9.20 usec
 PL1 -2.00 dB
 SFO1 400.1464009 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1440000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

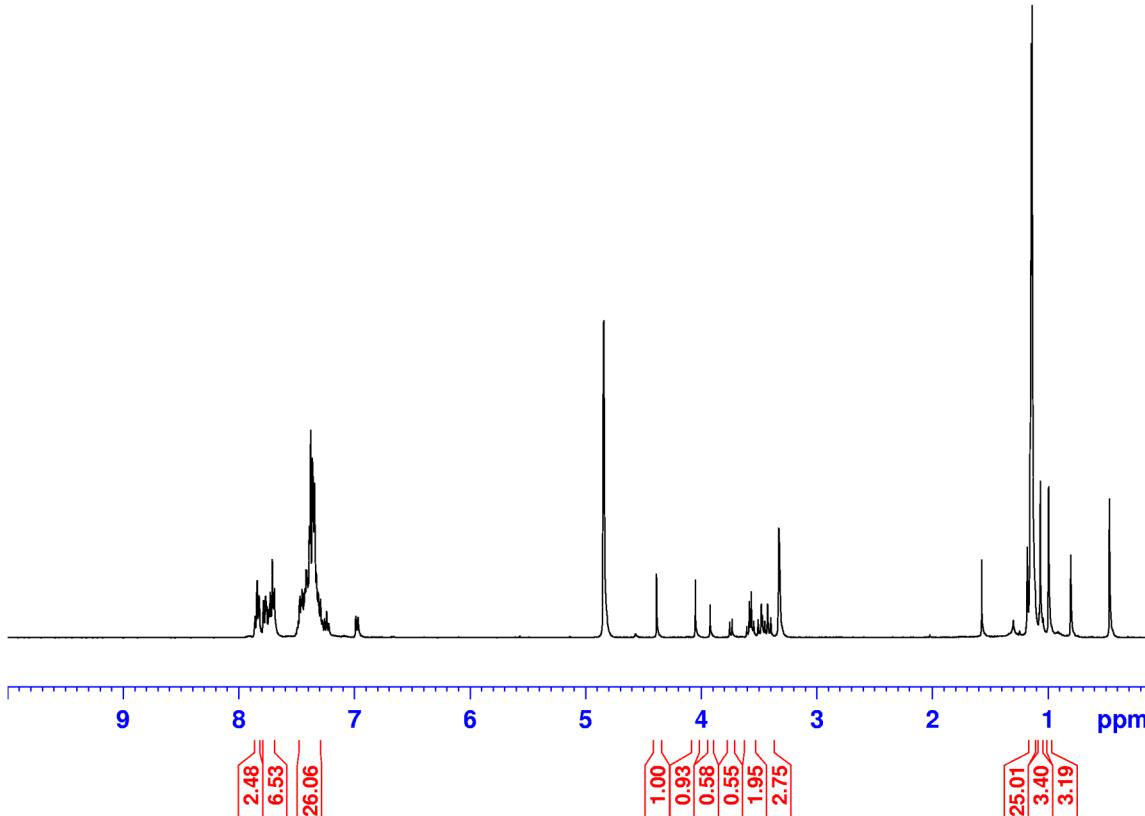


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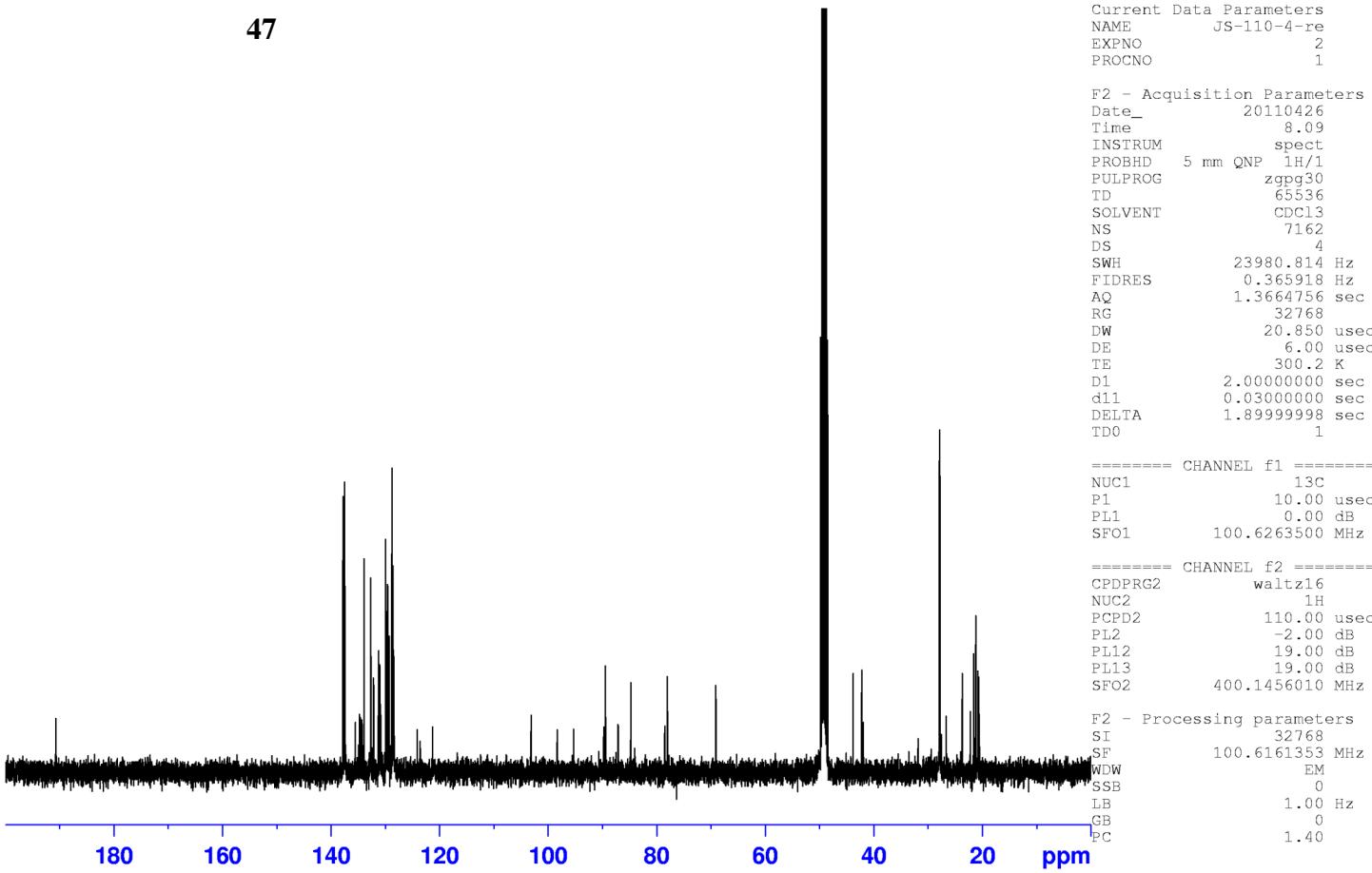
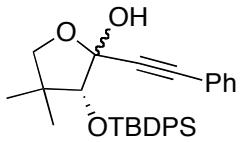


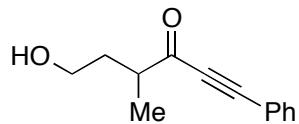
Current Data Parameters
 NAME JS-110-4
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110425
 Time 22.21
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 26
 DS 2
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 456.1
 DW 89.400 usec
 DE 6.00 usec
 TE 300.2 K
 D1 1.5000000 sec
 TDO 1

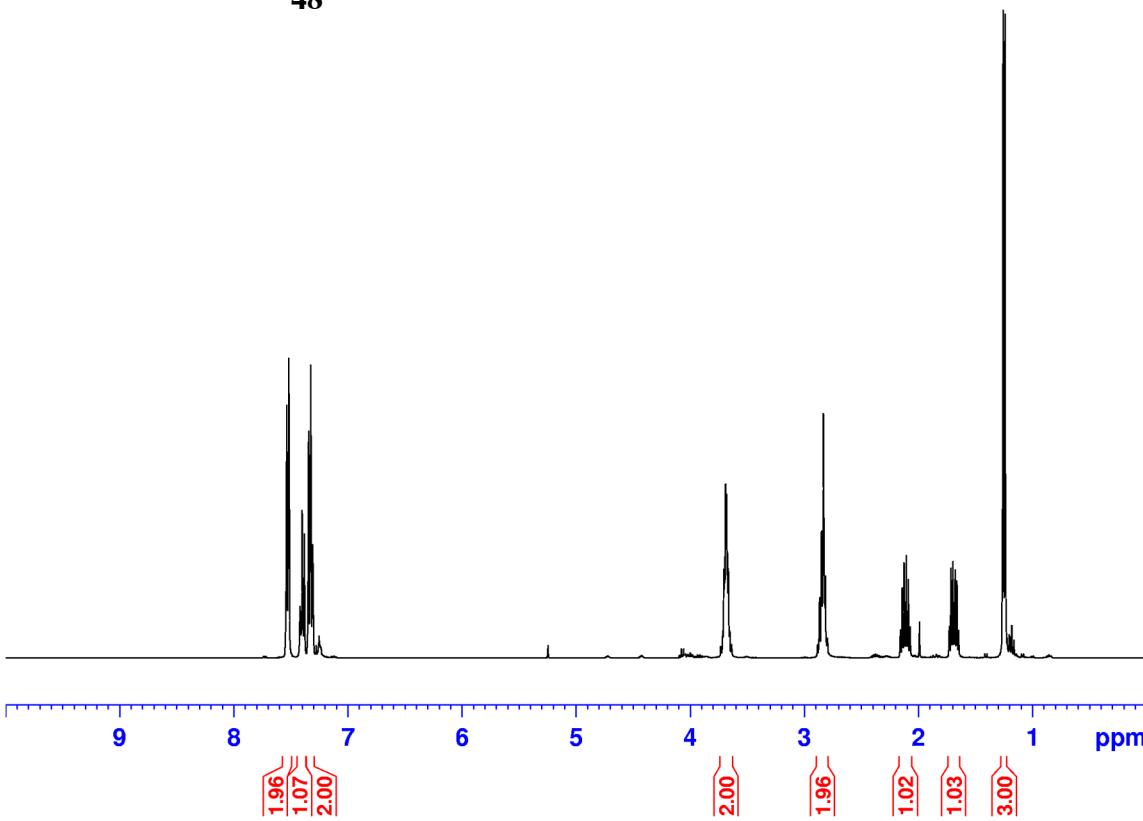
===== CHANNEL f1 =====
 NUC1 1H
 P1 9.20 usec
 PL1 -2.00 dB
 SFO1 400.1464009 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1440000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





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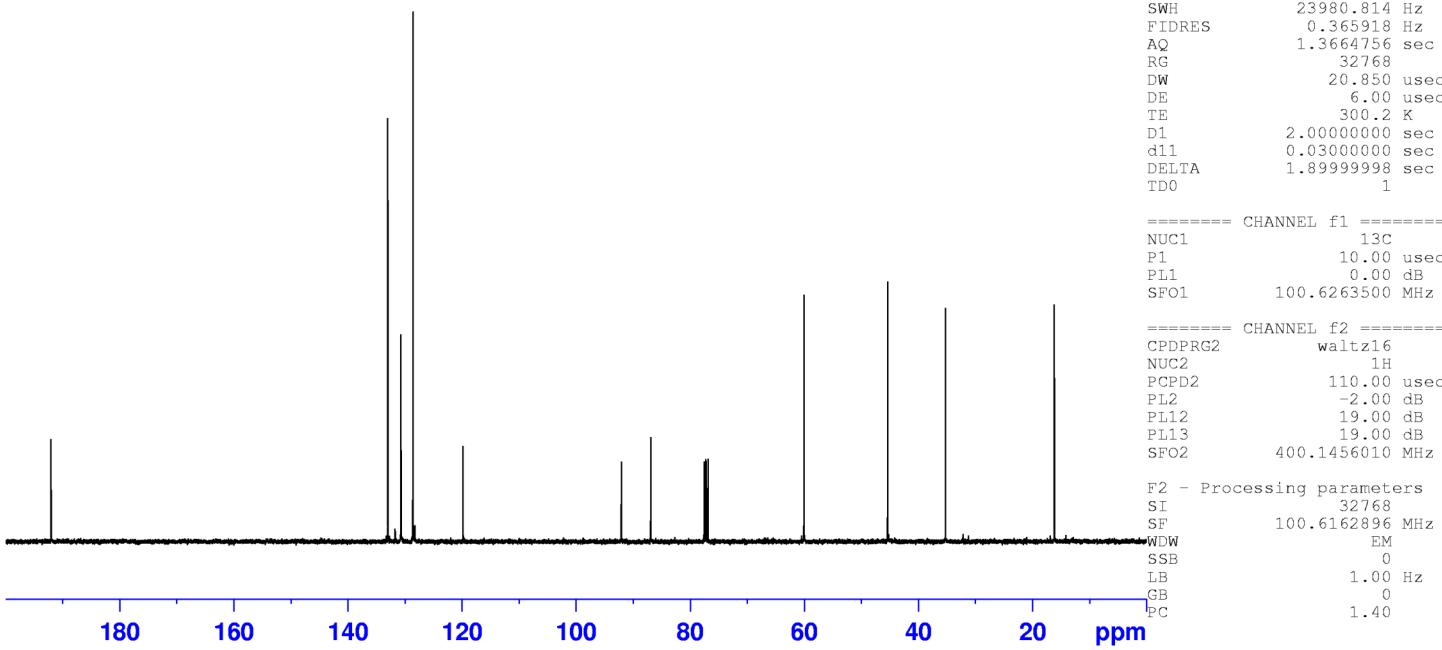
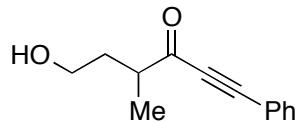


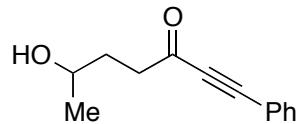
Current Data Parameters
NAME JS-97-4-B
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110328
Time 20.12
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5592.841 Hz
FIDRES 0.170680 Hz
AQ 2.9295092 sec
RG 25.4
DW 89.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.5000000 sec
TDO 1

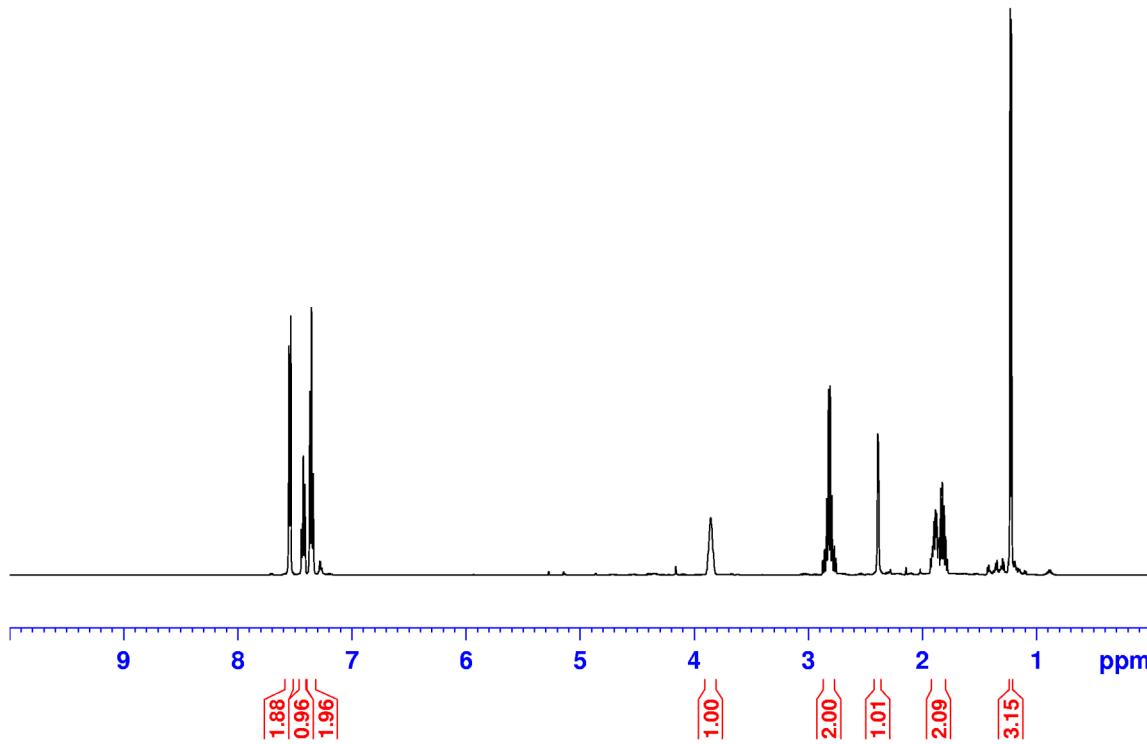
===== CHANNEL f1 ======
NUC1 1H
P1 9.20 usec
PL1 -2.00 dB
SFO1 400.1464009 MHz

F2 - Processing parameters
SI 32768
SF 400.1440000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





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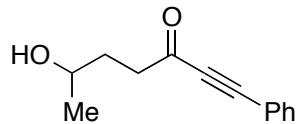


Current Data Parameters
NAME JS-97-4-A
Time 22.56
EXPNO 1
PROCNO 1

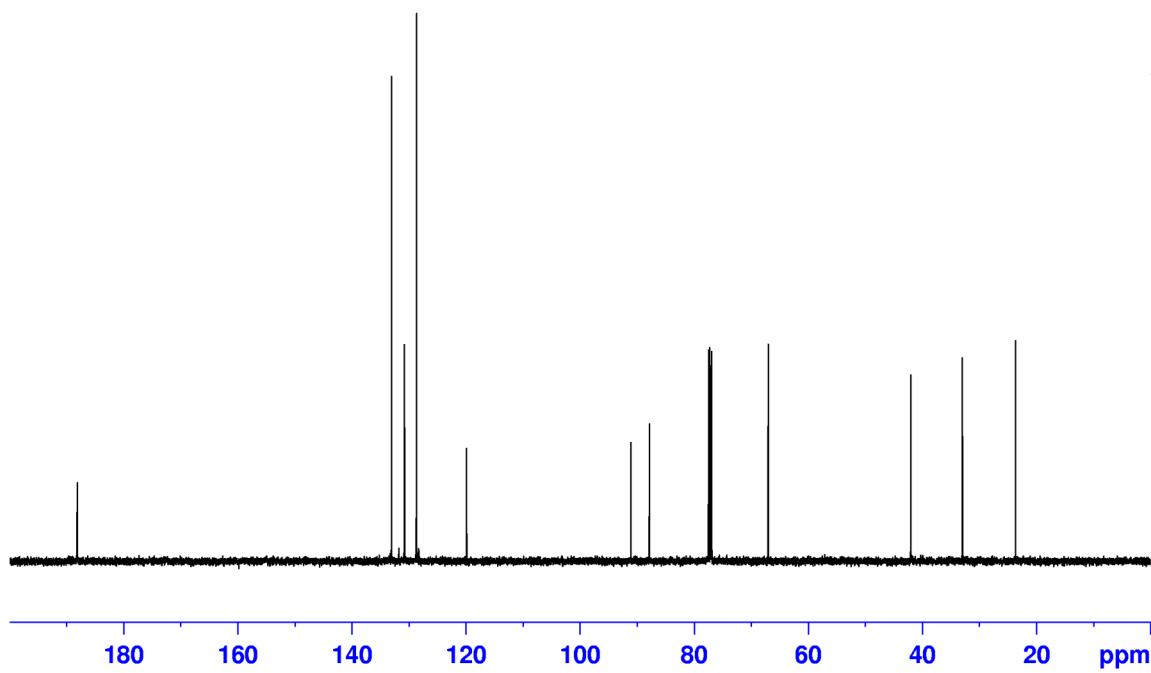
F2 - Acquisition Parameters
Date_ 20110329
Time 22.56
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 32
DW 48.400 usec
DE 6.00 usec
TE 298.2 K
D1 1.0000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 -1.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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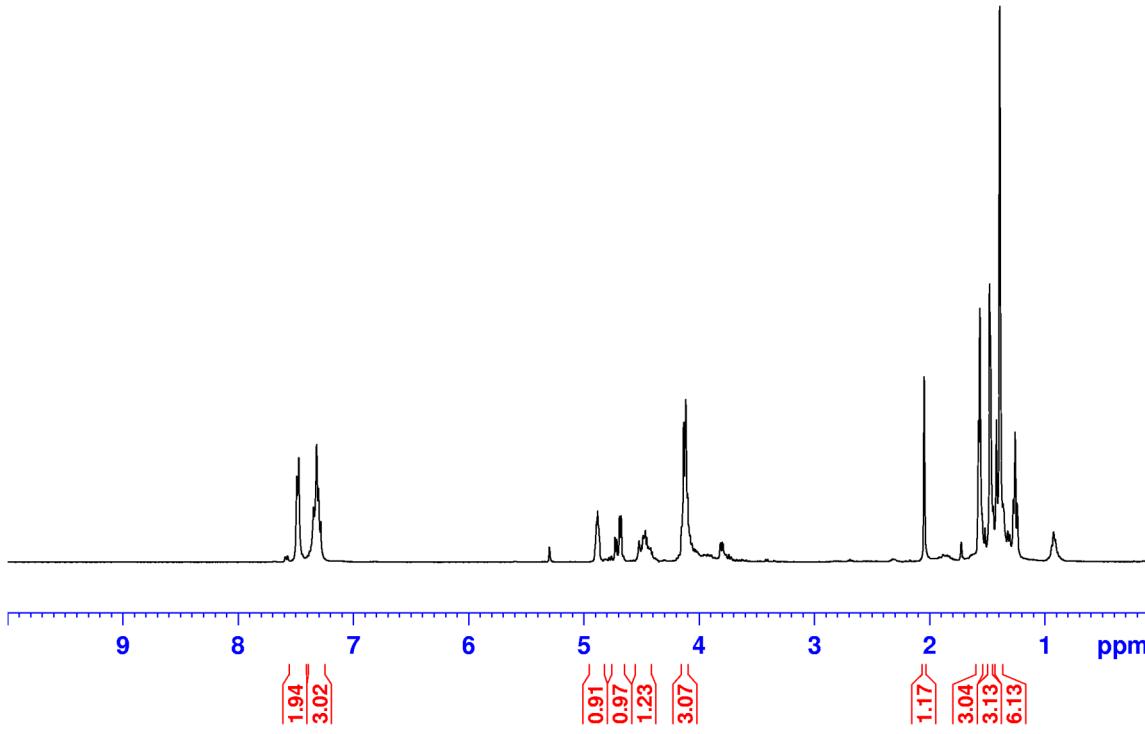
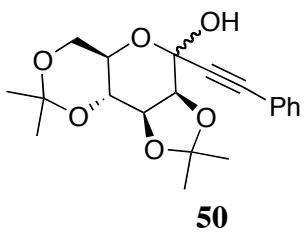
Current Data Parameters
NAME JS-97-4-A
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110329
Time 22.58
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 39
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 1.5000000 sec
D11 0.03000000 sec
TDO 1

===== CHANNEL f1 ======
NUC1 13C
P1 6.25 usec
PL1 0.00 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -1.00 dB
PL12 13.76 dB
PL13 70.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577816 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

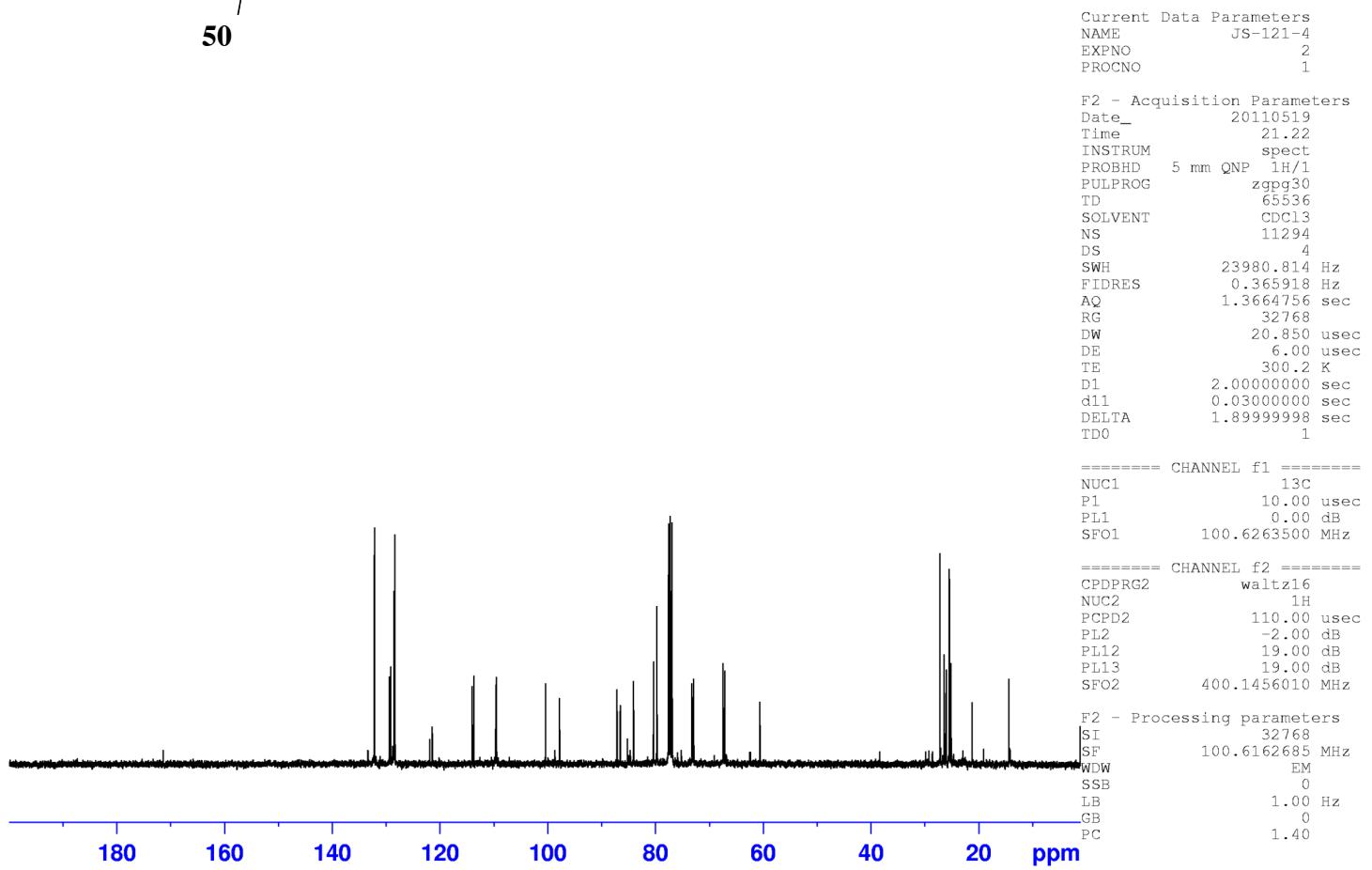
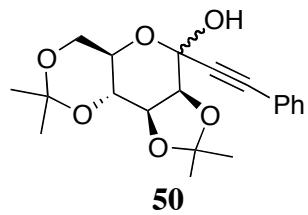


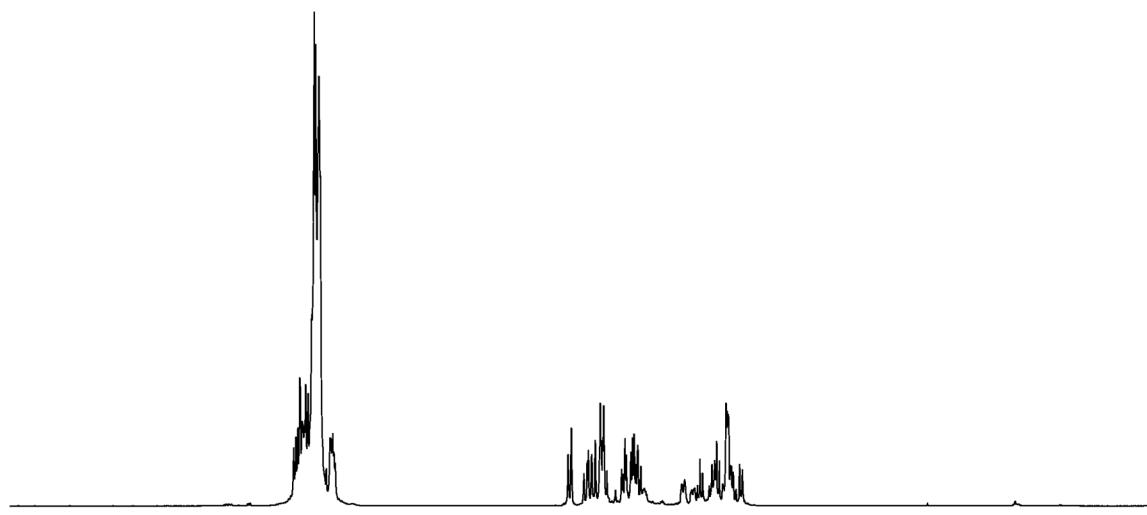
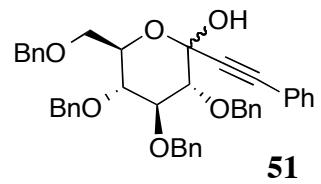
Current Data Parameters
NAME JS-121-4
Time 21.18
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110519
Time 21.18
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 27
DS 2
SWH 5592.841 Hz
FIDRES 0.170680 Hz
AQ 2.9295092 sec
RG 287.4
DW 89.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.5000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.20 usec
PL1 -2.00 dB
SFO1 400.1464009 MHz

F2 - Processing parameters
SI 32768
SF 400.1440000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



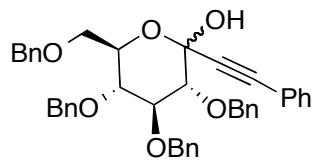


Current Data Parameters
NAME JS-116-4-re
Time 21.48
EXPNO 1
PROCNO 1

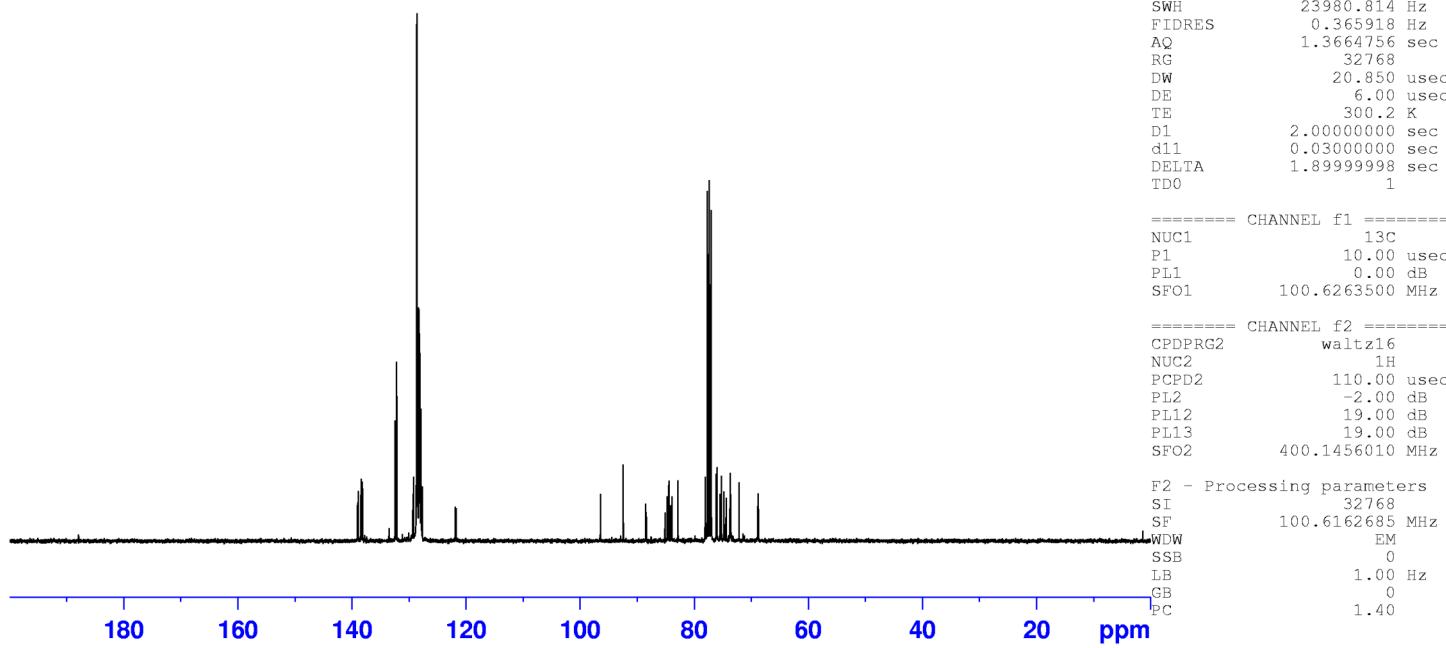
F2 - Acquisition Parameters
Date_ 20110509
Time 21.48
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5592.841 Hz
FIDRES 0.170680 Hz
AQ 2.9295092 sec
RG 57
DW 89.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.5000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.20 usec
PL1 -2.00 dB
SFO1 400.1464009 MHz

F2 - Processing parameters
SI 32768
SF 400.1440000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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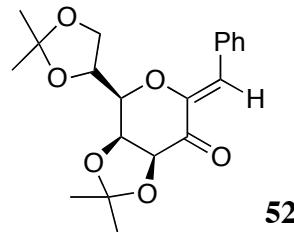
Current Data Parameters
 NAME JS-116-4-re
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110509
 Time 22.49
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1061
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 32768
 DW 20.850 usec
 DE 6.00 usec
 TE 300.2 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8999998 sec
 TDO 1

===== CHANNEL f1 ======
 NUC1 13C
 P1 10.00 usec
 PL1 0.00 dB
 SFO1 100.6263500 MHz

===== CHANNEL f2 ======
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 110.00 usec
 PL2 -2.00 dB
 PL12 19.00 dB
 PL13 19.00 dB
 SFO2 400.1456010 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6162685 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

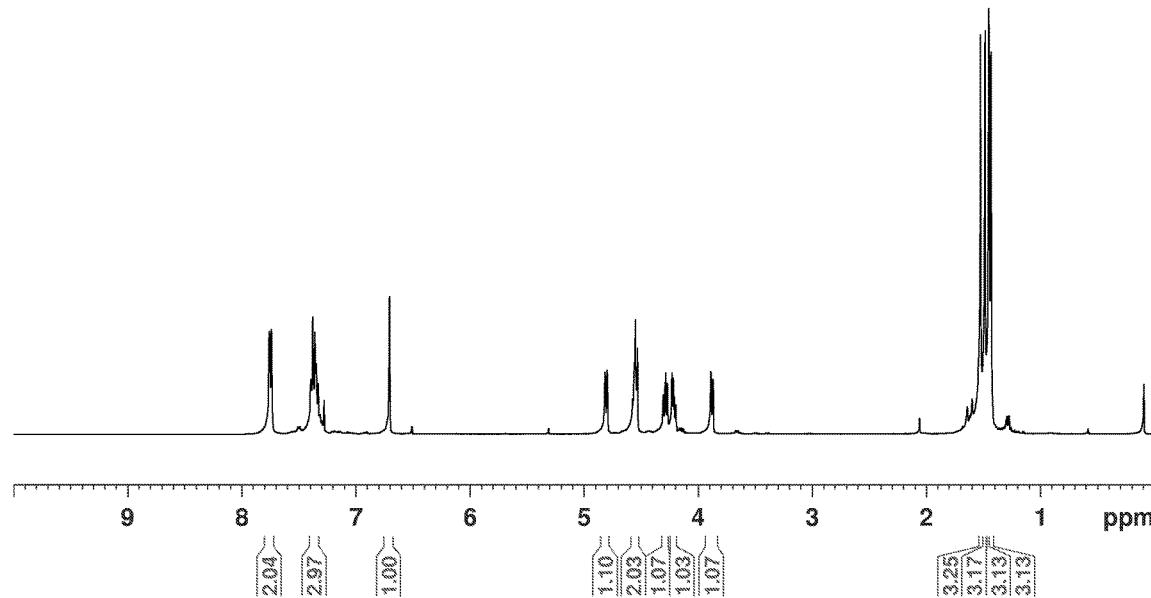


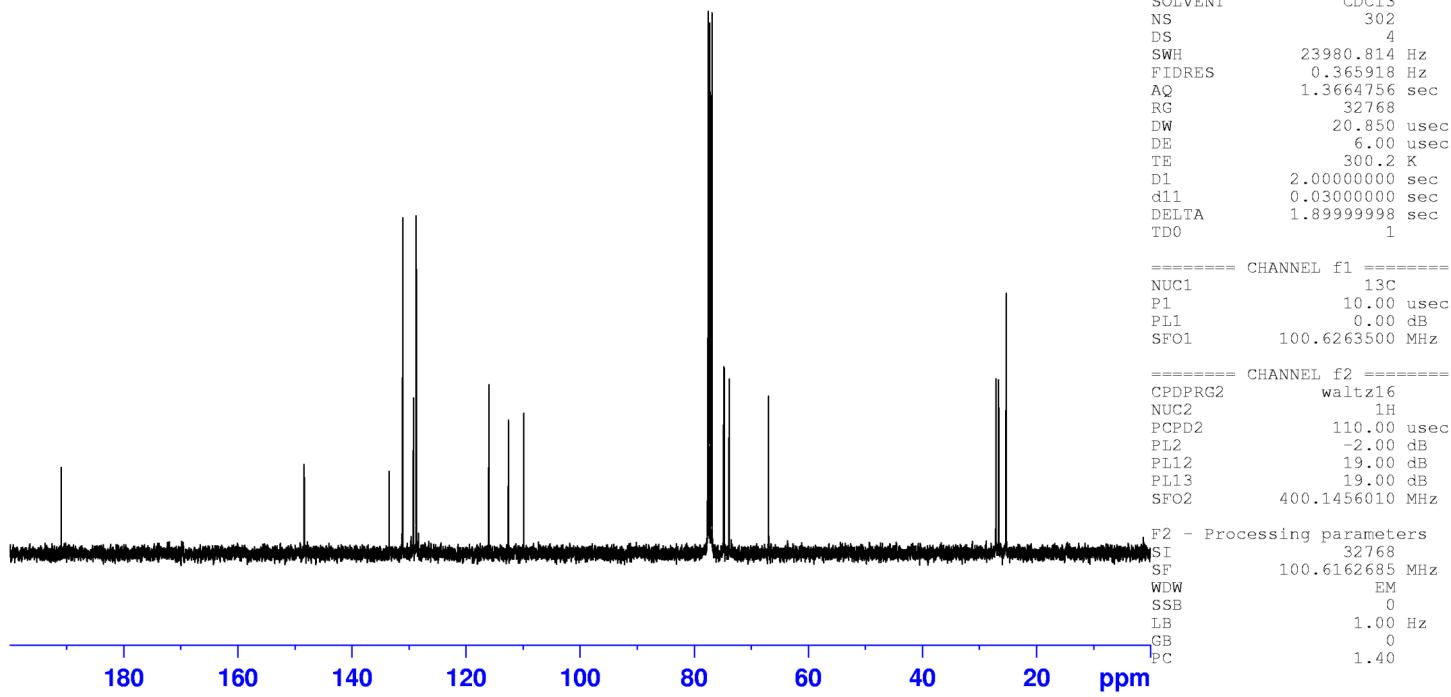
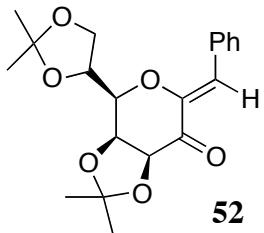
Current Data Parameters
 NAME JS-58-4-400-re
 Date_ 20110103
 Time 13.22
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 128
 DW 89.400 usec
 DE 6.00 usec
 TE 300.2 K
 D1 1.5000000 sec
 TDO 1

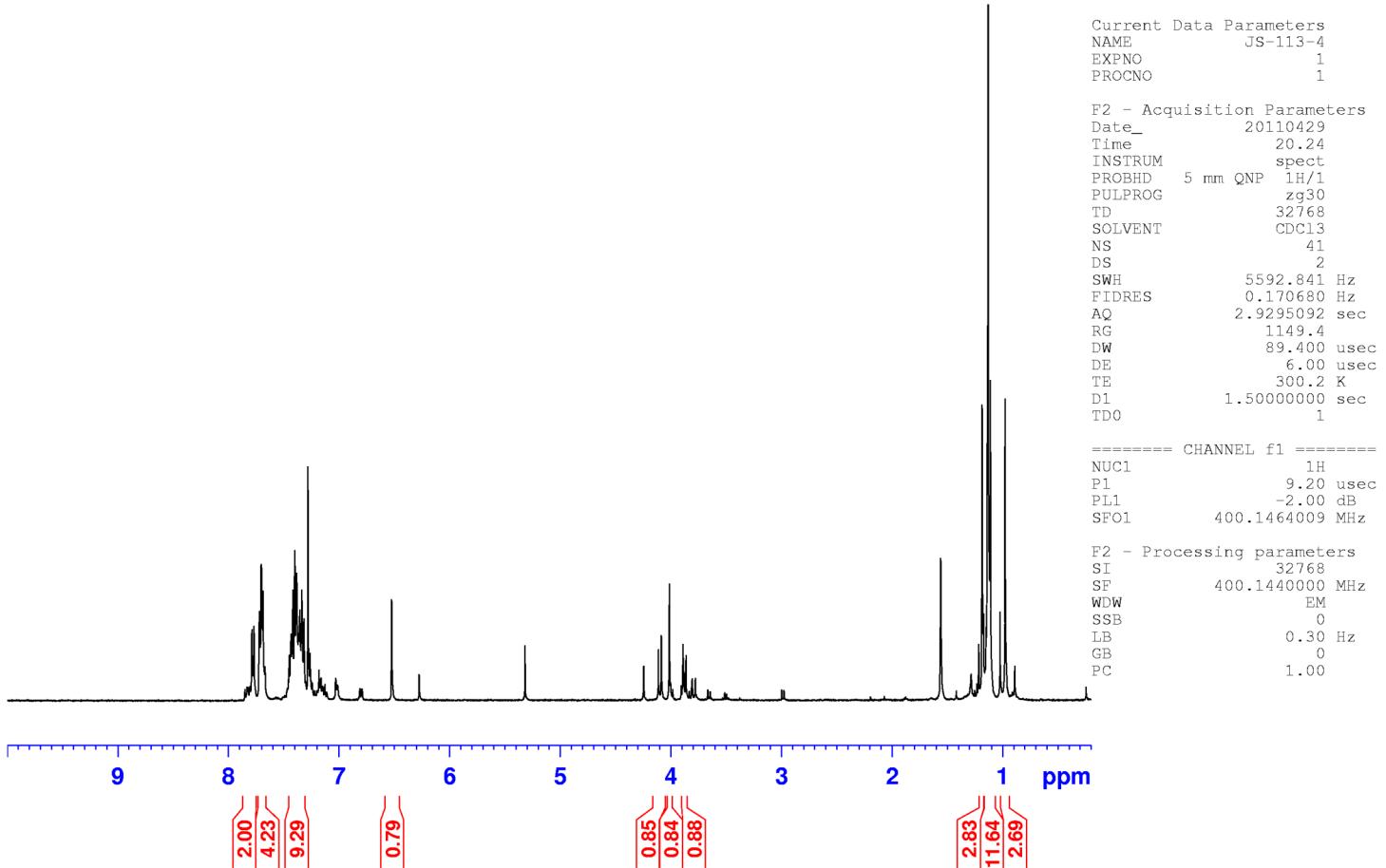
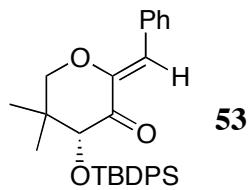
F2 - Acquisition Parameters
 Date_ 20110103
 Time 13.22
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 128
 DW 89.400 usec
 DE 6.00 usec
 TE 300.2 K
 D1 1.5000000 sec
 TDO 1

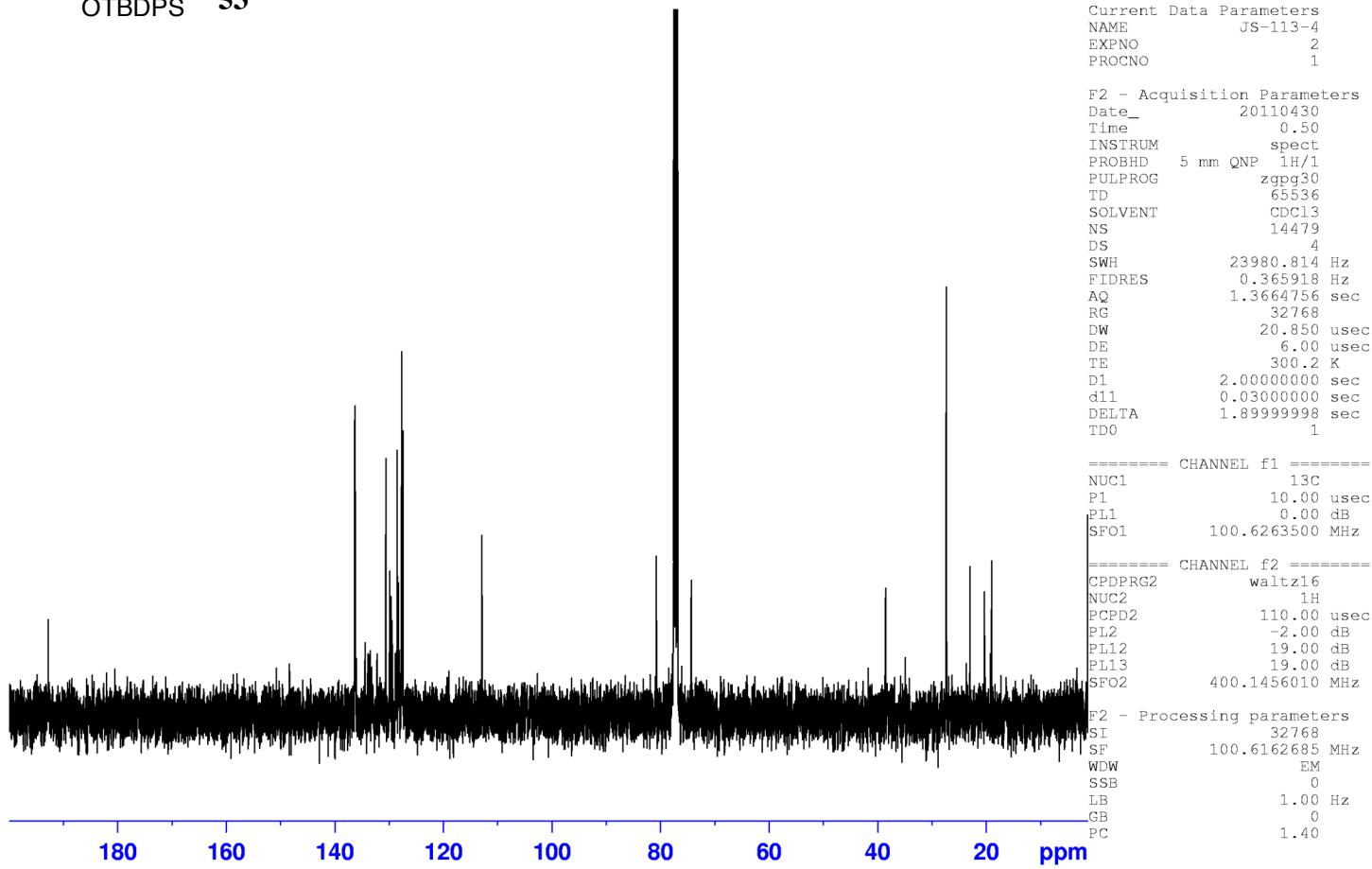
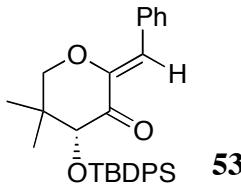
===== CHANNEL f1 =====
 NUC1 1H
 P1 9.20 usec
 PL1 -2.00 dB
 SFO1 400.1464009 MHz

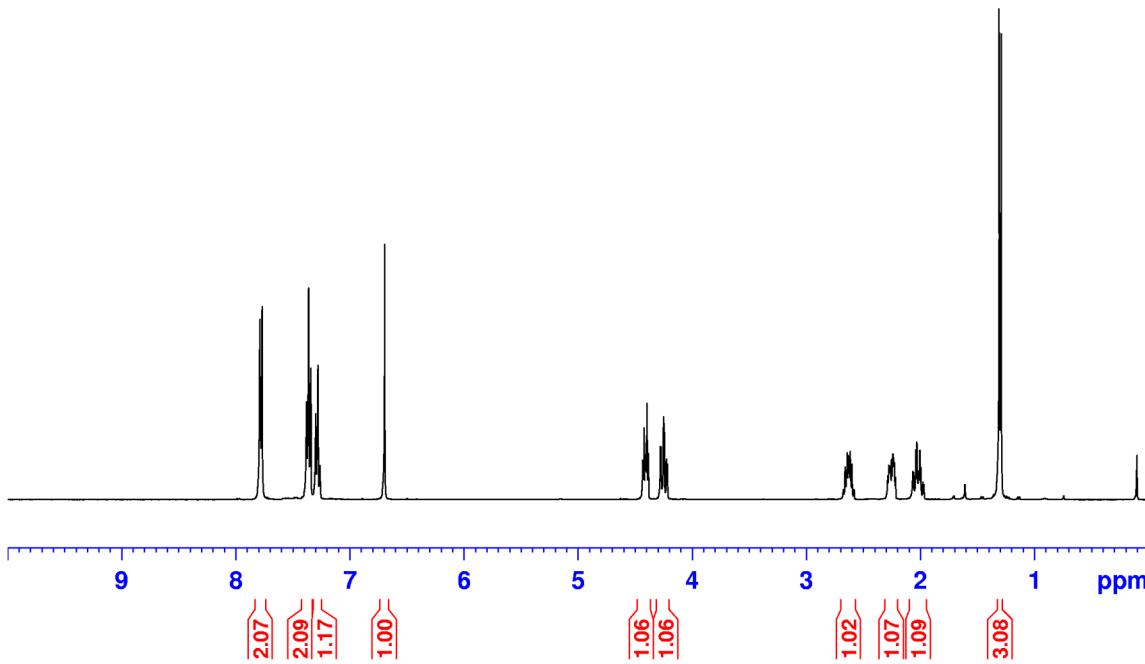
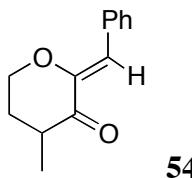
F2 - Processing parameters
 SI 32768
 SF 400.1440000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00









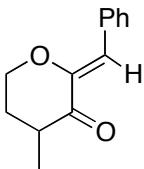


Current Data Parameters
NAME JS-100-4
Time 0.02
EXPNO 1
PROCNO 1

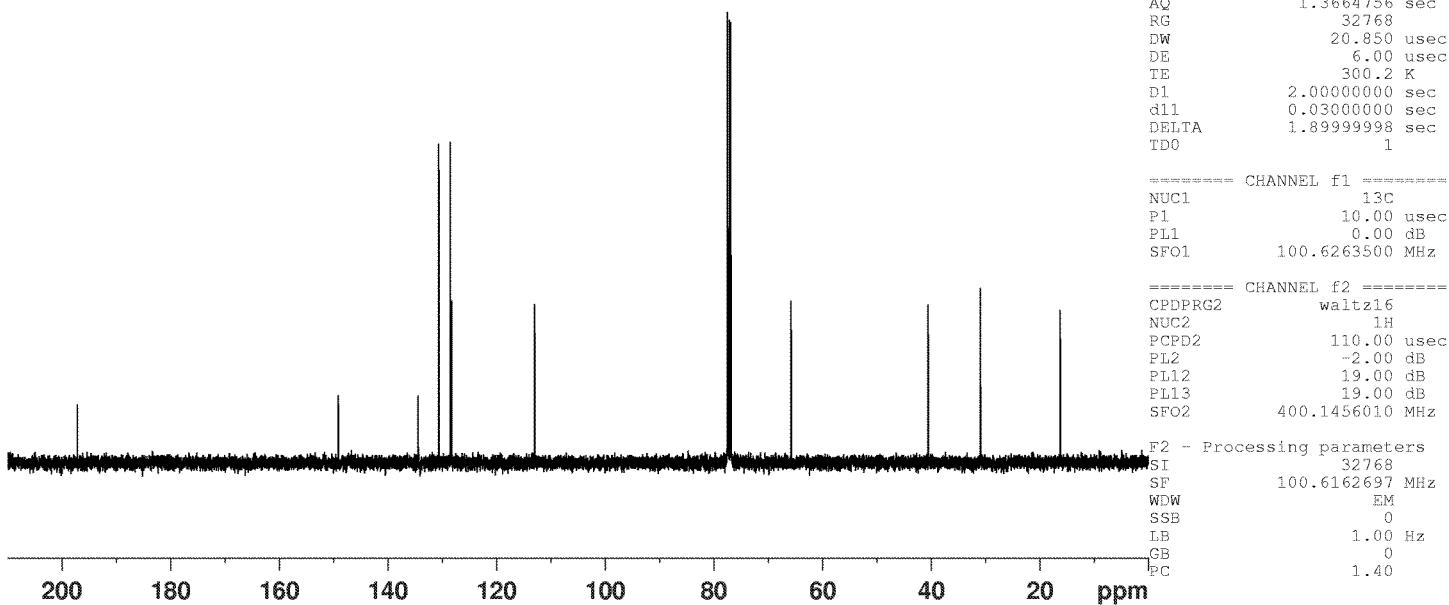
F2 - Acquisition Parameters
Date_ 20110331
Time 0.02
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5592.841 Hz
FIDRES 0.170680 Hz
AQ 2.9295092 sec
RG 203.2
DW 89.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.5000000 sec
TDO 1

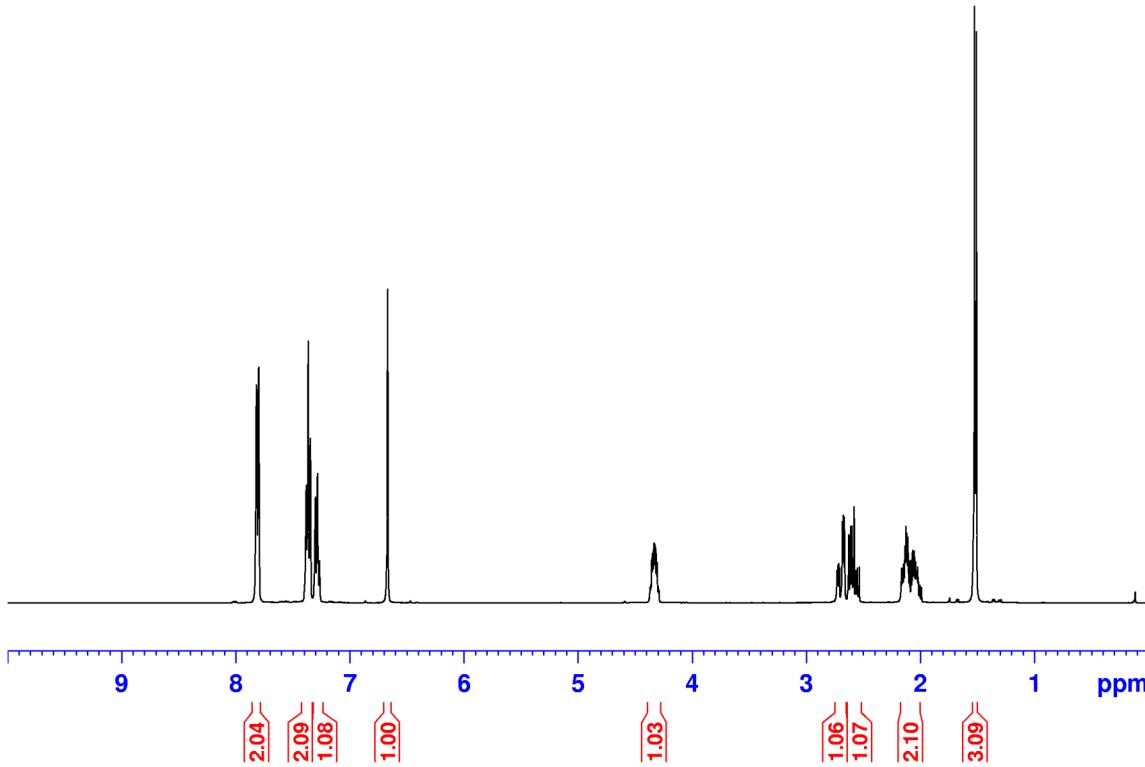
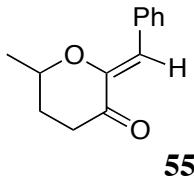
===== CHANNEL f1 =====
NUC1 1H
P1 9.20 usec
PL1 -2.00 dB
SFO1 400.1464009 MHz

F2 - Processing parameters
SI 32768
SF 400.1440000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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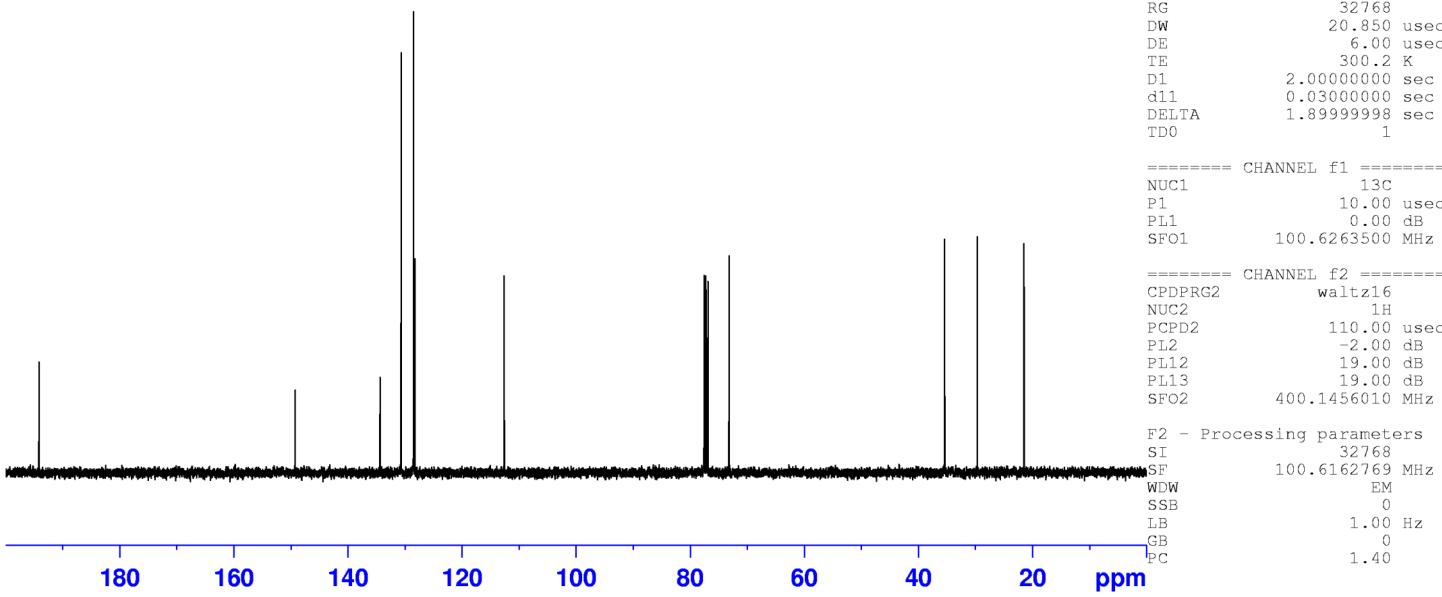
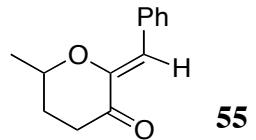


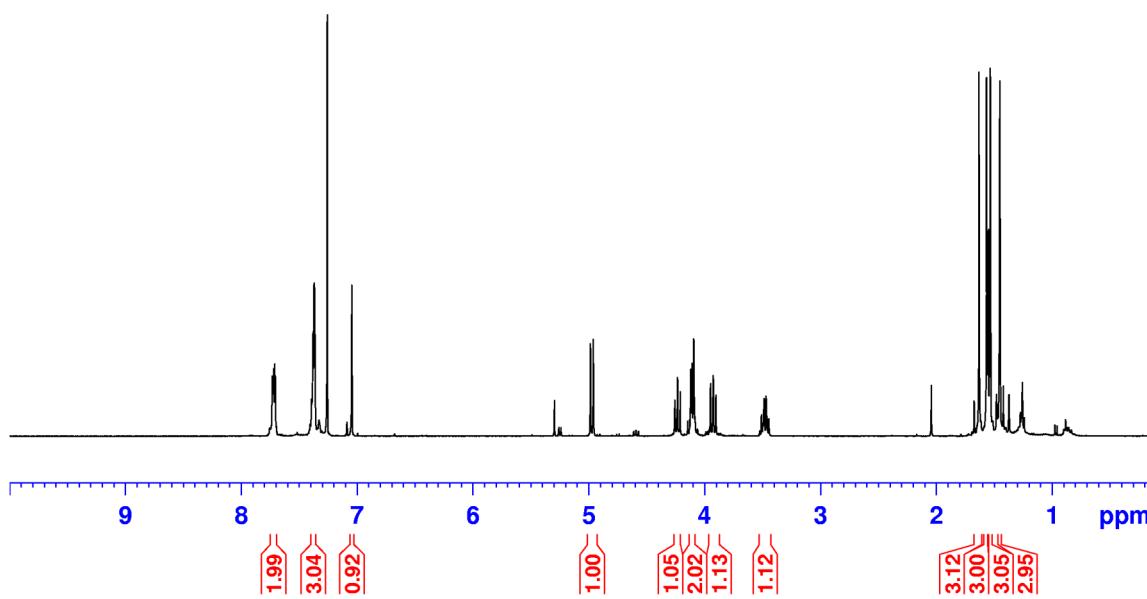
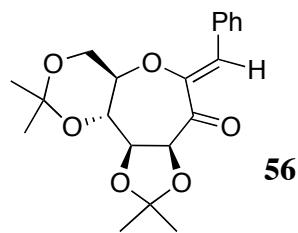
Current Data Parameters
NAME JS-101-4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110401
Time 11.03
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5592.841 Hz
FIDRES 0.170680 Hz
AQ 2.9295092 sec
RG 57
DW 89.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.5000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.20 usec
PL1 -2.00 dB
SFO1 400.1464009 MHz

F2 - Processing parameters
SI 32768
SF 400.1440000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



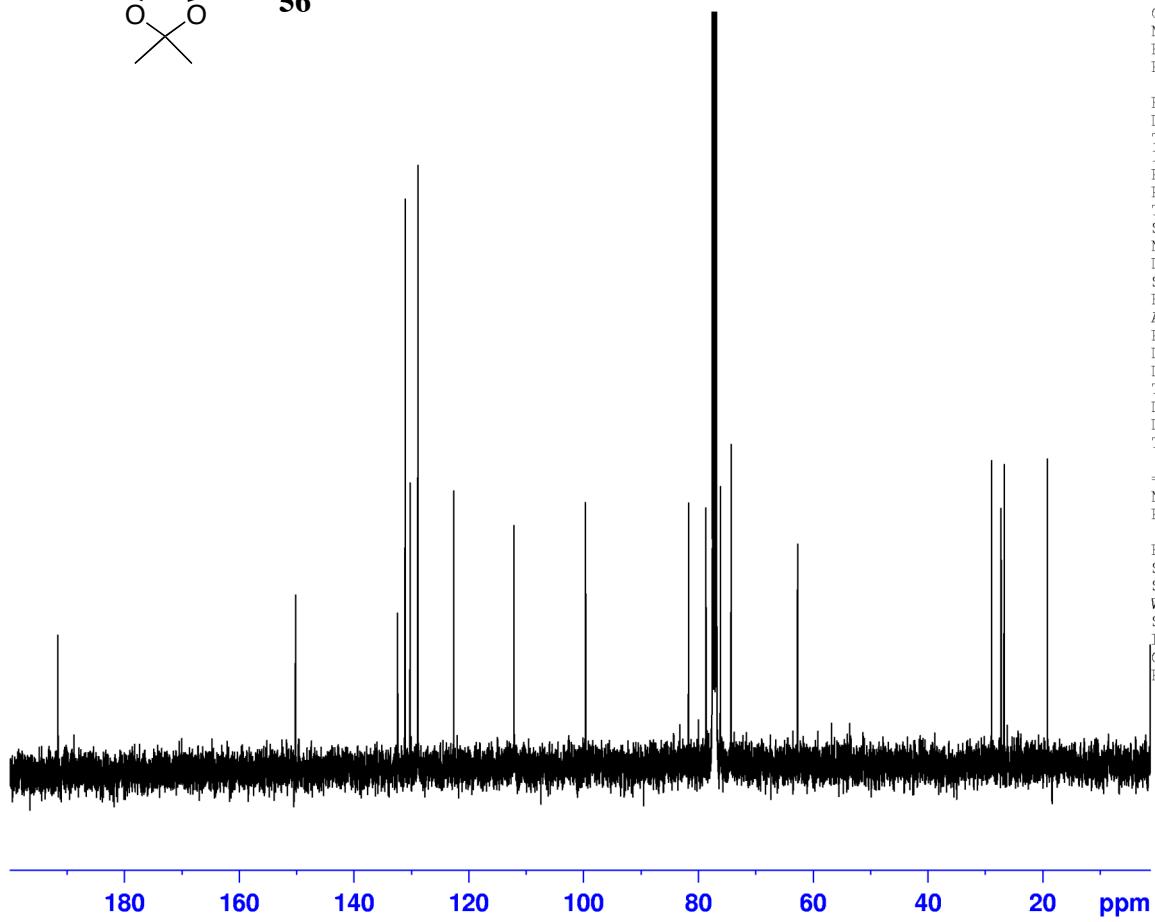
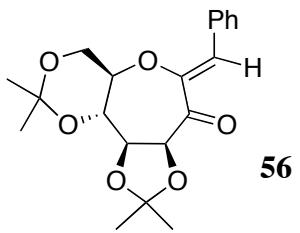


Current Data Parameters
NAME cl-1-121
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111108
Time 16.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 181
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.0000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec

F2 - Processing parameters
SI 65536
SF 400.1440094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

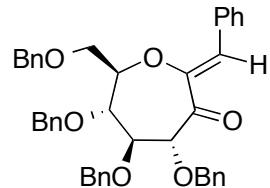


Current Data Parameters
NAME cl-1-121
EXPNO 2
PROCNO 1

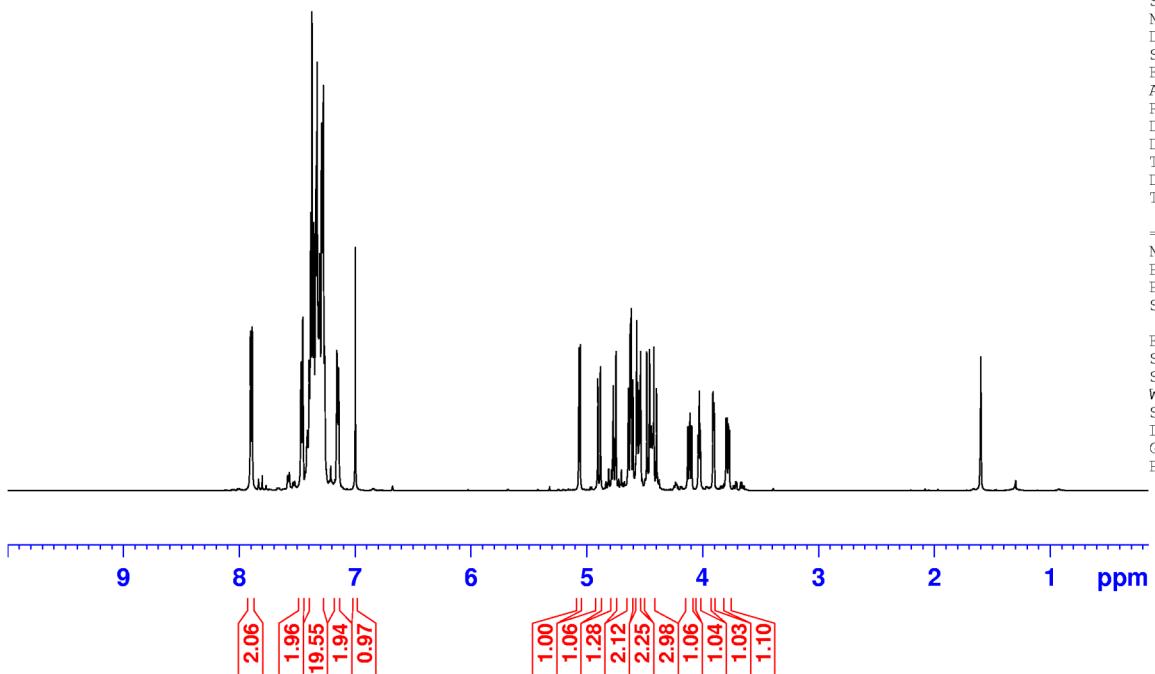
F2 - Acquisition Parameters
Date_ 20111108
Time 16.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 960
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TDO 1

===== CHANNEL f1 ======
NUC1 13C
P1 10.00 usec

F2 - Processing parameters
SI 32768
SF 100.6162683 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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Current Data Parameters
NAME JS-117-4-sp abv sm
EXPNO 1
PROCNO 1

```

F2 - Acquisition Parameters
Date_      20110526
Time       16.15
INSTRUM   spect
PROBHD   5 mm BBO BB-1H
PULPROG  zg30
TD        32768
SOLVENT    CDC13
NS         20
DS          2
SWH       10330.578 Hz
FIDRES   0.315264 Hz
AQ        1.586096 sec
RG        128
DW        48.400 usec
DE        6.00  usec
TE        298.2 K
D1        1.0000000 sec
TD0          1

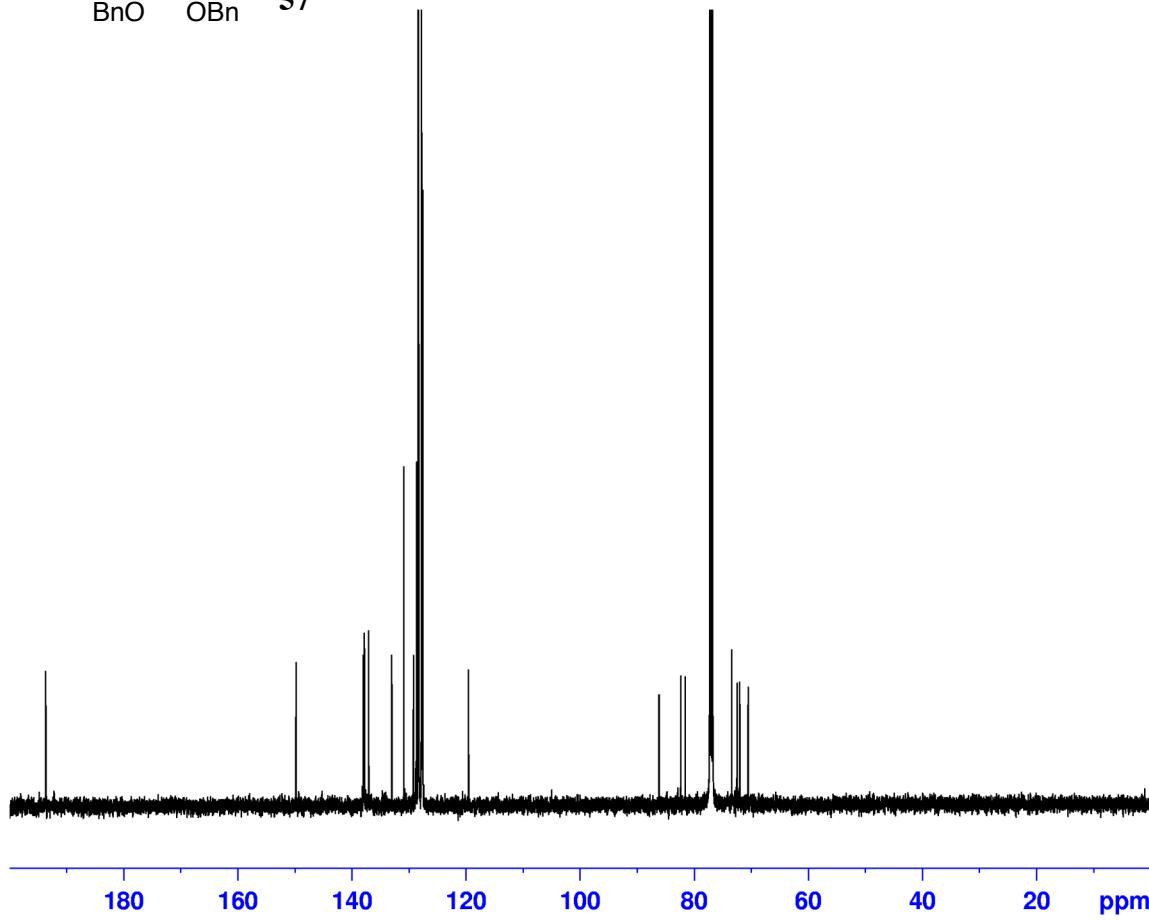
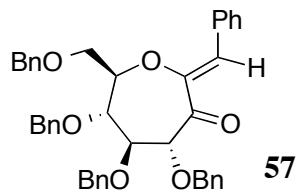
```

===== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 -1.00 dB
SEQ1 500.1330885 MHz

```

F2 - Processing parameters
SI          32768
SF         500.1300000 MHz
WDW          EM
SSB            0
LB           0.30 Hz
GB            0
PC          1.00

```



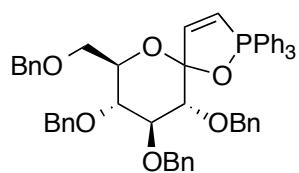
Current Data Parameters
NAME JS-117-4-sp abv sm-13C
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date 20110526
Time 17.44
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1949
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 1.5000000 sec
D11 0.03000000 sec
TDO 1

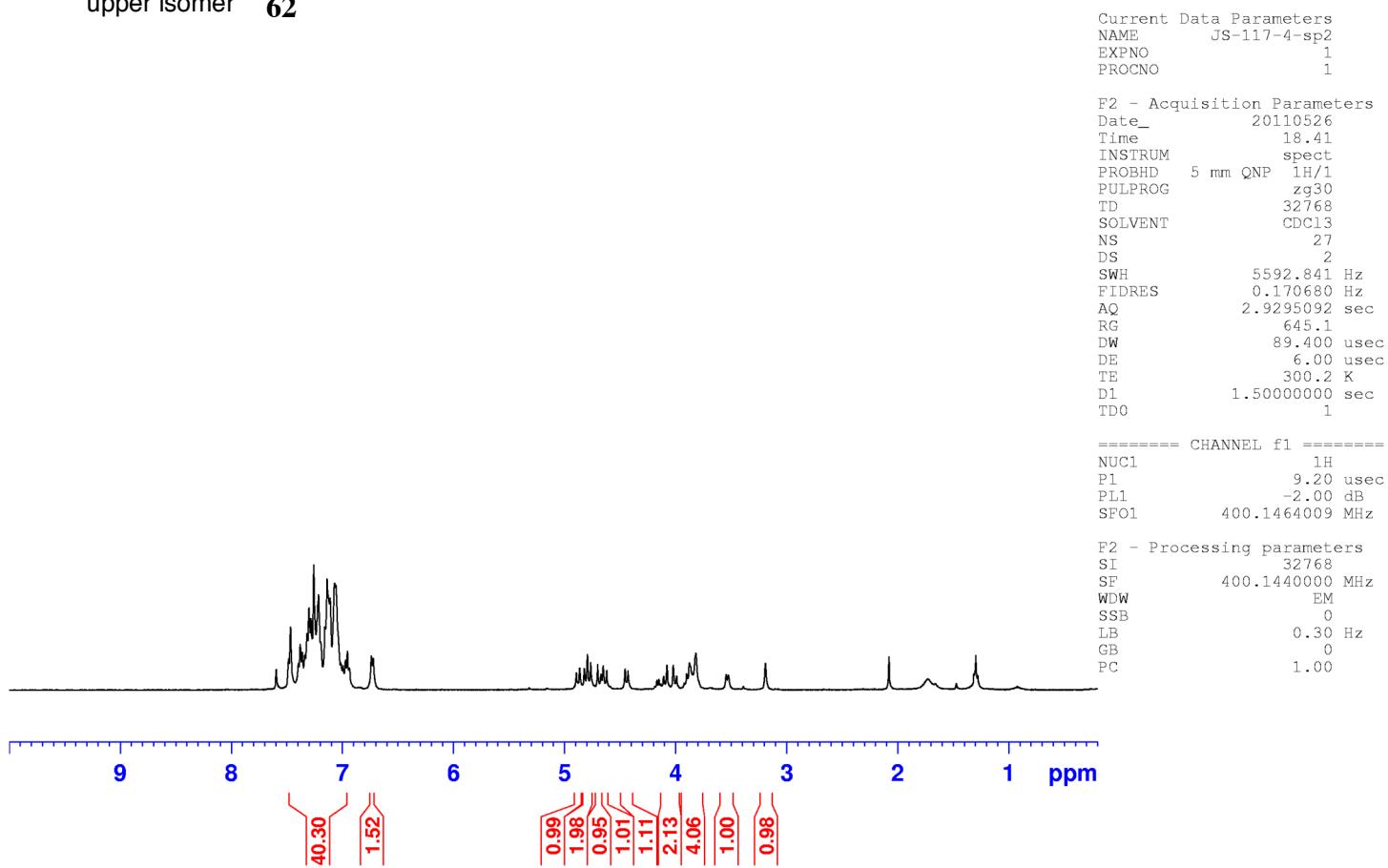
===== CHANNEL f1 =====
NUC1 13C
P1 6.25 usec
PL1 0.00 dB
SFO1 125.7703643 MHz

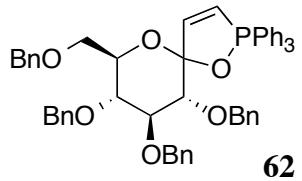
===== CHANNEL f2 =====
CPDPG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -1.00 dB
PL12 13.76 dB
PL13 70.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

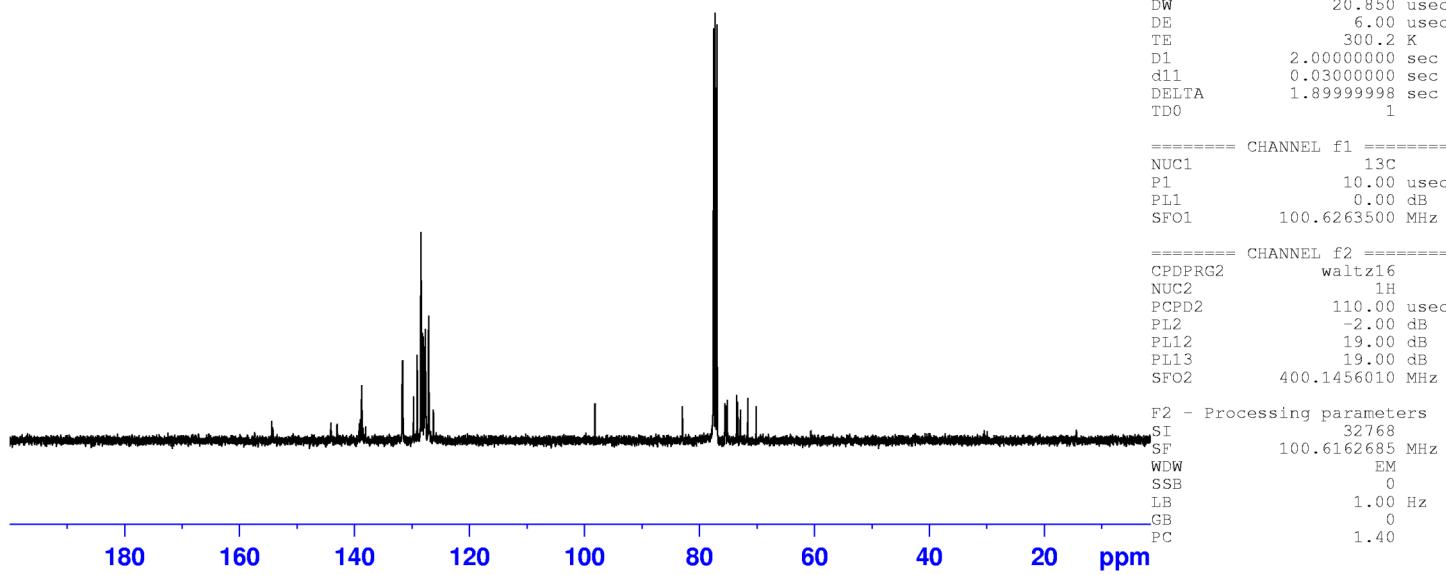


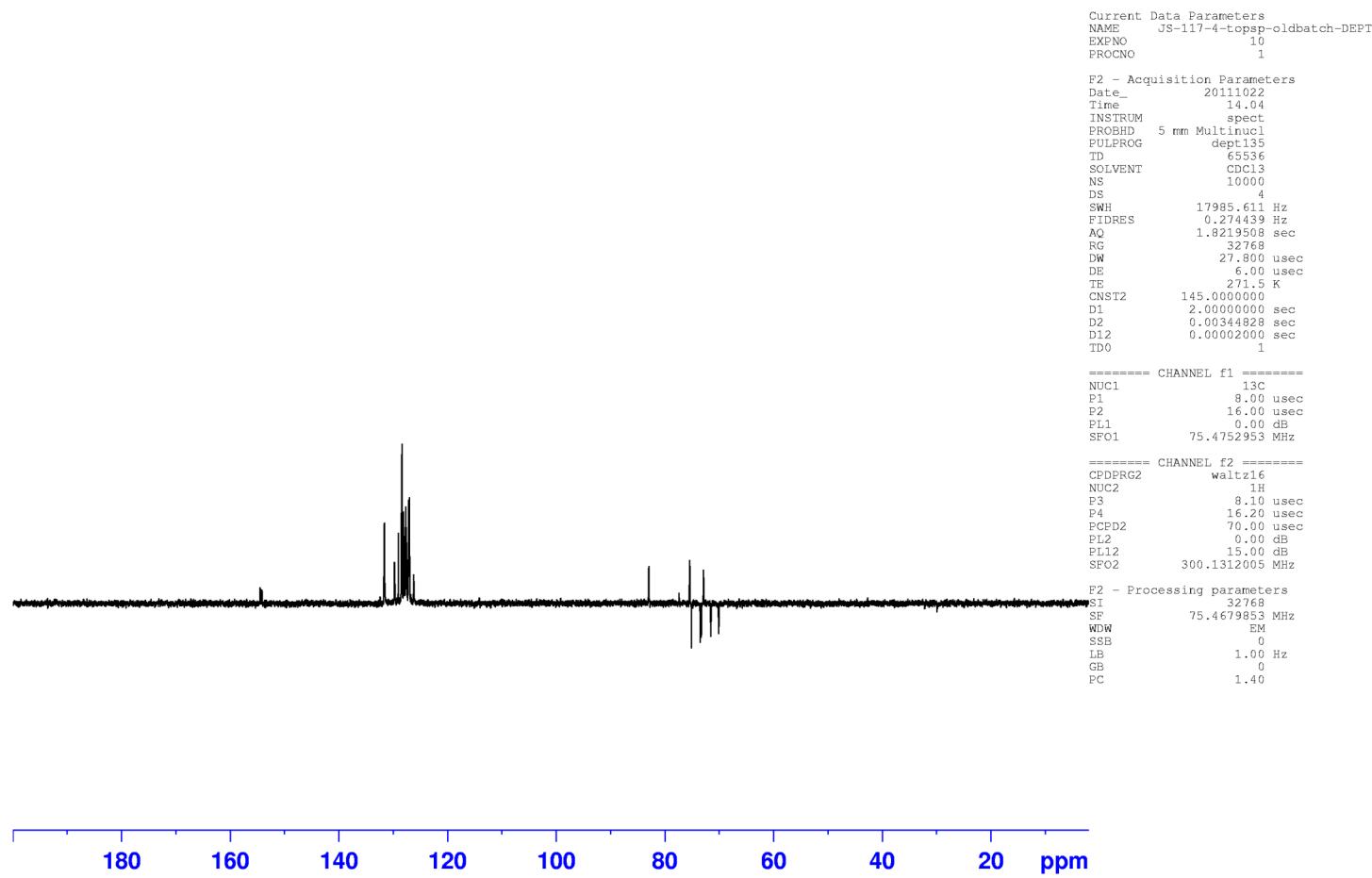
upper isomer **62**

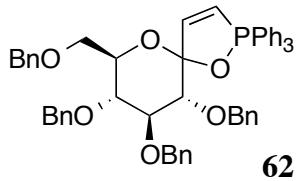




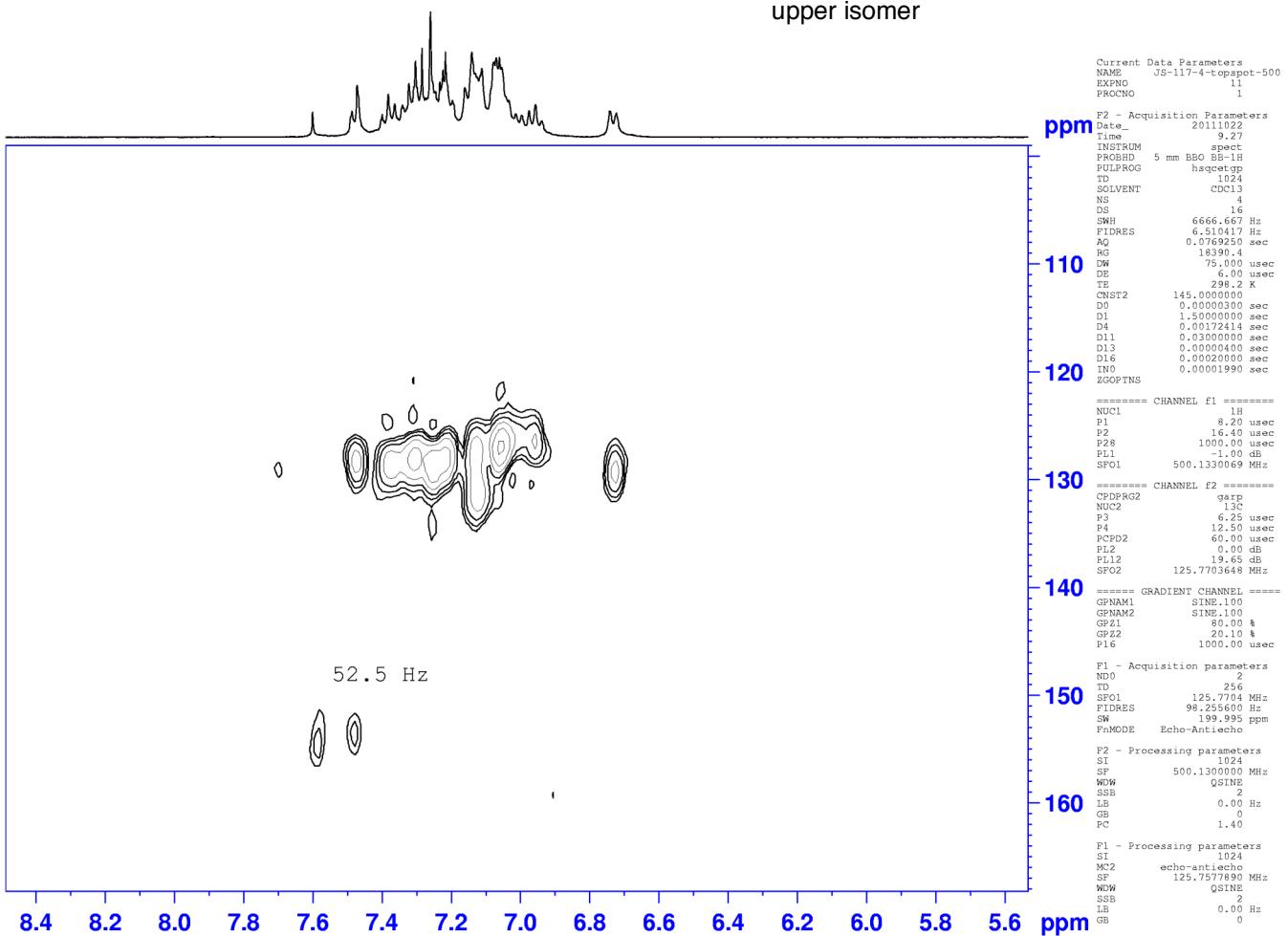
upper isomer

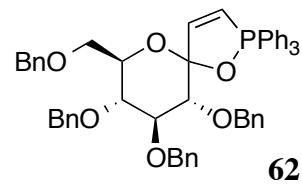




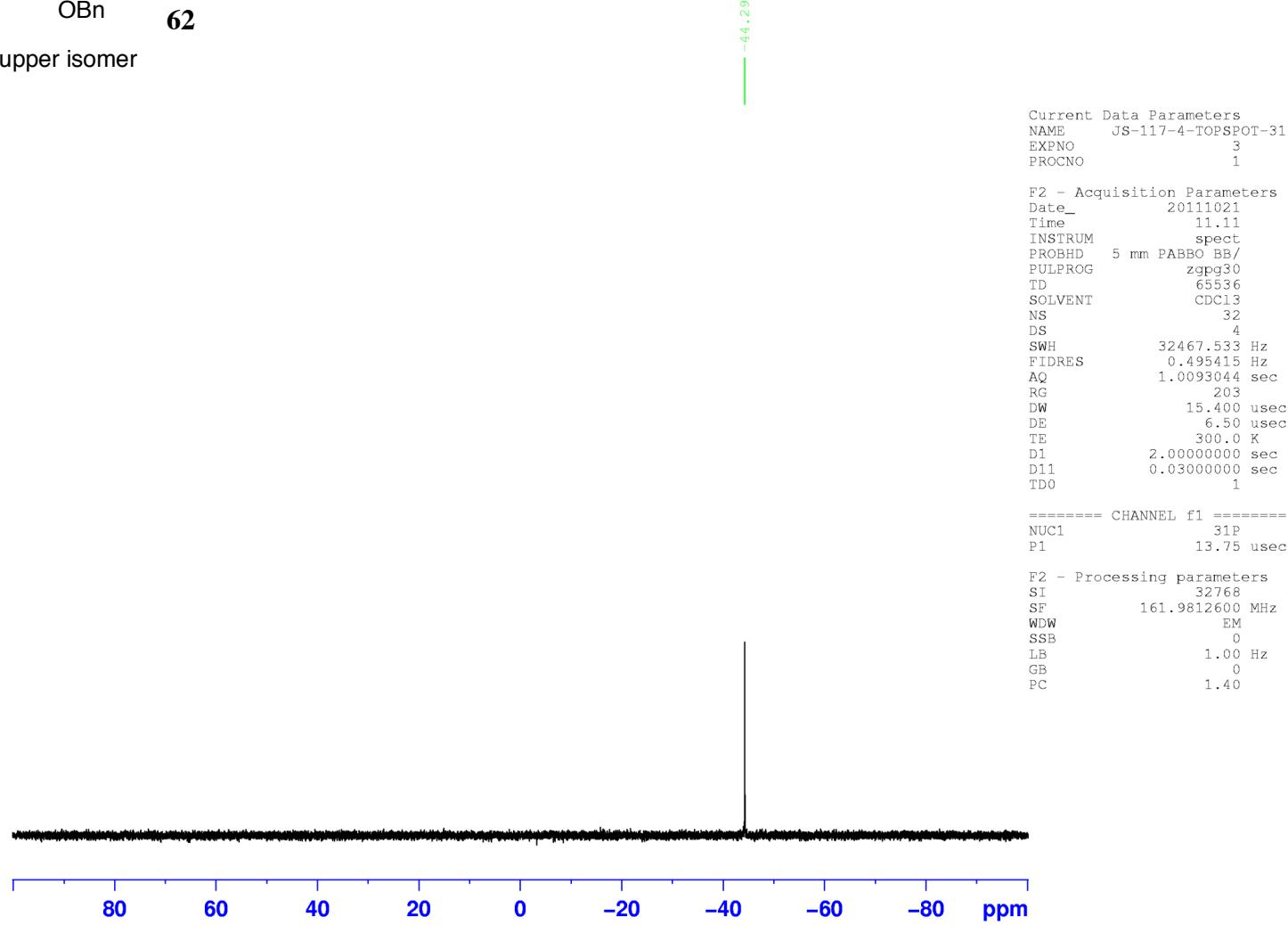


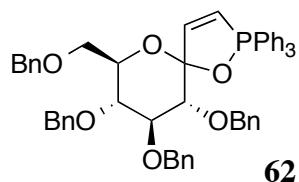
upper isomer



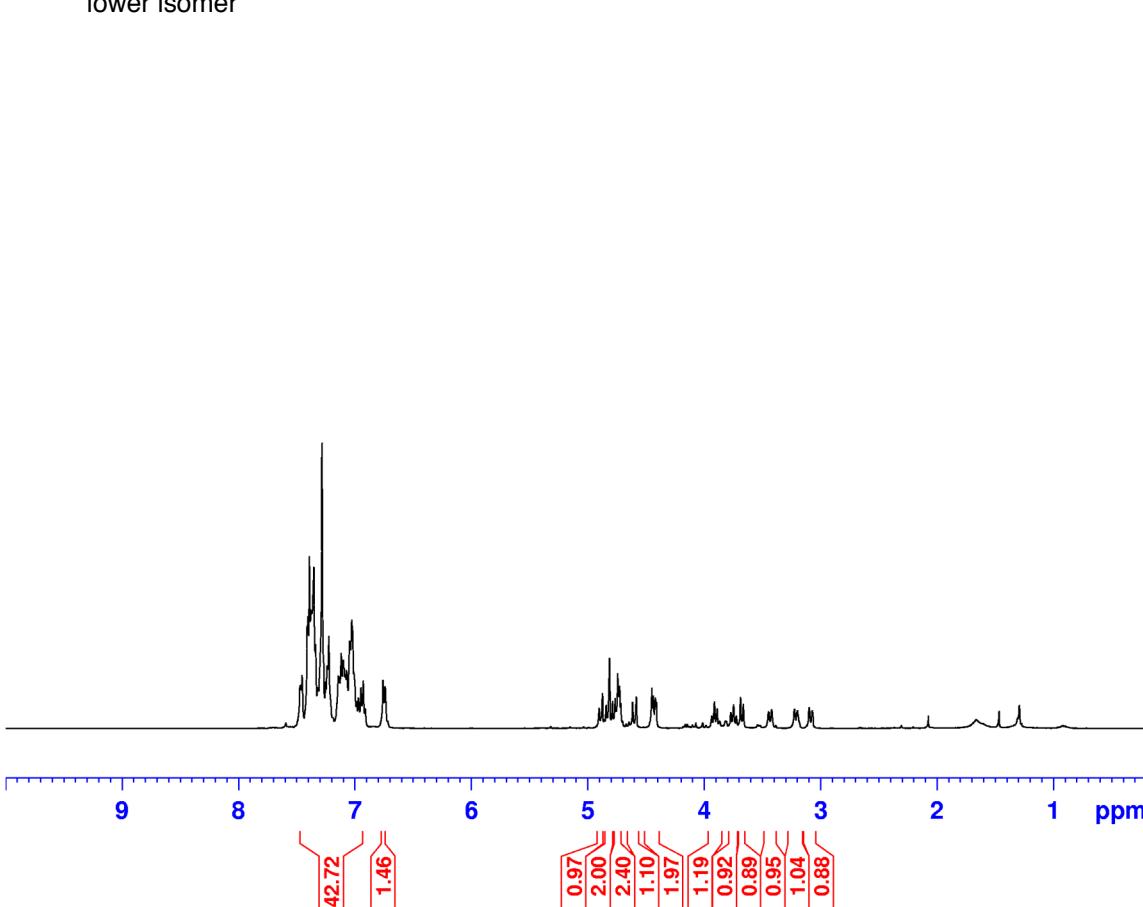


upper isomer





lower isomer

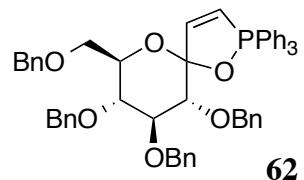


Current Data Parameters
 NAME JS-117-4-botmost-sp
 EXPNO 1
 PROCNO 1

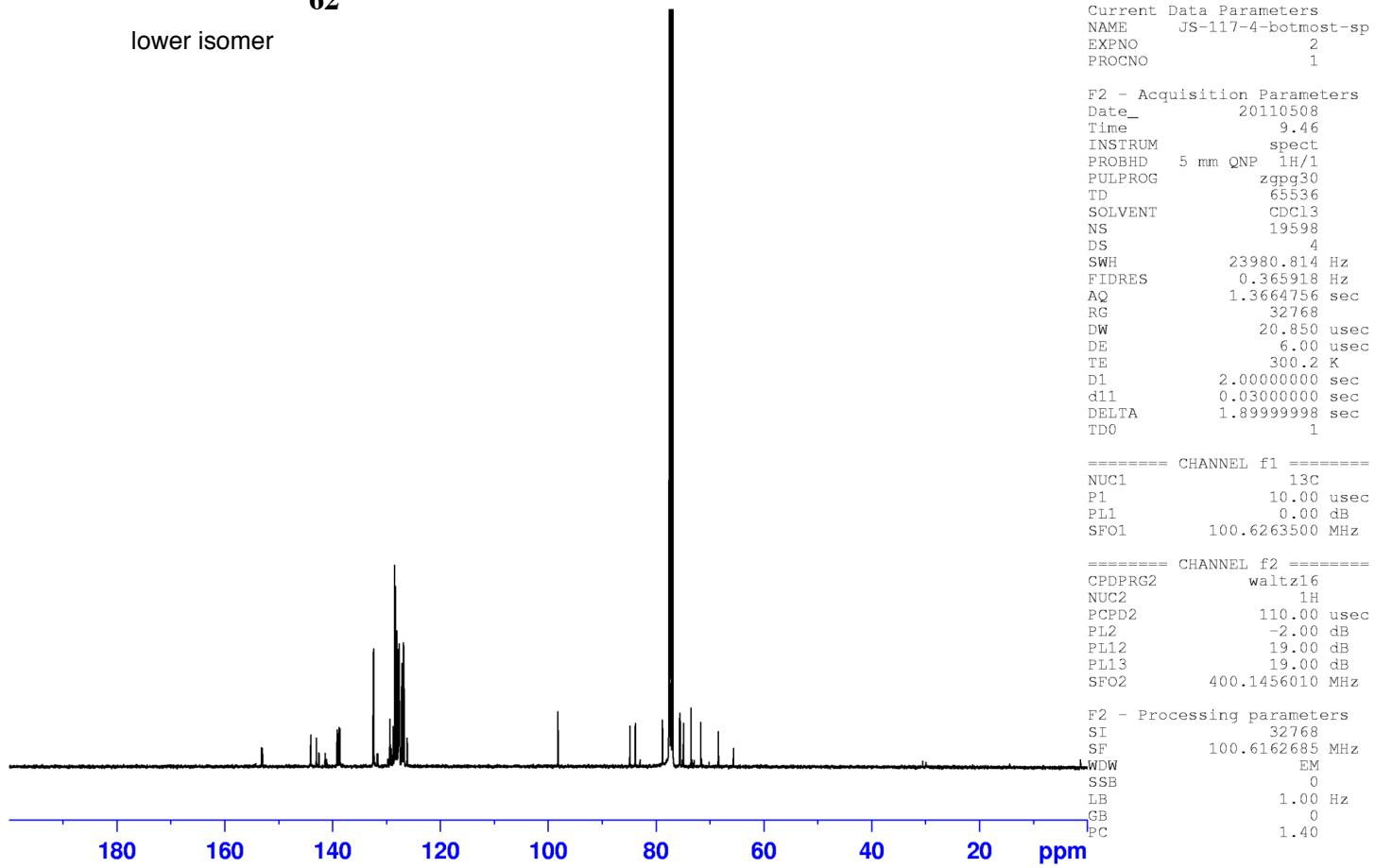
F2 - Acquisition Parameters
 Date_ 20110507
 Time 15.04
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 20
 DS 2
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 228.1
 DW 89.400 usec
 DE 6.00 usec
 TE 300.2 K
 D1 1.5000000 sec
 TDO 1

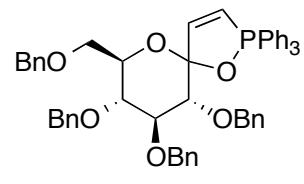
===== CHANNEL f1 =====
 NUC1 1H
 P1 9.20 usec
 PL1 -2.00 dB
 SFO1 400.1464009 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1440000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



lower isomer





upper+lower isomer

-45.41
-48.36

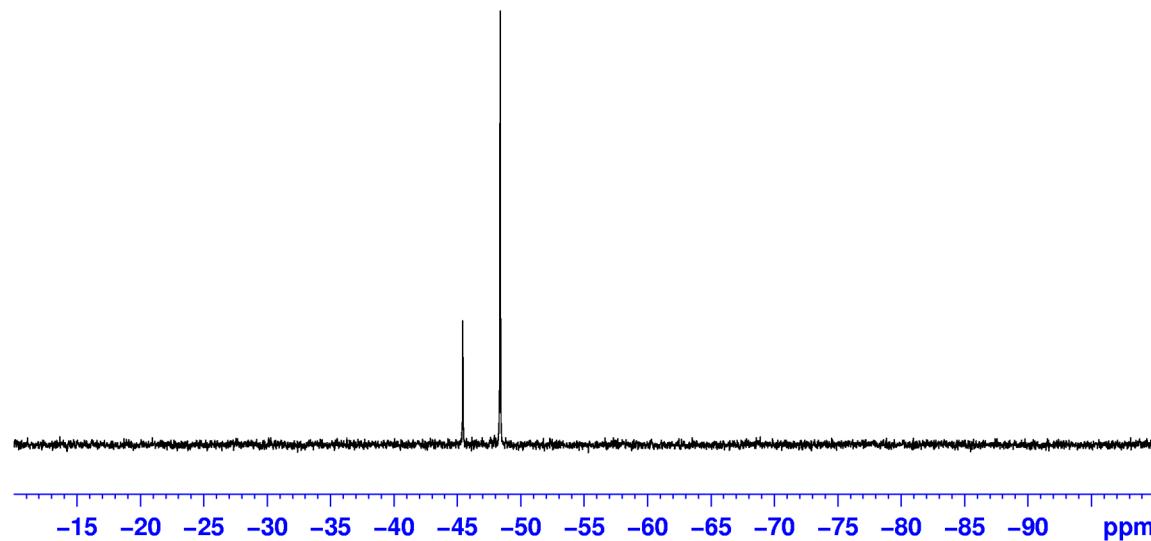
Current Data Parameters
NAME JS-117-4-i-sp3-300-31PHMBC
EXPNO 8
PROCNO 1

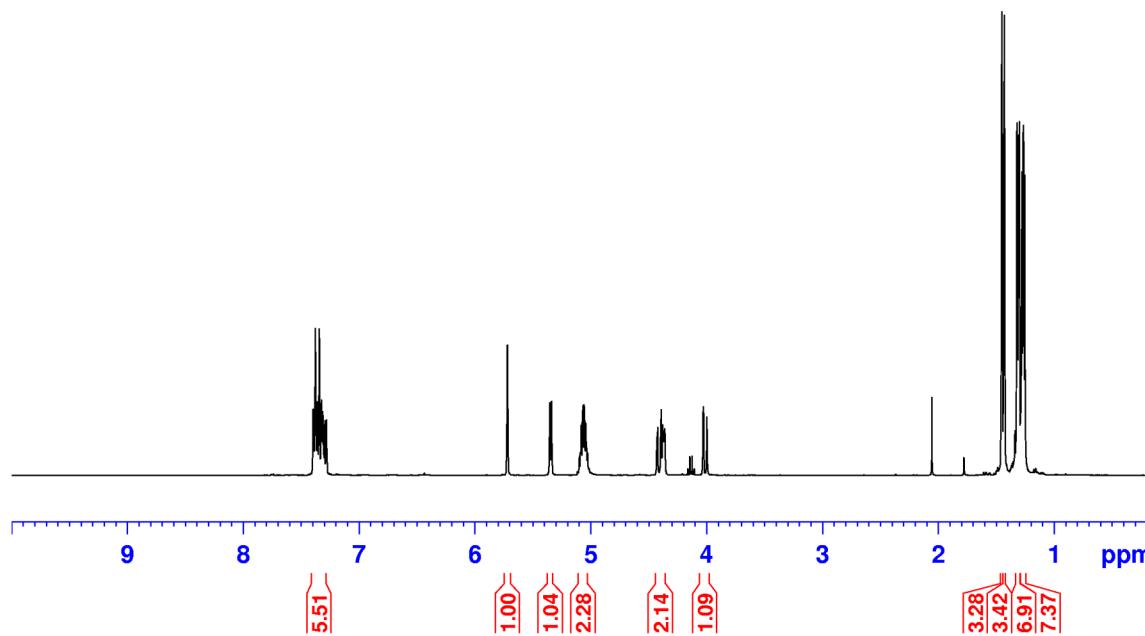
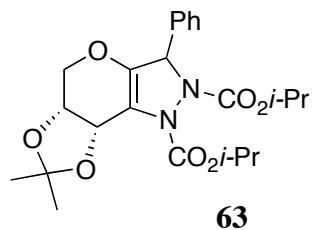
F2 - Acquisition Parameters
Date_ 20110601
Time 11.22
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 75187.969 Hz
FIDRES 1.147277 Hz
AQ 0.4358644 sec
RG 20642.5
DW 6.650 usec
DE 6.00 usec
TE 300.2 K
D1 2.0000000 sec
D11 0.0300000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 31P
P1 8.25 usec
PL1 -1.00 dB
SFO1 121.5130752 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 121.4953994 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



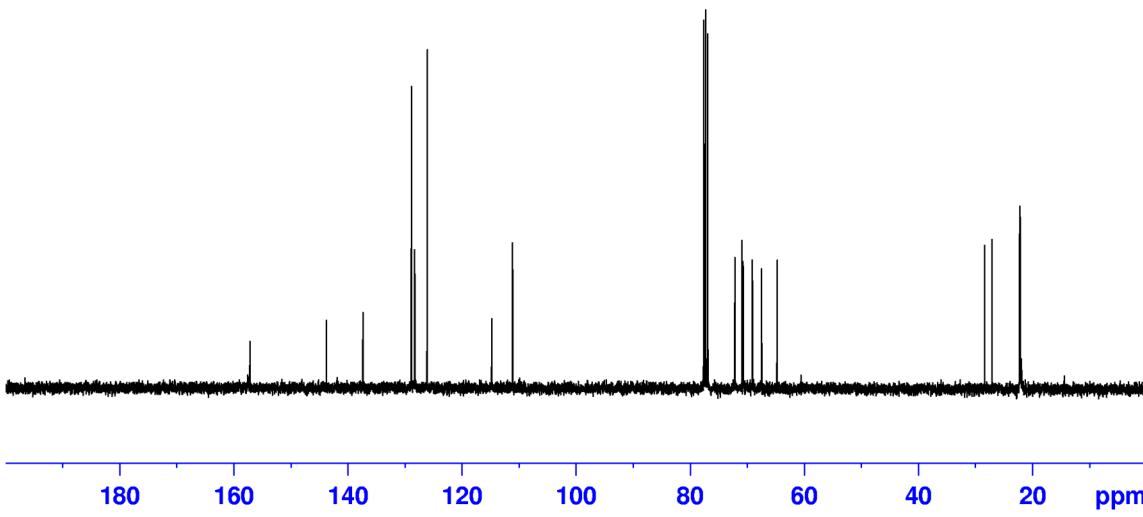
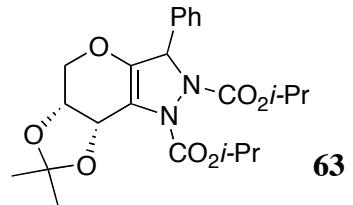


Current Data Parameters
 NAME JS-137-4-top
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110930
 Time 15.43
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894966 sec
 RG 28.5
 DW 62.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec

F2 - Processing parameters
 SI 65536
 SF 400.1440000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

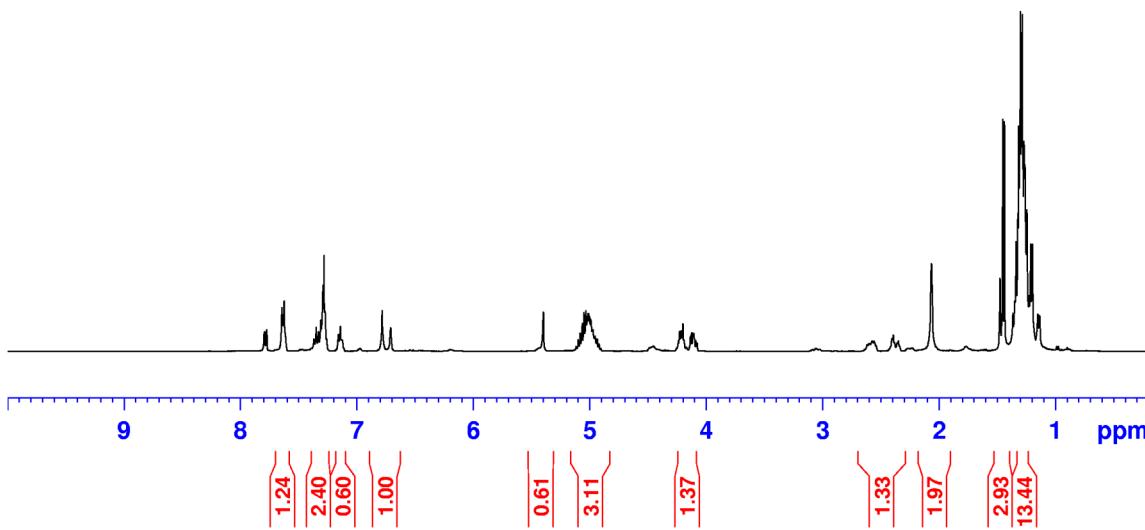
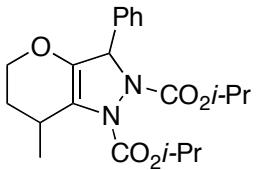


Current Data Parameters
 NAME JS-137-4-top
 EXPNO 2
 PROCNO 1

 F2 - Acquisition Parameters
 Date_ 20110930
 Time 15.48
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 86
 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 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

 ===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec

 F2 - Processing parameters
 SI 32768
 SF 100.6162656 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

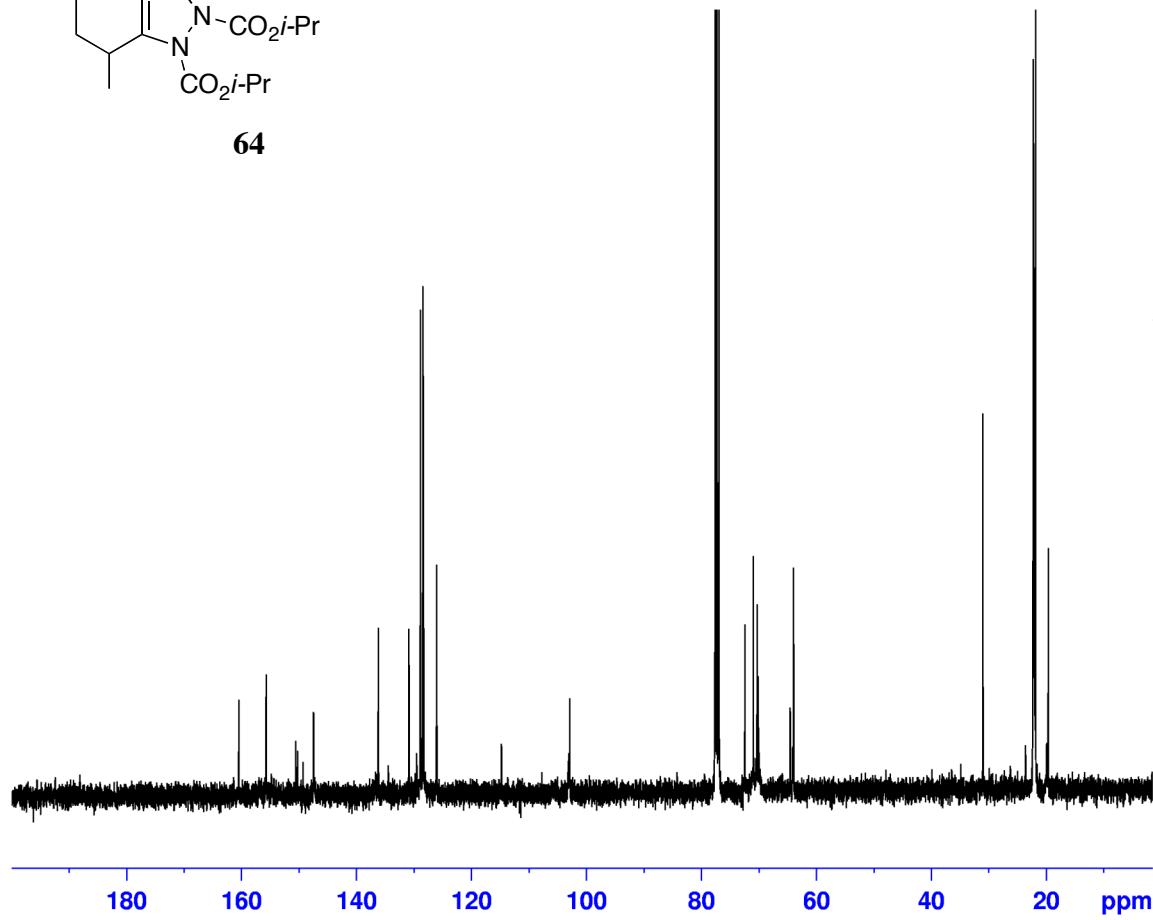
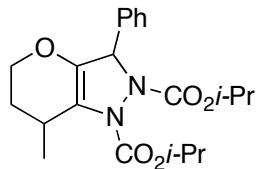


Current Data Parameters
 NAME JS-141-4-a
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20111020
 Time 16.58
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894966 sec
 RG 64
 DW 62.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 ======
 NUC1 1H
 P1 15.00 usec

F2 - Processing parameters
 SI 65536
 SF 400.1440000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



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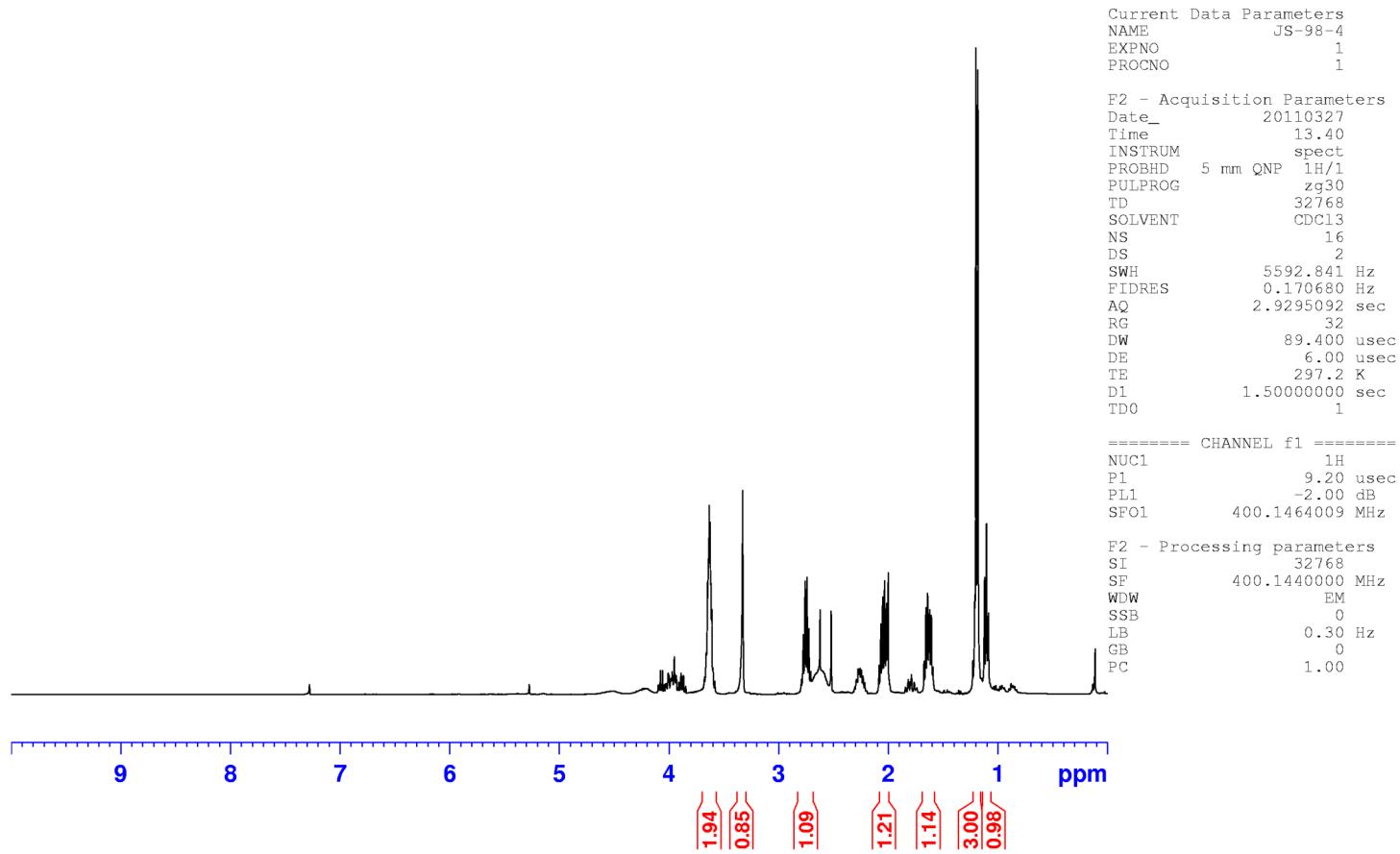
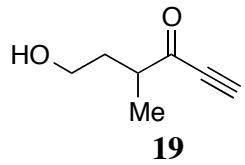
Current Data Parameters
NAME          JS-141-4-a
EXPNO         2
PROCNO        1

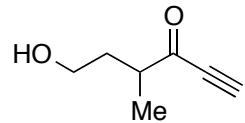
F2 - Acquisition Parameters
Date_        20111020
Time         17.04
INSTRUM      spect
PROBHD      5 mm PABBO BB/
PULPROG     zgpg30
TD           65536
SOLVENT      CDCl3
NS            641
DS             4
SWH          24038.461 Hz
FIDRES       0.366798 Hz
AQ           1.3631988 sec
RG            203
DW           20.800 usec
DE            6.50 usec
TE            300.0 K
D1          2.0000000 sec
D11          0.0300000 sec
TDO           1

===== CHANNEL f1 =====
NUC1          13C
P1           10.00 usec

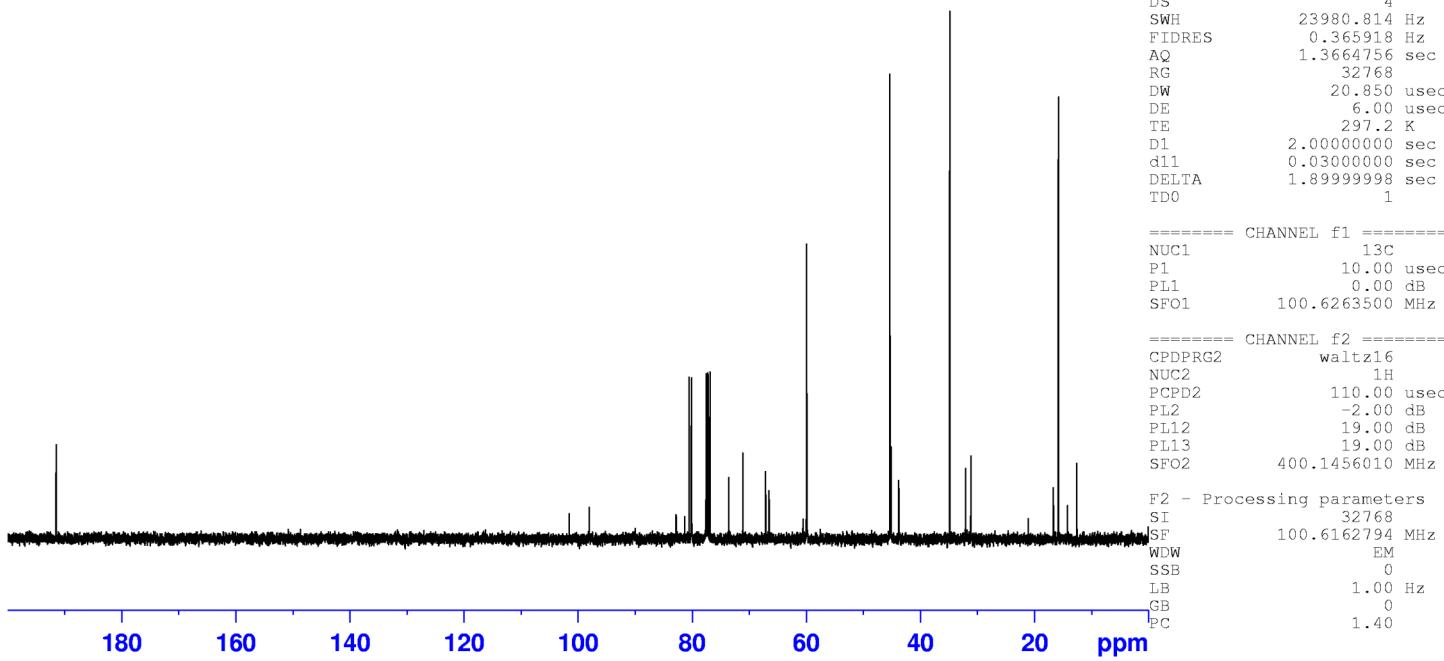
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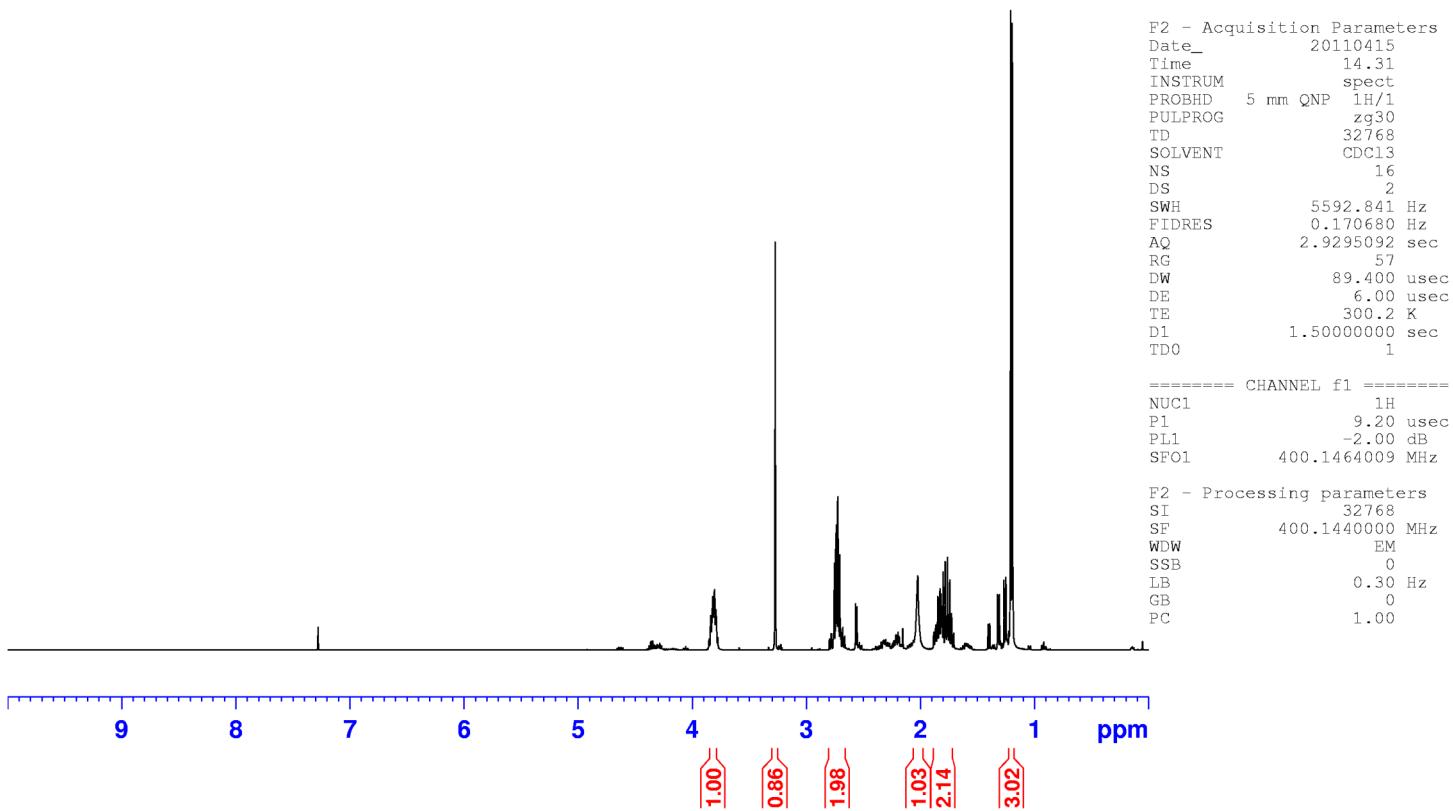
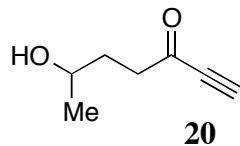
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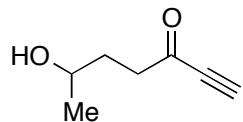




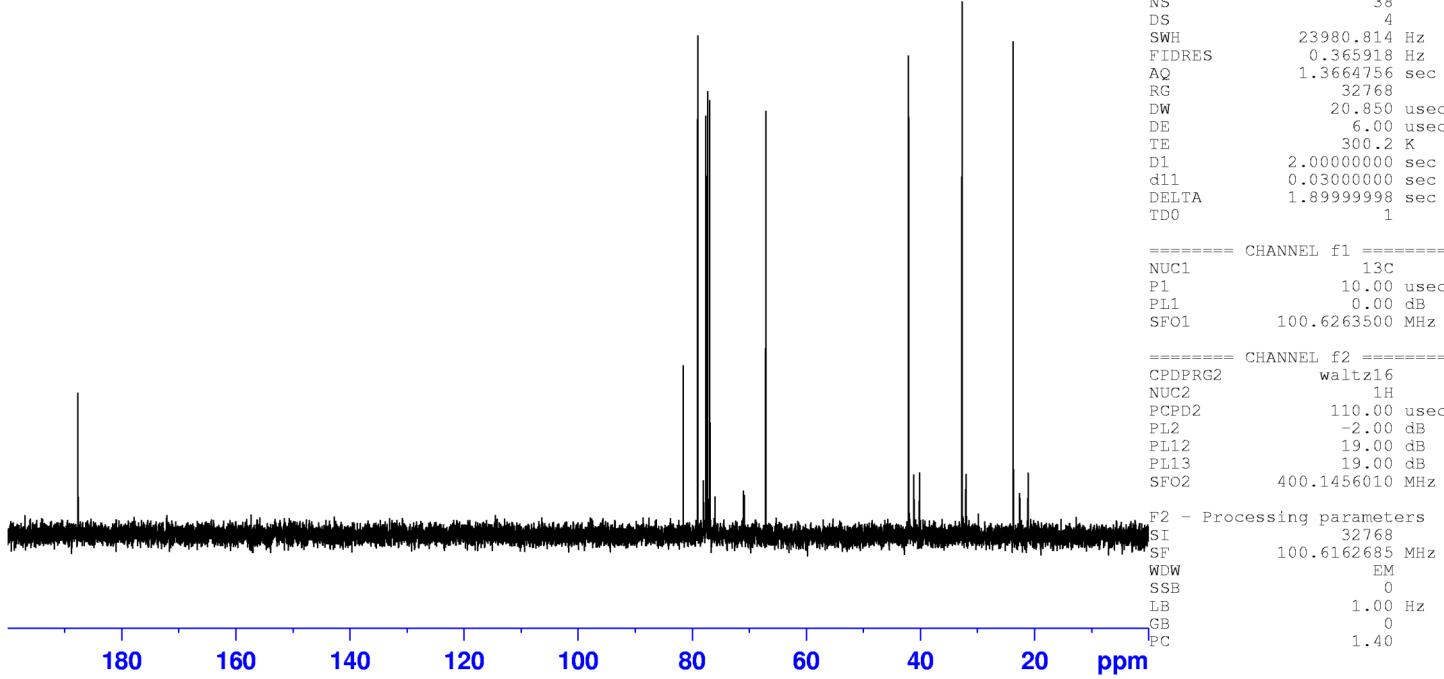
19







20



Sample: UCONN MP10

X-ray Structure Report

for

Mark Peczuh

University of Connecticut

March 19, 2010

Experimental

Data Collection

A colorless block crystal of $O_8C_{18}H_{24}$ having approximate dimensions of $0.30 \times 0.30 \times 0.20$ mm was mounted on a glass fiber. All measurements were made on a Rigaku Mercury2 CCD area detector with filtered Mo-K α radiation.

Indexing was performed from 1 images that were exposed for -10.0 seconds. The crystal-to-detector distance was 49.90 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive orthorhombic cell with dimensions:

$$\begin{aligned}a &= 10.150(6) \text{ \AA} \\b &= 13.718(8) \text{ \AA} \\c &= 26.178(15) \text{ \AA} \\V &= 3644.9(35) \text{ \AA}^3\end{aligned}$$

For $Z = 8$ and F.W. = 368.38, the calculated density is 1.343 g/cm³. The systematic absences of:

$$\begin{aligned}h00: h &\pm 2n \\0k0: k &\pm 2n \\00l: l &\pm 2n\end{aligned}$$

uniquely determine the space group to be:

$$P2_12_12_1 (\#19)$$

The data were collected at a temperature of $-50 \pm 1^\circ\text{C}$ to a maximum 2θ value of 55.0° . A total of 180 oscillation images were collected. A sweep of data was done using ω scans from -120.0 to 60.0° in 1.0° step, at $\chi=54.0^\circ$ and $\phi = 0.0^\circ$. The exposure rate was 40.0 [sec./°]. The detector swing angle was -28.40° . The crystal-to-detector distance was 49.90 mm. Readout was performed in the 0.146 mm pixel mode.

Data Reduction

Of the 11526 reflections that were collected, 7659 were unique ($R_{\text{int}} = 0.055$). Data were collected and processed using CrystalClear (Rigaku). Net intensities and sigmas were derived as follows:

$$F^2 = [\sum(P_i - mB_{\text{ave}})] \cdot L_p^{-1}$$

where P_i is the value in counts of the i^{th} pixel

m is the number of pixels in the integration area

B_{ave} is the background average

L_p is the Lorentz and polarization factor

$$B_{\text{ave}} = \sum(B_j)/n$$

where n is the number of pixels in the background area

B_j is the value of the j^{th} pixel in counts

$$\sigma^2(F^2_{\text{hkl}}) = [(\sum P_i) + m((\sum(B_{\text{ave}} - B_j)^2)/(n-1))] \cdot L_p \cdot \text{errmul} + (\text{erradd} \cdot F^2)^2$$

where $\text{erradd} = 0.00$

$\text{errmul} = 1.00$

The linear absorption coefficient, μ , for Mo-K α radiation is 1.056 cm^{-1} . An empirical absorption correction was applied which resulted in transmission factors ranging from 0.575 to 0.979. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods² and expanded using Fourier techniques³. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement⁴ on F^2 was based on 7655 observed reflections and 470 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \sum \|F_{\text{obs}} - F_{\text{cal}}\| / \sum |F_{\text{obs}}| = 0.0765$$

$$wR2 = [\sum (w(F_o^2 - F_c^2)^2) / \sum w(F_o^2)^2]^{1/2} = 0.1871$$

The standard deviation of an observation of unit weight⁵ was 1.06. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.24 and -0.28 e⁻/Å³, respectively. The absolute structure was deduced based on Flack parameter, 0.1(13), using 3159 Friedel pairs.⁶

Neutral atom scattering factors were taken from Cromer and Waber⁷. Anomalous dispersion effects were included in Fcalc⁸; the values for Δf' and Δf'' were those of Creagh and McAuley⁹. The values for the mass attenuation coefficients are those of Creagh and Hubbell¹⁰. All calculations were performed using the CrystalStructure¹¹ crystallographic software package except for refinement, which was performed using SHELXL-97¹².

References

- (1) CrystalClear: Rigaku Corporation, 1999. CrystalClear Software User's Guide, Molecular Structure Corporation, (c) 2000.J.W.Pflugrath (1999) Acta Cryst. D55, 1718-1725.
- (2) SHELX97: Sheldrick, G.M. (1997).
- (3) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
- (4) Least Squares function minimized: (SHELXL97)

$$\sum w(F_o^2 - F_c^2)^2 \quad \text{where } w = \text{Least Squares weights.}$$

- (5) Standard deviation of an observation of unit weight:

$$[\sum w(F_o^2 - F_c^2)^2 / (N_o - N_v)]^{1/2}$$

where N_o = number of observations

N_v = number of variables

- (6) Flack, H. D. (1983), *Acta Cryst.* A39, 876-881.
- (7) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).
- (8) Ibers, J. A. & Hamilton, W. C.; *Acta Crystallogr.*, 17, 781 (1964).
- (9) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).
- (10) Creagh, D. C. & Hubbell, J.H.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).
- (11) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2007). 9009 New Trails Dr. The Woodlands TX 77381 USA.
- (12) SHELX97: Sheldrick, G.M. (1997).

EXPERIMENTAL DETAILS

A. Crystal Data

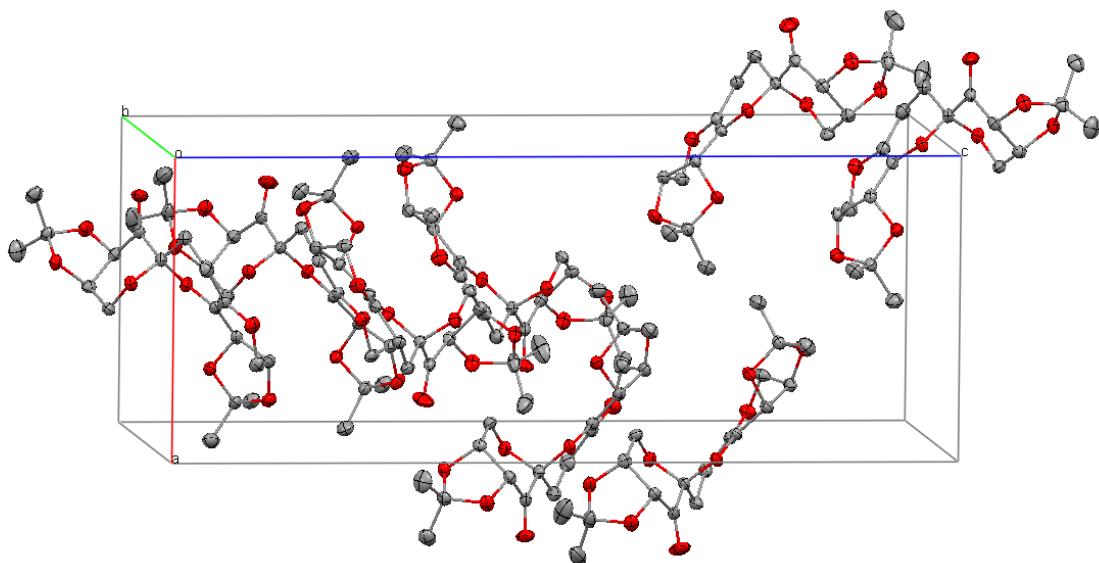
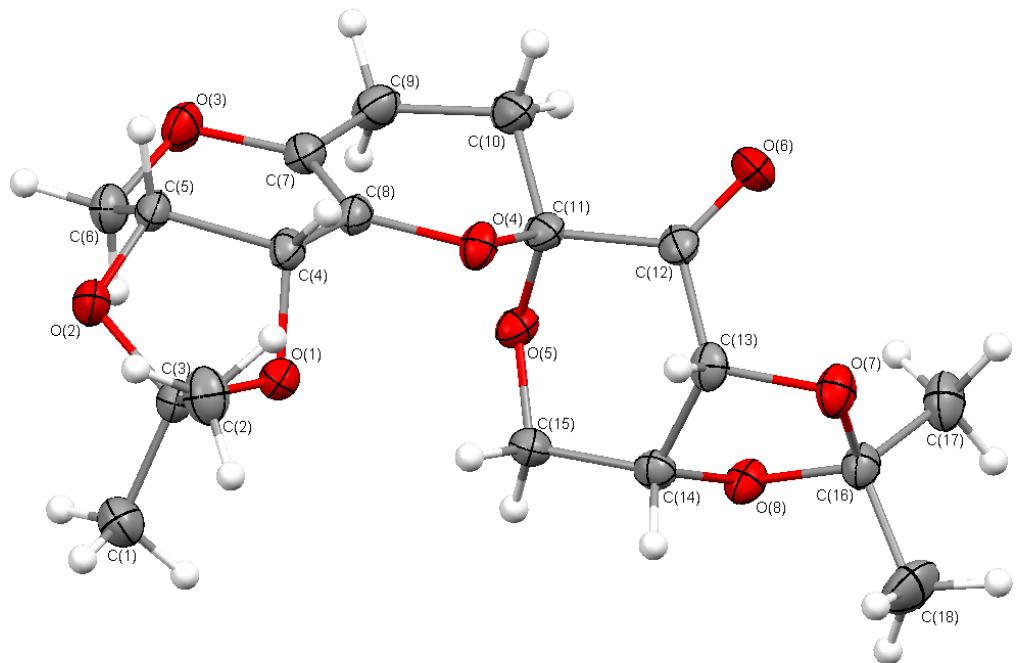
Empirical Formula	O ₈ C ₁₈ H ₂₄
Formula Weight	368.38
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.30 X 0.30 X 0.20 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Indexing Images	6 images @ 10.0 seconds
Detector Position	49.90 mm
Pixel Size	0.146 mm
Lattice Parameters	a = 10.150(6) Å b = 13.718(8) Å c = 26.178(15) Å V = 3644.9(35) Å ³
Space Group	P2 ₁ 2 ₁ 2 ₁ (#19)
Z value	8
D _{calc}	1.343 g/cm ³
F ₀₀₀	1568.00
μ(MoKα)	1.056 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku SCXmini
Radiation	MoK α ($\lambda = 0.71075 \text{ \AA}$)
Detector Aperture	75 mm round
Data Images	180 exposures
ω oscillation Range ($\chi=54.0$, $\phi=0.0$)	-120.0 - 60.0 $^{\circ}$
Exposure Rate	40.0 sec./ $^{\circ}$
Detector Swing Angle	-28.40 $^{\circ}$
Detector Position	49.90 mm
Pixel Size	0.146 mm
$2\theta_{\max}$	55.0 $^{\circ}$
No. of Reflections Measured	Total: 11526 Unique: 7655 ($R_{\text{int}} = 0.055$) Friedel pairs: 3159
Corrections	Lorentz-polarization Absorption (trans. factors: 0.575 - 0.979)

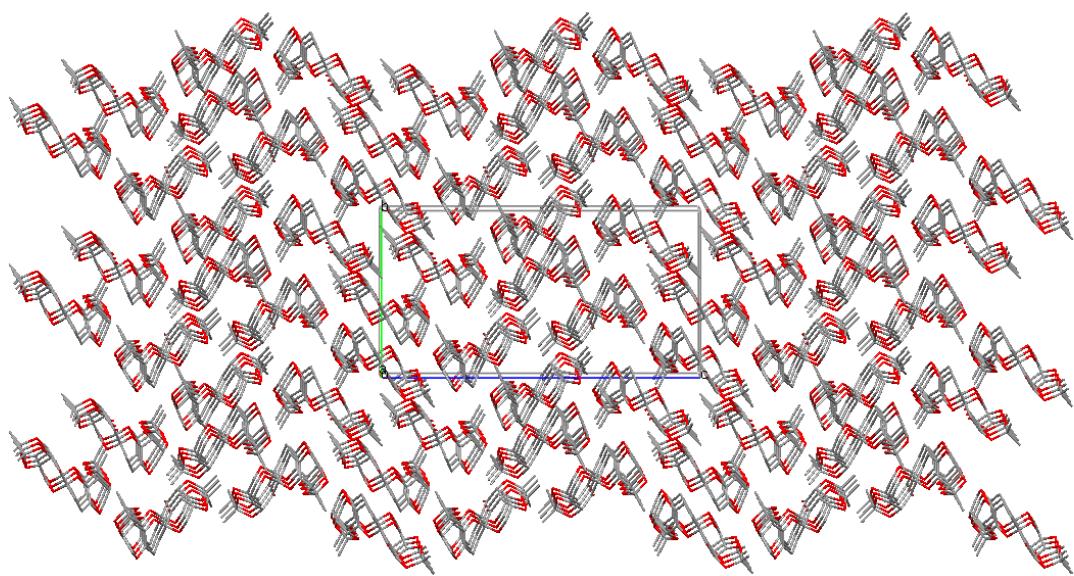
C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\sum w (F_o^2 - F_c^2)^2$
Least Squares Weights	$w = 1 / [\sigma^2(F_o^2) + (0.0747 \cdot P)^2 + 0.5392 \cdot P]$ where $P = (\text{Max}(F_o^2, 0) + 2F_c^2)/3$
$2\theta_{\max}$ cutoff	55.0°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	7655
No. Variables	470
Reflection/Parameter Ratio	16.29
Residuals: R1 ($ I > 2.00\sigma(I)$)	0.0765
Residuals: R (All reflections)	0.1200
Residuals: wR2 (All reflections)	0.1871
Goodness of Fit Indicator	1.062
Flack Parameter	0.1(13)
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.24 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.28 e ⁻ /Å ³

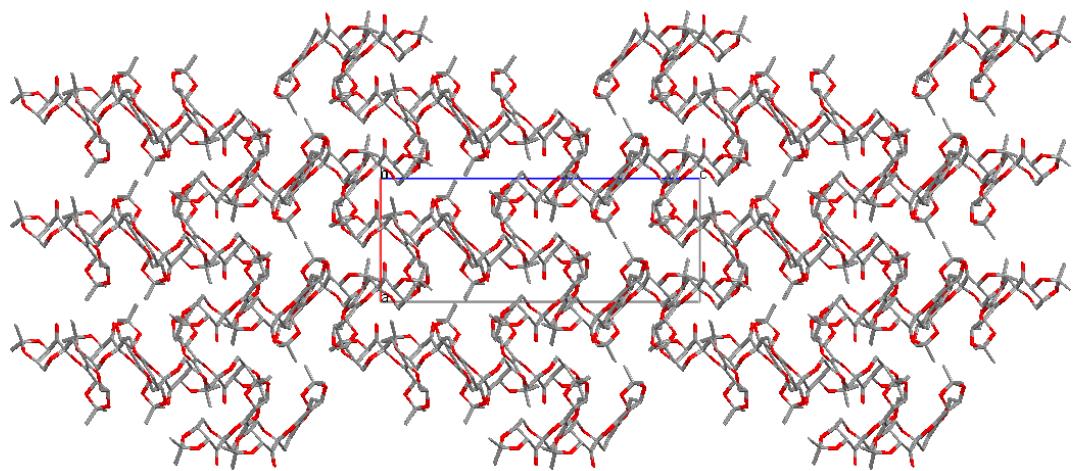




Packing diagram – View down the a -axis



Packing diagram – View down the *b*-axis



Packing diagram – View down the *c*-axis

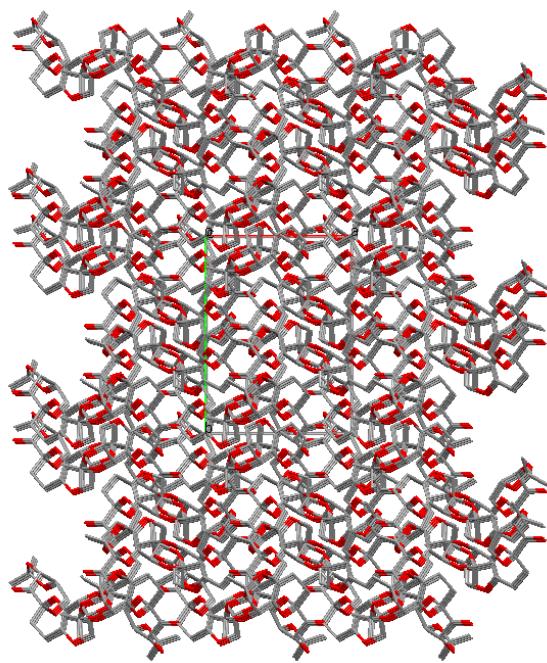


Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B _{eq}
O(1)	0.2500(2)	0.9409(2)	0.42256(10)	3.06(5)
O(2)	0.1565(2)	0.8712(2)	0.35204(11)	3.75(6)
O(3)	0.4237(3)	0.7034(2)	0.38562(11)	3.73(6)
O(4)	0.5272(2)	0.9230(2)	0.45480(9)	2.96(5)
O(5)	0.5442(2)	0.8355(2)	0.53161(9)	2.85(5)
O(6)	0.8127(3)	0.9639(3)	0.51288(12)	5.02(8)
O(7)	0.6860(3)	1.1020(2)	0.57143(10)	3.55(6)
O(8)	0.5938(2)	0.9781(2)	0.61776(9)	3.04(5)
O(9)	0.7456(2)	0.6854(2)	0.25452(9)	3.08(5)
O(10)	0.8177(3)	0.6116(2)	0.32813(10)	3.75(6)
O(11)	0.5888(2)	0.4368(2)	0.27020(11)	3.30(5)
O(12)	0.4638(2)	0.6658(2)	0.21560(10)	2.94(5)
O(13)	0.1783(3)	0.7101(2)	0.16689(14)	5.62(9)
O(14)	0.4541(2)	0.5893(2)	0.13567(10)	3.02(5)
O(15)	0.2800(3)	0.8383(2)	0.09609(11)	3.65(6)
O(16)	0.3899(2)	0.7201(2)	0.05077(10)	3.49(6)
C(1)	0.0166(4)	0.9264(3)	0.42130(19)	4.42(10)
C(2)	0.1304(5)	1.0440(3)	0.36250(18)	4.27(9)
C(3)	0.1378(4)	0.9465(3)	0.38977(14)	3.00(7)
C(4)	0.3568(4)	0.9071(3)	0.39145(14)	2.74(7)
C(5)	0.2877(4)	0.8366(3)	0.35455(14)	2.95(7)
C(6)	0.2907(4)	0.7346(3)	0.37663(17)	3.56(8)
C(7)	0.4930(4)	0.7657(3)	0.41681(15)	3.18(8)
C(8)	0.4605(4)	0.8592(3)	0.42196(14)	2.75(7)
C(9)	0.6067(4)	0.7207(3)	0.44371(17)	3.93(9)
C(10)	0.6967(4)	0.8000(3)	0.46576(16)	3.98(9)
C(11)	0.6144(4)	0.8788(3)	0.49063(13)	2.91(7)
C(12)	0.6946(4)	0.9637(3)	0.51291(14)	3.01(7)
C(13)	0.6102(4)	1.0415(3)	0.53863(14)	2.94(7)
C(14)	0.5116(4)	0.9944(3)	0.57402(15)	2.84(7)
C(15)	0.4508(4)	0.9009(3)	0.55507(15)	3.04(7)
C(16)	0.6754(4)	1.0622(3)	0.62286(15)	3.13(8)
C(17)	0.8069(4)	1.0308(3)	0.64220(18)	4.47(10)
C(18)	0.6096(6)	1.1380(4)	0.6560(2)	5.57(12)
C(19)	0.8492(5)	0.7831(3)	0.32102(18)	4.56(10)
C(20)	0.9769(4)	0.6656(4)	0.26570(19)	4.73(11)
C(21)	0.8485(4)	0.6870(3)	0.29165(14)	3.13(7)

Table 1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$ (continued)

atom	x	y	z	B_{eq}
C(22)	0.6316(3)	0.6453(3)	0.28006(14)	2.75(7)
C(23)	0.6945(4)	0.5691(3)	0.31480(15)	3.27(8)
C(24)	0.7137(3)	0.4757(3)	0.28518(16)	3.18(8)
C(25)	0.5372(3)	0.5990(3)	0.24450(14)	2.55(7)
C(26)	0.5123(3)	0.5036(3)	0.24348(15)	2.72(7)
C(27)	0.4004(4)	0.4584(3)	0.21560(15)	3.04(7)
C(28)	0.3043(4)	0.5379(3)	0.19880(16)	3.25(8)
C(29)	0.3800(3)	0.6240(3)	0.17772(13)	2.58(7)
C(30)	0.2943(4)	0.7094(3)	0.15923(14)	2.90(7)
C(31)	0.3670(4)	0.7882(3)	0.12913(14)	2.91(7)
C(32)	0.4702(4)	0.7463(3)	0.09315(15)	2.97(7)
C(33)	0.5421(4)	0.6595(3)	0.11299(16)	3.16(7)
C(34)	0.2864(5)	0.7911(4)	0.04639(16)	4.24(10)
C(35)	0.1603(5)	0.7379(5)	0.0352(2)	6.48(16)
C(36)	0.3216(7)	0.8675(5)	0.0074(2)	7.9(2)

$$B_{\text{eq}} = \frac{8}{3} \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B _{eq}
H(1)	-0.0023	0.8571	0.4208	5.30
H(2)	-0.0576	0.9619	0.4072	5.30
H(3)	0.0316	0.9474	0.4562	5.30
H(4)	0.0811	1.0898	0.3832	5.13
H(5)	0.0867	1.0357	0.3298	5.13
H(6)	0.2188	1.0688	0.3570	5.13
H(7)	0.3950	0.9624	0.3721	3.29
H(8)	0.3297	0.8383	0.3204	3.53
H(9)	0.2415	0.7334	0.4088	4.28
H(10)	0.2479	0.6894	0.3529	4.28
H(11)	0.6567	0.6801	0.4198	4.72
H(12)	0.5746	0.6788	0.4714	4.72
H(13)	0.7562	0.7712	0.4911	4.77
H(14)	0.7502	0.8283	0.4384	4.77
H(15)	0.5644	1.0814	0.5126	3.53
H(16)	0.4413	1.0416	0.5827	3.41
H(17)	0.4087	0.8676	0.5839	3.65
H(18)	0.3819	0.9167	0.5302	3.65
H(19)	0.8217	0.9631	0.6332	5.36
H(20)	0.8096	1.0379	0.6791	5.36
H(21)	0.8750	1.0709	0.6269	5.36
H(22)	0.6761	1.1799	0.6710	6.68
H(23)	0.5605	1.1059	0.6830	6.68
H(24)	0.5500	1.1768	0.6354	6.68
H(25)	0.9053	0.8296	0.3035	5.47
H(26)	0.8827	0.7722	0.3553	5.47
H(27)	0.7602	0.8086	0.3230	5.47
H(28)	0.9931	0.5959	0.2663	5.68
H(29)	1.0476	0.6990	0.2834	5.68
H(30)	0.9731	0.6879	0.2306	5.68
H(31)	0.5870	0.6961	0.3006	3.30
H(32)	0.6398	0.5578	0.3456	3.92
H(33)	0.7673	0.4886	0.2548	3.82
H(34)	0.7604	0.4281	0.3064	3.82
H(35)	0.3552	0.4117	0.2379	3.64
H(36)	0.4333	0.4232	0.1856	3.64
H(37)	0.2450	0.5121	0.1725	3.91

Table 2. Atomic coordinates and B_{iso} involving hydrogens/ B_{eq} (continued)

atom	x	y	z	B_{eq}
H(38)	0.2508	0.5587	0.2280	3.91
H(39)	0.4088	0.8348	0.1530	3.49
H(40)	0.5335	0.7976	0.0831	3.56
H(41)	0.5903	0.6285	0.0849	3.80
H(42)	0.6066	0.6806	0.1386	3.80
H(43)	0.1668	0.6714	0.0476	7.78
H(44)	0.1451	0.7372	-0.0014	7.78
H(45)	0.0877	0.7706	0.0520	7.78
H(46)	0.2418	0.8974	-0.0055	9.47
H(47)	0.3693	0.8373	-0.0205	9.47
H(48)	0.3766	0.9168	0.0233	9.47

$$B_{eq} = \frac{8}{3} \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos \gamma + 2U_{13}(aa^*cc^*)\cos \beta + 2U_{23}(bb^*cc^*)\cos \alpha)$$

Table 3. Anisotropic displacement parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	0.0387(16)	0.0421(18)	0.0355(14)	-0.0036(13)	0.0006(12)	-0.0054(13)
O(2)	0.0396(17)	0.054(2)	0.0486(16)	-0.0047(15)	-0.0061(14)	-0.0173(15)
O(3)	0.0542(19)	0.0354(18)	0.0519(17)	-0.0053(15)	-0.0041(15)	-0.0126(14)
O(4)	0.0474(17)	0.0347(16)	0.0303(13)	-0.0054(13)	-0.0069(12)	0.0022(11)
O(5)	0.0462(16)	0.0317(16)	0.0306(12)	-0.0003(14)	0.0040(12)	-0.0004(11)
O(6)	0.0313(18)	0.096(3)	0.064(2)	-0.0132(18)	0.0072(15)	-0.027(2)
O(7)	0.0565(18)	0.0355(17)	0.0429(15)	-0.0137(15)	-0.0059(14)	0.0038(13)
O(8)	0.0496(17)	0.0339(16)	0.0319(13)	-0.0039(14)	-0.0016(12)	0.0014(12)
O(9)	0.0377(15)	0.0438(18)	0.0356(13)	-0.0076(13)	-0.0075(12)	0.0056(13)
O(10)	0.0493(18)	0.0487(19)	0.0443(15)	-0.0080(15)	-0.0198(14)	0.0115(15)
O(11)	0.0445(17)	0.0315(16)	0.0492(16)	0.0004(13)	-0.0042(14)	0.0118(13)
O(12)	0.0416(15)	0.0360(16)	0.0341(13)	0.0070(13)	-0.0076(12)	-0.0002(12)
O(13)	0.0391(19)	0.090(3)	0.084(2)	0.026(2)	0.0159(17)	0.033(2)
O(14)	0.0451(16)	0.0342(16)	0.0355(13)	0.0080(14)	0.0026(12)	-0.0022(12)
O(15)	0.0586(19)	0.0369(18)	0.0434(14)	0.0179(15)	-0.0042(14)	-0.0020(13)
O(16)	0.0551(19)	0.0438(18)	0.0336(13)	0.0193(15)	-0.0028(14)	-0.0026(13)
C(1)	0.044(2)	0.060(3)	0.064(3)	-0.008(2)	0.003(2)	-0.008(2)
C(2)	0.053(2)	0.052(3)	0.058(2)	-0.003(2)	-0.013(2)	0.007(2)
C(3)	0.036(2)	0.042(2)	0.0354(19)	-0.006(2)	-0.0020(18)	-0.0082(18)
C(4)	0.041(2)	0.033(2)	0.0307(17)	-0.0109(19)	0.0026(18)	-0.0009(17)
C(5)	0.040(2)	0.041(2)	0.0308(19)	-0.009(2)	-0.0014(18)	-0.0078(18)
C(6)	0.046(2)	0.038(2)	0.051(2)	-0.009(2)	-0.001(2)	-0.005(2)
C(7)	0.040(2)	0.043(2)	0.038(2)	-0.004(2)	0.0034(19)	-0.0049(19)
C(8)	0.038(2)	0.034(2)	0.0324(19)	-0.0097(19)	0.0021(17)	-0.0051(17)
C(9)	0.056(2)	0.046(2)	0.047(2)	0.006(2)	0.003(2)	-0.011(2)
C(10)	0.041(2)	0.070(3)	0.040(2)	0.003(2)	-0.001(2)	-0.012(2)
C(11)	0.037(2)	0.046(2)	0.0267(17)	0.001(2)	0.0007(17)	-0.0031(18)
C(12)	0.039(2)	0.049(2)	0.0258(18)	-0.007(2)	0.0003(18)	0.0011(18)
C(13)	0.041(2)	0.036(2)	0.0347(19)	-0.001(2)	-0.0095(18)	0.0046(18)
C(14)	0.032(2)	0.036(2)	0.040(2)	0.0034(18)	0.0003(18)	-0.0010(18)
C(15)	0.033(2)	0.046(2)	0.037(2)	-0.003(2)	0.0057(18)	-0.0045(19)
C(16)	0.043(2)	0.037(2)	0.039(2)	-0.008(2)	-0.0038(19)	-0.0022(19)
C(17)	0.059(3)	0.052(3)	0.059(2)	-0.019(2)	-0.011(2)	0.000(2)
C(18)	0.095(4)	0.050(3)	0.066(3)	0.003(3)	0.005(3)	-0.022(2)
C(19)	0.061(3)	0.053(3)	0.059(2)	-0.003(2)	-0.016(2)	-0.007(2)
C(20)	0.043(2)	0.079(4)	0.057(2)	-0.005(2)	-0.008(2)	0.001(2)
C(21)	0.041(2)	0.044(2)	0.0345(19)	-0.002(2)	-0.0073(19)	0.0040(18)

Table 3. Anisotropic displacement parameters (continued)

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(22)	0.034(2)	0.038(2)	0.0328(18)	0.0037(19)	-0.0059(17)	0.0024(18)
C(23)	0.037(2)	0.053(3)	0.0340(19)	-0.004(2)	-0.0023(18)	0.009(2)
C(24)	0.034(2)	0.038(2)	0.048(2)	0.0011(19)	-0.0046(19)	0.015(2)
C(25)	0.030(2)	0.035(2)	0.0312(18)	0.0051(18)	-0.0055(17)	-0.0009(17)
C(26)	0.032(2)	0.037(2)	0.034(2)	0.0039(18)	0.0024(17)	0.0091(17)
C(27)	0.038(2)	0.040(2)	0.038(2)	-0.007(2)	-0.0006(18)	0.0022(19)
C(28)	0.035(2)	0.047(2)	0.041(2)	-0.004(2)	-0.0058(18)	0.002(2)
C(29)	0.032(2)	0.038(2)	0.0283(17)	0.0050(18)	-0.0043(16)	-0.0017(17)
C(30)	0.037(2)	0.046(2)	0.0275(17)	0.011(2)	-0.0008(17)	-0.0041(18)
C(31)	0.040(2)	0.034(2)	0.037(2)	0.008(2)	-0.0042(18)	-0.0065(18)
C(32)	0.040(2)	0.034(2)	0.039(2)	-0.003(2)	-0.0014(19)	-0.0010(18)
C(33)	0.034(2)	0.042(2)	0.044(2)	0.009(2)	0.0055(19)	0.0001(19)
C(34)	0.067(3)	0.059(3)	0.035(2)	0.026(2)	-0.000(2)	0.004(2)
C(35)	0.075(4)	0.103(5)	0.069(3)	0.038(4)	-0.025(3)	-0.032(3)
C(36)	0.143(6)	0.090(5)	0.067(3)	0.061(4)	0.024(4)	0.039(3)

The general temperature factor expression: $\exp(-2\pi^2(a^*2U_{11}h^2 + b^*2U_{22}k^2 + c^*2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O(1)	C(3)	1.429(4)	O(1)	C(4)	1.433(4)
O(2)	C(3)	1.442(5)	O(2)	C(5)	1.415(5)
O(3)	C(6)	1.435(5)	O(3)	C(7)	1.375(5)
O(4)	C(8)	1.401(4)	O(4)	C(11)	1.425(4)
O(5)	C(11)	1.419(4)	O(5)	C(15)	1.443(4)
O(6)	C(12)	1.198(5)	O(7)	C(13)	1.420(4)
O(7)	C(16)	1.456(4)	O(8)	C(14)	1.434(4)
O(8)	C(16)	1.427(5)	O(9)	C(21)	1.427(4)
O(9)	C(22)	1.445(4)	O(10)	C(21)	1.442(5)
O(10)	C(23)	1.424(5)	O(11)	C(24)	1.430(4)
O(11)	C(26)	1.390(4)	O(12)	C(25)	1.402(4)
O(12)	C(29)	1.427(4)	O(13)	C(30)	1.195(5)
O(14)	C(29)	1.416(4)	O(14)	C(33)	1.441(5)
O(15)	C(31)	1.414(4)	O(15)	C(34)	1.455(5)
O(16)	C(32)	1.422(4)	O(16)	C(34)	1.437(6)
C(1)	C(3)	1.507(6)	C(2)	C(3)	1.518(6)
C(4)	C(5)	1.537(5)	C(4)	C(8)	1.476(5)
C(5)	C(6)	1.515(6)	C(7)	C(8)	1.331(6)
C(7)	C(9)	1.487(6)	C(9)	C(10)	1.532(6)
C(10)	C(11)	1.513(6)	C(11)	C(12)	1.535(6)
C(12)	C(13)	1.525(6)	C(13)	C(14)	1.510(5)
C(14)	C(15)	1.506(5)	C(16)	C(17)	1.492(6)
C(16)	C(18)	1.509(7)	C(19)	C(21)	1.527(6)
C(20)	C(21)	1.498(6)	C(22)	C(23)	1.526(5)
C(22)	C(25)	1.479(5)	C(23)	C(24)	1.510(6)
C(25)	C(26)	1.333(5)	C(26)	C(27)	1.485(5)
C(27)	C(28)	1.527(6)	C(28)	C(29)	1.514(6)
C(29)	C(30)	1.537(5)	C(30)	C(31)	1.527(5)
C(31)	C(32)	1.522(5)	C(32)	C(33)	1.490(6)
C(34)	C(35)	1.502(8)	C(34)	C(36)	1.505(8)

Table 5. Bond lengths involving hydrogens (\AA)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.970	C(1)	H(2)	0.970
C(1)	H(3)	0.970	C(2)	H(4)	0.970
C(2)	H(5)	0.970	C(2)	H(6)	0.970
C(4)	H(7)	0.990	C(5)	H(8)	0.990
C(6)	H(9)	0.980	C(6)	H(10)	0.980
C(9)	H(11)	0.980	C(9)	H(12)	0.980
C(10)	H(13)	0.980	C(10)	H(14)	0.980
C(13)	H(15)	0.990	C(14)	H(16)	0.990
C(15)	H(17)	0.980	C(15)	H(18)	0.980
C(17)	H(19)	0.970	C(17)	H(20)	0.970
C(17)	H(21)	0.970	C(18)	H(22)	0.970
C(18)	H(23)	0.970	C(18)	H(24)	0.970
C(19)	H(25)	0.970	C(19)	H(26)	0.970
C(19)	H(27)	0.970	C(20)	H(28)	0.970
C(20)	H(29)	0.970	C(20)	H(30)	0.970
C(22)	H(31)	0.990	C(23)	H(32)	0.990
C(24)	H(33)	0.980	C(24)	H(34)	0.980
C(27)	H(35)	0.980	C(27)	H(36)	0.980
C(28)	H(37)	0.980	C(28)	H(38)	0.980
C(31)	H(39)	0.990	C(32)	H(40)	0.990
C(33)	H(41)	0.980	C(33)	H(42)	0.980
C(35)	H(43)	0.970	C(35)	H(44)	0.970
C(35)	H(45)	0.970	C(36)	H(46)	0.970
C(36)	H(47)	0.970	C(36)	H(48)	0.970

Table 6. Bond angles ($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C(3)	O(1)	C(4)	106.2(2)	C(3)	O(2)	C(5)	109.4(3)
C(6)	O(3)	C(7)	113.2(3)	C(8)	O(4)	C(11)	116.0(3)
C(11)	O(5)	C(15)	113.0(3)	C(13)	O(7)	C(16)	107.5(3)
C(14)	O(8)	C(16)	106.6(2)	C(21)	O(9)	C(22)	106.1(2)
C(21)	O(10)	C(23)	108.8(2)	C(24)	O(11)	C(26)	112.8(3)
C(25)	O(12)	C(29)	115.4(2)	C(29)	O(14)	C(33)	115.1(3)
C(31)	O(15)	C(34)	107.6(3)	C(32)	O(16)	C(34)	108.1(3)
O(1)	C(3)	O(2)	105.5(3)	O(1)	C(3)	C(1)	108.2(3)
O(1)	C(3)	C(2)	111.7(3)	O(2)	C(3)	C(1)	110.6(3)
O(2)	C(3)	C(2)	108.4(3)	C(1)	C(3)	C(2)	112.2(3)
O(1)	C(4)	C(5)	102.4(3)	O(1)	C(4)	C(8)	112.1(3)
C(5)	C(4)	C(8)	112.7(3)	O(2)	C(5)	C(4)	104.4(3)
O(2)	C(5)	C(6)	110.3(3)	C(4)	C(5)	C(6)	109.4(3)
O(3)	C(6)	C(5)	110.9(3)	O(3)	C(7)	C(8)	122.2(3)
O(3)	C(7)	C(9)	114.9(3)	C(8)	C(7)	C(9)	123.0(3)
O(4)	C(8)	C(4)	113.5(3)	O(4)	C(8)	C(7)	123.0(3)
C(4)	C(8)	C(7)	123.4(3)	C(7)	C(9)	C(10)	110.3(4)
C(9)	C(10)	C(11)	109.9(3)	O(4)	C(11)	O(5)	111.3(3)
O(4)	C(11)	C(10)	111.3(2)	O(4)	C(11)	C(12)	104.9(3)
O(5)	C(11)	C(10)	107.6(3)	O(5)	C(11)	C(12)	107.3(2)
C(10)	C(11)	C(12)	114.4(3)	O(6)	C(12)	C(11)	122.1(4)
O(6)	C(12)	C(13)	124.0(4)	C(11)	C(12)	C(13)	113.6(3)
O(7)	C(13)	C(12)	111.8(3)	O(7)	C(13)	C(14)	103.8(2)
C(12)	C(13)	C(14)	110.1(3)	O(8)	C(14)	C(13)	99.8(3)
O(8)	C(14)	C(15)	111.6(3)	C(13)	C(14)	C(15)	115.7(3)
O(5)	C(15)	C(14)	113.6(3)	O(7)	C(16)	O(8)	105.0(2)
O(7)	C(16)	C(17)	110.8(3)	O(7)	C(16)	C(18)	107.9(3)
O(8)	C(16)	C(17)	108.5(3)	O(8)	C(16)	C(18)	110.7(3)
C(17)	C(16)	C(18)	113.6(3)	O(9)	C(21)	O(10)	106.4(3)
O(9)	C(21)	C(19)	111.1(3)	O(9)	C(21)	C(20)	109.0(3)
O(10)	C(21)	C(19)	106.7(3)	O(10)	C(21)	C(20)	110.4(3)
C(19)	C(21)	C(20)	113.2(4)	O(9)	C(22)	C(23)	101.6(3)
O(9)	C(22)	C(25)	113.0(2)	C(23)	C(22)	C(25)	110.6(3)
O(10)	C(23)	C(22)	103.5(3)	O(10)	C(23)	C(24)	111.1(3)
C(22)	C(23)	C(24)	109.2(3)	O(11)	C(24)	C(23)	110.1(3)
O(12)	C(25)	C(22)	113.8(3)	O(12)	C(25)	C(26)	122.0(3)
C(22)	C(25)	C(26)	123.9(3)	O(11)	C(26)	C(25)	122.1(3)

Table 6. Bond angles ($^{\circ}$) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
O(11)	C(26)	C(27)	113.6(3)	C(25)	C(26)	C(27)	124.4(3)
C(26)	C(27)	C(28)	109.4(3)	C(27)	C(28)	C(29)	109.7(3)
O(12)	C(29)	O(14)	111.0(3)	O(12)	C(29)	C(28)	111.3(2)
O(12)	C(29)	C(30)	104.5(3)	O(14)	C(29)	C(28)	106.9(3)
O(14)	C(29)	C(30)	108.2(2)	C(28)	C(29)	C(30)	115.0(3)
O(13)	C(30)	C(29)	120.7(3)	O(13)	C(30)	C(31)	123.8(4)
C(29)	C(30)	C(31)	115.4(3)	O(15)	C(31)	C(30)	111.0(3)
O(15)	C(31)	C(32)	103.6(2)	C(30)	C(31)	C(32)	112.6(3)
O(16)	C(32)	C(31)	100.6(3)	O(16)	C(32)	C(33)	110.6(3)
C(31)	C(32)	C(33)	115.0(3)	O(14)	C(33)	C(32)	112.0(3)
O(15)	C(34)	O(16)	105.2(3)	O(15)	C(34)	C(35)	110.7(3)
O(15)	C(34)	C(36)	107.9(4)	O(16)	C(34)	C(35)	108.0(4)
O(16)	C(34)	C(36)	110.6(4)	C(35)	C(34)	C(36)	114.1(4)

Table 7. Bond angles involving hydrogens ($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C(3)	C(1)	H(1)	109.5	C(3)	C(1)	H(2)	109.5
C(3)	C(1)	H(3)	109.5	H(1)	C(1)	H(2)	109.5
H(1)	C(1)	H(3)	109.5	H(2)	C(1)	H(3)	109.5
C(3)	C(2)	H(4)	109.5	C(3)	C(2)	H(5)	109.5
C(3)	C(2)	H(6)	109.5	H(4)	C(2)	H(5)	109.5
H(4)	C(2)	H(6)	109.5	H(5)	C(2)	H(6)	109.5
O(1)	C(4)	H(7)	109.8	C(5)	C(4)	H(7)	109.8
C(8)	C(4)	H(7)	109.8	O(2)	C(5)	H(8)	110.9
C(4)	C(5)	H(8)	110.9	C(6)	C(5)	H(8)	110.9
O(3)	C(6)	H(9)	109.5	O(3)	C(6)	H(10)	109.5
C(5)	C(6)	H(9)	109.5	C(5)	C(6)	H(10)	109.5
H(9)	C(6)	H(10)	108.0	C(7)	C(9)	H(11)	109.6
C(7)	C(9)	H(12)	109.6	C(10)	C(9)	H(11)	109.6
C(10)	C(9)	H(12)	109.6	H(11)	C(9)	H(12)	108.1
C(9)	C(10)	H(13)	109.7	C(9)	C(10)	H(14)	109.7
C(11)	C(10)	H(13)	109.7	C(11)	C(10)	H(14)	109.7
H(13)	C(10)	H(14)	108.2	O(7)	C(13)	H(15)	110.3
C(12)	C(13)	H(15)	110.3	C(14)	C(13)	H(15)	110.3
O(8)	C(14)	H(16)	109.7	C(13)	C(14)	H(16)	109.7
C(15)	C(14)	H(16)	109.7	O(5)	C(15)	H(17)	108.8
O(5)	C(15)	H(18)	108.8	C(14)	C(15)	H(17)	108.8
C(14)	C(15)	H(18)	108.8	H(17)	C(15)	H(18)	107.7
C(16)	C(17)	H(19)	109.5	C(16)	C(17)	H(20)	109.5
C(16)	C(17)	H(21)	109.5	H(19)	C(17)	H(20)	109.5
H(19)	C(17)	H(21)	109.5	H(20)	C(17)	H(21)	109.5
C(16)	C(18)	H(22)	109.5	C(16)	C(18)	H(23)	109.5
C(16)	C(18)	H(24)	109.5	H(22)	C(18)	H(23)	109.5
H(22)	C(18)	H(24)	109.5	H(23)	C(18)	H(24)	109.5
C(21)	C(19)	H(25)	109.5	C(21)	C(19)	H(26)	109.5
C(21)	C(19)	H(27)	109.5	H(25)	C(19)	H(26)	109.5
H(25)	C(19)	H(27)	109.5	H(26)	C(19)	H(27)	109.5
C(21)	C(20)	H(28)	109.5	C(21)	C(20)	H(29)	109.5
C(21)	C(20)	H(30)	109.5	H(28)	C(20)	H(29)	109.5
H(28)	C(20)	H(30)	109.5	H(29)	C(20)	H(30)	109.5
O(9)	C(22)	H(31)	110.4	C(23)	C(22)	H(31)	110.4
C(25)	C(22)	H(31)	110.4	O(10)	C(23)	H(32)	110.9
C(22)	C(23)	H(32)	110.9	C(24)	C(23)	H(32)	110.9

Table 7. Bond angles involving hydrogens ($^{\circ}$) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
O(11)	C(24)	H(33)	109.7	O(11)	C(24)	H(34)	109.6
C(23)	C(24)	H(33)	109.6	C(23)	C(24)	H(34)	109.6
H(33)	C(24)	H(34)	108.2	C(26)	C(27)	H(35)	109.8
C(26)	C(27)	H(36)	109.8	C(28)	C(27)	H(35)	109.8
C(28)	C(27)	H(36)	109.8	H(35)	C(27)	H(36)	108.2
C(27)	C(28)	H(37)	109.7	C(27)	C(28)	H(38)	109.7
C(29)	C(28)	H(37)	109.7	C(29)	C(28)	H(38)	109.7
H(37)	C(28)	H(38)	108.2	O(15)	C(31)	H(39)	109.8
C(30)	C(31)	H(39)	109.8	C(32)	C(31)	H(39)	109.8
O(16)	C(32)	H(40)	110.1	C(31)	C(32)	H(40)	110.1
C(33)	C(32)	H(40)	110.1	O(14)	C(33)	H(41)	109.2
O(14)	C(33)	H(42)	109.2	C(32)	C(33)	H(41)	109.2
C(32)	C(33)	H(42)	109.2	H(41)	C(33)	H(42)	107.9
C(34)	C(35)	H(43)	109.5	C(34)	C(35)	H(44)	109.5
C(34)	C(35)	H(45)	109.5	H(43)	C(35)	H(44)	109.5
H(43)	C(35)	H(45)	109.5	H(44)	C(35)	H(45)	109.5
C(34)	C(36)	H(46)	109.5	C(34)	C(36)	H(47)	109.5
C(34)	C(36)	H(48)	109.5	H(46)	C(36)	H(47)	109.5
H(46)	C(36)	H(48)	109.5	H(47)	C(36)	H(48)	109.5

Table 8. Torsion Angles($^{\circ}$)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(3)	O(1)	C(4)	C(5)	-35.0(3)	C(3)	O(1)	C(4)	C(8)	-156.0(3)
C(4)	O(1)	C(3)	O(2)	30.7(3)	C(4)	O(1)	C(3)	C(1)	149.1(3)
C(4)	O(1)	C(3)	C(2)	-86.9(3)	C(3)	O(2)	C(5)	C(4)	-8.4(3)
C(3)	O(2)	C(5)	C(6)	109.0(3)	C(5)	O(2)	C(3)	O(1)	-13.0(4)
C(5)	O(2)	C(3)	C(1)	-129.8(3)	C(5)	O(2)	C(3)	C(2)	106.8(3)
C(6)	O(3)	C(7)	C(8)	-23.8(5)	C(6)	O(3)	C(7)	C(9)	156.7(3)
C(7)	O(3)	C(6)	C(5)	56.2(4)	C(8)	O(4)	C(11)	O(5)	-76.2(3)
C(8)	O(4)	C(11)	C(10)	43.9(4)	C(8)	O(4)	C(11)	C(12)	168.1(3)
C(11)	O(4)	C(8)	C(4)	170.9(3)	C(11)	O(4)	C(8)	C(7)	-12.7(5)
C(11)	O(5)	C(15)	C(14)	-56.1(4)	C(15)	O(5)	C(11)	O(4)	-51.4(4)
C(15)	O(5)	C(11)	C(10)	-173.6(3)	C(15)	O(5)	C(11)	C(12)	62.8(3)
C(13)	O(7)	C(16)	O(8)	0.7(4)	C(13)	O(7)	C(16)	C(17)	117.7(3)
C(13)	O(7)	C(16)	C(18)	-117.4(3)	C(16)	O(7)	C(13)	C(12)	-94.9(3)
C(16)	O(7)	C(13)	C(14)	23.7(4)	C(14)	O(8)	C(16)	O(7)	-26.6(3)
C(14)	O(8)	C(16)	C(17)	-145.2(3)	C(14)	O(8)	C(16)	C(18)	89.6(3)
C(16)	O(8)	C(14)	C(13)	40.0(3)	C(16)	O(8)	C(14)	C(15)	162.9(3)
C(21)	O(9)	C(22)	C(23)	-36.4(3)	C(21)	O(9)	C(22)	C(25)	-154.9(3)
C(22)	O(9)	C(21)	O(10)	25.7(3)	C(22)	O(9)	C(21)	C(19)	-90.0(3)
C(22)	O(9)	C(21)	C(20)	144.7(3)	C(21)	O(10)	C(23)	C(22)	-19.0(3)
C(21)	O(10)	C(23)	C(24)	98.1(3)	C(23)	O(10)	C(21)	O(9)	-3.2(4)
C(23)	O(10)	C(21)	C(19)	115.4(3)	C(23)	O(10)	C(21)	C(20)	-121.3(3)
C(24)	O(11)	C(26)	C(25)	-17.4(5)	C(24)	O(11)	C(26)	C(27)	164.2(3)
C(26)	O(11)	C(24)	C(23)	54.3(4)	C(25)	O(12)	C(29)	O(14)	-73.7(3)
C(25)	O(12)	C(29)	C(28)	45.2(4)	C(25)	O(12)	C(29)	C(30)	169.9(2)
C(29)	O(12)	C(25)	C(22)	174.8(3)	C(29)	O(12)	C(25)	C(26)	-12.2(5)
C(29)	O(14)	C(33)	C(32)	-60.2(4)	C(33)	O(14)	C(29)	O(12)	-53.4(4)
C(33)	O(14)	C(29)	C(28)	-174.9(3)	C(33)	O(14)	C(29)	C(30)	60.7(4)
C(31)	O(15)	C(34)	O(16)	-6.3(4)	C(31)	O(15)	C(34)	C(35)	110.2(4)
C(31)	O(15)	C(34)	C(36)	-124.3(4)	C(34)	O(15)	C(31)	C(30)	-93.8(3)
C(34)	O(15)	C(31)	C(32)	27.3(4)	C(32)	O(16)	C(34)	O(15)	-19.3(4)
C(32)	O(16)	C(34)	C(35)	-137.6(3)	C(32)	O(16)	C(34)	C(36)	97.0(4)
C(34)	O(16)	C(32)	C(31)	34.8(3)	C(34)	O(16)	C(32)	C(33)	156.8(3)
O(1)	C(4)	C(5)	O(2)	26.4(3)	O(1)	C(4)	C(5)	C(6)	-91.6(3)
O(1)	C(4)	C(8)	O(4)	-66.8(4)	O(1)	C(4)	C(8)	C(7)	116.8(4)
C(5)	C(4)	C(8)	O(4)	178.3(3)	C(5)	C(4)	C(8)	C(7)	1.9(5)
C(8)	C(4)	C(5)	O(2)	147.0(3)	C(8)	C(4)	C(5)	C(6)	29.0(4)
O(2)	C(5)	C(6)	O(3)	-172.5(3)	C(4)	C(5)	C(6)	O(3)	-58.2(4)

Table 8. Torsion angles ($^{\circ}$) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(3)	C(7)	C(8)	O(4)	177.8(3)	O(3)	C(7)	C(8)	C(4)	-6.1(6)
O(3)	C(7)	C(9)	C(10)	165.3(3)	C(8)	C(7)	C(9)	C(10)	-14.2(5)
C(9)	C(7)	C(8)	O(4)	-2.7(6)	C(9)	C(7)	C(8)	C(4)	173.3(3)
C(7)	C(9)	C(10)	C(11)	43.5(4)	C(9)	C(10)	C(11)	O(4)	-59.8(4)
C(9)	C(10)	C(11)	O(5)	62.4(4)	C(9)	C(10)	C(11)	C(12)	-178.5(3)
O(4)	C(11)	C(12)	O(6)	-126.2(4)	O(4)	C(11)	C(12)	C(13)	58.8(3)
O(5)	C(11)	C(12)	O(6)	115.4(4)	O(5)	C(11)	C(12)	C(13)	-59.7(4)
C(10)	C(11)	C(12)	O(6)	-3.9(5)	C(10)	C(11)	C(12)	C(13)	-179.0(3)
O(6)	C(12)	C(13)	O(7)	-12.6(5)	O(6)	C(12)	C(13)	C(14)	-127.4(4)
C(11)	C(12)	C(13)	O(7)	162.4(3)	C(11)	C(12)	C(13)	C(14)	47.5(4)
O(7)	C(13)	C(14)	O(8)	-38.6(3)	O(7)	C(13)	C(14)	C(15)	-158.5(3)
C(12)	C(13)	C(14)	O(8)	81.2(3)	C(12)	C(13)	C(14)	C(15)	-38.7(4)
O(8)	C(14)	C(15)	O(5)	-70.3(4)	C(13)	C(14)	C(15)	O(5)	42.9(4)
O(9)	C(22)	C(23)	O(10)	33.6(3)	O(9)	C(22)	C(23)	C(24)	-84.8(3)
O(9)	C(22)	C(25)	O(12)	-74.1(4)	O(9)	C(22)	C(25)	C(26)	113.0(4)
C(23)	C(22)	C(25)	O(12)	172.7(3)	C(23)	C(22)	C(25)	C(26)	-0.2(4)
C(25)	C(22)	C(23)	O(10)	153.9(3)	C(25)	C(22)	C(23)	C(24)	35.5(4)
O(10)	C(23)	C(24)	O(11)	-177.3(3)	C(22)	C(23)	C(24)	O(11)	-63.8(4)
O(12)	C(25)	C(26)	O(11)	176.9(3)	O(12)	C(25)	C(26)	C(27)	-4.9(6)
C(22)	C(25)	C(26)	O(11)	-10.8(6)	C(22)	C(25)	C(26)	C(27)	167.4(3)
O(11)	C(26)	C(27)	C(28)	166.1(3)	C(25)	C(26)	C(27)	C(28)	-12.2(5)
C(26)	C(27)	C(28)	C(29)	43.2(4)	C(27)	C(28)	C(29)	O(12)	-61.6(4)
C(27)	C(28)	C(29)	O(14)	59.7(3)	C(27)	C(28)	C(29)	C(30)	179.8(3)
O(12)	C(29)	C(30)	O(13)	-114.1(4)	O(12)	C(29)	C(30)	C(31)	68.5(3)
O(14)	C(29)	C(30)	O(13)	127.6(4)	O(14)	C(29)	C(30)	C(31)	-49.8(4)
C(28)	C(29)	C(30)	O(13)	8.2(5)	C(28)	C(29)	C(30)	C(31)	-169.2(3)
O(13)	C(30)	C(31)	O(15)	-22.6(5)	O(13)	C(30)	C(31)	C(32)	-138.2(4)
C(29)	C(30)	C(31)	O(15)	154.7(3)	C(29)	C(30)	C(31)	C(32)	39.1(4)
O(15)	C(31)	C(32)	O(16)	-37.8(3)	O(15)	C(31)	C(32)	C(33)	-156.6(3)
C(30)	C(31)	C(32)	O(16)	82.2(3)	C(30)	C(31)	C(32)	C(33)	-36.6(4)
O(16)	C(32)	C(33)	O(14)	-67.4(4)	C(31)	C(32)	C(33)	O(14)	45.6(4)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	C(36) ¹⁾	3.518(7)	O(2)	C(19) ²⁾	3.442(6)
O(3)	C(17) ³⁾	3.501(6)	O(3)	C(22)	3.567(4)
O(4)	O(14) ⁴⁾	3.294(3)	O(5)	C(6) ⁵⁾	3.599(5)
O(6)	C(1) ⁶⁾	3.209(5)	O(8)	O(10) ³⁾	3.372(4)
O(8)	C(6) ⁵⁾	3.540(5)	O(10)	O(8) ⁵⁾	3.372(4)
O(10)	C(14) ⁵⁾	3.542(4)	O(10)	C(15) ⁵⁾	3.347(4)
O(11)	C(31) ⁷⁾	3.363(4)	O(13)	C(20) ²⁾	3.353(6)
O(14)	O(4) ⁷⁾	3.294(3)	O(14)	C(4) ⁷⁾	3.229(4)
O(16)	C(13) ⁷⁾	3.389(4)	O(16)	C(35) ⁸⁾	3.595(6)
C(1)	O(6) ²⁾	3.209(5)	C(4)	O(14) ⁴⁾	3.229(4)
C(6)	O(5) ³⁾	3.599(5)	C(6)	O(8) ³⁾	3.540(5)
C(13)	O(16) ⁴⁾	3.389(4)	C(14)	O(10) ³⁾	3.542(4)
C(15)	O(10) ³⁾	3.347(4)	C(17)	O(3) ⁵⁾	3.501(6)
C(19)	O(2) ⁶⁾	3.442(6)	C(20)	O(13) ⁶⁾	3.353(6)
C(22)	O(3)	3.567(4)	C(24)	C(31) ⁷⁾	3.510(5)
C(31)	O(11) ⁴⁾	3.363(4)	C(31)	C(24) ⁴⁾	3.510(5)
C(35)	O(16) ⁹⁾	3.595(6)	C(36)	O(1) ¹⁰⁾	3.518(7)

Symmetry Operators:

- | | |
|---------------------------|--------------------------|
| (1) -X+1/2,-Y+2,Z+1/2 | (2) X-1,Y,Z |
| (3) X+1/2-1,-Y+1/2+1,-Z+1 | (4) -X+1,Y+1/2,-Z+1/2 |
| (5) X+1/2,-Y+1/2+1,-Z+1 | (6) X+1,Y,Z |
| (7) -X+1,Y+1/2-1,-Z+1/2 | (8) X+1/2,-Y+1/2+1,-Z |
| (9) X+1/2-1,-Y+1/2+1,-Z | (10) -X+1/2,-Y+2,Z+1/2-1 |

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O(1)	H(41) ¹⁾	3.048	O(1)	H(46) ²⁾	2.911
O(1)	H(47) ²⁾	3.598	O(1)	H(48) ²⁾	3.523
O(2)	H(25) ³⁾	2.905	O(2)	H(26) ³⁾	3.095
O(2)	H(29) ³⁾	3.167	O(2)	H(33) ¹⁾	3.318
O(3)	H(19) ⁴⁾	2.555	O(3)	H(31)	2.777
O(3)	H(32)	3.147	O(4)	H(41) ¹⁾	3.234
O(5)	H(1) ⁵⁾	2.959	O(5)	H(9) ⁵⁾	2.708
O(5)	H(13) ⁴⁾	3.322	O(6)	H(1) ⁶⁾	3.388
O(6)	H(2) ⁶⁾	3.065	O(6)	H(3) ⁶⁾	2.682
O(6)	H(9) ⁵⁾	3.471	O(6)	H(12) ⁵⁾	3.328
O(6)	H(43) ¹⁾	3.264	O(6)	H(48) ⁷⁾	3.563
O(7)	H(40) ⁷⁾	3.176	O(7)	H(43) ¹⁾	3.584
O(7)	H(44) ¹⁾	3.121	O(7)	H(45) ²⁾	3.322
O(7)	H(47) ¹⁾	3.538	O(8)	H(9) ⁵⁾	3.339
O(8)	H(10) ⁵⁾	2.884	O(8)	H(28) ⁴⁾	3.360
O(8)	H(29) ⁴⁾	3.580	O(9)	H(5) ⁸⁾	3.464
O(9)	H(6) ⁸⁾	3.348	O(9)	H(22) ⁹⁾	2.971
O(9)	H(35) ¹⁾	3.275	O(10)	H(11)	3.051
O(10)	H(16) ⁵⁾	3.383	O(10)	H(17) ⁵⁾	2.498
O(11)	H(8) ⁸⁾	2.853	O(11)	H(20) ⁴⁾	3.149
O(11)	H(21) ⁴⁾	3.460	O(11)	H(39) ⁸⁾	2.450
O(13)	H(4) ¹⁰⁾	3.372	O(13)	H(23) ¹¹⁾	3.525
O(13)	H(24) ¹¹⁾	2.909	O(13)	H(28) ³⁾	3.572
O(13)	H(29) ³⁾	3.330	O(13)	H(30) ³⁾	2.685
O(13)	H(34) ¹⁾	3.135	O(14)	H(6) ⁹⁾	3.337
O(14)	H(7) ⁸⁾	2.328	O(15)	H(16) ¹¹⁾	2.808
O(15)	H(24) ¹¹⁾	3.510	O(15)	H(32) ¹⁾	3.474
O(15)	H(34) ¹⁾	2.864	O(16)	H(15) ⁸⁾	2.567
O(16)	H(44) ¹²⁾	2.953	O(16)	H(45) ¹²⁾	3.360
C(1)	H(12) ⁴⁾	3.213	C(1)	H(14) ³⁾	3.053
C(1)	H(25) ³⁾	3.542	C(1)	H(26) ³⁾	3.052
C(1)	H(48) ²⁾	3.595	C(2)	H(30) ¹⁾	3.307
C(2)	H(33) ¹⁾	3.329	C(2)	H(41) ¹⁾	3.358
C(2)	H(42) ¹⁾	3.262	C(2)	H(47) ²⁾	3.468
C(4)	H(36) ¹⁾	2.942	C(4)	H(41) ¹⁾	3.146
C(5)	H(33) ¹⁾	3.584	C(5)	H(36) ¹⁾	3.246
C(6)	H(13) ⁴⁾	3.482	C(6)	H(19) ⁴⁾	2.741

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(6)	H(29) ³⁾	3.505	C(7)	H(13) ⁴⁾	3.441
C(7)	H(31)	3.328	C(7)	H(36) ¹⁾	3.524
C(8)	H(13) ⁴⁾	3.561	C(8)	H(36) ¹⁾	3.141
C(9)	H(3) ⁵⁾	3.572	C(9)	H(17) ⁵⁾	3.374
C(9)	H(18) ⁵⁾	3.439	C(9)	H(32)	3.422
C(10)	H(1) ⁶⁾	3.366	C(10)	H(9) ⁵⁾	3.346
C(10)	H(17) ⁵⁾	3.406	C(10)	H(18) ⁵⁾	3.519
C(10)	H(26)	3.475	C(11)	H(9) ⁵⁾	3.310
C(12)	H(9) ⁵⁾	3.425	C(12)	H(43) ¹⁾	3.551
C(13)	H(45) ²⁾	3.287	C(14)	H(45) ²⁾	3.427
C(15)	H(11) ⁴⁾	3.252	C(15)	H(13) ⁴⁾	3.307
C(15)	H(26) ⁴⁾	3.410	C(16)	H(10) ⁵⁾	3.586
C(17)	H(10) ⁵⁾	3.082	C(17)	H(35) ⁵⁾	3.274
C(17)	H(39) ⁷⁾	3.436	C(17)	H(40) ⁷⁾	3.249
C(18)	H(30) ⁷⁾	3.197	C(19)	H(1) ⁶⁾	3.183
C(19)	H(2) ⁶⁾	3.463	C(19)	H(11)	3.535
C(19)	H(14)	3.291	C(19)	H(17) ⁵⁾	3.292
C(19)	H(35) ¹⁾	3.130	C(19)	H(36) ¹⁾	3.457
C(19)	H(37) ¹⁾	3.288	C(20)	H(5) ⁸⁾	3.138
C(20)	H(10) ⁶⁾	3.589	C(20)	H(38) ⁶⁾	3.295
C(21)	H(17) ⁵⁾	3.399	C(23)	H(11)	3.165
C(23)	H(17) ⁵⁾	3.538	C(23)	H(39) ⁸⁾	3.484
C(24)	H(8) ⁸⁾	3.376	C(24)	H(39) ⁸⁾	2.811
C(25)	H(20) ⁴⁾	3.587	C(26)	H(7) ⁸⁾	3.219
C(26)	H(8) ⁸⁾	3.243	C(26)	H(20) ⁴⁾	2.945
C(27)	H(7) ⁸⁾	3.096	C(27)	H(8) ⁸⁾	3.333
C(27)	H(20) ⁴⁾	2.908	C(27)	H(27) ⁸⁾	2.811
C(28)	H(20) ⁴⁾	3.363	C(28)	H(25) ⁸⁾	3.562
C(28)	H(27) ⁸⁾	3.263	C(29)	H(7) ⁸⁾	3.440
C(30)	H(34) ¹⁾	3.182	C(31)	H(21) ⁹⁾	3.256
C(31)	H(34) ¹⁾	2.865	C(32)	H(15) ⁸⁾	3.592
C(32)	H(21) ⁹⁾	3.089	C(32)	H(44) ¹²⁾	2.995
C(33)	H(6) ⁸⁾	2.838	C(33)	H(7) ⁸⁾	2.806
C(33)	H(44) ¹²⁾	3.410	C(33)	H(46) ¹²⁾	3.554
C(34)	H(15) ⁸⁾	3.598	C(34)	H(16) ¹¹⁾	3.393
C(35)	H(15) ¹¹⁾	3.420	C(35)	H(16) ¹¹⁾	3.430
C(35)	H(24) ¹¹⁾	3.580	C(35)	H(40) ¹³⁾	3.387

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(35)	H(47) ¹³⁾	3.152	C(36)	H(3) ¹¹⁾	3.235
C(36)	H(4) ¹¹⁾	3.448	C(36)	H(16) ¹¹⁾	3.544
C(36)	H(41) ¹³⁾	3.370	C(36)	H(44) ¹²⁾	3.587
H(1)	O(5) ⁴⁾	2.959	H(1)	O(6) ³⁾	3.388
H(1)	C(10) ³⁾	3.366	H(1)	C(19) ³⁾	3.183
H(1)	H(12) ⁴⁾	2.969	H(1)	H(13) ³⁾	3.283
H(1)	H(14) ³⁾	2.584	H(1)	H(17) ⁴⁾	3.214
H(1)	H(25) ³⁾	3.233	H(1)	H(26) ³⁾	2.381
H(1)	H(27) ³⁾	3.580	H(2)	O(6) ³⁾	3.065
H(2)	C(19) ³⁾	3.463	H(2)	H(14) ³⁾	2.798
H(2)	H(25) ³⁾	3.285	H(2)	H(26) ³⁾	2.998
H(2)	H(27) ³⁾	3.564	H(2)	H(37) ¹⁴⁾	2.905
H(2)	H(43) ¹⁴⁾	3.301	H(3)	O(6) ³⁾	2.682
H(3)	C(9) ⁴⁾	3.572	H(3)	C(36) ²⁾	3.235
H(3)	H(12) ⁴⁾	2.604	H(3)	H(14) ³⁾	3.323
H(3)	H(46) ²⁾	3.290	H(3)	H(47) ²⁾	3.179
H(3)	H(48) ²⁾	2.724	H(4)	O(13) ¹⁴⁾	3.372
H(4)	C(36) ²⁾	3.448	H(4)	H(22) ¹⁵⁾	3.595
H(4)	H(24) ¹⁵⁾	3.254	H(4)	H(30) ¹⁾	3.315
H(4)	H(41) ¹⁾	3.479	H(4)	H(42) ¹⁾	3.454
H(4)	H(43) ¹⁴⁾	3.295	H(4)	H(45) ¹⁴⁾	3.457
H(4)	H(46) ²⁾	3.427	H(4)	H(47) ²⁾	2.756
H(5)	O(9) ¹⁾	3.464	H(5)	C(20) ¹⁾	3.138
H(5)	H(25) ³⁾	3.443	H(5)	H(28) ¹⁾	2.770
H(5)	H(30) ¹⁾	2.689	H(5)	H(33) ¹⁾	2.743
H(5)	H(37) ¹⁴⁾	3.382	H(6)	O(9) ¹⁾	3.348
H(6)	O(14) ¹⁾	3.337	H(6)	C(33) ¹⁾	2.838
H(6)	H(22) ¹⁵⁾	3.550	H(6)	H(30) ¹⁾	3.423
H(6)	H(33) ¹⁾	3.129	H(6)	H(41) ¹⁾	2.596
H(6)	H(42) ¹⁾	2.347	H(6)	H(47) ²⁾	3.569
H(7)	O(14) ¹⁾	2.328	H(7)	C(26) ¹⁾	3.219
H(7)	C(27) ¹⁾	3.096	H(7)	C(29) ¹⁾	3.440
H(7)	C(33) ¹⁾	2.806	H(7)	H(36) ¹⁾	2.369
H(7)	H(41) ¹⁾	2.547	H(7)	H(42) ¹⁾	3.006
H(8)	O(11) ¹⁾	2.853	H(8)	C(24) ¹⁾	3.376
H(8)	C(26) ¹⁾	3.243	H(8)	C(27) ¹⁾	3.333
H(8)	H(29) ³⁾	3.576	H(8)	H(31)	3.300

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(8)	H(33) ¹⁾	3.016	H(8)	H(36) ¹⁾	2.677
H(9)	O(5) ⁴⁾	2.708	H(9)	O(6) ⁴⁾	3.471
H(9)	O(8) ⁴⁾	3.339	H(9)	C(10) ⁴⁾	3.346
H(9)	C(11) ⁴⁾	3.310	H(9)	C(12) ⁴⁾	3.425
H(9)	H(13) ⁴⁾	2.625	H(9)	H(19) ⁴⁾	3.022
H(10)	O(8) ⁴⁾	2.884	H(10)	C(16) ⁴⁾	3.586
H(10)	C(17) ⁴⁾	3.082	H(10)	C(20) ³⁾	3.589
H(10)	H(19) ⁴⁾	2.251	H(10)	H(20) ⁴⁾	3.287
H(10)	H(29) ³⁾	2.730	H(11)	O(10)	3.051
H(11)	C(15) ⁵⁾	3.252	H(11)	C(19)	3.535
H(11)	C(23)	3.165	H(11)	H(17) ⁵⁾	2.641
H(11)	H(18) ⁵⁾	2.951	H(11)	H(26)	3.115
H(11)	H(27)	3.261	H(11)	H(31)	3.206
H(11)	H(32)	2.573	H(12)	O(6) ⁴⁾	3.328
H(12)	C(1) ⁵⁾	3.213	H(12)	H(1) ⁵⁾	2.969
H(12)	H(3) ⁵⁾	2.604	H(12)	H(13) ⁴⁾	3.447
H(12)	H(18) ⁵⁾	3.383	H(13)	O(5) ⁵⁾	3.322
H(13)	C(6) ⁵⁾	3.482	H(13)	C(7) ⁵⁾	3.441
H(13)	C(8) ⁵⁾	3.561	H(13)	C(15) ⁵⁾	3.307
H(13)	H(1) ⁶⁾	3.283	H(13)	H(9) ⁵⁾	2.625
H(13)	H(12) ⁵⁾	3.447	H(13)	H(17) ⁵⁾	3.141
H(13)	H(18) ⁵⁾	2.930	H(14)	C(1) ⁶⁾	3.053
H(14)	C(19)	3.291	H(14)	H(1) ⁶⁾	2.584
H(14)	H(2) ⁶⁾	2.798	H(14)	H(3) ⁶⁾	3.323
H(14)	H(17) ⁵⁾	3.185	H(14)	H(26)	2.671
H(14)	H(27)	3.035	H(15)	O(16) ¹⁾	2.567
H(15)	C(32) ¹⁾	3.592	H(15)	C(34) ¹⁾	3.598
H(15)	C(35) ²⁾	3.420	H(15)	H(41) ¹⁾	3.066
H(15)	H(43) ¹⁾	3.384	H(15)	H(44) ²⁾	3.293
H(15)	H(45) ²⁾	2.751	H(15)	H(46) ²⁾	3.157
H(15)	H(47) ¹⁾	3.580	H(16)	O(10) ⁴⁾	3.383
H(16)	O(15) ²⁾	2.808	H(16)	C(34) ²⁾	3.393
H(16)	C(35) ²⁾	3.430	H(16)	C(36) ²⁾	3.544
H(16)	H(34) ⁴⁾	3.460	H(16)	H(45) ²⁾	2.714
H(16)	H(46) ²⁾	3.080	H(17)	O(10) ⁴⁾	2.498
H(17)	C(9) ⁴⁾	3.374	H(17)	C(10) ⁴⁾	3.406
H(17)	C(19) ⁴⁾	3.292	H(17)	C(21) ⁴⁾	3.399

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(17)	C(23) ⁴⁾	3.538	H(17)	H(1) ⁵⁾	3.214
H(17)	H(11) ⁴⁾	2.641	H(17)	H(13) ⁴⁾	3.141
H(17)	H(14) ⁴⁾	3.185	H(17)	H(26) ⁴⁾	2.507
H(17)	H(32) ⁴⁾	3.451	H(18)	C(9) ⁴⁾	3.439
H(18)	C(10) ⁴⁾	3.519	H(18)	H(11) ⁴⁾	2.951
H(18)	H(12) ⁴⁾	3.383	H(18)	H(13) ⁴⁾	2.930
H(18)	H(46) ²⁾	2.992	H(18)	H(48) ²⁾	3.483
H(19)	O(3) ⁵⁾	2.555	H(19)	C(6) ⁵⁾	2.741
H(19)	H(9) ⁵⁾	3.022	H(19)	H(10) ⁵⁾	2.251
H(19)	H(32) ⁵⁾	3.288	H(20)	O(11) ⁵⁾	3.149
H(20)	C(25) ⁵⁾	3.587	H(20)	C(26) ⁵⁾	2.945
H(20)	C(27) ⁵⁾	2.908	H(20)	C(28) ⁵⁾	3.363
H(20)	H(10) ⁵⁾	3.287	H(20)	H(35) ⁵⁾	2.329
H(20)	H(38) ⁵⁾	2.834	H(20)	H(39) ⁷⁾	3.419
H(21)	O(11) ⁵⁾	3.460	H(21)	C(31) ⁷⁾	3.256
H(21)	C(32) ⁷⁾	3.089	H(21)	H(32) ⁵⁾	3.296
H(21)	H(35) ⁵⁾	3.553	H(21)	H(39) ⁷⁾	2.637
H(21)	H(40) ⁷⁾	2.330	H(21)	H(42) ⁷⁾	3.428
H(22)	O(9) ⁷⁾	2.971	H(22)	H(4) ¹⁶⁾	3.595
H(22)	H(6) ¹⁶⁾	3.550	H(22)	H(25) ⁷⁾	3.569
H(22)	H(30) ⁷⁾	2.831	H(22)	H(35) ⁵⁾	3.253
H(22)	H(42) ⁷⁾	3.041	H(23)	O(13) ²⁾	3.525
H(23)	H(25) ⁷⁾	3.296	H(23)	H(28) ⁴⁾	3.145
H(23)	H(30) ⁷⁾	3.110	H(23)	H(34) ⁴⁾	3.093
H(24)	O(13) ²⁾	2.909	H(24)	O(15) ²⁾	3.510
H(24)	C(35) ²⁾	3.580	H(24)	H(4) ¹⁶⁾	3.254
H(24)	H(30) ⁷⁾	3.117	H(24)	H(45) ²⁾	2.690
H(25)	O(2) ⁶⁾	2.905	H(25)	C(1) ⁶⁾	3.542
H(25)	C(28) ¹⁾	3.562	H(25)	H(1) ⁶⁾	3.233
H(25)	H(2) ⁶⁾	3.285	H(25)	H(5) ⁶⁾	3.443
H(25)	H(22) ⁹⁾	3.569	H(25)	H(23) ⁹⁾	3.296
H(25)	H(35) ¹⁾	3.071	H(25)	H(37) ¹⁾	2.997
H(26)	O(2) ⁶⁾	3.095	H(26)	C(1) ⁶⁾	3.052
H(26)	C(10)	3.475	H(26)	C(15) ⁵⁾	3.410
H(26)	H(1) ⁶⁾	2.381	H(26)	H(2) ⁶⁾	2.998
H(26)	H(11)	3.115	H(26)	H(14)	2.671
H(26)	H(17) ⁵⁾	2.507	H(27)	C(27) ¹⁾	2.811

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(27)	C(28) ¹⁾	3.263	H(27)	H(1) ⁶⁾	3.580
H(27)	H(2) ⁶⁾	3.564	H(27)	H(11)	3.261
H(27)	H(14)	3.035	H(27)	H(35) ¹⁾	2.431
H(27)	H(36) ¹⁾	2.526	H(27)	H(37) ¹⁾	2.795
H(28)	O(8) ⁵⁾	3.360	H(28)	O(13) ⁶⁾	3.572
H(28)	H(5) ⁸⁾	2.770	H(28)	H(23) ⁵⁾	3.145
H(28)	H(38) ⁶⁾	2.848	H(29)	O(2) ⁶⁾	3.167
H(29)	O(8) ⁵⁾	3.580	H(29)	O(13) ⁶⁾	3.330
H(29)	C(6) ⁶⁾	3.505	H(29)	H(8) ⁶⁾	3.576
H(29)	H(10) ⁶⁾	2.730	H(29)	H(38) ⁶⁾	3.173
H(30)	O(13) ⁶⁾	2.685	H(30)	C(2) ⁸⁾	3.307
H(30)	C(18) ⁹⁾	3.197	H(30)	H(4) ⁸⁾	3.315
H(30)	H(5) ⁸⁾	2.689	H(30)	H(6) ⁸⁾	3.423
H(30)	H(22) ⁹⁾	2.831	H(30)	H(23) ⁹⁾	3.110
H(30)	H(24) ⁹⁾	3.117	H(30)	H(38) ⁶⁾	3.331
H(31)	O(3)	2.777	H(31)	C(7)	3.328
H(31)	H(8)	3.300	H(31)	H(11)	3.206
H(31)	H(35) ¹⁾	3.179	H(31)	H(36) ¹⁾	3.143
H(32)	O(3)	3.147	H(32)	O(15) ⁸⁾	3.474
H(32)	C(9)	3.422	H(32)	H(11)	2.573
H(32)	H(17) ⁵⁾	3.451	H(32)	H(19) ⁴⁾	3.288
H(32)	H(21) ⁴⁾	3.296	H(32)	H(39) ⁸⁾	3.099
H(33)	O(2) ⁸⁾	3.318	H(33)	C(2) ⁸⁾	3.329
H(33)	C(5) ⁸⁾	3.584	H(33)	H(5) ⁸⁾	2.743
H(33)	H(6) ⁸⁾	3.129	H(33)	H(8) ⁸⁾	3.016
H(34)	O(13) ⁸⁾	3.135	H(34)	O(15) ⁸⁾	2.864
H(34)	C(30) ⁸⁾	3.182	H(34)	C(31) ⁸⁾	2.865
H(34)	H(16) ⁵⁾	3.460	H(34)	H(23) ⁵⁾	3.093
H(34)	H(39) ⁸⁾	2.391	H(35)	O(9) ⁸⁾	3.275
H(35)	C(17) ⁴⁾	3.274	H(35)	C(19) ⁸⁾	3.130
H(35)	H(20) ⁴⁾	2.329	H(35)	H(21) ⁴⁾	3.553
H(35)	H(22) ⁴⁾	3.253	H(35)	H(25) ⁸⁾	3.071
H(35)	H(27) ⁸⁾	2.431	H(35)	H(31) ⁸⁾	3.179
H(36)	C(4) ⁸⁾	2.942	H(36)	C(5) ⁸⁾	3.246
H(36)	C(7) ⁸⁾	3.524	H(36)	C(8) ⁸⁾	3.141
H(36)	C(19) ⁸⁾	3.457	H(36)	H(7) ⁸⁾	2.369
H(36)	H(8) ⁸⁾	2.677	H(36)	H(27) ⁸⁾	2.526

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(36)	H(31) ⁸⁾	3.143	H(37)	C(19) ⁸⁾	3.288
H(37)	H(2) ¹⁰⁾	2.905	H(37)	H(5) ¹⁰⁾	3.382
H(37)	H(25) ⁸⁾	2.997	H(37)	H(27) ⁸⁾	2.795
H(38)	C(20) ³⁾	3.295	H(38)	H(20) ⁴⁾	2.834
H(38)	H(28) ³⁾	2.848	H(38)	H(29) ³⁾	3.173
H(38)	H(30) ³⁾	3.331	H(39)	O(11) ¹⁾	2.450
H(39)	C(17) ⁹⁾	3.436	H(39)	C(23) ¹⁾	3.484
H(39)	C(24) ¹⁾	2.811	H(39)	H(20) ⁹⁾	3.419
H(39)	H(21) ⁹⁾	2.637	H(39)	H(32) ¹⁾	3.099
H(39)	H(34) ¹⁾	2.391	H(40)	O(7) ⁹⁾	3.176
H(40)	C(17) ⁹⁾	3.249	H(40)	C(35) ¹²⁾	3.387
H(40)	H(21) ⁹⁾	2.330	H(40)	H(44) ¹²⁾	2.466
H(41)	O(1) ⁸⁾	3.048	H(41)	O(4) ⁸⁾	3.234
H(41)	C(2) ⁸⁾	3.358	H(41)	C(4) ⁸⁾	3.146
H(41)	C(36) ¹²⁾	3.370	H(41)	H(4) ⁸⁾	3.479
H(41)	H(6) ⁸⁾	2.596	H(41)	H(7) ⁸⁾	2.547
H(41)	H(15) ⁸⁾	3.066	H(41)	H(44) ¹²⁾	2.911
H(41)	H(46) ¹²⁾	2.609	H(41)	H(47) ¹²⁾	3.328
H(42)	C(2) ⁸⁾	3.262	H(42)	H(4) ⁸⁾	3.454
H(42)	H(6) ⁸⁾	2.347	H(42)	H(7) ⁸⁾	3.006
H(42)	H(21) ⁹⁾	3.428	H(42)	H(22) ⁹⁾	3.041
H(43)	O(6) ⁸⁾	3.264	H(43)	O(7) ⁸⁾	3.584
H(43)	C(12) ⁸⁾	3.551	H(43)	H(2) ¹⁰⁾	3.301
H(43)	H(4) ¹⁰⁾	3.295	H(43)	H(15) ⁸⁾	3.384
H(43)	H(47) ¹³⁾	3.104	H(44)	O(7) ⁸⁾	3.121
H(44)	O(16) ¹³⁾	2.953	H(44)	C(32) ¹³⁾	2.995
H(44)	C(33) ¹³⁾	3.410	H(44)	C(36) ¹³⁾	3.587
H(44)	H(15) ¹¹⁾	3.293	H(44)	H(40) ¹³⁾	2.466
H(44)	H(41) ¹³⁾	2.911	H(44)	H(47) ¹³⁾	3.036
H(44)	H(48) ¹³⁾	3.496	H(45)	O(7) ¹¹⁾	3.322
H(45)	O(16) ¹³⁾	3.360	H(45)	C(13) ¹¹⁾	3.287
H(45)	C(14) ¹¹⁾	3.427	H(45)	H(4) ¹⁰⁾	3.457
H(45)	H(15) ¹¹⁾	2.751	H(45)	H(16) ¹¹⁾	2.714
H(45)	H(24) ¹¹⁾	2.690	H(45)	H(47) ¹³⁾	2.790
H(46)	O(1) ¹¹⁾	2.911	H(46)	C(33) ¹³⁾	3.554
H(46)	H(3) ¹¹⁾	3.290	H(46)	H(4) ¹¹⁾	3.427
H(46)	H(15) ¹¹⁾	3.157	H(46)	H(16) ¹¹⁾	3.080

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(46)	H(18) ¹¹⁾	2.992	H(46)	H(41) ¹³⁾	2.609
H(47)	O(1) ¹¹⁾	3.598	H(47)	O(7) ⁸⁾	3.538
H(47)	C(2) ¹¹⁾	3.468	H(47)	C(35) ¹²⁾	3.152
H(47)	H(3) ¹¹⁾	3.179	H(47)	H(4) ¹¹⁾	2.756
H(47)	H(6) ¹¹⁾	3.569	H(47)	H(15) ⁸⁾	3.580
H(47)	H(41) ¹³⁾	3.328	H(47)	H(43) ¹²⁾	3.104
H(47)	H(44) ¹²⁾	3.036	H(47)	H(45) ¹²⁾	2.790
H(48)	O(1) ¹¹⁾	3.523	H(48)	O(6) ⁹⁾	3.563
H(48)	C(1) ¹¹⁾	3.595	H(48)	H(3) ¹¹⁾	2.724
H(48)	H(18) ¹¹⁾	3.483	H(48)	H(44) ¹²⁾	3.496

Symmetry Operators:

- | | |
|----------------------------|---------------------------|
| (1) -X+1,Y+1/2,-Z+1/2 | (2) -X+1/2,-Y+2,Z+1/2 |
| (3) X-1,Y,Z | (4) X+1/2-1,-Y+1/2+1,-Z+1 |
| (5) X+1/2,-Y+1/2+1,-Z+1 | (6) X+1,Y,Z |
| (7) -X+1/2+1,-Y+2,Z+1/2 | (8) -X+1,Y+1/2-1,-Z+1/2 |
| (9) -X+1/2+1,-Y+2,Z+1/2-1 | (10) -X,Y+1/2-1,-Z+1/2 |
| (11) -X+1/2,-Y+2,Z+1/2-1 | (12) X+1/2,-Y+1/2+1,-Z |
| (13) X+1/2-1,-Y+1/2+1,-Z | (14) -X,Y+1/2,-Z+1/2 |
| (15) X+1/2-1,-Y+1/2+2,-Z+1 | (16) X+1/2,-Y+1/2+2,-Z+1 |