Synthesis of (+)-Coronafacic Acid

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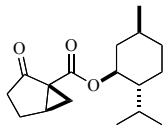
Supporting Information

Contents:

Experimental Section

General. ^{1}H NMR (at 400 MHz) and ^{13}C NMR (at 400 MHz) spectra were obtained as solutions in deuteriochloroform ($CDCl_3$). The infrared (IR) spectra were determined as neat oils. R_f values indicated refer to thin layer chromatography (TLC) on 5.0 x 10 cm, 250 μm analytical plates coated with silica gel 60 F_{254}, developed in the solvent system indicated. Column chromatography was carried out on using silica gel 60 particle size 0.015 – 0.040 mm. The solvent mixtures reported are volume/volume mixtures. EtOAc is ethyl acetate. All glassware was oven dried and reactions were carried out under a flow of nitrogen. All Wittig reactions were stirred magnetically, under dry N_2 unless otherwise noted. $Fe(CO)_5$ mediated carbonylation was performed in a Rayonet reactor using a tube inside a tube setup, such that the reaction mixture is a thin layer between the outer tube and the inner tube.

Diazo ester 1: 5-hexen-2-one (20 g, 200 mmol) was mixed with NaH (20 g) in toluene (200 mL) at rt. The mixture was heated to reflux for 10 min before dimethyl carbonate (56 g) was added dropwise over 30 min. Caution: the reaction is highly exothermic! After an additional 3 h at reflux, the mixture was partitioned between MTBE and 1N aquous HCl and brine. The combined organic extract was dried (Na_2SO_4) and concentrated. The residue was bulb to bulb distilled to afford the β-ketoester as a colorless oil (27.24 g, 86% yield). TLC R_f (PE/MTBE = 8/2) = 0.45. The β-ketoester (8.80 g, 56 mmol) was mixed with (+)-menthol (7.4 g), DMAP (1.15 g) and molecular sieve (4 Å, 12.5 g) in toluene (150 mL). The reaction mixture was heated to reflux overnight, then passed through a pad of silica gel. The eluant was concentrated and the residue was chromatographed to afford the menthol ester as a colorless oil (9.27 g, 60% yield). TLC R_f (PE/MTBE = 8/2) = 0.80. The β-ketoester (4.13 g, 14.7 mmol) was mixed with TEA (3.1 g, 30 mmol) and mesyl azide (2.7 g, 22 mmol) in MeCN (50 mL) at rt. After 8 h the solvent was removed *in vacuo*. The residue was partitioned between MTBE and 1N aqueous NaOH, saturated aqueous $NaHCO_3$ and brine. The combined organic extract was dried (Na_2SO_4) and concentrated. The residue was chromatographed to afford the diazo ester **1** as a colorless oil (4.36 g, 72% yield). TLC R_f (PE/MTBE = 8/2) = 0.50. The spectroscopic data was consistent with those that were earlier reported.[11a]



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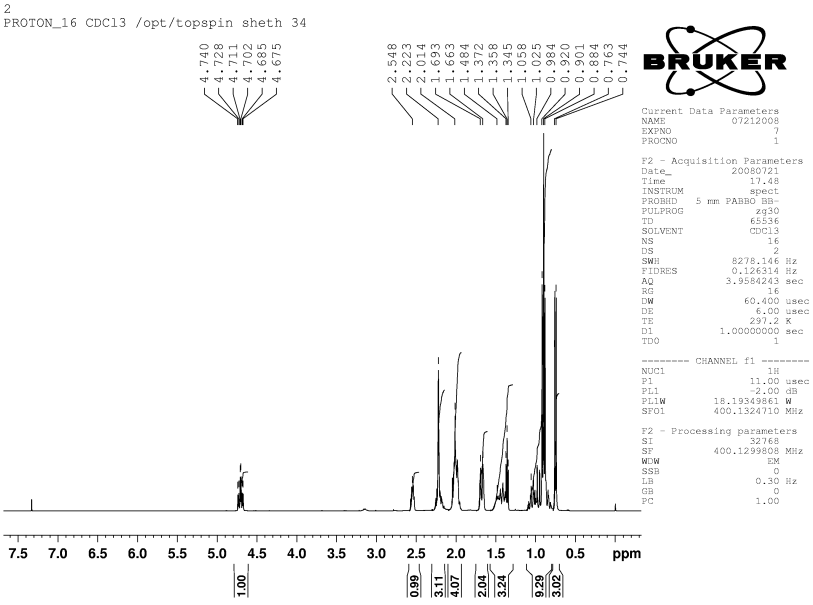
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1.693
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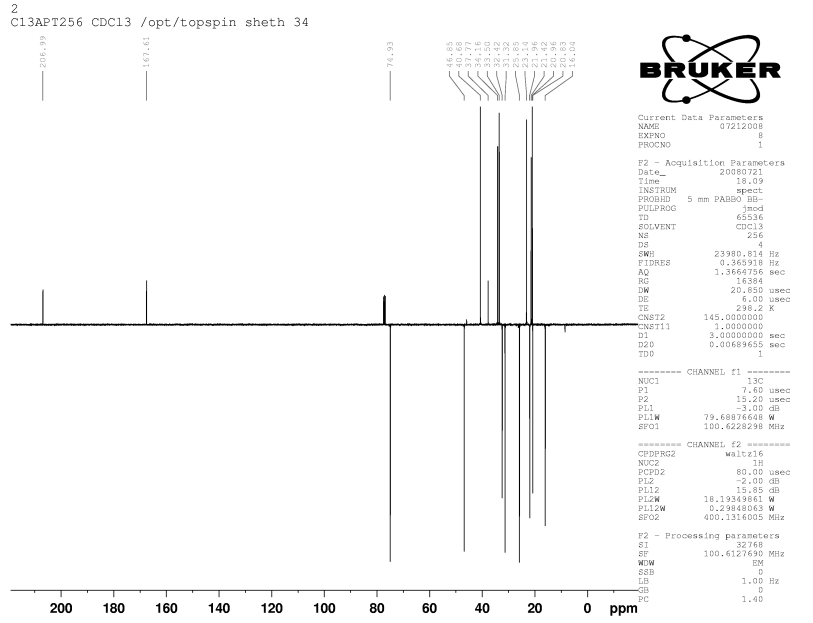
3.02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25.65
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21.96
21.36
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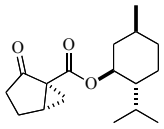

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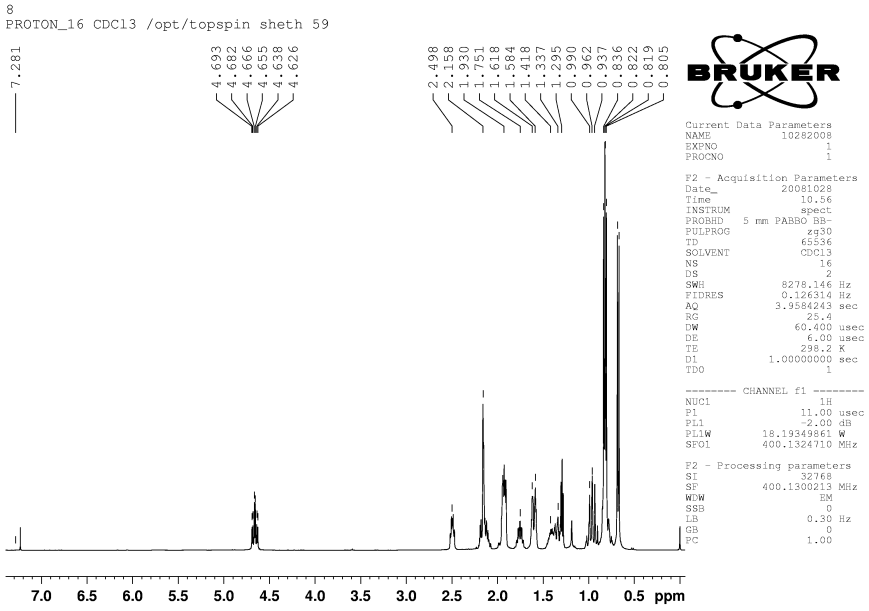
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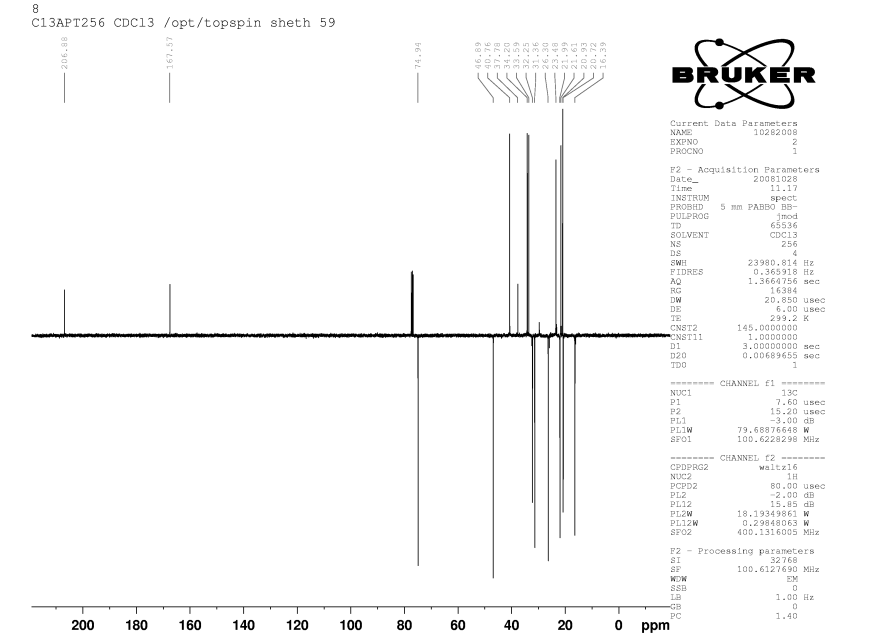
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P2 15.20 usec
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PCPD2 80.00 usec
PL2 -2.00 dB
PL12 15.85 dB
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PL12W 0.29848063 W
SFO2 400.1316005 MHz
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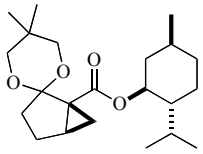

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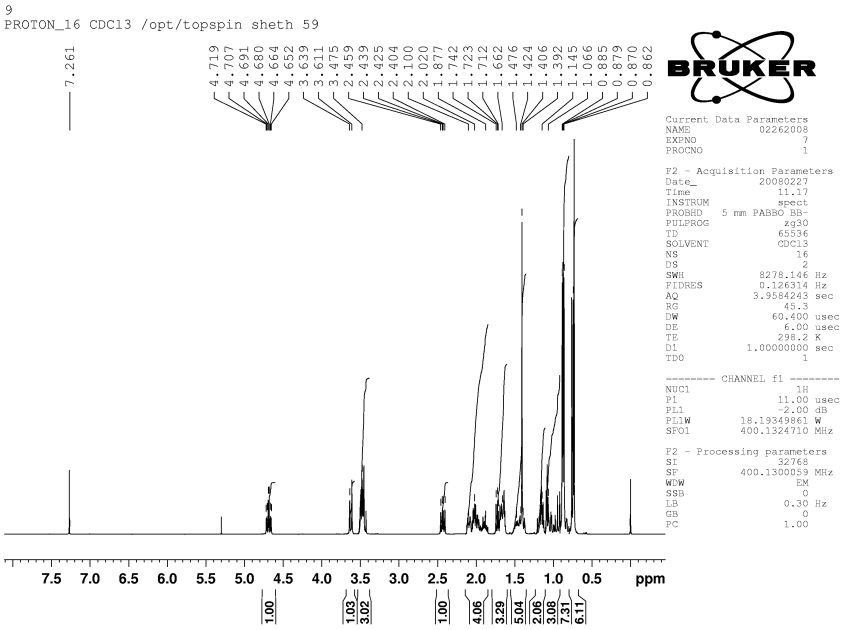
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3.475
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2.439
2.425
2.404
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TE 298.2 K
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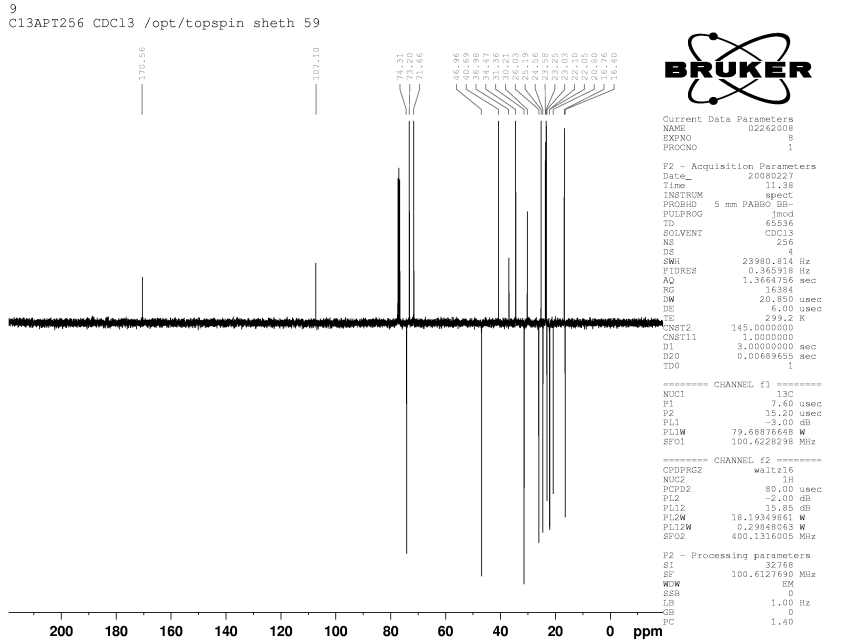
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28.03
24.56
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23.03
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DE 6.00 usec
TE 299.2 K
CNST2 145.0000000
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D20 0.00689655 sec
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P2 15.20 usec
PL1 -3.00 dB
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NUC2 1H
PCPD2 80.00 usec
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PL12 15.85 dB
PL2W 18.19349861 W
PL12W 0.29848063 W
SFO2 400.1316005 MHz
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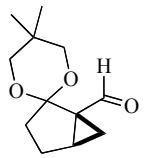

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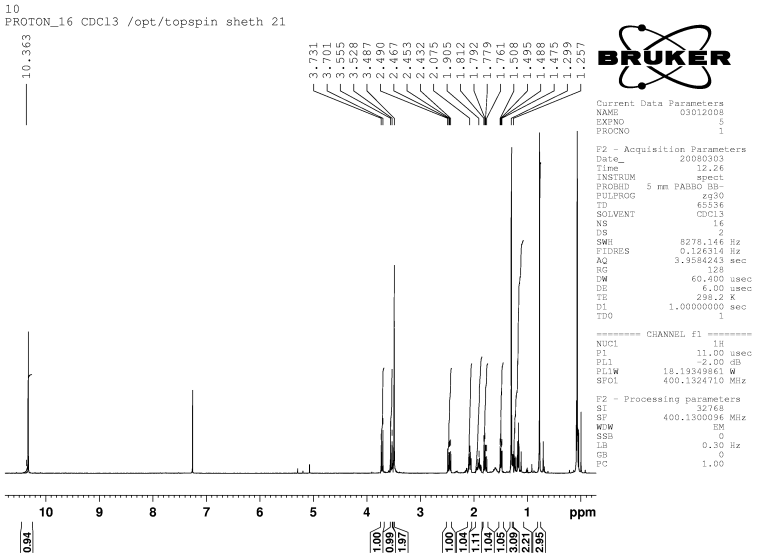
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TD0 1
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PL1W 18.19349861 W
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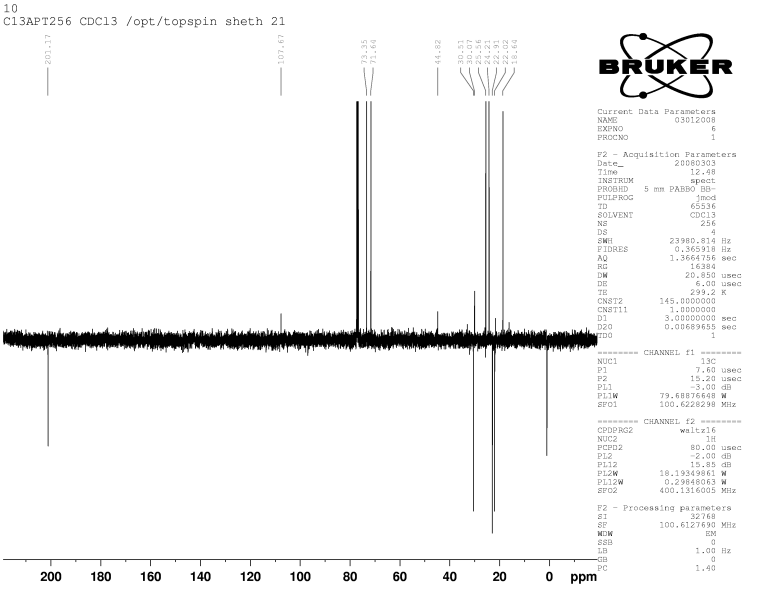
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AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 299.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 3.00000000 sec
D20 0.00689655 sec
TD0 1
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P1 7.60 usec
P2 15.20 usec
PL1 -3.00 dB
PL1W 79.68876648 W
SFO1 100.6228298 MHz
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NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 15.85 dB
PL2W 18.19349861 W
PL12W 0.29848063 W
SFO2 400.1316005 MHz
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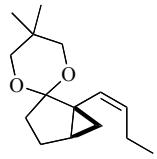

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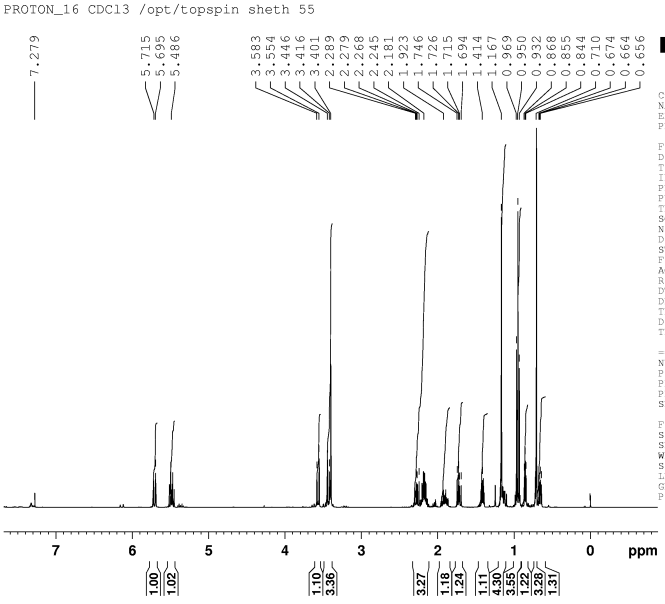

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D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
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F2 - Processing parameters
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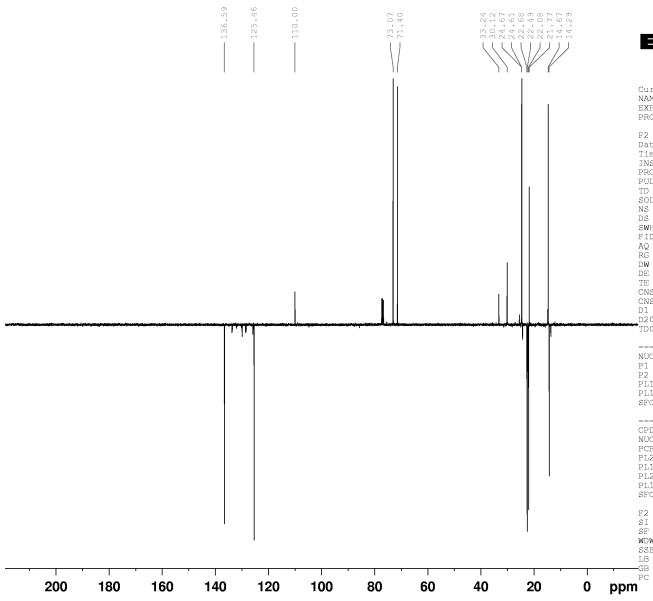

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RG 16384
DW 20.850 usec
DE 6.00 usec
TE 298.2 K
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CNST11 1.0000000
D1 3.00000000 sec
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TD0 1

======== CHANNEL f1 ========
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P2 15.20 usec
PL1 -3.00 dB
PL1W 79.68876648 W
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
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NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 15.85 dB
PL2W 18.19349861 W
PL12W 0.29848063 W
SFO2 400.1316005 MHz

F2 - Processing parameters
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SSB 0
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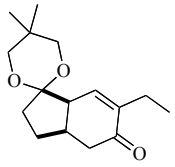

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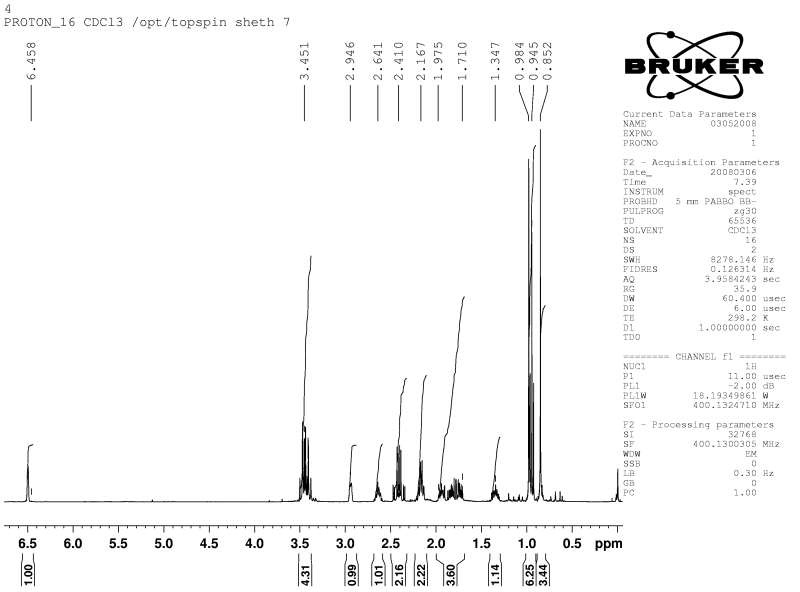
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2.410
2.167
1.975
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1.347
0.984
0.945
0.852
6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00 4.31 0.99 1.01 2.16 2.22 3.60 1.14 6.25 3.44
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FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 35.9
DW 60.400 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
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PL1 -2.00 dB
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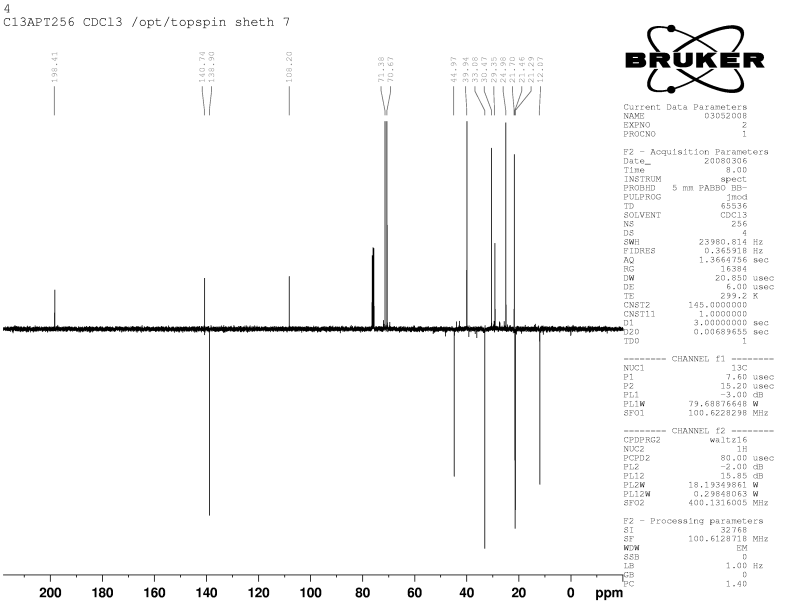
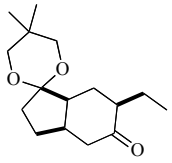
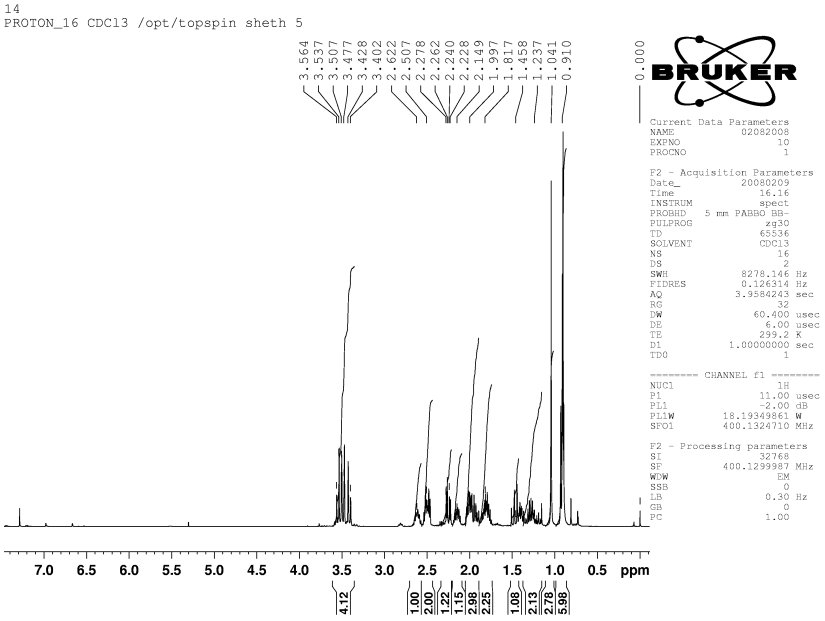
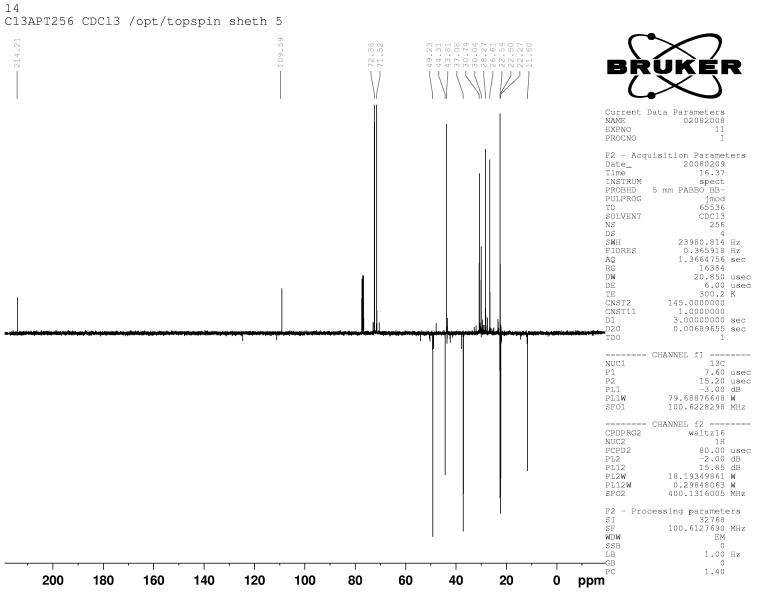
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300305 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

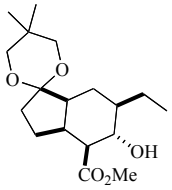

4
C13APT256 CDCl3 /opt/topspin sheth 7
198.41
140.74
138.90
108.20
77.38
76.67
44.97
39.84
33.58
30.47
29.35
28.88
21.70
21.46
21.19
12.07
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
Current Data Parameters
NAME 03052008
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080306
Time 8.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 299.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 3.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.60 usec
P2 15.20 usec
PL1 -3.00 dB
PL1W 79.68876648 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 15.85 dB
PL2W 18.19349861 W
PL12W 0.29848063 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128718 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



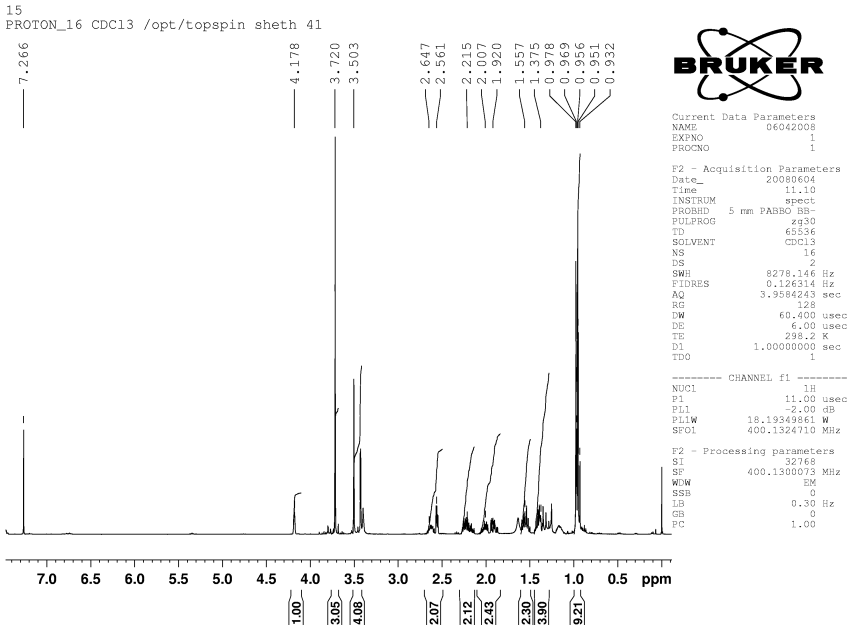
14

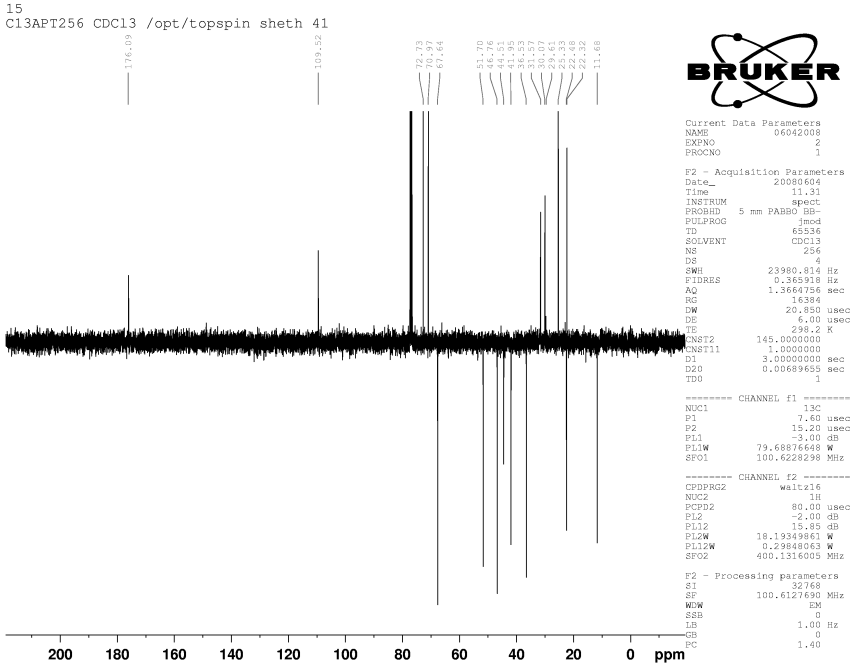

14
PROTON_16 CDCl3 /opt/topspin sheth 5
3.564
3.537
3.507
3.477
3.428
3.402
2.622
2.507
2.278
2.262
2.240
2.228
2.149
1.997
1.817
1.458
1.237
1.041
0.910
0.000
BRUKER
Current Data Parameters
NAME 02082008
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080209
Time 16.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 32
DW 60.400 usec
DE 6.00 usec
TE 299.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
PL1W 18.19349861 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1299987 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
4.12
1.00
2.00
1.22
1.15
2.98
2.25
1.08
2.13
2.78
5.98

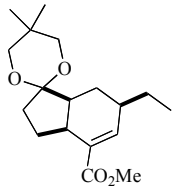

14
C13APT256 CDCl3 /opt/topspin sheth 5
214.21
169.59
72.38
71.52
49.23
43.31
42.70
37.08
30.74
28.48
26.27
25.54
22.50
22.37
11.60
BRUKER
Current Data Parameters
NAME 02082008
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080209
Time 16.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 3.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.60 usec
P2 15.20 usec
PL1 -3.00 dB
PL1W 79.68876648 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 15.85 dB
PL2W 18.19349861 W
PL12W 0.29848063 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm


O
O
OH
CO2Me

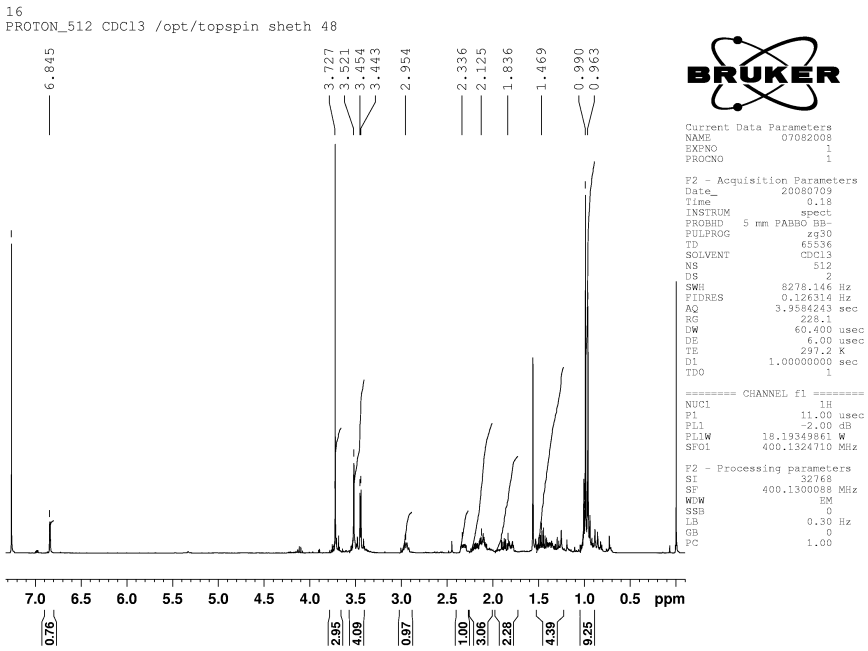
15

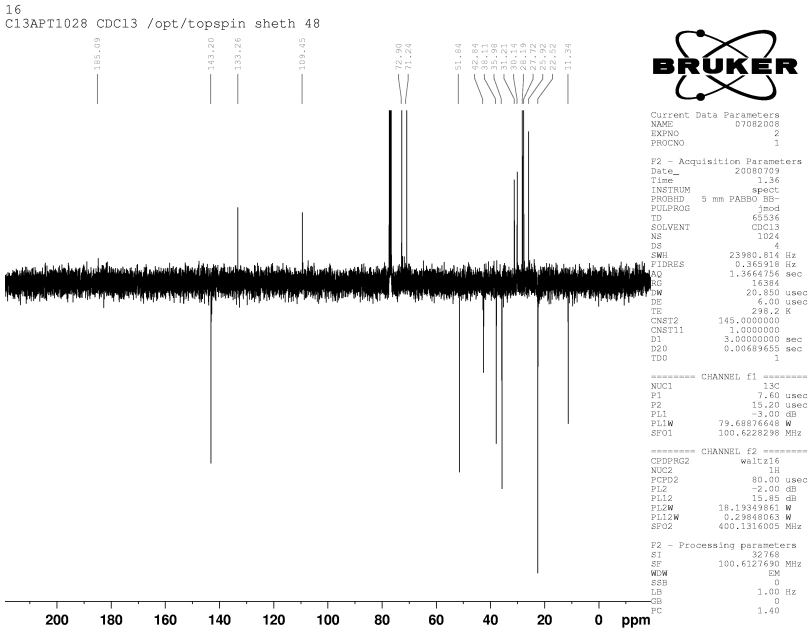

15
PROTON_16 CDCl3 /opt/topspin sheth 41
7.266
4.178
3.720
3.503
2.647
2.561
2.215
2.007
1.920
1.557
1.375
0.978
0.969
0.956
0.951
0.932
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
3.05
4.08
2.07
2.12
2.43
2.30
3.90
9.21
BRUKER
Current Data Parameters
NAME 06042008
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080604
Time 11.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.95842432 sec
RG 128
DW 60.400 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
PL1W 18.19349861 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300073 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


15
C13APT256 CDCl3 /opt/topspin sheth 41
176.09
109.52
78.73
70.97
67.64
51.70
46.76
44.51
41.95
36.53
31.57
31.07
29.61
23.33
22.38
11.68
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
Current Data Parameters
NAME 06042008
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080604
Time 11.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.36647568 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 298.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 3.00000000 sec
D20 0.00689655 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 7.60 usec
P2 15.20 usec
PL1 -3.00 dB
PL1W 79.68876648 W
SFO1 100.6228298 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 15.85 dB
PL2W 18.19349861 W
PL12W 0.29848063 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


O
O
CO2Me

16


16
PROTON_512 CDCl3 /opt/topspin sheth 48
6.845
3.727
3.521
3.454
3.443
2.954
2.336
2.125
1.836
1.469
0.990
0.963
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.76
2.95
4.09
0.97
1.00
3.06
2.28
4.39
9.25
BRUKER
Current Data Parameters
NAME 07082008
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080709
Time 0.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 512
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.00 usec
TE 297.2 K
D1 1.00000000 sec
TDO 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
PL1W 18.19349861 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


16
C13APT1028 CDCl3 /opt/topspin sheth 48
185.09
143.20
133.26
109.45
72.90
71.24
51.84
42.84
38.11
35.98
31.21
29.19
28.11
27.72
25.92
22.20
11.34
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
Current Data Parameters
NAME 07082008
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080709
Time 1.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 298.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 3.00000000 sec
D20 0.00689655 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.60 usec
P2 15.20 usec
PL1 -3.00 dB
PL1W 79.68876648 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 15.85 dB
PL2W 18.19349861 W
PL12W 0.29848063 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40