

Remarkable diastereomeric rearrangement of an  $\alpha$ -Acyloxy  $\beta$ -ketosulfide to an  $\alpha$ -acyloxy thioester. A novel approach to the synthesis of optically active (2*S*,3*S*)  $\beta$ -amino  $\alpha$ -hydroxy acid.

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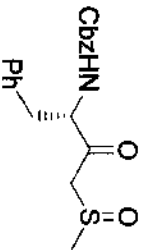
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## General Experimental Method

All reagents were purchased and used without further purification. Thin-layer chromatography (TLC) was conducted on precoated TLC plates. High-performance liquid chromatography (HPLC) was performed with a HPLC pump and an UV detector system using an ODS column. Melting points were measured with an automatic melting point apparatus, and are uncorrected. Optical rotations were measured using a cell with path length of 10 mm. NMR spectra were obtained on 400MHz or 300 MHz spectrometers. All proton NMR spectra were measured in CDCl<sub>3</sub> or DMSO-d<sub>6</sub> solvent, and chemical shifts are reported as  $\delta$  values in parts per million relative to tetramethylsilane ( $\delta$  0.00) or CDCl<sub>3</sub> ( $\delta$  7.26) as an internal standard. Data are reported as follows: chemical shift (integrated intensity or assignment, multiplicity, coupling constants in hertz, assignment). All carbon NMR spectra were measured in CDCl<sub>3</sub> or DMSO-d<sub>6</sub> solvent, and chemical shifts are reported as  $\delta$  values in parts per million relative to CDCl<sub>3</sub> ( $\delta$  77.0) or DMSO-d<sub>6</sub> ( $\delta$  39.5) as an internal standard. Infrared (IR) spectra were reported in wavenumber (cm<sup>-1</sup>). Mass spectra (MS) were obtained with ESI (electrospray) or FAB (fast atom bombardment) ionization.

<sup>1</sup>H NMR for 2a

T995002

ppm

7.3595  
7.3414  
7.3322  
7.3106  
7.2964  
7.2785  
7.2670  
7.2608  
7.2499  
7.1669

5.0764

4.5589

4.0626  
4.0276

3.5769  
3.5420

3.1712  
2.9925

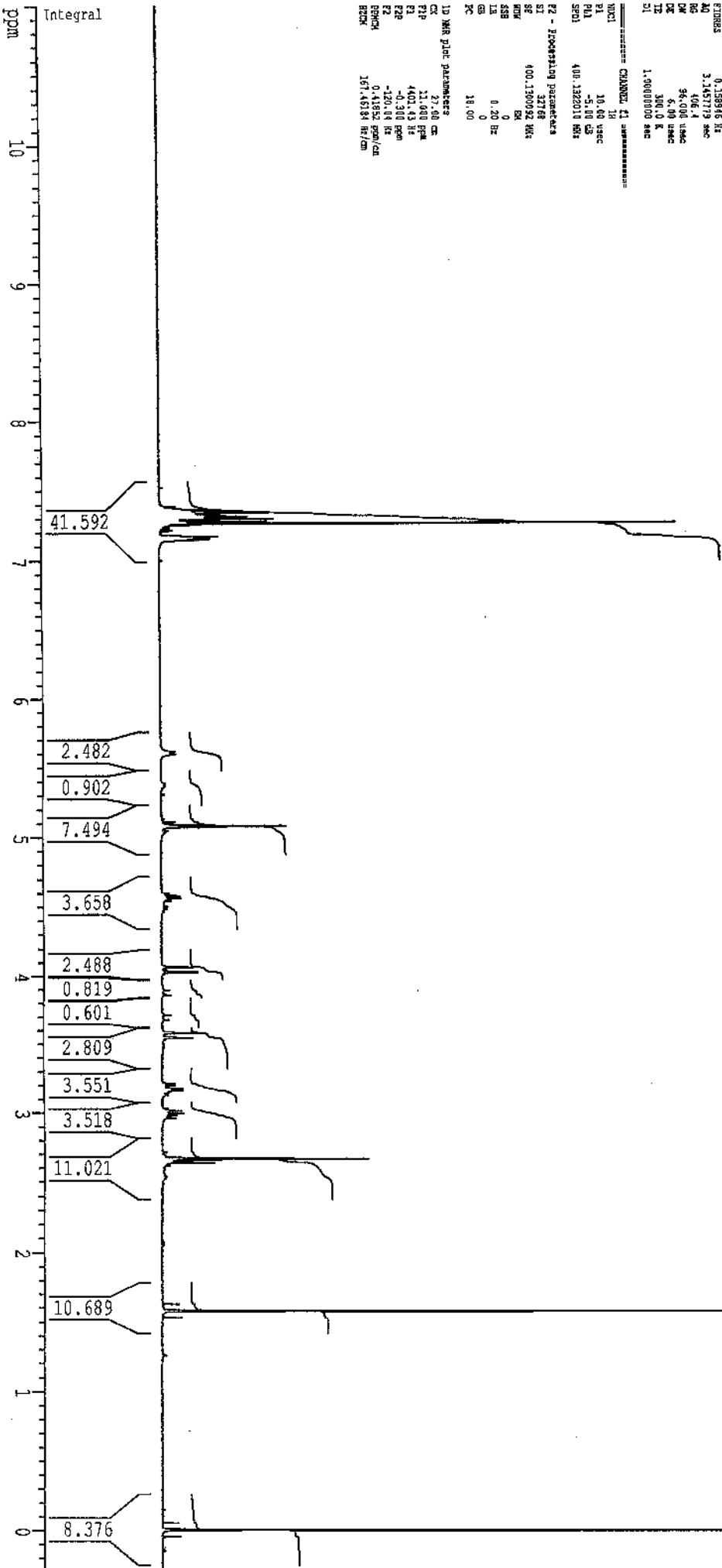
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1.6240  
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1.5239

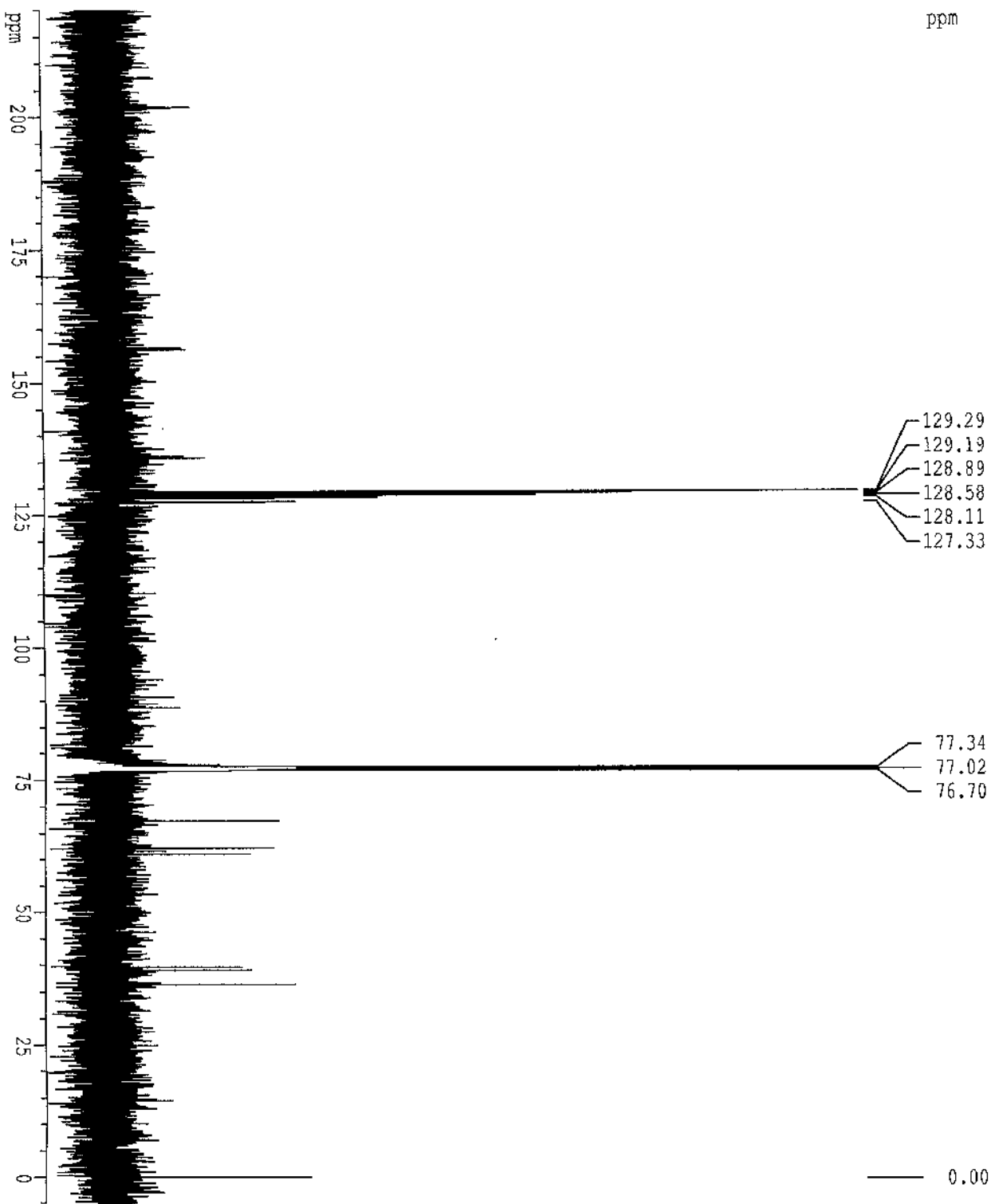
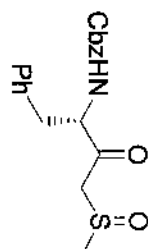
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LB 6.00 Hz  
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PIF 11.000 ppm  
F3 4401.43 Hz  
F2F 0.300 ppm  
F2 0.41852 ppm/cm  
BPMCH 161.46134 Hz/cm

Integral



TS95002

<sup>13</sup>C NMR for 2a

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EXPNO 20  
PROCNO 1

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SOLVENT CDCl3  
NS 4096

DS 4  
SWH 23148.148 Hz

FIDRES 0.353213 Hz  
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RG 16384  
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TE 300.0 K

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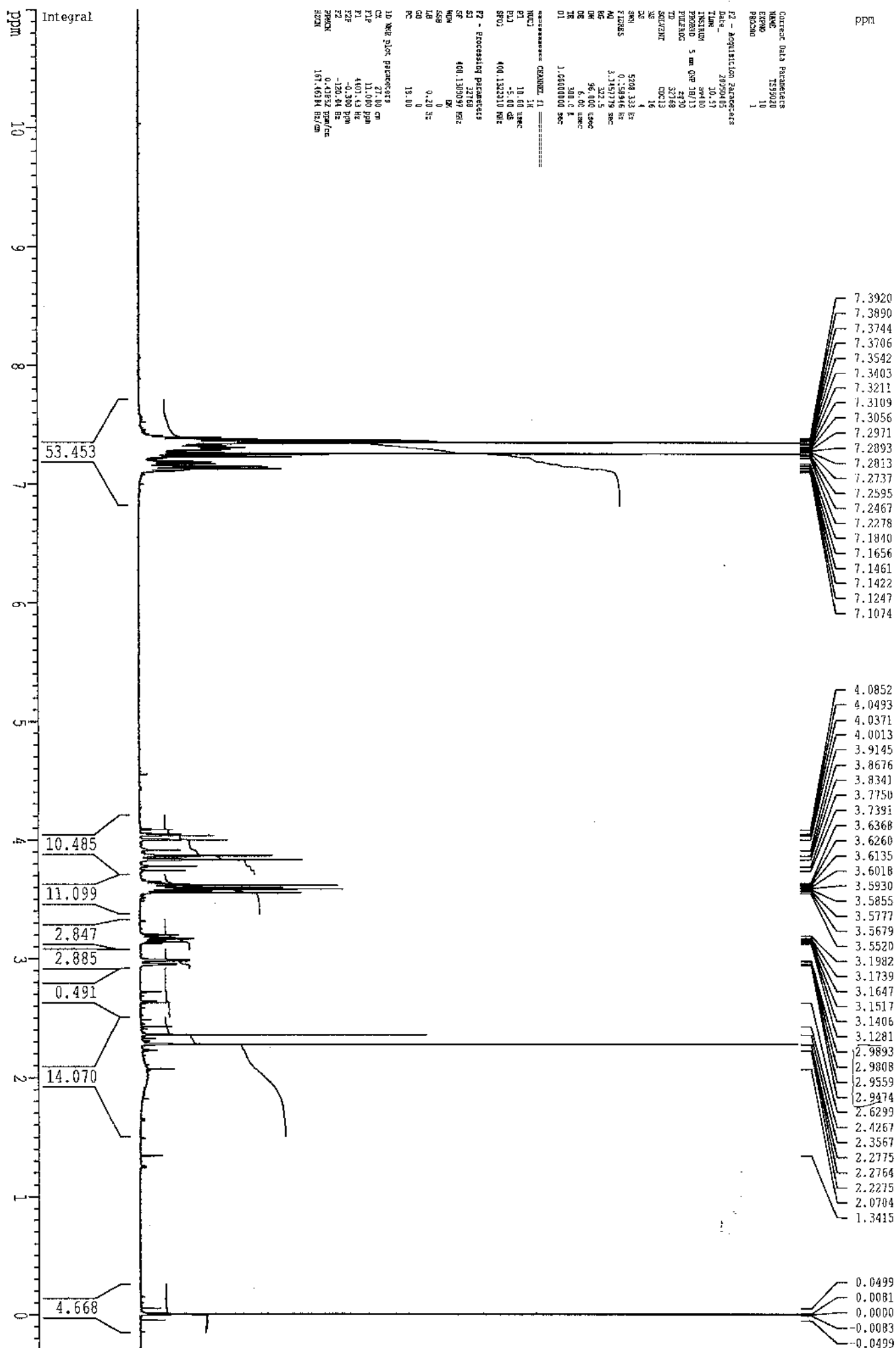
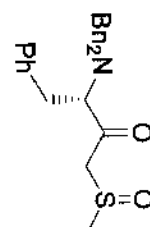
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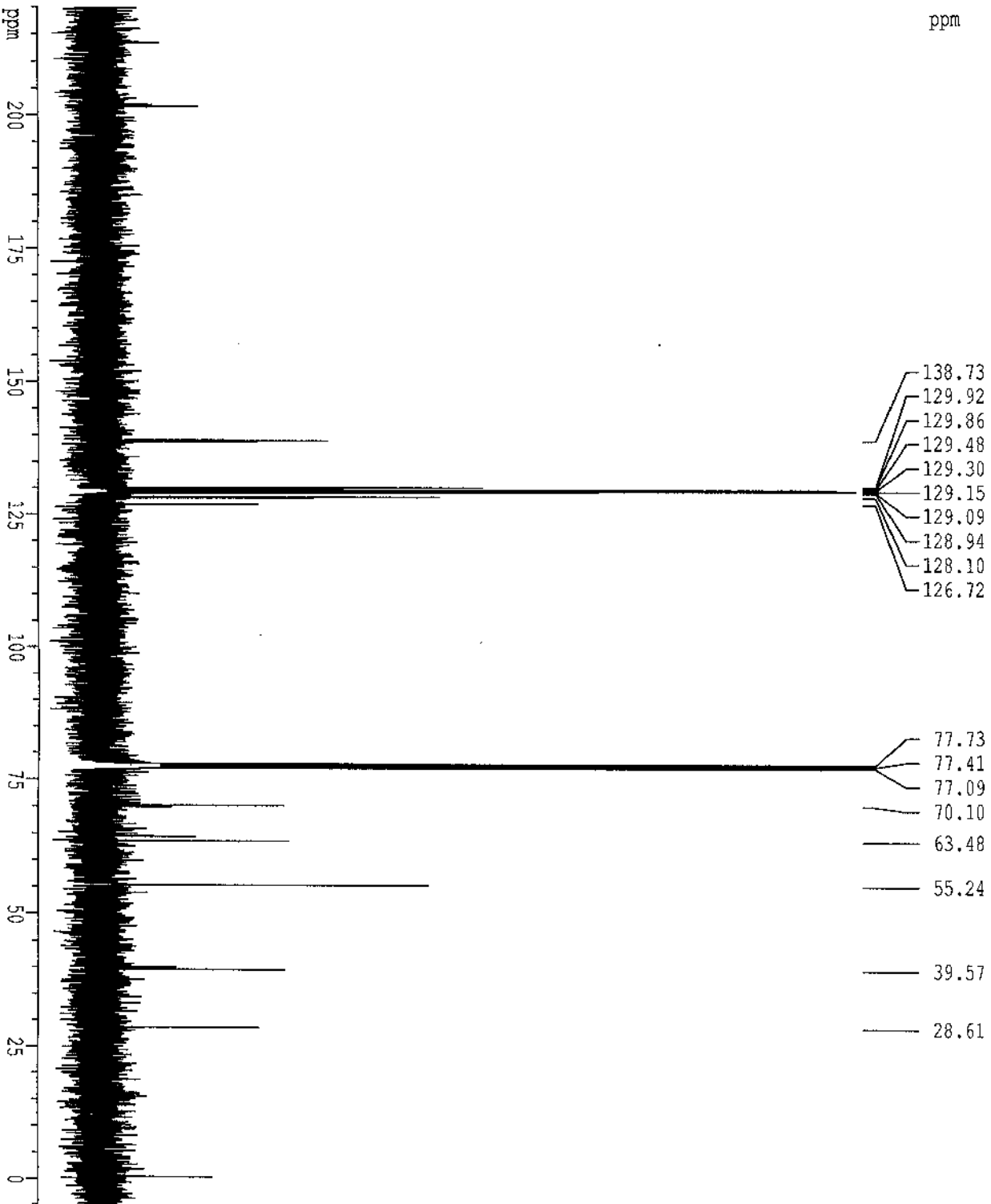
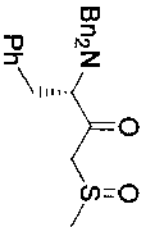
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NUC2 <sup>1</sup>H  
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PL12 12.50 dB  
PL13 12.50 dB  
SFO2 400.1316085 MHz

F2 - Processing Parameters  
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SF 100.6127676 MHz  
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SSB 0  
LB 1.00 Hz  
GB 0  
PC 3.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 226.000 ppm  
F1 22134.31 Hz  
F2P -5.000 ppm  
F2 -503.07 Hz  
PPMCM 11.25000 ppm/cm  
HZCM 1131.89366 Hz/cm

<sup>1</sup>H NMR for 2b

<sup>13</sup>C NMR for 2b

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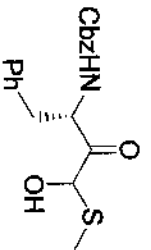
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NS 1536  
DS 4  
SWH 23148.148 Hz  
FIDRES 9.353213 Hz  
AQ 1.4156276 sec  
RG 16384  
DN 21.600 usec  
DE 28.00 usec  
TE 300.0 K  
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d11 0.03000000 sec  
d:2 0.00002800 sec

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SFO1 100.6237964 MHz

===== CHANNEL f2 =====  
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PL13 12.50 dB  
SFO2 400.1316005 MHz

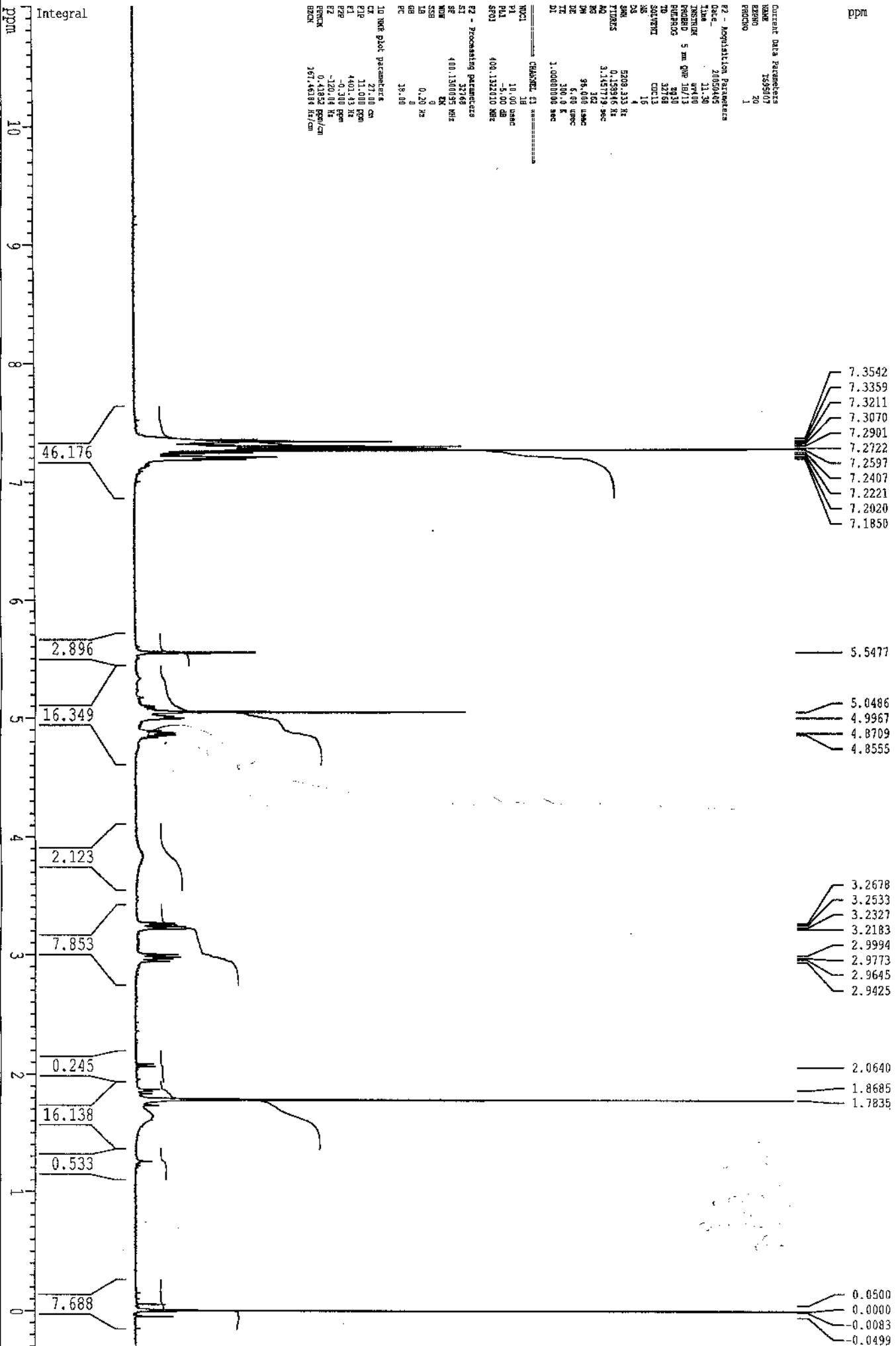
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PC 3.00

1D NMR plot parameters  
CX 20.00 cm  
FL2 220.000 ppm  
F1 22134.80 Hz  
F2P -5.000 ppm  
F2 -503.87 Hz  
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HZCM 1131.89319 Hz/cm

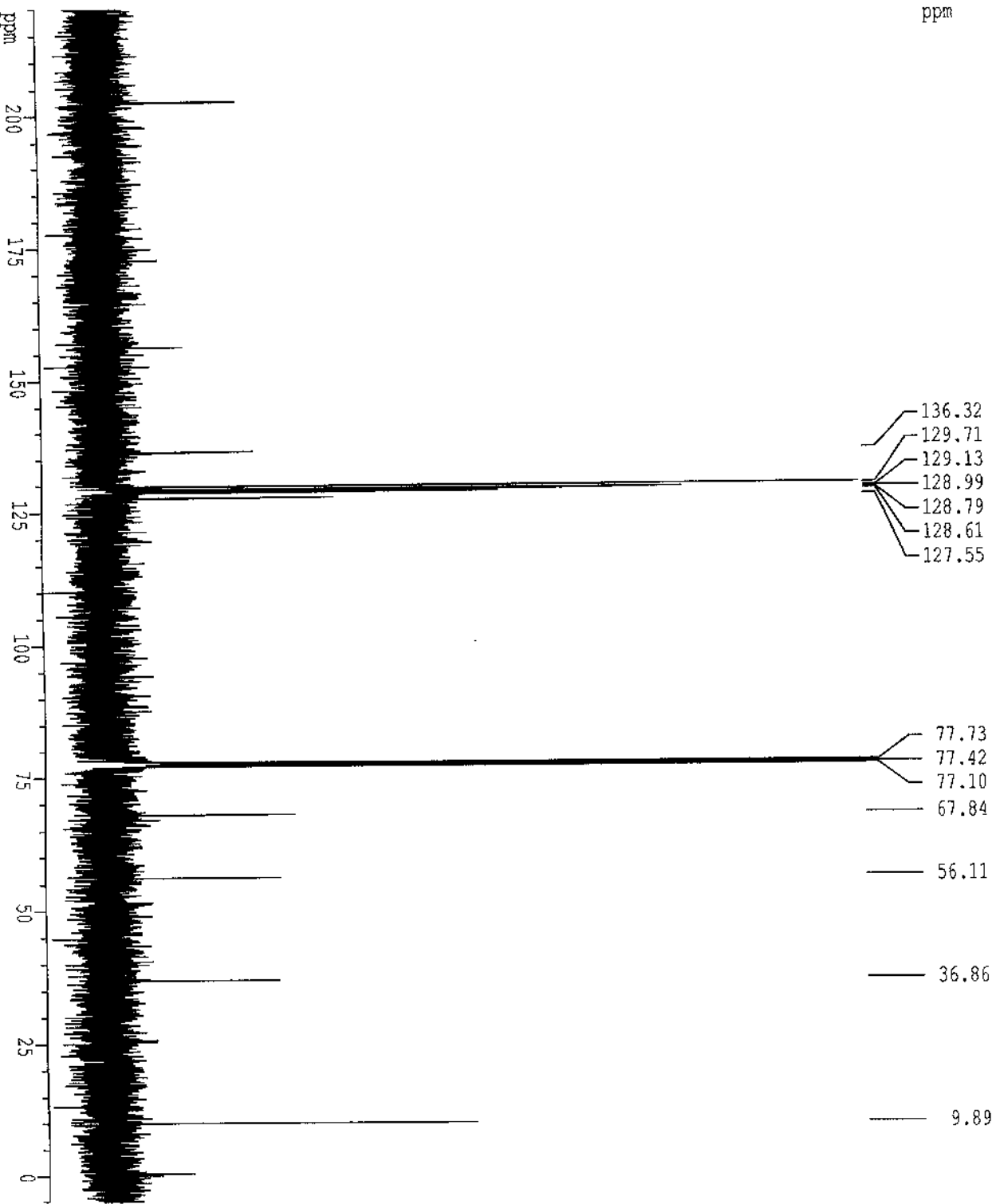
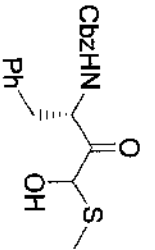
<sup>1</sup>H NMR for 3a

TS95007

ppm





<sup>13</sup>C NMR for 3a

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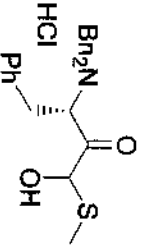
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FIDRES 0.353213 Hz  
AQ 1.4156276 sec  
RG 16384  
RG 16384  
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TE 300.0 K  
D1 1.00000000 sec  
d11 0.03000000 sec  
d12 0.00002000 sec

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PL1 -3.00 dB  
SFO1 100.6237964 MHz

===== CHANNEL f2 =====  
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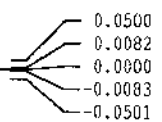
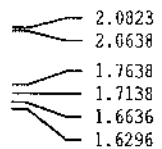
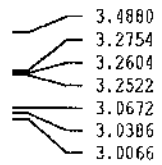
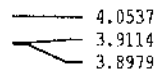
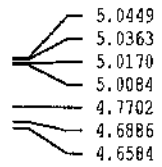
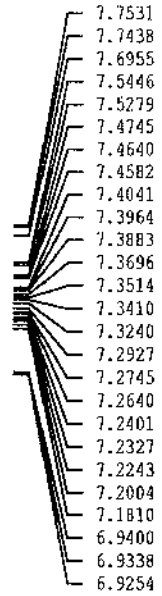
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F1 22134.80 Hz  
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<sup>1</sup>H NMR for 3b

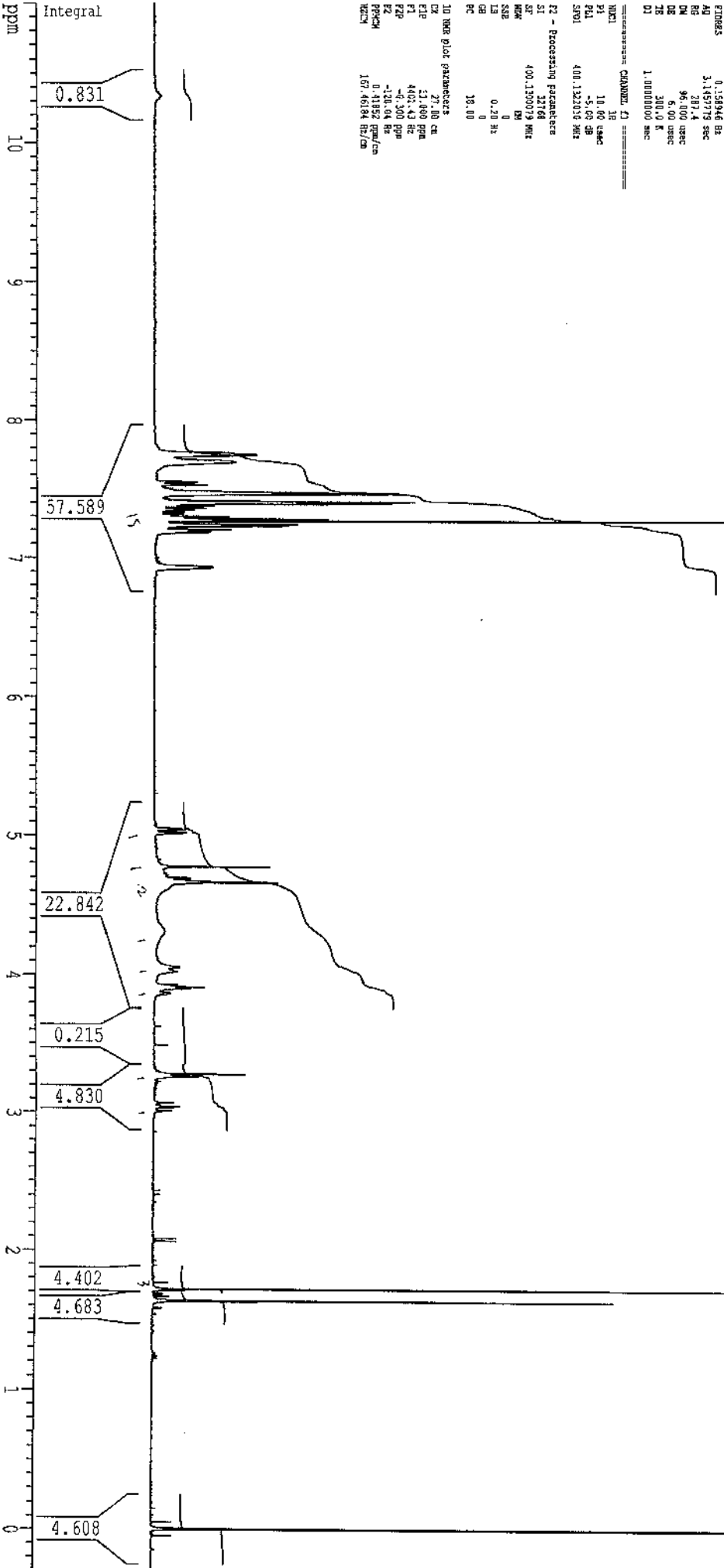
TS95182

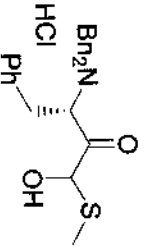
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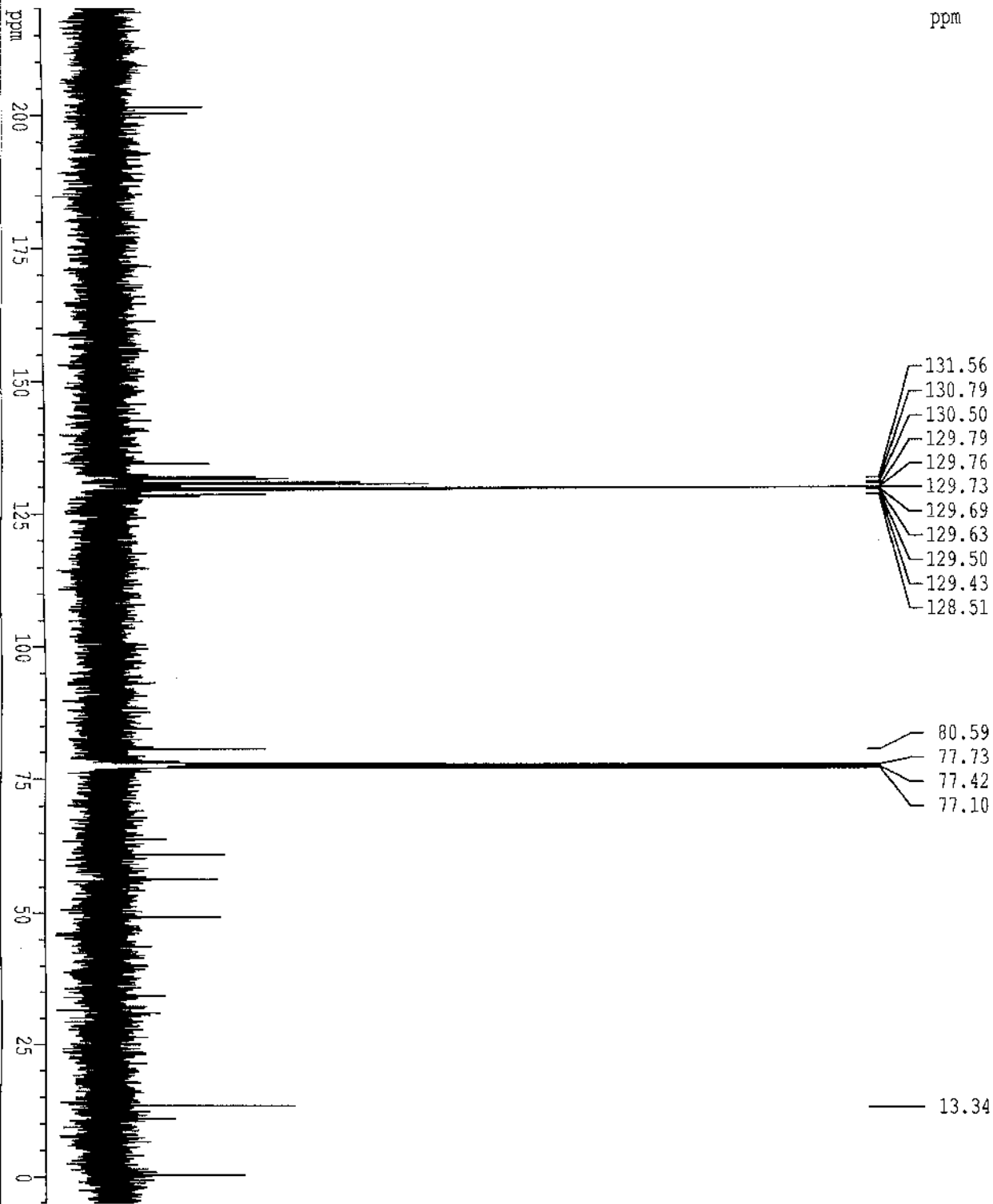
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 SSB 0  
 LB 0.20 Hz  
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 F2P -120.04 Hz  
 F2 -120.04 Hz  
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Integral



<sup>13</sup>C NMR for 3b

TS95182



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EXPNO 11  
PROCNO 1

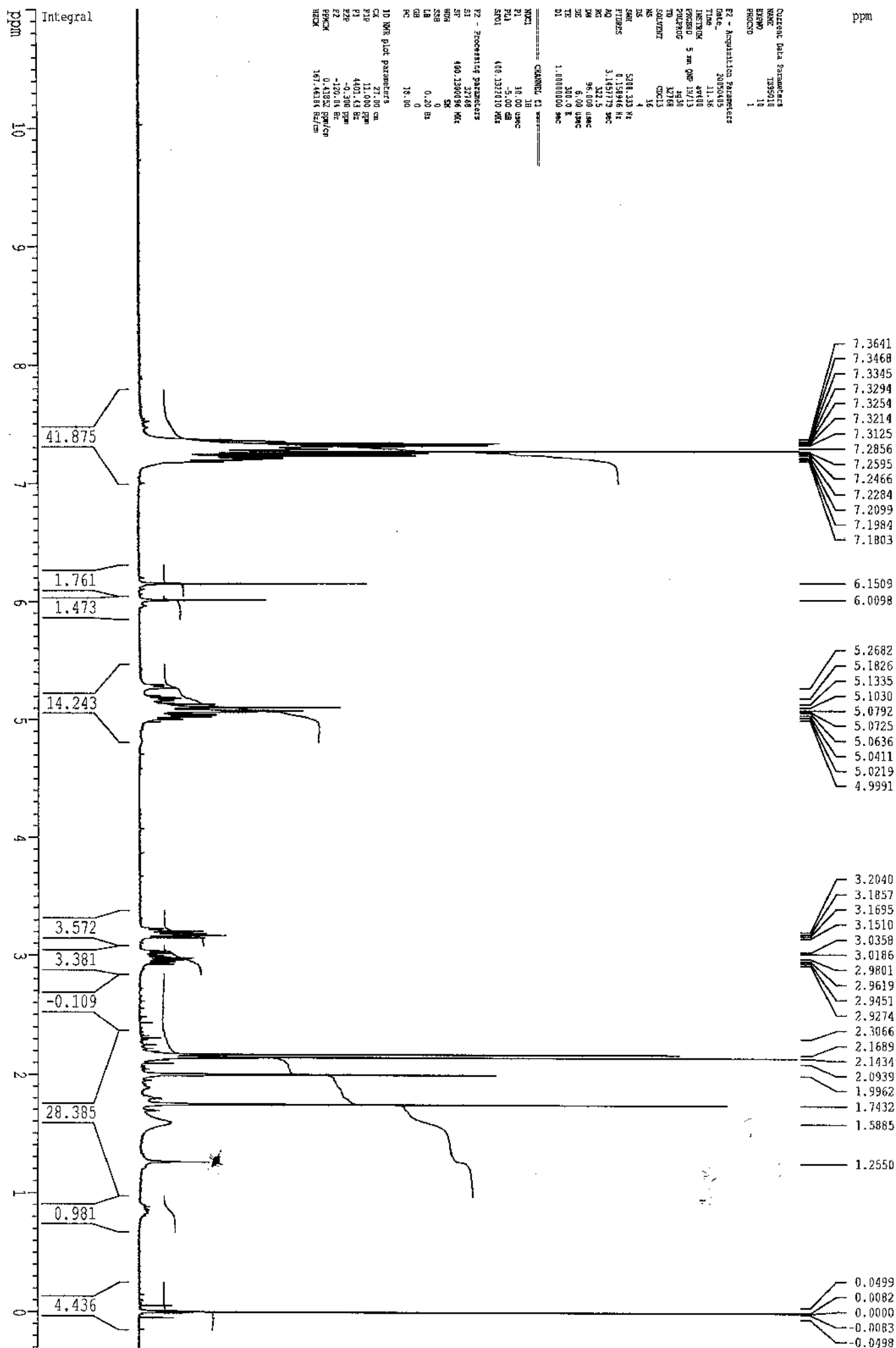
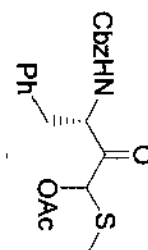
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FIDRES 0.35323 Hz  
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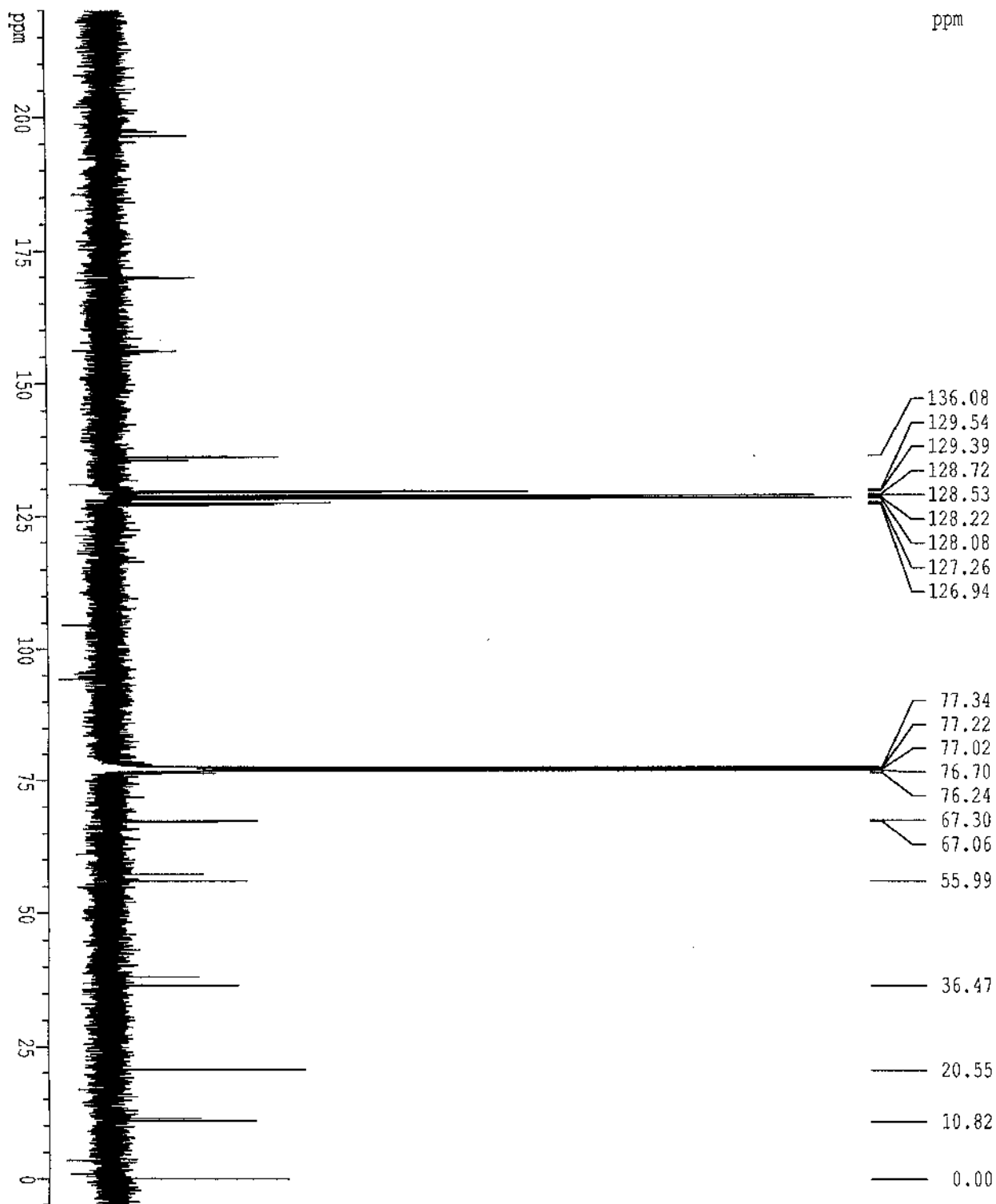
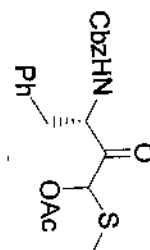
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NUC2 1H  
PCPD2 80.00 usec  
PL2 -5.00 dB  
PL12 12.50 dB  
PL13 12.50 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
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SF 100.6127290 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 3.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 220.000 ppm  
F1 22134.80 Hz  
F2P -5.000 ppm  
F2 -503.07 Hz  
PPHCA 11.25000 ppm/cm  
HZCM 1131.89319 Hz/cm

<sup>1</sup>H NMR for 4a

<sup>13</sup>C NMR for 4a

Current Data Parameters  
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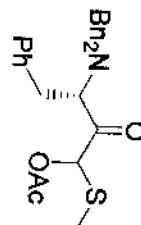
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DS 4  
SWH 23148.148 Hz  
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AQ 1.4156276 sec  
RG 16384  
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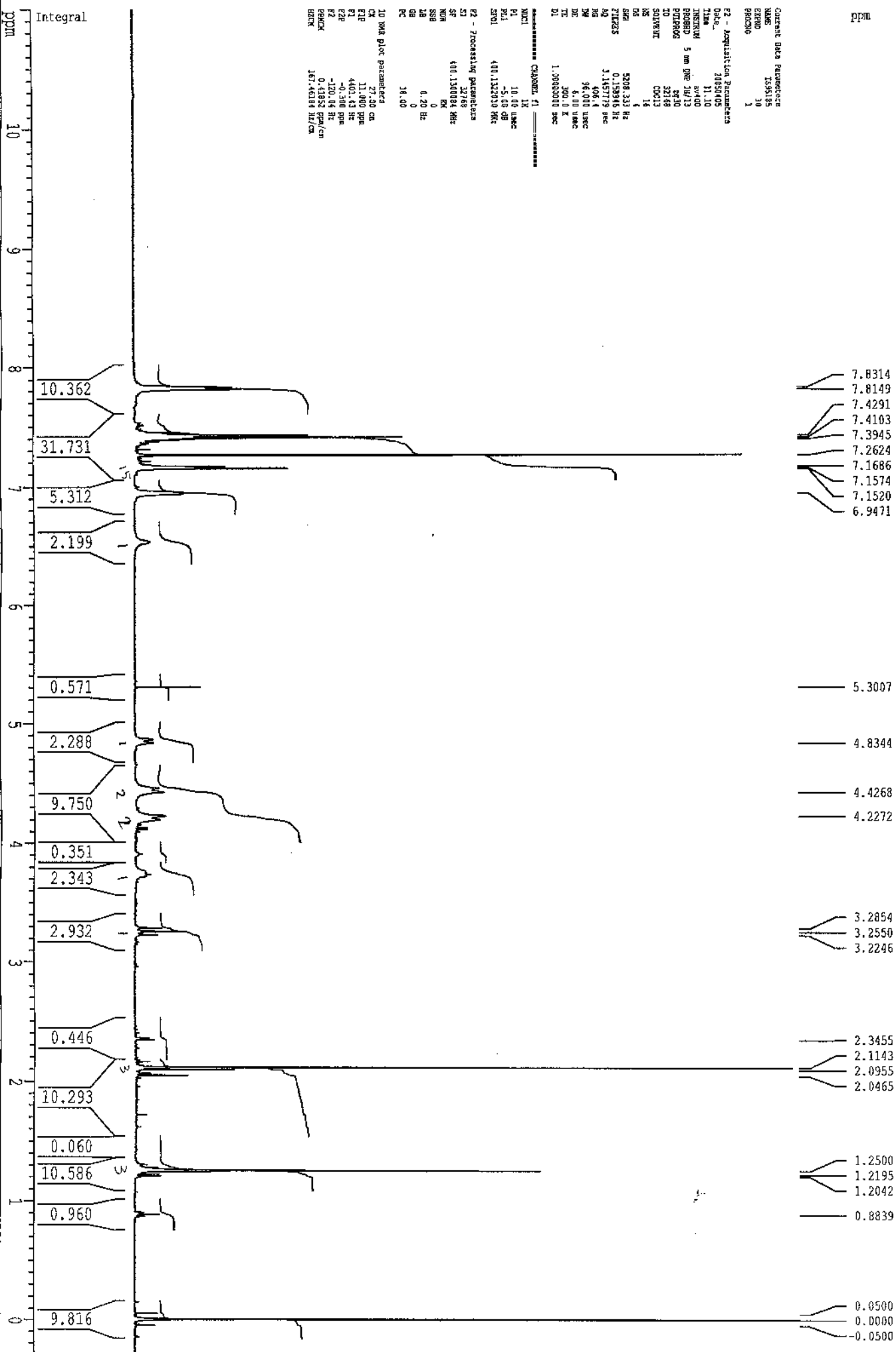
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SFO2 400.1316005 MHz

F2 - Processing parameters  
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1D NMR plot parameters  
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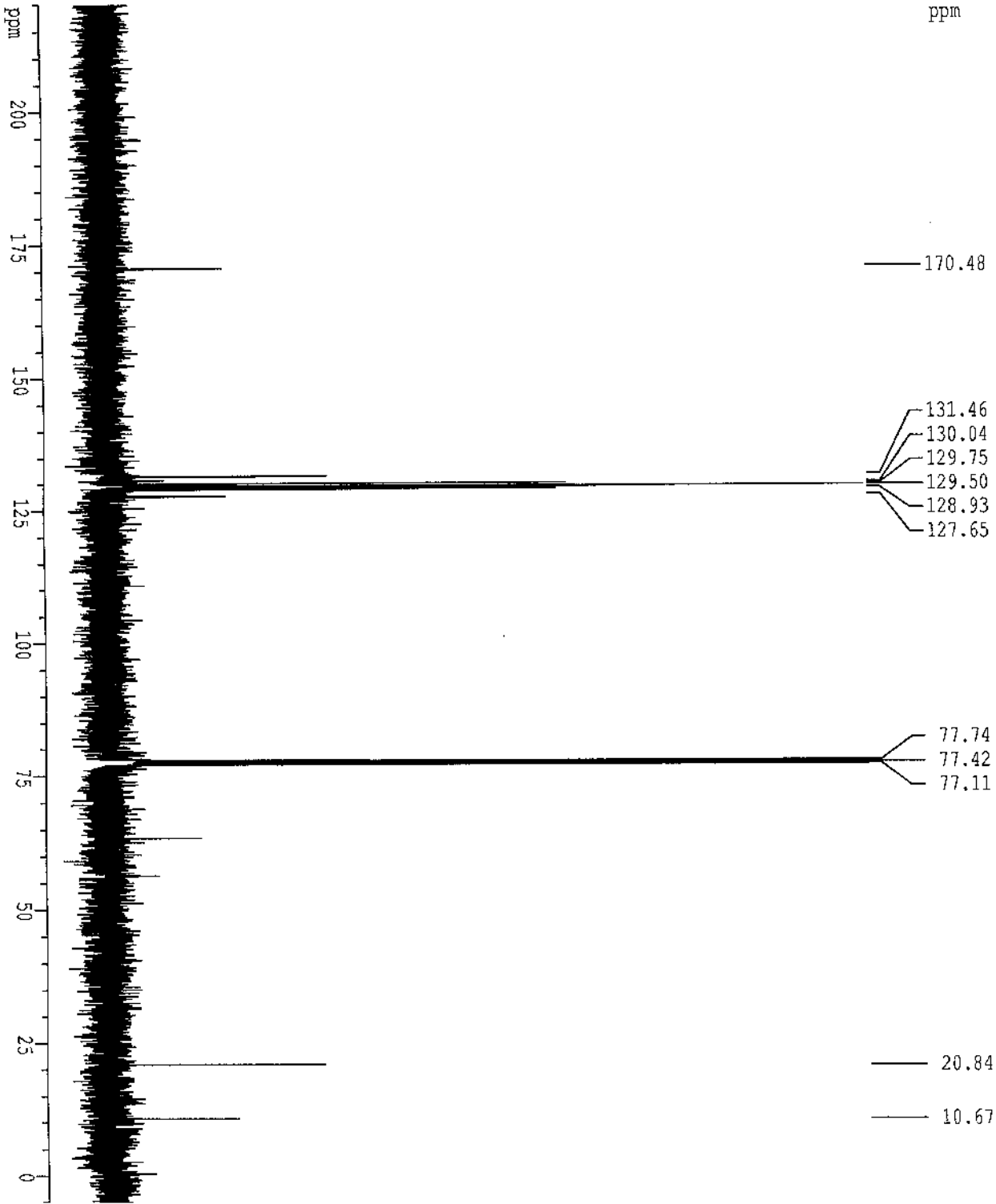
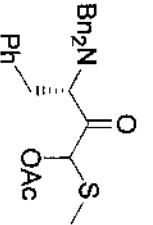


תפיל



<sup>13</sup>C NMR for 4b

TS95185



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PROCNO 1

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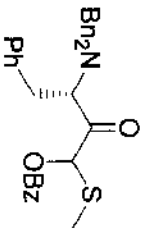
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FIDRES 0.353213 Hz  
AQ 1.4156276 sec  
RG 16384  
DE 21.600 usec  
TE 300.0 K  
D1 1.00000000 sec  
d11 0.03000000 sec  
d12 0.0002000 sec

===== CHANNEL f1 =====  
NUC1 <sup>13</sup>C  
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PL1 -3.00 dB  
SFO1 100.6207964 MHz

===== CHANNEL f2 =====  
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NUC2 <sup>1</sup>H  
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PL2 12.50 dB  
P13 12.50 dB  
SFO2 400.1316005 MHz

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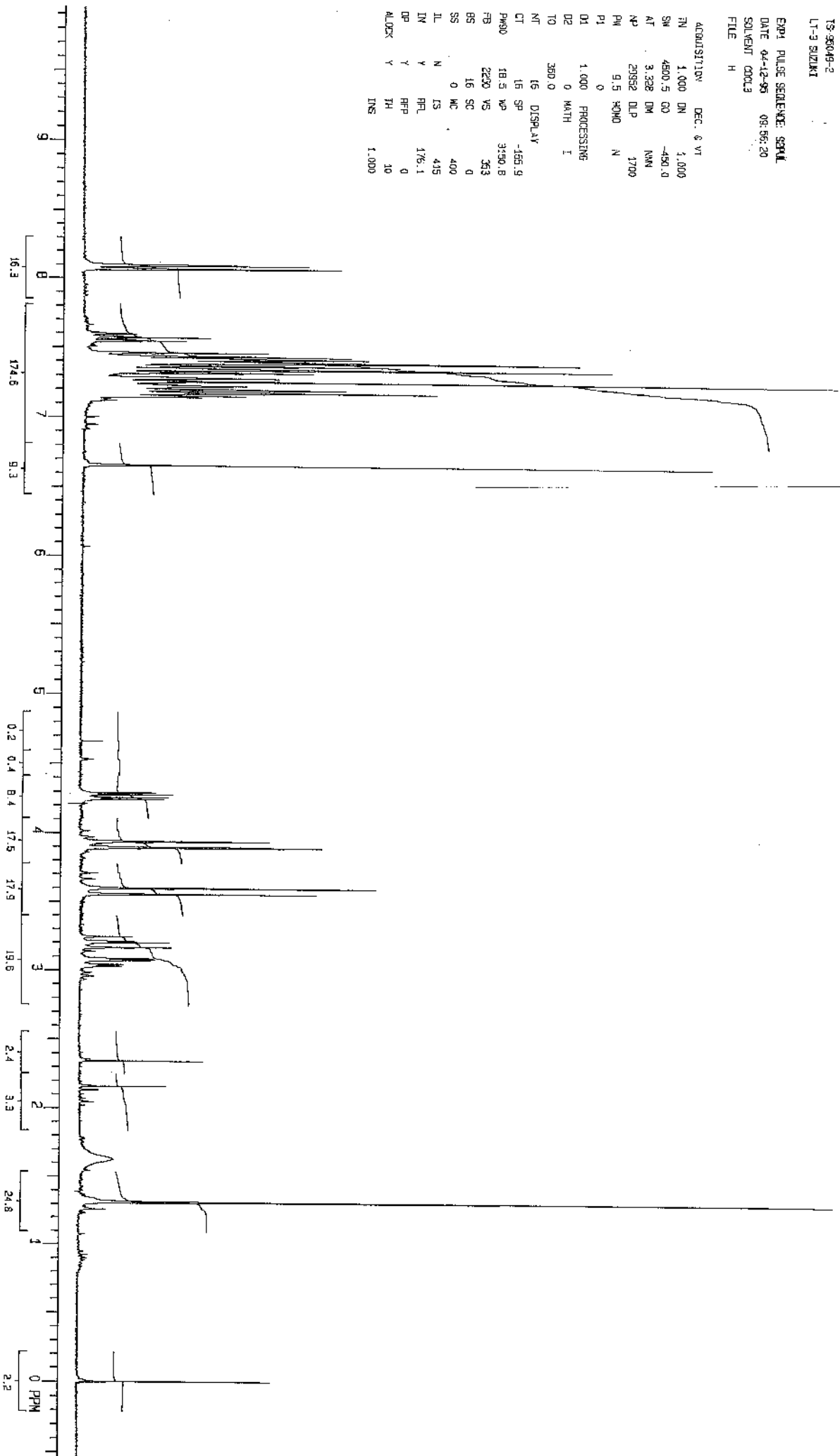
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<sup>1</sup>H NMR for 4c

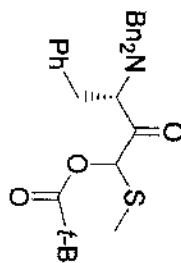
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D1 1.000 PROCESSING  
D2 0 MATH I  
TO 360.0  
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P190 18.5 W 3150.8  
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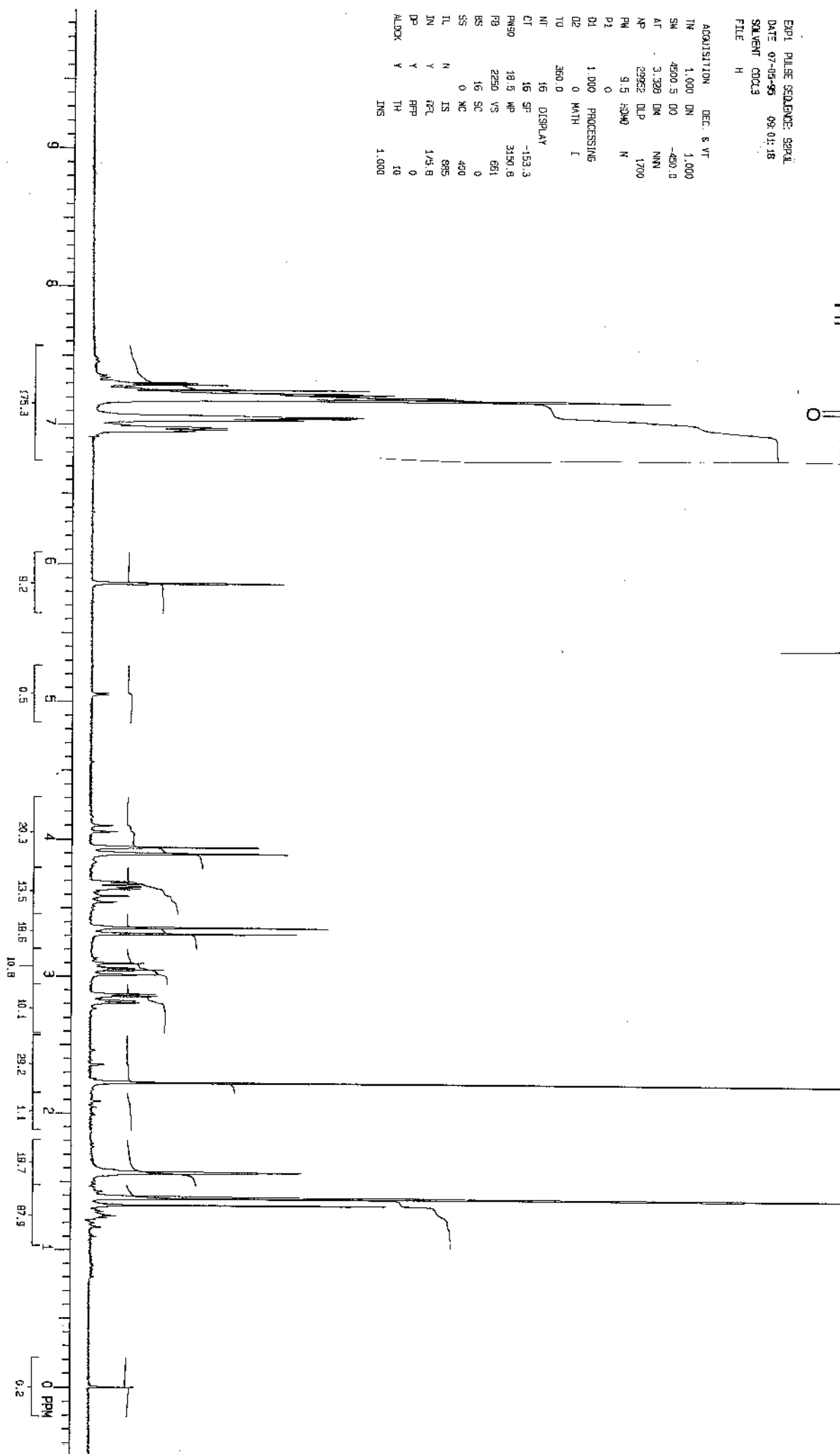


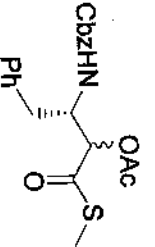


<sup>1</sup>H NMR for 4d

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PM 9.5 :3040 N  
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D2 0 MATH I  
T0 360.0  
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NT 16 DISPLAY  
CT 16 SF -153.3  
PM30 18.0 WP 3150.8  
P3 2250 VS 661  
BS 16 SC 0  
SS 0 MC 400  
TL N JS 895  
IN Y F2L 1/5 B  
DP Y FFP 0  
ALOCK Y TH 10  
INS 1.000



<sup>1</sup>H NMR for 5a

TS95024

ppm

7.3685  
7.3511  
7.3465  
7.3351  
7.3154  
7.2965  
7.2776  
7.2579  
7.2419  
7.2247  
7.1929  
7.1786  
7.1617

5.4329  
5.4231  
5.2112  
5.2054  
5.1305  
5.1060  
5.0866  
5.0557  
5.0491  
5.0193  
4.8090  
4.5547  
4.5352

2.9213  
2.9040  
2.8869  
2.8699  
2.8303  
2.8098  
2.7961  
2.7759  
2.3556  
2.3056  
2.2500  
2.2325  
2.1825  
2.1325  
1.5829

0.0499  
0.0081  
0.0000  
-0.0083  
-0.0500

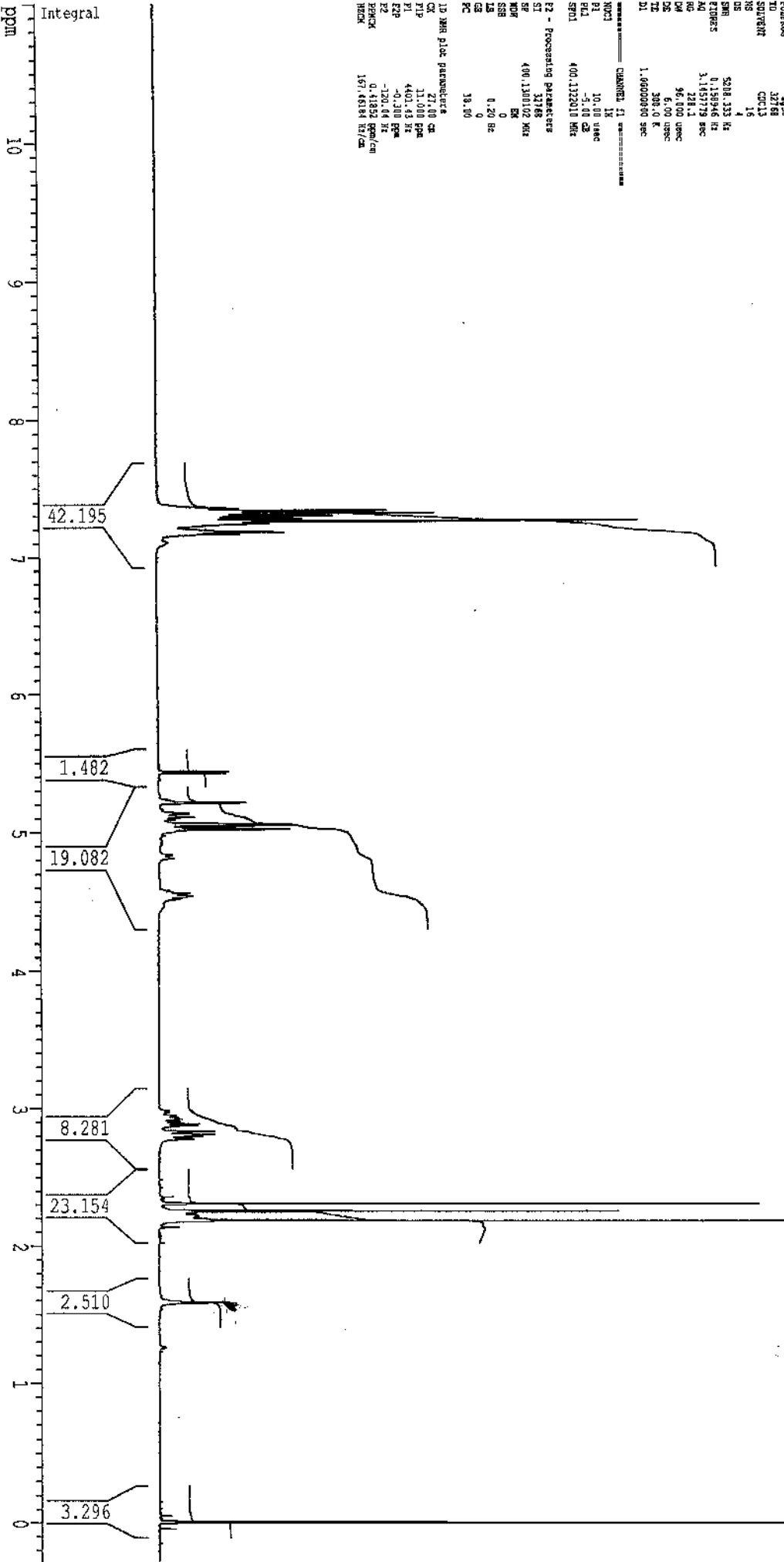
Current Data Parameters  
NAME TS95024  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20050405  
Time 11.43  
INSTRUM spect  
PROBHD 5 mm QNP 1H/13  
PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 1  
DS 4  
SWH 500.133 Hz  
FIDRES 0.15946 Hz  
AQ 3.1457779 sec  
RG 228.1  
DQ 96.000 umsec  
DE 6.00 umsec  
TE 300.0 K  
D1 1.00000000 sec

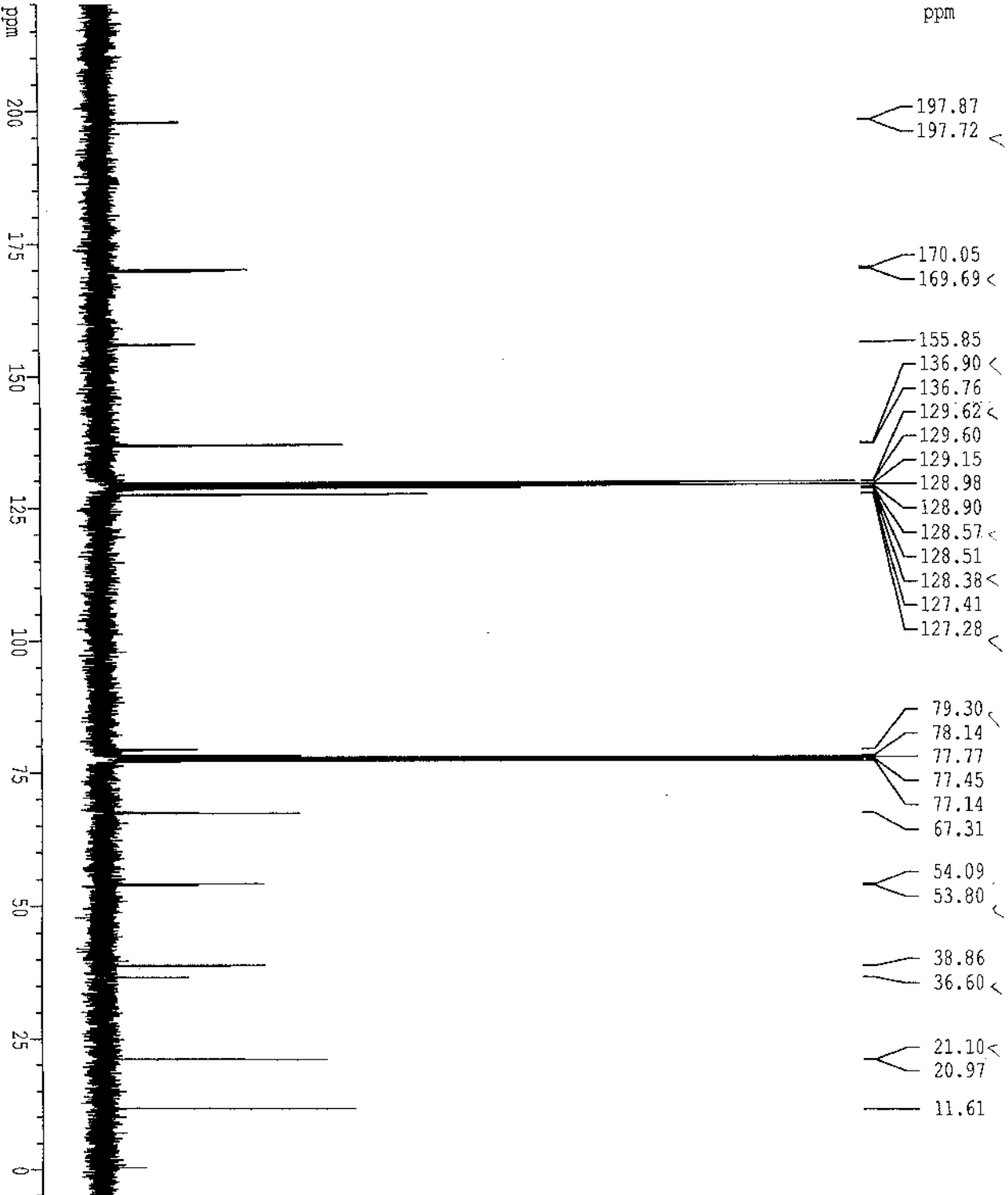
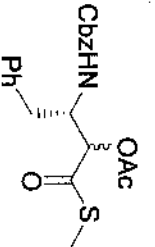
===== CHANNEL f1 =====  
NUC1 13  
P1 10.00 usec  
PL1 -5.00 dB  
SFO1 400.1122010 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1100002 MHz  
WDW EM  
SSB 0  
LB 0.20 Hz  
GB 0  
PC 18.10  
ID NMR file parameters  
CX 27.00 cm  
FID 11.000 ppm  
F1 4401.43 Hz  
F2 -0.300 ppm  
F2 -120.04 Hz  
BENCH 0.41852 ppm/cm  
HATCH 167.45184 Hz/cm

Integral



TS95024

<sup>13</sup>C NMR for 5a

Current Data Parameters  
NAME TS95024  
EXPNO 11  
PROCNO 1

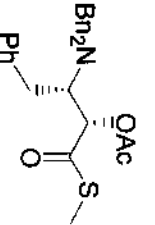
F2 - Acquisition Parameters  
Date\_ 20050407  
Time 13.57  
INSTRUM av400  
PROBHD 5 mm QNP 1H/13  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 512  
DS 4  
SWH 23148.148 Hz  
FIDRES 0.353213 Hz  
AQ 1.4156276 sec  
RG 16364  
DM 21.600 usec  
DE 20.00 usec  
TE 300.0 K  
D1 1.00000000 sec  
d11 0.03000000 sec  
d12 0.00020000 sec

CHANNEL F1  
NUC1 13C  
P1 9.40 usec  
PL1 -3.00 dB  
SFO1 100.6237964 MHz

CHANNEL F2  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -9.00 dB  
PL12 12.50 dB  
PL13 12.50 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 65536  
SF 100.6127290 MHz  
WDW 3M  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 3.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 220.000 ppm  
F1 22134.80 Hz  
F2P -5.000 ppm  
F2 -503.07 Hz  
PFCM 11.25000 ppm/cm  
H2CM 1131.89319 Hz/cm

<sup>1</sup>H NMR for 5b

TS95197

ppm

Current Data Parameters  
NAME TS95197  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20050405  
Time 11.17

INSTRUM spect  
PROBHD 5 mm QNP 1H/13  
PULPROG zgpg30  
SOLVENT CDCl3  
CONC 14

NS 4  
DS 4  
SWH 5208.333 Hz

FIDRES 0.159946 Hz  
AQ 3.1457779 sec  
RG 362

W 96.000 umax  
WDW 5.00 umax  
SSB 300.0 K

TE 300.2 K  
01 1.8000000 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.00 umax  
PL1 -5.00 dB  
SFO1 400.1322010 MHz

F2 - Processing parameters  
SI 32768  
SF 400.130002 MHz  
WDW 16  
SSB 0  
GB 0  
PC 15.00

10 NMR plot parameters  
CX 27.00 cm  
F1 11.000 ppm  
F2 400.143 Hz  
F3 0.300 ppm  
F4 -121.04 Hz  
PRGCM 0.41832 ppm/cm  
HSCM 167.46184 Hz/cm

7.7743  
7.4261  
7.4131  
7.4089  
7.3133  
7.2634  
7.2132  
7.1362

6.1758

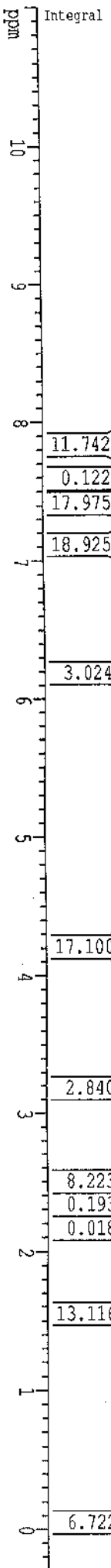
4.4993  
4.3719  
4.0674

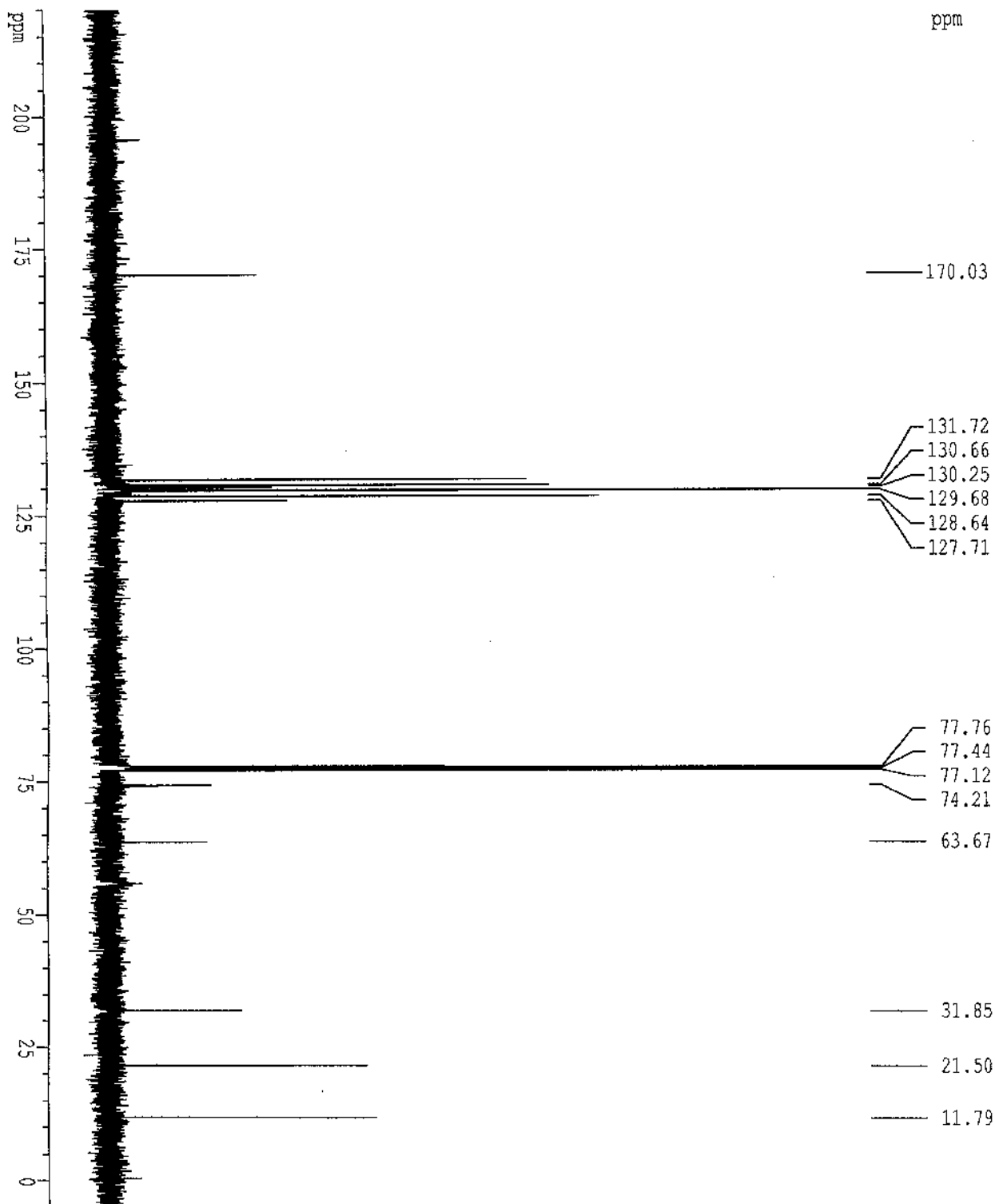
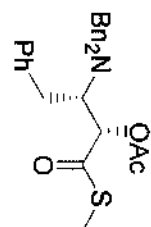
3.2118  
3.1838  
3.1513

2.4713  
2.4214  
2.3713

1.8194

0.0499  
0.0082  
0.0000  
-0.0083  
-0.0501



<sup>13</sup>C NMR for 5b

Current Data Parameters  
NAME TS95197  
EXPNO 31  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20050408  
Time 12.56  
INSTRUM av400  
PROBHD 5 mm QNP 1H/13  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 512  
DS 4  
SWE 23148.148 Hz  
FIDRES 0.353213 Hz  
AQ 1.4156276 sec  
RG 16384  
DM 21.600 usec  
DE 20.00 usec  
TE 300.0 K  
D1 1.00000000 sec  
d11 0.03000000 sec  
d12 0.00002000 sec

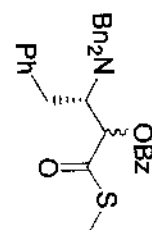
CHANNEL F1  
NUC1 13C  
P1 9.40 usec  
PL1 -3.00 dB  
SFO1 100.6237964 MHz

CHANNEL F2  
CPDPRG2 waltz16

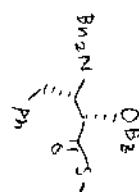
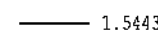
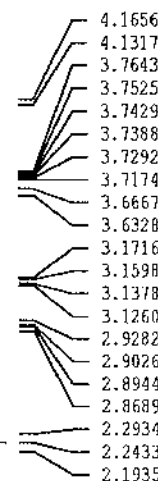
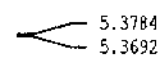
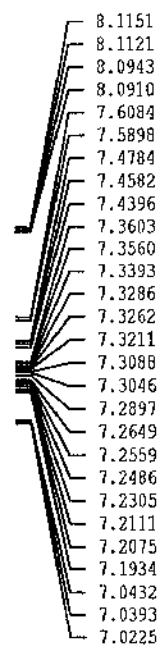
NUC2 1H  
PCPD2 80.00 usec  
PL2 -5.00 dB  
PL12 12.50 dB  
PL13 12.50 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 65536  
SF 100.6127290 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 3.00

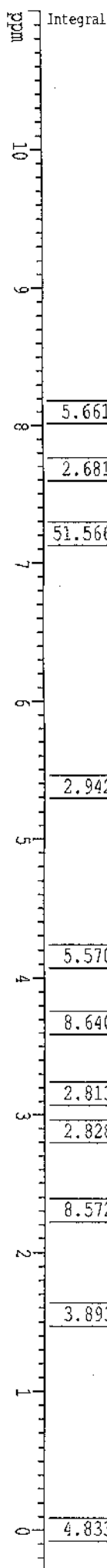
1D NMR plot parameters  
CX 20.00 cm  
F1P 220.000 ppm  
F1 22334.80 Hz  
F2P -5.000 ppm  
F2 -503.07 Hz  
PPMKM 11.25090 ppm/cm  
HZCM 1131.89319 Hz/cm

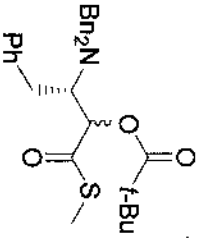
<sup>1</sup>H NMR for 5c

ppm



Current Data Parameters  
 NAME: 135033  
 EXPNO: 10  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20050405  
 Time: 11.56  
 INSTRUM: spect  
 PROBRW: 5 mm CP 1H/13  
 PULPROG: zgpg30  
 TD: 32768  
 SOLVENT: CDCl3  
 NS: 16  
 DS: 4  
 SWH: 5208.333 Hz  
 FIDRES: 0.184946 Hz  
 AQ: 3.119717 sec  
 RG: 313  
 AC: 32768  
 ZM: 36.000 umsec  
 DE: 5.00 umsec  
 TE: 300.0 K  
 D1: 1.0000000 sec  
 CHANNEL: C1  
 NUC1: 13C  
 P1: 10.00 usec  
 PL1: -5.00 dB  
 SFO1: 400.132610 MHz  
 F2 - Processing parameters  
 SI: 32768  
 SF: 400.130012 MHz  
 WDW: EM  
 SSB: 0  
 LB: 0.20 Hz  
 GB: 0  
 PC: 18.00  
 IN NMR plot parameters  
 G1: 17.00 cm  
 G2: 17.00 cm  
 F1: 11.00 ppm  
 F2: 4.0143 ppm  
 F3: -8.100 ppm  
 F4: -12.04 Hz  
 PRNU: 0.01852 ppm/cm  
 RECK: 167.45184 Hz/cm

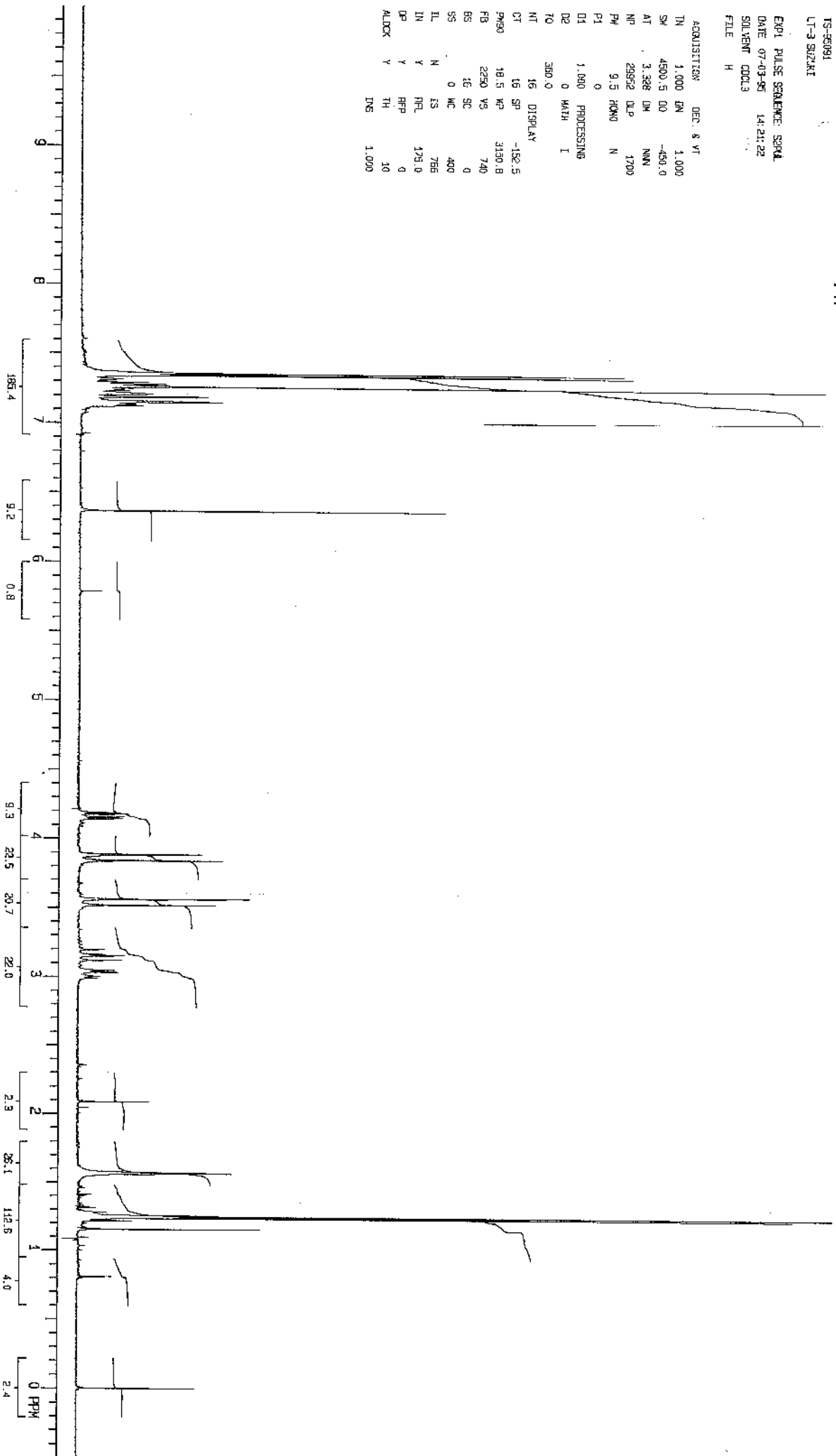


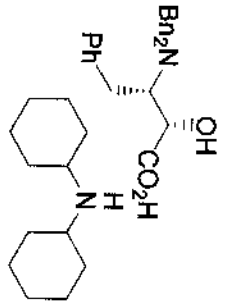
<sup>1</sup>H NMR for 5d

15-00091  
LT-3 SUZUKI

EXP1 PULSE SEQUENCE: zgpg30  
DATE: 07-03-95 14:21:22  
SOLVENT: CDCl3  
FILE: H

ACQUISITION DEC. 8 VT  
 TN 1.000 EN 1.000  
 SM 4500.5 D0 -450.0  
 AT 3.368 DM NMR  
 NP 29952 DLP 1700  
 PH 9.5 PGM N  
 P1 0  
 D1 1.080 PROCESSING  
 D2 0 MATH I  
 TO 360.0  
 NT 16 DISPLAY  
 CT 16 SP -152.5  
 PWB 18.5 WP 3130.8  
 FB 2250 VS 740  
 BS 16 SC 0  
 SS 0 MC 400  
 EL N 13 766  
 IN Y PFL 129.0  
 DP Y PFP 0  
 TH 10  
 ALLOC Y DMS 1.000



<sup>1</sup>H NMR for 6b

opu

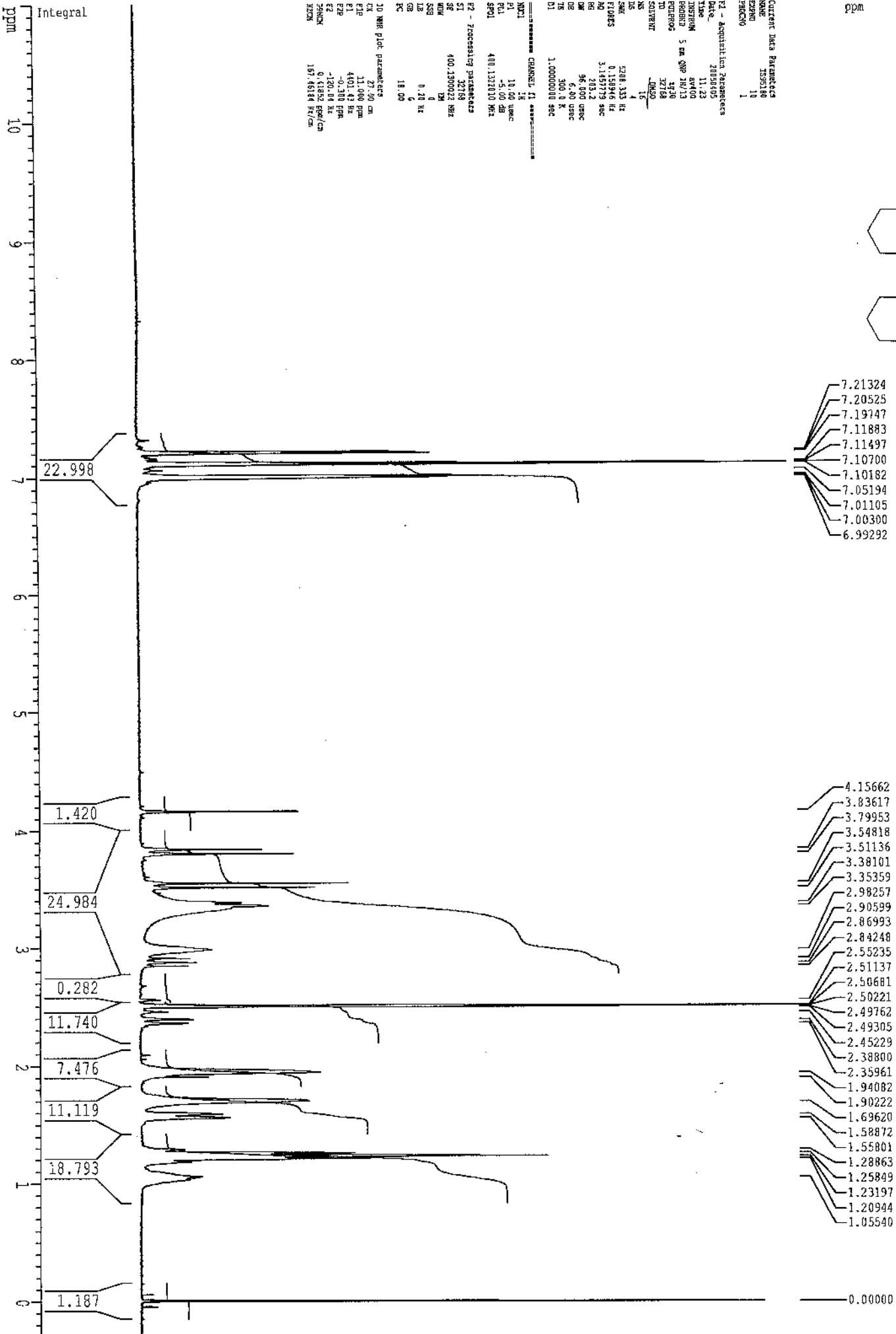
Current Data Parameters  
NAME 1595180  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100505  
Time 11:23  
INSTRUM spect  
PROBHD 5 mm QNP 1H/13  
PULPROG zgpg30  
TD 32768  
SOLVENT DMSO  
NS 16  
DS 4  
SWH 5208.333 Hz  
FIDRES 0.158946 Hz  
AQ 3.145773 sec  
RG 203.2  
WM 96.000 usec  
DE 6.00 usec  
TE 300.2 K  
D1 1.0000000 sec

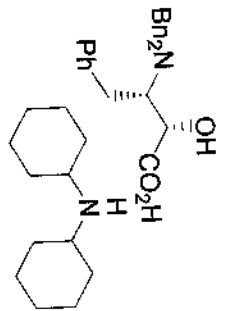
===== CHANNEL f1 =====  
NUC1 13  
P1 10.00 usec  
PL 0  
PC 1  
SFO1 400.137800 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1378002 MHz  
WDW EM  
SSB 0  
LB 0.20 Hz  
GB 6  
CB 18.00

10 NMR plot parameters  
CX 27.30 cm  
FIP 11.060 ppm  
F1 4401.43 Hz  
F2 -0.300 ppm  
FZ -120.04 Hz  
PCNCK 0.41852 ppm/cm  
HZCN 167.46144 Hz/cm



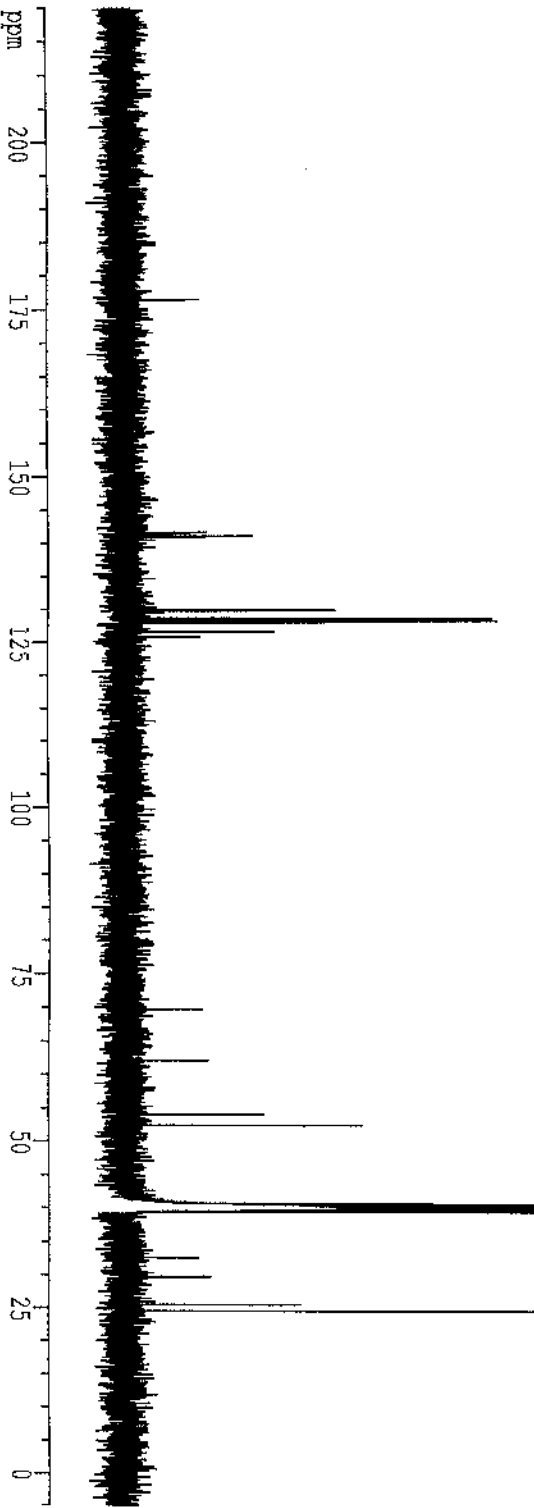


<sup>13</sup>C NMR for 6b

ppm

140.96  
129.83  
128.48  
128.07  
127.85  
126.55

53.92  
52.24  
40.51  
40.30  
40.09  
39.88  
39.67  
39.47  
39.26  
25.29  
24.39



Current Data Parameters  
NAME TS95180  
EXNO 11  
PROCNO 1

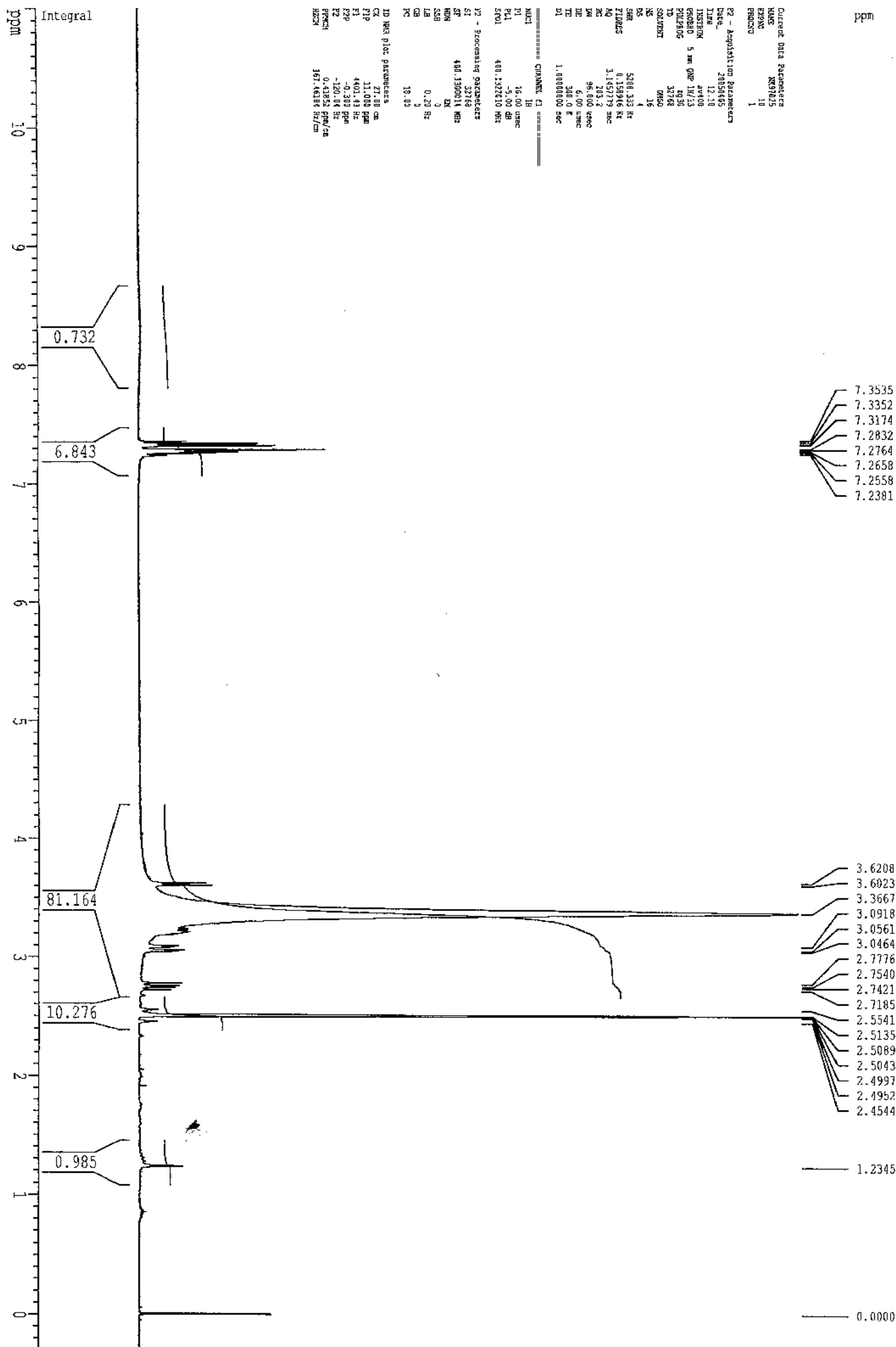
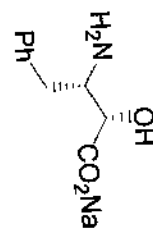
F2 - Acquisition Parameters  
Date\_ 20050406  
Time 4.37  
INSTRUM av400  
PROBHD 5 mm QNP 1H/13  
PULPROG zgpg30  
TD 65536  
SOLVENT DMSO  
NS 1536  
DS 4  
SFR 23148.148 Hz  
FIDRES 0.353213 Hz  
AQ 1.4156276 sec  
RG 16384  
DM 21.600 usec  
DE 20.00 usec  
TE 300.0 K  
D1 1.00000000 sec  
d11 0.03000000 sec  
d12 0.00002000 sec

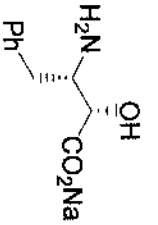
CHANNEL F1  
NUC1 13C  
P1 9.40 usec  
PL -3.00 dB  
SFO1 100.6237964 MHz

CHANNEL F2  
CPRPG2 Waltz16  
NUC2 1H  
PCPD2 80.00 usec  
P12 -5.00 dB  
P12 12.50 dB  
P13 12.50 dB  
SFO2 400.1316005 MHz

F2 - Processing Parameters  
SI 65536  
SF 100.6127793 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 3.00

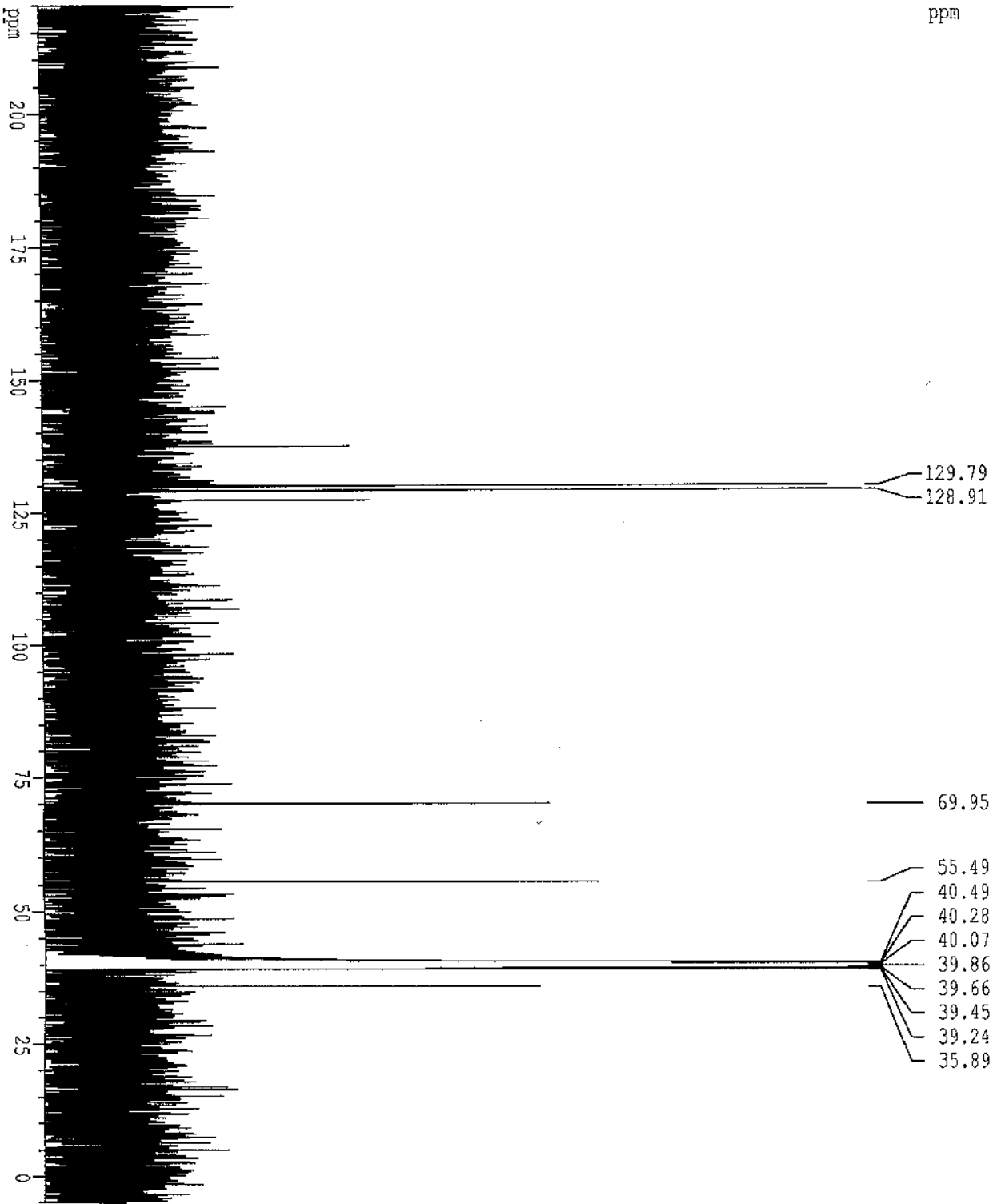
1D NMR plot parameters  
CX 20.00 cm  
F1P 220.000 ppm  
F1 22334.81 Hz  
F2P -5.000 ppm  
F2 -503.07 Hz  
PPKCM 11.25000 ppm/cm  
HZCM 1131.89380 Hz/cm

<sup>1</sup>H NMR for 6c

<sup>13</sup>C NMR for 6c

NK97025

ppm



Current Data Parameters  
NAME NK97025  
EXPNO 11  
PROCNO 1

F2 - Acquisition Parameters

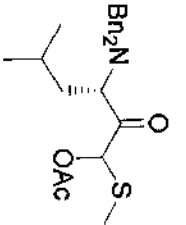
Date\_ 20050407  
Time 3.09  
INSTRUM av400  
PROBHD 5 mm QNP 1H/13  
PULPROG zgpg30  
TD 65536  
SOLVENT DMSO  
NS 4096  
DS 4  
SWH 23148.148 Hz  
FIDRES 0.353213 Hz  
AQ 1.4156276 sec  
RG 16384  
DM 21.600 usec  
DE 20.00 usec  
TE 300.0 K  
D1 1.00000000 sec  
d11 0.03000000 sec  
d12 0.00002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 9.40 usec  
PL1 -3.00 dB  
SFO1 100.6237964 MHz

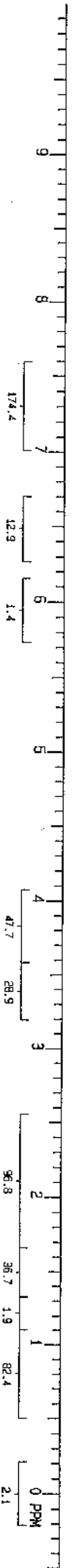
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -5.00 dB  
PL12 12.50 dB  
PL13 12.50 dB  
SFO2 400.1316005 MHz

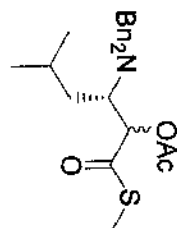
F2 - Processing parameters  
SI 65536  
SF 100.6127793 MHz  
WDW EM  
SS3 0  
LB 1.00 Hz  
GB 0  
PC 3.00

ID RMS plot parameters  
CX 20.00 cm  
F1P 220.000 ppm  
F1 22134.81 Hz  
F2P -5.000 ppm  
F2 -503.07 Hz  
PPMCM 11.25000 ppm/cm  
R2CM 1131.89380 Hz/cm

<sup>1</sup>H NMR for 4e

NAME: 4e-CH (OAc) SMe  
15-96107  
LT-3 SUZUKI  
EXP: PULSE SEQUENCE: zgpg30  
DATE: 08-26-96 15:16:55  
SOLVENT: CDCl3  
FILE: H  
ACQUISITION DEC: 6 VT  
IN 1.000 DV 1.000  
SM 4500.5 DD -400.0  
AT 3.328 SM NNN  
NP 29882 DLP 1700  
PW 9.5 H2O N  
P1 0  
D1 1.000 PROCESSING  
D2 0 MATH L  
TO 360.0  
NT 15 DISPLAY  
CT 16 SP -153.0  
PHE0 19.0 NP 3150.8  
FR 2250 VS 289  
BS 16 SC 0  
SS 0 MC 408  
IL N IS 703  
IN Y REL 175.5  
DP Y FFP 0  
ALOCK Y TH 10  
INS 1.000



<sup>1</sup>H NMR for 5c

ppm

7.4540  
7.4374  
7.3779  
7.3542  
7.3284  
7.3224  
7.3141  
7.2995  
7.2931  
7.2784  
7.2575  
7.2511  
7.2433  
7.2356  
7.2205  
6.5665

5.8313  
5.8276

4.4165  
4.3196  
4.2669  
3.9298  
3.9127  
3.8961  
3.8272  
3.5391  
3.3203  
3.2865  
3.2499  
2.5985  
2.3438  
2.2919  
2.2790  
2.2753  
2.2609  
2.2254  
2.1978  
2.0791  
2.0122  
1.8454  
1.8377  
1.8281  
1.8106  
1.7826  
1.0371  
1.0297  
1.0103  
1.0018  
0.9937  
0.9773  
0.9652  
0.9438  
0.9272  
0.9180  
0.9019  
0.8769  
0.8603  
0.3716  
0.3556  
0.0000

Current Data Parameters  
NAME TS96109  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters

Date\_ 20050407  
Time 16.49  
INSTRUM spect  
PROBHD 5 mm QNP 1H/13  
PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 4  
DS 4  
SWH 5018.333 Hz  
FIDRES 0.438946 Hz  
AQ 3.1457779 sec  
RG 161.1  
WM 96.010 umsc  
DB 6.00 umsc  
DE 300.0 K  
TE 1.00000000 sec

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

NUC1 1H  
P1 10.00 umsc  
PL1 -5.00 dB  
SFO1 400.1327110 MHz

F2 - Processing parameters

SF 400.1300102 MHz  
WDW EM  
SSB 0  
LB 0  
GB 0  
PC 18.00

1D NMR PLOT PARAMETERS  
CX 27.00 cm  
CT 11.000 ppm  
F1 440.43 Hz  
F2 -0.300 ppm  
F3 -320.04 Hz  
SFREQ 0.43832 ppm/cm  
ZPCN 167.46184 Hz/cm  
ZPCN 320K

Integral

31.850

2.188

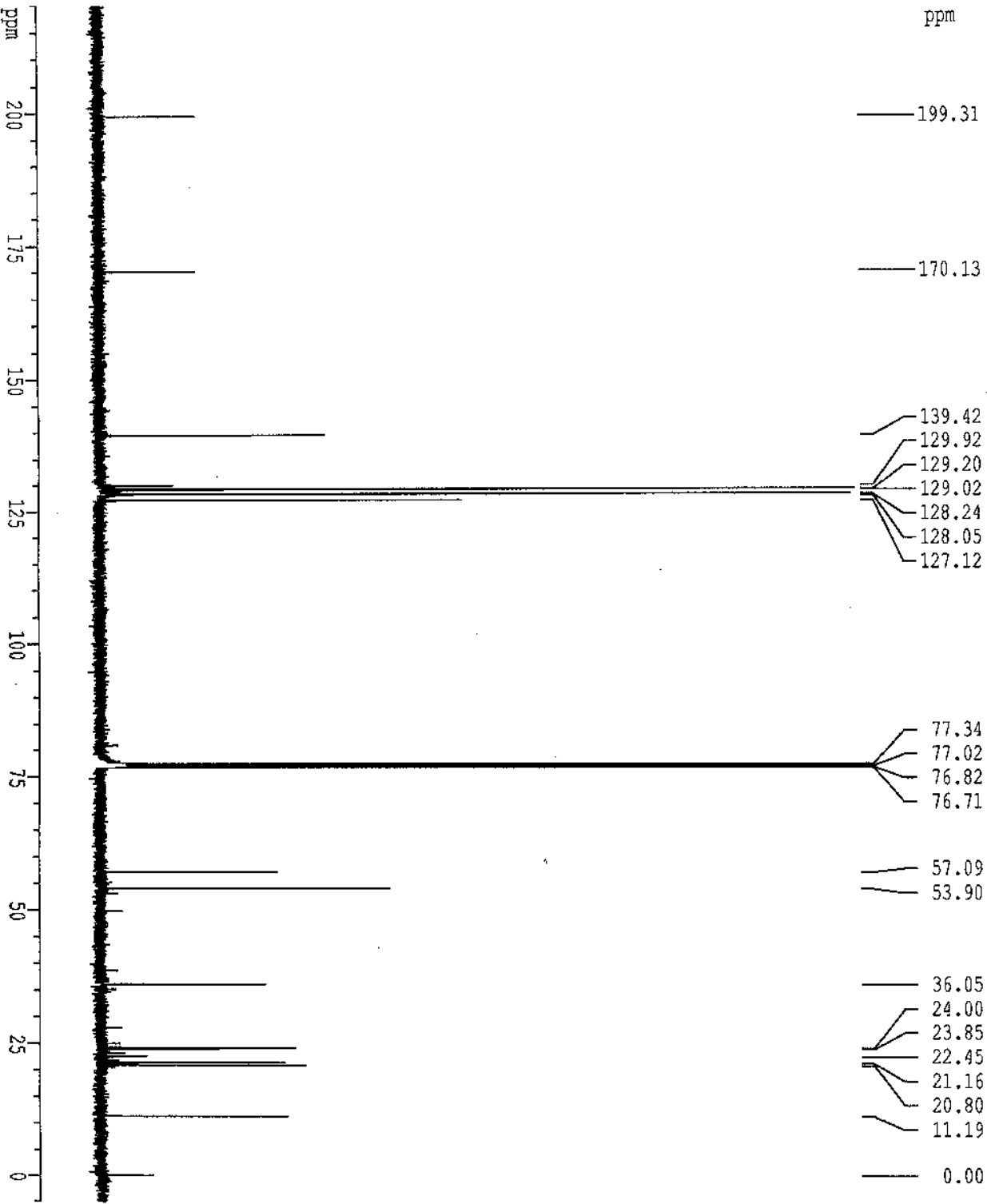
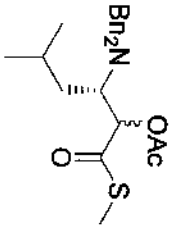
15.128

41.660

6.737

2.437

ppm  
10  
9  
8  
7  
6  
5  
4  
3  
2  
1  
0

<sup>13</sup>C NMR for 5e

Current Data Parameters  
NAME TS96108  
EXPNO 11  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20050408  
Time 3.30  
INSTRUM av400  
PROBHD 5 mm QNP 1H/13  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 5120  
DS 4  
SWH 23148.148 Hz  
FIDRES 0.353213 Hz  
AQ 1.4156276 sec  
RG 16384  
DM 21.600 usec  
DE 20.00 usec  
TE 300.0 K  
B1 1.00000000 sec  
d11 0.03000000 sec  
d12 0.0002009 sec

CHANNEL f1  
NUC1 13C  
P1 9.40 usec  
PL1 -3.00 dB  
SFO1 100.6237964 MHz

CHANNEL f2  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -5.00 dB  
PL12 12.50 dB  
PL13 12.50 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 65536  
SF 100.6127681 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 3.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 220.000 ppm  
F1 22134.81 Hz  
F2P -5.000 ppm  
F2 -503.07 Hz  
PPMCM 11.25000 ppm/cm  
HZCM 1131.89368 Hz/cm

### Analysis for the optical purity of 6b

Column: Chiralcel OD 4.6 x 250 mm, Temp: Ambient

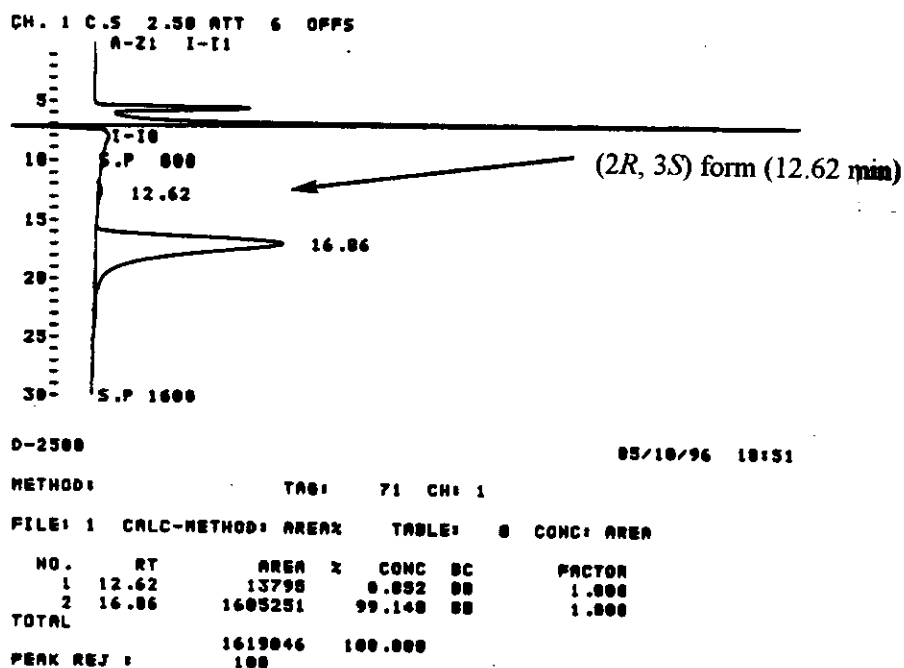
Eluent: *n*-hexane / isopropanol / formic acid = 74 / 25 / 1

Wave length : 254 nm,

Flow rate : 0.5 mL / min

Injected volume: 10  $\mu$ L

### Chromatogram for the sample



### Chromatogram for the racemic mixture

