

Design, Synthesis and Functional Activity of Labeled CD1d Glycolipid Agonists

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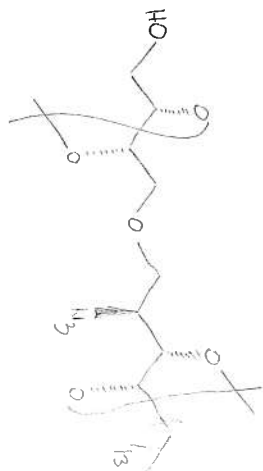
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Supporting Information II

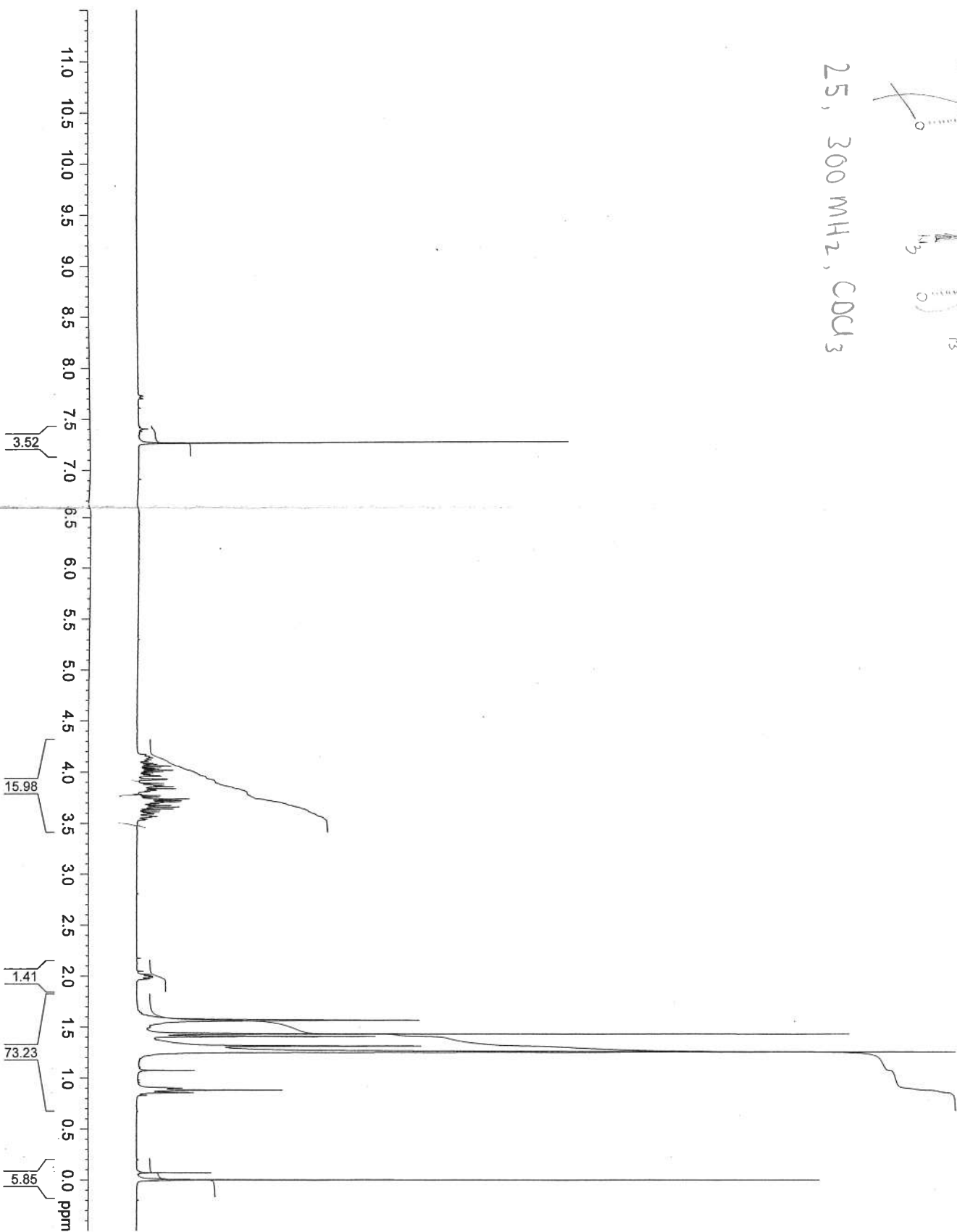
Justyna Wojno - JW3-65



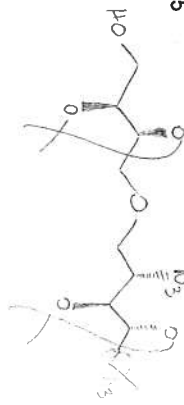
25, 300 MHz, CDCl₃

NAME 08-18-Beazra-18
EXPNO 10
PROCNO 1
Date_ 20090818
Time 12:04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 32
DS 2
SWMH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 512
DWT 83.200 usec
DE 13.19 usec
TE 283.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.80 usec
PL1 1.00 dB
PL1W 9.57725906 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300055 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



JustynaWojno - JW3-65



25, 100 MHz, CDCl₃

- 134.8
- 129.6
- 127.7

- 109.4
- 108.3

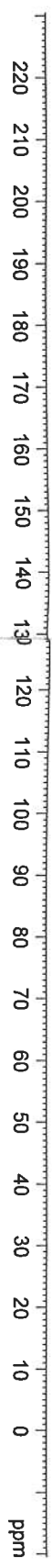
- 79.4
- 77.8
- 77.4
- 77.0
- 76.7
- 76.0
- 75.6
- 72.9
- 71.9
- 62.3
- 59.9

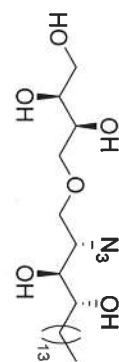
- 31.9
- 29.7
- 29.6
- 29.4
- 29.4
- 28.1
- 27.0
- 27.0
- 26.4
- 25.6
- 22.7
- 14.1

NAME 08-18-Besra-4
EXPNO 10
PROCNO 1
Date_ 20090818
Time 12:17
INSTRUM spect
PROBHD 5 mm PABUL 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 512
DS 4
SWH 25252.525 Hz
FIDRES 0.366323 Hz
AQ 1.2876629 sec
RG 2050
DW 19.800 usec
DE 19.800 usec
TE 296.3 K
CNST72 146.0000000
CNST73 1.0000000
CNST74 5.0000000
D1 1.5000000 sec
D2 0.00172414 sec
D3 0.00437034 sec
DT2 0.00002000 sec
DT3 0.00009400 sec
TD0 1

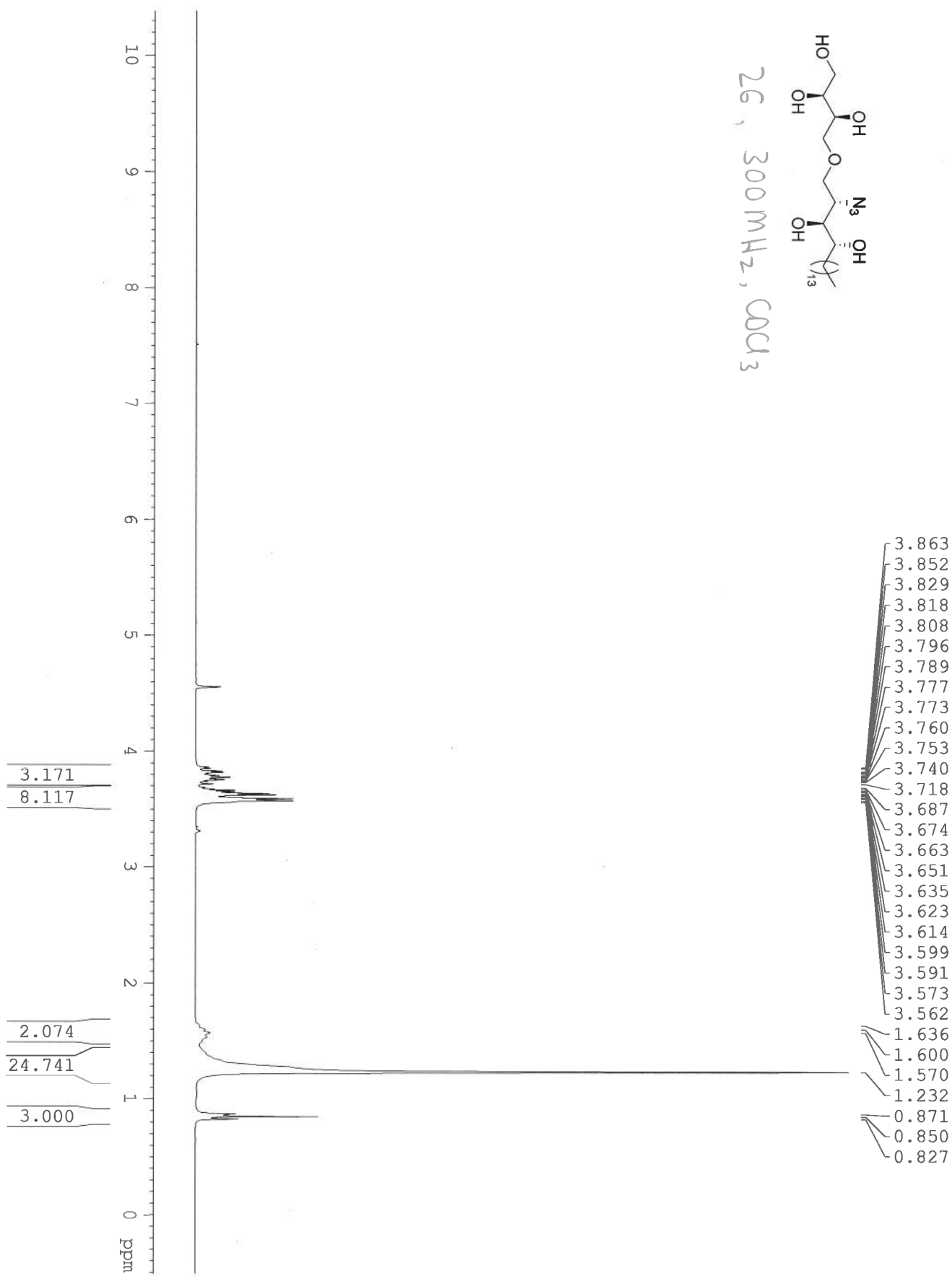
===== CHANNEL f1 =====
NUC1 ¹³C
P1 8.80 usec
PL1 -17.60 usec
PL1W -3.00 dB
SFO1 58.63890457 W
SFO1 100.6233333 MHz

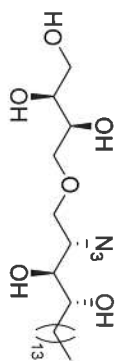
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ¹H
P3 9.70 usec
P4 19.40 usec
PCPD2 90.00 usec
PL2 -4.00 dB
PL12 15.35 dB
PL2W 24.29165867 W
PL12W 0.28213742 W
SFO2 400.1316005 MHz
SI 65536
SE 100.6127690 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.00



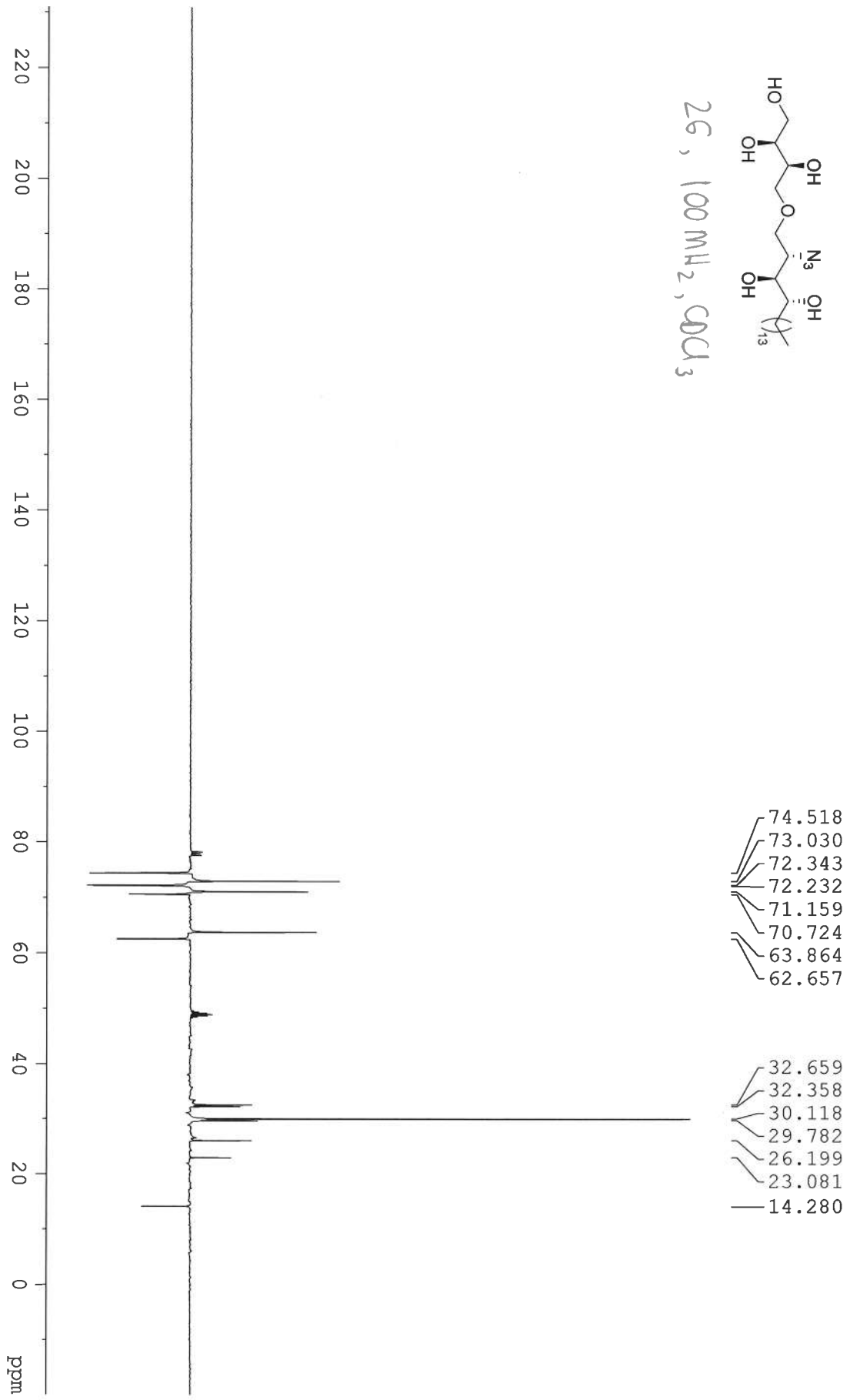


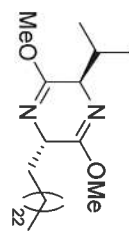
26, 300 MHz, CDCl₃





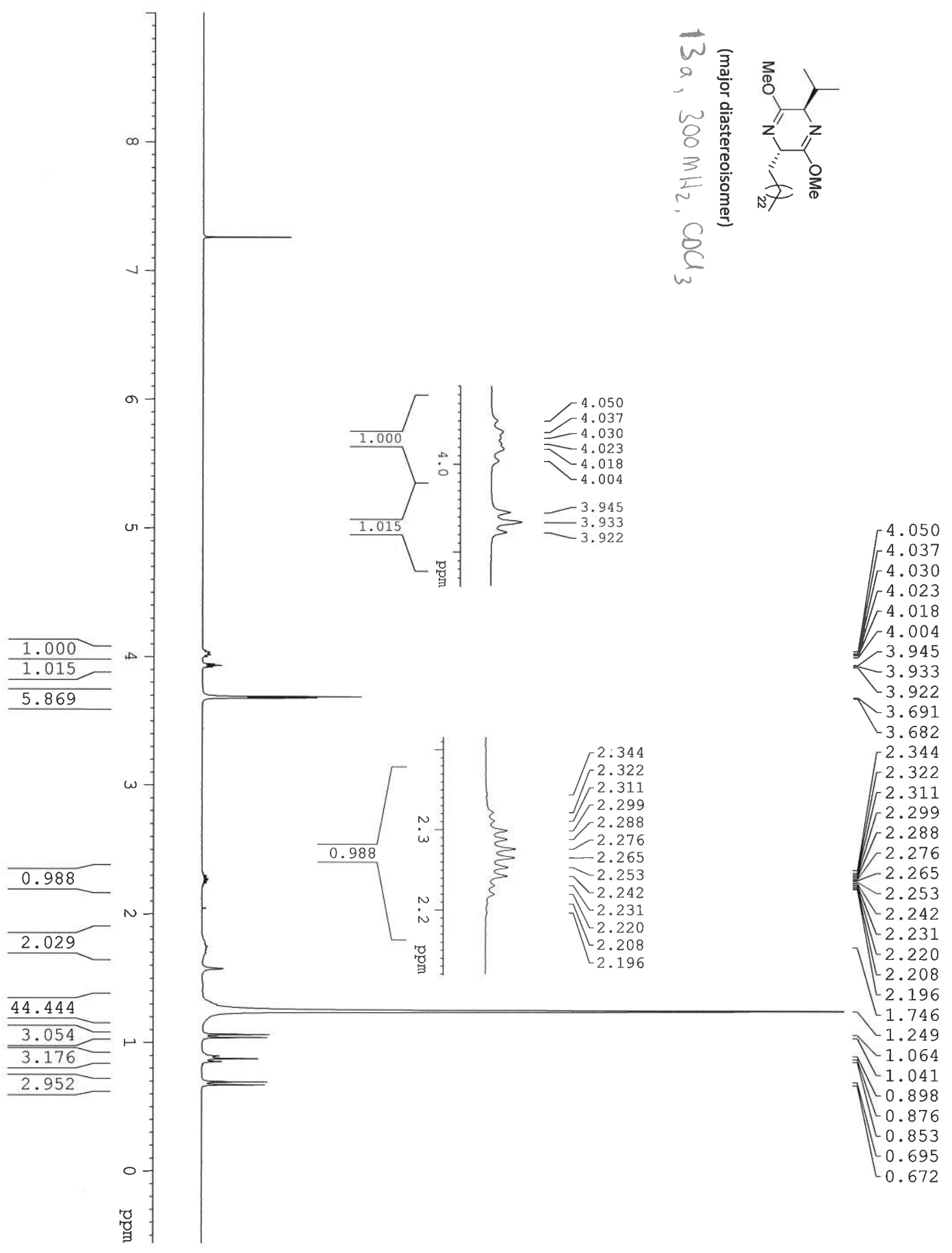
26, 100 MHz, CDCl₃

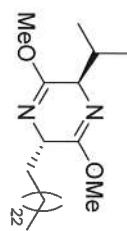




(major diastereoisomer)

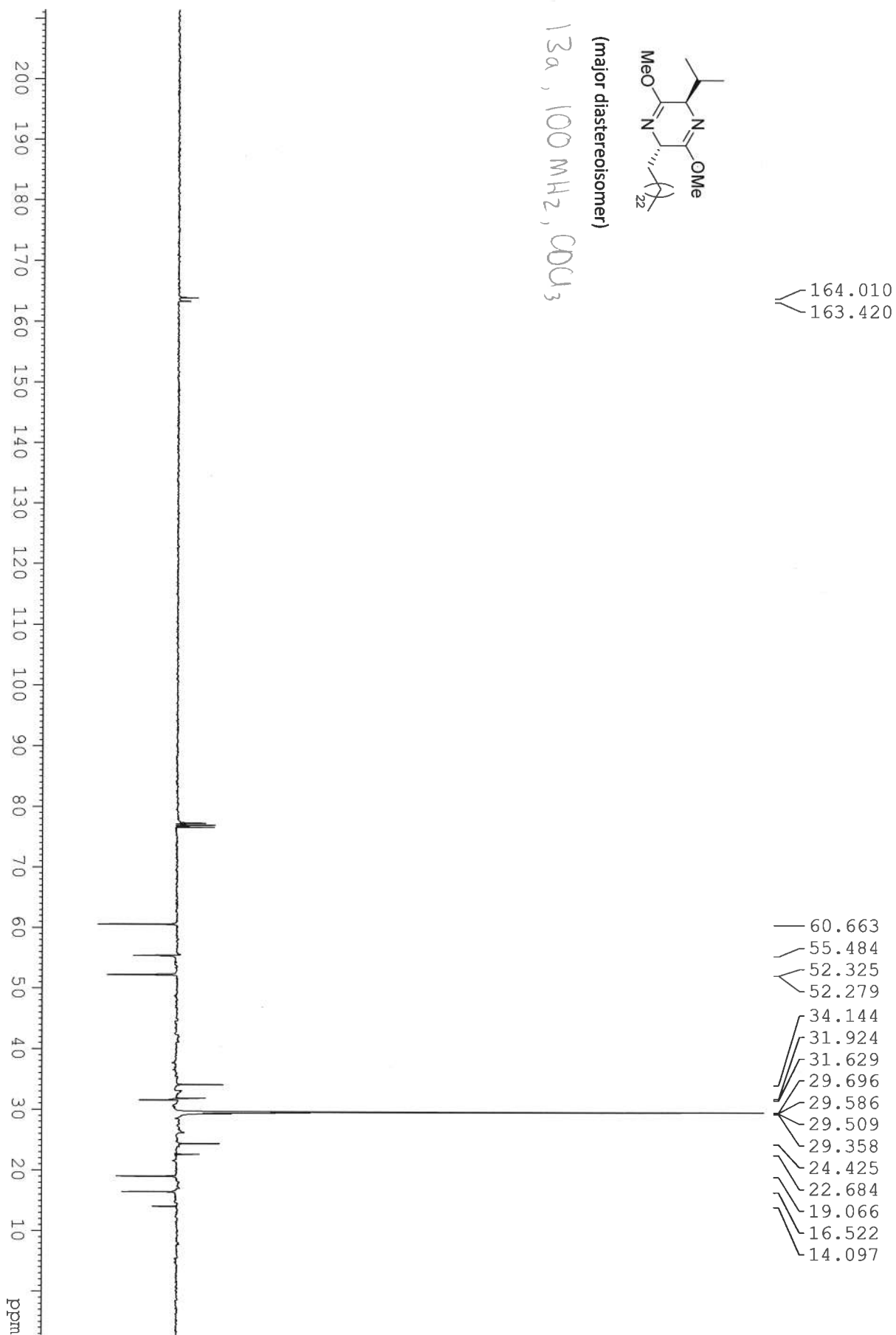
13a, 300 MHz, CDCl₃

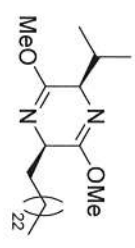




(major diastereoisomer)

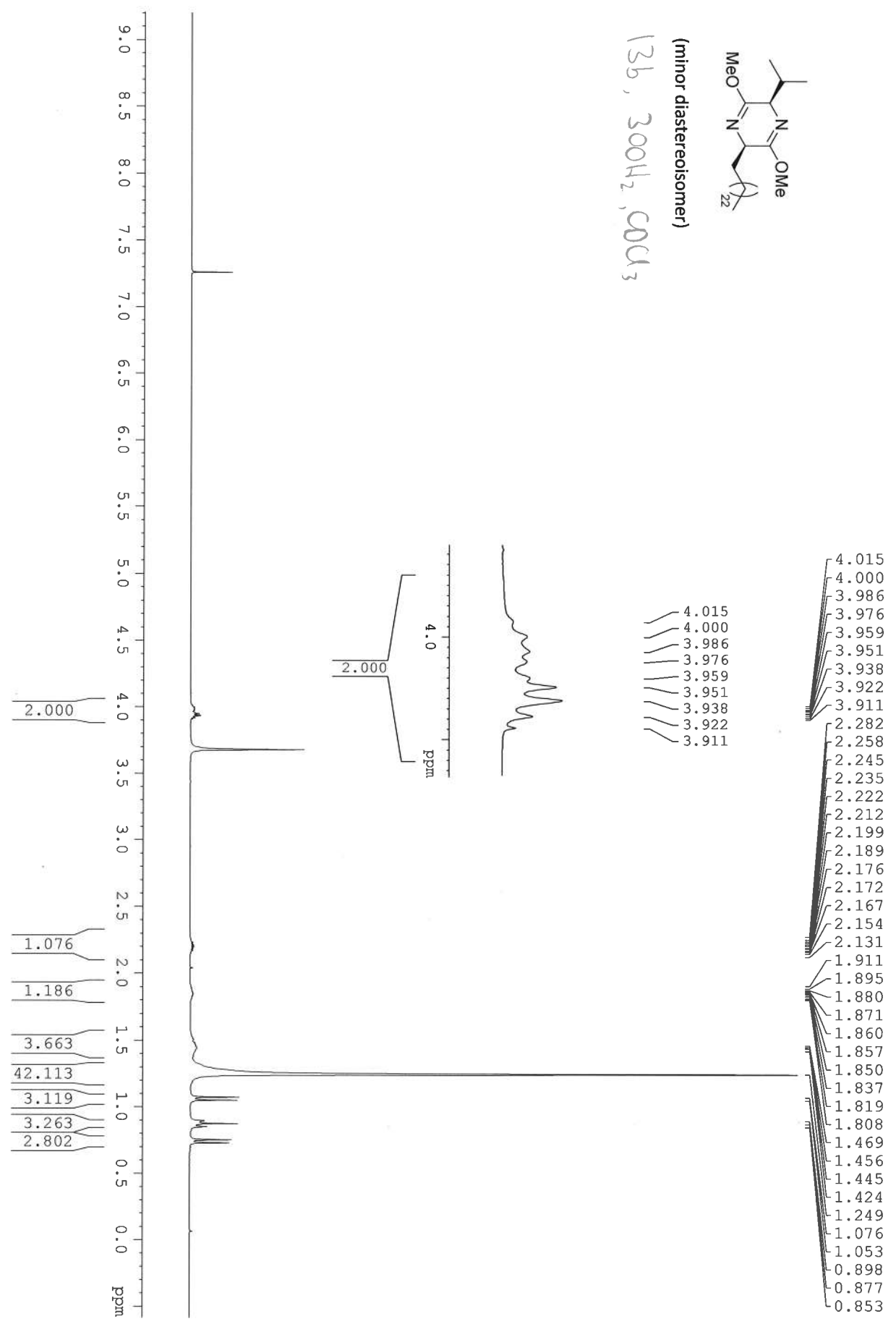
13a, 100 MHz, CDCl₃

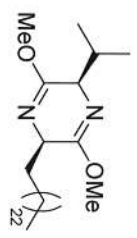




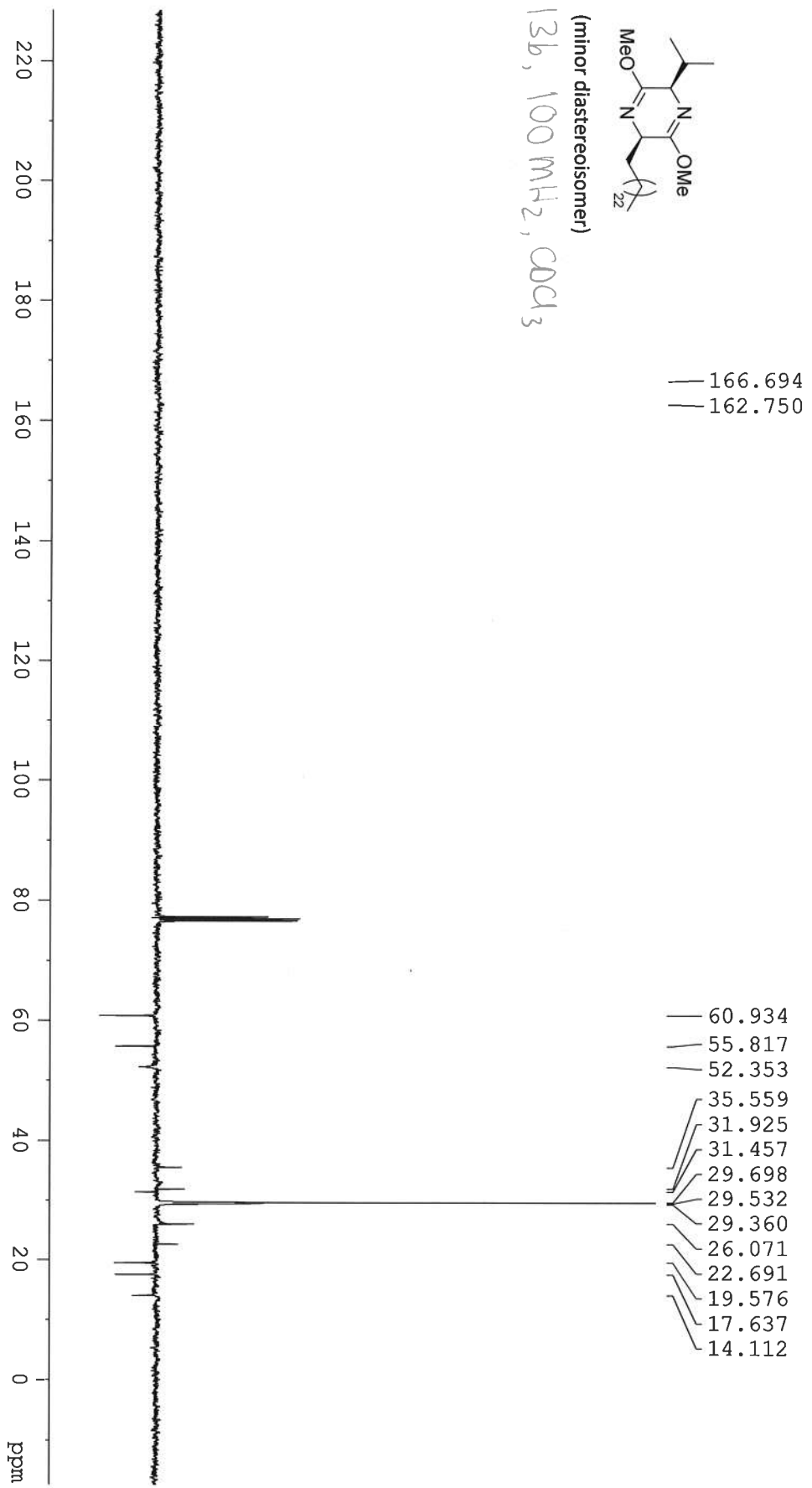
(minor diastereoisomer)

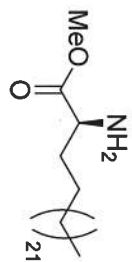
136, 300K, CDCl₃



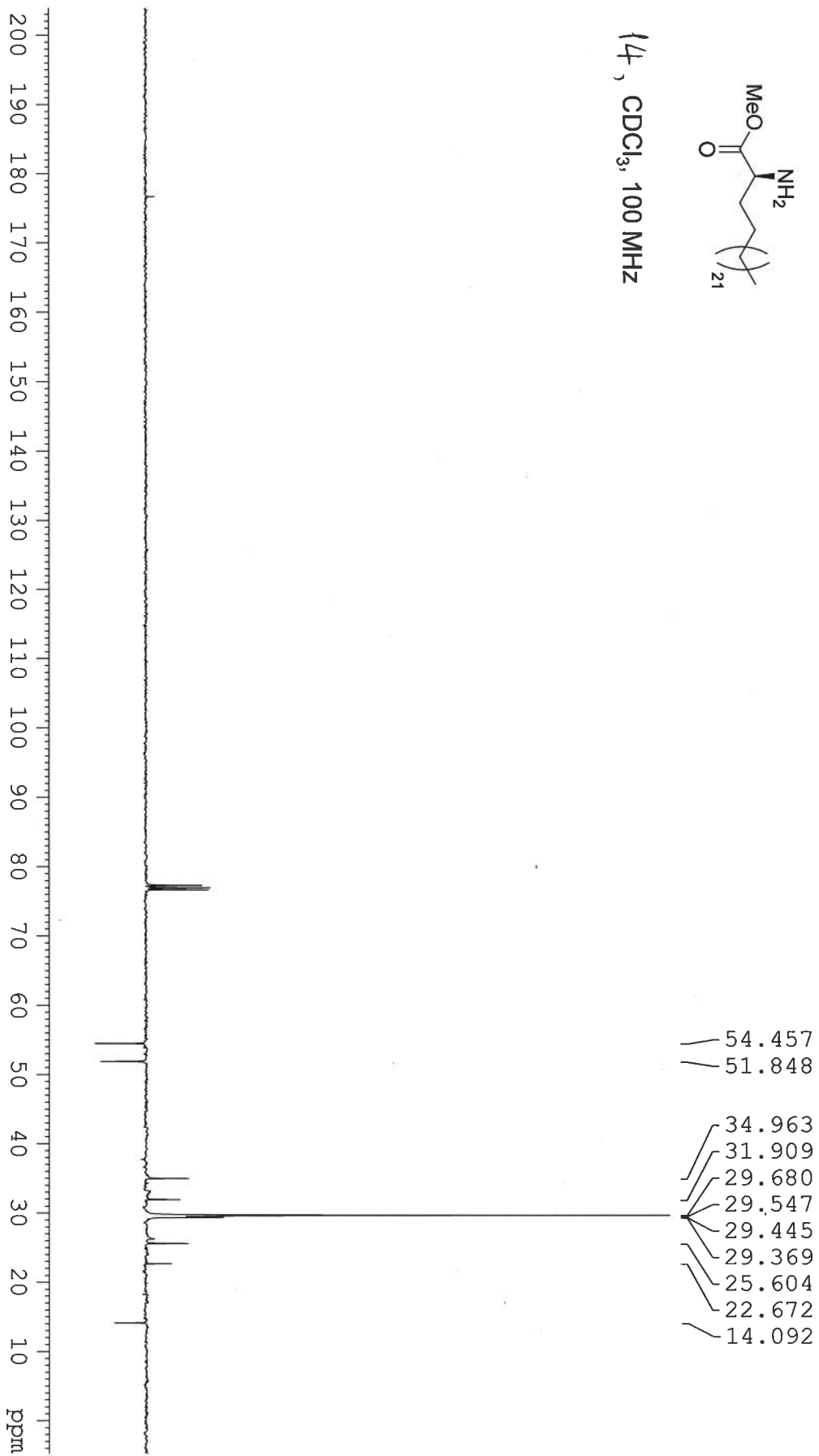


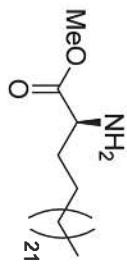
(minor diastereoisomer)
13b, 100 MHz, CDCl₃



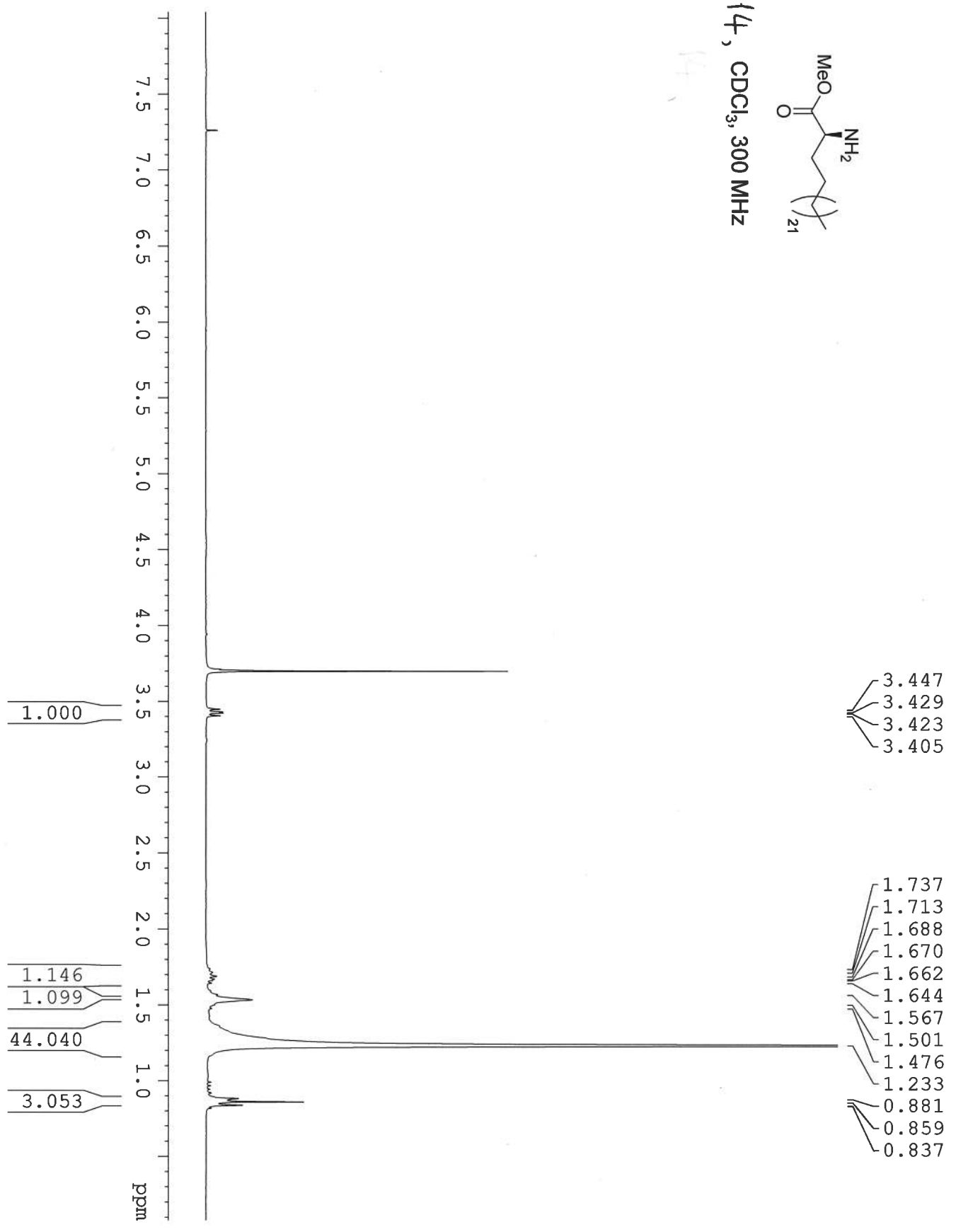


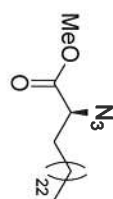
14, CDCl₃, 100 MHz



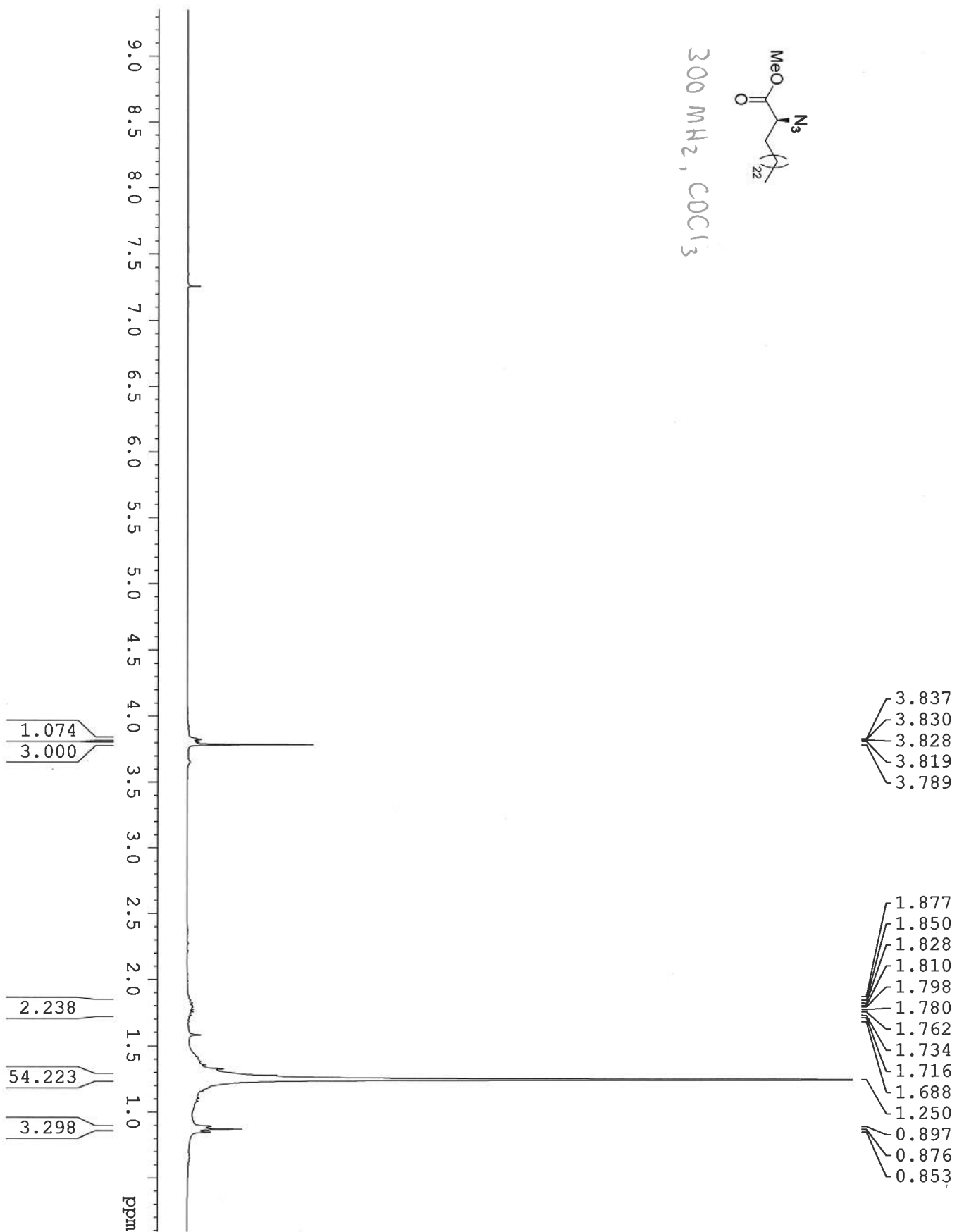


14, CDCl₃, 300 MHz

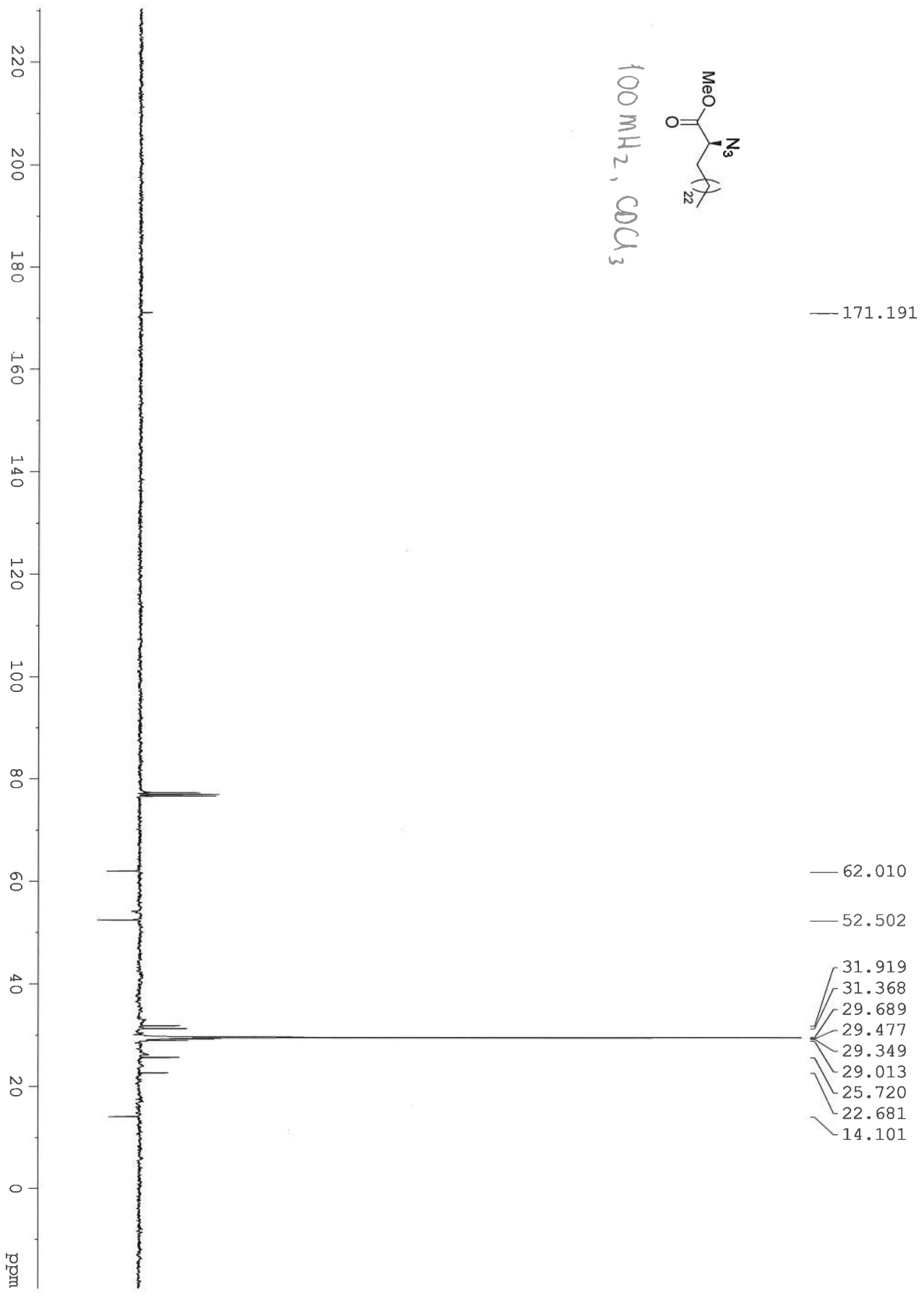
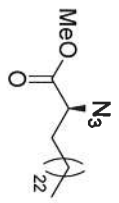




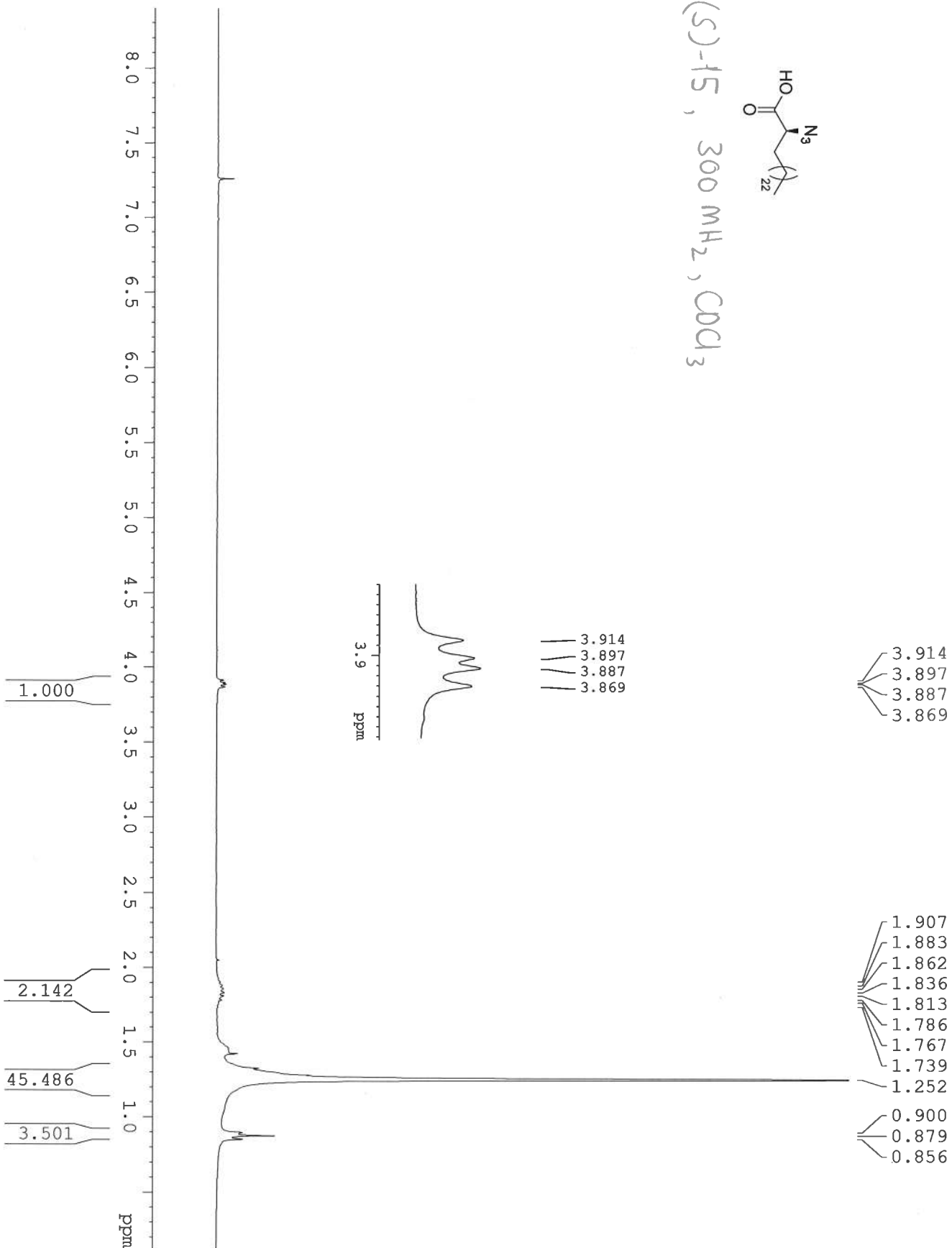
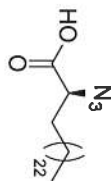
300 MHz, CDCl₃



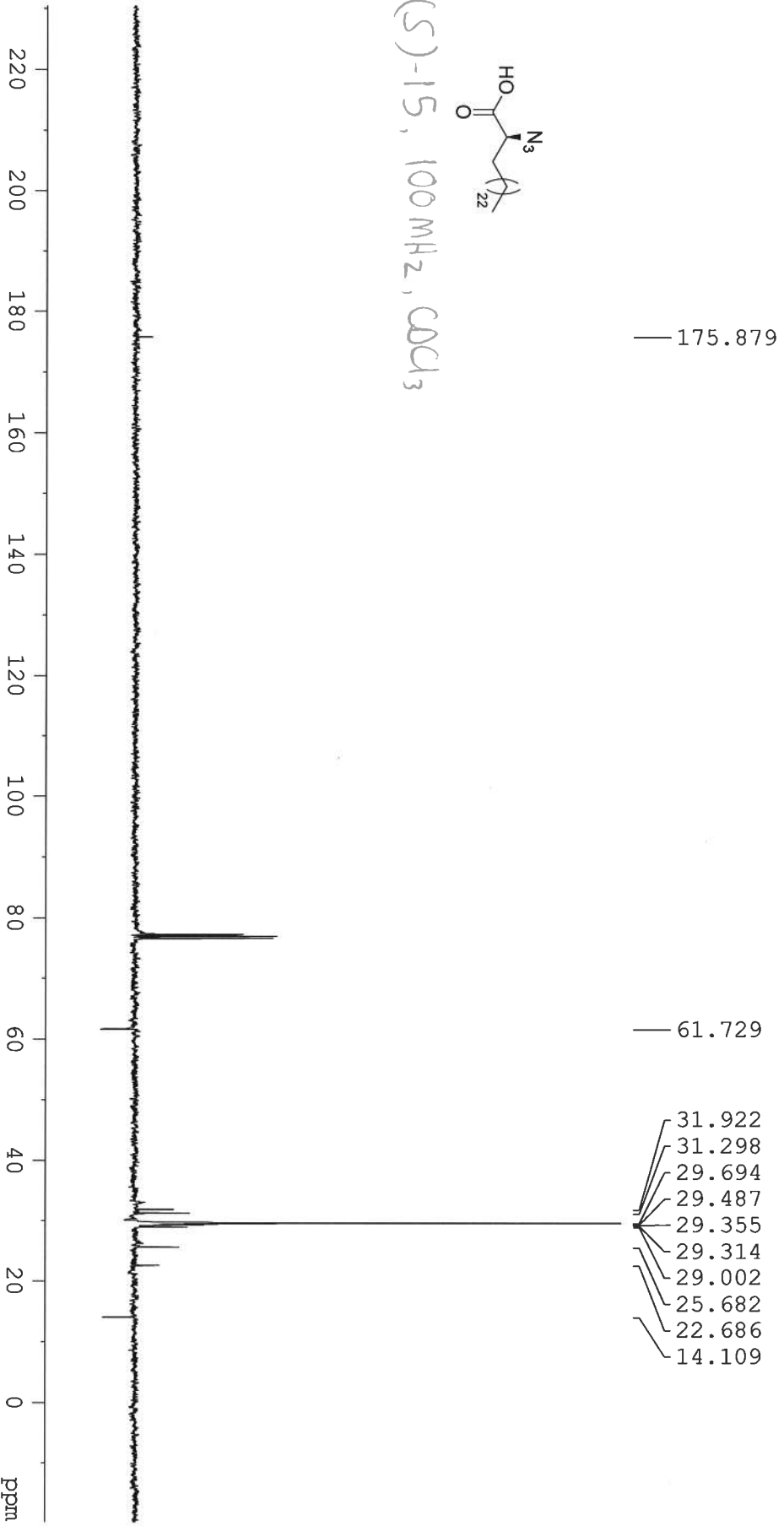
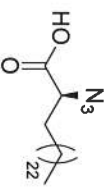
100 MHz, CDCl₃



(S)-15, 300 MHz, CDCl₃

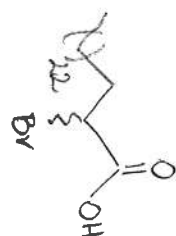


(S)-15, 100 MHz, CDCl₃



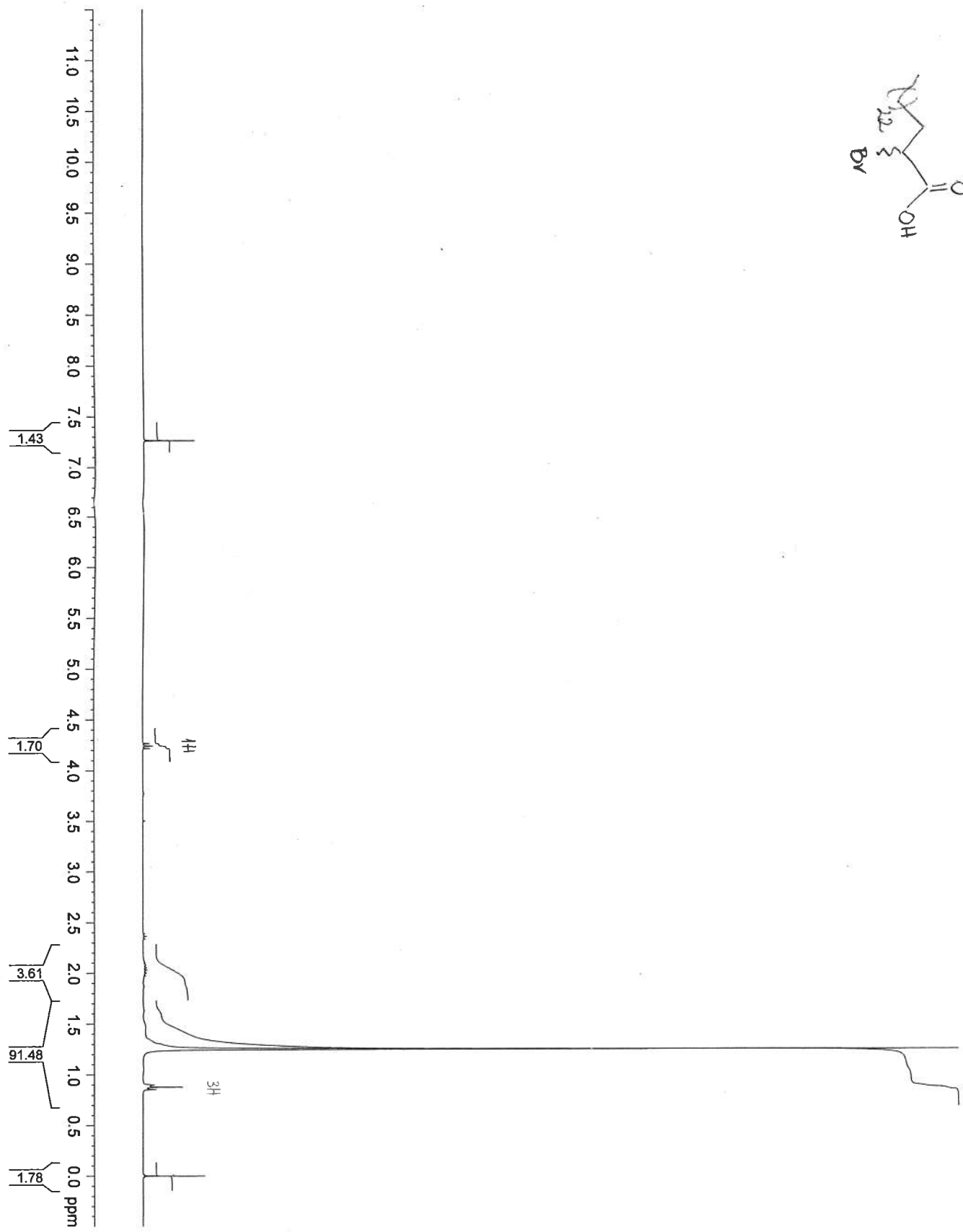
JN3-31

Justy Wojno - C26 alfa bromination



NAME 06-03-Besra-26
EXPNO 10
PROCNO 1
Date_ 20090603
Time_ 16.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 32
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 256
DWM 83.200 usec
DE 13.19 usec
TE 283.2 K
D1 1.00000000 sec
TD0 1

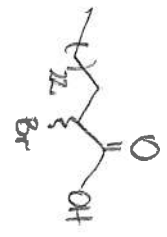
===== CHANNEL f1 =====
NUC1 1H
P1 12.80 usec
PL1 1.00 dB
PL1W 9.57725906 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300057 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



JN3-31

Justyna Wojno, CDCI3, +27C, AV400
13C-PENDANT, 1H-decoupled
Barcode label 1277

*Mon on est
d'origine
Nully*

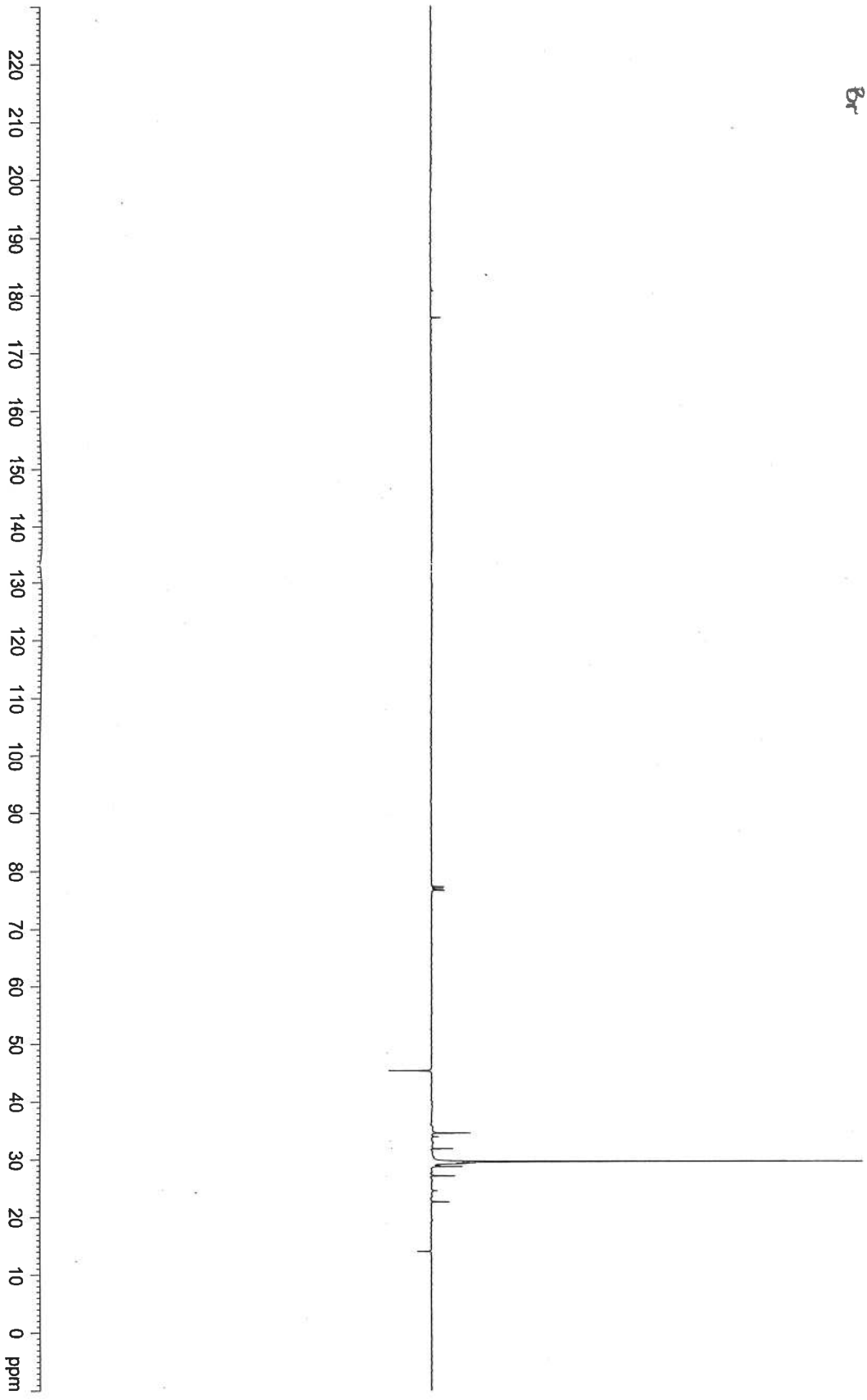


NAME 06-03 5.FAC
 EXPNO 10
 PROCNO 1
 Date 20090603
 Time 17.11
 INSTRUM av400
 PROBHD 5 mm BBO BB-1H
 PULPROG gpcpradms
 TD 65536
 SOLVENT CDCI3
 NS 512
 DS 16
 SWH 25125.629 Hz
 FIDRES 0.383387 Hz
 AQC 1.3042164 sec
 RG 16384
 DW 19.900 usec
 DE 6.50 usec
 TE 301.1 K
 CNST2 145.0000000
 CNST3 1.0000000
 CNST4 5.0000000
 D1 1.500000000 sec
 D2 0.00172414 sec
 D3 0.00431034 sec
 D12 0.00002000 sec
 D13 0.00000400 sec
 TD0 1

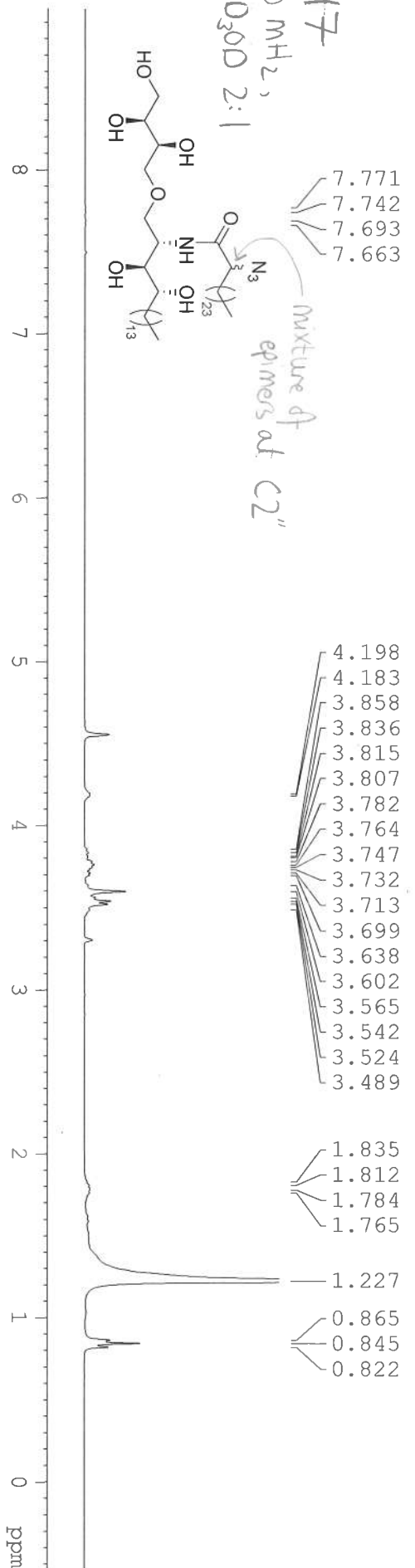
===== CHANNEL f1 =====
 NUC1 13C
 P1 10.60 usec
 P2 21.20 usec
 PL1 6.00 dB
 SFO1 100.6086820 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P3 10.50 usec
 P4 21.00 usec
 PCPD2 100.00 usec
 PL2 2.00 dB
 PL12 23.20 dB
 SF02 400.0723000 MHz
 SI 65536
 SF 100.5976820 MHz
 W/DW EM
 SSB 0
 LB 5.00 Hz
 GB 0
 PC 1.00

29.7
29.5



(R/S)-17
 300 MHz,
 CDCl₃/CD₃OD 2:1



(S)-17
 300 MHz,
 CDCl₃/CD₃OD
 2:1

