

Supporting Information
for
Preparation of mixed trialkyl alkylcarbonate derivatives of
etidronic acid via an unusual route

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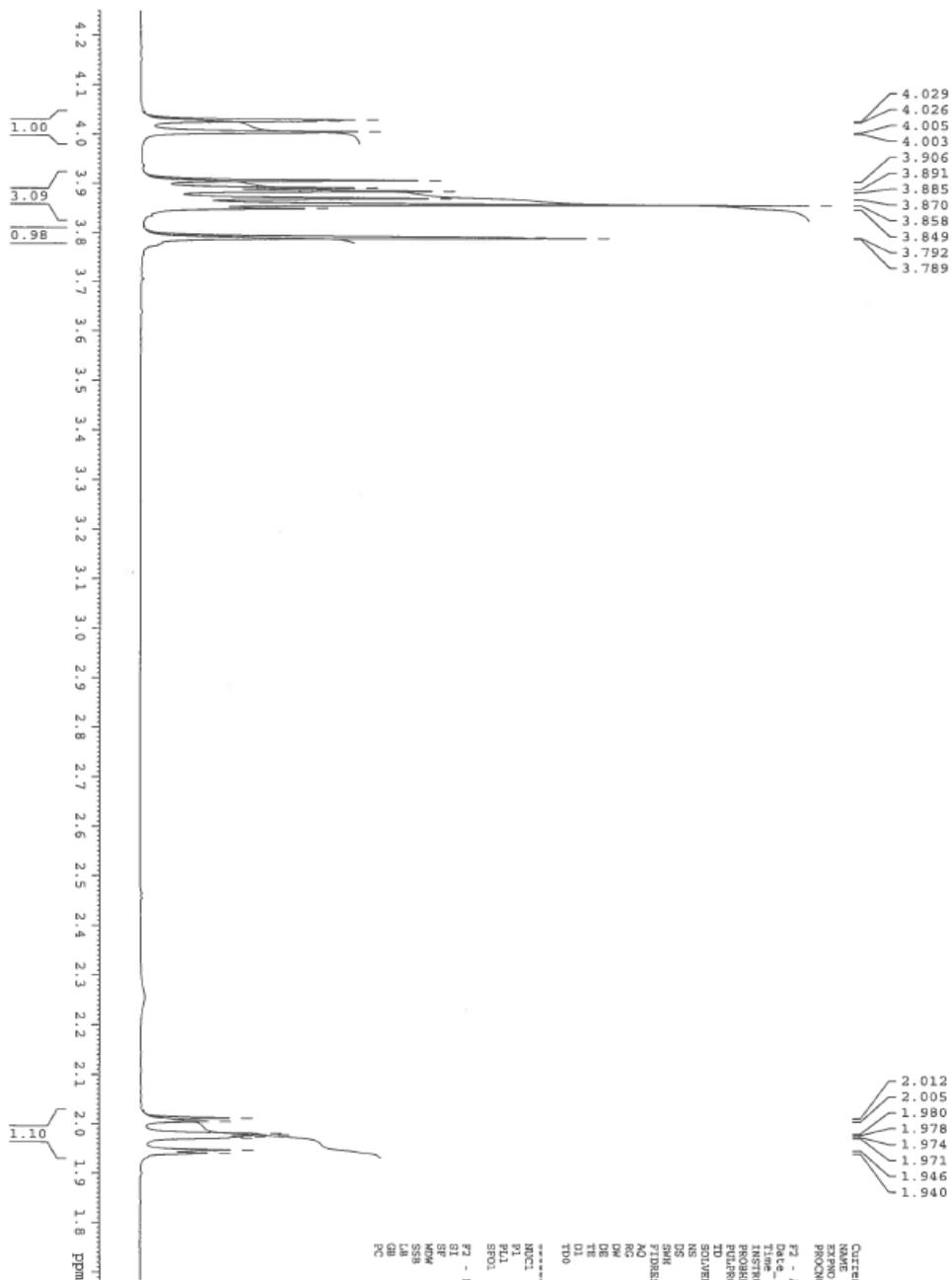
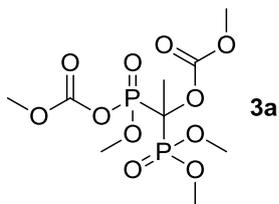
*Corresponding author

¹H, ¹³C and ³¹P NMR spectra for the compounds **2, **3a–d** and **4****

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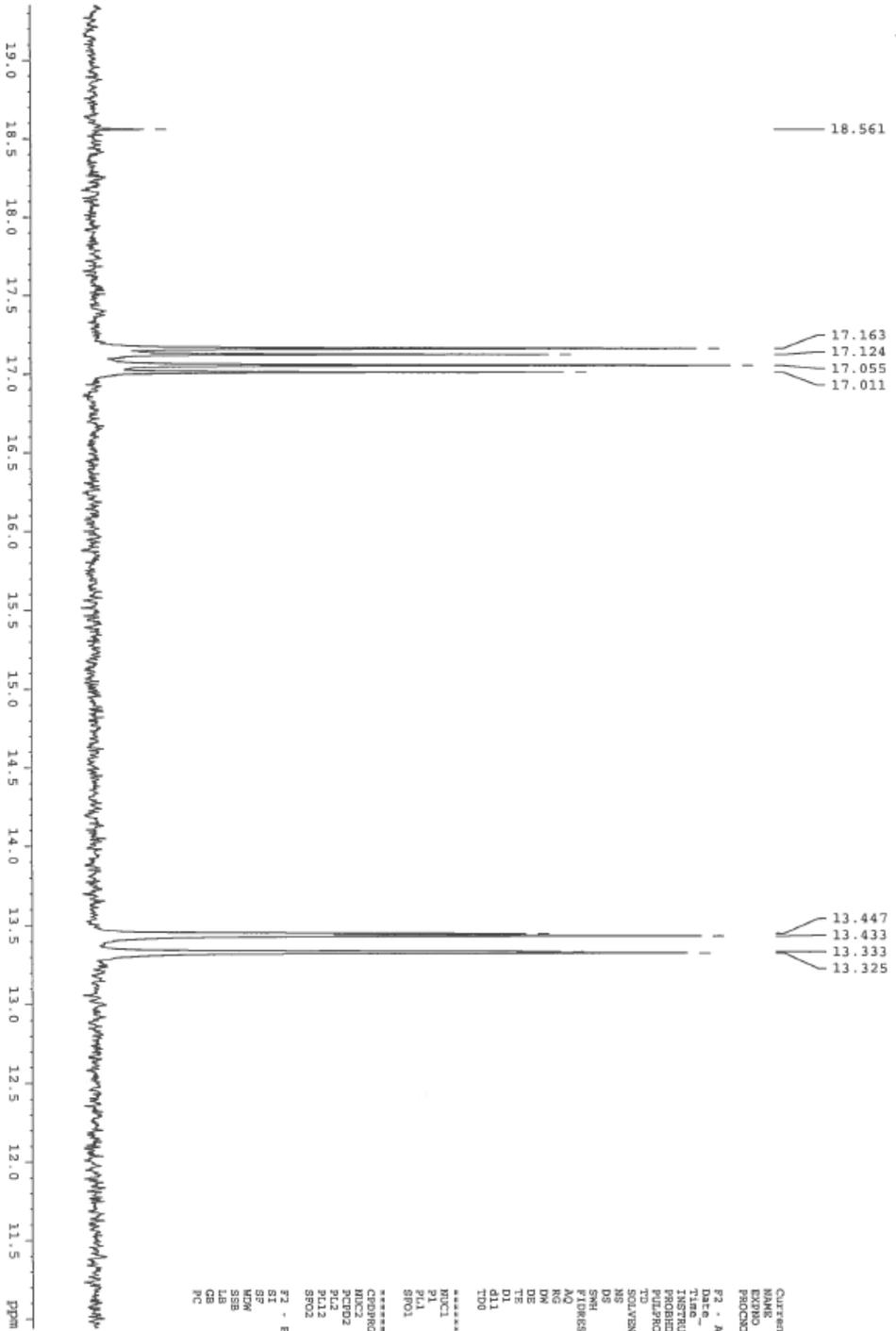
PT/MS-160810-2a cdcl3



Current Data Parameters
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 PULPROG zgpg30
 TD 65536
 SFO 500.1360010 MHz
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 10000.000 Hz
 FWHM 32.520 Hz
 AQ 3.2769001 sec
 RG 64
 DW 50.000 usec
 DE 6.000 usec
 TE 300.2 K
 D1 1.50000000 sec
 TDO 1

----- CHANNEL f1 -----
 NUC1 31P
 P1 9.36 usec
 PL1 -1.00 dB
 SFO1 500.1360010 MHz
 F2 - Processing parameters
 SI 262144
 SF 506.1300137 MHz
 NDM EN
 SSB 0.30 Hz
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PT/MS-160810-2a cdcl3



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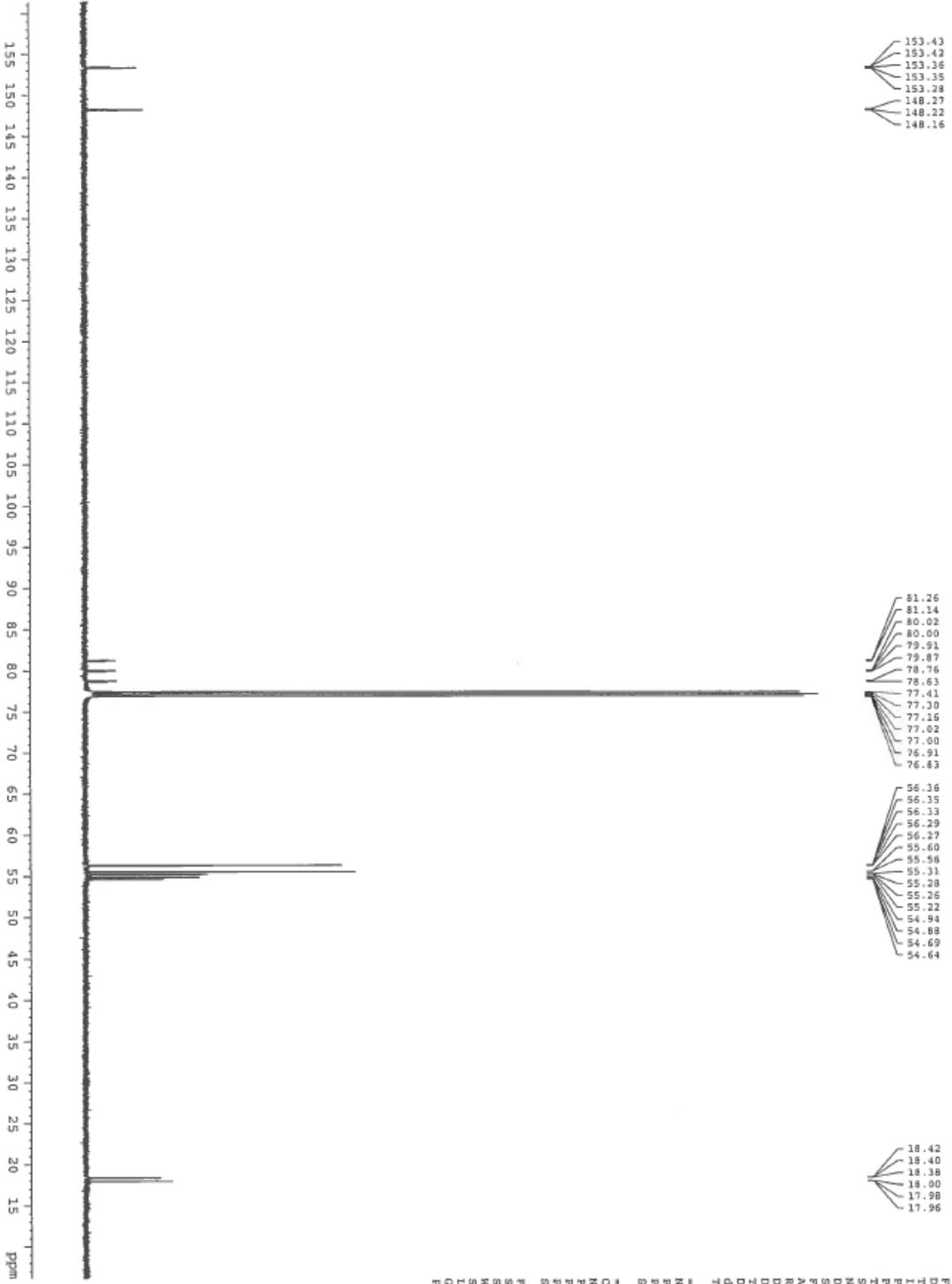
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SOLVENT  CDCl3
NS       20
DS       0
SWH      40650.406 Hz
FIDRES   0.830229 Hz
AQ       0.889228 sec
RG        2048
AQ       12.300 usec
DE        6.00 usec
TE        300.0 K
D1        2.0000000 sec
d11       0.0300000 sec
TD0       1

***** CHANNEL f1 *****
NUC1      13C
P1        8.00 usec
PL1       0.00 dB
SFO1     202.458930 MHz

***** CHANNEL f2 *****
CPDPRG2  WALTZ16
NUC2      1H
P2        90.00 usec
PL2       -1.00 dB
PULPROG  zgpg30
SFO2     500.136000 MHz

F2 - Processing parameters
SI        524288
SF        202.458930 MHz
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SSB       0
GB        0
PC        1.40
    
```

PT/MS-160810-2a cdcl3



153.43
153.42
153.36
153.35
153.28
148.27
148.22
148.19

81.26
81.14
80.02
80.00
79.91
79.87
78.76
78.63
77.41
77.30
77.16
77.02
77.00
76.91
76.83

56.36
56.35
56.33
56.29
56.27
55.60
55.58
55.31
55.28
55.26
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54.92
54.69
54.64

18.42
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18.38
18.35
17.98
17.96

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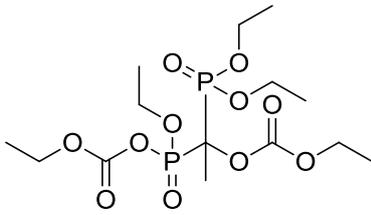
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PULPROG  zgpg30
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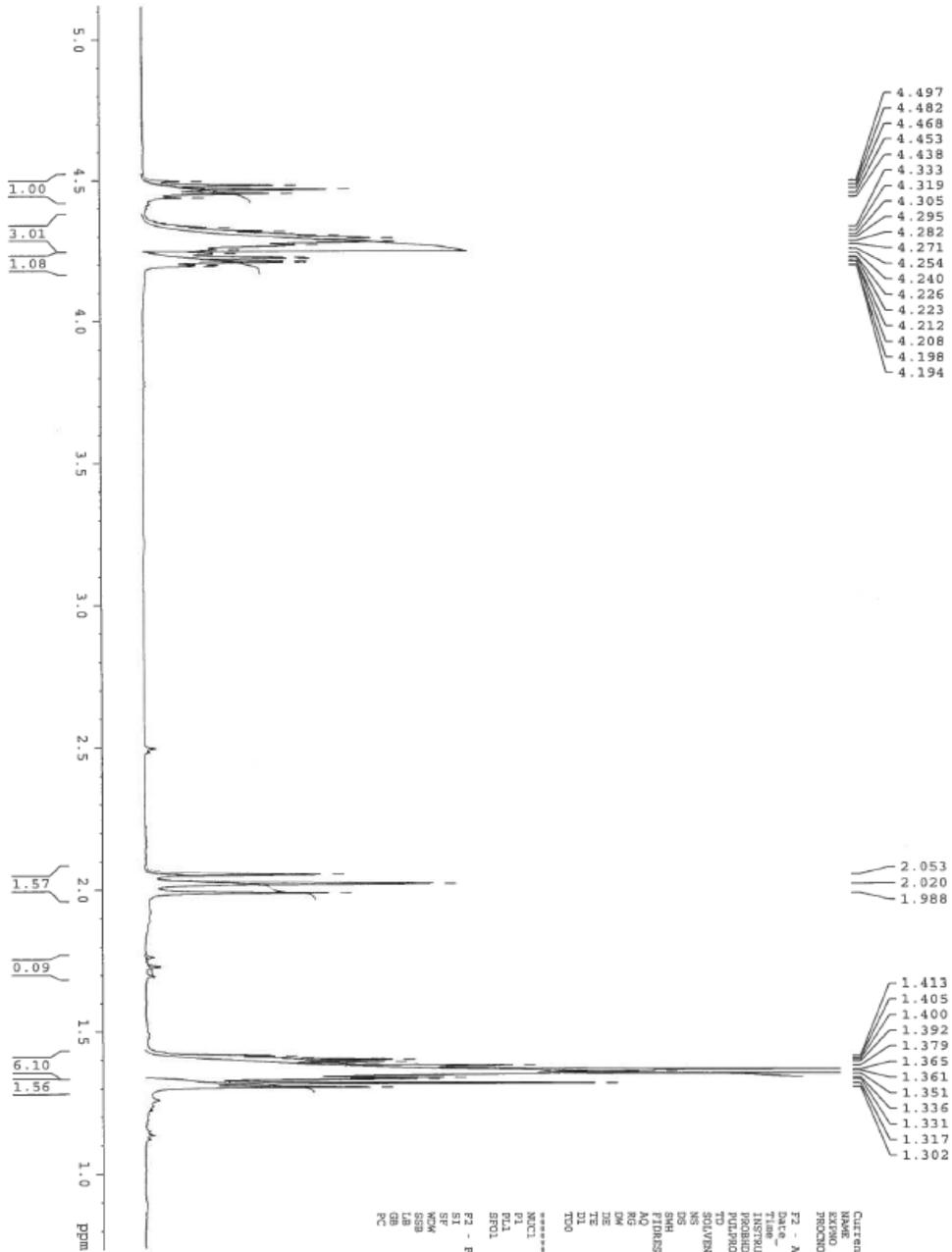
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NUC1      13C
P1        7.75 usec
PL1       6.00 dB
SFO1      125.7715724 MHz

***** CHANNEL f2 *****
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NUC2      13C
PCPD2     90.00 usec
PL2       -1.00 dB
PL12      18.56 dB
SFO2      500.1322566 MHz

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GB        0
PC        0.50
    
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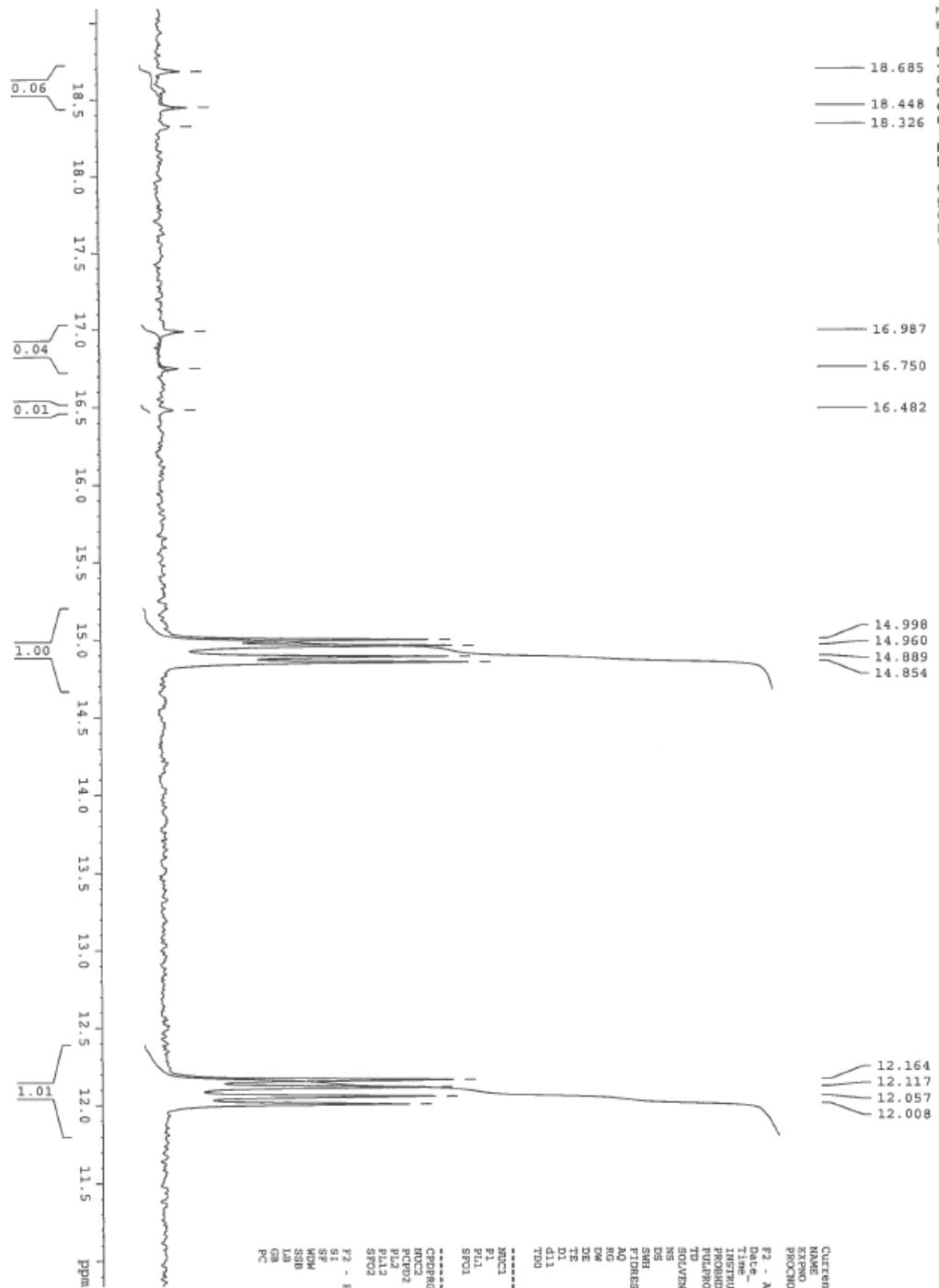


3b



PF-270308-1a cdCl3

PT-270308-1a cdcl3



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Current Data Parameters
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EXPNO    1
PROCNO   1

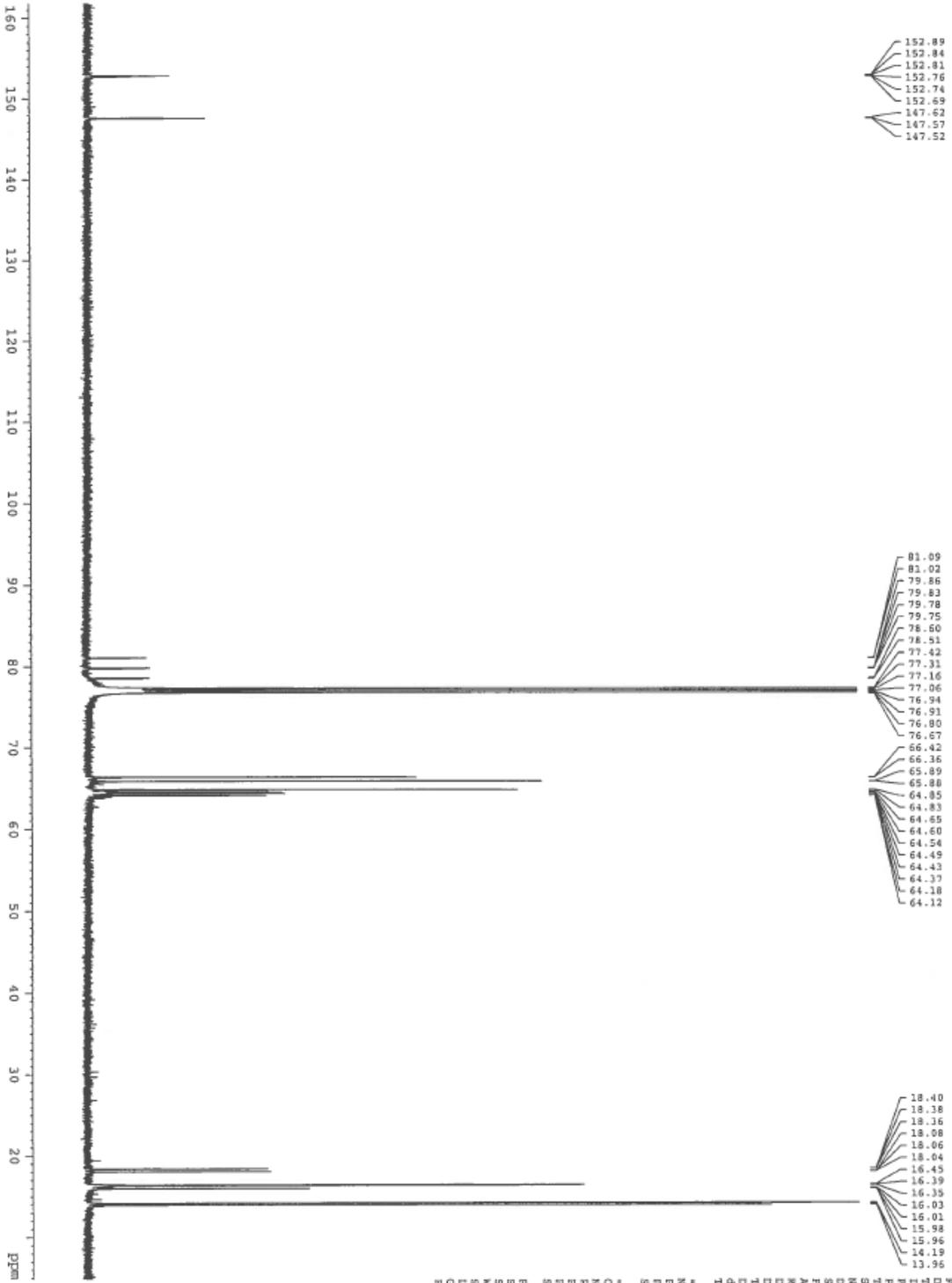
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PULPROG  zgpg30
TD        32768
SOLVENT  CDCl3
NS        16
DS        4
SSB       0
AQ        0.250007 Hz
RG        1.9999993 sec
RG        20642.5
DM        6.150 usec
DE        150.0 usec
DI        2.0000000 sec
D11       0.0300000 sec
TD0       1

***** CHANNEL f1 *****
NUC1      13C
P1        8.50 usec
PL1       10.00 dB
SFO1      202.498930 MHz

***** CHANNEL f2 *****
CPDPRG2   waltz16
NUC2      1H
P2        90.00 usec
PL2       12.00 dB
SFO2      500.135007 MHz

F2 - Processing parameters
SI        262244
SF        202.498930 MHz
RG        1.9999993 sec
DE        6.150 usec
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PT-270308-1a cdcl3



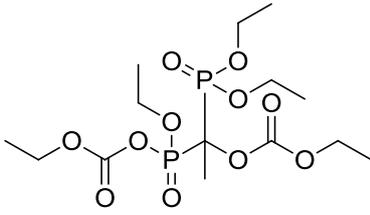
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 METHOD 1
 PROCNM 1

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 INSTRUM spect
 PULPROG zgpg30
 TD 131072
 SOLVENT CDCl3
 NS 3310
 DS 0
 SWH 3090.031 Hz
 FWHM 0.14144 Hz
 AQ 2.162756 sec
 RG 8192
 DM 16.500 usec
 DE 6.00 usec
 TE 300.0 K
 D1 18.00000000 sec
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 TD0 1

***** CHANNEL f1 *****
 NUCL1 13C
 P1 7.75 usec
 PL1 6.00 dB
 SFO1 125.7715724 MHz

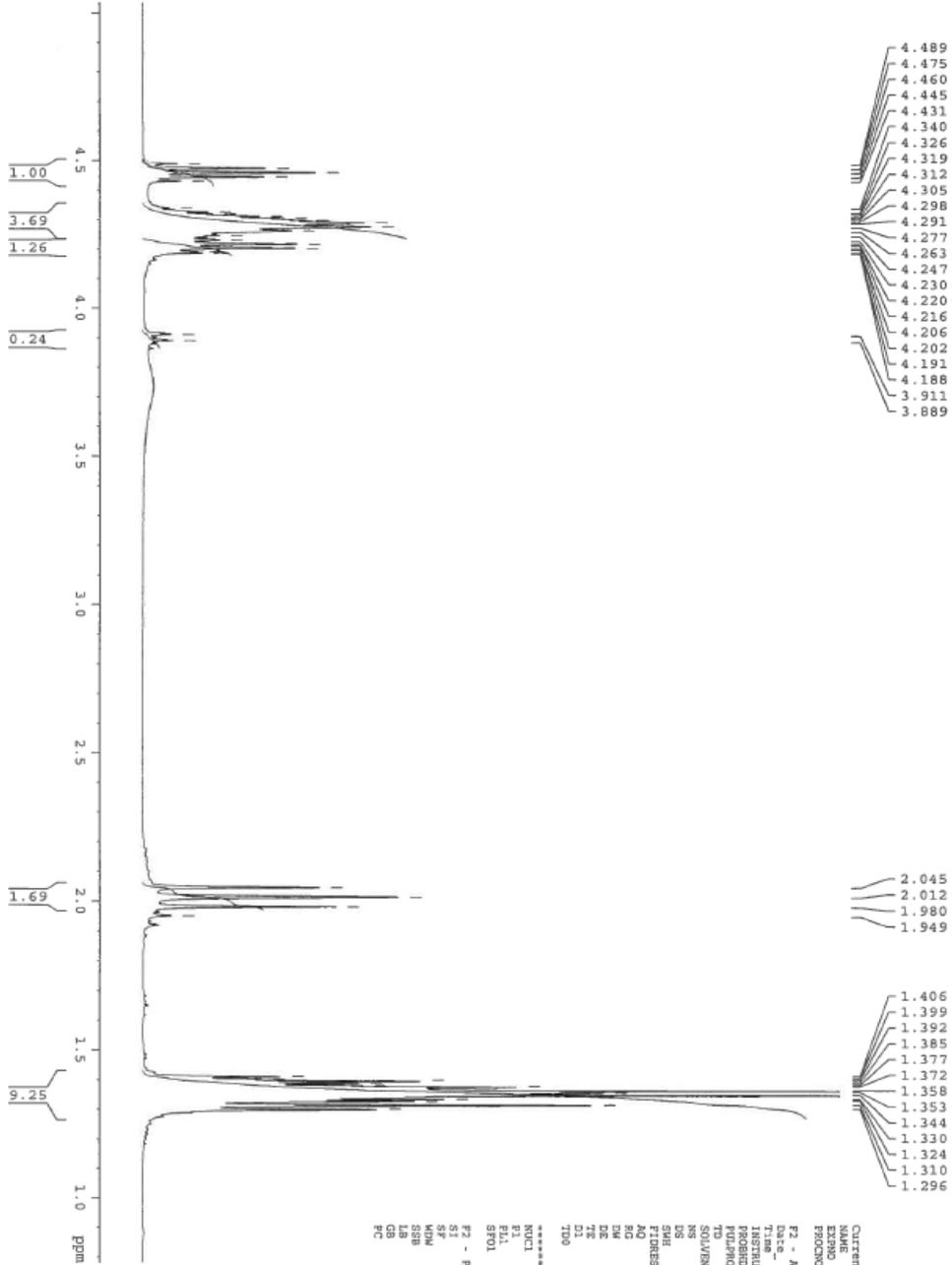
***** CHANNEL f2 *****
 CPROBPG2 waltz16
 NUCL2 1H
 PCPD2 90.00 usec
 PL2 -2.00 dB
 PL12 19.00 dB
 SFO2 500.1322506 MHz

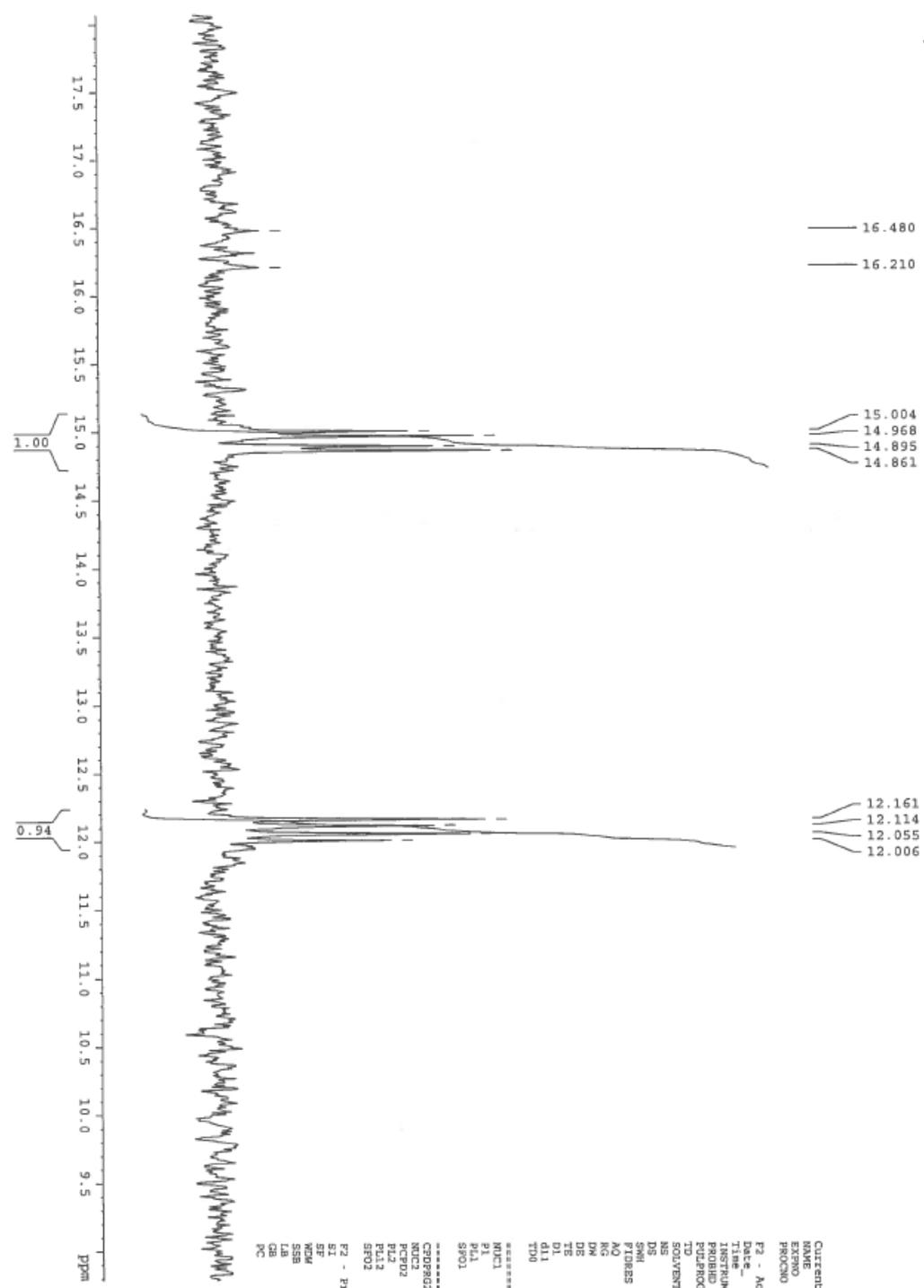
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 SF 125.7715724 MHz
 MDW 0
 SSB 0
 LB 0.60 Hz
 GB 0
 PC 0.10



3b

PT/MS-180810-2 cdCl3





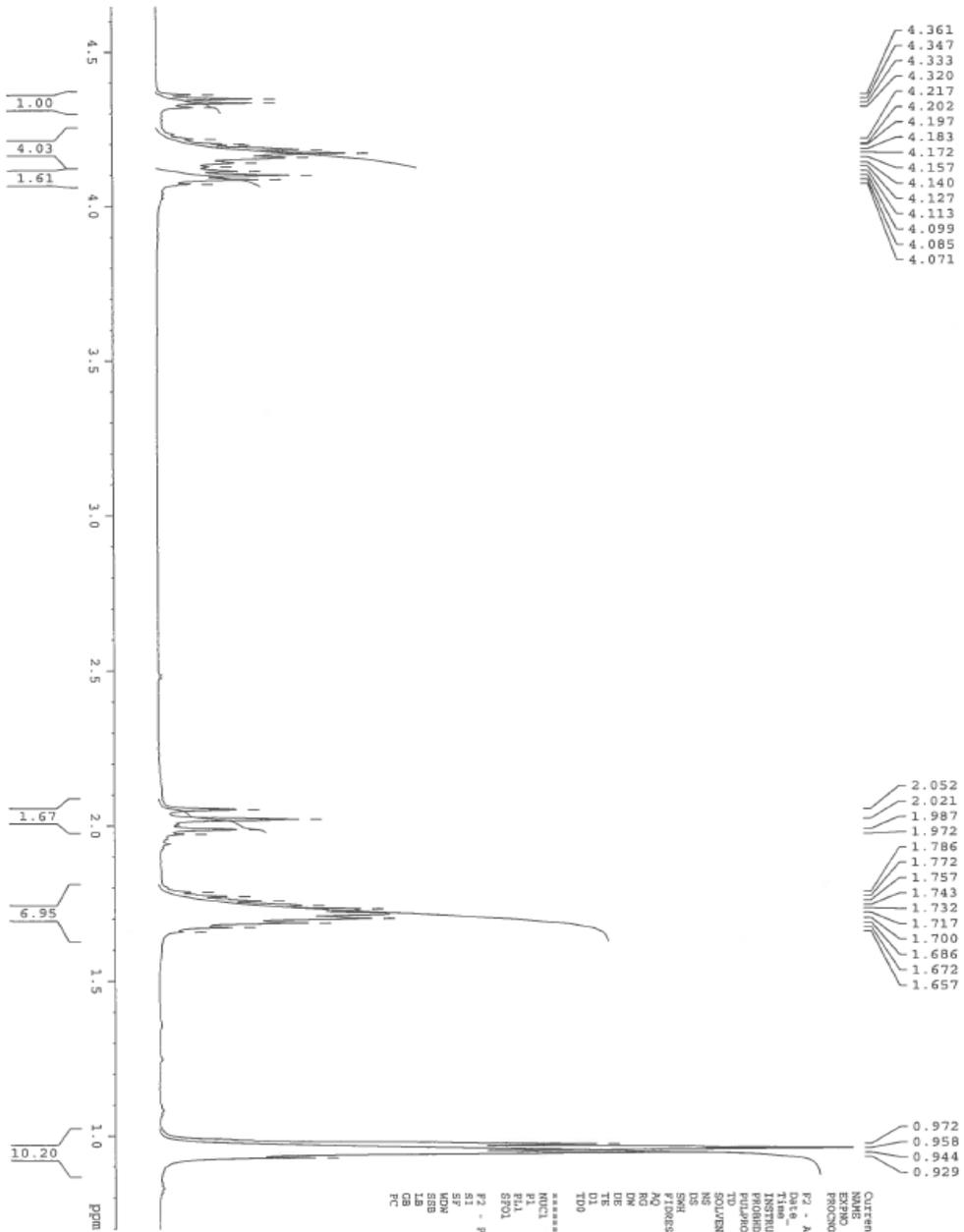
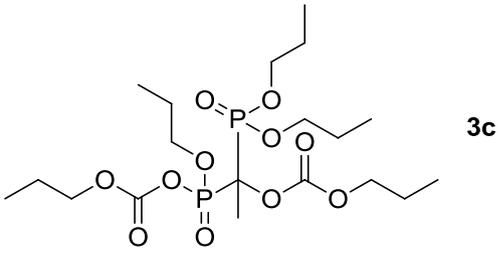
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 EXPTNO 1
 PROCNO 1

F2 - Acquisition Parameters
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 Time 12.03
 INSTRUM spect
 PROBRD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 55
 DS 0
 SWH 40650.405 Hz
 FIDRES 0.620276 Hz
 AQ 0.18087263 sec
 RG 2048
 DN 12.300 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000001
 TDO 1

***** CHANNEL F1 *****
 NUC1 31P
 P1 8.50 usec
 PL1 18.00 dB
 SFO1 202.4566930 MHz

***** CHANNEL F2 *****
 CPDPRG2 WALTZ16
 VPP2 90.00 usec
 PCPD2 -1.00 dB
 PL2 18.56 dB
 PL12 500.1325007 MHz
 SFO2

F2 - Processing Parameters
 SI 32768
 SF 202.4563481 MHz
 WDM EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40



PT/MS-290212-1 cdcl3

4.361
4.347
4.333
4.320
4.217
4.202
4.197
4.183
4.172
4.157
4.140
4.127
4.113
4.099
4.085
4.071

2.052
2.021
1.987
1.972
1.786
1.772
1.757
1.743
1.732
1.717
1.700
1.686
1.672
1.657

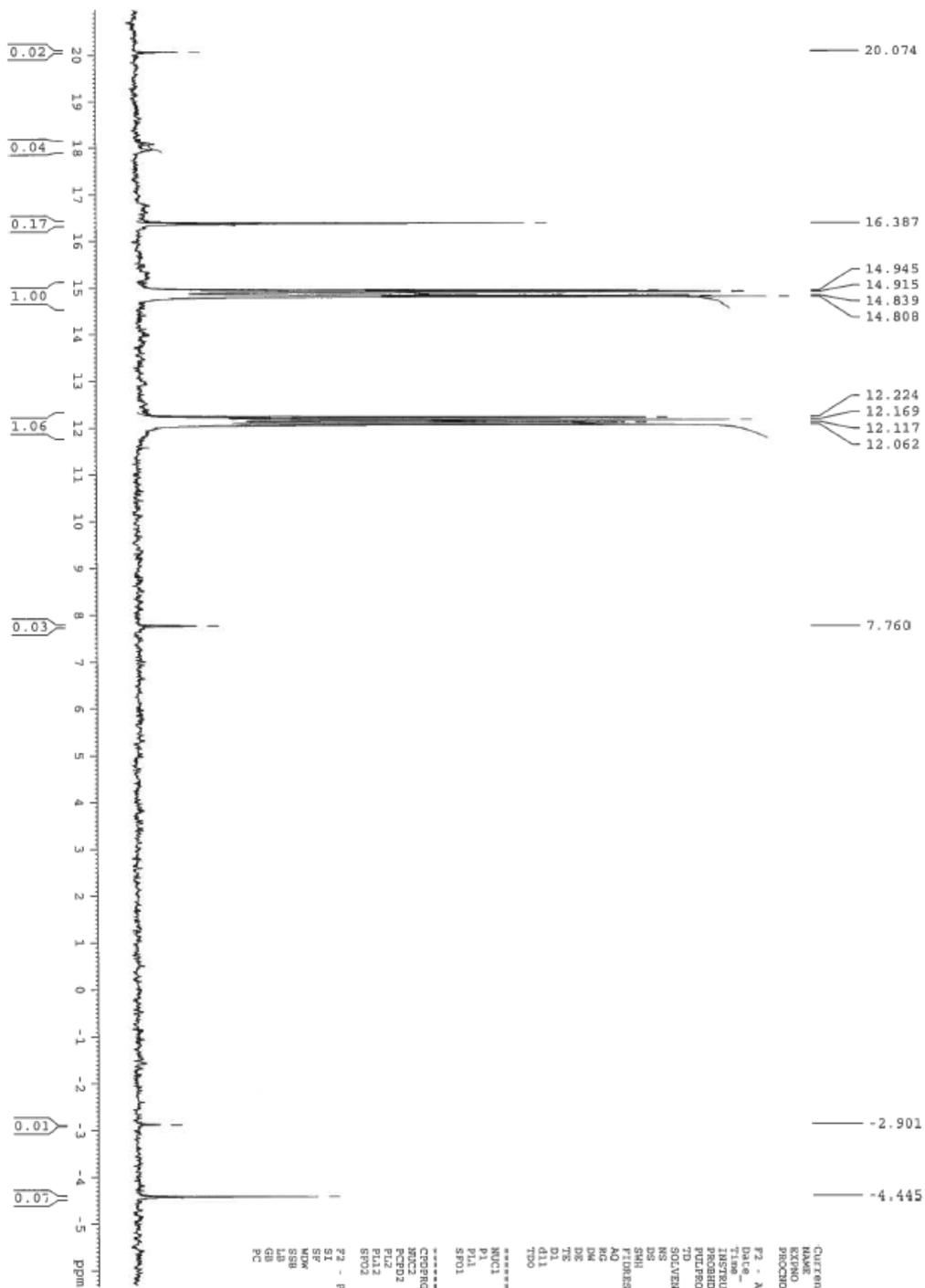
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0.929

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PROCNO 1
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Time 12:22
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2789012 sec
RG 50.000 usec
DE 6.00 usec
TE 297.2 K
D1 1.50000000 sec
D10 1

***** CHANNEL f1 *****
NUC1 1H IR
P1 10.06 usec
PL1 0.00 dB
SFO1 500.1340010 MHz

F2 - Processing parameters
SI 32768
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 5.00

PT/MS-290212-1 cdcl3



Current Data Parameters
 NAME MS-290212-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120301
 Time 12.19
 INSTRUM spect
 PROBRD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SFO1 CDCl3
 SOLVENT CDCl3
 NS 16
 DS 0

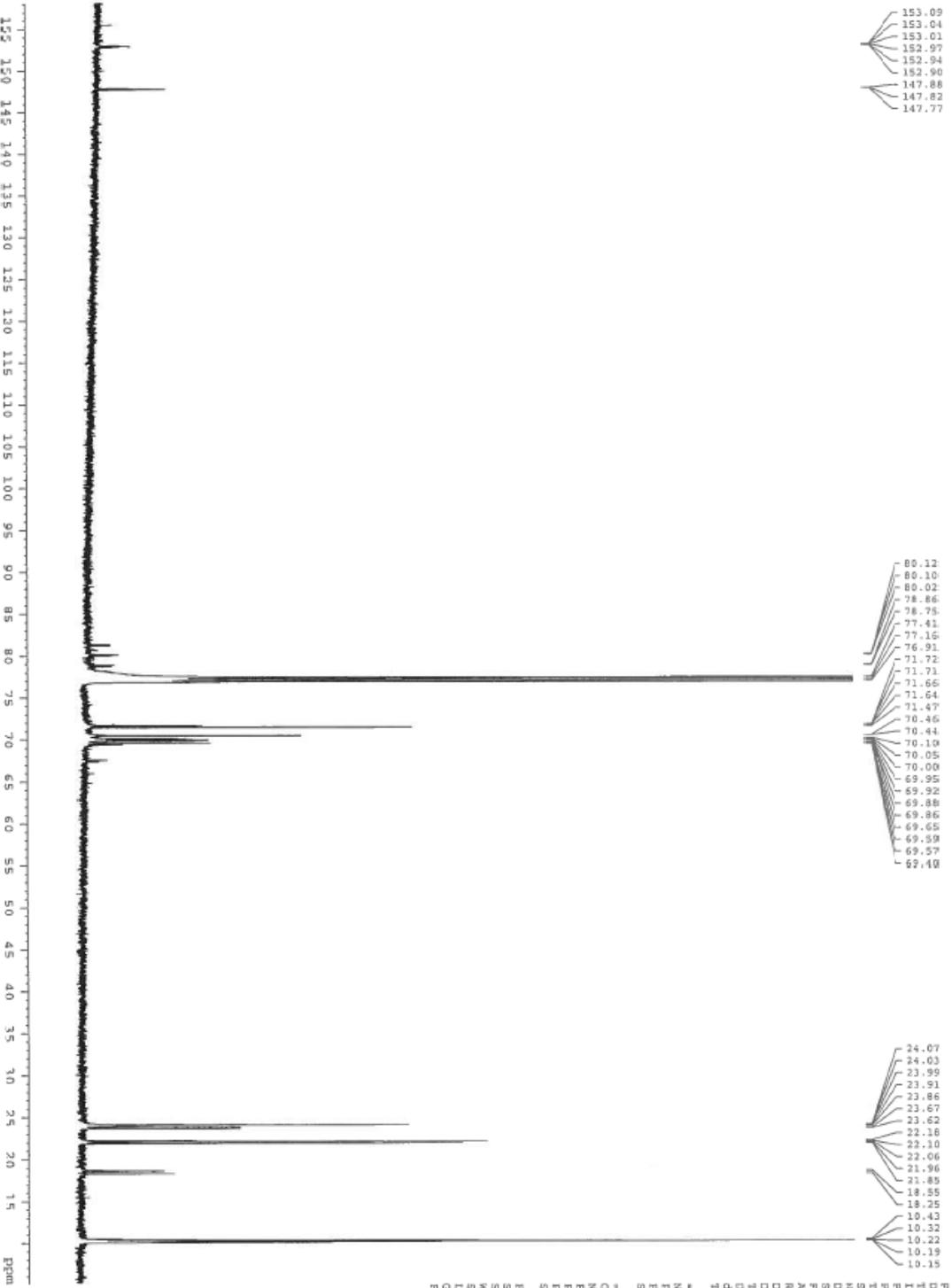
SI 90909.094 Hz
 SH 13.62012 Hz
 F2 13650215 sec
 RG 2048
 DW 5.500 usec
 DE 6.00 usec
 TE 297.15 K
 D11 0.03000000 sec
 TDO 1

***** CHANNEL F1 *****
 NUC1 13C
 P1 8.50 usec
 PL1 10.00 dB
 SFO1 202.458930 MHz

***** CHANNEL F2 *****
 CROPRG2 waltz16
 NUC2 1H
 P2 90.00 usec
 PL2 1.00 dB
 PL12 18.00 dB
 SFO2 500.132607 MHz

F2 - Processing parameters
 SI 131072
 SF 202.459478 MHz
 MD 64
 AS 2
 LSI 2.00 Hz
 GB 0
 PC 1.40

PT/MS-290212-1 cdcl3 tripropyl propylcarbonate BP



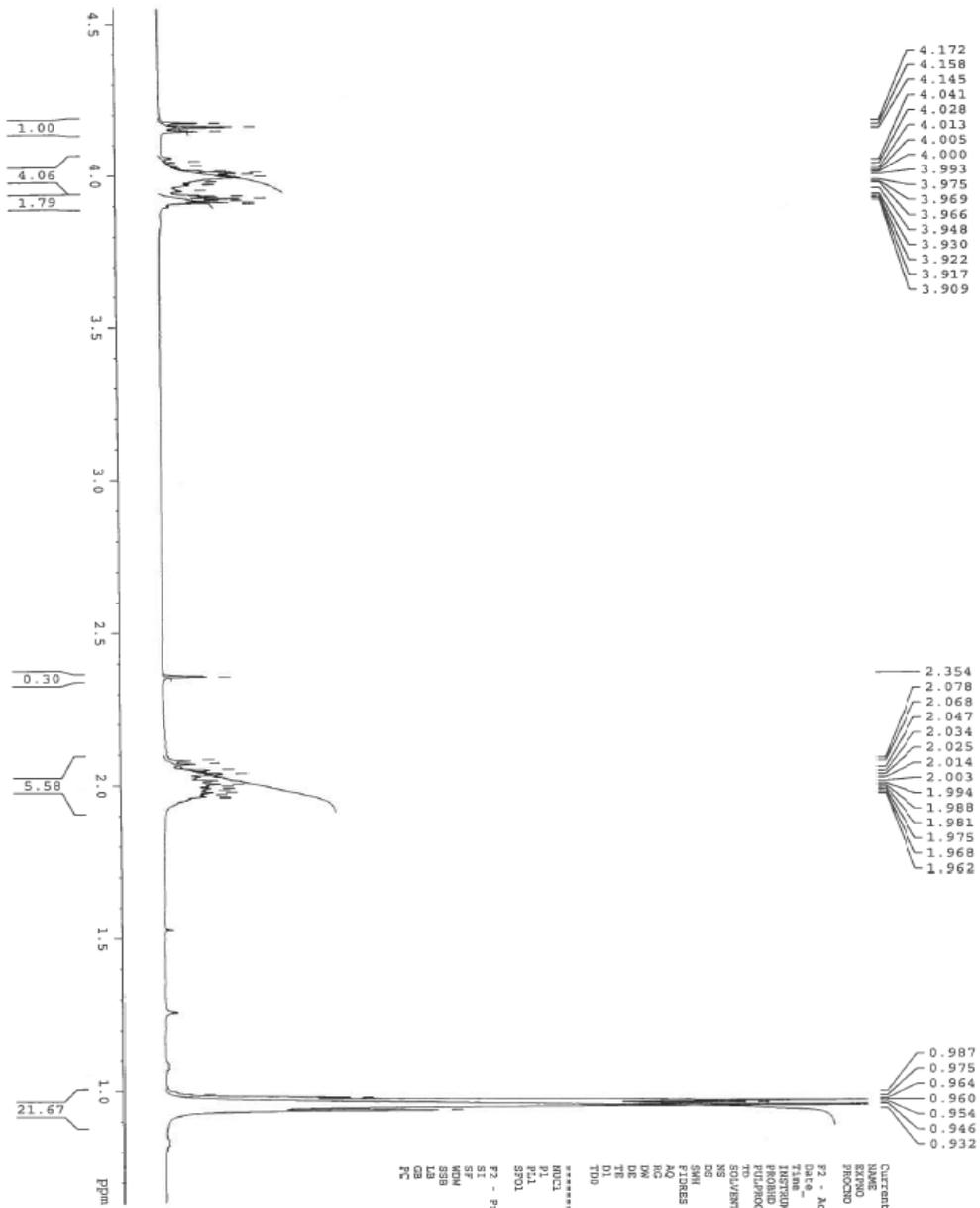
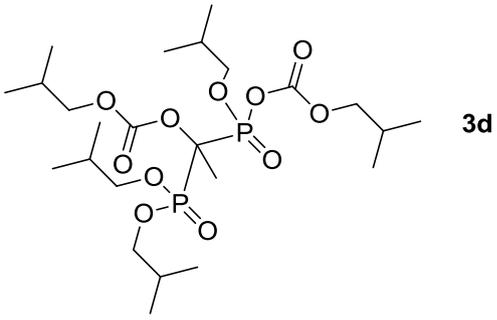
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 EXPNO: 1
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20120215
 Time: 13:01:15
 INSTRUM: spect
 PROBRD: 5 mm QNP 1H/1
 PULPROG: zgpg30
 TD: 131072
 SOLVENT: CDCl3
 DE: 410
 DM: 0
 FIDRES: 30303.031 Hz
 AQ: 0.231194 Hz
 RG: 2.1627948 sec
 AC: 8192
 DM: 16.500 usec
 DE: 3000 Hz
 DS: 12.0000000 sec
 D11: 0.03000000 sec
 TDO: 1

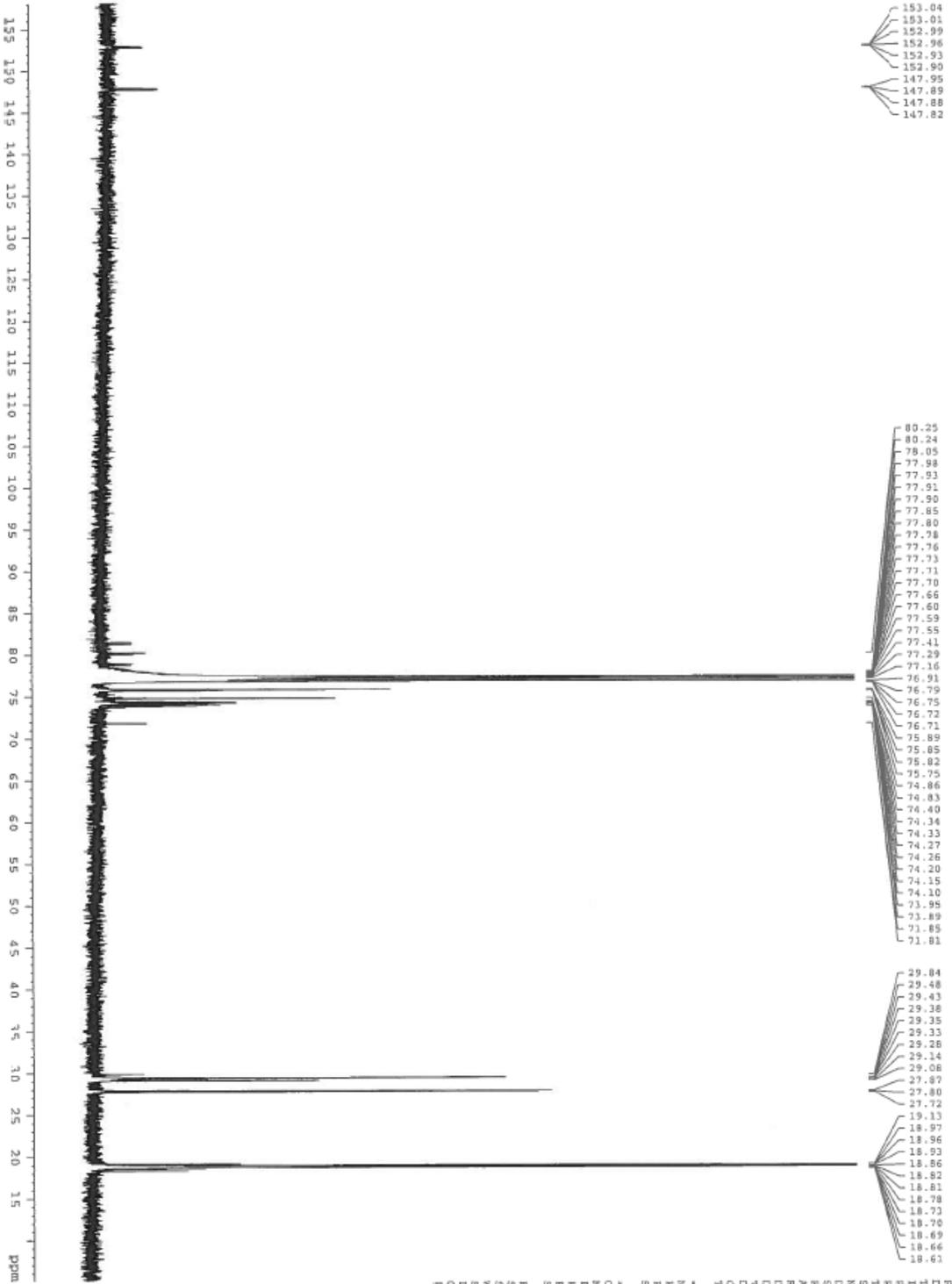
***** CHANNEL f1 *****
 NUQ1 13C
 P1 130 usec
 PL1 0.00 dB
 SFO1 125.7715724 MHz

***** CHANNEL f2 *****
 CPOPRG2 waltz16
 NUQ2 1H
 PCPD2 90.00 usec
 PL12 18.00 dB
 SFO2 500.132806 MHz

F2 - Processing parameters
 SI 524288
 SF 125.757717 MHz
 RG 4096
 SSB 0
 LB 1.20 Hz
 GB 0
 PC 0.60



PT/MS-310511-1 cdcl3 haihd. korkkarissa 23.3.2012



Current Data Parameters
 NAME M6-310511-1
 EXPNO 33
 PROCNO 1

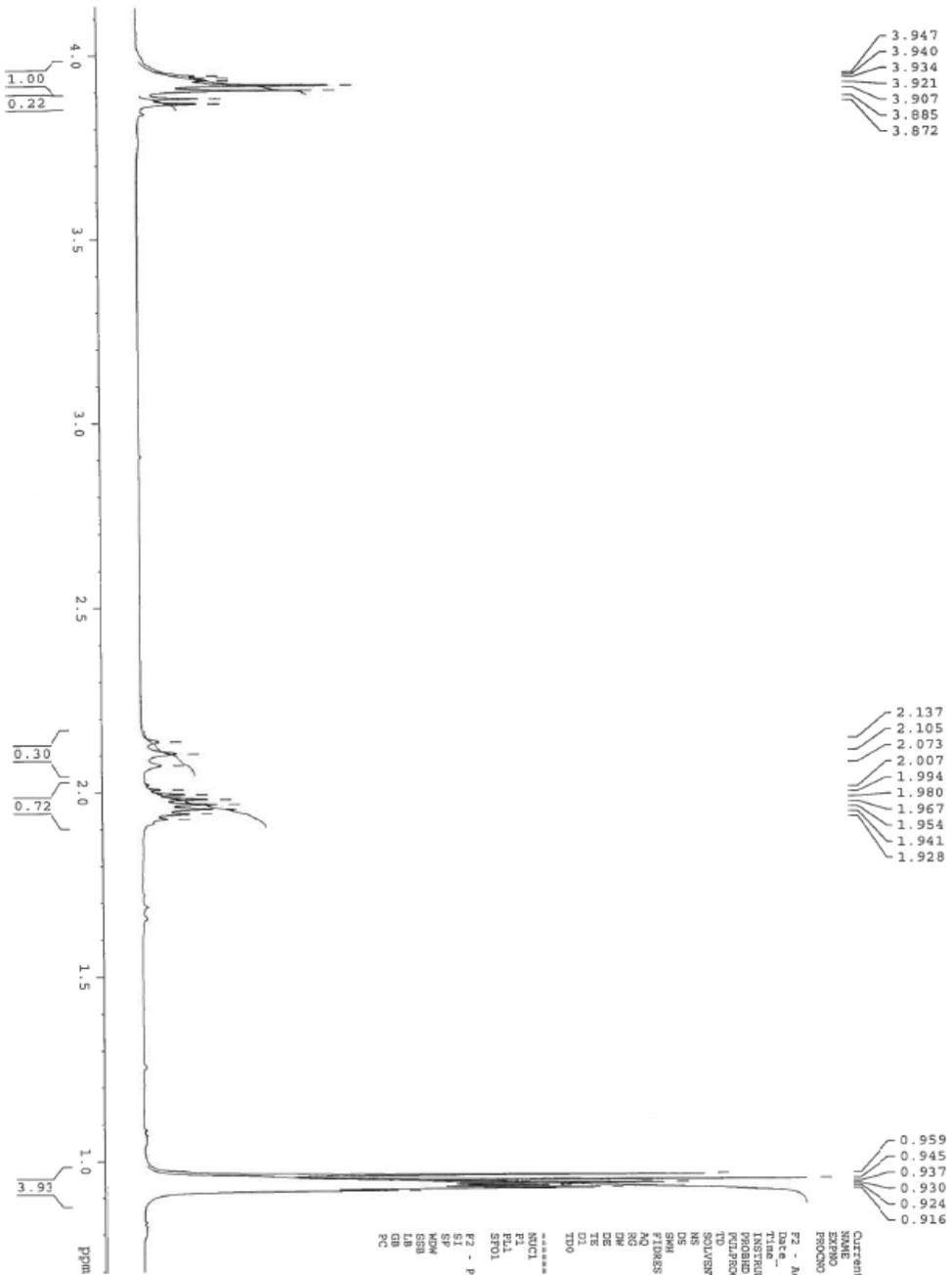
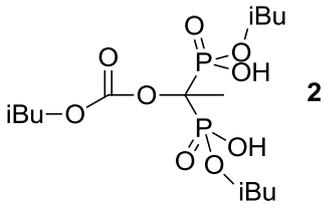
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Date_ 20120324
 Time 11.55
 C1 13C
 INSTRUM spect
 PROBRD BR1
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 3967
 DS 0
 SWH 30303.031 Hz
 FIDRES 0.231194 Hz
 AQ 2.162945 sec
 RG 655
 RB 0.80 Hz
 RR 16.500 usec
 DR 6.00 usec
 TR 300.0 K
 D1 18.00000000 sec
 D11 0.03000000 sec
 T00 1

***** CHANNEL F1 *****
 NUCL 13C
 P1 8.75 usec
 PL1 6.00 dB
 SFO1 125.7715734 MHz

***** CHANNEL F2 *****
 CHRG2 WAIT218
 NUCL2 1H
 P2 90.00 usec
 PCPD2 -1.00 dB
 PL2 18.00 dB
 SFO2 500.1322506 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577708 MHz
 WDW EM
 SSB 0
 LB 0.80 Hz
 GB 0
 PC 0.13



Current Data Parameters

NAME MS-230810-2

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20100924

Time 14:24

INSTRUM spect

PROBHD 5 mm QNP 1H/1

PULPROG zgpg30

TD 65536

TOUVRNT 16

DS 0

SWH 10000.000 Hz

FIDRES 0.152588 Hz

AQ 2.179581 sec

RG 64

DW 50.000 usec

DE 6.00 usec

TE 300.0 K

TU 1.5000000 sec

TD0 1

***** CHANNEL f1 *****

NUC1 31P

P1 9.21 usec

PL1 -1.00 dB

SFO1 500.1340010 MHz

F2 - Processing parameters

SI 32768

SF 500.1300136 MHz

WDW EM

SSB 0

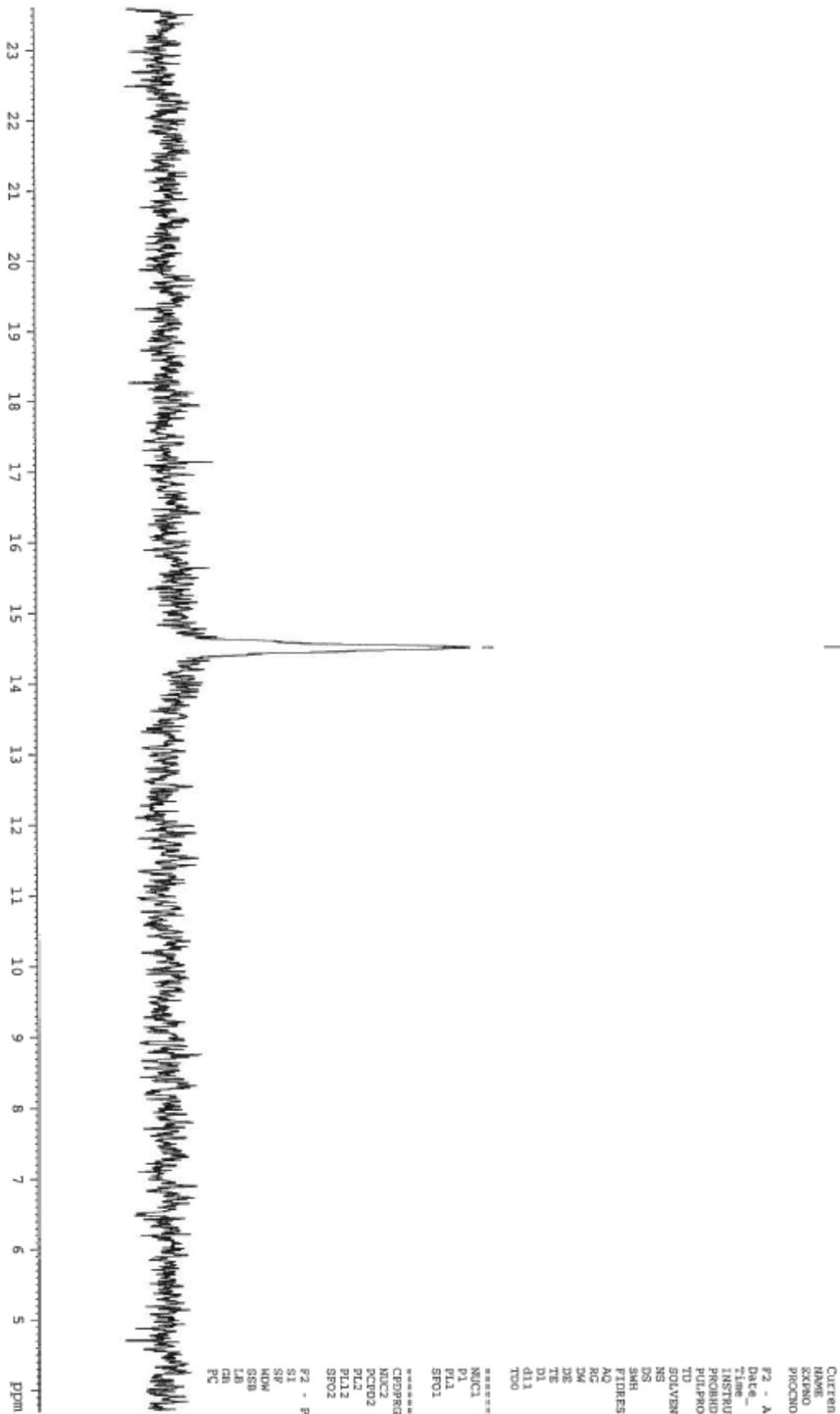
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PC 5.00

PT/MS-230810-3 cdcl3

14.513



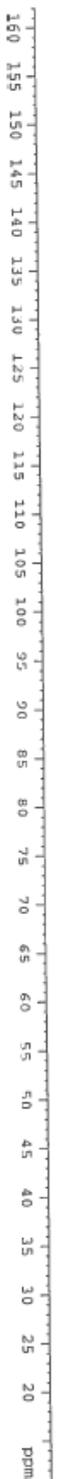
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 EXCNO 1
 PROCNO 1

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 Time_ 12:22:22
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 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 140
 DS 2
 SWH 40650.406 Hz
 FIDRES 0.620276 Hz
 AQ 0.8061551 sec
 RG 2048
 DM 12.300 usec
 DE 16.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 TD0 1

***** CHANNEL F1 *****
 NUCL 31P
 P1 8.50 usec
 PL1 10.00 dB
 SFO1 202.45869310 MHz

***** CHANNEL F2 *****
 CPDPRG2 waltz16
 NUCL 1H
 PCPDZ 90.00 usec
 PL2 -1.00 dB
 PL12 18.56 dB
 SFO2 500.1325007 MHz

F2 - Processing parameters
 SI 262144
 SF 202.4584297 MHz
 MW 6W
 SSB 0
 GB 0
 CB 0
 PC 1.40



154.79
154.73
154.67

82.66
81.47
80.27
75.53
75.38
75.24
75.18
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74.90
74.84
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74.65
74.54
74.51
74.49
74.29
74.26
74.23

49.51
49.45
49.34
49.28
49.17
49.11
49.00
48.93
48.66
48.49

30.55
30.53
30.51
30.47
30.46
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19.12
19.05
19.04
19.01

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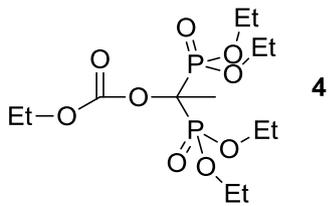
Current Data Parameters
NAME      MS-120111-2
EXPNO     33
PROCNO    1

F2 - Acquisition Parameters
Date_     20120208
Time      18.43
INSTRUM   spect
PROBHD    5 mm QNP 1H/1
PULPROG   zgpg30
TD         131072
SOLVENT   H2O
NS         3544
DS         0
SWH        30303.031 Hz
FIDRES     0.231194 Hz
AQ         2.1697545 sec
RG         8132
DM         16.400 usec
DE         4.400 usec
TE         300.0 K
D1         18.00000000 sec
d11        0.03000000 sec
T200       1

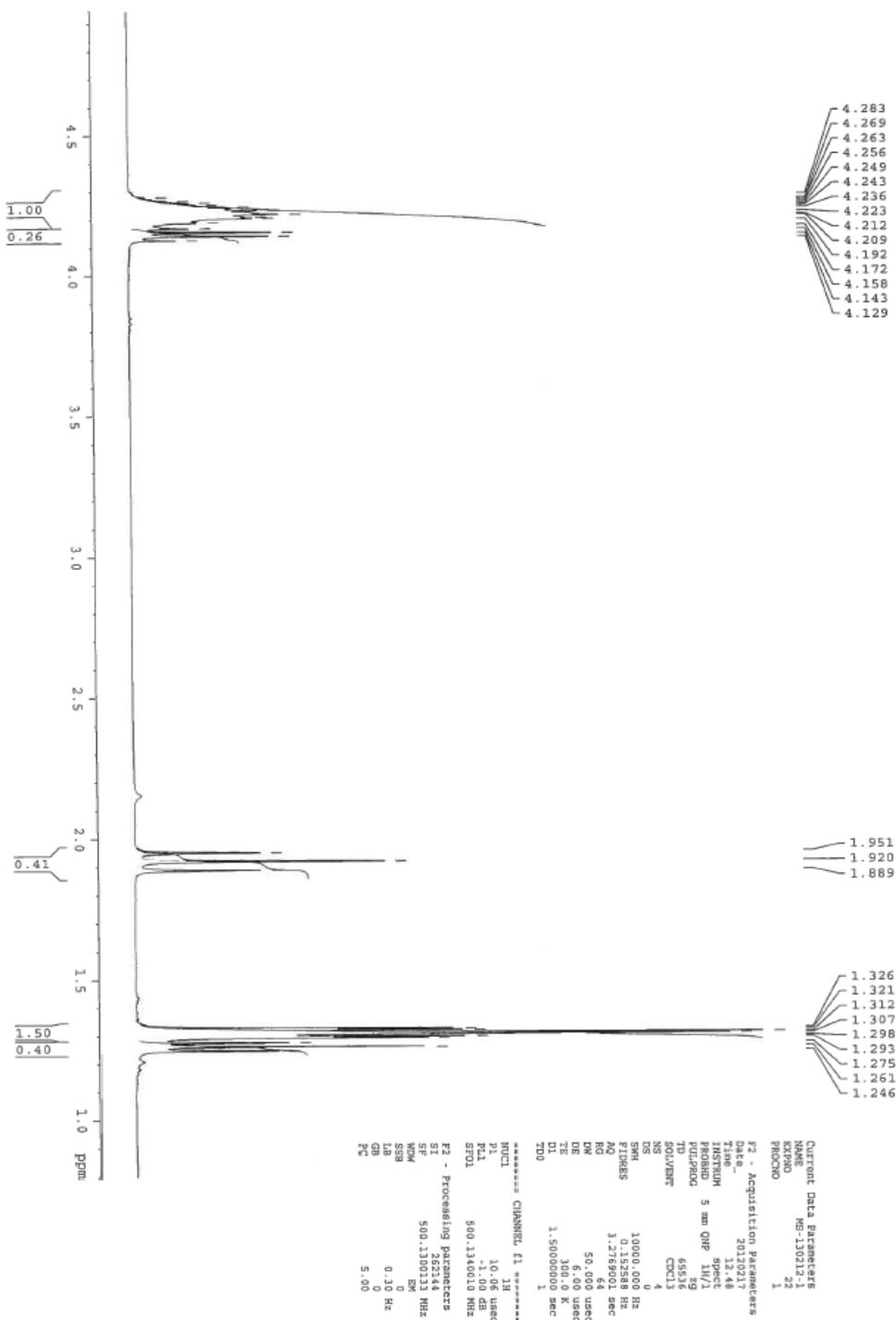
***** CHANNEL F1 *****
NUC1       13C
P1         12.5 usec
PL1        0.00 dB
SFO1       125.7715724 MHz

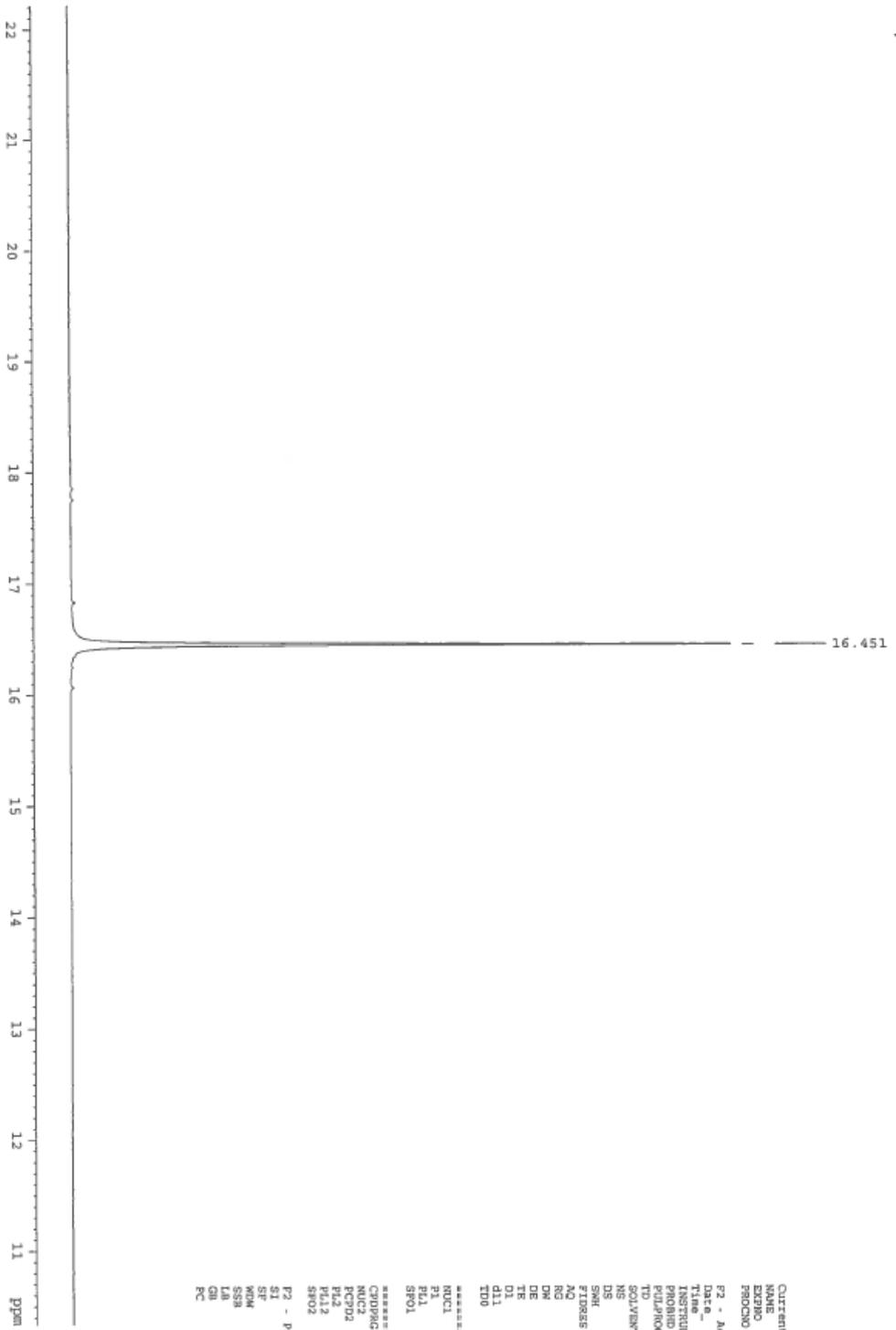
***** CHANNEL F2 *****
CPDPRG2   waltz16
NUC2       1H
PCPD2     90.00 usec
PL2        1.00 dB
SFO2       500.132506 MHz

F2 - Processing parameters
SI         524288
SF         125.7574079 MHz
WDW        EM
SSB        0
GB         0
PC         1.00
  
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PT/MS-130212-1 cdcl3 fr. 17-27





```

Current Data Parameters
NAME      MS-130212-1
EXPNO     11
PROCNO    1

F2 - Acquisition Parameters
Date_     20120217
Time      12.46
INSTRUM   spect
PROBHD    5 mm QNP 1H/1
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         7
DS         0
SWH        40650.406 Hz
FIDRES     0.620276 Hz
AQ         0.8502048 sec
RG         2048
DE         12.300 usec
TE         300.1 K
D1         2.00000000 sec
d11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         8.50 usec
PL1        0.00 dB
SFO1       202.450230 MHz

===== CHANNEL f2 =====
CYPUPRG2   waltz16
NUC2        1H
PCPD2       90.00 usec
PL2         1.00 dB
SFO2       500.135007 MHz

F2 - Processing parameters
SI         262144
SF         202.4563491 MHz
WDW         1
SSB         0
LBI         2.00 Hz
GB          0
PC         1.40
    
```

