OL020158j

Experimental Section for Previously Unknown Compound **2**

To Accompany:

Synthesis of 1,3-difluoroaromatics in One Step by Addition of Difluorocarbene to 1,2-Disubstituted Cyclobutenes

Heather M. Morrison, James E. Rainbolt and Scott B. Lewis*

Made from Ph-Hg-CF₃

To a 25 mL 3-neck round bottom flask, excess NaI was added and dried under vacuum at 150 °C for 24 hr. To this was added 0.753 g (2.16 mmol) of Ph-Hg-CF_3 and drying continued at 25 °C for an additional 24 hr. under vacuum. Finally, 0.100 g (0.725 mmol) 1,2-di-*n*-propylcyclobutene in 8 mL of benzene (K/benzophenone) was added and the mixture brought to reflux for 48 hr. After that time, the mixture was cooled, filtered, washed with water and the solvent removed under reduced pressure. Hexane was added and the mixture was filtered again followed by solvent removal under reduced pressure. The material was chromatographed on silica gel using cyclohexane which yielded 0.0861 g (60%) of 1,3-difluoro-2,4-di-*n*-propylcyclobutene (**2**). ^{1}H NMR ($CDCl_3$ δ relative to TMS at 0.0 ppm) δ 6.94 (m, 1H), 6.74 (m 1H), 2.65 (t, J=7.35 Hz, 2H), 2.58 (t, J=7.50 Hz, 2H), 1.6 (m), 0.93 (m); ^{13}C NMR δ 159.8 (dd, $^1J_{CF}$=243.17, $^3J_{CF}$=8.83 Hz), 159.5 (dd, $^1J_{CF}$=244.94, $^3J_{CF}$=8.83 Hz), 127.5 (dd, $^3J_{CF}$=9.71, $^3J_{CF}$=7.06), 124.7 (dd, $^2J_{CF}$=17.84, $^4J_{CF}$=3.71 Hz), 117.4 (dd, $^2J_{CF}$=21.72, $^2J_{CF}$=20.49 Hz), 110.1 (dd, $^2J_{CF}$=22.43, $^4J_{CF}$=3.71 Hz), 30.8 (d, $^3J_{CF}$=2.29 Hz), 24.4 (t, $^3J_{CF}$=2.47 Hz), 23.4, 27.8, 13.79, 13.76; ^{19}F NMR (δ relative to $CFCl_3$ at 0.0 ppm) δ –120.35 (m), -121.43 (m); IR 2963, 1626, 1596, 1489 cm^{-1}; HRMS (EI) *m/z* calc. for $C_{12}H_{16}F_2$: 198.1220, found: 198.1225.

Made from Acetate Salt

To a 50 mL RBF, 2.0 g (0.013 mol) of $NaCClF_2CO_2$ was added and placed under vacuum for 24 hours. To this, 20 mL of dry diglyme (distilled from CaH_2) was added under argon and stirred until the salt dissolved. In a separate 50 mL RBF, 0.121 g (8.77×10^{-4} mol) of 1,2-di-*n*-propylcyclobutene was added along with 5 mL of the diglyme salt solution. This solution was brought to reflux under argon, with additional 5 mL aliquots of the diglyme salt solution added by syringe every 15 minutes until all had been added. The solution was refluxed an additional 15 minutes, cooled and the majority of the diglyme distilled off through a short path distillation head. The remaining material was columned through silica gel using cyclohexane to afford 0.0719 g (41%) of 1,3-difluoro-2,4-di-*n*-propylcyclobutene (**2**). ^{1}H NMR ($CDCl_3$ δ relative to TMS at 0.0 ppm) δ 6.94 (m, 1H), 6.74 (m 1H), 2.65 (t, J=7.35 Hz, 2H), 2.58 (t, J=7.50 Hz, 2H), 1.6 (m), 0.93 (m); ^{13}C NMR δ 159.8 (dd, $^1J_{CF}$=243.17, $^3J_{CF}$=8.83 Hz), 159.5 (dd, $^1J_{CF}$=244.94, $^3J_{CF}$=8.83 Hz), 127.5 (dd, $^3J_{CF}$=9.71, $^3J_{CF}$=7.06), 124.7 (dd, $^2J_{CF}$=17.84, $^4J_{CF}$=3.71 Hz), 117.4 (dd, $^2J_{CF}$=21.72, $^2J_{CF}$=20.49 Hz), 110.1 (dd, $^2J_{CF}$=22.43, $^4J_{CF}$=3.71 Hz), 30.8 (d, $^3J_{CF}$=2.29 Hz), 24.4 (t, $^3J_{CF}$=2.47 Hz), 23.4, 27.8, 13.79, 13.76; ^{19}F NMR (δ relative to $CFCl_3$ at 0.0 ppm) δ –120.35 (m), -121.43 (m).

Spectra for 1,3-difluoro-2,4-diphenylbenzene (**1**) and 1,3-difluoro-2,4-di-n-propylbenzene (**2**)

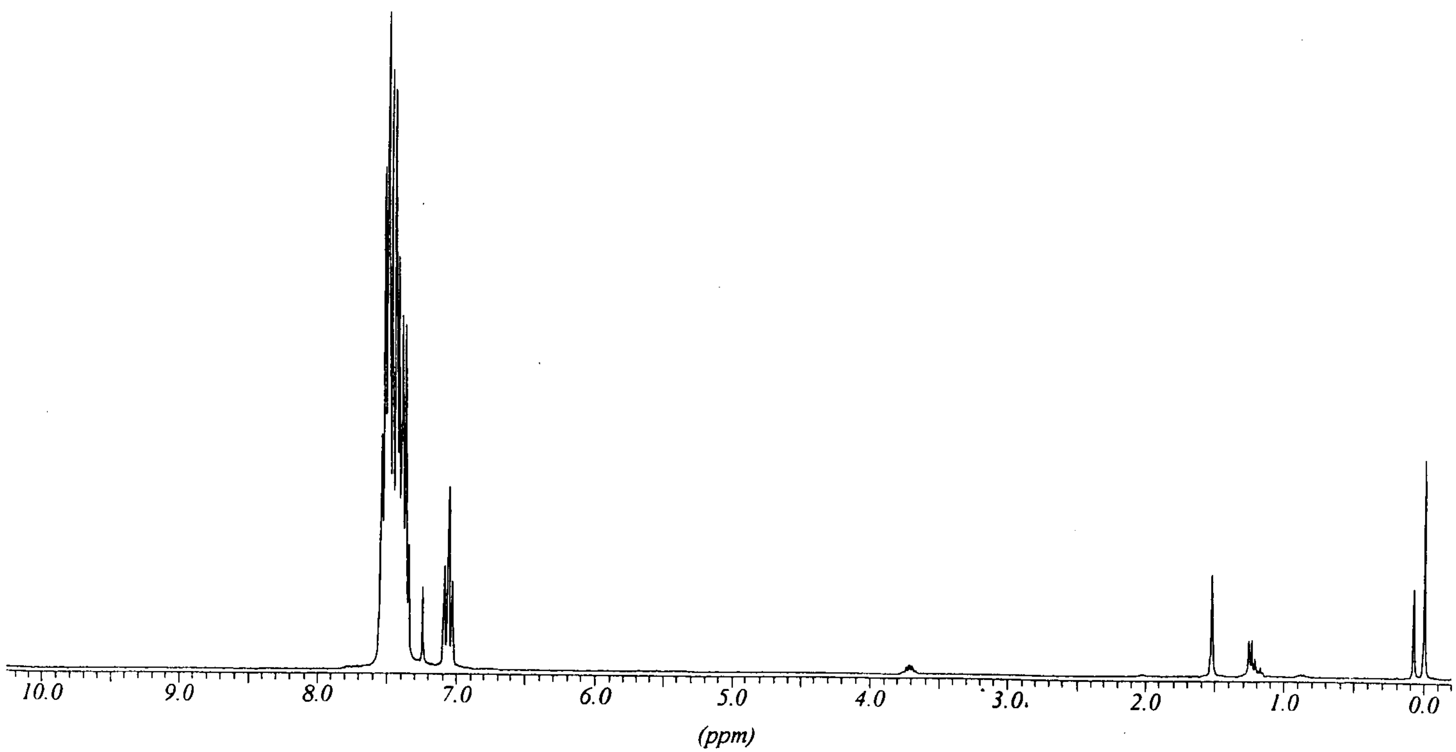
To Accompany:
Synthesis of 1,3-difluoroaromatics in One Step by Addition of Difluorocarbene to 1,2-Disubstituted Cyclobutenes.
Heather M. Morrison, James E. Rainbolt and Scott B. Lewis*

Compound **1**: Prepared by using acetate salt (see reference 8 for original prep. of this compound)

Compound **2**: Prepared by using Ph-Hg-CF_3

Compound **2**: Prepared by using acetate salt

35. ^{1}H NMR 0.0 to 7.5 ppm
36. ^{1}H NMR 6.6 to 7.0 ppm
37. ^{1}H NMR 0.8 to 2.8 ppm
38. ^{19}F NMR 50 to −180 ppm
39. ^{19}F NMR −119.2 to −122 ppm
40. ^{13}C NMR 0.0 to 220 ppm
41. ^{13}C NMR 108 to 164 ppm
42. ^{13}C NMR 13 to 32 ppm
43. GC/MS



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PULPROG	:	zg30
RG	:	512.0000000
SOLVENT	:	CDCl3
SW	:	16.0495 ppm

*** Processing Parameters ***

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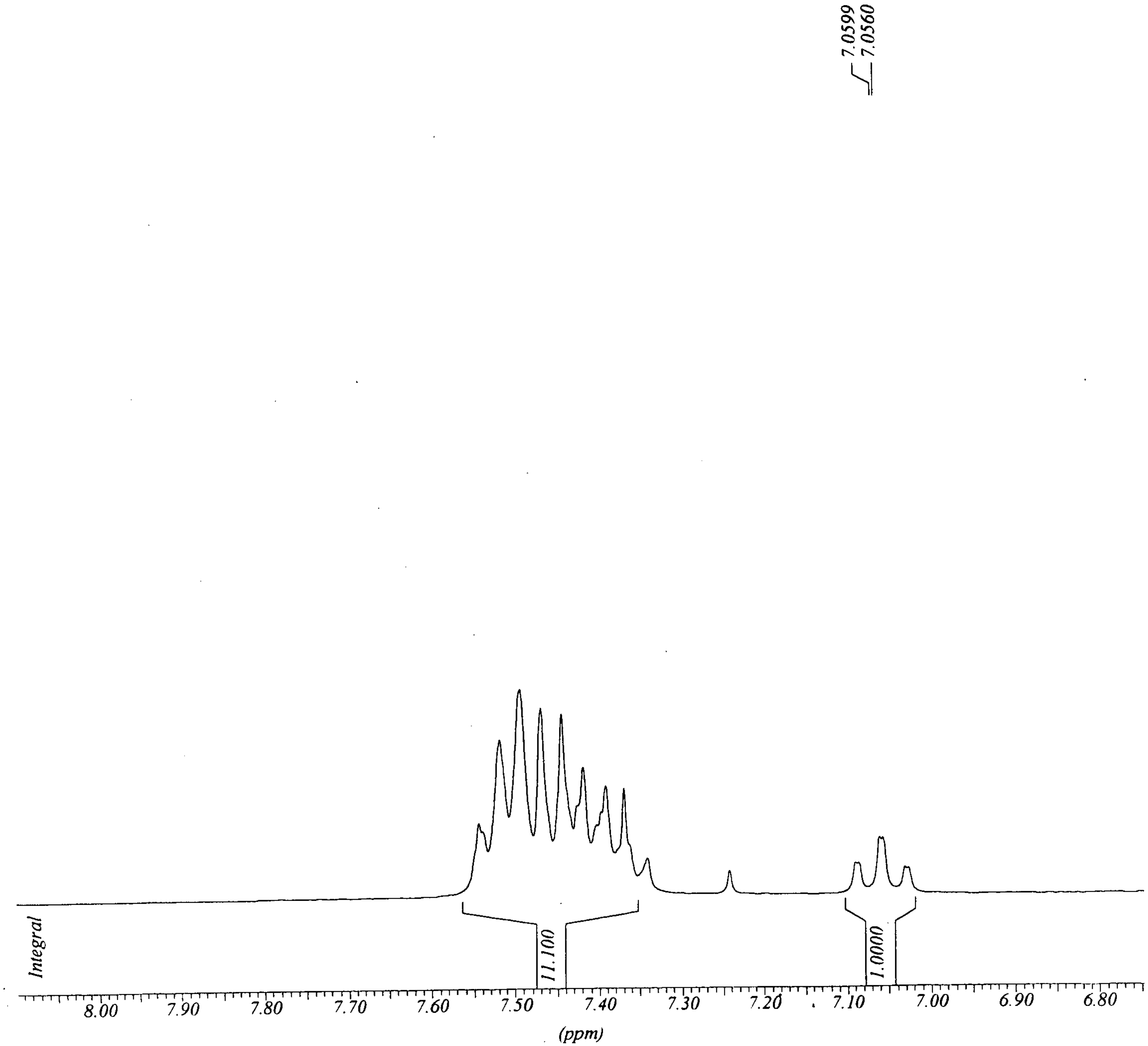
MI	:	0.00 cm
PC	:	1.00
SI	:	32768

*** 1D NMR Plot Parameters ***

SOLVENT	:	?

1



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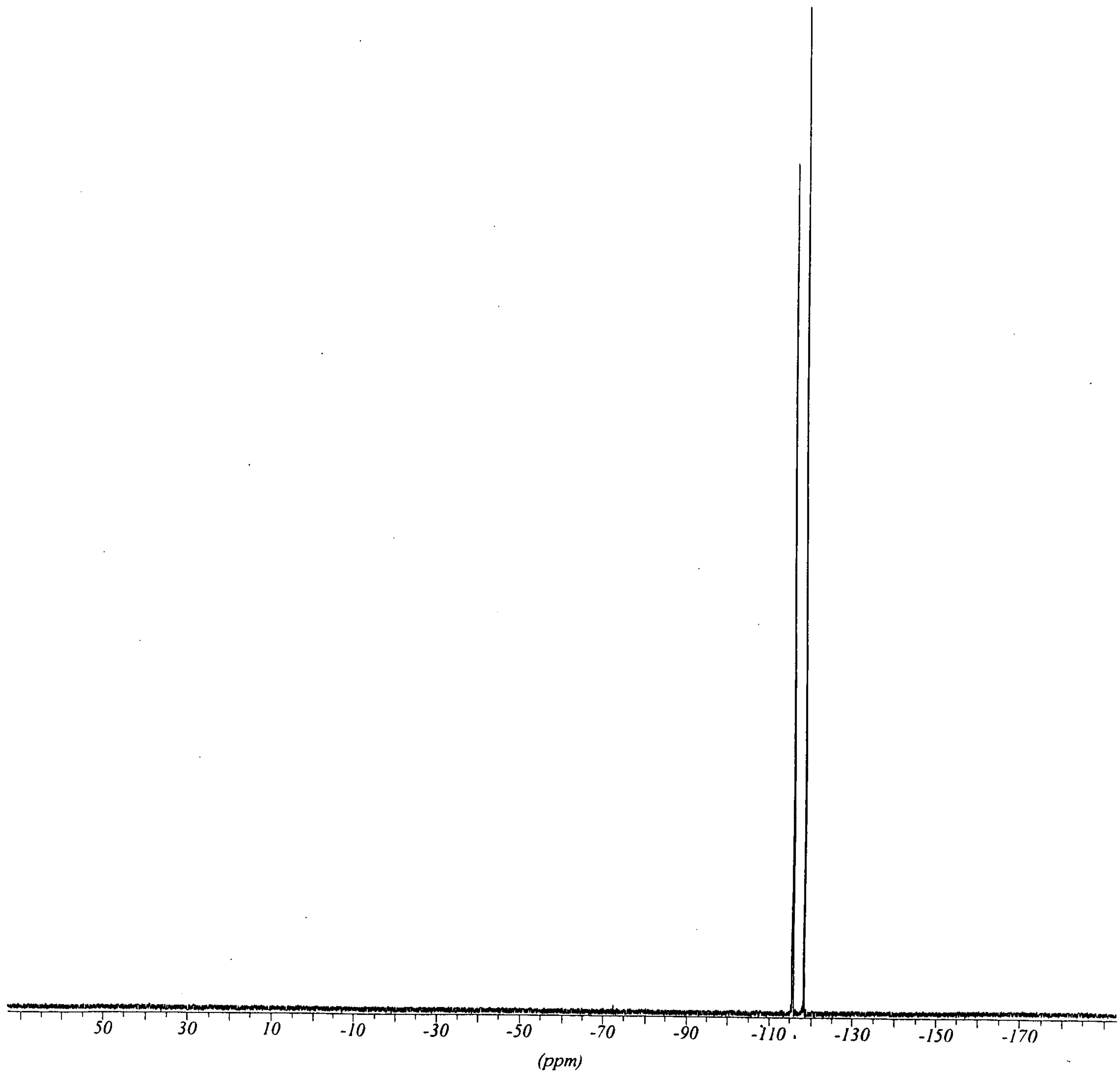
SOLVENT	:	CDCl3
SW	:	16.0495 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
MI	:	0.00 cm
PC	:	1.00
SI	:	32768

*** 1D NMR Plot Parameters ***

SOLVENT	:	?



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NUC1	:	19F
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PULPROG	:	zg30
RG	:	3251.0000000
SOLVENT	:	CDCl3
SW	:	266.2581 ppm

*** Processing Parameters ***

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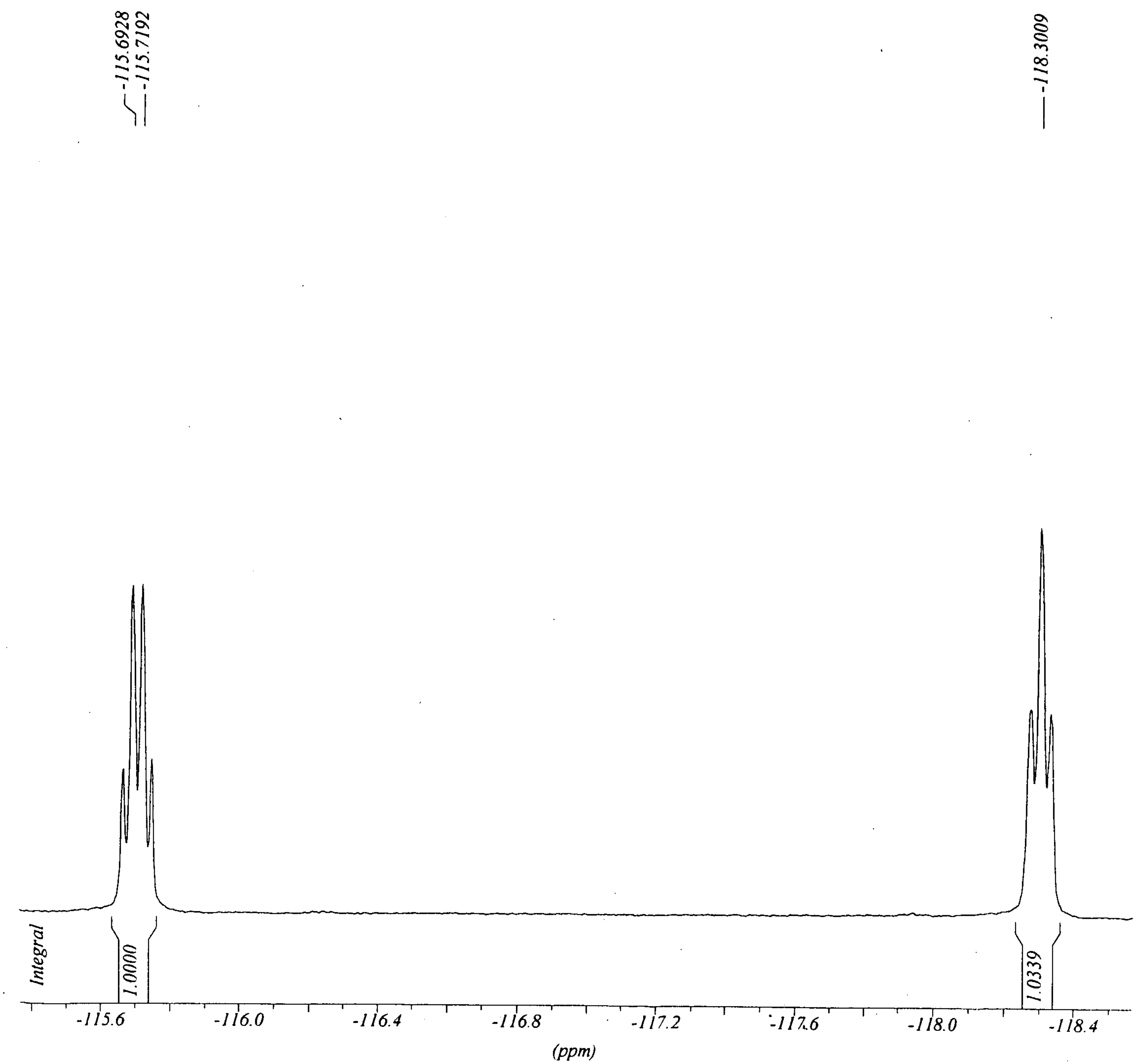
PC	:	1.00
SI	:	131072

*** 1D NMR Plot Parameters ***

SOLVENT	:	?

3



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NUC1	:	19F
O1	:	-16944.27 Hz
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PULPROG	:	zg30
RG	:	3251.0000000
SOLVENT	:	CDCl3
SW	:	266.2581 ppm

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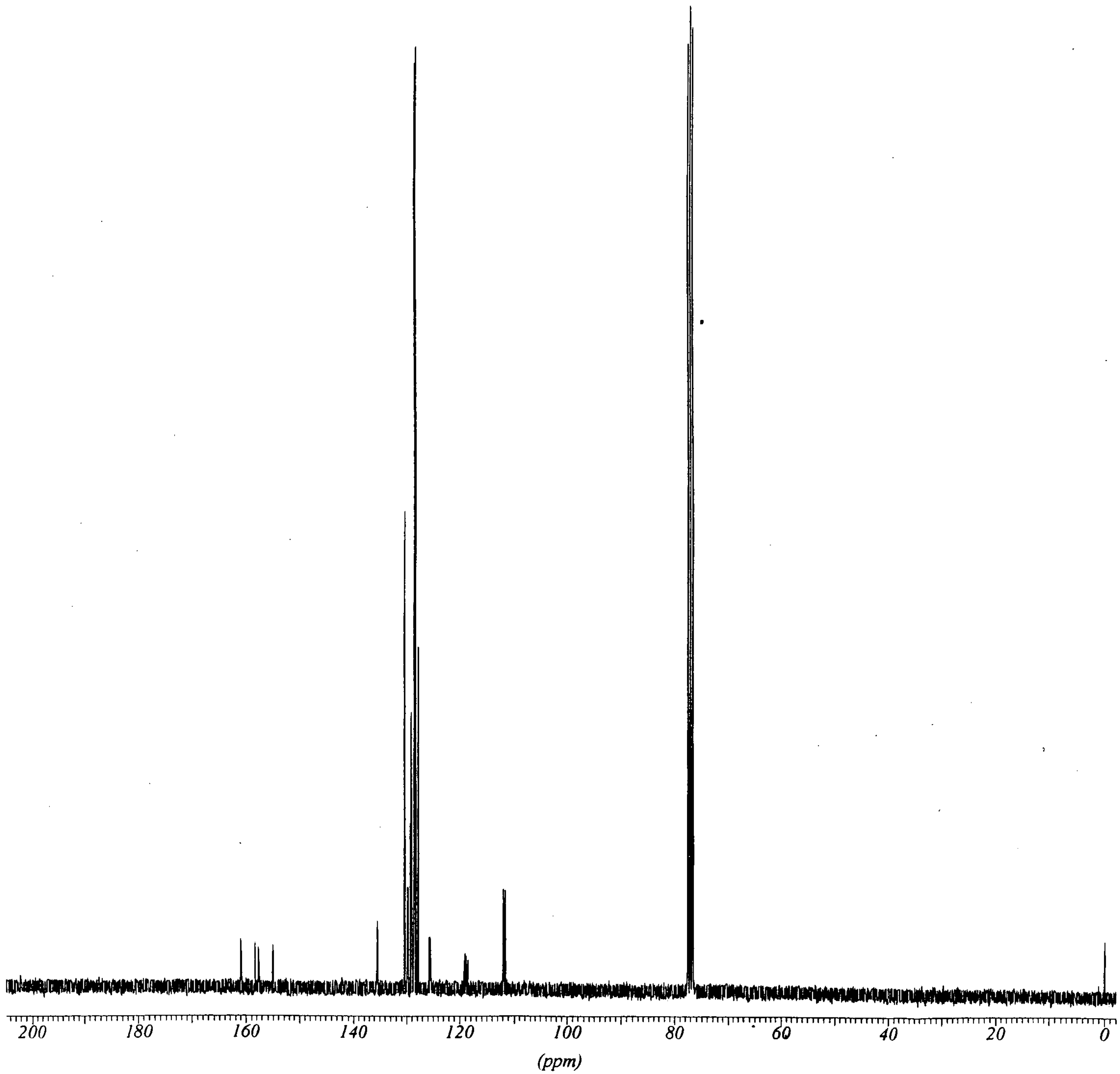
*** 1D NMR Plot Parameters ***

SOLVENT	:	?



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PULPROG	:	zgdc30
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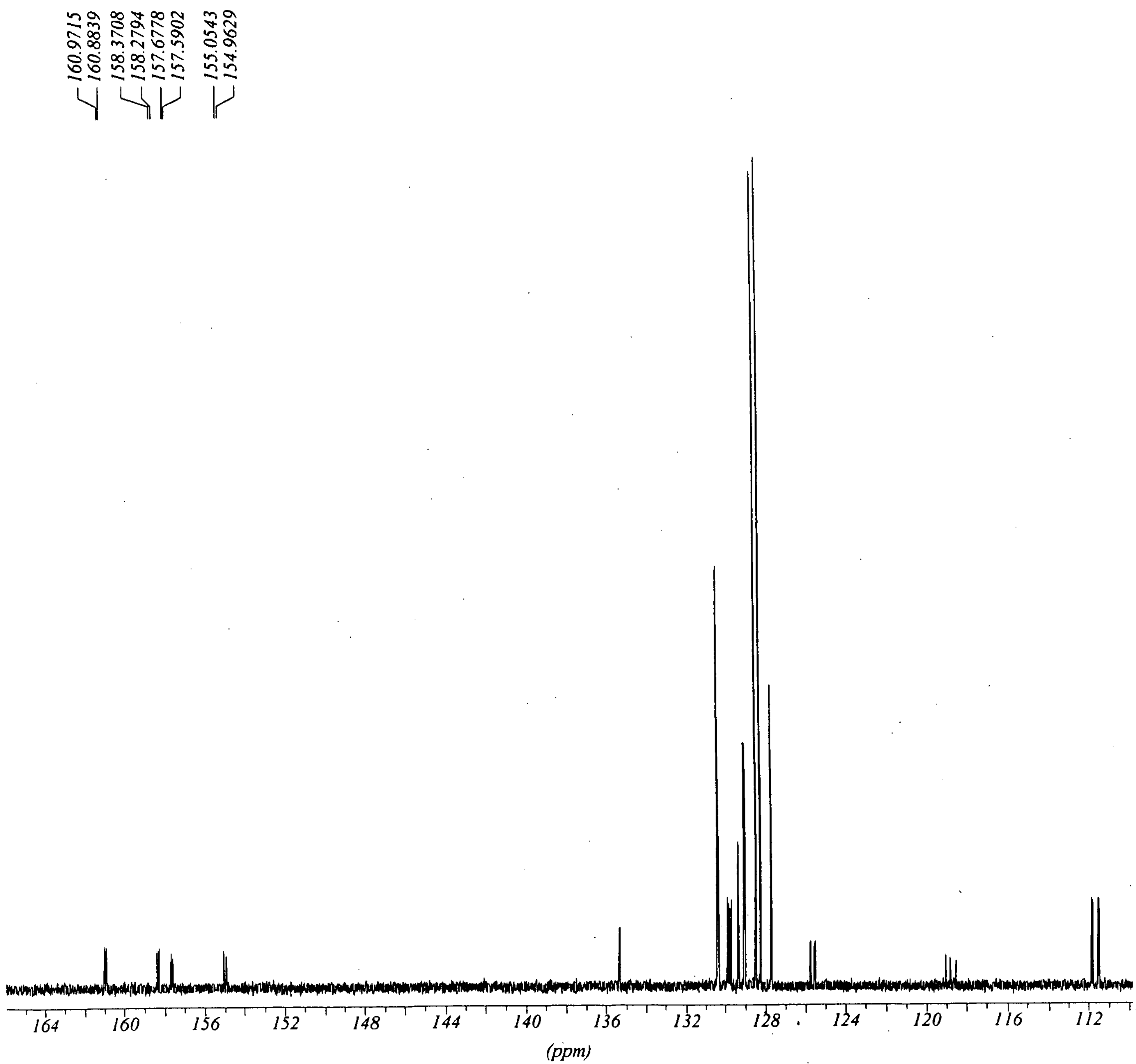
*** 1D NMR Plot Parameters ***		
SOLVENT	:	?

5



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DATE_d	:	Jul 02 2002
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NS	:	7926
NUC1	:	13C
O1	:	9056.13 Hz
PROBHD	:	5 mm QNP 1H/13C/31 9F -
PULPROG	:	zgdc30
RG	:	574.7000122
SOLVENT	:	CDCl3
SW	:	249.5124 ppm

*** Processing Parameters ***

LB	:	0.00 Hz
MI	:	0.00 cm
PC	:	1.00
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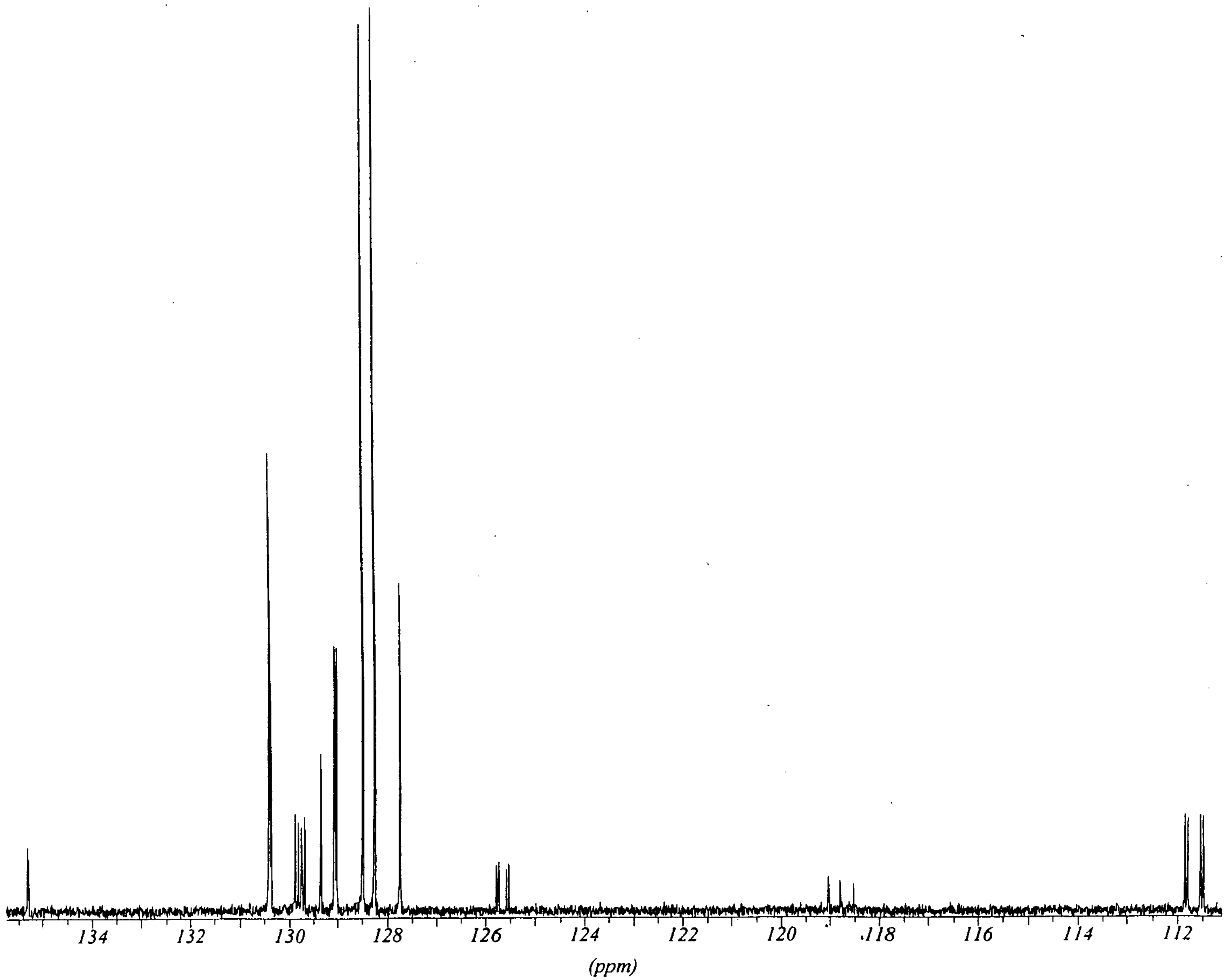
*** 1D NMR Plot Parameters ***

SOLVENT	:	?



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FW	:	90000.00 Hz
NS	:	7926
NUC1	:	13C
O1	:	9056.13 Hz
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PULPROG	:	zgdc30
RG	:	574.7000122
SOLVENT	:	CDCl3
SW	:	249.5124 ppm
*** Processing Parameters ***		
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MI	:	0.00 cm
PC	:	1.00
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*** 1D NMR Plot Parameters ***		
SOLVENT	:	?

*** Current Data Parameters ***

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PULPROG	:	zgdc30
RG	:	4597.6000977
SOLVENT	:	CDCl3
SW	:	266.2581 ppm

*** Processing Parameters ***

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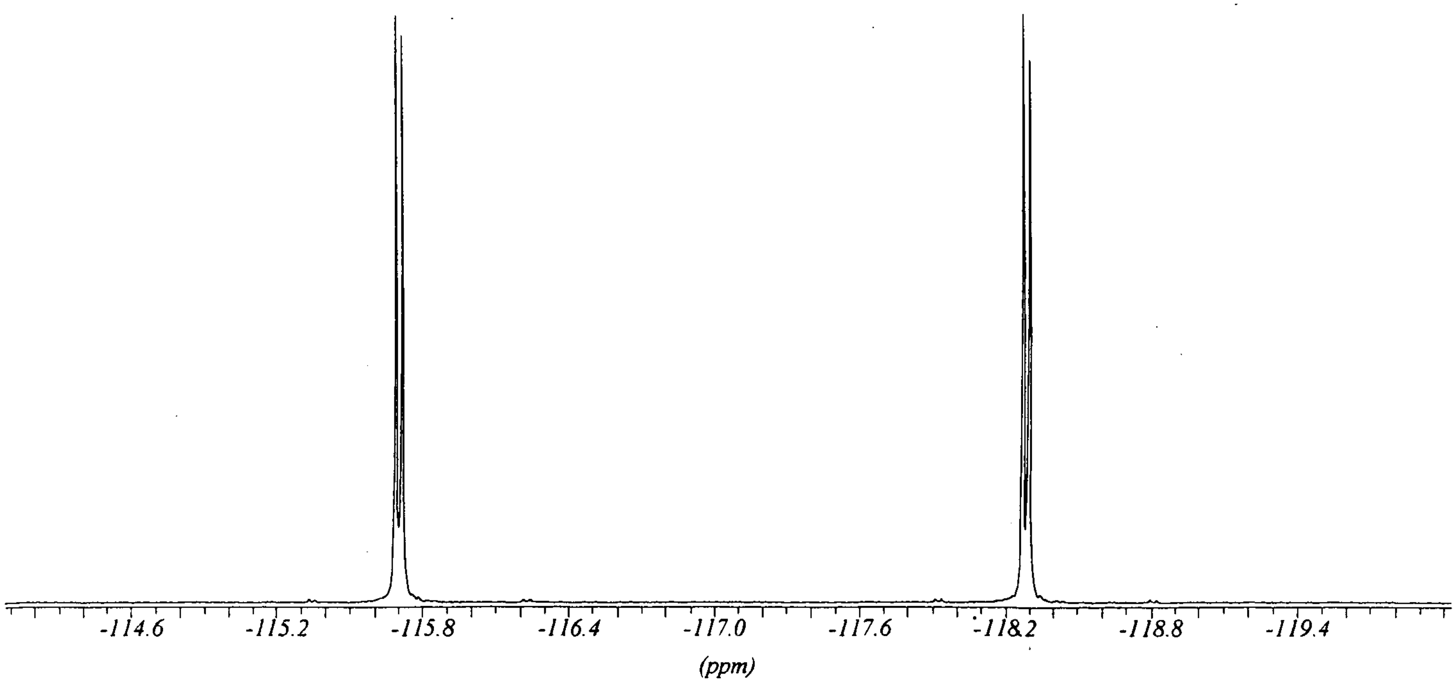
*** 1D NMR Plot Parameters ***

SOLVENT	:	?



8

*** Current Data Parameters ***

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RG	:	16384.0000000
SOLVENT	:	CDCl3
SW	:	80.0051 ppm

*** Processing Parameters ***

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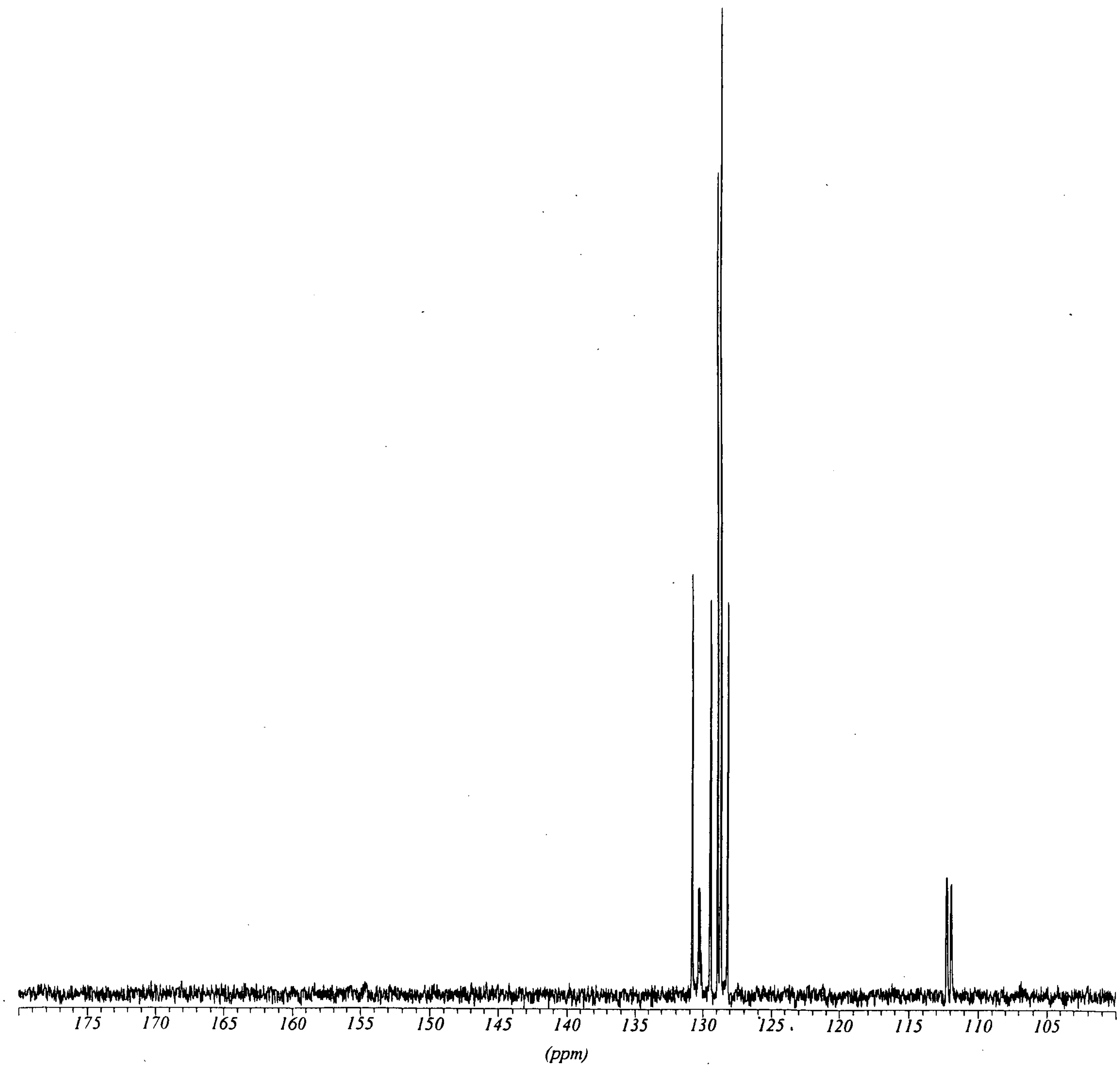
*** 1D NMR Plot Parameters ***

SOLVENT	:	?



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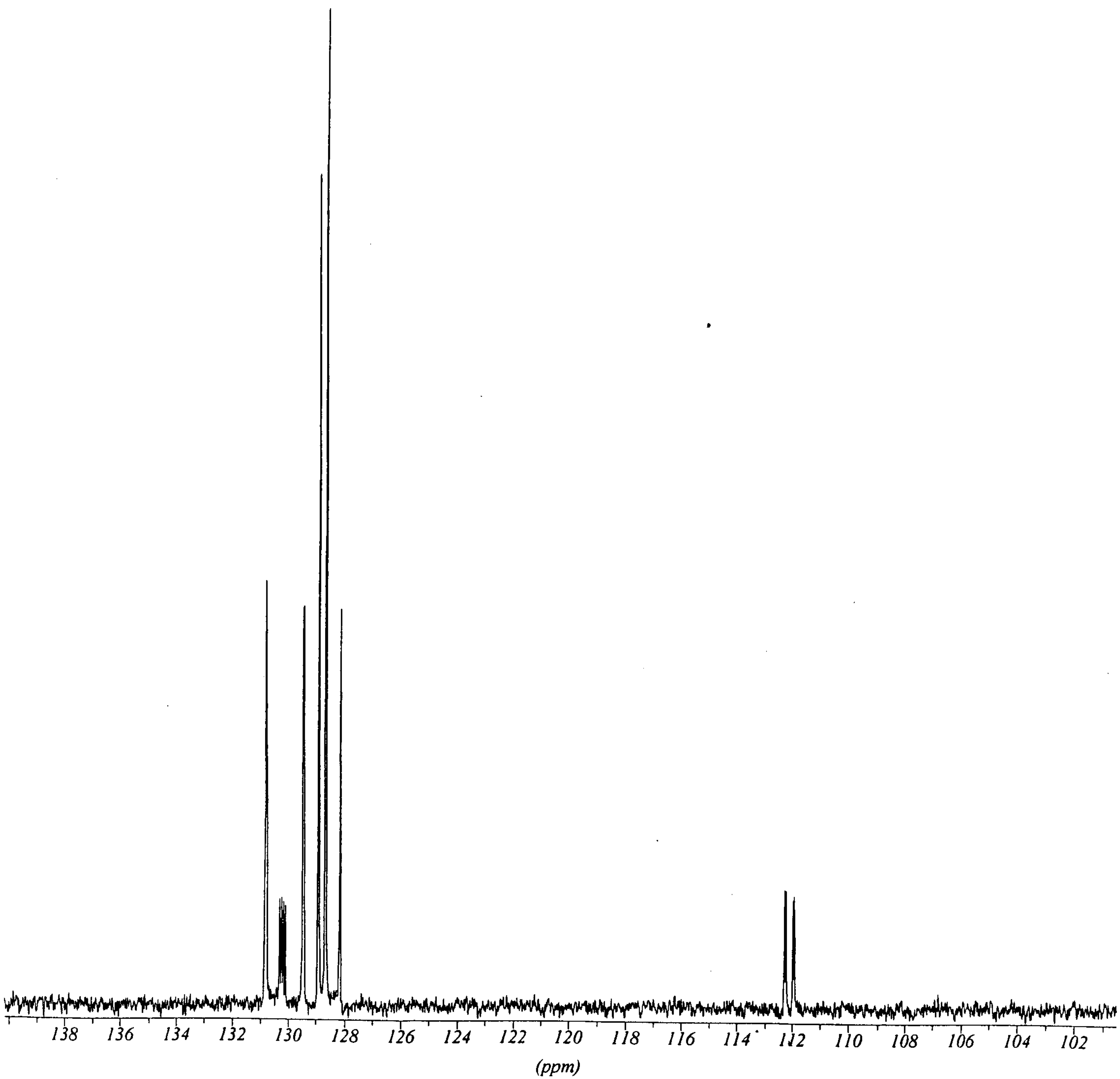

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NUC1	:	13C
O1	:	10565.48 Hz
PROBHD	:	5 mm QNP 1H/13C/31P/19F -
PULPROG	:	dept135
RG	:	16384.0000000
SOLVENT	:	CDCl3
SW	:	80.0051 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
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*** 1D NMR Plot Parameters ***

SOLVENT	:	?

10

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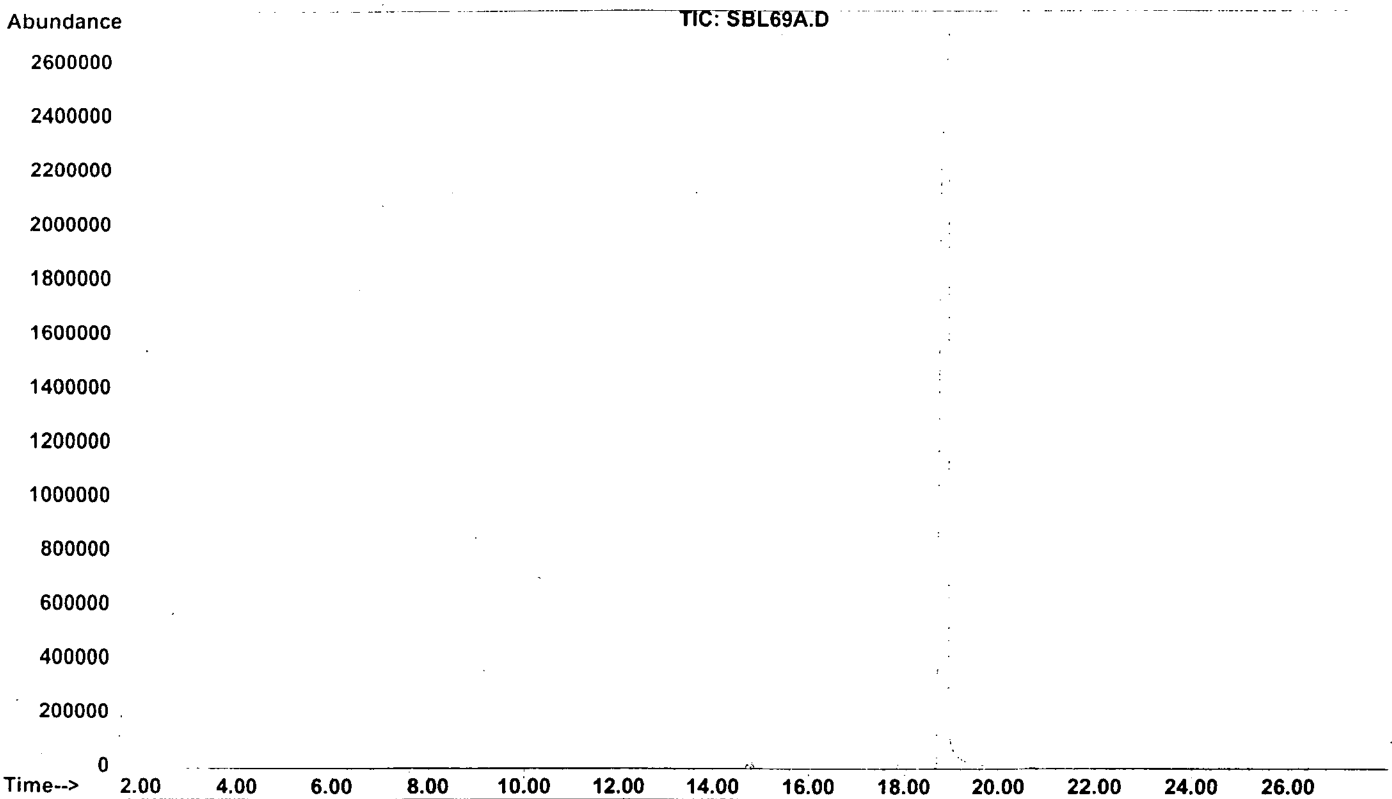

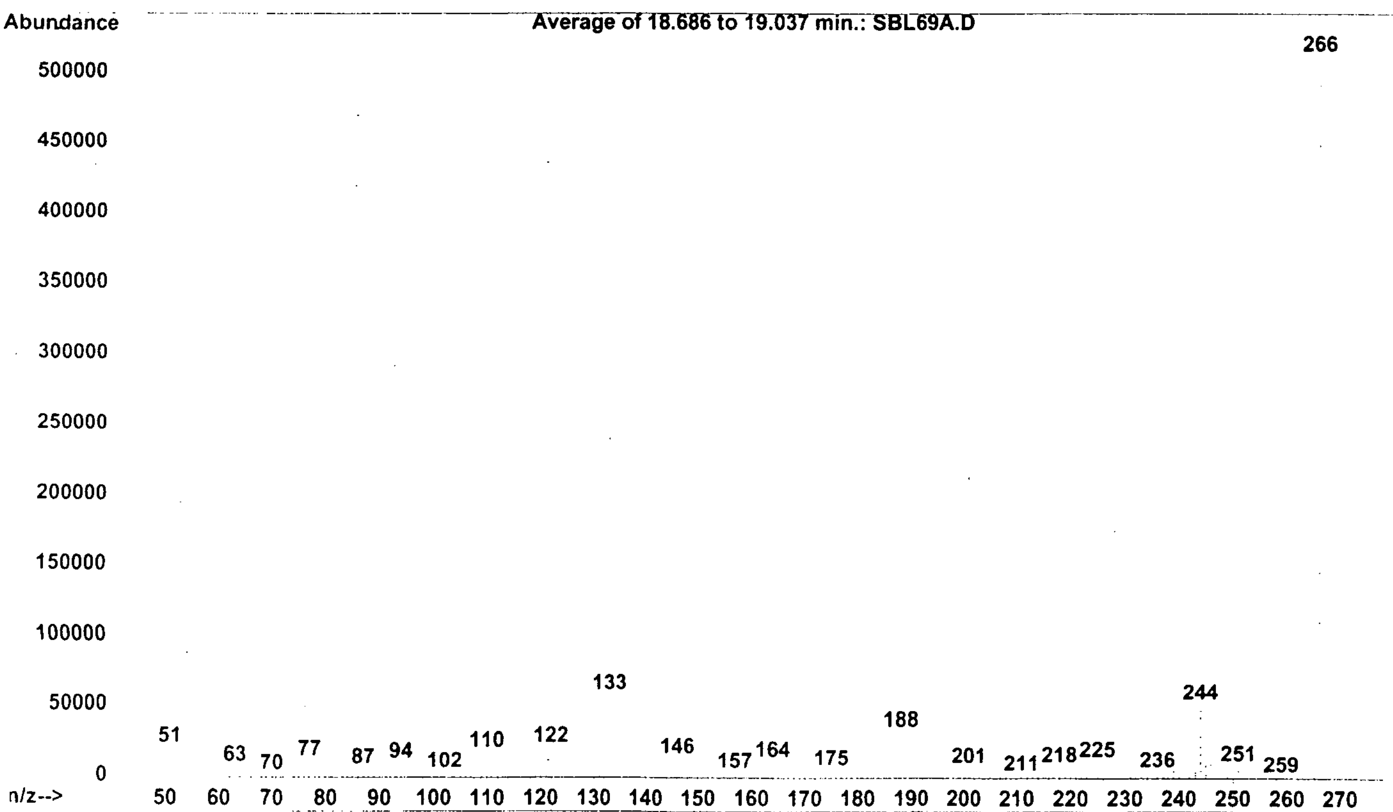

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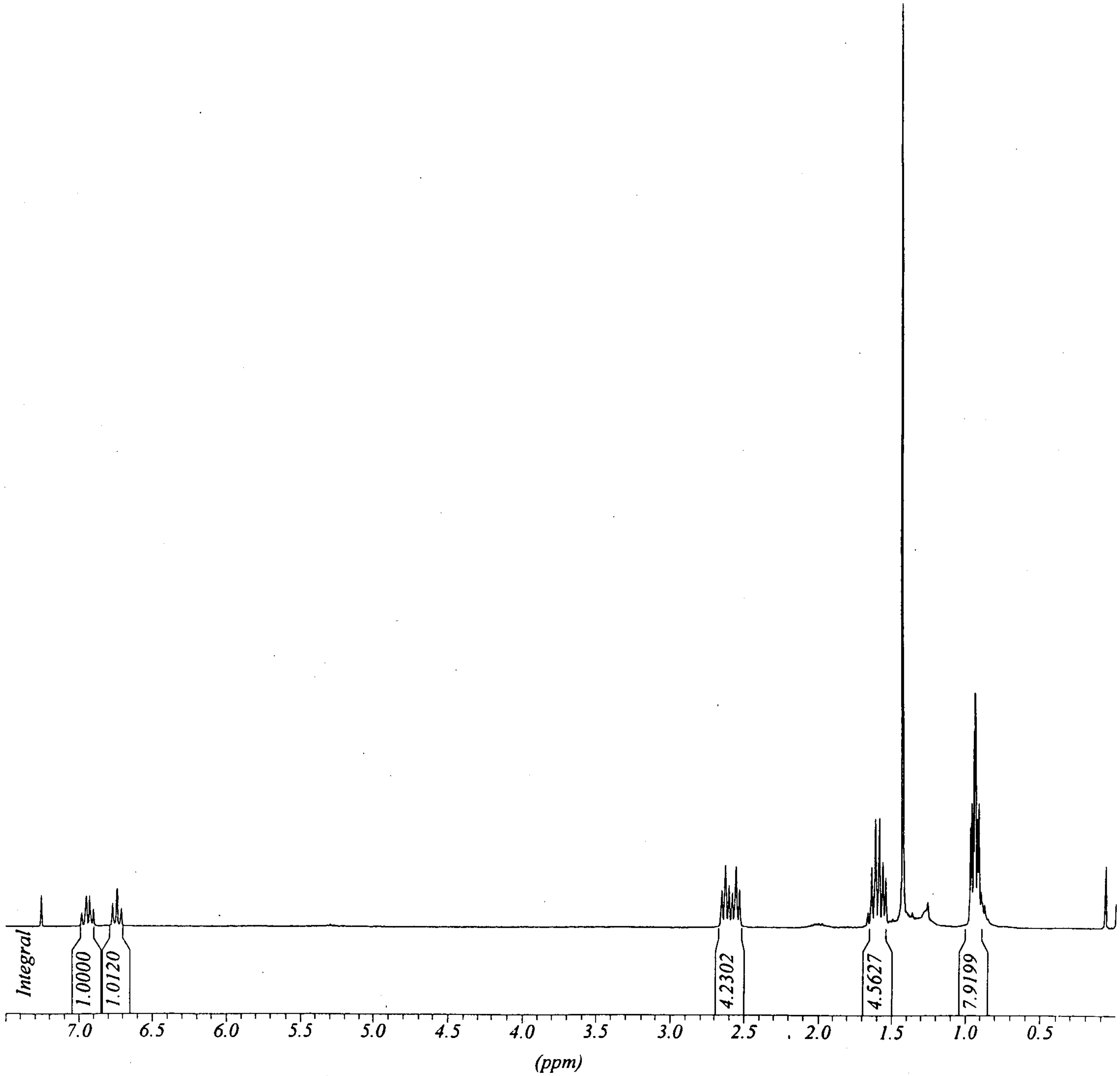

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O1 : 2117.60 Hz
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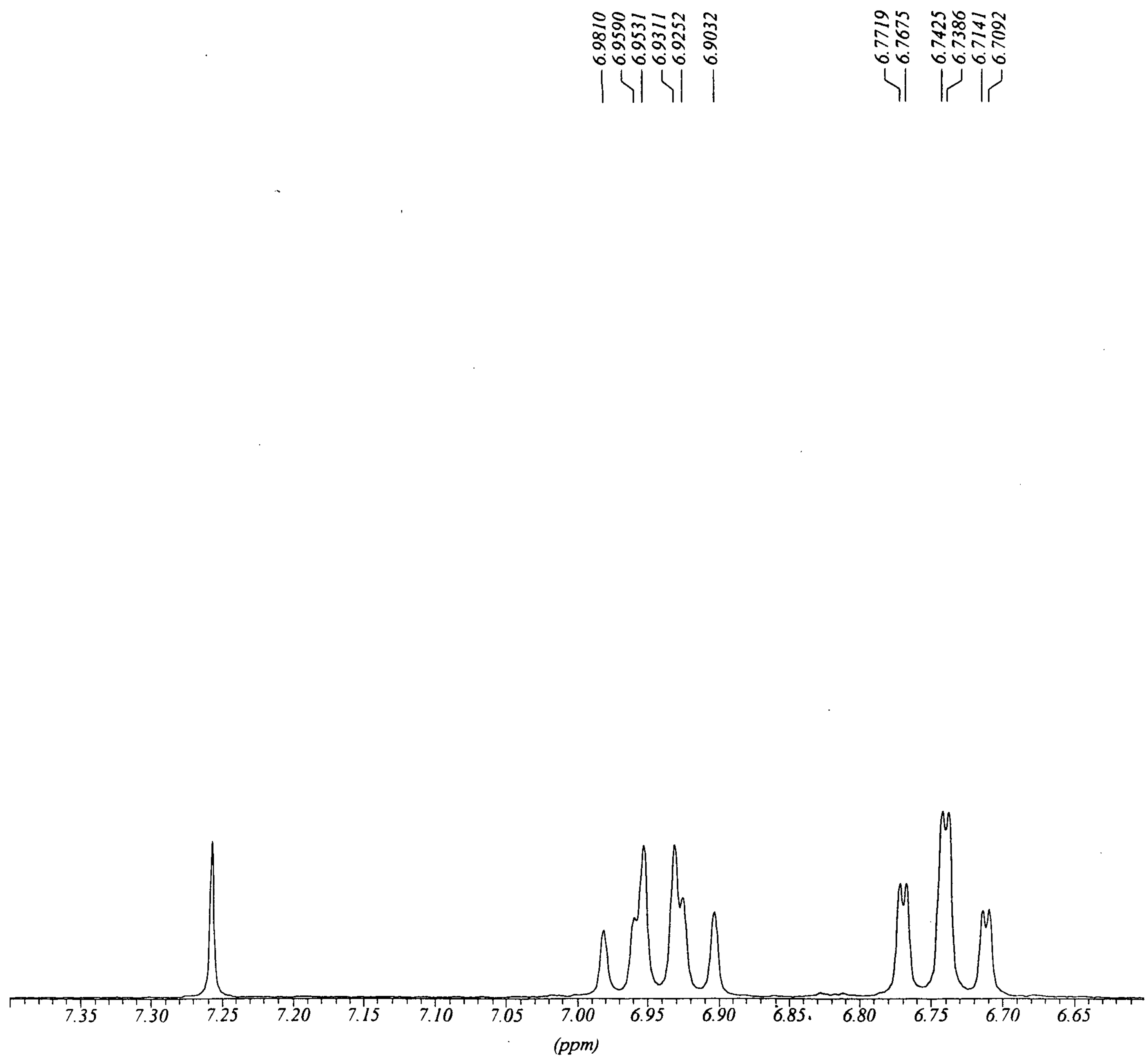
*** 1D NMR Plot Parameters
SOLVENT : ?

12



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RG	:	161.3000031
SOLVENT	:	CDCl3
SW	:	16.0495 ppm

*** Processing Parameters ***

LB	:	0.30 Hz
MI	:	0.00 cm
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SI	:	32768

*** 1D NMR Plot Parameters ***

SOLVENT	:	?

*** Current Data Parameters ***

NAME	:	hmm45a
EXPNO	:	1
PROCNO	:	1

*** Acquisition Parameters ***

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O1	:	2117.60 Hz
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SOLVENT	:	CDCl3
SW	:	16.0495 ppm

*** Processing Parameters ***

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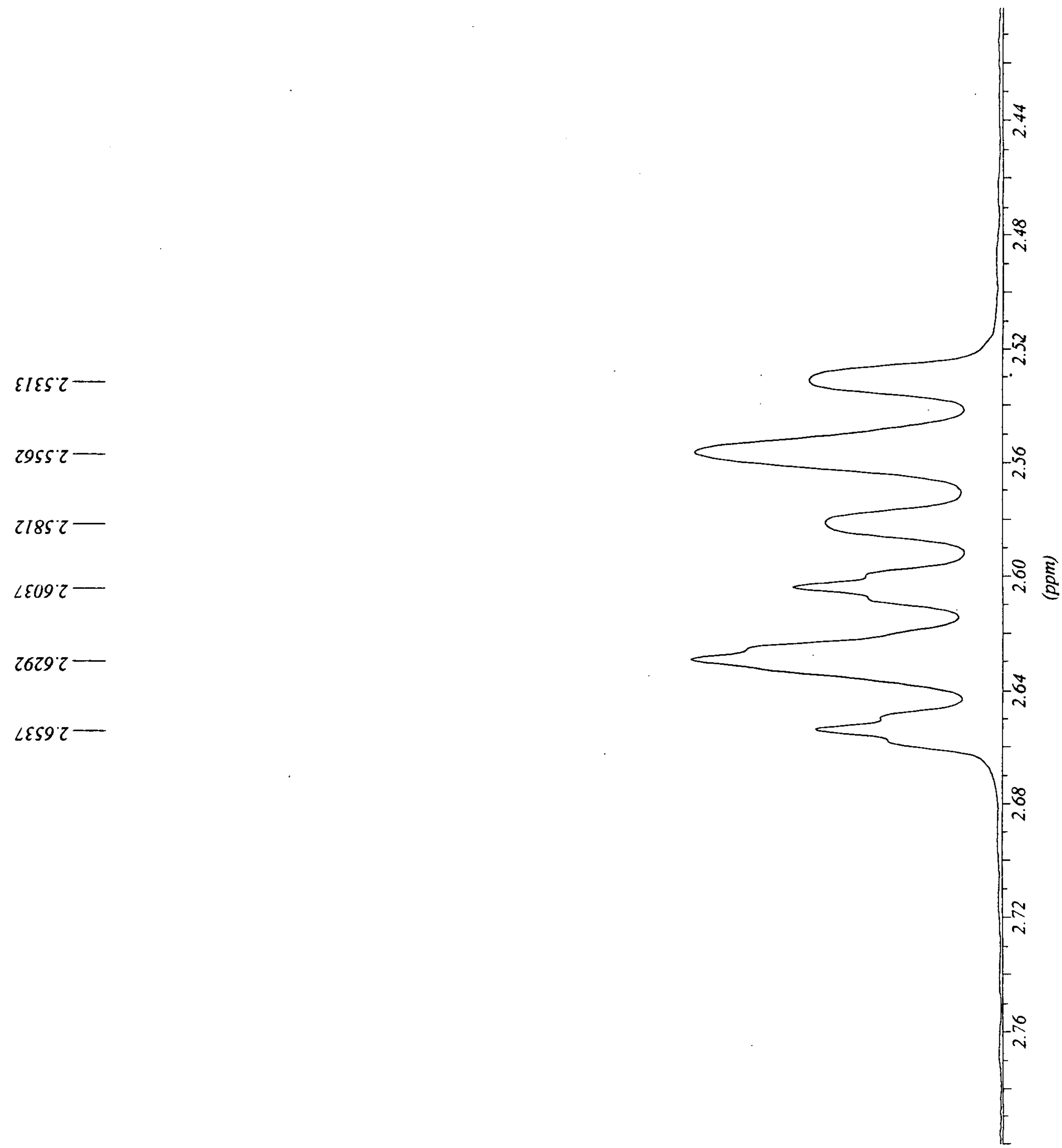
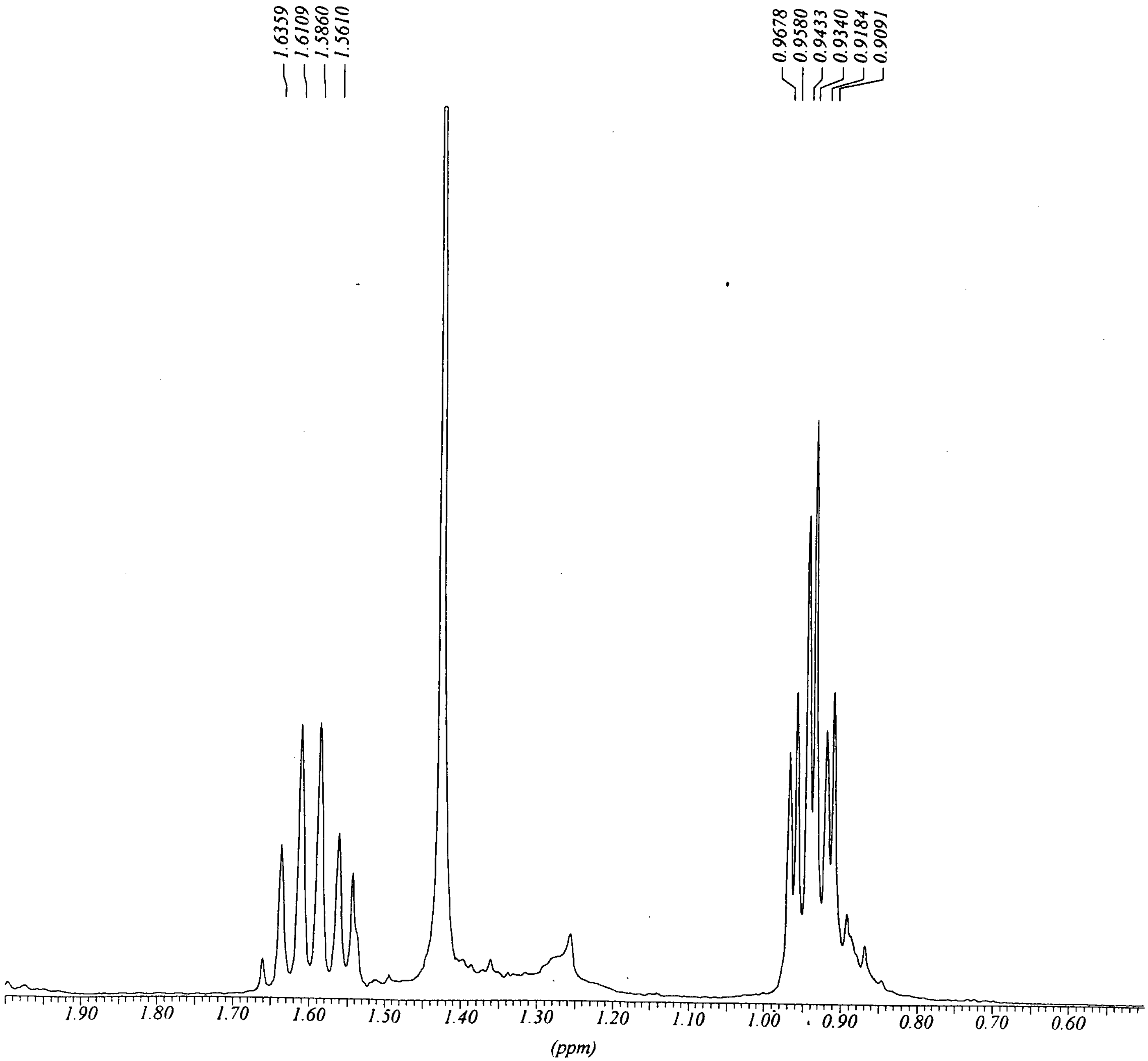
*** 1D NMR Plot Parameters ***

SOLVENT	:	?





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PROBHD	:	5 mm QNP 1H/13C/31 19F -
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SOLVENT	:	CDCl3
SW	:	16.0495 ppm

*** Processing Parameters ***

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*** 1D NMR Plot Parameters ***

SOLVENT	:	?

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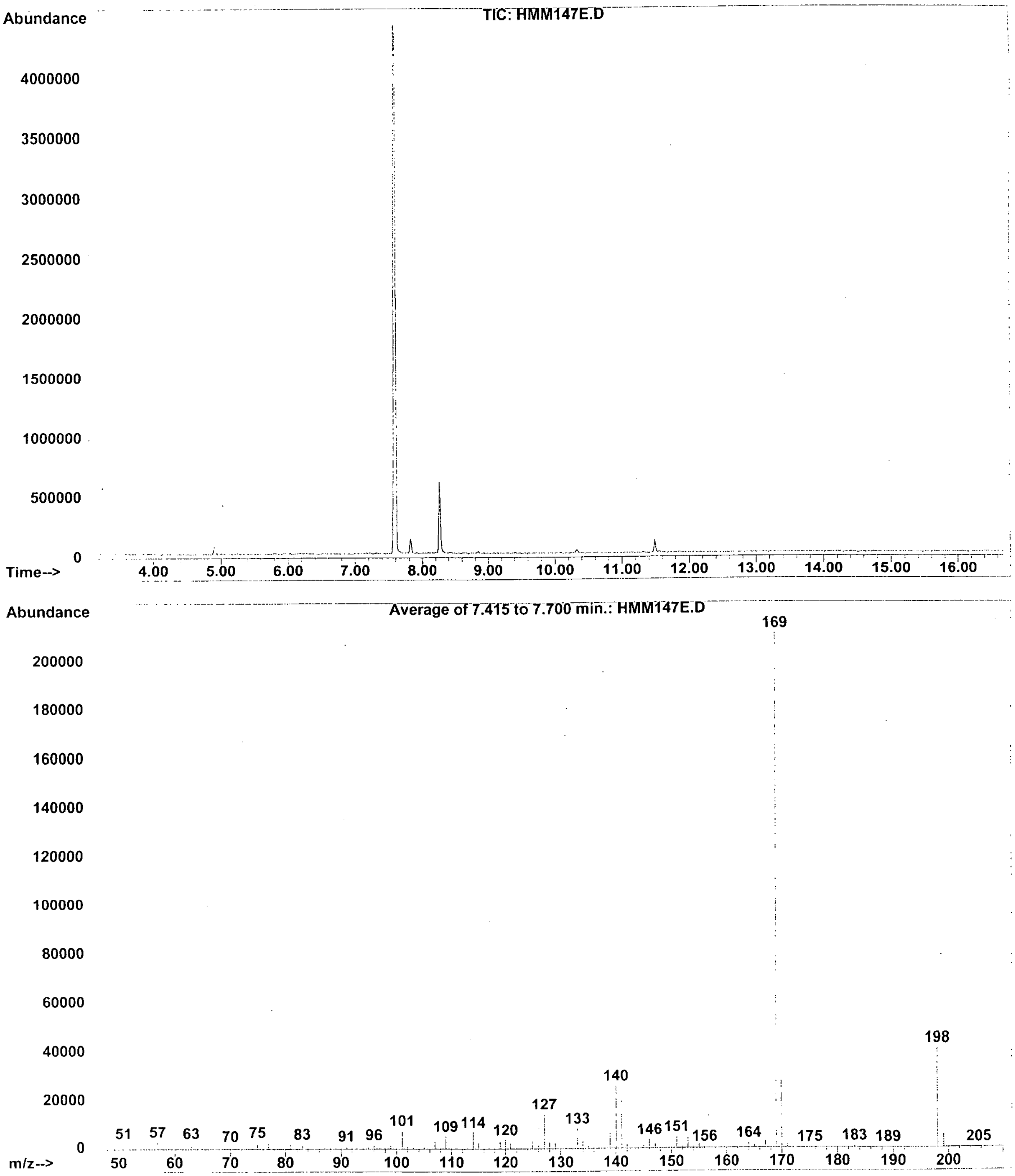

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PULPROG	:	zg30
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SOLVENT	:	CDCl3
SW	:	266.2581 ppm

*** Processing Parameters ***

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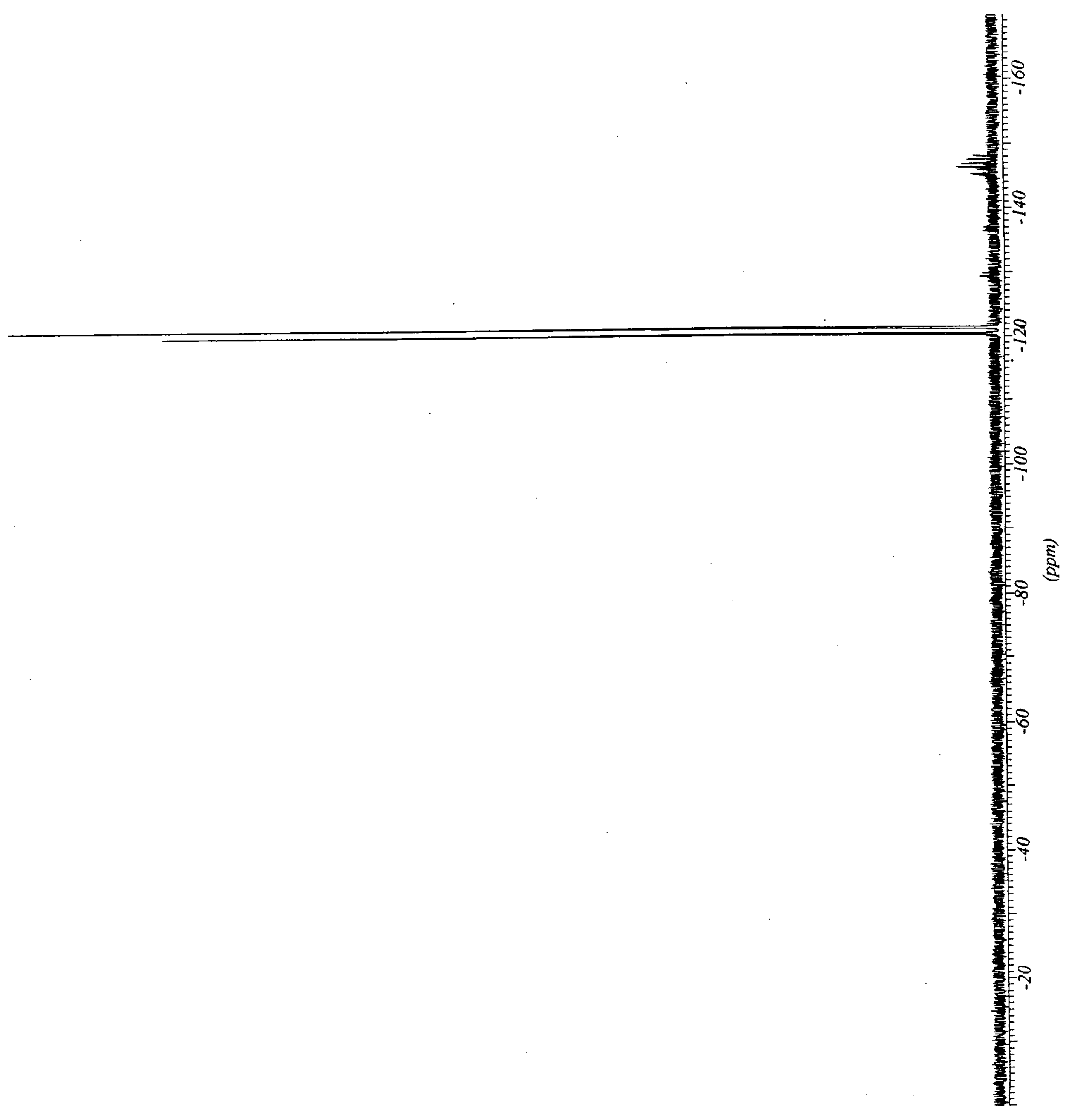
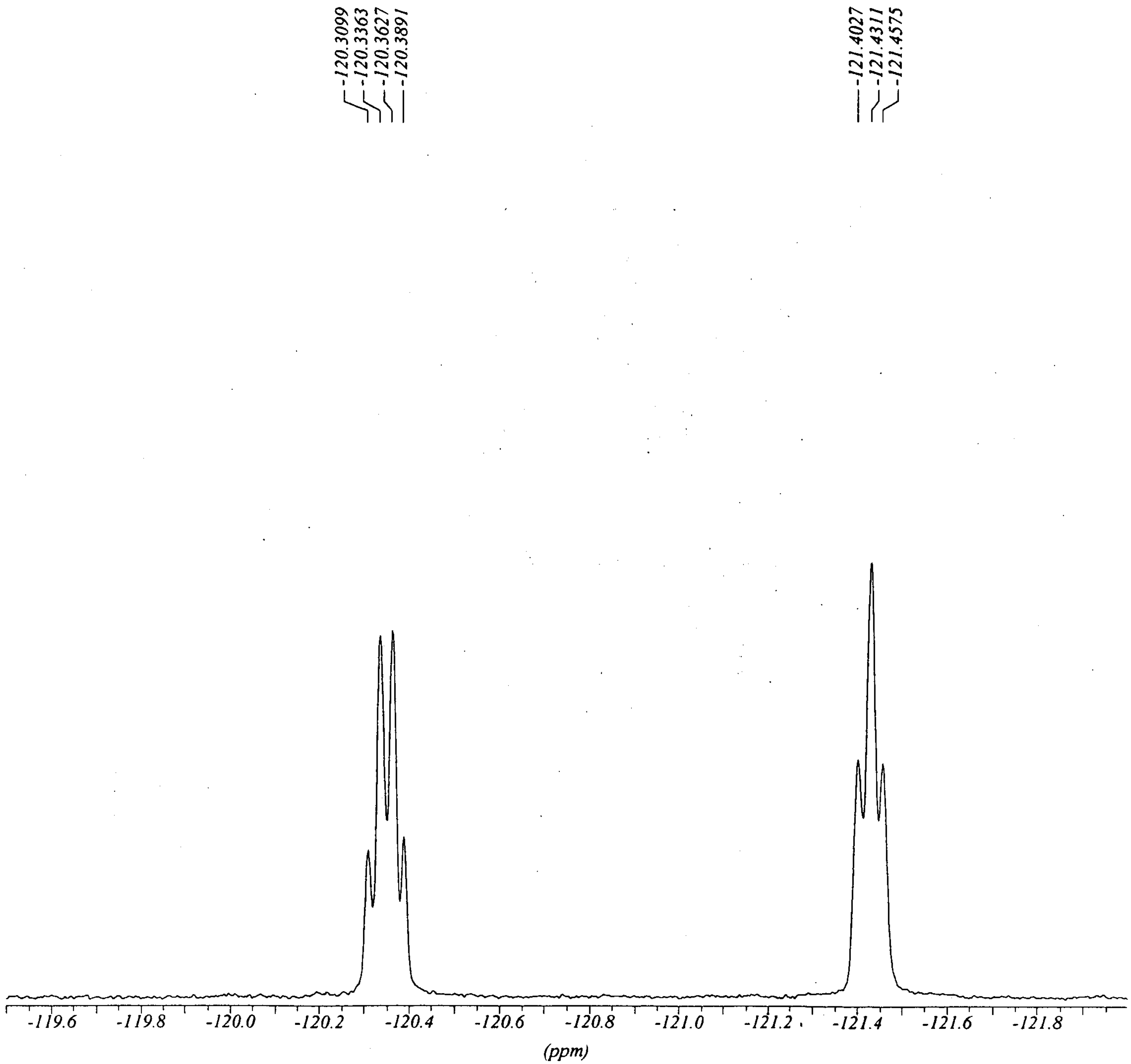
*** 1D NMR Plot Parameters ***

SOLVENT	:	?





*** Current Data Parameters ***

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PROCNO	:	1

*** Acquisition Parameters ***

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O1	:	-16944.27 Hz
PROBHD	:	5 mm QNP 1H/13C/31P/19F -
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SOLVENT	:	CDCl3
SW	:	266.2581 ppm

*** Processing Parameters ***

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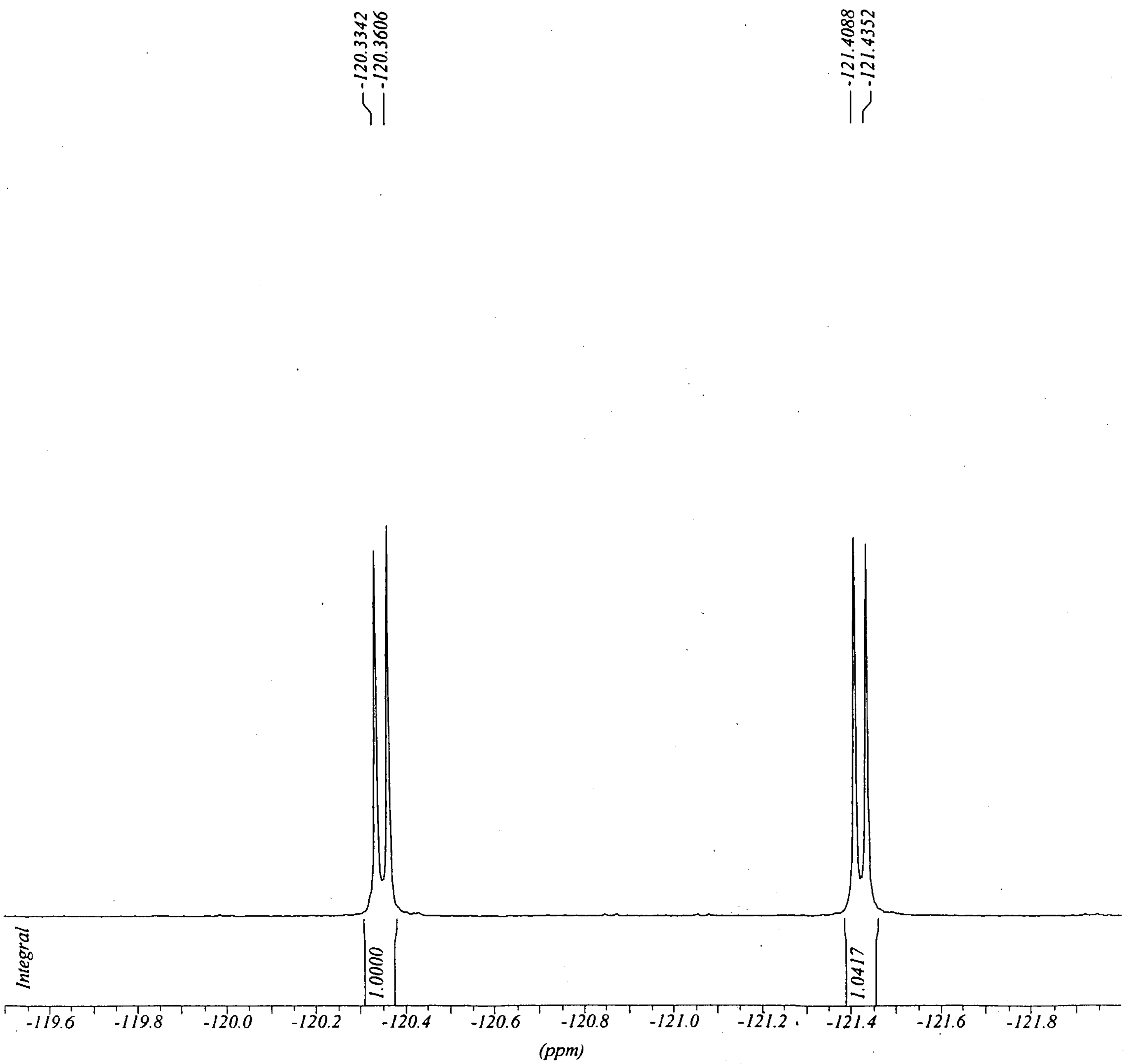
SI	:	131072

*** 1D NMR Plot Parameters ***

SOLVENT	:	?



*** Current Data Parameters ***

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PROCNO	:	1

*** Acquisition Parameters ***

AQ_mod	:	qsim
AUNM	:	au_zg
BF1	:	282.4044810 MHz
DATE_t	:	11:27:15
DATE_d	:	Dec 04 2001
DS	:	2
FW	:	90000.00 Hz
NS	:	16
NUC1	:	19F
O1	:	-16944.27 Hz
PROBHD	:	5 mm QNP 1H/13C/31P F -
PULPROG	:	zgdc30
RG	:	5792.6000977
SOLVENT	:	CDCl3
SW	:	266.2581 ppm

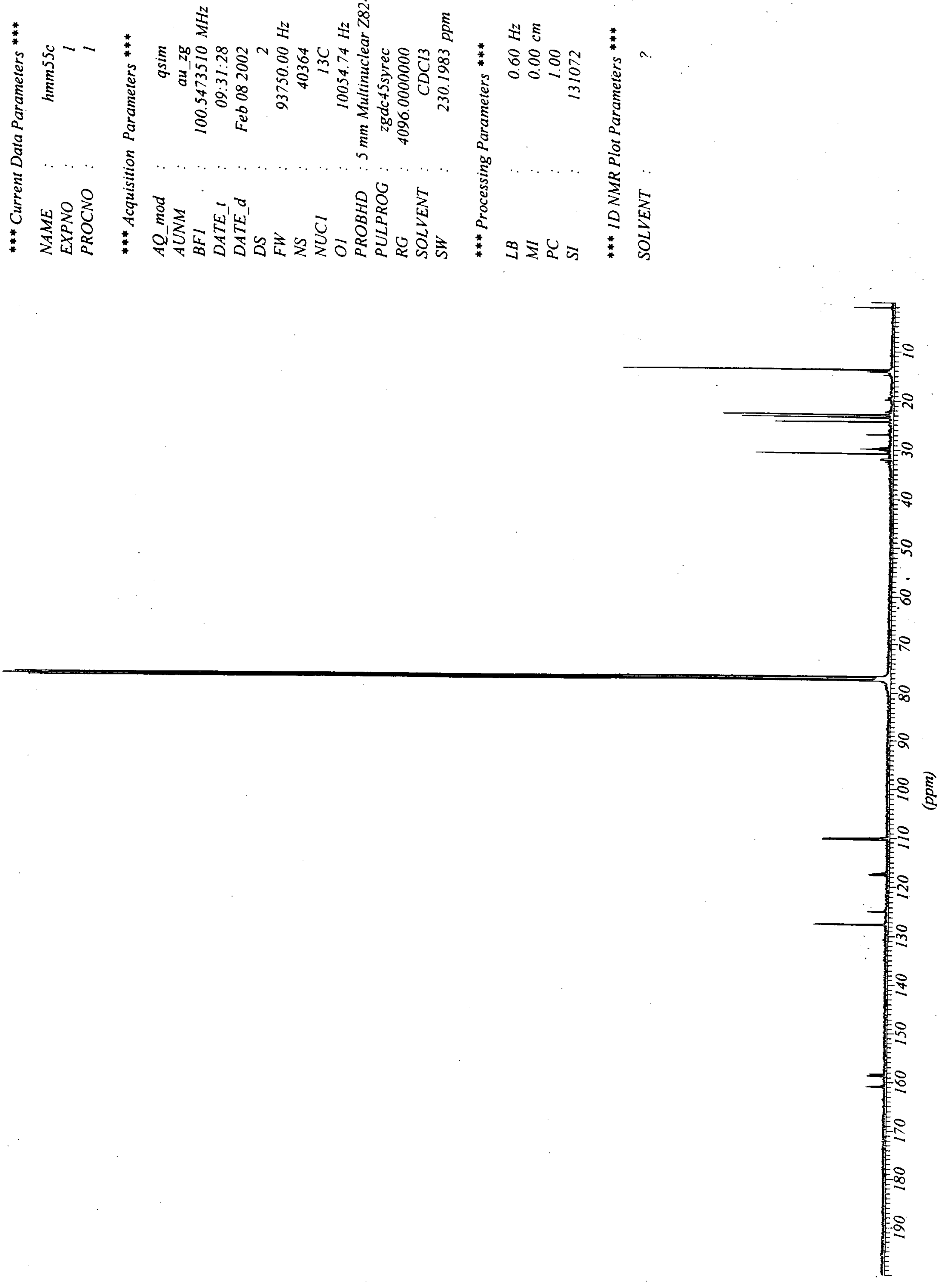
*** Processing Parameters ***

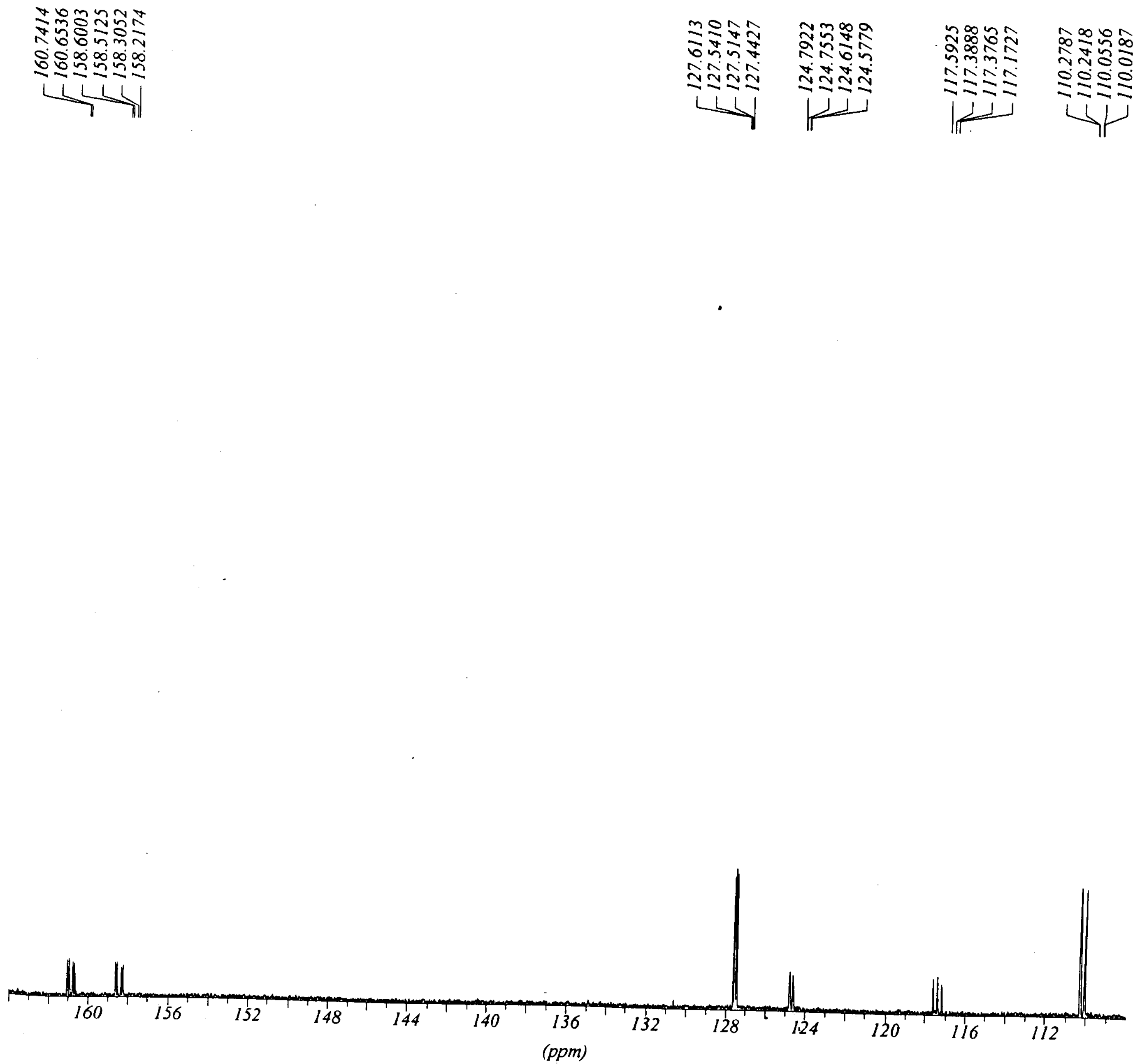
LB	:	1.00 Hz
MI	:	0.00 cm
PC	:	1.00
SI	:	131072

*** 1D NMR Plot Parameters ***

SOLVENT	:	?

20


*** Current Data Parameters ***
NAME : hmm55c
EXPNO : 1
PROCNO : 1
*** Acquisition Parameters ***
AQ_mod : qsim
AUNM : au_zg
BF1 : 100.5473510 MHz
DATE_t : 09:31:28
DATE_d : Feb 08 2002
DS : 2
FW : 93750.00 Hz
NS : 40364
NUC1 : 13C
O1 : 10054.74 Hz
PROBHD : 5 mm Multinuclear Z824
PULPROG : zgdc45syrec
RG : 4096.0000000
SOLVENT : CDCl3
SW : 230.1983 ppm
*** Processing Parameters ***
LB : 0.60 Hz
MI : 0.00 cm
PC : 1.00
SI : 131072
*** 1D NMR Plot Parameters ***
SOLVENT : ?
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
(ppm)



*** Current Data Parameters ***

NAME	:	hmm55c
EXPNO	:	1
PROCNO	:	1

*** Acquisition Parameters ***

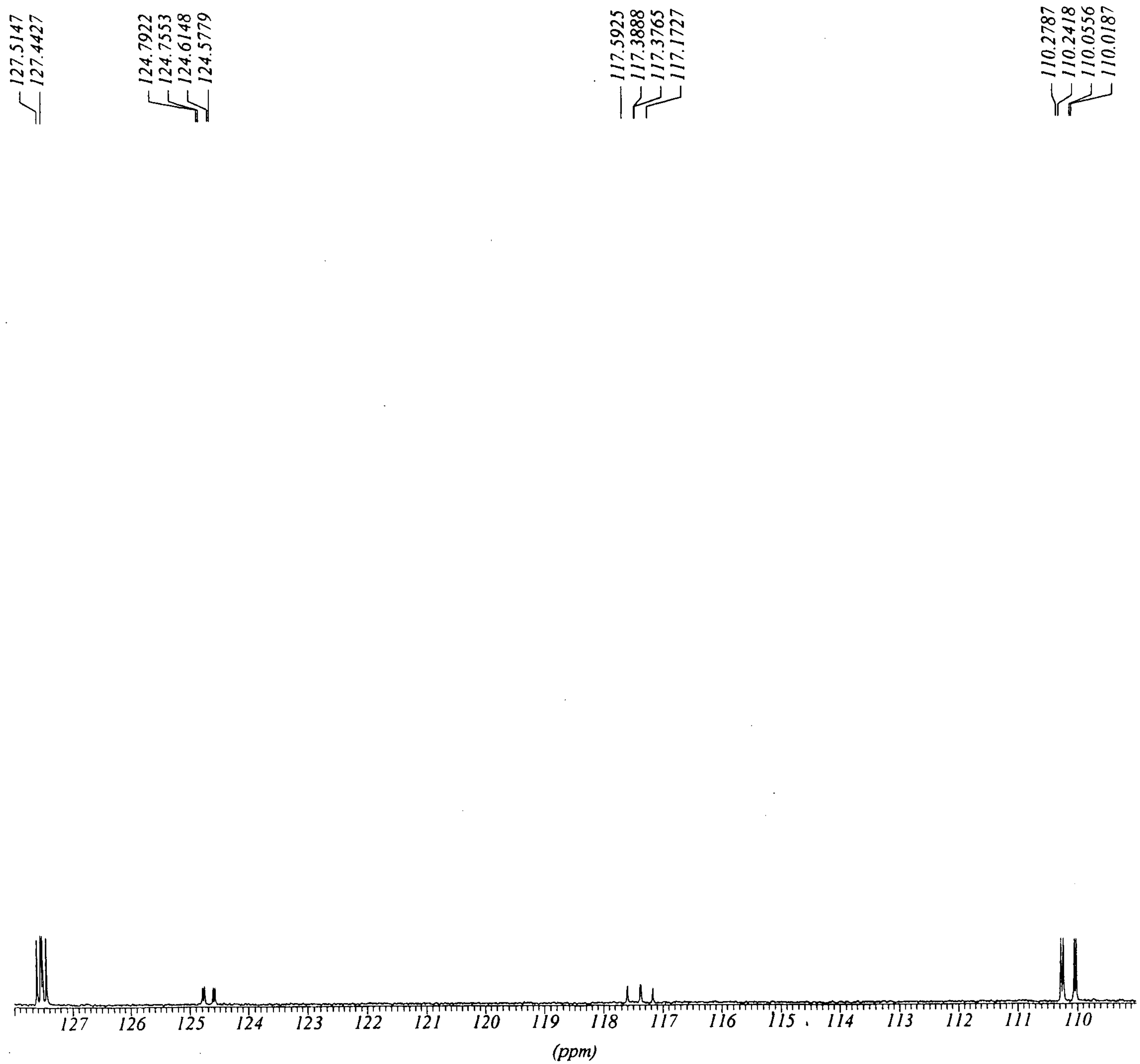
AQ_mod	:	qsim
AUNM	:	au_zg
BF1	:	100.5473510 MH
DATE_t	:	09:31:28
DATE_d	:	Feb 08 2002
DS	:	2
FW	:	93750.00 Hz
NS	:	40364
NUC1	:	13C
O1	:	10054.74 Hz
PROBHD	:	5 mm Multinuclear Z8
PULPROG	:	zgdc45syrec
RG	:	4096.0000000
SOLVENT	:	CDCl3
SW	:	230.1983 ppm

*** Processing Parameters ***

LB	:	0.60 Hz
MI	:	0.00 cm
PC	:	1.00
SI	:	131072

*** 1D NMR Plot Parameters ***

SOLVENT	:	?



*** Current Data Parameters ***

NAME	:	hmm55c
EXPNO	:	1
PROCNO	:	1

*** Acquisition Parameters ***

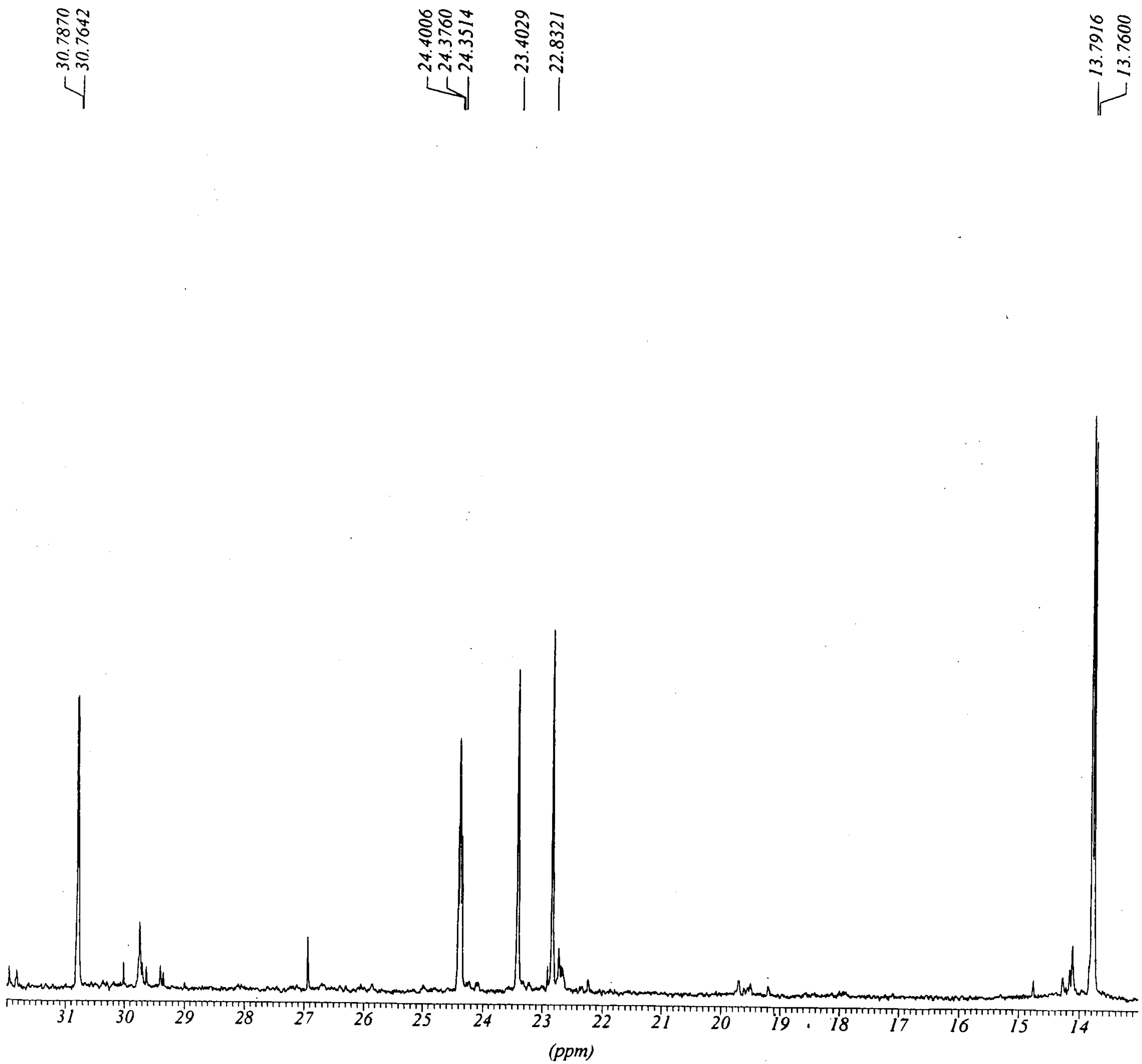
AQ_mod	:	qsim
AUNM	:	au_zg
BF1	:	100.5473510 MHz
DATE_t	:	09:31:28
DATE_d	:	Feb 08 2002
DS	:	2
FW	:	93750.00 Hz
NS	:	40364
NUC1	:	13C
O1	:	10054.74 Hz
PROBHD	:	5 mm Multinuclear Z82 '001
PULPROG	:	zgdc45syrec
RG	:	4096.0000000
SOLVENT	:	CDCl3
SW	:	230.1983 ppm

*** Processing Parameters ***

LB	:	0.60 Hz
MI	:	0.00 cm
PC	:	1.00
SI	:	131072

*** 1D NMR Plot Parameters ***

SOLVENT	:	?



*** Current Data Parameters ***

NAME	:	hmm55c
EXPNO	:	1
PROCNO	:	1

*** Acquisition Parameters ***

AQ_mod	:	qsim
AUNM	:	au_zg
BF1	:	100.5473510 MHz
DATE_t	:	09:31:28
DATE_d	:	Feb 08 2002
DS	:	2
FW	:	93750.00 Hz
NS	:	40364
NUC1	:	13C
O1	:	10054.74 Hz
PROBHD	:	5 mm Multinuclear Z82 0001
PULPROG	:	zgdc45syrec
RG	:	4096.0000000
SOLVENT	:	CDCl3
SW	:	230.1983 ppm

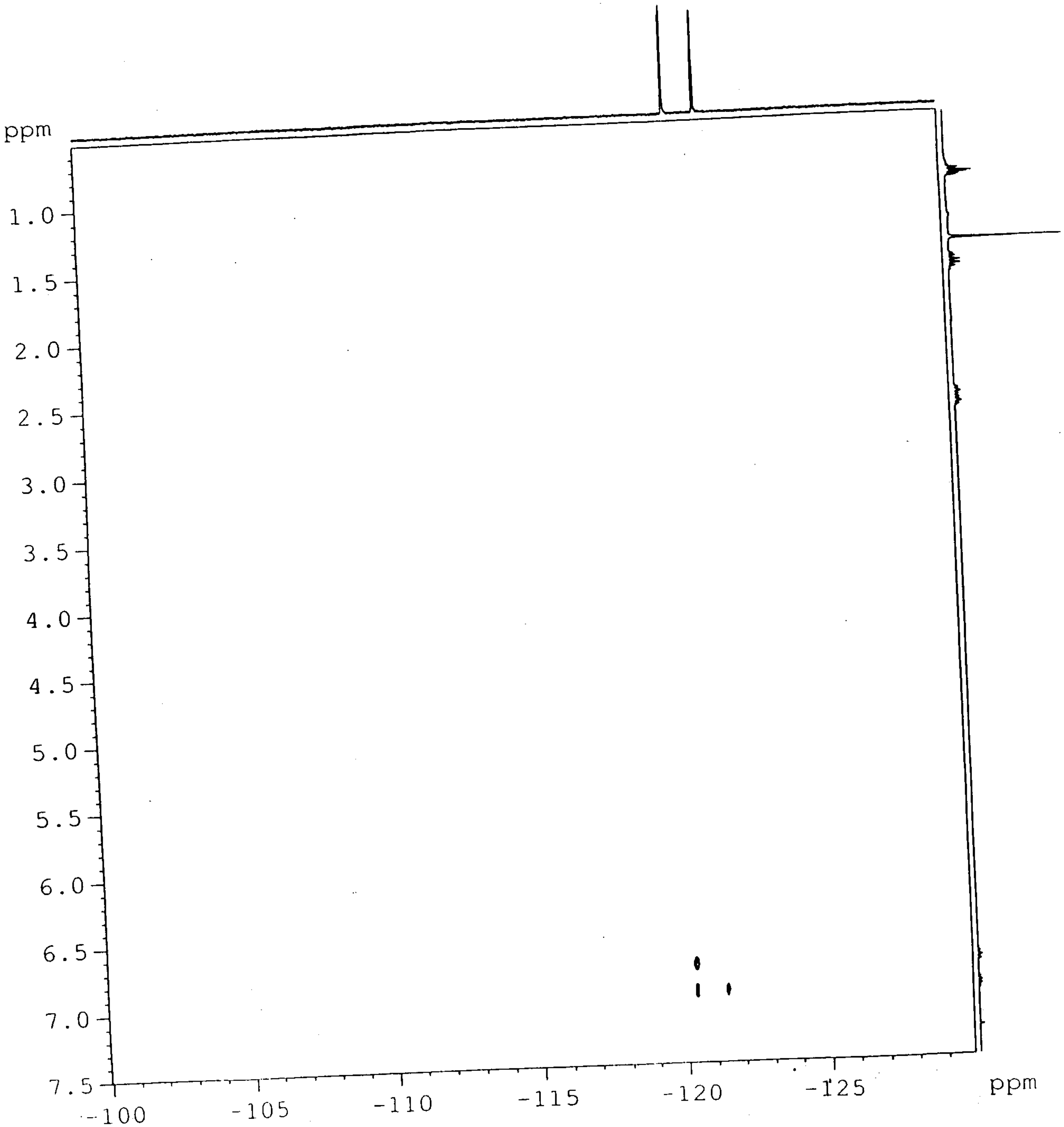
*** Processing Parameters ***

LB	:	0.60 Hz
MI	:	0.00 cm
PC	:	1.00
SI	:	131072

*** 1D NMR Plot Parameters ***

SOLVENT	:	?

Proton Decoupled Fluorine Spectrum Used



Current Data Parameters

NAME	hmm45b
EXPNO	4
PROCNO	1

F2 – Acquisition Parameters

Date_	20011204
Time	16.24
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	hxcoqf
TD	4096
SOLVENT	CDCl3
NS	8
DS	16
SWH	8445.946 Hz
FIDRES	2.061999 Hz
AQ	0.2425332 sec
RG	13004
DW	59.200 usec
DE	6.00 usec
TE	300.0 K
CNST2	7.0000000
CNST11	3.0000000
d0	0.00000300 sec
D1	3.00000000 sec
d2	0.07142857 sec
d3	0.04761905 sec
d11	0.03000000 sec
d12	0.00002000 sec
IN0	0.00023799 sec
MCREST	0.00000000 sec
MCWRK	0.03000000 sec

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

NUC1	19F
P1	5.90 usec
p2	11.80 usec
PL1	3.00 dB
SFO1	282.3720045 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
P3	8.30 usec
PCPD2	80.00 usec
PL2	3.50 dB
PL12	16.00 dB
SFO2	300.1312005 MHz

F1 – Acquisition parameters

ND0	2
TD	256
SFO1	300.1312 MHz
FIDRES	8.206839 Hz
SW	7.000 ppm

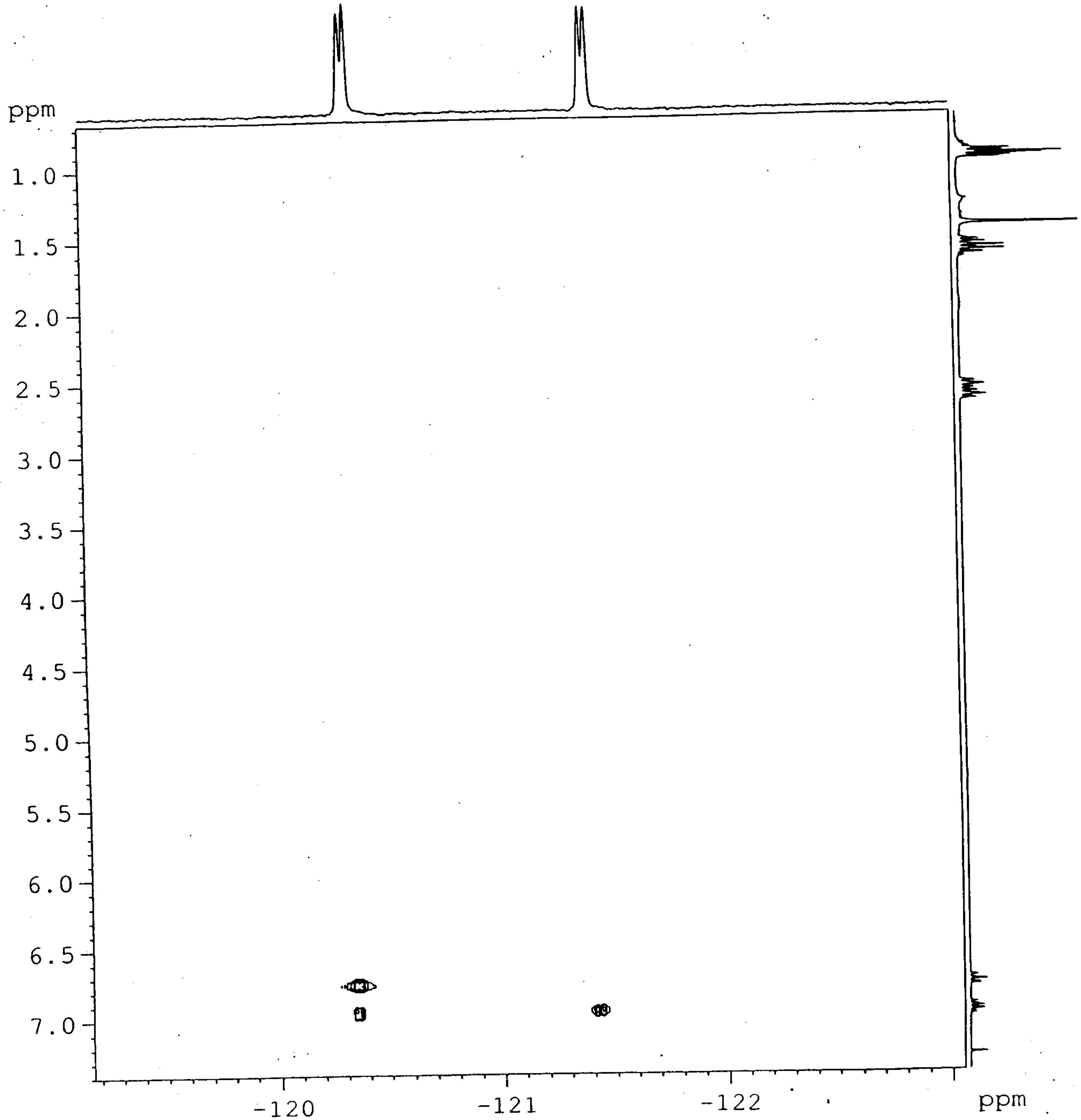
F2 – Processing parameters

SI	4096
SF	282.4044516 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00

F1 – Processing parameters

SI	1024
MC2	QF
SF	300.1300000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

Proton Decoupled Fluorine Spectrum Used



Current Data Parameters

NAME	hmm45b
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20011204
Time	16.24
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	hxcoqf
TD	4096
SOLVENT	CDCl3
NS	8
DS	16
SWH	8445.946 Hz
FIDRES	2.061999 Hz
AQ	0.2425332 sec
RG	13004
DW	59.200 usec
DE	6.00 usec
TE	300.0 K
CNST2	7.0000000
CNST11	3.0000000
d0	0.00000300 sec
D1	3.00000000 sec
d2	0.07142857 sec
d3	0.04761905 sec
d11	0.03000000 sec
d12	0.00002000 sec
IN0	0.00023799 sec
MCREST	0.00000000 sec
MCWRK	0.03000000 sec

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

NUC1	19F
P1	5.90 usec
p2	11.80 usec
PL1	3.00 dB
SFO1	282.3720045 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
P3	8.30 usec
PCPD2	80.00 usec
PL2	3.50 dB
PL12	16.00 dB
SFO2	300.1312005 MHz

F1 - Acquisition parameters

ND0	2
TD	256
SFO1	300.1312 MHz
FIDRES	8.206839 Hz
SW	7.000 ppm

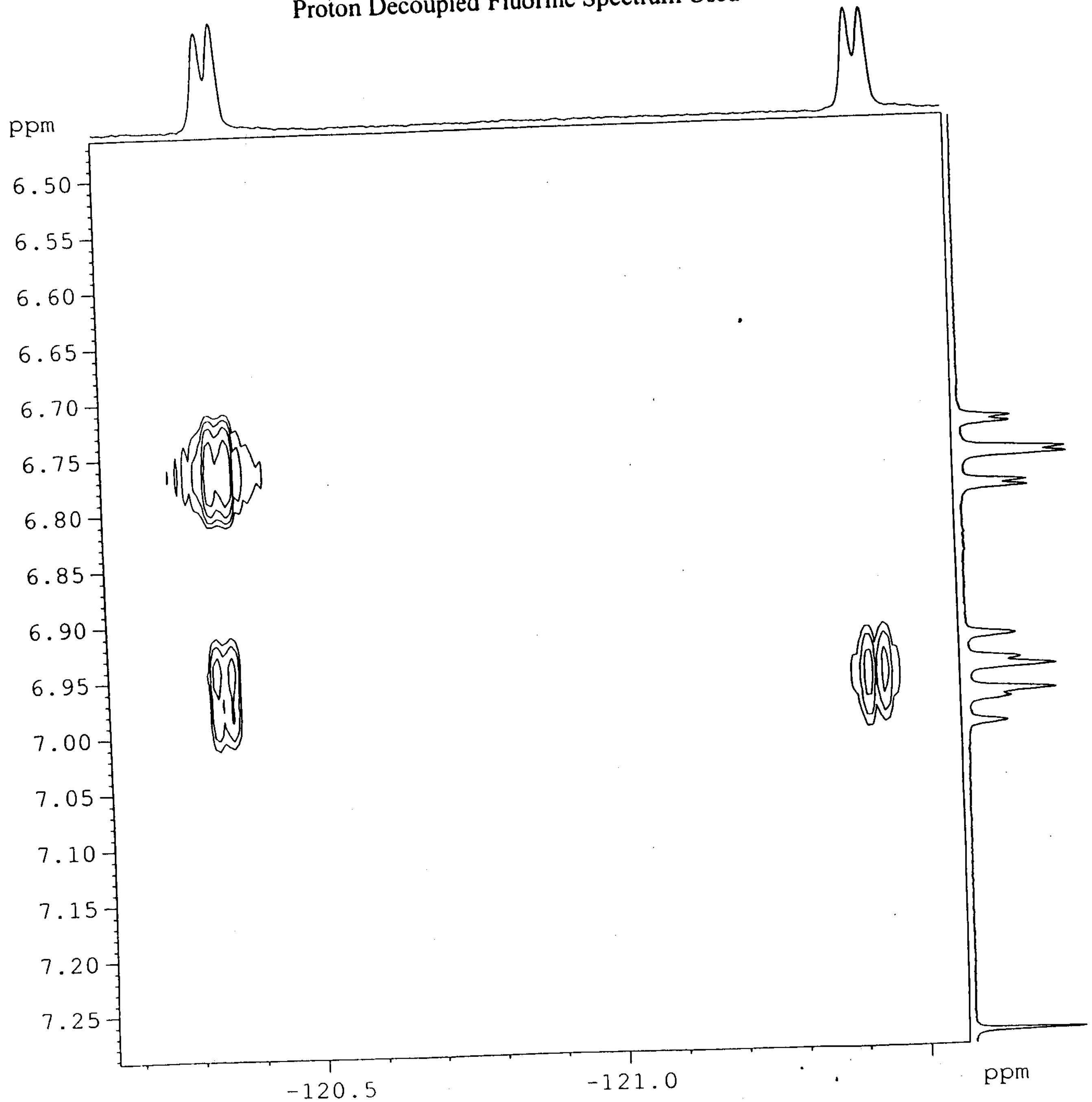
F2 - Processing parameters

SI	4096
SF	282.4044516 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00

F1 - Processing parameters

SI	1024
MC2	QF
SF	300.1300000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

Proton Decoupled Fluorine Spectrum Used



Current Data Parameters
NAME hmm45b
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011204
Time 16.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG hxcoqf
TD 4096
SOLVENT CDCl3
NS 8
DS 16
SWH 8445.946 Hz
FIDRES 2.061999 Hz
AQ 0.2425332 sec
RG 13004
DW 59.200 usec
DE 6.00 usec
TE 300.0 K
CNST2 7.0000000
CNST11 3.0000000
d0 0.00000300 sec
D1 3.00000000 sec
d2 0.07142857 sec
d3 0.04761905 sec
d11 0.03000000 sec
d12 0.00002000 sec
IN0 0.00023799 sec
MCREST 0.00000000 sec
MCWRK 0.03000000 sec

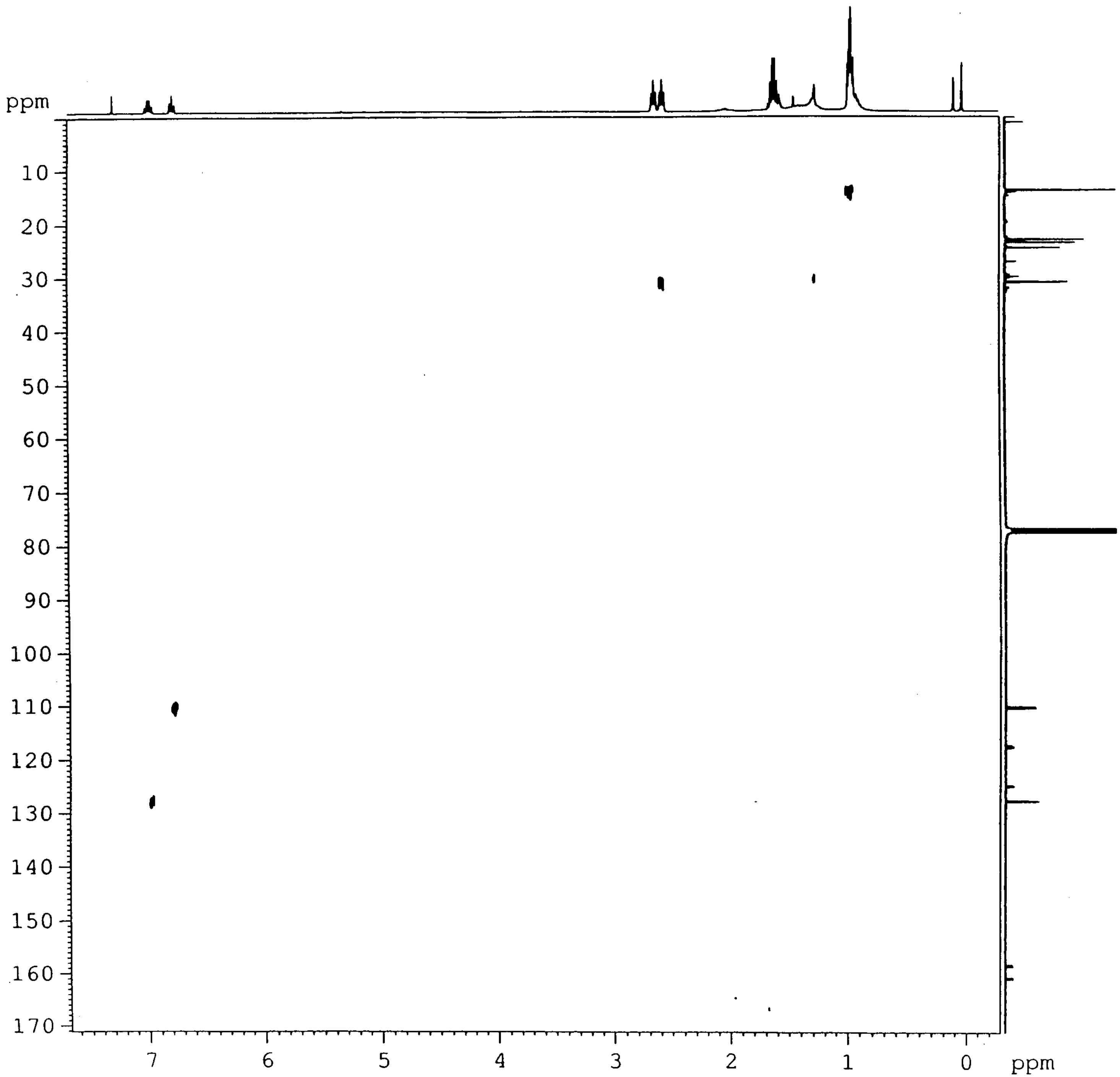
======== CHANNEL f1 ========
NUC1 19F
P1 5.90 usec
p2 11.80 usec
PL1 3.00 dB
SFO1 282.3720045 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 8.30 usec
PCPD2 80.00 usec
PL2 3.50 dB
PL12 16.00 dB
SFO2 300.1312005 MHz

F1 - Acquisition parameters
ND0 2
TD 256
SFO1 300.1312 MHz
FIDRES 8.206839 Hz
SW 7.000 ppm

F2 - Processing parameters
SI 4096
SF 282.4044516 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 QF
SF 300.1300000 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0



Current Data Parameters
NAME hmm55d
EXPNO 4
PROCNO 1

F2 – Acquisition Parameters
Date_ 20020215
Time 12.28
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG inv4gs
TD 2048
SOLVENT CDCl3
NS 4
DS 8
SWH 4032.258 Hz
FIDRES 1.968876 Hz
AQ 0.2540020 sec
RG 9195.2
DW 124.000 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
d0 0.00000300 sec
D1 1.50000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
d13 0.00000300 sec
D16 0.00025000 sec
d20 0.00242528 sec
IN0 0.00002240 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
p2 23.00 usec
PL1 0.00 dB
SFO1 399.8718118 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 13C
P3 7.50 usec
PCPD2 65.00 usec
PL2 -3.00 dB
PL12 16.00 dB
SFO2 100.5573516 MHz

====== GRADIENT CHANNEL =====
P16 1000.00 usec

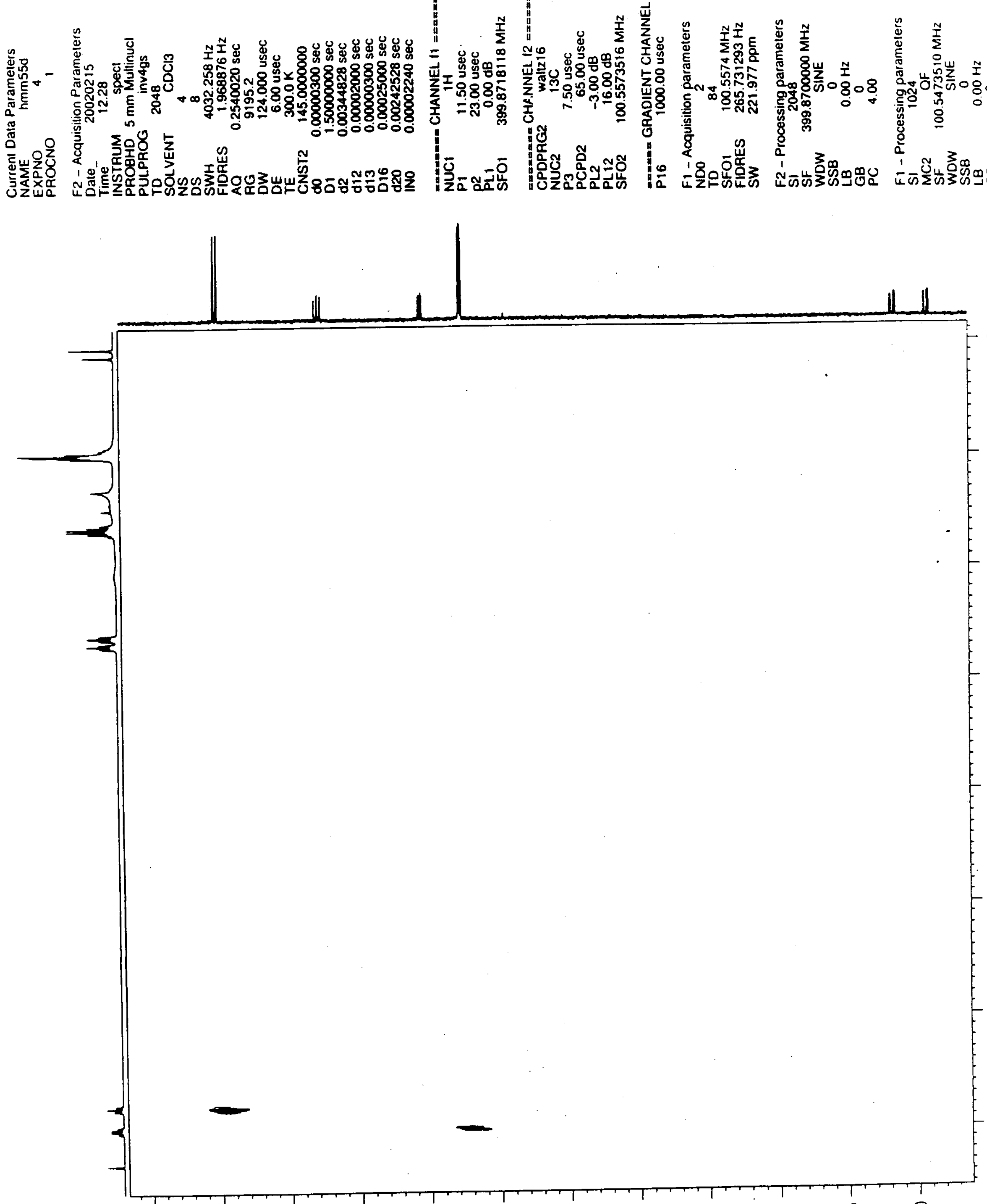
F1 – Acquisition parameters
ND0 2
TD 84
SFO1 100.5574 MHz
FIDRES 265.731293 Hz
SW 221.977 ppm

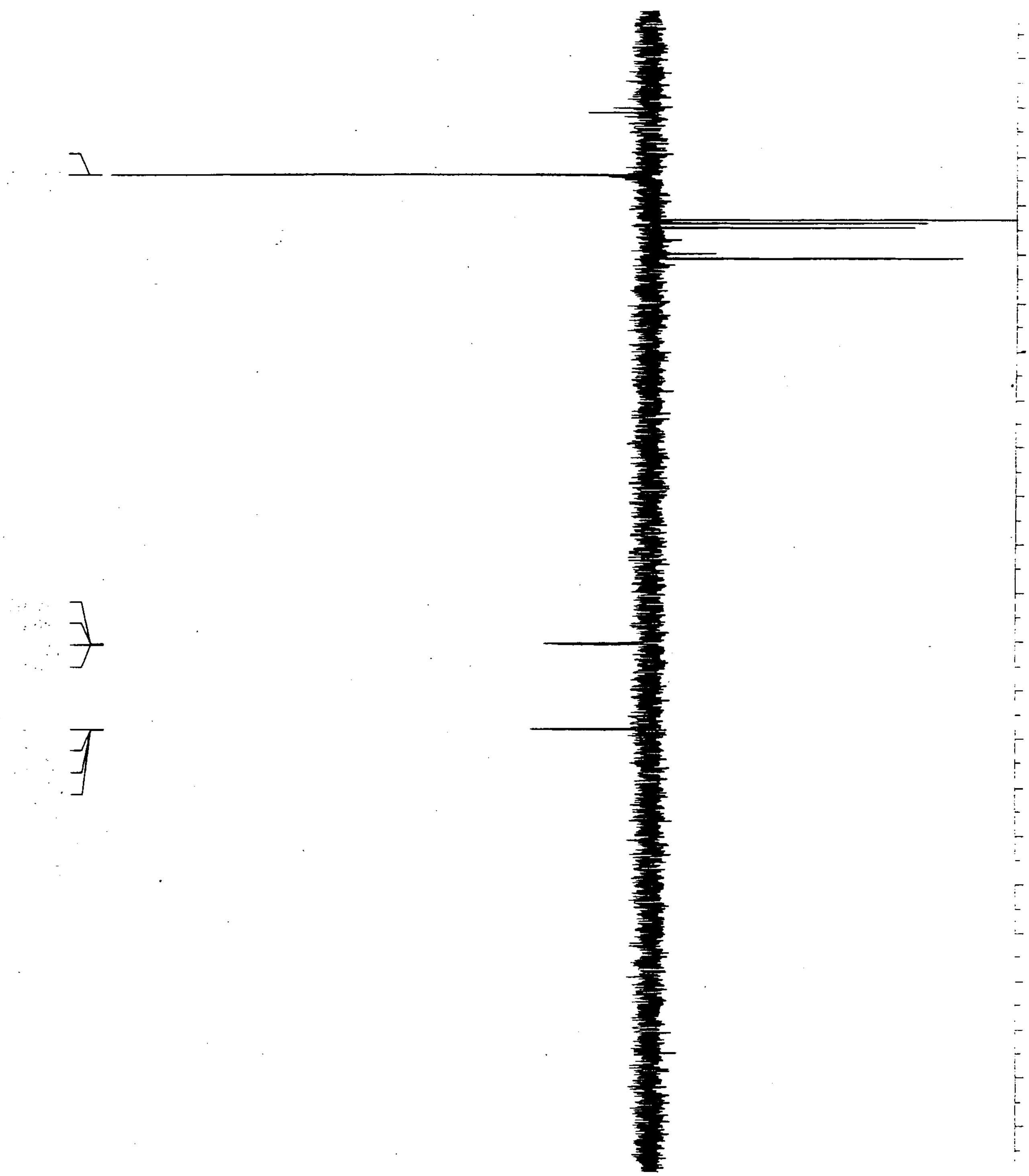
F2 – Processing parameters
SI 2048
SF 399.8700000 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 4.00

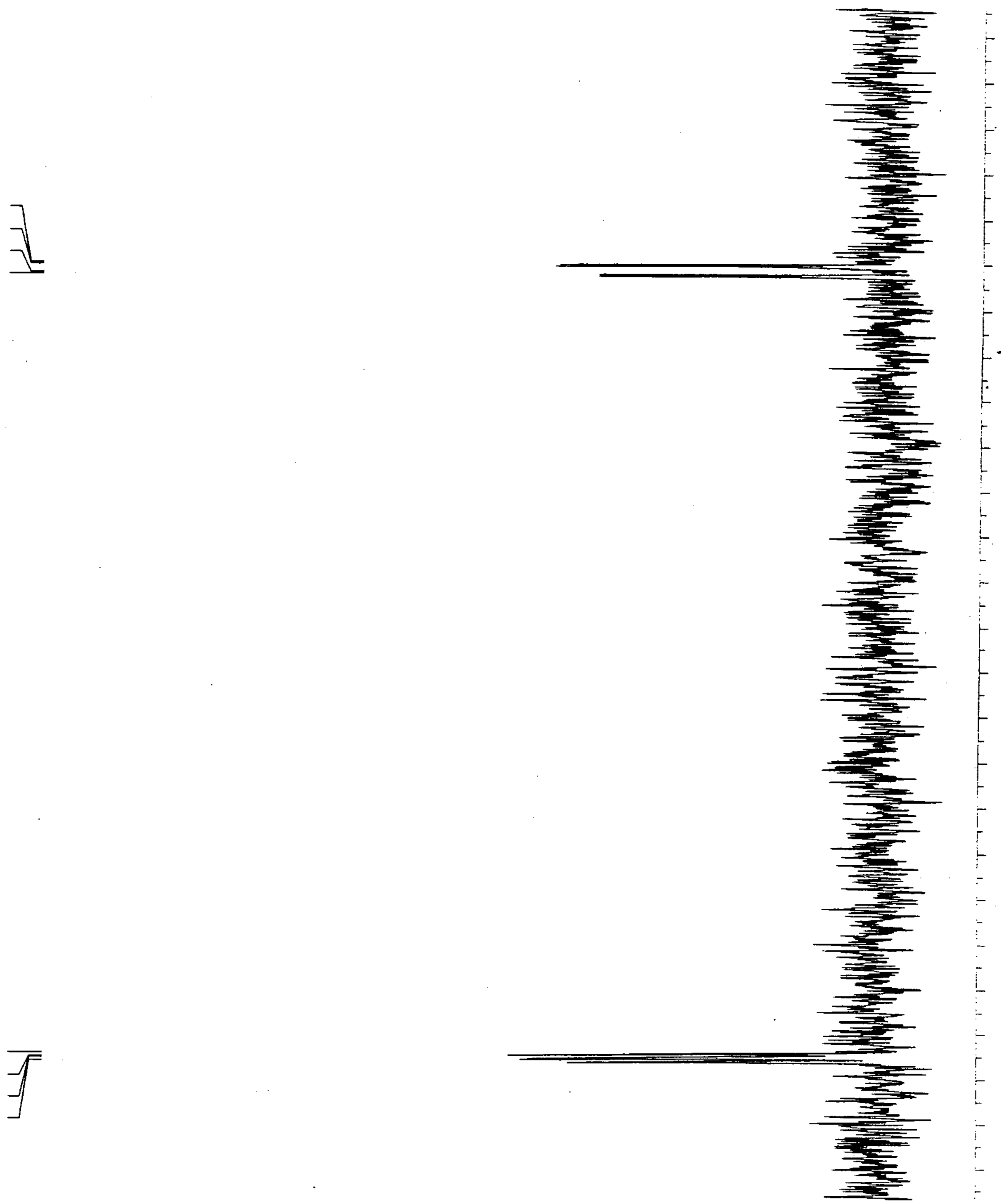
F1 – Processing parameters
SI 1024
MC2 QF
SF 100.5473510 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

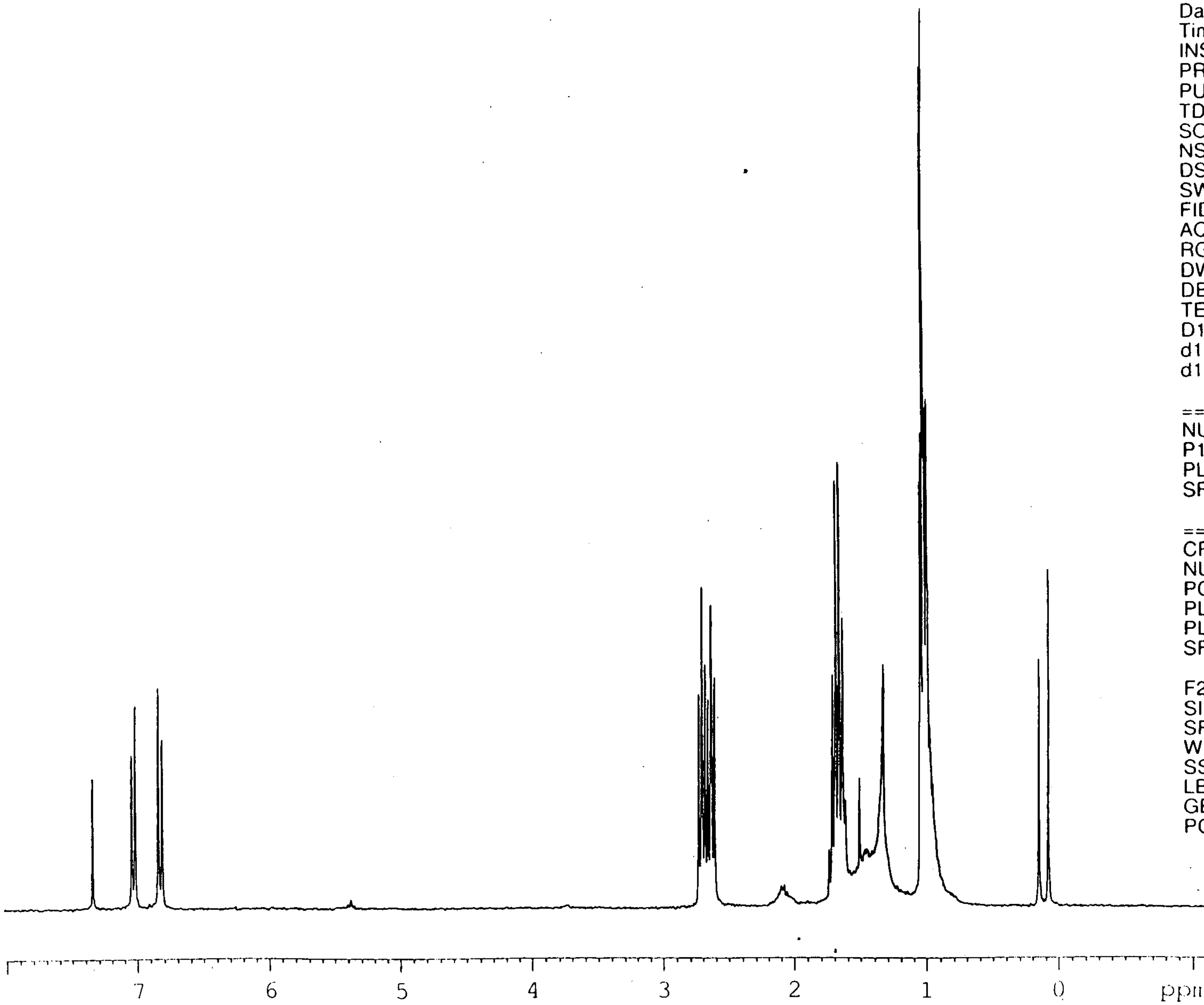
27

28


Current Data Parameters
NAME hmm55d
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020215
Time 12.28
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG inv4gs
TD 2048
SOLVENT CDCl3
NS 4
DS 8
SWH 4032.258 Hz
FIDRES 1.968876 Hz
AQ 0.2540020 sec
RG 9195.2
DW 124.000 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
d0 0.00000300 sec
D1 1.50000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
d13 0.00000300 sec
D16 0.00025000 sec
d20 0.00242528 sec
IN0 0.00002240 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
p2 23.00 usec
PL1 0.00 dB
SFO1 399.8718118 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 13C
P3 7.50 usec
PCPD2 65.00 usec
PL2 -3.00 dB
PL12 16.00 dB
SFO2 100.5573516 MHz
====== GRADIENT CHANNEL =====
P16 1000.00 usec
F1 - Acquisition parameters
ND0 2
TD 84
SFO1 100.5574 MHz
FIDRES 265.731293 Hz
SW 221.977 ppm
F2 - Processing parameters
SI 2048
SF 399.8700000 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
F1 - Processing parameters
SI 1024
MC2 QF
SF 100.5473510 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
ppm
0
1
2
3
4
5
6
7
ppm
105
110
115
120
125
130
135
140
145
150
155
160







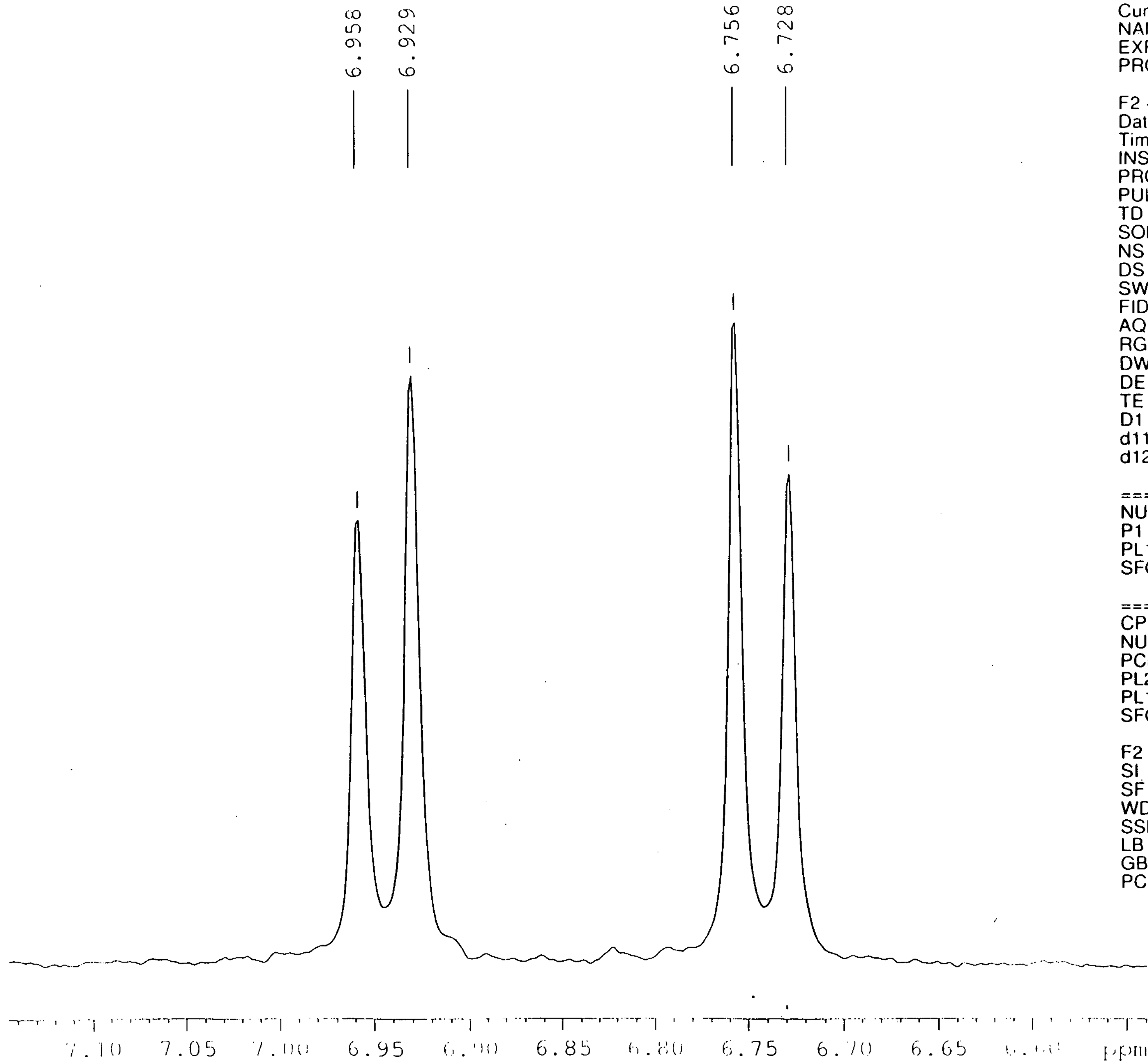
Current Data Parameters
NAME junkf
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020218
Time 14.26
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zghfigqn
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 6265.664 Hz
FIDRES 0.191213 Hz
AQ 2.6149364 sec
RG 1149.4
DW 79.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.30 usec
PL1 3.50 dB
SFO1 300.1318766 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 19F
PCPD2 80.00 usec
PL2 3.00 dB
PL12 25.64 dB
SFO2 282.3762408 MHz

F2 - Processing parameters
SI 16384
SF 300.1300000 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



Current Data Parameters

NAME	junkf
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20020218
Time	14.26
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zghfigqn
TD	32768
SOLVENT	CDCl3
NS	8
DS	2
SWH	6265.664 Hz
FIDRES	0.191213 Hz
AQ	2.6149364 sec
RG	1149.4
DW	79.800 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

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

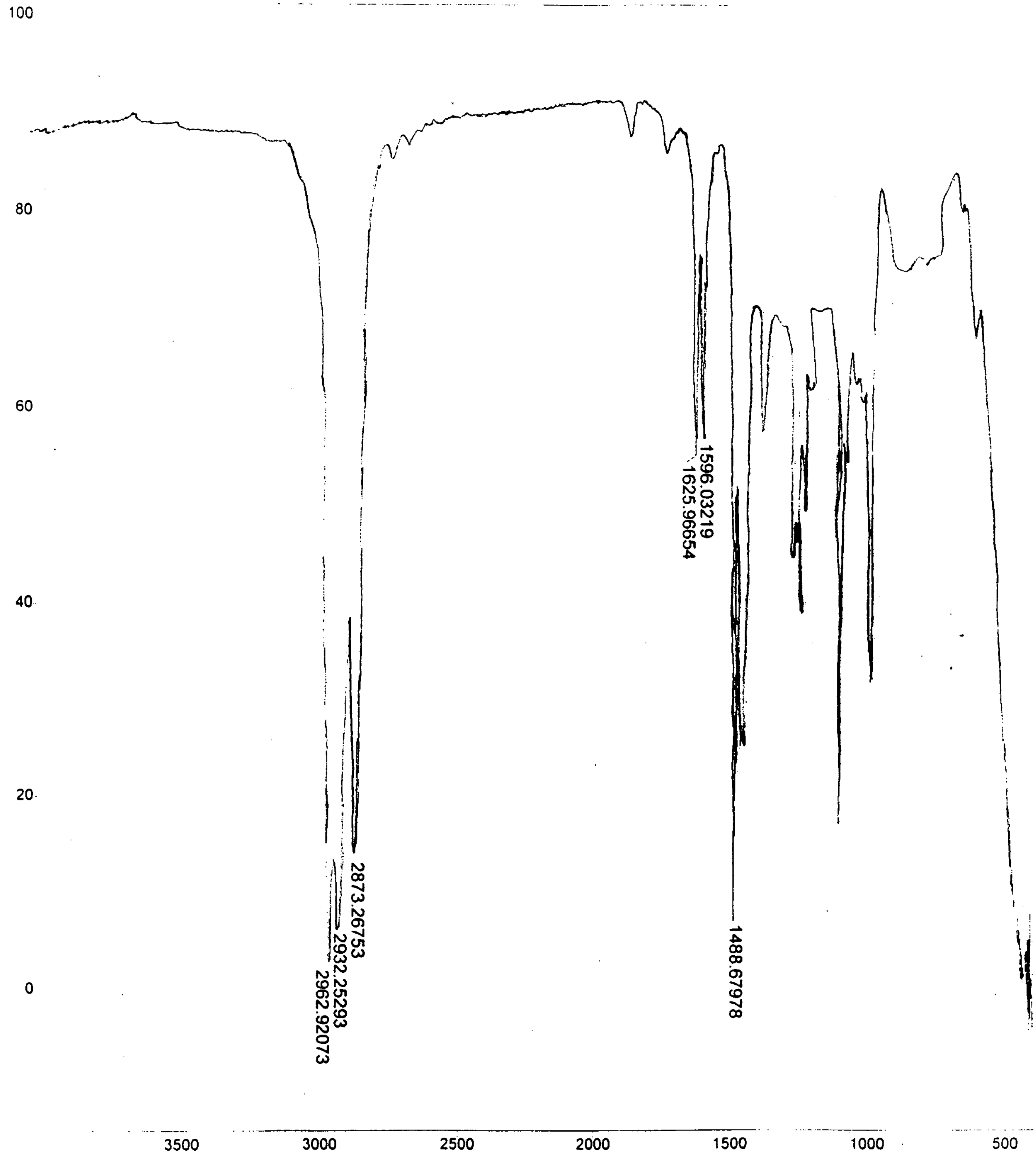
NUC1	1H
P1	8.30 usec
PL1	3.50 dB
SFO1	300.1318766 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	19F
PCPD2	80.00 usec
PL2	3.00 dB
PL12	25.64 dB
SFO2	282.3762408 MHz

F2 - Processing parameters

SI	16384
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00

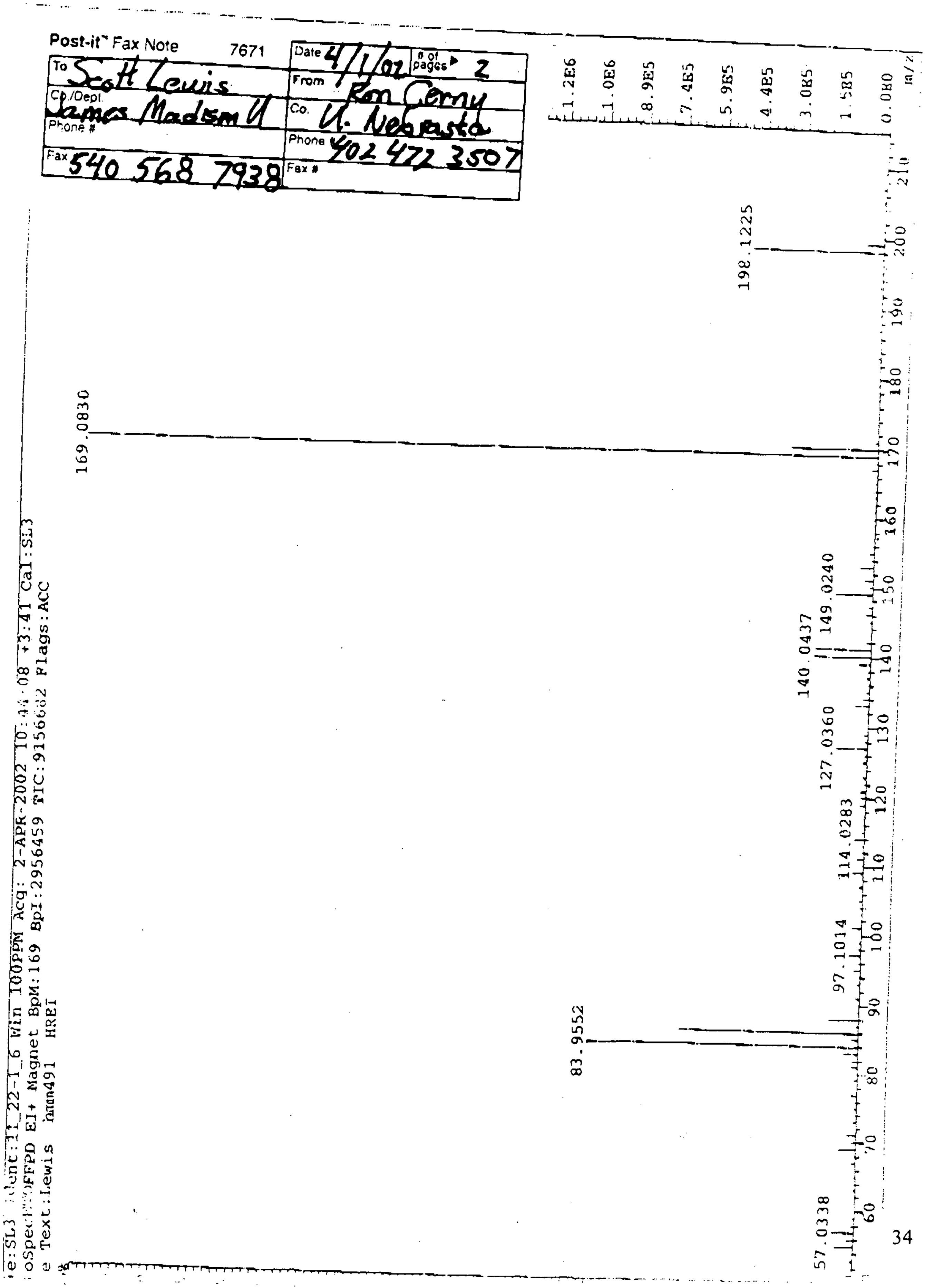


Transmittance / Wavenumber (cm-1)

Paged X-Zoom CURSOR

File # 1 = HMMPH1

2/26/2002 5:24 PM Res=4 cm-1

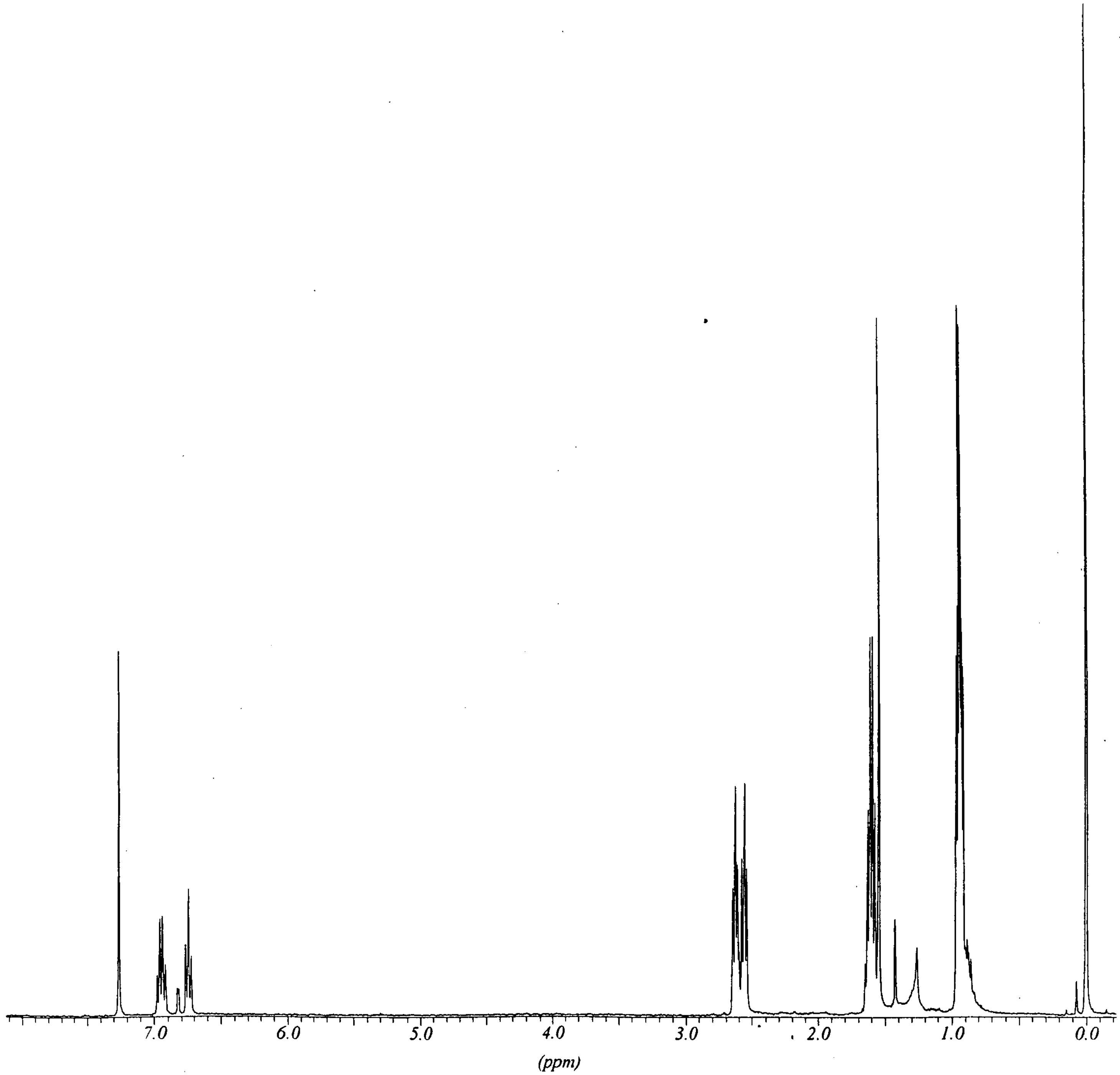

Post-it™ Fax Note 7671
Date 4/1/02
# of pages 2
To Scott Lewis
From Ron Cerny
Co./Dept. James Madison U
Co. U. Nebraska
Phone #
Phone 402 472 3507
Fax 540 568 7938
Fax #
File:SL3 Ident:31_22-1_6 Win 100PPM Acq: 2-APR-2002 10:44:08 +3:41 Cal:SL3
AutoSpec EI+ Magnet BpM:169 BpI:2956459 TIC:9156682 Flags:ACC
File Text:Lewis hmm491 HREI
169.0830
198.1225
83.9552
57.0338
97.1014
114.0283
127.0360
140.0437
149.0240
1.2E6
1.0E6
8.9E5
7.4E5
5.9E5
4.4E5
3.0E5
1.5E5
0.0E0
m/z

Elemental Composition

Date : 2-APR-2002

File:SL3 Ident:11_22-1_6 Win 100PPM Acq: 2-APR-2002 10:44:08 +3:41 Cal:SL3
AutoSpecETOFFPD EI+ Magnet BpM:169 BpI:2956459 TIC:9156682 Flags:ACC
File Text:Lewis hmm491 HREI
Heteroatom Max: 20 Ion: Both Even and Odd
Limits:

50.000	0.4							-0.5	0	0	0	0
215.235	100.0			20.0				20.0	12	1	20	2
Mass	**%RA**	**Pks**	**Std**	**PPM**	**mDa**	**Calc. Mass**		**DBE**	**C**	**13C**	**H**	**F**
199.125911	2.2	12	2.5	-2.8	-0.5	199.125362		4.0	11	1	16	2
				19.7	3.9	199.129832		3.5	12		17	2
[illegible]	16.4	12	3.2	-2.6	-0.5	198.122007		4.0	12		15	2
170.086293	10.9	12	2.3	-0.3	-0.1	170.086237		4.5	9	1	11	2
169.082954	100.0	12	1.2	-0.4	-0.1	169.082882		4.5	10		11	2
167.067330	0.8	12	4.1	0.1	0.0	167.067232		5.5	10		9	2
155.067332	0.7	12	2.4	-0.6	-0.1	155.067232		4.5	9		9	2
153.051724	1.8	12	3.0	-0.9	-0.1	153.051582		5.5	9		7	2
151.035747	1.1	12	3.6	1.2	0.2	151.035932		6.5	9		5	2
150.027422	0.5	12	4.5	4.6	0.7	150.028107		7.0	9		4	2
149.076457	1.0	12	4.0	1.3	0.2	149.076654		5.5	10		10	1
147.060192	0.5	12	4.8	5.5	0.8	147.061004		6.5	10		8	1
146.052879	0.8	12	3.1	2.0	0.3	146.053179		7.0	10		7	1
142.055029	0.6	12	2.2	-0.6	-0.1	142.054937		4.5	7	1	7	2
141.051349	6.9	12	2.3	1.6	0.2	141.051582		4.5	8		7	2
140.043722	7.2	12	2.6	0.2	0.0	140.043757		5.0	8		6	2
139.035788	1.5	12	3.5	1.0	0.1	139.035932		5.5	8		5	2
134.052398	0.8	12	2.7	5.8	0.8	134.053179		6.0	9		7	1
133.045447	1.8	12	2.7	-0.7	-0.1	133.045353		6.5	9		6	1
129.070187	0.5	12	3.6	1.8	0.2	129.070425		6.5	10		9	
127.035963	4.0	12	2.5	-0.2	0.0	127.035932		4.5	7		5	2
125.020112	0.5	12	3.0	1.4	0.2	125.020282		5.5	7		3	2
123.117215	0.5	12	4.6	1.3	0.2	123.117376		2.5	9		15	
121.045276	0.6	12	2.1	0.6	0.1	121.045353		5.5	8		6	1
120.037354	0.7	12	2.6	1.5	0.2	120.037528		6.0	8		5	1
119.029760	0.9	12	1.9	-0.5	-0.1	119.029703		6.5	8		4	1
114.028313	1.3	12	2.2	-1.8	-0.2	114.028107		4.0	6		4	2
111.117538	0.9	12	2.6	-1.5	-0.2	111.117376		1.5	8		15	
109.102097	0.7	12	2.4	-3.4	-0.4	109.101726		2.5	8		13	
109.045769	1.4	12	1.9	-3.8	-0.4	109.045353		4.5	7		6	1
107.029919	0.5	12	3.5	-2.0	-0.2	107.029703		5.5	7		4	1
101.039340	1.2	12	2.5	-2.1	-0.2	101.039125		6.5	8		5	
101.020558	0.4	12	2.3	-2.7	-0.3	101.020282		3.5	5		3	2
97.101433	1.5	12	2.1	3.0	0.3	97.101726		1.5	7		13	
95.085883	0.9	12	2.4	2.0	0.2	95.086075		2.5	7		11	
91.055472	0.6	12	3.4	-7.6	-0.7	91.054775		4.5	7		7	
69.070488	2.2	12	2.5	-0.9	-0.1	69.070425		1.5	5		9	
67.051463	0.6	12	3.4	-17.3	-1.2	67.050305		3.0	4	1	6	



*** Current Data Parameters ***

NAME	:	SBL75a
EXPNO	:	1
PROCNO	:	1

*** Acquisition Parameters ***

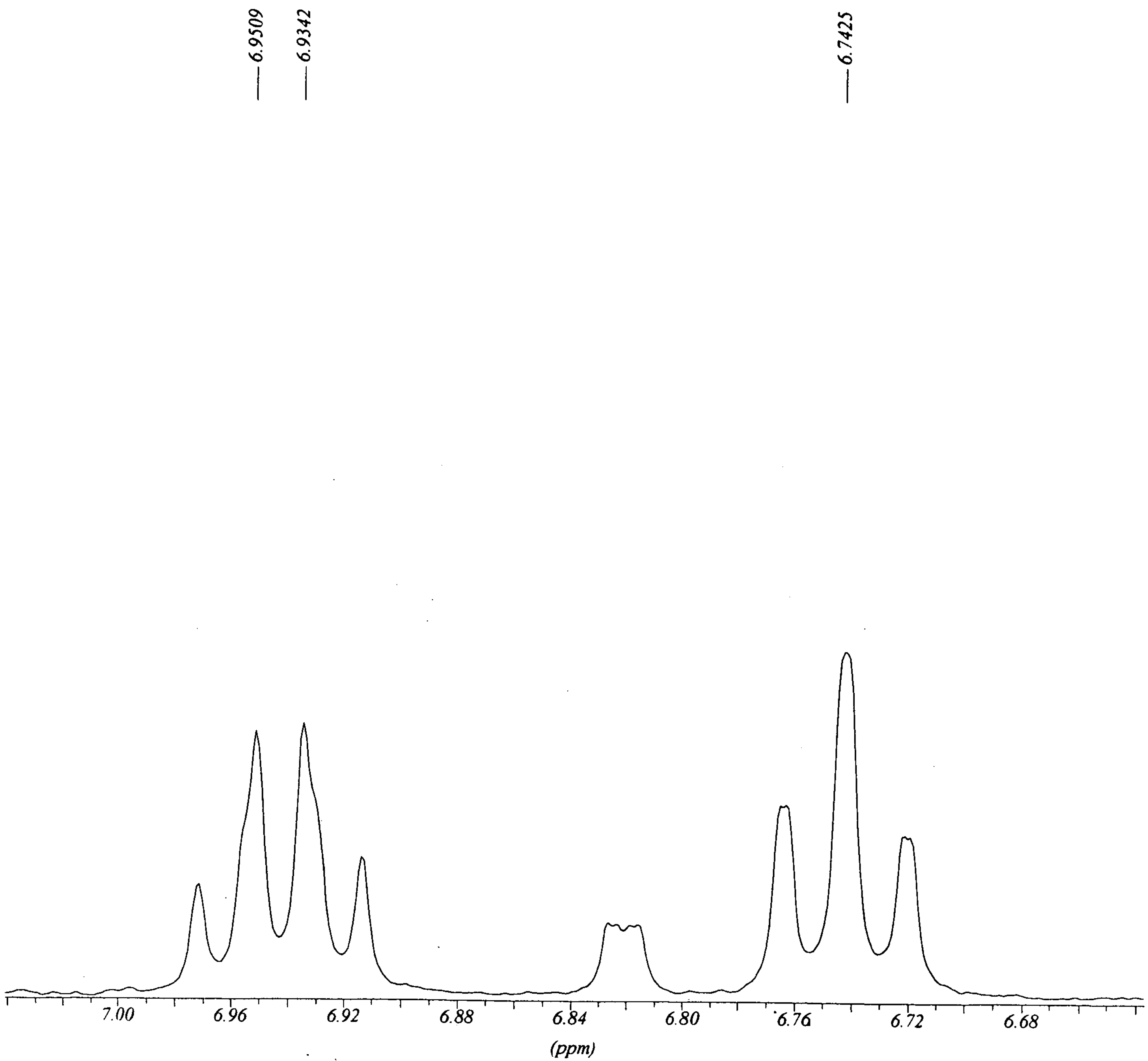
AQ_mod	:	dqd
AUNM	:	au_zg
BF1	:	399.8700000 MHz
DATE_t	:	11:36:14
DATE_d	:	Jul 25 2002
DS	:	2
FW	:	93750.00 Hz
NS	:	8
NUC1	:	1H
O1	:	2399.22 Hz
PROBHD	:	5 mm Multinuclear Z82-001
PULPROG	:	zg30
RG	:	456.1000061
SOLVENT	:	CDCl3
SW	:	16.0308 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
MI	:	0.15 cm
PC	:	4.00
SI	:	16384

*** 1D NMR Plot Parameters ***

SOLVENT	:	?



*** Current Data Parameters ***

NAME	:	SBL75a
EXPNO	:	1
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*** Acquisition Parameters ***

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O1	:	2399.22 Hz
PROBHD	:	5 mm Multinuclear Z82001
PULPROG	:	zg30
RG	:	456.1000061
SOLVENT	:	CDCl3
SW	:	16.0308 ppm

*** Processing Parameters ***

LB	:	1.00 Hz
MI	:	0.15 cm
PC	:	4.00
SI	:	16384

*** 1D NMR Plot Parameters ***

SOLVENT	:	?