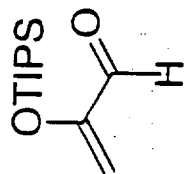


Preparation of **4**: An oven-dried flask equipped with a stir bar, rubber septum and N₂ inlet was charged with **3** (1.0g, 6.85 mmol) and triethylamine (1.33 mL, 10.27 mmol) and dry benzene (10 mL). Neat TIPSOTf (2.31 g, 7.54 mmol) was added over a few minutes and the solution was allowed to stir at room temperature for 12 h. The reaction was quenched with water and extracted with ether (3x25 mL). The combined organic extracts were washed with brine, dried over Na₂SO₄ and the solvent was removed *in vacuo*. Flash chromatographic purification over silica gel using hexane-EtOAc (20:1) afforded 1.12 g (72%) of **4** as colorless oil which was analytically pure. IR: 1707 cm⁻¹. ¹H NMR (250 MHz, CDCl₃) δ: 9.31(s, 1H), 5.48(d, J=1.7 Hz, 1H), 5.23(d, J=1.7 Hz, 1H), 1.32-1.16(m, 3H), 1.1-1.05(m, 18H). ¹³C NMR (62.5 MHz, CDCl₃) δ: 189.4, 156.2, 112.0, 17.8, 12.6. Anal. Calcd. for C₁₂H₂₄O₂Si: C, 63.1; H, 10.59. Found: C, 63.30; H, 10.56.

Preparation of **7**: A 10 mL oven-dried, roundbottom flask equipped with a stir bar, rubber septum and N₂ inlet was charged with aldehyde **4** (0.075 g, 0.328 mmol) and dry CH₂Cl₂ (1.5 mL). The flask was placed in an ice bath and furan (0.056g, 0.82 mmol) was added and the solution was stirred for 5 min. Solid Sc(OTf)₃ (Aldrich, 0.016g, 0.033 mmol) was added in one portion. The mixture was stirred for 2 h as it slowly warmed to room temperature. TLC showed complete consumption of starting material. The reaction mixture was transferred to a separatory funnel with the aid of 20 mL ether and washed with water (2x15 mL) and brine (15 mL). Drying (Na₂SO₄) and removal of the solvent *in vacuo* afforded crude product which was purified over silica gel by flash chromatography using hexane-EtOAc (20:1) to give **7** (0.087 g, 90%) as colorless oil. IR: 1732 cm⁻¹. ¹H NMR (250 MHz, CDCl₃) δ: 6.32-6.26(m, 2H), 4.98(d of t, J₁=1.2 Hz, J₂=4.8 Hz, 1H), 4.92(dd, J₁=1.5 Hz, J₂=5.1 Hz, 1H), 4.45(d, J=5.1 Hz, 1H), 2.73(dd, J₁=4.8 Hz, J₂=15.4 Hz, 1H), 2.35(d, J=15.4 Hz, 1H), 1.18-1.00(m, 21H). ¹³C NMR (62.5 MHz, CDCl₃) δ: 204.21, 134.49, 131.91, 81.52, 79.88, 78.41, 45.82, 17.84, 12.28. Anal. Calcd. for C₁₆H₂₈O₃Si: C, 64.82; H, 9.52. Found: C, 64.66; H, 9.70.



4

9.31379

7.25946

5.48540
5.47843
5.23850
5.23176

1.26556
1.25148
1.23975
1.23370
1.22124
1.20584
1.18146
1.15555
1.09593
1.08517
1.07087
1.06178
1.04941

US-II-26B-A1

OTIPS
(100)

Current Data Parameters
NAME US-II-26B-A1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 13.09
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TO 32768
SOLVENT CDC13
NS 16
DS 2
SWH 5208.333 Hz
FIDRES 0.158945 Hz
AQ 3.1457779 sec
RG 715
DW 96.000 usec
DE 137.14 usec
TE 300.0 K
D1 1.0000000 sec
P1 8.70 usec
DE 137.14 usec
SF01 250.1315321 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 16384
SF 250.130006 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.50

10 NMR plot parameters
CX 20.00 cm
F1P 10.039 ppm
F1 2511.17 Hz
F2P 0.148 ppm
F2 -37.05 Hz
PPMCM 0.50938 ppm/cm
HZCM 127.41089 Hz/cm

3.451
20.722

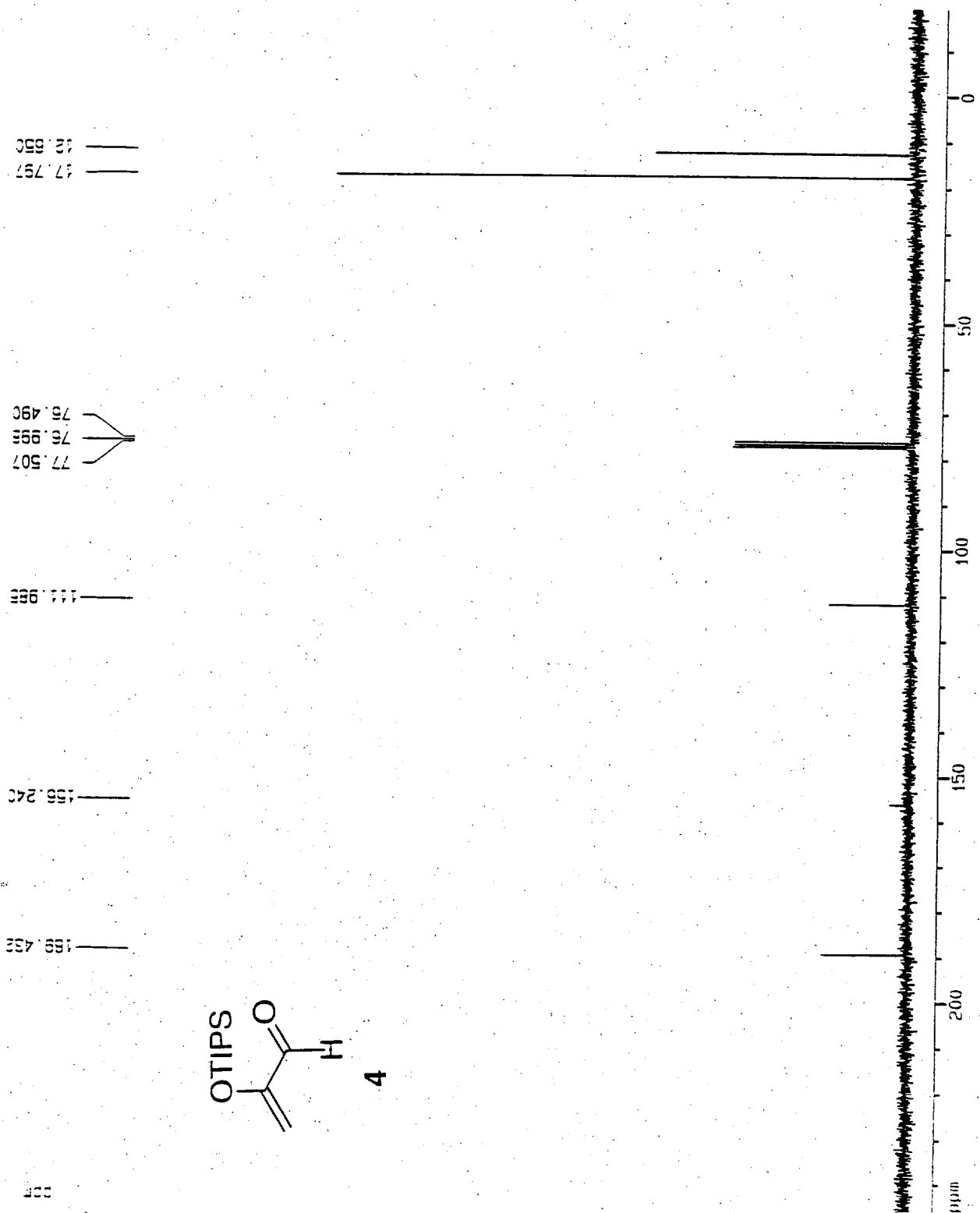
1.040
1.039

1.000



US-II-26B-A1

Carbon



Current Data Parameters

NAME US-II-26B-A1

EXPNO 2

PROCNO 1

F2 - Acquisition Parameters

Date_ 981028

Time 13 17

INSTRUM spect

PULPROG zgpg30

TD 36864

SOLVENT CHCl3

NS 185

DS 4

SWH 16666.666 Hz

FIDRES 0.452112 Hz

AQ 1.1059700 sec

RG 22800

DW 30.000 usec

DE 42.86 usec

TE 300.0 K

D12 0.00002000 sec

OL5 23.00 dB

CPUPRG waltz16

P31 103.00 usec

D1 1.00000000 sec

P1 5.35 usec

DE 42.86 usec

SFO1 62.9023694 MHz

NUCLEUS 13C

D11 0.03000000 sec

F2 Processing parameters

SF 37.708

SF 62.8952402 MHz

WDW FM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX 20.00 cm

FIP 245.846 ppm

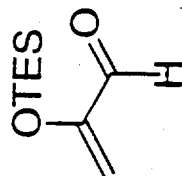
F1 15482.54 Hz

F2 -19.145 ppm

F2 -1204.12 Hz

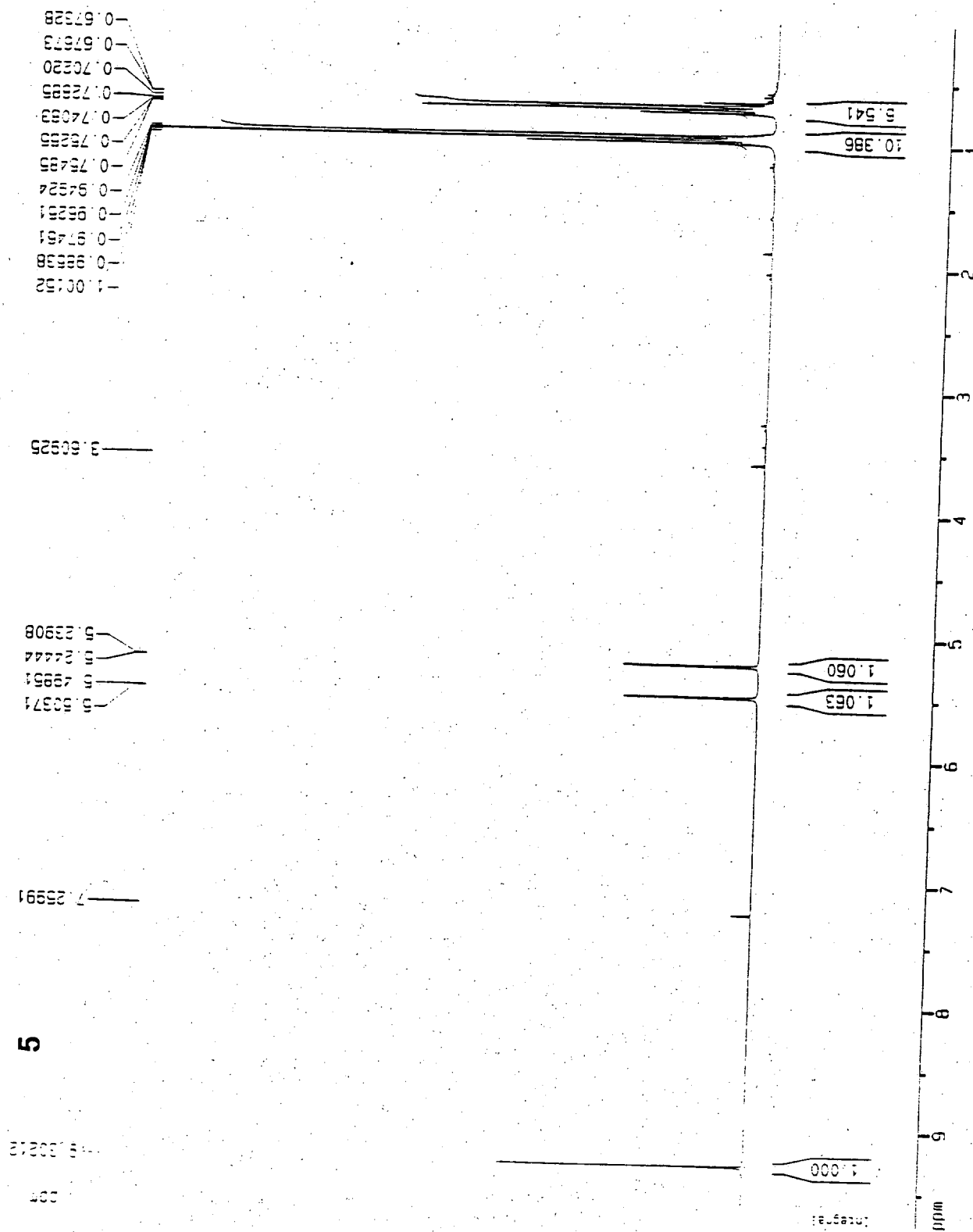
PPMCM 13.24955 ppm/cm

HZCM 833.33331 Hz/cm



5

US-11-300
Proton

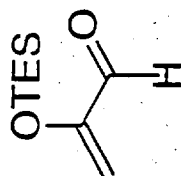


Current Data Parameters
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 PROCNO: 1

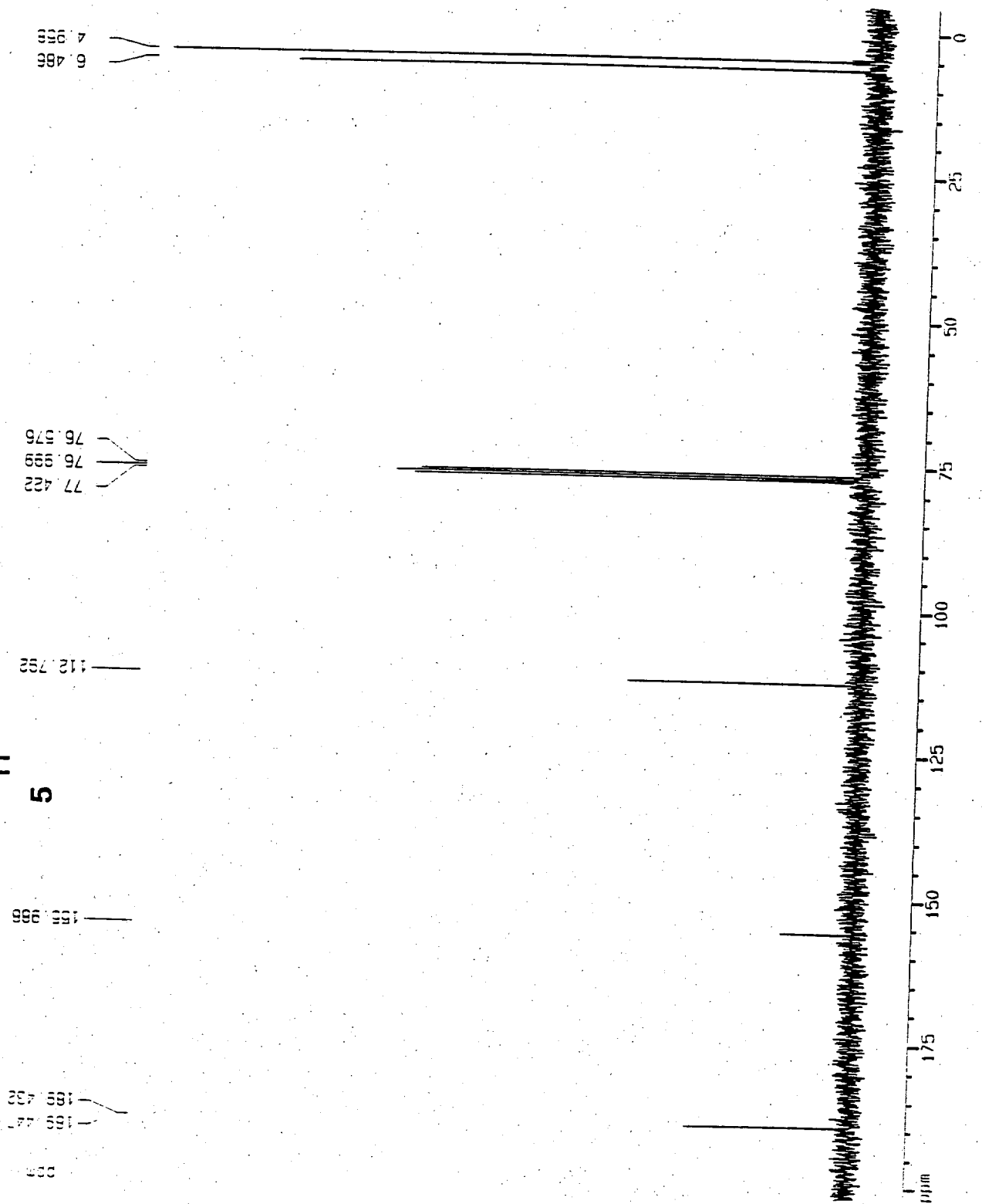
F2 Acquisition Parameters
 Date_: 990524
 Time: 10.42
 INSTRUM: OFX300
 PULPROG: zgpg30
 FIDRES: 0.100000 Hz
 AQ: 6.542000 sec
 RG: 170
 DW: 81.000 usec
 DE: 6.00 usec
 TE: 300.0 K
 O1: 1.0000000 sec
 P1: 7.50 usec
 DE: 6.00 usec
 SF01: 300.131700 MHz
 NUC1: 1H
 PC: 0.00 dB

F2 Processing parameters
 SI: 16384
 SF: 300.1300063 MHz
 RG: 64
 SSF: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00

1D NMR plot parameters
 CX: 20.00 cm
 FIP: 9.775 ppm
 F1: 293.170 Hz
 F2: 0.025 ppm
 F2: 7.62 Hz
 P1/MCM: 0.40740 ppm/cm
 HZCN: 146.30817 Hz/cm



5



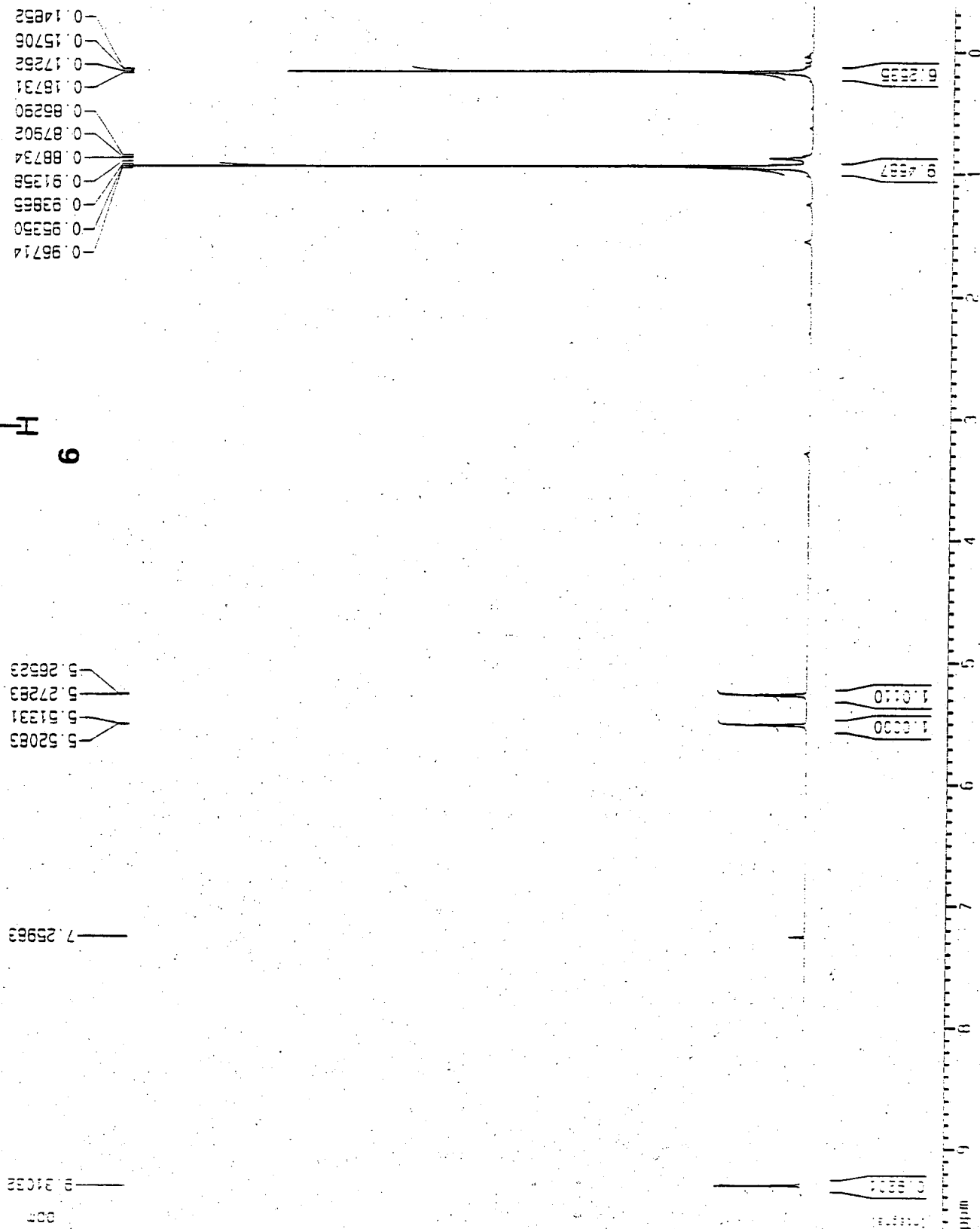
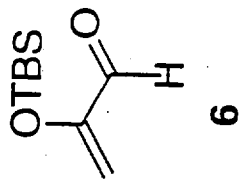
Current Data Parameters	
NAME	05 11 001
EXPO	?
PHONO	1

Acquisition Parameters	
Date	0000-0-0
Time	10 47
INSTRUM	000-300
PHOTOD	5 mm 030 00 10
PULPHOS	2900 30
TD	655 36
SOLVENT	CHL 13
MS	120-
DS	4
SWH	21045 02 1 10
FIDUES	0 3002 77 10
AD	1 50 39 116 000
RG	1000
OW	25 100 000
TE	6 00 000
U1	300 0 8
U11	0 0 000000 000
P1 P2	25 10 00
CHRGNG2	wait 2 16
PCUP02	100 00 USEC
SP 02	300 1310000 000
NUC2	10
P1 P2	0 00 00
D1	1 00000000 SEC
P1	7 25 USEC
DE	6 00 USEC
SP 01	25 470000 000
NUC1	100
P1 P2	6 00 00

Processing parameters	
SI	32768
SP	25 4677490 000
MDH	10
SSU	0
UB	1 00 10
GB	0
PC	1 40

10 NMR plot parameters	
X	20 00 cm
UP	201 992 000
1	15243 09 10
2p	4 567 000
2	344 03 10
PMCM	10 32797 000/cm
VMCM	779 47000 102/cm

US-IV-20
H1 spectrum

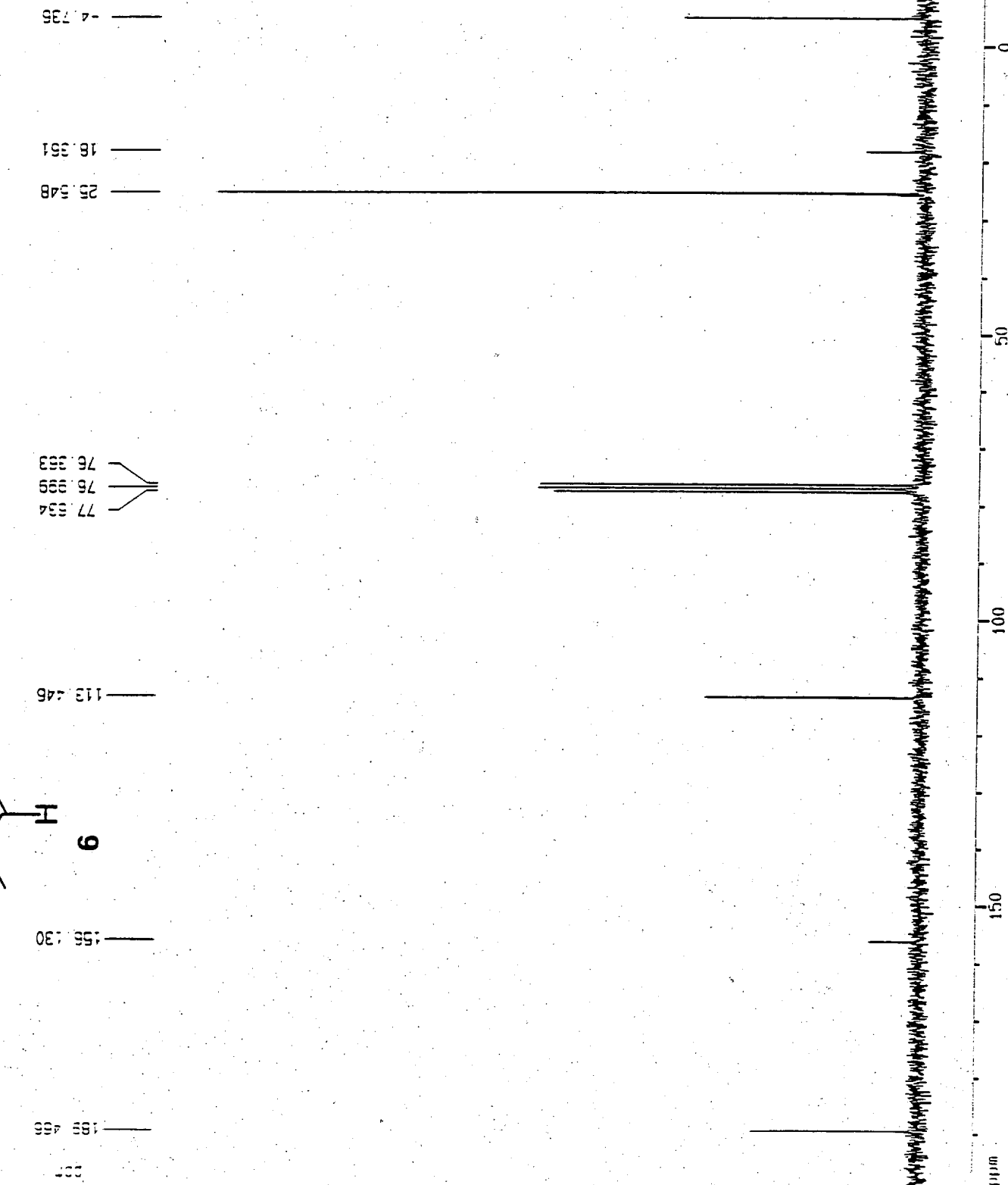
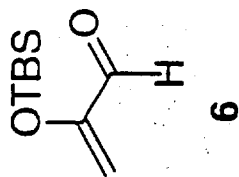


Current Data Parameters
NAME US-IV-20
EXPNO 1
PROCNO 1

F2 Acquisition Parameters
Date 990628
Time 15:55
INSTRUM AMX-200
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 3623.100 Hz
FIDRES 0.22142 Hz
AQ 2.2610471 sec
RG 1024
DM 130.000 usec
DE 197.14 usec
TE 300.0 K
H1 1.00
H1 1.00
D1 1.00000000 sec
P1 6.80 usec
DE 197.14 usec
SFO 200.133000 MHz
NUC1H5 1H

F2 Processing parameters
SI 16384
SF 200.133000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 21.00 cm
FJP 9.665 ppm
F1 1934.33 Hz
F2 -0.376 ppm
F2 -75.17 Hz
PQMCN 0.47814 ppm
H1CM 95.63044 Hz



Current Data Parameters	
NAME	US-1V-20
EXPRO	?
PRODNO	1

F2 - Acquisition Parameters	
Date..	940628
Time	16:00
INSTRUM	AMX-200
PROBHD	5 mm Molt 1mm
PULPROG	zgpg30
TO	16.384
SOLVENT	CDCl3
NS	2179
DS	?
SWH	12820.513 Hz
F1FREQ	0.70750717
AQ	0.6300940 sec
RG	32768
DW	39.000 usec
DE	55.71 usec
DE	300.0 K
D11	0.0300000 sec
CGPRG	waltz16
PP31	100.00 usec
SF	26.00
NUC1	31.00
D1	1.0000000 sec
P1	/ 00 usec
DE	55.71 usec
SF01	50.3200000 MHz
NUCLEUS	13C

F2 - Processing parameters	
SI	16384
SF	50 3233161 Hz
WIN	1M
SSB	0
LB	1.00 Hz
GB	0
PC	1.00

10-MHz plot parameters	
CX	21.00 cm
F1P	198 006 ppm
F1	10009 61 Hz
F2P	-14 665 ppm
F2	-738.00 Hz
PTMCM	10 16911 ppm
HZCM	511.74319 Hz

US-II-42B

Current Data Parameters
NAME US-II-42B
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

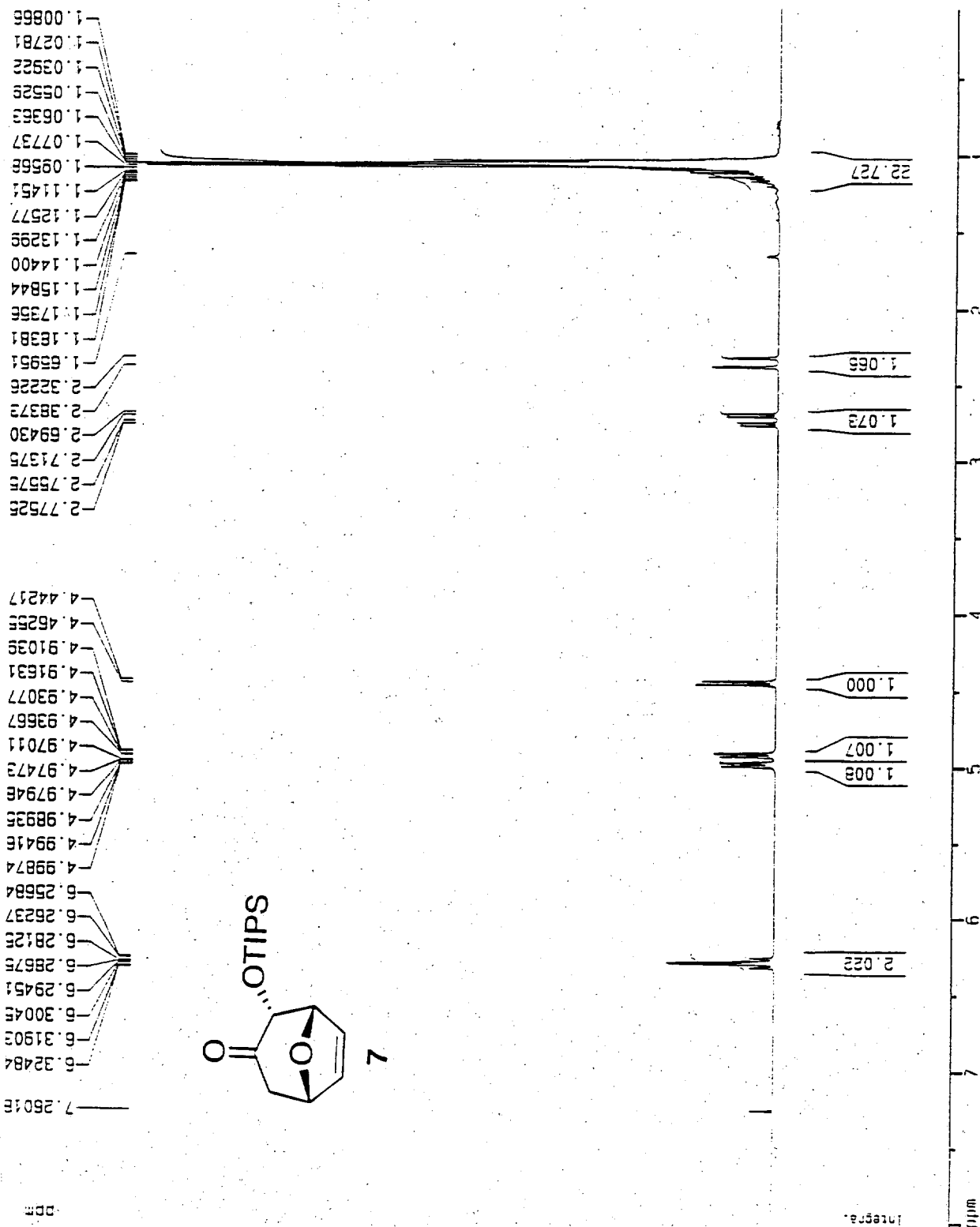
Date_ 981124
Time 14.37
INSTRUM arx250
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 12
DS 2
SWH 5200.171 Hz
FIDRES 0.150946 Hz
AQ 3.145779 sec
RG 256
DM 96.000 use
DE 137.14 use
TE 300.0 K
D1 1.00000000 sec
P1 8.70 use
DE 137.14 use
SF01 250.1315321 MHz
NUCLEUS 1H

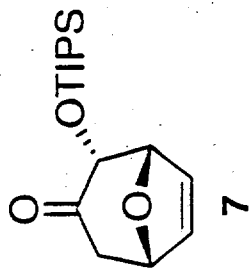
F2 - Processing parameters

SI 16384
SF 250.1300073 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.50

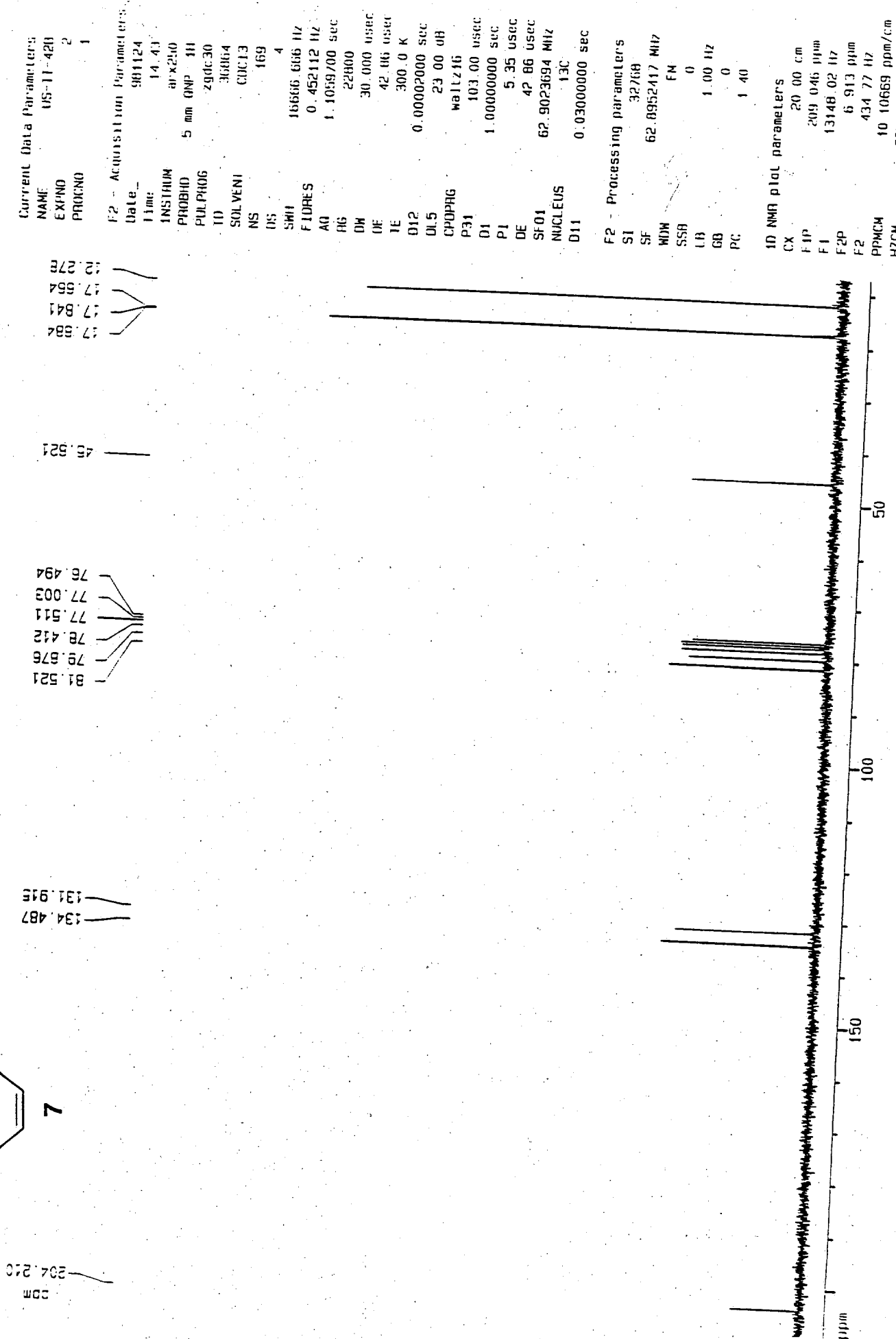
1D NMR plot parameters

CX 20.00 cm
F1P 8.022 ppm
F1 2006.63 Hz
F2P 0.050 ppm
F2 12.49 Hz
PPMCM 0.3862 ppm/cm
HZCM 99.70678 Hz/cm

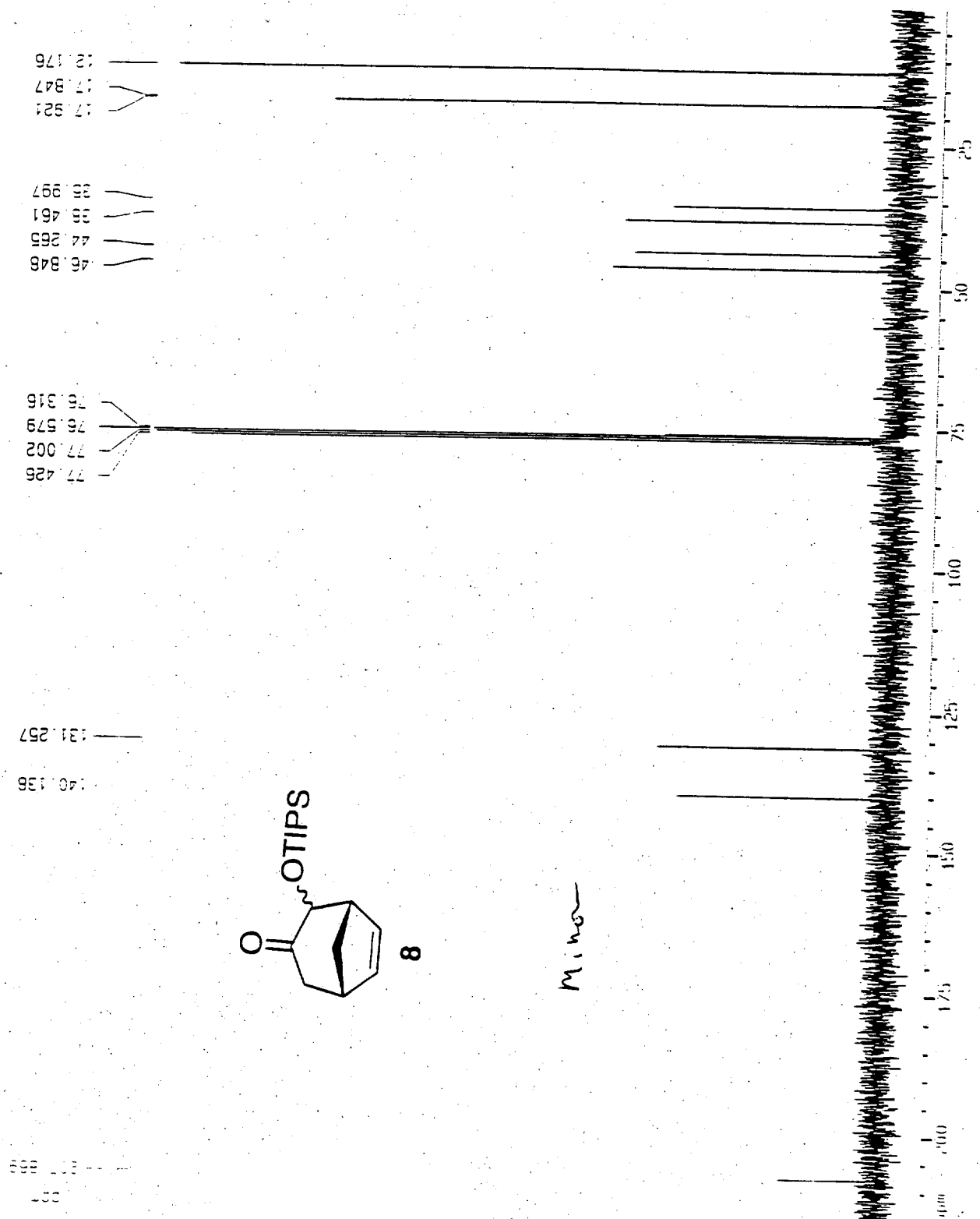




US-11-42B

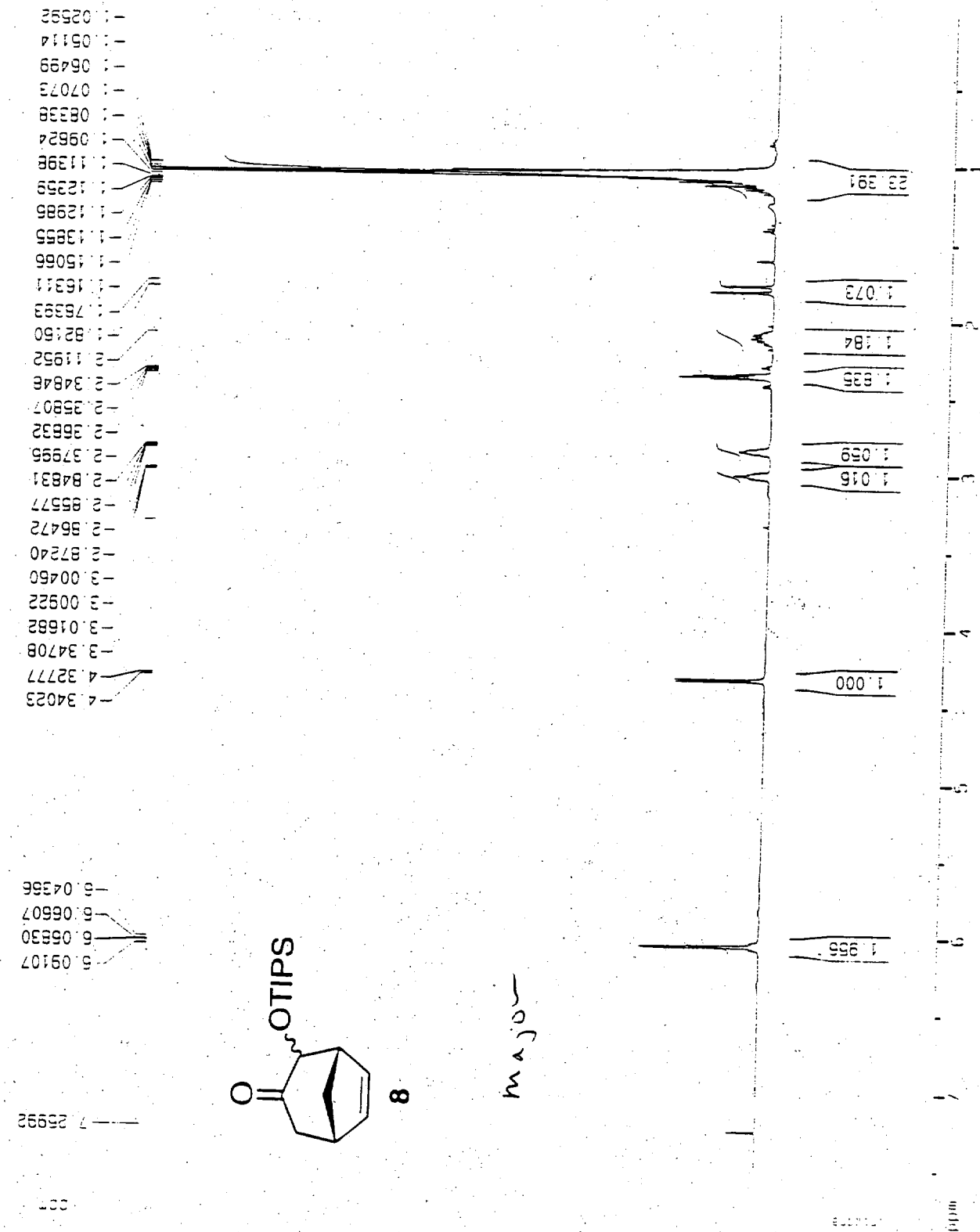


05 111 30101



Current Data Parameters	
NAME	05 111 30101
EXP#	1
PROCNO	1
F2 Acquisition Parameters	
Date	990211
Time	14 35
INSTRUM	400 MHz
PULPROG	zgpg30
TD	65536
SI	32768
RG	1024
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40
F2 Processing Parameters	
SI	32768
SF	75 467491 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40
1D 1H NMR Parameters	
EX	20.00 cm
FL	214 963 mm
FI	16.775 / 1.0
FO	0.001 mm
FR	51.41 Hz
FW	10.71407 mm/cm
FT	800.346341 Hz/cm

US-111-300A



Current Data Parameters

NAME US-111-300A

EXPNO 1

PROCNO 1

F2 Acquired 100.000 MHz

Time 00:00:00

INSTRUM 00.000

PROBHD 5 mm BBO HX 1H

PULPROG zgpg30

TD 32768

SF 500.130

RG 16

DS 16

SWH 63.172 MHz

FIDRES 0.38000 Hz

AQ 2.000000 sec

RG 1.20

DW 03.000 msec

DE 6.00 msec

TE 300.2 K

03 1.0000000 sec

P1 7.50 msec

DE 6.00 msec

SF 100.131700 MHz

NUC1 1H

P1 0.00 sec

F2 Processing parameters

SF 16.404

SI 300.100001 MHz

WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 20.00 cm

F1 7.877 ppm

F2 2363.98 Hz

F3 0.025 ppm

F4 7.62 Hz

PPM 0.38000 ppm/cm

HZ 117.61014 Hz/cm

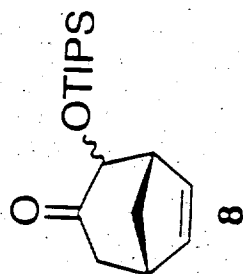
125.111 MHz

136.671
133.622

81.961
77.422
76.999
76.975

48.152
45.463
41.303
39.353

17.997
17.955
12.434

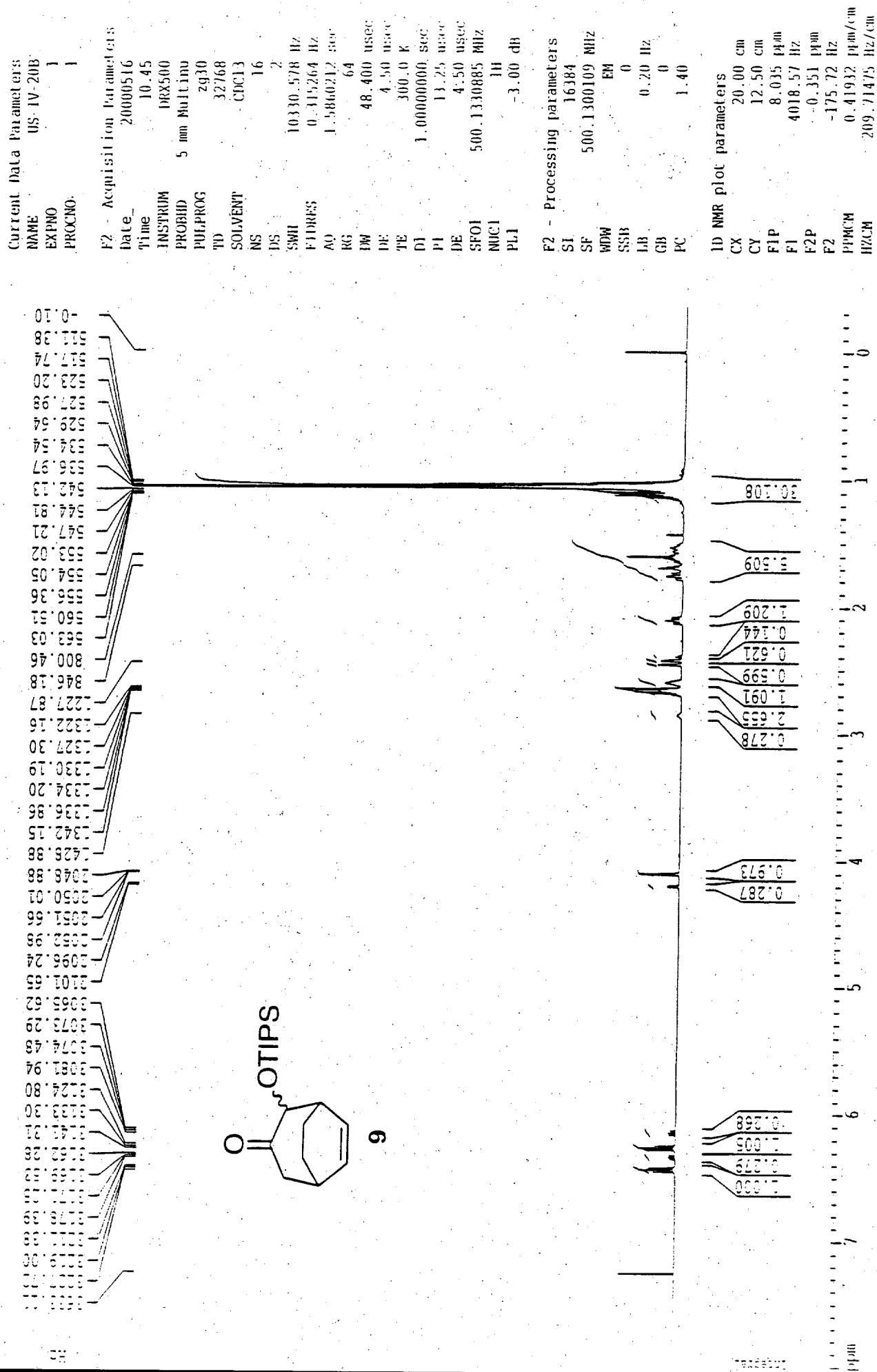


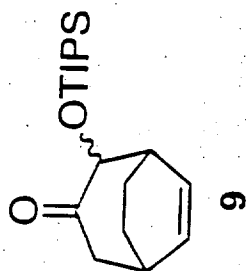
Major

Current Data Parameters	
NAME	125.111 MHz
EXPER	1
PROCNO	1
F2 Acquired Parameters	
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Time	15.11
INSTRUM	DPX 400
PROBHD	5 mm BBO (H) 1H
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	203
DS	4
SWH	21645.071 Hz
FIDRES	0.30677 Hz
AQ	1.539116 Sec
RG	2048
DM	23.109 Hz
DE	6.00 Hz
TE	300.2 K
NUC1	0.000000 Sec
NUC2	24.960 Hz
CPDPRG2	waltz16
PCPD2	100.00 Hz
SCD2	300.1315007 MHz
NUC2	1H
NUC1	0.000000 Sec
NUC2	24.960 Hz
NUC1	0.000000 Sec
NUC2	24.960 Hz
NUC1	0.000000 Sec
NUC2	24.960 Hz
Processing parameters	
SI	16384
SI	16384
SI	16384
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40
1D NMR plot parameters	
EX	20.00 cm
GP	214.145 ppm
F1	16.2141 Hz
F2	6.454 ppm
F3	407.15 Hz
PPMCM	10.4540 ppm/cm
HZCM	206.71 Hz/cm

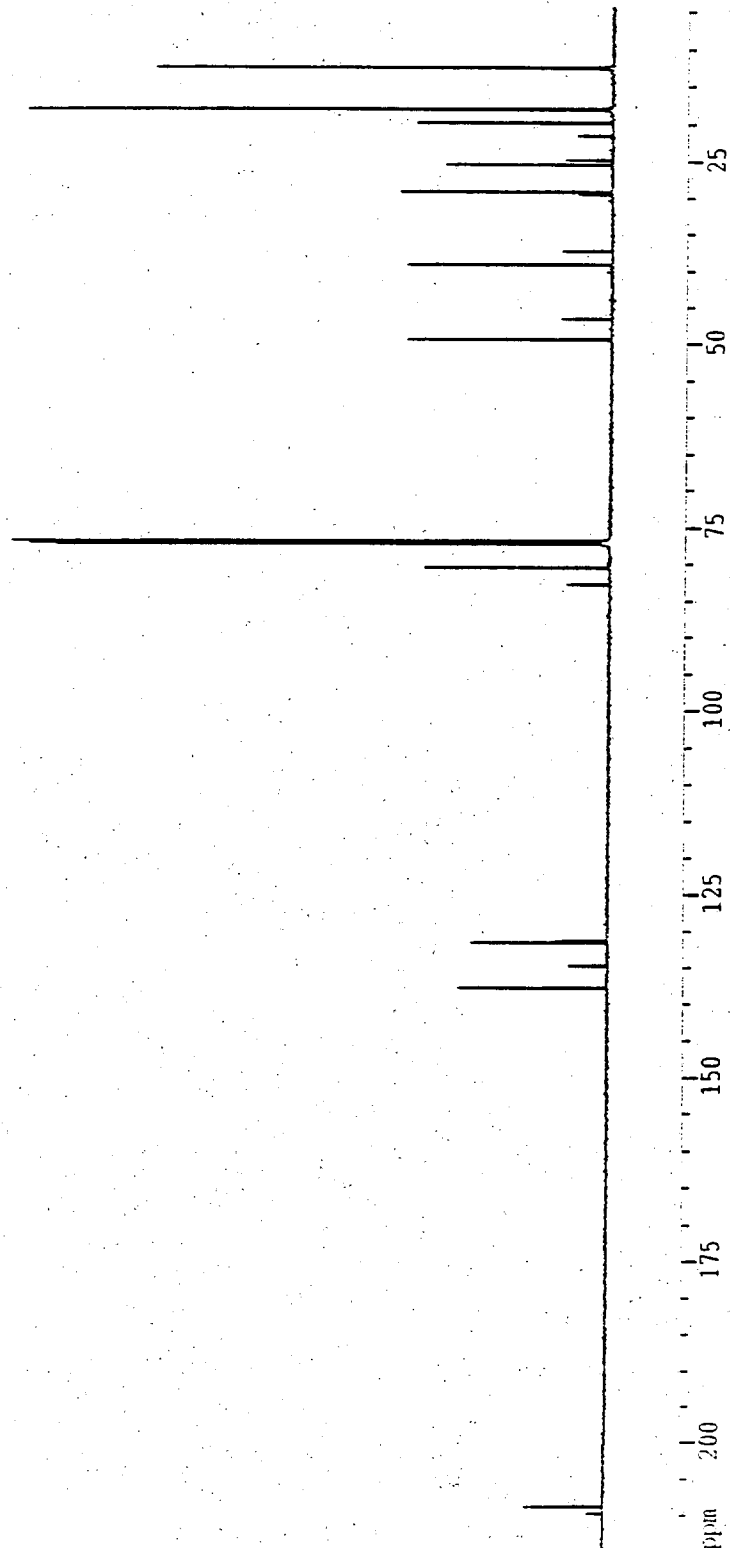
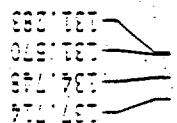
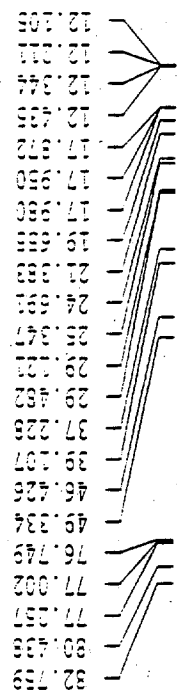


US-IV-20B





US-IV-20B

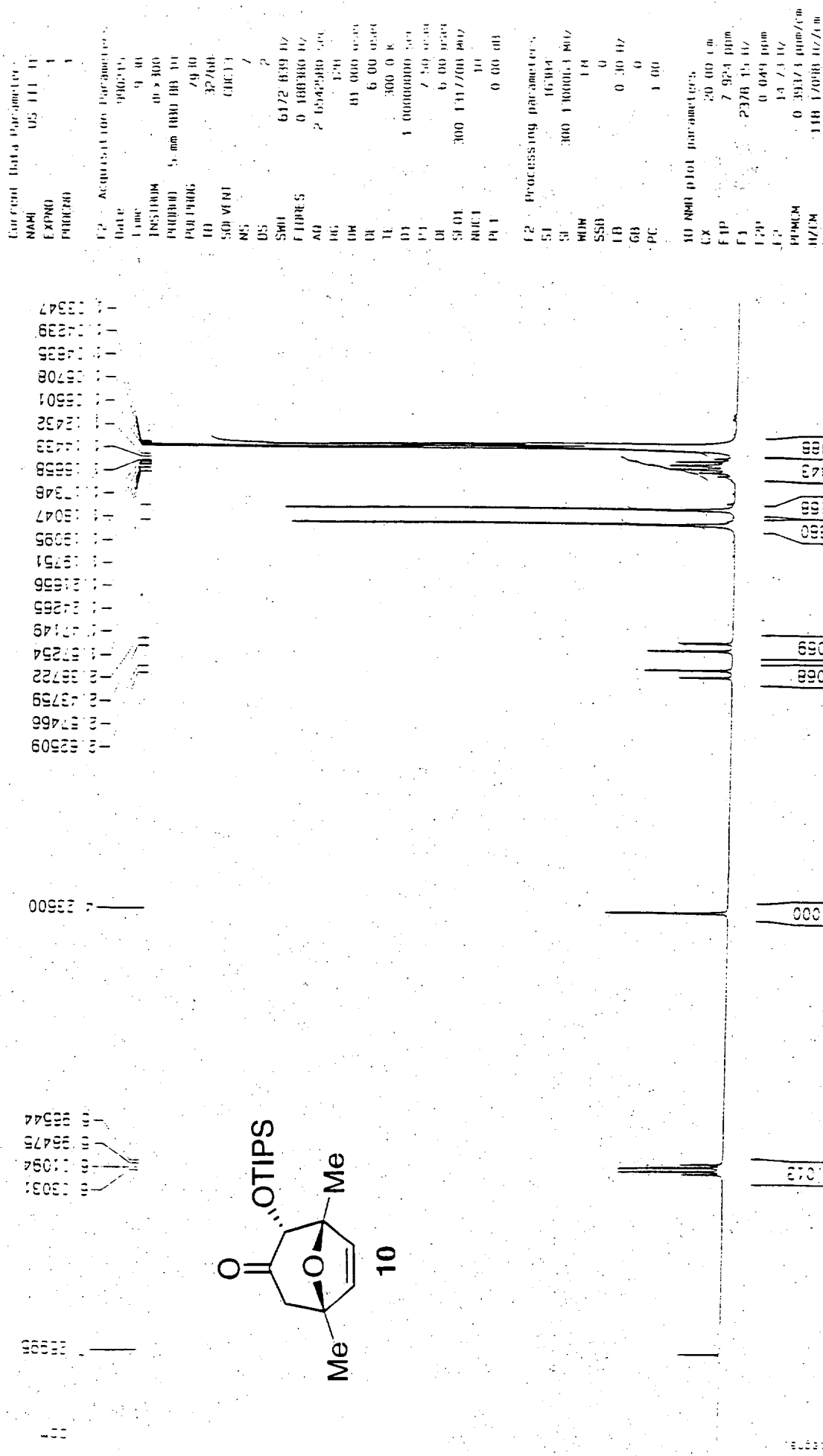
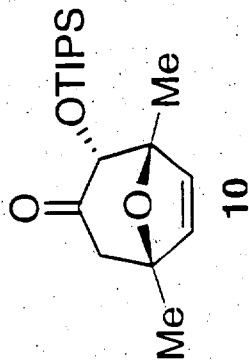


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PRGNO	1

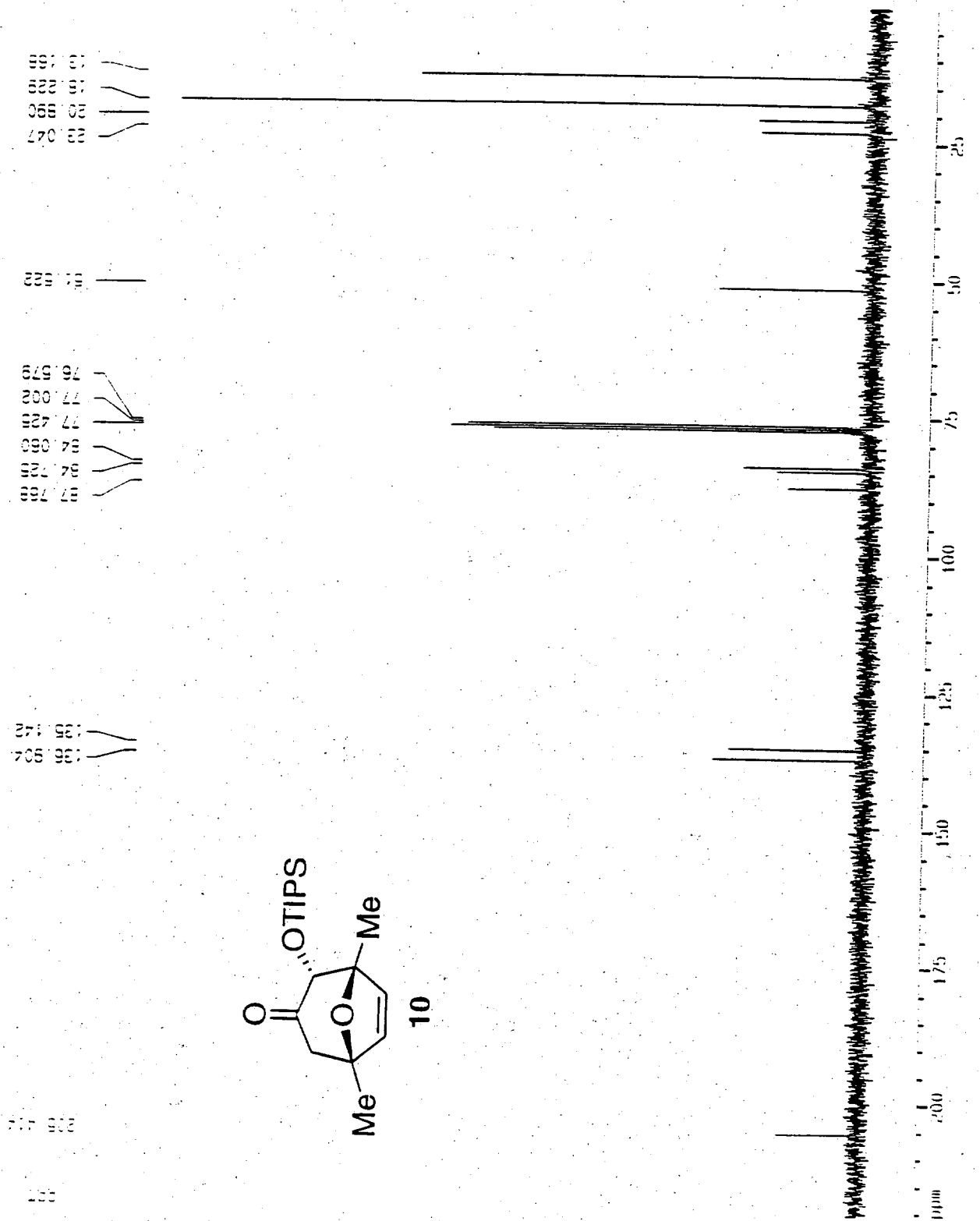
F2 - Acquisition Parameters	
Date	20000516
Time	10:59
INSTRUM	DPX500
PROBHD	5 mm Mult inn
PULPROG	zgpg30
TD	65536
SOLVENT	(1X)13
NS	2465
DS	4
SWH	39682.539 Hz
FIDRES	0.605507 Hz
RG	0.8258036 sec
RG	4096
AW	12.600 msec
DE	4.50 usec
TE	298.0 K
d11	0.0300000 sec
PL12	21.00 dB
CPDPRG2	waltz16
PCPD2	107.00 usec
SFO2	500.1320005 MHz
NUC2	¹ H
PUL2	0.00 dB
PL11	1.000000000 sec
PL1	8.24 usec
DE	4.50 usec
SFO1	125.7713108 MHz
NUC1	¹³ C
PL1	3.00 dB

F2 - Processing parameters	
SI	32768
SF	125.7577922 MHz
EM	0
SSB	1.00 Hz
LB	0
GB	1.40
PC	

1D NMR plot parameters	
CCX	20.00 cm
CY	8.00 cm
F1F1P	214.820 ppm
F1F1	27015.11 Hz
F2P	4.150 ppm
F2	521.83 Hz
PPMCH	10.53353 ppm/cm
HZCM	1324.67383 Hz/cm



05.111.31



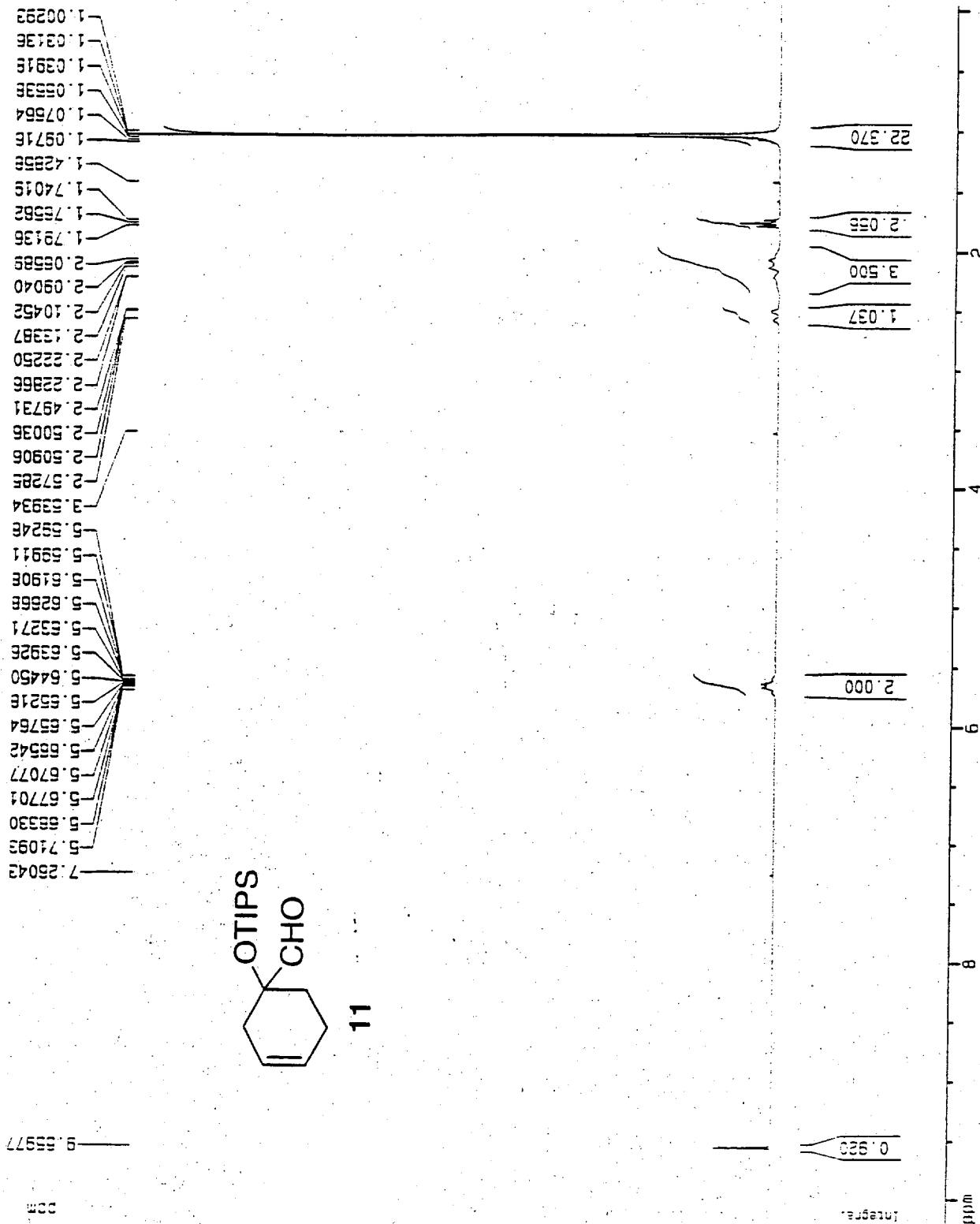
US-IV-18B

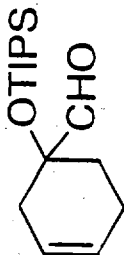
Current Data Parameters
 NAME US-IV-18B
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 9/03/02
 Time 13.53
 INSTRUM apx250
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 5208.333 Hz
 FIDRES 0.158946 Hz
 AQ 3.1457779 sec
 RG 256
 DW 96.000 usec
 DE 137.14 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 8.70 usec
 DE 137.14 usec
 SF01 250.1315321 MHz
 NUCLEUS 1H

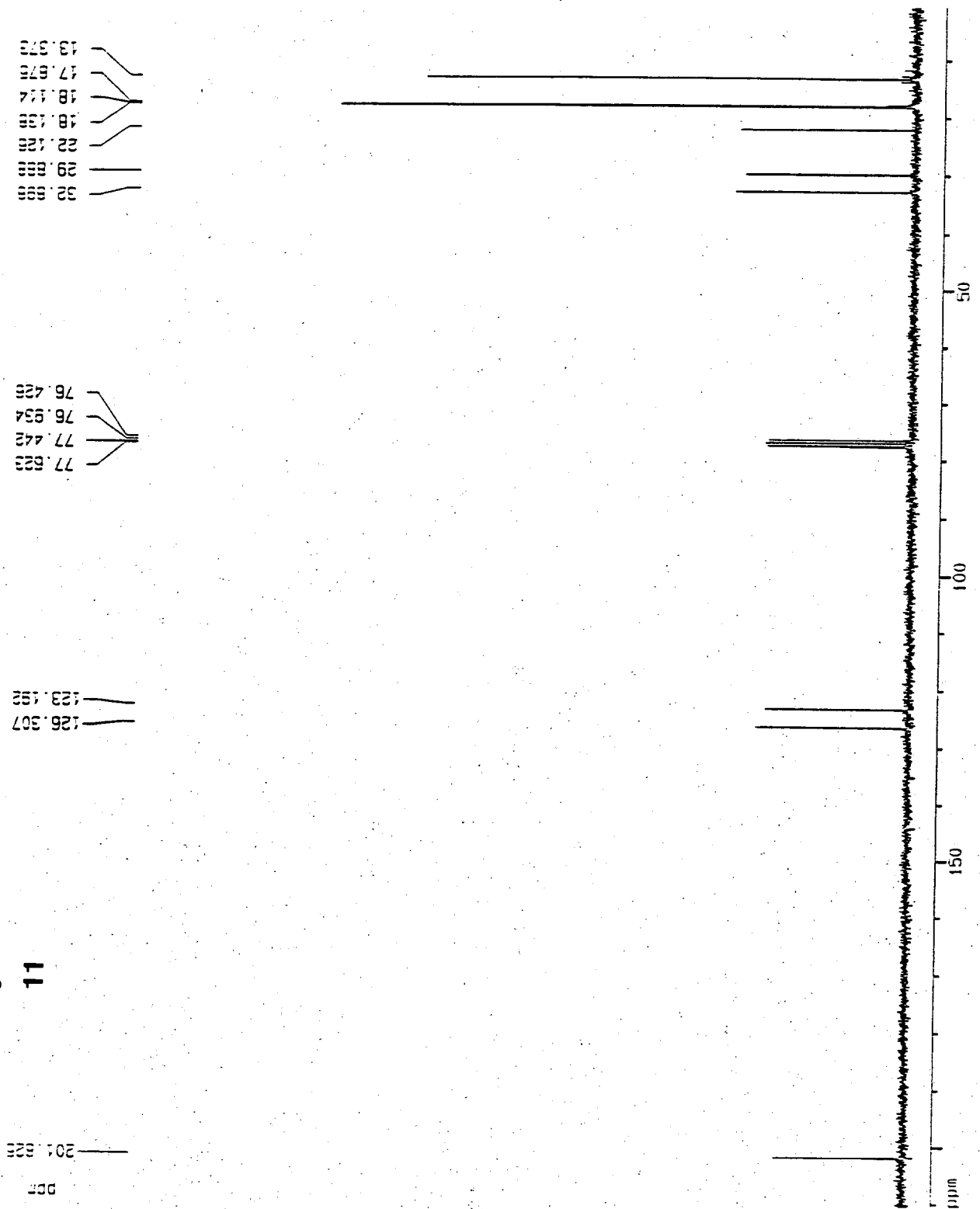
F2 - Processing parameters
 SI 16384
 SF 250.130073 MHz
 WDW EM
 SSB 0
 LB 0.20 Hz
 GB 0
 PC 1.50

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.203 ppm
 F1 2552.18 Hz
 F2P -0.045 ppm
 F2 -11.30 Hz
 PPMCM 0.51243 ppm/cm
 HZCM 128.17383 Hz/cm





二



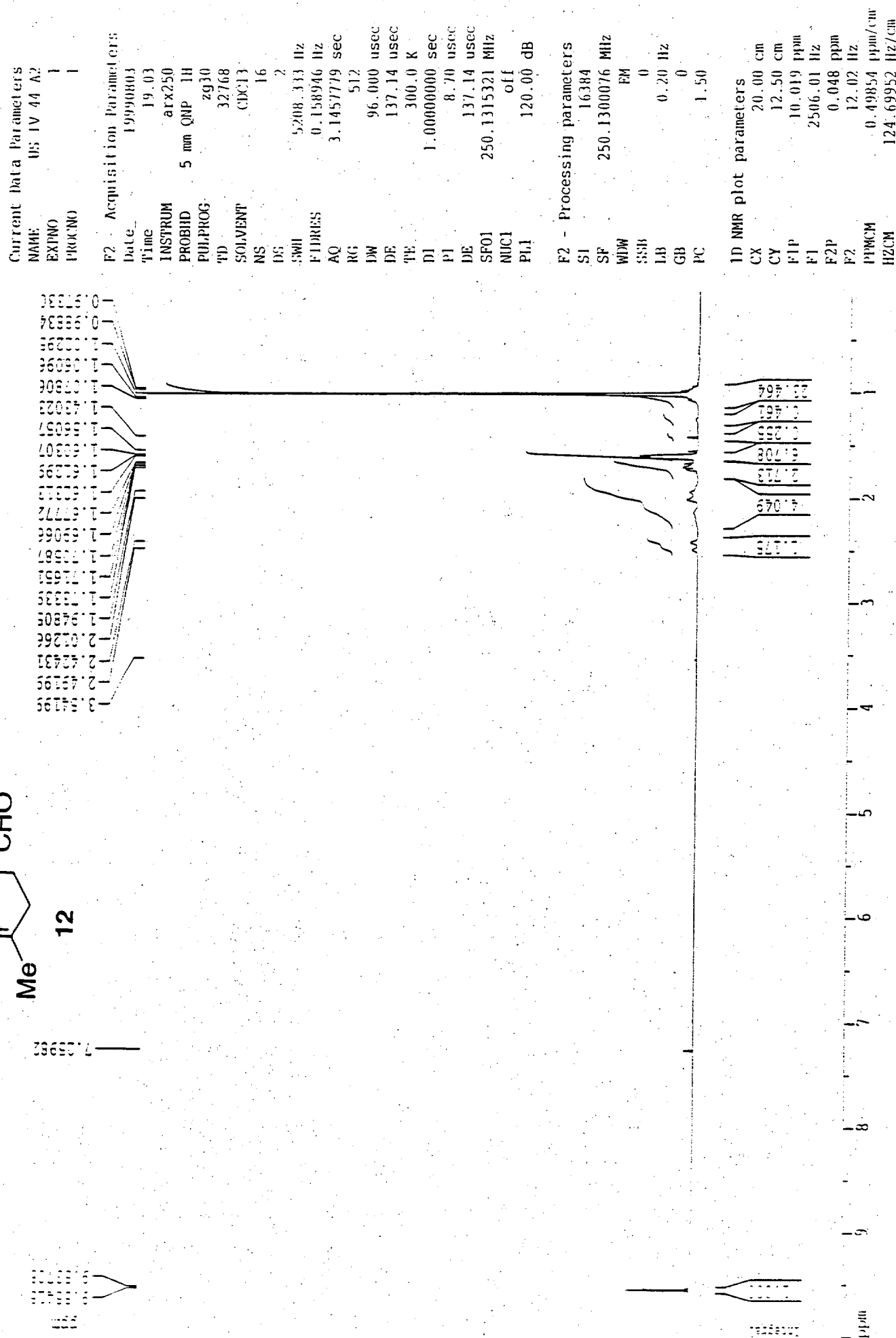
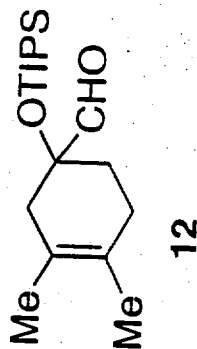
Current Data Parameters	
NAME	US-IV-100
EXNO	2
PHONO	1

F2 - Acquisition Parameters	
Date_	930502
Time	14 03
INSTRUM	apx250
PROBHD	5 mm QNP 1H
PULPROG	zgpg30
TD	36864
SOLVENT	CUC13
NS	256
DS	4
SWH	16666.666 Hz
FIDRES	0.452112 Hz
AQ	1.1059700 sec
RG	22800
QW	30.000 usec
DE	42.86 usec
TE	300.0 K
D12	0.00002000 sec
D15	23.00 dB
CPDPRG	waltz16
P31	103.00 usec
D1	1.00000000 sec
P1	5.35 usec
DE	42.86 usec
SF01	62.9023694 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 Processing parameters	
SI	32768
SF	62.0952440 MHz
WDW	FM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

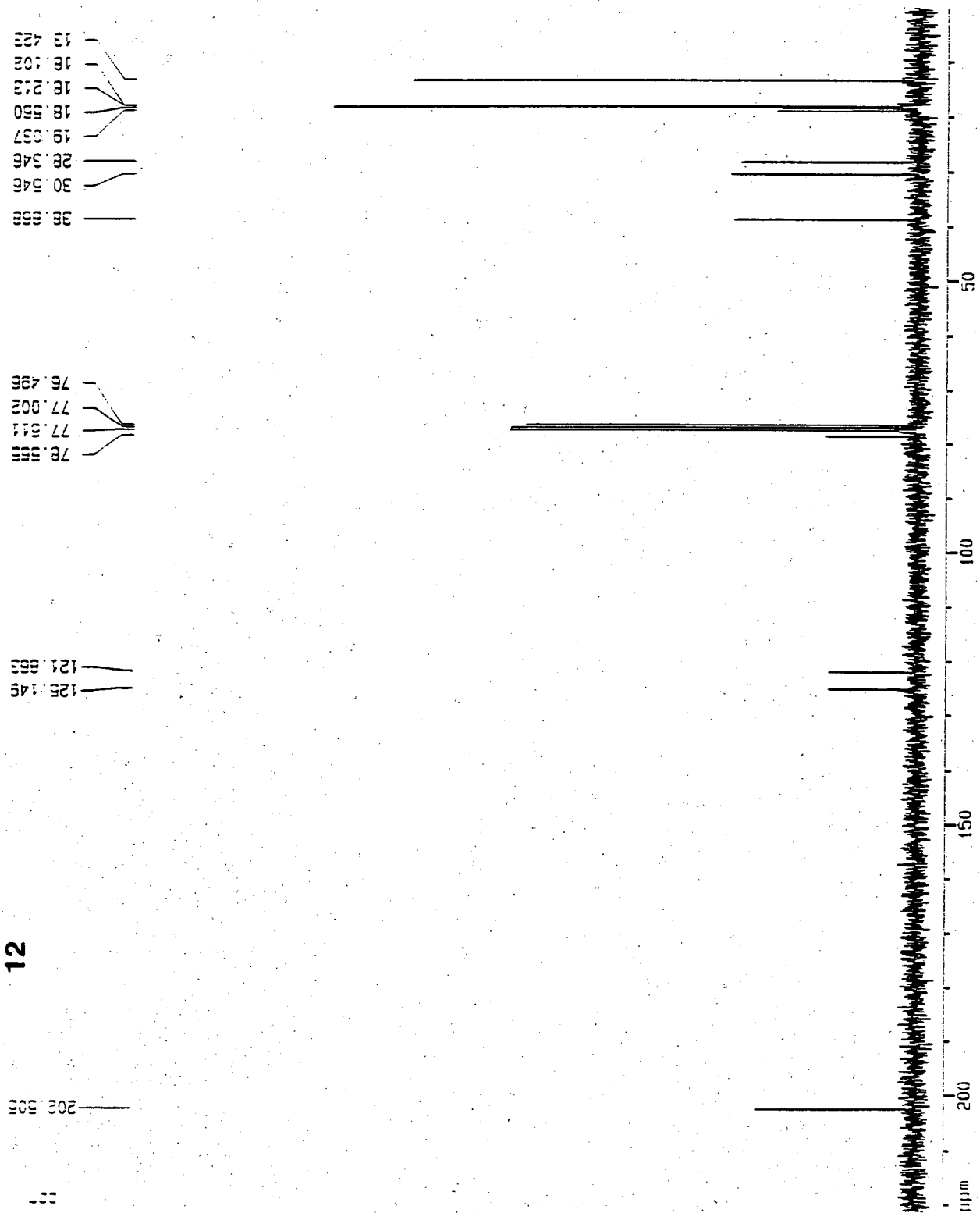
10 MHz pilot parameters	
CX	20 00 cm
FIP	210 591 ppm
F1	13245 15 Hz
2P	0 979 ppm
F2	61 56 Hz
ppmCM	10 40060 ppm/cm
HZCM	659 17969 Hz/cm

USIV-44-A2



US-IV-44-A2

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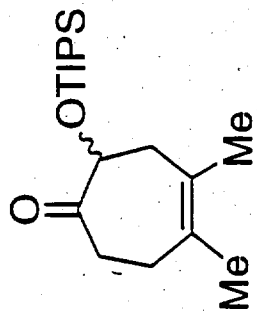
Current Data Parameters

NAME	US-IV 44-A?
EXPNO	2
PROCNO	1

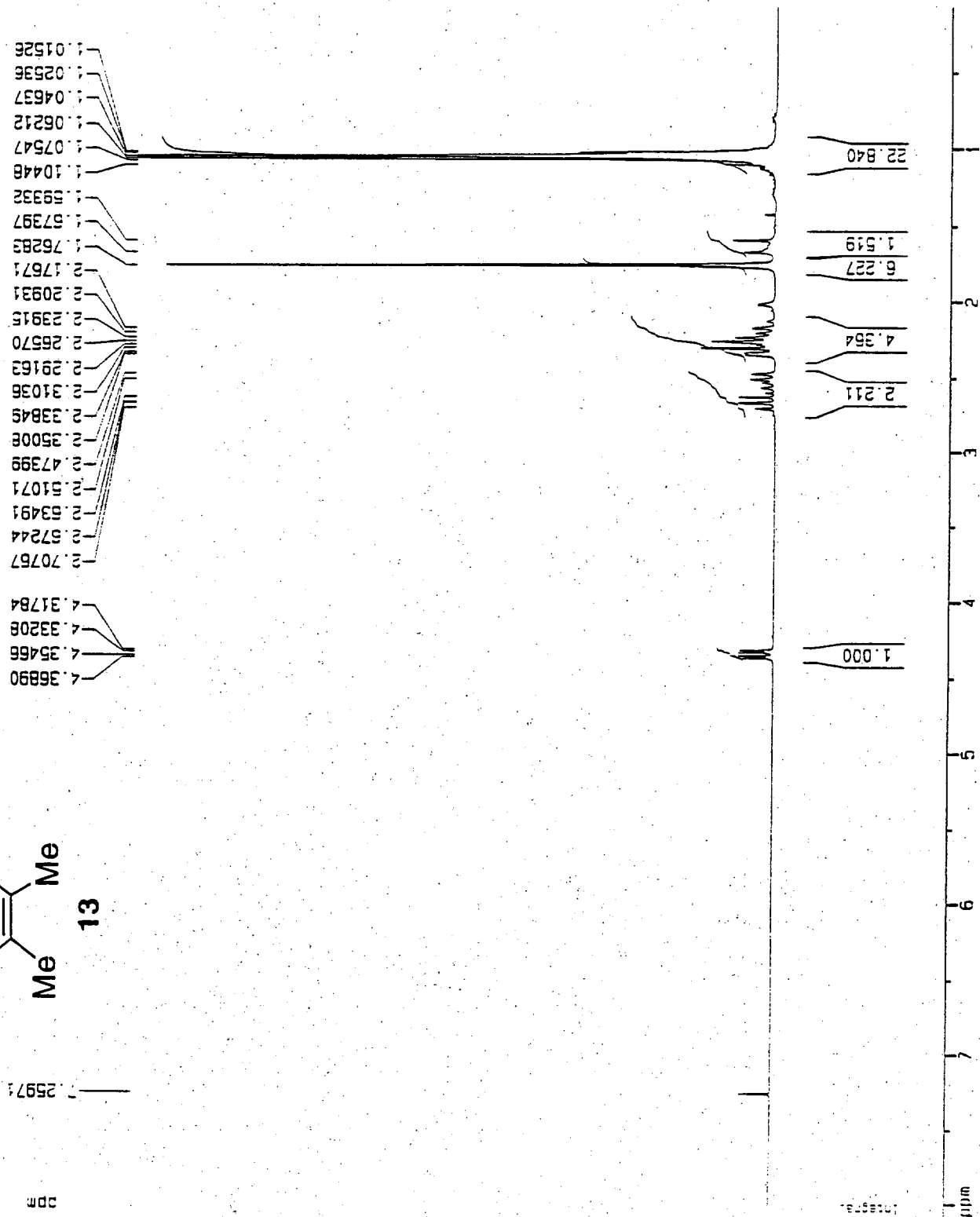
F2 - Acquisition Parameters	
Date:	990803
Time	19 07
INSTRUM	APX250
PROBHD	5 mm QNP 1H
PULPROG	zgdc30
TD	32764
SOLVENT	CDCl3
NS	293
DS	4
SWH	17241.379 Hz
FIDRES	0.467702 Hz
AQ	1.06391060 sec
RG	22800
AW	29.000 usec
DE	41.43 usec
TE	300.0 K
D12	0.0002000 sec
D15	23.00 dB
CPDPRG	waltz16
PP31	103.00 usec
D1	1.00000000 sec
P1	5.35 usec
DE	41.43 usec
SF01	62.9023694 MHz
NUC1	13C
D11	0.0300000 sec

F2 - Processing parameters	
SI	32768
SE	62 0932.980 MHz
MD	FM
SSB	0
FB	1 00 Hz
GB	0
PC	1.40

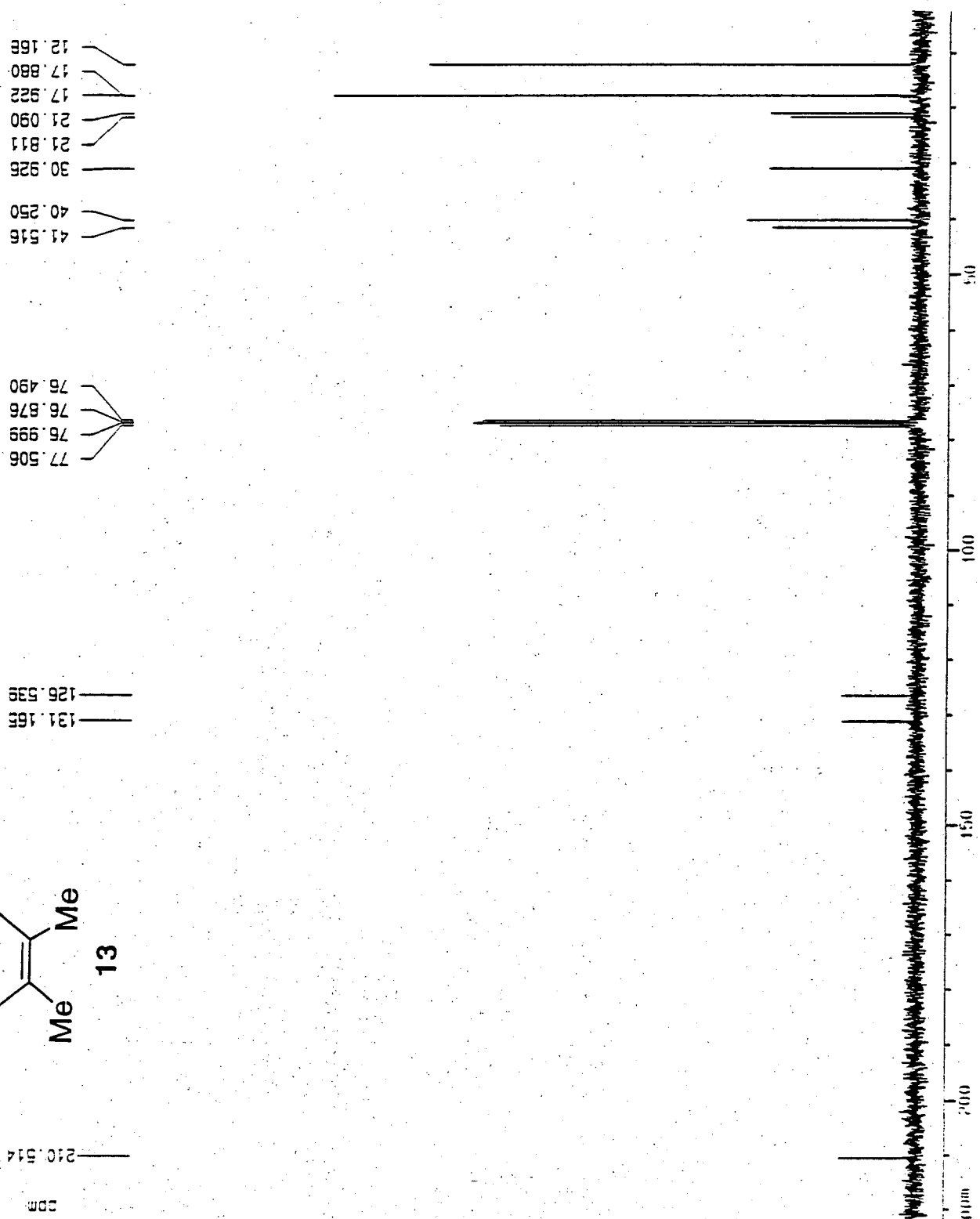
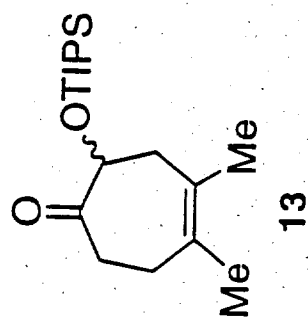
10 NH ₄ plot parameters	
CX	20.00 cm
FIP	221.211 ppm
F1	13913.14 Hz
F2P	0.167 ppm
F2	10.51 Hz
PMCM	11.05624 ppm/cm
HZCM	689.1314 Hz/cm



US-IV-44-A3



Current Data Parameters	
NAME	US-IV-44-A3
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
Date_	990804
Time	8.34
INSTRUM	arx250
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	5208.333 Hz
FIDRES	0.150946 Hz
AQ	3.145779 sec
RG	715
DW	96.000 usec
DE	137.14 usec
TE	300.0 K
D1	1.00000000 sec
P1	8.70 usec
DE	137.14 usec
SFO1	250.1315321 MHz
NUCLEUS	1H
F2 - Processing parameters	
SI	16384
SF	250.1300076 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.50
1D NMR plot parameters	
CX	20.00 cm
FIP	8.079 ppm
F1	2020.72 Hz
F2	0.057 ppm
PPMCM	14.23 Hz
HZCM	0.40109 ppm/cr
	100 32446 Hz/cm



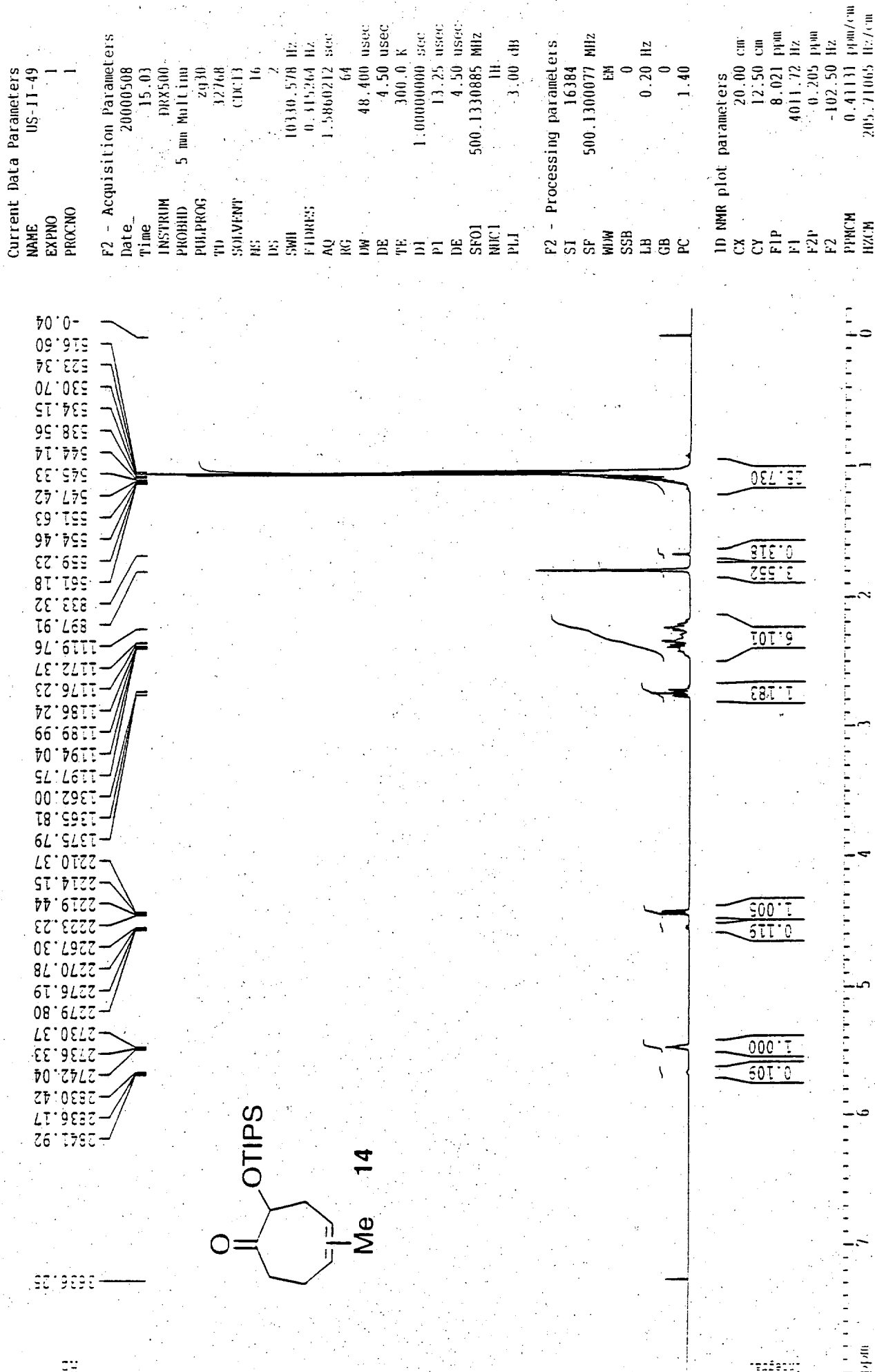
Current Data Parameters	
NAME	US-IV-44-A3
EXPNO	2
PROCNO	1

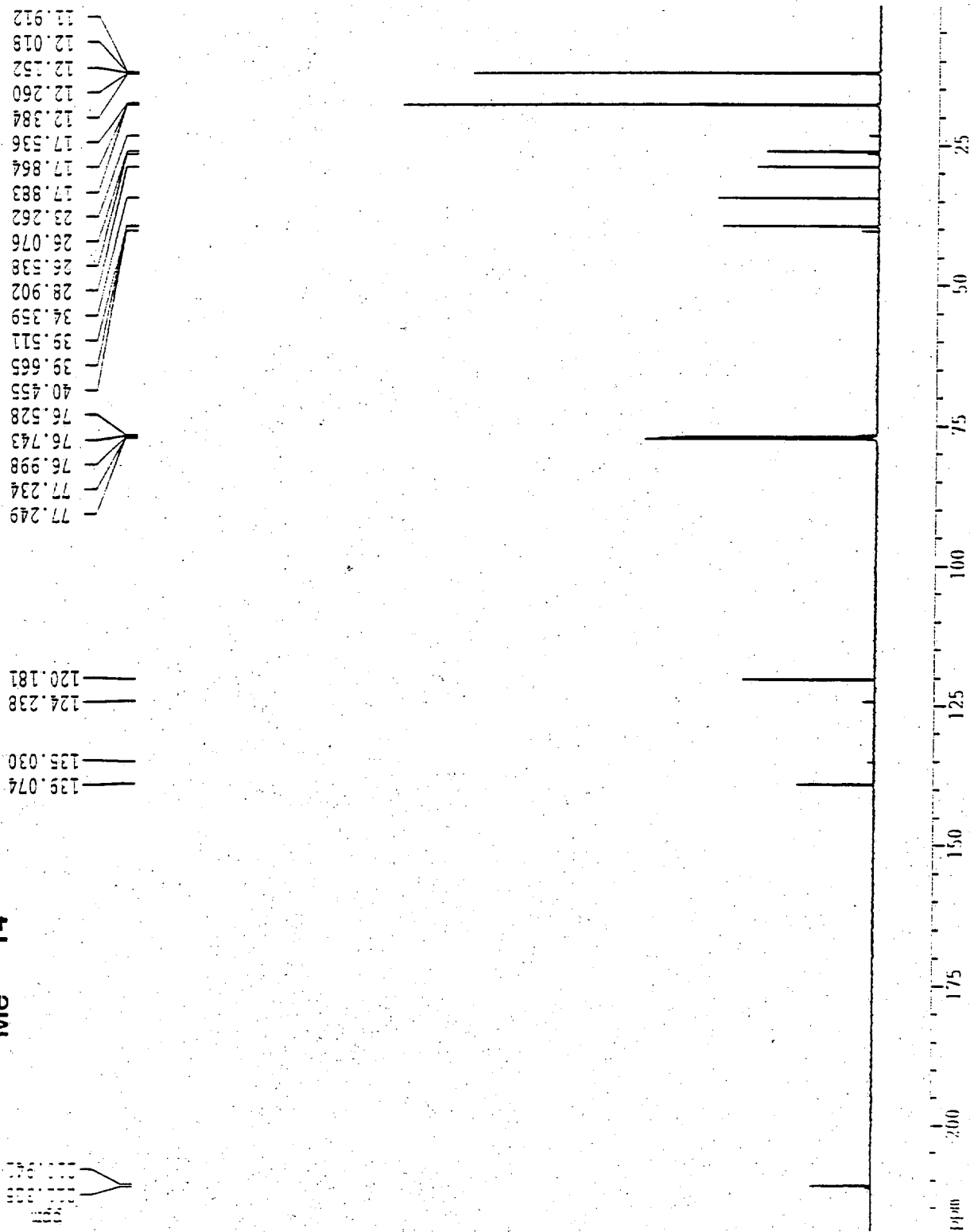
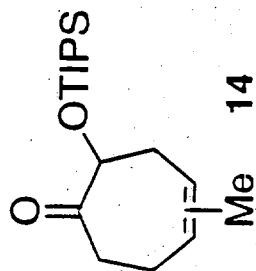
F2 - Acquisition Parameters	
Date_	990804
Time	0 40
INSTRUM	APX250
PROBHD	5 mm QNP 1H
PULPROG	zgpg30
TD	36864
SOLVENT	CDCl3
NS	5/8
DS	4
SWH	17241.379 Hz
FIDUHS	0.46702 Hz
AQ	1.0691060 sec
RG	22800
RGW	29.000 usec
DE	41.43 usec
TE	300.0 K
D12	0.0000200 sec
D15	23.00 dB
PCPRPG	waltz16
P31	103.00 usec
D1	1.0000000 sec
P1	5.35 usec
DE	41.43 usec
SF01	62.9023694 MHz
NUCLEUS	13C
D11	0.0300000 sec

F2 - Processing parameters	
SI	32768
SF	62.0952403 MHz
WDM	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters	
CX	20.00 cm
F1P	221.765 ppm
F1	13947.595 Hz
F2P	2.406 ppm
F2	151.31 Hz
PPMCM	10.96795 ppm/
HzCM	689.83324 Hz/cm

US-II-49





Current Data Parameters
NAME US-II-49
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date 20000508
Time 15.12
INSTRUM DRX500
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 1047
DS 4
SWH 39682.519 Hz
FIDRES 0.605507 Hz
AQ 0.8258036 sec
RG 4096
IM 12.600 usec
DE 4.50 usec
TE 298.0 K
d11 0.0300000 sec
PL12 21.00 dB
CPDPRG2 waltz16
PCPD2 107.00 usec
SF02 500.1320005 MHz
NUC2 1H
PL2 0.00 dB
D1 1.0000000 sec
P1 8.24 usec
DE 4.50 usec
SF01 125.7713108 MHz
NUC1 13C
PL1 3.00 dB

F2 - Processing parameters
SI 32768
SF 125.7577934 MHz
WIM EX
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 219.397 ppm
F1 27590.88 Hz
F2P 0.165 ppm
F2 20.74 Hz
PPHM 10.96160 ppm/cm
HZCM 1378.50671 Hz/cm