JOC The Journal of Organic Chemistry

J. Org. Chem., 1998, 63(1), 129-136, DOI:10.1021/jo971591n

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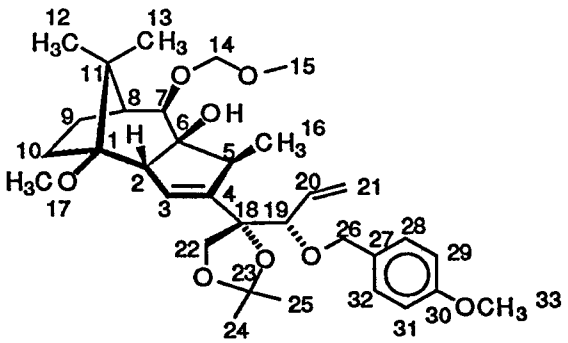
Spectroscopic Details for 11. The IR spectrum indicated the absence of a carbonyl group and the presence of a hydroxyl group at 3510 cm^{-1}. The ^{1}H NMR and ^{13}C/DEPT spectra suggested that a methoxy group had been incorporated and an alkene had disappeared. A quaternary carbon at 80.9 ppm suggested that a C-O bond had formed at C-1. Cross peaks observed in the COSY spectrum between H-3 and H-5 and H-2 suggested that the double bond at C3-C4 had remained intact. NOE interactions observed between H-2/H-3 and H-2/H-17 also supported the above assignment. The above data also showed that the sidechain (C18-C33) was unaltered. The methyl group at C-5 was assigned as β due to the NOE observed between H-5 and H-7. Therefore, the addition of a methoxy group to the bridgehead carbon followed by a transannular C-C bond formation between C-2 and C-6 had occurred.



The structure has been elucidated by means of ^{1}H, ^{13}C/DEPT, C-H correlation, COSY, NOE, and IR experiments.

^{1}H NMR ($CDCl_3$)

Chemical shift	# of hydrogen	multiplicity	Coupling constants (Hz)	Assigned Carbon #
7.17-7.24	2	m	-	31,32
6.78-6.87	2	m	-	28,29
5.90	1	ddd	7.1, 10.5, 17.2	20
5.81	1	t	2.7	3
5.36	1	d	10.2	21
5.32	1	d	17.1	21
4.75	1	d	6.8	14
4.61	1	d	6.8	14
4.55	1	d	11.3	26
4.31	1	d	11.3	26
4.13	1	d	8.7	22
3.87	1	d	8.7	22
3.85	1	d	7.2	19
3.80	3	s	-	33
3.75	1	br s	-	OH
3.45	1	s	-	7
3.40	3	s	-	15
3.34	3	s	-	17
3.11	1	s	-	2
2.40-2.48	1	br q	7.2	5
1.74-1.97	3	m	-	8,9
1.50-1.55	1	m	-	10
1.48	3	s	-	24
1.34	3	s	-	25
1.15	3	s	-	12
1.02-1.13	1	m	-	10
0.98	3	s	-	13
0.97	3	d	7.1	16

^{13}C/DEPT

Chemical shift	types of carbon	Assigned carbon #
159.0	q	30
146.9	q	27
134.7	CH,CH3	20
130.1	q	4
129.7(X2)	CH,CH3	31,32
126.6	CH,CH3	3
119.2	CH2	21
113.6(X2)	CH,CH3	28,29
109.3	q	23
96.2	CH2	14
85.8	q	18
85.1	q	6
83.0	CH,CH3	19
81.2	CH,CH3	7
80.9	q	1
70.9(X2)	CH2	22,26
60.1	CH,CH3	2
55.9	CH,CH3	17
55.2	CH,CH3	33
53.5	CH,CH3	15
52.3	CH,CH3	5
49.4	CH,CH3	8
45.1	q	11
26.7	CH,CH3	(12,13,16,24,25)
25.6	CH,CH3	(12,13,16,24,25)
25.4	CH2	9
24.3	CH2	10
23.4	CH,CH3	(12,13,16,24,25)
21.5	CH,CH3	(12,13,16,24,25)
10.1	CH,CH3	(12,13,16,24,25)

C-H Correlation

Chemical Shift (13C, ppm)	Cross peak (1H, ppm)	Assigned carbon #
134.7	5.9	20
130.1	-	-
129.7	7.17-1.24	28,29
126.6	5.81	3
119.2	5.32, 5.36	21
109.3	-	-
113.6	6.78-6.87	31,32
96.2	4.75, 4.61	14
85.8	-	-
85.1	-	-
83.0	3.85	19
81.2	3.45	7
80.9	-	-
70.9	3.87, 4.13	
	4.61, 4.75	22,26
60.1	3.11	2
55.9	3.40	15
55.2	3.80	33
53.5	3.34	17
52.3	2.40-2.48	5
49.4	1.74-1.97	8
45.1	-	-

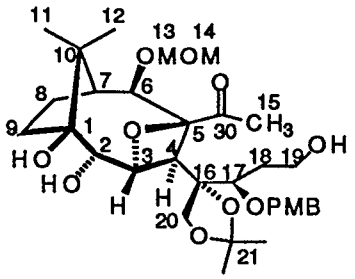
COSY

Chemical Shift (ppm)	Cross Peak (ppm)	Assigned carbon #
5.81	2.40-2.48	5
	3.11	2

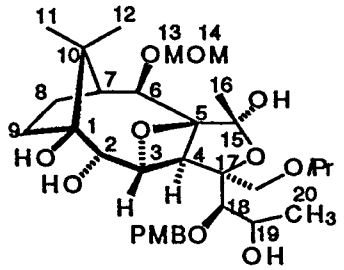
NOE

Irradiation (ppm)	carbon #	Observed NOE (ppm, assigned carbon#)		(% enhancement)
2.40-2.48	C-5	3.47,	C-7	8.54
3.11	C-2	5.81,	C-3	10.37
		3.34,	C-17	positive NOE
		1.15,	C-13	7.14
3.45	C-7	4.61, 4.75,	C-14	4.20, 2.46
		2.40-2.48,	C-5	7.69
		1.93,	C-8	3.37

Spectroscopic Details for the Hydroboration Products of 20.



For the primary alcohol: IR (neat, cm^{-1}) 3428, 1712, 1614, 1515, 1249, 1074, 1017; ^1H NMR (300 MHz, $CDCl_3$) δ 7.22 (d, *J* = 8.5 Hz, 2 H), 6.86 (d, *J* = 8.5 Hz, 2 H), 5.01 (dd, *J* = 5.3, 3.8 Hz, 1 H), 4.71 (d, *J* = 10.6 Hz, 1 H), 4.51 (d, *J* = 6.8 Hz, 1 H), 4.49 (d, *J* = 10.6 Hz, 1H), 4.36 (d, *J* = 6.8 Hz, 1 H), 3.98 (dd, *J* = 10.0, 1.9 Hz, 1 H), 3.95 (dd, *J* = 10.0, 3.8 Hz, 1 H), 3.91 (d, *J* = 8.3 Hz, 2 H), 3.80 (s, 3 H), 3.79 (d, *J* = 5.7 Hz, 2 H), 3.88-3.71 (m, 2 H), 3.52 (d, *J* = 8.3 Hz, 1 H), 3.40 (d, *J* = 5.3 Hz, 1 H), 3.31 (s, 3 H), 3.16 (d, *J* = 10.0 Hz, 1 H), 2.86 (s, 1 H), 2.51 (dd, *J* = 3.8, 0.3 Hz, 1 H), 2.33 (s, 3 H), 2.36-2.21 (m, 1 H), 2.18-2.07 (m, 1 H), 2.03-1.94 (m, 1 H), 1.45 (s, 3 H), 1.41 (s, 3 H), 1.40 (s, 3 H), 1.45-1.20 (series of m, 4 H), 1.13 (s, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) ppm 213.8, 159.8, 130.2, 129.2, 114.0, 111.7, 97.4, 90.3, 86.9, 85.9, 83.8, 80.8, 76.8, 76.6, 75.1, 70.2, 60.4, 56.2, 55.3, 50.9, 46.5, 39.1, 33.3, 30.8, 30.7, 29.9, 29.0, 25.4, 25.2, 20.1; FAB MS *m/z* (M^++H) calcd 609.33, obsd 609.65; $[\alpha]_D^{22}$ +11.9 (*c* 0.85, $CHCl_3$).



For the secondary alcohol: IR (neat, cm^{-1}) 3360, 1613, 1514; ^1H NMR (300 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.6 Hz, 2 H), 6.88 (d, *J* = 8.6 Hz, 2 H), 5.61 (s, 1 H), 5.00 (d, *J* = 10.6 Hz, 1 H), 4.87 (d, *J* = 6.9 Hz, 1 H), 4.73 (dd, *J* = 3.5, 3.5 Hz, 1 H), 4.65 (d, *J* = 6.9 Hz, 1 H), 4.61 (d, *J* = 10.6 Hz, 1 H), 4.07 (br d, *J* = 4.0 Hz, 1 H), 3.92 (d, *J* = 4.4 Hz, 1

H), 3.86-3.81 (m, 2 H), 3.81 (s, 3 H), 3.77-3.63 (m, 2 H), 3.56-3.43 (series of m, 4 H), 3.45 (s, 3 H), 2.67 (dd, J = 10.0, 4.4 Hz, 1 H), 2.42-2.27 (m, 2 H), 2.04 (s, 1 H), 1.75-1.59 (m, 2 H), 1.55 (s, 3 H), 1.48 (s, 3 H), 1.22 (d, J = 6.4 Hz, 9 H), 1.18 (s, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) ppm 159.3, 130.7, 130.2, 113.7, 106.5, 98.5, 95.5, 87.5, 86.9, 86.1, 81.9, 75.8, 75.2, 74.9, 73.1, 70.8, 69.2, 55.9, 55.3, 52.6, 46.8, 43.7, 31.5, 30.7, 23.9, 22.0, 21.88, 21.86, 21.6, 20.2; FAB MS *m/z* (M^++H) calcd 611.34, obsd 611.50; $[\alpha]_D^{22}$ +71.3 (*c* 0.70, $CHCl_3$).

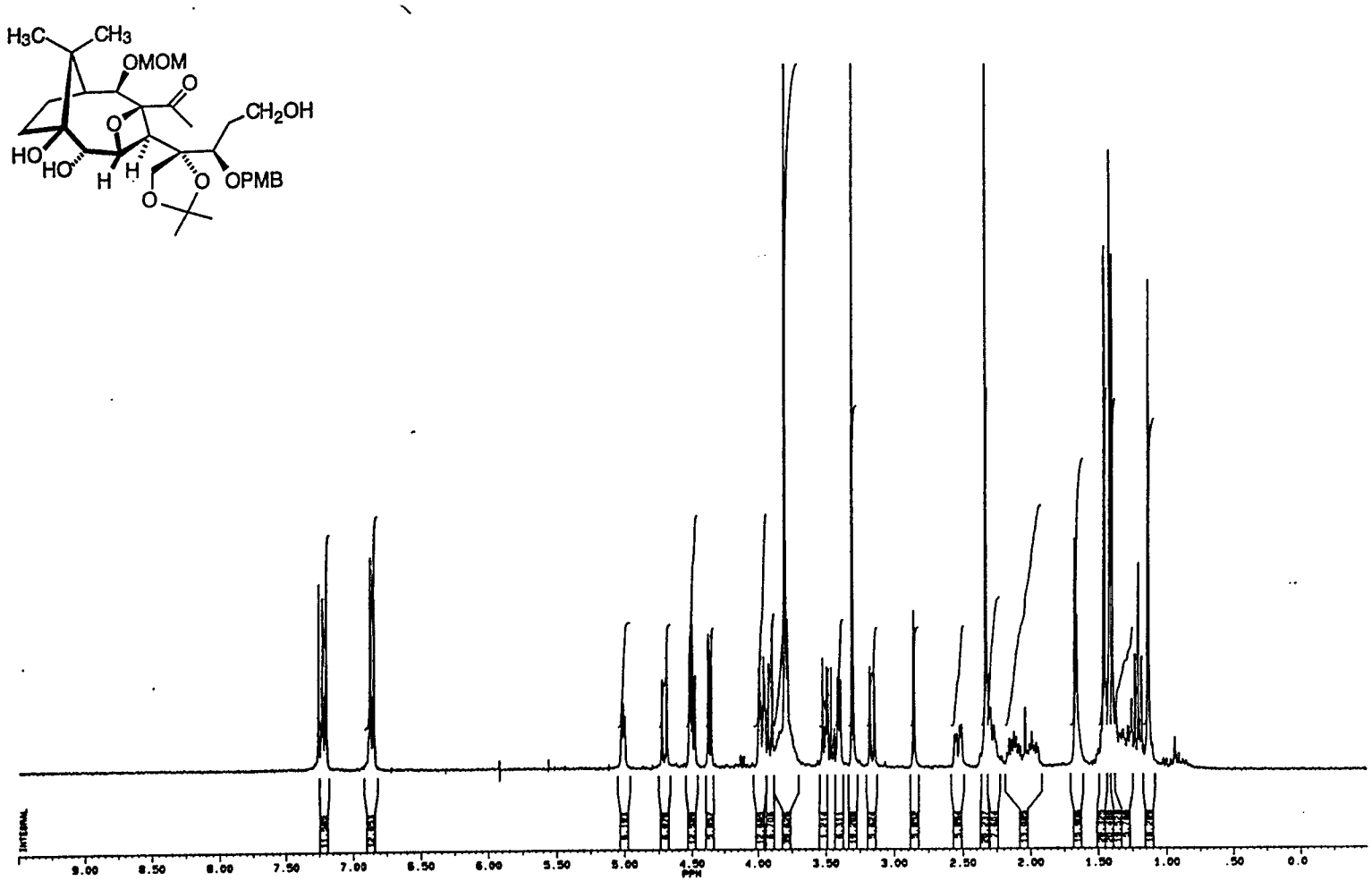


Figure 1. Proton NMR spectrum of Compound 1 in $CDCl_3$ at 500 MHz.

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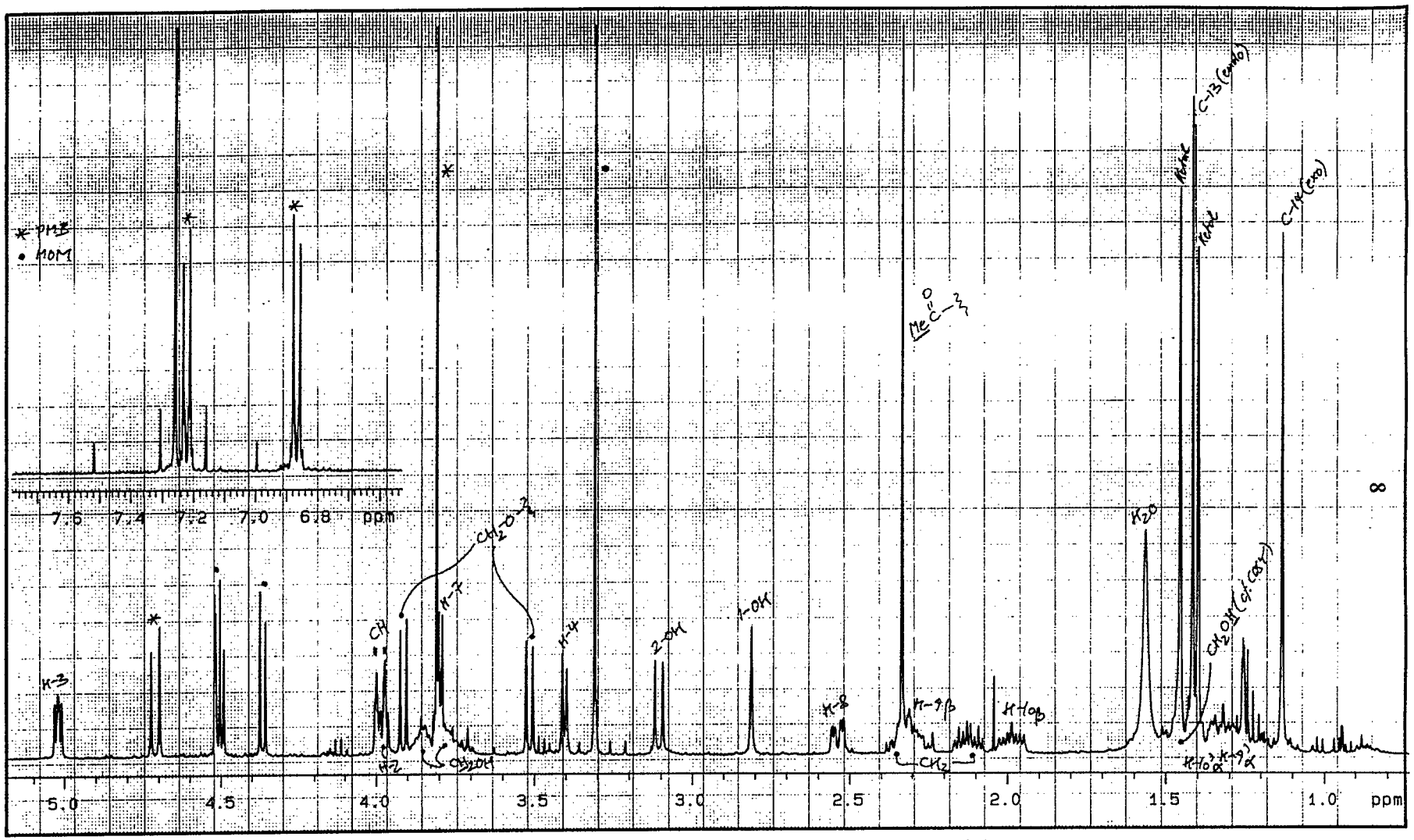

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Modulation Mode	ccc	Freq.	not used Hz
Pulse Width	— μsec	Power Mode	30

PLOT/PROCESSING			
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LB	not used Hz	GB	not used sec CCD
Width	1779.8 Hz/ppm	Start	291.3 Hz/ppm
Reference			

EXPERIMENT			
Pulse Sequence	s2pul		
Tube O.D.	mm		
Temp.	28.0°C		
Solvent	CDCl3		

SAMPLE

JNJ2A
IV-JNJ-288B Johnston
Solvent: CDCl3
Operator: DF
gain=52/ct=256

2 of 2

Number	
File	JNJ2A
Date	Jun 26 97
XL	400

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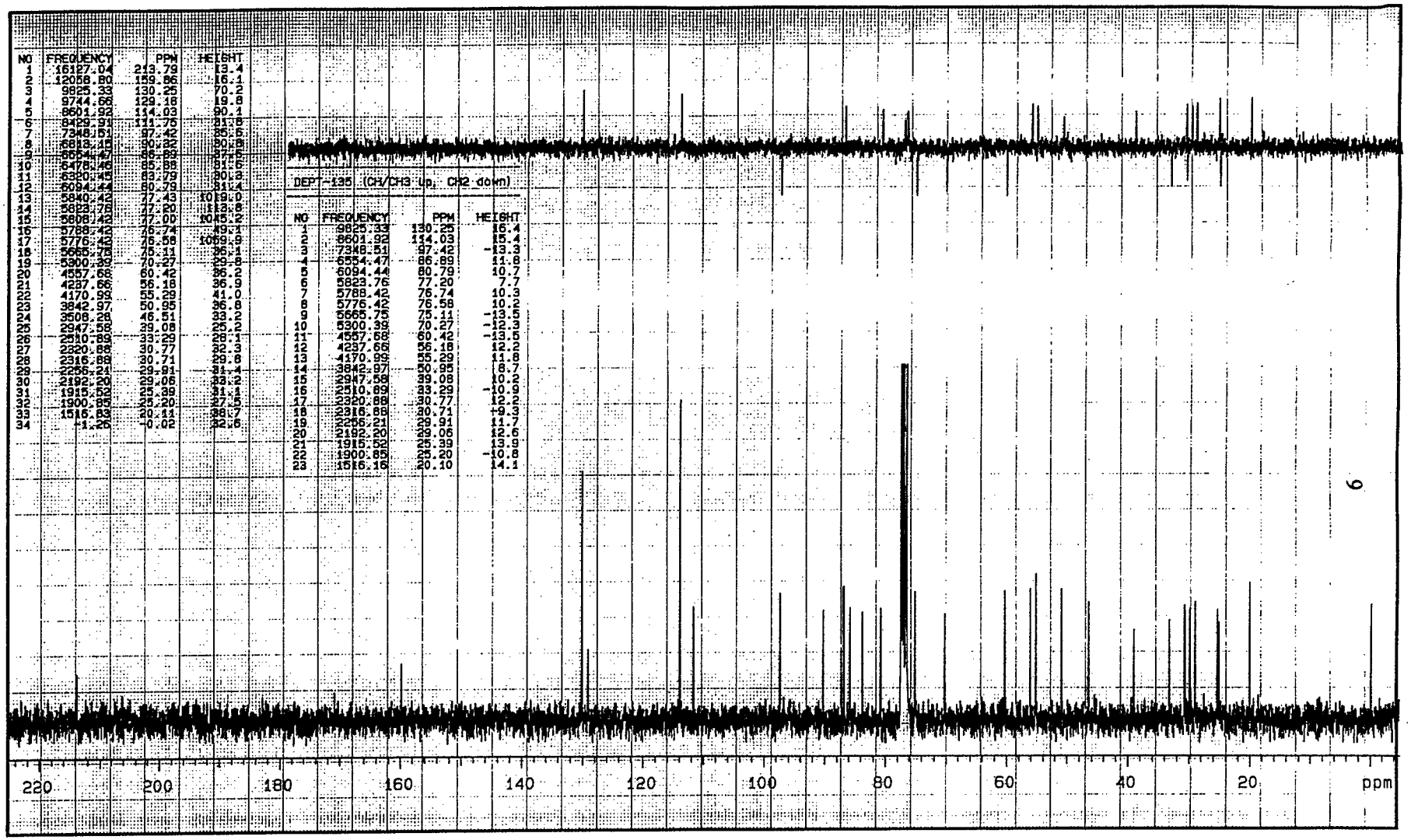
NO	FREQUENCY	PPM	HEIGHT
1	16127.04	213.79	13.4
2	12058.80	159.86	16.1
3	9825.33	130.25	70.2
4	9744.66	129.18	19.8
5	8601.92	114.03	90.1
6	8429.91	111.75	31.8
7	7348.51	97.42	35.5
8	6813.45	90.32	30.8
9	6554.47	86.89	37.5
10	6478.46	85.88	31.6
11	6320.45	83.79	30.3
12	6094.44	80.79	31.4
13	5840.42	77.43	1019.0
14	5823.76	77.20	1113.8
15	5808.42	77.00	1045.2
16	5788.42	76.74	49.1
17	5776.42	76.58	1059.9
18	5665.75	75.11	36.1
19	5300.39	70.27	29.8
20	4557.68	60.42	36.2
21	4237.66	56.18	36.9
22	4170.99	55.29	41.0
23	3842.97	50.95	36.8
24	3508.28	46.51	33.2
25	2947.58	39.08	25.2
26	2510.89	33.29	28.1
27	2320.88	30.77	32.3
28	2316.88	30.71	29.8
29	2256.21	29.91	31.4
30	2192.20	29.06	33.2
31	1915.52	25.39	31.1
32	1900.85	25.20	27.5
33	1516.83	20.11	38.7
34	-1.26	-0.02	32.6

DEPT-135 (CH/CH3 Up, CH2 down)

NO	FREQUENCY	PPM	HEIGHT
1	9825.33	130.25	16.4
2	8601.92	114.03	15.4
3	7348.51	97.42	-13.3
4	6554.47	86.89	11.8
5	6094.44	80.79	10.7
6	5823.76	77.20	7.7
7	5788.42	76.74	10.3
8	5776.42	76.58	10.2
9	5665.75	75.11	-13.5
10	5300.39	70.27	-12.3
11	4557.68	60.42	-13.5
12	4237.66	56.18	12.2
13	4170.99	55.29	11.8
14	3842.97	50.95	8.7
15	2947.58	39.08	10.2
16	2510.89	33.29	-10.9
17	2320.88	30.77	12.2
18	2316.88	30.71	+9.3
19	2256.21	29.91	11.7
20	2192.20	29.06	12.6
21	1915.52	25.39	13.9
22	1900.85	25.20	-10.8
23	1516.16	20.10	14.1



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Pulse Width: 10.5 μsec — Transients: 16000

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Nucleus: H1 — Offset: -300.0 Hz
Mode: nyy — Power: not used db
Modulation Mode: CWW — Freq.: 11800 Hz
Pulse Width: 14.0 μsec — Power Mode: 41

PLOT/PROCESSING
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LB: 1.000 Hz — Not used sec — CCD:
Width: 17349.0 Hz/ppm — Start: -377.3 Hz/ppm
Reference:

EXPERIMENT
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Tube O.D.: mm
Temp.: 25.0 °C
Solvent: CDCl3

SAMPLE
JNJ2B
IV-JNJ-288B Johnston
Solvent: CDCl3
Operator: DF
gain=60/ct=16000

Number:
File: JNJ2B
Date: Jun 27 97
XL: 400

PART NO. 997239
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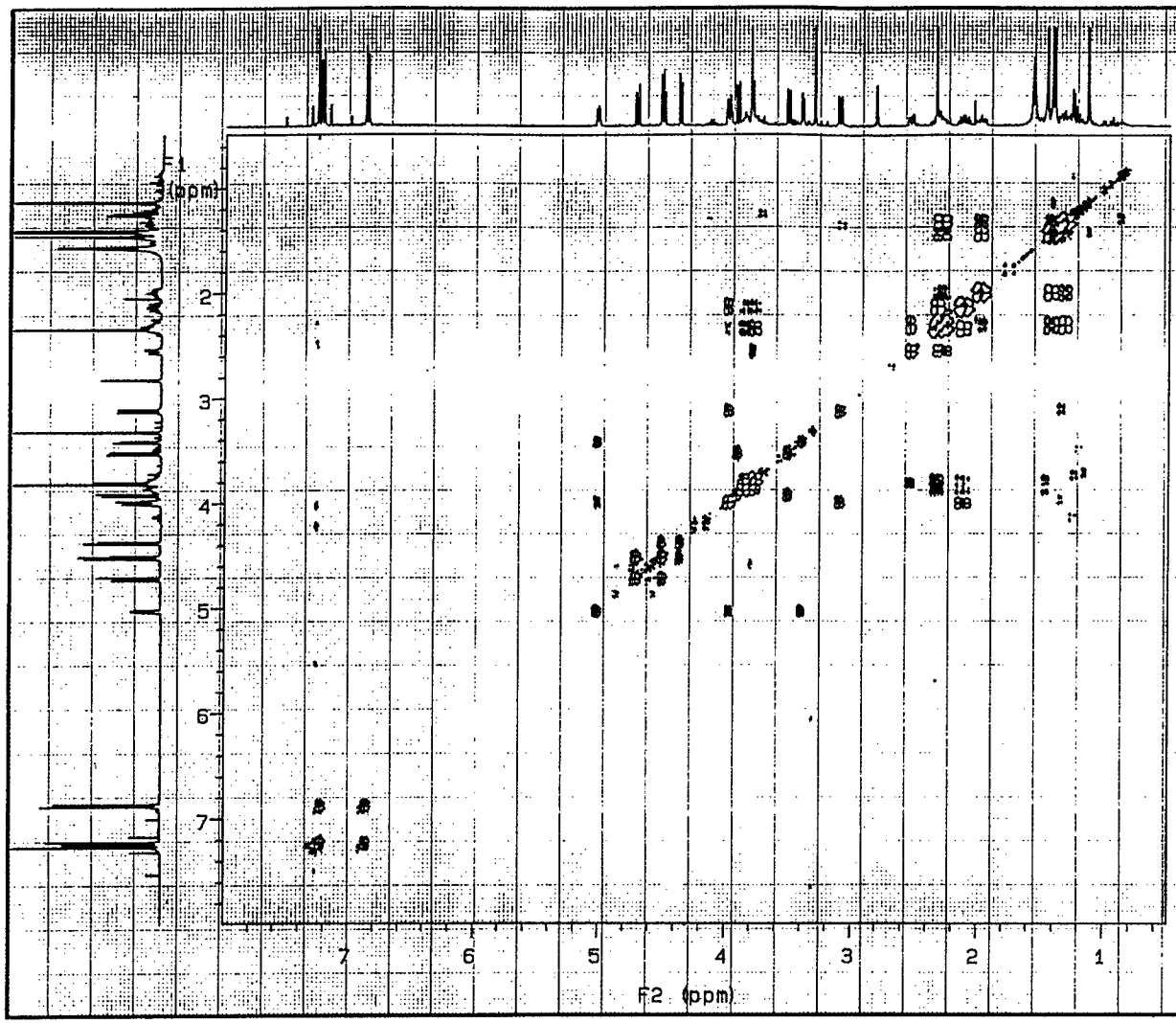


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JNJ2C
IV-JNJ-288B  Johnston        ****************
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Operator: DF                 ****************

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NJ-Cpds/JNJ2/JNJ2C      dmm            c
    ACQUISITION         dmf       not used
sfrq        399.937     dpwr           30
tn               H1     temp         28.0
at            0.256        PROCESSING
np             2048     sb          -0.256
sw           3999.2     sbs         -0.256
fb             2200     phfid        267.0
tpwr             54     wtfile
pw             12.1     proc           ft
d1            1.000     fn           2048
tof          -817.9     math            f
nt               64
ct                0     werr
alock             y     wexp
gain             42     wbs
        FLAGS           wnt
il                n       2D PROCESSING
in                n     sb1         -0.064
dp                y     sbs1        -0.064
hs                n     wtfile1
  2D ACQUISITION        proc1          ft
sw1          3999.2     fn1          1024
ni              256         DISPLAY
    2D DISPLAY          sp           198.3
sp1          195.0      wp          2998.4
wp1         2997.4      vs             200
sc2               0     sc             130
wc2             180     wc             220
rfl1        399.8       hzmm         13.63
rfp1              0     is           93.96
                        rfl          399.8
                        rfp              0
                        th               2
                        ins        100.000
                        ai   cdc   ph
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OBSERVE			
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Pulse Width	12.1 μsec	Transients	0

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Mode	n	Power	not used db
Modulation: Mode	c	Freq.	not used Hz
Pulse Width	μsec	Power Mode	30

PLOT/PROCESSING			
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LB	not used Hz	not used sec	CCD
Width	2998.4 Hz/ppm	Start	198.3 Hz/ppm
Reference			

EXPERIMENT	
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Tube O.D.	mm
Temp.	28.0 °C
Solvent	CDCl3

SAMPLE

JNJ2C
IV-JNJ-288B Johnston
Solvent: CDCl3
Operator: DF

lot2

Number	
File	JNJ2C
Date	Jun 27 97
XL	400

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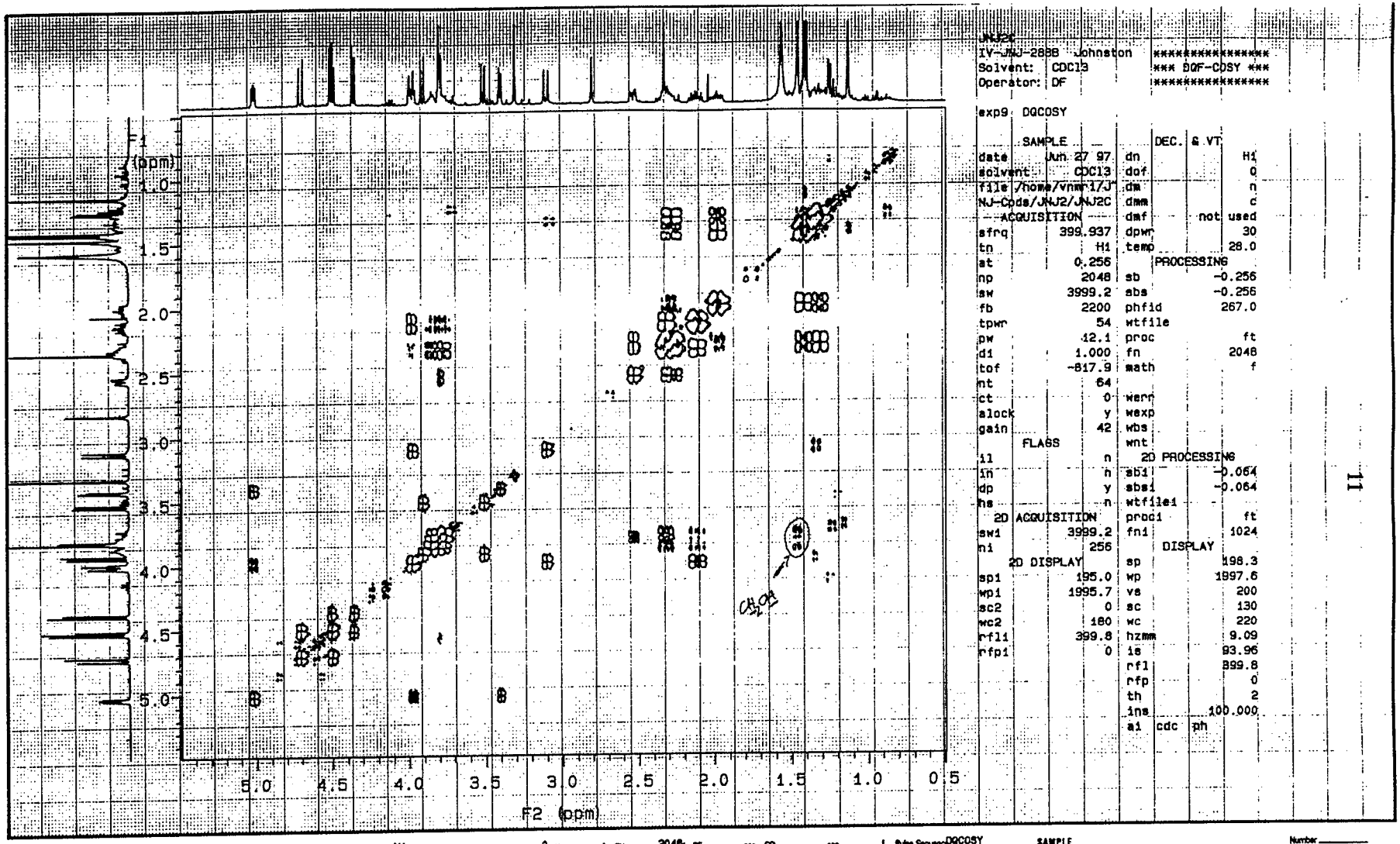

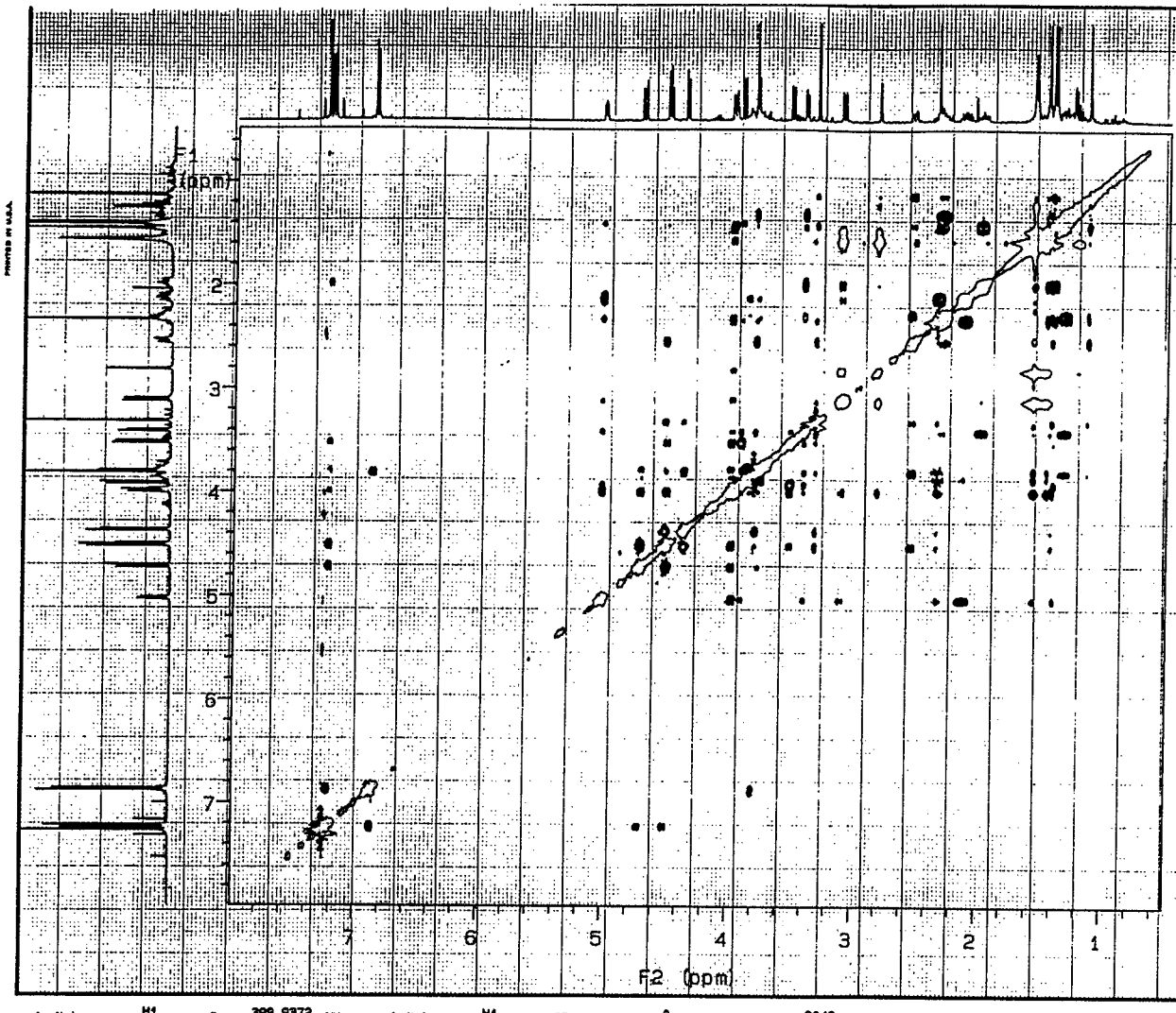


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IV-JNJ-2888 Johnston      ************
Solvent:  CDCl3           *** NOESY ***
Operator: DF              ************

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NJ-Cpds/JNJ2/JNJ2D      dmm                c
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sfrq          399.937   dpwr              30
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sw           3999.2     sbs           -0.256
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ss                8     proc             lp
tpwr             54     fn             2048
pw             12.1     math              f
d1            1.000
tof          -817.9    werr
nt               64    wexp
ct                0    wbs
alock             y    wnt
gain             42      2D PROCESSING
        FLAGS           sbi          -0.064
il                n     sbs1         -0.064
in               -n     wtfile1
dp                y     proc1            ft
hs                n     fn1            1024
  2D ACQUISITION            DISPLAY
sw1          3999.2     sp             198.3
ni              256     wp            2998.4
     2D DISPLAY         vs               333
sp1            195.0    sc               130
wp1          2997.4     wc               220
sc2                0    hzmm           13.63
wc2              160    is             93.96
rfl1           399.8    rfl            399.8
rfp1               0    rfp                0
                        th                 2
                        ins          100.000
                        ai   cdc   ph
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OBSERVE: Nucleus H1; Freq. 399.9372 MHz; Spec. Width 3999.2 Hz; Offset -817.9 Hz; Acq. Time 0.256 sec; Delay 1.000 sec; Pulse Width 12.1 μsec; Transients 0

DECOUPLE: Nucleus H1; Offset 0 Hz; Mode n; Power not used db; Modulation Mode c; Freq. not used Hz; Pulse Width --- μsec; Power Mode 30

PLOT/PROCESSING: FN 2048K; RE sec; CD sec; La not used Hz; RAt used sec; CCD; Width 2998.4 Hz/ppm; Start 198.3 Hz/ppm; Reference

EXPERIMENT: Pulse Sequence NOESY; Tube O.D. mm; Temp. 26.0 °C; Solvent CDCl3

SAMPLE
JNJ2D
IV-JNJ-2888 Johnston
Solvent: CDCl3
Operator: DF

1 of 2

Number; File JNJ2D; Date Jun 28 97; XL 400

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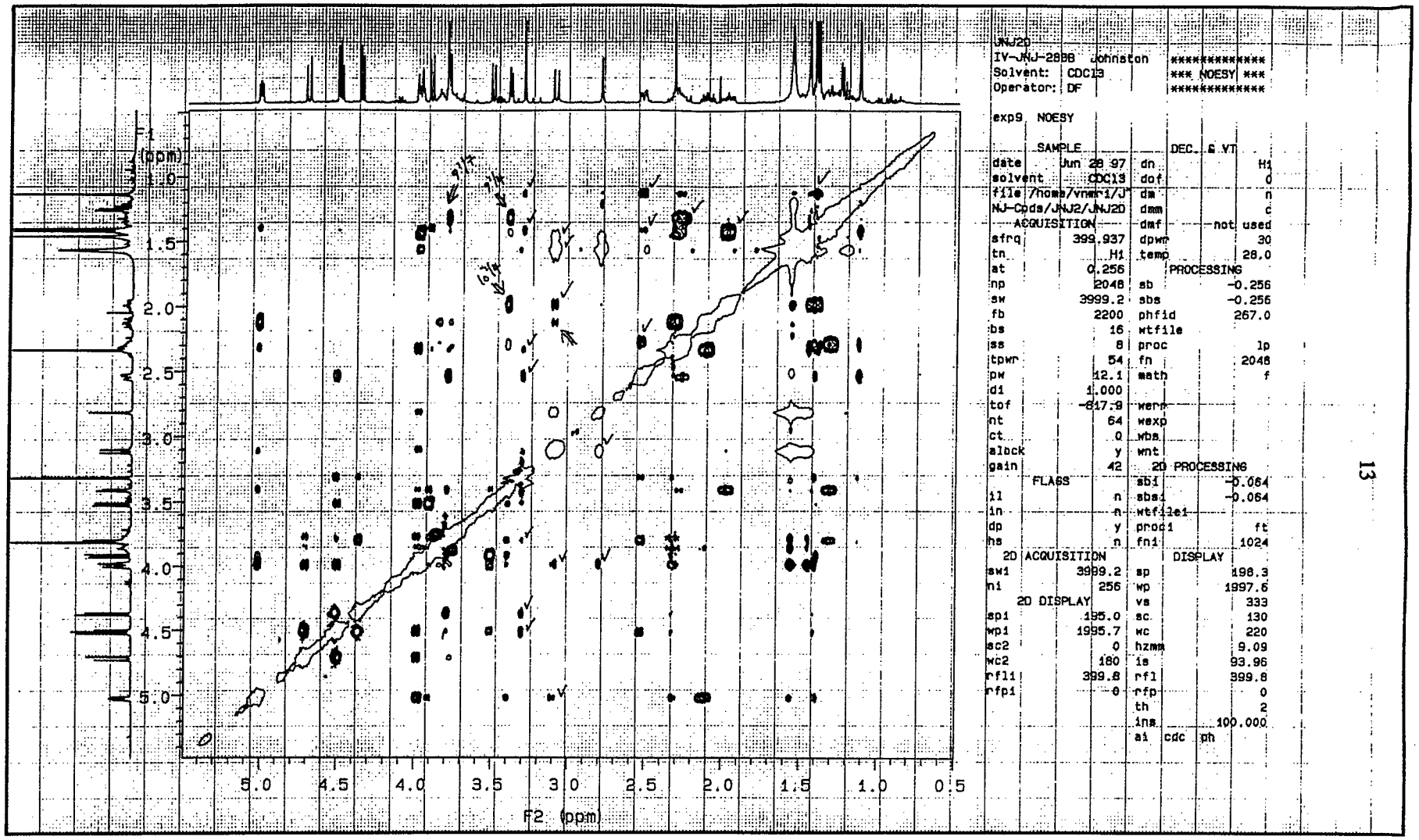

OBSERVE: Nucleus H1; Freq. 399.9372 MHz; Spec. Width 3999.2 Hz; Offset -817.9 Hz; Acq. Time 0.256 sec; Delay 1.000 sec; Pulse Width 12.1 μsec; Transients 0

DECOUPLE: Nucleus H1; Offset 0 Hz; Mode n; Power not used db; Modulation Mode c; Freq. not used Hz; Pulse Width ___ μsec; Power Mode 30

PLOT/PROCESSING: FN 2048; RE ___ sec; CD ___ sec; LB not used Hz; GF not used sec; CCD ___; Width 1997.6 Hz/ppm; Start 198.3 Hz/ppm; Reference ___

EXPERIMENT: Pulse Sequence NOESY; Tube O.D. ___ mm; Temp. 28.0 °C; Solvent CDCl3

SAMPLE
JNJ2D
IV-JNJ-2888 Johnston
Solvent: CDCl3
Operator: DF

2 of 2

Number ___; File JNJ2D; Date Jun 28 97; XL 400

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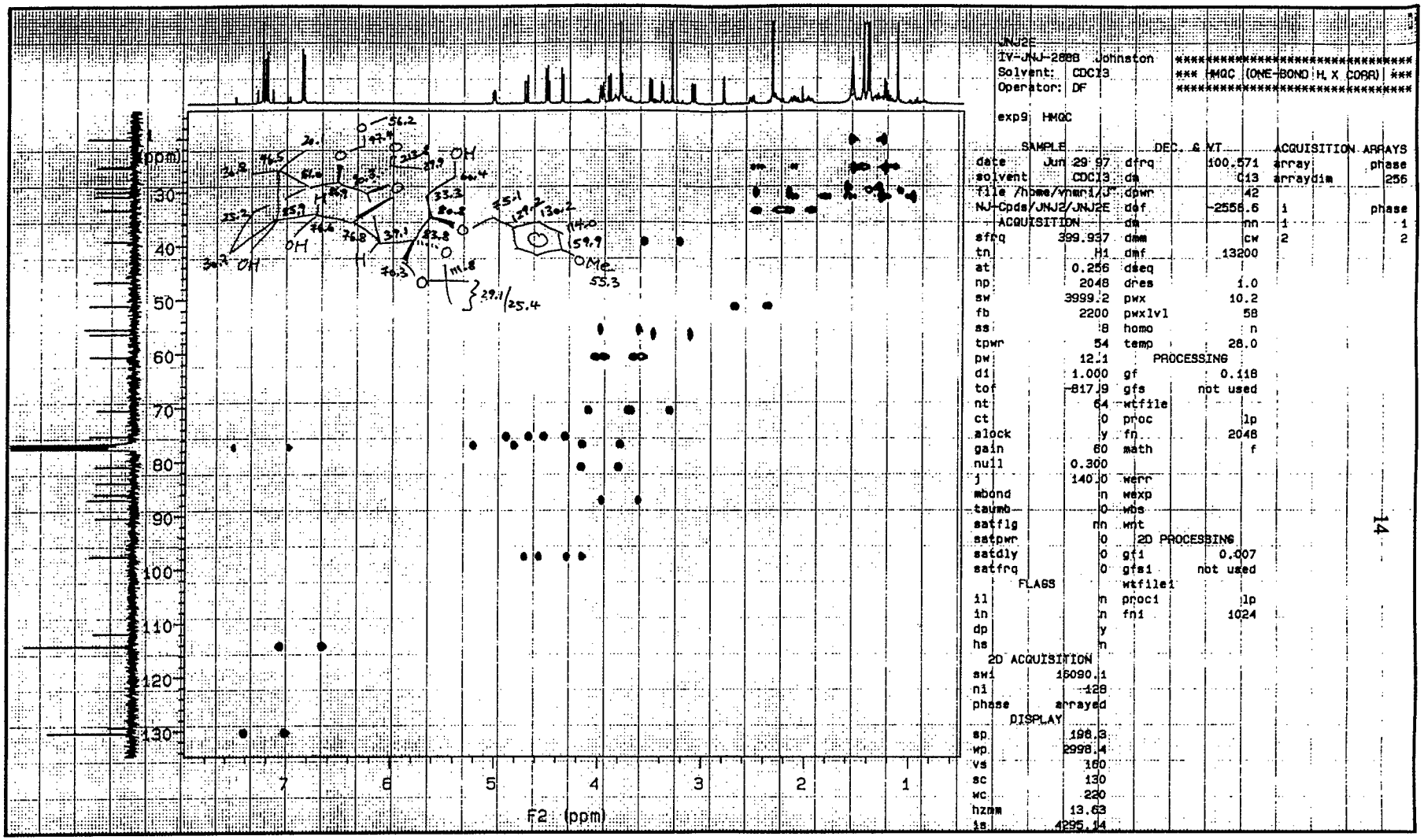

OBSERVE: Nucleus H1; Freq 399.9372 MHz; Spec. Width 3999.2 Hz; Offset -817.9 Hz; Acq. Time 0.256 sec; Delay 1.000 sec; Pulse Width 12.1 μsec; Transients 0

DECOUPLE: Nucleus C13; Offset -2558.6 Hz; Mode nn; Power not used db; Modulation: Mode CW; Freq 13200 Hz; Pulse Width ---- μsec; Power Mode 42

PLOT/PROCESSING: FN 2048; RE sec; CD sec; LB not used Hz; AF 0.118 sec; CCD; Width 2998.4 Hz/ppm; Start 198.3 Hz/ppm; Reference

EXPERIMENT: Pulse Sequence HMQC; Tube O.D. mm; Temp. 28.0°C; Solvent CDCl3

SAMPLE: JNJ2E; IV-JNJ-2888 Johnston; Solvent: CDCl3; Operator: DF

Number; File JNJ2E; Date Jun 29 97; XL 400

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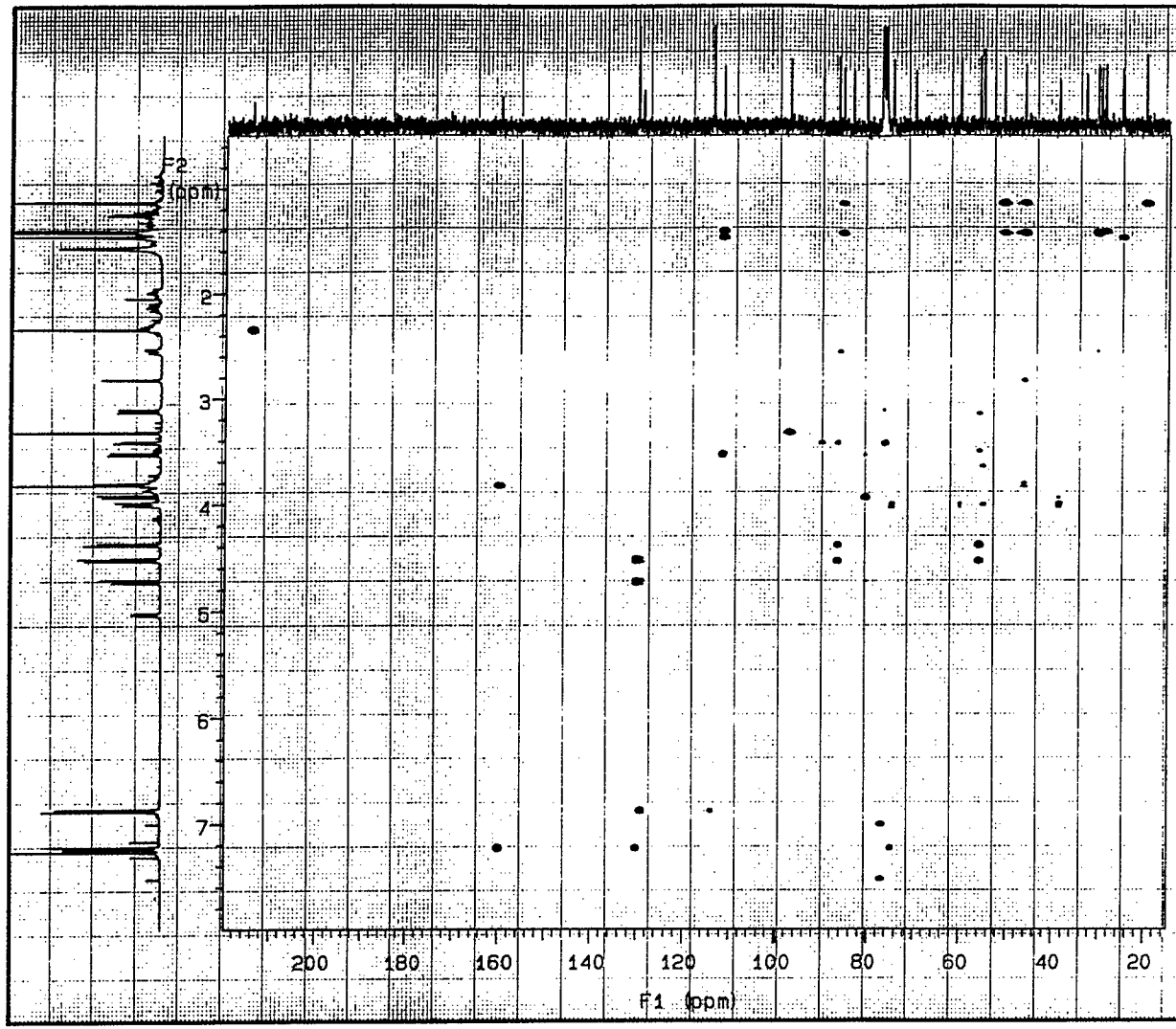

JNJ2F
IV-JNJ-288B Johnston
Solvent: CDCl3
Operator: DF

*** HMBC (LONG-RANGE H,X CORR) ***

exp9: HMQC

SAMPLE		DEC. & VT		ACQUISITION ARRAYS	
date	Jun 29 97	dirq	100.575	array	phase
solvent	CDCl3	dn	C13	arraydim	256
file /home/vnmr1/J		dpwr	42		
NJ-Cpds/JNJ2/JNJ2F		dof	1464.3	1	phase
ACQUISITION		dm	nn	1	1
sfrq	399.937	dmm	cw	2	2
tn	H1	dmf	13200		
at	0.256	dseq			
np	2048	dres	1.0		
sw	3999.2	pwx	10.2		
fb	2200	pwxlvl	58		
ss	8	homo	n		
tpwr	54	temp	28.0		
pw	12.1	PROCESSING			
d1	1.000	gf	0.118		
tof	-817.9	gfs	not used		
nt	256	wtfile			
ct	0	proc	lp		
alock	y	fn	2048		
gain	53	math	f		
null	0				
j	140.0	werr			
wbcnd	y	wexp			
taumb	0.060	wbs			
satflg	nn	wnt			
satpwr	0	2D PROCESSING			
satdly	0	gf1	0.005		
satfrq	0	gfs1	not used		
FLAGS		wtfile1			
il	n	proc1	lp		
in	n	fn1	1024		
dp	y				
hs	n				
2D ACQUISITION					
sw1	24140.0				
ni	128				
phase	arrayed				
DISPLAY					
sp	198.3				
wp	2998.4				
vs	2204				
sc	130				
wc	220				
hzmm	13.63				
is	4295.14				

15

OBSERVE: Nucleus H1; Spec. Width 3999.2 Hz; Acq. Time 0.256 sec; Pulse Width 12.1 μsec; Freq. 399.9372 MHz; Offset -817.9 Hz; Delay 1.000 sec; Transients 0

DECOUPLE: Nucleus C13; Mode nn; Modulation Mode cw; Pulse Width --- μsec; Offset 1464.3 Hz; Power not used db; Freq. 13200 Hz; Power Mode 42

PLOT/PROCESSING: FN 2048; RE sec; CD sec; LB not used Hz; AF 0.118 sec; CCD; Width 2998.4 Hz/ppm; Start 198.3 Hz/ppm; Reference

EXPERIMENT: Pulse Sequence HMQC; Tube O.D. mm; Temp. 28.0 °C; Solvent CDCl3

SAMPLE
JNJ2F
IV-JNJ-288B Johnston
Solvent: CDCl3
Operator: DF

1 of 2

Number; File JNJ2F; Date Jun 29 97; XL 400

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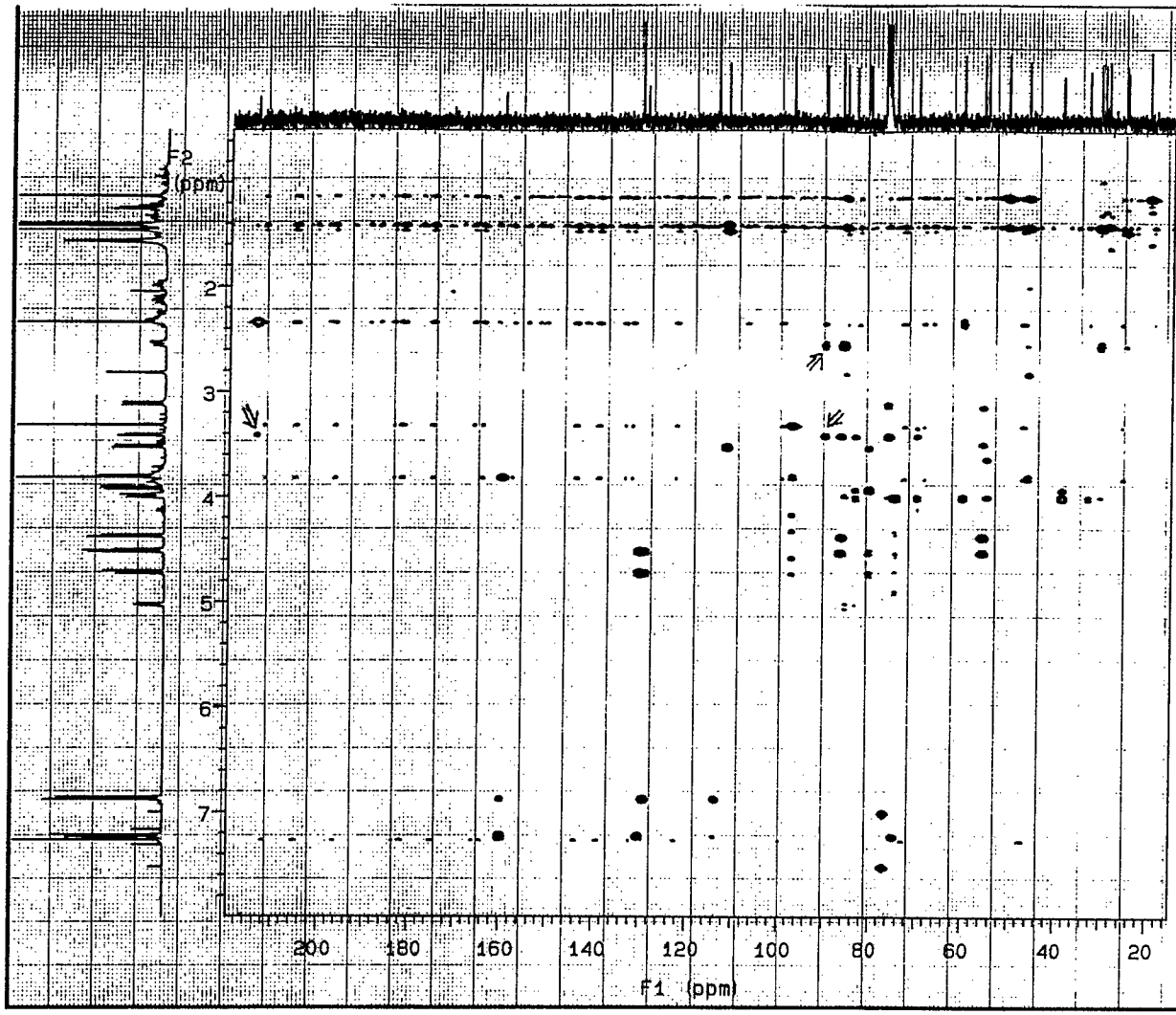

JNJ2F
IV-JNJ-288B Johnston
Solvent: CDCl3
Operator: DF

exp9 HMQC

SAMPLE		DEC. & VT		ACQUISITION ARRAYS	
date	Jun 29 97	dfrq	100.575	array	phase
solvent	CDCl3	dn	C13	arraydim	256
file /home/vnmr1/v		dpwr	42		
NJ-Cpds/JNJ2/JNJ2F		dof	1464.3	1	phase
ACQUISITION		dm	nn	1	1
sfrq	399.937	dmm	cw	2	2
tn	H1	dmf	13200		
at	0.256	dseq			
np	2048	dres	1.0		
sw	3999.2	pwx	10.2		
fb	2200	pwxlvl	58		
ss	8	homo	n		
tpwr	54	temp	28.0		
pw	12.1	PROCESSING			
d1	1.000	gf	0.118		
tof	-817.9	gfs	not used		
nt	256	wtfile			
ct	0	proc	lp		
alock	y	fn	2048		
gain	53	math	f		
null	0				
j	140.0	werr			
mbond	y	wexp			
taumb	0.060	wbs			
satflg	nn	wnt			
satpwr	0	2D PROCESSING			
satdly	0	gf1	0.005		
satfrq	0	gfs1	not used		
FLAGS		wtfile1			
il	n	proc1	lp		
in	n	fn1	1024		
dp	y				
hs	n				
2D ACQUISITION					
sw1	24140.0				
ni	128				
phase	arrayed				
DISPLAY					
sp	198.3				
wp	2998.4				
vs	6400				
sc	130				
wc	220				
hzmm	13.63				
is	4295.14				

16

OBSERVE			
Nucleus	H1	Freq.	399.9372 MHz
Spec. Width	3999.2 Hz	Offset	-817.9 Hz
Acq. Time	0.256 sec	Delay	1.000 sec
Pulse Width	12.1 μsec	Transients	0

DECOUPLE			
Nucleus	C13	Offset	1464.3 Hz
Mode	nn	Power	not used db
Modulation Mode	cw	Freq.	13200 Hz
Pulse Width	--- μsec	Power Mode	42

PLOT/PROCESSING			
FN	2048k	RE	sec
LB	not used Hz	AF	0.118 sec
Width	2998.4 Hz/ppm	Start	198.3 Hz/ppm
Reference			

EXPERIMENT	
Pulse Sequence	HMQC
Tube O.D.	mm
Temp.	28.0°C
Solvent	CDCl3

SAMPLE
JNJ2F
IV-JNJ-288B Johnston
Solvent: CDCl3
Operator: DF

2 of 2

Number
File JNJ2F
Date Jun 29 97
XL 400

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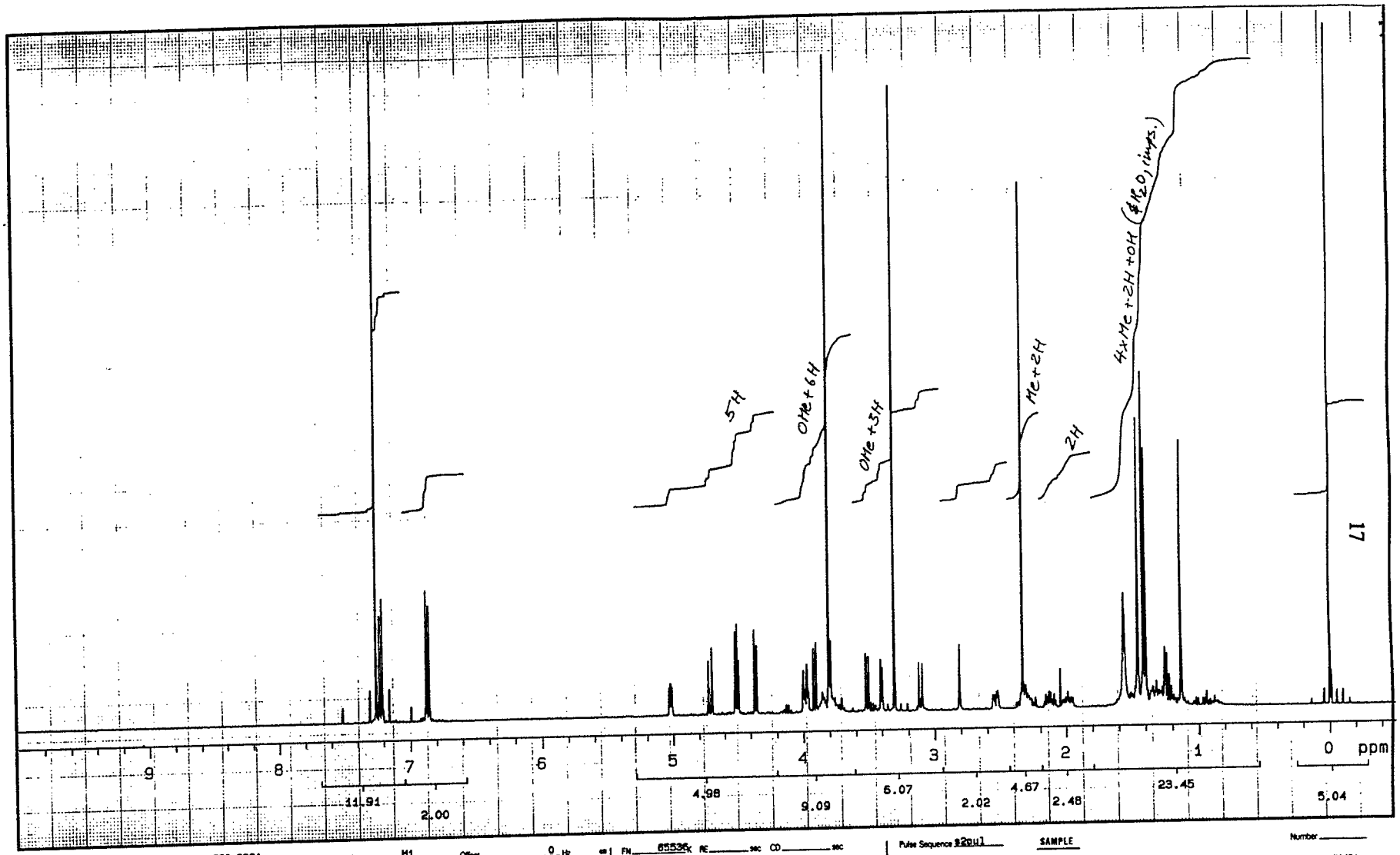


OBSERVE
Nucleus H1 — Freq. 399.9381 MHz
Spec. Width 8000.0 Hz — Offset 0 Hz
Acq. Time 4.096 sec — Delay 0 sec
Pulse Width 4.5 sec — Transients 256

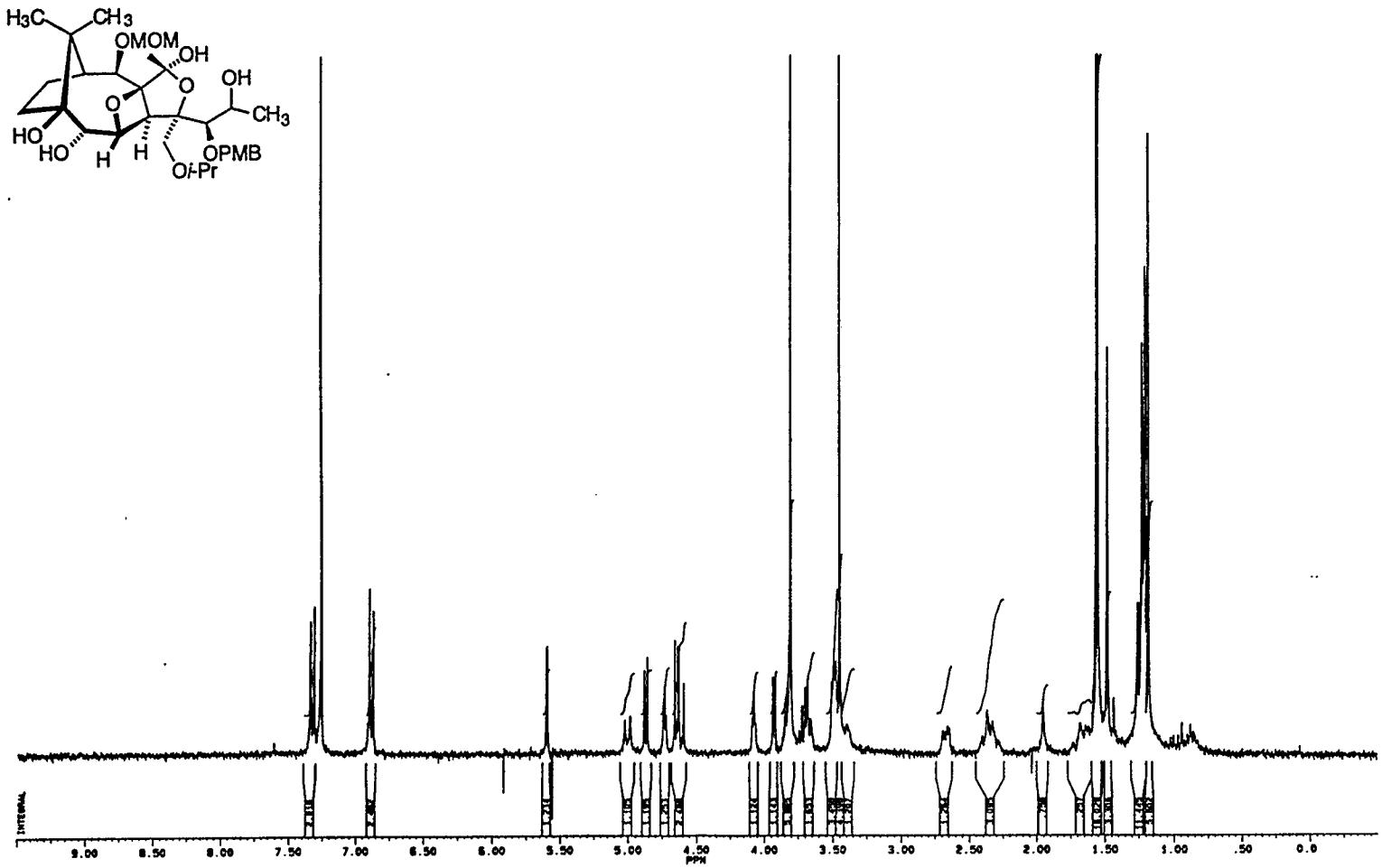
DECOUPLE
Nucleus H1 — Offset 0 Hz
Mode nnn — Power not used db
Modulation: Mode ccc — Freq. not used Hz
Pulse Width ___ μsec — Power Mode 30

PLOT/PROCESSING
FN 65536K RE ___ sec CD ___ sec
LB not used Hz Mat used sec CCD ___
Width 4199.1 Hz/ppm Start -200.2 Hz/ppm
Reference ___

EXPERIMENT
Pulse Sequence s2pul
Tube O.D. ___ mm
Temp. 28.0 °C
Solvent CDCl3

SAMPLE
JNJ2A
IV-JNJ-2888 Johnston
Solvent: CDCl3
Operator: DF
gain=52/ct=256

Number ___
File JNJ2A
Date Jun 26 97
XL 400


H3C
CH3
OMOM
OH
O
OH
CH3
O
HO
HO
H
H
OPMB
Oi-Pr
INTEGRAL
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8.50
8.00
7.50
7.00
6.50
6.00
5.50
5.00
4.50
PPM
4.00
3.50
3.00
2.50
2.00
1.50
1.00
.50
0.0

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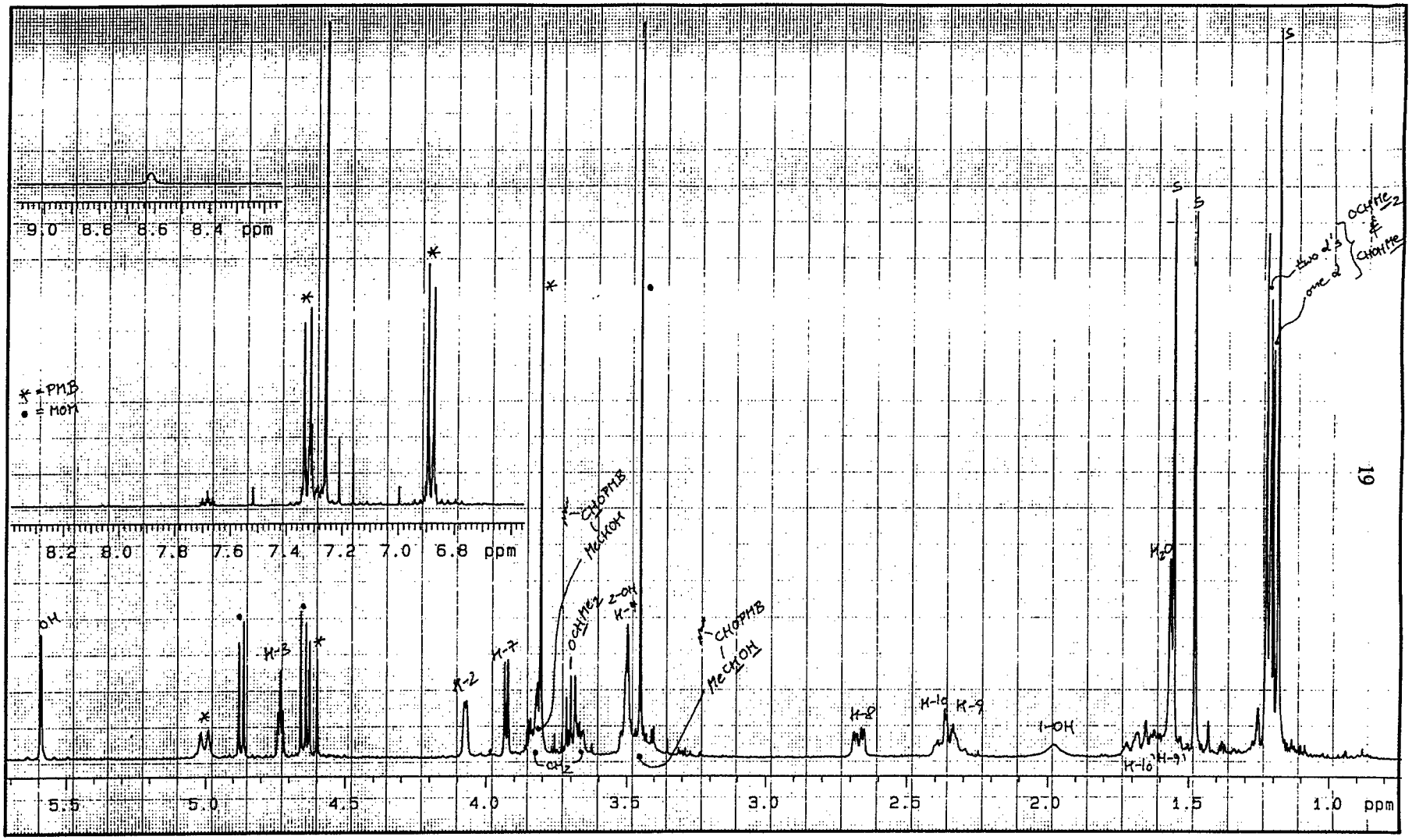

OBSERVE			
Nucleus	H1	Freq.	399.9361 MHz
Spec. Width	8000.0 Hz	Offset	0 Hz
Acq. Time	4.096 sec	Delay	0 sec
Pulse Width	4.5 μsec	Transients	256

DECOUPLE			
Nucleus	H1	Offset	0 Hz
Mode	nnn	Power	not used db
Modulation: Mode	ccc	Freq.	not used Hz
Pulse Width	— μsec	Power Mode	30

PLOT/PROCESSING	
FN 65536	RE sec CD sec
LB not used Hz	GF not used sec CCD
Width 1991.8 Hz/ppm	Start 294.0 Hz/ppm
Reference	

EXPERIMENT	
Pulse Sequence s2pul	
Tube O.D. mm	
Temp. 28.0 °C	
Solvent CDCl3	

SAMPLE

JNJ1A
IV-JNJ-286A Johnston
Solvent: CDCl3
Operator: DF
gain=48/ct=256

2 of 2

Number	
File	JNJ1A
Date	Jun 26 97
XL	400

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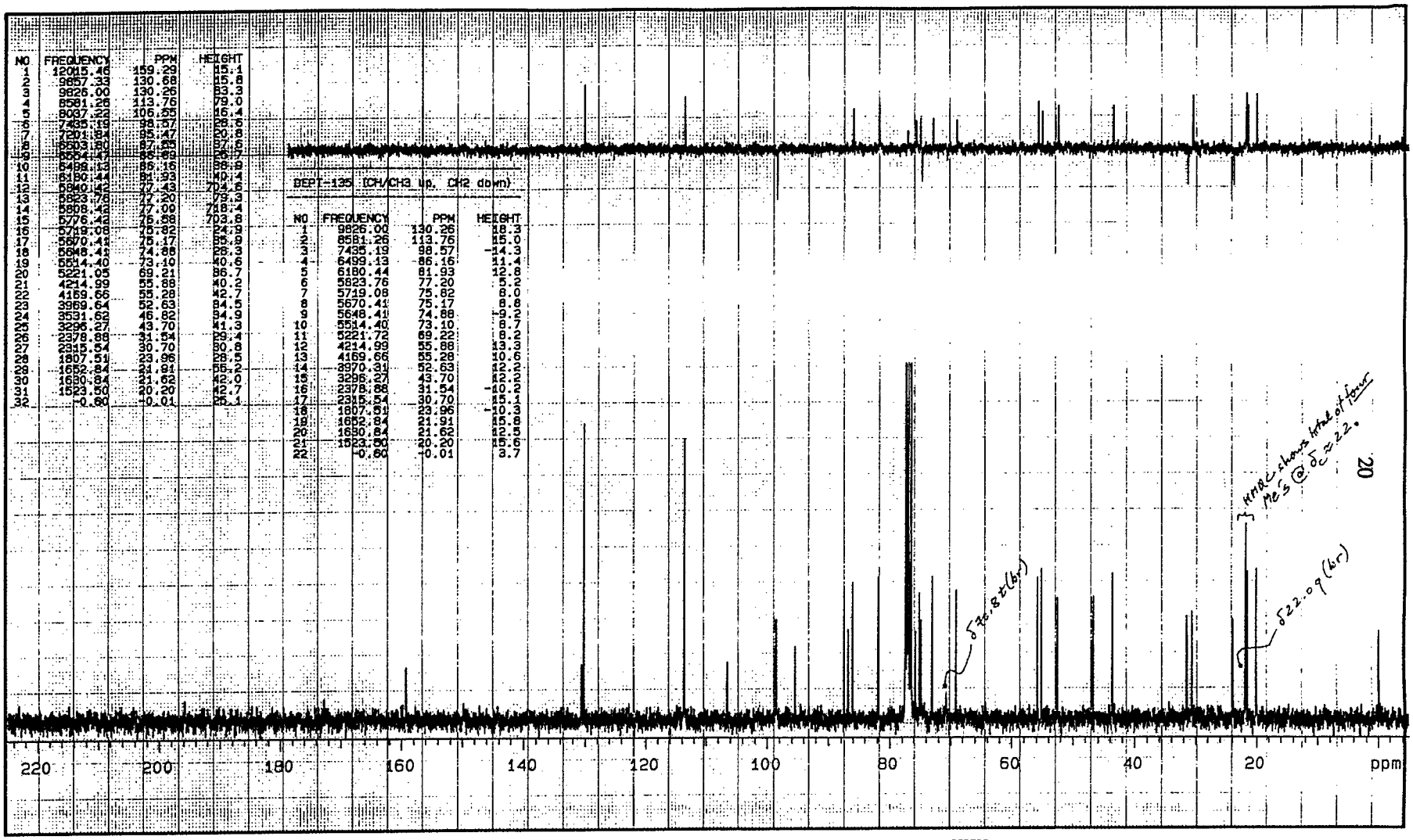

NO	FREQUENCY	PPM	HEIGHT
1	12015.46	159.29	15.1
2	9857.33	130.68	15.8
3	9826.00	130.26	83.3
4	8581.26	113.76	79.0
5	8037.22	106.55	16.4
6	7435.19	98.57	28.5
7	7201.84	95.47	20.8
8	6603.80	87.55	97.5
9	6554.47	86.89	26.7
10	6499.13	86.16	38.8
11	6180.44	81.93	40.4
12	5840.42	77.43	704.5
13	5823.76	77.20	79.3
14	5808.42	77.00	718.4
15	5776.42	76.58	703.8
16	5719.08	75.82	24.9
17	5670.41	75.17	35.9
18	5648.41	74.88	28.3
19	5514.40	73.10	40.6
20	5221.05	69.21	86.7
21	4214.99	55.88	40.2
22	4169.66	55.28	42.7
23	3969.64	52.63	84.5
24	3531.62	46.82	34.9
25	3296.27	43.70	41.3
26	2378.88	31.54	29.4
27	2315.54	30.70	30.8
28	1807.51	23.96	28.5
29	1652.84	21.91	56.2
30	1630.84	21.62	42.0
31	1523.50	20.20	42.7
32	-0.60	-0.01	25.1

DEPT-135 (CH/CH3 up, CH2 down)

NO	FREQUENCY	PPM	HEIGHT
1	9826.00	130.26	18.3
2	8581.26	113.76	15.0
3	7435.19	98.57	-14.3
4	6499.13	86.16	11.4
5	6180.44	81.93	12.8
6	5823.76	77.20	5.2
7	5719.08	75.82	8.0
8	5670.41	75.17	8.8
9	5648.41	74.88	-9.2
10	5514.40	73.10	8.7
11	5221.72	69.22	8.2
12	4214.99	55.88	13.3
13	4169.66	55.28	10.6
14	3970.31	52.63	12.2
15	3296.27	43.70	12.2
16	2378.88	31.54	-10.2
17	2315.54	30.70	15.1
18	1807.51	23.96	-10.3
19	1652.84	21.91	15.8
20	1630.84	21.62	12.5
21	1523.50	20.20	15.6
22	-0.60	-0.01	3.7

OBSERVE
Nucleus C13 — Freq. 75.4340 MHz
Spec. Width 21846.0 Hz — Offset 0 Hz
Acq. Time 1.500 sec — Delay 0.020 sec
Pulse Width 10.5 μsec — Transients 16000

DECOUPLE
Nucleus H1 — Offset -300.0 Hz
Mode nyy — Power not used db
Modulation Mode CWW — Freq. 11500 Hz
Pulse Width 14.0 μsec — Power Mode 41

PLOT/PROCESSING
FN 65536 RE sec CD sec
LB 1.000 Hz not used sec CCD
Width 17349.0 Hz/ppm Start -377.3 Hz/ppm
Reference

EXPERIMENT
Pulse Sequence DEPTB8 — Number
Tube O.D. mm — File JNJ1B
Temp. 25.0°C — Date Jun 26 97
Solvent CDCl3 — XL 400

SAMPLE
JNJ1B
IV-JNJ-268A Johnston
Solvent: CDCl3
Operator: DF
gain=60/ct=16000

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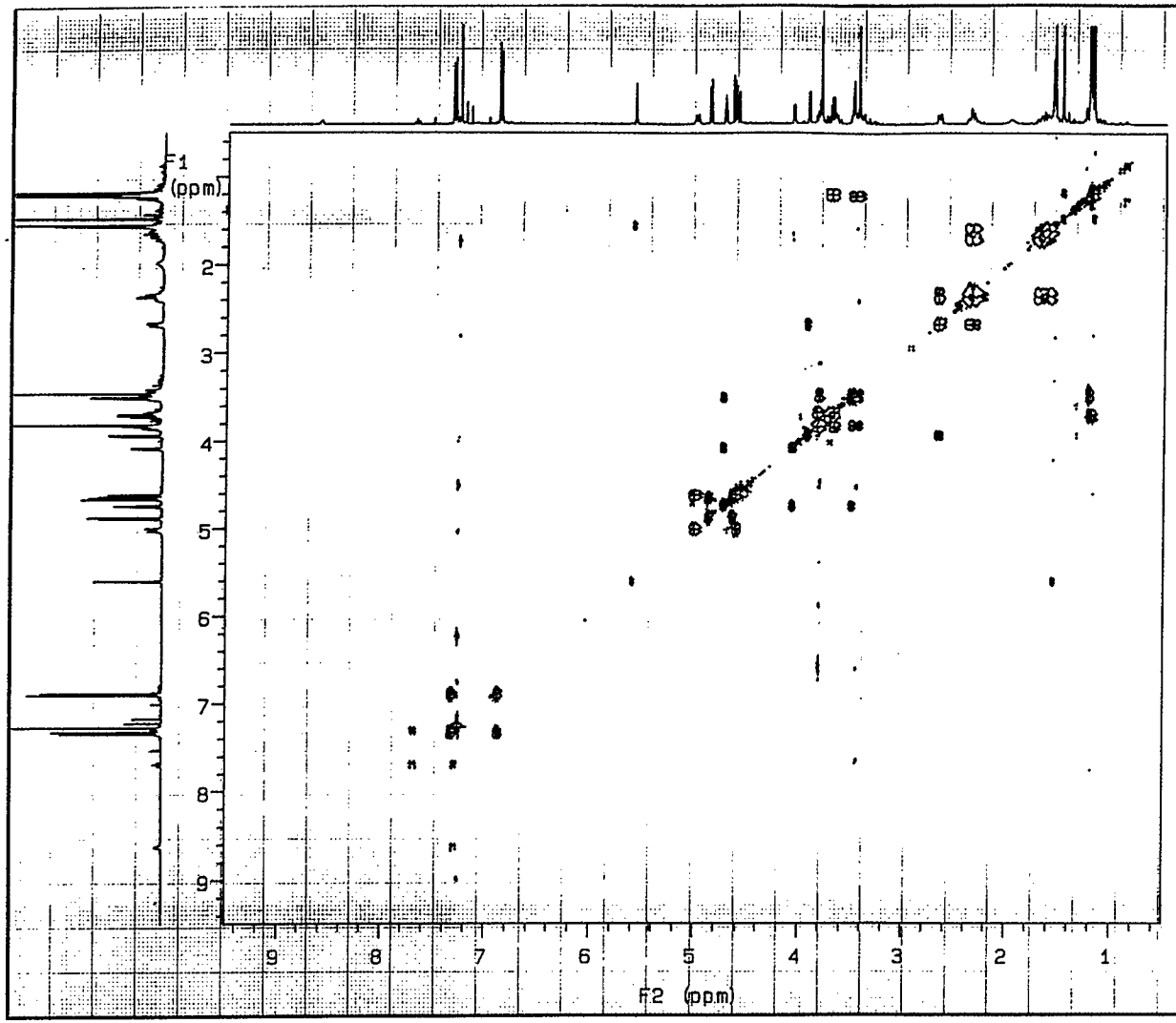

JNJ1C
IV-JNJ-266A Johnston
Solvent: CDCl3
Operator: DF
gain=30/ct=32

****
*** DQF-COSY ***
****

exp9 DQCOSY

SAMPLE		DEC. & VT	
date	Jul 1 97	dn	H1
solvent	CDCl3	dof	0
file	/home/vnmr1/J~	dm	n
	NJ-Cpds/JNJ1/JNJ1C	dmm	c
ACQUISITION		dmf	not used
sfrq	399.937	dpwr	30
tn	H1	temp	28.0
at	0.256	PROCESSING	
np	2048	sb	-0.256
sw	3999.2	sbs	-0.256
fb	2200	phfid	267.0
tpwr	54	wtfile	
pw	12.1	proc	ft
d1	1.000	fn	2048
tof	-618.0	math	f
nt	32		
ct	32	werr	
alock	y	wexp	AUTOSAVE
gain	30	wbs	
FLAGS		wnt	
il	n	2D PROCESSING	
in	n	sb1	-0.064
dp	y	sbs1	-0.064
hs	n	wtfile1	
2D ACQUISITION		proc1	ft
sw1	3999.2	fn1	1024
ni	256	DISPLAY	
2D DISPLAY		sp	198.8
sp1	199.2	wp	3596.5
wp1	3592.2	vs	833
sc2	0	sc	130
wc2	180	wc	220
rfl1	199.9	hzmm	16.35
rfp1	0	is	93.96
		rfl	199.9
		rfp	0
		th	2
		ins	100.000
		ai cdc ph	

OBSERVE			
Nucleus	H1	Freq.	399.9374 MHz
Spec. Width	3999.2 Hz	Offset	-618.0 Hz
Acq. Time	0.256 sec	Delay	1.000 sec
Pulse Width	12.1 μsec	Transients	32

DECOUPLE			
Nucleus	H1	Offset	0 Hz
Mode	n	Power	not used db
Modulation: Mode	c	Freq.	not used Hz
Pulse Width	μsec	Power Mode	30

PLOT/PROCESSING			
FN	2048k	RE sec	CD sec
LB	not used Hz	not used sec	CCD
Width	3596.5 Hz/ppm	Start	198.8 Hz/ppm
Reference			

EXPERIMENT			
Pulse Sequence	DQCOSY	SAMPLE	
Tube O.D.	mm	JNJ1C	
Temp.	28.0 °C	IV-JNJ-266A Johnston	
Solvent	CDCl3	Solvent: CDCl3	
		Operator: DF	
		gain=30/ct=32	

1 of 2

Number	
File	JNJ1C
Date	Jul 1 97
XL	400

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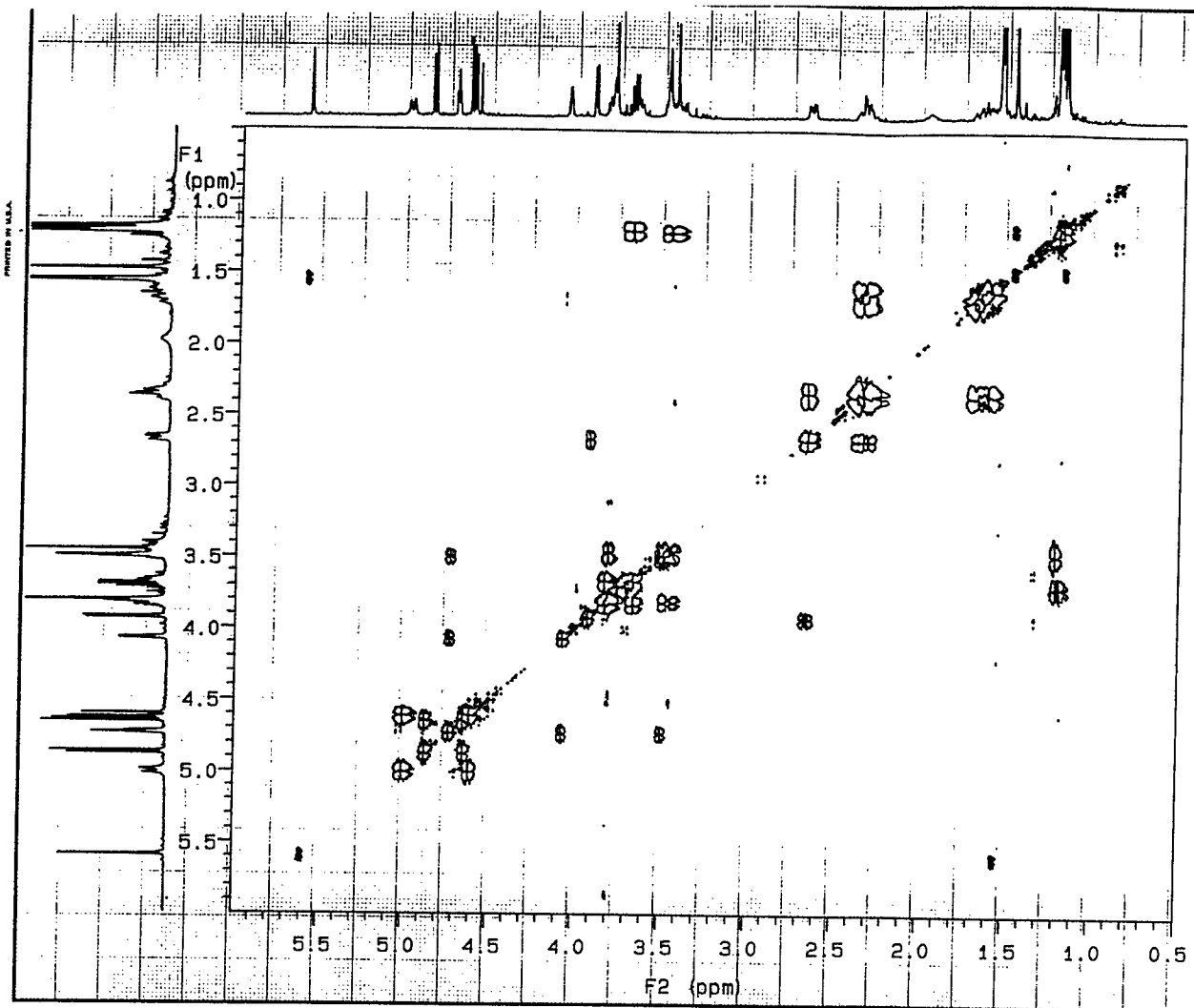

JNJ1C
IV-JNJ-266A Johnston
Solvent: CDCl3
Operator: DF
gain=30/ct=32

****
*** DQF-COSY ***
****

exp9 DQCOSY

SAMPLE	
date	Jul 1 97
solvent	CDCl3
file	/home/vnmr1/J~
	NJ-Cpds/JNJ1/JNJ1C
ACQUISITION	
sfrq	399.937
tn	H1
at	0.256
np	2048
sw	3999.2
fb	2200
tpwr	54
pw	12.1
d1	1.000
tof	-618.0
nt	32
ct	32
alock	y
gain	30
FLAGS	
il	n
in	n
dp	y
hs	n
2D ACQUISITION	
sw1	3999.2
ni	256
2D DISPLAY	
spi	199.2
wpi	2199.2
sc2	0
wc2	180
rfl1	199.9
rfp1	0

DEC. & VT	
dn	H1
dof	0
dm	n
dmm	c
dmf	not used
dpwr	30
temp	28.0
PROCESSING	
sb	-0.256
sbs	-0.256
phfid	267.0
wtfile	
proc	ft
fn	2048
math	f
werr	
wexp	AUTOSAVE
wbs	
wnt	
2D PROCESSING	
sbi	-0.064
sbs1	-0.064
wtfile1	
proc1	ft
fn1	1024
DISPLAY	
sp	198.8
wp	2197.0
vs	833
sc	130
wc	220
hzmm	9.99
is	93.96
rfl	199.9
rfp	0
th	2
ins	100.000
ai cdc ph	

22

OBSERVE			
Nucleus	H1	Freq.	399.9874 MHz
Spec. Width	3999.2 Hz	Offset	-618.0 Hz
Acq. Time	0.256 sec	Delay	1.000 sec
Pulse Width	12.1 μsec	Transients	32

DECOUPLE			
Nucleus	H1	Offset	0 Hz
Mode	n	Power	not used db
Modulation: Mode	c	Freq.	not used Hz
Pulse Width	μsec	Power Mode	30

PLOT/PROCESSING			
FN	2048	RE sec	CD sec
LB	not used Hz	GAT used sec	CCD
Width	2197.0 Hz/ppm	Start	198.8 Hz/ppm
Reference			

EXPERIMENT			
Pulse Sequence	DQCOSY		
Tube O.D.	mm		
Temp.	28.0 °C		
Solvent	CDCl3		

SAMPLE
JNJ1C
IV-JNJ-266A Johnston
Solvent: CDCl3
Operator: DF
gain=30/ct=32

2 of 2

Number
File JNJ1C
Date Jul 1 97
XL 400

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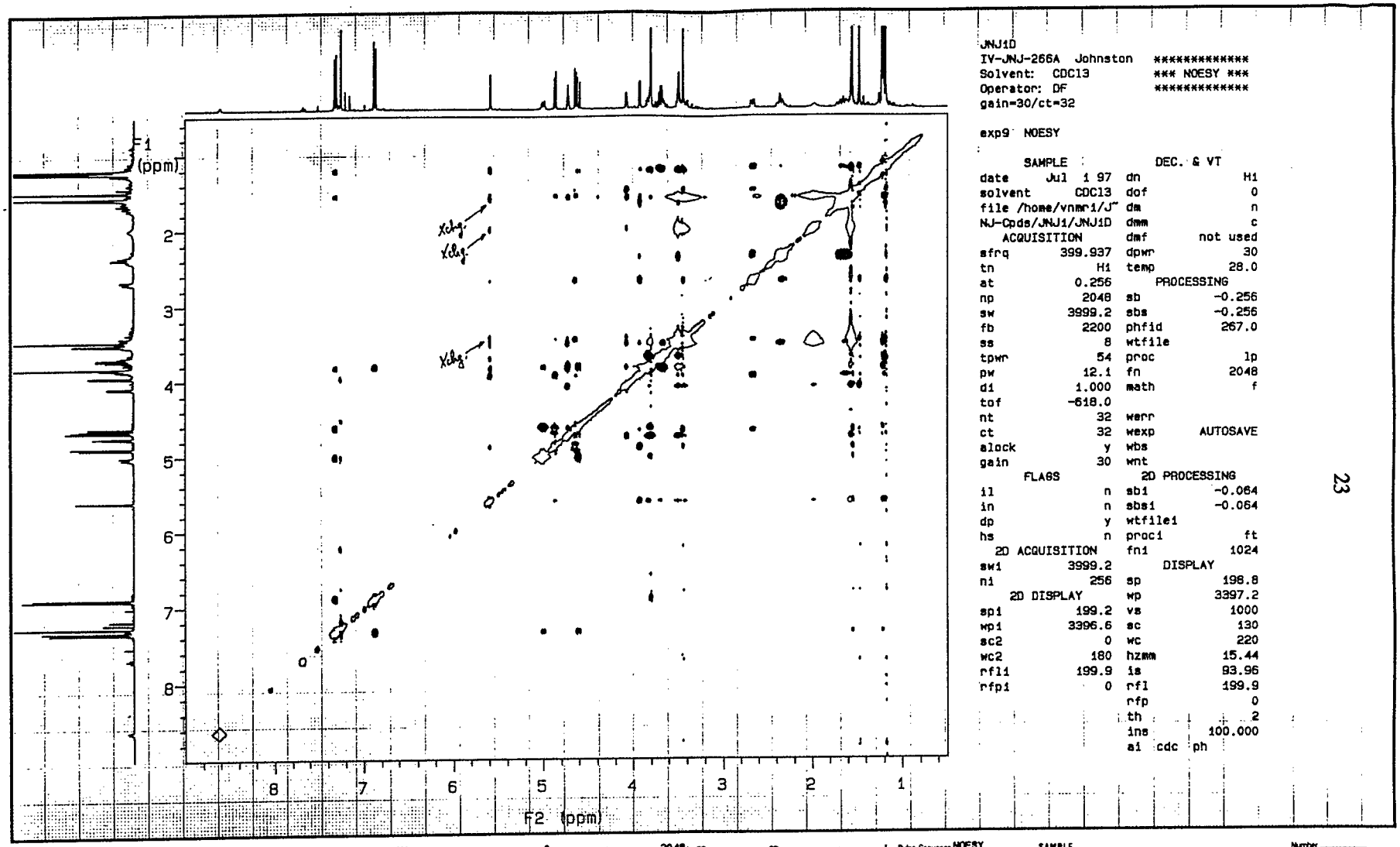

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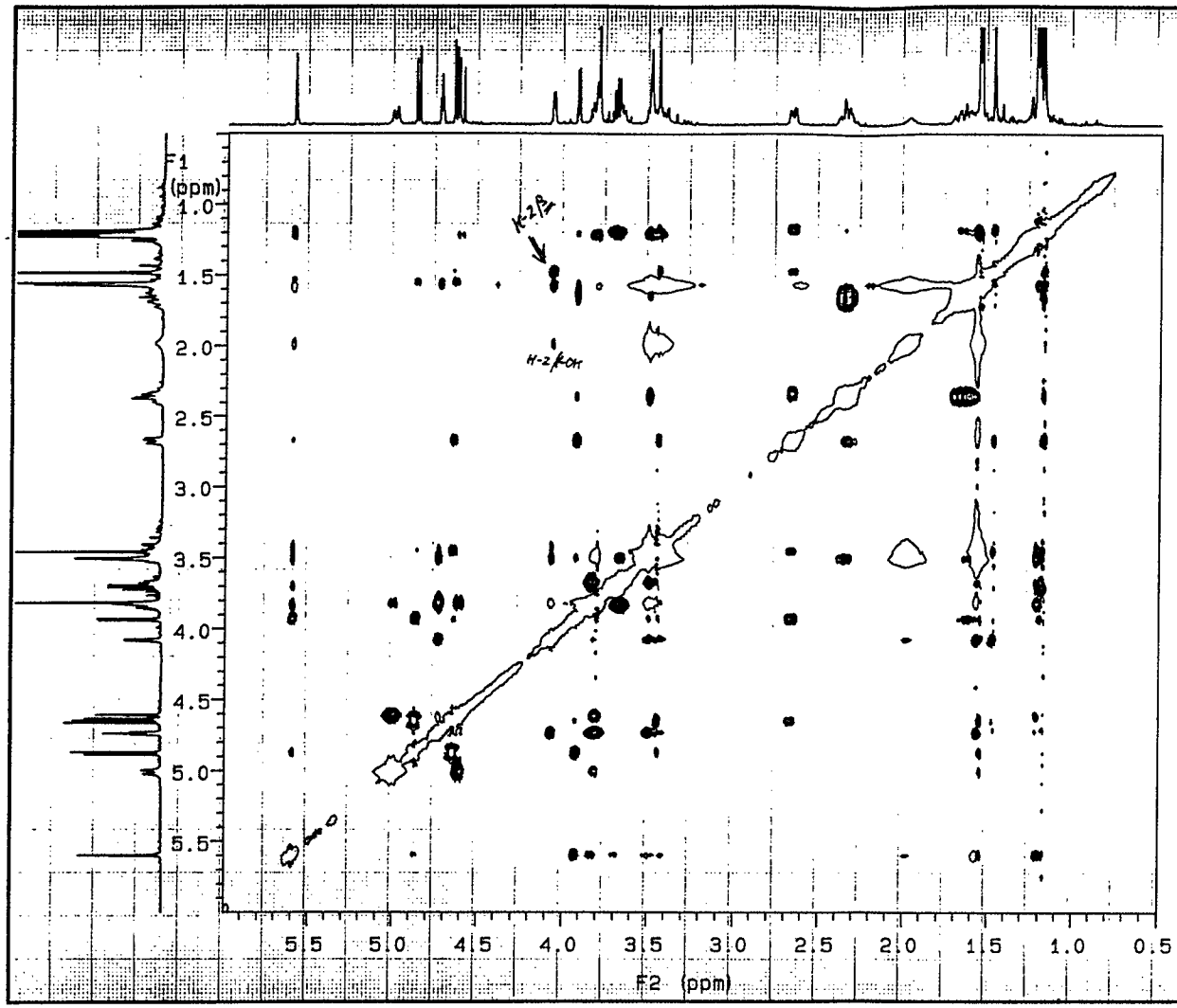


```
JNJ1D
IV-JNJ-266A  Johnston      *************
Solvent:  CDCl3            *** NOESY ***
Operator: DF               *************
gain=30/ct=32

exp9  NOESY

      SAMPLE                 DEC. & VT
date      Jul  1 97   dn                H1
solvent        CDCl3  dof                0
file /home/vnmr1/J~   dm                 n
NJ-Cpds/JNJ1/JNJ1D    dmm                c
   ACQUISITION        dmf        not used
sfrq        399.937   dpwr             30
tn               H1   temp           28.0
at            0.256       PROCESSING
np             2048   sb           -0.256
sw           3999.2   sbs          -0.256
fb             2200   phfid        267.0
ss                8   wtfile
tpwr             54   proc            lp
pw             12.1   fn            2048
d1            1.000   math             f
tof          -618.0
nt               32   werr
ct               32   wexp      AUTOSAVE
alock             y   wbs
gain             30   wnt
      FLASS              2D PROCESSING
il                n   sbi          -0.064
in                n   sbsi         -0.064
dp                y   wtfile1
hs                n   proc1           ft
  2D ACQUISITION      fn1           1024
swi          3999.2       DISPLAY
ni              256   sp            198.8
    2D DISPLAY        wp           2197.0
sp1           199.2   vs             1000
wp1          2199.2   sc              130
sc2               0   wc              220
wc2             180   hzmm           9.99
rfl1          199.9   is            93.96
rfp1              0   rfl           199.9
                      rfp               0
                      th                2
                      ins         100.000
                      ai  cdc  ph
```

OBSERVE			
Nucleus	H1	Freq.	399.9374 MHz
Spec. Width	3999.2 Hz	Offset	-618.0 Hz
Acq. Time	0.256 sec	Delay	1.000 sec
Pulse Width	12.1 sec	Transients	32

DECOUPLE			
Nucleus	H1	Offset	0 Hz
Mode	n	Power	not used db
Modulation: Mode	c	Freq.	not used Hz
Pulse Width	--- μsec	Power Mode	30

PLOT/PROCESSING			
FN	2048	RE	sec CD sec
LB	not used Hz	GB	not used sec CCD
Width	2197.0 Hz/ppm	Start	198.8 Hz/ppm
Reference			

EXPERIMENT			
Pulse Sequence	NOESY	SAMPLE	
Tube O.D.	mm	JNJ1D	
Temp.	28.0°C	IV-JNJ-266A Johnston	
Solvent	CDCl3	Solvent: CDCl3	
		Operator: DF	
		gain=30/ct=32	

2/2

Number	
File	JNJ1D
Date	Jul 1 97
XL	400

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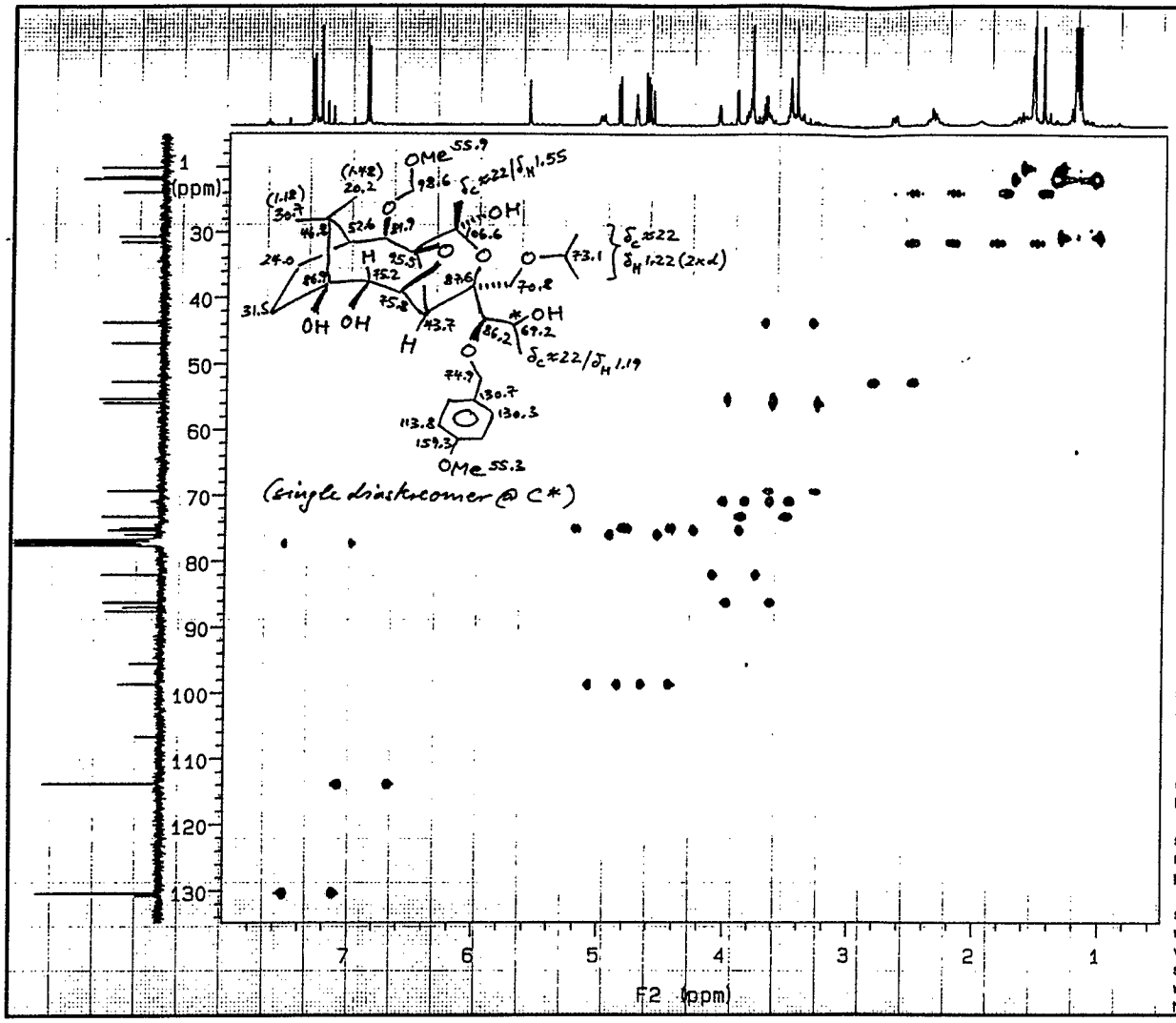

JNJ1E
IV-JNJ-266A Johnston
Solvent: CDCl3
Operator: DF
gain=60/ct=32

****
*** HMQC (ONE-BOND H,X CORR) ***
****

exp9 HMQC

SAMPLE		DEC. & VT		ACQUISITION ARRAYS	
date	Jul 2 97	dfrq	100.571	array	phase
solvent	CDCl3	dn	C13	arraydim	256
file /home/vnmr1/J~		dpwr	42		
NJ-Cpds/JNJ1/JNJ1E		dof	-2558.1	i	phase
ACQUISITION		dm	nn	i	1
sfrq	399.937	dmm	cw	2	2
tn	H1	dmf	13200		
at	0.256	dseq			
np	2048	dres	1.0		
sw	3999.2	pwx	10.2		
fb	2200	pwxlvl	58		
ss	8	homo	n		
tpwr	54	temp	28.0		
pw	12.1	PROCESSING			
d1	1.000	gf	0.118		
tof	-618.0	gfs	not used		
nt	32	wtfile			
ct	32	proc	lp		
alock	y	fn	2048		
gain	60	math	f		
null	0.300				
j	140.0	werr			
mbond	n	wexp	AUTOSAVE		
taumb	0	wbs			
satflg	nn	wnt			
satpwr	0	2D PROCESSING			
satdly	0	gf1	0.008		
satfrq	0	gfs1	not used		
FLAGS		wtfile1			
il	n	proc1	lp		
in	n	fn1	1024		
dp	y				
hs	n				
2D ACQUISITION					
sw1	15083.0				
ni	128				
phase	arrayed				
DISPLAY					
sp	198.8				
wp	2998.4				
vs	114				
sc	130				
wc	220				
hzmm	13.63				

25

OBSERVE: Nucleus H1; Freq 399.9374 MHz; Spec. Width 3999.2 Hz; Offset -618.0 Hz; Acq. Time 0.256 sec; Delay 1.000 sec; Pulse Width 12.1 μsec; Transients 32

DECOUPLE: Nucleus C13; Offset -2558.1 Hz; Mode nn; Power not used db; Modulation Mode CW; Freq 13200 Hz; Pulse Width --- μsec; Power Mode 42

PLOT/PROCESSING: FN 2048 k; RE sec; CD sec; LB not used Hz; AF 0.118 sec; CCD; Width 2998.4 Hz/ppm; Start 198.8 Hz/ppm; Reference

EXPERIMENT: Pulse Sequence HMQC; Tube O.D. mm; Temp 28.0 °C; Solvent CDCl3

SAMPLE
JNJ1E
IV-JNJ-266A Johnston
Solvent: CDCl3
Operator: DF
gain=60/ct=32

Number; File JNJ1E; Date Jul 2 97; XL 400

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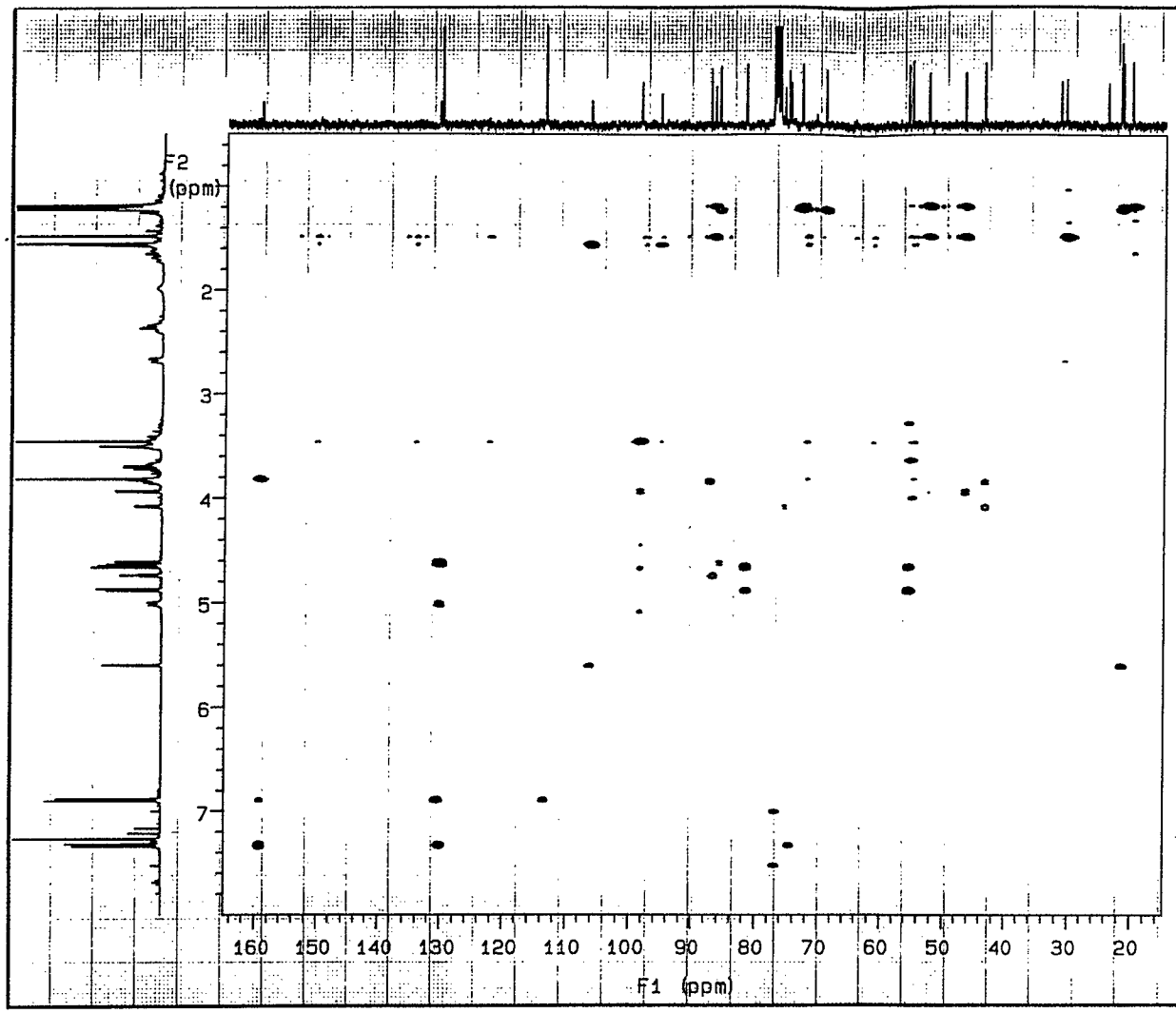


```
JNJ1F
IV-JNJ-266A  Johnston   ********************************
Solvent:  CDCl3         *** HMBC (LONG-RANGE H, X CORR) ***
Operator: DF            ********************************
gain=52/ct=160

exp9  HMQC

      SAMPLE              DEC. & VT          ACQUISITION ARRAYS
date      Jul  2 97  dfrq        100.575  array        phase
solvent       CDCl3  dn              C13  arraydim       256
file /home/vnmr1/J~  dpwr             42
NJ-Cpds/JNJ1/JNJ1F   dof           1464.7  i            phase
   ACQUISITION       dm              nn    1                1
sfrq        399.937  dmm             cw    2                2
tn               H1  dmf           13200
at            0.256  dseq
np             2048  dres            1.0
sw           3999.2  pwx            10.2
fb             2200  pwxlvl           58
ss                8  homo             n
tpwr             54  temp           28.0
pw             12.1      PROCESSING
d1            1.000  gf            0.118
tof          -618.0  gfs        not used
nt              160  wtfile
ct              160  proc             lp
alock             y  fn             2048
gain             52  math              f
null              0
j             140.0  werr
mbond             y  wexp       AUTOSAVE
taumb         0.060  wbs
satflg           nn  wnt
satpwr            0    2D PROCESSING
satdly            0  gf1           0.006
satfrq            0  gfs1       not used
       FLAGS         wtfile1
il                n  proc1            lp
in                n  fn1            1024
dp                y
hs                n
  2D ACQUISITION
sw1         21119.3
ni              128
phase       arrayed
      DISPLAY
sp            198.8
wp           2998.4
vs             2500
sc              130
wc              220
hzmm          13.63
```

OBSERVE			
Nucleus	H1	Freq.	399.9374 MHz
Spec. Width	3999.2 Hz	Offset	-618.0 Hz
Acq. Time	0.256 sec	Delay	1.000 sec
Pulse Width	12.1 μsec	Transients	160

DECOUPLE			
Nucleus	C13	Offset	1464.7 Hz
Mode	nn	Power	not used db
Modulation: Mode	CW	Freq.	13200 Hz
Pulse Width	μsec	Power Mode	42

PLOT/PROCESSING			
FN	2048k	RE sec	CD sec
LB	not used Hz	AF 0.118 sec	CCD
Width	2998.4 Hz/ppm	Start 198.8 Hz ppm	
Reference			

EXPERIMENT			
Pulse Sequence	HMQC	SAMPLE	
Tube O.D.	mm	JNJ1F	
Temp.	28.0 °C	IV-JNJ-266A Johnston	
Solvent	CDCl3	Solvent: CDCl3	
		Operator: DF	
		gain=52/ct=160	

1 of 2

Number	
File	JNJ1F
Date	Jul 2 97
XL	400

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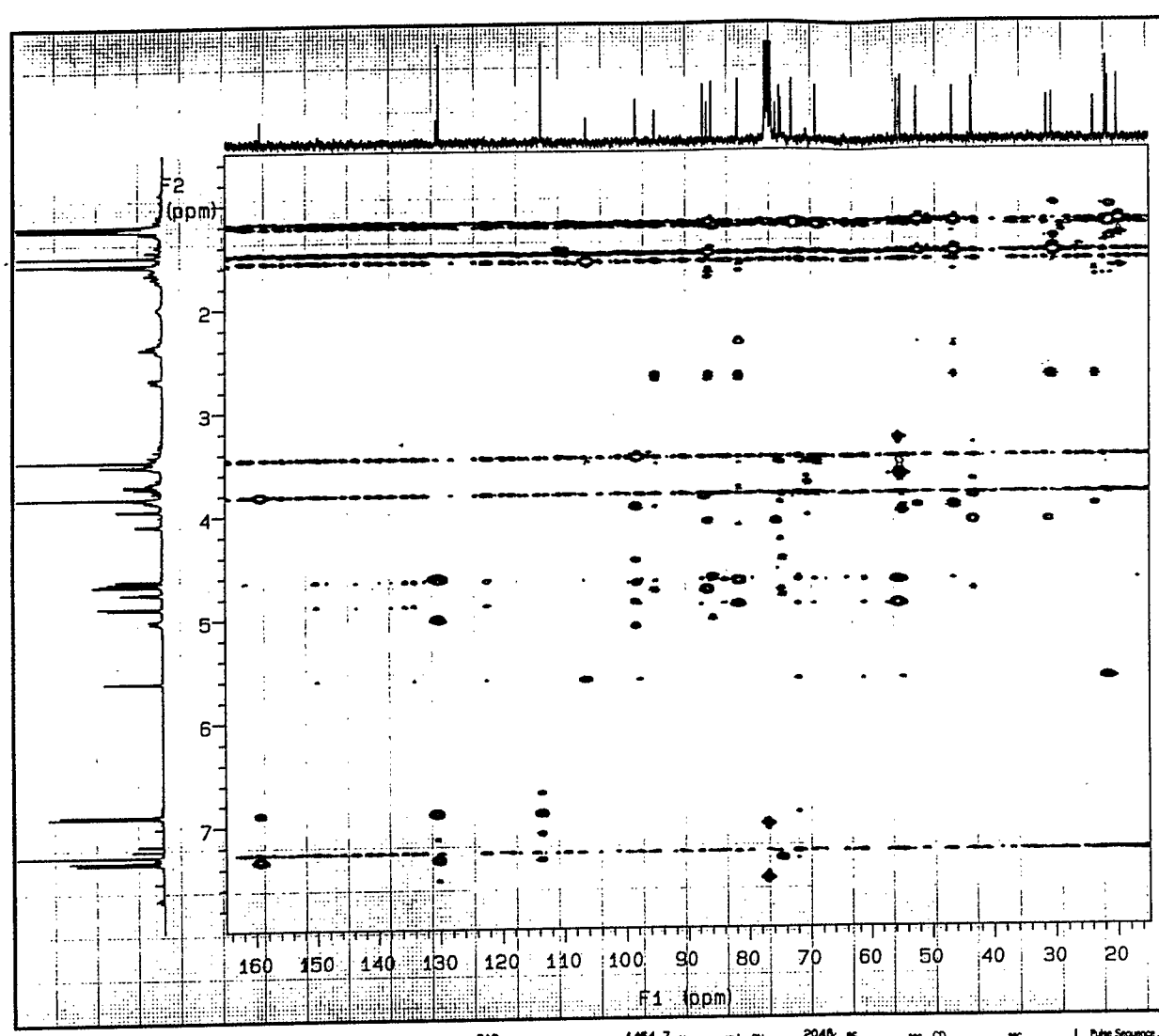

JNJ1F
IV-JNJ-266A Johnston
Solvent: CDCl3
Operator: DF
gain=52/ct=160

****
*** HMBC (LONG-RANGE H, X CORR) ***
****

exp9 HMQC

SAMPLE		DEC. & VT		ACQUISITION ARRAYS	
date	Jul 2 97	dfrq	100.575	array	phase
solvent	CDCl3	dn	C13	arraydim	256
file /home/vnmr1/J~		dpwr	42		
NJ-Cpds/JNJ1/JNJ1F		dof	1464.7	i	phase
ACQUISITION		dm	nn	1	1
sfrq	399.937	dmm	cw	2	2
tn	H1	dmf	13200		
at	0.256	dseq			
np	2048	dres	1.0		
sw	3999.2	pwx	10.2		
fb	2200	pwxlvl	58		
ss	8	homo	n		
tpwr	54	temp	28.0		
pw	12.1	PROCESSING			
d1	1.000	gf	0.118		
tof	-618.0	gfs	not used		
nt	160	wtfile			
ct	160	proc	lp		
alock	y	fn	2048		
gain	52	math	f		
null	0				
j	140.0	werr			
mbond	y	wexp	AUTOSAVE		
taumb	0.060	wbs			
satflg	nn	wnt			
satpwr	0	2D PROCESSING			
satdly	0	gf1	0.006		
satfrq	0	gfs1	not used		
FLAGS		wtfile1			
il	n	proc1	lp		
in	n	fn1	1024		
dp	y				
hs	n				
2D ACQUISITION					
sw1	21119.3				
ni	128				
phase	arrayed				
DISPLAY					
sp	198.8				
wp	2998.4				
vs	10000				
sc	130				
wc	220				
hzmm	13.63				

27

OBSERVE: Nucleus H1; Freq. 399.9374 MHz; Spec. Width 3999.2 Hz; Offset -618.0 Hz; Acq. Time 0.256 sec; Delay 1.000 sec; Pulse Width 12.1 µsec; Transients 160

DECOUPLE: Nucleus C13; Offset 1464.7 Hz; Mode nn; Power not used db; Modulation: Mode cw; Freq. 13200 Hz; Pulse Width ___ µsec; Power Mode 42

PLOT/PROCESSING: FN 2048; RE ___ sec; CD ___ sec; LB not used Hz; AF 0.118 sec; CCD ___; Width 2998.4 Hz/ppm; Start 198.8 Hz/ppm; Reference ___

EXPERIMENT: Pulse Sequence HMQC; Tube O.D. ___ mm; Temp. 28.0 °C; Solvent CDCl3

SAMPLE
JNJ1F
IV-JNJ-266A Johnston
Solvent: CDCl3
Operator: DF
gain=52/ct=160

2 of 2

Number ___; File JNJ1F; Date Jul 2 97; XL 400

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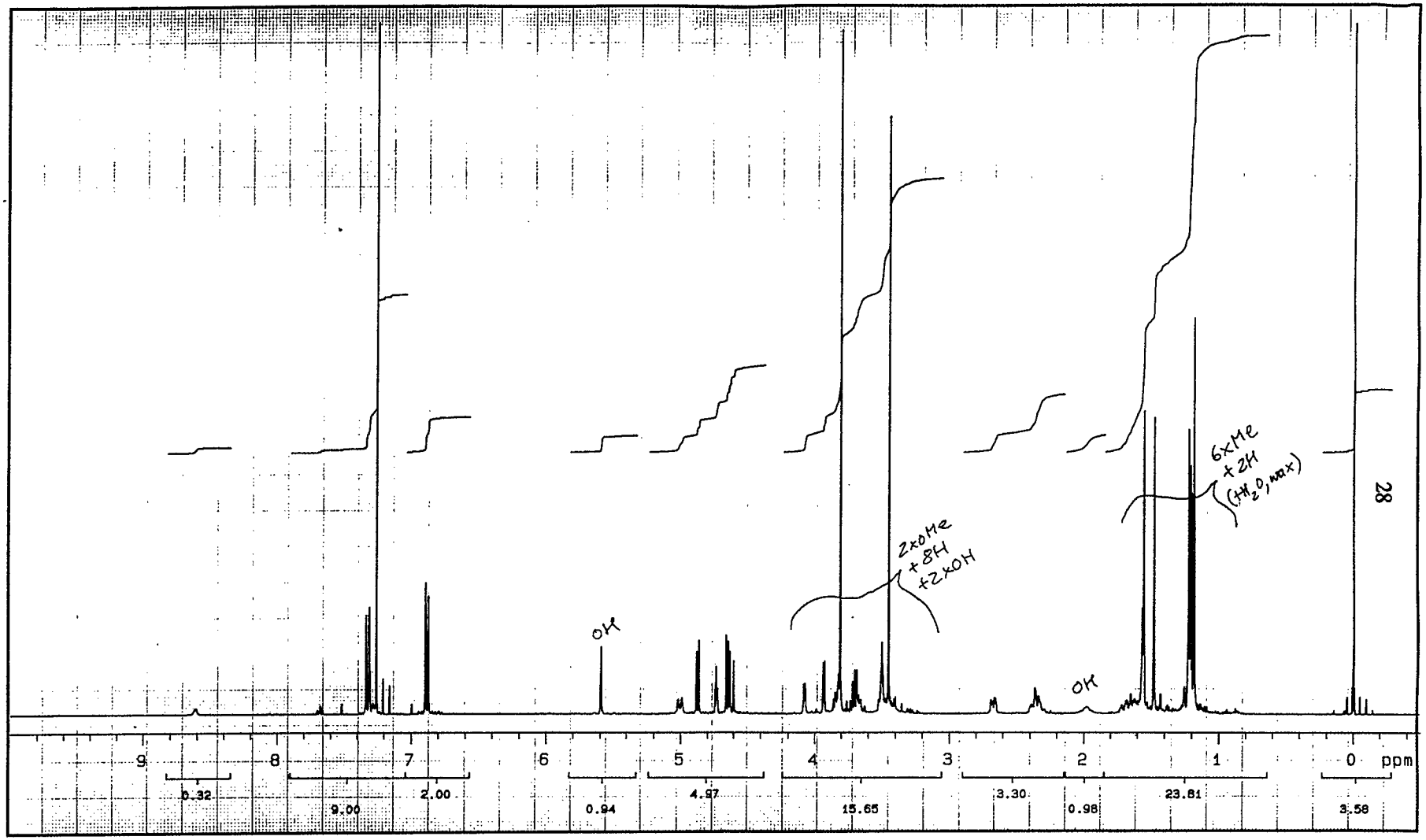

OBSERVE			DECOUPLE			PLOT/PROCESSING			EXPERIMENT		
Nucleus H1	Freq. 399.9361 MHz		Nucleus H1	Offset 0 Hz		FN 65536	RE sec	CD sec	Pulse Sequence s2pul	SAMPLE	
Spec. Width 8000.0 Hz	Offset 0 Hz		Mode nnn	Power not used db		LB not used Hz	RF not used sec	CCD	Tube O.D. mm	JNJ1A	
Acq. Time 4.096 sec	Delay 0 sec		Modulation: Mode ccc	Freq. not used Hz		Width 4199.1 Hz/ppm	Start -200.2 Hz/ppm		Temp. 28.0 °C	IV-JNJ-288A Johnston	
Pulse Width 4.5 μsec	Transients 256		Pulse Width μsec	Power Mode 30		Reference			Solvent CDCl3	Solvent: CDCl3	
										Operator: DF	
										gain=48/ct=256	

Number
File JNJ1A
Date Jun 26 97
XL 400

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Table 1. Crystal data and Structure Refinement for **14b.**

Color / Shape	colorless / fragment
Empirical formula	$C_{28}H_{38}F_6O_9$
Formula weight	632.58
Temperature	173(2) K
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
Unit cell dimensions	a = 9.1680(9) Å $\alpha = 90^\circ$
(2233 reflections	b = 12.1237(12) Å $\beta = 90^\circ$
in full θ range)	c = 27.611(3) Å $\gamma = 90^\circ$
Volume	3069.0(5) $Å^3$
Z	4
Density (calculated)	1.369 Mg/m^3
Absorption coefficient	0.123 mm^{-1}
Diffractometer / scan	Siemens SMART / CCD area detector
Radiation / wavelength	MoKα (graphite monochrom.) / 0.71073 Å
F(000)	1328
Crystal size	0.10 x 0.20 x 0.30 mm
θ range for data collection	1.48 to 19.99°
Index ranges	$-8 \le h \le 8$, $-10 \le k \le 11$, $-26 \le \ell \le 26$
Reflections collected	9947
Independent / observed refls.	2850(R_{int} = 0.0942) / 2085([I>2σ(I)])
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Computing	SHELXTL, Ver. 5[1]
Data / restraints / parameters	2846 / 0 / 396
Goodness-of-fit on F^2	1.050
SHELX-93 weight parameters	0.0520, 0.0000
Final R indices [I>2σ(I)]	R1 = 0.0495, wR2 = 0.0939
R indices (all data)	R1 = 0.0858, wR2 = 0.1123
Absolute structure parameter	1(2)
Extinction coefficient	0.0052(8)
Largest diff. peak and hole	0.187 and -0.209 $eÅ^{-3}$

Table 2. Atomic Coordinates [x 10^4] and Equivalent Isotropic Displacement Parameters [$Å^2$ x 10^3] for **14b**.

Atom	x/a	y/b	z/c	U(eq)
F(1)	-3566(6)	-8872(3)	-710(2)	83(2)
F(2)	-5395(6)	-9906(3)	-816(2)	98(2)
F(3)	-4412(5)	-9763(4)	-118(2)	90(2)
F(4)	-2324(7)	-5786(4)	-3324(2)	100(2)
F(5)	-574(6)	-6142(4)	-2855(2)	92(2)
F(6)	-2225(6)	-7332(4)	-2973(2)	84(2)
O(1)	-1700(4)	-3303(3)	-532(1)	30(1)
O(2)	-3394(4)	-3498(3)	-1132(1)	25(1)
O(3)	-5513(5)	-3373(3)	-23(2)	39(1)
O(4)	-5766(4)	-7507(4)	-726(2)	33(1)
O(5)	-6588(5)	-8307(4)	-30(2)	51(2)
O(6)	-8468(5)	-7567(4)	-1114(2)	46(1)
O(7)	-10553(5)	-7515(4)	-1603(2)	56(1)
O(8)	-3989(5)	-6351(4)	-2389(2)	39(1)
O(9)	-2403(5)	-4920(4)	-2305(2)	48(1)
C(1)	-4875(6)	-5002(5)	-871(2)	21(2)
C(2)	-5890(7)	-5545(5)	-496(2)	27(2)
C(3)	-6776(7)	-6539(5)	-683(2)	30(2)
C(4)	-7737(7)	-6509(5)	-1140(2)	32(2)
C(5)	-7402(8)	-6269(5)	-1675(2)	34(2)
C(6)	-7147(7)	-5011(5)	-1804(2)	34(2)
C(7)	-5559(7)	-4925(5)	-2014(2)	33(2)
C(8)	-4860(8)	-6080(5)	-1951(2)	32(2)
C(9)	-3901(7)	-6267(5)	-1531(2)	30(2)
C(10)	-3861(7)	-5841(5)	-1091(2)	25(2)
C(11)	-4016(7)	-3958(5)	-696(2)	22(2)
C(12)	-2701(7)	-4134(5)	-372(2)	30(2)
C(13)	-1922(7)	-3164(5)	-1042(2)	28(2)
C(14)	-1740(7)	-1956(5)	-1163(2)	39(2)
C(15)	-910(7)	-3898(6)	-1332(2)	43(2)
C(16)	-4989(7)	-3036(5)	-480(2)	24(2)

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Table 2 (continued)

C(17)	-6178(8)	-2671(5)	-813(2)	29(2)
C(18)	-7580(9)	-2660(5)	-718(2)	44(2)
C(19)	-5167(7)	-5857(5)	-21(2)	31(2)
C(20)	-5858(8)	-8272(6)	-383(3)	35(2)
C(21)	-4780(10)	-9210(7)	-514(3)	49(2)
C(22)	-9985(8)	-7563(7)	-1141(3)	60(2)
C(23)	-10419(9)	-8510(8)	-1863(3)	83(3)
C(24)	-6216(7)	-6872(5)	-1984(2)	32(2)
C(25)	-6802(7)	-6943(5)	-2506(2)	42(2)
C(26)	-5814(7)	-8057(5)	-1827(2)	38(2)
C(27)	-2837(10)	-5763(7)	-2489(3)	40(2)
C(28)	-1996(11)	-6273(7)	-2914(3)	58(2)

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 3. Bond Lengths [Å] and Angles [°] for **14b**.

F(1)-C(21)	1.304(8)	F(2)-C(21)	1.313(8)
F(3)-C(21)	1.325(8)	F(4)-C(28)	1.312(9)
F(5)-C(28)	1.323(9)	F(6)-C(28)	1.311(8)
O(1)-C(12)	1.432(6)	O(1)-C(13)	1.435(7)
O(2)-C(13)	1.431(7)	O(2)-C(11)	1.445(6)
O(3)-C(16)	1.411(6)	O(4)-C(20)	1.328(8)
O(4)-C(3)	1.500(7)	O(5)-C(20)	1.184(8)
O(6)-C(22)	1.392(8)	O(6)-C(4)	1.449(7)
O(7)-C(22)	1.379(8)	O(7)-C(23)	1.410(9)
O(8)-C(27)	1.304(8)	O(8)-C(8)	1.485(7)
O(9)-C(27)	1.208(8)	C(1)-C(10)	1.506(8)
C(1)-C(2)	1.540(8)	C(1)-C(11)	1.567(8)
C(2)-C(19)	1.516(8)	C(2)-C(3)	1.542(8)
C(3)-C(4)	1.540(8)	C(4)-C(5)	1.537(8)
C(5)-C(24)	1.564(8)	C(5)-C(6)	1.583(8)
C(6)-C(7)	1.571(8)	C(7)-C(8)	1.550(9)
C(8)-C(9)	1.473(8)	C(8)-C(24)	1.573(8)
C(9)-C(10)	1.321(8)	C(11)-C(12)	1.516(8)
C(11)-C(16)	1.549(8)	C(13)-C(14)	1.511(8)
C(13)-C(15)	1.514(8)	C(16)-C(17)	1.492(8)
C(17)-C(18)	1.312(8)	C(20)-C(21)	1.550(10)
C(24)-C(25)	1.539(8)	C(24)-C(26)	1.545(8)
C(27)-C(28)	1.535(10)		

C(12)-O(1)-C(13)	107.1(4)	C(13)-O(2)-C(11)	109.7(4)
C(20)-O(4)-C(3)	116.8(5)	C(22)-O(6)-C(4)	117.1(6)
C(22)-O(7)-C(23)	113.7(6)	C(27)-O(8)-C(8)	119.1(5)
C(10)-C(1)-C(2)	110.9(5)	C(10)-C(1)-C(11)	111.1(5)
C(2)-C(1)-C(11)	116.2(4)	C(19)-C(2)-C(1)	115.0(5)
C(19)-C(2)-C(3)	108.9(5)	C(1)-C(2)-C(3)	115.3(5)
O(4)-C(3)-C(4)	107.8(5)	O(4)-C(3)-C(2)	108.3(4)
C(4)-C(3)-C(2)	123.9(5)	O(6)-C(4)-C(5)	107.9(5)
O(6)-C(4)-C(3)	101.7(5)	C(5)-C(4)-C(3)	132.7(6)
C(4)-C(5)-C(24)	125.1(5)	C(4)-C(5)-C(6)	115.3(5)

Table 3 (continued)

C(24)-C(5)-C(6)	103.1(5)	C(7)-C(6)-C(5)	106.5(5)
C(8)-C(7)-C(6)	106.4(5)	C(9)-C(8)-O(8)	106.6(5)
C(9)-C(8)-C(7)	118.3(5)	O(8)-C(8)-C(7)	109.4(5)
C(9)-C(8)-C(24)	115.1(5)	O(8)-C(8)-C(24)	104.1(5)
C(7)-C(8)-C(24)	102.6(5)	C(10)-C(9)-C(8)	133.0(6)
C(9)-C(10)-C(1)	128.2(6)	O(2)-C(11)-C(12)	103.4(5)
O(2)-C(11)-C(16)	105.6(4)	C(12)-C(11)-C(16)	109.5(5)
O(2)-C(11)-C(1)	104.7(4)	C(12)-C(11)-C(1)	117.9(5)
C(16)-C(11)-C(1)	114.3(5)	O(1)-C(12)-C(11)	103.2(5)
O(2)-C(13)-O(1)	105.7(5)	O(2)-C(13)-C(14)	109.9(5)
O(1)-C(13)-C(14)	108.4(5)	O(2)-C(13)-C(15)	108.7(5)
O(1)-C(13)-C(15)	111.2(5)	C(14)-C(13)-C(15)	112.7(6)
O(3)-C(16)-C(17)	112.9(5)	O(3)-C(16)-C(11)	109.3(5)
C(17)-C(16)-C(11)	113.5(5)	C(18)-C(17)-C(16)	126.5(7)
O(5)-C(20)-O(4)	130.4(7)	O(5)-C(20)-C(21)	121.8(7)
O(4)-C(20)-C(21)	107.8(7)	F(1)-C(21)-F(2)	107.8(8)
F(1)-C(21)-F(3)	106.5(7)	F(2)-C(21)-F(3)	107.9(6)
F(1)-C(21)-C(20)	114.2(6)	F(2)-C(21)-C(20)	110.3(6)
F(3)-C(21)-C(20)	109.9(7)	O(7)-C(22)-O(6)	115.3(6)
C(25)-C(24)-C(26)	107.1(5)	C(25)-C(24)-C(5)	107.1(5)
C(26)-C(24)-C(5)	116.6(5)	C(25)-C(24)-C(8)	111.4(5)
C(26)-C(24)-C(8)	111.3(5)	C(5)-C(24)-C(8)	103.4(5)
O(9)-C(27)-O(8)	129.8(7)	O(9)-C(27)-C(28)	119.8(8)
O(8)-C(27)-C(28)	110.4(7)	F(6)-C(28)-F(4)	107.3(7)
F(6)-C(28)-F(5)	106.9(8)	F(4)-C(28)-F(5)	106.1(8)
F(6)-C(28)-C(27)	114.1(7)	F(4)-C(28)-C(27)	111.3(7)
F(5)-C(28)-C(27)	110.6(7)		

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Table 4. Anisotropic Displacement Parameters [Å^2 x 10^3] for 14b.

Atom	U11	U22	U33	U23	U13	U12
F(1)	58(3)	43(3)	147(5)	26(3)	27(3)	22(3)
F(2)	111(4)	32(3)	151(5)	-39(3)	-57(4)	18(3)
F(3)	104(4)	56(3)	111(4)	38(3)	-12(3)	31(3)
F(4)	184(6)	72(4)	45(3)	6(3)	29(4)	36(4)
F(5)	72(4)	95(4)	111(4)	-38(3)	37(3)	-4(4)
F(6)	122(4)	38(3)	93(3)	-20(3)	53(3)	-5(3)
O(1)	31(3)	19(3)	41(3)	0(2)	-7(2)	-6(2)
O(2)	22(3)	21(2)	32(3)	4(2)	2(2)	-7(2)
O(3)	61(4)	19(3)	38(3)	-8(2)	11(3)	9(3)
O(4)	35(3)	19(3)	44(3)	3(3)	-2(3)	-2(3)
O(5)	67(4)	31(3)	56(3)	16(3)	15(3)	-1(3)
O(6)	43(4)	38(3)	57(3)	18(3)	-15(3)	-21(3)
O(7)	38(3)	40(3)	91(4)	3(3)	-19(3)	9(3)
O(8)	48(3)	26(3)	43(3)	-8(2)	13(3)	-16(3)
O(9)	52(4)	32(3)	59(3)	-9(3)	13(3)	-11(3)
C(1)	23(4)	13(4)	26(4)	-4(3)	-1(3)	2(4)
C(2)	34(5)	13(4)	33(4)	-1(3)	5(4)	8(4)
C(3)	19(4)	26(4)	45(5)	5(4)	-8(4)	-1(4)
C(4)	28(4)	18(4)	50(5)	9(4)	2(4)	1(4)
C(5)	45(5)	19(4)	39(5)	2(3)	2(4)	-8(4)
C(6)	39(5)	29(4)	34(4)	4(4)	-11(4)	-6(4)
C(7)	36(5)	27(4)	35(4)	0(4)	-10(4)	-6(4)
C(8)	46(5)	17(4)	32(5)	-5(3)	15(5)	-5(4)
C(9)	32(5)	20(4)	37(5)	0(4)	-2(4)	-5(4)
C(10)	24(5)	19(4)	32(5)	2(4)	2(4)	-7(3)
C(11)	26(4)	19(4)	20(4)	0(3)	3(4)	2(4)
C(12)	27(5)	24(4)	40(5)	0(4)	0(4)	-5(4)
C(13)	26(5)	29(5)	29(5)	2(4)	-2(4)	-3(4)
C(14)	50(5)	27(4)	42(5)	7(4)	-7(4)	-14(4)
C(15)	22(4)	48(5)	59(5)	-7(4)	3(4)	-6(4)
C(16)	36(5)	11(4)	24(4)	-4(3)	-3(4)	0(4)
C(17)	32(5)	15(4)	39(5)	-2(3)	-1(4)	3(4)
C(18)	44(6)	36(5)	50(5)	-11(4)	-3(4)	-2(4)

Table 4 (continued)

C(19)	42(5)	14(4)	38(4)	5(3)	1(4)	-6(4)
C(20)	41(6)	21(5)	44(5)	5(5)	-16(5)	-9(5)
C(21)	40(6)	29(5)	78(7)	13(5)	-13(5)	-2(5)
C(22)	42(6)	70(6)	67(6)	-23(5)	15(5)	-31(5)
C(23)	63(6)	100(8)	86(7)	-20(7)	-8(5)	-13(7)
C(24)	41(5)	20(4)	34(5)	1(4)	-5(4)	-2(4)
C(25)	54(5)	29(4)	43(5)	-4(4)	-7(4)	-11(4)
C(26)	43(5)	23(4)	50(5)	-8(4)	-7(4)	-9(4)
C(27)	61(7)	24(5)	35(5)	-1(4)	11(5)	8(5)
C(28)	56(7)	42(6)	75(7)	-8(6)	21(6)	-9(5)

The anisotropic displacement factor exponent takes the form:
$-2\pi^2 [(ha^*)^2 U_{11} + \ldots + 2hka^* b^* U_{12}]$.

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Table 5. Hydrogen Coordinates [x 10^4] and Isotropic Displacement Parameters [$Å^2$ x 10^3] for **14b**.

Atom	x	y	z	U(eq)
H(3A)	-5944(61)	-2844(16)	111(10)	47
H(1A)	-5520(6)	-4745(5)	-1140(2)	25
H(2A)	-6628(7)	-4969(5)	-411(2)	32
H(3B)	-7456(7)	-6731(5)	-413(2)	36
H(4A)	-8520(7)	-5966(5)	-1057(2)	39
H(5A)	-8335(8)	-6452(5)	-1844(2)	41
H(6A)	-7869(7)	-4760(5)	-2046(2)	41
H(6B)	-7245(7)	-4549(5)	-1510(2)	41
H(7A)	-5588(7)	-4717(5)	-2361(2)	39
H(7B)	-4991(7)	-4363(5)	-1835(2)	39
H(9A)	-3163(7)	-6801(5)	-1588(2)	36
H(10A)	-3096(7)	-6096(5)	-888(2)	30
H(12A)	-2958(7)	-4031(5)	-27(2)	36
H(12B)	-2287(7)	-4882(5)	-417(2)	36
H(14A)	-2435(26)	-1520(6)	-973(9)	47
H(14B)	-1922(35)	-1841(7)	-1509(3)	47
H(14C)	-743(13)	-1723(8)	-1085(11)	47
H(15A)	-1042(26)	-4666(6)	-1230(9)	52
H(15B)	102(7)	-3675(18)	-1275(10)	52
H(15C)	-1137(25)	-3829(21)	-1677(3)	52
H(16A)	-4346(7)	-2382(5)	-424(2)	28
H(17A)	-5890(8)	-2421(5)	-1125(2)	34
H(18B)	-8338(9)	-2414(5)	-1017(2)	53
H(18A)	-7805(9)	-2813(5)	-452(2)	53
H(19A)	-4787(30)	-5193(6)	136(6)	38
H(19B)	-4364(23)	-6371(21)	-85(2)	38
H(19C)	-5885(11)	-6211(23)	191(5)	38
H(22A)	-10355(8)	-8239(7)	-981(3)	72
H(22B)	-10354(8)	-6923(7)	-955(3)	72
H(23A)	-10929(42)	-8443(15)	-2173(8)	100
H(23B)	-10849(44)	-9113(10)	-1675(8)	100
H(23C)	-9385(9)	-8665(20)	-1922(14)	100

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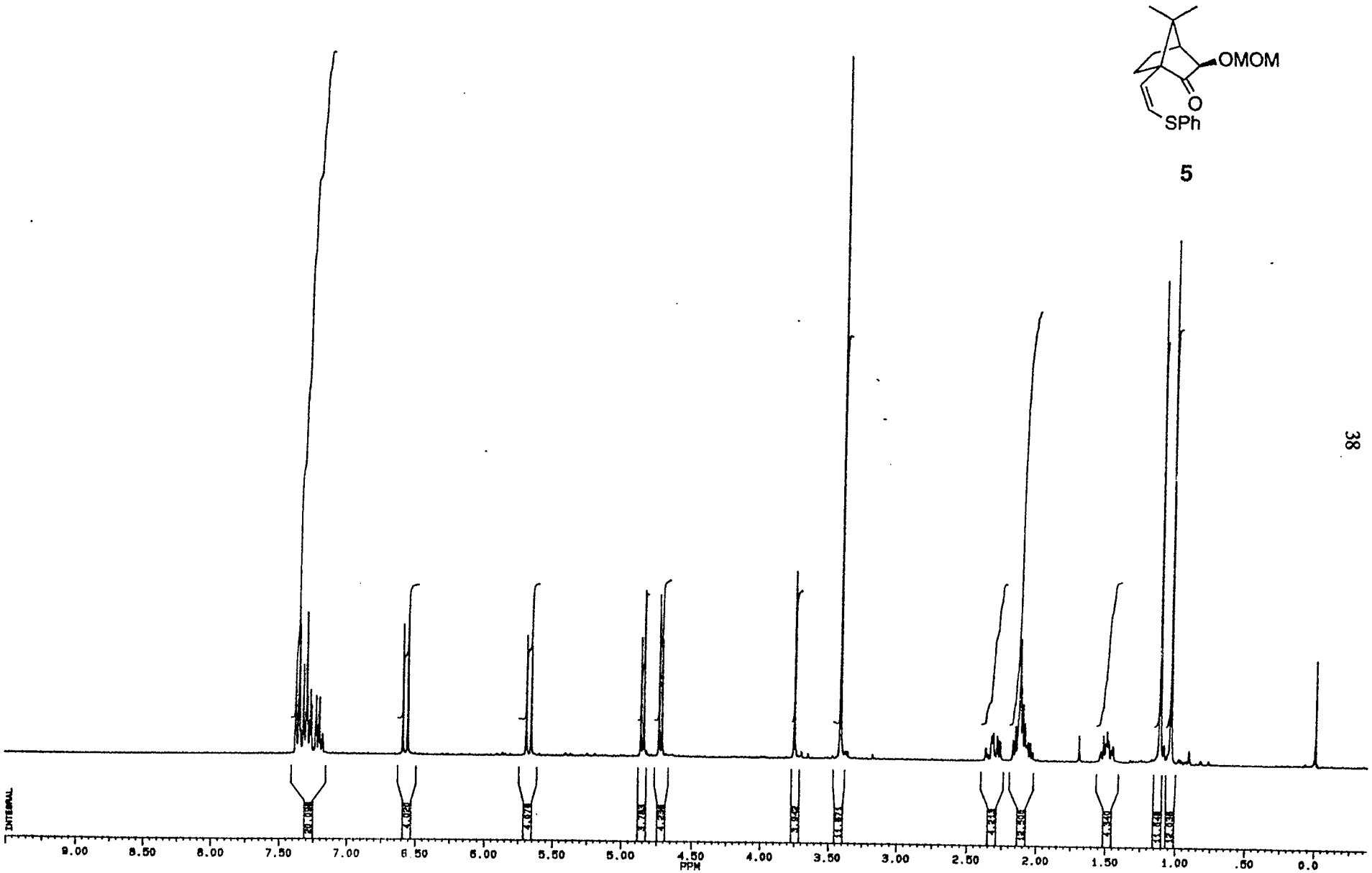
Table 5 (continued)

H(25A)	-7711(21)	-7368(25)	-2508(3)	50
H(25B)	-6079(18)	-7309(25)	-2712(3)	50
H(25C)	-6989(35)	-6198(5)	-2629(5)	50
H(26A)	-6662(12)	-8540(7)	-1869(11)	46
H(26B)	-5519(35)	-8055(7)	-1486(4)	46
H(26C)	-5007(24)	-8328(11)	-2027(8)	46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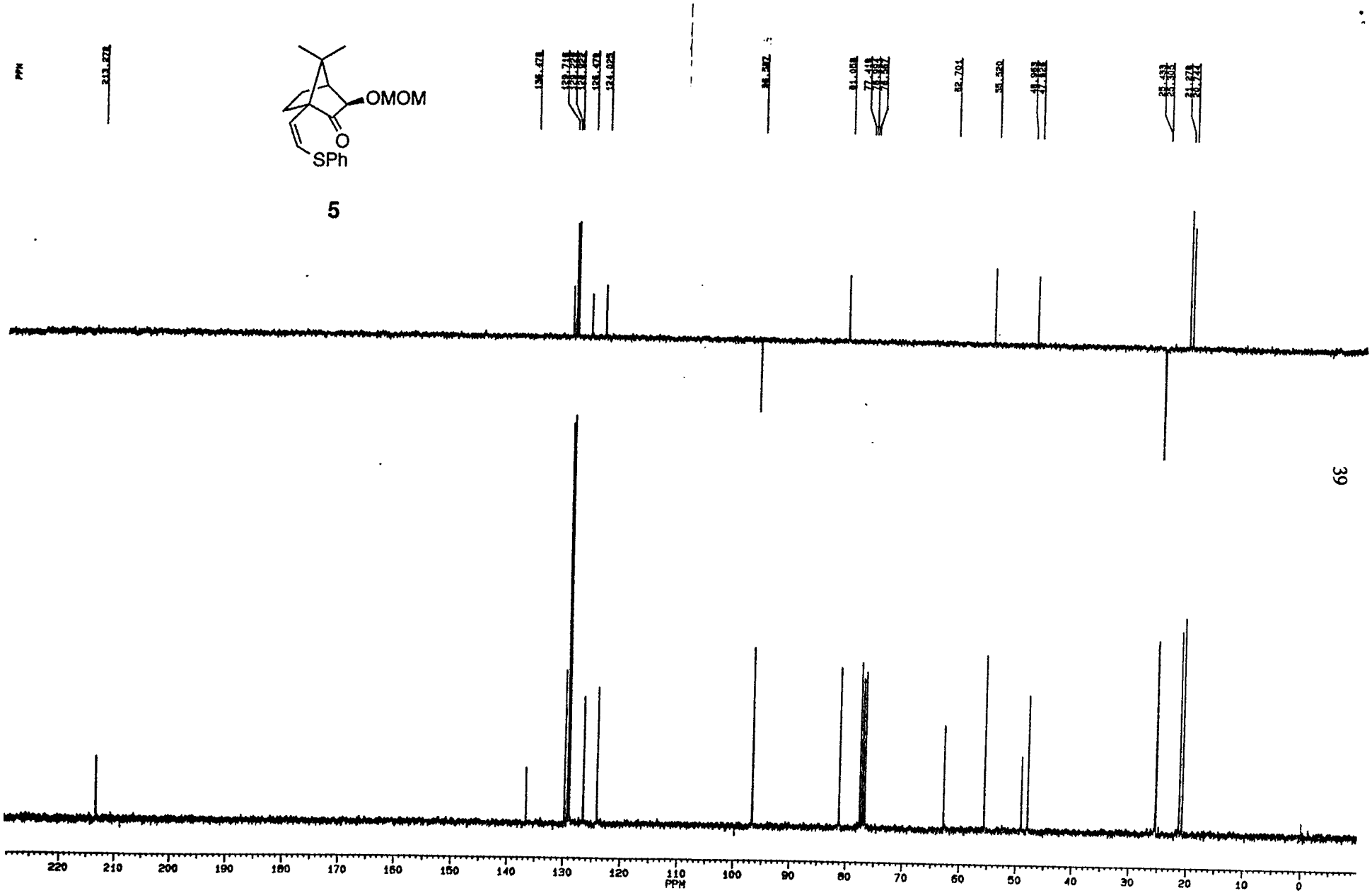
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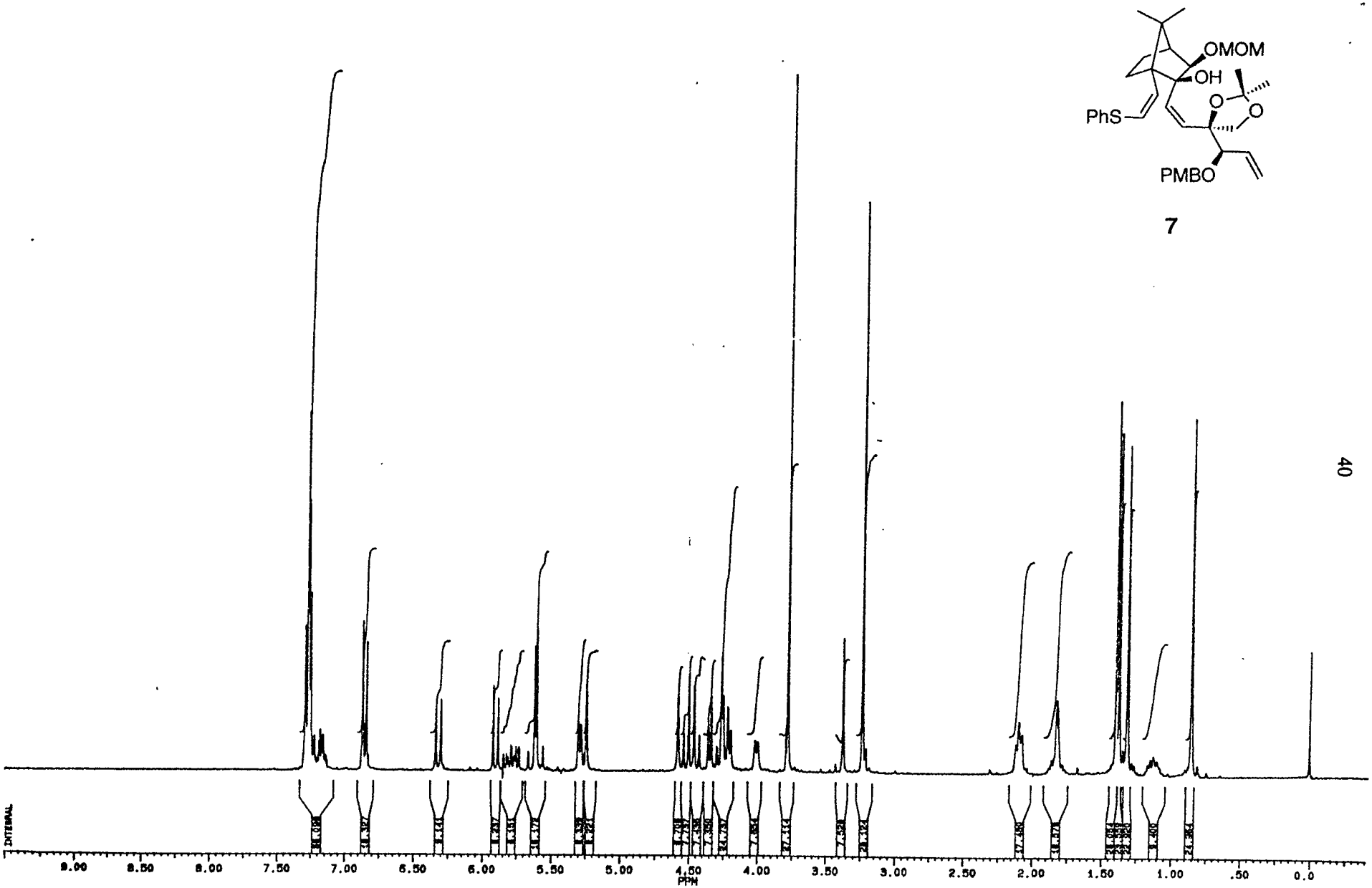
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