SUPPORTING INFORMATION

A Practical Asymmetric Synthesis of Aprepitant, a Potent Human NK-1 Receptor Antagonist, via a Stereoselective Lewis Acid-Catalyzed *trans*-Acetalization Reaction

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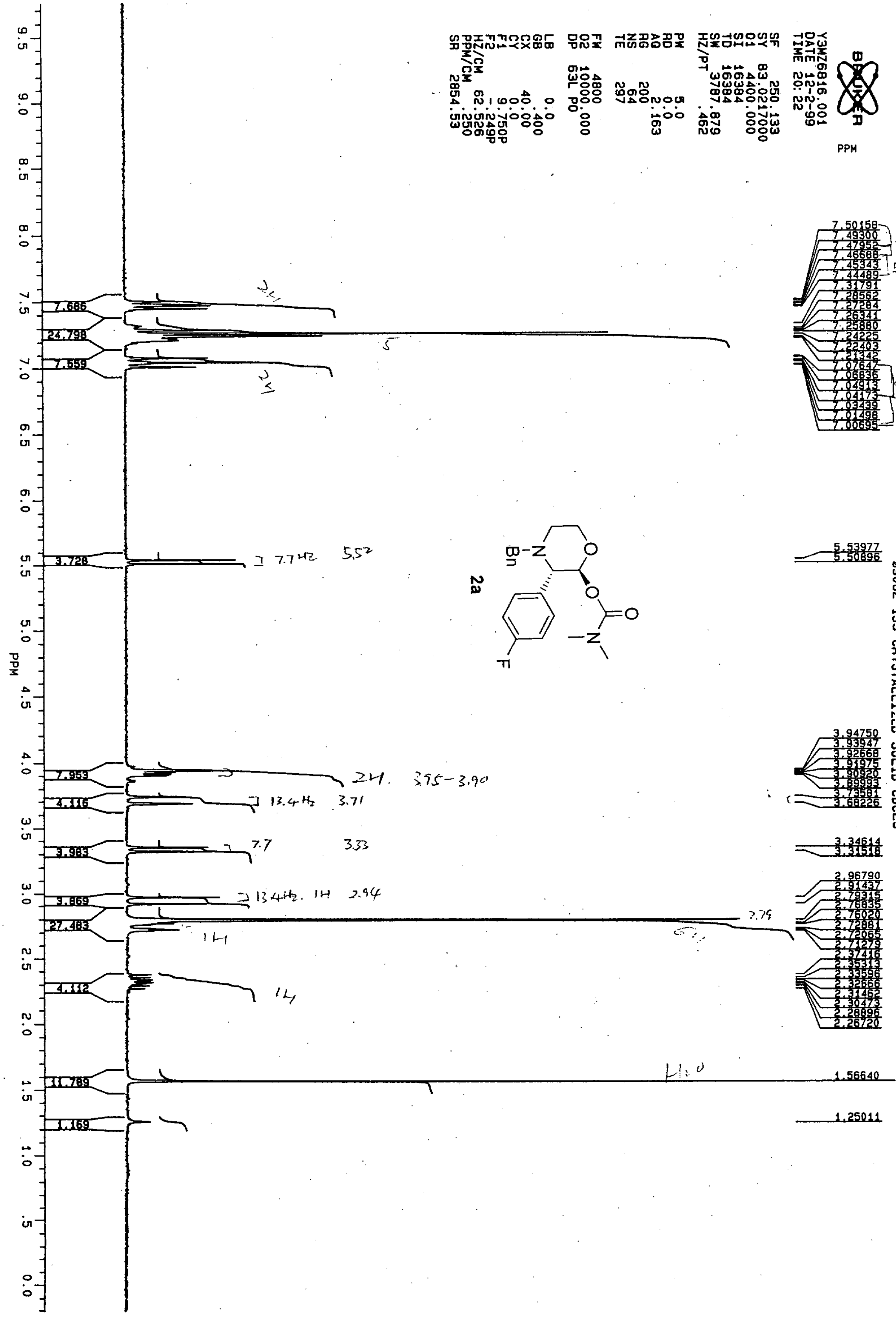
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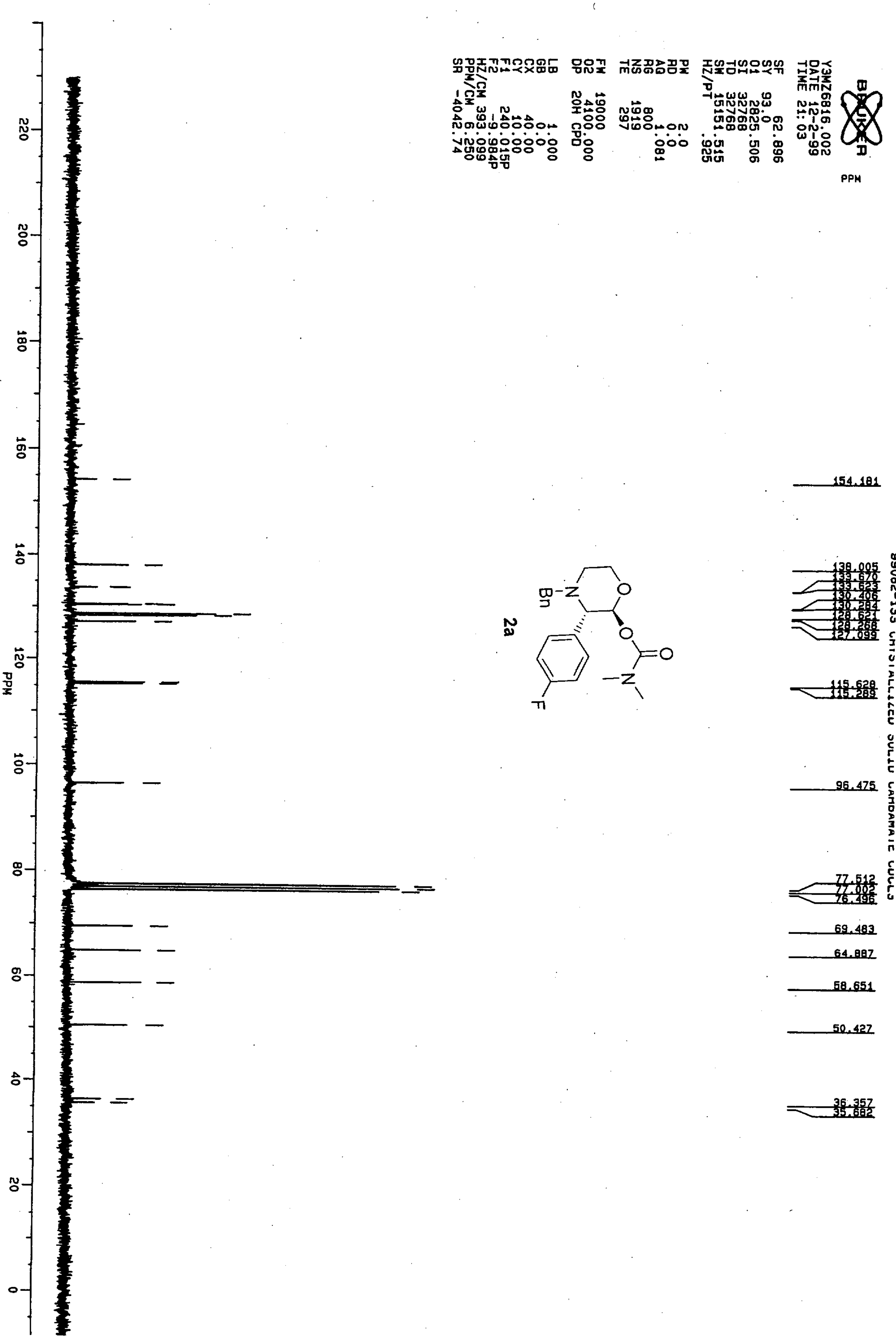

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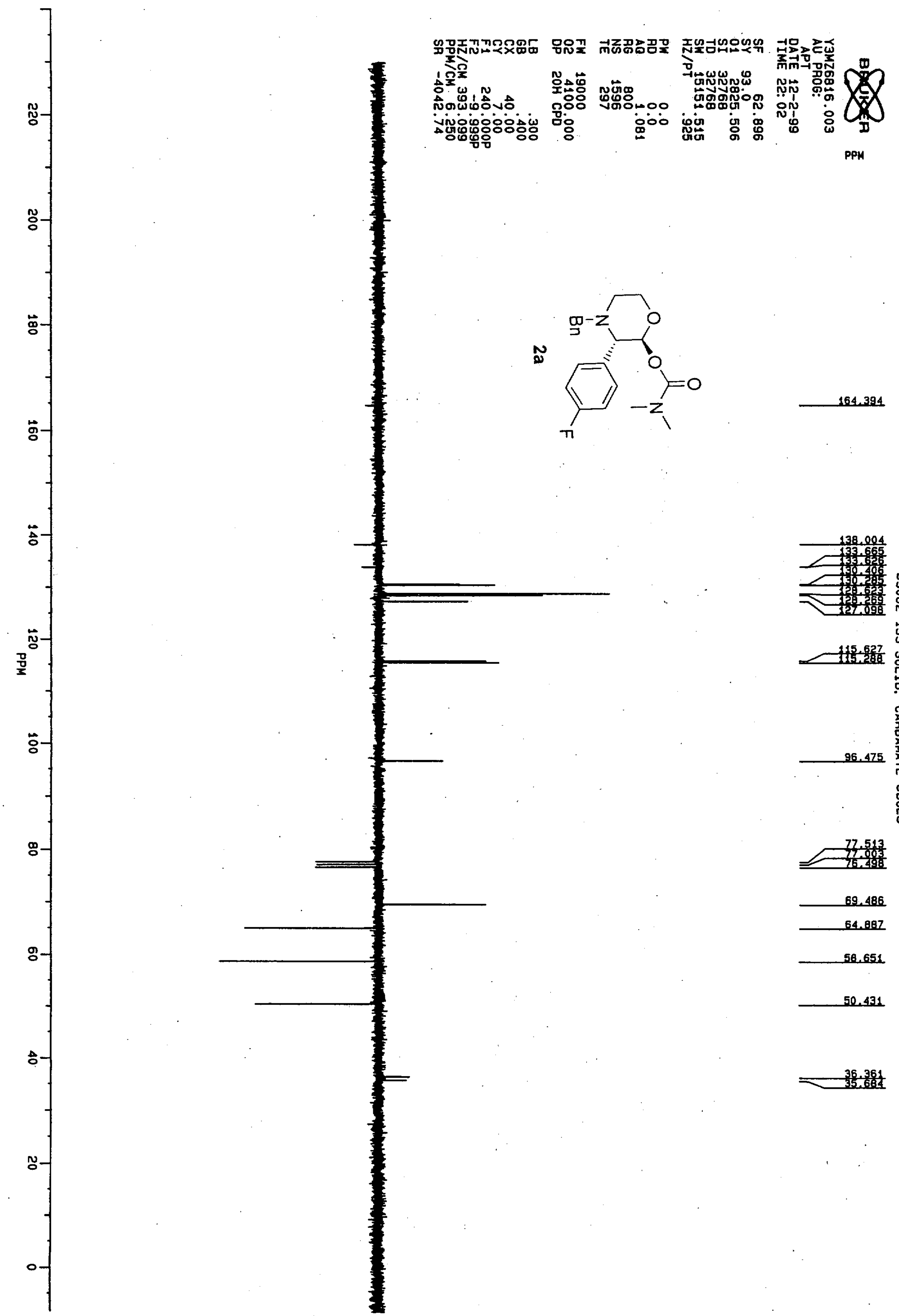
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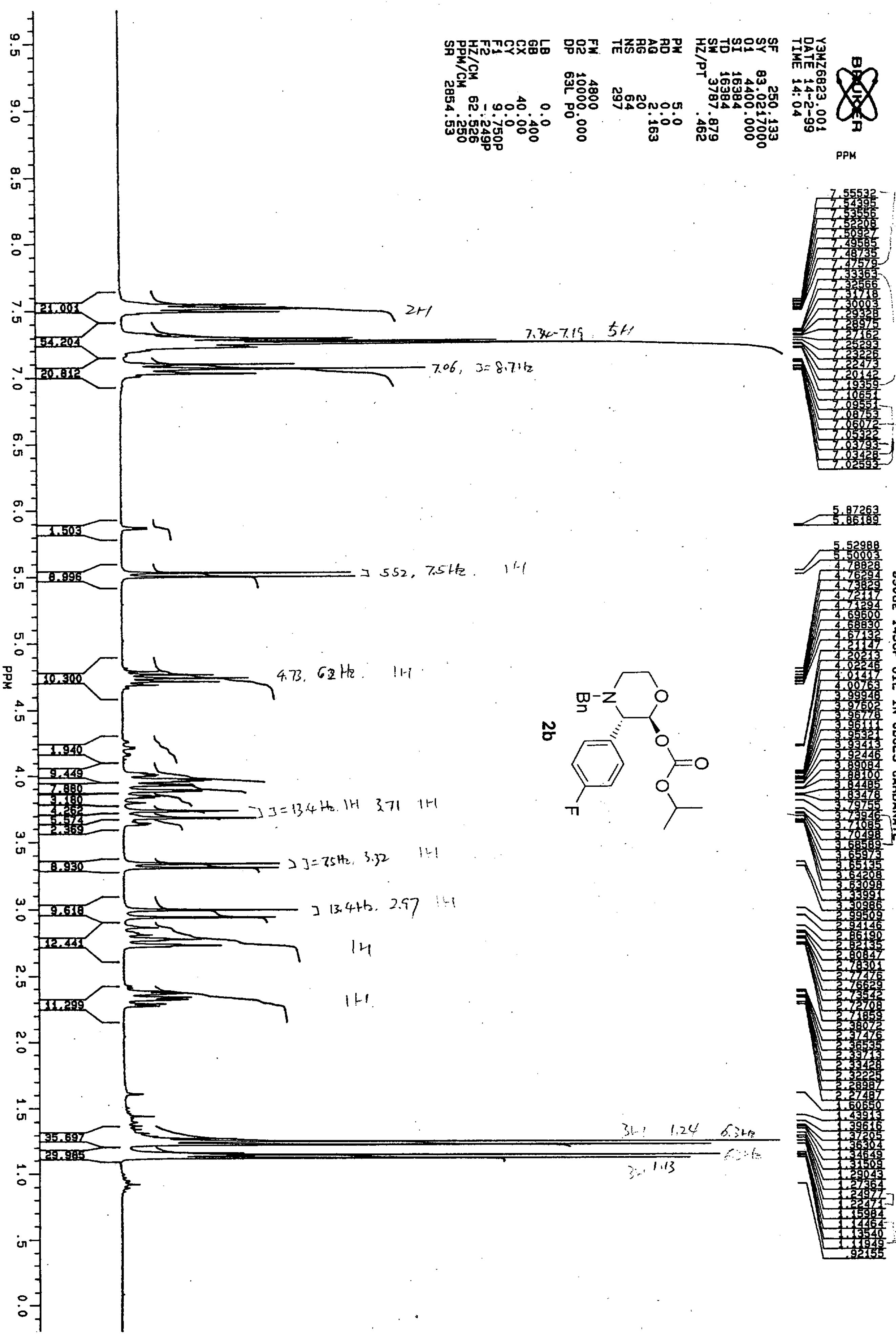

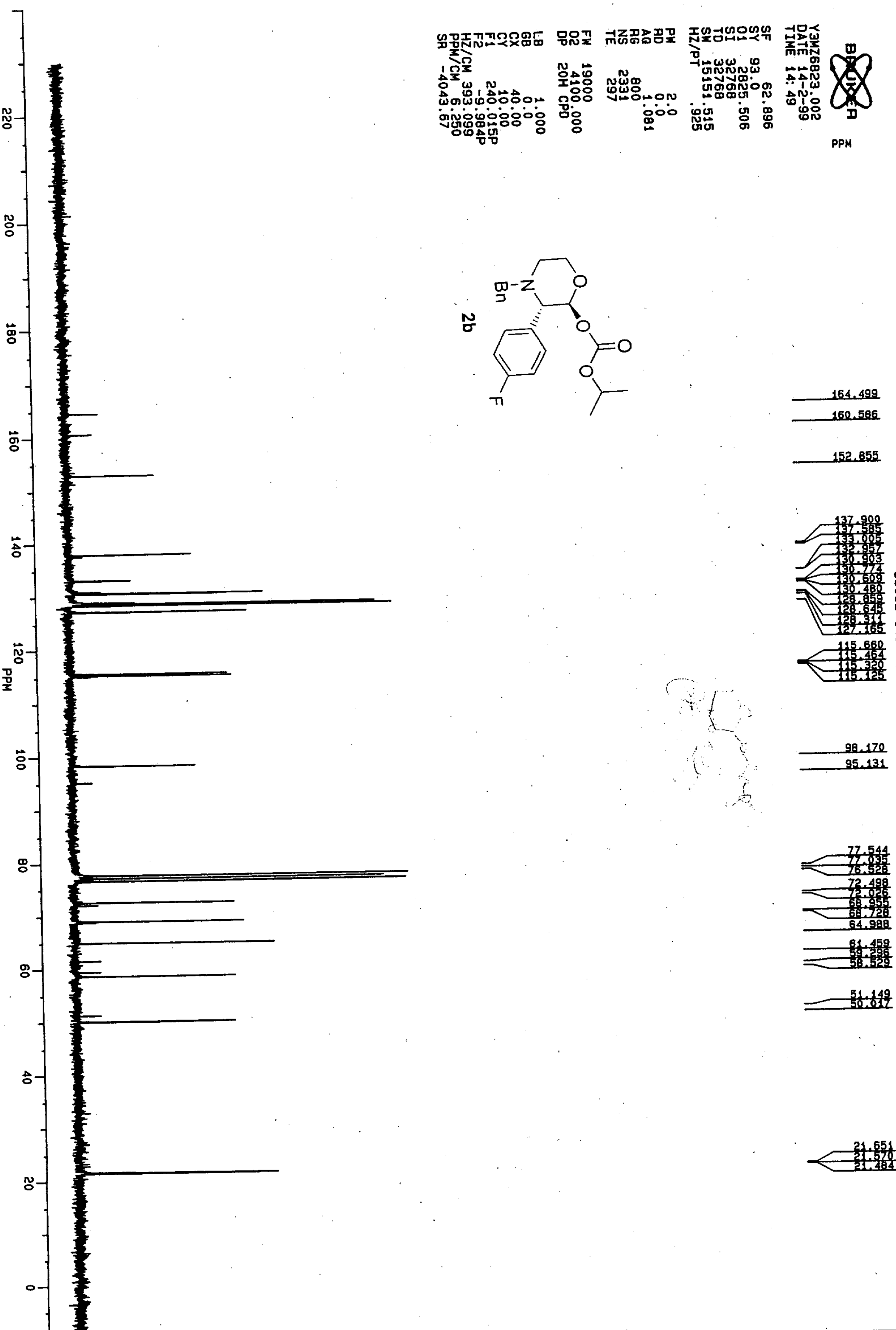
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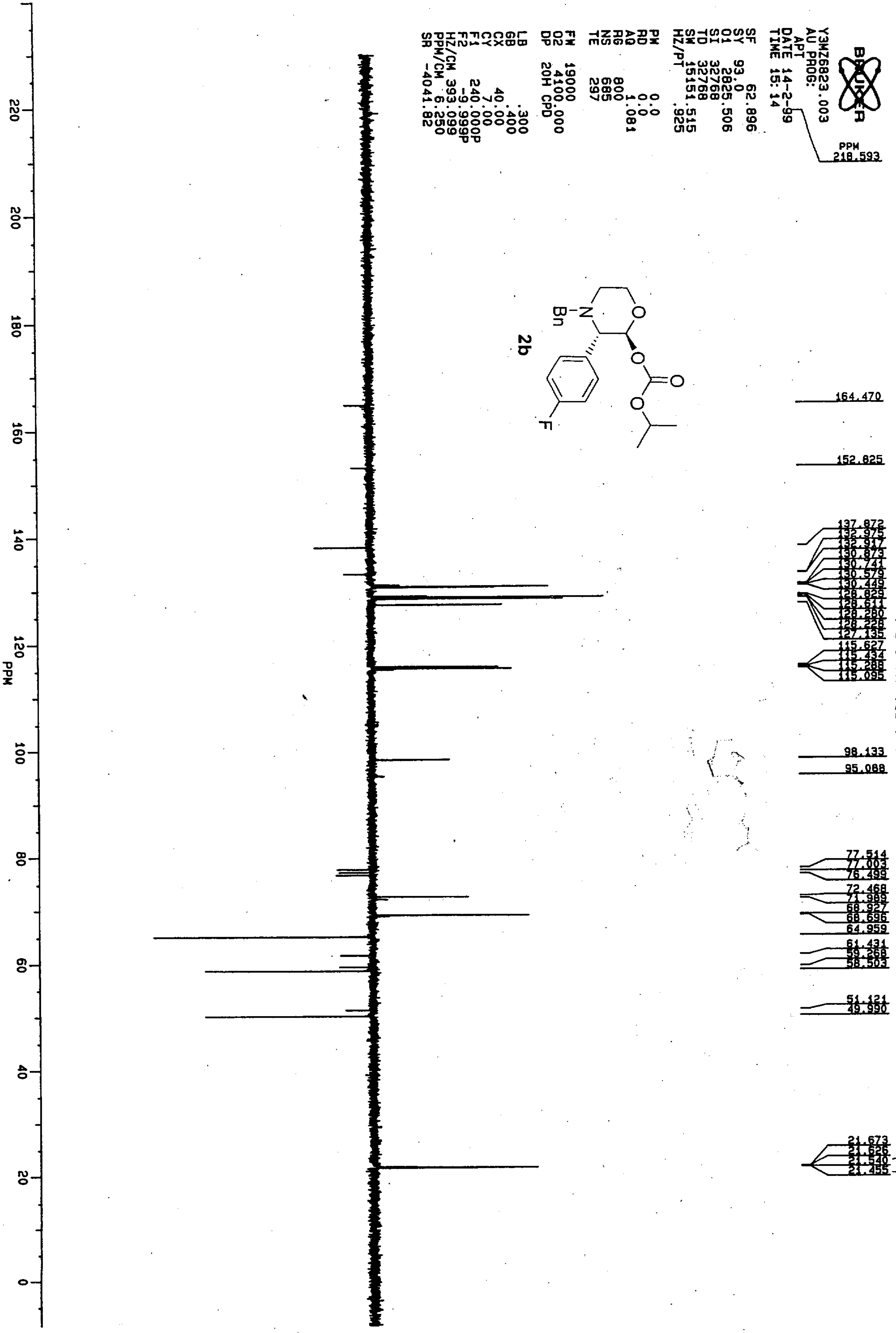
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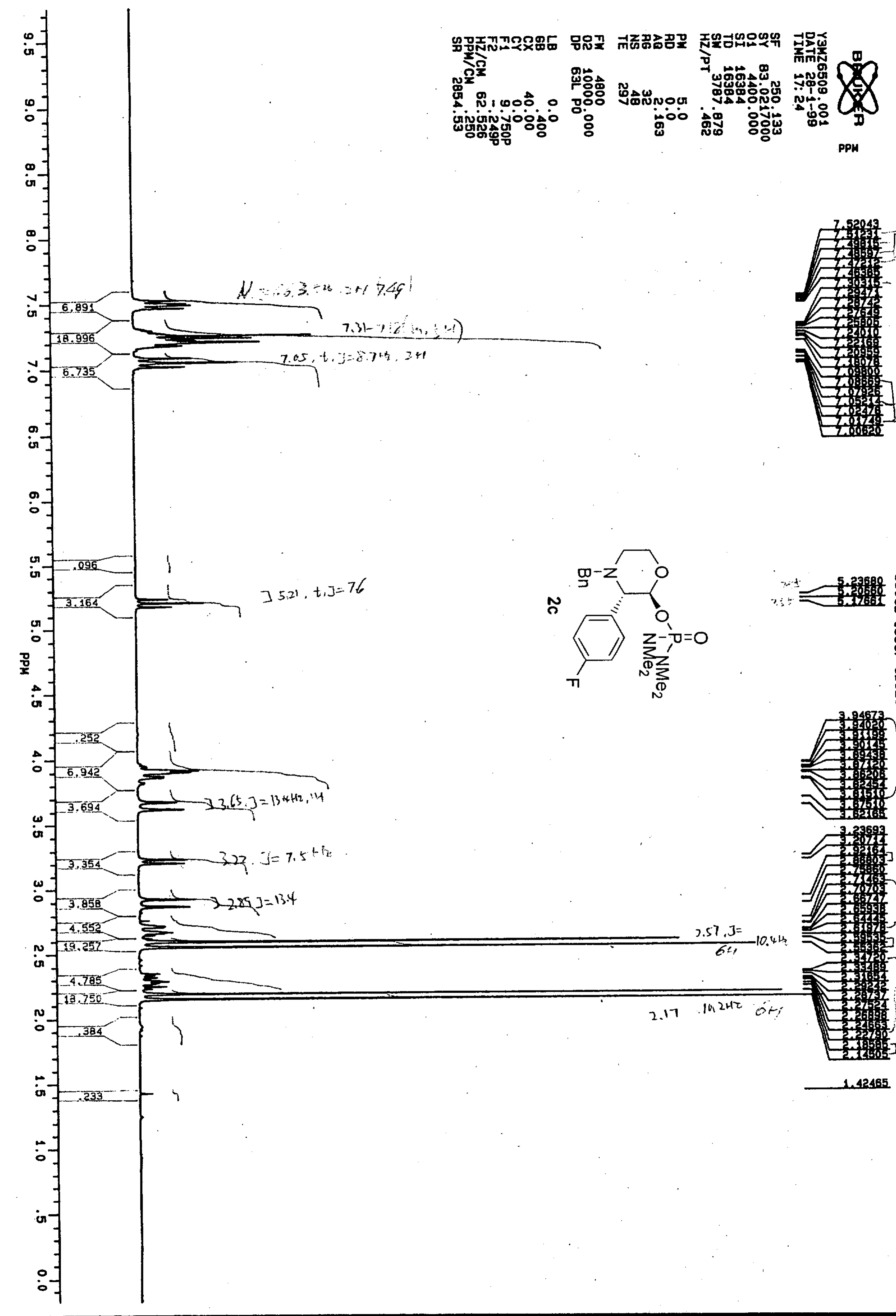

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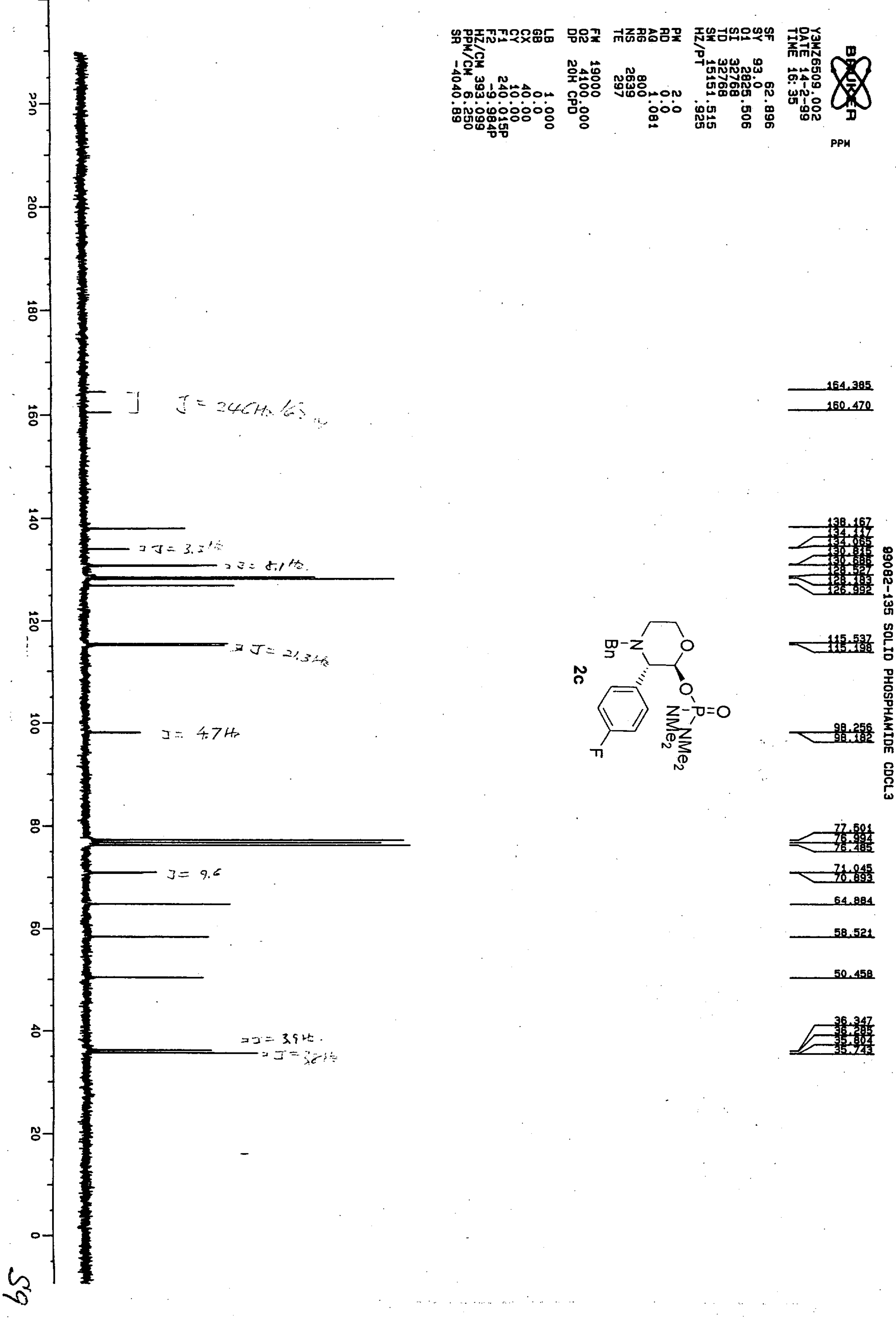
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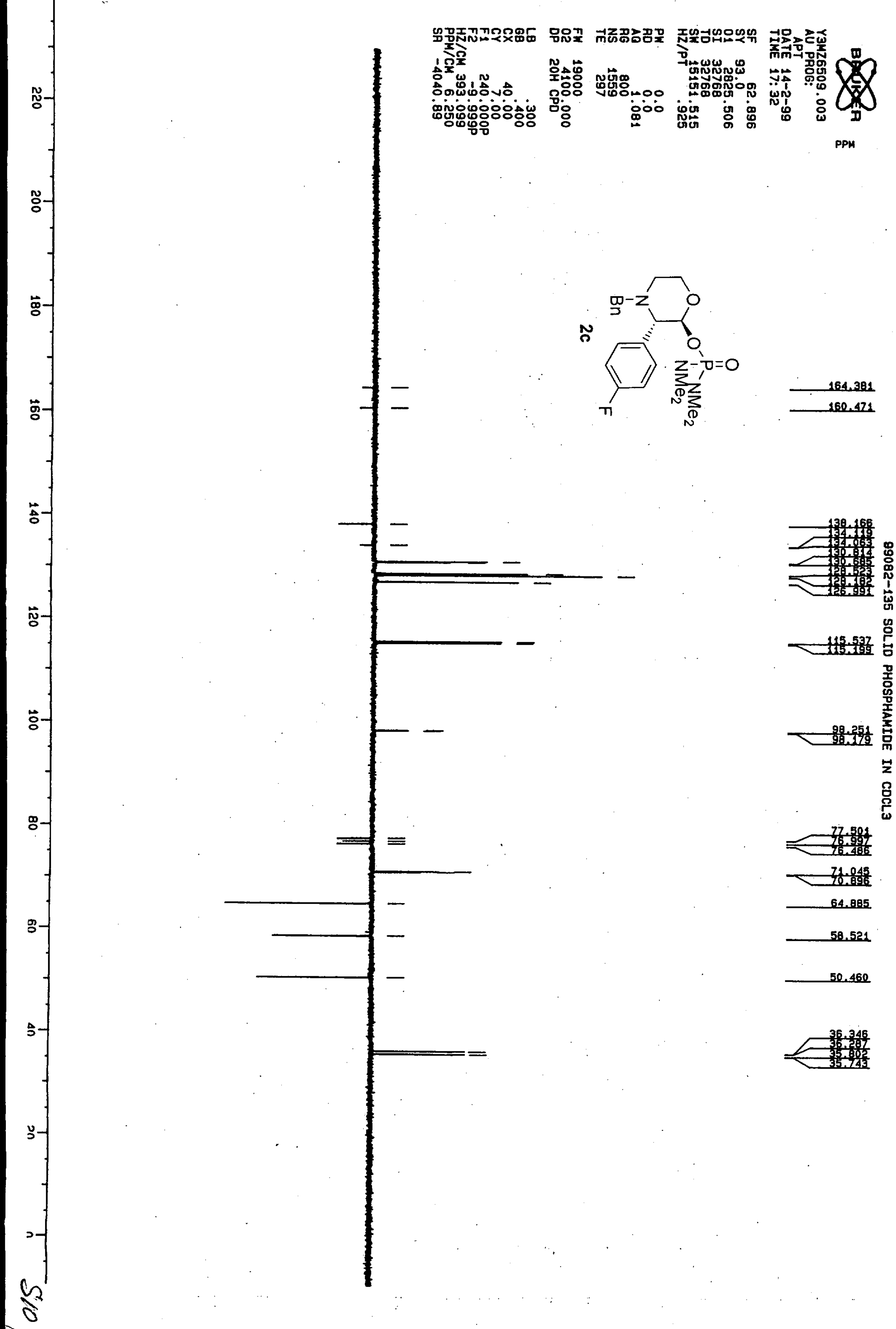
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CY 7.00
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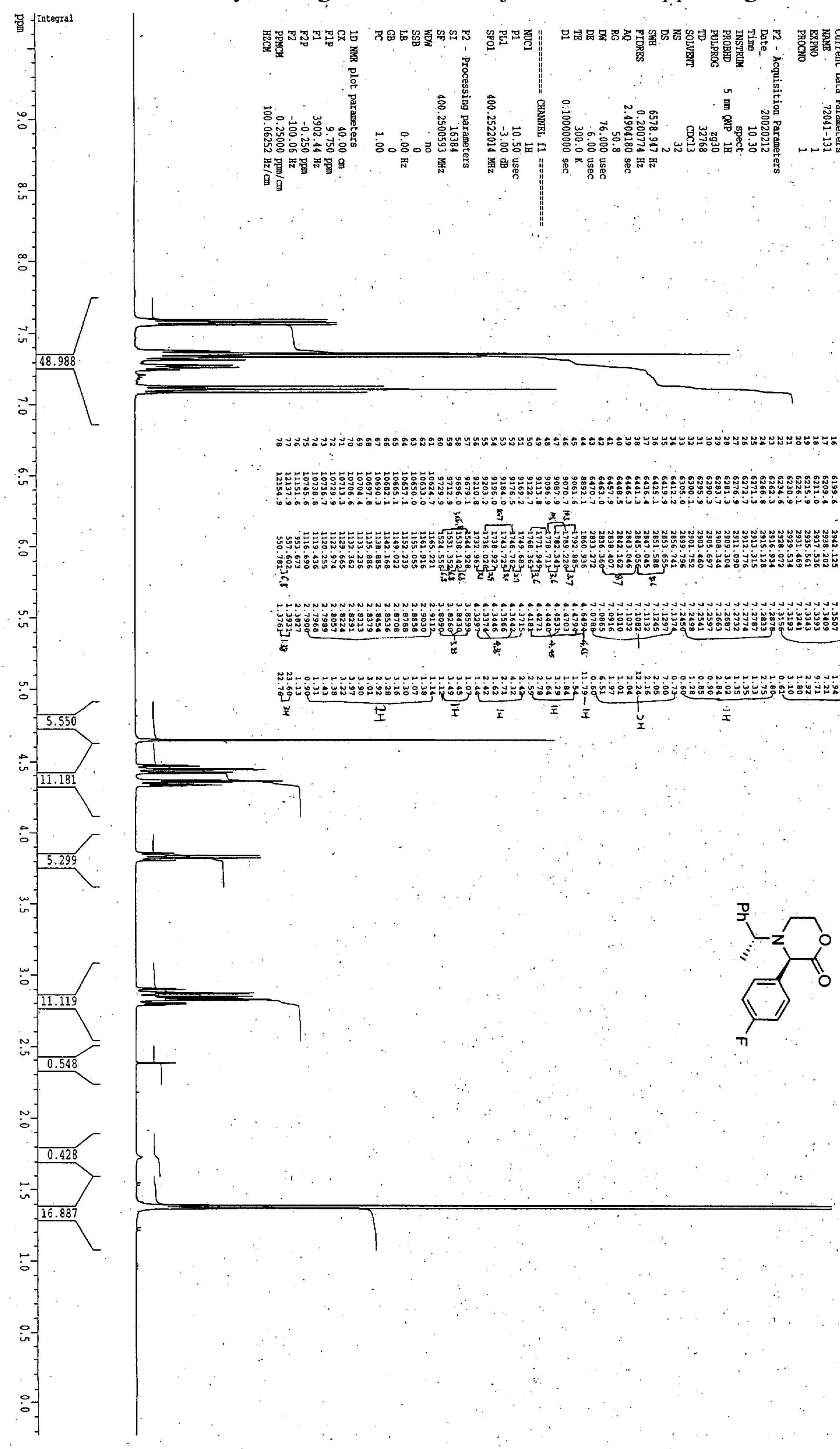
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TD 32768
SOLVENT CDCl3
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FIDRES 0.200774 Hz
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RG 50.8
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz
F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
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SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
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F2P -0.250 ppm
F2 -100.06 Hz
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HZCM 100.06252 Hz/cm
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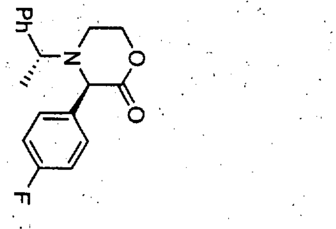
nmr400a h-1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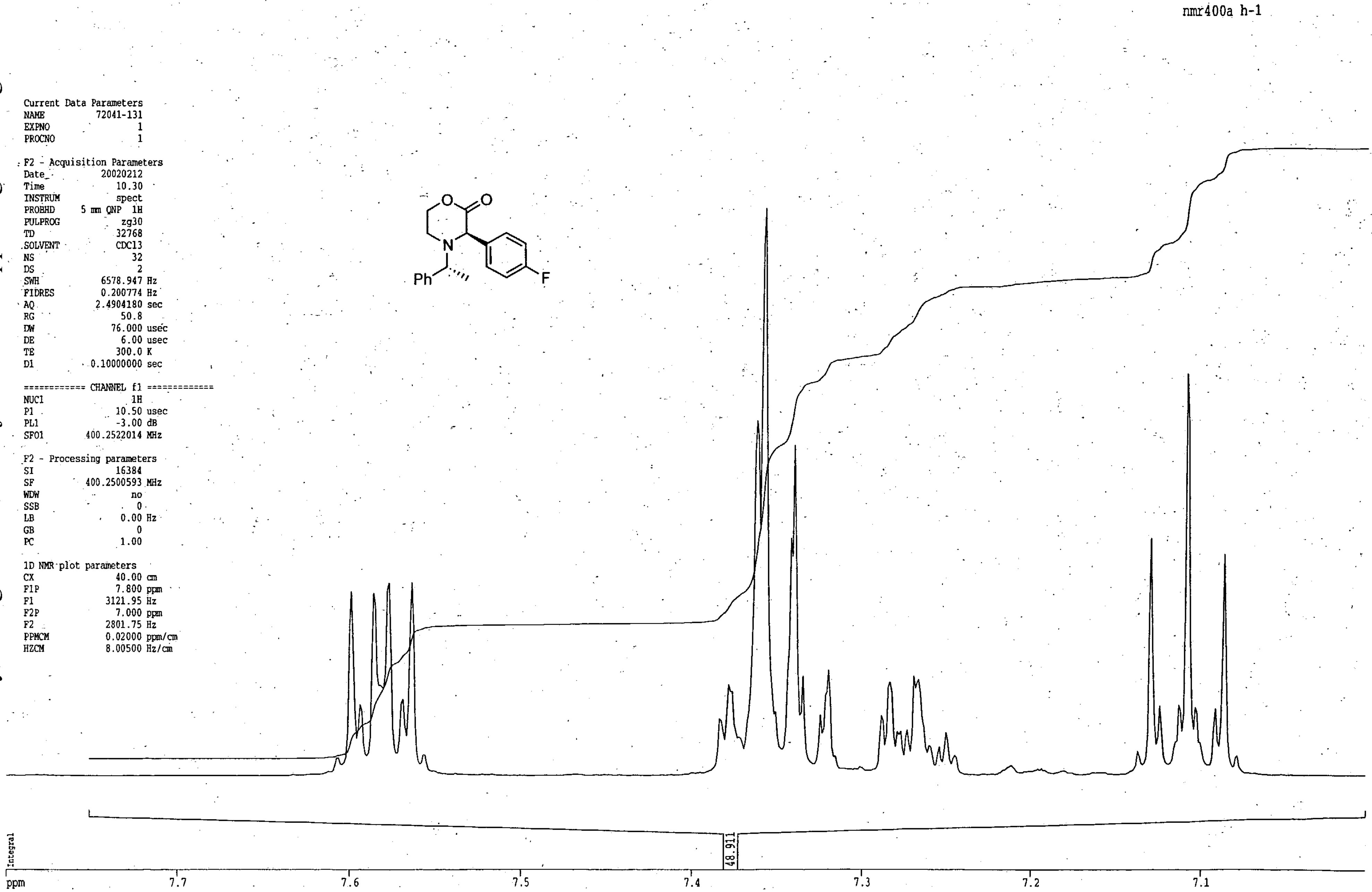
S11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PROCNO 1
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Time 10.30
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 50.8
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
============ CHANNEL f1 ============
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz
F2 - Processing parameters
SI 16384
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WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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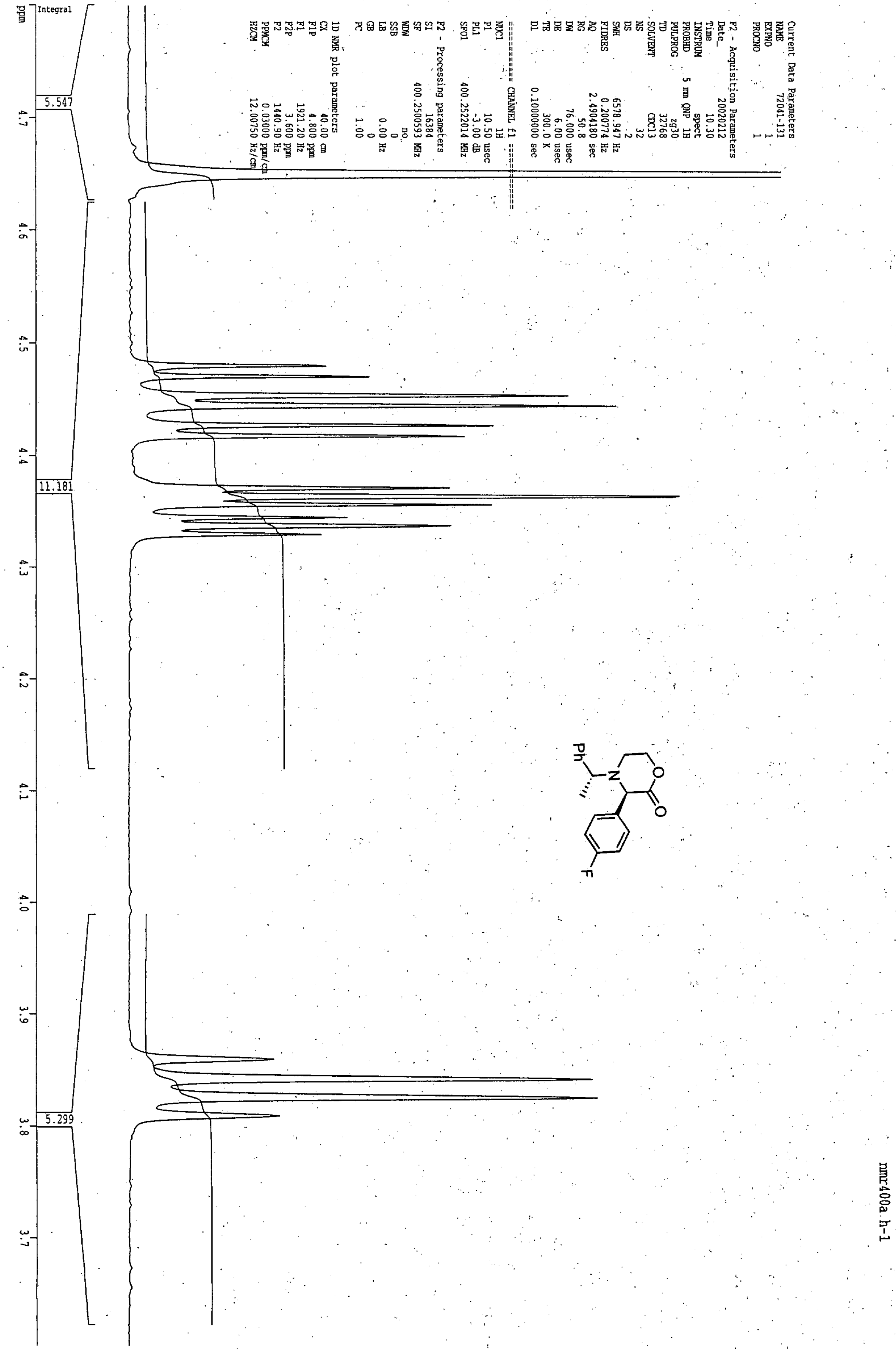
7.2
7.1

S12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TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 50.8
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
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P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz
F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 40.00 cm
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F2P 3.600 ppm
F2 1440.90 Hz
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HZCM 12.00750 Hz/cm
Integral
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ppm
4.7
4.6
4.5
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4.3
4.2
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4.0
3.9
3.8
3.7
Ph
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O
O
F
nmr400a h-1

nmr400a h-1

Current Data Parameters
NAME 72041-131
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020212
Time 10.30
INSTRUM spect
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
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FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 50.8
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

============ CHANNEL f1 ============
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
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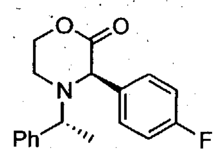

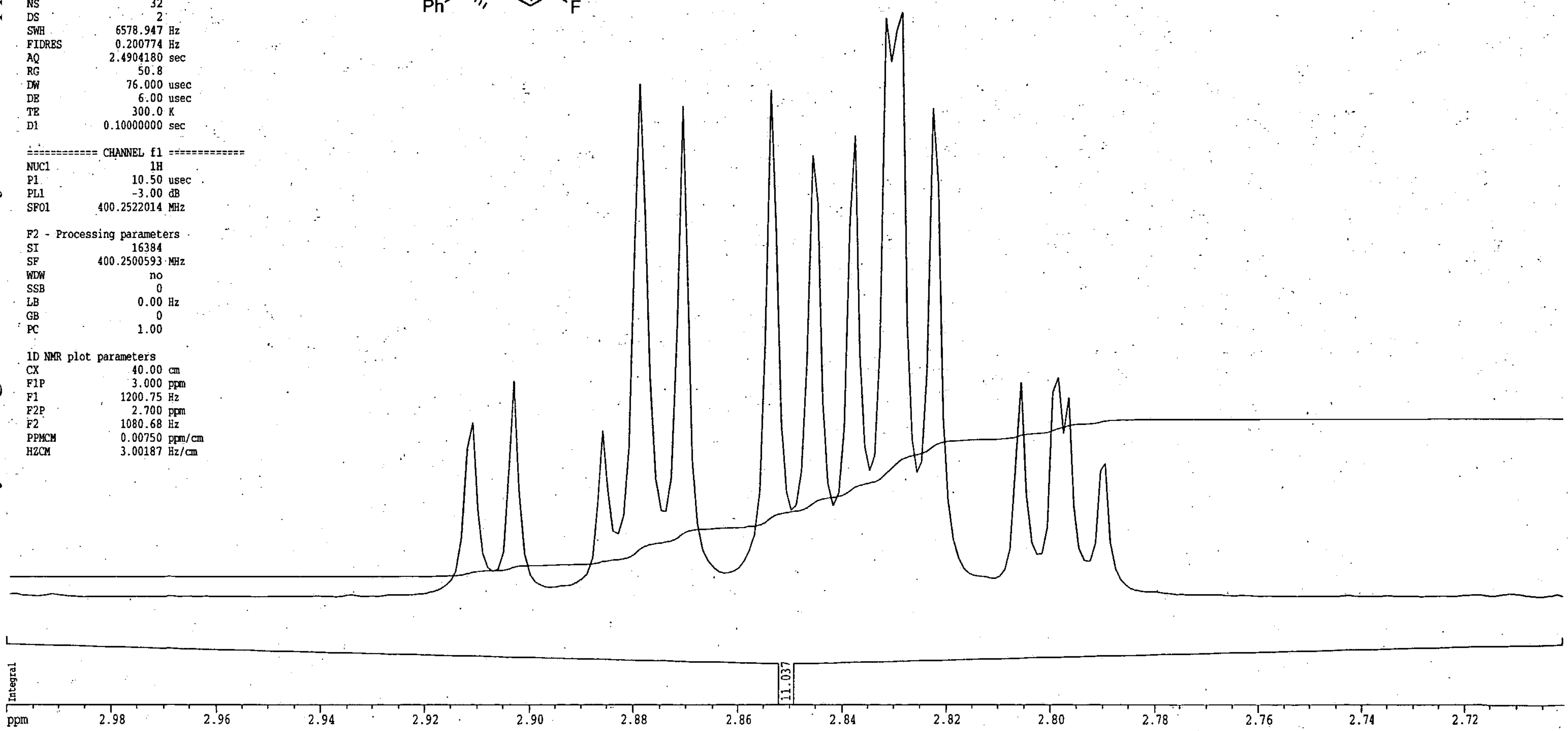

S/4

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SOLVENT	CDCl3	
NS	460	
DS	0	
SWH	26315.789	Hz
FIDRES	0.401547	Hz
AQ	1.2452340	sec
RG	8192	
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DE	6.00	usec
TE	300.0	K
D1	0.10000000	sec
d11	0.03000000	sec
============ CHANNEL f1 ============		
NUC1	13C	
P1	2.00	usec
PL1	0.00	dB
SFO1	100.6539737	MHz
============ CHANNEL f2 ============		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	93.00	usec
PL2	120.00	dB
PL12	18.00	dB
SFO2	400.2520012	MHz
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1D NMR plot parameters		
CX	40.00	cm
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F1	24154.31	Hz
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F2	-1006.42	Hz
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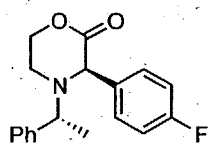
HZCM	629.01849	Hz/cm



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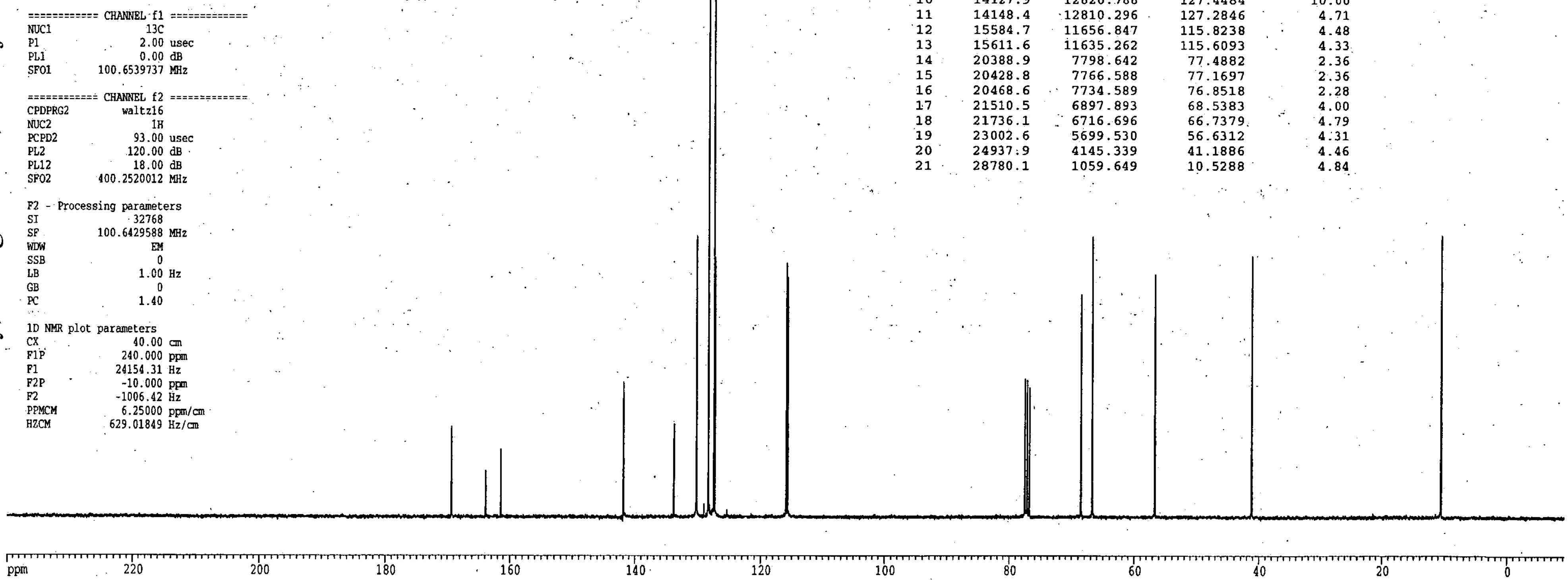
3	9870.0	16246.284	161.4249	1.15
4	12306.5	14289.519	141.9823	2.44
5	13312.2	13481.880	133.9575	1.22
6	13316.1	13478.772	133.9266	1.59
7	13768.6	13115.300	130.3151	4.35
8	13778.9	13107.092	130.2336	4.83
9	14012.2	12919.727	128.3719	9.41
10	14127.9	12826.788	127.4484	10.00
11	14148.4	12810.296	127.2846	4.71
12	15584.7	11656.847	115.8238	4.48
13	15611.6	11635.262	115.6093	4.33
14	20388.9	7798.642	77.4882	2.36
15	20428.8	7766.588	77.1697	2.36
16	20468.6	7734.589	76.8518	2.28
17	21510.5	6897.893	68.5383	4.00
18	21736.1	6716.696	66.7379	4.79
19	23002.6	5699.530	56.6312	4.31
20	24937.9	4145.339	41.1886	4.46
21	28780.1	1059.649	10.5288	4.84



S15

nmr400a apt

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9870.0	16246.306	161.4252	-0.44
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13312.1	13481.944	133.9581	-0.49
13316.1	13478.739	133.9263	-0.52
13768.6	13115.348	130.3156	4.17
13778.9	13107.103	130.2337	4.58
13925.9	12989.008	129.0603	0.08
14012.1	12919.748	128.3721	9.50
14027.0	12907.839	128.2538	0.12
14127.9	12826.771	127.4483	10.00
14148.4	12810.312	127.2847	4.70
15584.7	11656.856	115.8239	3.94
15611.5	11635.289	115.6096	3.76
20388.9	7798.642	77.4882	-0.36
20428.9	7766.508	77.1689	-0.33
20468.6	7734.596	76.8518	-0.34
21510.3	6897.990	68.5392	-4.07
21736.1	6716.704	66.7379	3.78
23002.4	5699.754	56.6334	3.71
24937.9	4145.314	41.1883	-4.46
28780.3	1059.487	10.5272	2.88

Current Data Parameters

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PROCNO	1	

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PULPROG	jmod	
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SOLVENT	CDCl3	
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FIDRES	0.401547	Hz
AQ	1.2452340	sec
RG	32768	
DW	19.000	usec
DE	6.00	usec
TE	300.0	K
CNST2	145.0000000	
CNST11	1.0000000	
D1	1.00000000	sec
d13	0.00000300	sec
d20	0.00689655	sec
DELTA	6366.18261719	sec

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

NUC1	13C	
P1	5.60	usec
p2	11.20	usec
PL1	0.00	dB
SFO1	100.6539737	MHz

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

CPDPRG2	waltz16	
NUC2	1H	
PCPD2	93.00	usec
PL2	120.00	dB
PL12	18.00	dB
SFO2	400.2520012	MHz

F2 - Processing parameters

SI	32768	
SF	100.6429588	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

1D NMR plot parameters

CX	40.00	cm
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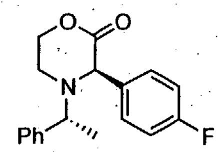
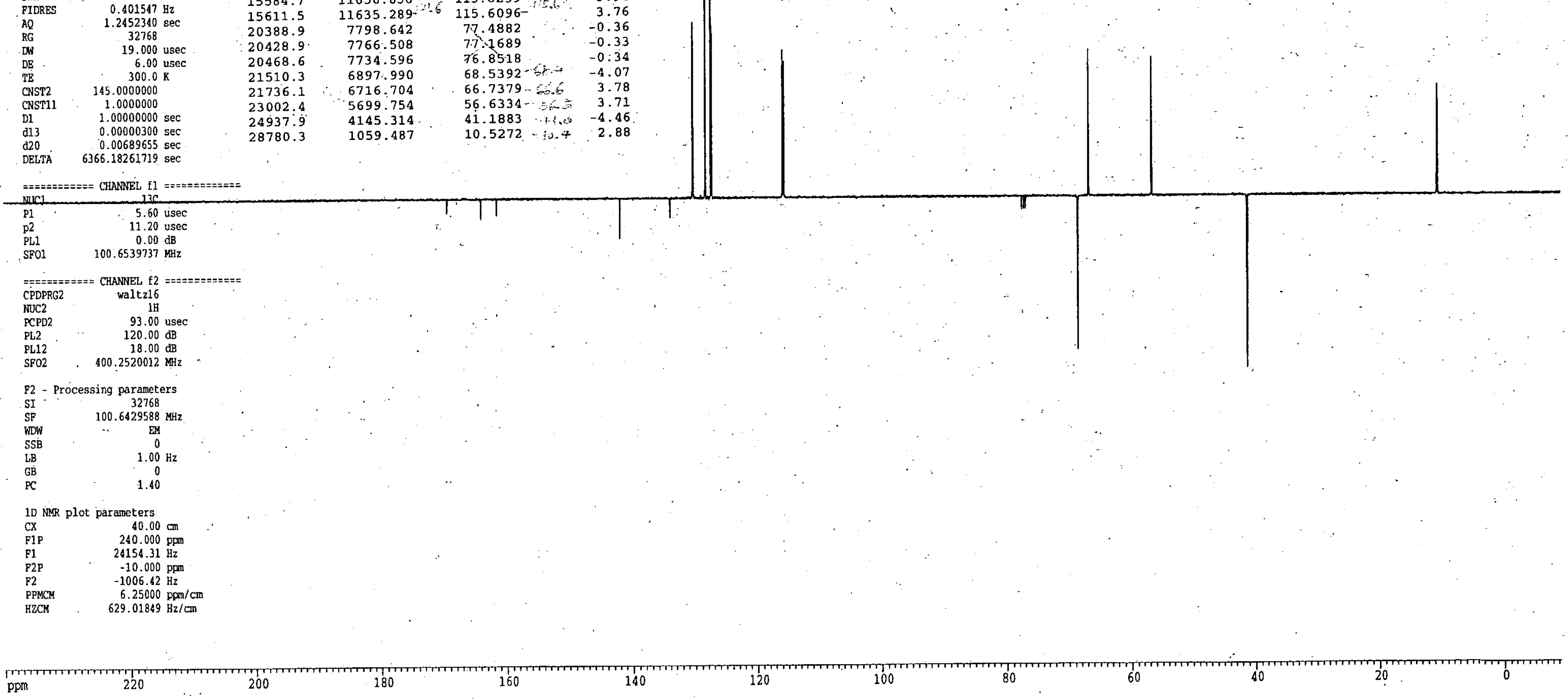
HZCM	629.01849	Hz/cm





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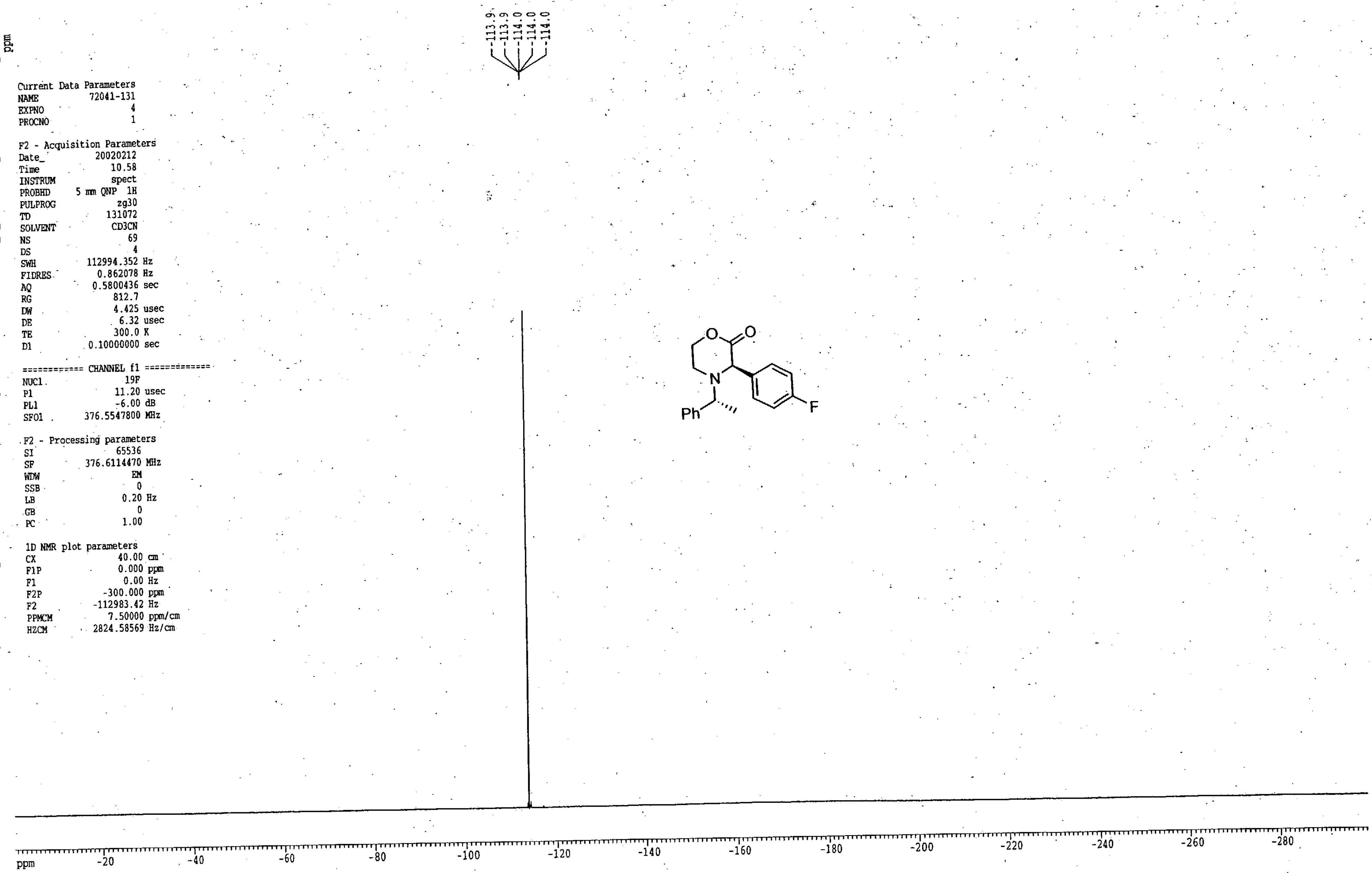

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TD 131072
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RG 812.7
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DE 6.32 usec
TE 300.0 K
D1 0.10000000 sec
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PL1 -6.00 dB
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F2 - Processing parameters
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SF 376.6114470 MHz
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LB 0.20 Hz
GB 0
PC 1.00
1D NMR plot parameters
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HZCM 2824.58569 Hz/cm
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-280
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O
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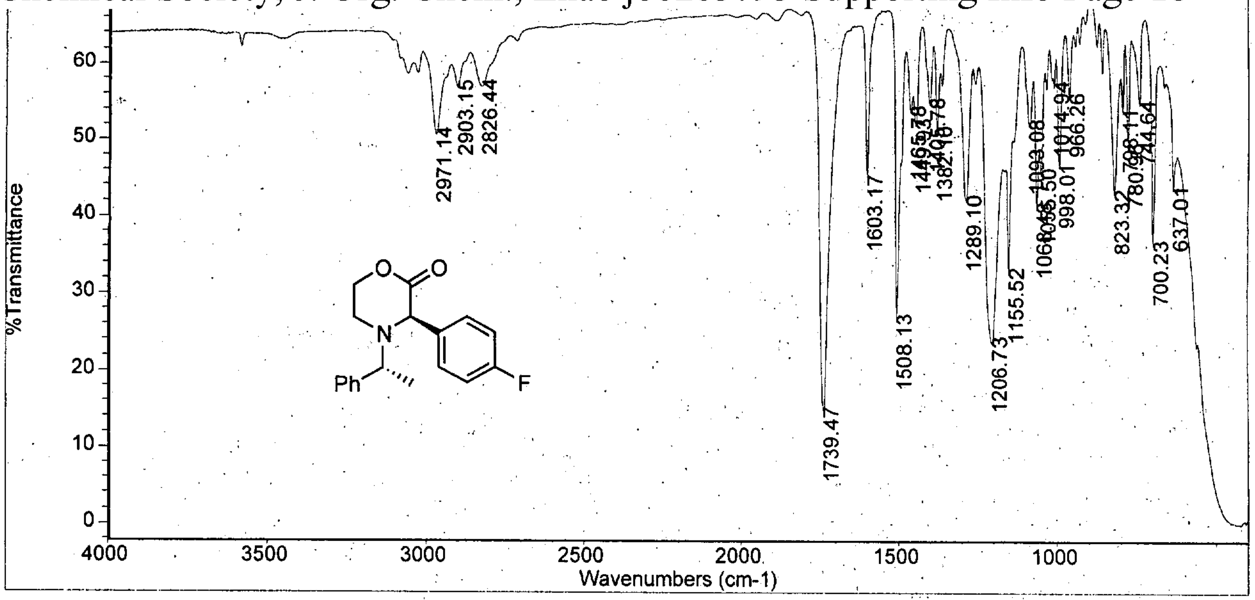
F

S17



Wed Feb 13 16:31:34 2002
FIND PEAKS:
Spectrum: 72041-131 oxazinone free base
Region: 4000.00 400.00
Absolute threshold: 57.446
Sensitivity: 50
Peak list:

Position:	1739.47	Intensity:	14.249
Position:	1206.73	Intensity:	23.390
Position:	1508.13	Intensity:	26.283
Position:	1155.52	Intensity:	32.342
Position:	700.23	Intensity:	36.016
Position:	1068.48	Intensity:	40.859
Position:	1289.10	Intensity:	41.709
Position:	823.32	Intensity:	42.563
Position:	637.01	Intensity:	43.075
Position:	1603.17	Intensity:	44.240
Position:	1055.50	Intensity:	45.084
Position:	998.01	Intensity:	45.953
Position:	780.96	Intensity:	48.655
Position:	2971.14	Intensity:	50.493
Position:	1382.10	Intensity:	50.682
Position:	1093.08	Intensity:	51.598
Position:	1449.93	Intensity:	51.687
Position:	798.11	Intensity:	53.203
Position:	1465.78	Intensity:	53.360
Position:	744.64	Intensity:	54.237
Position:	1405.78	Intensity:	54.390
Position:	966.26	Intensity:	55.180
Position:	2903.15	Intensity:	56.609
Position:	1014.94	Intensity:	56.562
Position:	2826.44	Intensity:	56.750

S18

nmr400a h-1

Current Data Parameters
NAME 24937-268
EXPNO 1
PROCNO 1

lacted

F2 - Acquisition Parameters
Date_ 20020204
Time 12.18
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 90.5
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

============ CHANNEL f1 ============
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
F1 3902.44 Hz
F2P -0.250 ppm
F2 -100.06 Hz
PPMCM 0.25000 ppm/cm
HZCM 100.06252 Hz/cm

~w/data/zhaom/nmr/24937-268/1/pdata/1/screen Mon F

XU=/w, USER=zhaom, NAME=24937-268, EXPNO=1, PROCNO=1
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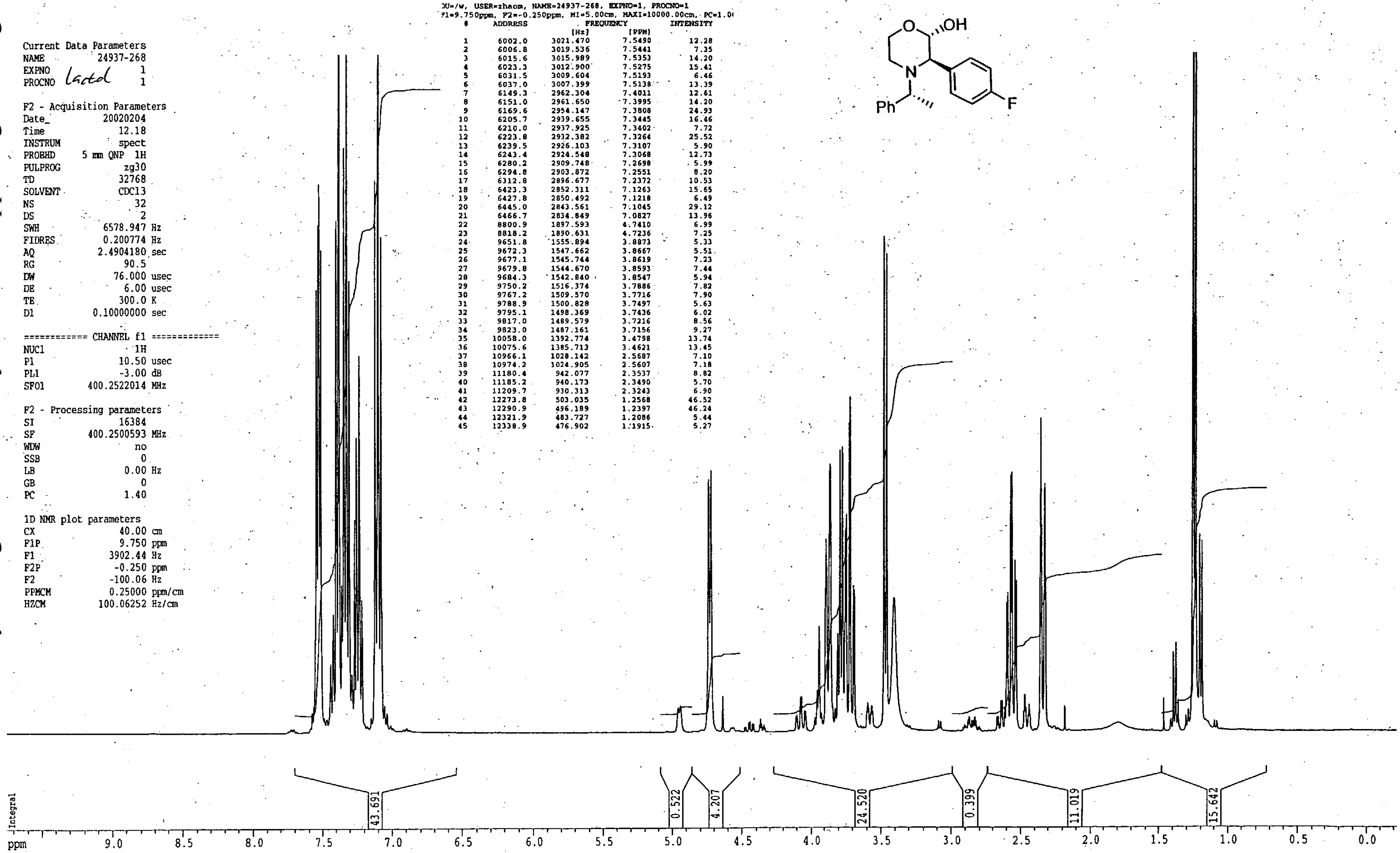
#	ADDRESS	FREQUENCY [Hz]	[PPM]	INTENSITY
1	6002.0	3021.470	7.5490	12.28
2	6006.8	3019.536	7.5441	7.35
3	6015.6	3015.989	7.5353	14.20
4	6023.3	3012.900	7.5275	15.41
5	6031.5	3009.604	7.5193	6.46
6	6037.0	3007.399	7.5138	13.39
7	6149.3	2962.304	7.4011	12.61
8	6151.0	2961.650	7.3995	14.20
9	6169.6	2954.147	7.3808	24.93
10	6205.7	2939.655	7.3445	16.46
11	6210.0	2937.925	7.3402	7.72
12	6223.8	2932.382	7.3264	25.52
13	6239.5	2926.103	7.3107	5.90
14	6243.4	2924.548	7.3068	12.73
15	6280.2	2909.748	7.2698	5.99
16	6294.8	2903.872	7.2551	8.20
17	6312.8	2896.677	7.2372	10.53
18	6423.3	2852.311	7.1263	15.65
19	6427.8	2850.492	7.1218	6.49
20	6445.0	2843.561	7.1045	29.12
21	6466.7	2834.849	7.0827	13.96
22	8800.9	1897.593	4.7410	6.99
23	8818.2	1890.631	4.7236	7.25
24	9651.8	1555.894	3.8873	5.33
25	9672.3	1547.662	3.8667	5.51
26	9677.1	1545.744	3.8619	7.23
27	9679.8	1544.670	3.8593	7.44
28	9684.3	1542.840	3.8547	5.94
29	9750.2	1516.374	3.7886	7.82
30	9767.2	1509.570	3.7716	7.90
31	9788.9	1500.828	3.7497	5.63
32	9795.1	1498.369	3.7436	6.02
33	9817.0	1489.579	3.7216	8.56
34	9823.0	1487.161	3.7156	9.27
35	10058.0	1392.774	3.4798	13.74
36	10075.6	1385.713	3.4621	13.45
37	10966.1	1028.142	2.5687	7.10
38	10974.2	1024.905	2.5607	7.18
39	11180.4	942.077	2.3537	8.82
40	11185.2	940.173	2.3490	5.70
41	11209.7	930.313	2.3243	6.90
42	12273.8	503.035	1.2568	46.52
43	12290.9	496.189	1.2397	46.24
44	12321.9	483.727	1.2086	5.44
45	12338.9	476.902	1.1915	5.27



S19

nmr400a h-1

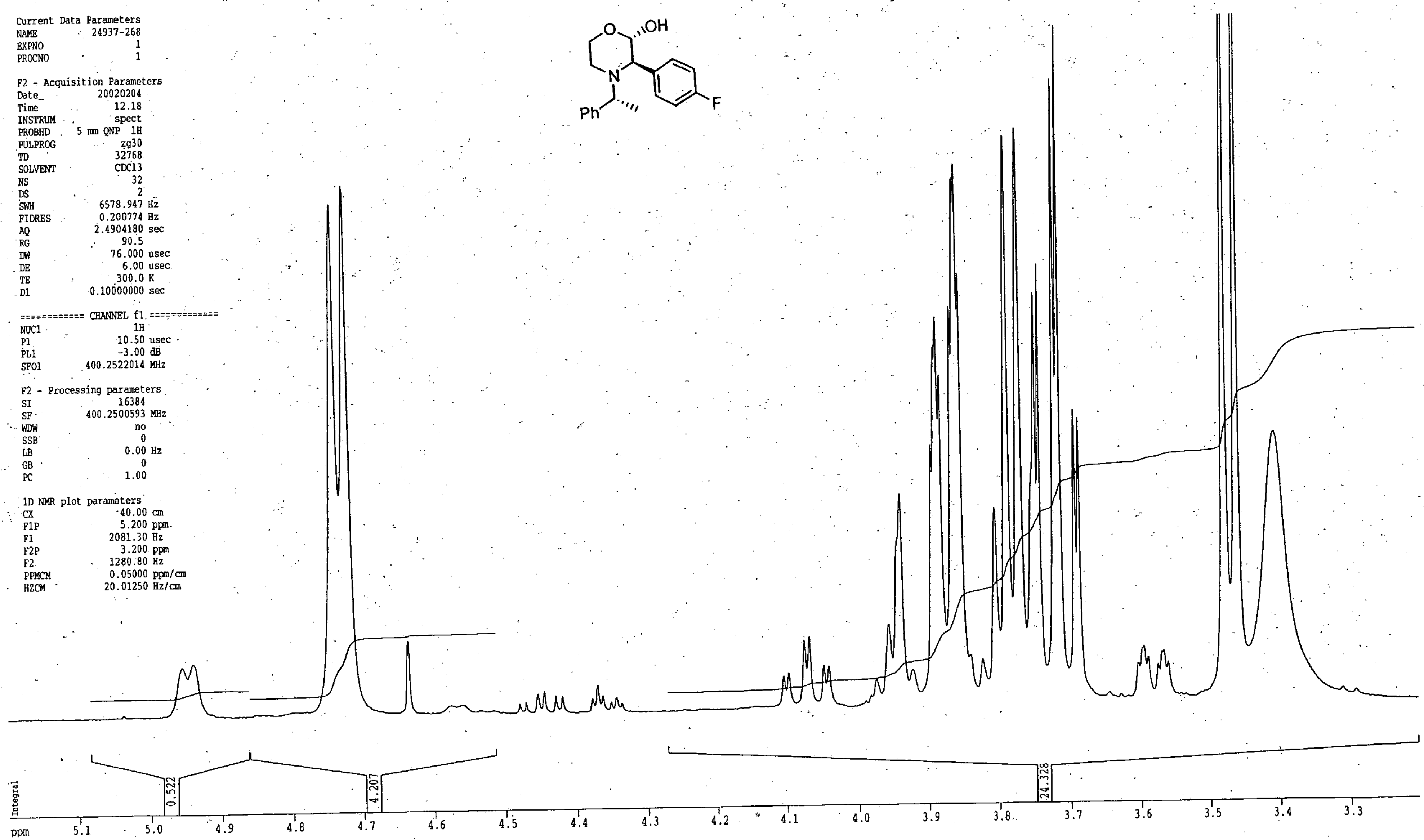
Current Data Parameters
NAME 24937-268
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020204
Time 12.18
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 90.5
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

============ CHANNEL f1 ============
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
F1P 5.200 ppm
F1 2081.30 Hz
F2P 3.200 ppm
F2 1280.80 Hz
PPMCM 0.05000 ppm/cm
HZCM 20.01250 Hz/cm



S20

nmr400a c-13

```
Current Data Parameters
NAME          24937-268
EXPNO                 2
PROCNO  lactol        1

F2 - Acquisition Parameters
Date_          20020204
Time              12.25
INSTRUM           spect
PROBHD     5 mm QNP 1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  346
DS                    0
SWH           26315.789 Hz
FIDRES         0.401547 Hz
AQ            1.2452340 sec
RG                 8192
DW               19.000 usec
DE                 6.00 usec
TE                300.0 K
D1           0.10000000 sec
d11          0.03000000 sec

============ CHANNEL f1 ============
NUC1                13C
P1                 2.00 usec
PL1                0.00 dB
SFO1        100.6539737 MHz

============ CHANNEL f2 ============
CPDPRG2         waltz16
NUC2                 1H
PCPD2             93.00 usec
PL2              120.00 dB
PL12              18.00 dB
SFO2        400.2520012 MHz

F2 - Processing parameters
SI                32768
SF          100.6429588 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                40.00 cm
F1P             240.000 ppm
F1             24154.32 Hz
F2P             -10.000 ppm
F2             -1006.42 Hz
PPMCM           6.25000 ppm/cm
HZCM          629.01849 Hz/cm
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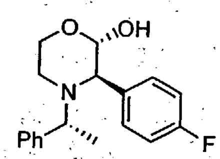
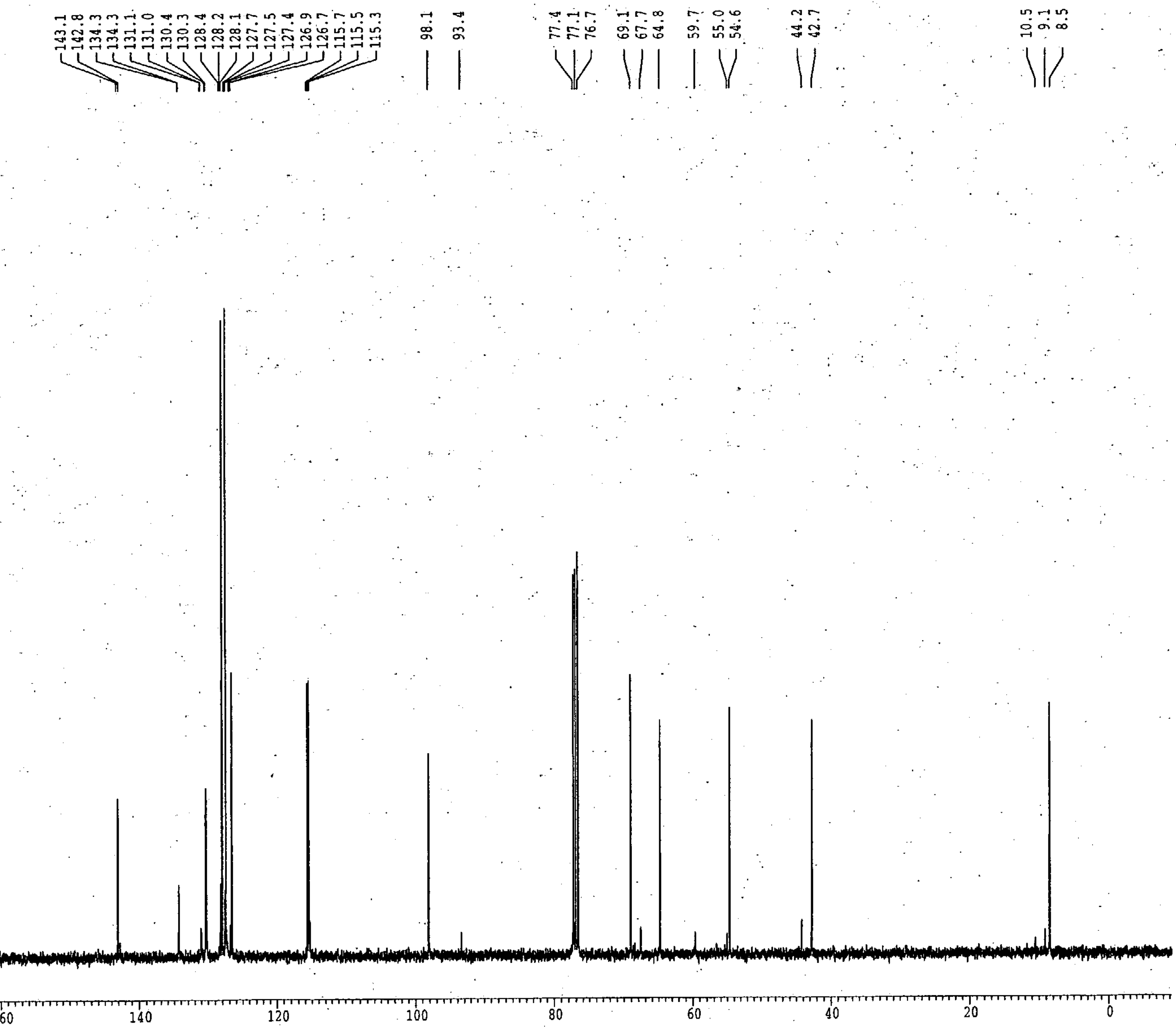
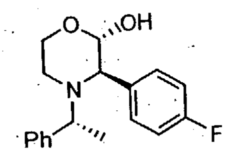
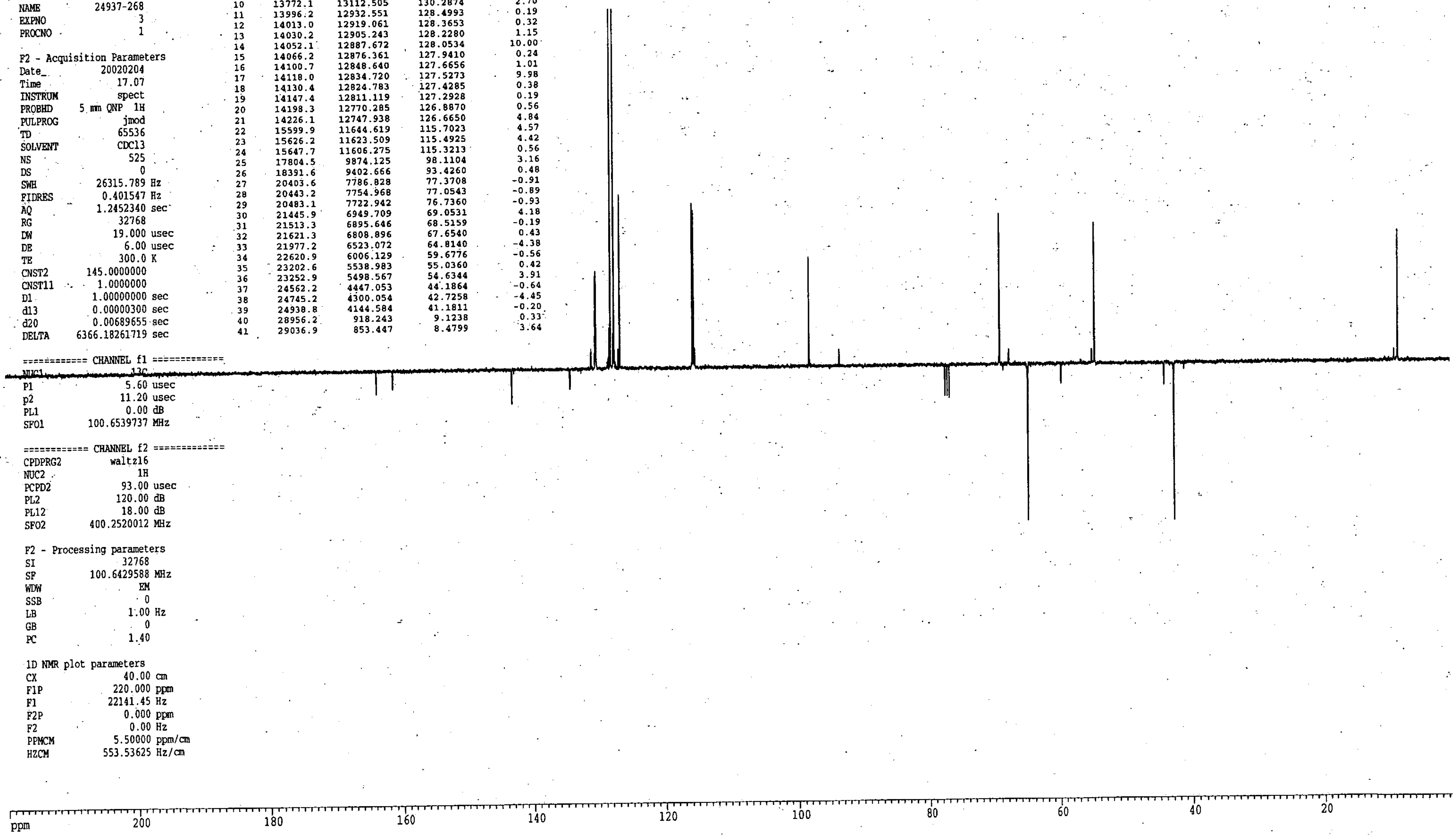




S21

nmr400a apt





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F1=220.000ppm, F2=0.000ppm, MI=-0.20cm, MAXI=10000.00cm, PC=1

#	ADDRESS	FREQUENCY [Hz]	FREQUENCY [PPM]	INTENSITY
1	9592.2	16469.377	163.6416	-0.66
2	9899.0	16222.981	161.1934	-0.51
3	12170.9	14398.405	143.0642	-0.97
4	13269.2	13516.371	134.3002	-0.57
5	13273.1	13513.263	134.2693	-0.57
6	13474.2	13351.790	132.6649	-0.13
7	13674.4	13191.014	131.0674	0.47
8	13684.2	13183.119	130.9890	0.55
9	13762.3	13120.410	130.3659	2.61
10	13772.1	13112.505	130.2874	2.70
11	13996.2	12932.551	128.4993	0.19
12	14013.0	12919.061	128.3653	0.32
13	14030.2	12905.243	128.2280	1.15
14	14052.1	12887.672	128.0534	10.00
15	14066.2	12876.361	127.9410	0.24
16	14100.7	12848.640	127.6656	1.01
17	14118.0	12834.720	127.5273	9.98
18	14130.4	12824.783	127.4285	0.38
19	14147.4	12811.119	127.2928	0.19
20	14198.3	12770.285	126.8870	0.56
21	14226.1	12747.938	126.6650	4.84
22	15599.9	11644.619	115.7023	4.57
23	15626.2	11623.509	115.4925	4.42
24	15647.7	11606.275	115.3213	0.56
25	17804.5	9874.125	98.1104	3.16
26	18391.6	9402.666	93.4260	0.48
27	20403.6	7786.828	77.3708	-0.91
28	20443.2	7754.968	77.0543	-0.89
29	20483.1	7722.942	76.7360	-0.93
30	21445.9	6949.709	69.0531	4.18
31	21513.3	6895.646	68.5159	-0.19
32	21621.3	6808.896	67.6540	0.43
33	21977.2	6523.072	64.8140	-4.38
34	22620.9	6006.129	59.6776	-0.56
35	23202.6	5538.983	55.0360	0.42
36	23252.9	5498.567	54.6344	3.91
37	24562.2	4447.053	44.1864	-0.64
38	24745.2	4300.054	42.7258	-4.45
39	24938.8	4144.584	41.1811	-0.20
40	28956.2	918.243	9.1238	0.33
41	29036.9	853.447	8.4799	3.64

Current Data Parameters
NAME 24937-268
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020204
Time 17.07
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 525
DS 0
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 32768
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 1.00000000 sec
d13 0.00000300 sec
d20 0.00689655 sec
DELTA 6366.18261719 sec

============ CHANNEL f1 ============
NUC1 13C
P1 5.60 usec
p2 11.20 usec
PL1 0.00 dB
SFO1 100.6539737 MHz

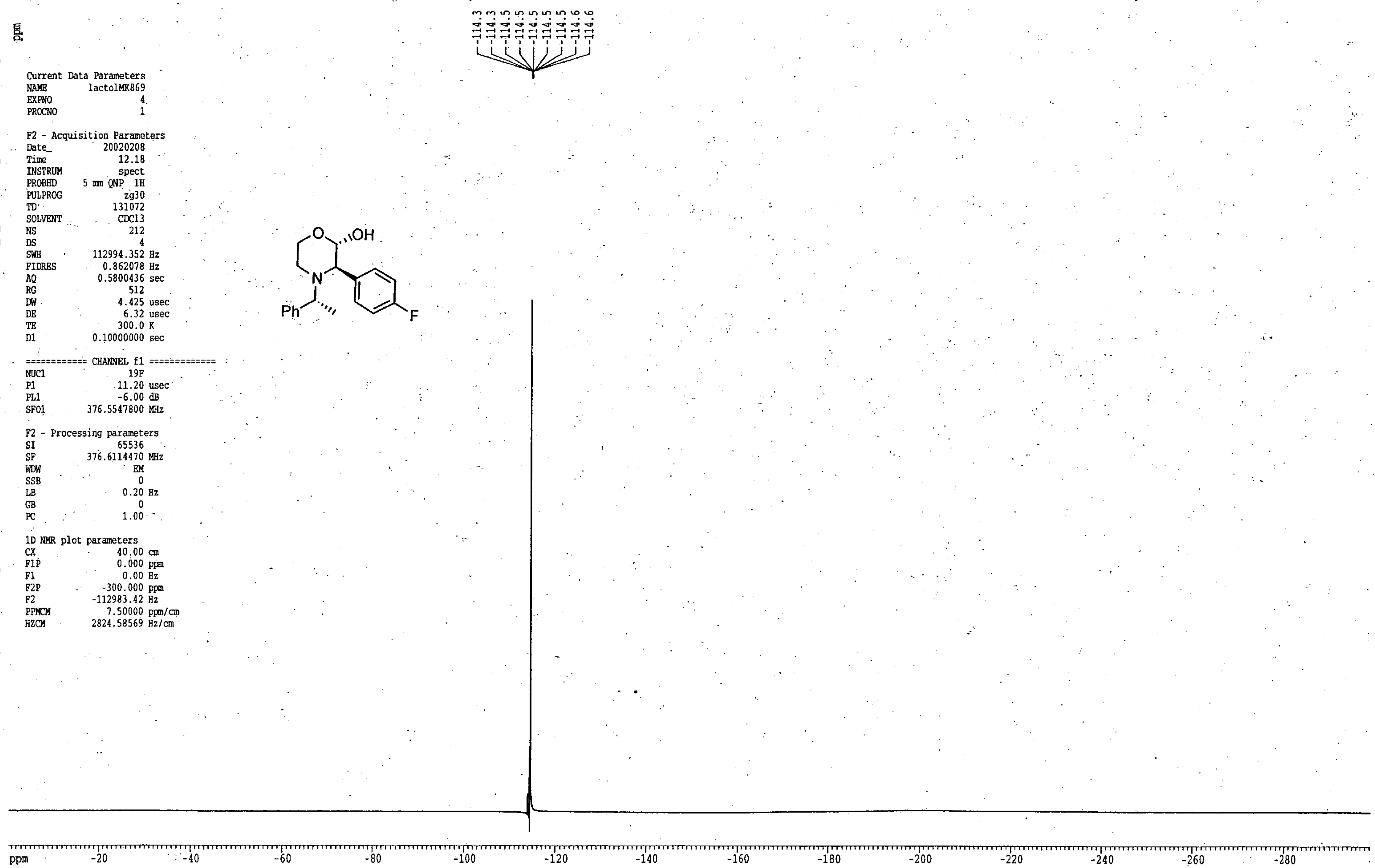
============ CHANNEL f2 ============
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz

F2 - Processing parameters
SI 32768
SF 100.6429588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

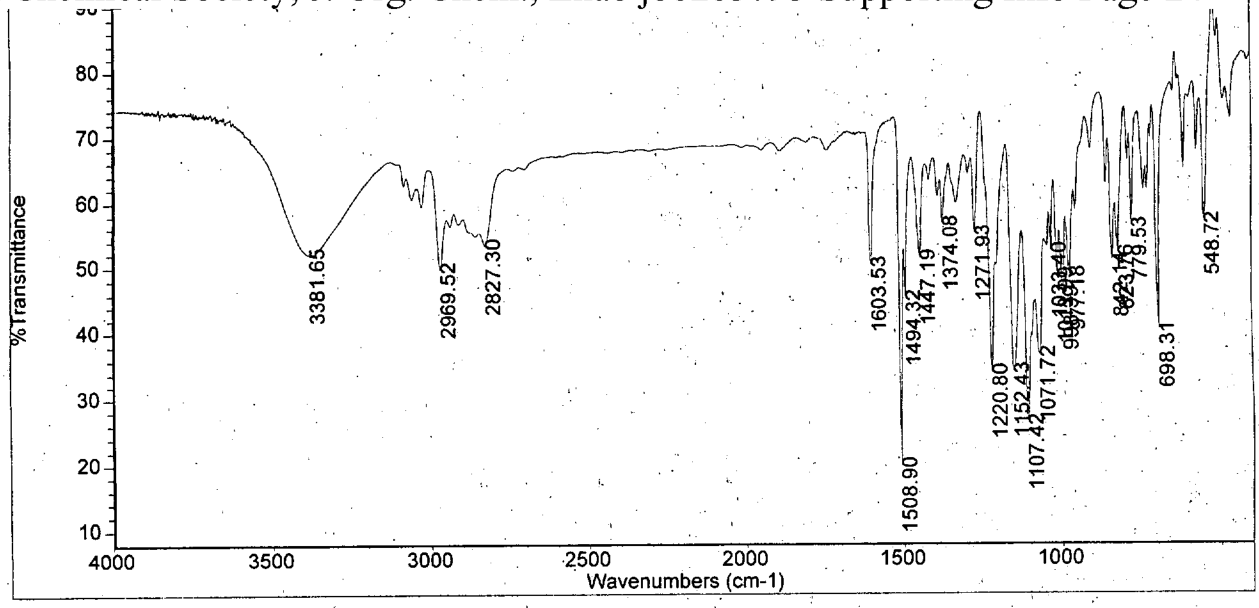
1D NMR plot parameters
CX 40.00 cm
F1P 220.000 ppm
F1 22141.45 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 5.50000 ppm/cm
HZCM 553.53625 Hz/cm

S22

nmr400a f-19

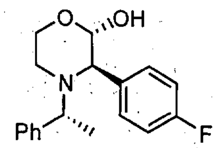


S23



Mon Feb 04 15:45:29 2002
FIND PEAKS:

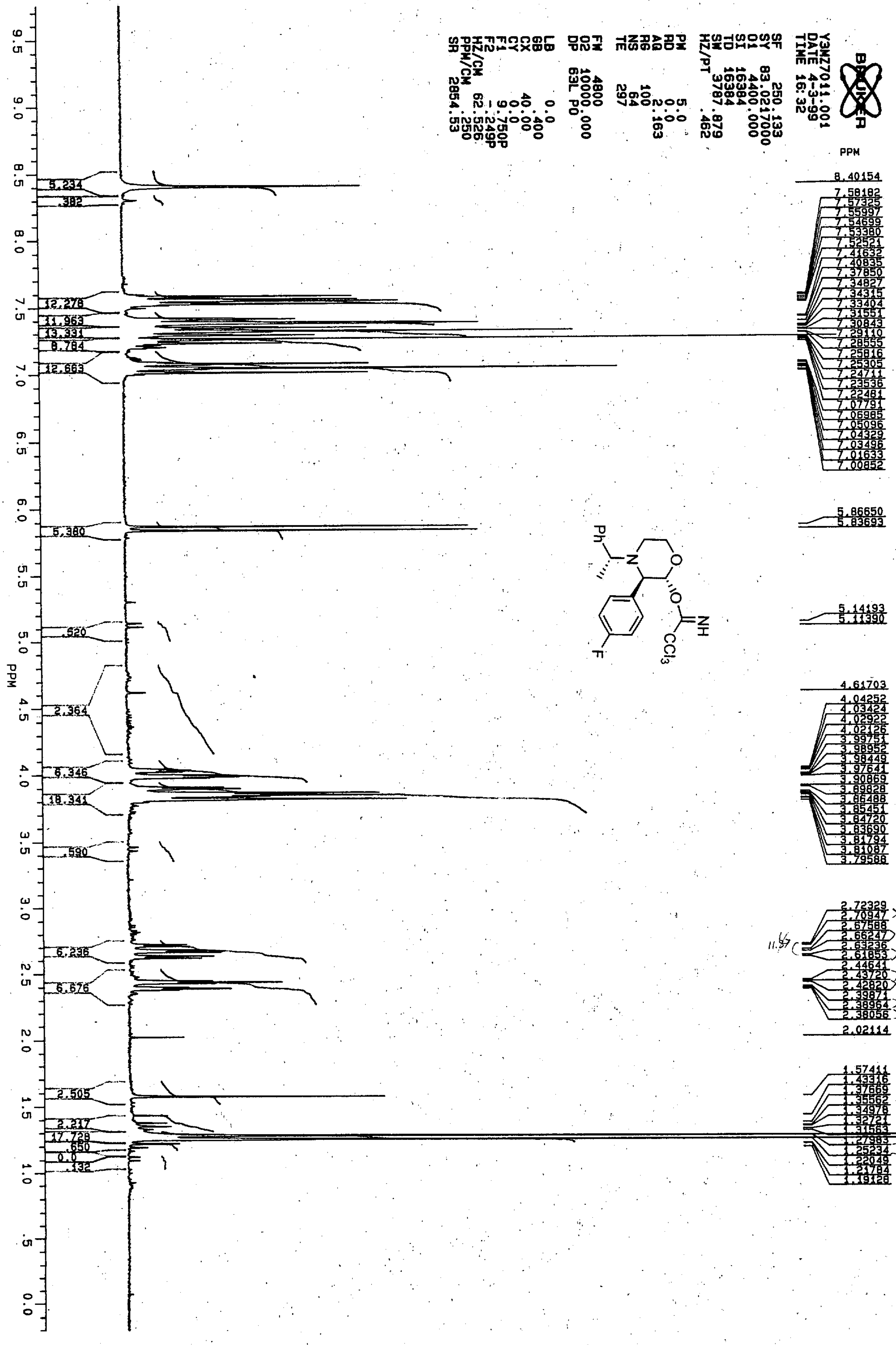
Spectrum: 24937-268 lactol (MK-869)
Region: 4000.00 400.00
Absolute threshold: 57.712
Sensitivity: 50
Peak list:



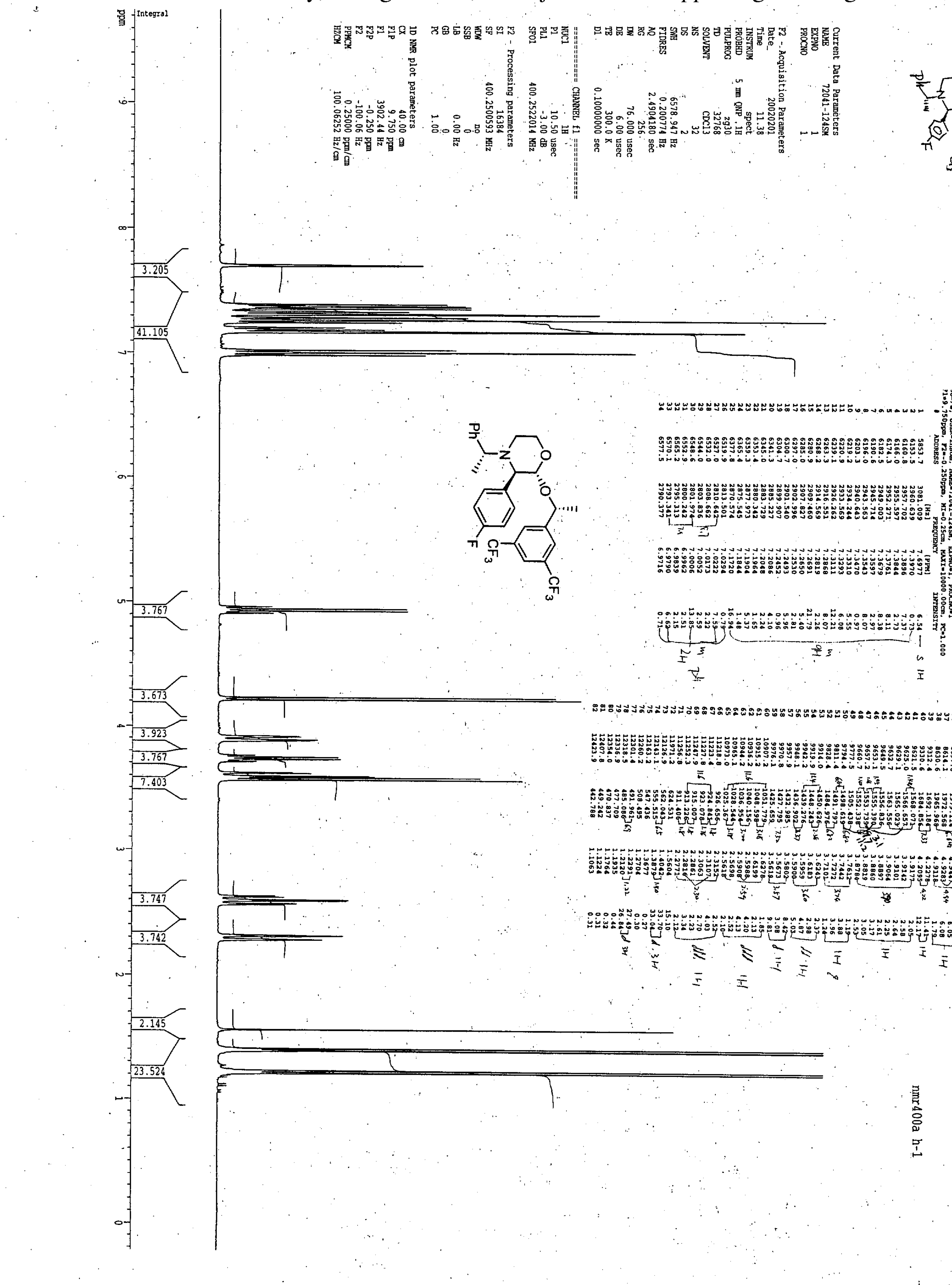
Position:	1508.90	Intensity:	19.871
Position:	1107.42	Intensity:	26.220
Position:	1220.80	Intensity:	34.033
Position:	1152.43	Intensity:	34.169
Position:	1071.72	Intensity:	36.543
Position:	698.31	Intensity:	40.268
Position:	1494.32	Intensity:	45.229
Position:	998.39	Intensity:	45.798

Position:	1013.99	Intensity:	48.347
Position:	977.18	Intensity:	48.789
Position:	2969.52	Intensity:	49.660
Position:	1603.53	Intensity:	50.209
Position:	842.14	Intensity:	50.757
Position:	1447.19	Intensity:	51.242
Position:	3381.65	Intensity:	51.950
Position:	823.76	Intensity:	51.958
Position:	1033.40	Intensity:	52.215
Position:	2827.30	Intensity:	53.208
Position:	1271.93	Intensity:	54.986
Position:	779.53	Intensity:	56.341
Position:	1374.08	Intensity:	56.512
Position:	548.72	Intensity:	57.052

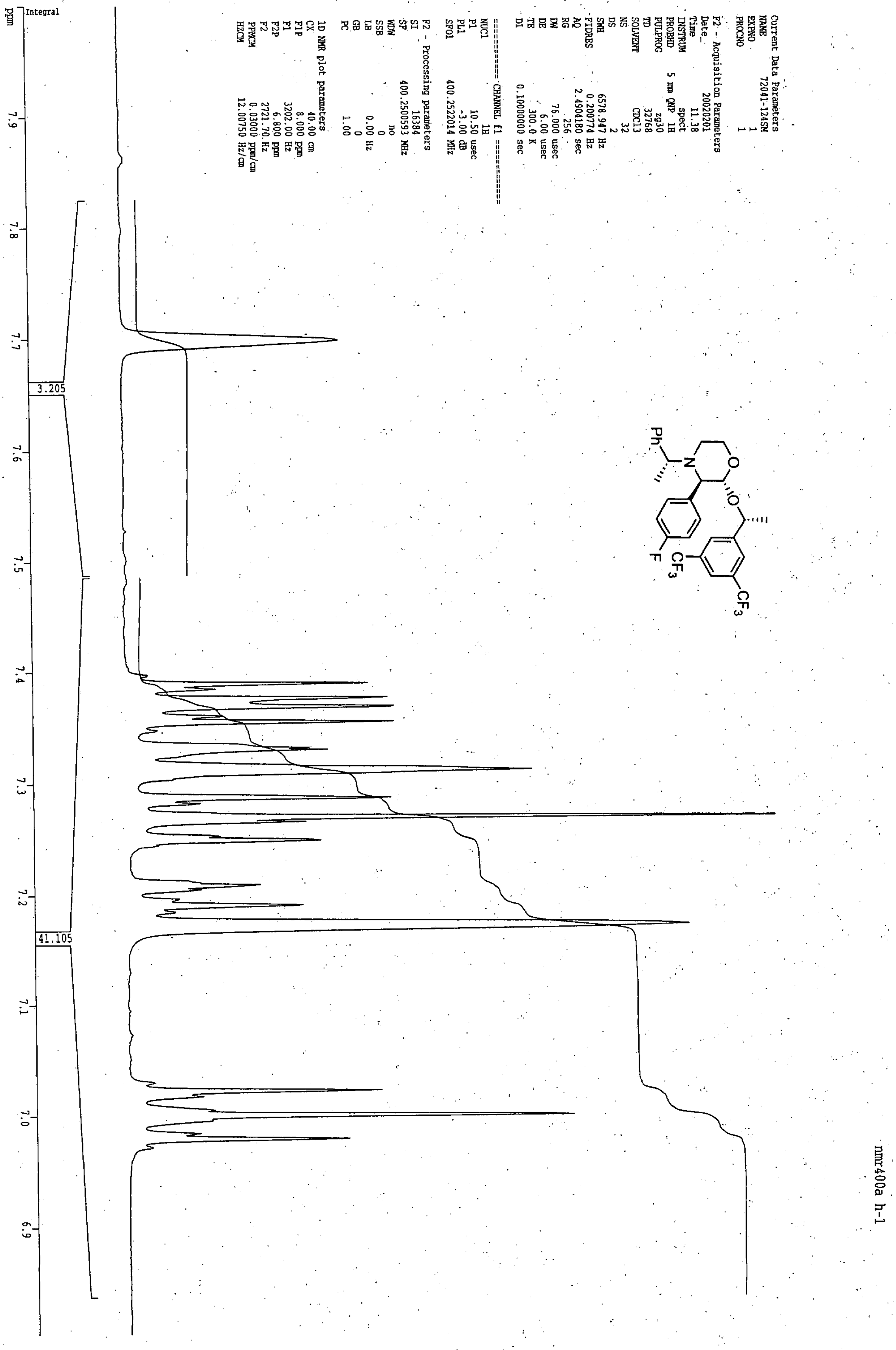
S24



S25


Current Data Parameters
NAME 72041-124SM
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020201
Time 11.38
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 256
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
============ CHANNEL f1 ============
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz
F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 40.00 cm
F1P 9.750 ppm
F1 3902.44 Hz
F2P -0.250 ppm
F2 -100.06 Hz
PPMCM 0.25000 ppm/cm
HZCM 100.06252 Hz/cm
Integral
ppm
3.205
41.105
3.767
3.673
3.923
3.767
7.403
3.747
3.742
2.145
23.524
CF3
CF3
Ph
F
nmr400a h-1

S26


Current Data Parameters
NAME 72041-124SM
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020201
Time 11.38
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 256
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz
F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 40.00 cm
F1P 8.000 ppm
F1 3202.00 Hz
F2P 6.800 ppm
F2 2721.70 Hz
PPMCM 0.03000 ppm/cm
HZCM 12.00750 Hz/cm
Ph
N
O
O
CF_3
CF_3
F
Integral
3.205
41.105
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
ppm
nmr400a h-1

S27

nmr400a h-1

```
Current Data Parameters
NAME        72041-124SM
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20020201
Time              11.38
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    2
SWH           6578.947 Hz
FIDRES        0.200774 Hz
AQ           2.4904180 sec
RG                  256
DW               76.000 usec
DE                 6.00 usec
TE                300.0 K
D1          0.10000000 sec

============ CHANNEL f1 ============
NUC1                 1H
P1                10.50 usec
PL1               -3.00 dB
SFO1        400.2522014 MHz

F2 - Processing parameters
SI                16384
SF          400.2500593 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                40.00 cm
F1P               5.200 ppm
F1              2081.30 Hz
F2P               3.400 ppm
F2              1360.85 Hz
PPMCM           0.04500 ppm/cm
HZCM           18.01125 Hz/cm
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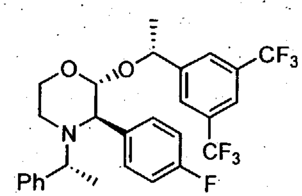
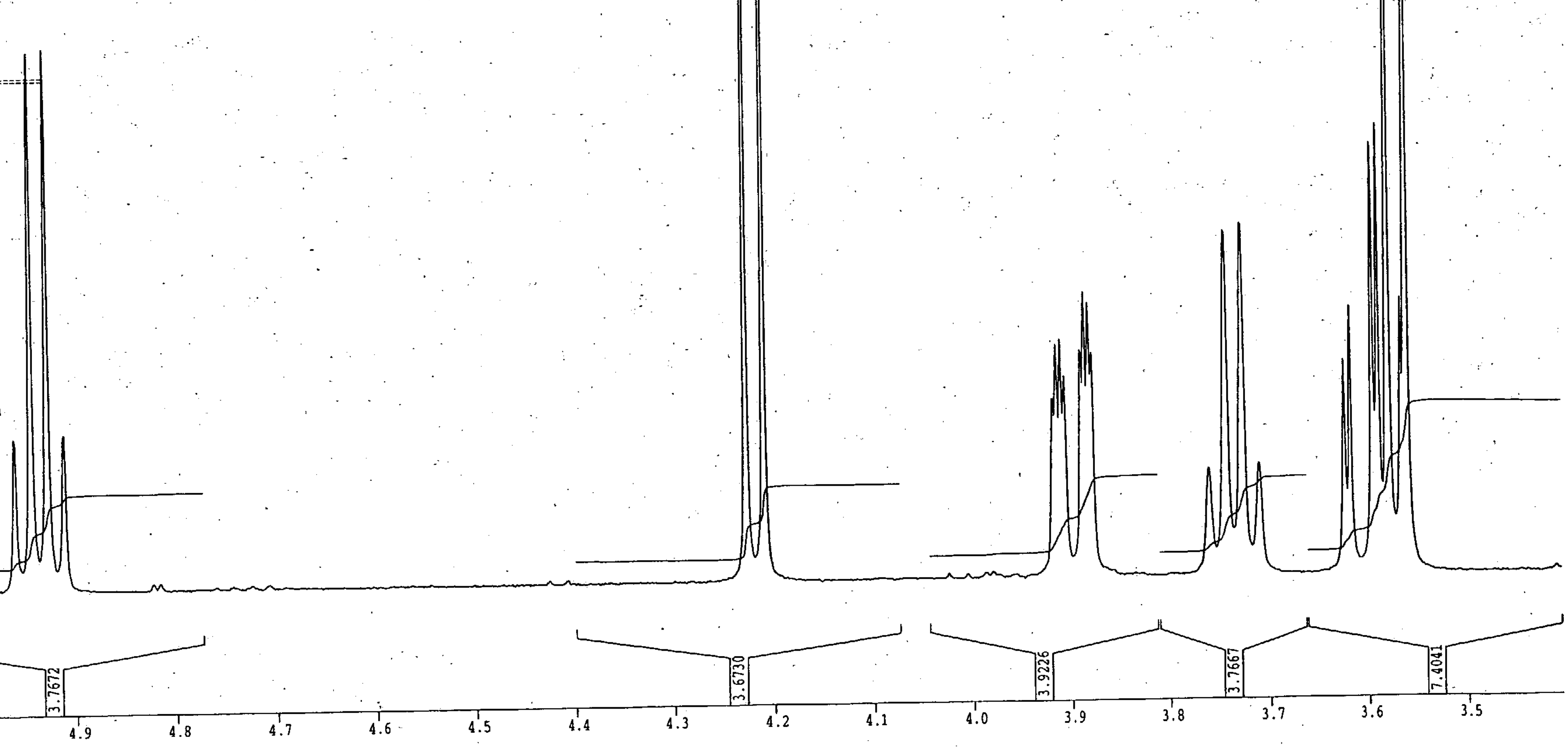
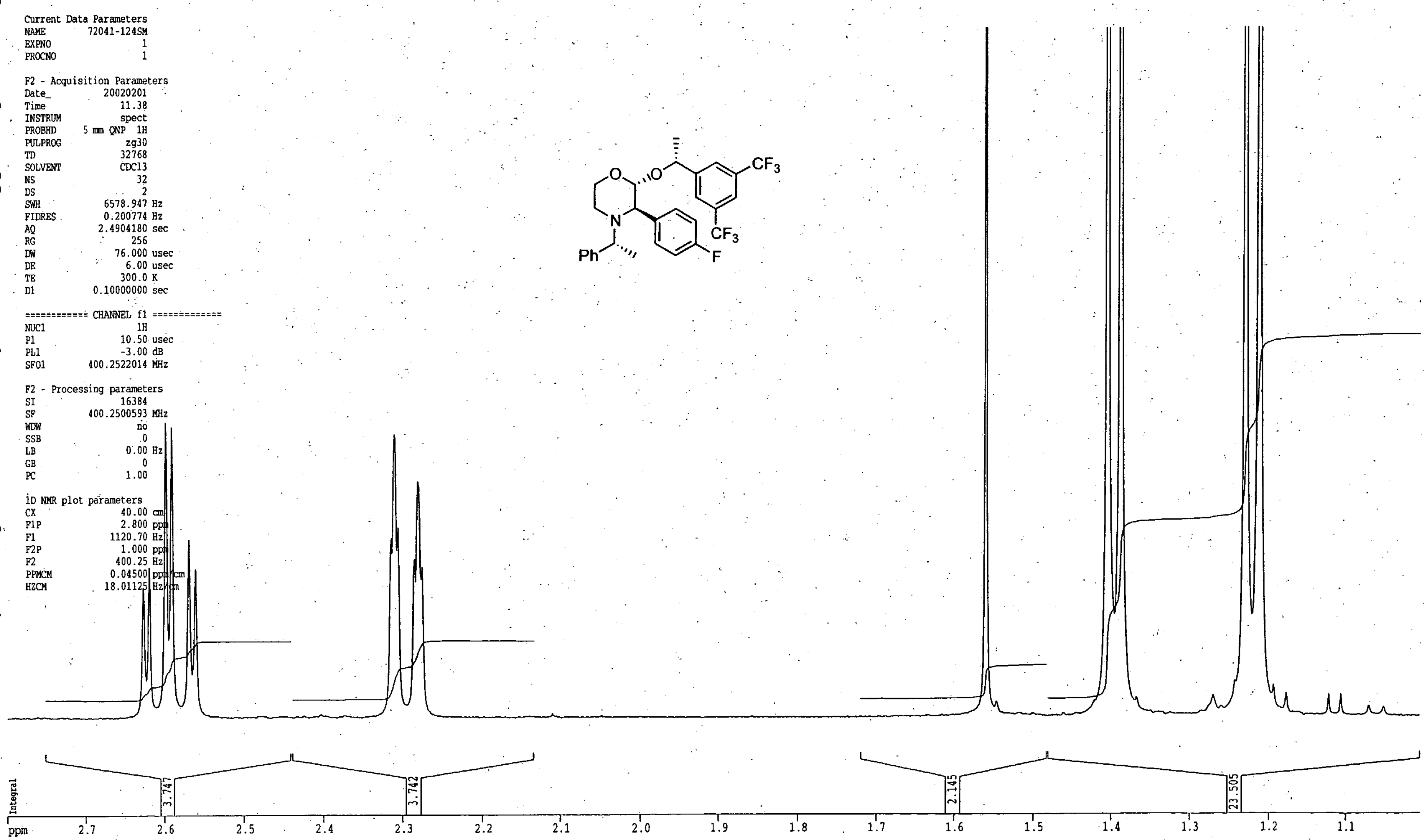




S28

nmr400a h-1



S29

nmr400a c-13

```
Current Data Parameters
NAME        72041-124SM
EXPNO                 2
PROCNO                1

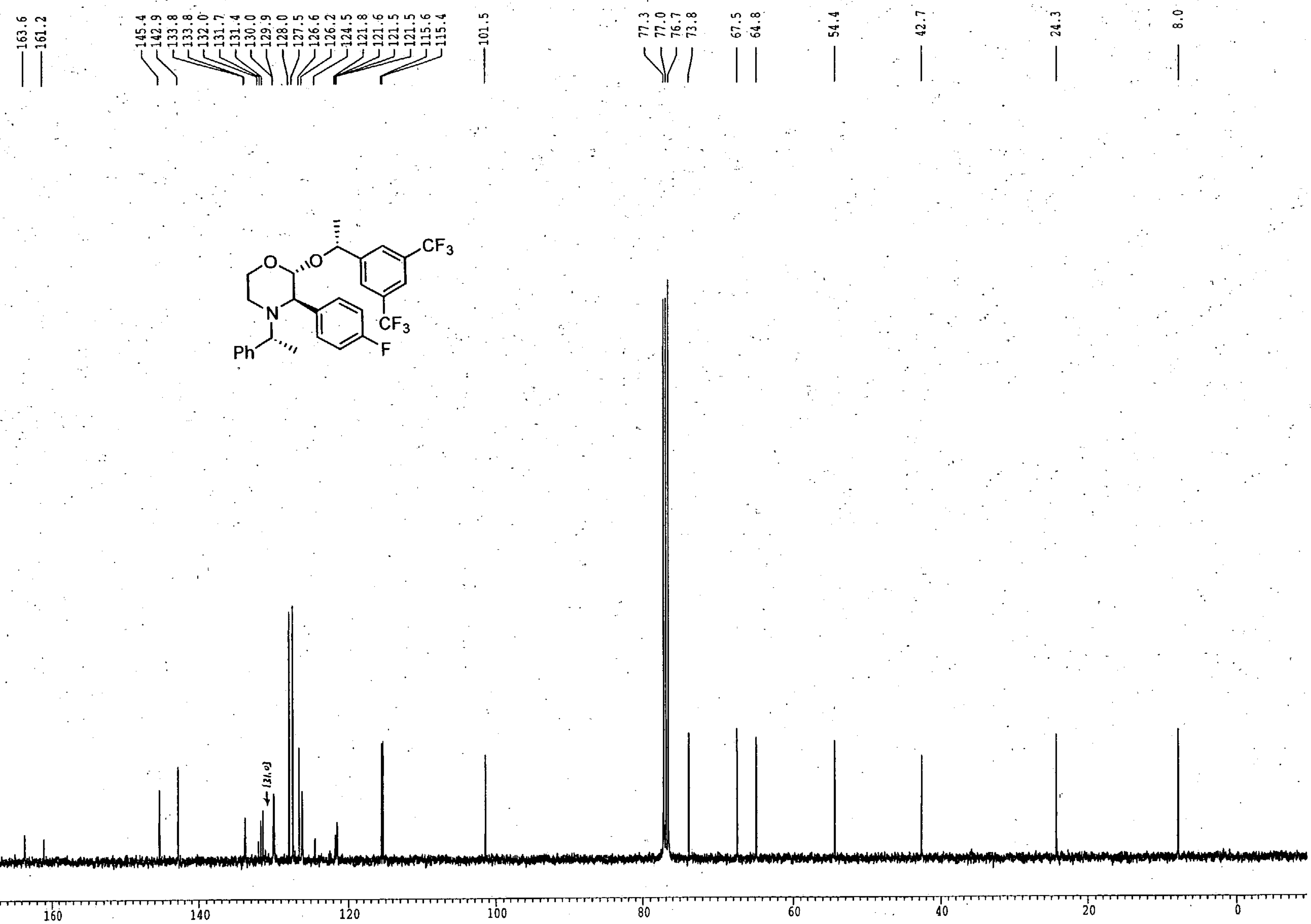
F2 - Acquisition Parameters
Date_          20020201
Time              12.05
INSTRUM           spect
PROBHD     5 mm QNP 1H
PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                 1102
DS                    0
SWH           26315.789 Hz
FIDRES         0.401547 Hz
AQ            1.2452340 sec
RG                 8192
DW               19.000 usec
DE                 6.00 usec
TE                300.0 K
D1           0.10000000 sec
d11          0.03000000 sec

============ CHANNEL f1 ============
NUC1                13C
P1                 2.00 usec
PL1                0.00 dB
SFO1        100.6539737 MHz

============ CHANNEL f2 ============
CPDPRG2         waltz16
NUC2                 1H
PCPD2             93.00 usec
PL2              120.00 dB
PL12              18.00 dB
SFO2        400.2520012 MHz

F2 - Processing parameters
SI                32768
SF          100.6429588 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                40.00 cm
F1P             200.000 ppm
F1             20128.59 Hz
F2P             -10.000 ppm
F2             -1006.43 Hz
PPMCM           5.25000 ppm/cm
HZCM          528.37549 Hz/cm
```



nmr400a apt

/w/date/zhaom/nmr/t-glycosylatio/3/pdata/1/screen

DU=/w, USER=zhaom, NAME=t-glycosylatio, EXPNO=3, PROCNO=1
F1=240.000ppm, F2=-10.000ppm, MI=-0.10cm, MAXI=10000.00cm,

#	ADDRESS	FREQUENCY [Hz]	[PPM]	INTENSITY
1	9589.5	16471.561	163.6633	-0.60
2	9896.9	16224.678	161.2103	-0.55
3	11870.4	14639.805	145.4628	-1.27
4	12184.0	14387.883	142.9597	-1.40
5	13324.0	13472.423	133.8635	-0.77
6	13327.9	13469.273	133.8322	-0.81
7	13551.2	13289.957	132.0505	-0.23
8	13592.7	13256.562	131.7187	-0.61
9	13634.0	13223.404	131.3893	-0.73
10	13675.3	13190.228	131.0596	-0.23
11	13808.7	13083.144	129.9956	2.13
12	13818.4	13075.308	129.9178	2.11
13	14044.3	12893.940	128.1157	0.14
14	14062.8	12879.074	127.9680	9.60
15	14120.9	12832.401	127.5042	10.00
16	14156.9	12803.500	127.2170	-0.17
17	14235.2	12740.606	126.5921	4.74
18	14282.4	12702.718	126.2157	2.48
19	14286.0	12699.794	126.1866	2.51
20	14496.5	12530.755	124.5070	-0.31
21	14836.1	12258.042	121.7973	-0.32
22	14860.9	12238.089	121.5991	0.45
23	14865.4	12234.514	121.5635	1.01
24	14870.1	12230.738	121.5260	1.37
25	14874.9	12226.911	121.4880	1.08
26	14879.5	12223.178	121.4509	0.47
27	15175.9	11985.114	119.0855	-0.15
28	15609.8	11636.652	115.6231	4.03
29	15636.4	11615.306	115.4110	3.83
30	17371.1	10222.159	101.5686	3.80
31	20408.5	7782.869	77.3315	-0.53
32	20448.3	7750.914	77.0140	-0.50
33	20488.1	7718.958	76.6964	-0.59
34	20841.9	7434.793	73.8730	4.25
35	21636.4	6796.777	67.5336	4.55
36	21973.2	6526.294	64.8460	-4.27
37	23276.1	5479.955	54.4495	4.38
38	24745.6	4299.810	42.7234	-3.92
39	27051.0	2448.326	24.3268	4.59
40	29102.4	800.885	7.9577	3.70

Current Data Parameters
NAME t-glycosylatio
EXPNO 3
PROCNO 1

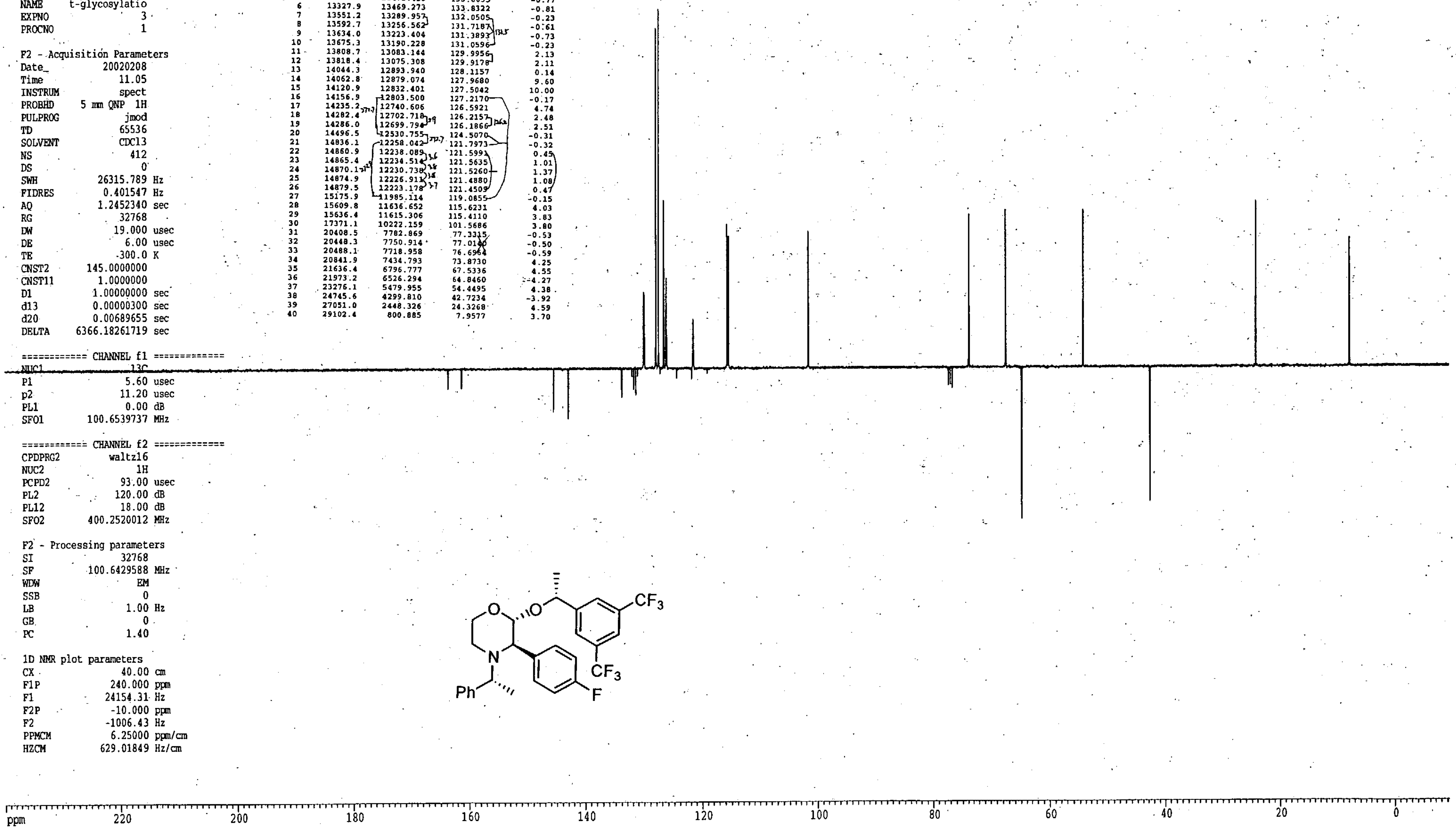
F2 - Acquisition Parameters
Date_ 20020208
Time 11.05
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 412
DS 0
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 32768
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 1.00000000 sec
d13 0.00000300 sec
d20 0.00689655 sec
DELTA 6366.18261719 sec

============ CHANNEL f1 ============
NUC1 13C
P1 5.60 usec
p2 11.20 usec
PL1 0.00 dB
SFO1 100.6539737 MHz

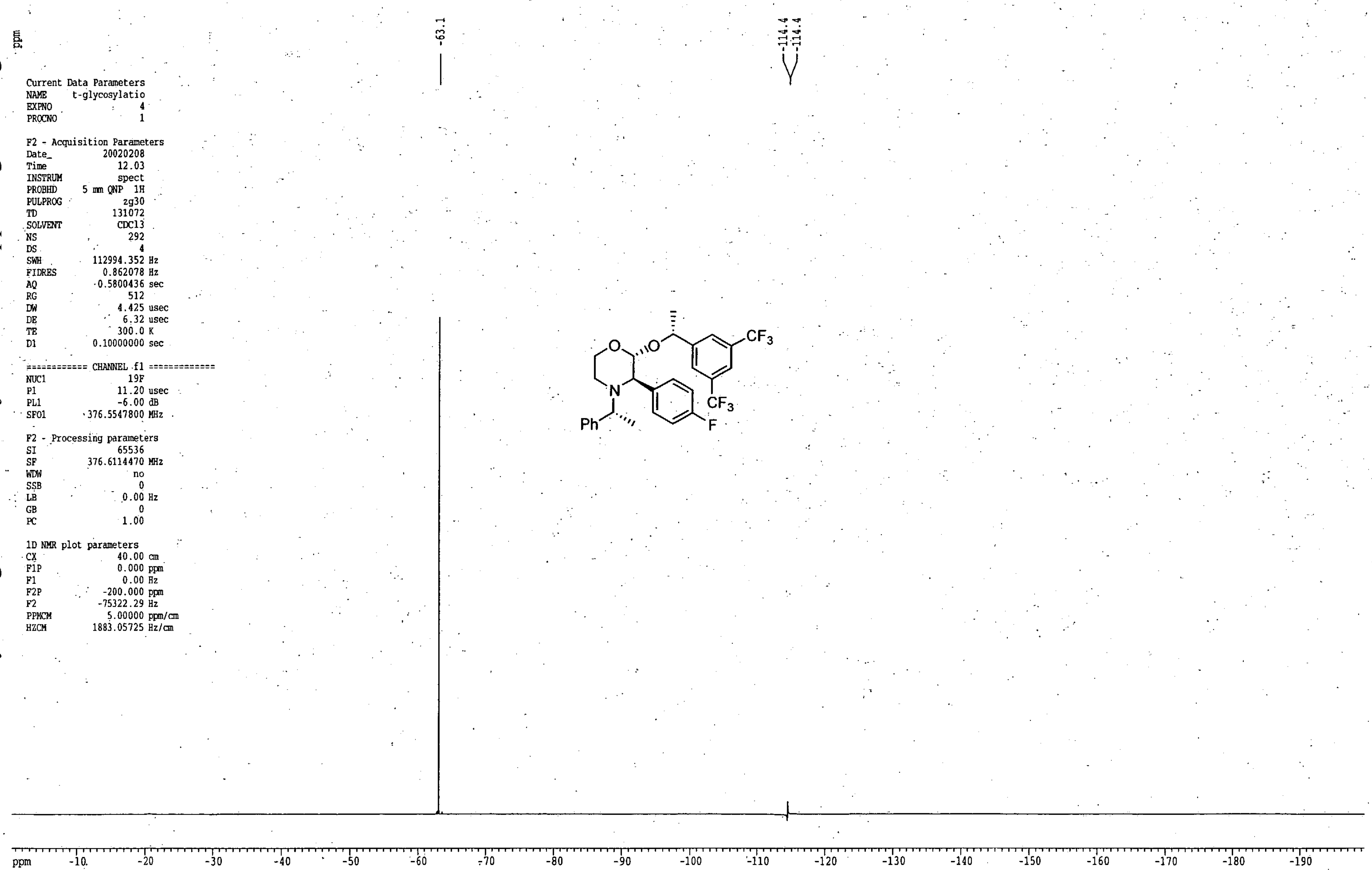
============ CHANNEL f2 ============
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz

F2 - Processing parameters
SI 32768
SF 100.6429588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 240.000 ppm
F1 24154.31 Hz
F2P -10.000 ppm
F2 -1006.43 Hz
PPMCM 6.25000 ppm/cm
HZCM 629.01849 Hz/cm



S31



Current Data Parameters
NAME t-glycosylatio
EXPNO 4
PROCNO 1

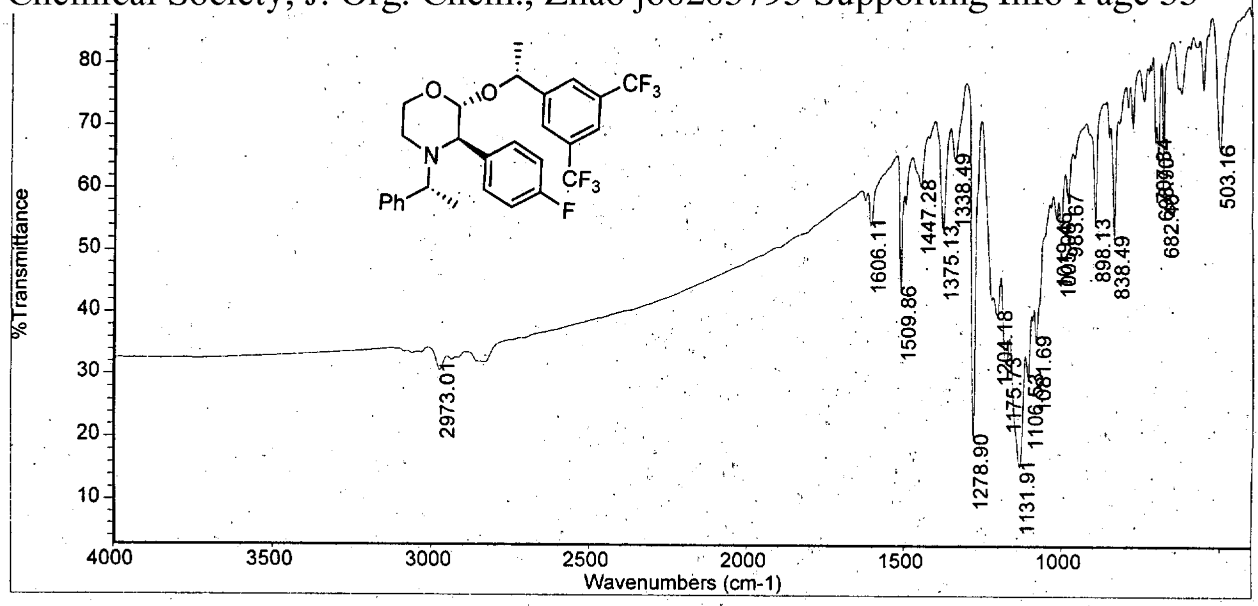
F2 - Acquisition Parameters
Date_ 20020208
Time 12.03
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 292
DS 4
SWH 112994.352 Hz
FIDRES 0.862078 Hz
AQ 0.5800436 sec
RG 512
DW 4.425 usec
DE 6.32 usec
TE 300.0 K
D1 0.10000000 sec

============ CHANNEL f1 ============
NUC1 19F
P1 11.20 usec
PL1 -6.00 dB
SFO1 376.5547800 MHz

F2 - Processing parameters
SI 65536
SF 376.6114470 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
F1P 0.000 ppm
F1 0.00 Hz
F2P -200.000 ppm
F2 -75322.29 Hz
PPMCM 5.00000 ppm/cm
HZCM 1883.05725 Hz/cm

S32



Mon Feb 04 15:12:35 2002
FIND PEAKS:
Spectrum: 72041-124SM' t-glycosylation
Region: 4000.00 400.00
Absolute threshold: 68.052
Sensitivity: 50
Peak list:

Position:	1131.91	Intensity:	15.332
Position:	1278.90	Intensity:	19.450
Position:	1106.53	Intensity:	29.081
Position:	2973.01	Intensity:	30.528
Position:	1175.73	Intensity:	31.678
Position:	1081.69	Intensity:	35.206
Position:	1204.18	Intensity:	39.222
Position:	1509.86	Intensity:	43.105

Position:	838.49	Intensity:	51.487
Position:	1375.13	Intensity:	52.797
Position:	1005.96	Intensity:	53.344
Position:	1606.11	Intensity:	53.885
Position:	898.13	Intensity:	54.195
Position:	1019.46	Intensity:	54.920
Position:	983.67	Intensity:	58.268
Position:	682.46	Intensity:	58.208
Position:	1447.28	Intensity:	60.205
Position:	1338.49	Intensity:	64.438
Position:	698.90	Intensity:	64.122
Position:	503.16	Intensity:	66.077
Position:	707.34	Intensity:	67.077

S33

Current Data Parameters
NAME 72041-124.HCl
EXPNO *t-Sec-amine* 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020202
Time 17.25
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 114
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

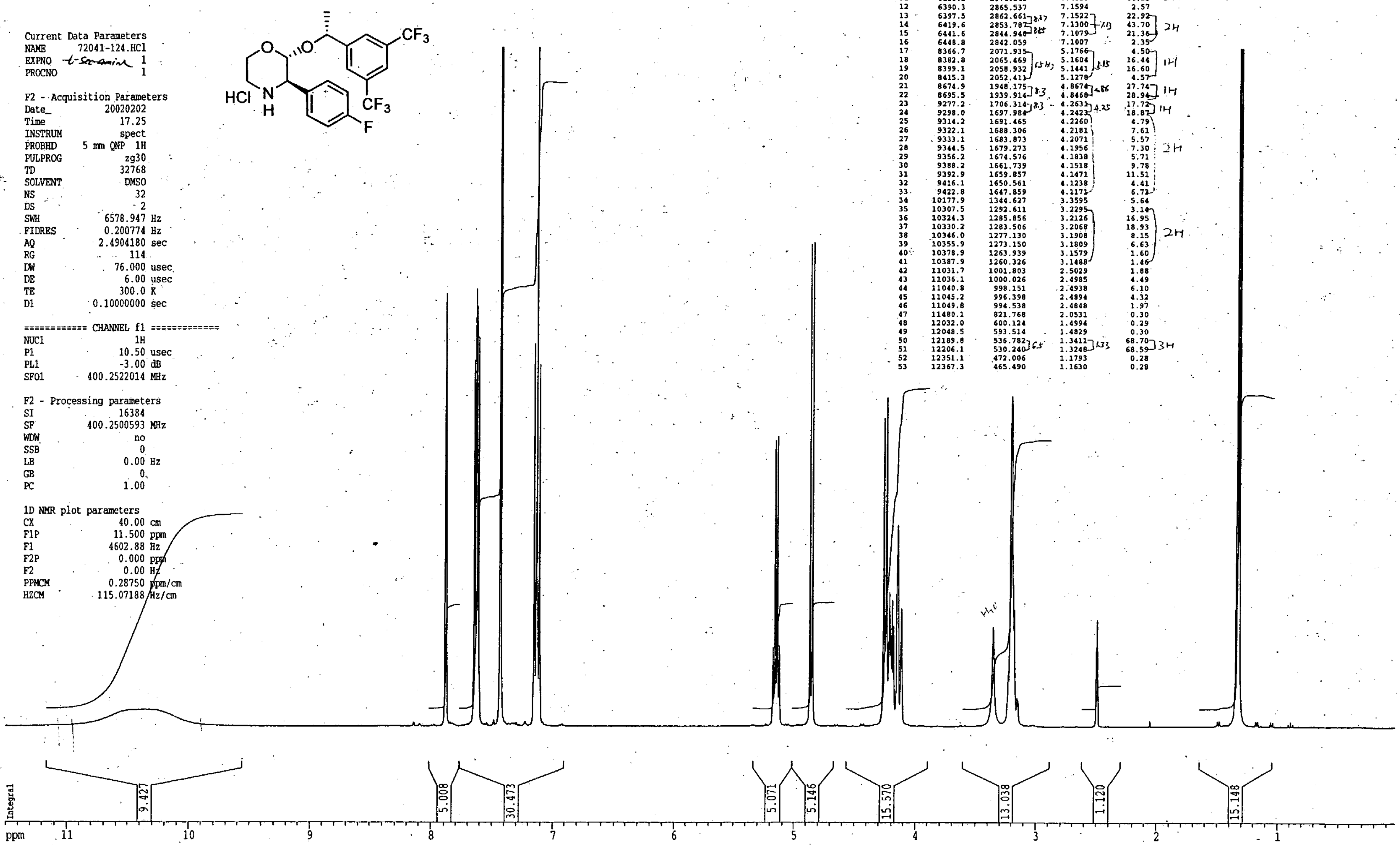
============ CHANNEL f1 ============
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

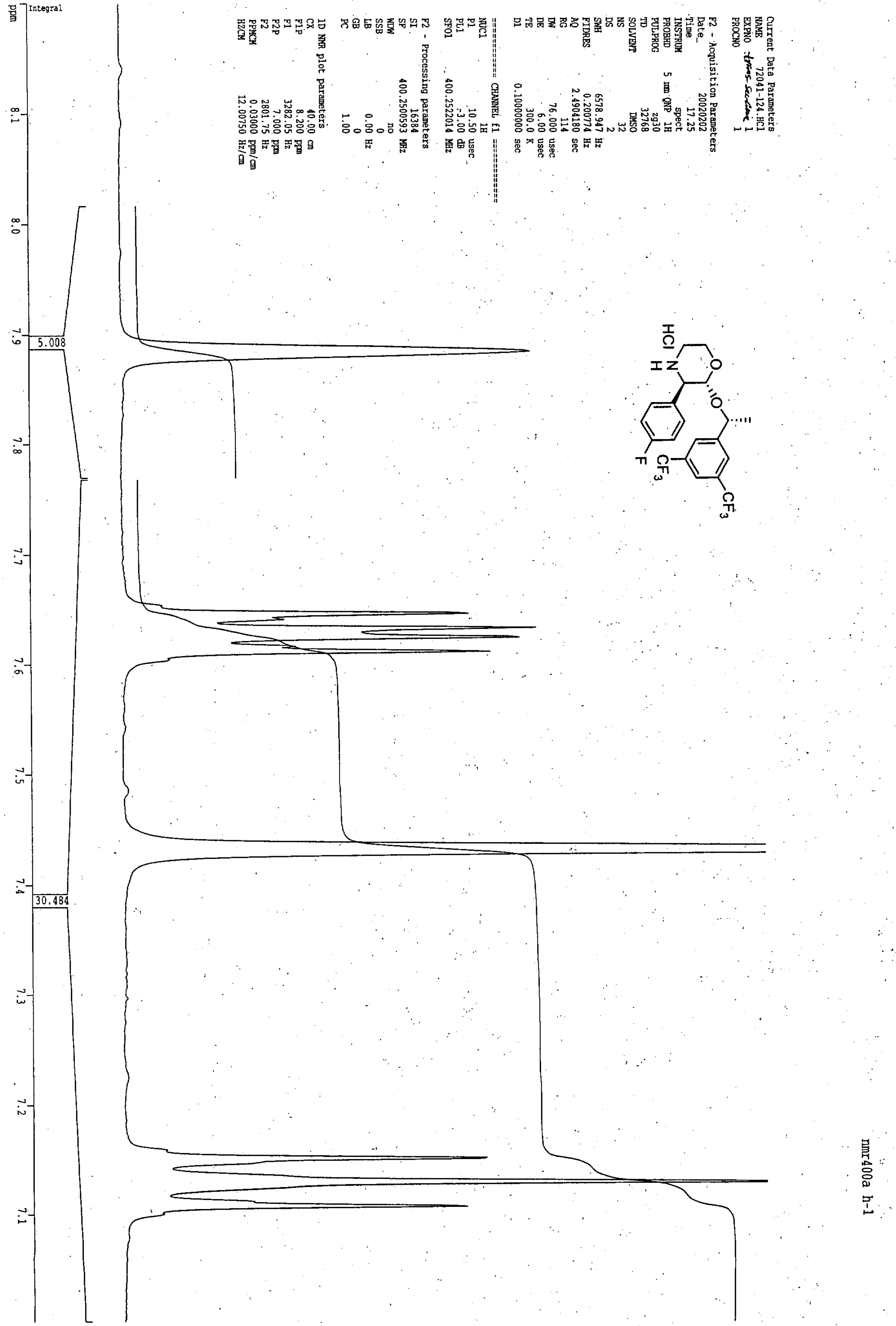
1D NMR plot parameters
CX 40.00 cm
F1P 11.500 ppm
F1 4602.88 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28750 ppm/cm
HZCM 115.07188 Hz/cm

		[Hz]	[PPM]		
1	3177.0	4155.834	10.3831	0.90	11–10
2	5667.7	3155.710	7.8843	24.88	
3	5904.7	3060.553	7.6466	21.34	
4	5909.7	3058.521	7.6415	9.85	
5	5918.0	3055.178	7.6332	25.06	2H
6	5926.6	3051.754	7.6246	24.76	
7	5934.8	3048.431	7.6163	10.49	
8	5939.8	3046.433	7.6113	22.41	
9	5947.0	3043.567	7.6042	2.83	
10	6065.7	2995.884	7.4850	0.32	
11	6118.1	2974.845	7.4325	56.23	2H
12	6390.3	2865.537	7.1594	2.57	
13	6397.5	2862.661	7.1522	22.92	
14	6419.6	2853.787	7.1300	43.70	2H
15	6441.6	2844.940	7.1079	21.36	
16	6448.8	2842.059	7.1007	2.35	
17	8366.7	2071.935	5.1766	4.50	
18	8382.8	2065.469	5.1604	16.44	1H
19	8399.1	2058.932	5.1441	16.60	
20	8415.3	2052.411	5.1278	4.57	
21	8674.9	1948.175	4.8674	27.74	1H
22	8695.5	1939.914	4.8468	28.94	
23	9277.2	1706.314	4.2631	17.72	1H
24	9298.0	1697.984	4.2423	18.87	
25	9314.2	1691.465	4.2260	4.79	
26	9322.1	1688.306	4.2181	7.61	
27	9333.1	1683.873	4.2071	5.57	
28	9344.5	1679.273	4.1956	7.30	2H
29	9356.2	1674.576	4.1838	5.71	
30	9388.2	1661.739	4.1518	9.78	
31	9392.9	1659.857	4.1471	11.51	
32	9416.1	1650.561	4.1238	4.41	
33	9422.8	1647.859	4.1171	6.73	
34	10177.9	1344.627	3.3595	5.64	
35	10307.5	1292.611	3.2295	3.14	
36	10324.3	1285.856	3.2126	16.95	
37	10330.2	1283.506	3.2068	18.93	
38	10346.0	1277.130	3.1908	8.15	2H
39	10355.9	1273.150	3.1809	6.63	
40	10378.9	1263.939	3.1579	1.60	
41	10387.9	1260.326	3.1488	1.46	
42	11031.7	1001.803	2.5029	1.88	
43	11036.1	1000.026	2.4985	4.49	
44	11040.8	998.151	2.4938	6.10	
45	11045.2	996.398	2.4894	4.32	
46	11049.8	994.538	2.4848	1.97	
47	11480.1	821.768	2.0531	0.30	
48	12032.0	600.124	1.4994	0.29	
49	12048.5	593.514	1.4829	0.30	
50	12189.8	536.782	1.3411	68.70	3H
51	12206.1	530.240	1.3248	68.59	
52	12351.1	472.006	1.1793	0.28	
53	12367.3	465.490	1.1630	0.28	

nmr400a h-1



S34



Current Data Parameters
NAME 72041-124.HCl
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020202
Time 17.25
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 114
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz
F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 40.00 cm
F1P 8.200 ppm
F1 3282.05 Hz
F2P 7.000 ppm
F2 2801.75 Hz
PPMCM 0.03000 ppm/cm
HZCM 12.00750 Hz/cm
HCl
H
N
O
O
CF3
CF3
F
Integral
5.008
30.484
ppm
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
nmr400a h-1

S35

nmr400a h-1

```
Current Data Parameters
NAME        72041-124.HCl
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20020202
Time                17.25
INSTRUM             spect
PROBHD      5 mm QNP  1H
PULPROG              zg30
TD                  32768
SOLVENT              DMSO
NS                     32
DS                      2
SWH              6578.947 Hz
FIDRES           0.200774 Hz
AQ              2.4904180 sec
RG                    114
DW                 76.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             0.10000000 sec

============ CHANNEL f1 =============
NUC1                   1H
P1                  10.50 usec
PL1                 -3.00 dB
SFO1          400.2522014 MHz

F2 - Processing parameters
SI                  16384
SF            400.2500593 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  40.00 cm
F1P                 5.500 ppm
F1                2201.38 Hz
F2P                 4.000 ppm
F2                1601.00 Hz
PPMCM             0.03750 ppm/cm
HZCM             15.00938 Hz/cm
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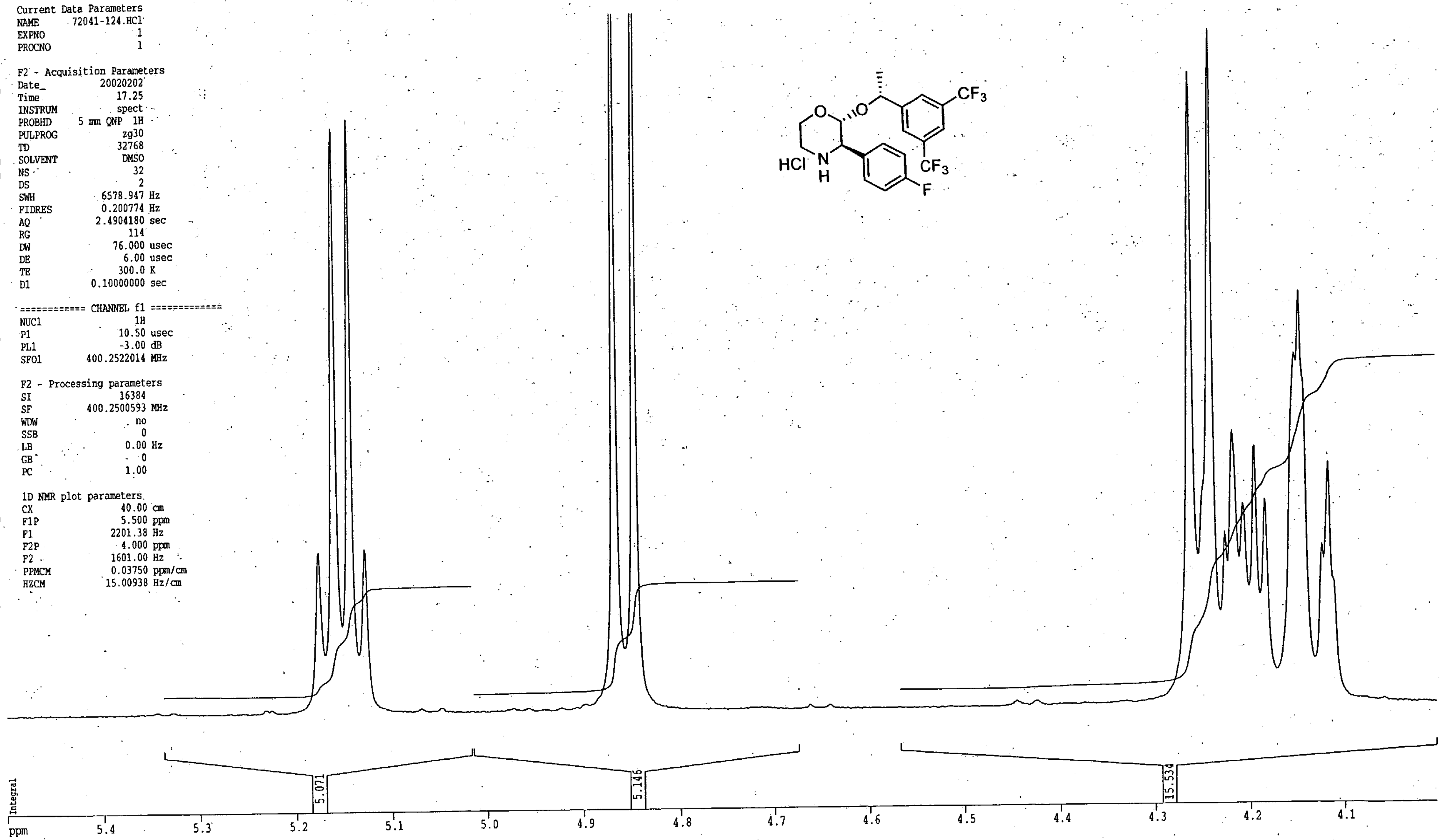


S36

nmr400a c-13

Current Data Parameters
NAME 72041-124.HCl
EXPNO t-Sa-amine 2
PROCNO 1

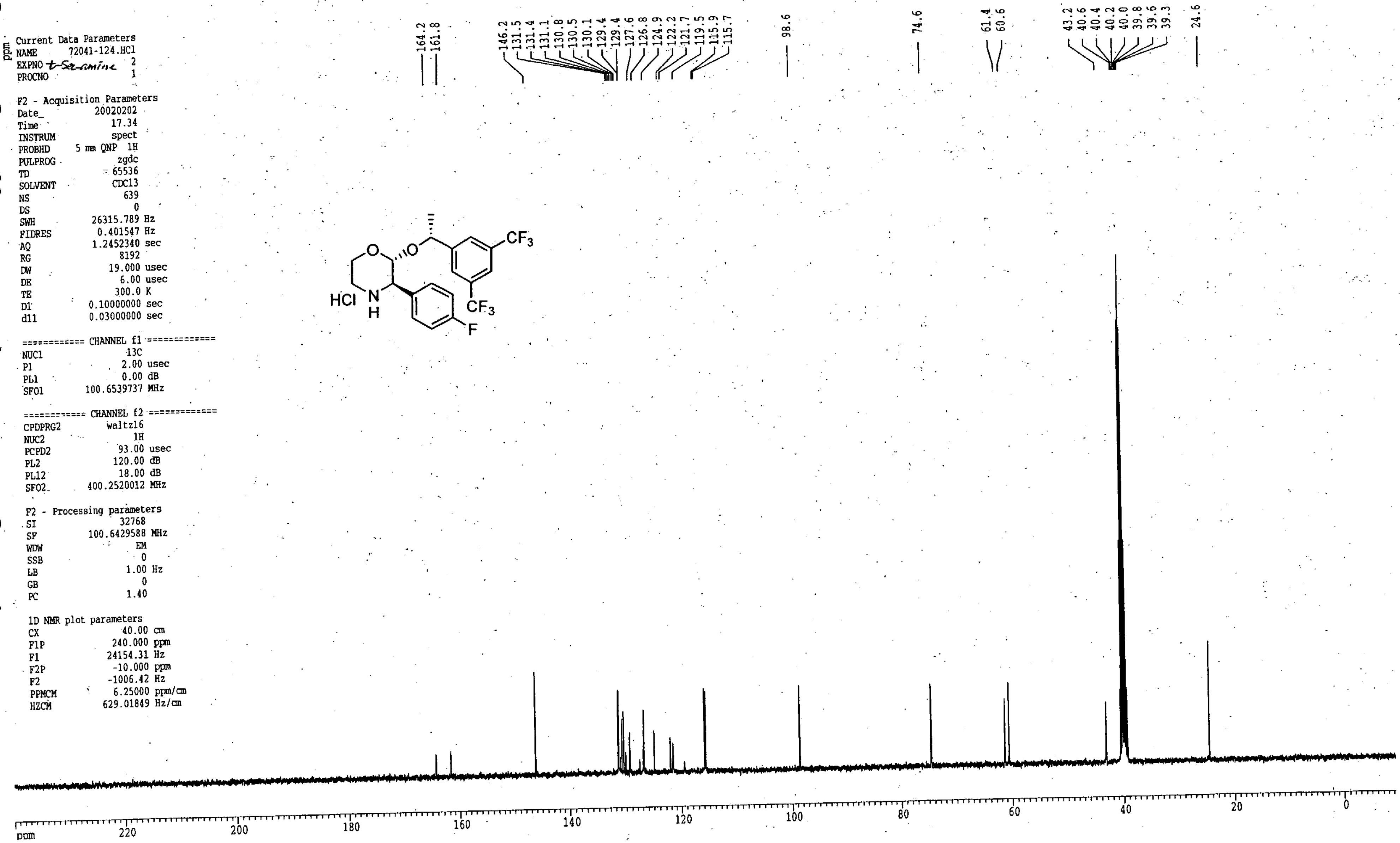
F2 - Acquisition Parameters
Date_ 20020202
Time 17.34
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 639
DS 0
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec

============ CHANNEL f1 ============
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 100.6539737 MHz

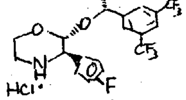
============ CHANNEL f2 ============
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz

F2 - Processing parameters
SI 32768
SF 100.6429588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 240.000 ppm
F1 24154.31 Hz
F2P -10.000 ppm
F2 -1006.42 Hz
PPMCM 6.25000 ppm/cm
HZCM 629.01849 Hz/cm



S37



:240.000ppm, F2=-10.000ppm, MI=0.10cm, MAXI=10000.00cm, I

nmr400a apt

#	ADDRESS	FREQUENCY [Hz]	[PPM]	INTENSITY
1	9521.6	16526.070	164.2049	-2.50
2	9828.6	16279.534	161.7553	-2.78
3	11782.0	14710.733	146.1675	-8.50
4	13622.8	13232.427	131.4789	9.34
5	13633.3	13224.025	131.3954	9.20
6	13669.5	13194.903	131.1061	-1.18
7	13710.3	13162.190	130.7810	-3.59
8	13751.1	13129.366	130.4549	-4.09
9	13792.1	13096.441	130.1277	-1.40
10	13884.8	13022.011	129.3882	-3.41
11	13888.3	13019.181	129.3601	-3.54
12	14111.1	12840.303	127.5827	-0.69
13	14211.9	12759.349	126.7784	6.49
14	14450.9	12567.408	124.8712	-1.68
15	14790.8	12294.384	122.1584	-1.41
16	14850.5	12246.495	121.6826	2.67
17	15130.3	12021.786	119.4499	-0.67
18	15574.9	11664.744	115.9022	8.90
19	15601.9	11643.043	115.6866	8.92
20	17744.0	9922.709	98.5932	10.00
21	20748.3	7509.948	74.6197	8.94
22	22400.6	6182.988	61.4349	-6.06
23	22504.7	6099.391	60.6043	8.40
24	24682.3	4350.601	43.2281	-6.30
25	25011.6	4086.158	40.6005	-1.02
26	25038.2	4064.805	40.3884	-3.54
27	25064.3	4043.840	40.1801	-7.20
28	25090.5	4022.812	39.9711	-8.83
29	25116.6	4001.795	39.7623	-7.49
30	25142.8	3980.764	39.5533	-3.77
31	25168.7	3960.000	39.3470	-1.27
32	27014.3	2477.807	24.6198	7.79

2465 163.9
8.4 131.44
32.74 32.8 32.9 130.6
2.8 129.4
270.1 271.0 272.6 123.5
21.70 115.8

Current Data Parameters
NAME 72041-124.HCl
EXPNO 3
PROCNO 1
DMSO

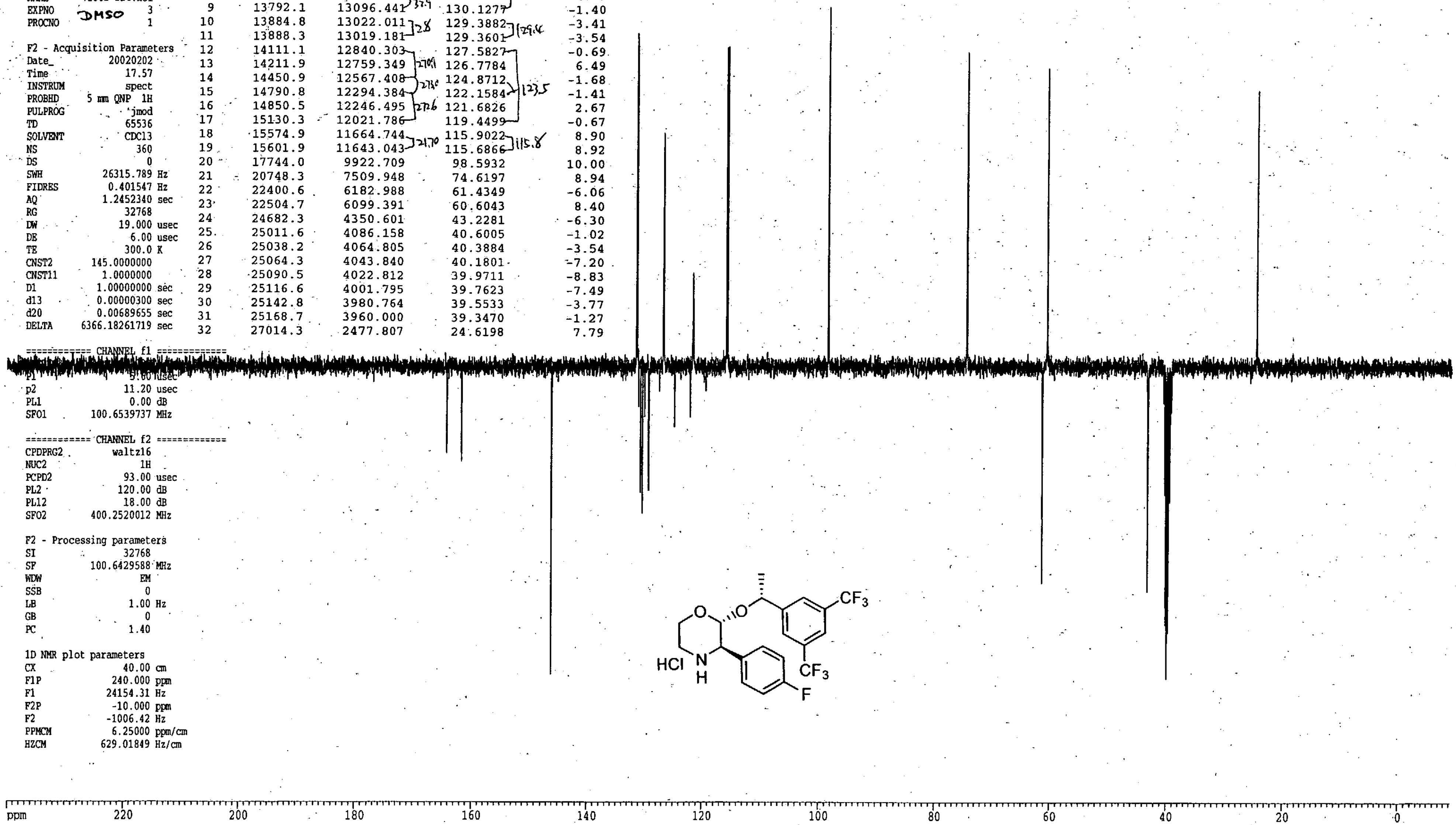
F2 - Acquisition Parameters
Date_ 20020202
Time 17.57
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 360
DS 0
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 32768
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 1.00000000 sec
d13 0.00000300 sec
d20 0.00689655 sec
DELTA 6366.18261719 sec

============ CHANNEL f1 ============
P1 5.00 usec
p2 11.20 usec
PL1 0.00 dB
SFO1 100.6539737 MHz

============ CHANNEL f2 ============
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz

F2 - Processing parameters
SI 32768
SF 100.6429588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 40.00 cm
F1P 240.000 ppm
F1 24154.31 Hz
F2P -10.000 ppm
F2 -1006.42 Hz
PPMCM 6.25000 ppm/cm
HZCM 629.01849 Hz/cm



S38

nmr400a f-19

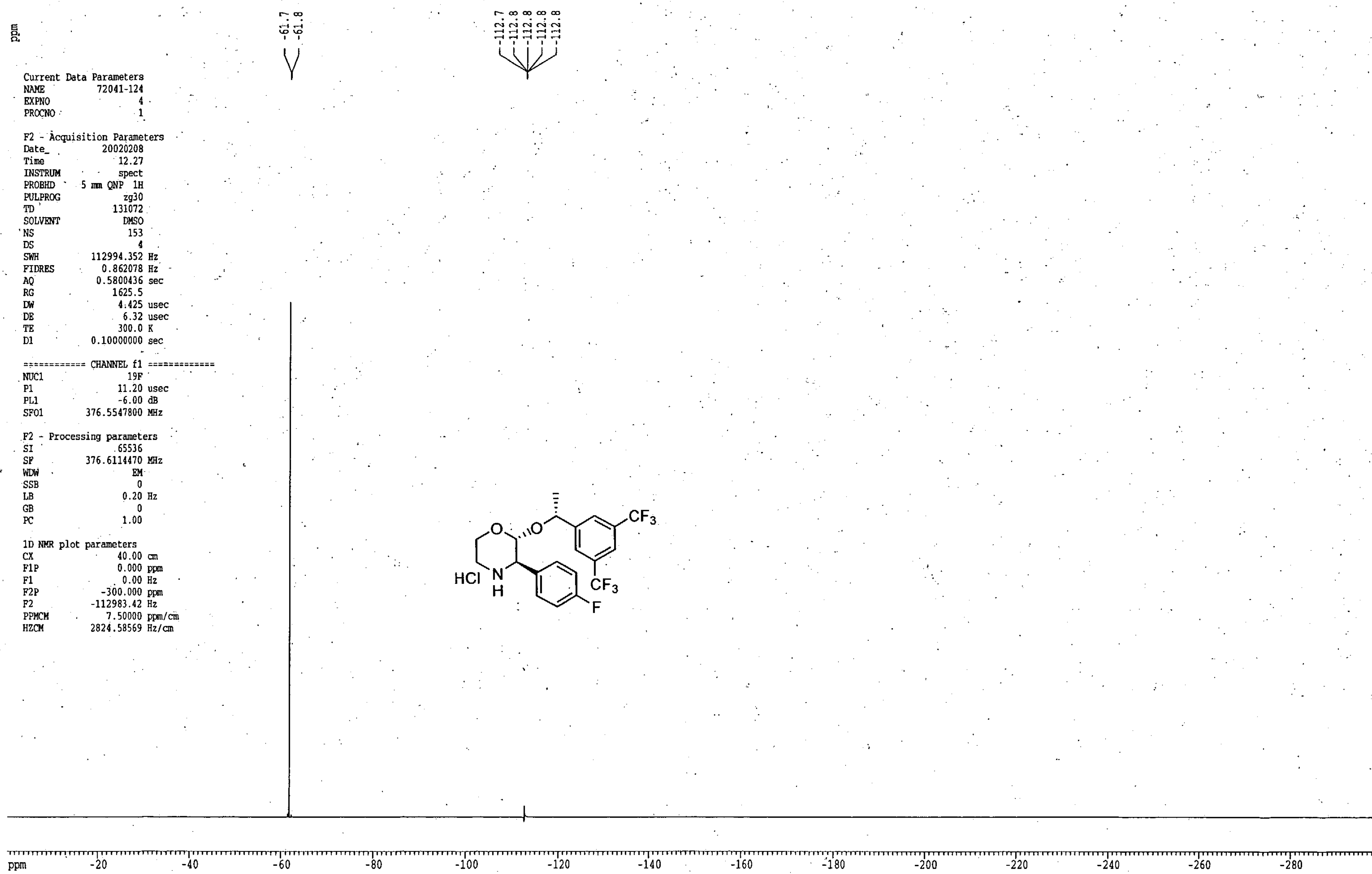
Current Data Parameters
NAME 72041-124
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020208
Time 12.27
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 131072
SOLVENT DMSO
NS 153
DS 4
SWH 112994.352 Hz
FIDRES 0.862078 Hz
AQ 0.5800436 sec
RG 1625.5
DW 4.425 usec
DE 6.32 usec
TE 300.0 K
D1 0.10000000 sec

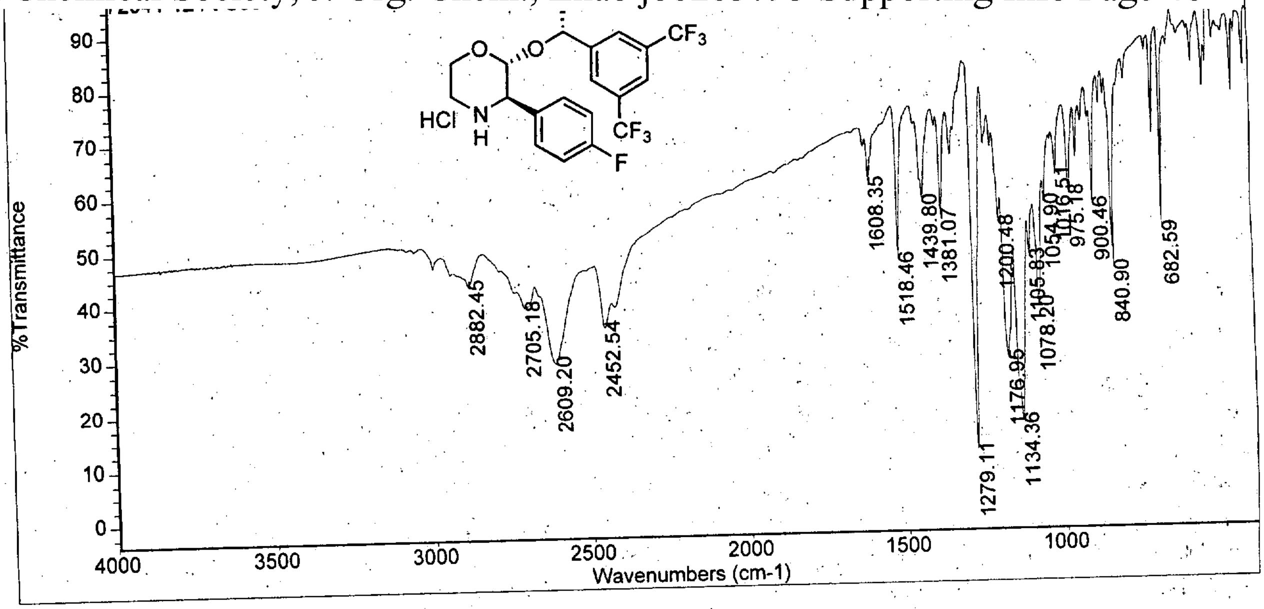
============ CHANNEL f1 ============
NUC1 19F
P1 11.20 usec
PL1 -6.00 dB
SFO1 376.5547800 MHz

F2 - Processing parameters
SI 65536
SF 376.6114470 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
F1P 0.000 ppm
F1 0.00 Hz
F2P -300.000 ppm
F2 -112983.42 Hz
PPMCM 7.50000 ppm/cm
HZCM 2824.58569 Hz/cm



639



Mon Feb 04 15:16:25 2002
FIND PEAKS:
Spectrum: 72041-124 t-Sec-amine.HCl
Region: 4000.00 400.00
Absolute threshold: 63.072
Sensitivity: 50
Peak list:

Position:	1279.11	Intensity:	10.617
Position:	1134.36	Intensity:	16.061
Position:	1176.95	Intensity:	27.438
Position:	2609.20	Intensity:	28.326
Position:	2452.54	Intensity:	34.645
Position:	1078.20	Intensity:	37.170
Position:	2705.18	Intensity:	38.584
Position:	2882.45	Intensity:	42.777
Position:	840.90	Intensity:	43.792
Position:	1105.83	Intensity:	45.986
Position:	1518.46	Intensity:	45.990
Position:	682.59	Intensity:	50.653
Position:	1200.48	Intensity:	52.260
Position:	1381.07	Intensity:	53.737
Position:	900.46	Intensity:	55.380
Position:	1054.90	Intensity:	55.931
Position:	1439.80	Intensity:	56.393
Position:	975.18	Intensity:	57.828
Position:	1608.35	Intensity:	59.995
Position:	1016.51	Intensity:	60.883

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