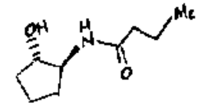
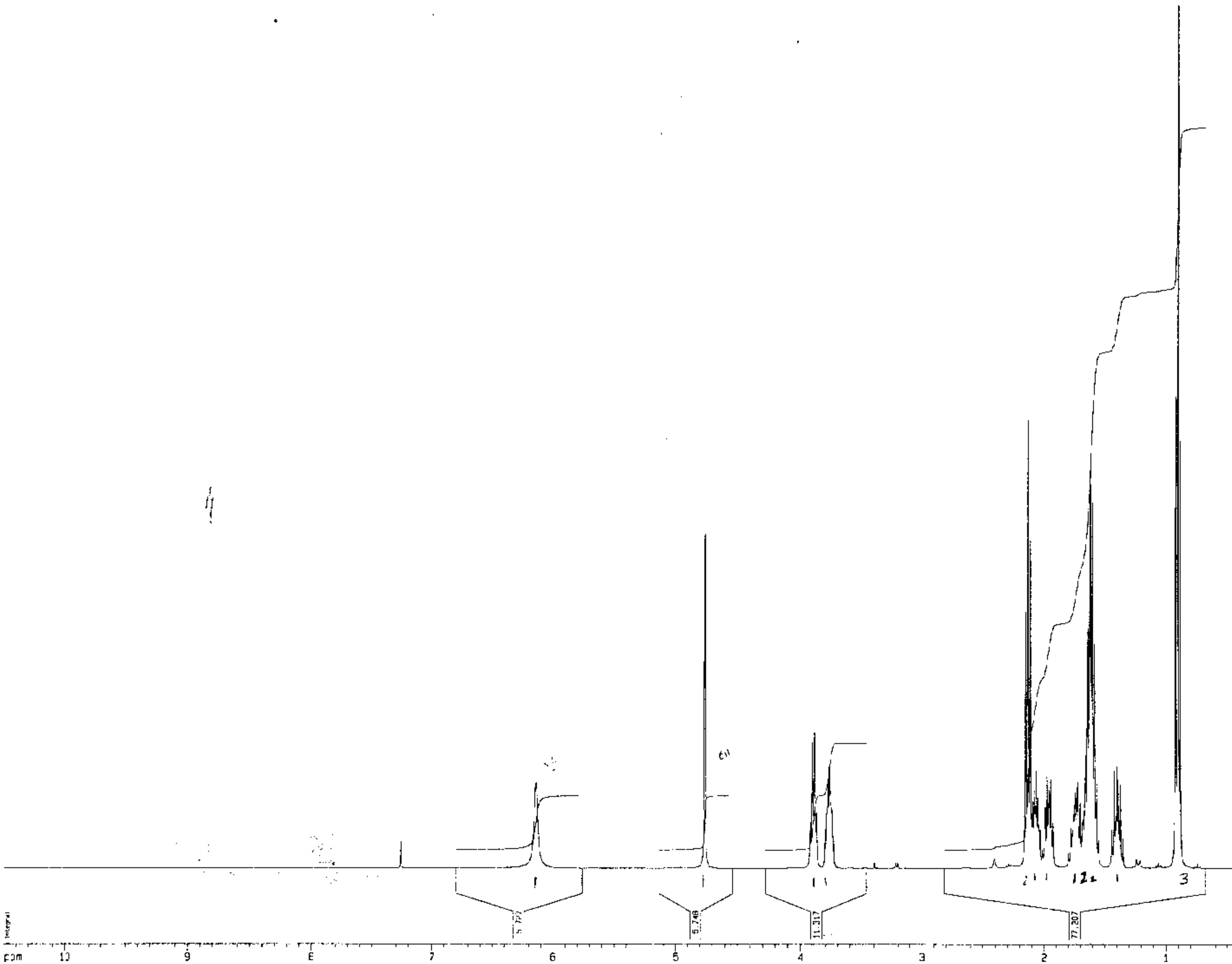
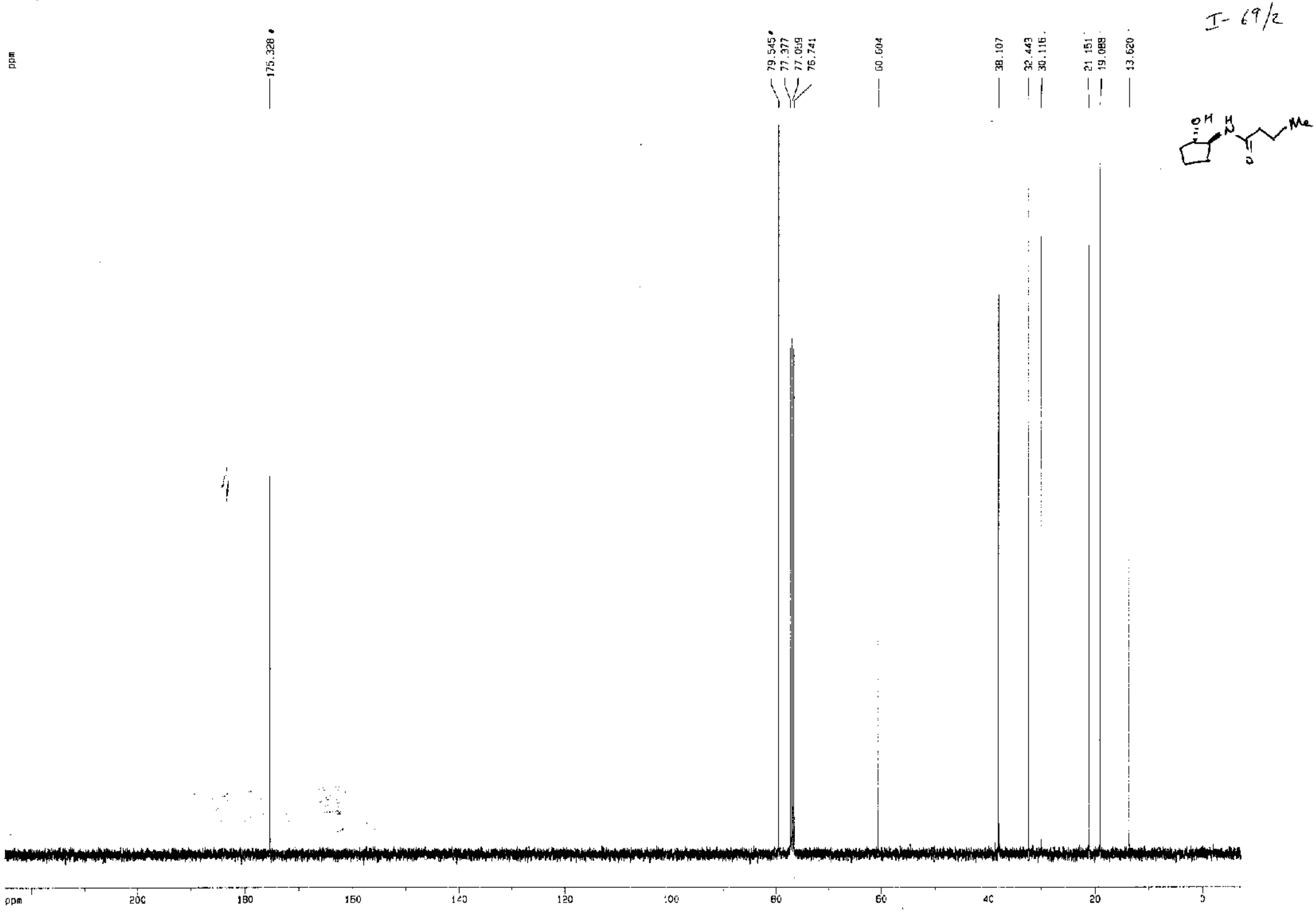
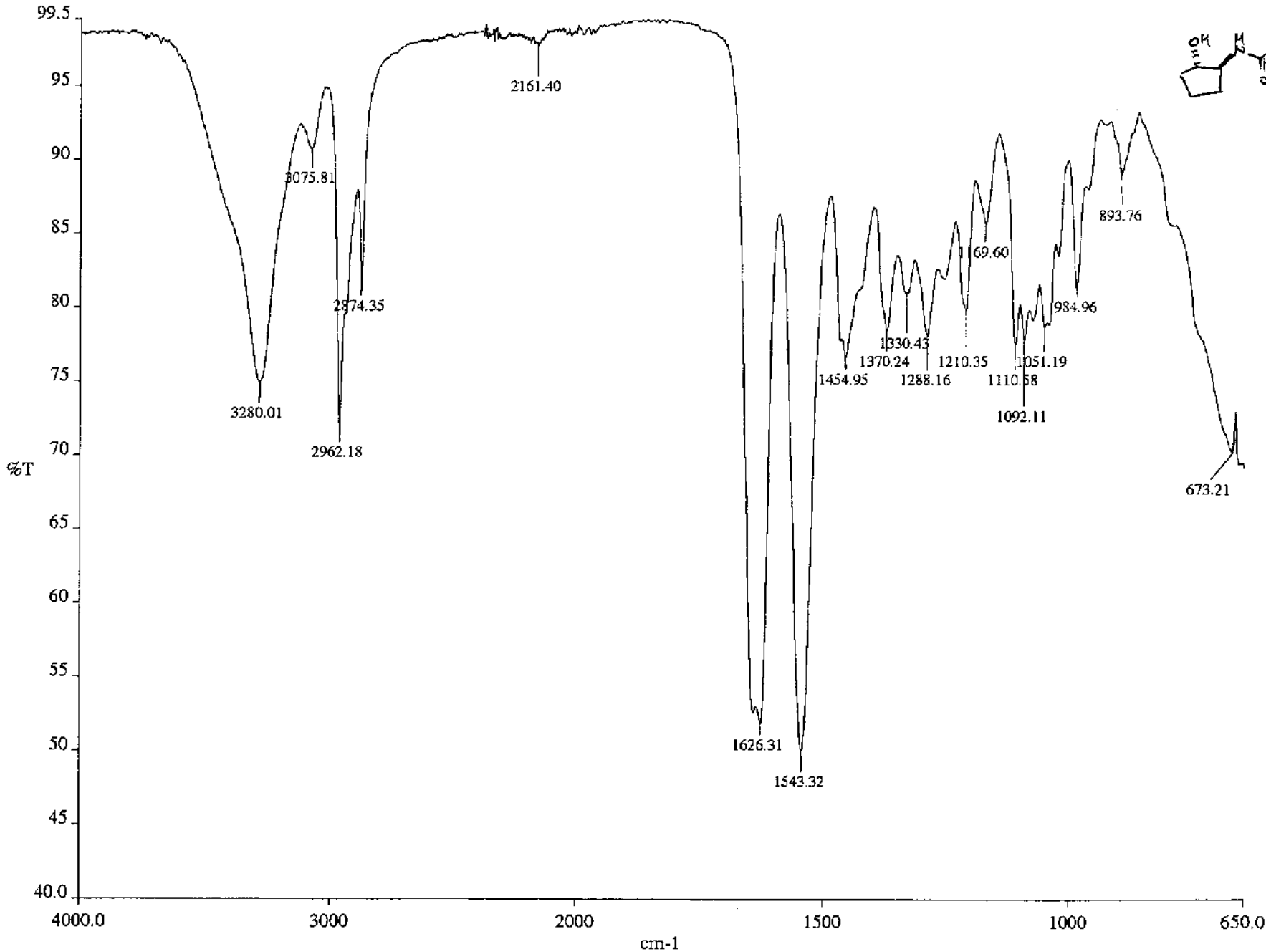
I-69/2


OH
H
N
O
Me


1
1
1 1
2 1 1
1 2 2
1
3
5.722
5.748
11.317
77.207
Integral
ppm
10
9
8
7
6
5
4
3
2
1
0


I-69/2
ppm
175.328
79.545
77.377
77.059
76.741
60.604
38.107
32.443
30.116
21.151
19.088
13.620
OH
H
N
O
Me
ppm
200
180
160
140
120
100
80
60
40
20
0

I-69/2

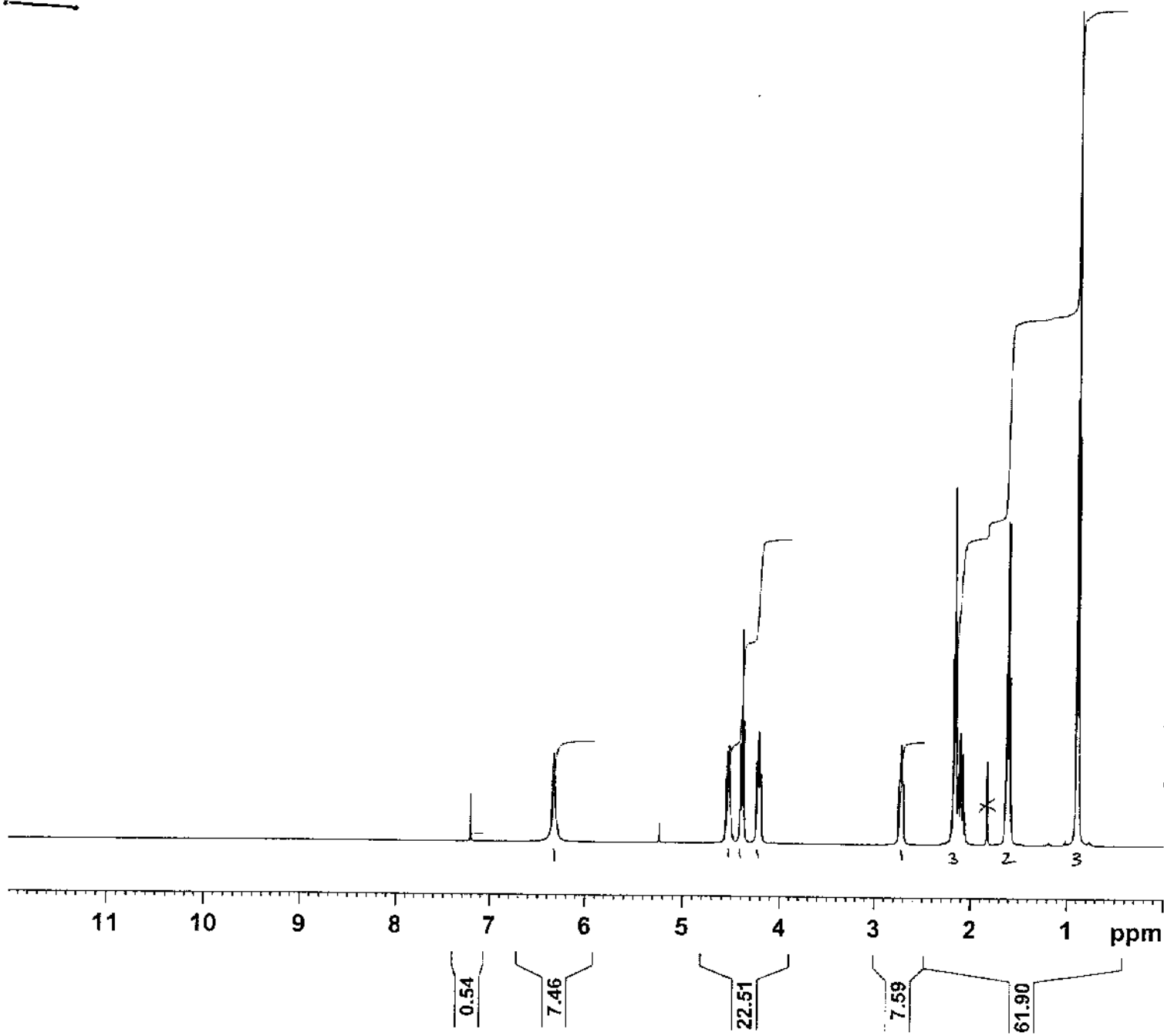


d:\group data\drs-i-69-2.sp

get expansion!

proton.std CDCl3 {d:\data\drs} dss29 11

BHL



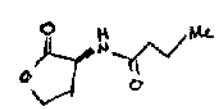
BRUKER

Current Data Parameters
NAME I1592
EXPNO 11
PROCNO 1

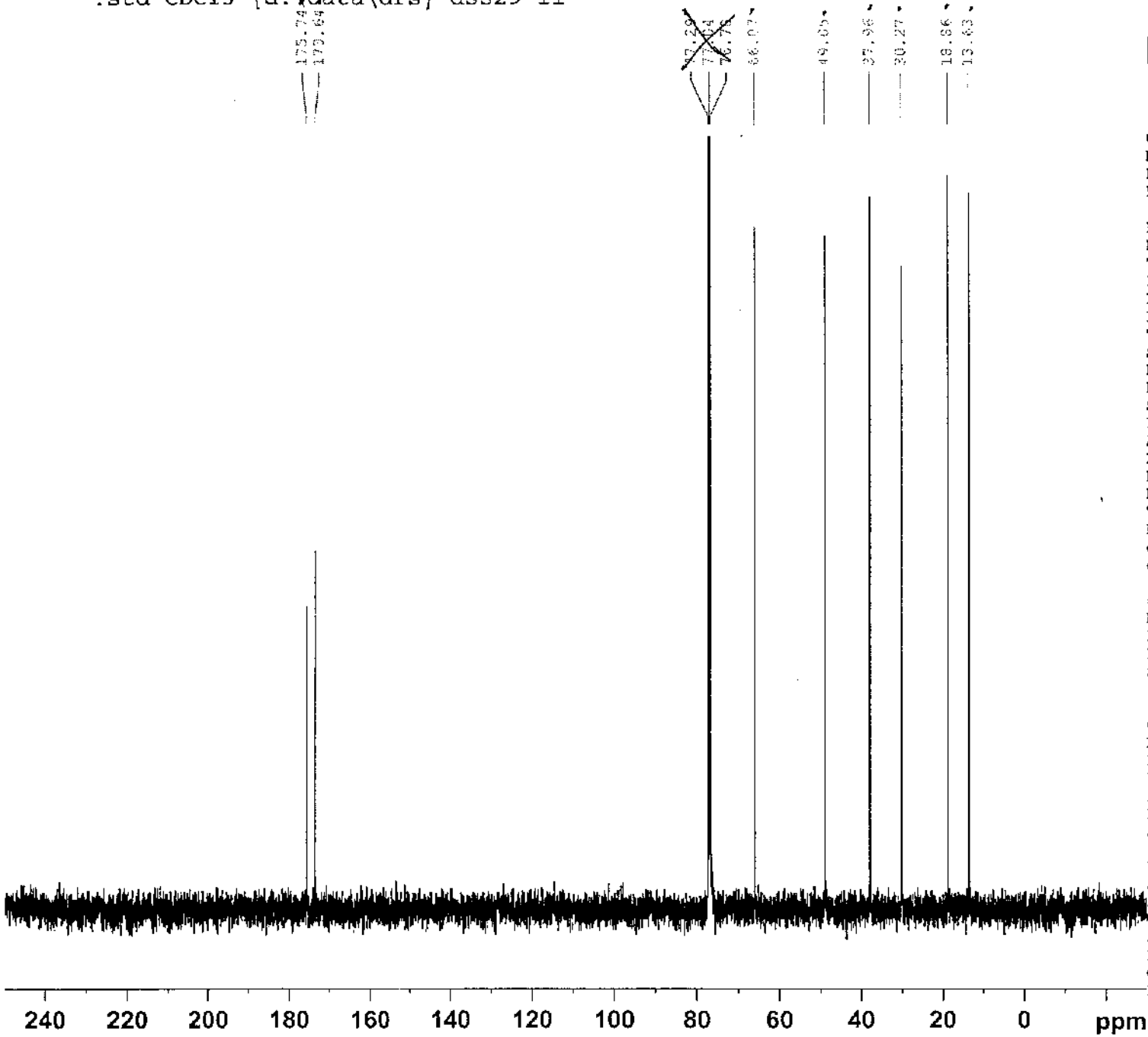
F2 - Acquisition Parameters
Date_ 20040325
Time 7.14
INSTRUM AV500
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 128
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 10.75 usec
PL1 -3.00 dB
SFO1 500.2030889 MHz

F2 - Processing parameters
SI 131072
SF 500.2000334 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



.std CDCl3 {d:\data\drs} dss29 11



BHL

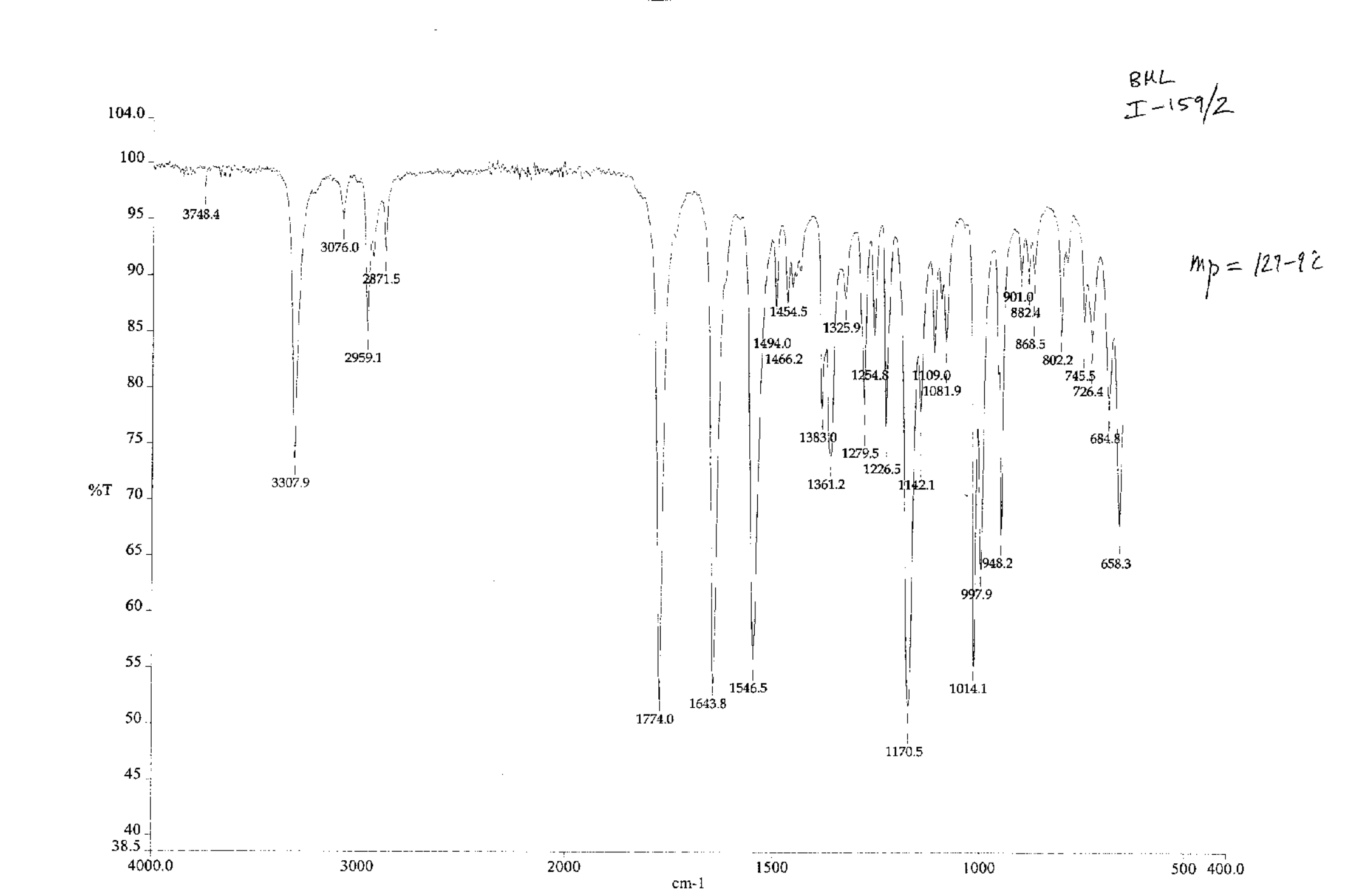
Current Data Parameters
NAME I1592
EXPNO 10
PROCNO 1

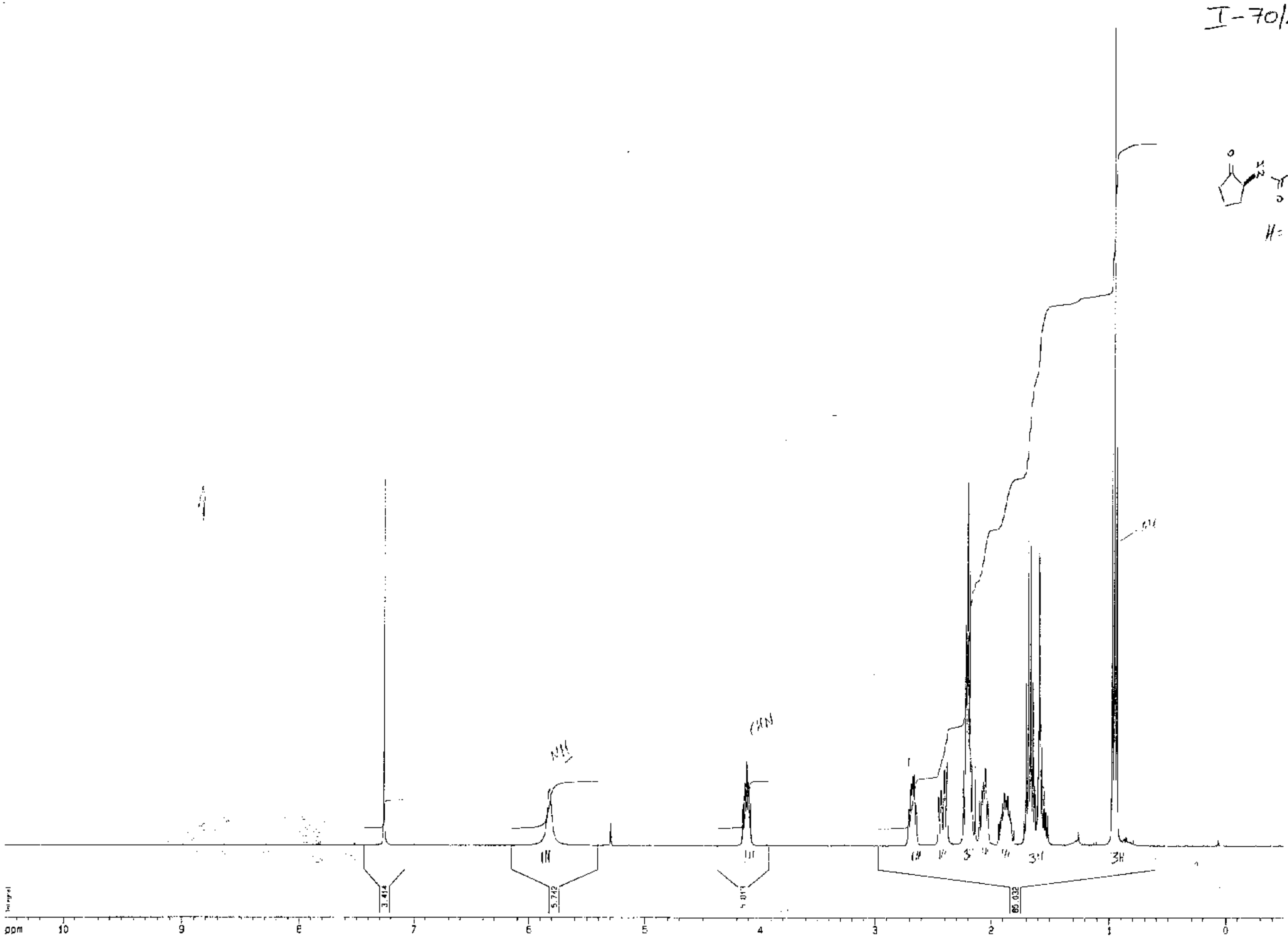
F2 - Acquisition Parameters
Date_ 20040325
Time 7.11
INSTRUM AV500
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 4
SWH 35211.270 Hz
FIDRES 0.537281 Hz
AQ 0.9306754 sec
RG 23170.5
DW 14.200 usec
DE 6.00 usec
TE 300.0 K
D1 4.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

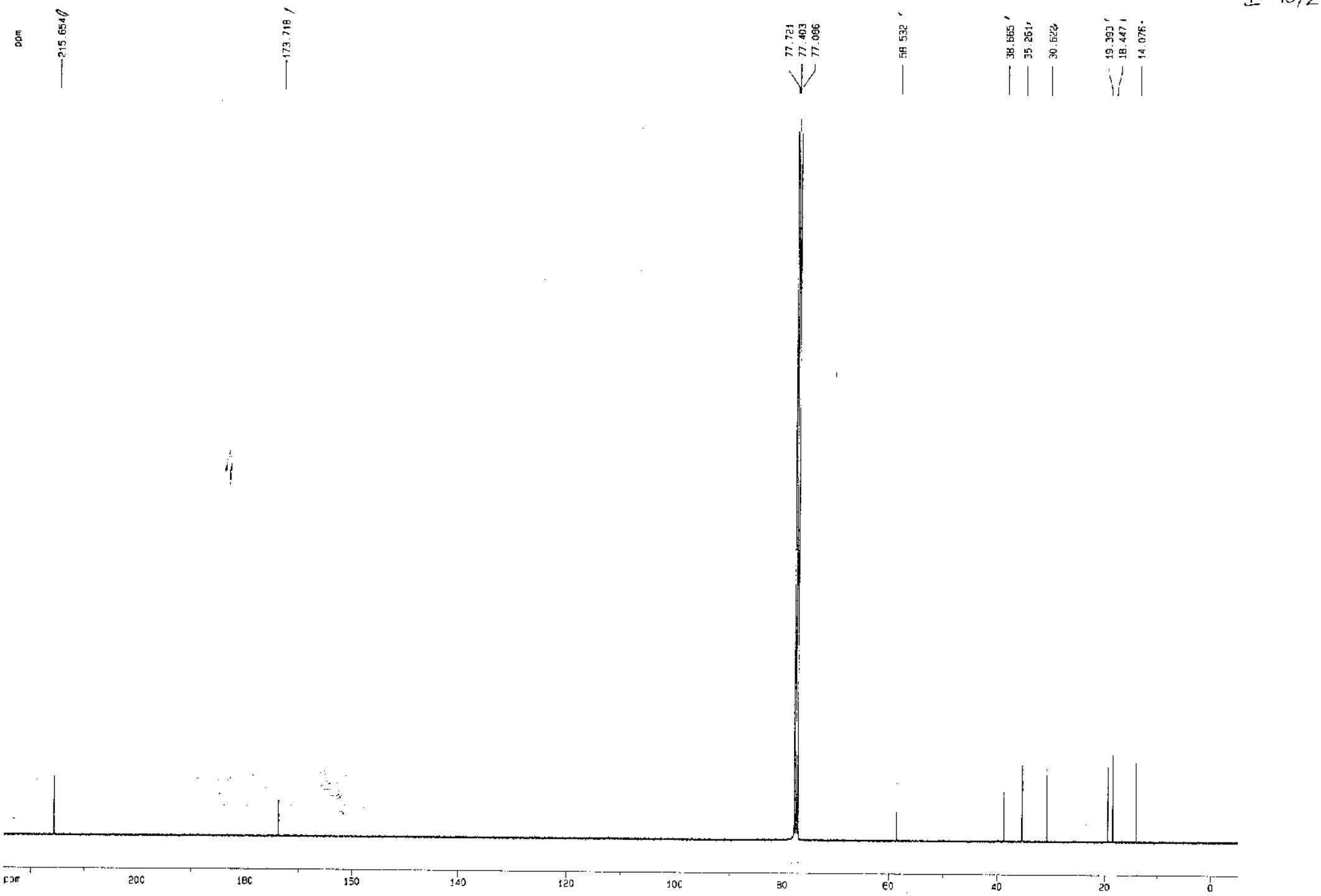
======== CHANNEL f1 ========
NUC1 13C
P1 8.45 usec
PL1 4.00 dB
SFO1 125.7892253 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 65.00 usec
PL2 -3.00 dB
PL12 13.00 dB
PL13 13.00 dB
SFO2 500.2020008 MHz

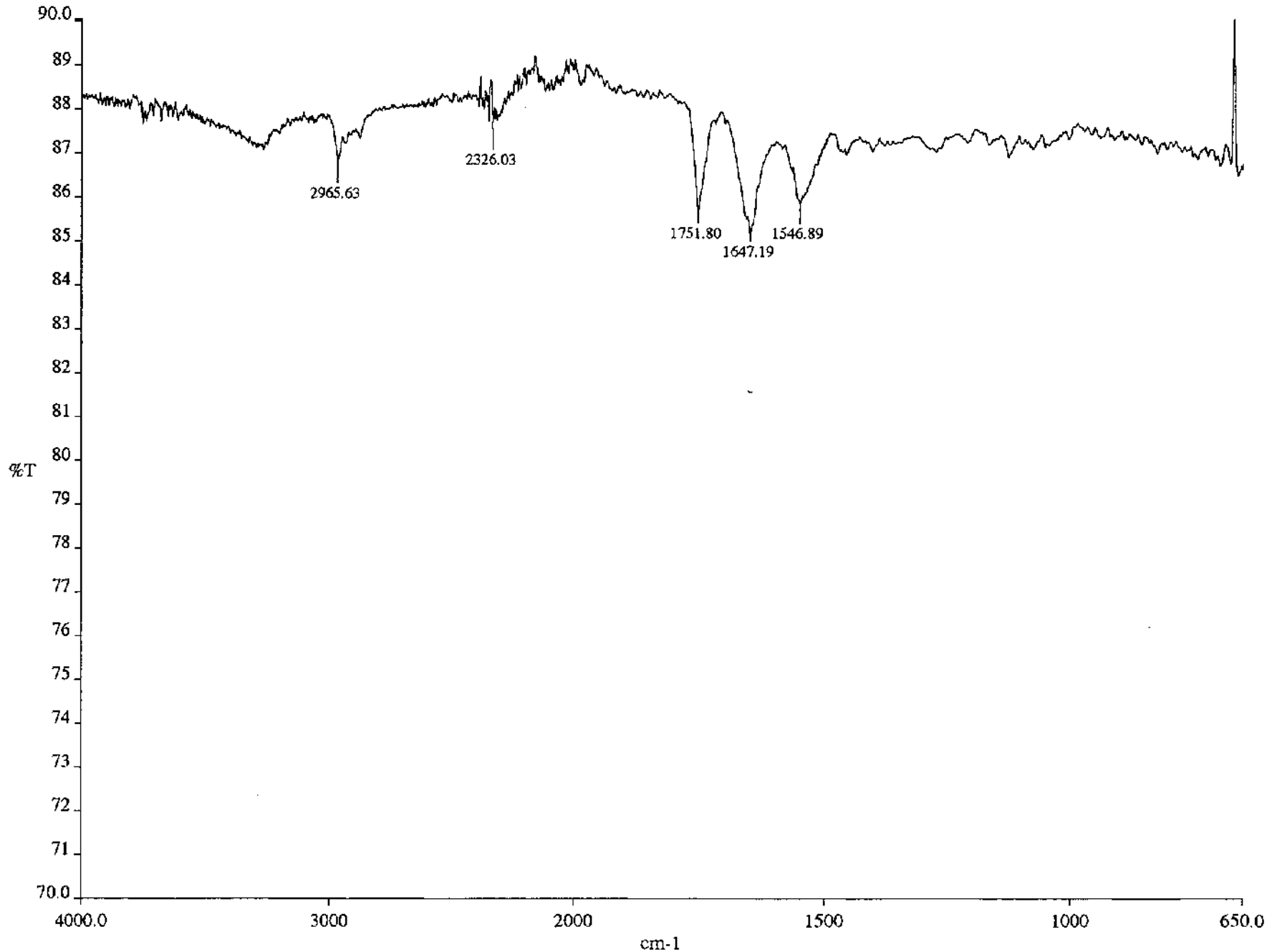
F2 - Processing parameters
SI 65536
SF 125.7753900 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40


BHL
I-159/2
mp = 127-9°C
104.0
100
95
90
85
80
75
70
65
60
55
50
45
40
38.5
%T
3748.4
3076.0
2871.5
2959.1
3307.9
1774.0
1643.8
1546.5
1454.5
1494.0
1466.2
1325.9
1383.0
1361.2
1279.5
1254.8
1226.5
1170.5
1142.1
1109.0
1081.9
1014.1
997.9
948.2
901.0
882.4
868.5
802.2
745.5
726.4
684.8
658.3
4000.0
3000
2000
1500
1000
500
400.0
cm-1
c:\pel_data\spectra\drs-i--2.sp

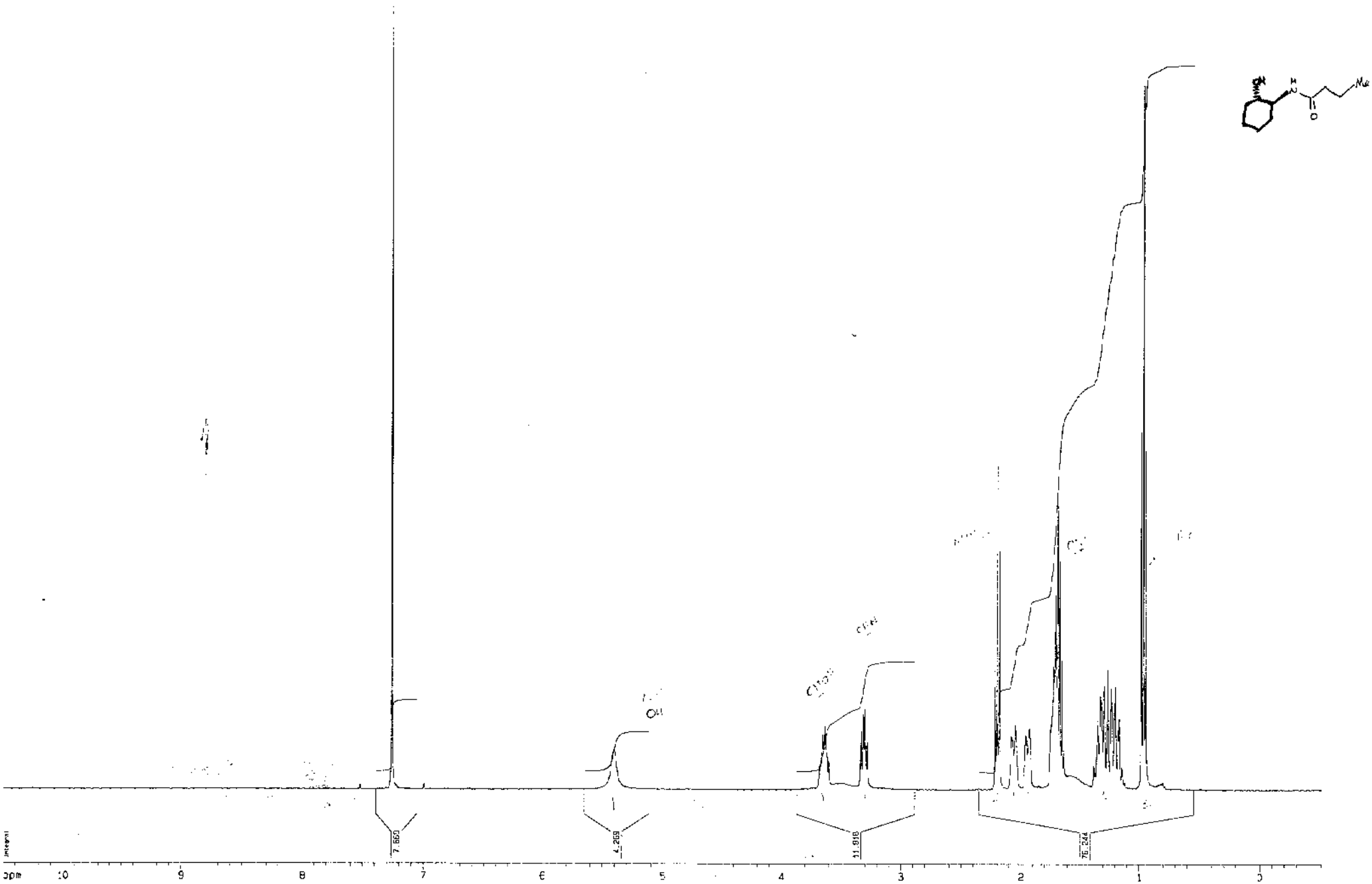

I-70/2
H=15
NH
CHN
1H
3H
3.414
5.742
85.032
ppm
10
9
8
7
6
5
4
3
2
1
0

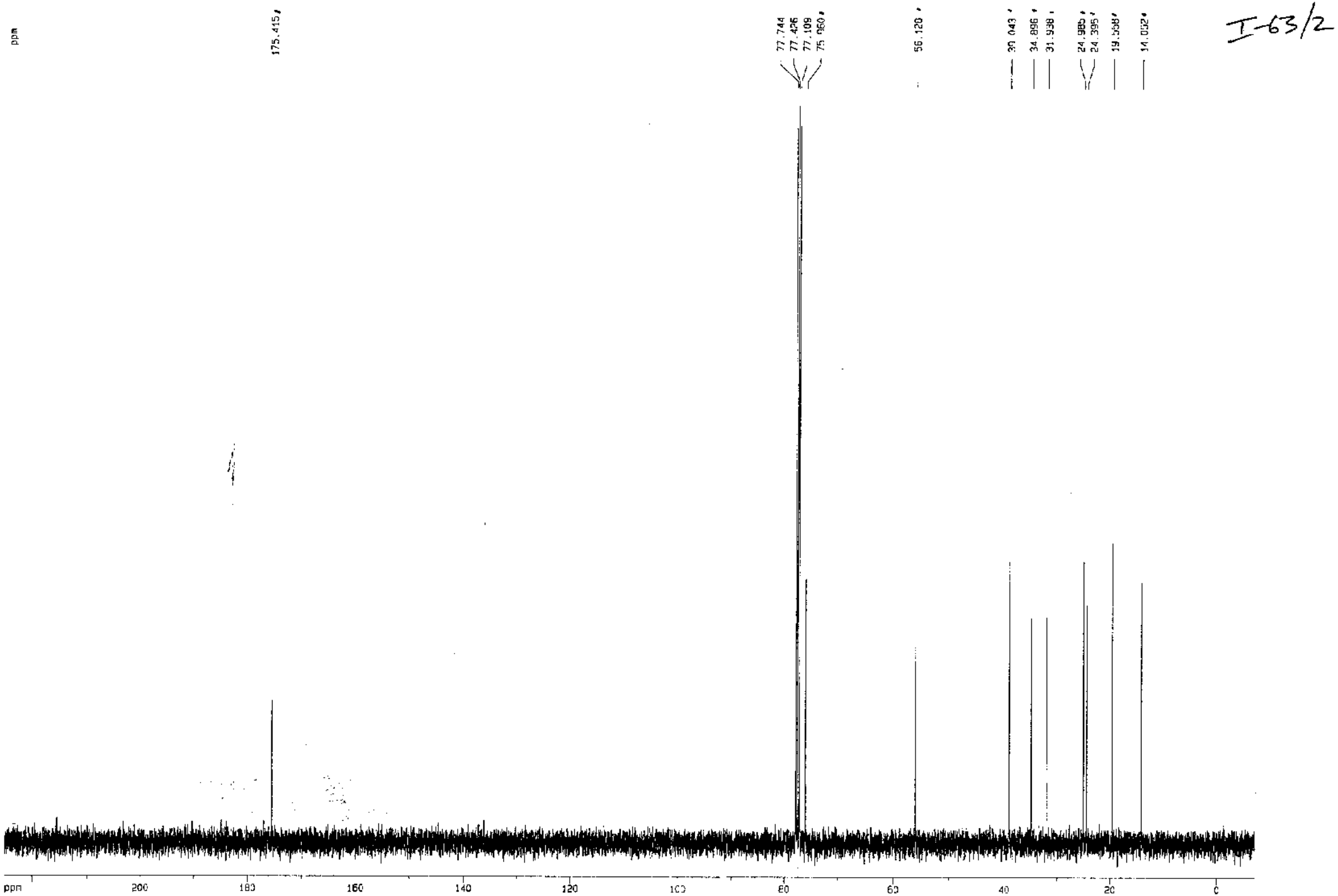

ppm
215.654
173.718
77.721
77.403
77.096
58.532
38.665
35.261
30.622
19.393
18.447
14.076
ppm
200
180
160
140
120
100
80
60
40
20
0

I-70/2


90.0
89
88
87
86
85
84
83
82
81
80
79
78
77
76
75
74
73
72
71
70.0
%T
2965.63
2326.03
1751.80
1647.19
1546.89
4000.0
3000
2000
1500
1000
650.0
cm-1
d:\group data\drs-i-70-2.sp

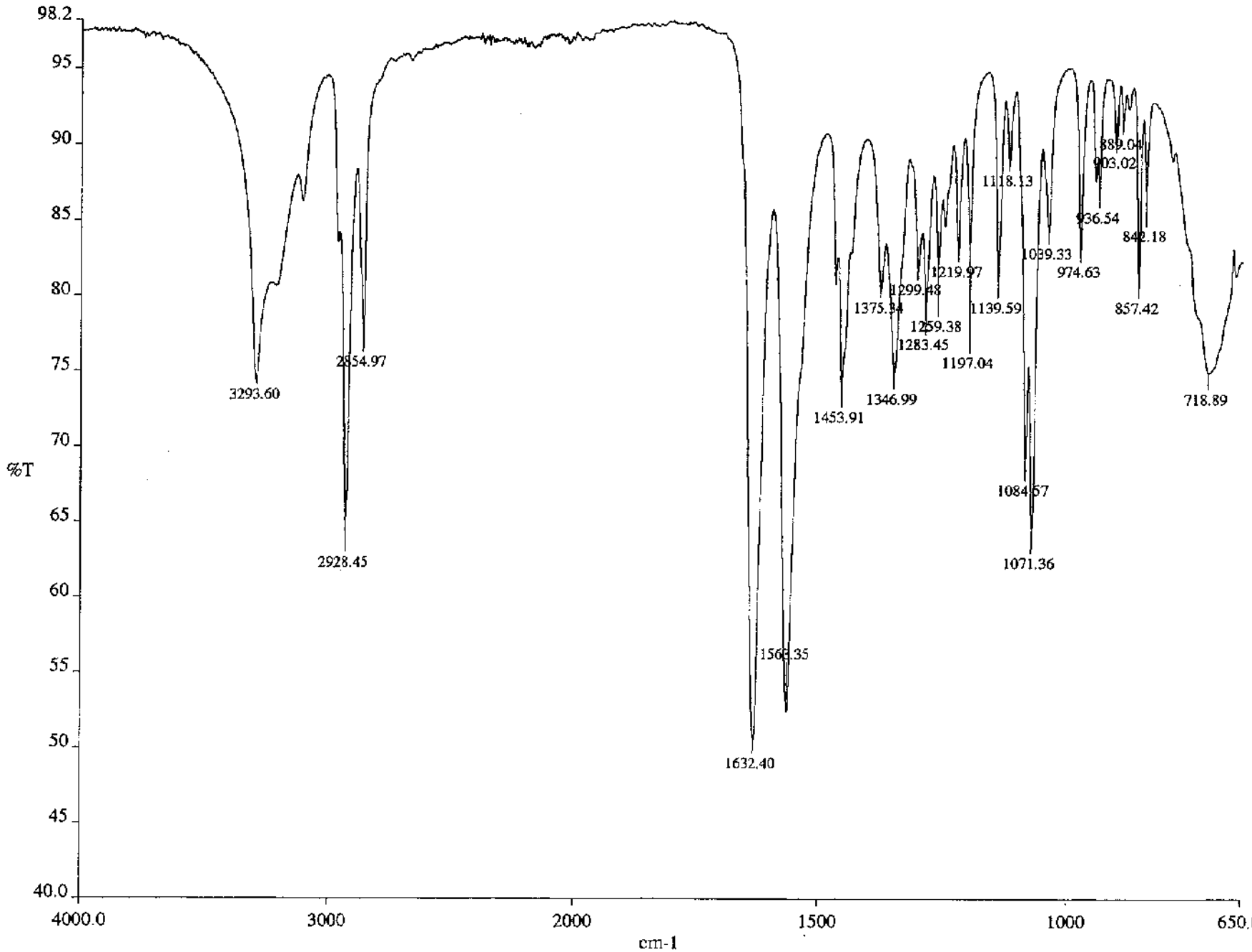
I-63/2



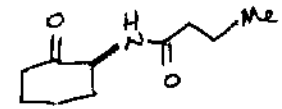

ppm
175.415
77.744
77.426
77.109
75.960
56.128
39.043
34.896
31.938
24.985
24.395
19.538
14.032
ppm
200
180
160
140
120
100
80
60
40
20
0

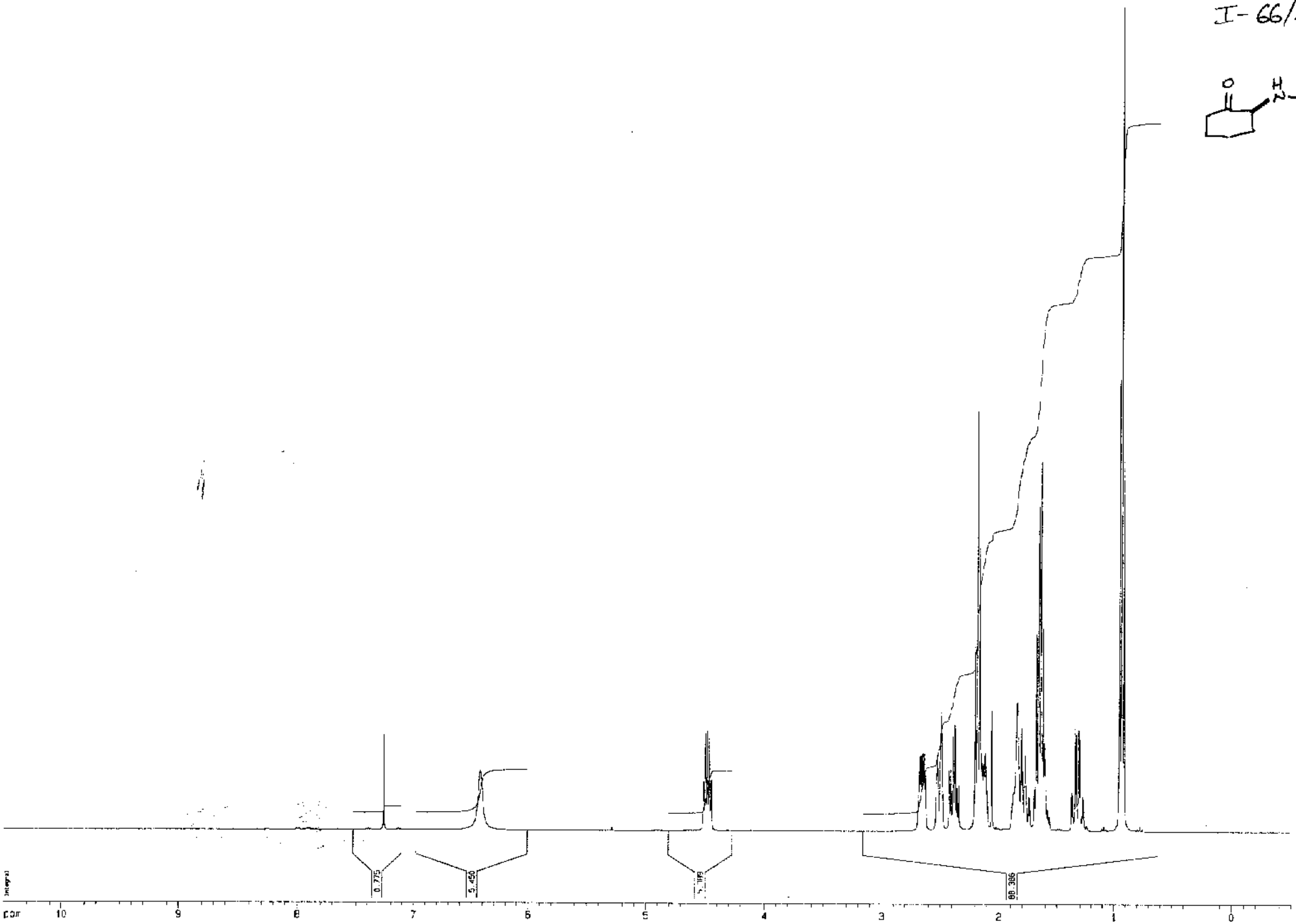
I-63/2

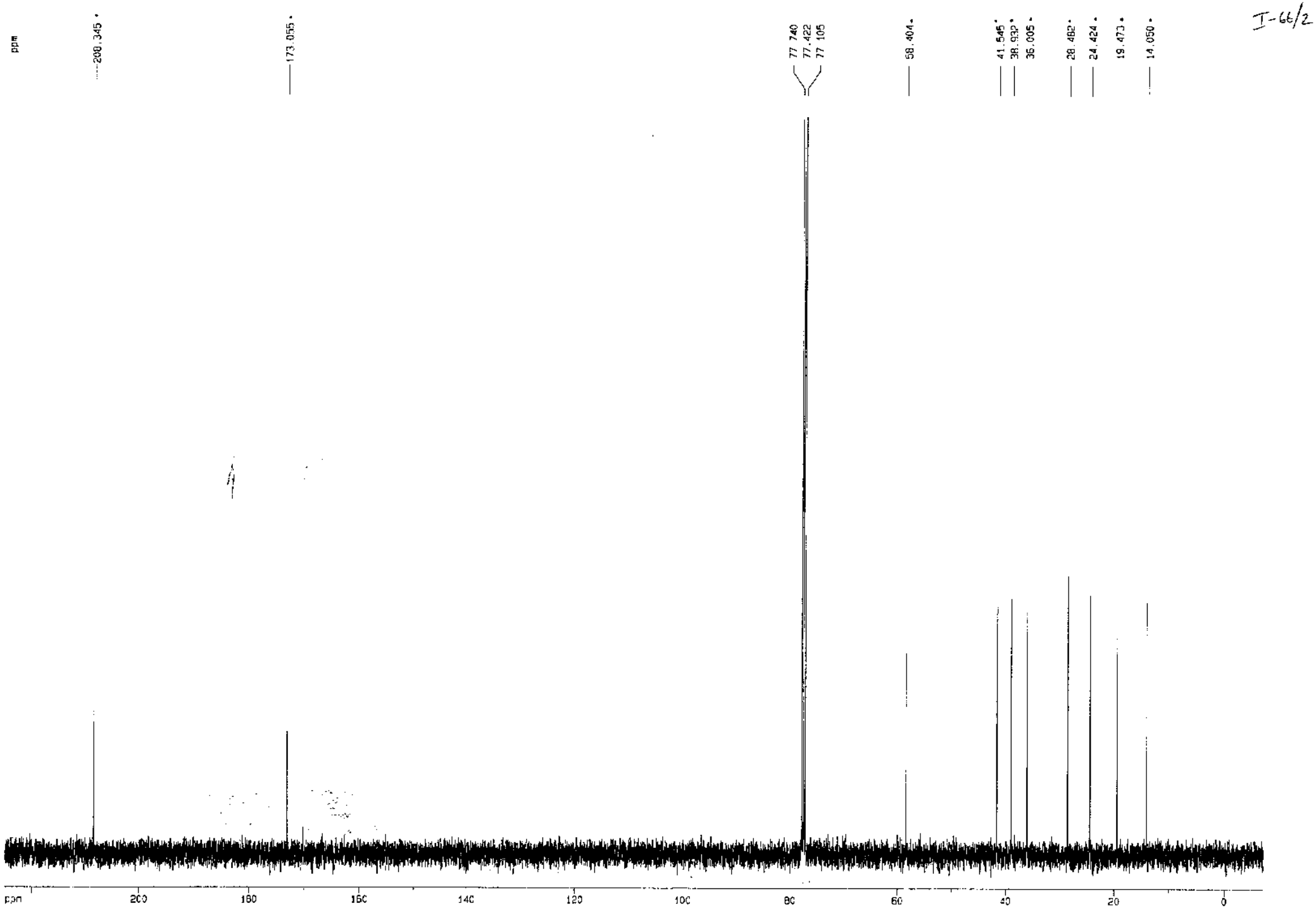
I-63/2


98.2
95
90
85
80
75
70
65
60
55
50
45
40.0
%T
4000.0
3000
2000
1500
1000
650.0
cm-1
3293.60
2954.97
2928.45
1632.40
1568.35
1453.91
1375.34
1346.99
1299.48
1283.45
1259.38
1219.97
1197.04
1139.59
1118.13
1084.67
1071.36
1039.33
974.63
936.54
903.02
889.04
857.42
842.18
718.89
d:\group data\drs-i-63-2.sp

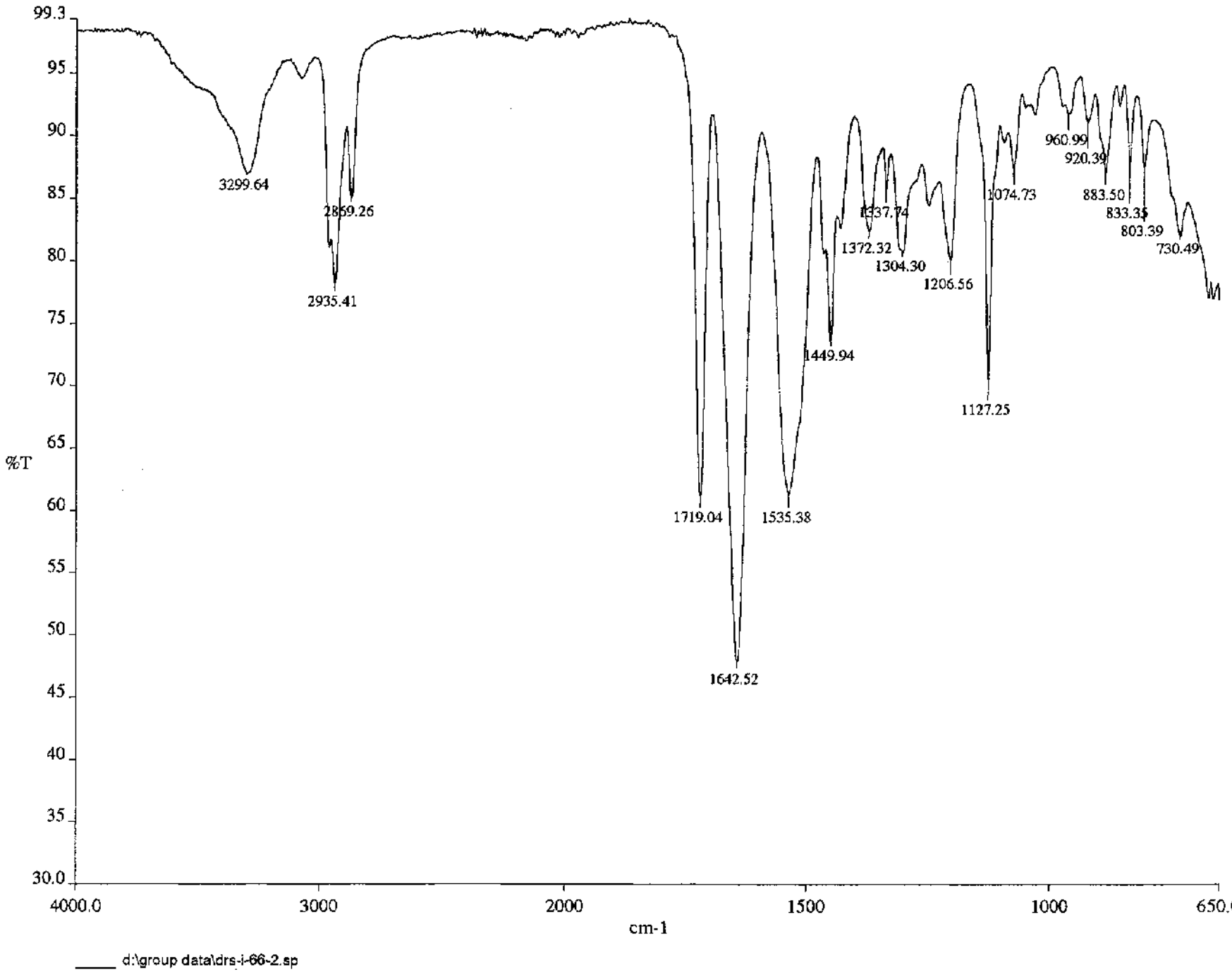
I-66/2



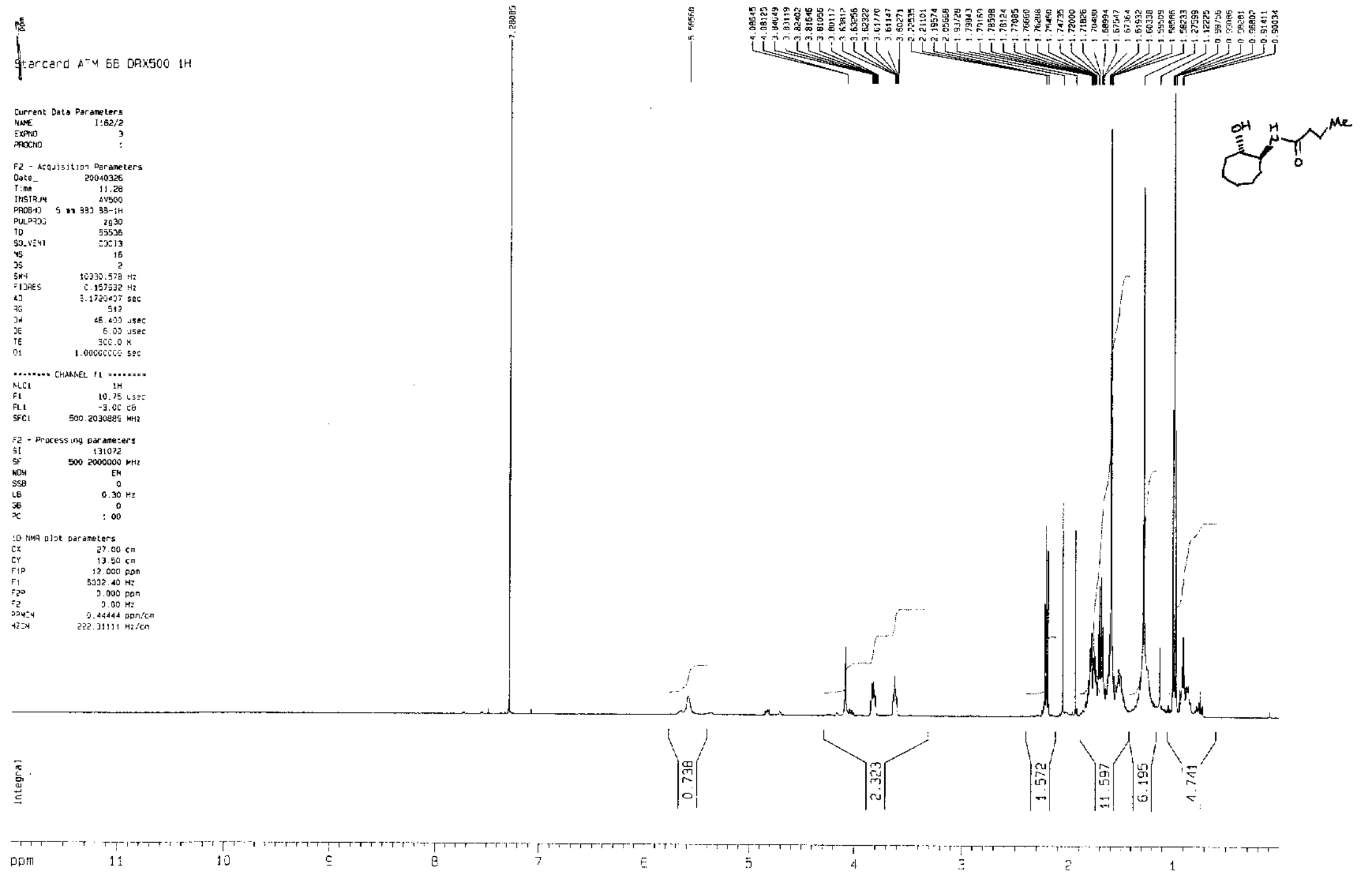


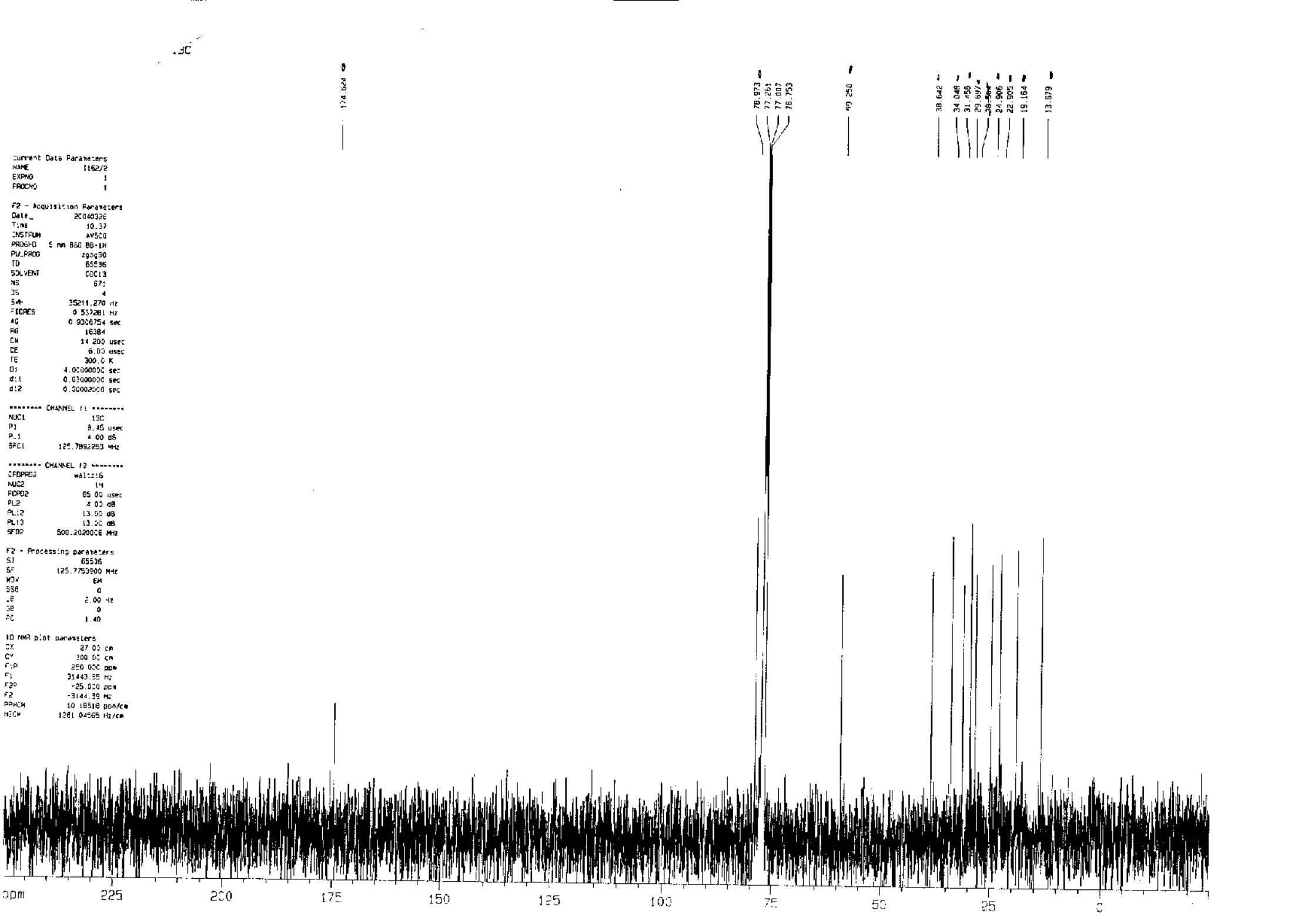

ppm
208.345
173.055
77.740
77.422
77.105
58.404
41.545
38.932
36.005
28.482
24.424
19.473
14.050
I-66/2
ppm
200
180
160
140
120
100
80
60
40
20
0

I-66/2

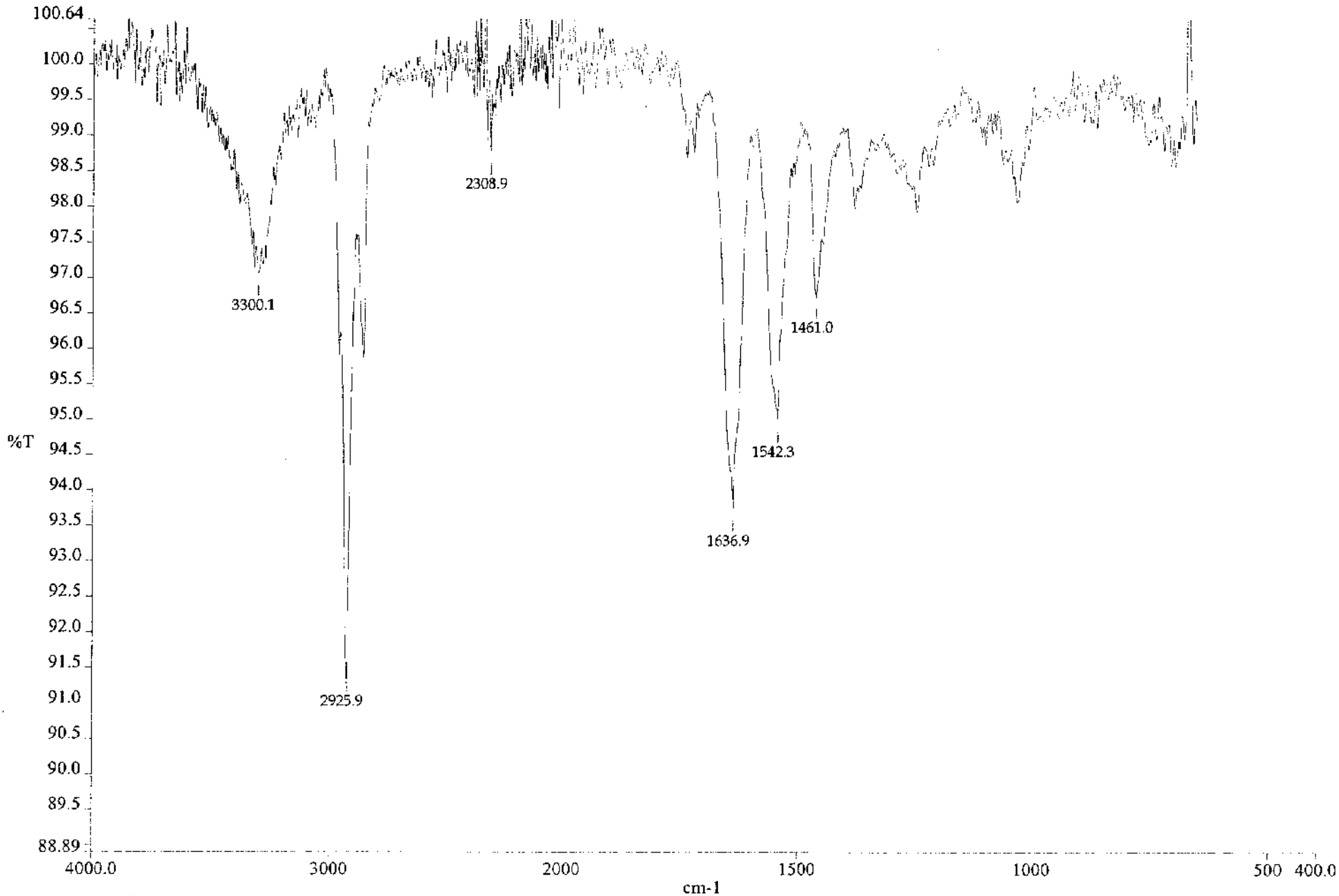

99.3
95
90
85
80
75
70
65
60
55
50
45
40
35
30.0
%T
3299.64
2859.26
2935.41
1719.04
1642.52
1535.38
1449.94
1372.32
1337.74
1304.30
1206.56
1127.25
1074.73
960.99
920.39
883.50
833.35
803.39
730.49
4000.0
3000
2000
1500
1000
650.0
cm-1
d:\group data\drs-i-66-2.sp

I-162/2

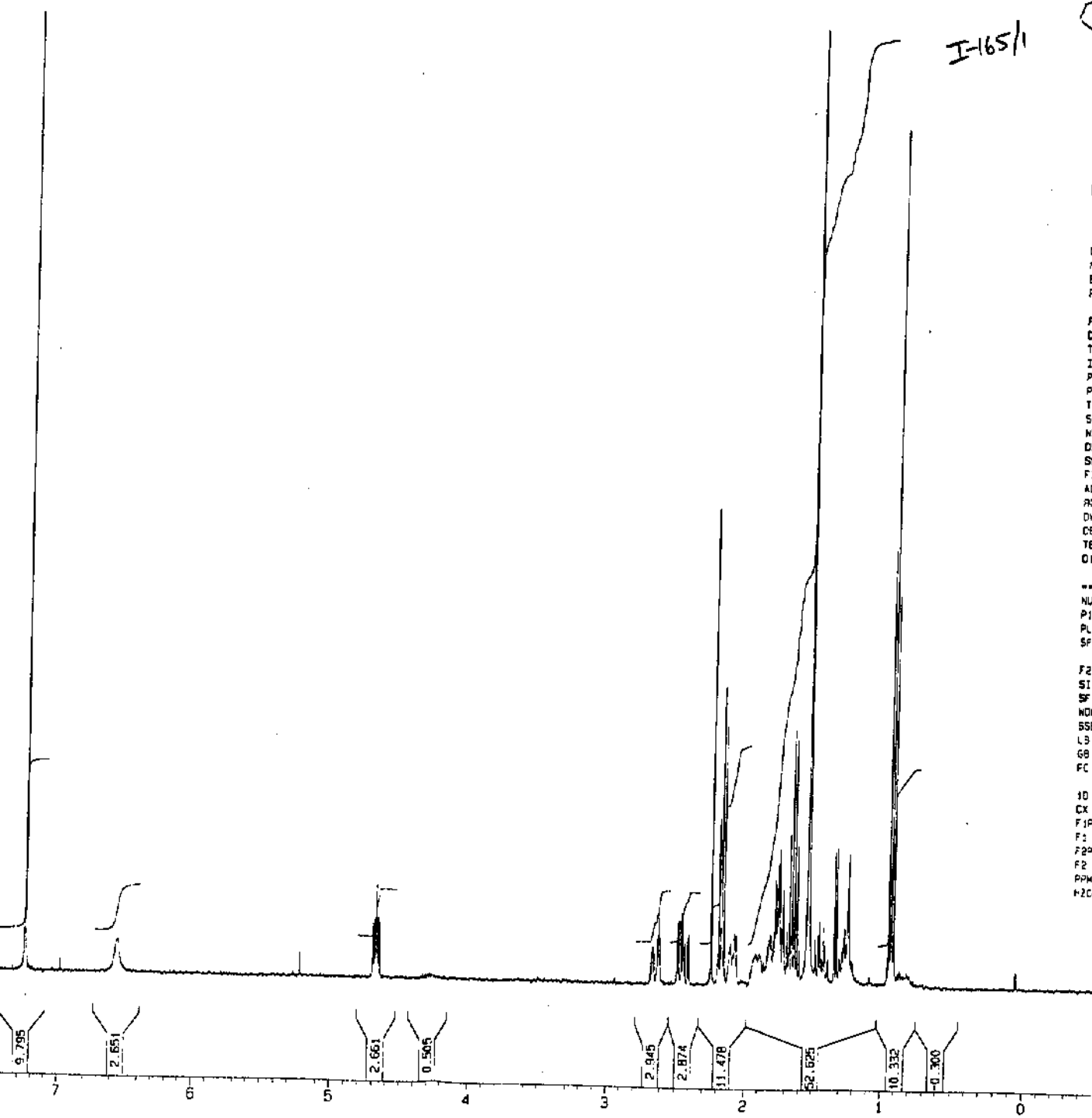

Starcard ARX 6B DRX500 1H
Current Data Parameters
NAME I162/2
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040326
Time 11.28
INSTRUM AV500
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157532 Hz
AQ 3.1720407 sec
RG 512
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.75 usec
PL1 -3.00 dB
SFO1 500.2030889 MHz
F2 - Processing parameters
SI 131072
SF 500.2000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 27.00 cm
CY 13.50 cm
F1P 12.000 ppm
F1 6002.40 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.44444 ppm/cm
HZCM 222.31111 Hz/cm
7.28085
5.66560
4.08645
4.08125
3.84049
3.83119
3.82402
3.81646
3.81056
3.80117
3.63813
3.63256
3.62322
3.61770
3.61147
3.60271
2.22535
2.21101
2.19574
2.05668
1.93728
1.79043
1.79160
1.78598
1.78124
1.77085
1.76660
1.76288
1.75450
1.74735
1.72000
1.71826
1.70480
1.68994
1.67547
1.67364
1.61932
1.60338
1.59509
1.58566
1.58233
1.27599
1.12225
0.99756
0.99086
0.96281
0.96802
0.91411
0.90034
OH
H
Me
O
Integral
0.738
2.323
1.572
11.597
6.195
4.741
ppm
11
10
9
8
7
6
5
4
3
2
1


13C
174.624
78.973
77.261
77.007
76.753
69.250
38.642
34.048
31.456
29.697
28.366
24.906
22.905
19.164
13.679
Current Data Parameters
NAME 1162/2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040326
Time 10.37
INSTRUM AV500
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 671
DS 4
SWH 35211.270 Hz
FIDRES 0.537281 Hz
AQ 0.9306754 sec
RG 16384
DW 14.200 usec
DE 6.00 usec
TE 300.0 K
D1 4.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.45 usec
PL1 4.00 dB
SFO1 125.7892253 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 65.00 usec
PL2 4.00 dB
PL12 13.00 dB
PL13 13.00 dB
SFO2 500.2020028 MHz
F2 - Processing parameters
SI 65536
SF 125.7753900 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 27.00 cm
CY 200.00 cm
F1P 250.000 ppm
F1 31443.85 Hz
F2P -25.000 ppm
F2 -3144.39 Hz
PPMCM 10.18510 ppm/cm
HZCM 1281.04565 Hz/cm
ppm
225
200
175
150
125
100
75
50
25
0

I-162/2



c:\pel_data\spectra\drs-i-~3.sp



V62897
DS/I-165-1
David Spring
1H
CDCl3
Position: 14

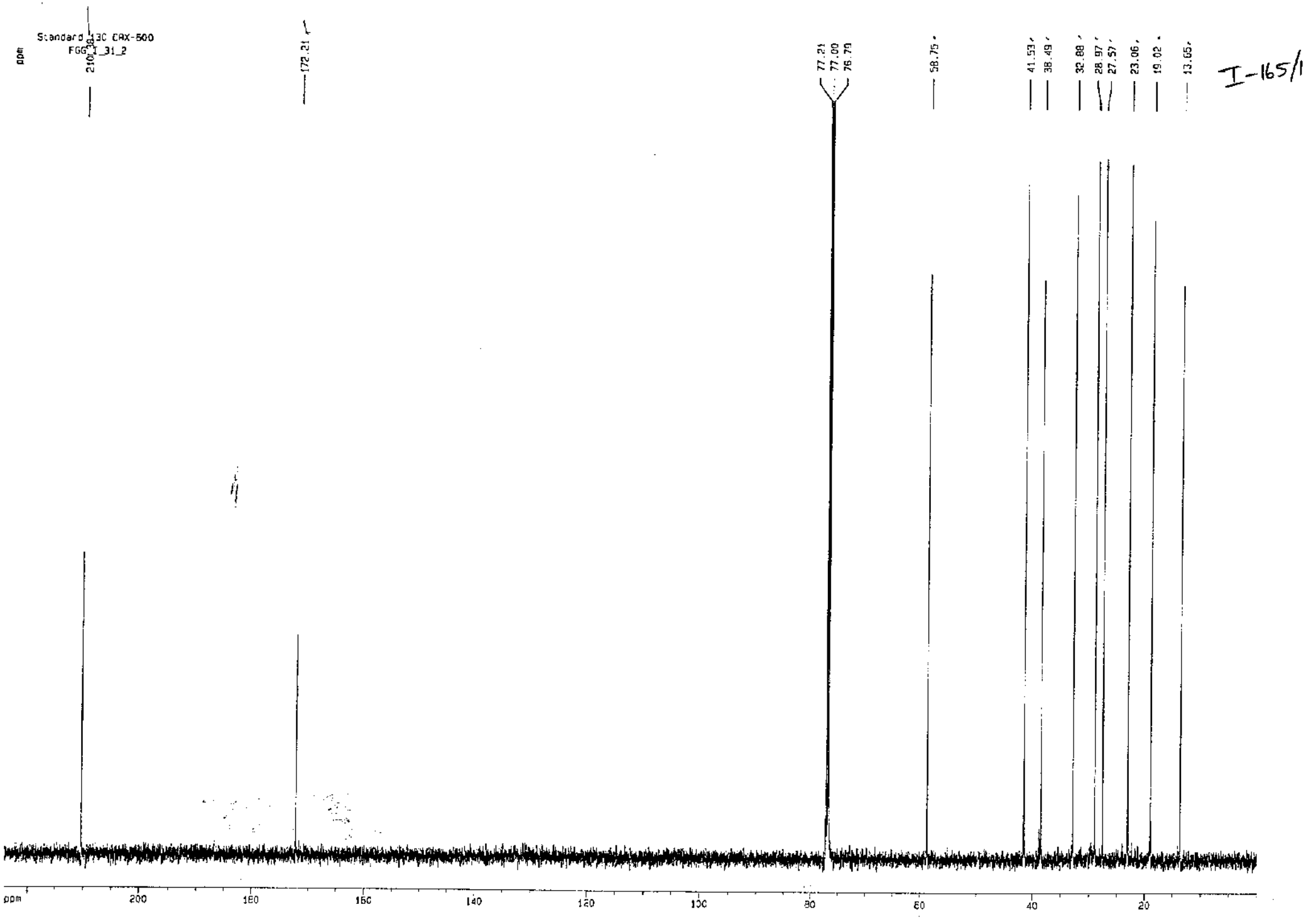
Current Data Parameters
NAME V62897
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040402
Time 17.38
INSTRUM cpx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 912.3
DW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

========== CHANNEL f1 =====
NUC1 1H
P1 9.40 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

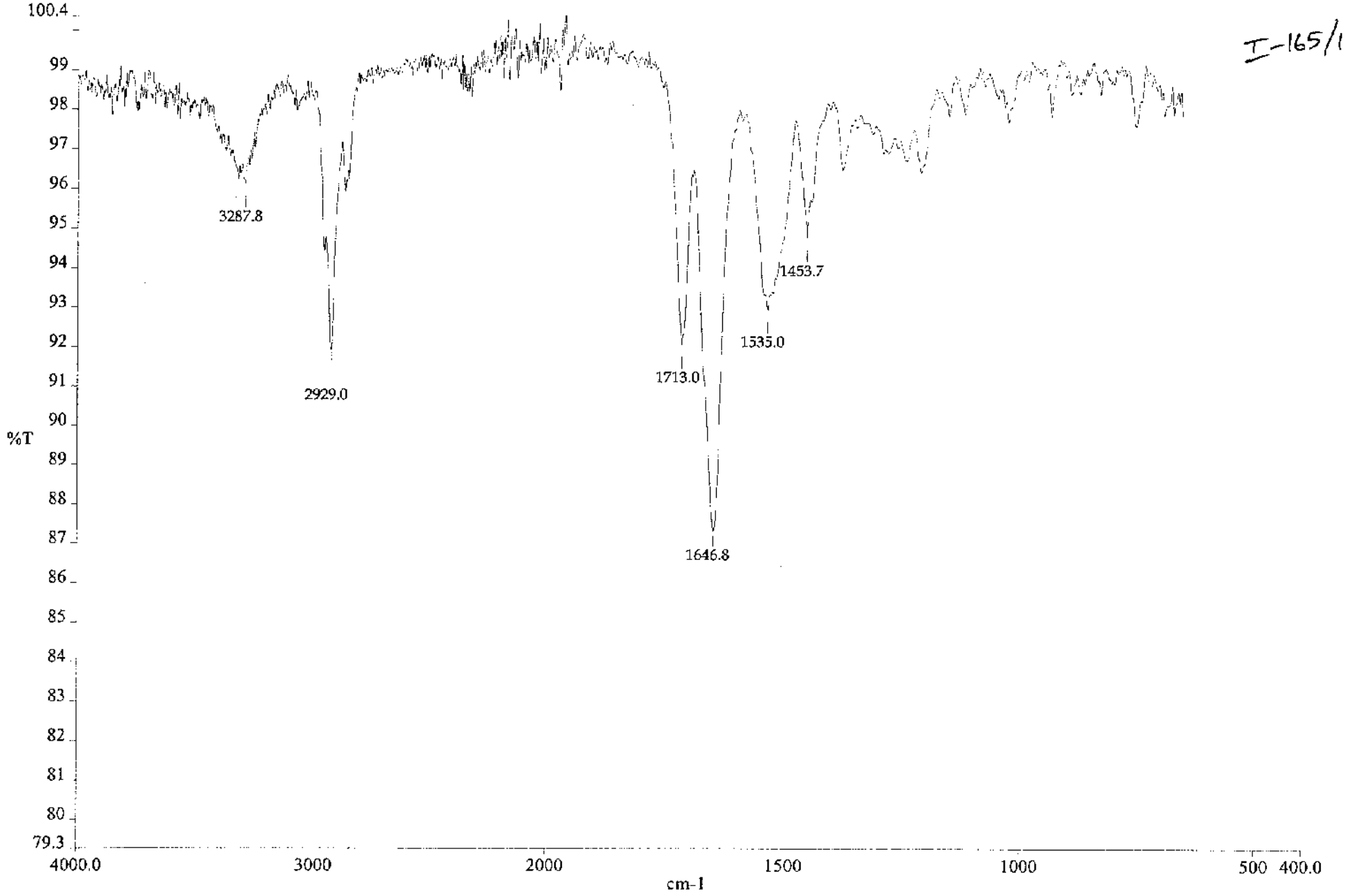
F2 - Processing parameters
SI 32768
SF 400.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 35.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.30000 ppm/cm
HZCM 120.03900 Hz/cm

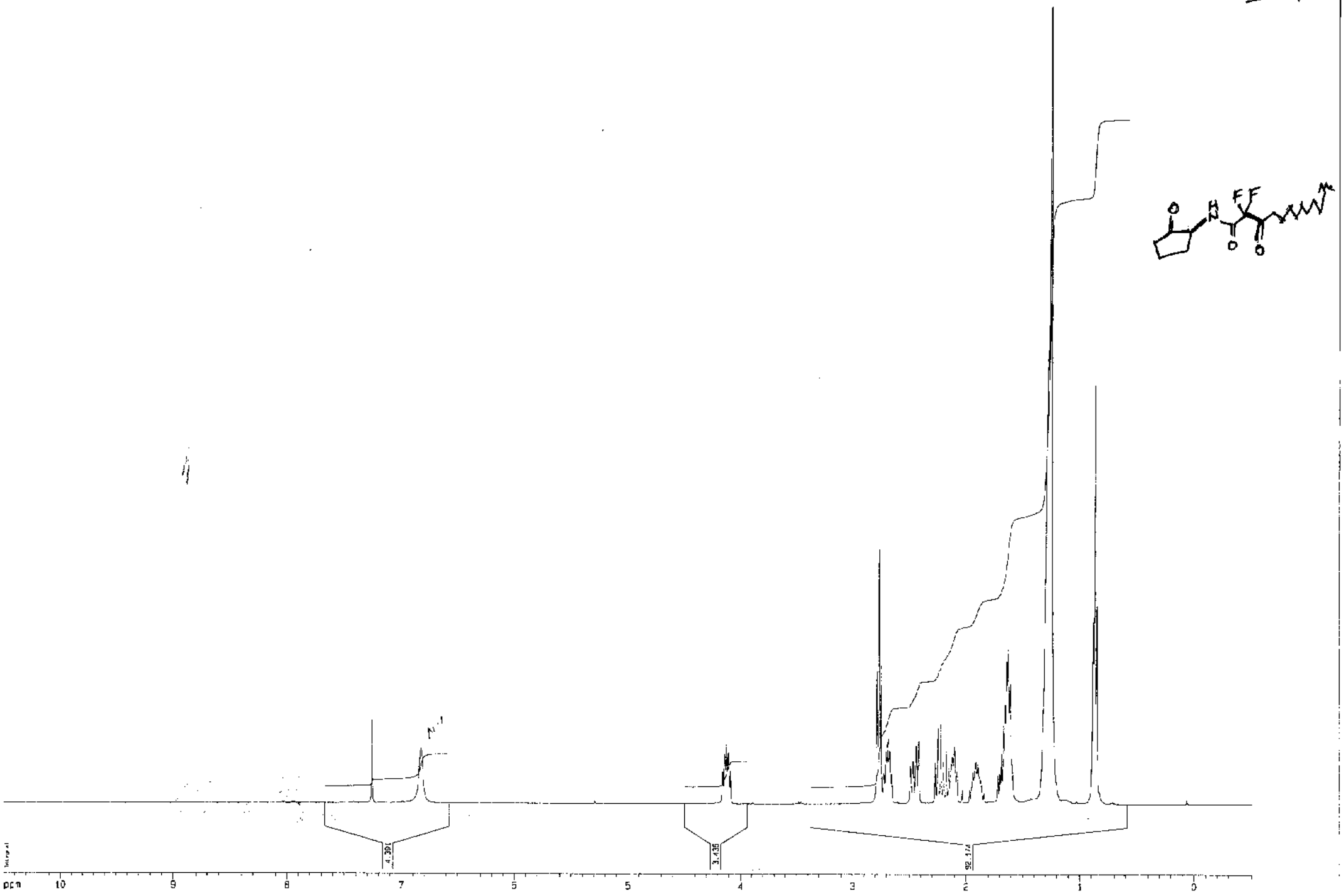

Standard 13C CRX-500
FGG_I_31_2
ppm
210.38
172.21
77.21
77.00
76.79
58.75
41.53
38.49
32.88
28.97
27.57
23.06
19.02
13.65
I-165/1
200
180
160
140
120
100
80
60
40
20

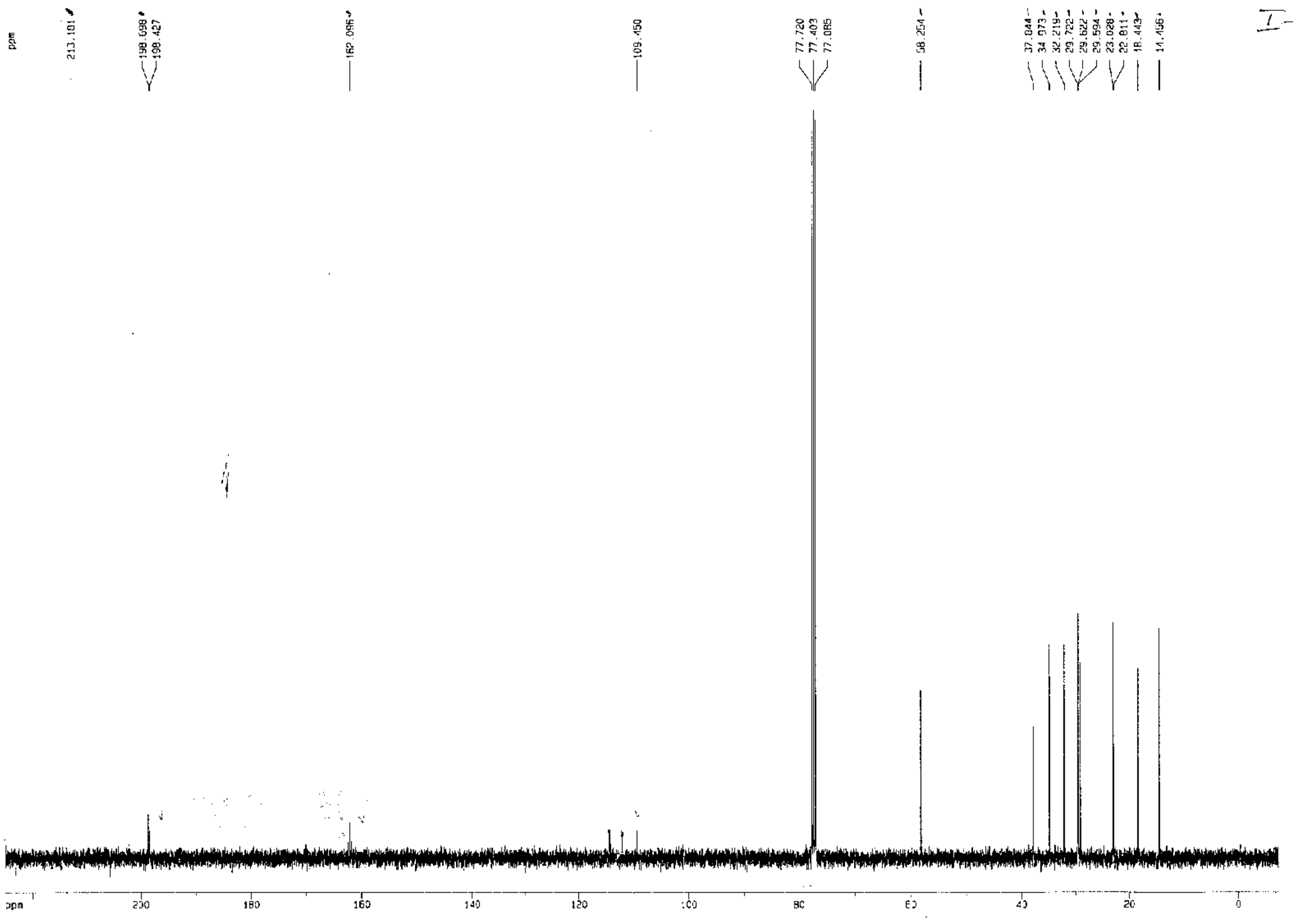
FGG-I-31/2

I-165/1



I-107/2




I-107/2
ppm
213.191
198.698
198.427
162.096
109.450
77.720
77.403
77.085
58.254
37.844
34.973
32.219
29.722
29.622
29.594
23.028
22.811
18.443
14.496
ppm
200
180
160
140
120
100
80
60
40
20
0

DPX400 fluorine
fluorineCPD.std CDCl3 /disk2 service 62

I-107/2

^{19}F NMR

Current Data Parameters
NAME ayl-ds-107-2
EXPNO 10
PROCNO 1

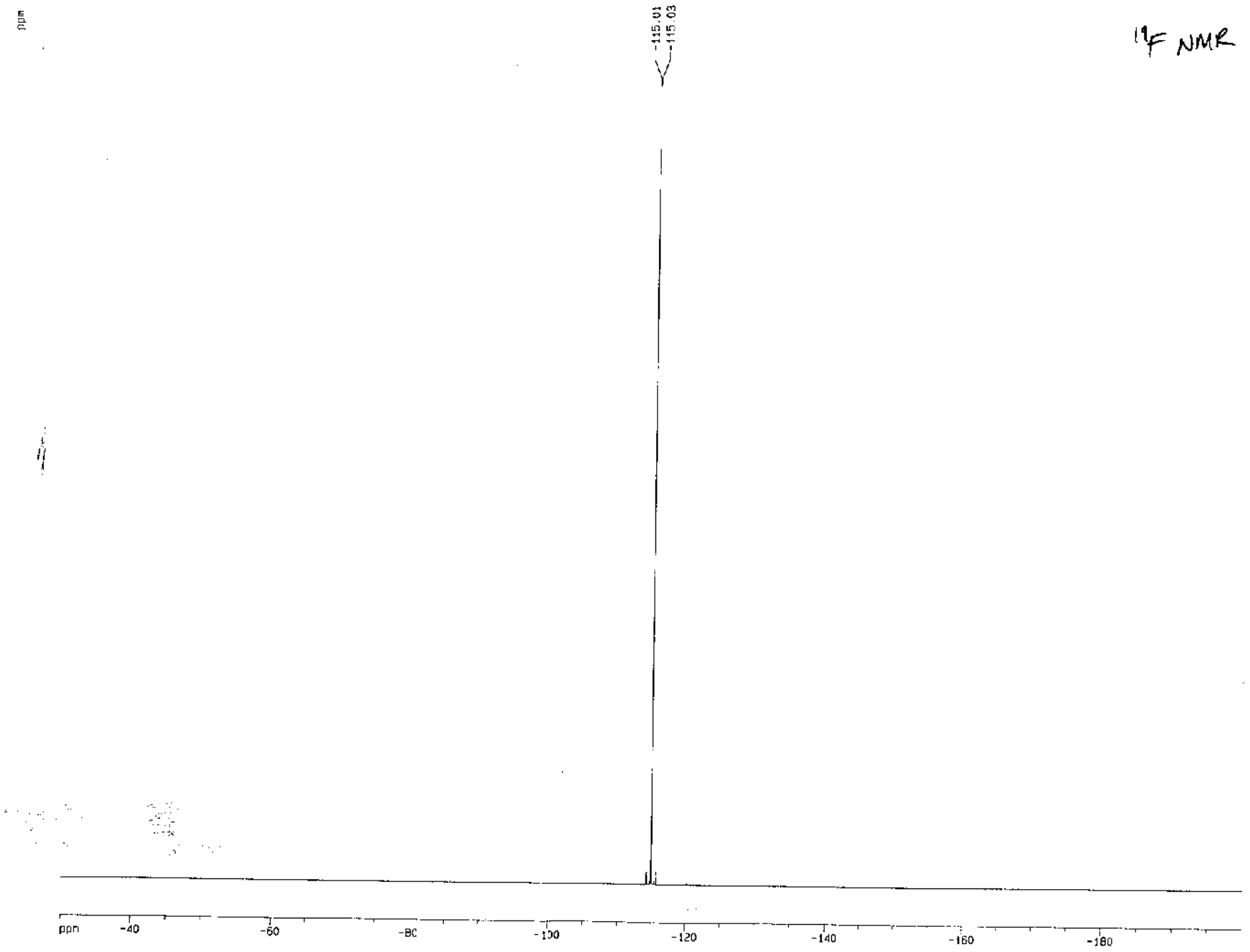
F2 - Acquisition Parameters
Date_ 20120701
Time 18.08
INSTRUM dpx400
PROBHD 5 mm SNP 1H
PULPROG zgfhigqn
TD 262144
SOLVENT CDCl3
NS 256
DS 5
SWH 25187.963 Hz
FIDRES 0.256813 Hz
AQ 1.7439076 sec
RG 512
DW 6.650 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

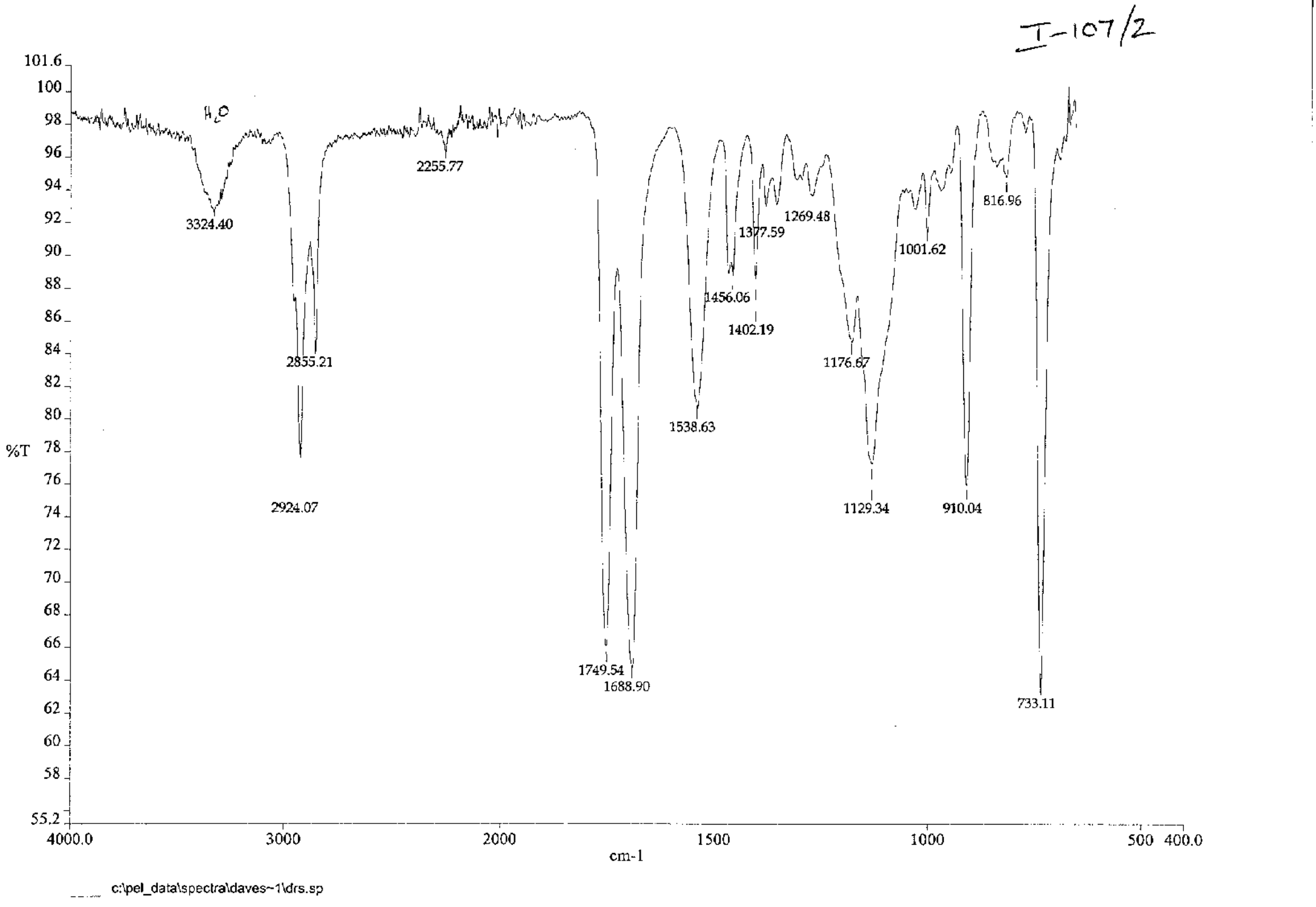
========== CHANNEL f1 ==========
NUC1 19F
P1 13.00 usec
PL1 -6.02 dB
SFO1 376.4495892 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 262144
SF 376.4984577 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 38.00 cm
F1P 16.000 ppm
F1 3764.98 Hz
F2P -200.000 ppm
F2 -75299.60 Hz
PPMCM 5.52632 ppm/cm
HZCM 2080.64917 Hz/cm




I-107/2
H2O
3324.40
2855.21
2924.07
2255.77
1749.54
1688.90
1538.63
1456.06
1402.19
1377.59
1269.48
1176.67
1129.34
1001.62
910.04
816.96
733.11
%T
cm-1
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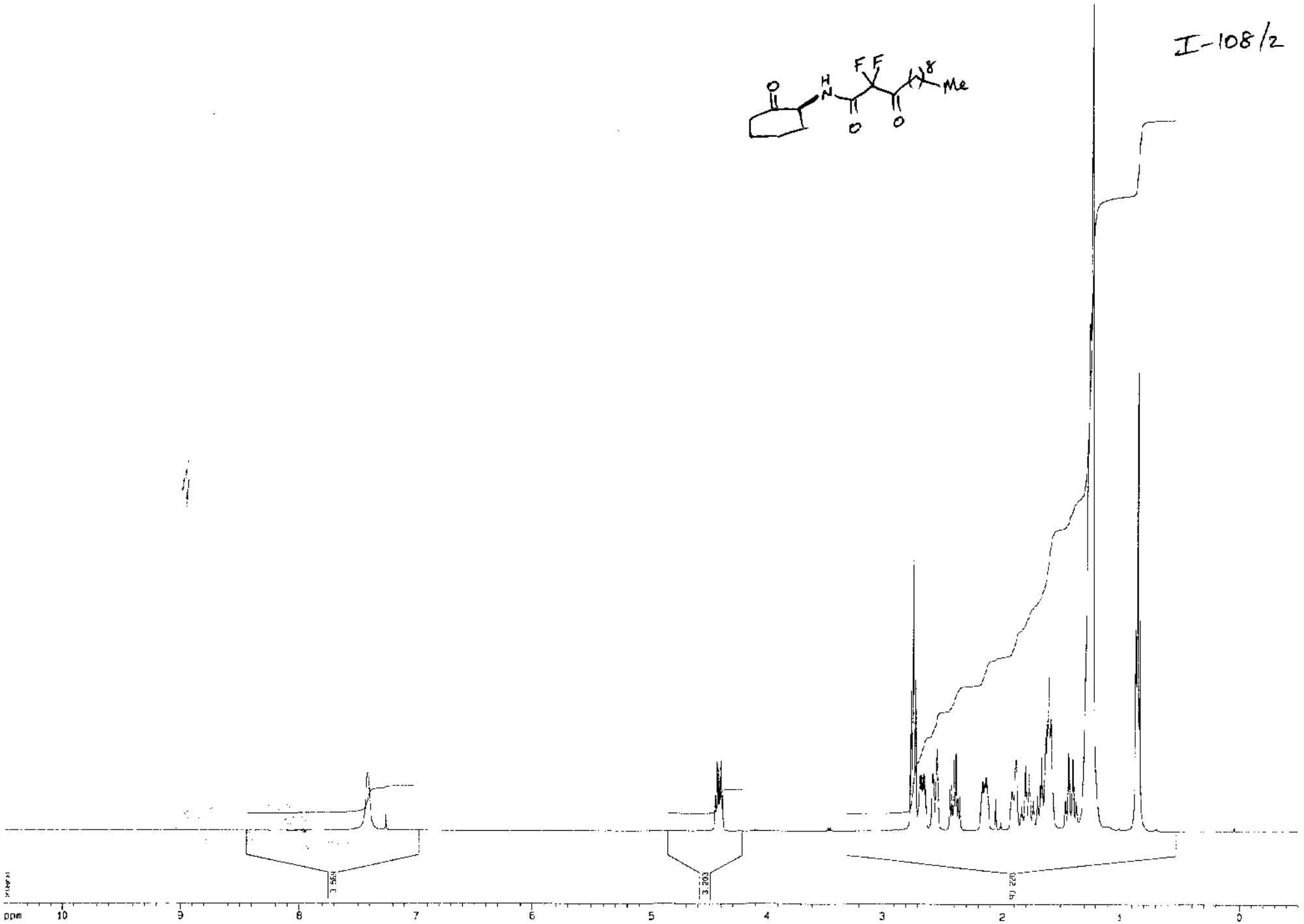
I-108/2



I-108/2

^{19}F NMR

DPX400 fluorine
fluorineCPD.std CDCl3 /disk2 service 63

ppm

-115.12
-115.15

Current Data Parameters
NAME sv1-ds-108-2
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020701
Time 18.25
INSTRUM dpx400
PROBHD 5 mm QNP 1H
PULPROG zgfhigqn
TD 262144
SOLVENT CDCl3
NS 256
DS 0
SWH 75187.969 Hz
FIDRES 0.286819 Hz
AQ 1.7431076 sec
RG 512
DW 6.650 usec
DE 6.00 usec
TE 303.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

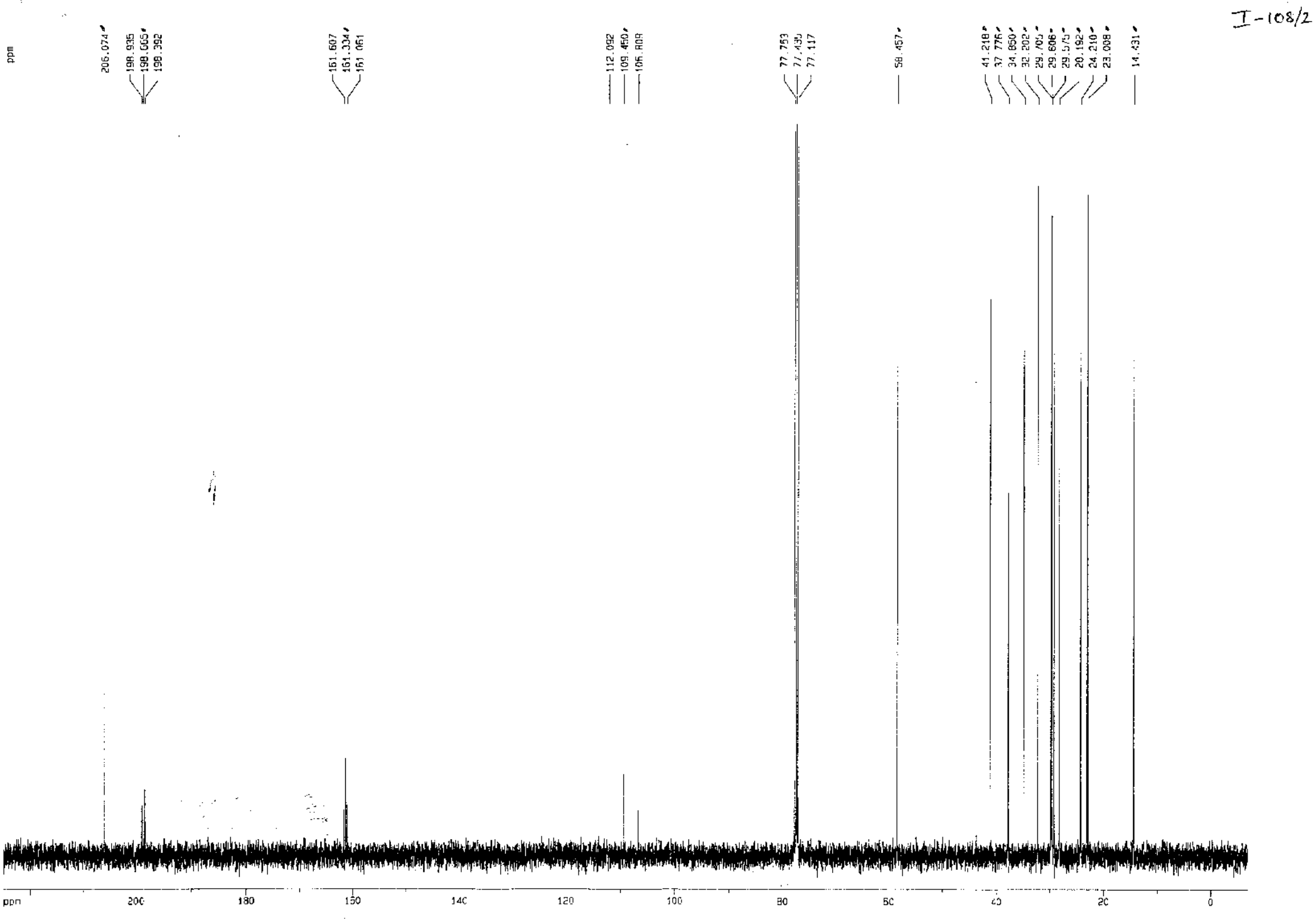
------------ CHANNEL f1 ------------
NUC1 19F
P1 13.00 usec
PL1 -6.00 dB
SFO1 376.4455892 MHz

------------ CHANNEL f2 ------------
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 20.00 dB
SFO2 400.1316005 MHz

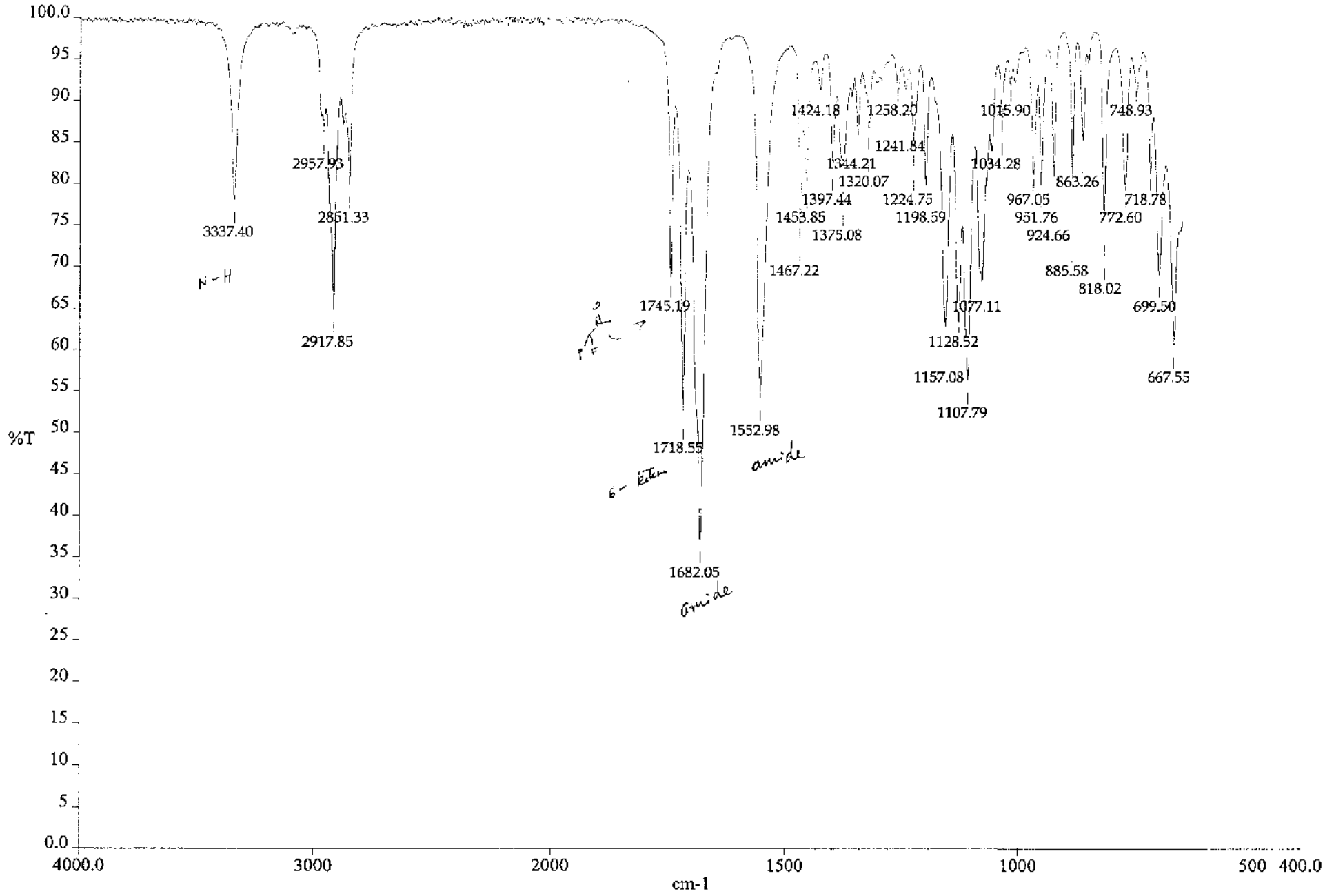
F2 - Processing parameters
SI 262144
SF 376.4984577 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

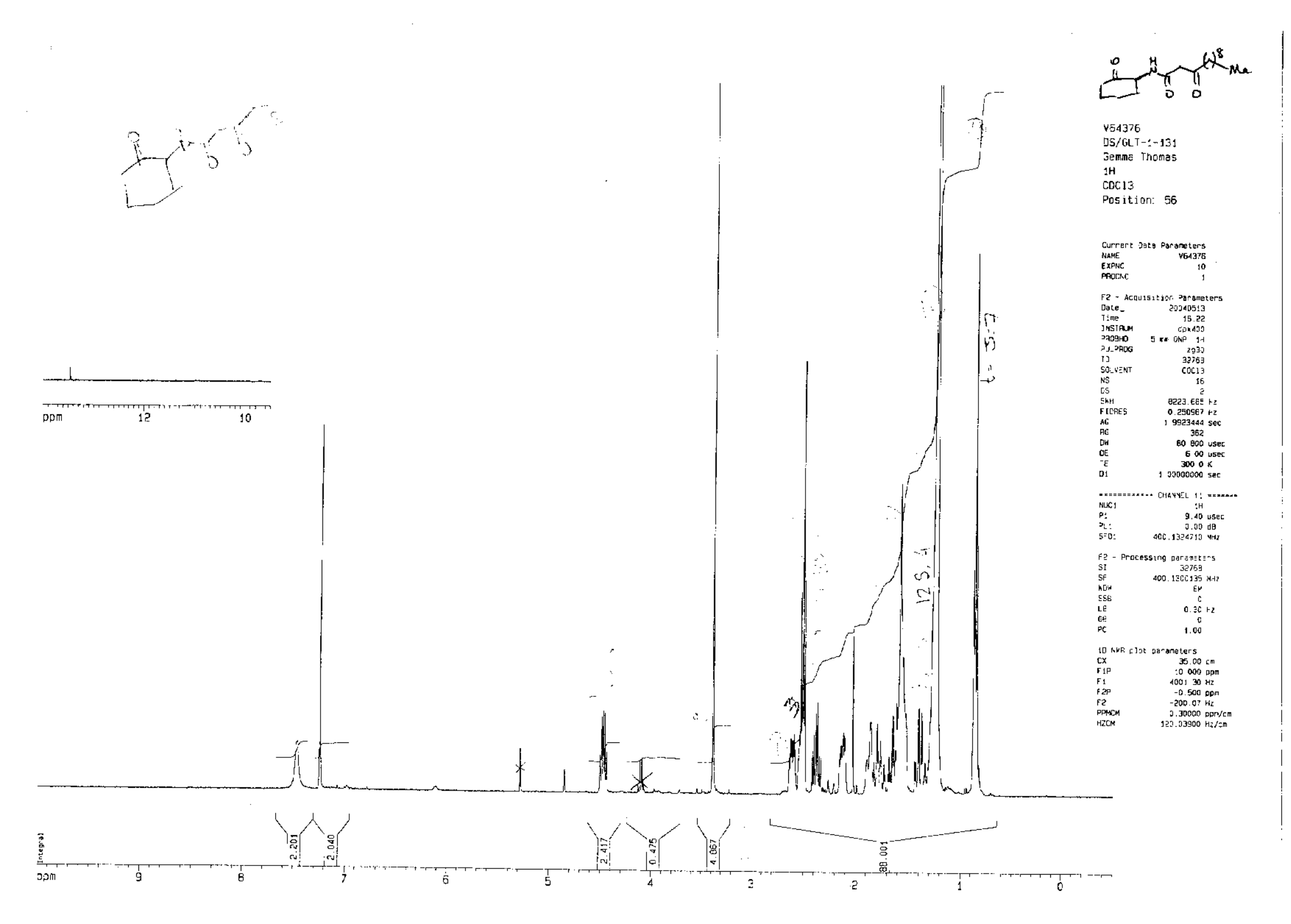
1D NMR plot parameters
CX 38.00 cm
F1P -77.325 ppm
F1 -29131.68 Hz
F2P -154.760 ppm
F2 -58267.01 Hz
PPMCM 2.03645 ppm/cm
HZCM 766.21936 Hz/cm

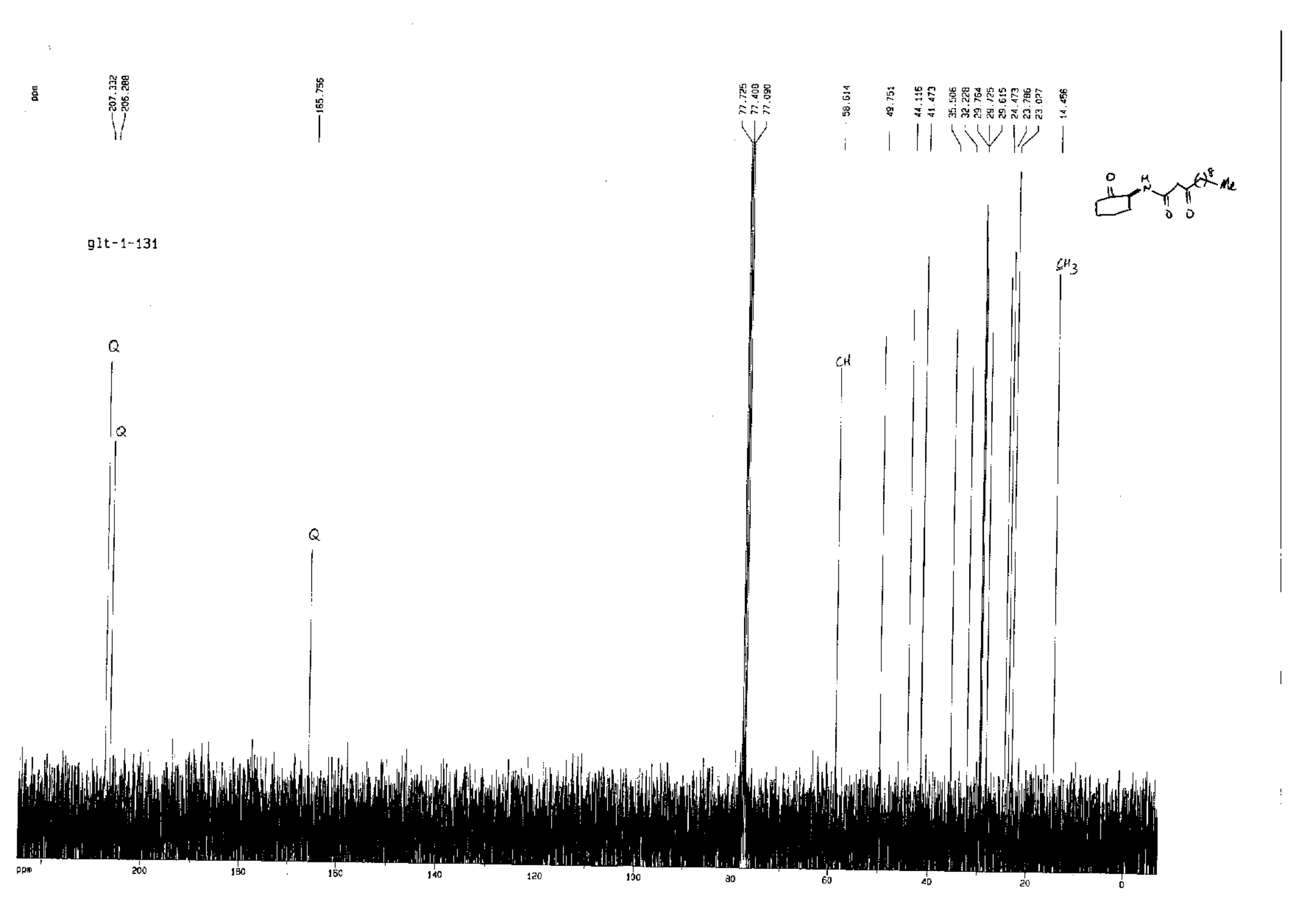
ppm -80 -90 -100 -110 -120 -130 -140 -150

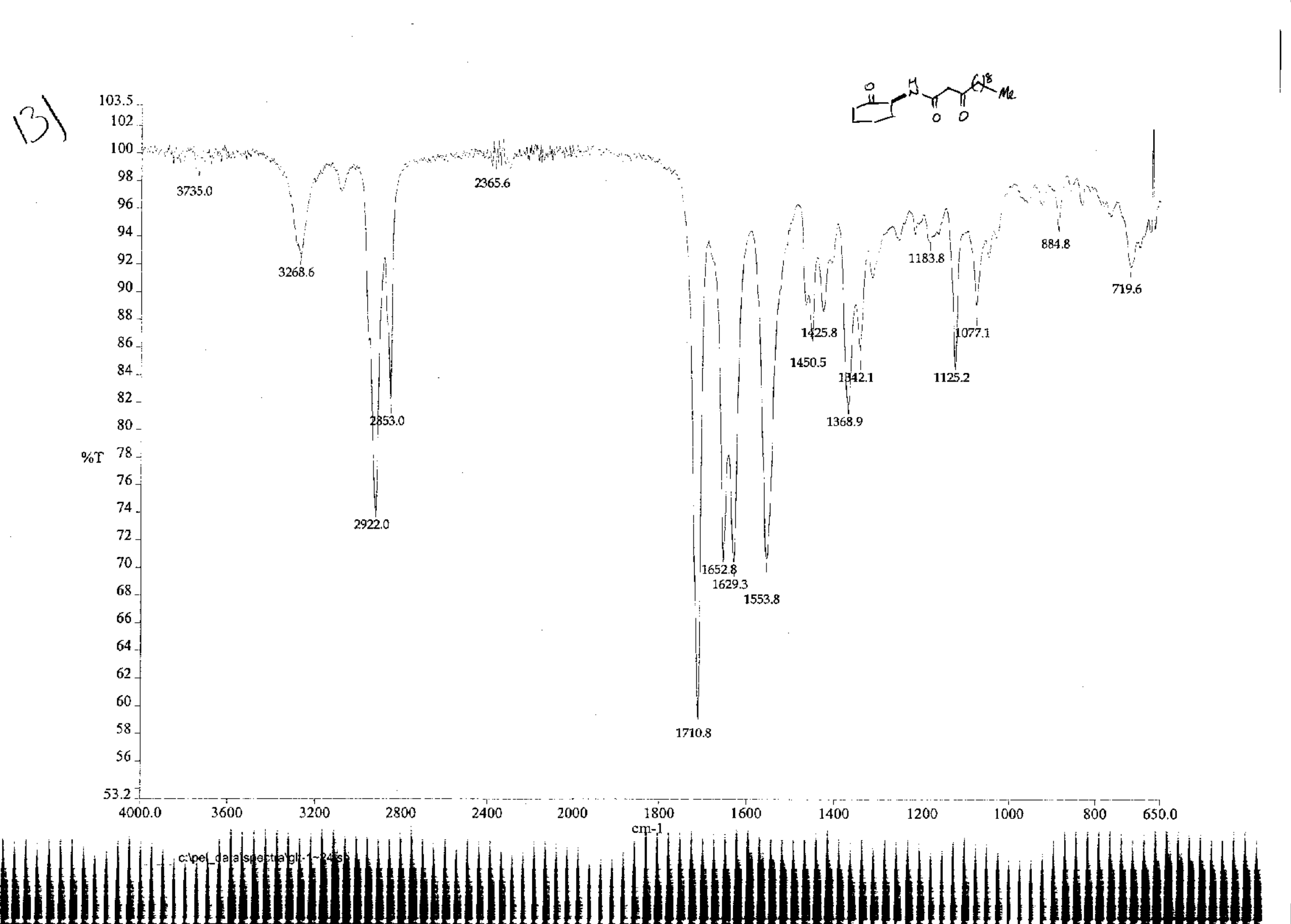

I-108/2
ppm
206.074
198.935
198.665
198.392
161.607
161.334
161.061
112.092
109.450
106.809
77.753
77.436
77.117
58.457
41.218
37.776
34.850
32.202
29.705
29.606
29.575
20.192
24.210
23.008
14.431
ppm
200
180
160
140
120
100
80
60
40
20
0

I-108/2

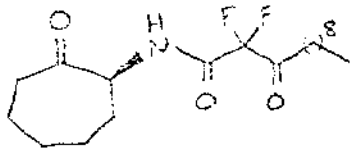

100.0
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0.0
%T
4000.0
3000
2000
1500
1000
500
400.0
cm-1
3337.40
N-H
2957.93
2851.33
2917.85
1745.19
1718.55
6-ketone
1682.05
amide
1552.98
amide
1467.22
1453.85
1424.18
1397.44
1375.08
1344.21
1320.07
1258.20
1241.84
1224.75
1198.59
1157.08
1128.52
1107.79
1077.11
1034.28
1015.90
967.05
951.76
924.66
885.58
863.26
818.02
772.60
748.93
718.78
699.50
667.55
c:\pel_data\spectra\drs.sp


V64376
DS/GLT-1-131
Gemma Thomas
1H
CDCl3
Position: 56
Current Data Parameters
NAME V64376
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040513
Time 15.22
INSTRUM cpx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 362
DW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 9.40 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 35.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.30000 ppm/cm
HZCM 120.03900 Hz/cm
ppm
12
10
Integral
2.201
2.040
2.417
0.475
4.067
88.001
9
8
7
6
5
4
3
2
1
0


ppm
207.332
206.288
165.756
77.725
77.408
77.090
58.614
49.751
44.116
41.473
35.506
32.228
29.764
29.125
29.615
24.473
23.786
23.077
14.456
glt-1-131
Q
Q
Q
CH
CH3
Me
ppm
200
180
160
140
120
100
80
60
40
20
0


3735.0
3268.6
2922.0
2853.0
2365.6
1710.8
1652.8
1629.3
1553.8
1450.5
1425.8
1368.9
1342.1
1183.8
1125.2
1077.1
884.8
719.6
%T
cm-1
4000.0
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
650.0
103.5
53.2
Me

I-33/2



V53615
DS/FGG/I-33/2
Freija Glansdorp
1H
CDCl3
Position: 54

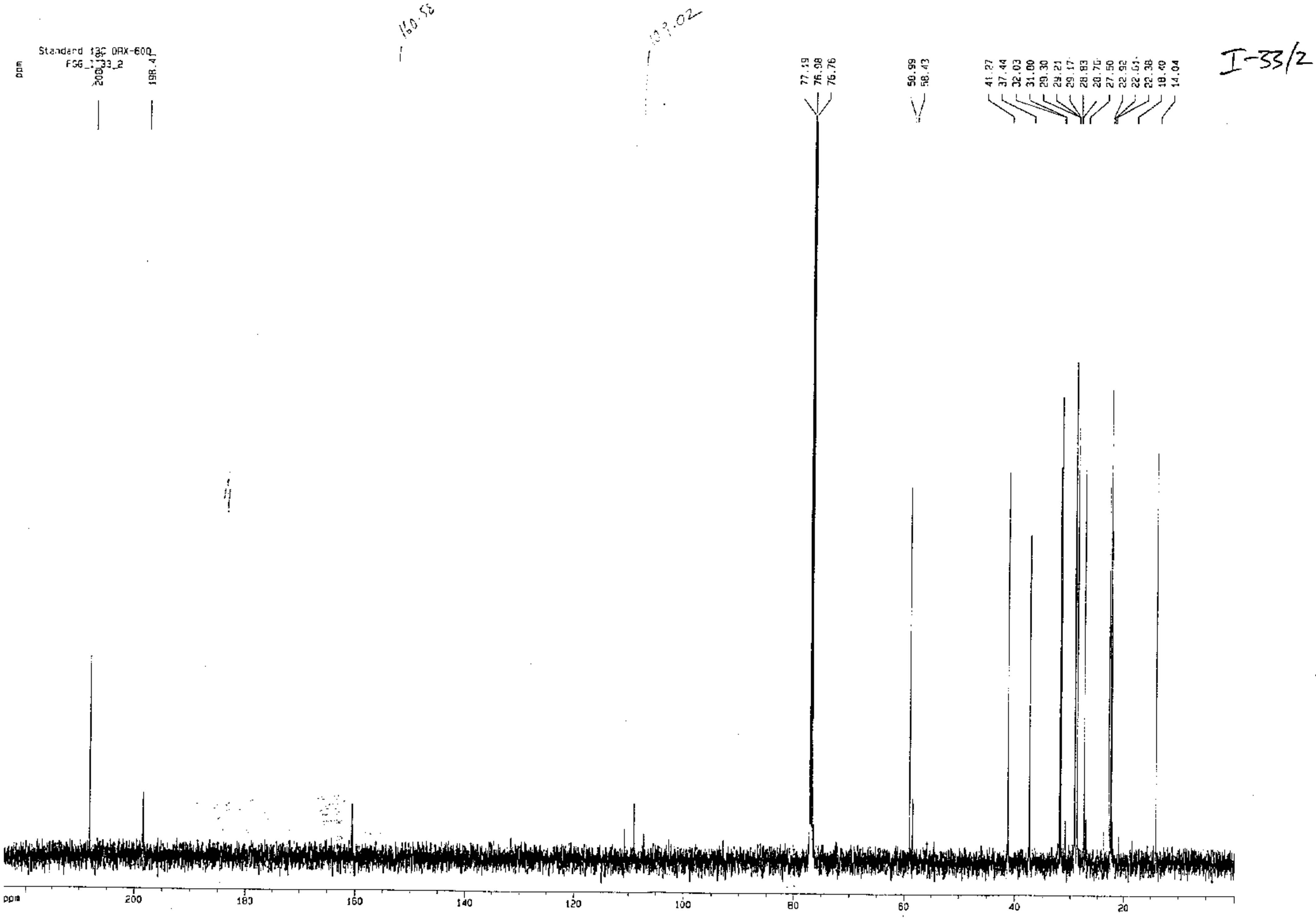
Current Data Parameters
NAME V53615
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030909
Time 14.45
INSTRUM dpx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 287.4
DW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

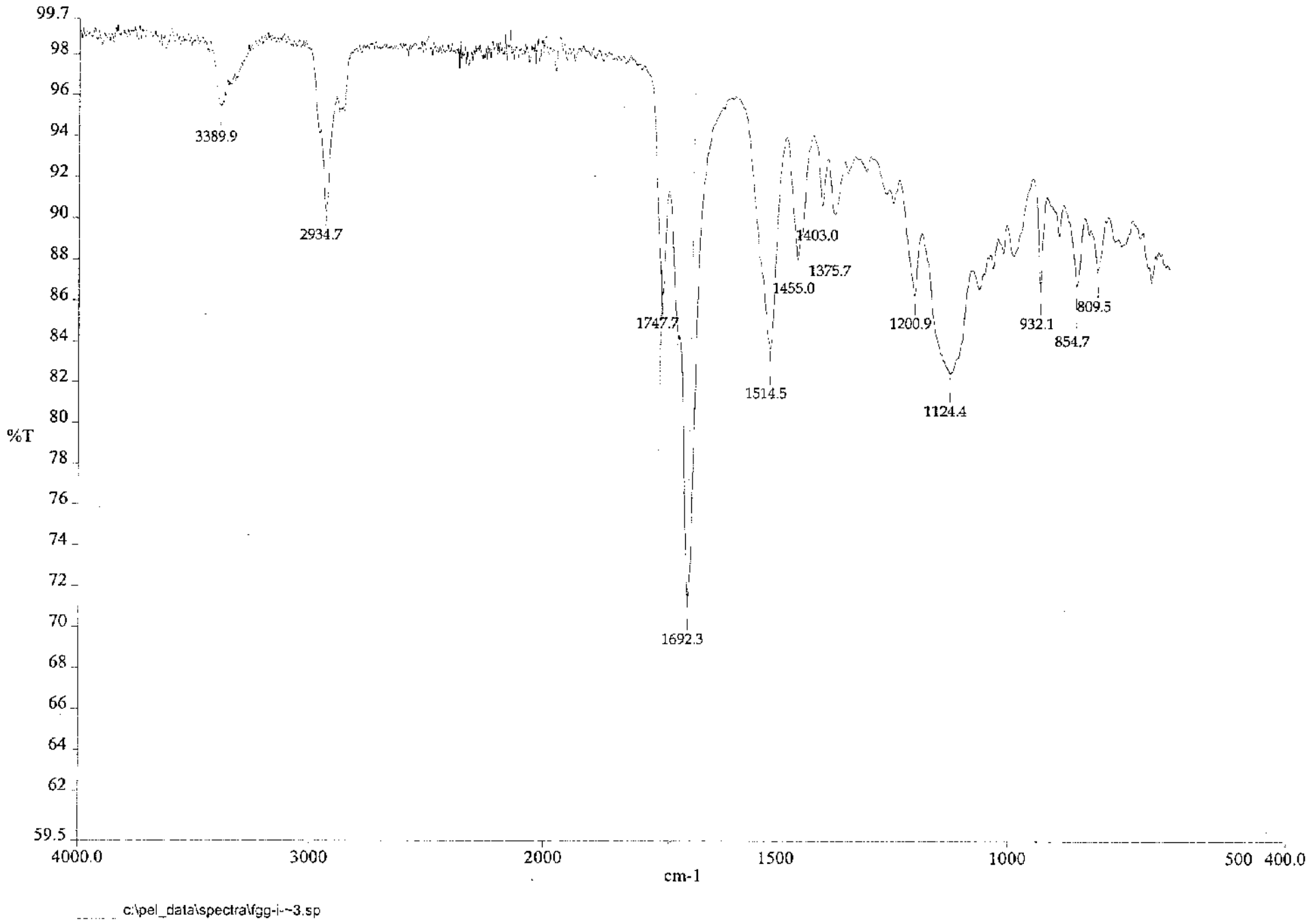
========== CHANNEL f1 =======
NUC1 1H
P1 9.40 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

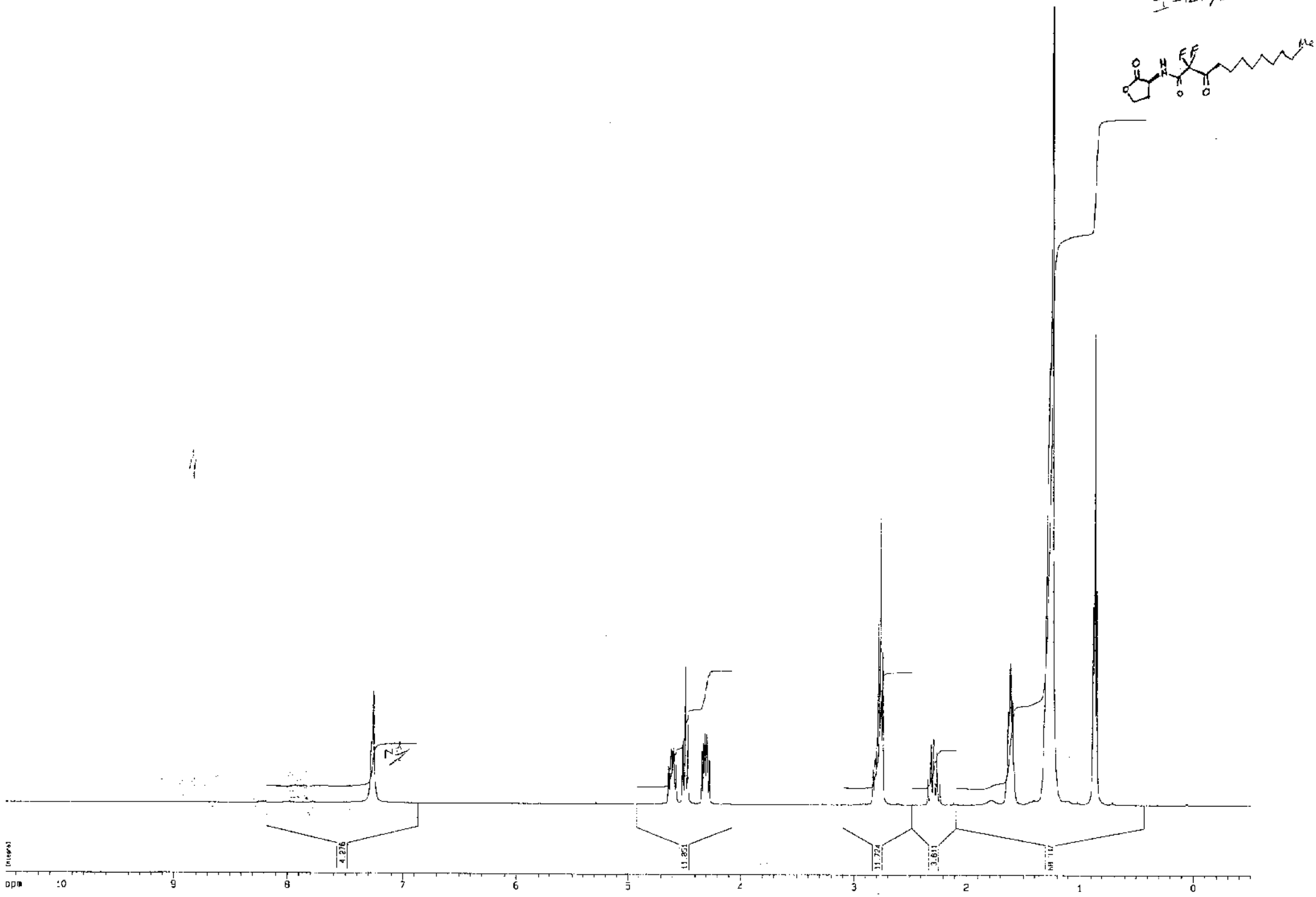
F2 - Processing parameters
SI 32768
SF 400.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

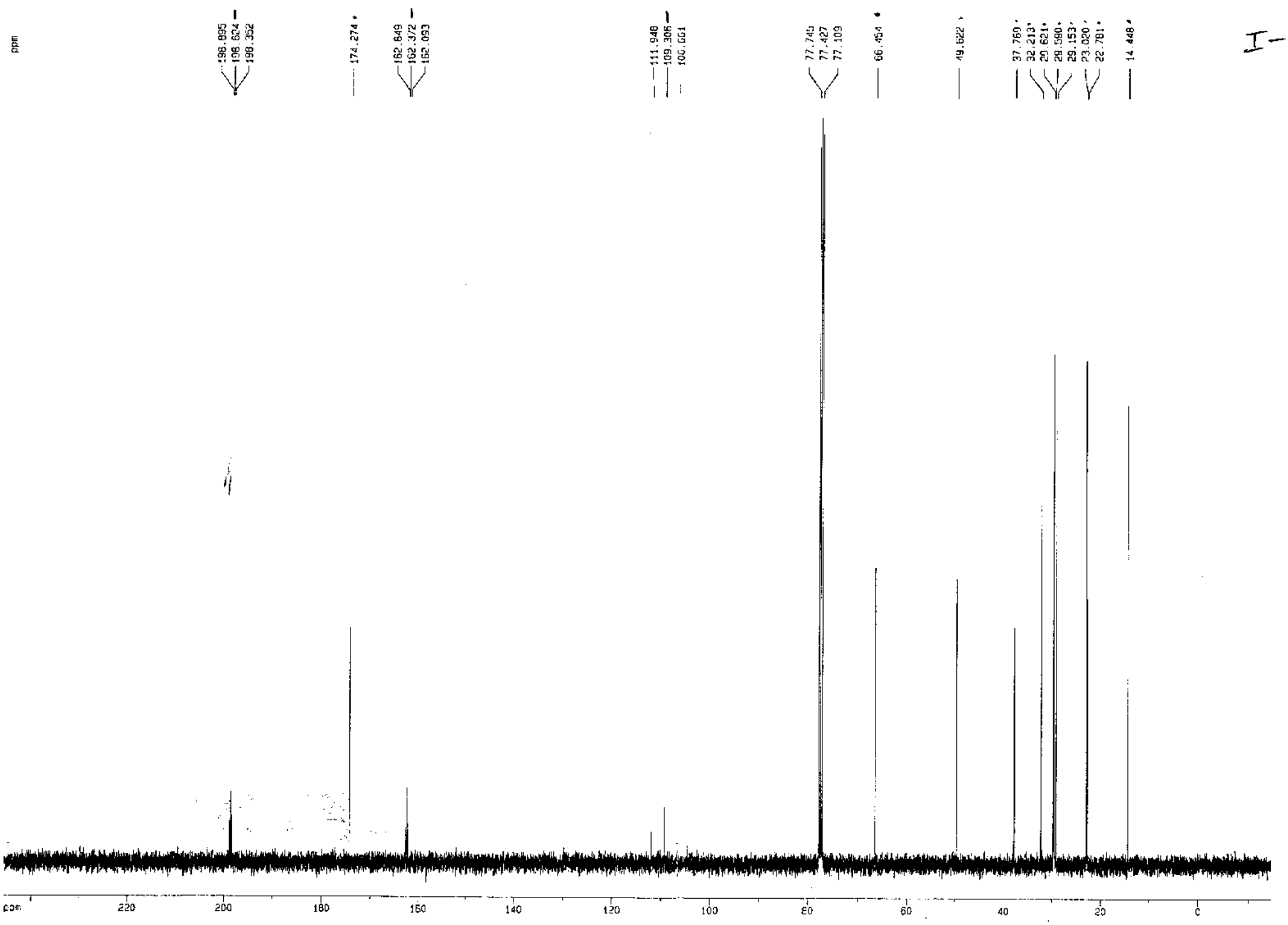
1D NMR plot parameters
CX 35.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.30000 ppm/cm
HZCM 120.03900 Hz/cm


Standard 13C DRX-600_
ppm
198.47
160.53
109.02
77.19
76.98
76.76
58.99
58.43
41.27
37.44
32.03
31.80
29.30
29.21
29.17
28.83
28.70
27.50
22.92
22.61
22.38
18.49
14.04
I-33/2
ppm
200
180
160
140
120
100
80
60
40
20

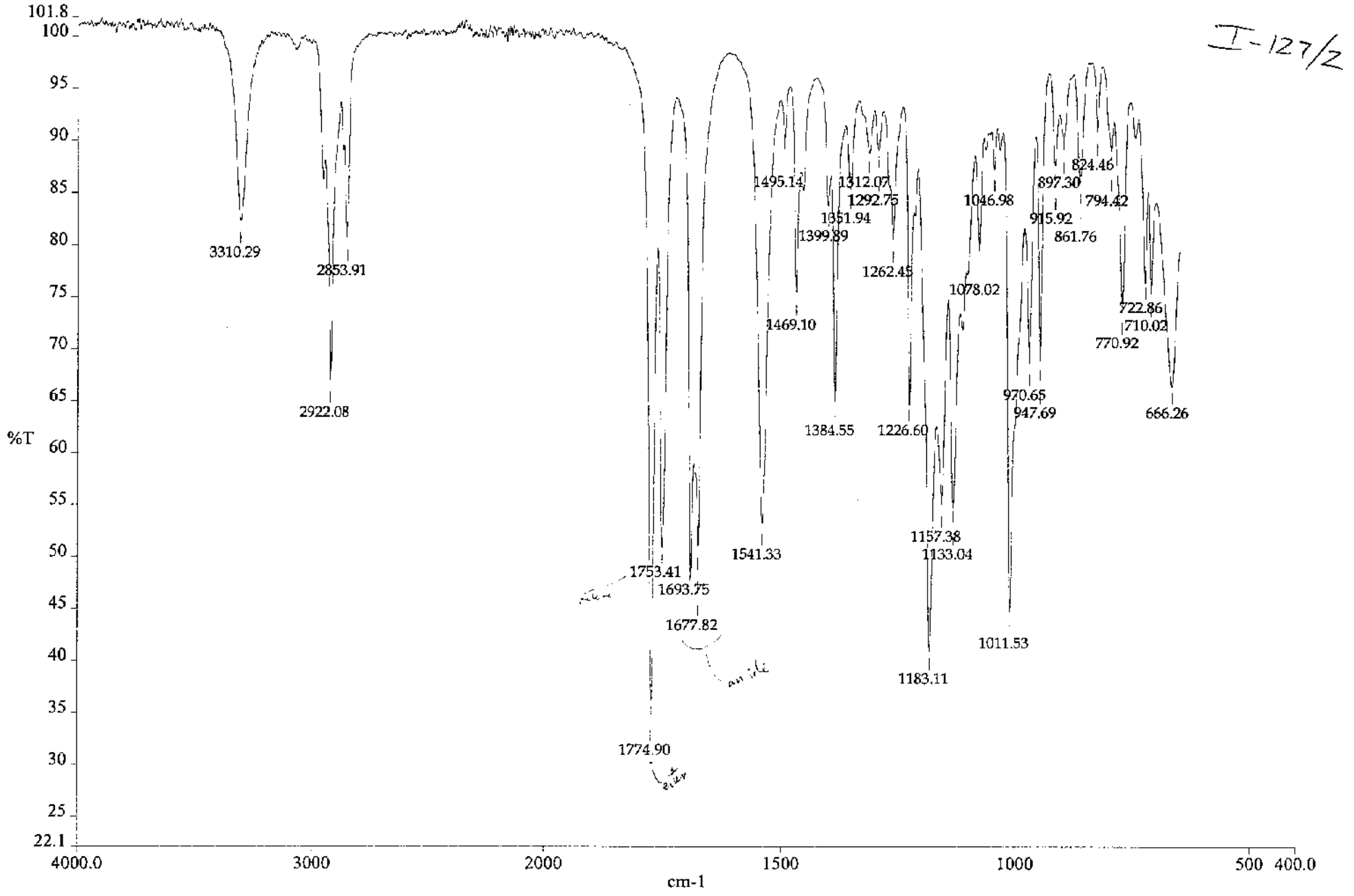
FGG-I-33/2

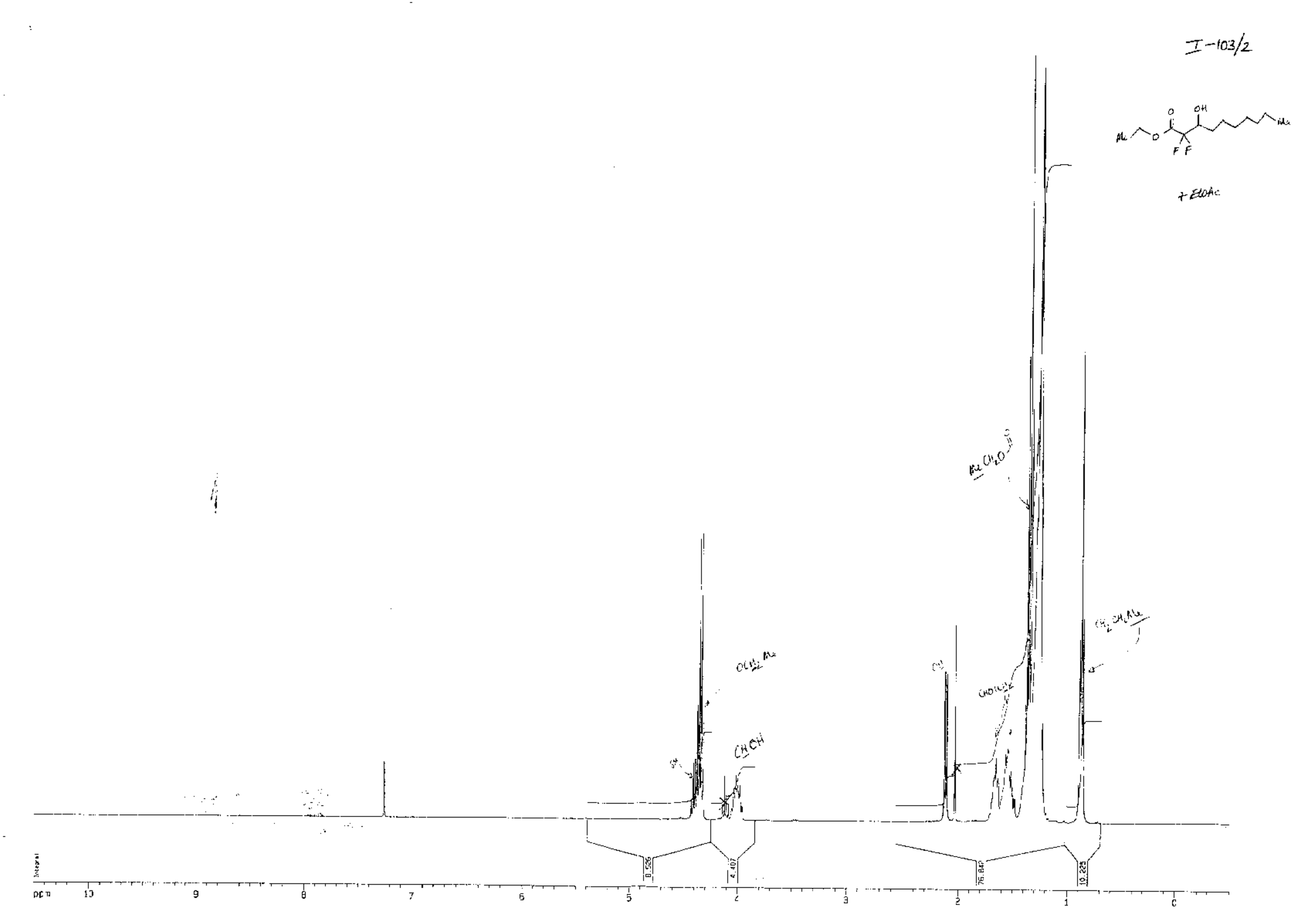

99.7
98
96
94
92
90
88
86
84
82
80
78
76
74
72
70
68
66
64
62
59.5
%T
3389.9
2934.7
1747.7
1692.3
1514.5
1455.0
1403.0
1375.7
1200.9
1124.4
932.1
854.7
809.5
4000.0
3000
2000
1500
1000
500
400.0
cm-1
c:\pel_data\spectra\fgg-i-~3.sp

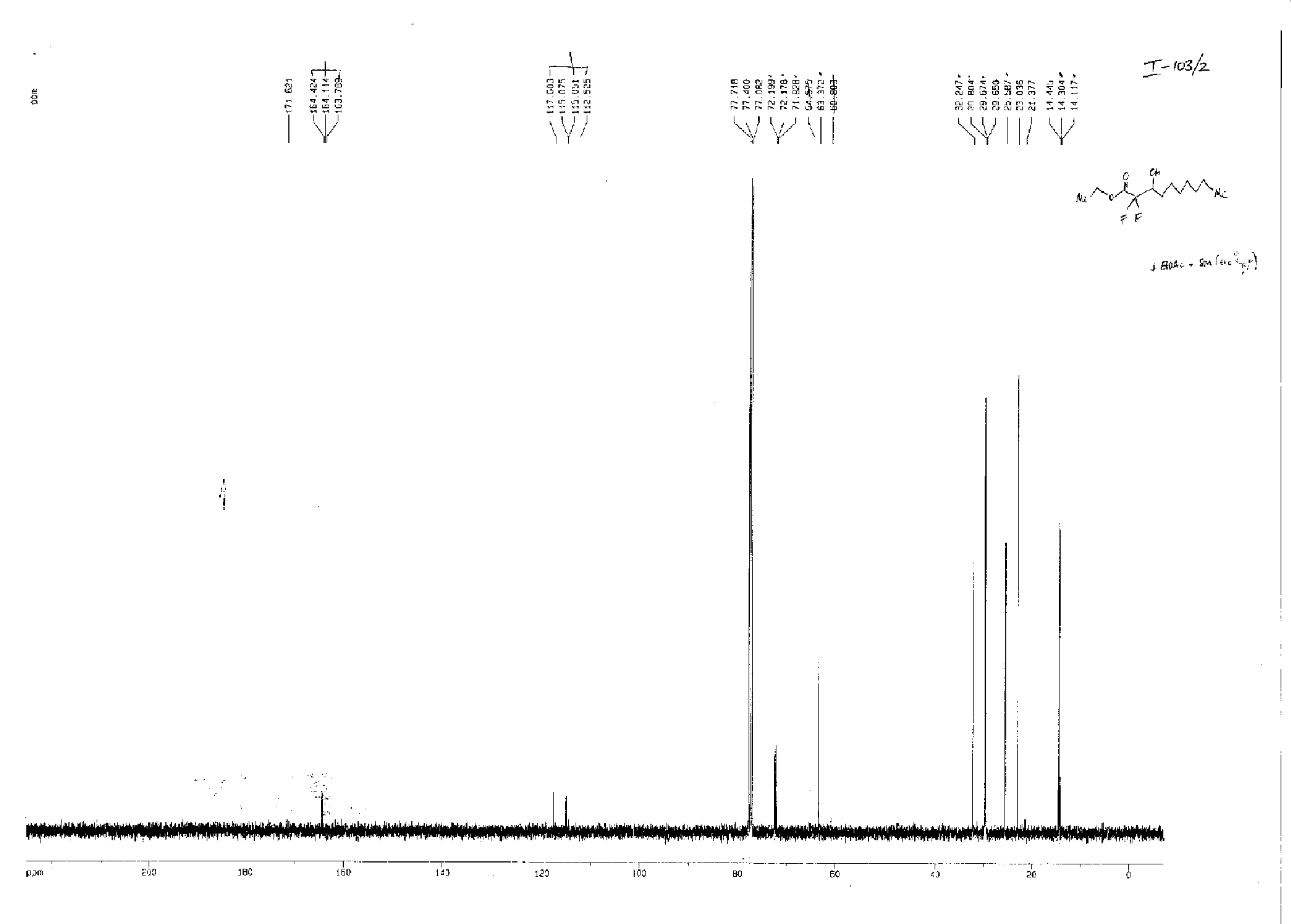

I-127/2
4.276
11.801
11.724
3.611
ppm


ppm
198.895
198.624
198.352
174.274
162.649
162.372
162.093
111.948
109.306
106.661
77.745
77.427
77.109
66.454
49.622
37.769
32.213
29.621
29.590
29.153
23.020
22.781
14.448
220
200
180
160
140
120
100
80
60
40
20
0

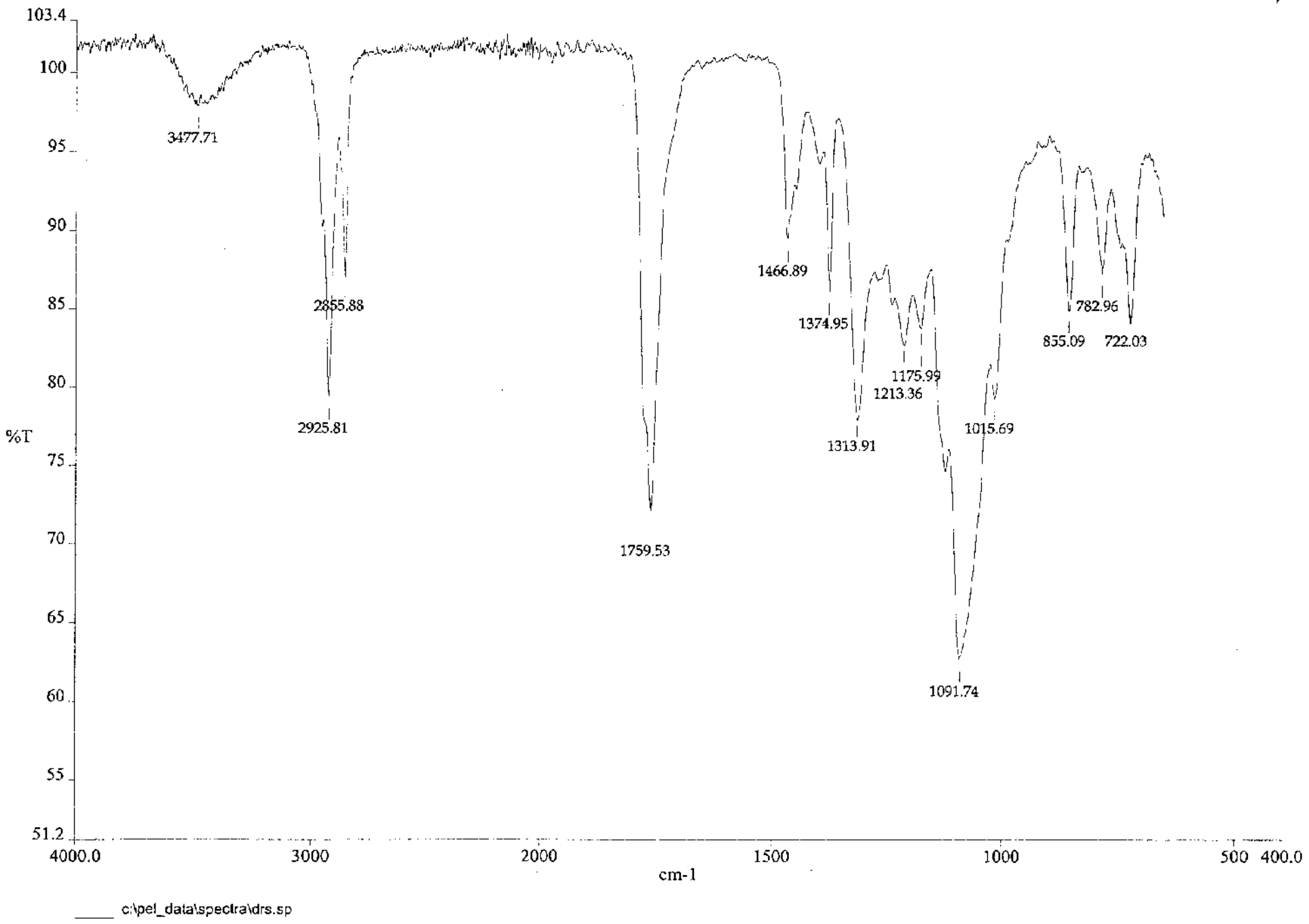
I-127/2

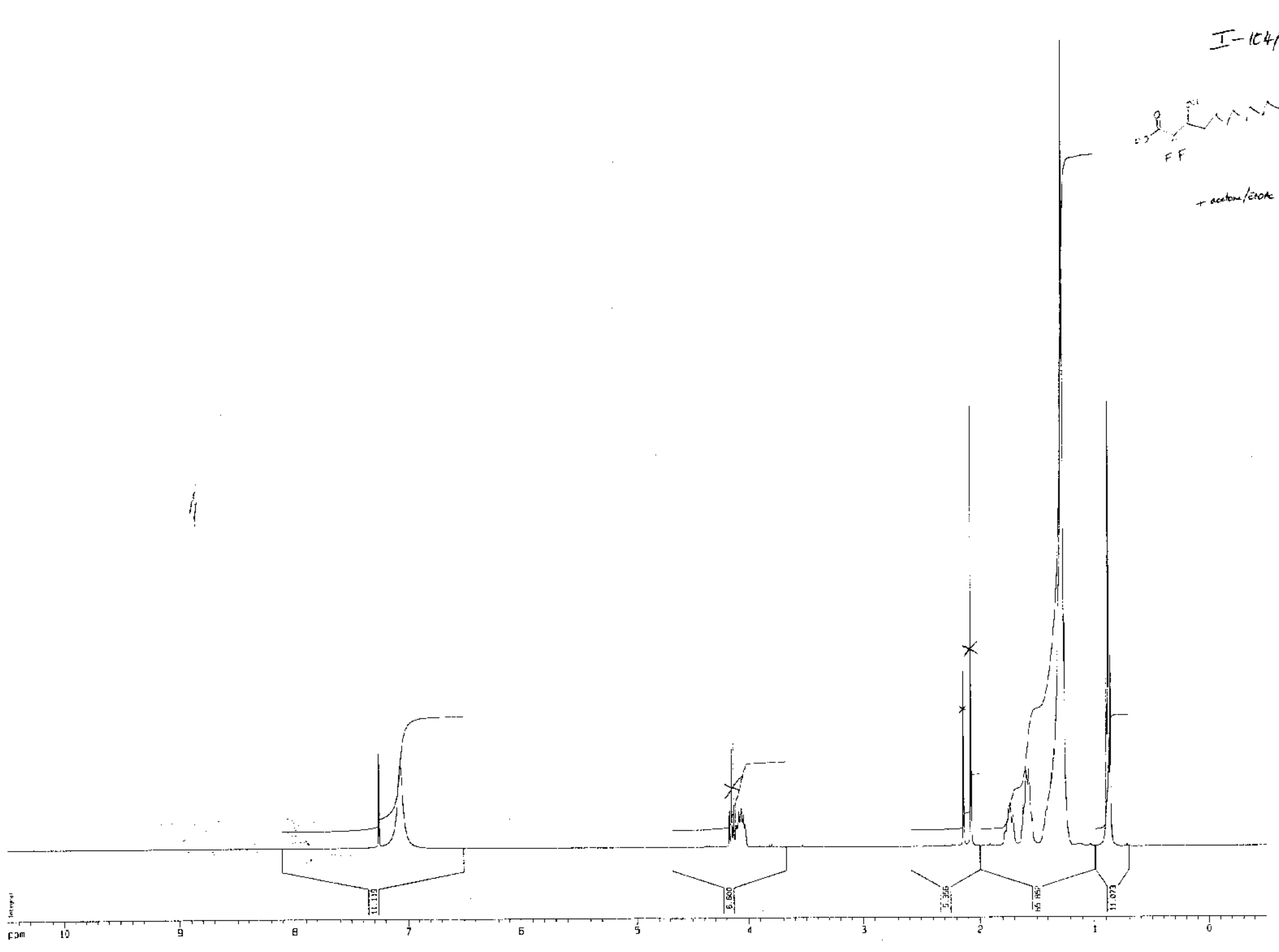

I-127/2
101.8
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
22.1
%T
4000.0
3000
2000
1500
1000
500
400.0
cm-1
3310.29
2853.91
2922.08
1774.90
1753.41
1693.75
1677.82
1541.33
1495.14
1469.10
1399.89
1351.94
1312.07
1292.75
1262.45
1384.55
1226.60
1183.11
1157.38
1133.04
1078.02
1046.98
1011.53
970.65
947.69
915.92
897.30
861.76
824.46
794.42
770.92
722.86
710.02
666.26
c:\pel_data\spectra\drs.sp


I-103/2
+ EtOAc
0.586
4.407
76.840
10.228
ppm
10
9
8
7
6
5
4
3
2
1
0

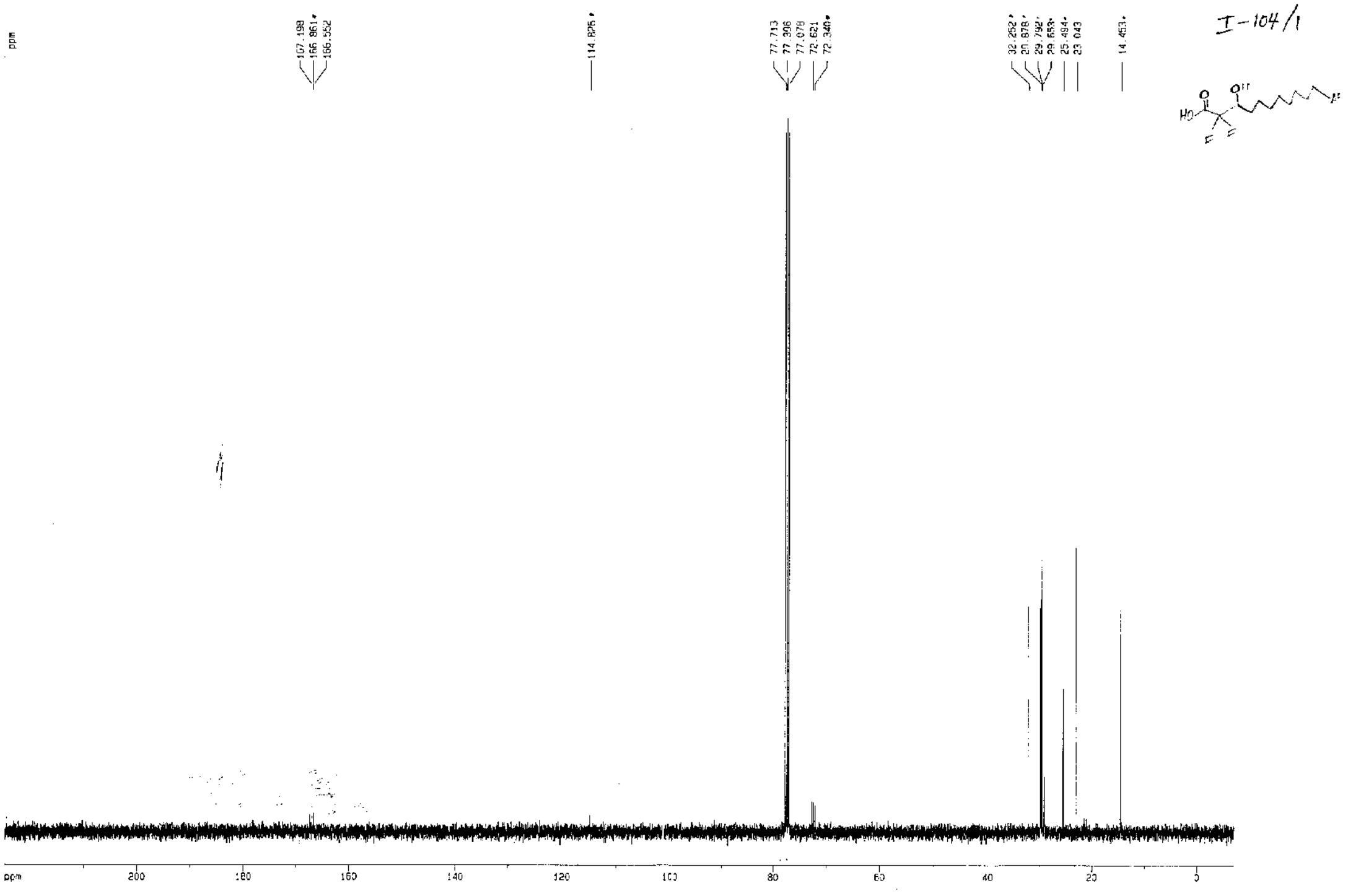

I-103/2
171.621
164.424
164.114
163.789
117.603
115.075
112.525
77.718
77.400
77.082
72.199
72.178
71.928
64.575
63.372
60.803
32.247
29.674
29.650
25.387
23.036
21.377
14.440
14.304
14.117
ppm
200
180
160
140
120
100
80
60
40
20
0

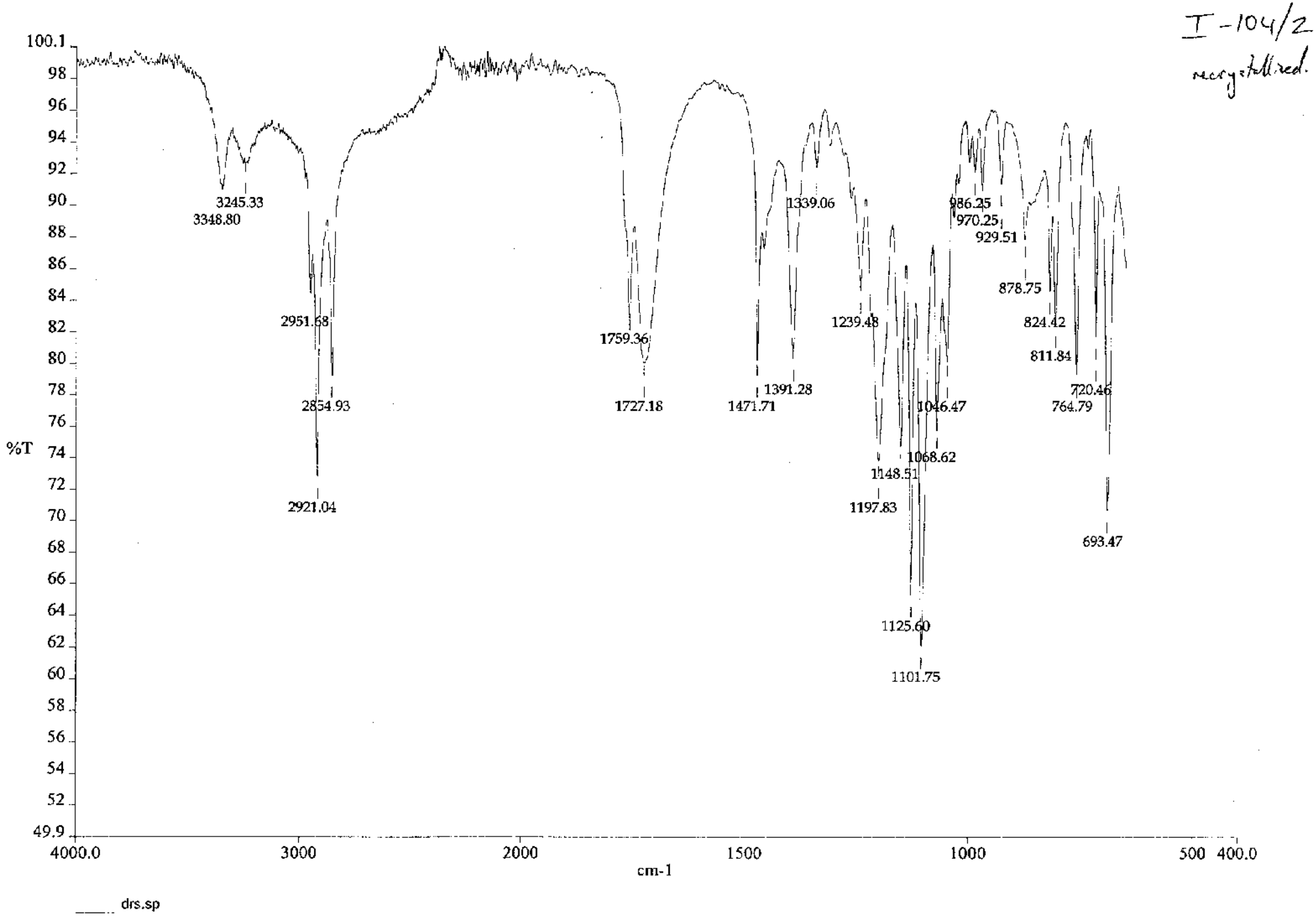
J-103/2


103.4
100
95
90
85
80
75
70
65
60
55
51.2
%T
3477.71
2855.88
2925.81
1759.53
1466.89
1374.95
1313.91
1213.36
1175.99
1091.74
1015.69
855.09
782.96
722.03
4000.0
3000
2000
1500
1000
500
400.0
cm-1
c:\pel_data\spectra\drs.sp



I-104/1
+ acetone/EtOAc
11.110
6.600
5.356
11.073
ppm
10
9
8
7
6
5
4
3
2
1
0

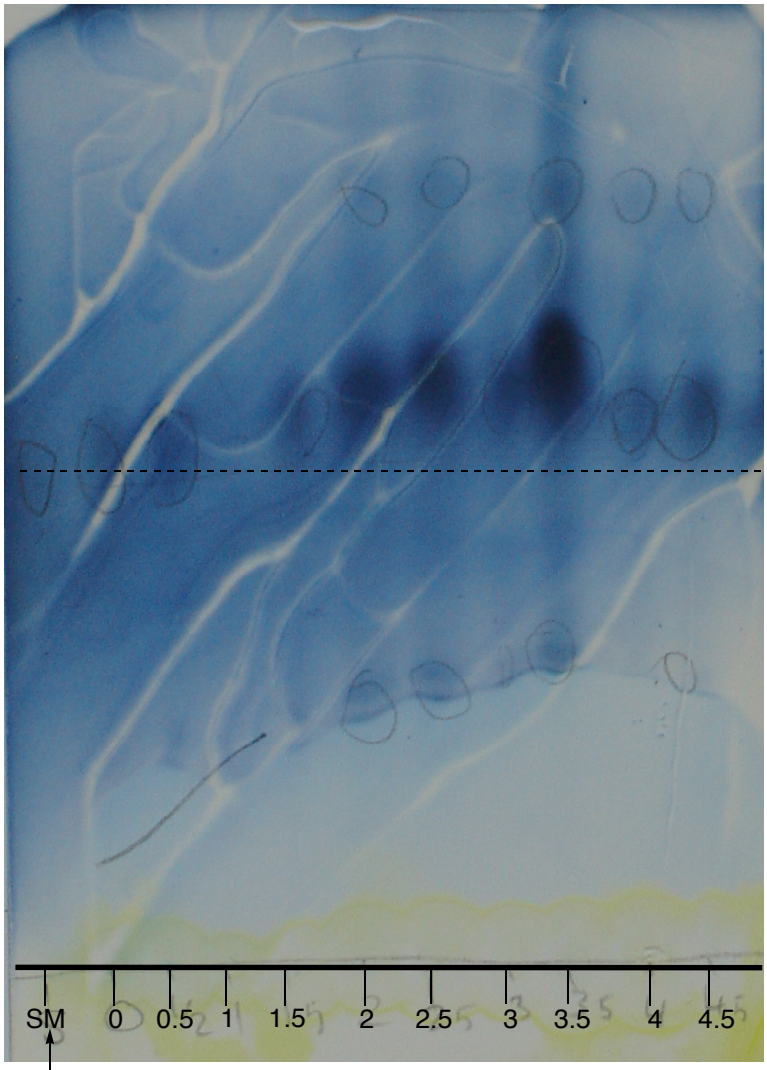

I-104/1
ppm
167.198
166.861
166.552
114.826
77.713
77.396
77.078
72.621
72.349
32.252
30.878
29.792
29.653
25.494
23.043
14.453
200
180
160
140
120
100
80
60
40
20
0


I-104/2
recrystallized.
100.1
98
96
94
92
90
88
86
84
82
80
78
76
74
72
70
68
66
64
62
60
58
56
54
52
49.9
%T
3348.80
3245.33
2951.68
2854.93
2921.04
1759.36
1727.18
1471.71
1391.28
1339.06
1239.48
1197.83
1148.51
1125.60
1101.75
1068.62
1046.47
986.25
970.25
929.51
878.75
824.42
811.84
764.79
720.46
693.47
4000.0
3000
2000
1500
1000
500
400.0
cm-1
drs.sp

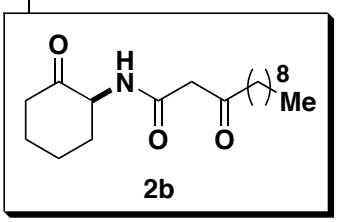
TLC TIME COURSE EXPERIMENT

Compound 2b (2 mg) in DMSO (100 μl) was mixed with aqueous phosphate buffer (pH 7.0, 1 ml) and incubated at either 25 ˚C or 37 ˚C. Aliquots from the mixture were spotted onto a silica TLC plate at the times indicated after initial mixing. At 25 ˚C the starting material has almost completely disappeared after 2 hours and many spots appear, at 37 ˚C starting material has disappeared after 1.5 hours. Pencil circles were drawn around spots that quench the fluorescence of silica gel under UV light. After 3.5 hours at 37 ˚C the solution is cloudy and a red-brown immiscible oil has formed.

25 ˚C

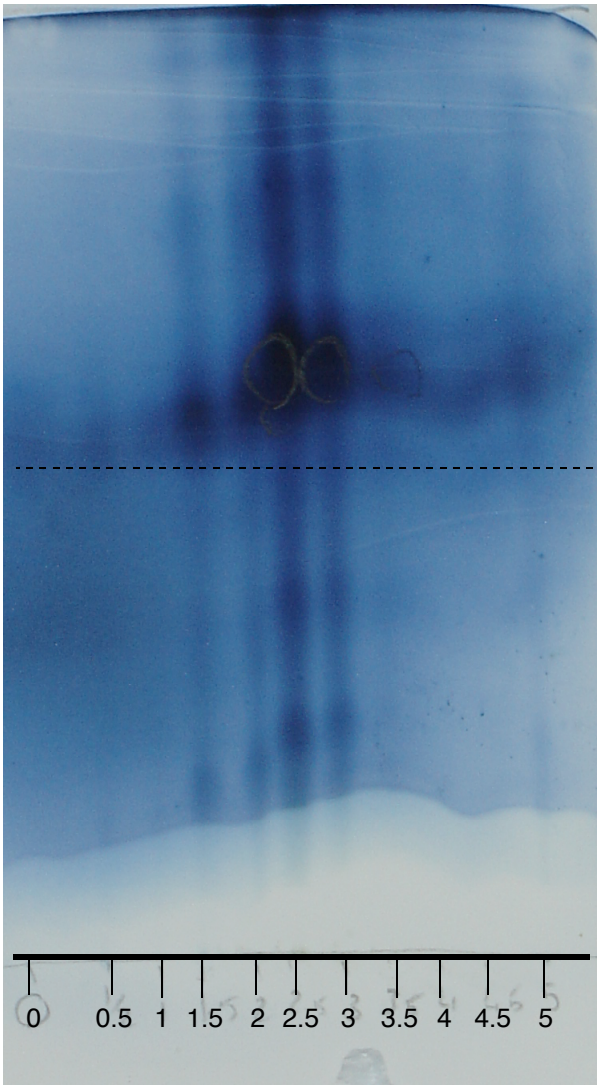


Time (h)



2b

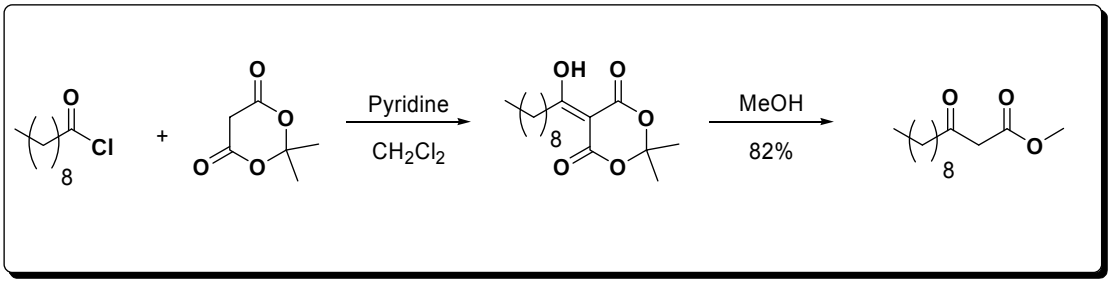
37 ˚C



Time (h)

Biochemistry Project

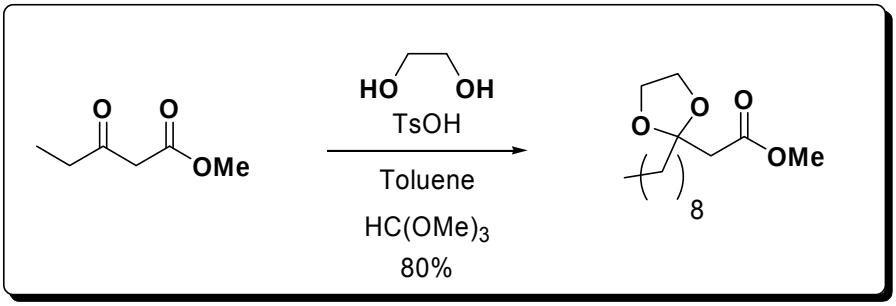
3-Oxo-dodecanoic acid methyl ester



To a solution of Meldrums acid (2.0 g, 13.88 mmol) in CH_2Cl_2 (20 ml) at 0 ºC under nitrogen was added pyridine (2.24 ml, 27.76 mmol) dropwise over 20 minutes, followed by decanoyl chloride (3.16 ml, 15.26 mmol) and the reaction stirred for one hour at 0 ºC and the allowed to warm to room temperature for one hour. The orange reaction was diluted with CH_2Cl_2 (20 ml) and poured into ice and HCl (2 N, 20 ml). The organic layer was separated, washed with HCl, brine, dried ($MgSO_4$) and the solvent removed in vacuo. The crude product was refluxed in methanol (55 ml) under nitrogen for 4 hours and then the solvent was removed in vacuo. The yellow oil was purified by vacuum distillation (125 ºC, 2 mbar) and then column chromatography to yield the title compound as a colourless liquid (2.6 g, 82 %)

R*f* 0.31 (SiO_2; 9:1 Hexane: Ethyl acetate); υ*max* (neat)/cm^{-1} 1748s (ester), 1717 (ketone); δH (400 MHz; $CDCl_3$) 3.76 (3H, s, OMe), 3.43 (2H, s, C(O)C$\underline{H}_2$C(O)), 2.53-2.49 (2H, t, *J* 7.5 Hz, C$\underline{H}_2$C(O)), 1.59-1.58 (2H, m, C$\underline{H}_2$$CH_2$CH(O)), 1.35-1.2 (12H, br m, alkyl CH_2), 0.88-0.84 (3H, t, *J* 6.5 Hz, CH_3) ; LCMS (MeCN) 4.23 min, 229.1 (MH+)
Spectral data consistent with literature values[1]

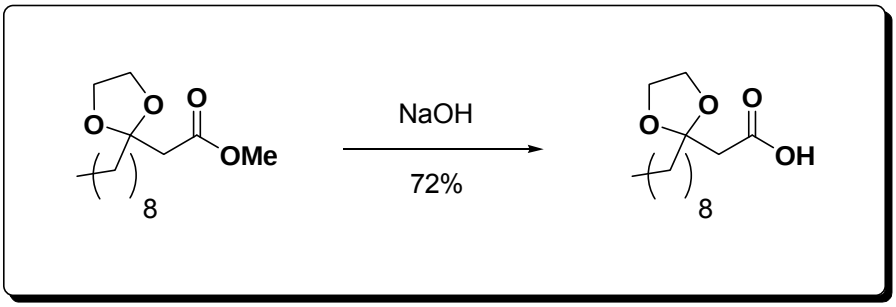
(2-Nonyl-[1,3]dioxolan-2-yl)-acetic acid methyl ester[2]:



To a 3-Oxo-dodecanoic acid methyl ester (2.5 g, 10.96 mmol) and *para*-toluene sulfonic acid (0.19 g) was added methyl orthoformate (3.45 ml, 20 mmol) and ethylene glycol (2.9 ml, 52 mmol) under nitrogen. The reaction was stirred for 4 hours, until LCMS showed no starting material. The reaction was quenched by addition of 5% NaH_2PO_4 solution (3 ml) and stirred for 15 minutes. Ether (100 ml) was added and the reaction washed with water, brine, dried ($MgSO_4$) and purified by column chromatography to yield the title compound as a colourless oil (2.4 g, 80 %).

R*f* 0.34 (SiO_2; 10:1 Hexane: Ethyl acetate); υ*max* (neat)/cm^{-1} 1741s (ester), 1124s (C-O); δH (400 MHz; $CDCl_3$) 4.00-3.82 (4H, m, $OC\underline{H}_2C\underline{H}_2O$), 3.68 (2H, s, $CO_2C\underline{H}_3$), 2.65 (2H, s, $C\underline{H}_2CO_2CH_3$), 1.78-1.75 (2H, m, CH_2), 1.40-1.30 (2H, br m, alkyl CH_2), 1.30-1.20 (12H, br m, alkyl CH_2), 0.88-0.84 (3H, t, *J* 6.5 Hz, CH_3). LCMS 4.41 min, 273 $(M+H)^+$

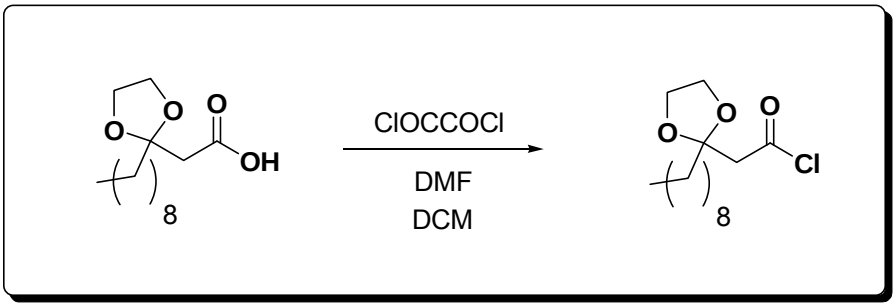
(2-Nonyl-[1,3]dioxolan-2-yl)-acetic acid



(2-Nonyl-[1,3]dioxolan-2-yl)-acetic acid methyl ester (1.0 g, 3.68 mmol) was stirred with 10% sodium hydroxide (1.47 ml, 3.68 mmol) for 3 hours. The reaction was monitored by TLC (10:1 hexane: ethyl acetate). The aqueous layer diluted with water (5 ml) and was washed with ether (x 3). The aqueous layer was acidified and extracted using ether (x 3). The organic layer was dried ($MgSO_4$) and solvent removed in vacuo to yield the title compound as a white solid (683 mg, 72%)

υmax (neat)/cm^{-1} 1706s (ester) 1056s (C-O); δH (400 MHz; $CDCl_3$) 4.06-3.98 (4H, m, $OC\underline{H_2}C\underline{H_2}O$), 2.70 (2H, s, $C\underline{H_2}CO_2H$), 1.78-1.74 (2H, m, CH_2), 1.40-1.30 (2H, br m, alkyl CH_2), 1.30-1.20 (12H, br m, alkyl CH_2), 0.88-0.85 (3H, t, *J* 7.0 Hz, CH_3); LCMS 4.43 min 199 $(M-57)^+$; mp 38-42^{0}C.

(2-Nonyl-[1,3]dioxolan-2-yl)-acetyl chloride

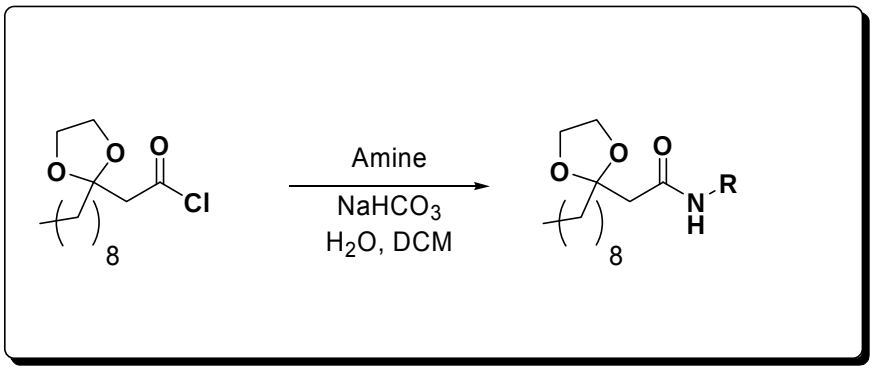


To a solution of (2-Nonyl-[1,3]dioxolan-2-yl)-acetic acid (600mg, 2.3 mmol) in CH_2Cl_2 (5 ml) was added oxalyl chloride (0.31 ml, 3.5 mmol) and DMF (cat.) at 0 ^{0}C. The reaction was stirred for 30 minutes then put under nitrogen for 90

minutes. Solvent was removed *in vacuo* to yield (2-Nonyl-[1,3]dioxolan-2-yl)-acetyl chloride as a brown oil (655 mg, 100 %) which was used in the next reaction without purification.

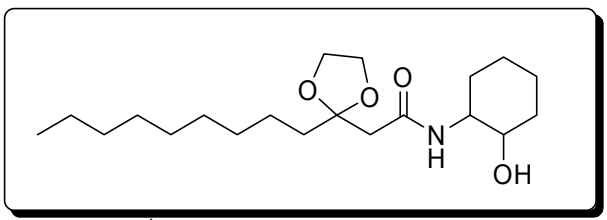
δH (400 MHz; $CDCl_3$) 4.06-3.92 (4H, m, $OC\underline{H_2}C\underline{H_2}O$), 3.21 (2H, s, $C\underline{H_2}COCl$), 1.78-1.70 (2H, m, CH_2), 1.35-1.20 (14H, br m, alkyl CH_2), 0.88-0.80 (3H, t, *J* 6.5 Hz, CH_3).

General Amide Coupling:



To a solution of the amine (1.5 equiv.) and sodium carbonate (3 equiv.) in water was added (2-Nonyl-[1,3]dioxolan-2-yl)-acetyl chloride (1 equiv.) in dry CH_2Cl_2. The reaction was stirred overnight and monitored by TLC. The organic layer was separated and washed with $Na_2CO_{3(aq)}$ (x 2), Brine (x 2), dried ($MgSO_4$) and solvent removed *in vacuo* to yield a brown solid.

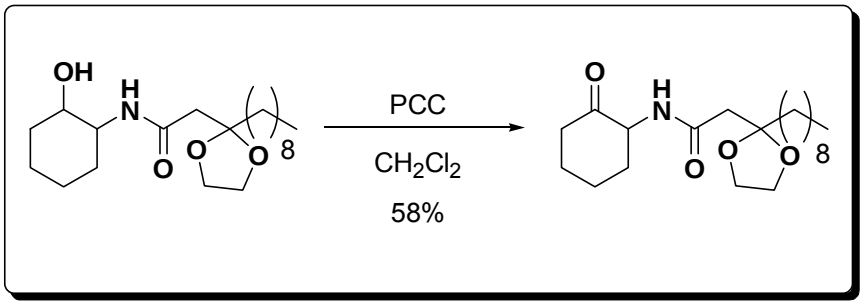
N-(2-Hydroxy-cyclohexyl)-2-(2-nonyl-[1,3]dioxolan-2-yl)-acetamide



isolated as a white solid by recrystalisation from ether (400 mg, 60 %) R*f* 0.60 (SiO_2; 10:1 Hexane: Ethyl acetate); υ*max* (neat)/cm^{-1} 3440w (NH), 3356m (OH), 1641s (amide); δH (400 MHz; $CDCl_3$) 6.41-6.35 (1H, d, *J* = 6.5 Hz, N<u>H</u>), 4.02-3.98 (4H, m, $CO\underline{CH_2}\underline{CH_2}CO$), 3.67-3.59 (1H, m,), 3.44-3.43 (1H, d, *J* = 4.0 Hz,), 3.34-3.27 (1H, m,), 2.63-2.55 (2H, d, *J* = 4.5 Hz, $C(O)C\underline{H_2}$), 2.07-2.00 (1H, m,), 1.92-1.89 (1H, m,), 1.74-

1.65 (4H, m,), 1.36-1.34 (3H, m, alkyl CH_2), 1.31-1.18 (14H, m, alkylCH_2), 0.88-0.85 (3H, t, *J*=6.5 Hz, C<u>H</u>$_3$); LCMS (MeCN) 4.11 min, 356 ($M+H^+$).

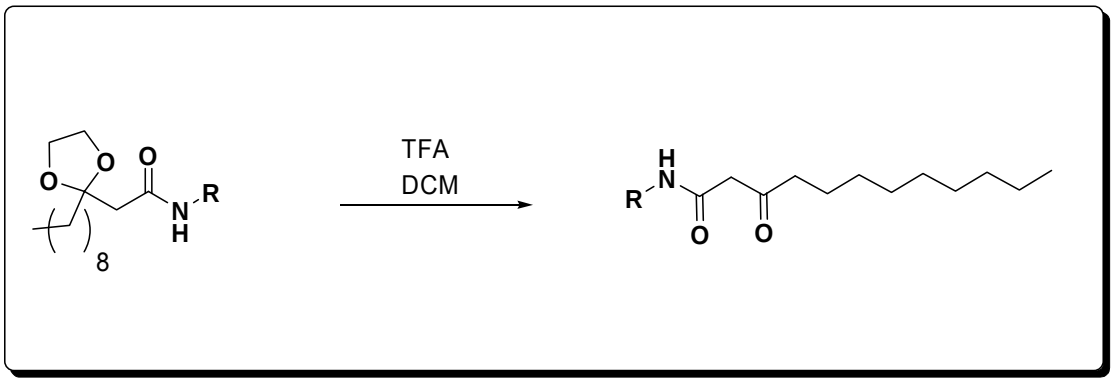
PCC oxidation



To a solution of N-(2-Hydroxy-cyclohexyl)-2-(2-nonyl-[1,3]dioxolan-2-yl)-acetamide (50 mg, 0.14 mmol) in CH_2Cl_2 (1 ml) was added PCC (30 mg, 0.14 mmol), and the reaction stirred at room temperature under nitrogen for 4 hours, a further portion of PCC (30 mg, 0.14 mmol) was added and the reaction stirred overnight. The black gum was washed with ether and filtered through a pad of silica to yield a colourless oil, which was used in the next reaction without further purification.

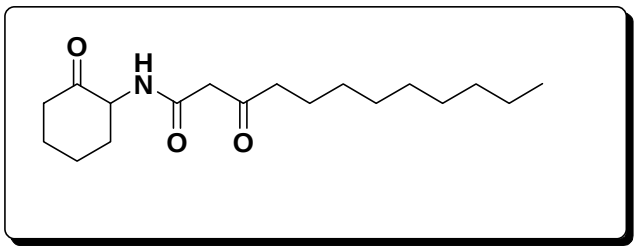
R*f* 0.42 (SiO_2; 1:2 hexane: ethyl acetate); υ*max* (neat)/cm^{-1} 3377w (amide NH), 1719 (ketone), 1667 (amide), 1514 (); δH (500 MHz; $CDCl_3$) 9.40-9.22 (1H, br s, N<u>H</u>), 8.32-8.23 (1H, d, *J* 5.0 Hz, H4), 8.15-8.10 (1H, br d, *J* 8.0 Hz, H1), 7.71-7.65 (1H, dt, *J* 8.0,1.5 Hz, H2), 7.05-7.01 (1H, m, H3), 3.56 (2H, s, H5), 2.58-2.46 (2H, t, *J* 7.5 Hz, H6), 1.64-1.54 (2H, m, H7), 1.33-1.17 (14H, m, alkyl C<u>H</u>$_2$), 0.90-0.81 (3H, t, *J* 6.5 Hz, C<u>H</u>$_3$);; LCMS (MeCN) 4.49 min, 354 (MH+)

General Procedure for Acetyl Deprotection



To a solution of the acetyl protected ketone (1 equiv.) in CH_2Cl_2 was added TFA (95%), the yellow reaction was stirred for 2.5 hours and quenched using sodium bicarbonate solution. The aqueous layer was extracted using DCM (x 3) and the combined organic layers washed with brine (x 2), dried ($MgSO_4$) and the solvent removed *in vacuo*. The crude product was purified by column chromatography to yield :

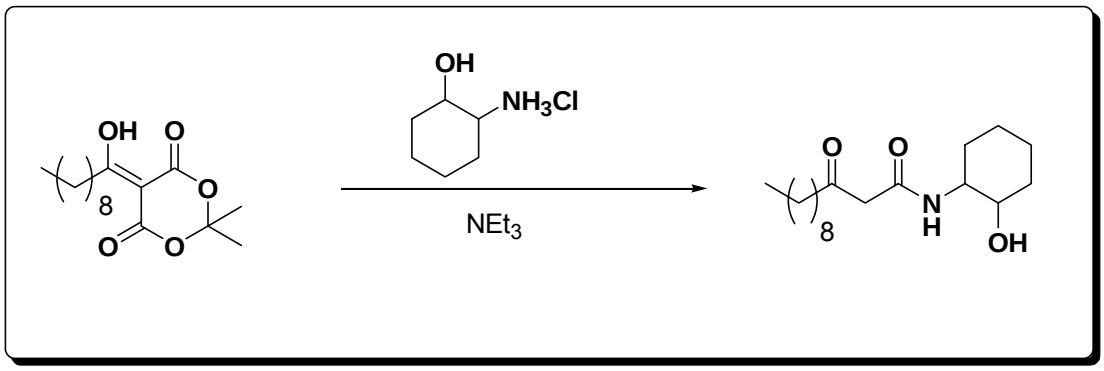
N-(2-oxocyclohexyl)-3-oxododecamide as a white solid (16 mg, 63 %).



R*f* 0.38 (SiO_2; 1:2 hexane: ethyl acetate); υ*max* (neat)/cm^{-1} 3268w (amide NH), 1710 (ketone), 1652 (amide), 1629 (ketone), 1514; δH (500 MHz; $CDCl_3$) 7.53 (1H, br s, N<u>H</u>), 4.43-4.55 (1H, quintet, *J* 6.0 Hz,), 3.39 (2H, s), 2.56-2.66 (1H, m), 2.48-2.58 (3H, m), 2.33-2.43 (1H, dt, *J* 13.5, 6.0 Hz,), 2.07-2.11 (1H, m), 1.83-1.94 (1H, m), 1.71-1.82 (1H, m), 1.52-1.67 (3H, m), 1.33-1.44 (1H, dq, *J* 12.5, 4.0 Hz), 1.19-1.33 (12H, m, alkyl CH_2), 0.83-0.88 (3H, t, *J* 7 Hz, C<u>H</u>$_3$); δC (125 MHz, $CDCl_3$) 207.33 (C), 206.28 (C), 165.75 (C), 58.61 (CH), 49.75 (CH_2), 44.11 (CH_2), 41.47 (CH_2), 35.50 (CH_2), 32.22 (CH_2), 29.76 (CH_2), 29.72 (CH_2), 29.61 (CH_2), 24.47 (CH_2), 23.79 (CH_2), 23.03 (CH_2), 14.03 (CH_3); LCMS (MeCN) 3.97 min, 310 (MH+)[3]

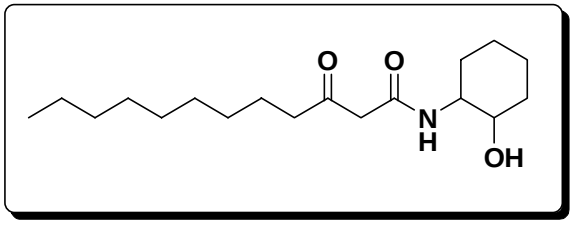
1) Data agrees with literature values: Calvet S, David O, Vanucci-Bacqué C, Fargeau-Bellassoued MC, Lhommet G: **Chiral heterocyclic β-enamino esters: convenient synthesis and diastereoselective reduction** *Tetrahedron.* 2003, **59**:6333-6339
2) Pirrung MC, Liu H, Morehead AT; **Rhodium Chemzymes: Michaelis-Menten Kinetics in Dirhodium(II) Carboxylate-Catalyzed Carbenoid Reactions** *J.Amer.Chem.Soc.* 2002, **124**: 1014 - 1023.
3) Data agrees with literature values: Smith KM, Bu Y, Suga H: **Induction and inhibition of *Pseudomonas aeruginosa* Quorum Sensing by Synthetic Autoinducer Analogs** *Chem. Biol.* 2003, **10**:81-89

3-Oxo-dodecanoic acid (2-hydroxy-cyclohexyl)-amide



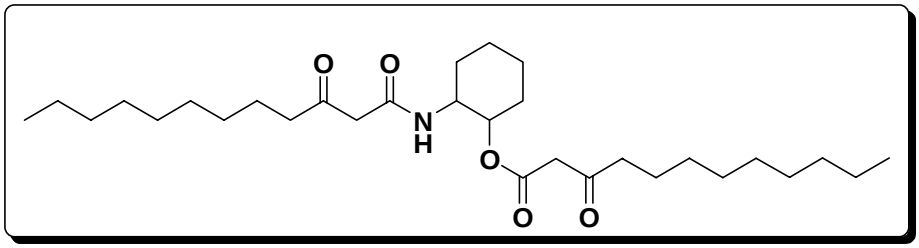
To a solution of acylated meldrums acid (1 g, 3 mmol) in toluene (50 ml) was added potassium carbonate (4.8 g ml, 30 mmol) and *trans*-2aminocyclohexanol hydrogen chloride salt (4.0 g, 30 mmol). The reaction was heated to reflux under nitrogen for 1 hour. The reaction was cooled, reduced in vacuo and taken up in ethyl acetate. The organic layer was washed with sodium hydrogen carbonate solution, brine, dried ($MgSO_4$) and reduced in vacuo to yield a yellow gum. The product was purified by column chromatography to yield:

3-Oxo-dodecanoic acid (2-hydroxy-cyclohexyl)-amide: as a white solid



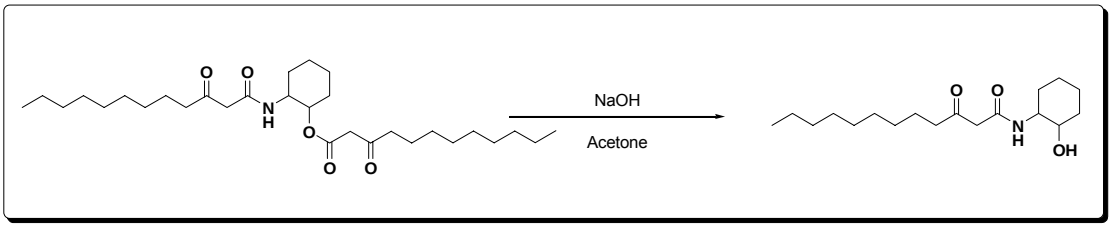
(514 mg,19 %) R*f* 0.08 (SiO_2; 10:1 CH_2Cl_2: ethyl acetate); υ*max* (neat)/cm^{-1} 1718m (ketone) 1669m (amide); δH (500 MHz; $CDCl_3$) 7.16-7.15 (1H, br s, NH), 3.68-3.66 (1H, m,), 3.44 (1H, s, C(O)CH_2C(O)), 3.37-3.33 (2H, m), 2.55-2.52 (2H, t, *J*= , CH_2C(O)), 2.06-2.04 (1H, m), 1.96-1.96 (1H, m), 1.76-1.71 (2H, m), 1.67-1.61 (2H,m), 1.40-1.22 (16H, m, alkyl CH_2), 0.91-0.88 (3H, t, *J* 6.5 Hz, CH_3); δC (125 MHz, $CDCl_3$) 207.43 (C), 167.24 (C), 75.27 (CH), 55.79 (CH), 48.36 (CH_2), 44.00 (CH_2), 36.83 (CH_2), 34.51 (CH_2), 34.30 (CH_2), 31.84 (CH_2), 31.58 (CH_2), 31.26 (CH_2), 29.37 (CH_2), 29.36 (CH_2), 29.32 (CH_2), 29.22 (CH_2), 28.98 (CH_2), 25.73 (CH_2), 24.54 (CH_2), 23.96 (CH_2), 23.37 (CH_2), 22.64 (CH_2), 14.08 (CH_3); LCMS (MeCN) 5.79 min, 312 (MH^+)

3-Oxo-dodecanoic acid 2-(3-oxo-dodecanoylamino)-cyclohexyl ester: as



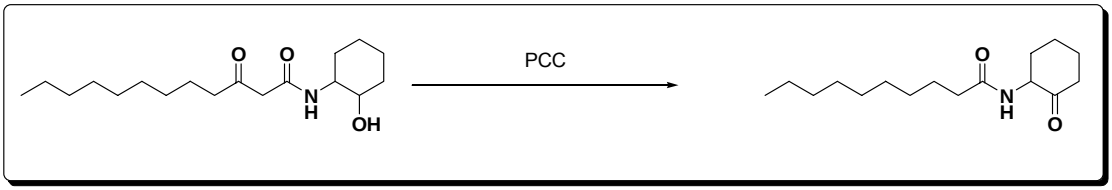
a yellow gum (2.5 g, 45 %) R*f* 0.48 (SiO_2; 10:1 CH_2Cl_2: ethyl acetate); υ*max* (neat)/cm^{-1} 1731m (ketone) 1634m (amide); δH (500 MHz; $CDCl_3$) 6.83-6.81 (1H, br d, *J*=8.0 Hz, NH), 4.74-4.69 (1H, dd, *J*= 10.5, 4.0 Hz, CHNH), 3.92-3.87 (1H, m, CHOC(O)), 3.43-3.30 (4H, m, 2x C(O)CH_2C(O)), 2.51-2.44 (4H, m, 2x CH_2C(O)), 2.08-1.97 (3H, m), 1.74-1.67 (3H, m), 1.67-1.50 (3H, m), 1.32-1.16 (26H, m, alkyl CH_2), 0.87-0.83 (6H, t, *J* 7.0 Hz, 2x CH_3); LCMS (MeCN) 6.81 min, 508 (MH^+)

Hydrolysis of 3-Oxo-dodecanoic acid 2-(3-oxo-dodecanoylamino)-cyclohexyl ester



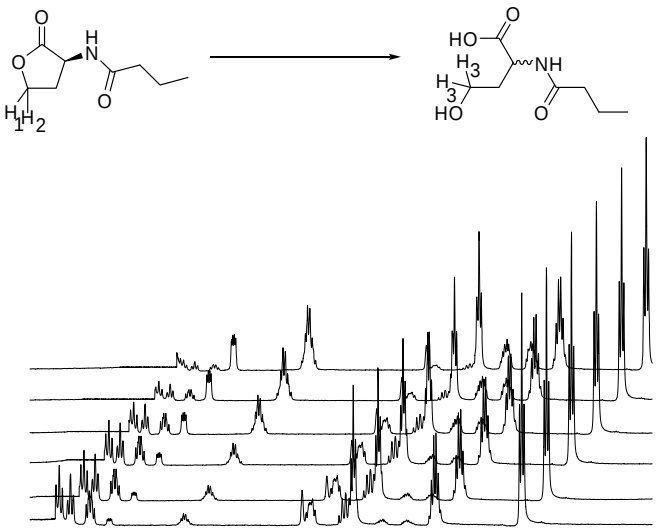
To a solution of 3-Oxo-dodecanoic acid 2-(3-oxo-dodecanoylamino)-cyclohexyl ester (50 mg, 98 µmol) in acetone (0.5 ml) was added 10 % sodium hydroxide (8 ml) and the reaction stirred overnight. The reaction was extracted with ethyl acetate and the aqueous layer acidified, then extracted with ethyl acetate. The organic layer was dried ($MgSO_4$) the product isolated as a cream solid. Data as above.

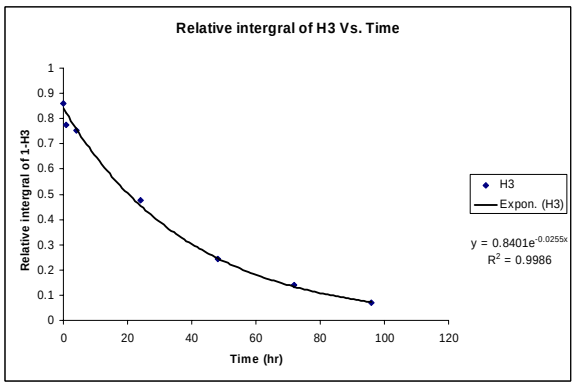
PCC oxidation



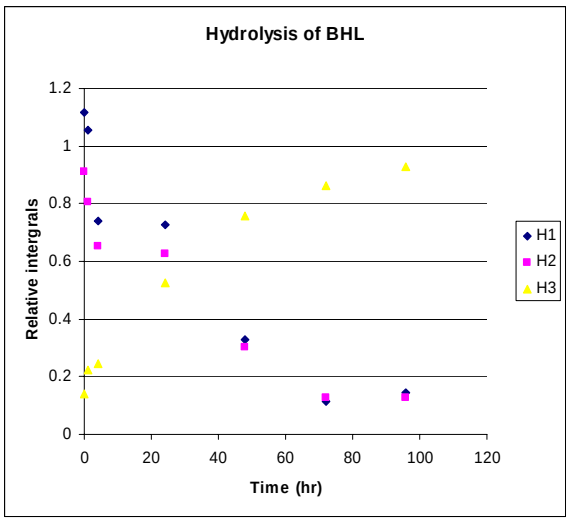
To a solution of 3-Oxo-dodecanoic acid (2-hydroxy-cyclohexyl)-amide (100 mg, 0.32 mmol) in CH_2Cl_2 (5 ml) was added PCC (138 mg, 0.64 mmol), and the reaction stirred at room temperature under nitrogen for 18 hours, The black gum was washed with ether and filtered through a pad of silica to yield yellow oil, which was purified by column chromatography to yield Decanoic acid (2-oxo-cyclohexyl)-amide as a white solid (20 mg, 20 %)

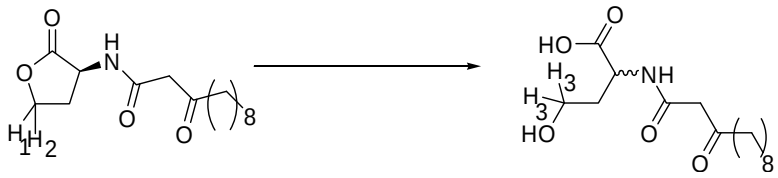
R*f* 0.28 (SiO_2; 3:1 CH_2Cl_2: ethyl acetate); υ*max* (neat)/cm^{-1} 3298 (amide NH), 1707m (ketone) 1638m (amide); δH (500 MHz; $CDCl_3$) 6.38 (1H, br s, N<u>H</u>), 4.51-4.44 (1H, m, C<u>H</u>NH), 2.70-2.61 (1H, m), 2.54-2.47 (1H, m), 2.43-2.35 (1H, dt, *J*= 13.5, 6.0 Hz), 2.21-2.16 (2H, t, *J*= 8.0 Hz, C(O)C<u>H_2</u>), 2.15-2.11 (1H, m), 1.88-1.80 (1H, m), 1.66-1.62 (3H, m), 1.33-1.26 (14H, m, alkyl C<u>H_2</u>), 0.87-0.84 (3H, t, *J* 7.0 Hz, C<u>H_3</u>); δC (125 MHz, $CDCl_3$) 208.35 (C), 173.16 (C), 58.42 (CH), 41.56 (CH_2), 37.10 (CH_2), 36.03 (CH_2), 32.24 (CH_2), 29.81 (CH_2), 27.97 (CH_2), 29.62 (CH_2), 26.06 (CH_2), 24.43 (CH_2), 23.04 (CH_2), 14.47 (CH_3); LCMS (MeCN) 6.05 min, 268 (MH^+), HRMS found 290.23840 $C_{18}H_{30}NO_3Na$, required 290.20960

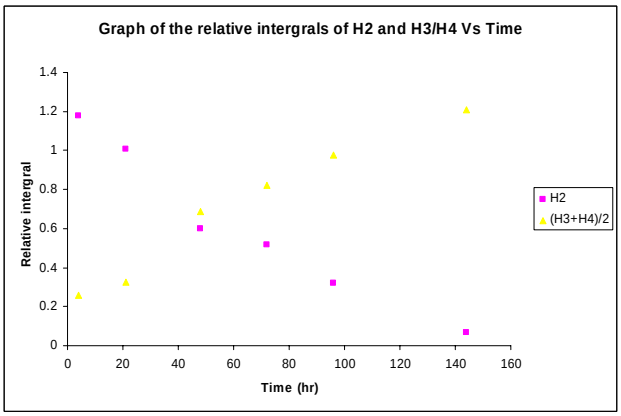


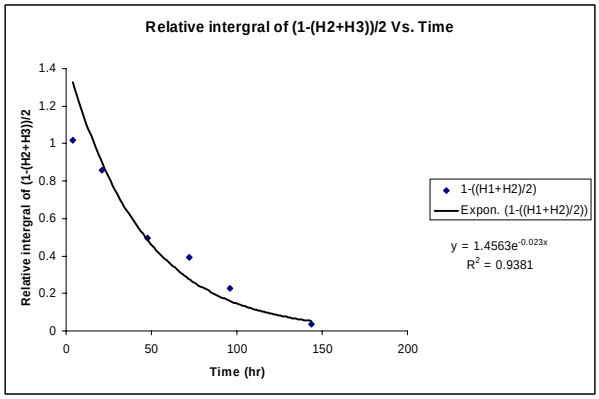


Half Life 20 hours

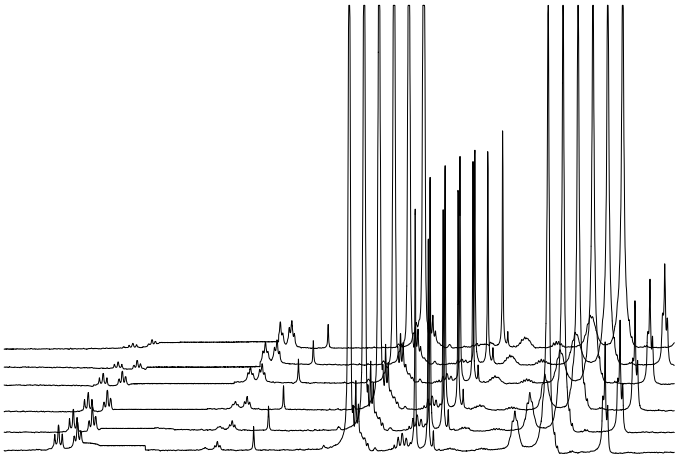


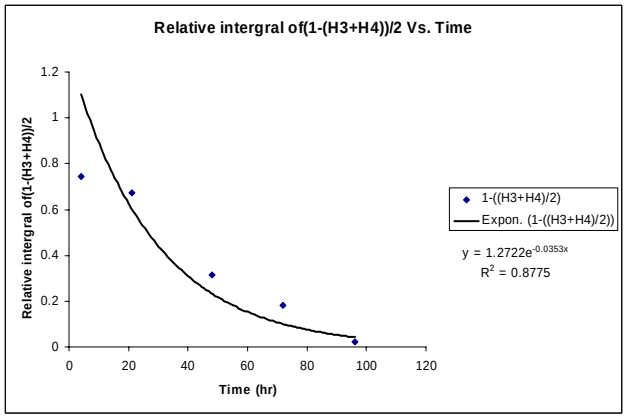




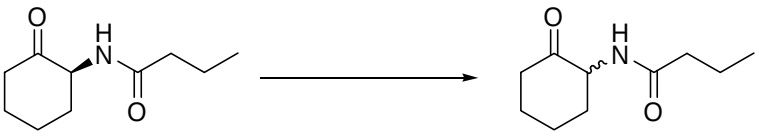


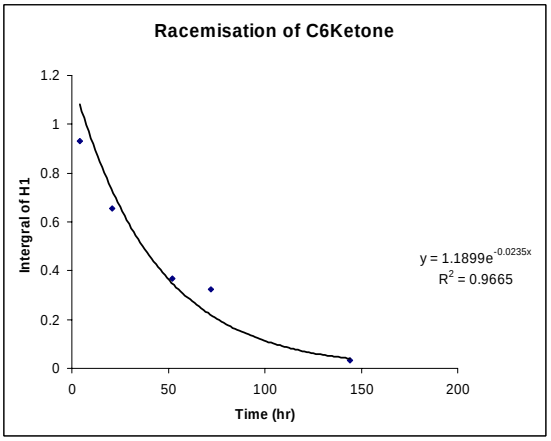
Half Life 46 hours

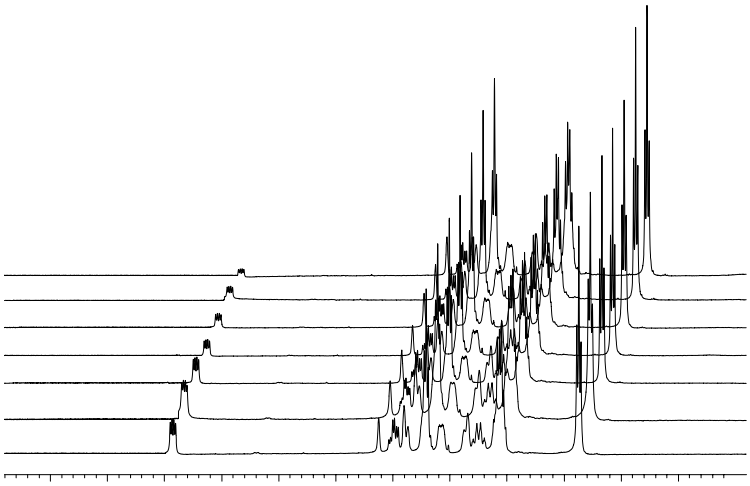




Half Life 31 hours

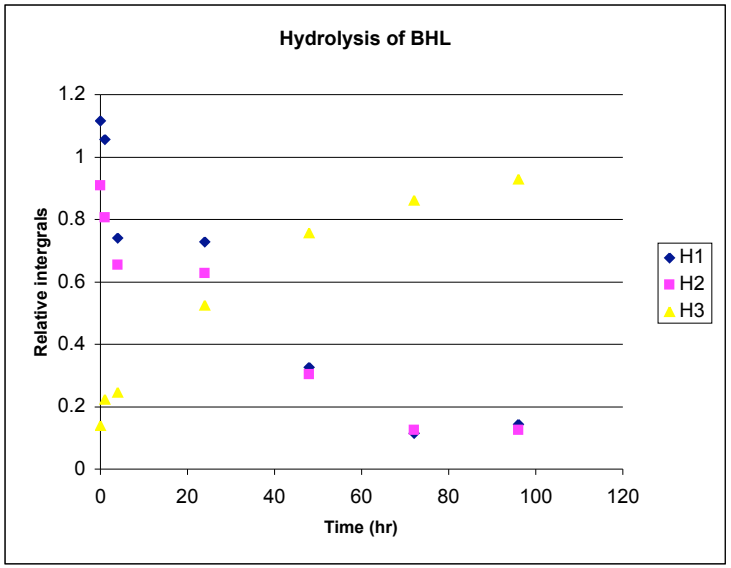


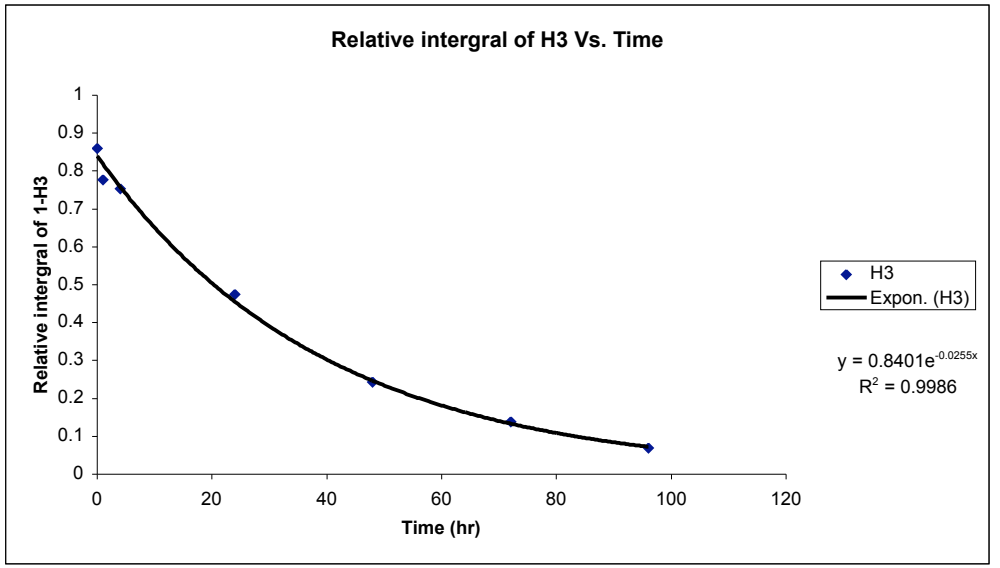




Half Life 37 hours

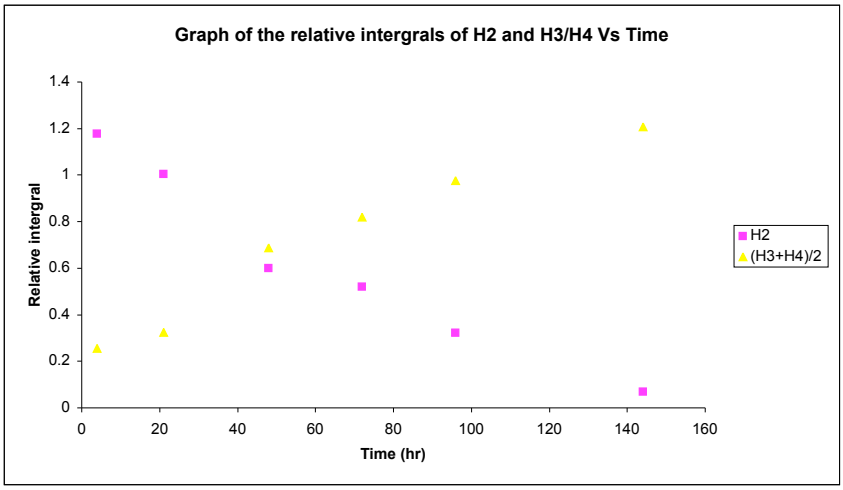
Time	Intergral of	1	2	3	4	Relative intergrals					
						1	2	3	4		(1-H3)
Time						H1	H2			H3	H3
0	100	37.2	30.3	4.7	9.3	1.116	0.909	0.141	0.279	0.1395	0.8605
1	100	35.2	26.9	7.3	14.9	1.056	0.807	0.219	0.447	0.2235	0.7765
4	100	24.7	21.8	5.3	16.4	0.741	0.654	0.159	0.492	0.246	0.754
24	100	24.3	20.9	18.8	35	0.729	0.627	0.564	1.05	0.525	0.475
48	100	10.9	10.1	23.1	50.5	0.327	0.303	0.693	1.515	0.7575	0.2425
72	100	3.8	4.2	24.8	57.4	0.114	0.126	0.744	1.722	0.861	0.139
96	100	4.8	4.2	27.3	62	0.144	0.126	0.819	1.86	0.93	0.07

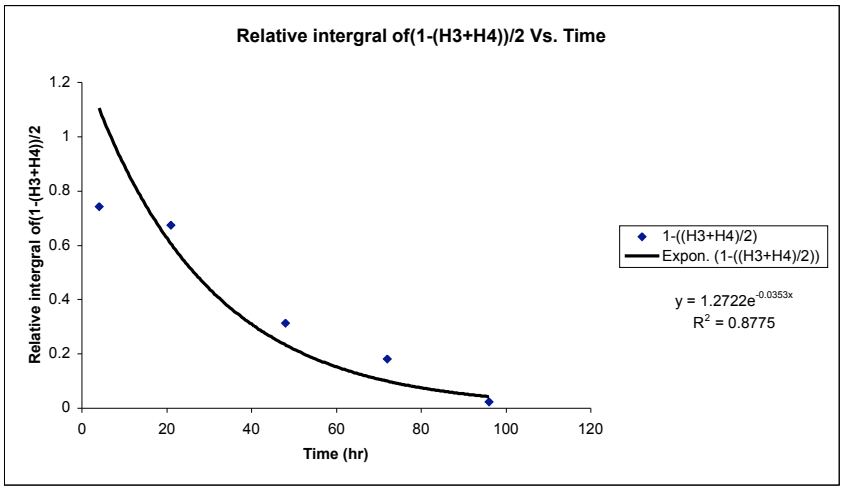




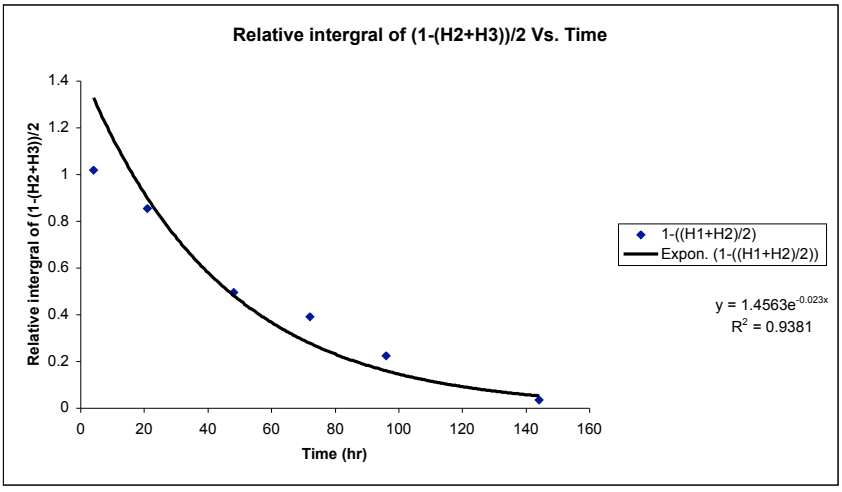
	0.595167
	-0.518913
Half Life	20.34952

Time	Intergral of					Relative intergrals						
		1	2	3	4	1	2	3	4			
Time		H1	H2	H3	H4	H1	H2	H3	H4	(H3+H4)/2	1-((H3+H4)/2)	1-((H1+H2)/2)
4	100	28.7	39.2	6.1	11	0.861	1.176	0.183	0.33	0.2565	0.7435	1.0185
21	100	23.5	33.5	9	12.7	0.705	1.005	0.27	0.381	0.3255	0.6745	0.855
48	100	13	20	21.1	24.7	0.39	0.6	0.633	0.741	0.687	0.313	0.495
72	100	8.9	17.3	26.2	28.4	0.267	0.519	0.786	0.852	0.819	0.181	0.393
96	100	4.3	10.7	30.6	34.5	0.129	0.321	0.918	1.035	0.9765	0.0235	0.225
144	100	0.1	2.3	39.8	40.7	0.003	0.069	1.194	1.221	1.2075	-0.2075	0.036





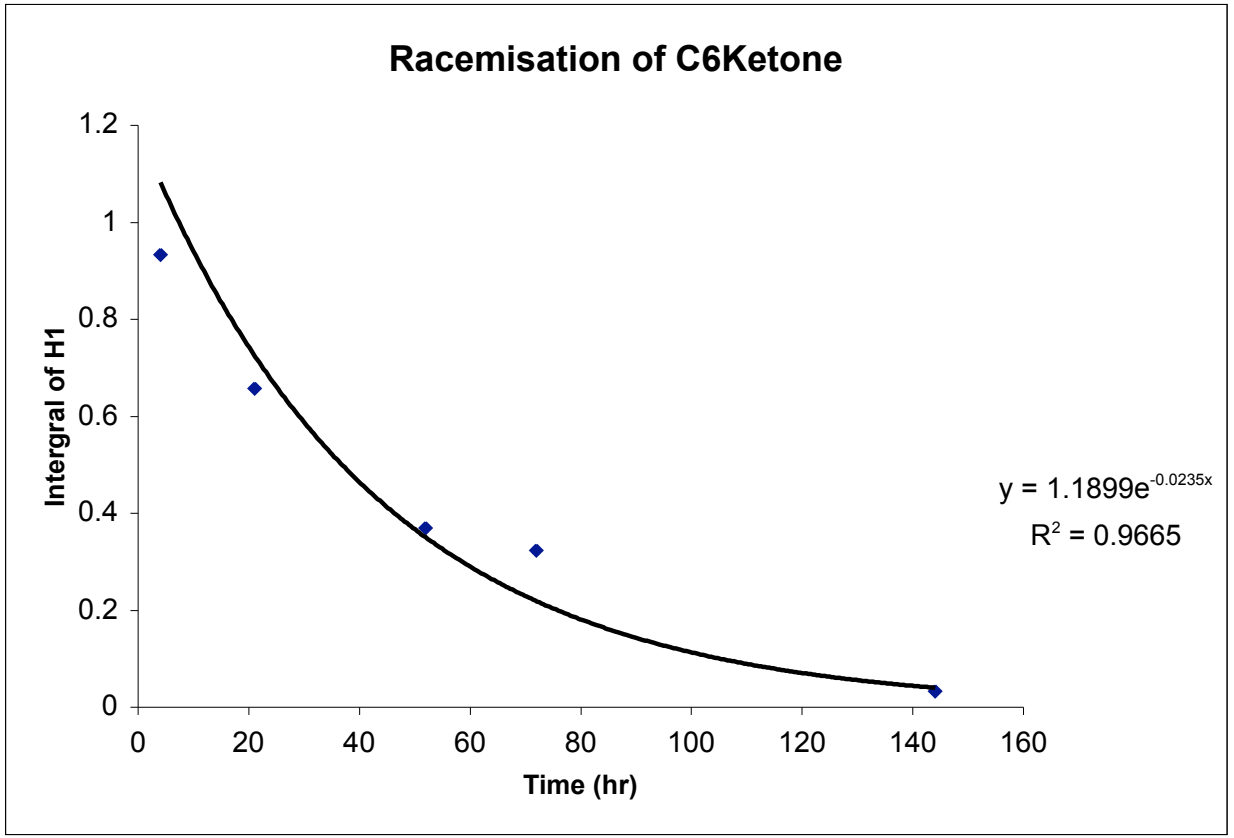
0.4560795
-0.785088
31.403522



0.3433359
-1.069046
46.480268

Time	Intergral of	1	2	3	4	Relative intergrals 1
0	100	27.6				0.828
4	100	31.1				0.933
21	100	21.9				0.657
52	100	12.3				0.369
72	100	10.8				0.324
144	100	1.1				0.033

Racemistation



	0.420203
	-0.86702
Half Life	36.89432

Pseudomonas aeruginosa Pigmentation Assay

PAO-JP2 incubated at 37 ˚C for 48 h with the starting concentration of each compound at 500 μM, and each step to the right the sample is diluted 2-fold. OdDHL is present at a constant concentration of 10 μM.