

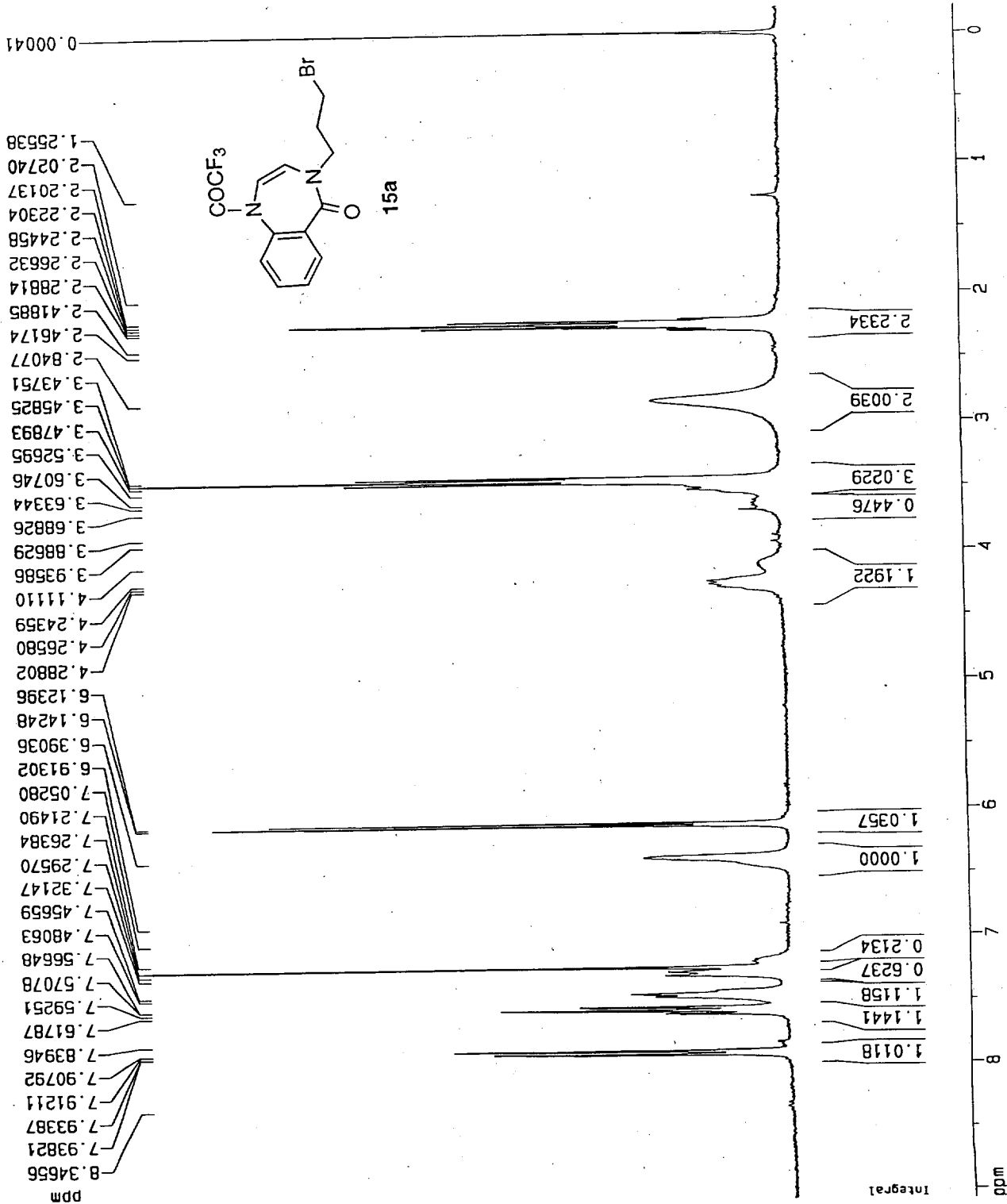
Current Data Parameters
 NAME tsw-213-15a
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date 990907
 Time 13.28
 PULPROG zg30
 SOLVENT CDC13
 AQ 2.1627080 sec
 FIDRES 0.231194 Hz
 DK 66.0 usec
 RG 1024
 NUCLEUS 1H
 HL1 1 dB
 D1 1.0000000 sec
 P1 8.0 usec
 DE 94.3 usec
 SF01 300.1351622 MHz
 SMH 7575.76 Hz
 TD 32768
 NS 8
 DS 2

F2 - Processing parameters
 SI 16384
 SF 300.1333663 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

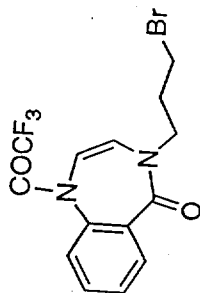
1D NMR plot parameters
 CX 20.00 cm
 F1P 9.090 ppm
 F1 2728.07 Hz
 F2P -0.210 ppm
 F2 -63.00 Hz
 PPMCM 0.46497 ppm/cm
 HZCM 139.55344 Hz/cm

S-41



S-42

13C NMR (CDCl3)



15a

141.285
139.885
138.743
132.101
133.881
129.453
129.087
128.498
127.230
125.609
124.815
124.573
119.239
117.851
116.954
114.027
110.205

154.952
155.447

158.915

PPM

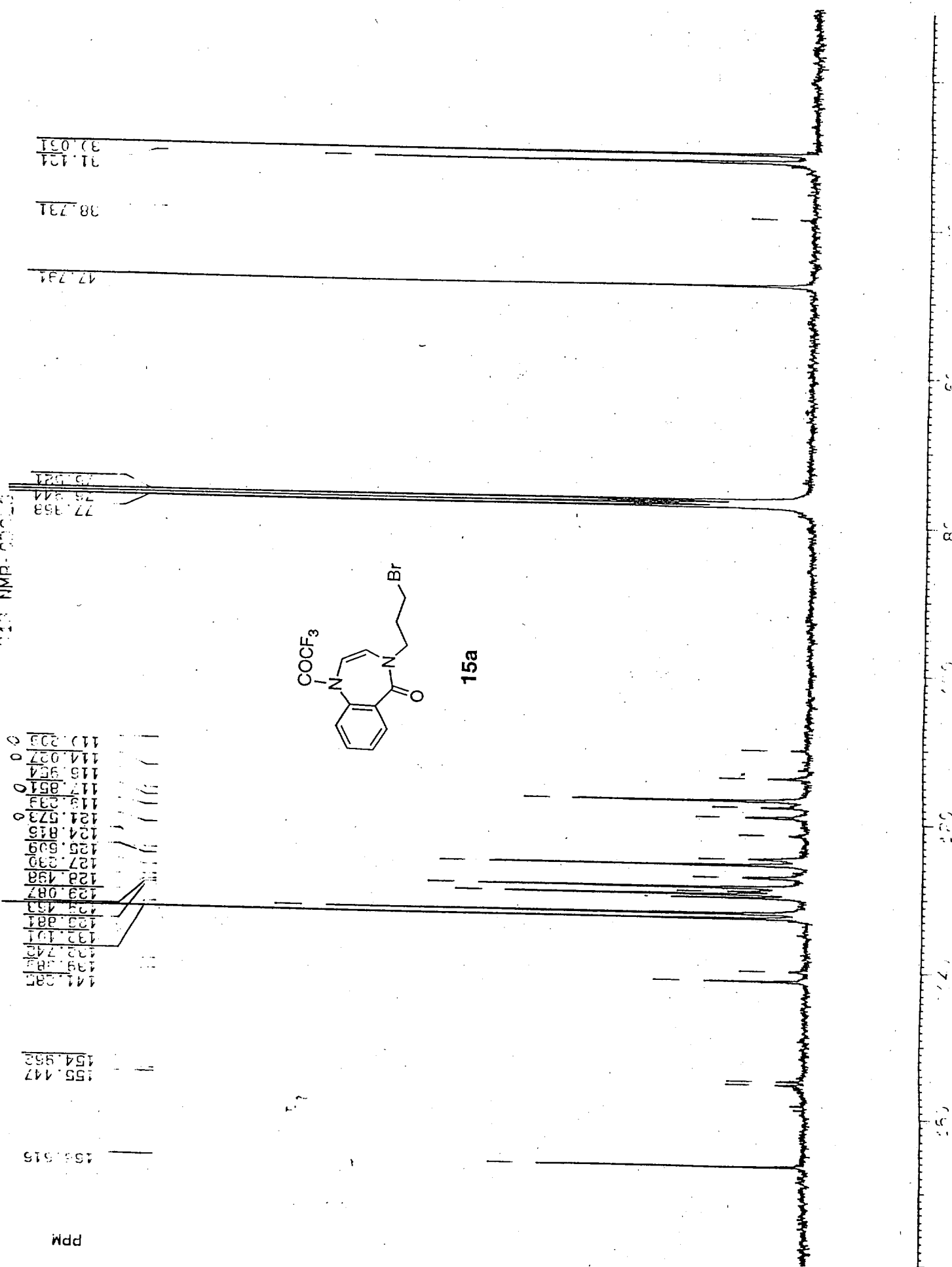
31.424
31.051

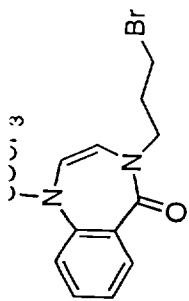
38.731

47.734

73.821

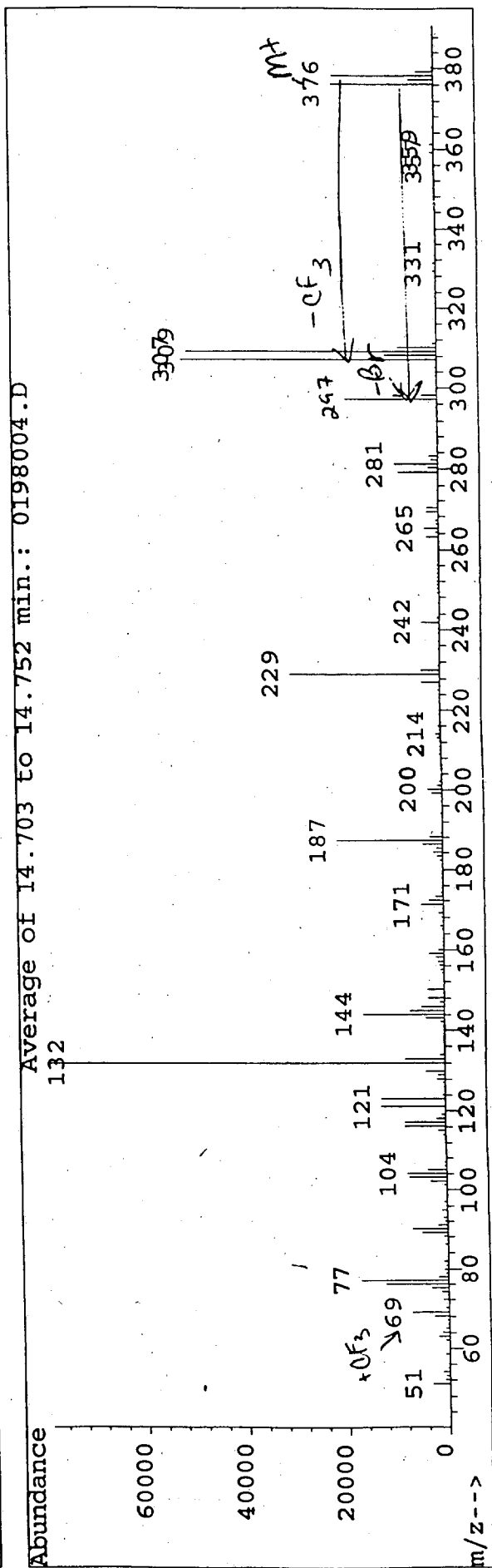
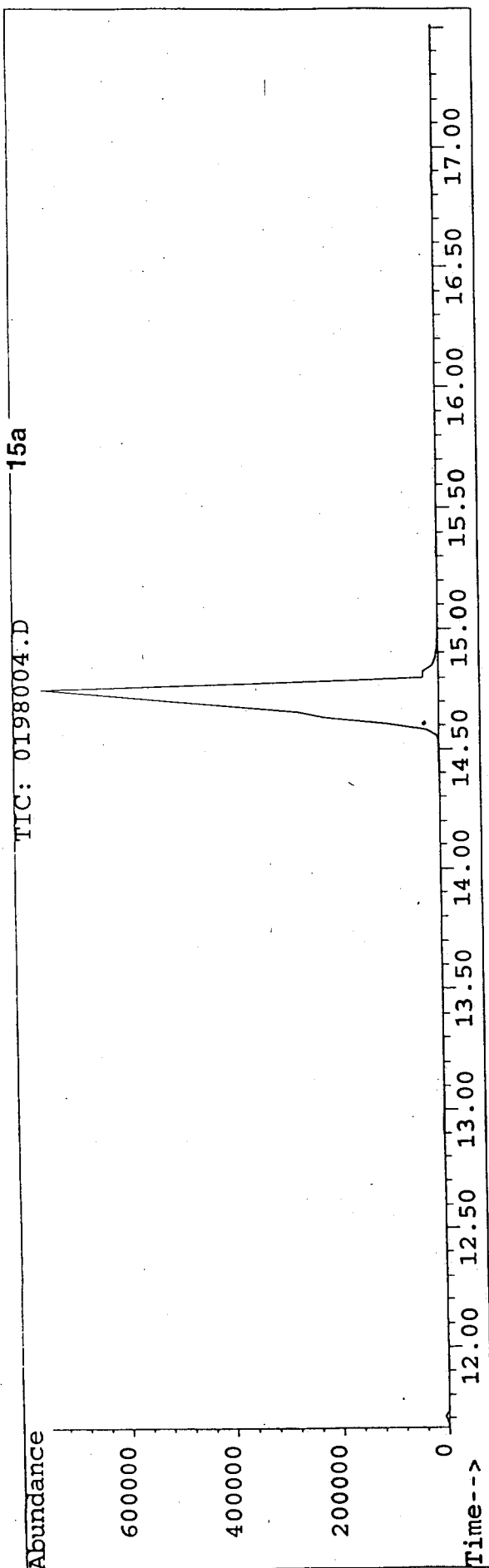
75.971
77.458



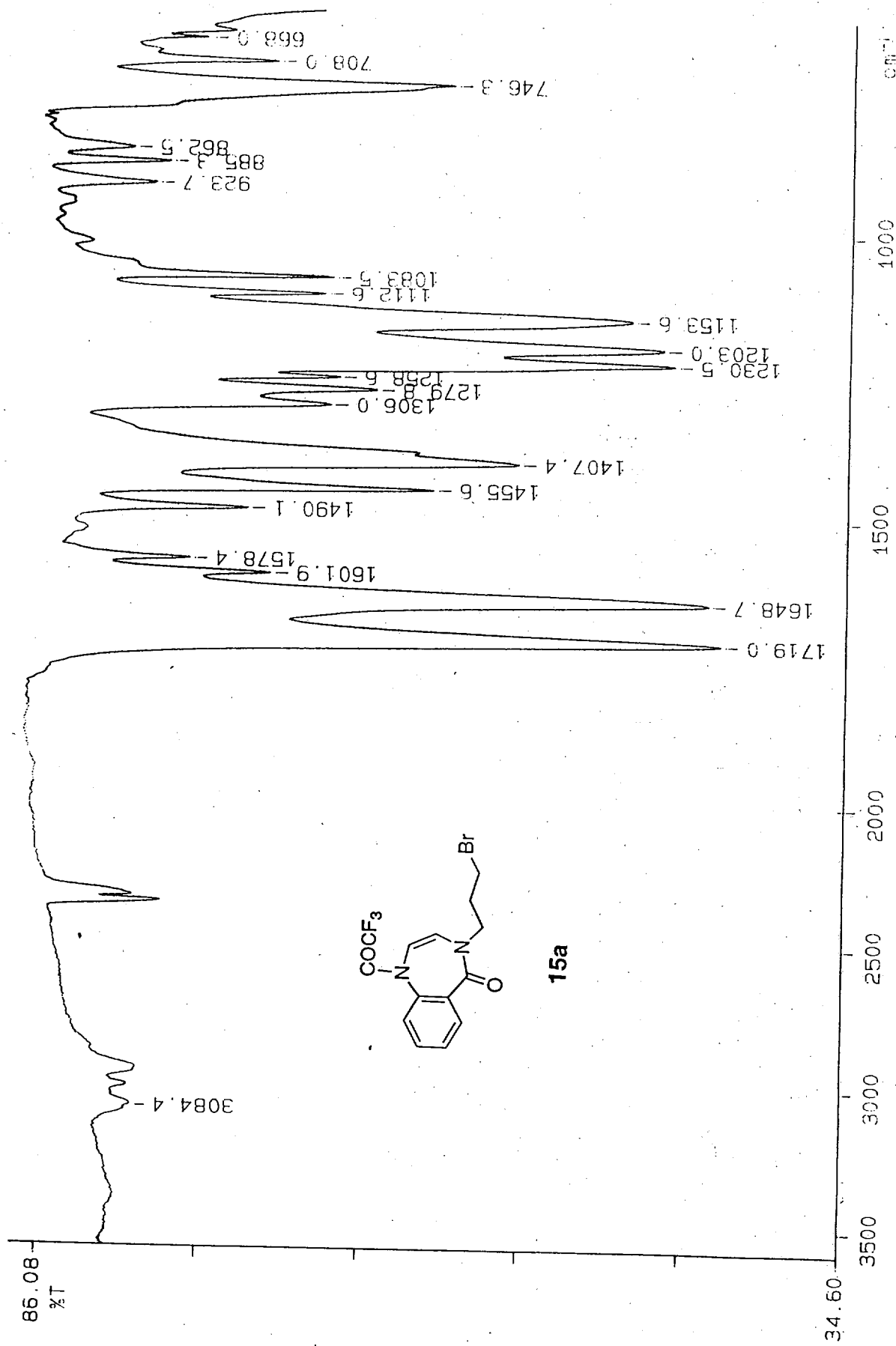


Operator : ACS
 Acquired : 21 Jan 98 10:50 am using AcqMethod GC100EI
 Instrument : 5989x - I
 Sample Name: ar0198004 SCAN 50-500
 Misc Info : GC/EI/100/300/10 30M DB-5
 Vial Number: 2

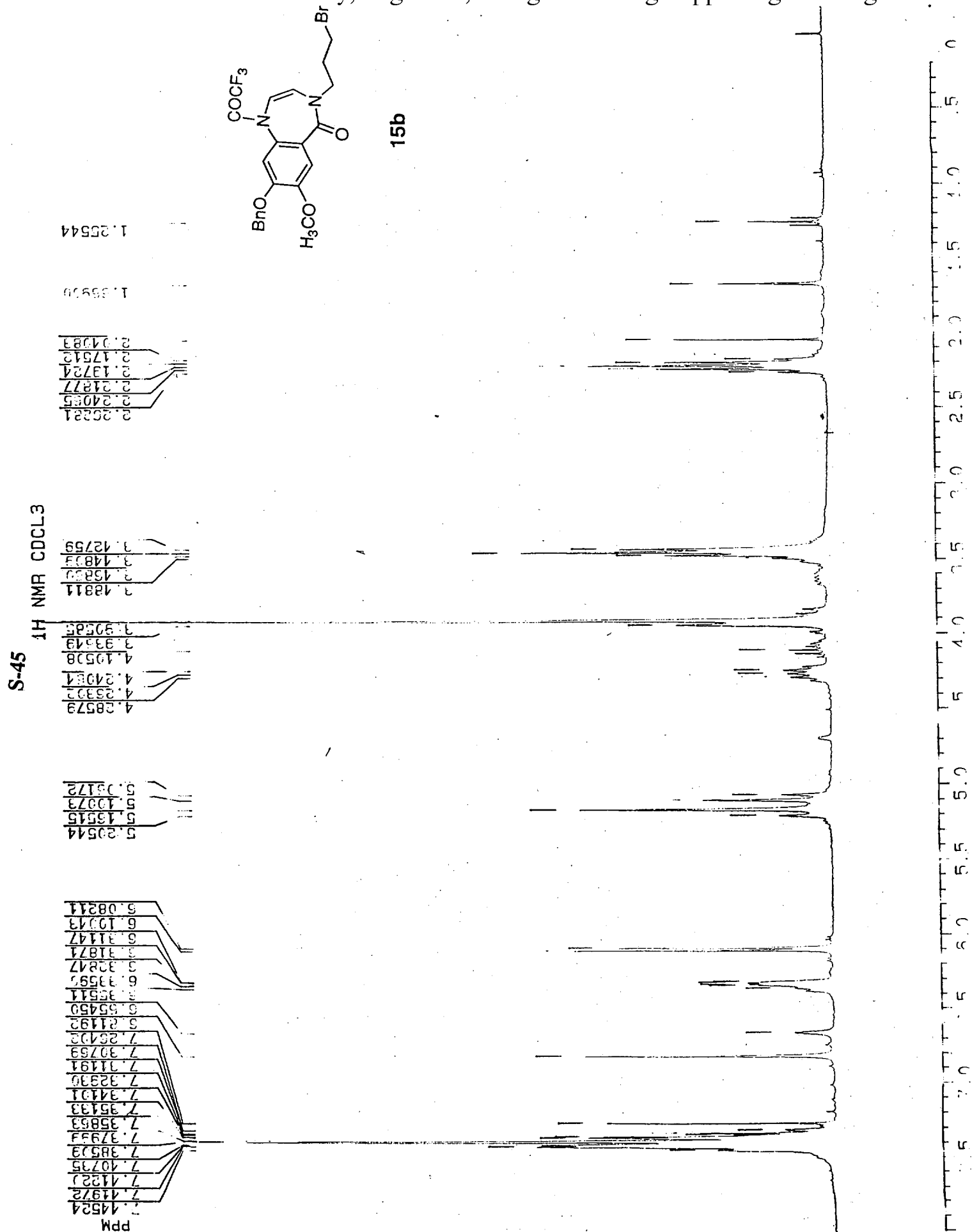
S-43



PERKIN ELMER S-44



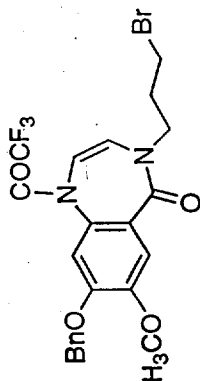
98/04/29 14.13
X. 4 scans, 4.0cm-1



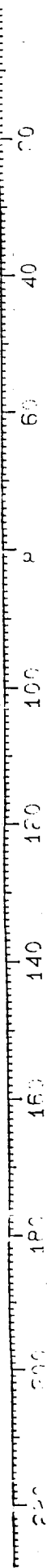
S-46 C13 NMR-CDCL3

167.545
166.012
155.530
155.043
154.551
154.534
151.254
149.683
149.254
135.575
134.570
133.028
132.353
130.779
129.855
129.484
128.527
128.603
128.444
128.222
127.455
127.259
127.044
125.850
122.554
122.234
121.725
119.048
117.893
116.575
114.074
113.240
109.710
77.372
77.152
76.948
76.525
71.090
68.057
55.249
56.158
47.252
38.643
31.213
30.279
30.104
28.831
29.660
22.885
13.950
11.837
5.509

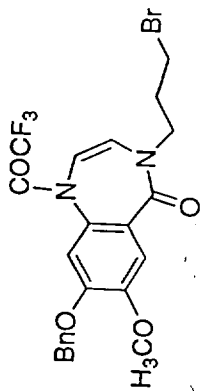
ppm



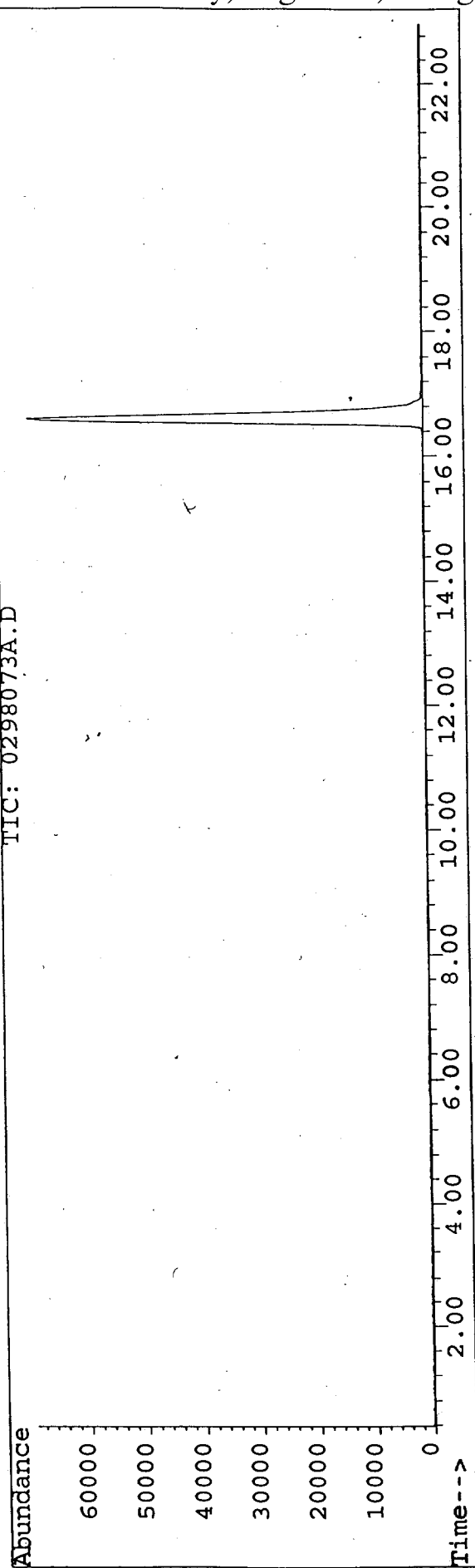
15b



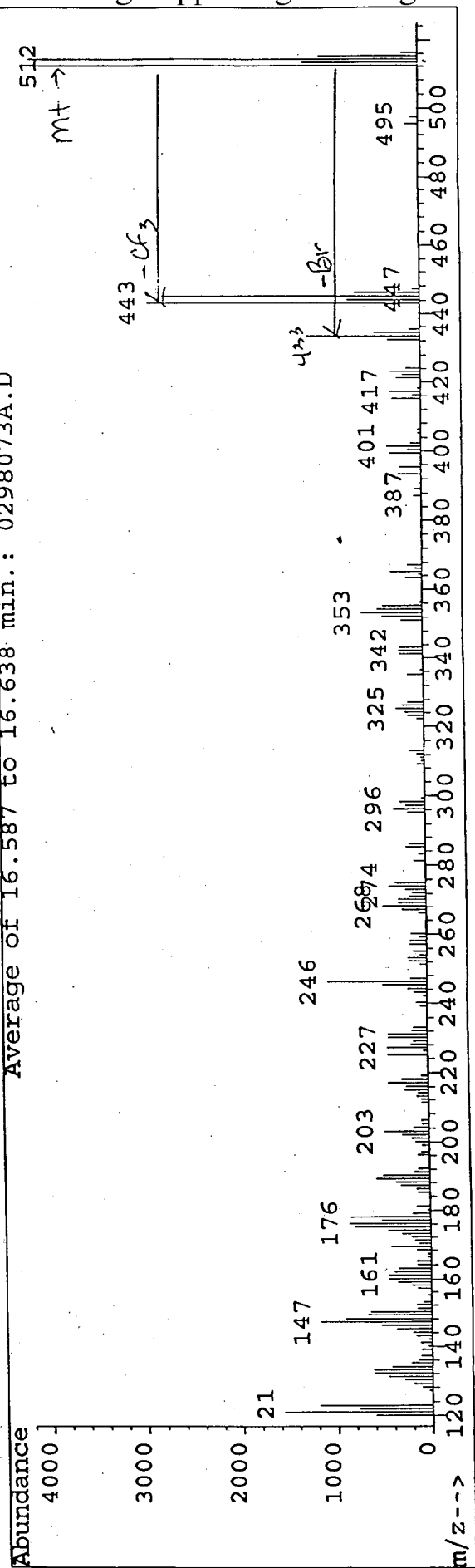
File : C:\HPCHEM\1\DATA\0298073A.D S-47
 Operator : acs
 Acquired : 11 Mar 98 9:21 am using AcqMethod PBGEI1
 Instrument : 5989x - I
 Sample Name: AR0298073 scan 100-600
 Misc Info : pb/ei/col 10/75/15-85/0/15
 Vial Number: 1



TIC: 0298073A.D

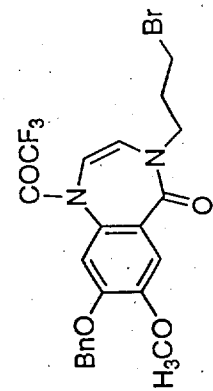
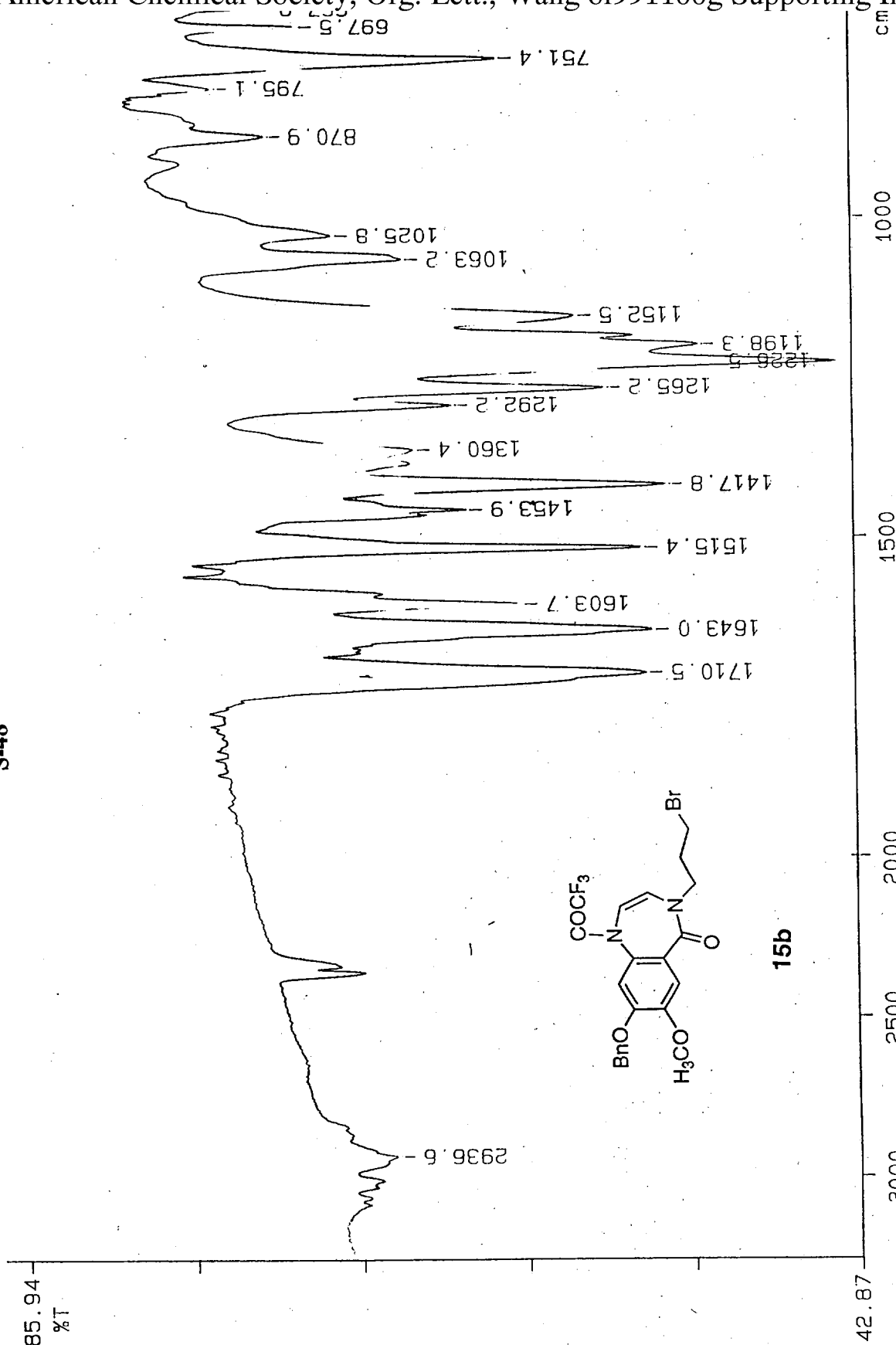


Average of 16.587 to 16.638 min.: 0298073A.D

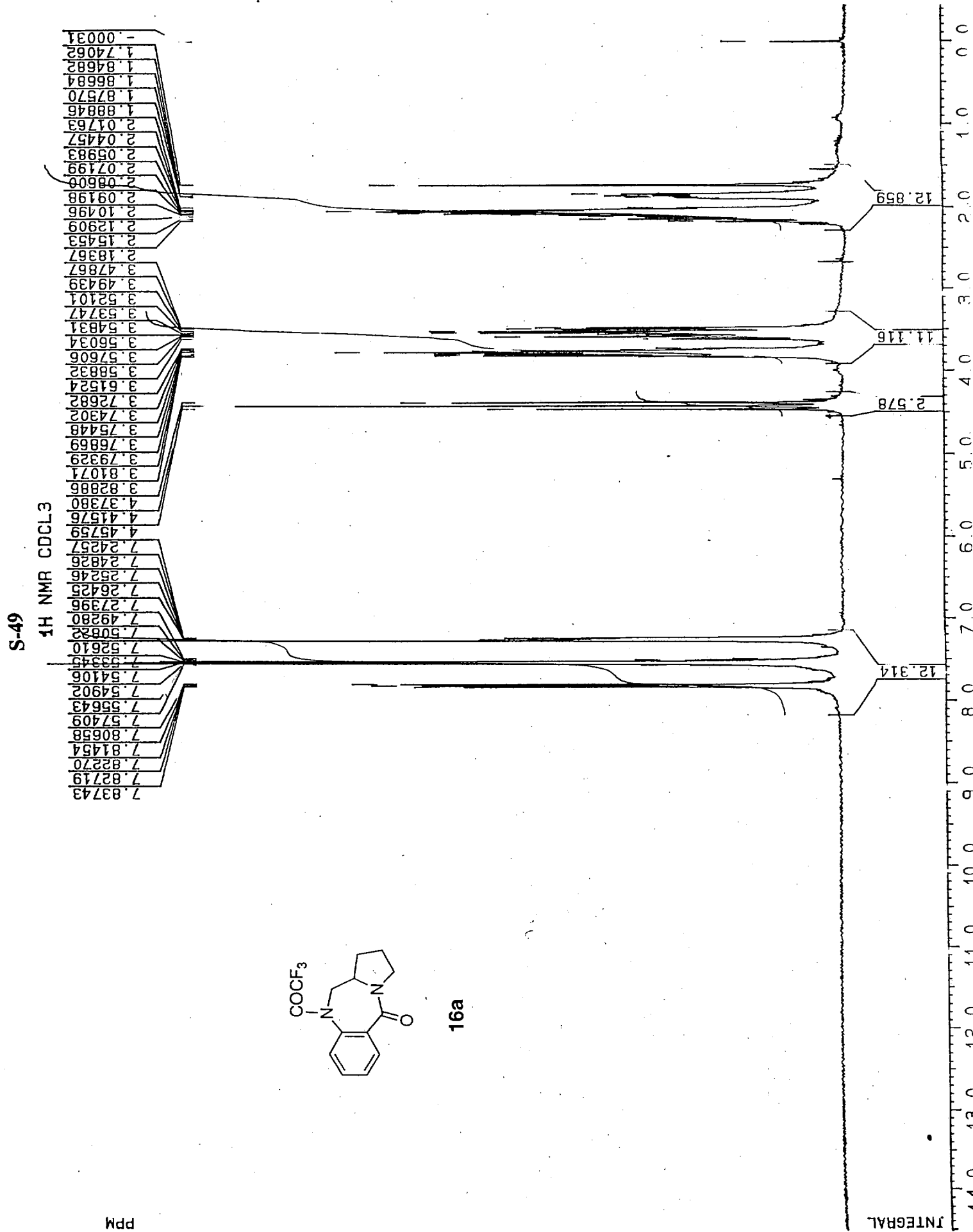


S-48

PERKIN ELMER

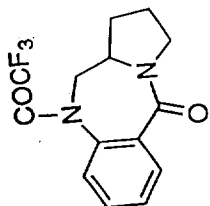


98/05/28 17:33
X: 4 scans, 4.0cm-1



S-50

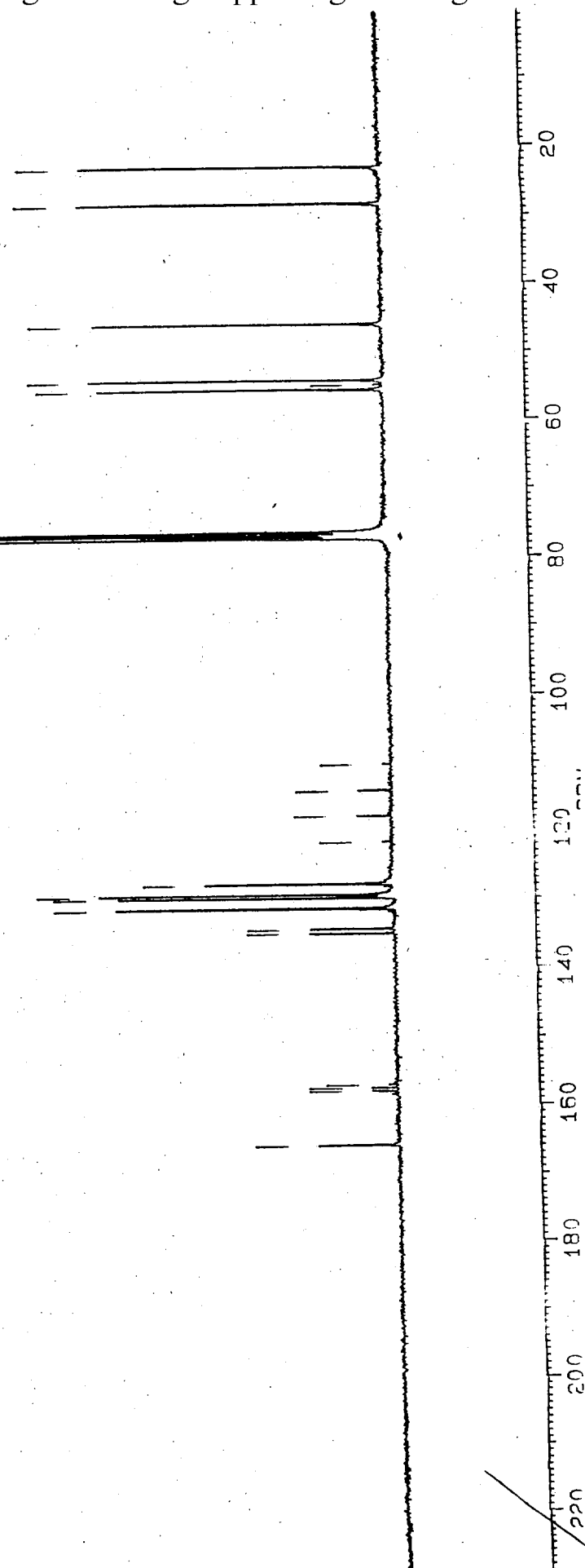
C13 NMR-CDCL3



16a

PPM

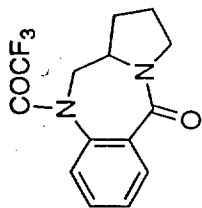
| |
|---------|
| 165.945 |
| 157.985 |
| 157.504 |
| 157.023 |
| 135.040 |
| 134.332 |
| 131.477 |
| 129.912 |
| 129.503 |
| 127.852 |
| 121.653 |
| 117.844 |
| 114.026 |
| 110.205 |
| 77.364 |
| 77.143 |
| 76.910 |
| 76.517 |
| 55.790 |
| 55.060 |
| 54.109 |
| 46.124 |
| 28.496 |
| 23.092 |



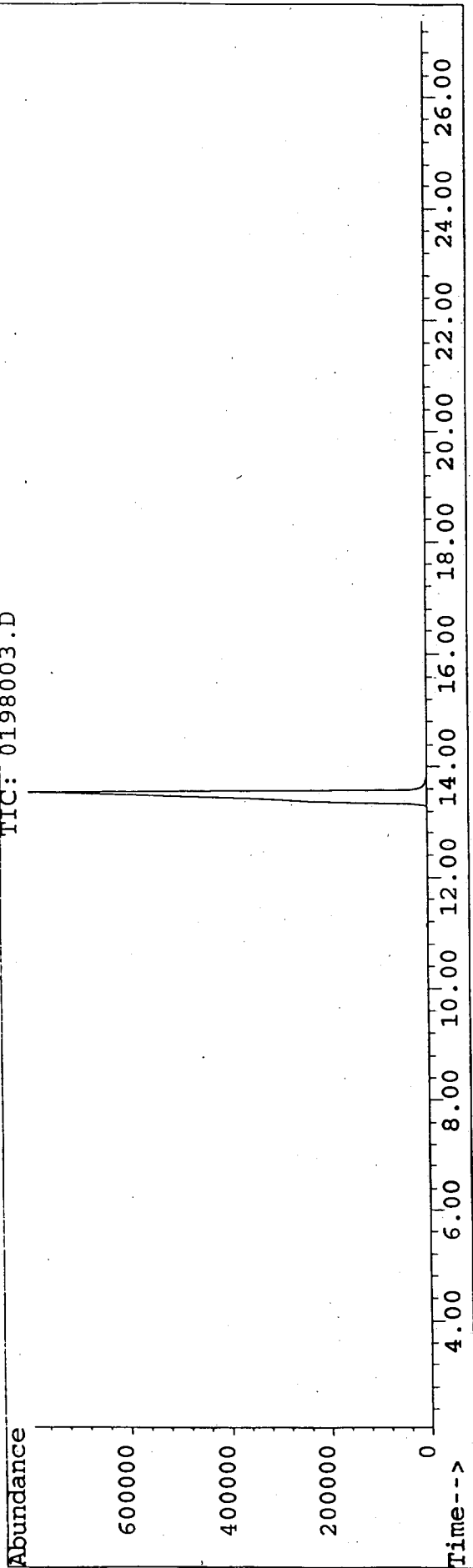
File : C:\HPCHEM\1\DATA\0198003.D

S-51

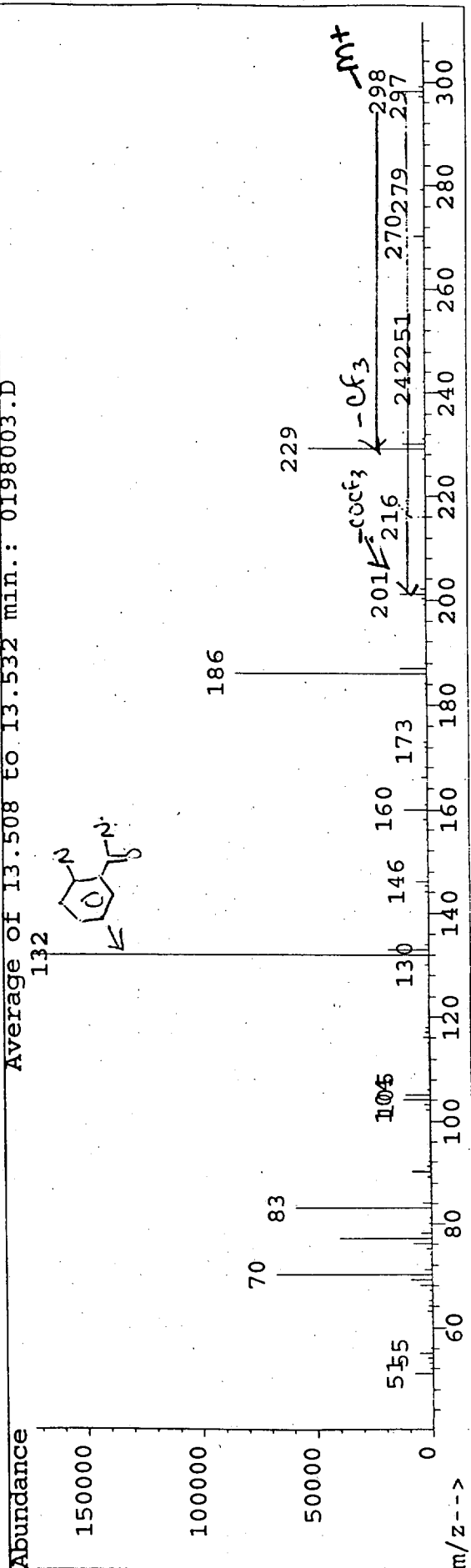
Operator : ACS
 Acquired : 21 Jan 98 10:16 am using AcqMethod GC100EI
 Instrument : 5989x - I
 Sample Name : ar0198003
 Misc Info : GC/EI/10//300/10
 Vial Number: 1

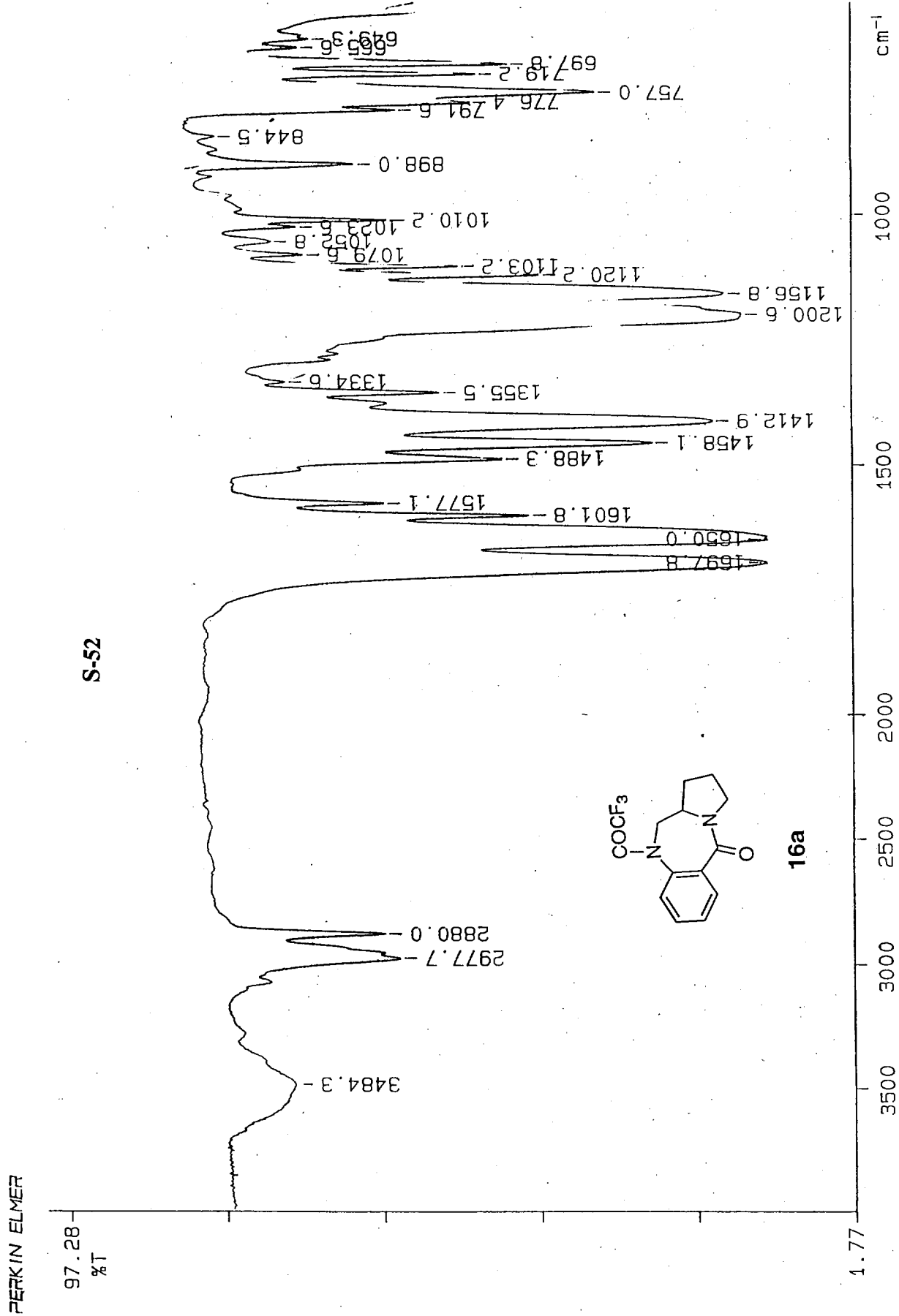


TIC: 0198003.D



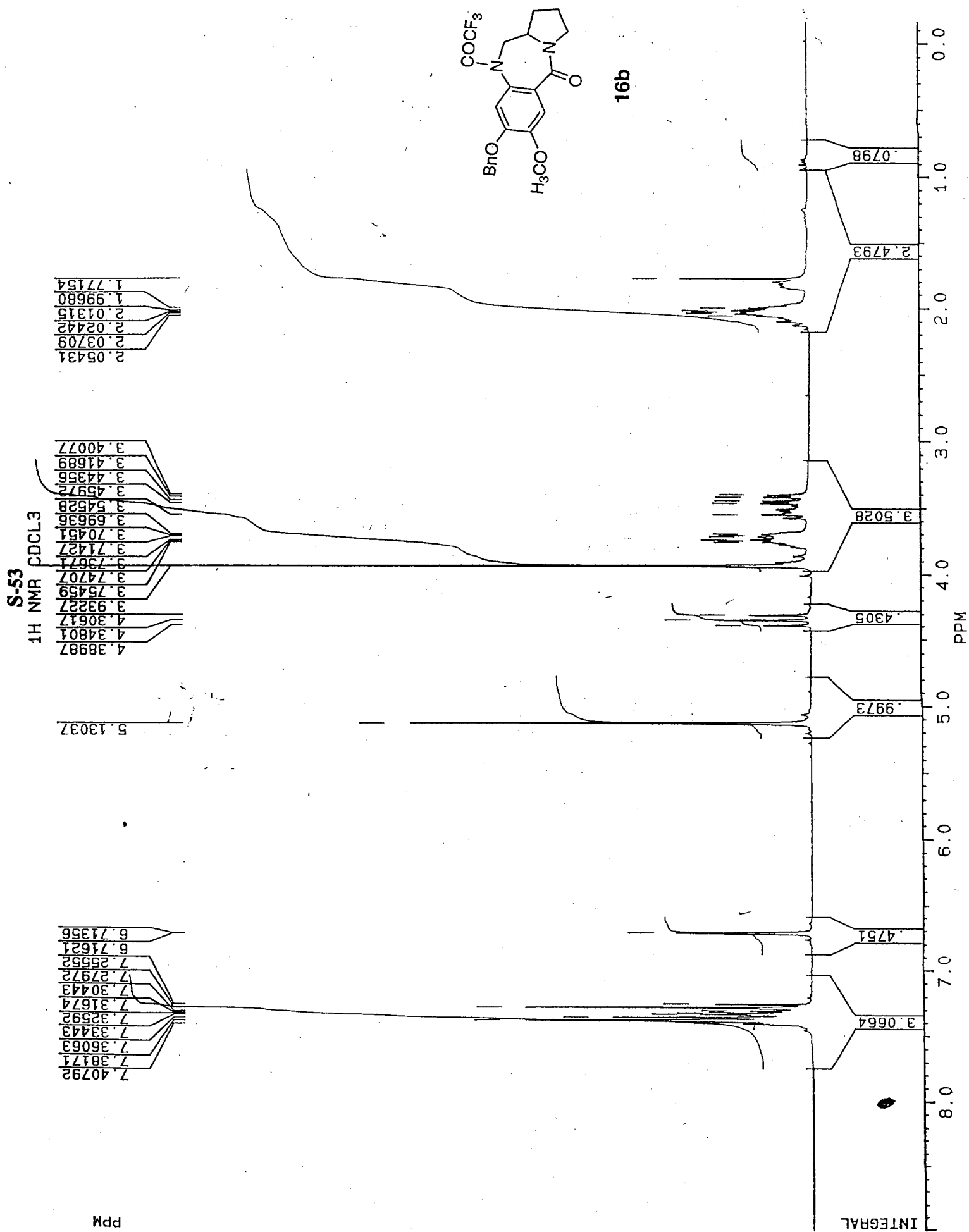
Average of 13.508 to 13.532 min.: 0198003.D





98/05/28 12:06

X: 4 scans, 4.0cm-1



S-54
6
G13 NMR-CDCL3

22.740
22.997
23.140
28.383
31.483

46.191
54.701
55.267
56.038
56.164

71.306
76.546
76.970
77.170
77.393

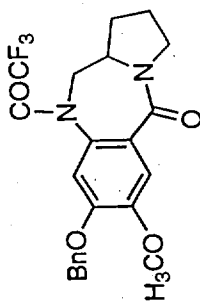
110.218
111.559
112.609
113.241
114.038
117.859
121.679
127.121
127.247
127.479
127.793
128.202
128.643
135.667

149.838
150.252
157.075
157.553
158.030
158.508
166.194

115.95

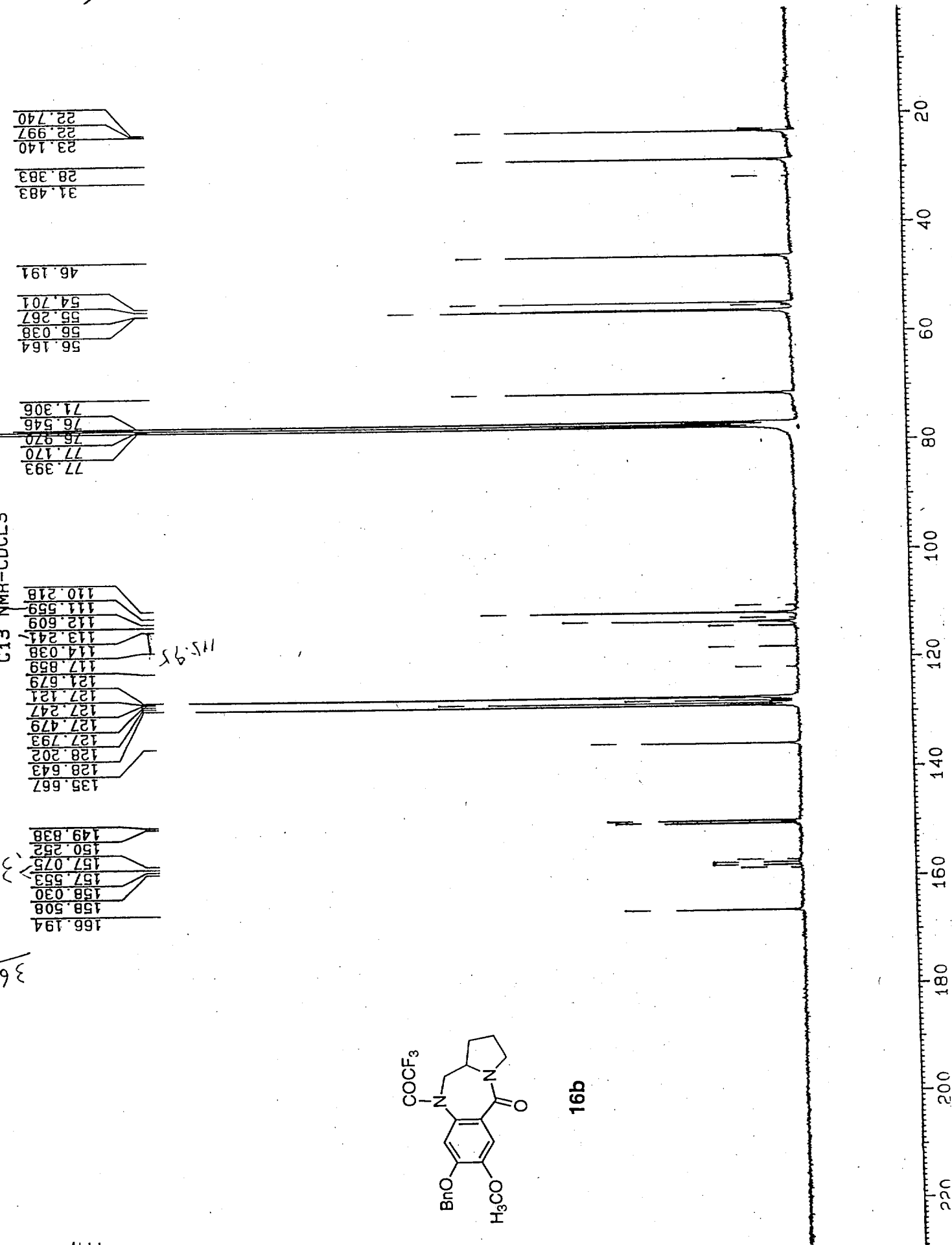
31

36



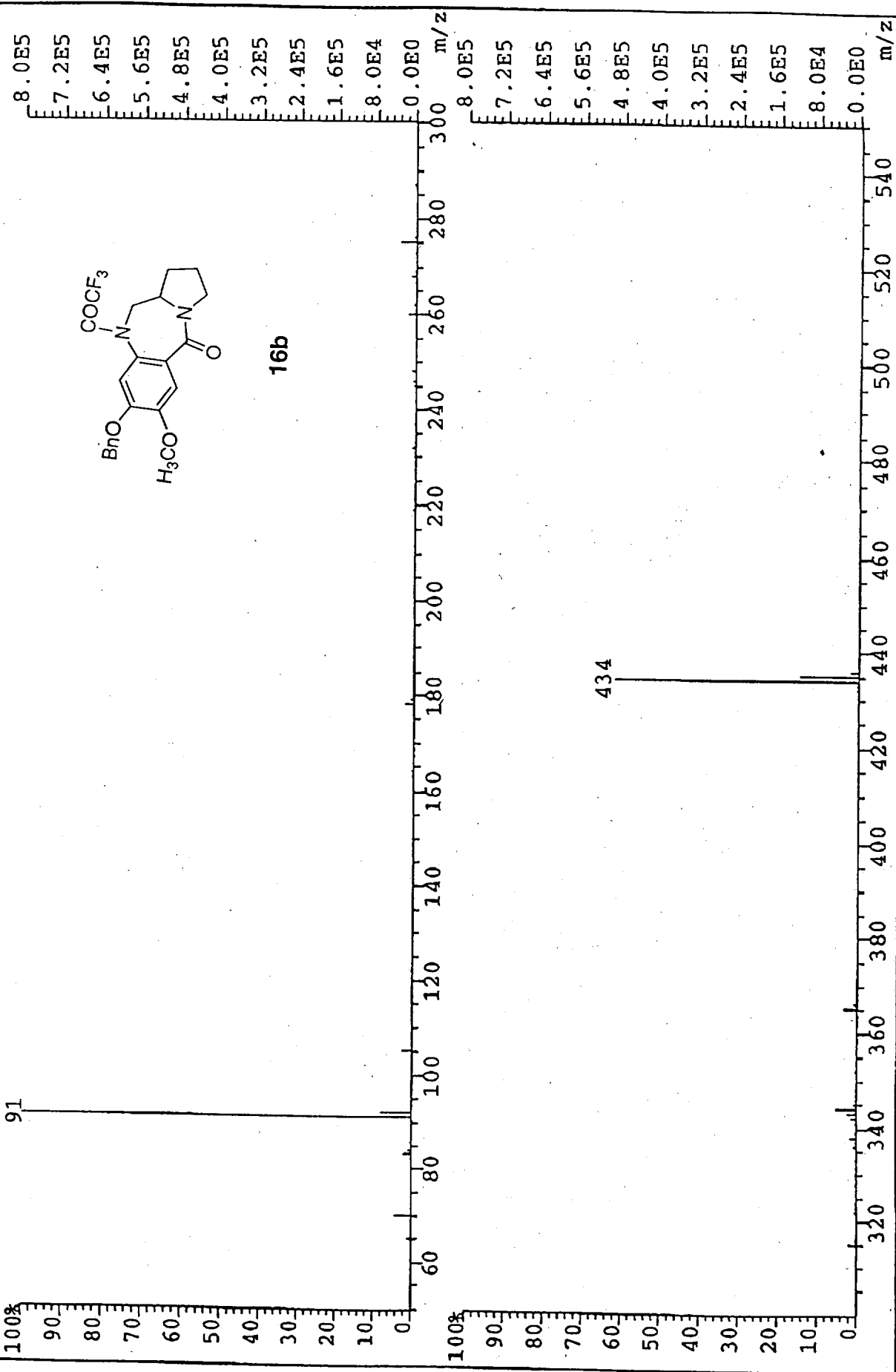
16b

Ppm



S-55

File: P6330 Ident: 32_39 Mer Def 0.25 Acq: 27-APR-1998 09:47:36 +4:28 Cal: P6330_1
ProSpec EI+ Magnet BpM: 91 BpI: 804416 TIC: 1945833 Flags: HALL
File Text: SJOLANDER AR0398035

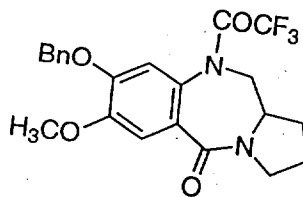


File: P6332 Ident: 11_23 Win 100PPM Acq: 27-APR-1998 10:56:14 +1:39 Cal: P6332
 ProSpec EI+ Voltage BpM: 434 BpI: 990996 TIC: 3217669 Flags: ACC
 File Text: SJOLANDER AR0398035

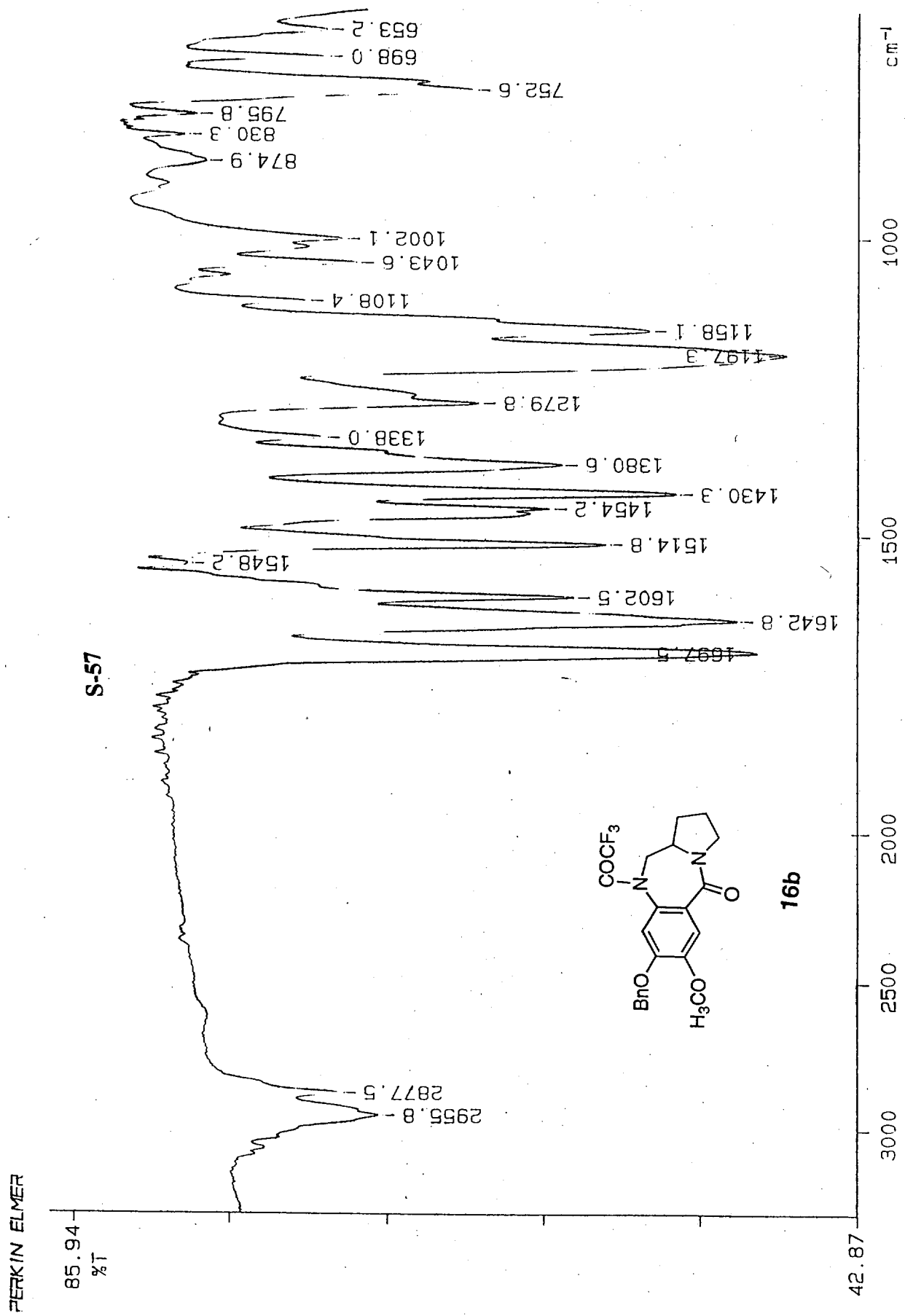
Heteroatom Max: 20 Ion: Both Even and Odd
 Limits:

| Mass | ΔRA | mDa | PPM | Calc. Mass | DBE | C | 13C | H | N | O | F |
|------------|-------|------|-------|------------|------|-----|-----|-----|---|---|---|
| 433.652 | 5.0 | | | | -0.5 | 0 | 0 | 0 | 0 | 0 | 3 |
| 435.502 | 100.0 | 10.0 | | | 20.0 | 200 | 1 | 400 | 4 | 6 | 3 |
| 435.148990 | 21.3 | 0.2 | 0.4 | 435.149145 | 7.5 | 17 | | 22 | 4 | 6 | 3 |
| | | -0.3 | -0.7 | 435.148697 | 12.0 | 21 | 1 | 21 | 2 | 4 | 3 |
| | | 2.4 | 5.5 | 435.151377 | 16.5 | 24 | 1 | 19 | 3 | 1 | 3 |
| | | 3.7 | 8.6 | 435.152720 | 16.0 | 26 | 1 | 21 | 2 | 2 | 3 |
| | | 4.2 | 9.6 | 435.153167 | 11.5 | 22 | | 22 | 2 | 4 | 3 |
| | | -4.3 | -9.9 | 435.144674 | 8.0 | 16 | 1 | 21 | 4 | 6 | 3 |
| | | -4.4 | -10.1 | 435.144614 | 16.0 | 26 | | 20 | 1 | 2 | 3 |
| | | -5.7 | -13.1 | 435.143271 | 16.5 | 24 | | 18 | 4 | 1 | 3 |
| | | 6.9 | 15.8 | 435.155847 | 16.0 | 25 | | 20 | 3 | 1 | 3 |
| | | -7.1 | -16.2 | 435.141934 | 11.5 | 23 | | 22 | | 5 | 3 |
| | | 8.2 | 18.8 | 435.157190 | 15.5 | 27 | | 22 | | 2 | 3 |
| | | 8.3 | 19.0 | 435.157250 | 7.5 | 17 | 1 | 23 | 3 | 6 | 3 |
| | | -8.4 | -19.3 | 435.140591 | 12.0 | 21 | | 20 | 3 | 4 | 3 |
| | | -8.8 | -20.3 | 435.140144 | 16.5 | 25 | 1 | 19 | 1 | 2 | 3 |
| 434.146275 | 100.0 | -0.9 | -2.1 | 434.145342 | 12.0 | 22 | | 21 | 2 | 4 | 3 |
| | | -1.4 | -3.2 | 434.144895 | 16.5 | 26 | 1 | 20 | 2 | 2 | 3 |
| | | 1.7 | 4.0 | 434.148022 | 16.5 | 25 | | 19 | 3 | 1 | 3 |
| | | -2.7 | -6.3 | 434.143552 | 17.0 | 24 | 1 | 18 | 3 | 1 | 3 |
| | | 3.1 | 7.1 | 434.149365 | 16.0 | 27 | | 21 | | 2 | 3 |
| | | 3.2 | 7.3 | 434.149425 | 8.0 | 17 | 1 | 22 | 3 | 6 | 3 |
| | | -5.0 | -11.4 | 434.141319 | 8.0 | 17 | | 21 | 4 | 6 | 3 |
| | | -5.4 | -12.4 | 434.140872 | 12.5 | 21 | 1 | 20 | 2 | 4 | 3 |
| | | 5.8 | 13.4 | 434.152105 | 12.5 | 20 | 1 | 20 | 4 | 3 | 3 |
| | | 7.2 | 16.5 | 434.153448 | 12.0 | 22 | 1 | 22 | 1 | 4 | 3 |
| | | 7.6 | 17.6 | 434.153896 | 7.5 | 18 | | 23 | 3 | 6 | 3 |
| | | -9.4 | -21.7 | 434.136849 | 8.5 | 16 | 1 | 20 | 4 | 6 | 3 |
| | | -9.5 | -21.9 | 434.136789 | 16.5 | 26 | | 19 | 1 | 2 | 3 |
| | | 9.9 | 22.7 | 434.156128 | 16.5 | 25 | 1 | 20 | 2 | 1 | 3 |

S-56

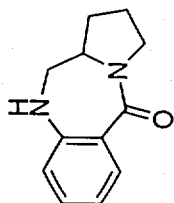


16b



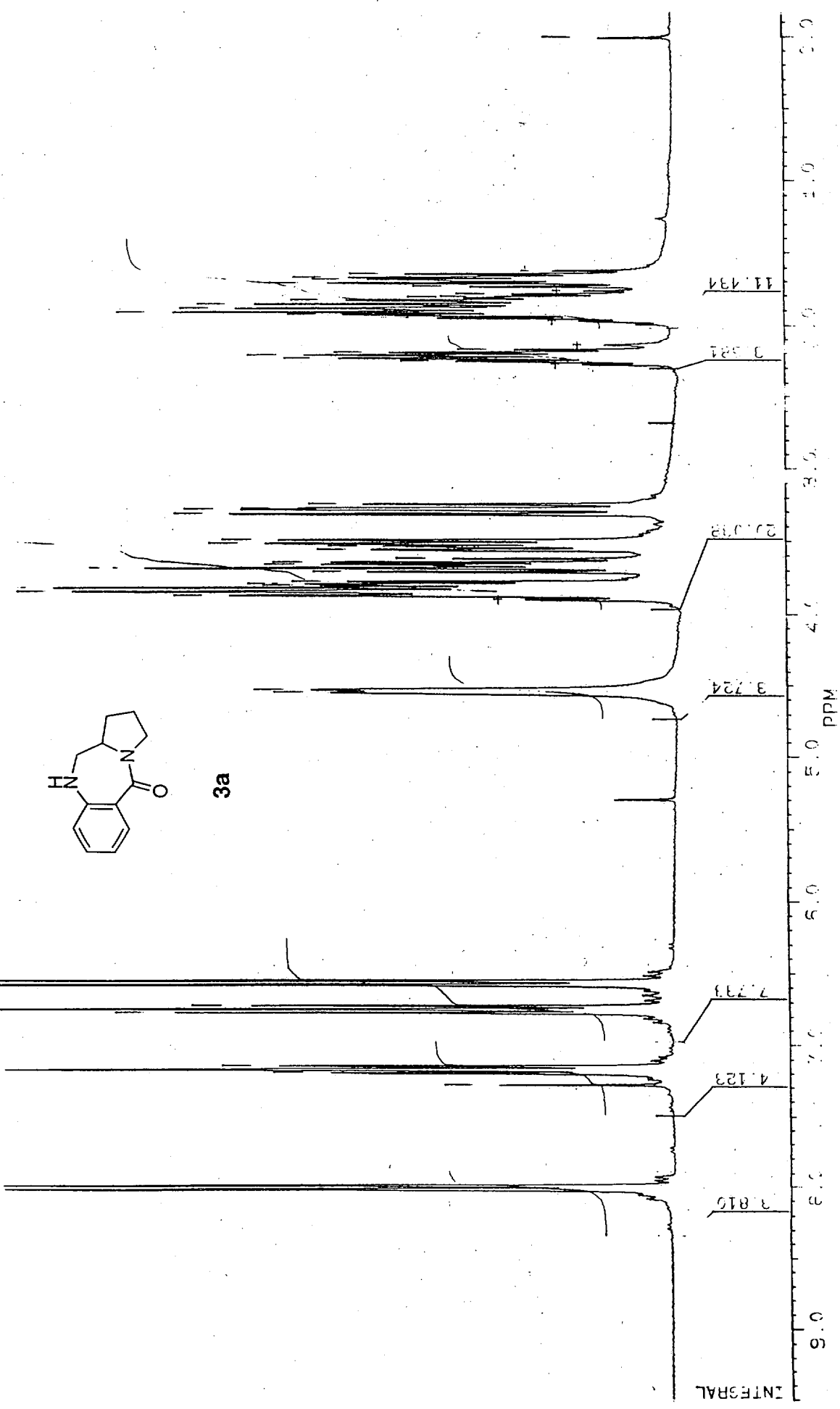
98/05/28 17: 24
X: 4 scans, 4.0cm-1

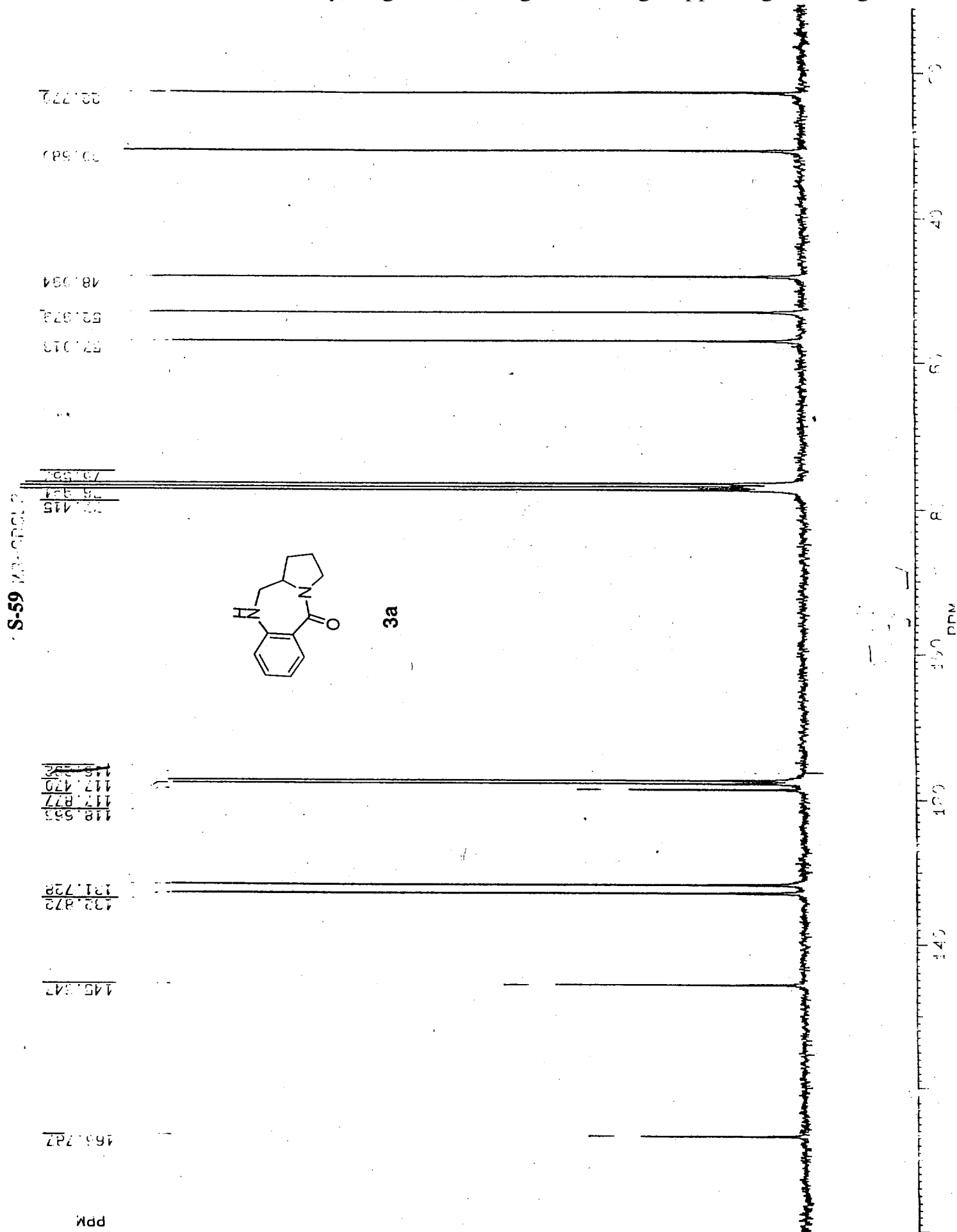
41 S-58 2D012



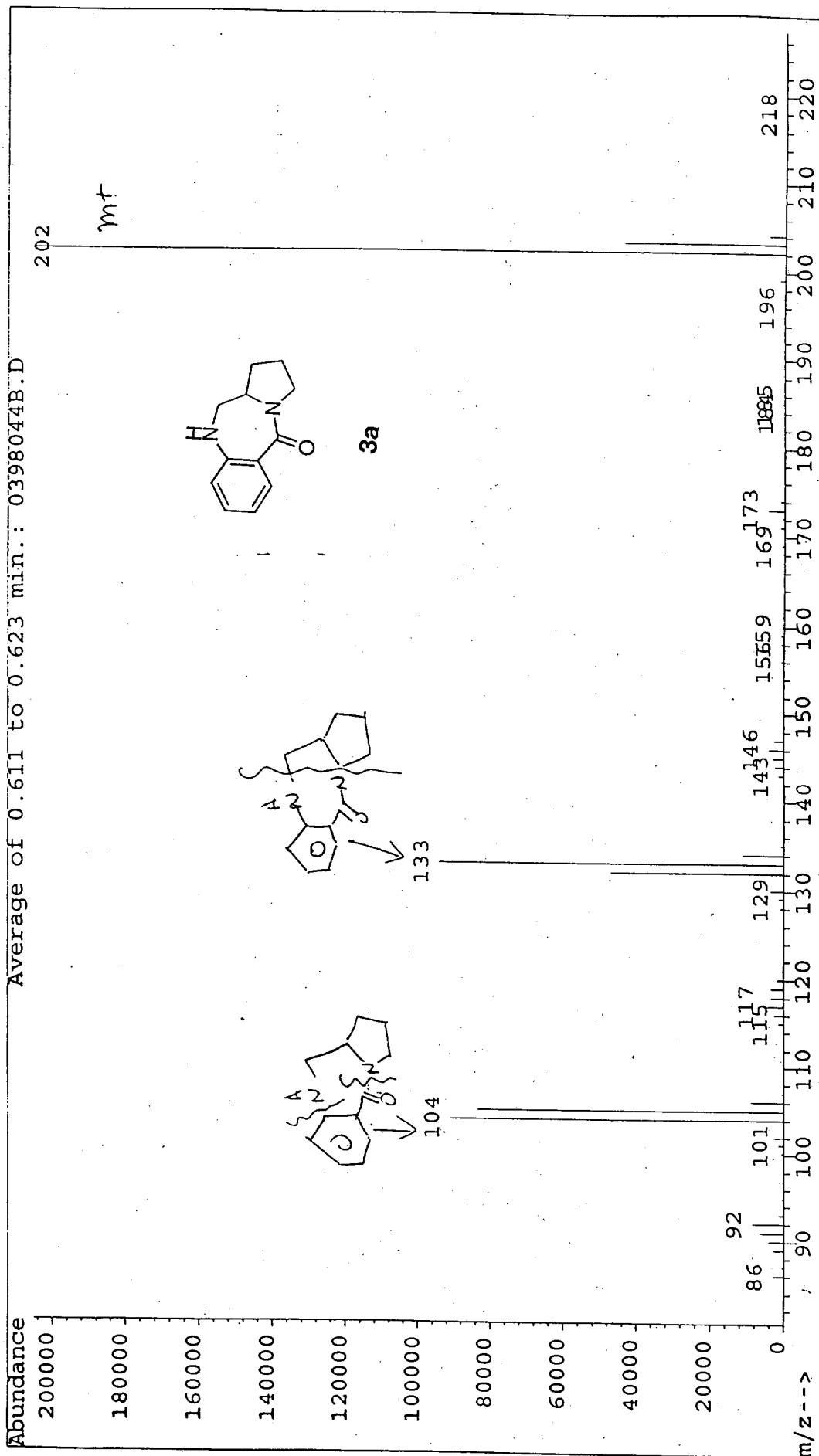
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| 4.54508 | 4.52583 | 3.85830 | 3.84081 | 3.81453 | 3.79852 | 3.79125 | 3.77400 | 3.70439 | 3.67950 | 3.65387 | 3.55487 | 3.53924 | 3.51428 | 3.50155 | 3.52874 | 3.51014 | 3.48735 | 3.31082 | 3.28075 | 3.27359 | 3.23978 | 2.25053 | 2.23154 | 2.20935 | 2.19119 | 2.16798 | 1.34023 | 1.32187 | 1.90477 | 1.87955 | 1.85555 | 1.85082 | 1.83555 | 1.80871 | 1.80302 | 1.78525 | 1.73178 | 1.70822 | 1.68270 | 1.66723 | 1.63927 |
|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|

| | | | | | | | | | | | |
|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| 8.01242 | 7.98554 | 7.27512 | 7.19018 | 7.15589 | 7.13953 | 6.75714 | 6.74847 | 6.74131 | 6.71595 | 6.57559 | 6.54847 |
|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|





File : C:\HPCHEM\1\DATA\0398044B.D S-60
 Operator : acs
 Acquired : 31 Mar 98 2:43 pm using AcqMethod PBGE11
 Instrument : 5989x - I
 Sample Name: ar0398044
 Misc Info : PB/EI/LOOP
 Vial Number: 3

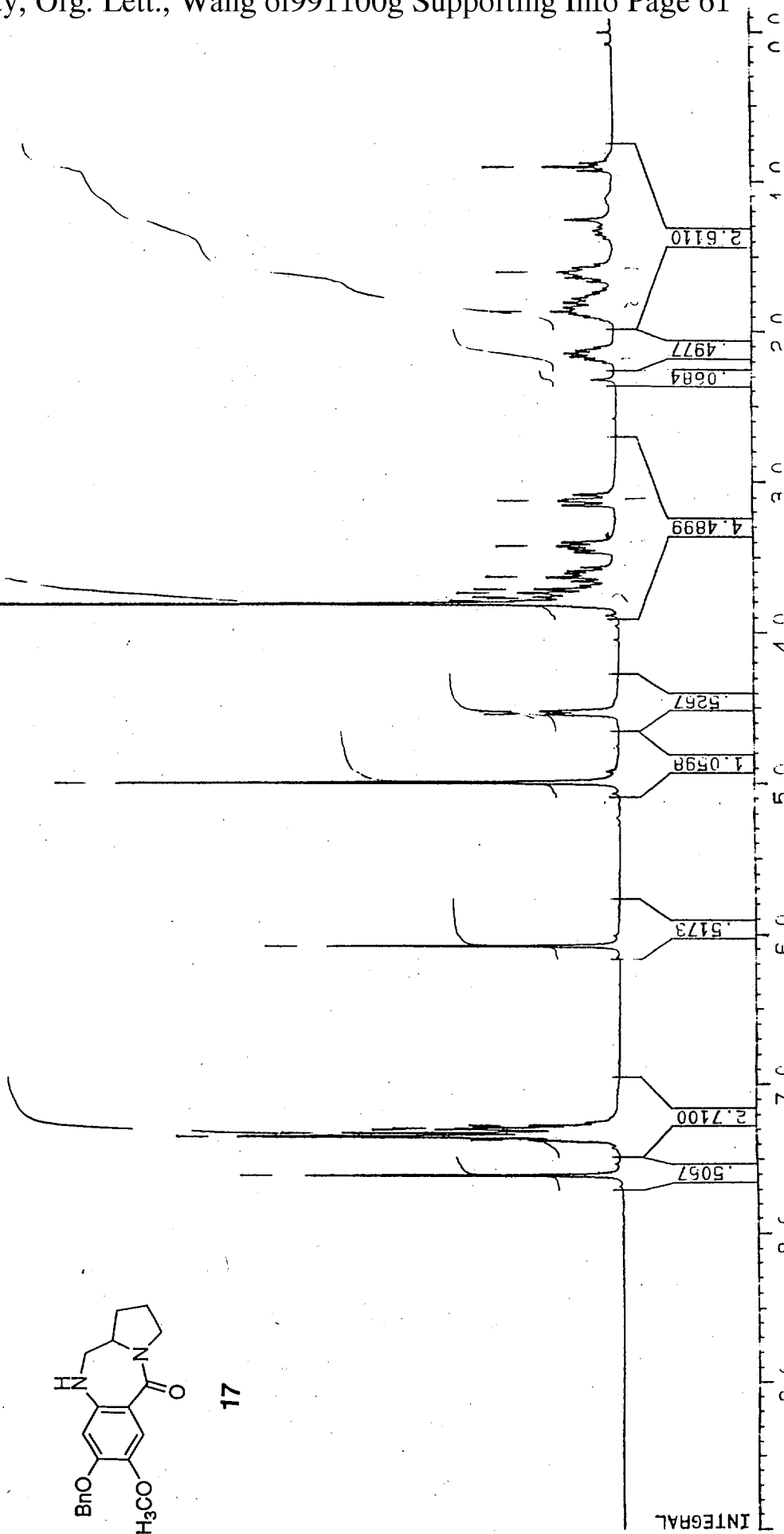
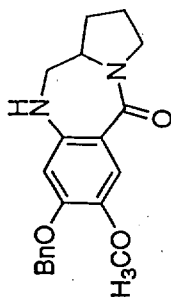


S-61

¹H NMR CDCL₃

| |
|---------|
| 7.59814 |
| 7.36677 |
| 7.34150 |
| 7.31924 |
| 7.29418 |
| 7.28731 |
| 7.27838 |
| 7.26602 |
| 6.07408 |
| 4.98862 |
| 4.53824 |
| 4.51813 |
| 3.80683 |
| 3.78816 |
| 3.77212 |
| 3.76168 |
| 3.73412 |
| 3.70908 |
| 3.62819 |
| 3.41870 |
| 3.11567 |
| 1.85983 |
| 1.60195 |
| 0.90900 |

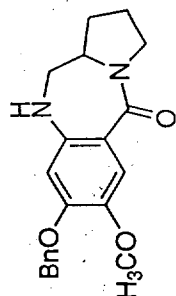
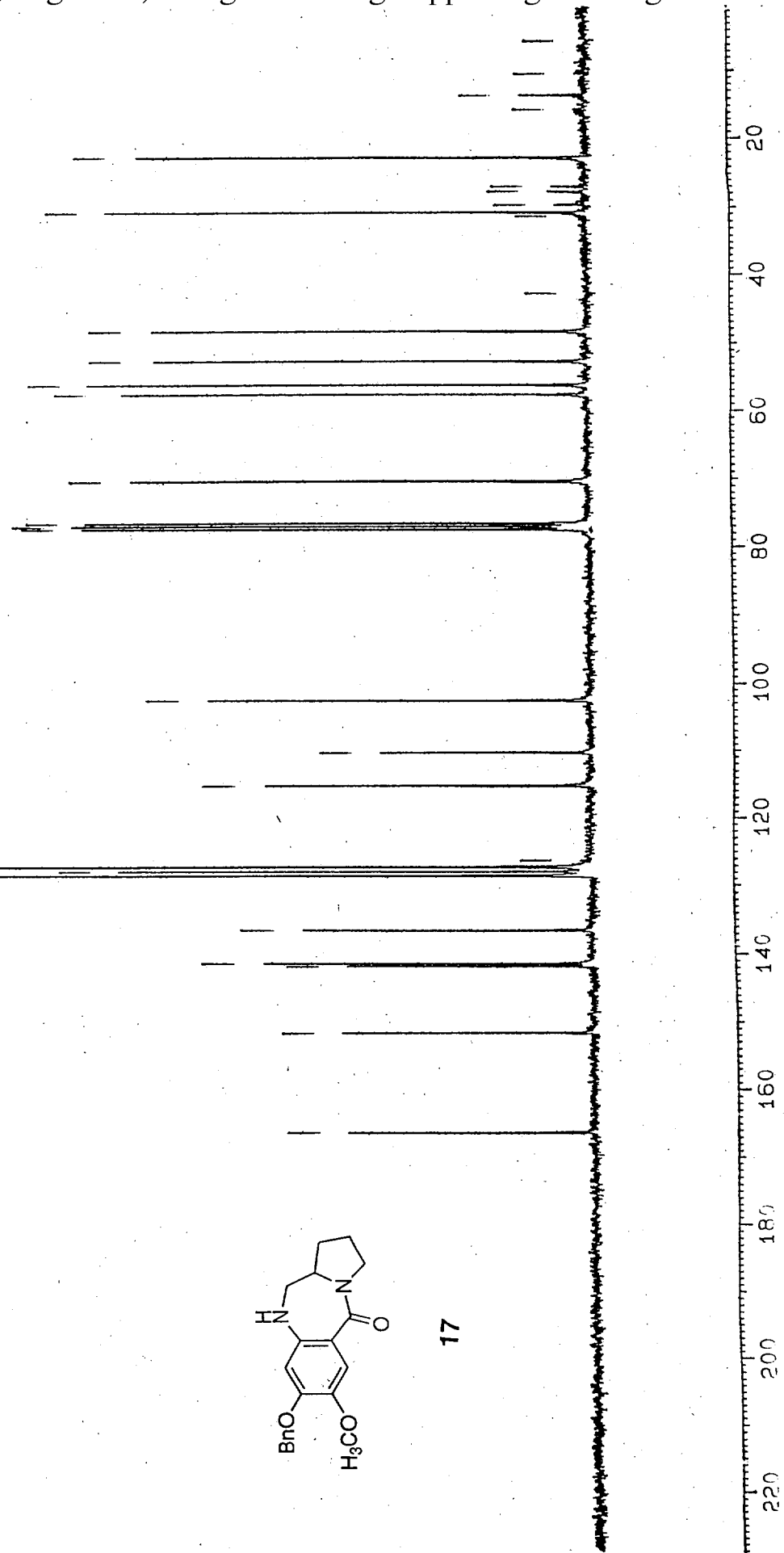
ppm



INTEGRAL

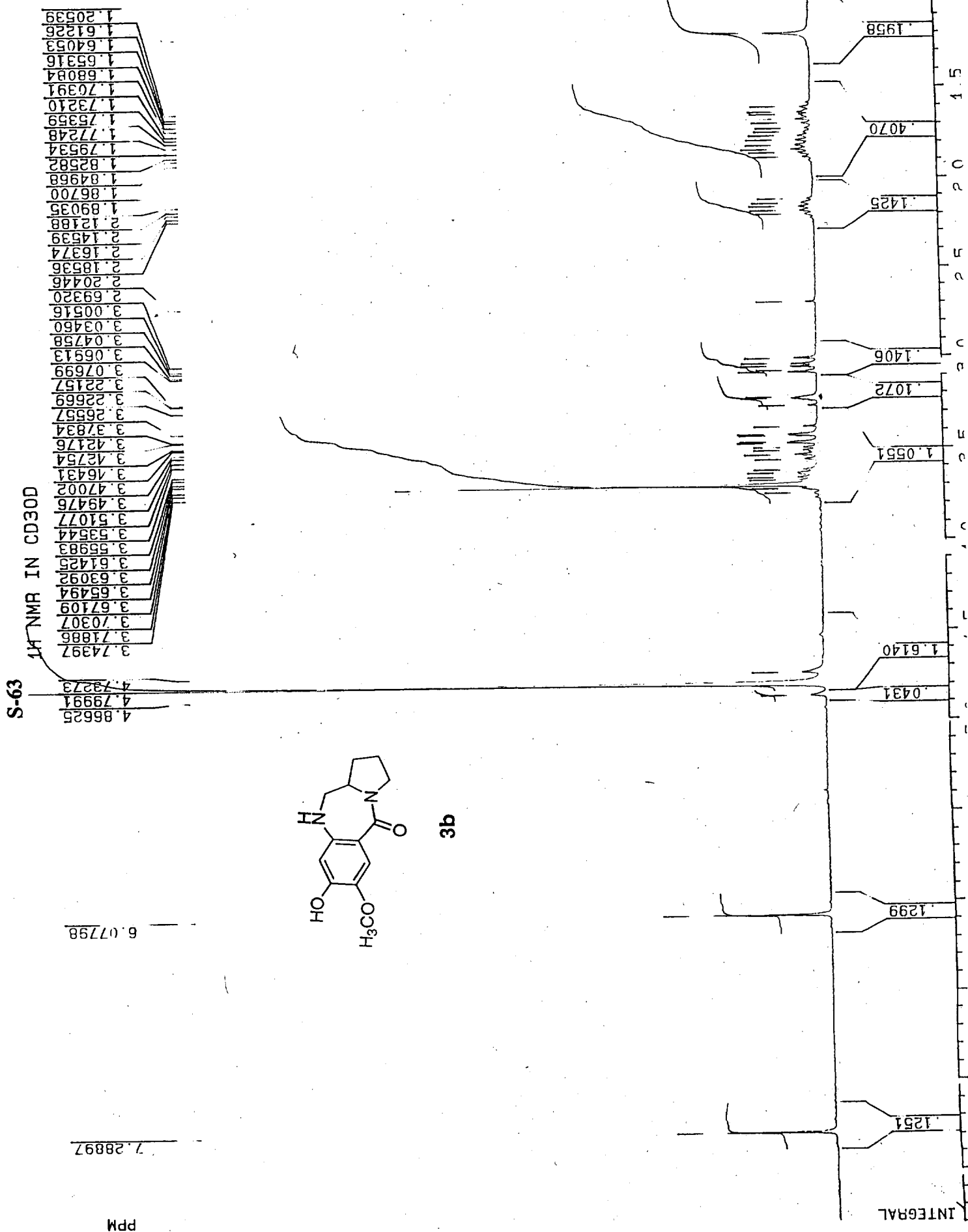
S-62
 C13 NMR-CDCL3

| |
|---------|
| 166.254 |
| 151.627 |
| 144.795 |
| 141.126 |
| 136.469 |
| 128.453 |
| 127.811 |
| 127.124 |
| 126.209 |
| 115.193 |
| 110.239 |
| 102.529 |
| 77.513 |
| 77.088 |
| 76.664 |
| 70.363 |
| 57.655 |
| 56.215 |
| 52.621 |
| 48.325 |
| 42.567 |
| 31.331 |
| 30.760 |
| 29.590 |
| 27.750 |
| 27.029 |
| 22.723 |
| 15.586 |
| 13.595 |
| 10.407 |
| 5.501 |



17

PPM



S-64
C13 IN METHANOL D4

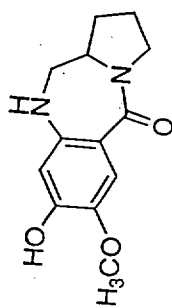
59.834
57.128
54.091
50.455
49.872
49.589
49.375
48.738
48.454
31.821
31.355
24.052

118.385
118.041
110.215
105.421

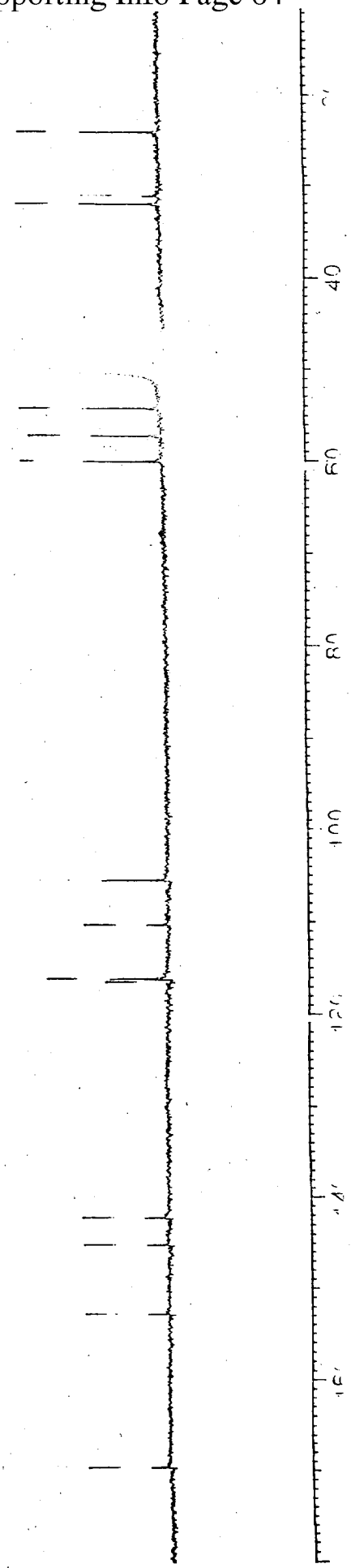
152.754
145.104
142.178

153.500

ppm



3b



File:P6366 Ident:30_46 Win 100PPM Acq: 6-MAY-1998 13:26:22 +3:35 Cal:P6366
 ProSpec EI+ Voltage BpM:248 BpI:728937 TIC:1620127 Flags:ACC
 File Text: SJOLANDER AR0498030
 Heteroatom Max: 20 Ion: Both Even and Odd
 Limits: .

| Mass | %RA | mDa | PPM | Calc. Mass | DBE | C | 13C | H | N | O |
|------------|-------|------|-------|------------|------|-----|-----|-----|---|---|
| 247.602 | 5.0 | | | | -0.5 | 0 | 0 | 0 | 0 | 0 |
| 249.452 | 100.0 | 10.0 | | | 20.0 | 200 | 1 | 400 | 4 | 6 |
| 249.118766 | 13.4 | 0.7 | 2.7 | 249.119447 | 7.0 | 12 | 1 | 16 | 2 | 3 |
| | | 1.1 | 4.5 | 249.119895 | 2.5 | 8 | | 17 | 4 | 5 |
| | | -2.0 | -8.0 | 249.116767 | 2.5 | 9 | 1 | 18 | 1 | 6 |
| | | 2.5 | 9.9 | 249.121238 | 2.0 | 10 | | 19 | 1 | 6 |
| | | -3.3 | -13.4 | 249.115425 | 3.0 | 7 | 1 | 16 | 4 | 5 |
| | | 3.4 | 13.5 | 249.122127 | 11.5 | 15 | 1 | 14 | 3 | |
| | | -3.4 | -13.7 | 249.115364 | 11.0 | 17 | | 15 | 1 | 1 |
| | | 4.7 | 18.9 | 249.123470 | 11.0 | 17 | 1 | 16 | | 1 |
| | | -4.7 | -19.0 | 249.114022 | 11.5 | 15 | | 13 | 4 | |
| | | 5.2 | 20.7 | 249.123918 | 6.5 | 13 | | 17 | 2 | 3 |
| | | -6.1 | -24.4 | 249.112684 | 6.5 | 14 | | 17 | | 4 |
| | | -7.4 | -29.8 | 249.111342 | 7.0 | 12 | | 15 | 3 | 3 |
| | | 7.8 | 31.4 | 249.126598 | 11.0 | 16 | | 15 | 3 | |
| | | -7.9 | -31.6 | 249.110894 | 11.5 | 16 | 1 | 14 | 1 | 1 |
| | | 9.2 | 36.8 | 249.127940 | 10.5 | 18 | | 17 | | 1 |
| | | -9.2 | -37.0 | 249.109551 | 12.0 | 14 | 1 | 12 | 4 | |
| 248.115965 | 100.0 | 0.1 | 0.5 | 248.116093 | 7.0 | 13 | | 16 | 2 | 3 |
| | | -0.3 | -1.3 | 248.115645 | 11.5 | 17 | 1 | 15 | | 1 |
| | | -1.7 | -6.7 | 248.114302 | 12.0 | 15 | 1 | 13 | 3 | |
| | | -2.6 | -10.3 | 248.113413 | 2.5 | 10 | | 18 | 1 | 6 |
| | | 2.8 | 11.3 | 248.118773 | 11.5 | 16 | | 14 | 3 | |
| | | -3.9 | -15.7 | 248.112070 | 3.0 | 8 | | 16 | 4 | 5 |
| | | 4.1 | 16.7 | 248.120115 | 11.0 | 18 | | 16 | | 1 |
| | | 4.2 | 17.0 | 248.120176 | 3.0 | 8 | 1 | 17 | 3 | 5 |
| | | -4.3 | -17.5 | 248.111622 | 7.5 | 12 | 1 | 15 | 2 | 3 |
| | | 5.6 | 22.4 | 248.121518 | 2.5 | 10 | 1 | 19 | | 6 |
| | | 6.9 | 27.8 | 248.122856 | 7.5 | 11 | 1 | 15 | 4 | 2 |
| | | -7.0 | -28.3 | 248.108942 | 3.0 | 9 | 1 | 17 | 1 | 6 |
| | | 8.2 | 33.2 | 248.124198 | 7.0 | 13 | 1 | 17 | 1 | 3 |
| | | -8.4 | -33.7 | 248.107600 | 3.5 | 7 | 1 | 15 | 4 | 5 |
| | | -8.4 | -34.0 | 248.107539 | 11.5 | 17 | | 14 | 1 | 1 |
| | | 8.7 | 35.0 | 248.124646 | 2.5 | 9 | | 18 | 3 | 5 |
| | | -9.8 | -39.4 | 248.106197 | 12.0 | 15 | | 12 | 4 | |

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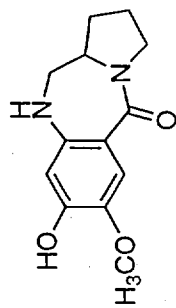


- 1 -

3b

S-66

File: P6358 Ident: 16_27 Mer Def 0.25 Acq: 6-MAY-1998 10:55:33 +2:39 Cal: P6357_1
ProSpec EI+ Magnet BpM: 248 BpI: 192082 TIC: 543109 Flags: HALL
File Text: SJOLANDER AR0498030



3b

