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Synthesis of enantiomerically pure thiocrown ethers derived from 1,1'-binaphthalene-2,2'-diol

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Published in:
Journal of Organic Chemistry

DOI:
[10.1021/jo952107o](https://doi.org/10.1021/jo952107o)

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Document Version
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Publication date:
1996

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Citation for published version (APA):

Stock, H. T., & Kellogg, R. M. (1996). Synthesis of enantiomerically pure thiocrown ethers derived from 1,1'-binaphthalene-2,2'-diol. *Journal of Organic Chemistry*, 61(9), 3093-3105.
<https://doi.org/10.1021/jo952107o>

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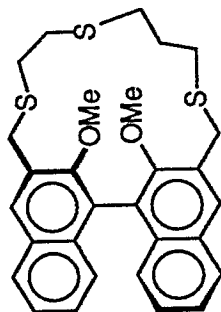
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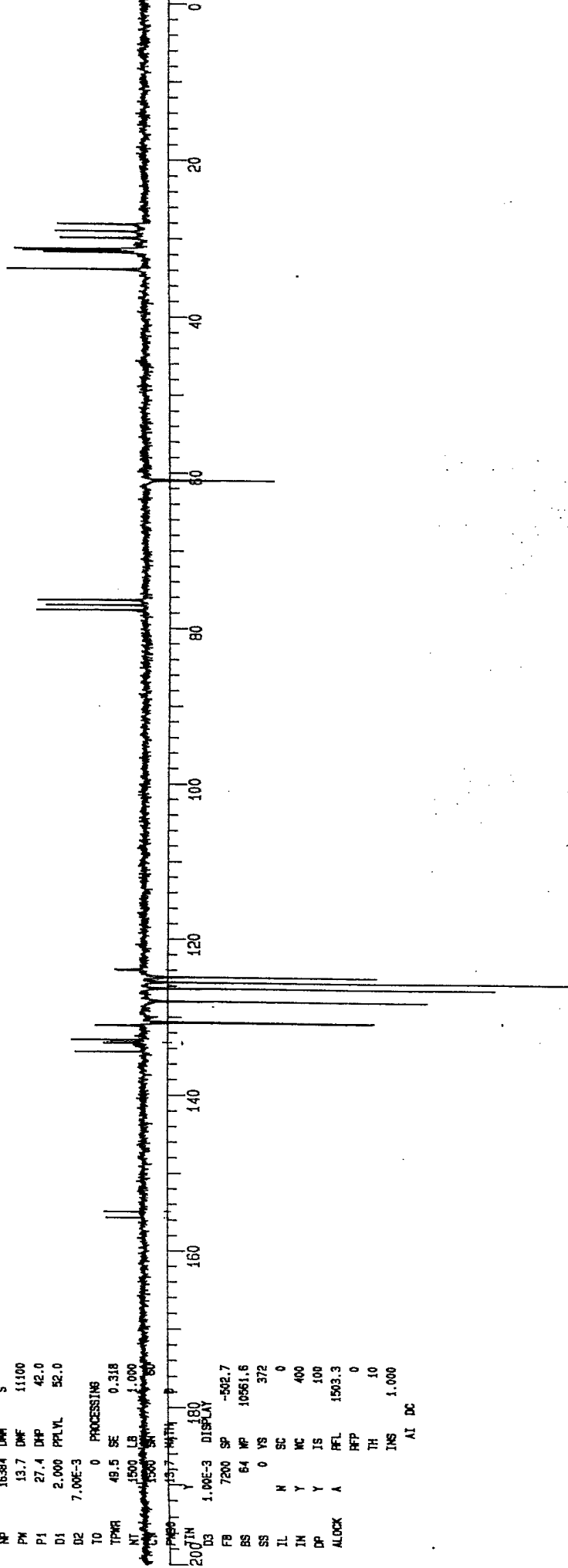
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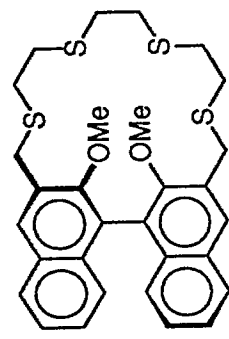
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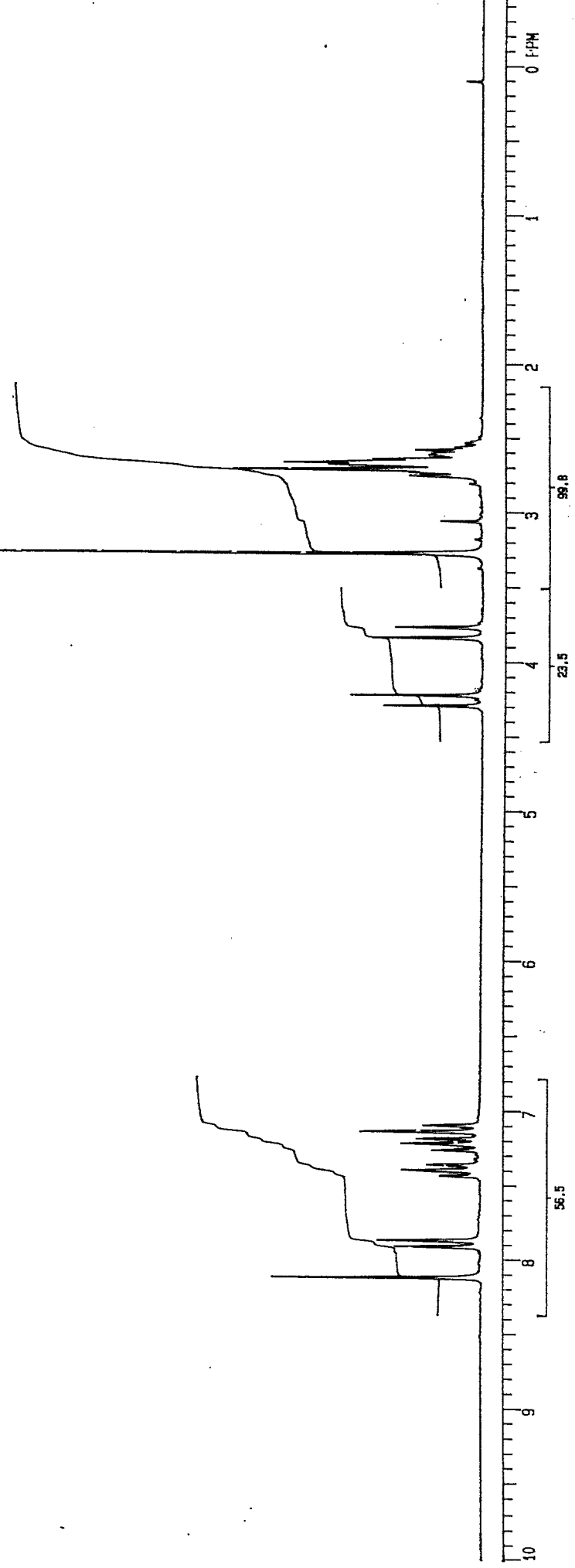
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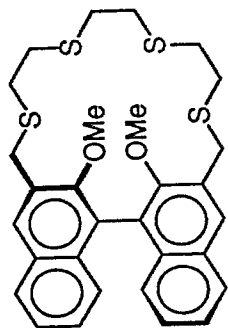
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BS 64 WP 10561.6
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42

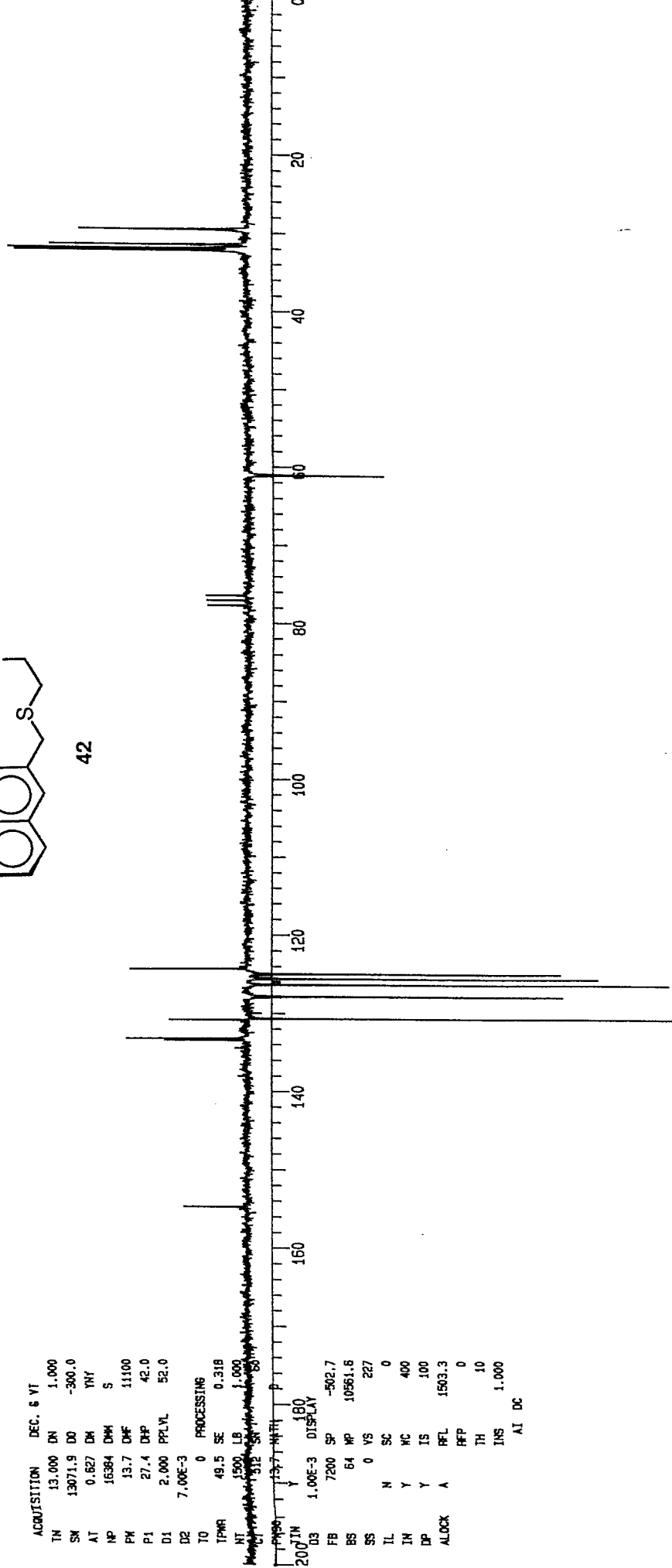




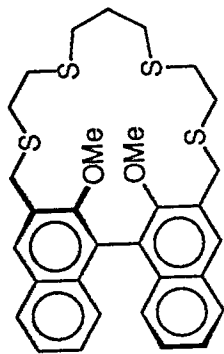
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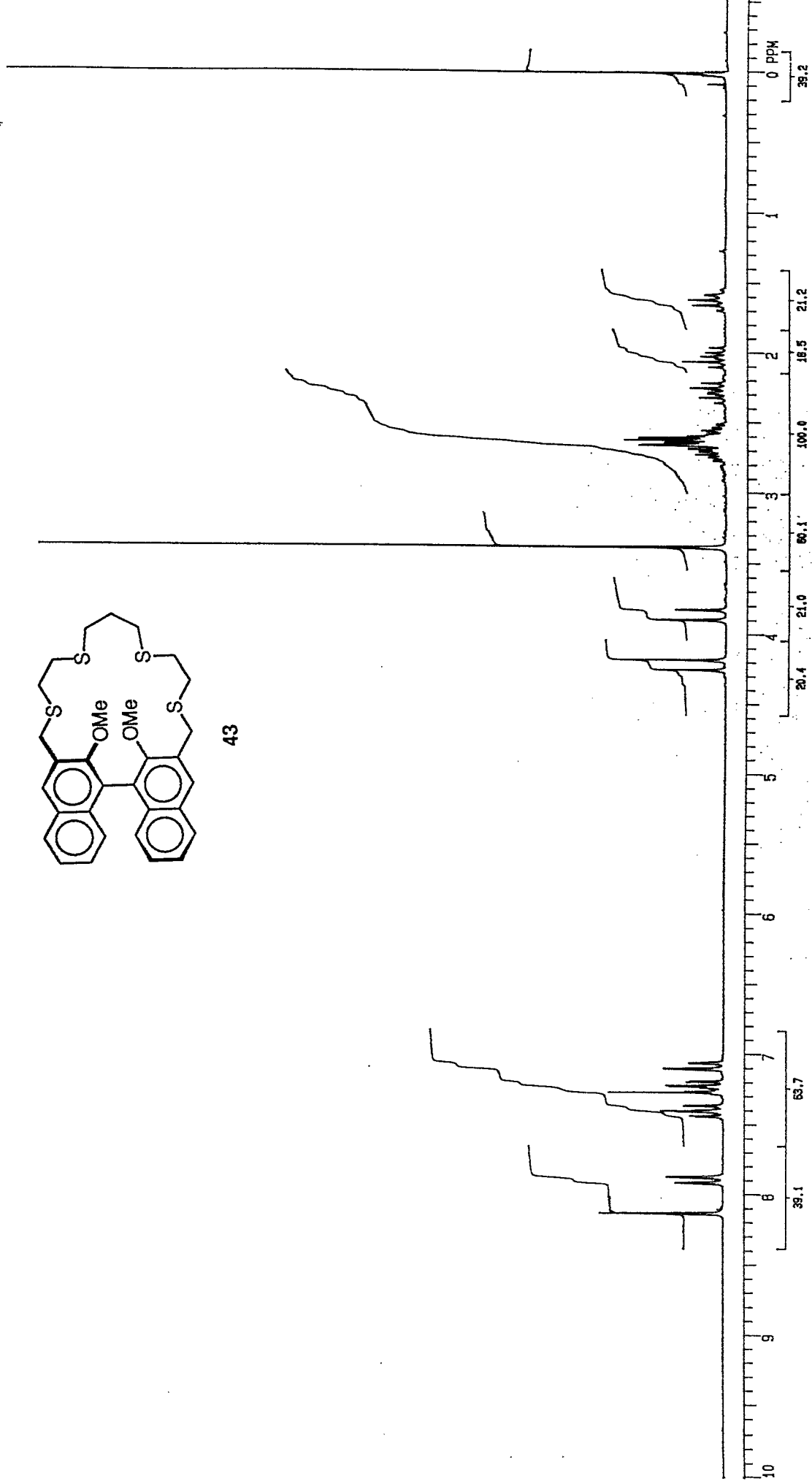
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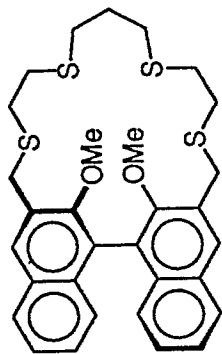


^1H NMR (CDCl_3 , 200 MHz)



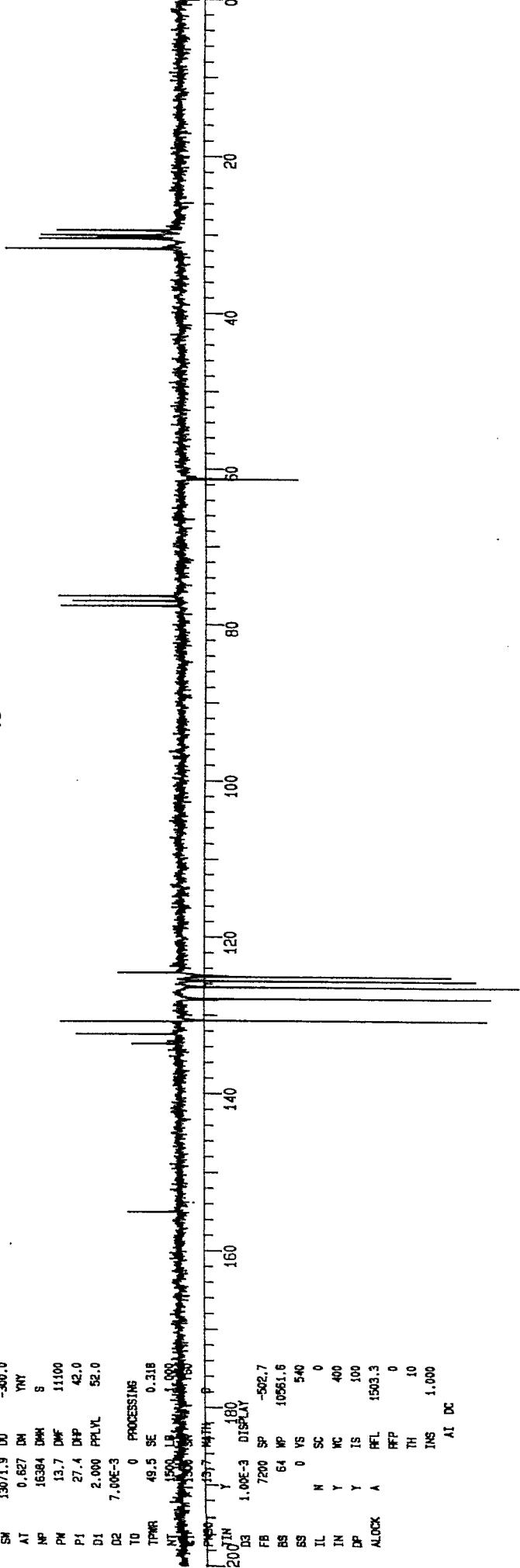
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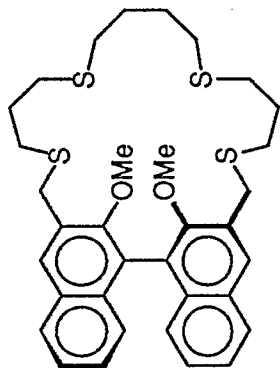


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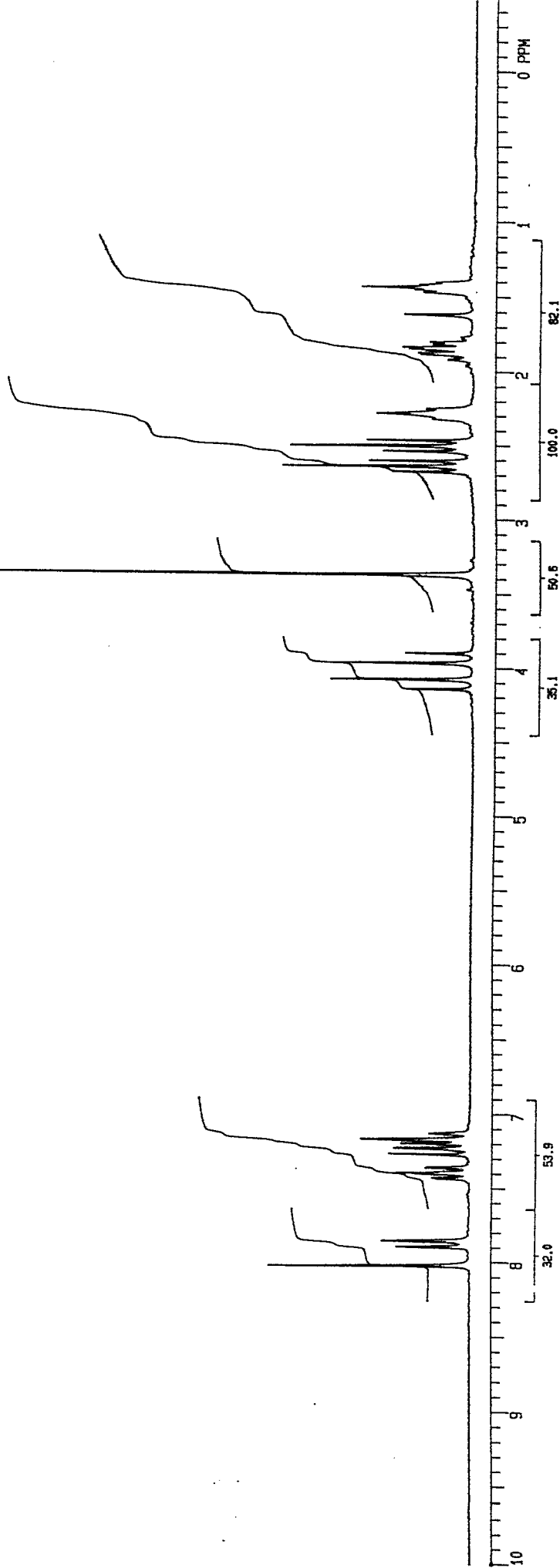
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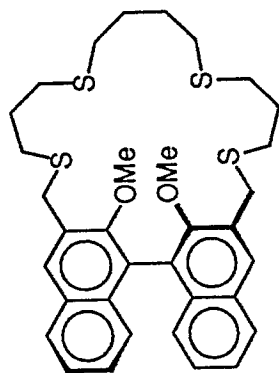


$^1\text{H NMR}$ (CDCl_3 , 200 MHz)

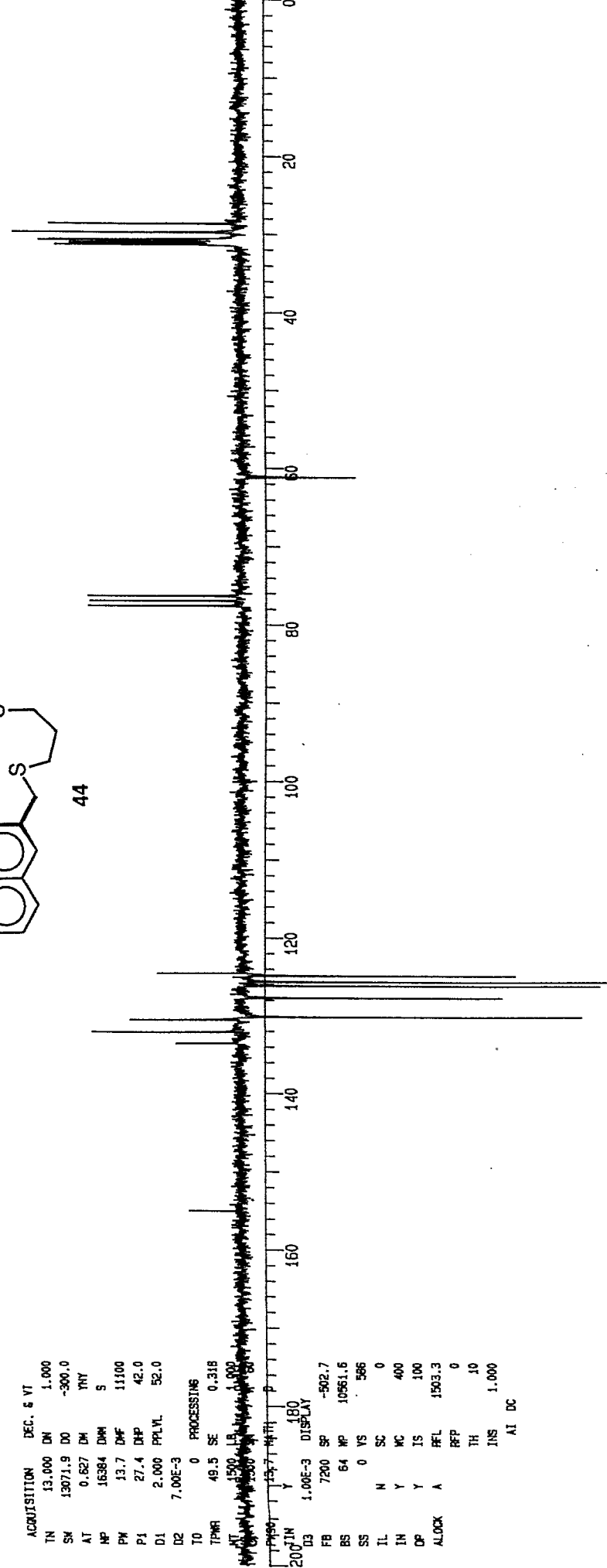


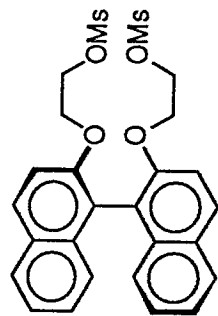
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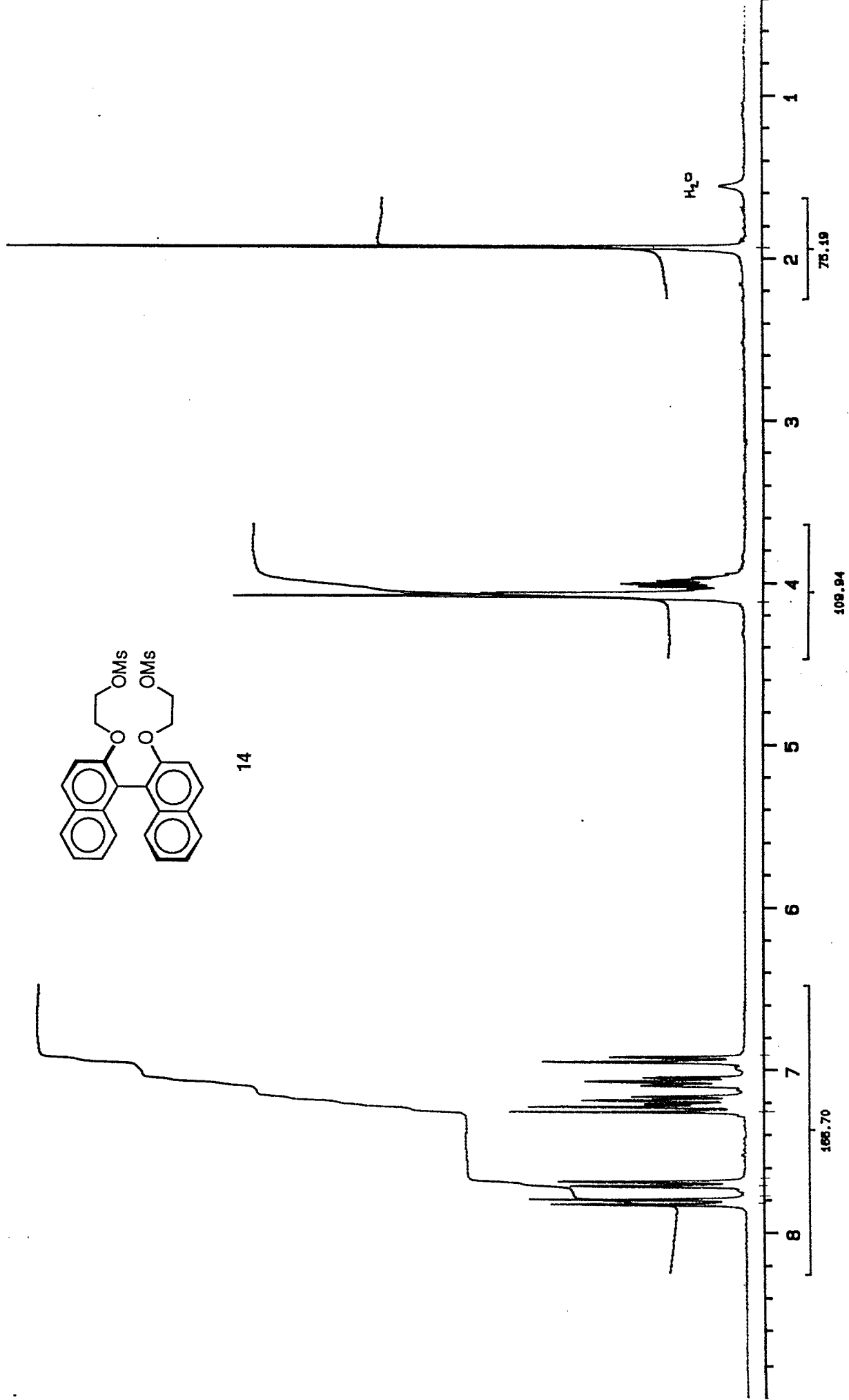


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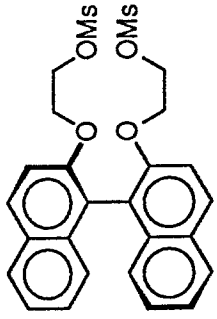




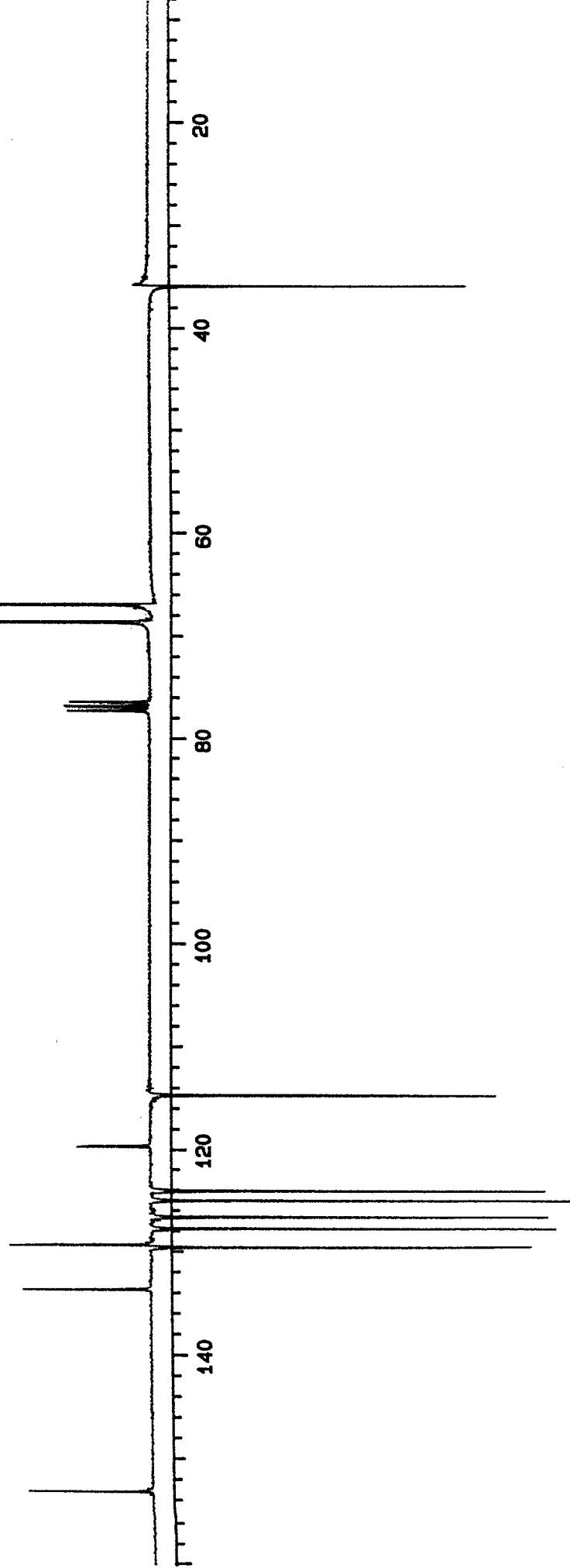
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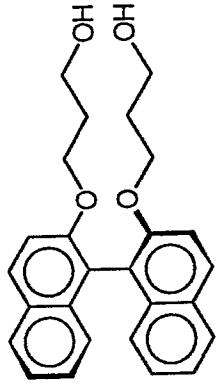
^{13}C NMR (CDCl_3 , 75.4 MHz)



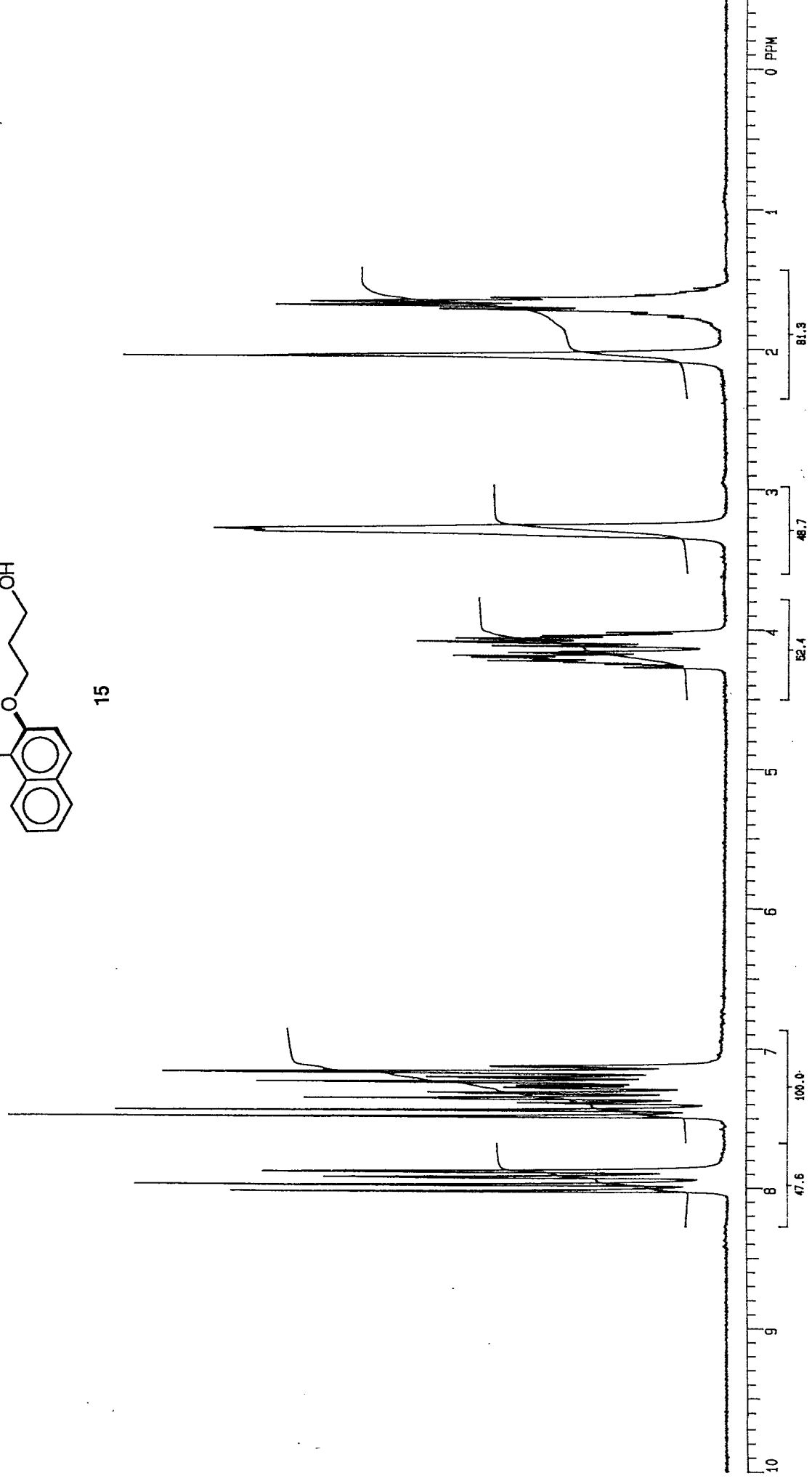
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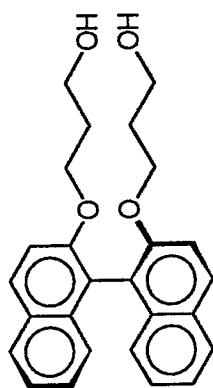
¹H NMR (CDCl₃, 200 MHz)



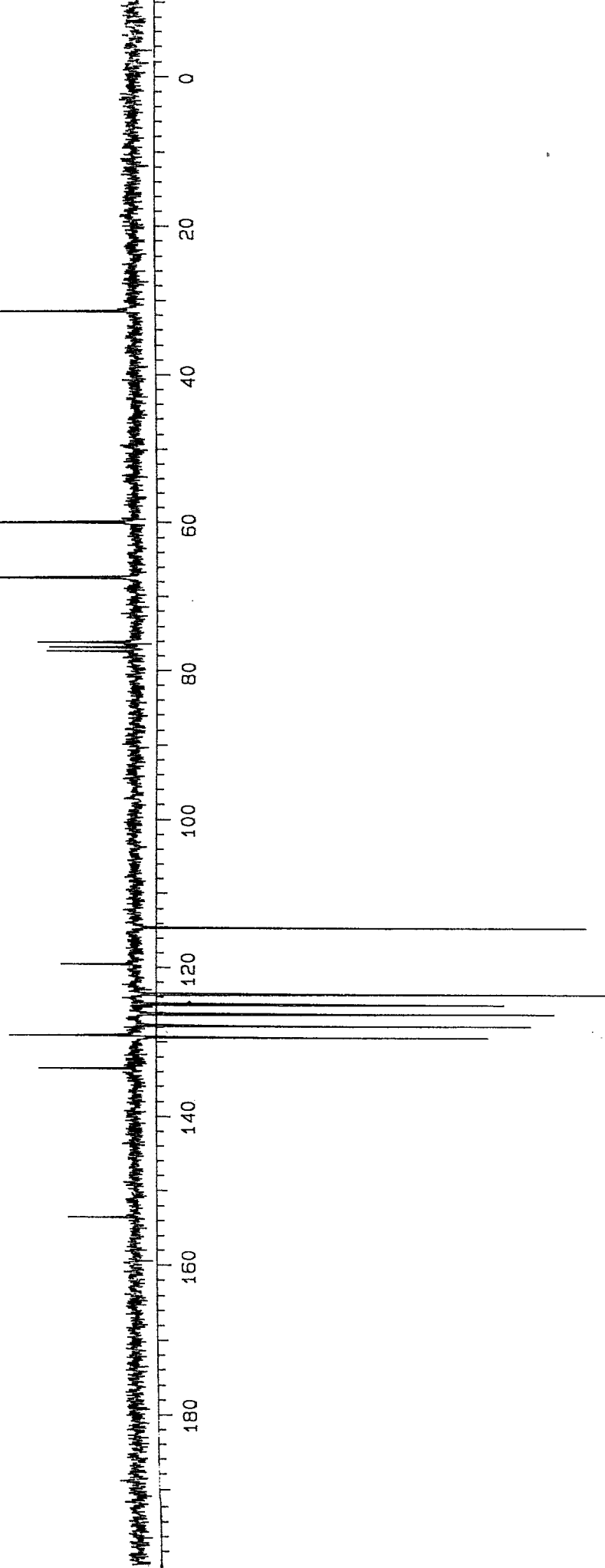
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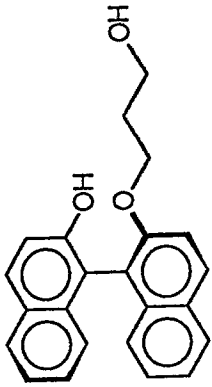
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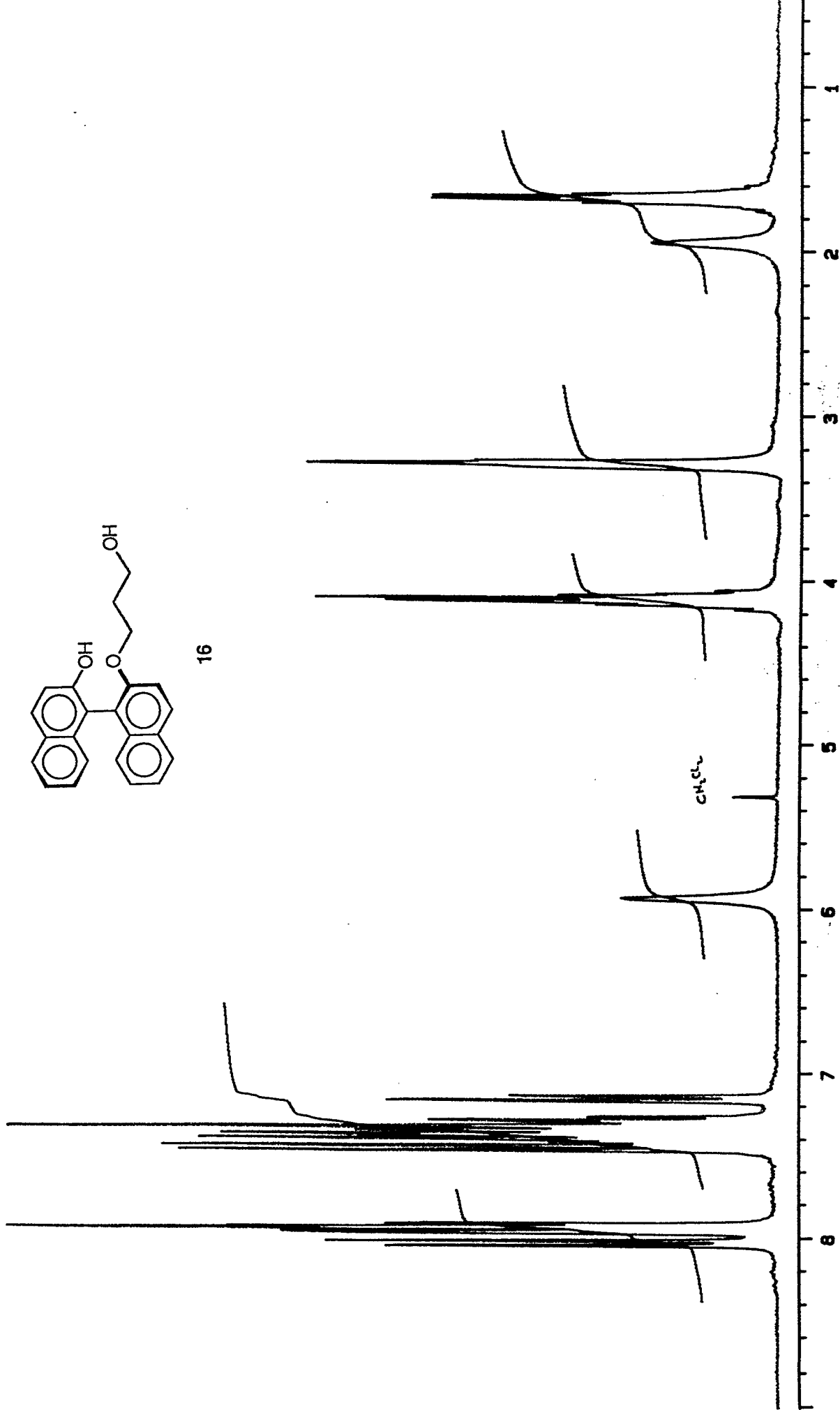
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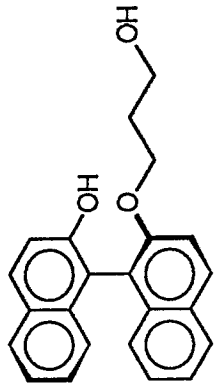
¹H NMR (CDCl₃, 300 MHz)



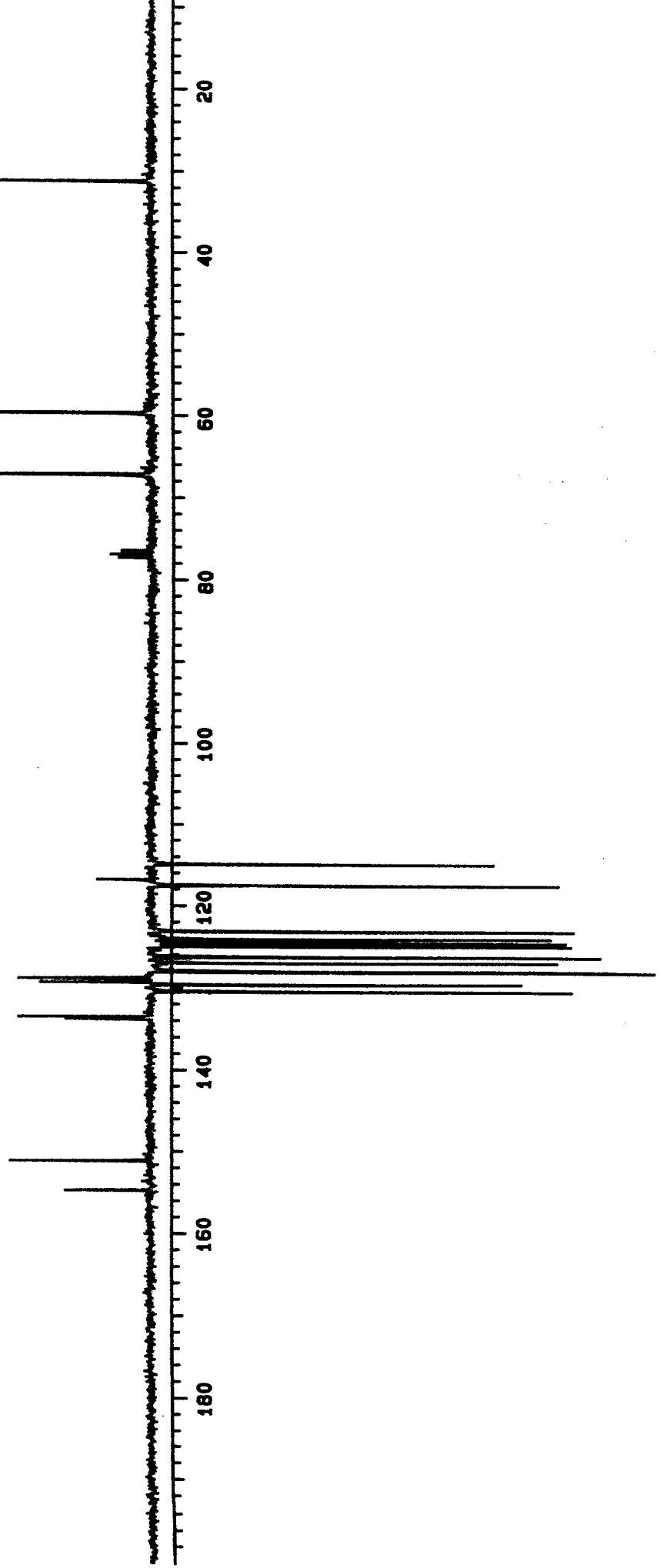
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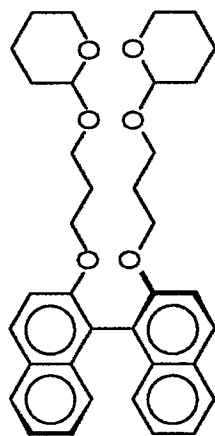


^{13}C NMR (CDCl_3 , 75.4 MHz)

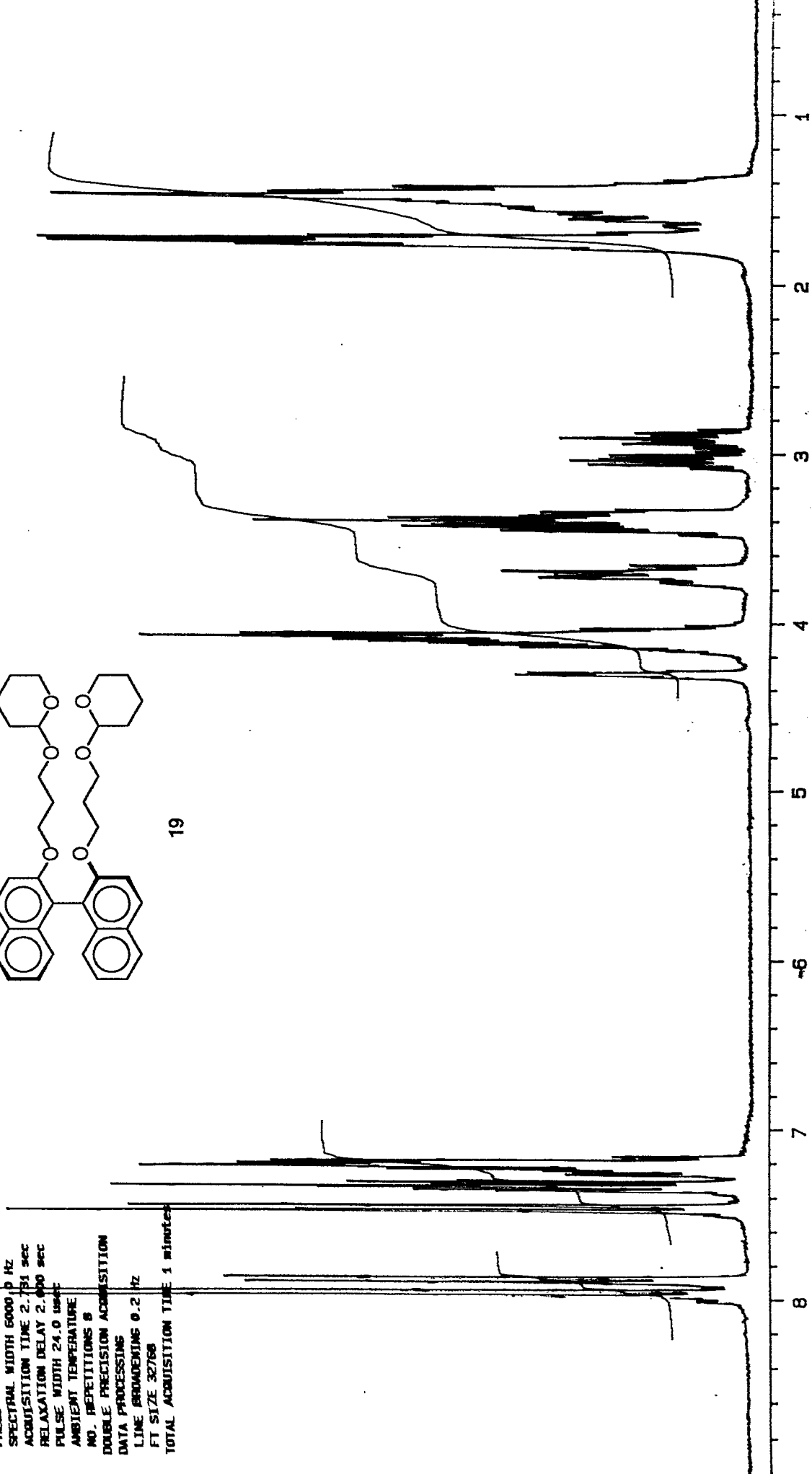


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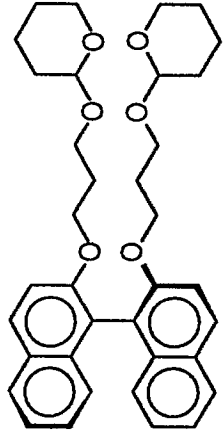




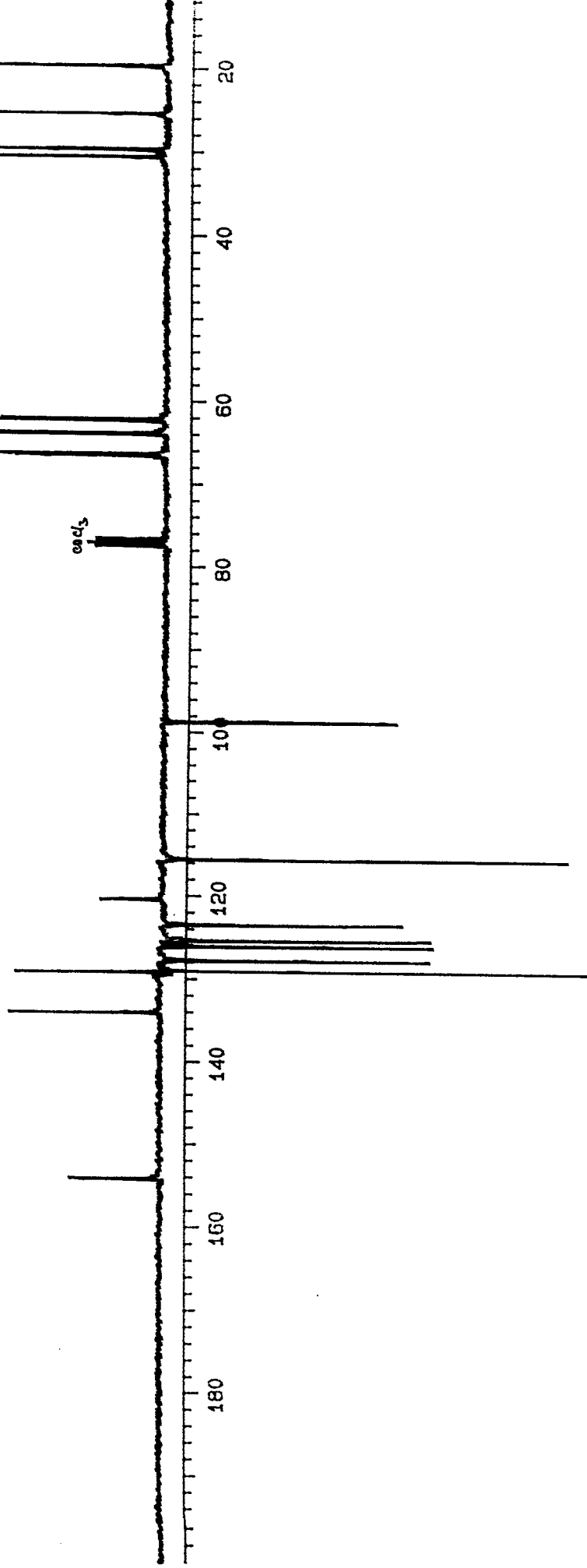
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SPECTRAL WIDTH 6000.0 Hz
ACQUISITION TIME 2.731 sec
RELAXATION DELAY 2.000 sec
PULSE WIDTH 24.0 usec
AMBIENT TEMPERATURE
NO. REPETITIONS 8
DOUBLE PRECISION ACQUISITION
DATA PROCESSING
LINE BROADENING 0.2 Hz
FT SIZE 32768
TOTAL ACQUISITION TIME 1 minutes



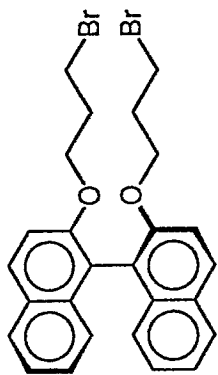
^{13}C NMR (CDCl_3 , 75.4 MHz)



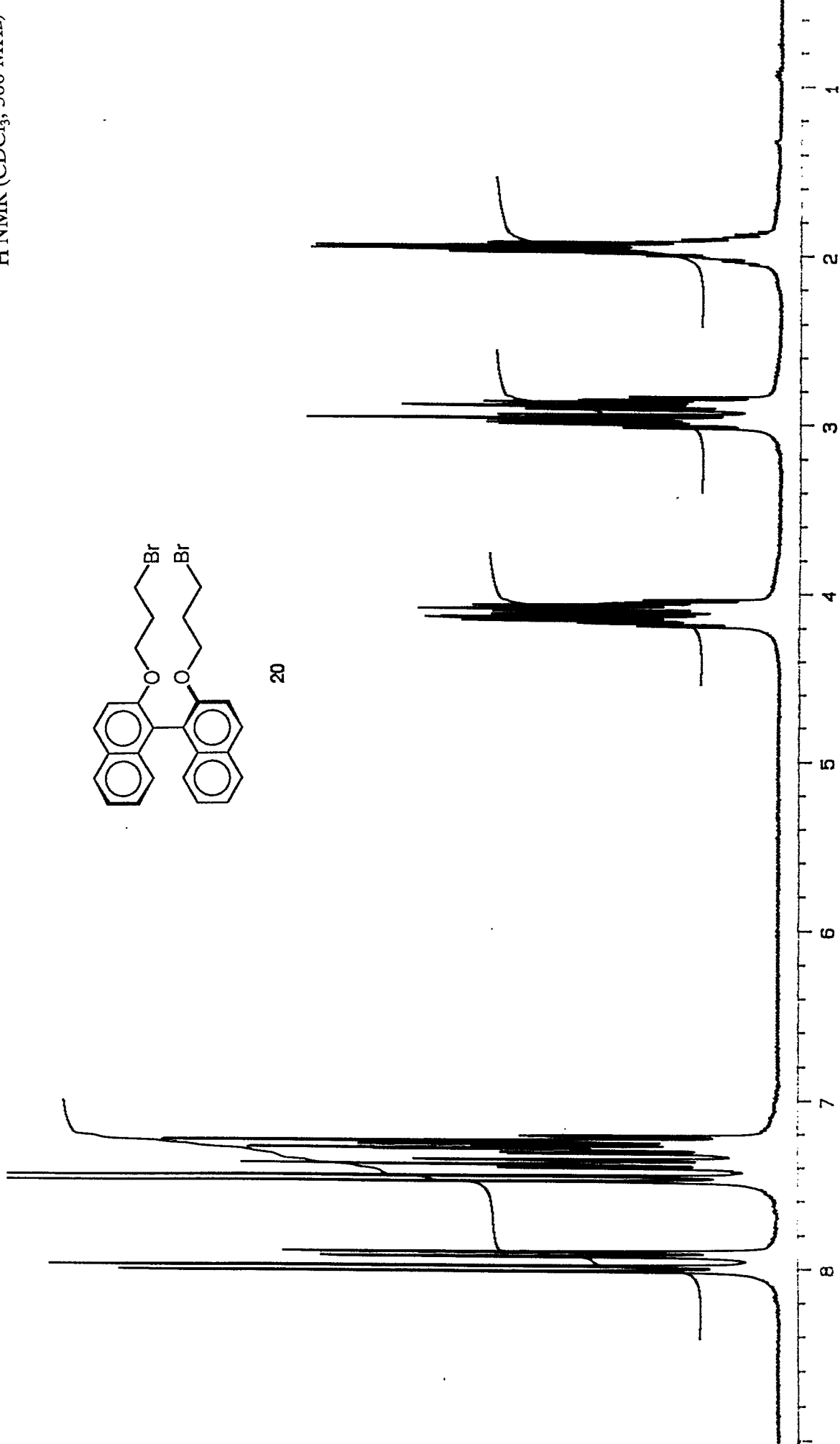
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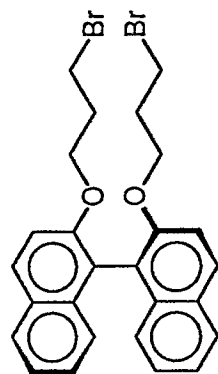


$^1\text{H NMR}$ (CDCl_3 , 300 MHz)

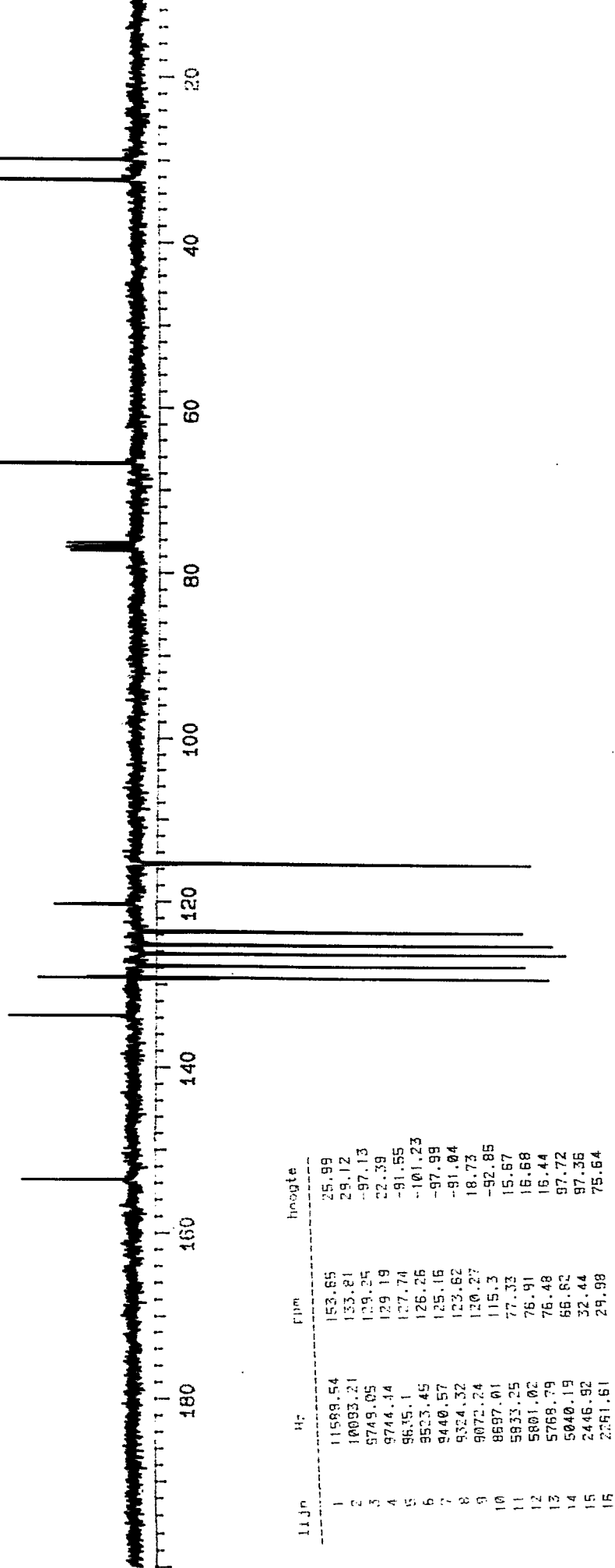


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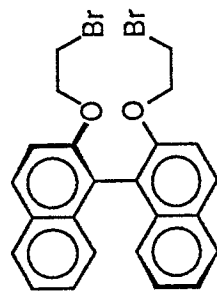




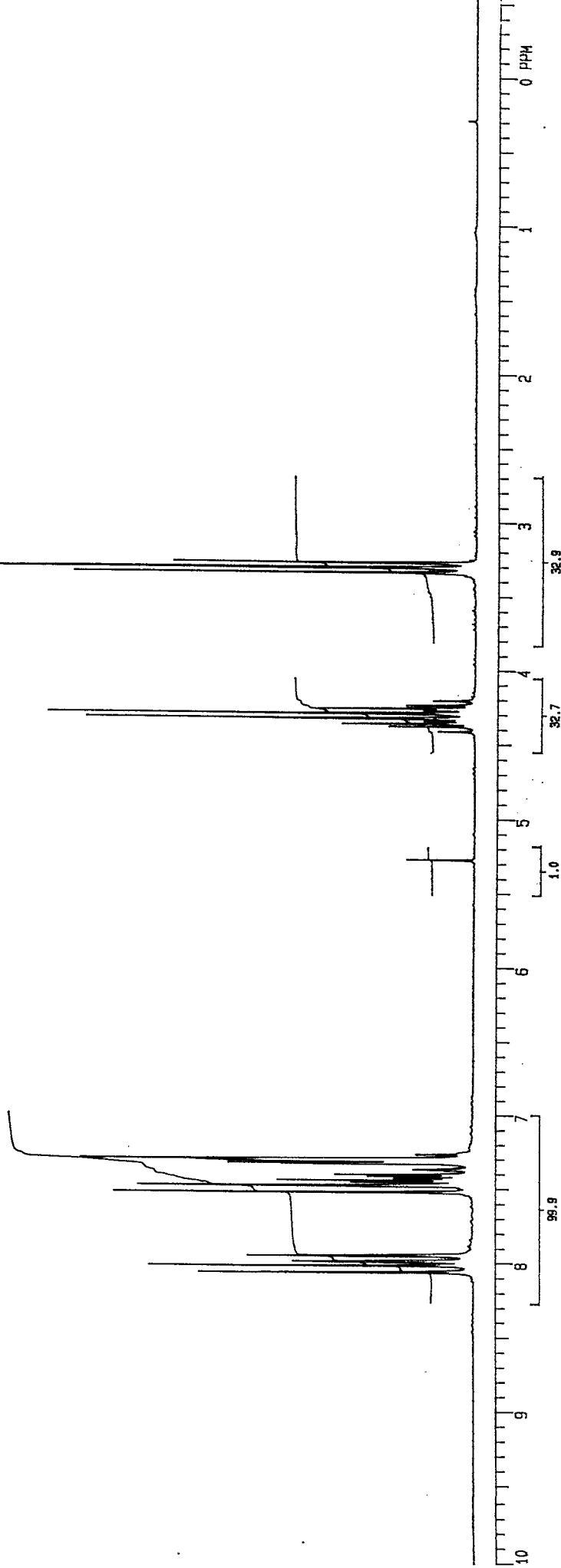
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$^1\text{H NMR}$ (CDCl_3 , 200 MHz)

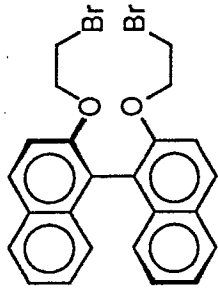


21



¹³C NMR (CDCl₃, 50.3 MHz)

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RFL= 1503.3 RFP= 0



21

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02	6746.0	134.144	21.049
03	6532.2	129.991	25.304
04	6529.1	129.931	-83.118
05	6444.2	128.143	-98.918
06	6370.8	126.583	-99.998
07	6313.7	125.547	-83.973
08	6243.7	124.274	-92.988
09	6090.3	121.105	16.764
10	5954.1	116.115	-76.843
11	3519.0	69.974	61.150
12	1492.6	29.491	54.032

ACQUISIT 10
TH 11
SW 43
AT

16384 DMH S

13.7 DMF 11100

27.4 DMF 42.0

2.000 PPLVL 52.0

D2 7.00E-3

TO 0 PROCESSING

IPMR 49.5 SE 0.318

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BS 64 RP 10561.6

SS 0 VS 67

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IH Y MC 400

IP Y TS 100

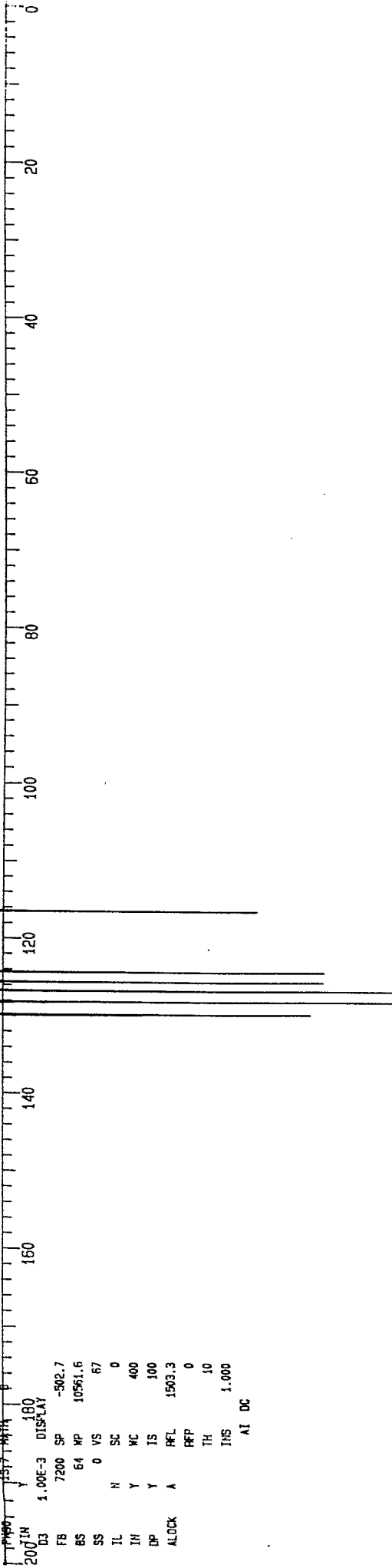
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PFP 0

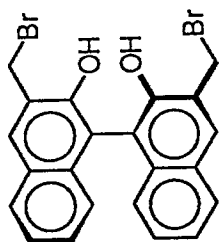
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INS 1.000

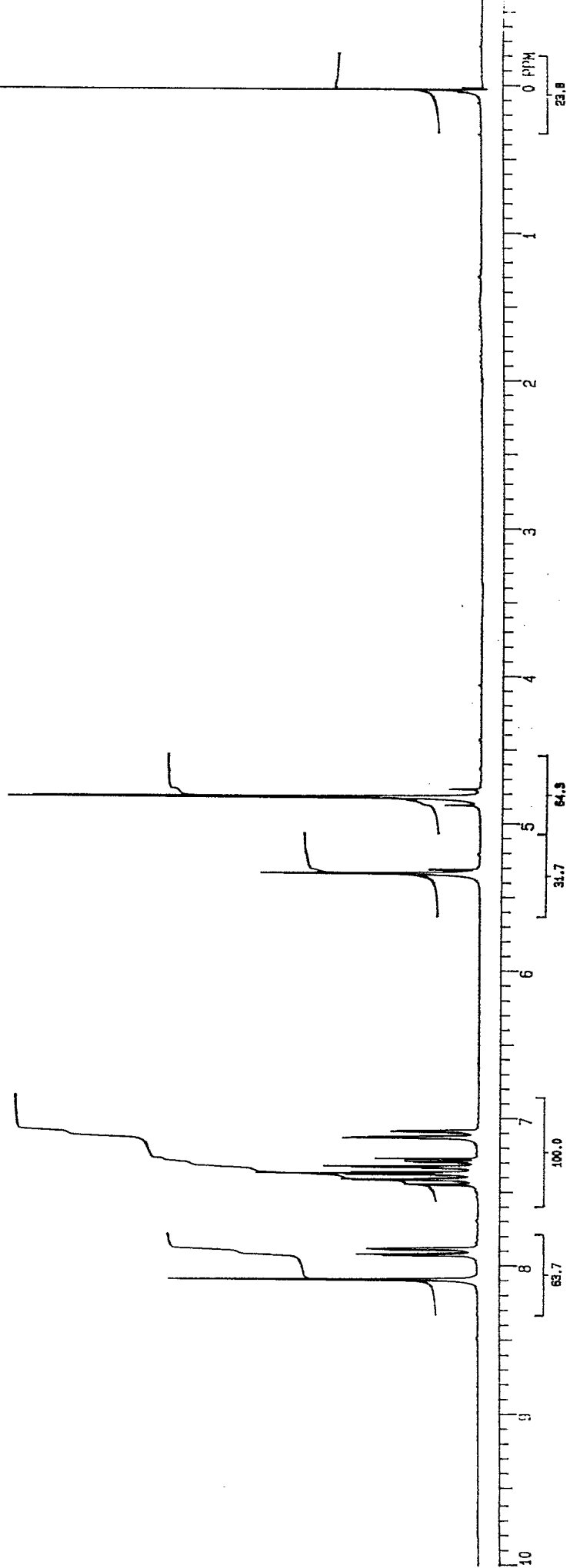
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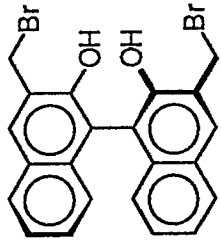


^1H NMR (CDCl_3 , 200 MHz)



45

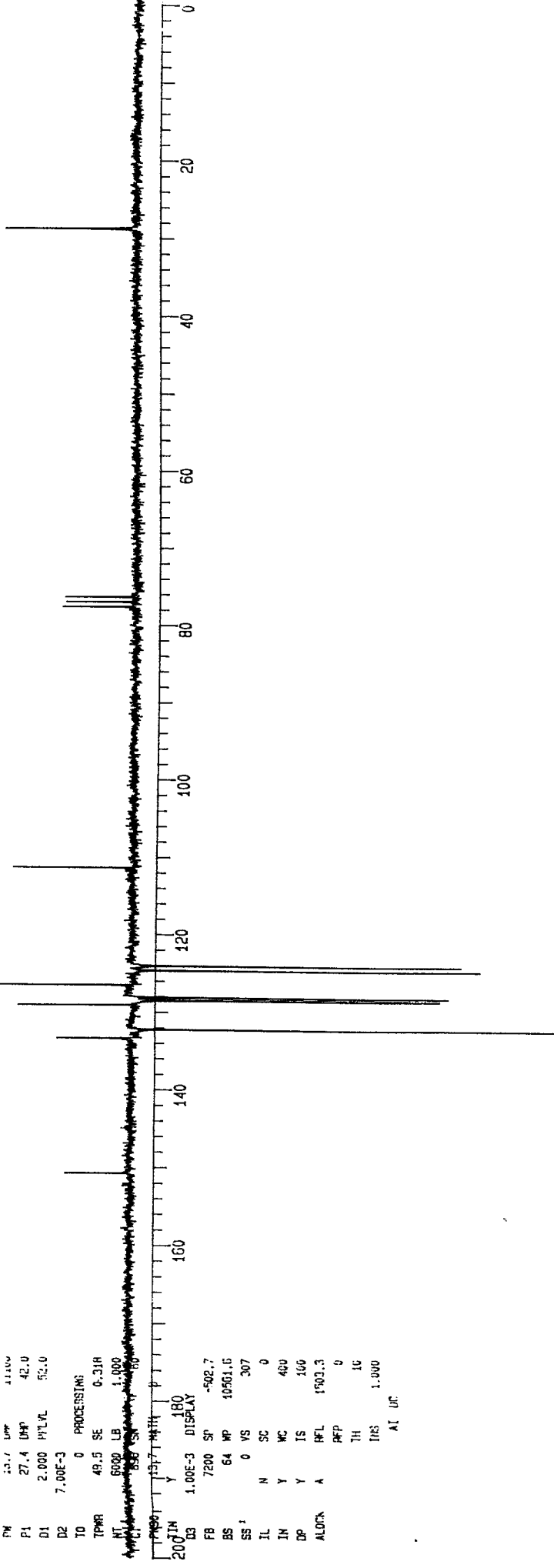




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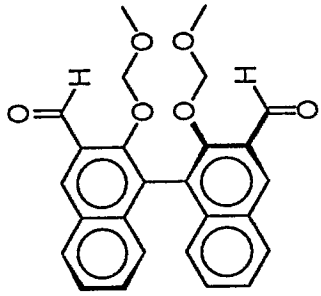
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 REFERENCE 100.625000

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03	148.384	148.384	19.304
04	148.000	148.000	21.120
05	147.616	147.616	23.856
06	147.232	147.232	26.592
07	146.848	146.848	29.328
08	146.464	146.464	32.064
09	146.080	146.080	34.800
10	145.696	145.696	37.536
11	145.312	145.312	40.272
12	144.928	144.928	43.008
13	144.544	144.544	45.744
14	144.160	144.160	48.480
15	143.776	143.776	51.216
16	143.392	143.392	53.952
17	143.008	143.008	56.688
18	142.624	142.624	59.424
19	142.240	142.240	62.160
20	141.856	141.856	64.896
21	141.472	141.472	67.632
22	141.088	141.088	70.368
23	140.704	140.704	73.104
24	140.320	140.320	75.840
25	139.936	139.936	78.576
26	139.552	139.552	81.312
27	139.168	139.168	84.048
28	138.784	138.784	86.784
29	138.400	138.400	89.520
30	138.016	138.016	92.256
31	137.632	137.632	95.000
32	137.248	137.248	97.744
33	136.864	136.864	100.488
34	136.480	136.480	103.232
35	136.096	136.096	105.976
36	135.712	135.712	108.720
37	135.328	135.328	111.464
38	134.944	134.944	114.208
39	134.560	134.560	116.952
40	134.176	134.176	119.696
41	133.792	133.792	122.440
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44	132.640	132.640	130.672
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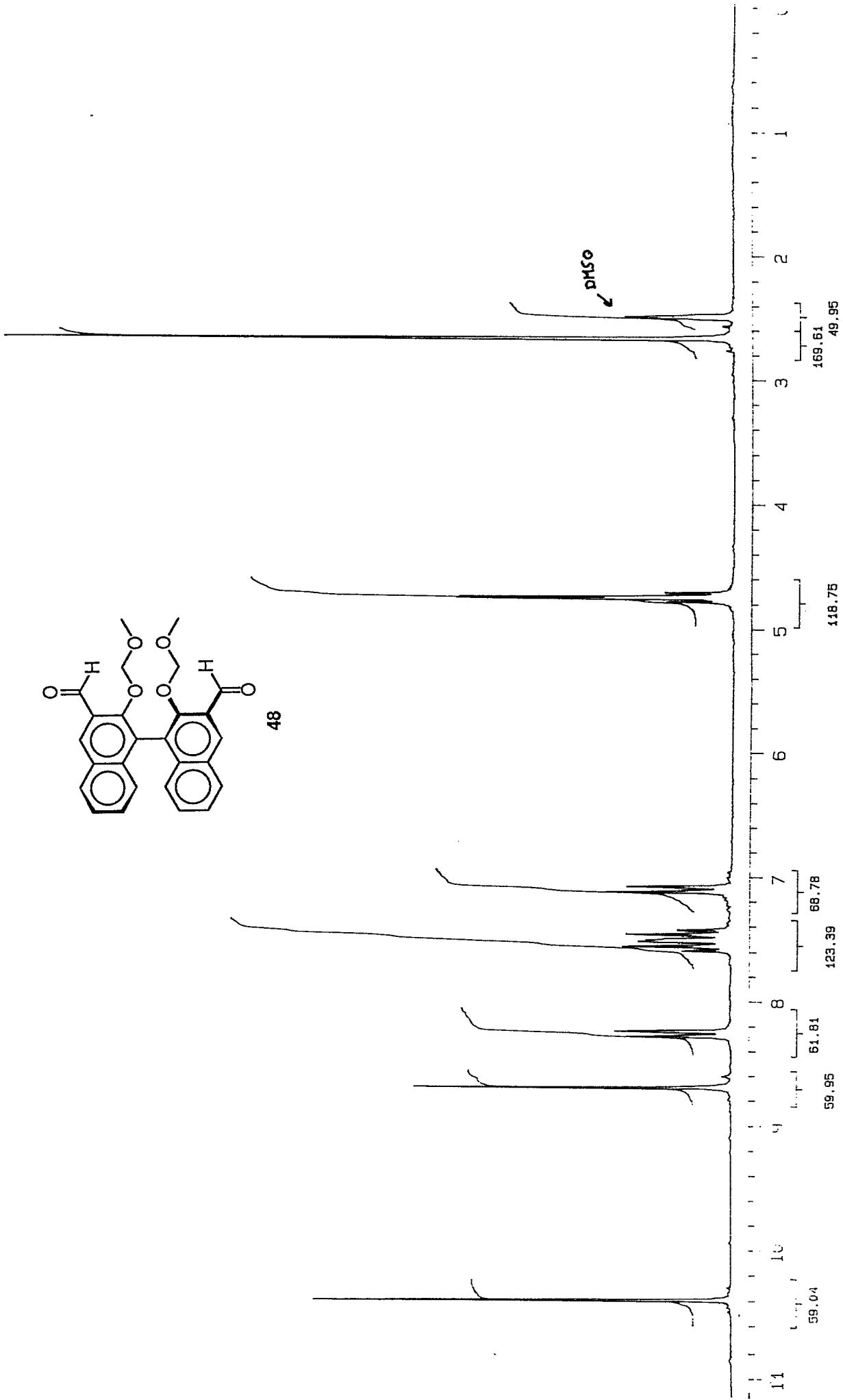
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 P1 27.4 DMP 42.0
 D1 2.000 PTLVL 52.0
 D2 7.00E-3
 TO 0 PROCESSING
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 AT 6000 LB 1.000
 AC 1000000
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 2007IN Y
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 BS 64 MP 10501.6
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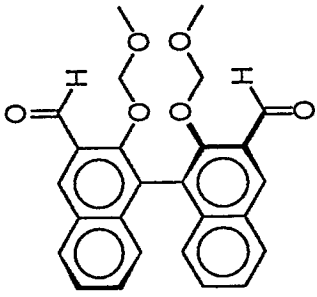
¹H NMR (DMSO-d₆, 200 MHz)



48

DMSO

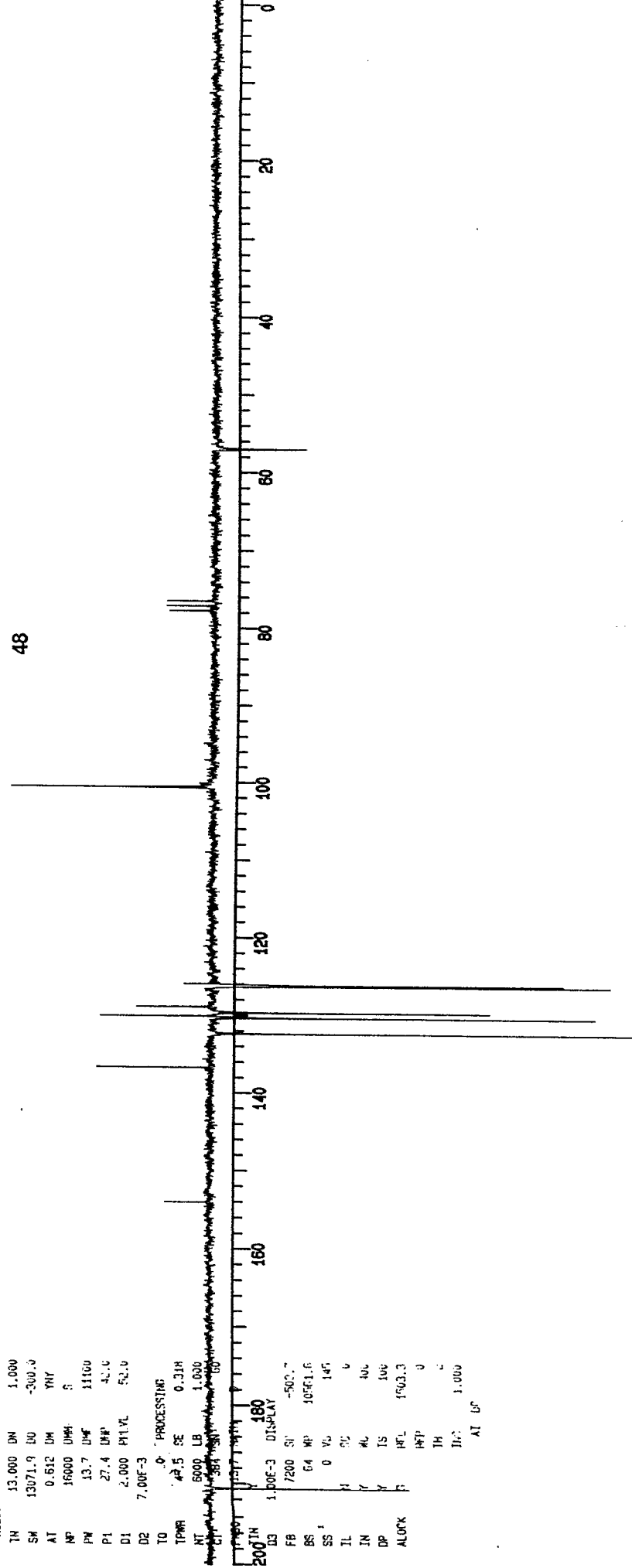




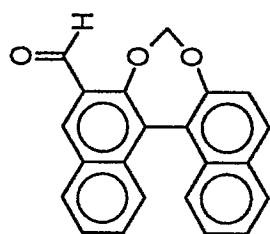
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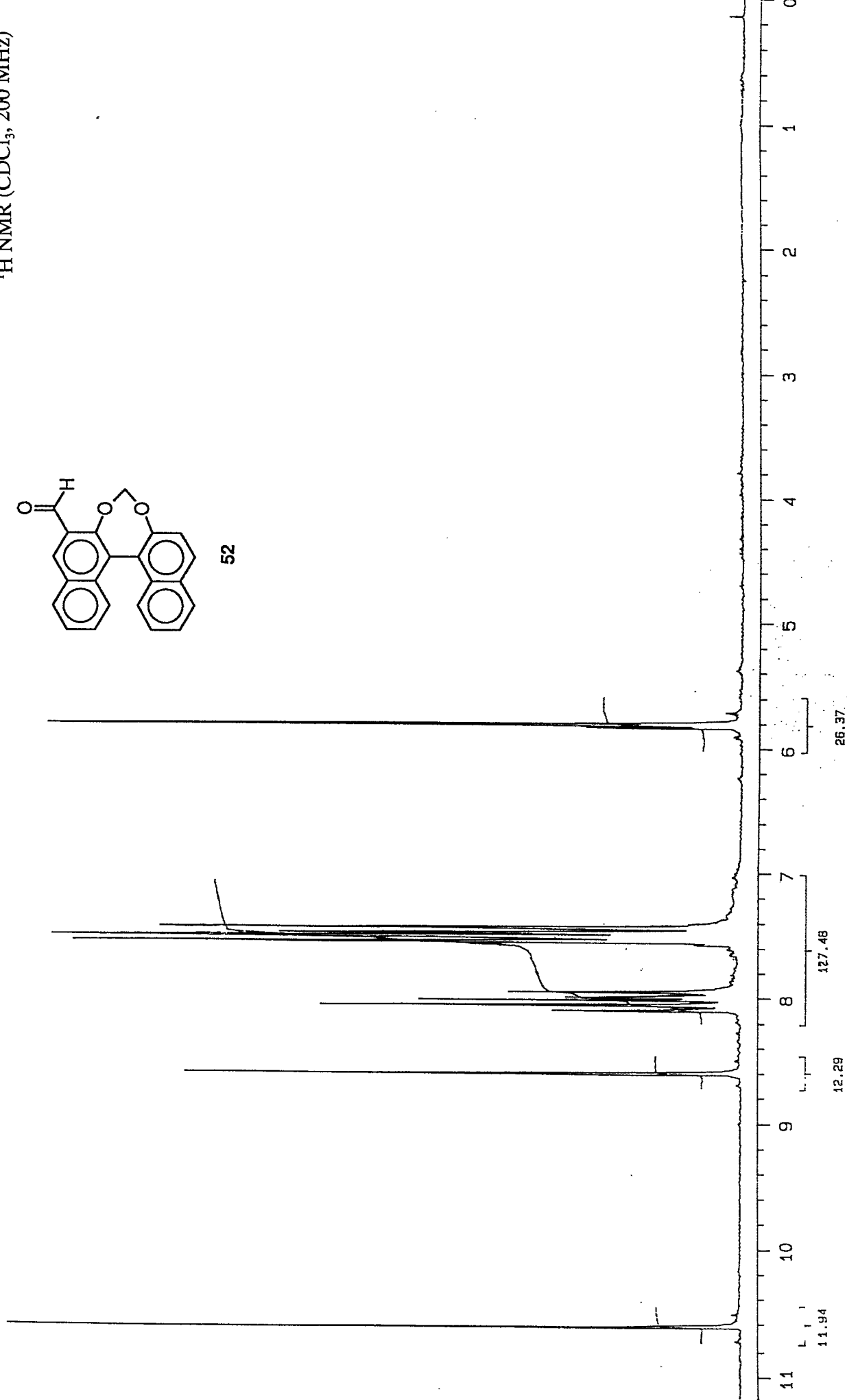
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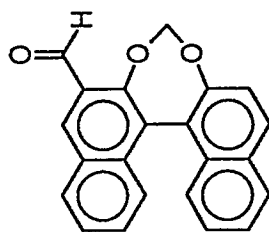


¹H NMR (CDCl₃, 200 MHz)



52

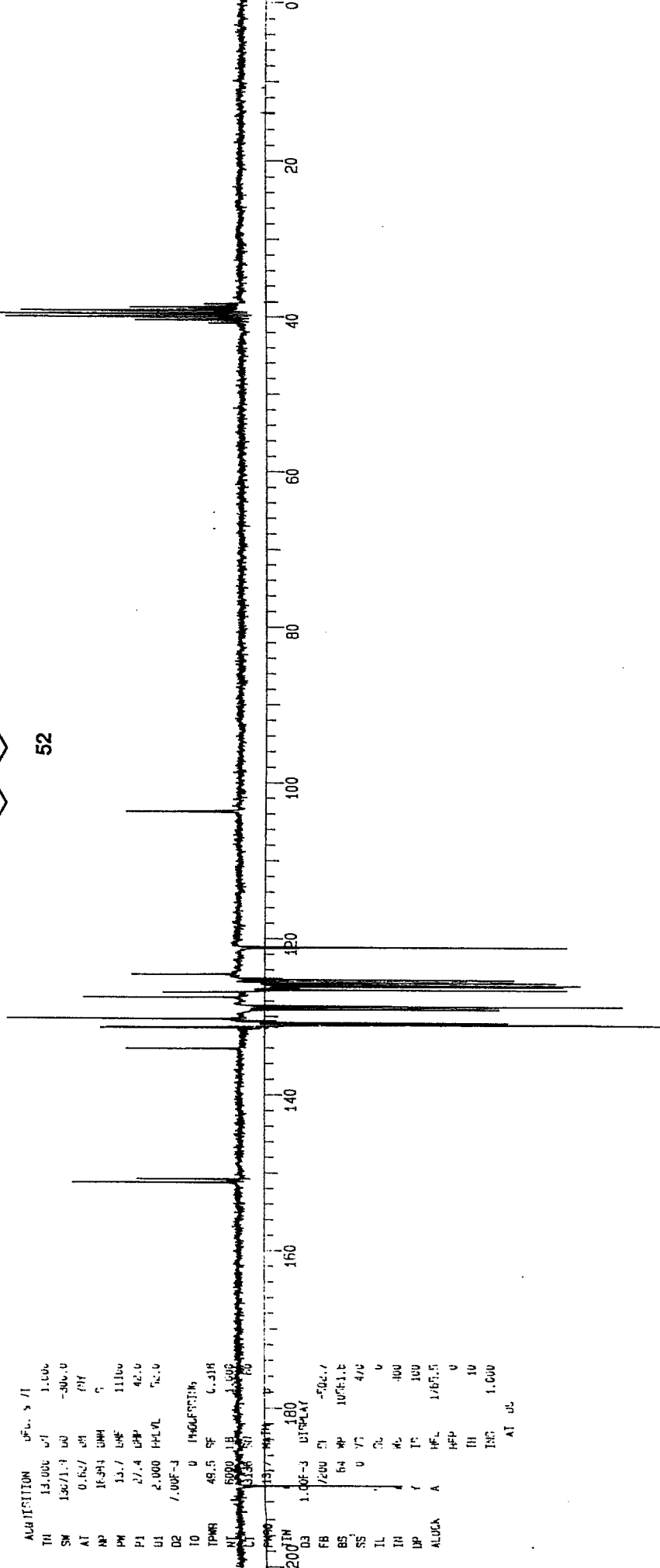




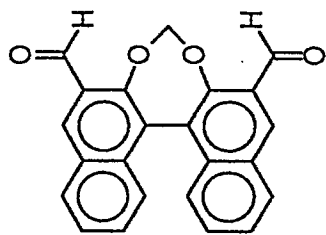
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 PW 13.7 0MF 11100
 P1 27.4 0PF 42.0
 Q1 2.000 141.0L 50.0
 D2 7.00F-3
 TO 0 140.0F0110
 TPWR 46.5 0F 6.318
 AT 5000 LH 1.000
 133.8 57 80

FB 7200 0 -502.7
 BS 64 0P 10751.5
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 ALLOCK A HFL 1/87.5
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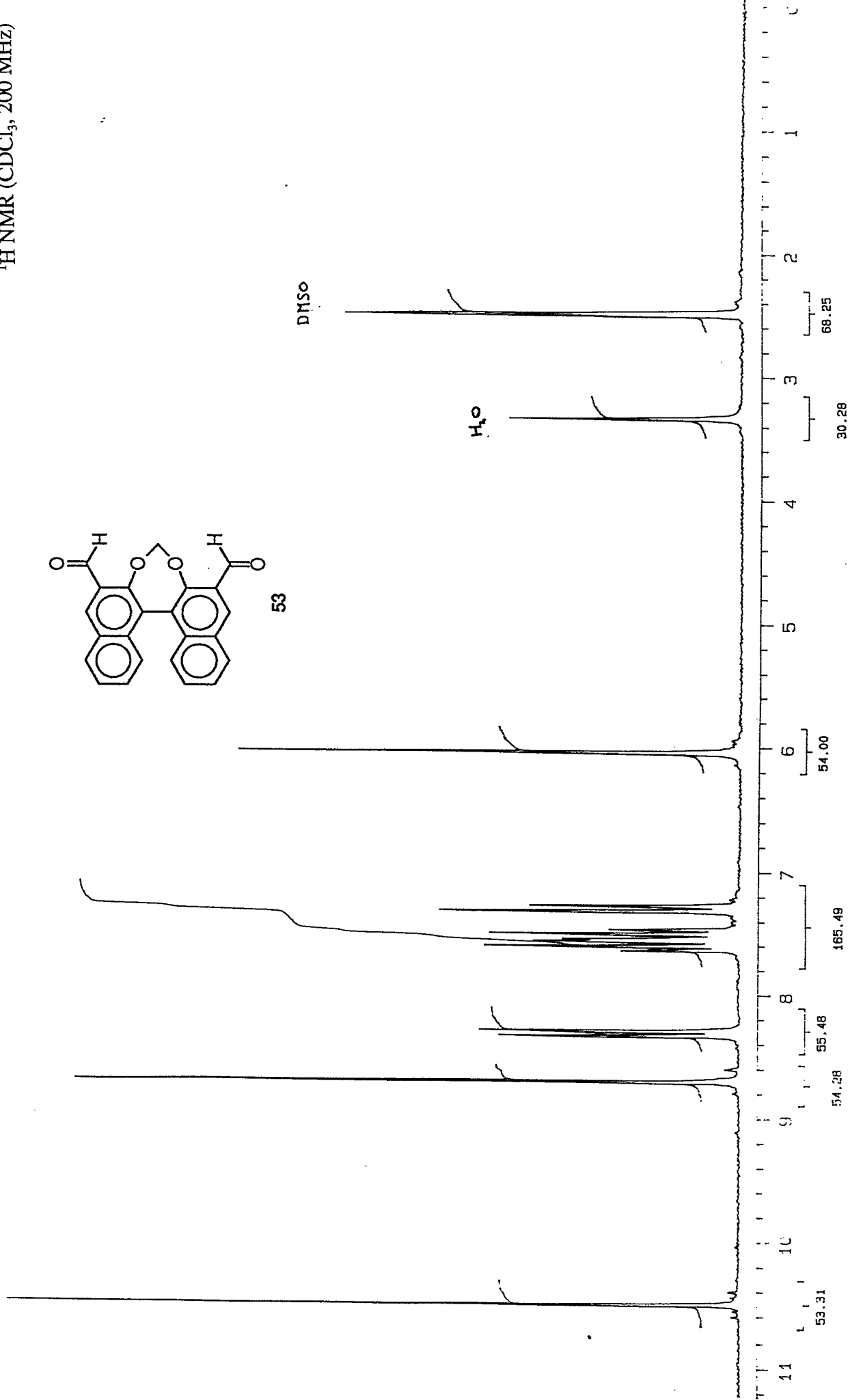
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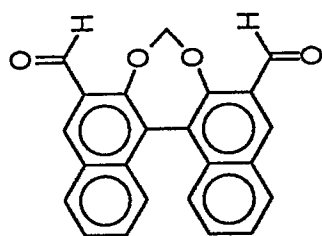
53

DMSO

H₂O

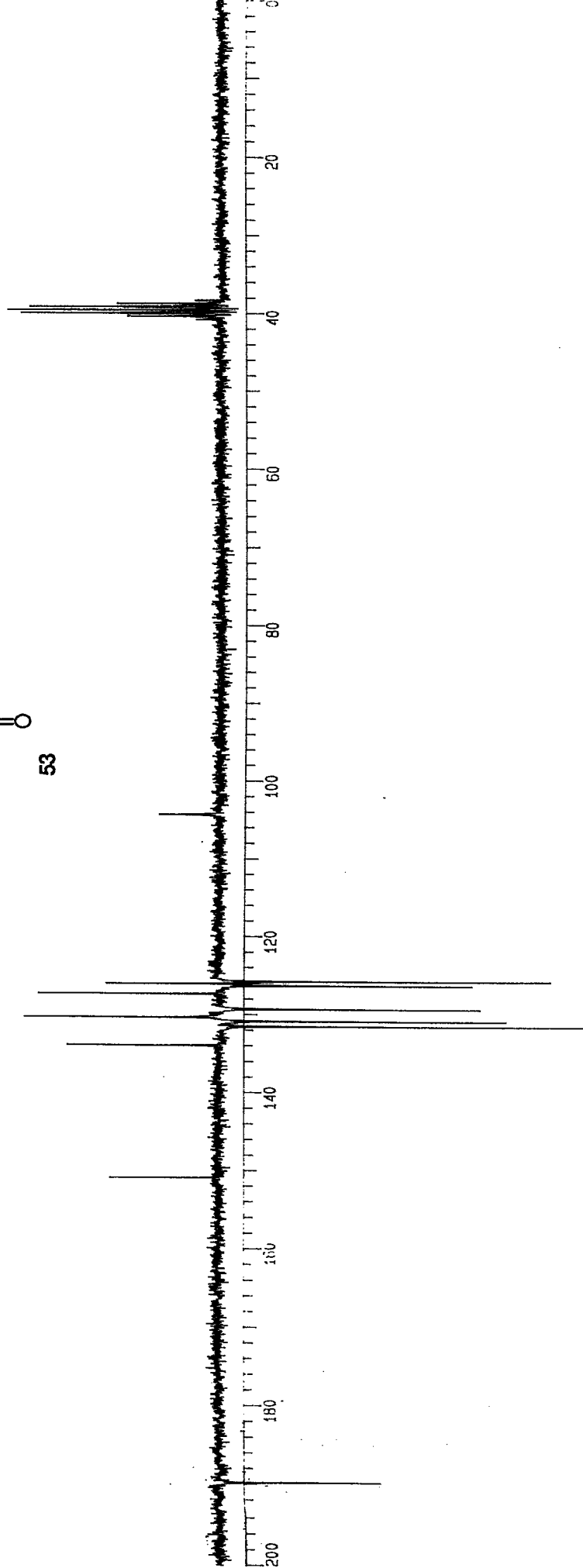


^{13}C NMR (CDCl_3 , 50.3 MHz)

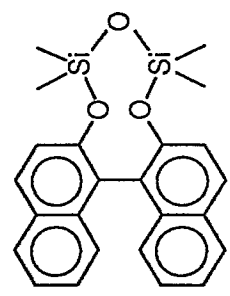


53

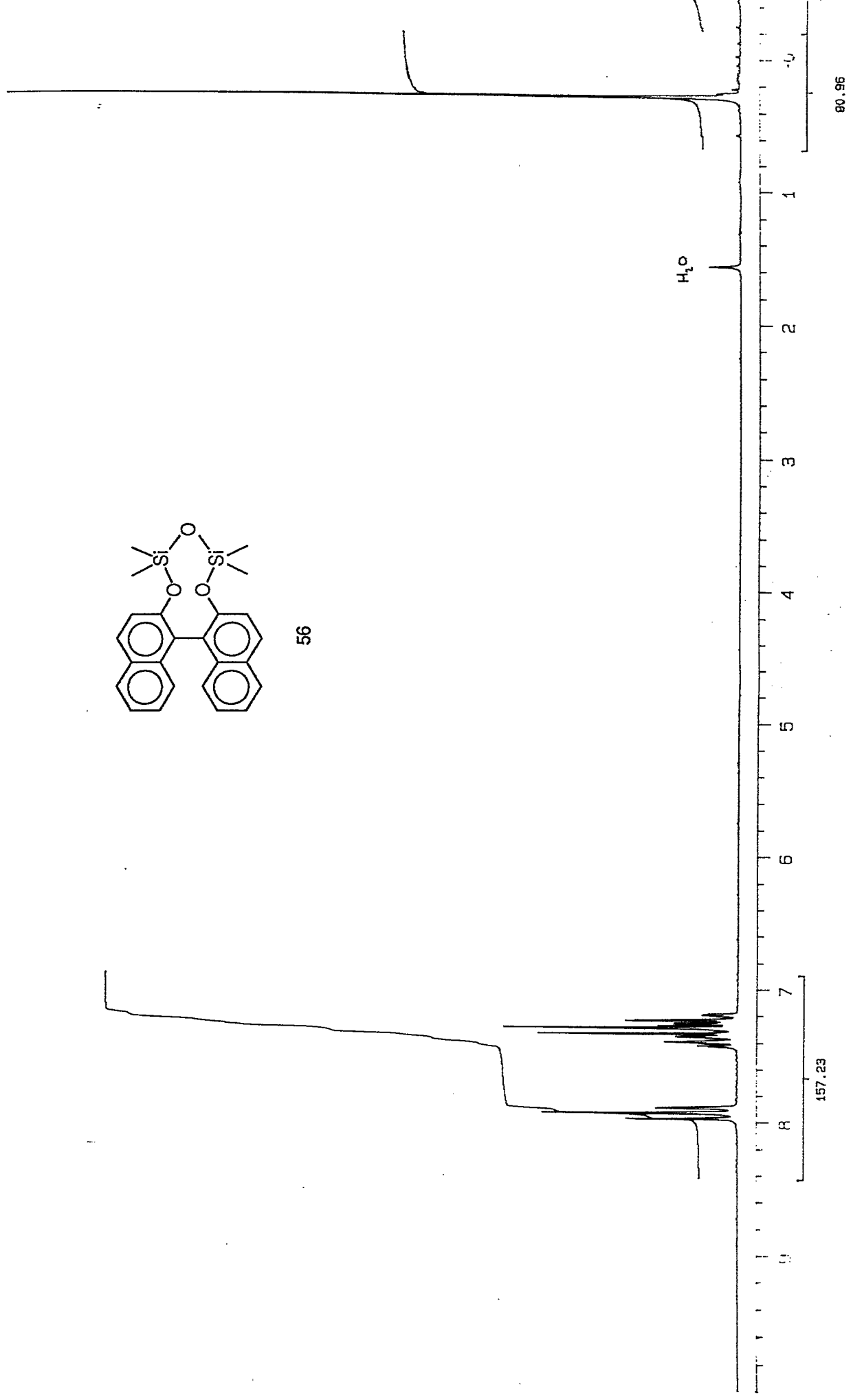
DMF d_6



¹H NMR (CDCl₃, 200 MHz)



56



157.23

8

7

6

5

4

3

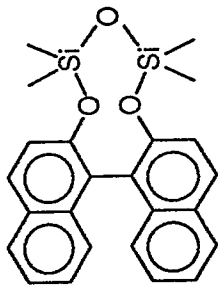
2

1

0

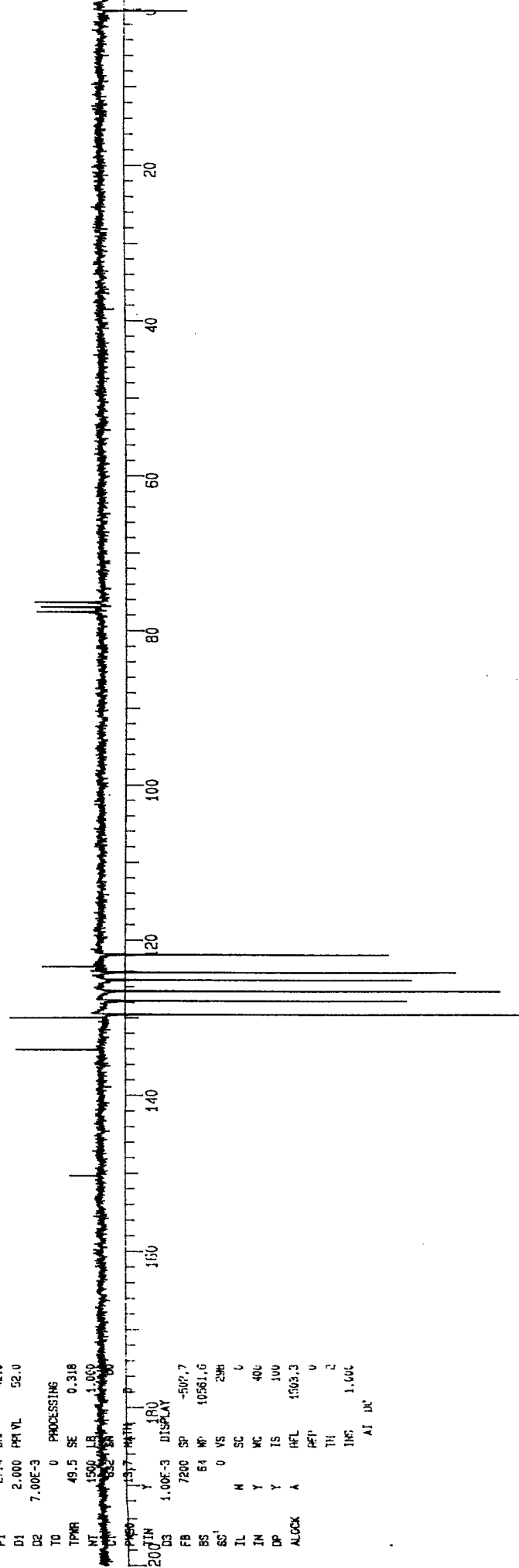
H₂O

80.96

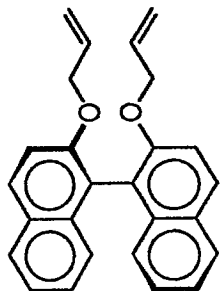


56

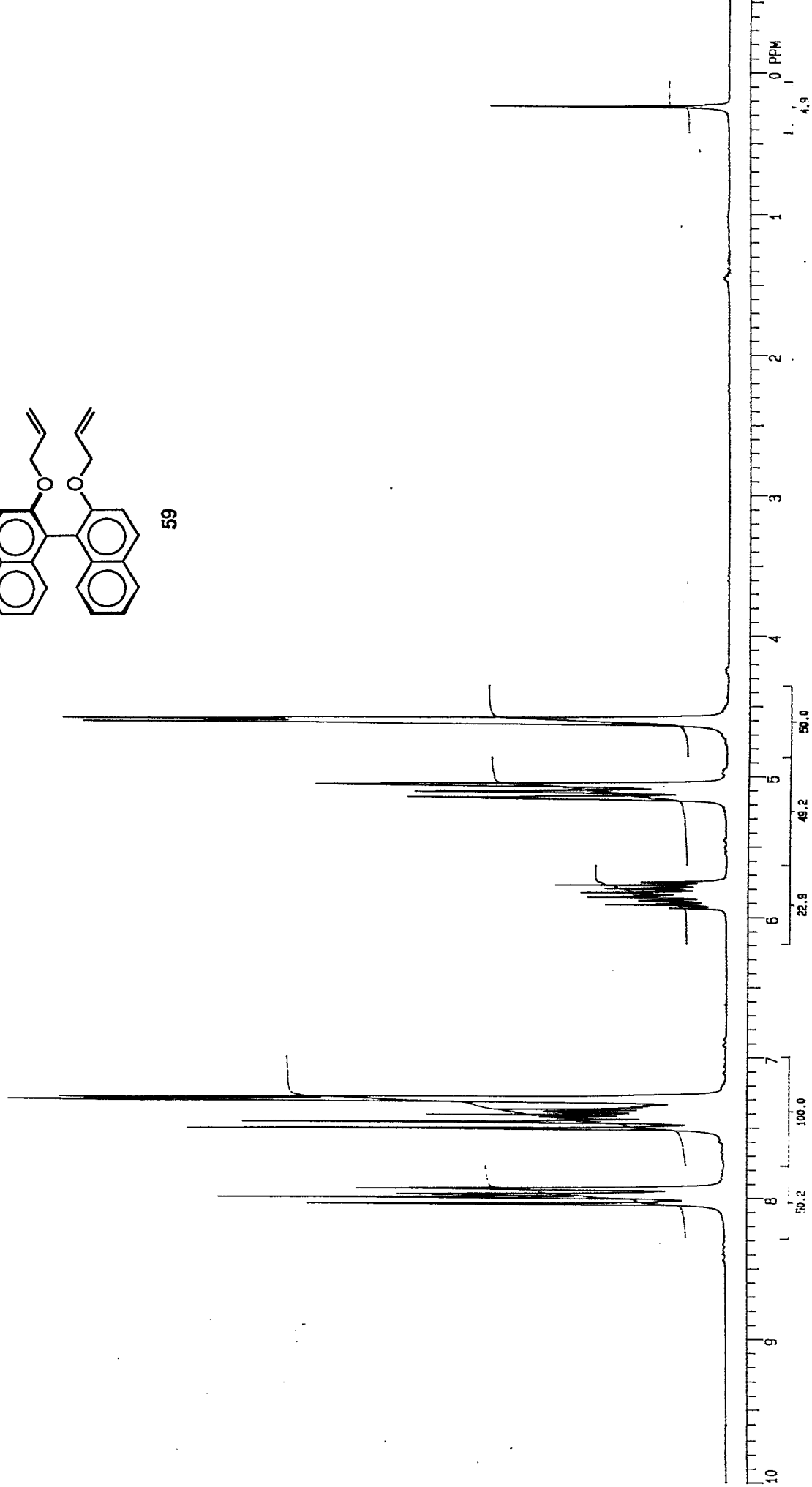
ACQUISITION JFC. 5 VI
 TN 13.006 DN 1.000
 SN 13071.9 DO -360.0
 AT 0.627 DM YNY
 NP 16384 DMH S
 PW 13.7 DM 11100
 P1 27.4 DHP 42.0
 D1 2.000 PPI VL 52.0
 D2 7.00E-3
 TO 0 PROCESSING
 TMR 49.5 SE 0.318
 NT 1500 LB 1.000
 CH 52.38
 FB 7200 SP -507.7
 BS 64 MC 10581.0
 SS' 0 VS 248
 IL N SC 0
 IN Y MC 400
 DP Y IS 100
 ALLOCK A HFL 1503.3
 PET' 0
 TH 2
 IMC 1.000
 AT DC



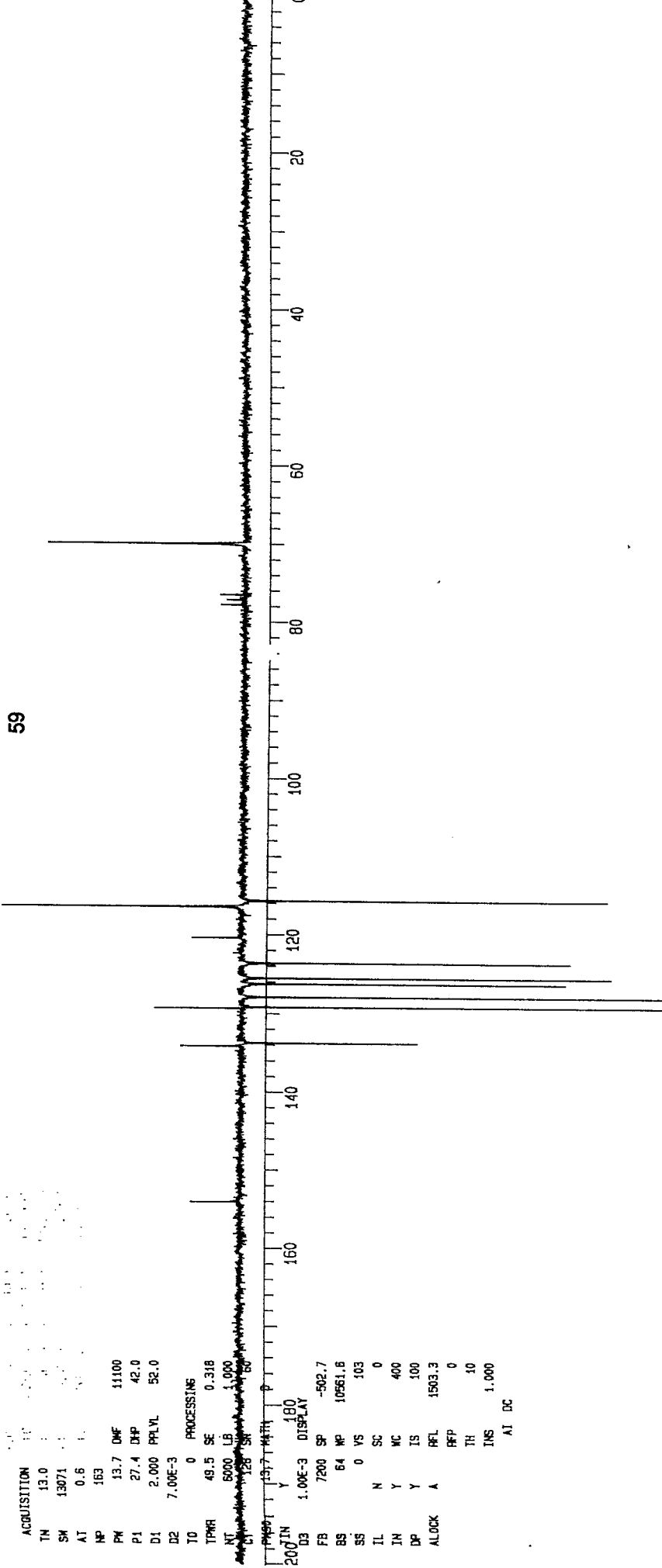
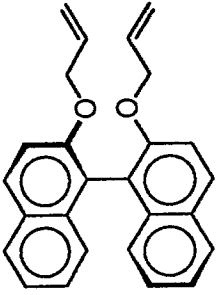
^1H NMR (CDCl_3 , 200 MHz)



59



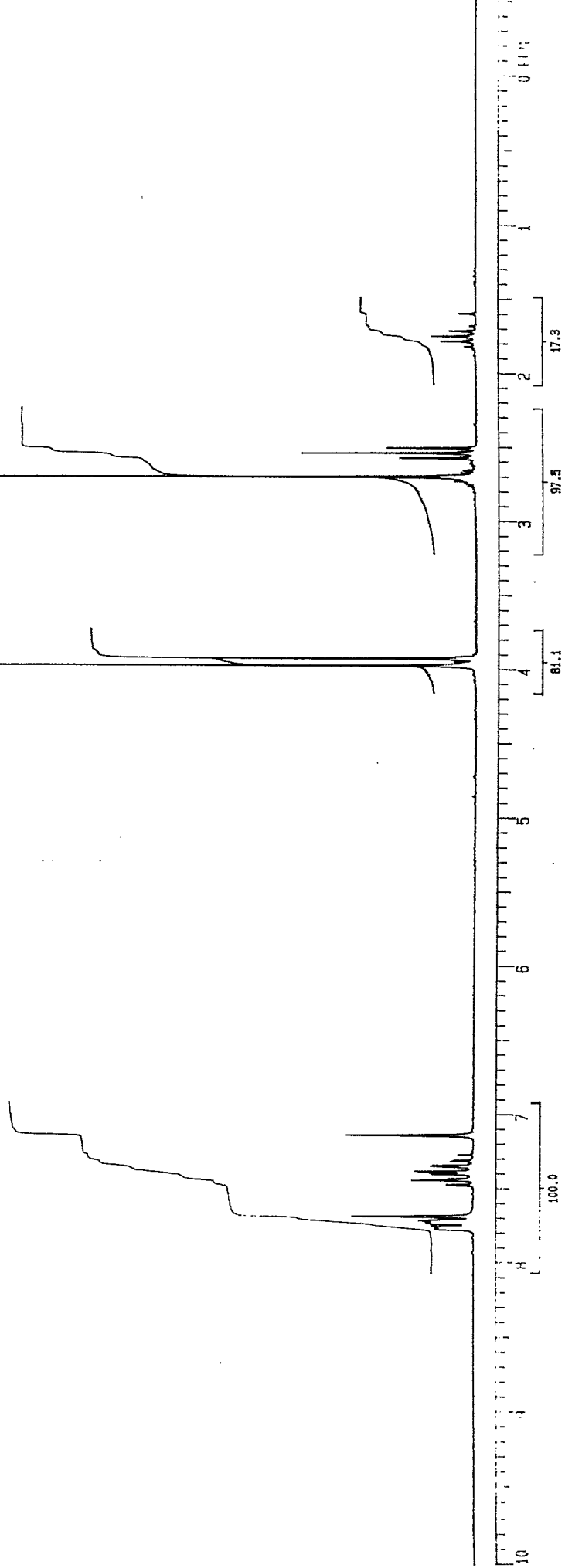
¹³C NMR (CDCl₃, 50.3 MHz)



¹H NMR (CDCl₃, 200 MHz)



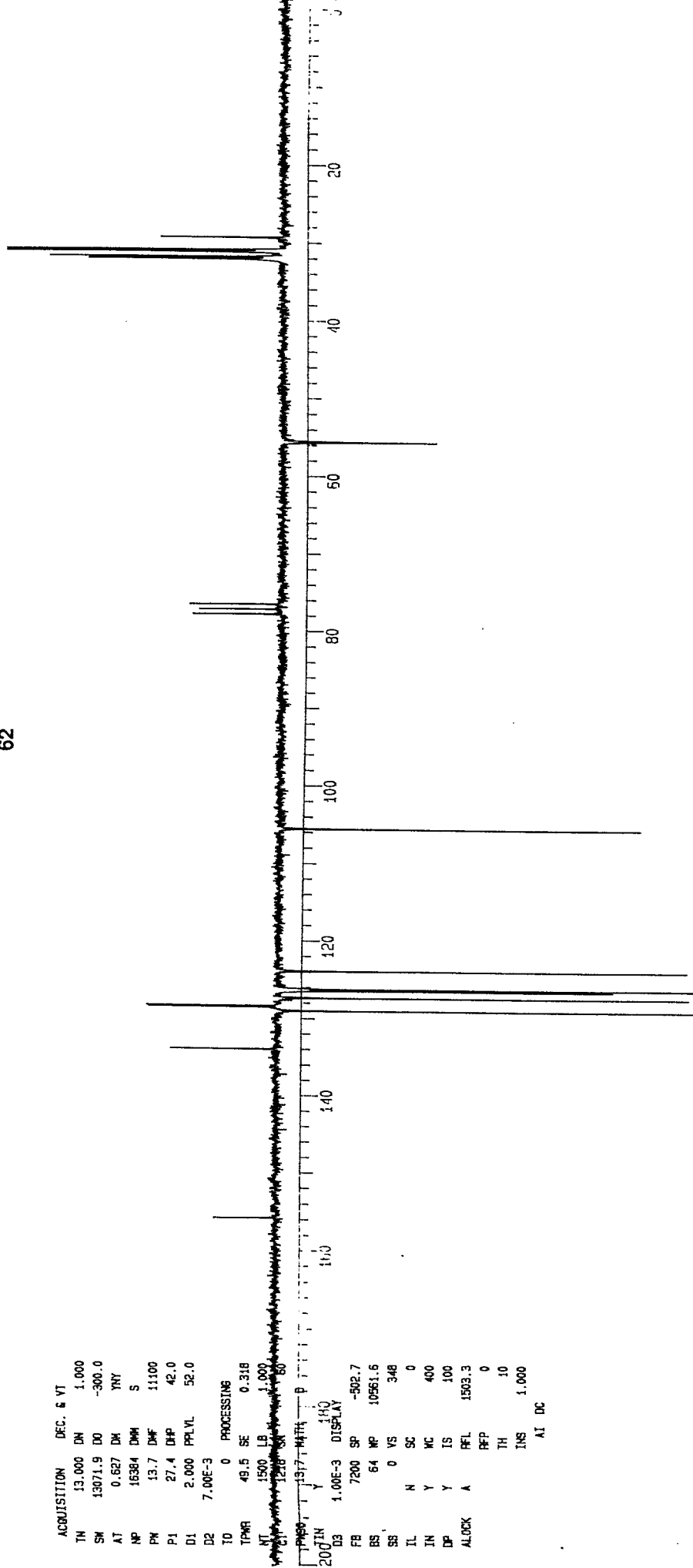
62





62

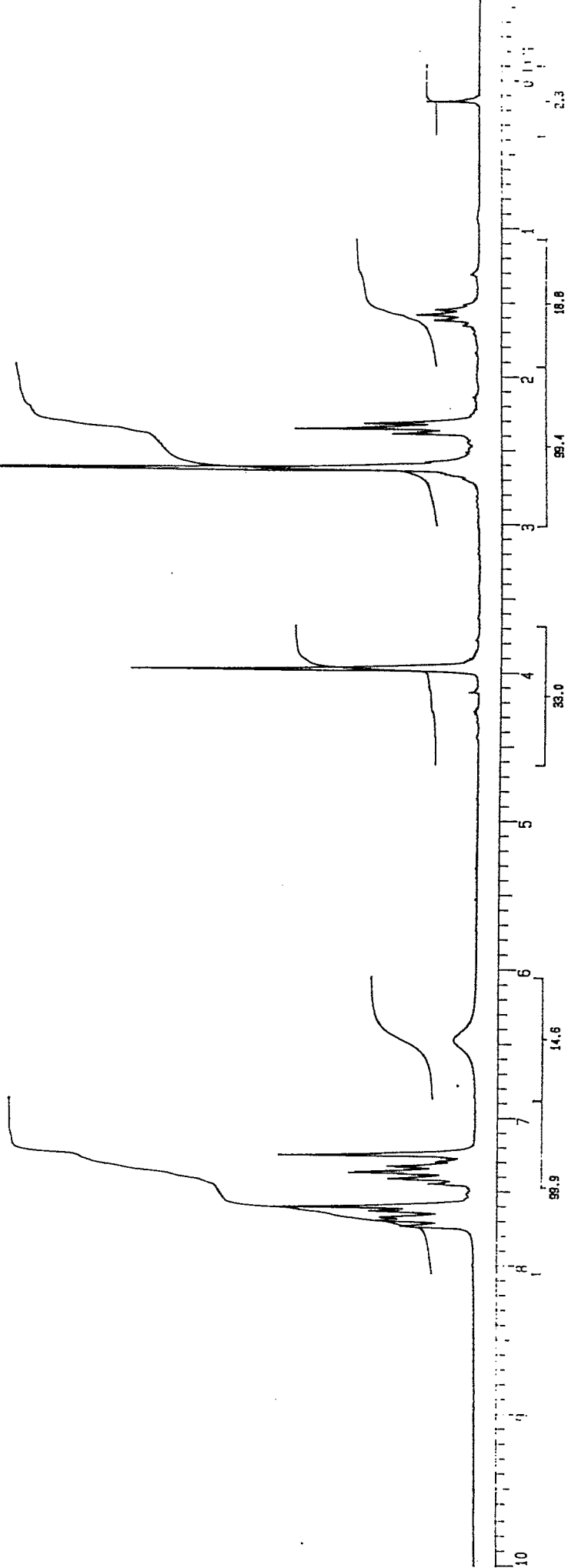
ACQUISITION DEC. & VT
TN 13.000 DM 1.000
SM 13071.9 D0 -300.0
AT 0.627 DM YNY
NP 16384 DM S
PW 13.7 DMF 11.00
P1 27.4 DMF 42.0
D1 2.000 PPLVL 52.0
D2 7.00E-3
TD 0 PROCESSING
TPMR 49.5 SE 0.318
NT 1500 LB 1.000
V-C 1218 SR 80
PMSO, Y 13.7-MATH P
D3 1.00E-3 DISPLA
FB 7200 SP -502.7
BS 64 WP 10561.6
SS 0 VS 348
IL N SC 0
IN Y WC 400
DP Y IS 100
ALOCK A PFL 1503.3
RFP 0
TH 10
INS 1.000
AI DC

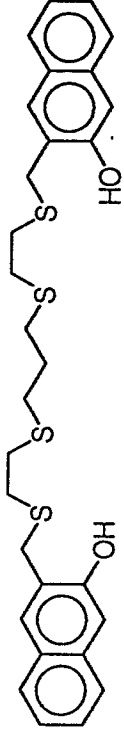


¹H NMR (CDCl₃, 200 MHz)



63





63

ACQUISITION DEC. & VT
 TN 13.000 DN 1.000
 SM 13071.9 D0 -300.0
 AI 0.627 DM YNY
 NP 16384 DMH S
 PM 13.7 DMF 11100
 P1 27.4 DHP 42.0
 D1 2.000 PPLVL 52.0
 D2 7.00E-3
 TD 0 PROCESSING
 TMR 49.5 SE 0.318
 NT 1500 LB 1.000
 157.7 141.8 89.7 50.3
 193.7 141.8 89.7 50.3
 1.00E-3 DISPLAY
 FB 7200 SF -502.7
 BS 64 NP 10561.6
 SS 0 VS 183
 IL N SC 0
 IN Y MC 400
 DP Y IS 100
 ALOCK A PFL 1503.3
 PFP 0
 TH 10
 INS 1.000
 AT DC

