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Report No. GR-141-xxx / Date : May 27, 1994

TITLE : X-ray Analysis of PG-394

Study duration : Mar. 18, 1994 - May 16, 1994

Reported by : Y.Furukawa

Work done by : Y.Furukawa

Directed by : Y.Furukawa

Abstract: Yoji

In order to confirm the structure of PG-394, X-ray analysis was carried out. A crystal with approximate dimensions of 0.5x0.5x0.3 mm was used for the X-ray analysis.

Crystal data:  $C_{18}H_{20}N_2O$ , M.W = 280.37, monoclinic, space group  $P2_1/n$ ,  $a = 10.920(1)\text{\AA}$ ,  $b = 8.341(1)\text{\AA}$ ,  $c = 15.685(2)\text{\AA}$ ,  $\beta = 92.59(1)^\circ$ ,  $V = 1427.1(3)\text{\AA}^3$ ,  $Z = 4$ ,  $D_c = 1.31 \text{ g}\cdot\text{cm}^{-3}$ .

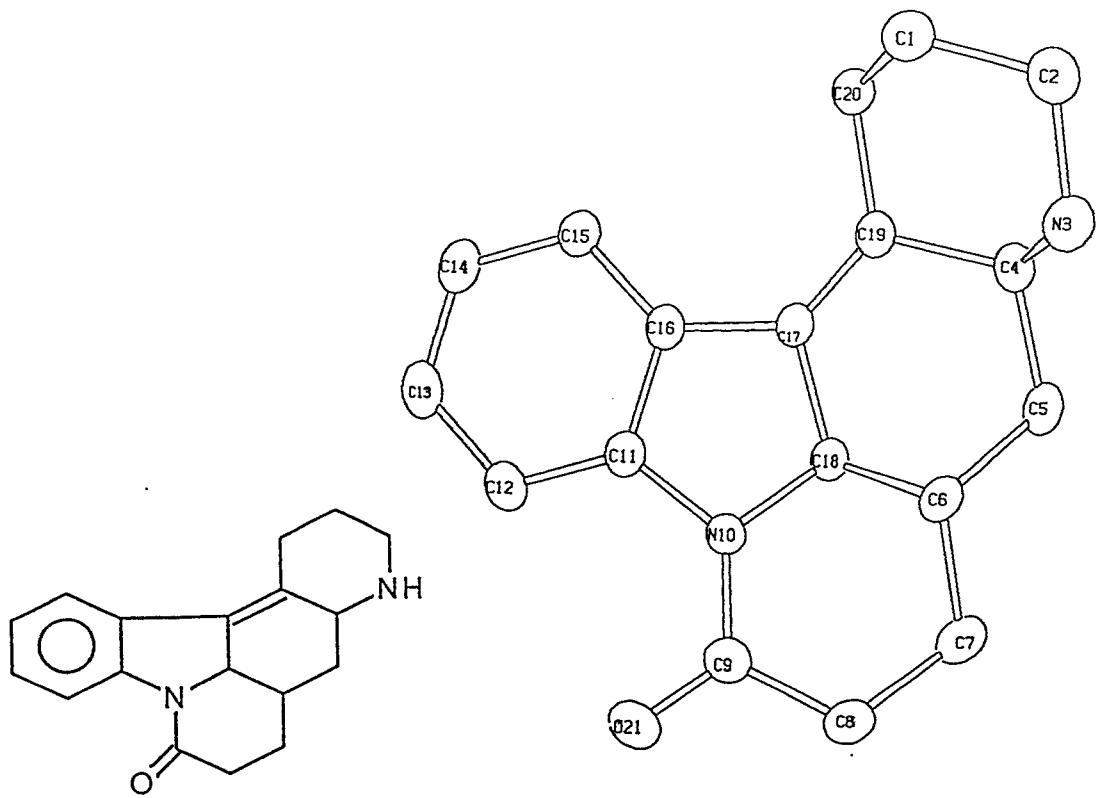
The structure was determined by routine direct methods and cascade block-diagonal least-squares refinement, giving  $R = 0.043$  for 1923 observed reflections ( $\geq 3\sigma(F_o)$ ). Structure of PG-394 was determined.

Diffractometter = RIGAKU Electronic AFC-5  
graphite-monochromated Cu-K $\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ )

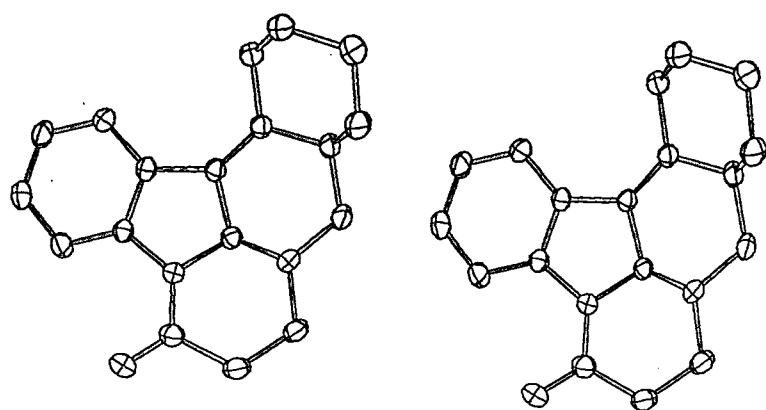
Cell parameters were determined and refined from 20 reflections in the range  $35^\circ < 2\theta < 46^\circ$ .

Data Collection :  $w - 2\theta$  scan Technique  
 $2^\circ < 2\theta < 130^\circ$   
2433 reflections.

Program : MULTAN 78



(a)



(b)

Fig. 1. (a) Perspective view of PG-394 with the atom-numbering scheme.  
(b) Stereoscopic view of PG-394.

Table 1 - 1 . Fractional atomic coordinates ( $\times 10^4$ ) and thermal parameters ( $\text{\AA}^2$ ) of PG-394, with estimated standard deviations in parentheses

ATOM	X	Y	Z	BEQUE
C1	-1327( 2)	-425( 3)	7591( 1)	4.43( 7)
C2	-1276( 2)	-481( 4)	8558( 2)	5.13( 8)
N3	-28( 2)	-213( 3)	8914( 1)	4.50( 6)
C4	481( 2)	1342( 3)	8651( 1)	3.41( 6)
C5	1739( 2)	1588( 3)	9104( 1)	3.71( 6)
C6	2774( 2)	912( 3)	8606( 1)	3.05( 5)
C7	4061( 2)	1148( 3)	8997( 1)	4.12( 6)
C8	5005( 2)	483( 3)	8405( 2)	4.10( 6)
C9	4757( 2)	639( 3)	7445( 1)	3.59( 6)
N10	3636( 1)	1186( 2)	7180( 1)	3.11( 4)
C11	3110( 2)	1189( 3)	6339( 1)	2.94( 5)
C12	3692( 2)	1006( 3)	5582( 1)	3.72( 6)
C13	2971( 2)	1015( 3)	4827( 1)	4.09( 6)
C14	1725( 2)	1234( 3)	4839( 1)	4.02( 6)
C15	1146( 2)	1436( 3)	5601( 1)	3.54( 6)
C16	1838( 2)	1407( 2)	6368( 1)	2.87( 5)
C17	1515( 2)	1533( 2)	7271( 1)	2.74( 5)
C18	2718( 2)	1753( 2)	7762( 1)	2.87( 5)
C19	480( 2)	1389( 3)	7677( 1)	3.02( 5)
C20	-778( 2)	1127( 3)	7271( 1)	3.78( 6)
O21	5528( 1)	217( 2)	6951( 1)	4.91( 5)

Table 1 - 2. Fractional atomic coordinates ( $\times 10^3$ ) and isotropic thermal parameters ( $\text{\AA}^2$ ) for hydrogen atoms of PG-394 with estimated standard deviations in parentheses

ATOM	X	Y	Z	B
H1A	-216( 2)	-52( 3)	739( 1)	4.7( 5)
H1B	-87( 2)	-142( 3)	737( 2)	6.7( 7)
H2A	-156( 2)	-169( 3)	873( 2)	7.2( 7)
H2B	-186( 2)	30( 3)	877( 2)	6.8( 7)
HN3	11( 5)	-46( 6)	943( 3)	19.0(18)
H4	-8( 2)	226( 2)	885( 1)	3.6( 5)
H5A	173( 2)	106( 3)	968( 1)	3.9( 5)
H5B	187( 2)	274( 3)	920( 1)	4.5( 5)
H6	264( 2)	-19( 2)	850( 1)	2.3( 4)
H7A	413( 2)	64( 3)	959( 1)	5.1( 6)
H7B	419( 2)	225( 3)	911( 1)	5.2( 6)
H8A	512( 2)	-69( 3)	848( 1)	5.4( 6)
H8B	582( 2)	103( 3)	852( 1)	5.1( 6)
H12	459( 2)	97( 3)	559( 1)	4.4( 5)
H13	339( 2)	96( 3)	427( 1)	4.6( 5)
H14	123( 2)	121( 3)	430( 1)	4.7( 5)
H15	24( 2)	162( 3)	559( 1)	3.8( 5)
H18	288( 2)	288( 2)	786( 1)	3.0( 4)
H20A	-129( 2)	206( 3)	744( 1)	5.0( 6)
H20B	-77( 2)	115( 3)	661( 1)	5.0( 5)

Table 2 . Bond lengths (Å) and bond angles (°) of PG-394,  
with estimated standard deviations in parentheses

ATOM		DISTANCE	ATOM		DISTANCE
C1	-C2	1.515( 3)	N10	-C11	1.415( 2)
C1	-C20	1.521( 4)	N10	-C18	1.464( 3)
C2	-N3	1.467( 3)	C11	-C12	1.379( 3)
N3	-C4	1.477( 3)	C11	-C16	1.403( 3)
C4	-C5	1.531( 3)	C12	-C13	1.393( 3)
C4	-C19	1.528( 3)	C13	-C14	1.374( 3)
C5	-C6	1.513( 3)	C14	-C15	1.387( 3)
C6	-C7	1.520( 3)	C15	-C16	1.392( 3)
C6	-C18	1.497( 3)	C16	-C17	1.480( 3)
C7	-C8	1.523( 3)	C17	-C18	1.503( 3)
C8	-C9	1.523( 3)	C17	-C19	1.328( 3)
C9	-N10	1.355( 3)	C19	-C20	1.504( 3)
C9	-O21	1.222( 3)			
ATOM		ANGLE	ATOM		ANGLE
C2	-C1	111.1( 2)	N10	-C11	109.3( 2)
C1	-C2	111.6( 2)	C12	-C11	122.5( 2)
C2	-N3	112.5( 2)	C11	-C12	117.8( 2)
N3	-C4	109.2( 2)	C12	-C13	120.8( 2)
N3	-C4	108.5( 2)	C13	-C14	121.2( 2)
C5	-C4	114.9( 2)	C14	-C15	119.5( 2)
C4	-C5	112.7( 2)	C11	-C16	118.3( 2)
C5	-C6	116.2( 2)	C11	-C16	108.7( 2)
C5	-C6	106.3( 2)	C15	-C16	133.0( 2)
C7	-C6	107.2( 2)	C16	-C17	104.9( 2)
C6	-C7	110.2( 2)	C16	-C17	134.2( 2)
C7	-C8	118.4( 2)	C13	-C17	120.6( 2)
C8	-C9	116.8( 2)	C6	-C18	113.5( 2)
C8	-C9	120.4( 2)	C6	-C18	113.3( 2)
N10	-C9	122.7( 2)	N10	-C18	104.4( 2)
C9	-N10	127.6( 2)	C4	-C19	121.3( 2)
C9	-N10	123.5( 2)	C4	-C19	112.3( 2)
C11	-N10	108.7( 1)	C17	-C19	126.3( 2)
N10	-C11	128.2( 2)	C1	-C20	110.5( 2)

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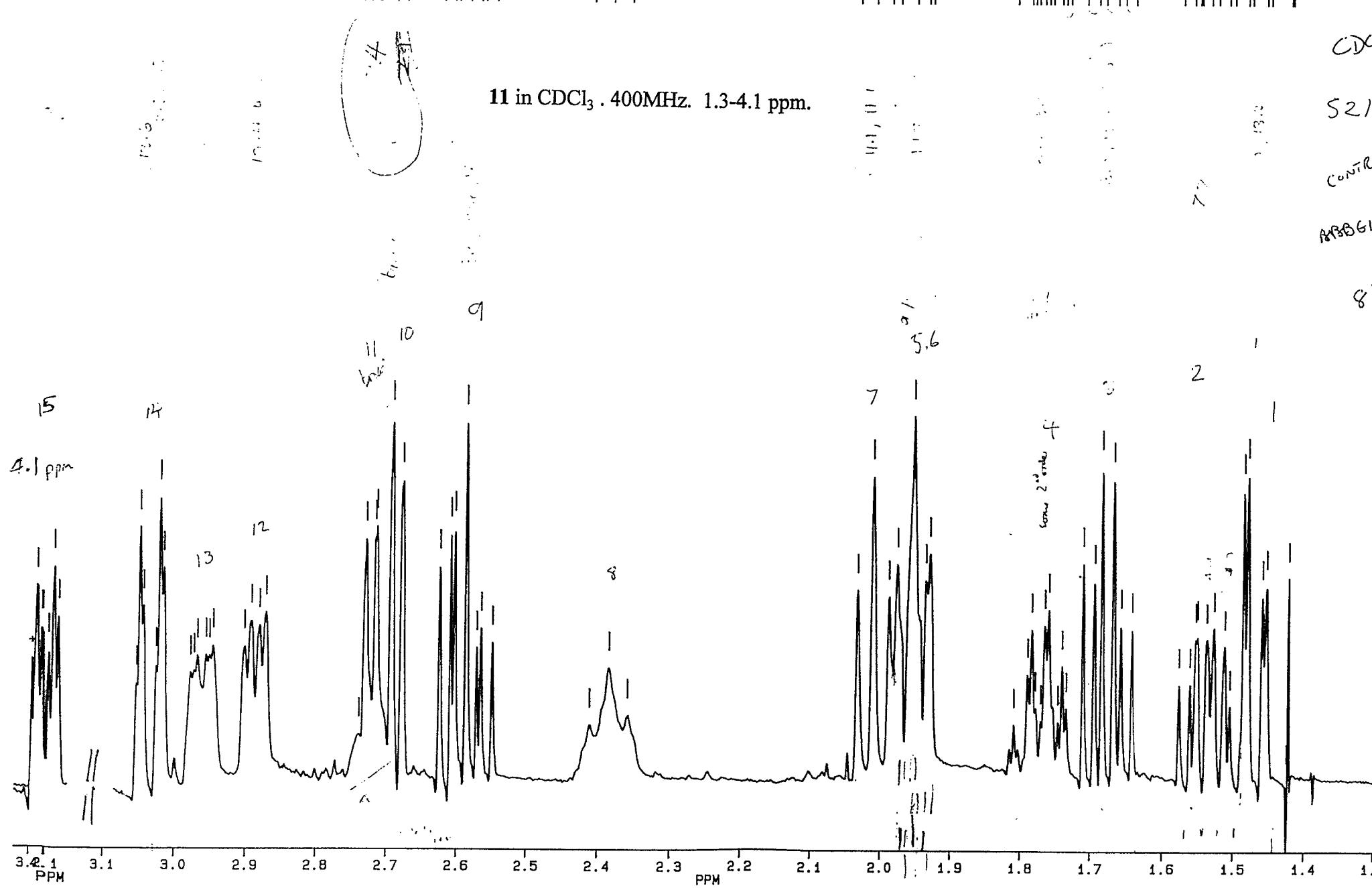
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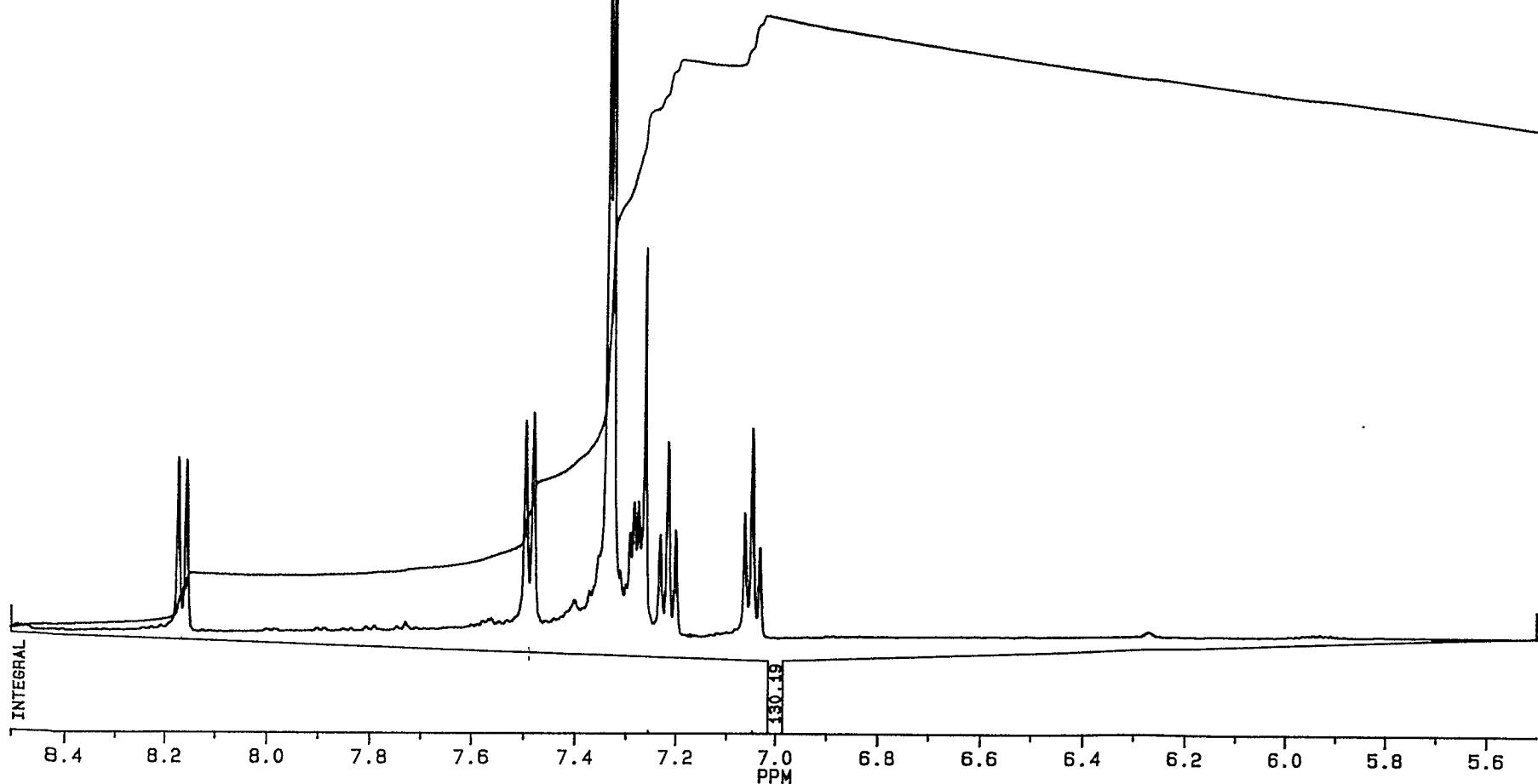
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11 in  $\text{CDCl}_3$ . 400MHz. 1.3-4.1 ppm.

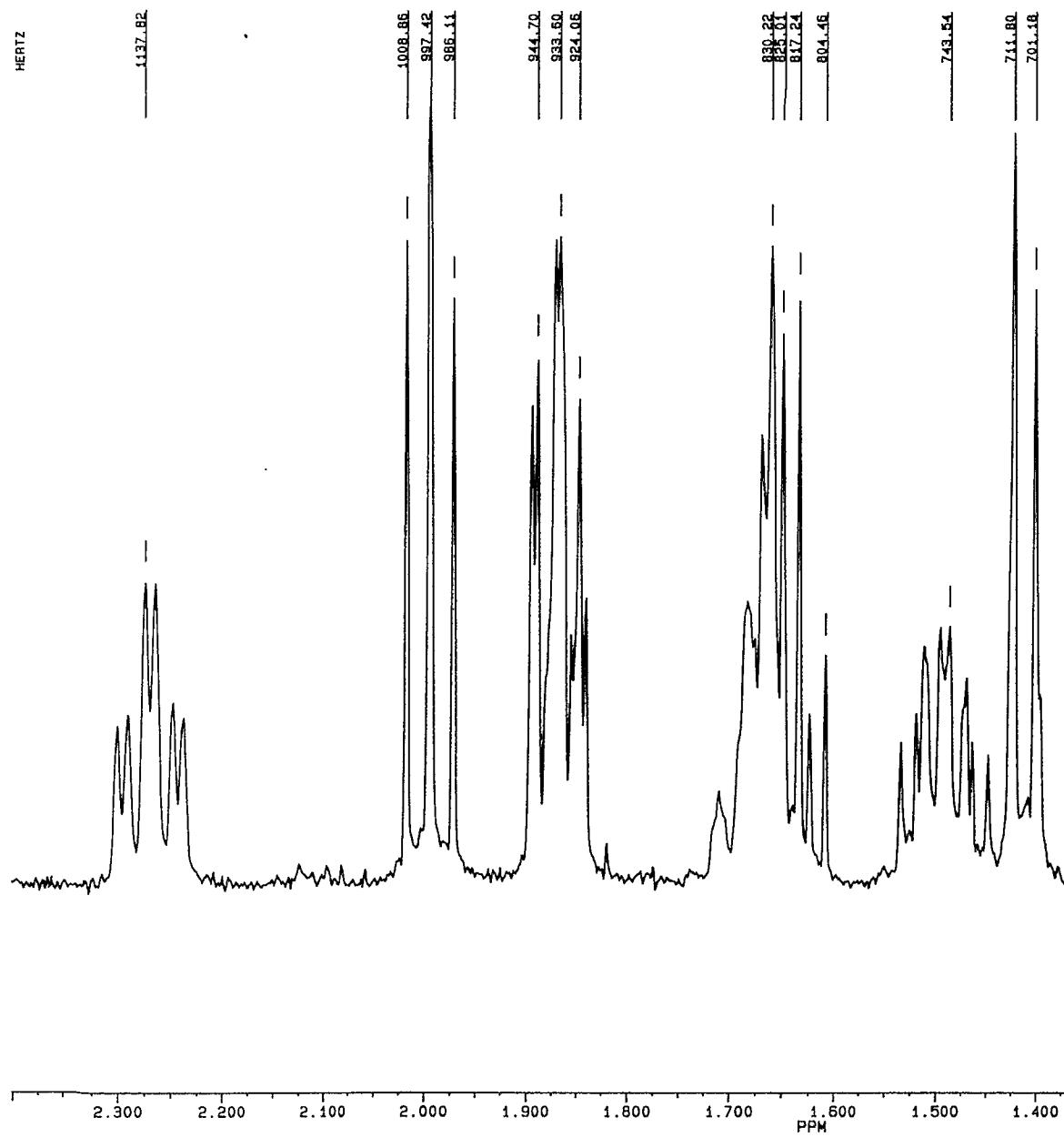


11 in  $\text{CDCl}_3$ . 400MHz. 5.6-8.4 ppm.



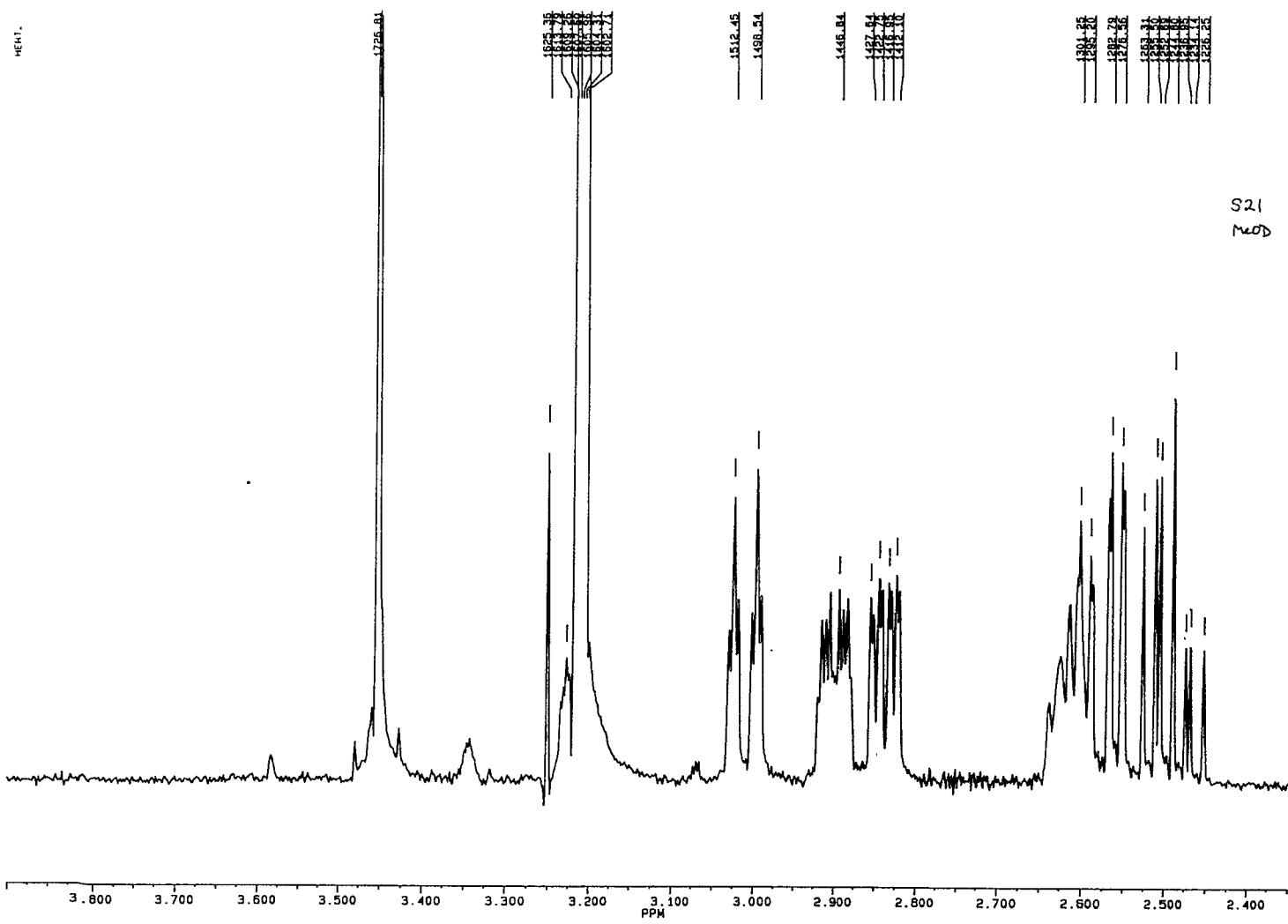
**11 in CD<sub>3</sub>OD. 400MHz. 1.4-2.4 ppm.**

HERTZ

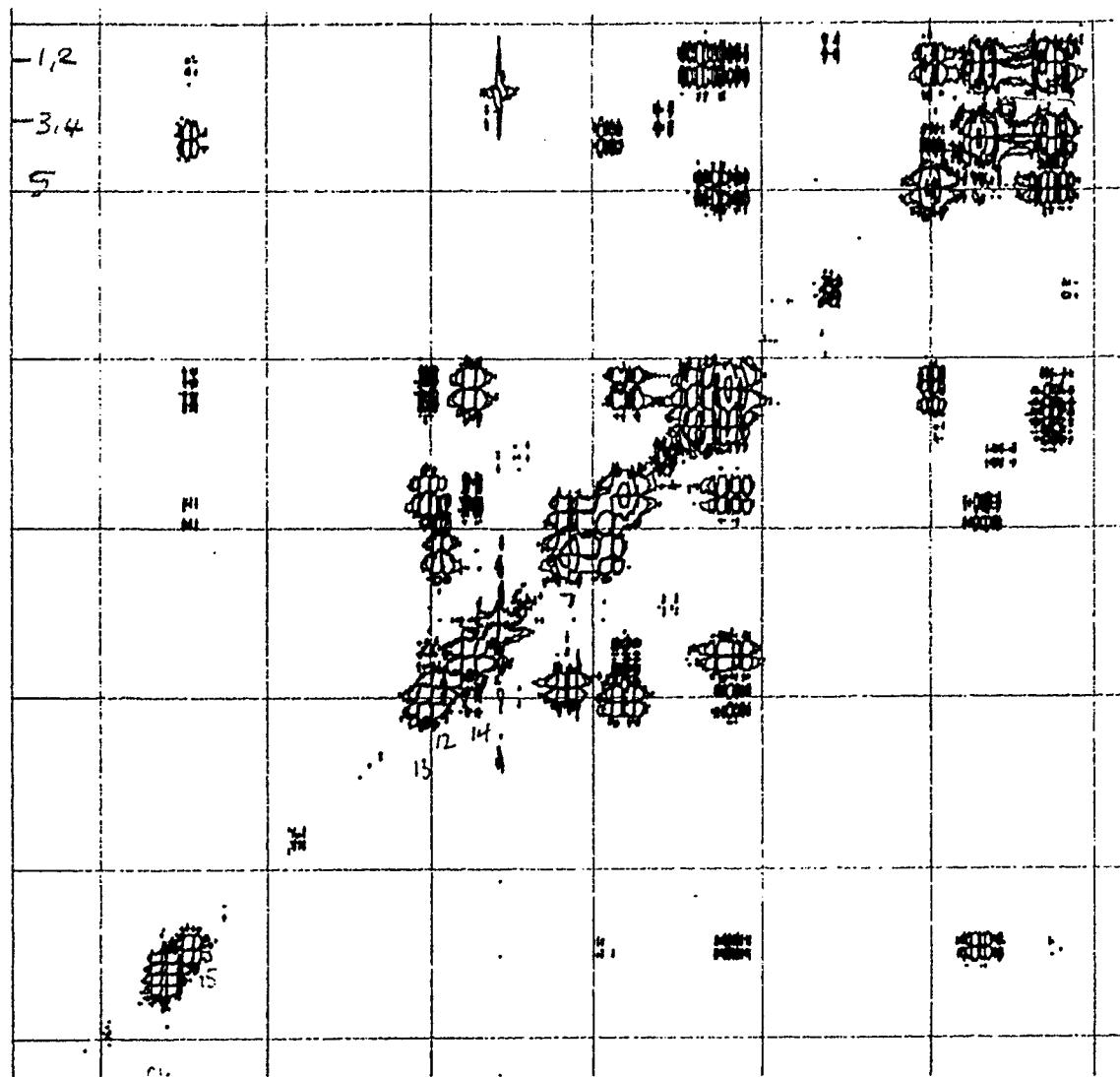


**11** in CD<sub>3</sub>OD. 400MHz. 2.4-3.6 ppm.

MHz.

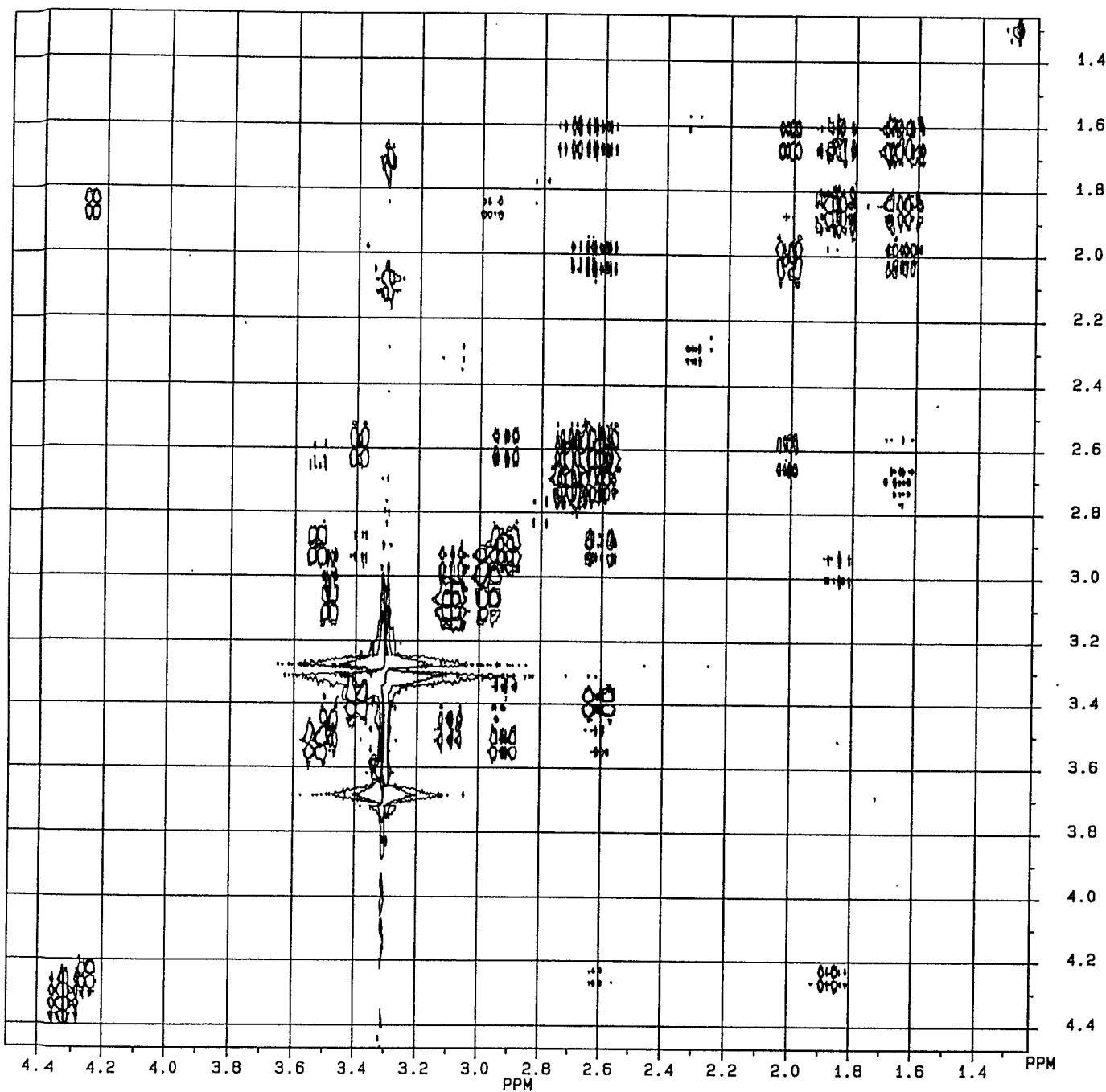


11 in CD<sub>3</sub>OD. 400MHz COSY spectrum. 1.4-4.4 ppm



BB191 S21+DCL COSY NMR 400MHz 298K

11 in  $\text{CD}_3\text{OD} + \text{DCl}$ . 400MHz COSY spectrum. 1.4-4.4 ppm



WD=Q

SSB = 6,6

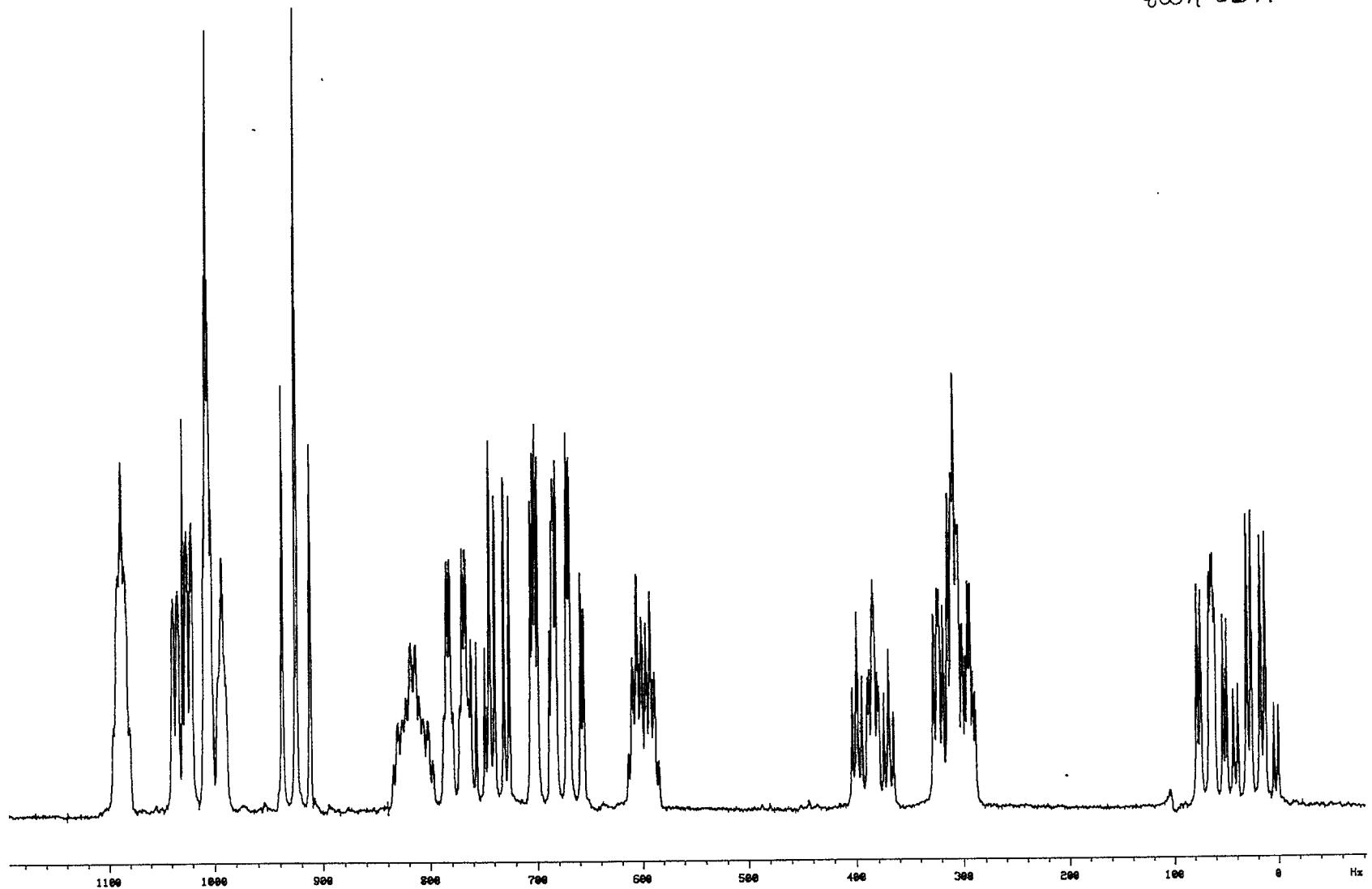
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PC0 -90 , O XFP

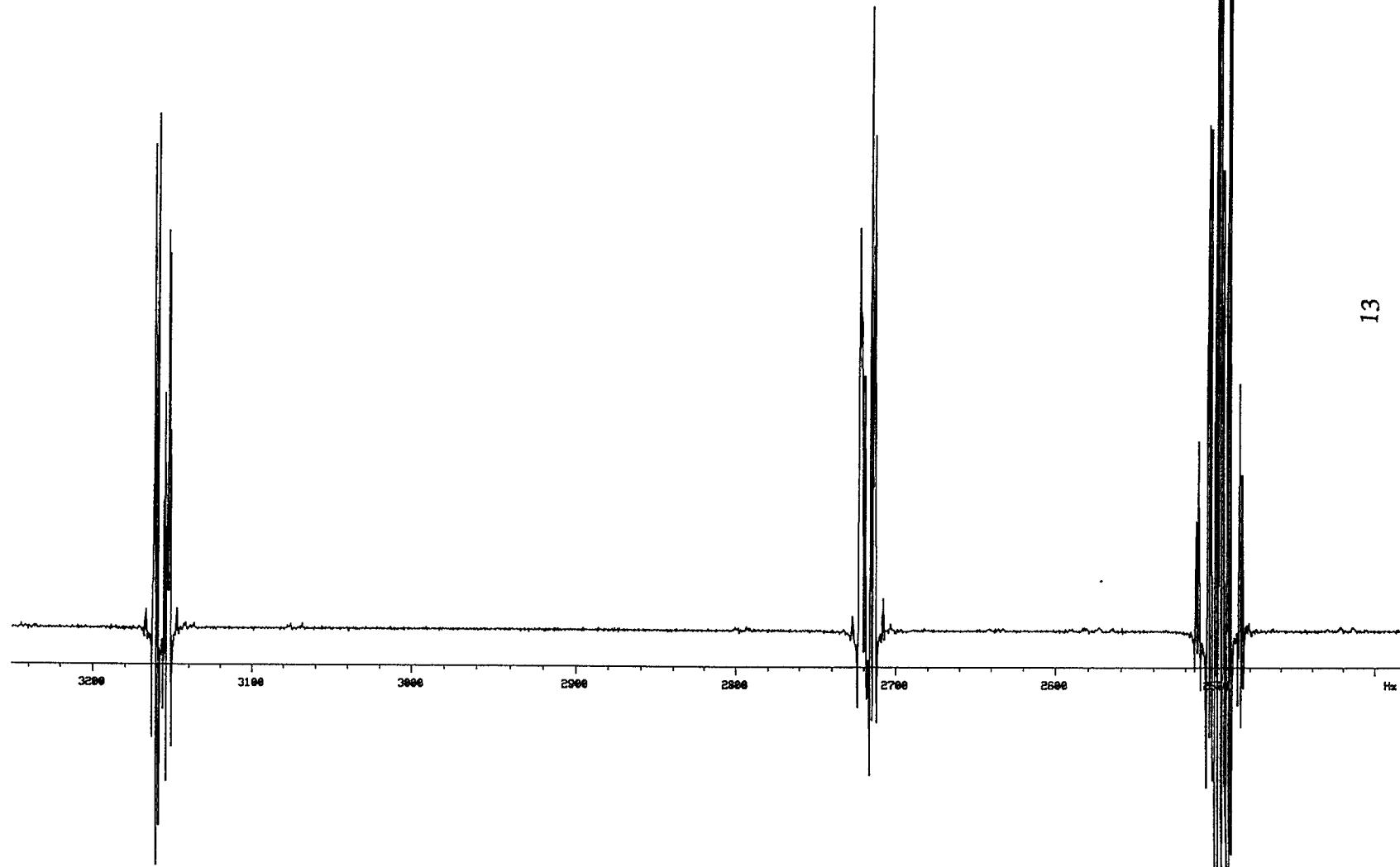
13 in CD<sub>3</sub>OD. 600MHz. 1.3-3-3 ppm

66h 56.1

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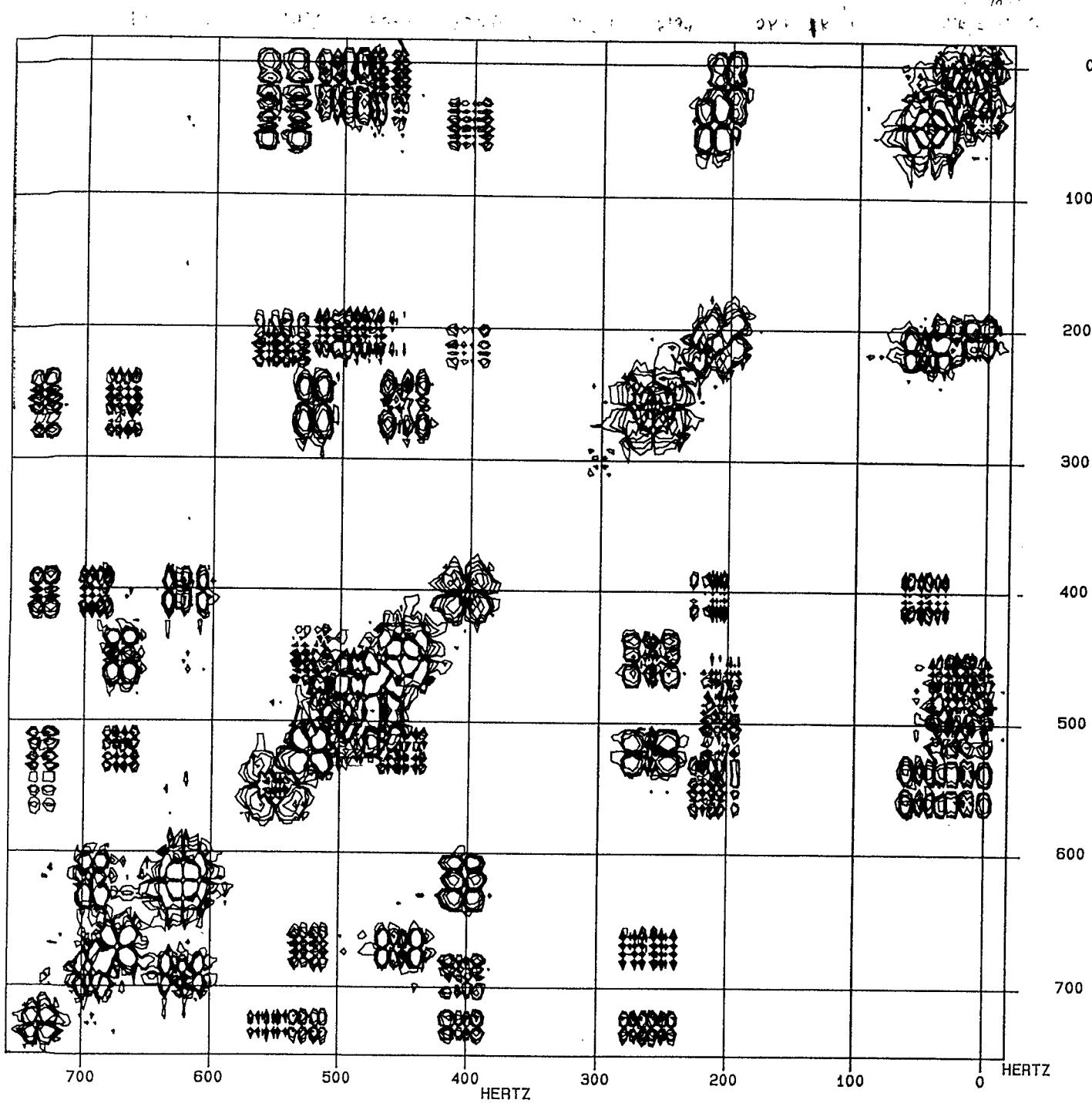


13 in CD<sub>3</sub>OD. 600MHz. Aromatic proton resonances.



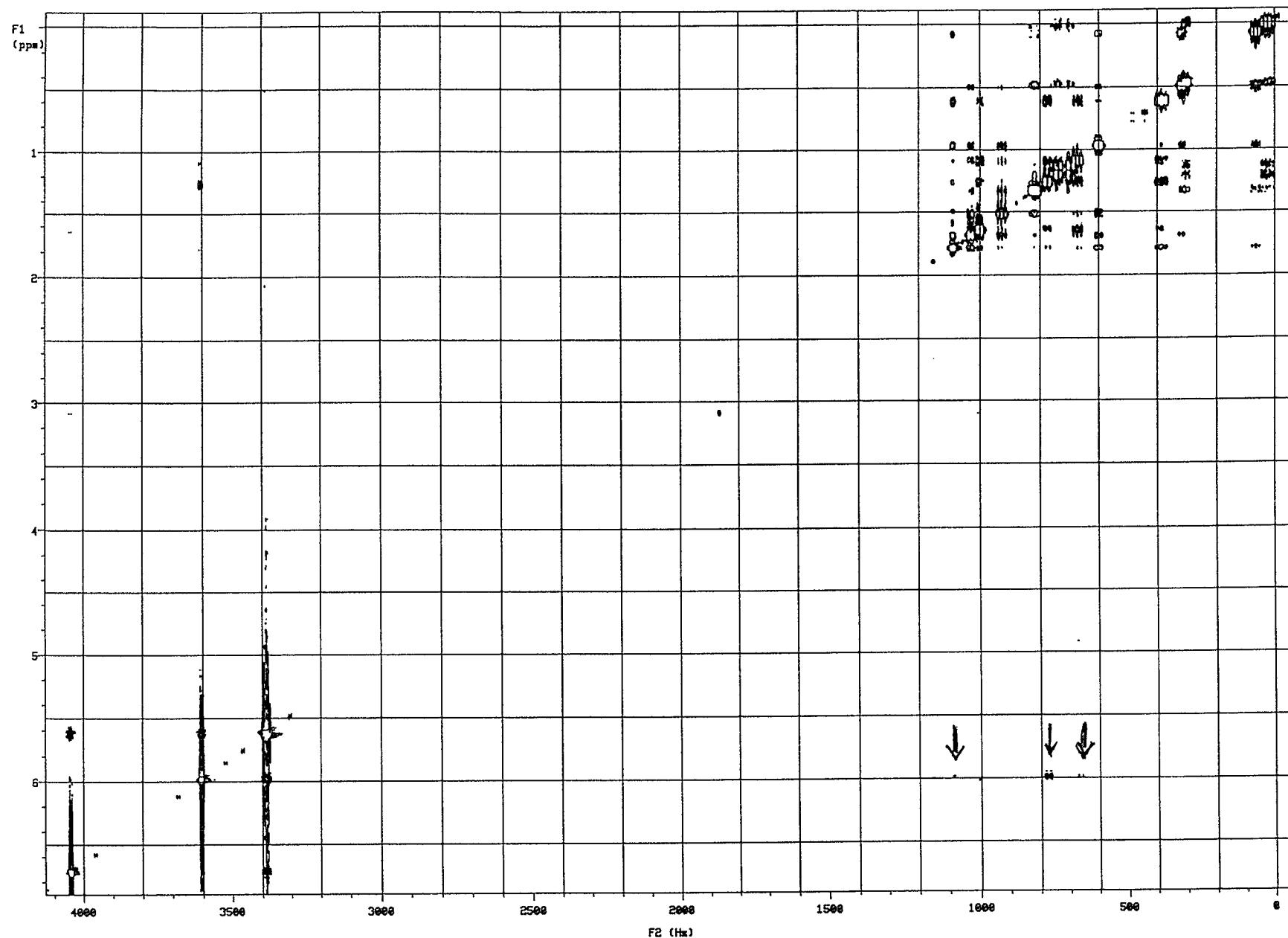
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$^{13}\text{C}$  in  $\text{CD}_3\text{OD}$ . 400MHz. COSY spectrum. Aliphatic resonances.



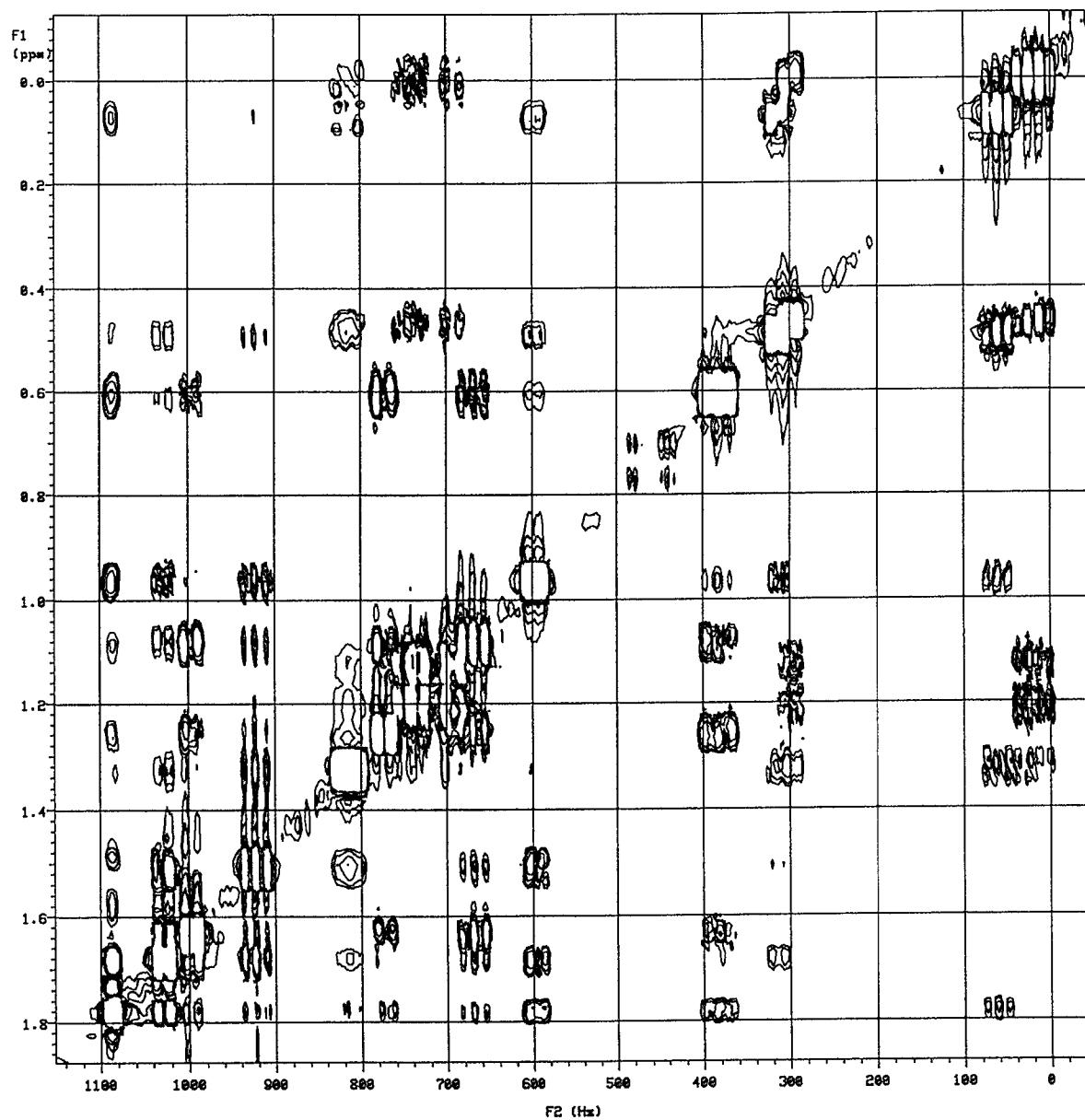
**13** in  $\text{CD}_3\text{OD}$ . 600MHz. ROESY spectrum showing crosspeaks (arrowed) between the  $\text{H}_{13}$  proton and three aliphatic resonances ( $\text{H}_{4b}$  and both 1-protons).

S47

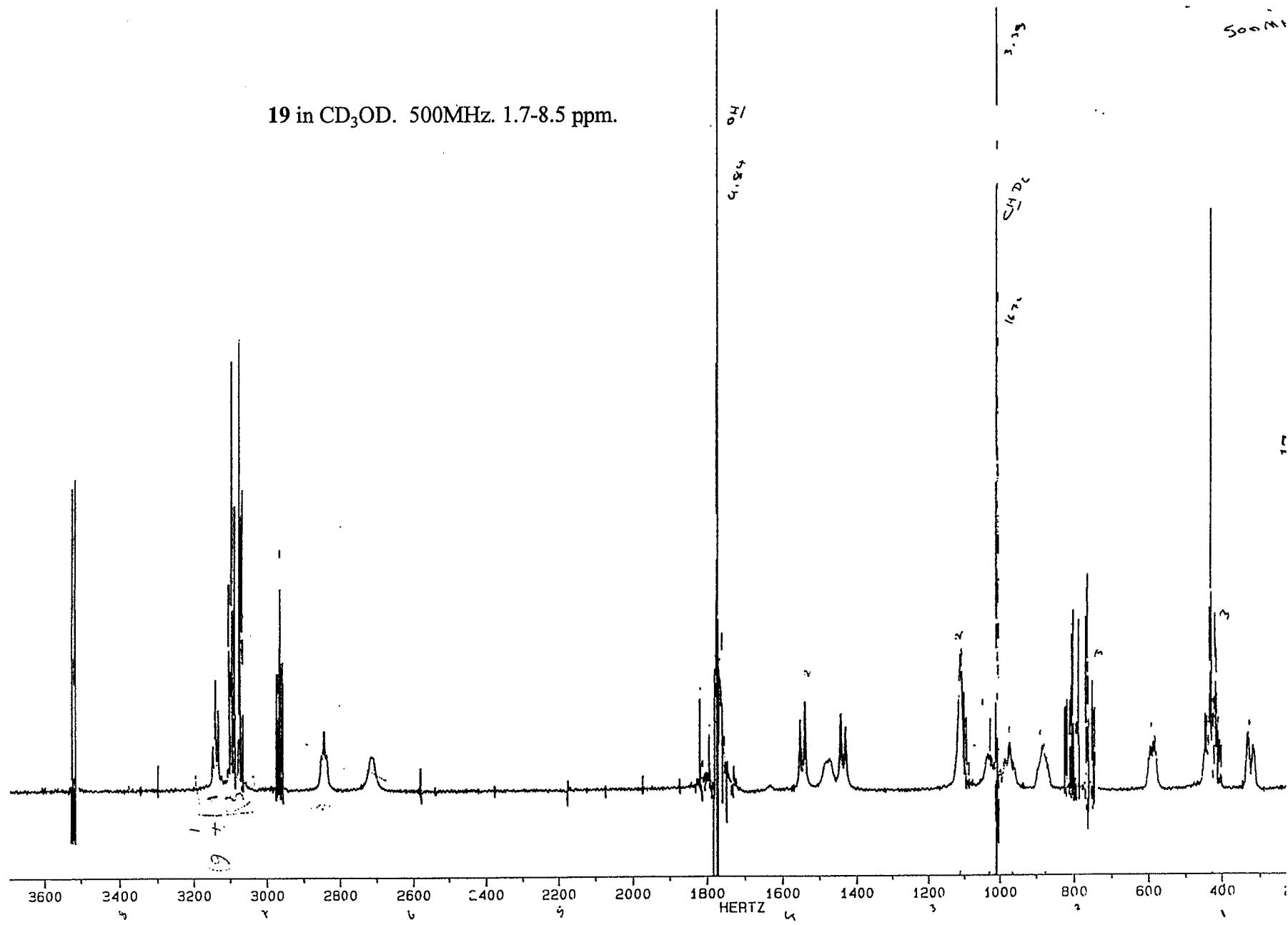


S1

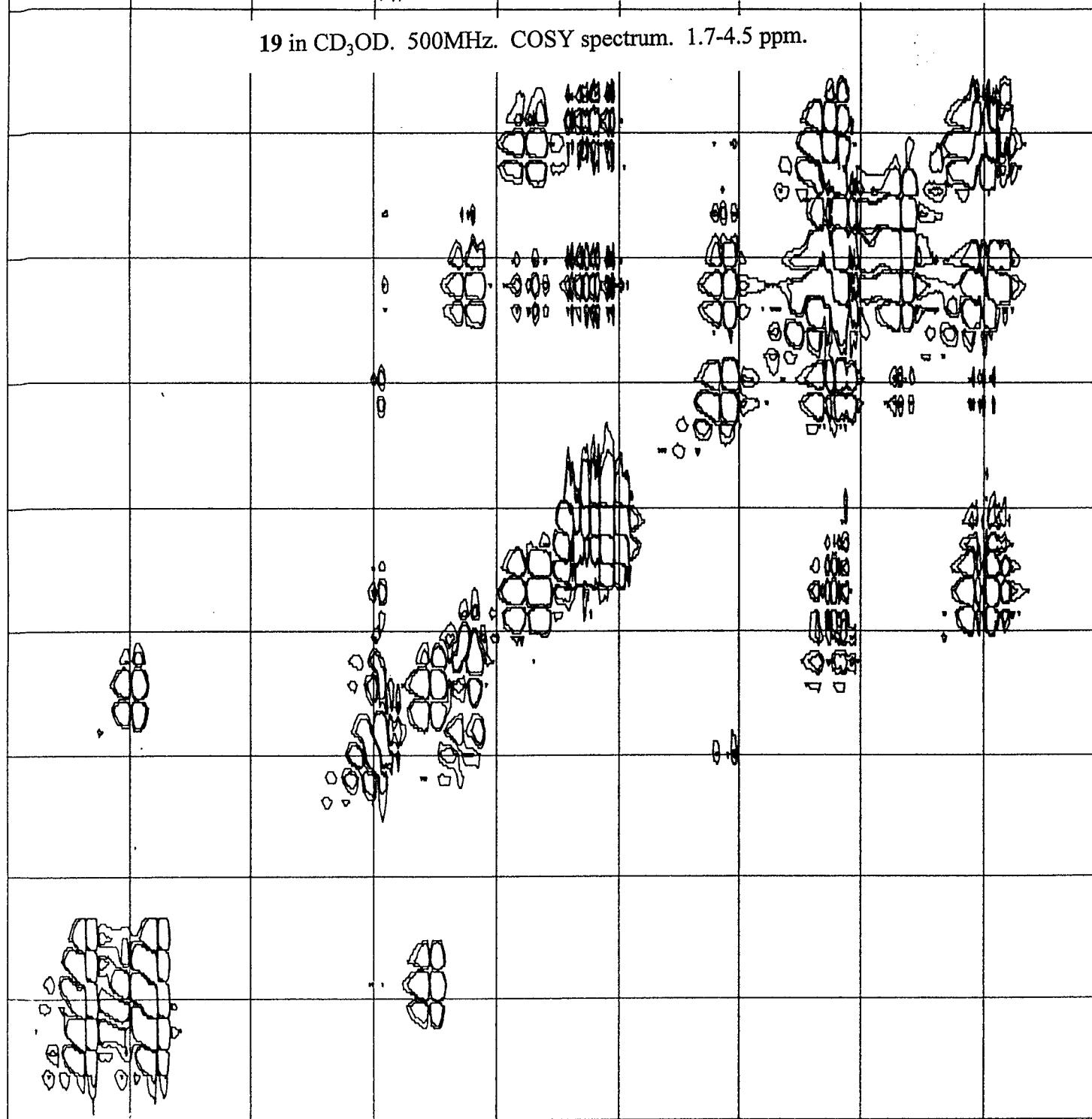
<sup>13</sup>C in CD<sub>3</sub>OD. 600MHz. Expanded region of the ROESY spectrum showing the crosspeaks between the aliphatic proton resonances.



19 in CD<sub>3</sub>OD. 500MHz. 1.7-8.5 ppm.



$^{19}\text{F}$  in  $\text{CD}_3\text{OD}$ . 500MHz. COSY spectrum. 1.7-4.5 ppm.



20 in CD<sub>3</sub>OD. 500MHz. 1.4-8.6 ppm.

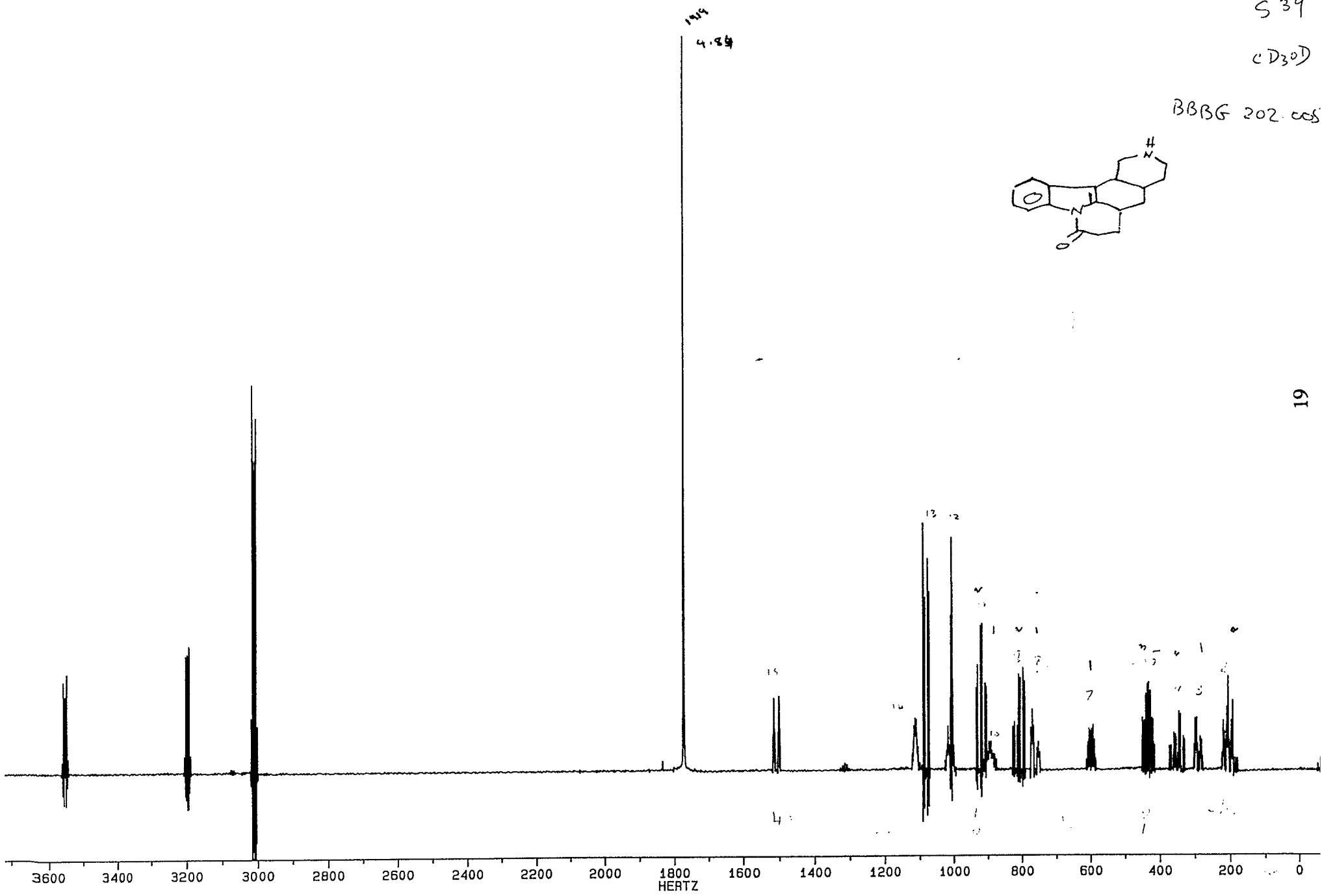
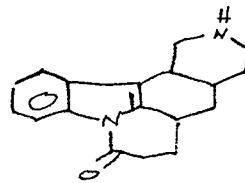
300°C

500MHz

S 39

CD<sub>3</sub>OD

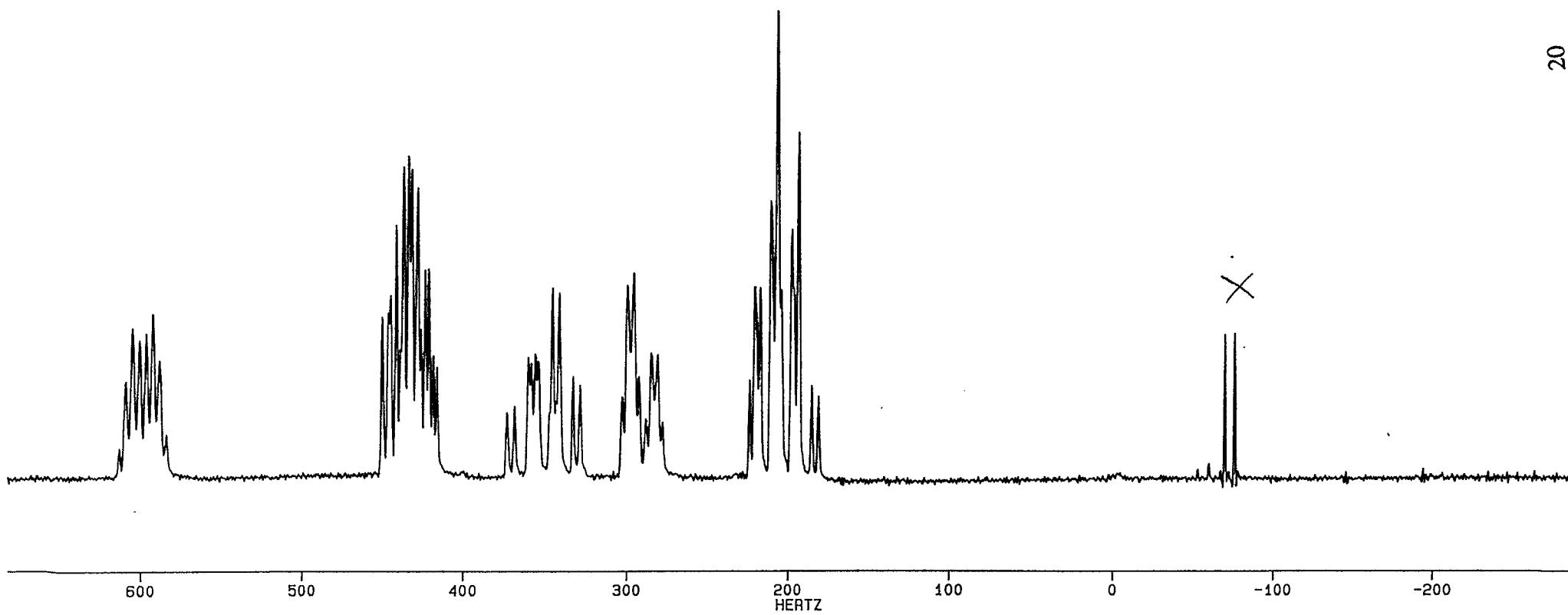
BBBG 202.005



SB9

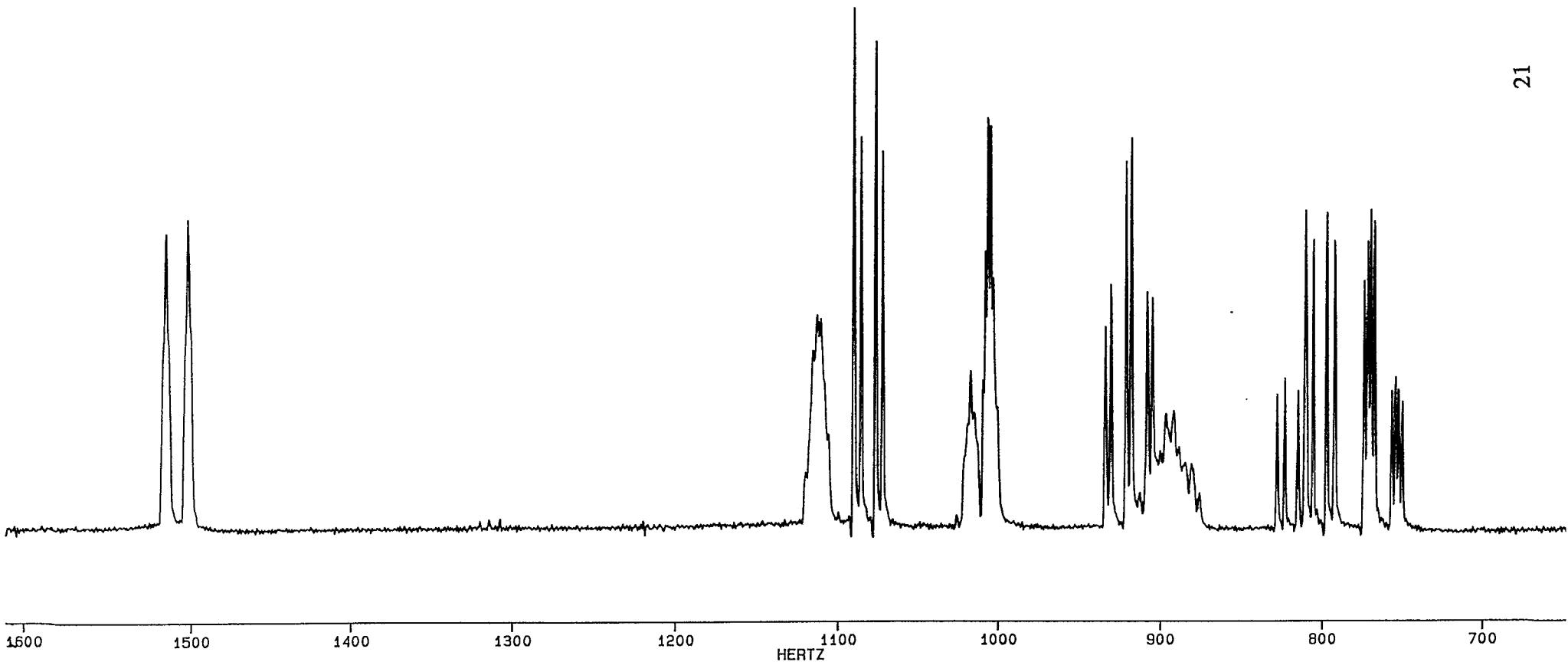
20 in CD<sub>3</sub>OD. 500MHz. 0.8-2.5 ppm.

CONT'D



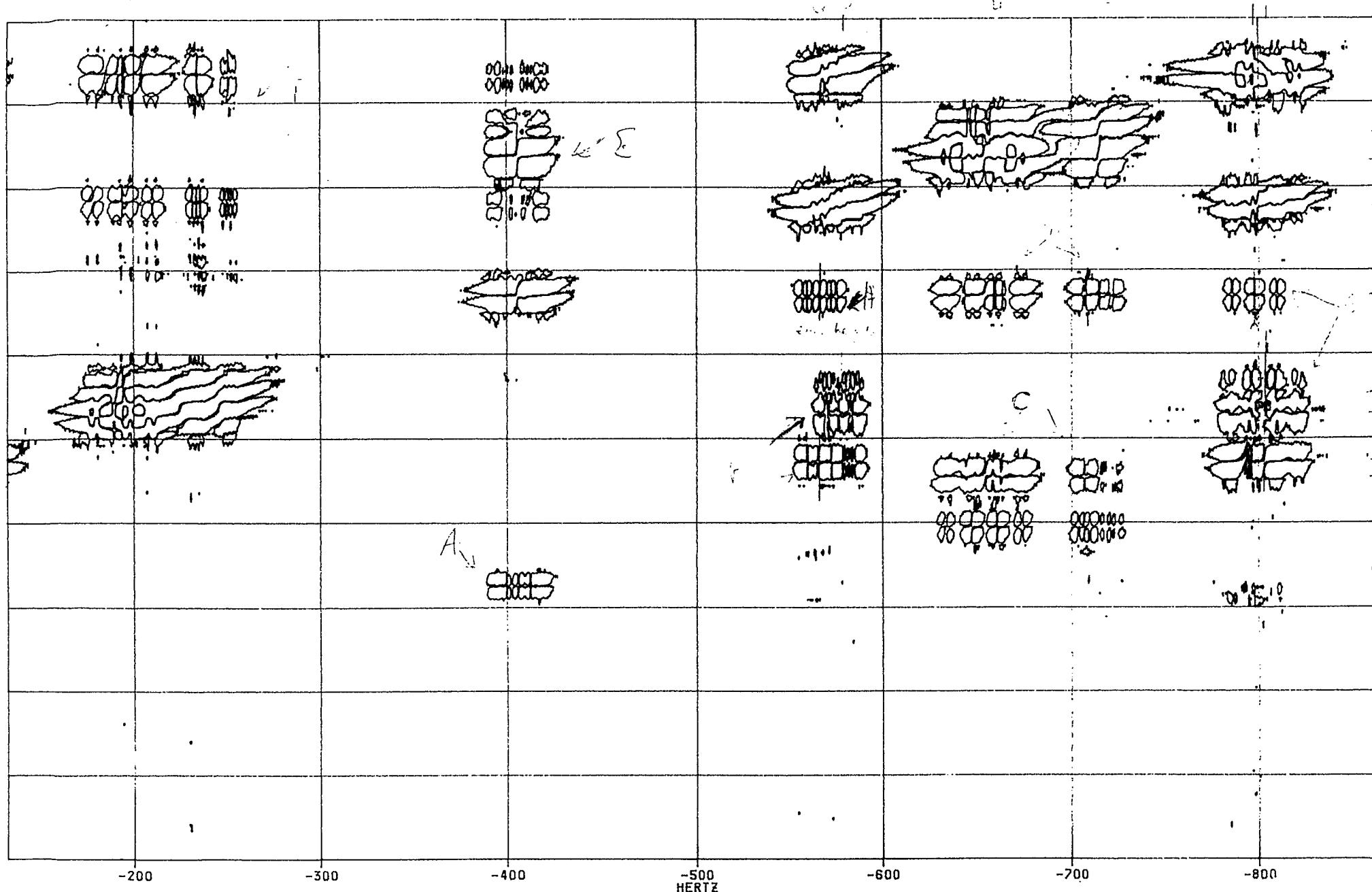
5367  
Corresp. 21

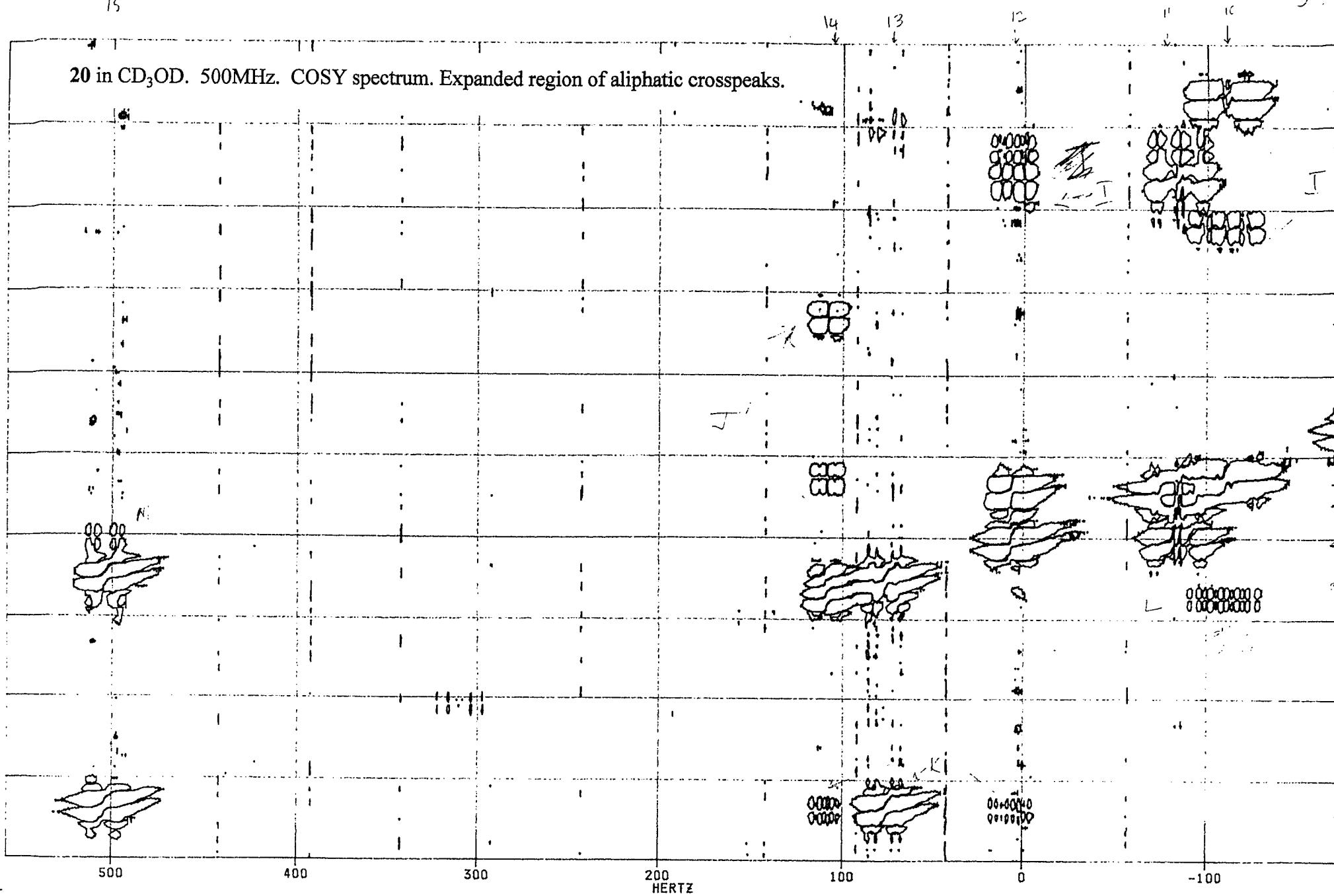
20 in CD<sub>3</sub>OD. 500MHz. 2.5-4.2 ppm.



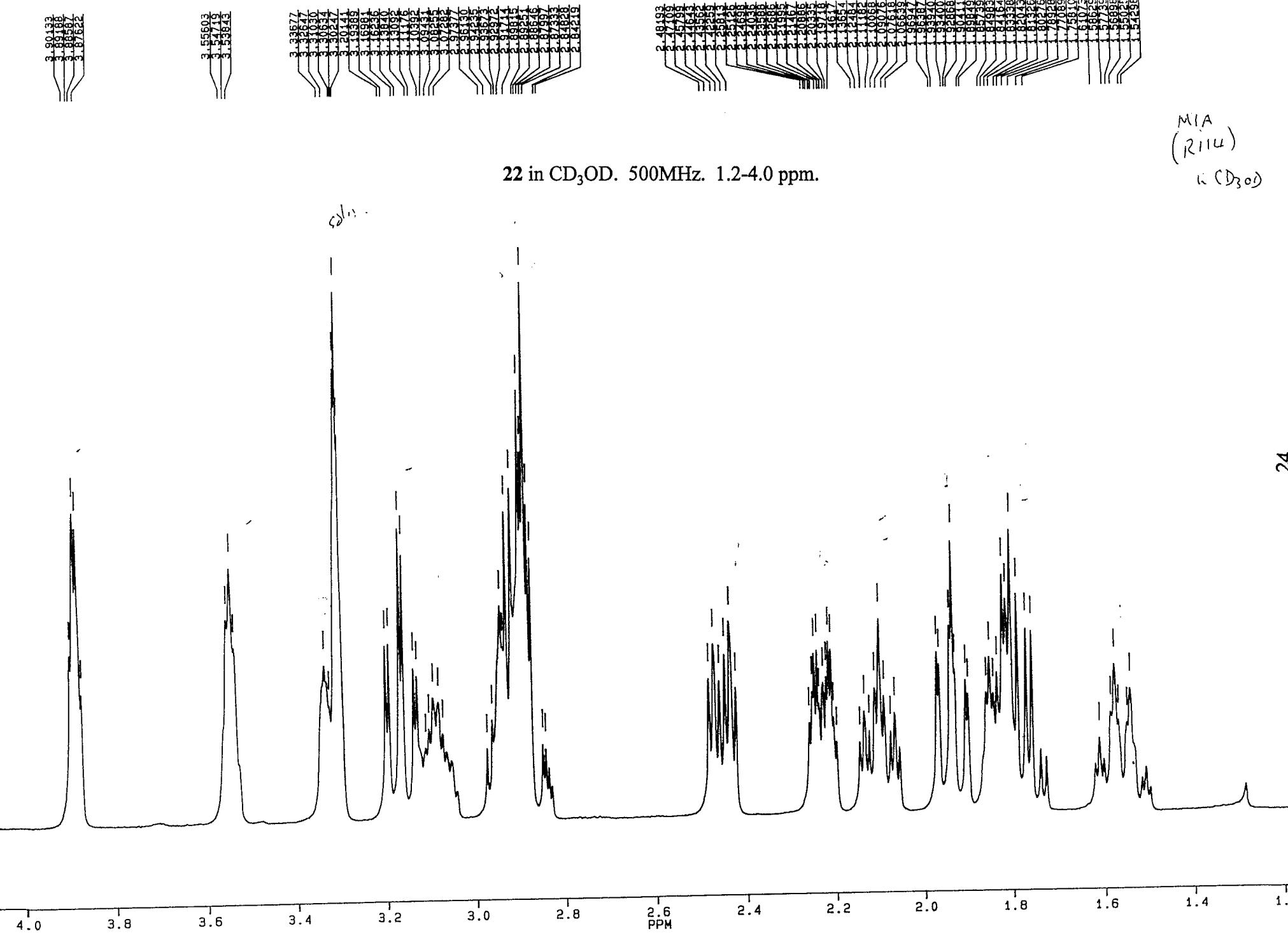
<sup>20</sup> in CD<sub>3</sub>OD. 500MHz. COSY spectrum. Expanded region of aliphatic crosspeaks.

S 29



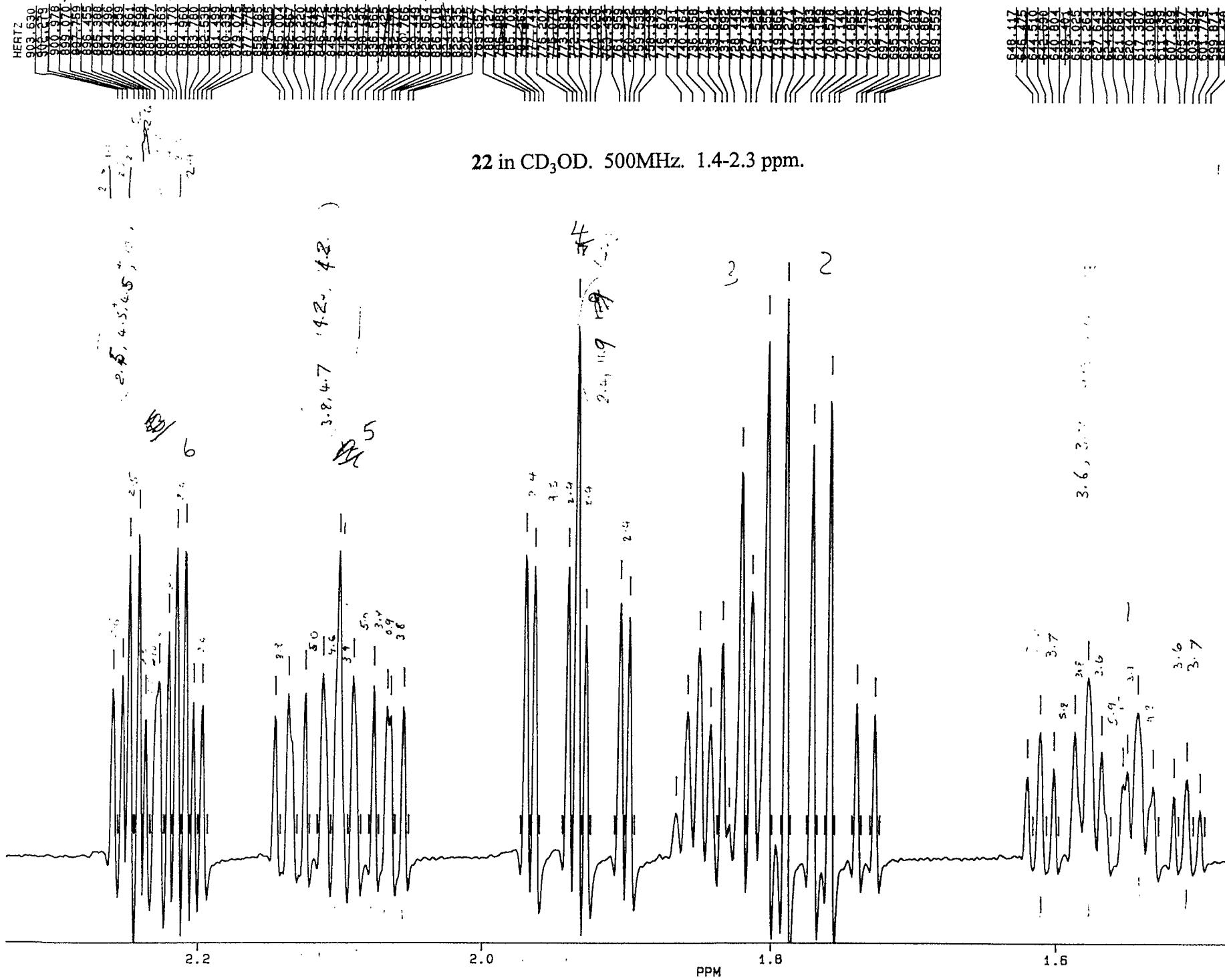


PPM



HERTZ

HEH 1/2  
903.630



**22** in CD<sub>3</sub>OD. 500MHz. 1.4-2.3 ppm.

HERTZ  
1338.66  
1337.18  
1336.71  
1335.24  
1334.77  
1333.30  
1332.83  
1331.36  
1330.89  
1329.42  
1328.95  
1327.48  
1326.01  
1324.54  
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1321.60  
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1314.72  
1313.25  
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1307.37  
1305.90  
1304.43  
1302.96  
1301.49  
1300.02  
1298.55  
1296.08  
1294.61  
1293.14  
1291.67  
1290.20  
1288.73  
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1281.38  
1280.91  
1279.44  
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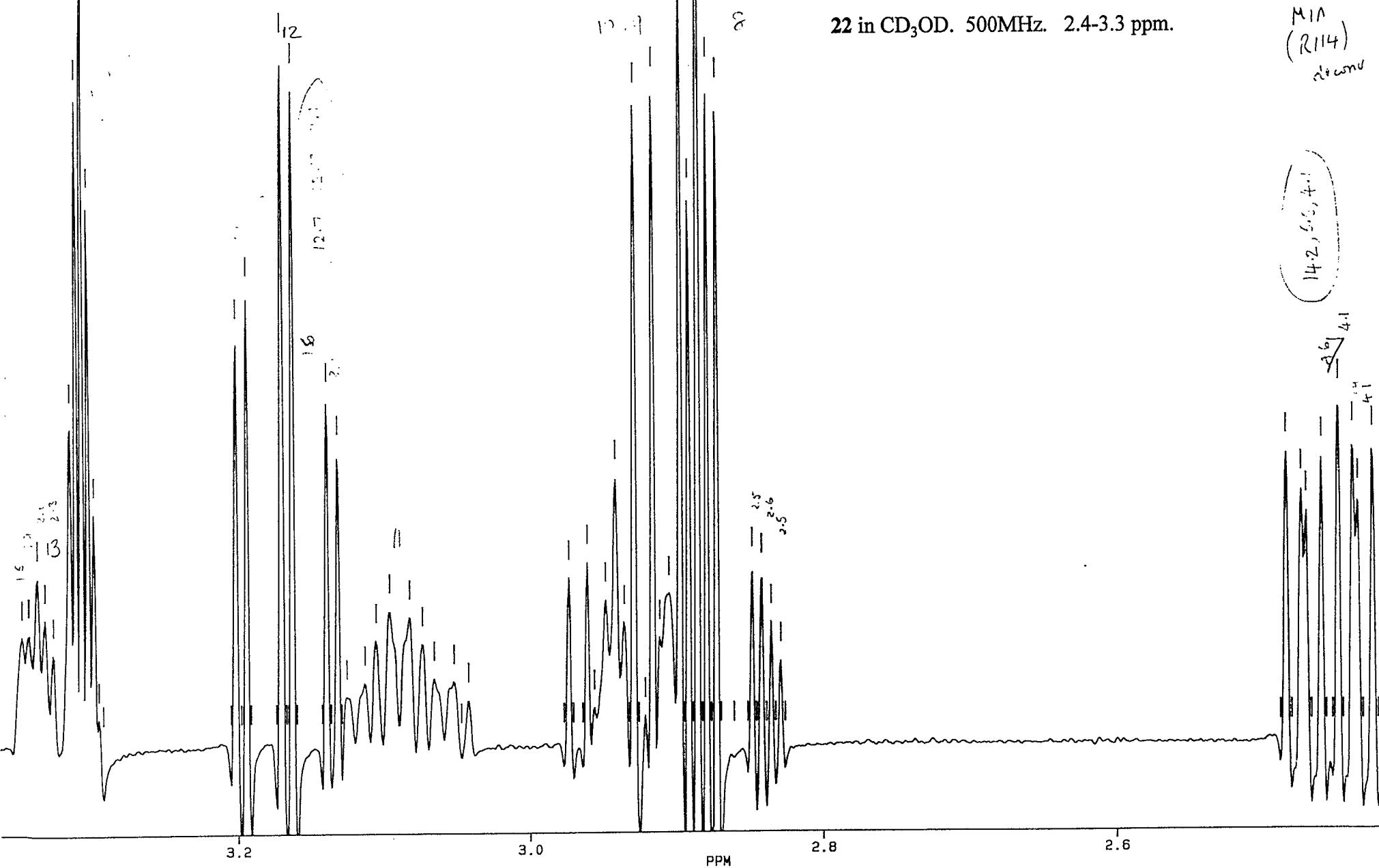
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1011.55  
1010.08  
1008.61  
1007.14  
1005.67  
1004.20  
1002.73  
1001.26  
1000.79  
1000.00

22 in CD<sub>3</sub>OD. 500MHz. 2.4-3.3 ppm.

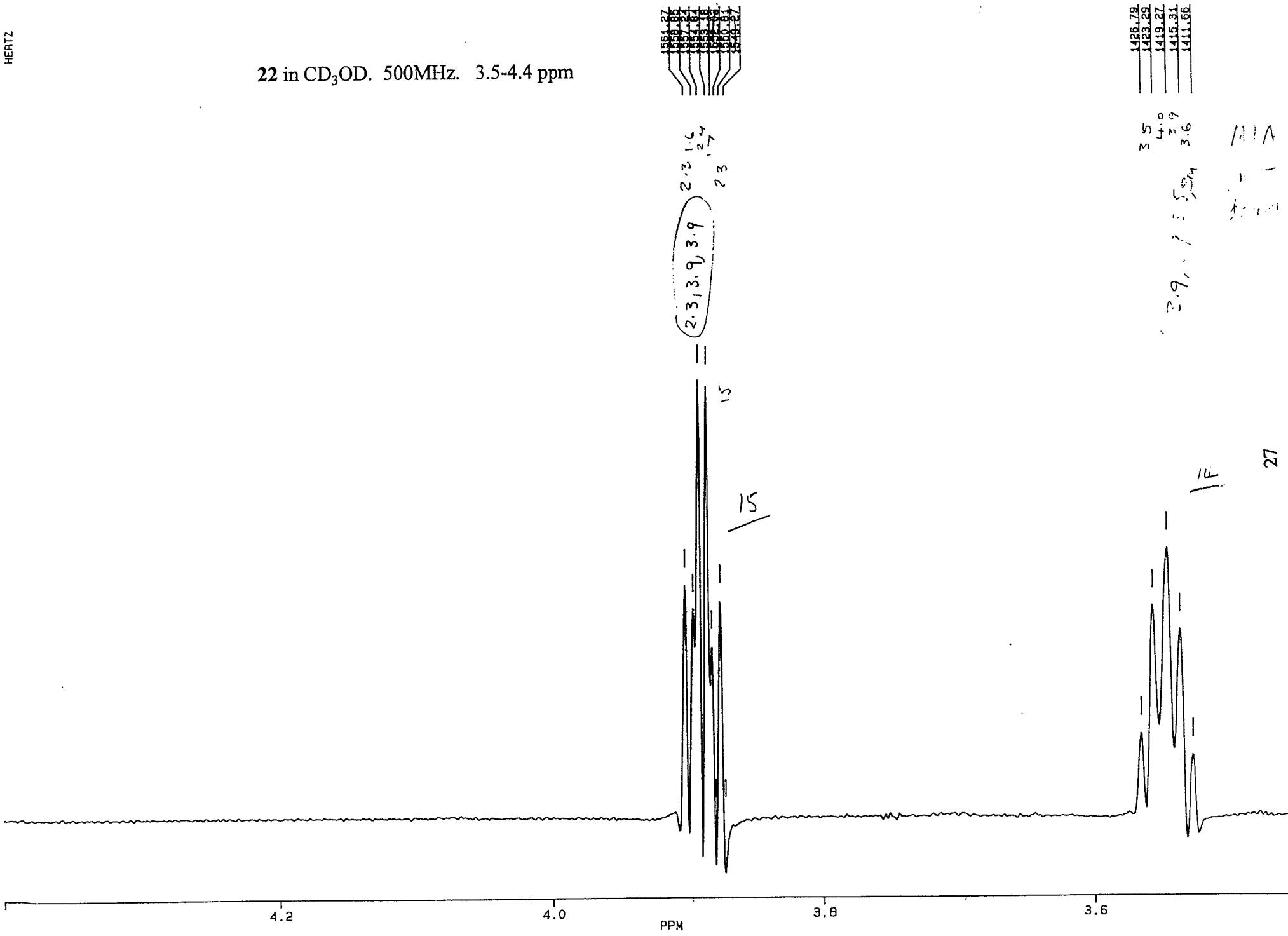
M11  
(R114)  
2.4-3.3 ppm

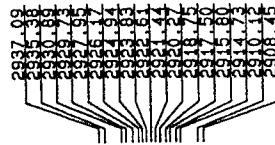
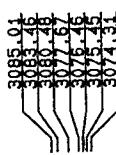
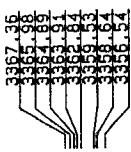
14.2, 6.6, 4.1

4.6  
4.1



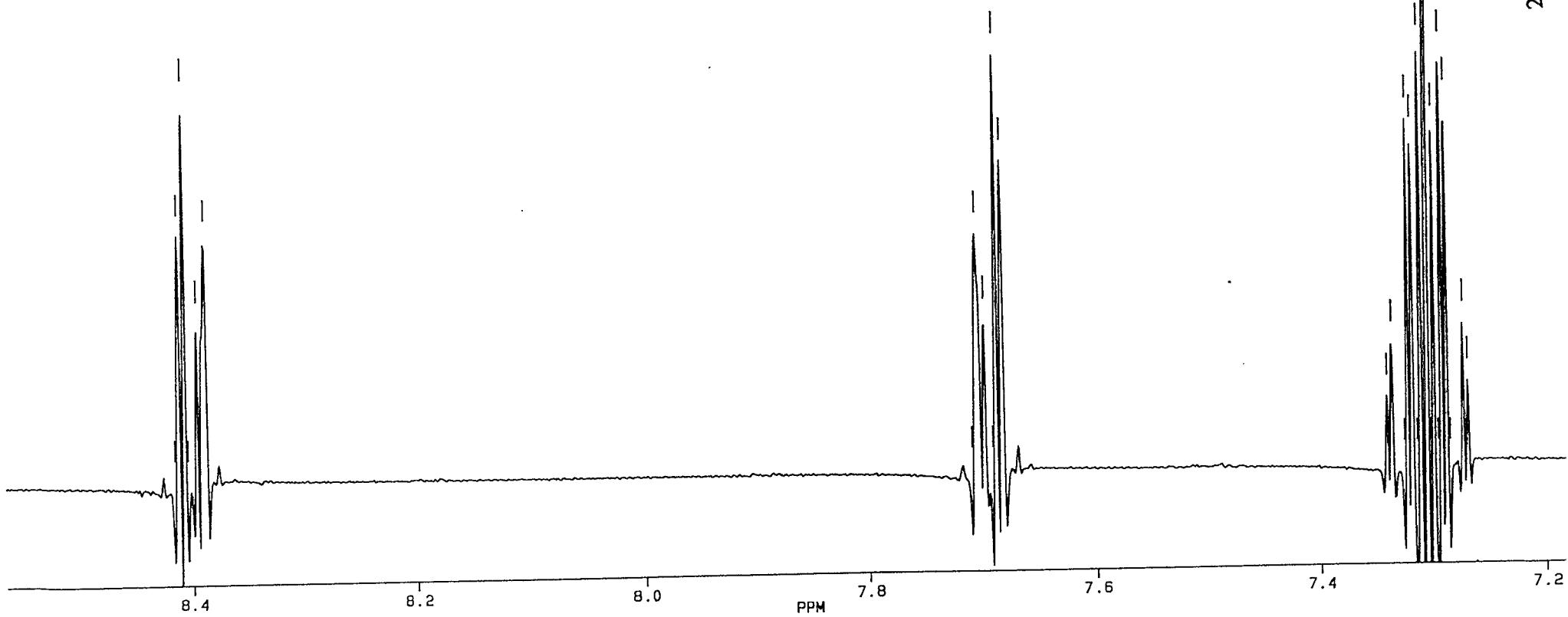
22 in CD<sub>3</sub>OD. 500MHz. 3.5-4.4 ppm



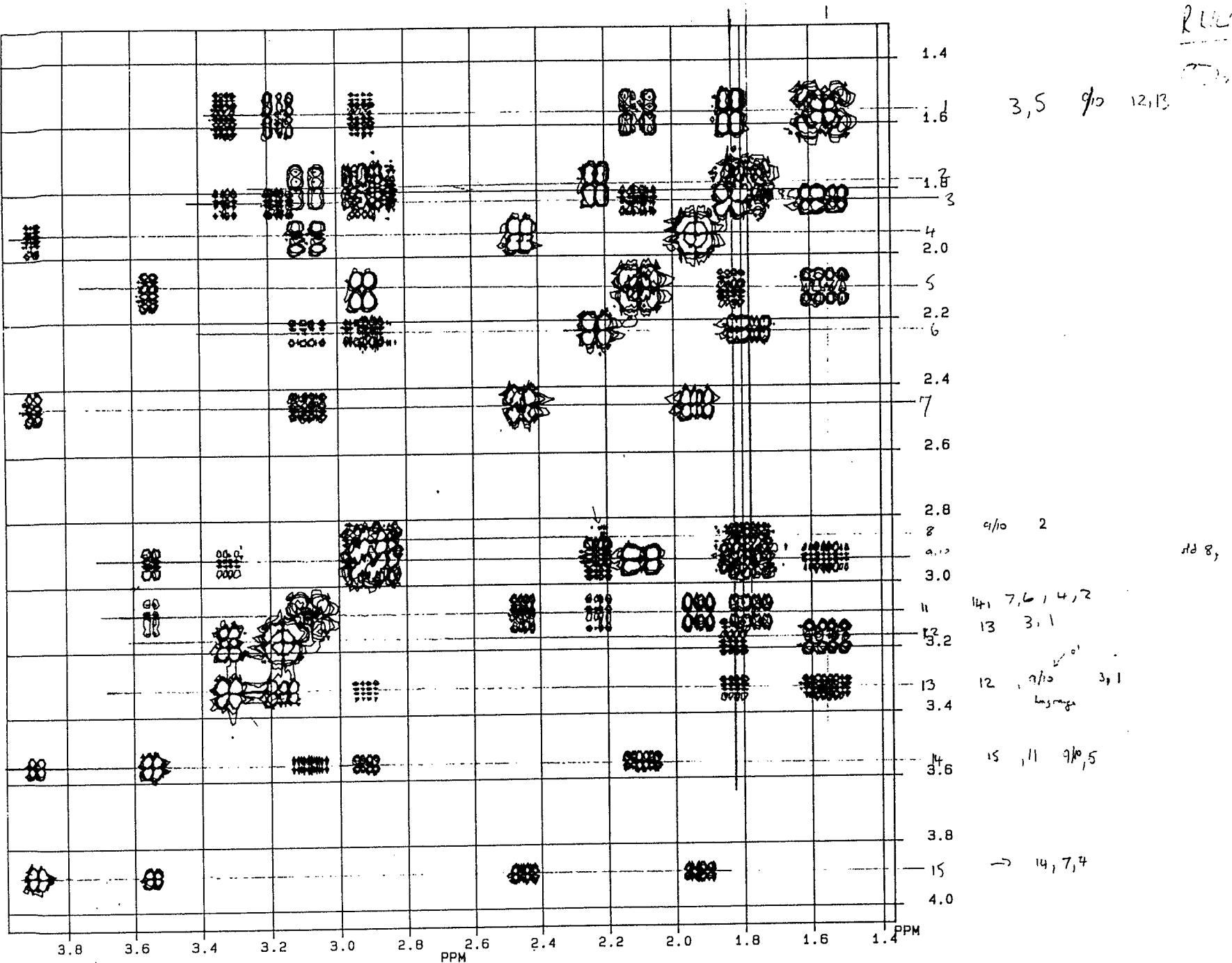


22 in CD<sub>3</sub>OD. 500MHz. 7.2-8.5 ppm.

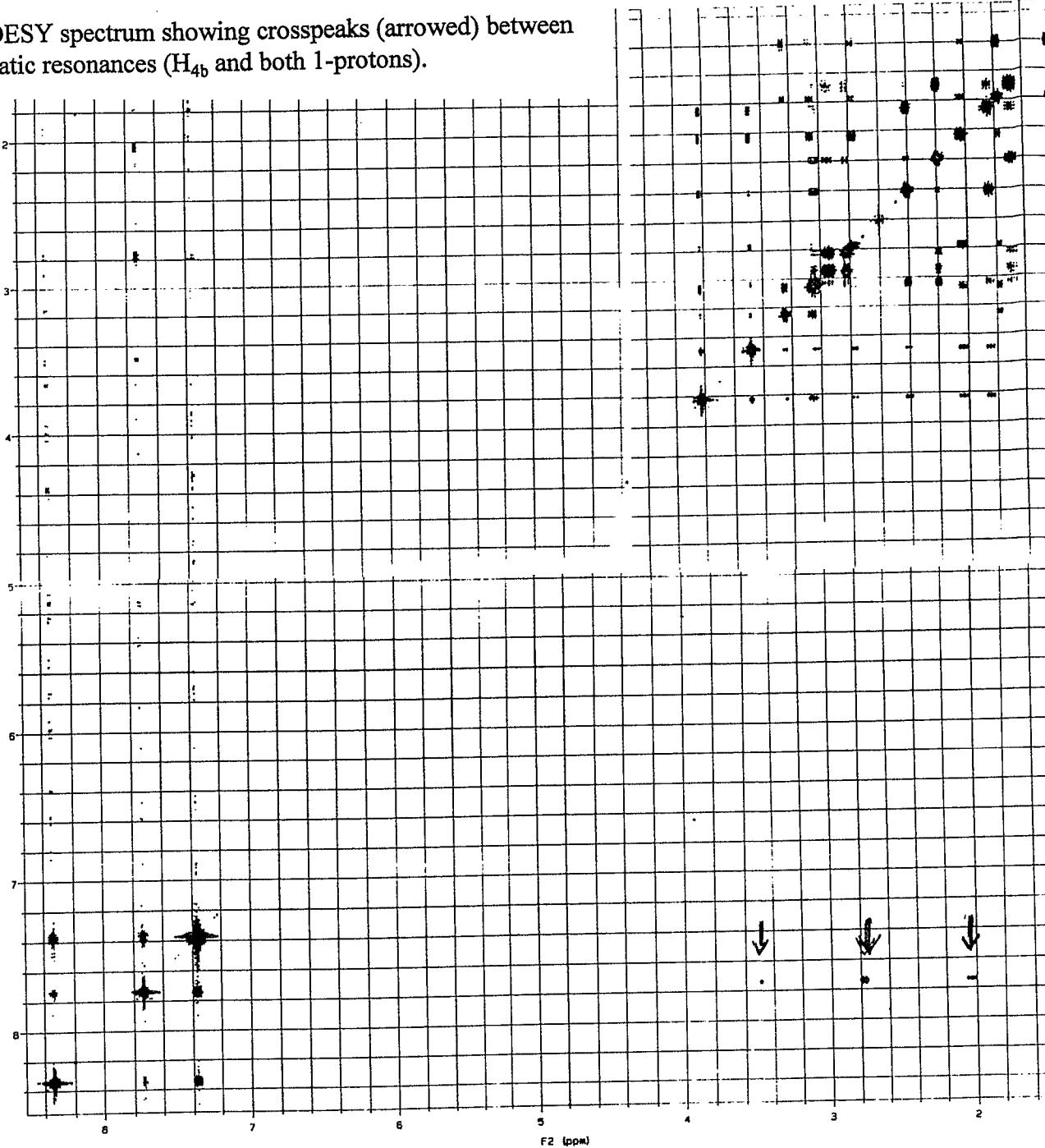
MIA  
(R<sub>114</sub>)  
DECONV



22 in CD<sub>3</sub>OD. 500MHz. COSY spectrum. 1.4-4.0 ppm region.



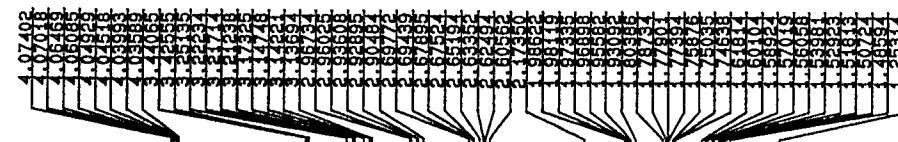
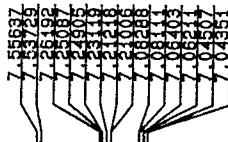
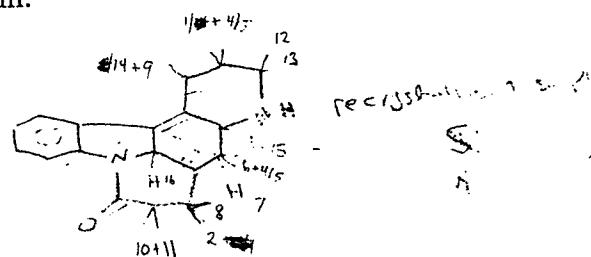
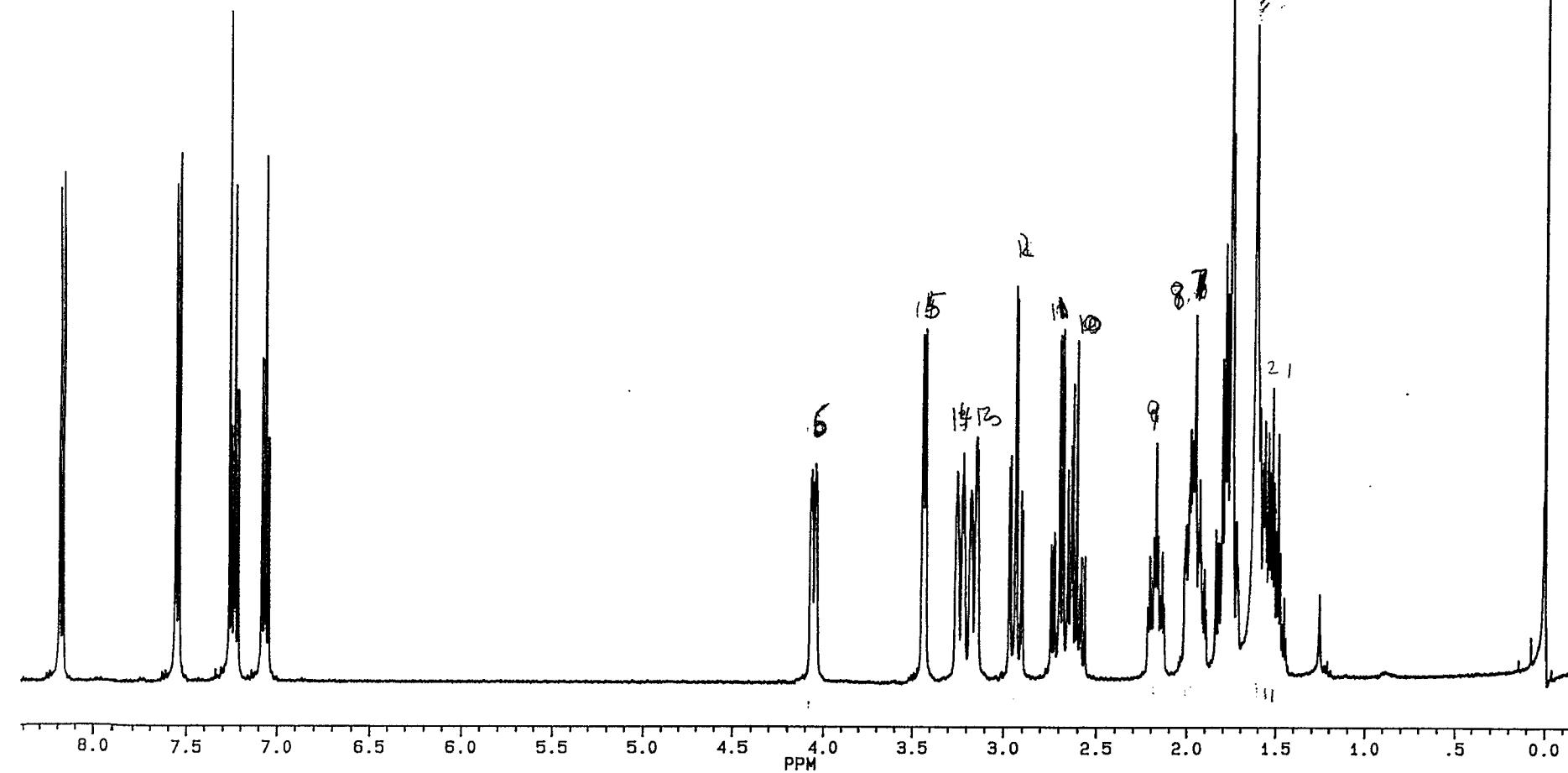
**22** in CD<sub>3</sub>OD. 500MHz. ROESY spectrum showing crosspeaks (arrowed) between the H<sub>13</sub> proton and three aliphatic resonances (H<sub>4b</sub> and both 1-protons).



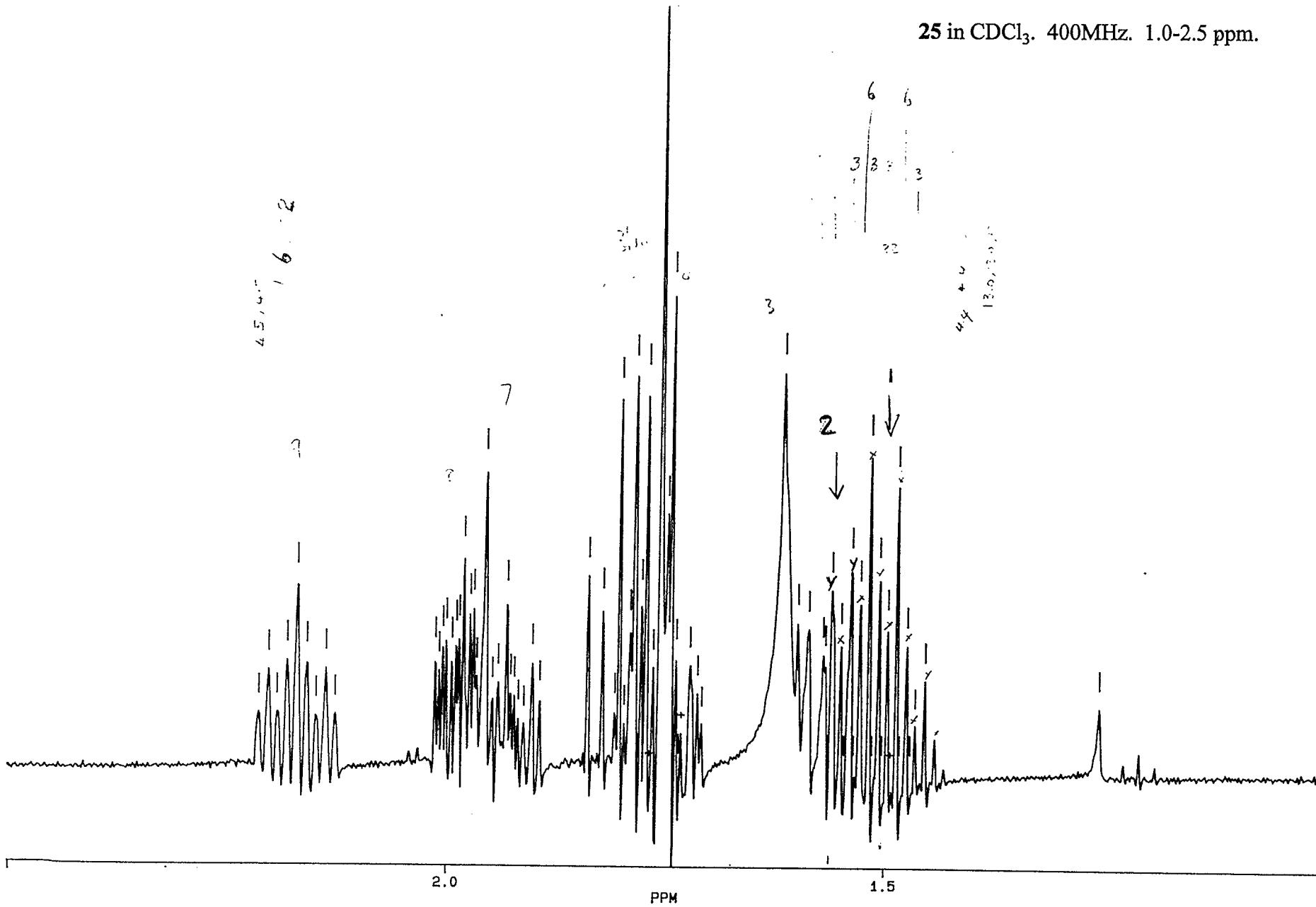
21

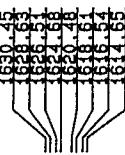
S99A

PPM

8.18638  
8.1663325 in  $\text{CDCl}_3$ . 400MHz. 0-8.4 ppm.~~BRUKER~~S99A  
DATE 17-2-94SF 400.134  
SI 32768

HERTZ





1377.34

1370.70

1305.25

1303.04

1305.25

1303.04

1305.25

1303.04

1305.25

1303.04

1305.25

1303.04

1305.25

1303.04

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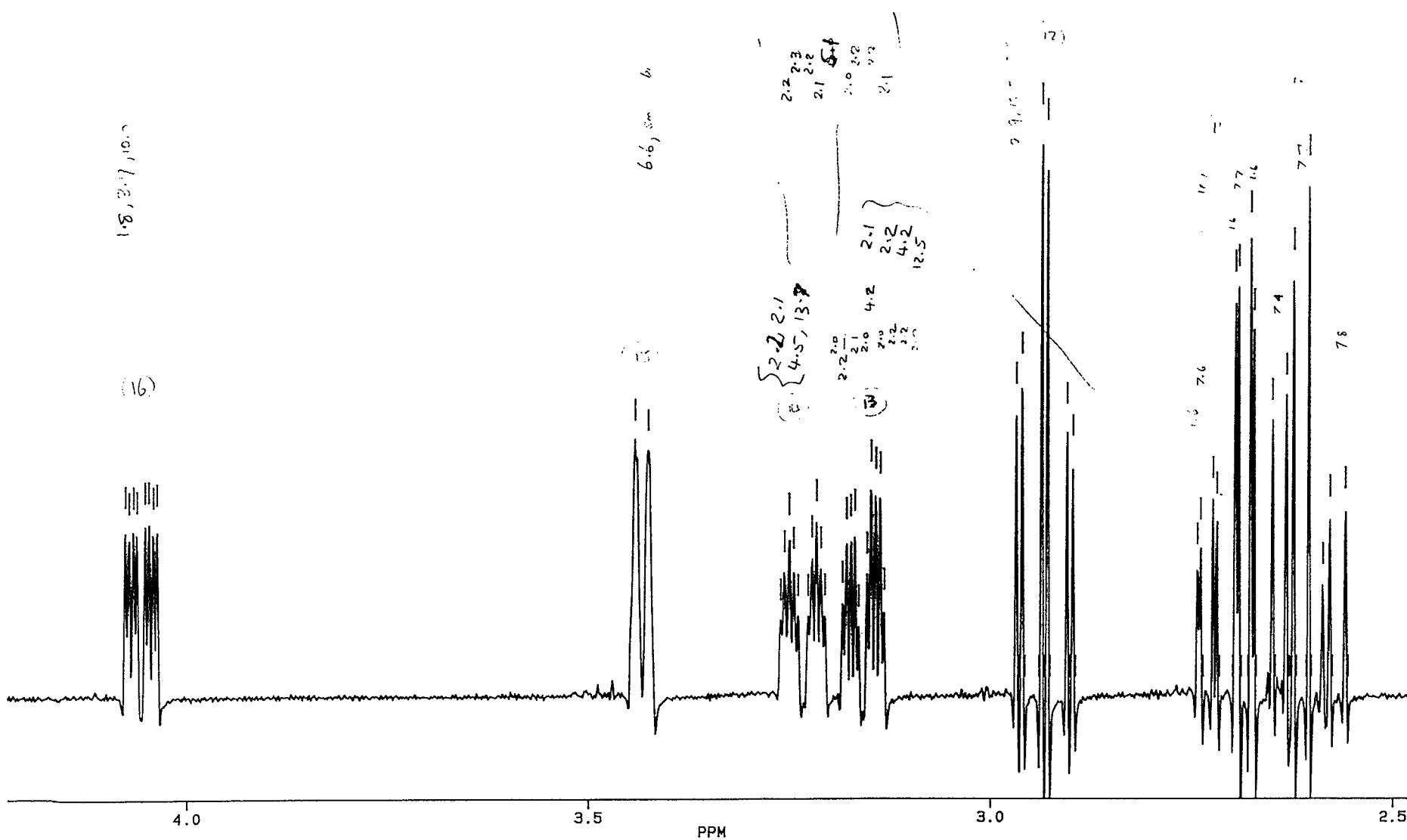
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1305.25

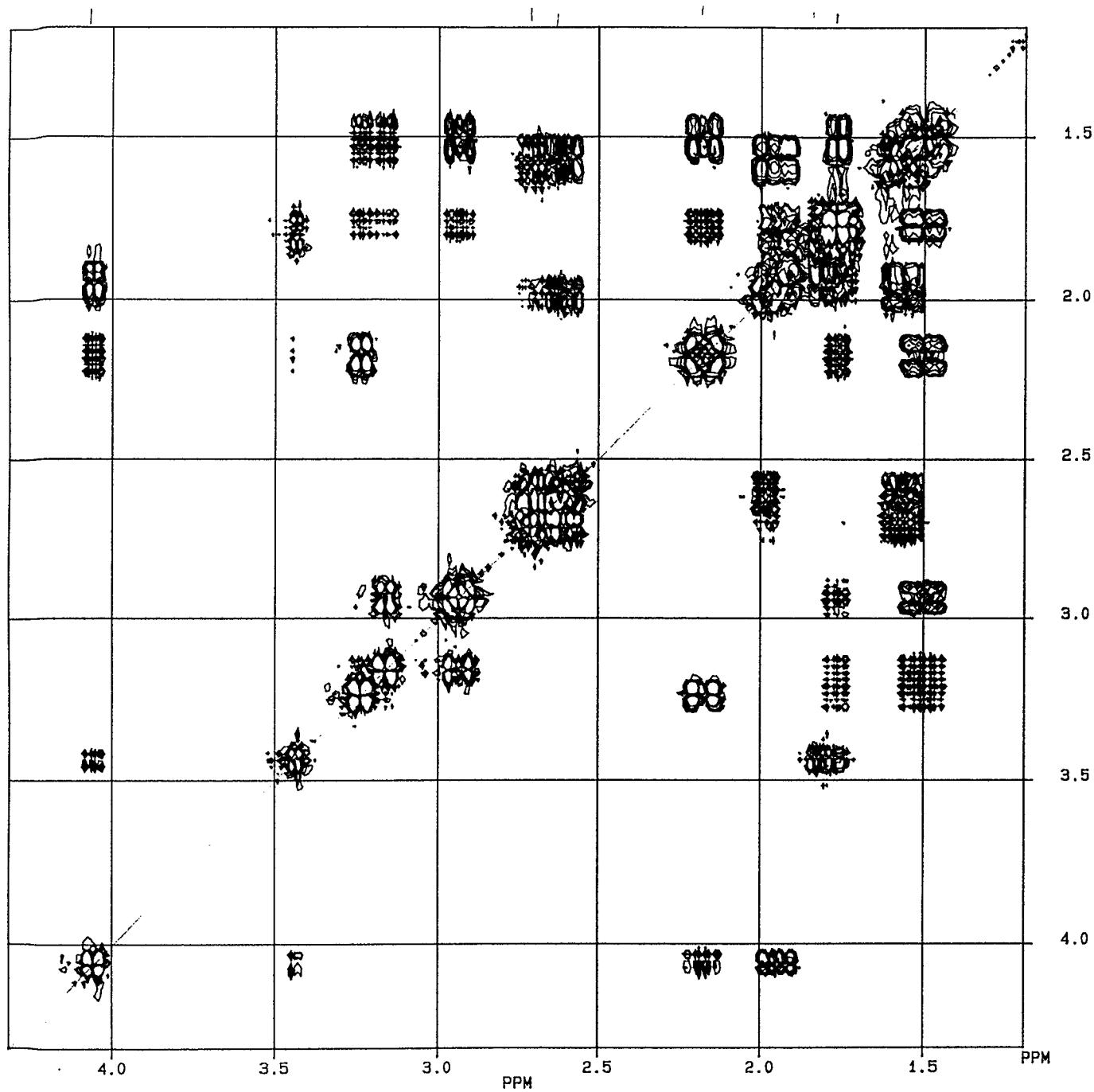
1303.04

1305.25

1303.04

25 in  $\text{CDCl}_3$ . 400MHz. 2.5-4.2 ppm.

25 in  $\text{CDCl}_3$ . 400MHz. COSY spectrum. Aliphatic region.



25 in  $\text{CDCl}_3$ . 400MHz. COSY spectrum. Aromatic region.

