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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.

Diffractometer = RIGAKU Electronic AFC-5

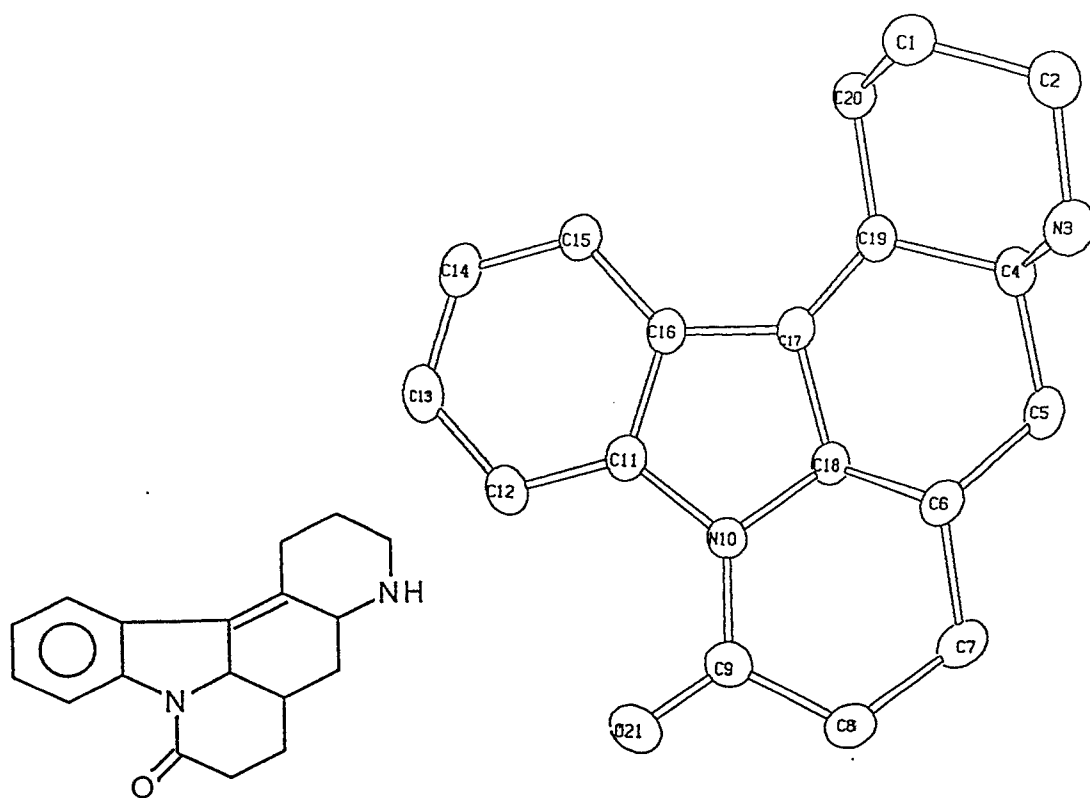
graphite-monochromated Cu-K α radiation ($\lambda = 1.5418\text{\AA}$)

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

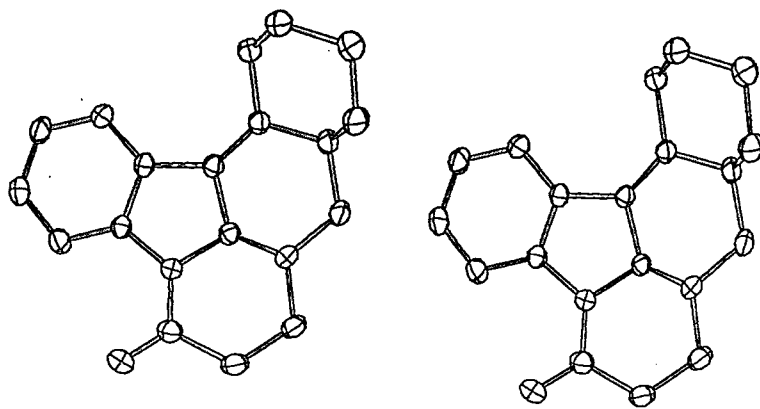
Data Collection : ω - 2θ scan technique
 $2^\circ < 2\theta < 130^\circ$

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)	5532(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	5523(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

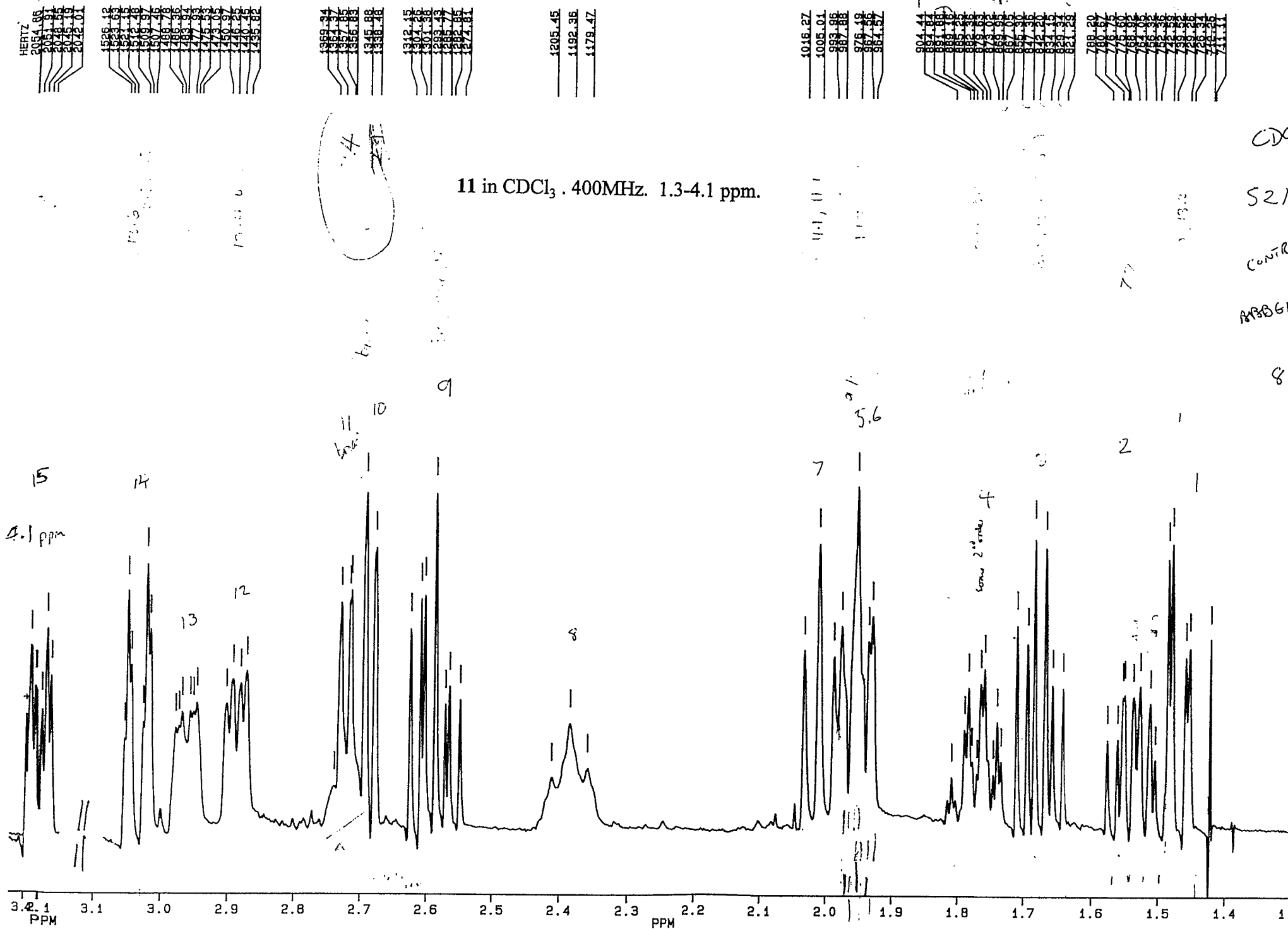
ATOM	X	Y	Z	B
H1A	-216(2)	-52(3)	739(1)	4.7(5)
H1B	-87(2)	-142(3)	737(2)	6.7(7)
H2A	-156(2)	-169(3)	873(2)	7.2(7)
H2B	-186(2)	30(3)	877(2)	6.8(7)
HN3	11(5)	-46(6)	943(3)	19.0(18)
H4	-8(2)	226(2)	885(1)	3.6(5)
H5A	173(2)	106(3)	968(1)	3.9(5)
H5B	187(2)	274(3)	920(1)	4.5(5)
H6	264(2)	-19(2)	850(1)	2.3(4)
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)	843(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)

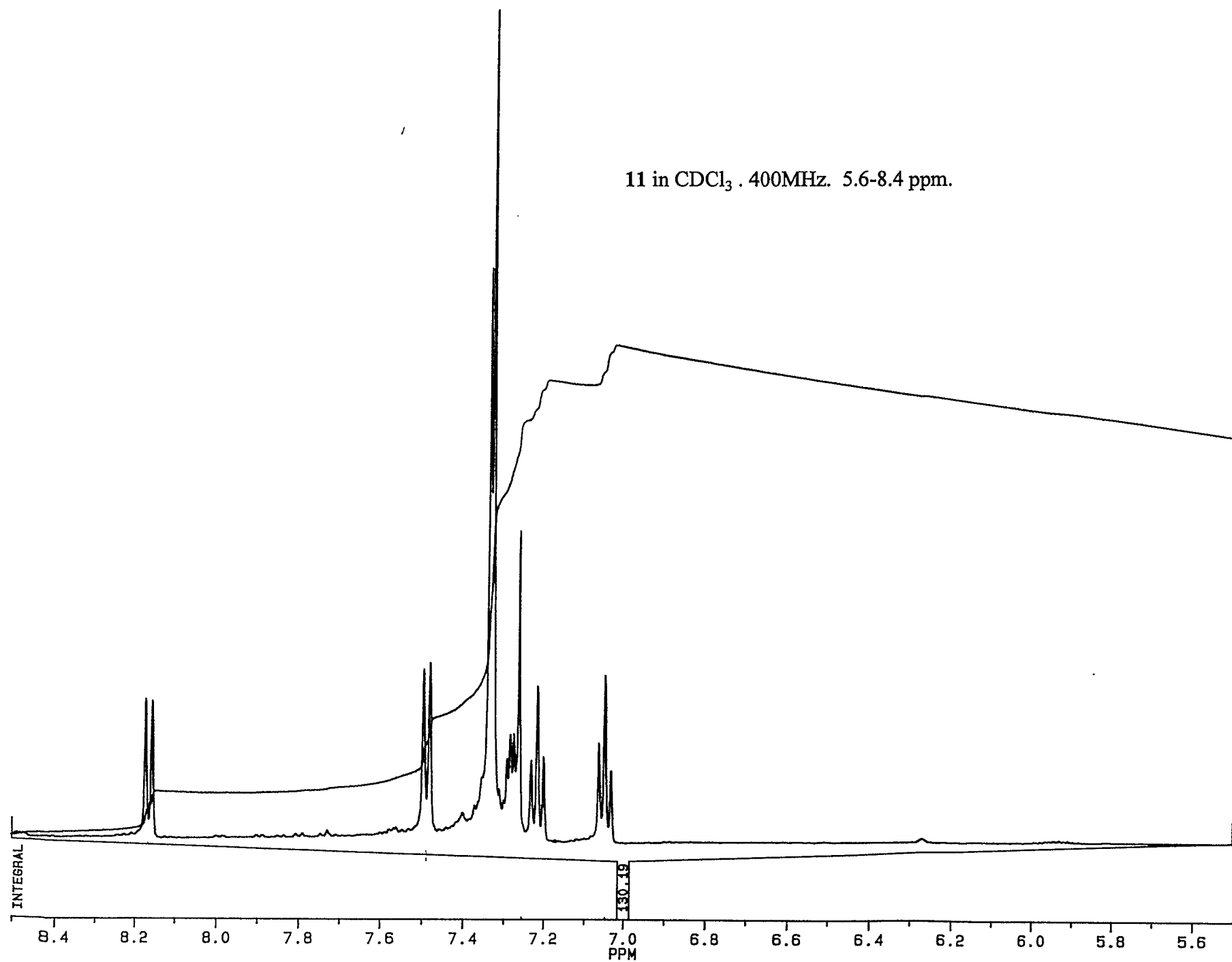
Table 2 . Bond lengths (Å) and bond angles (°) of PG-394,
with estimated standard deviations in parentheses

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)				

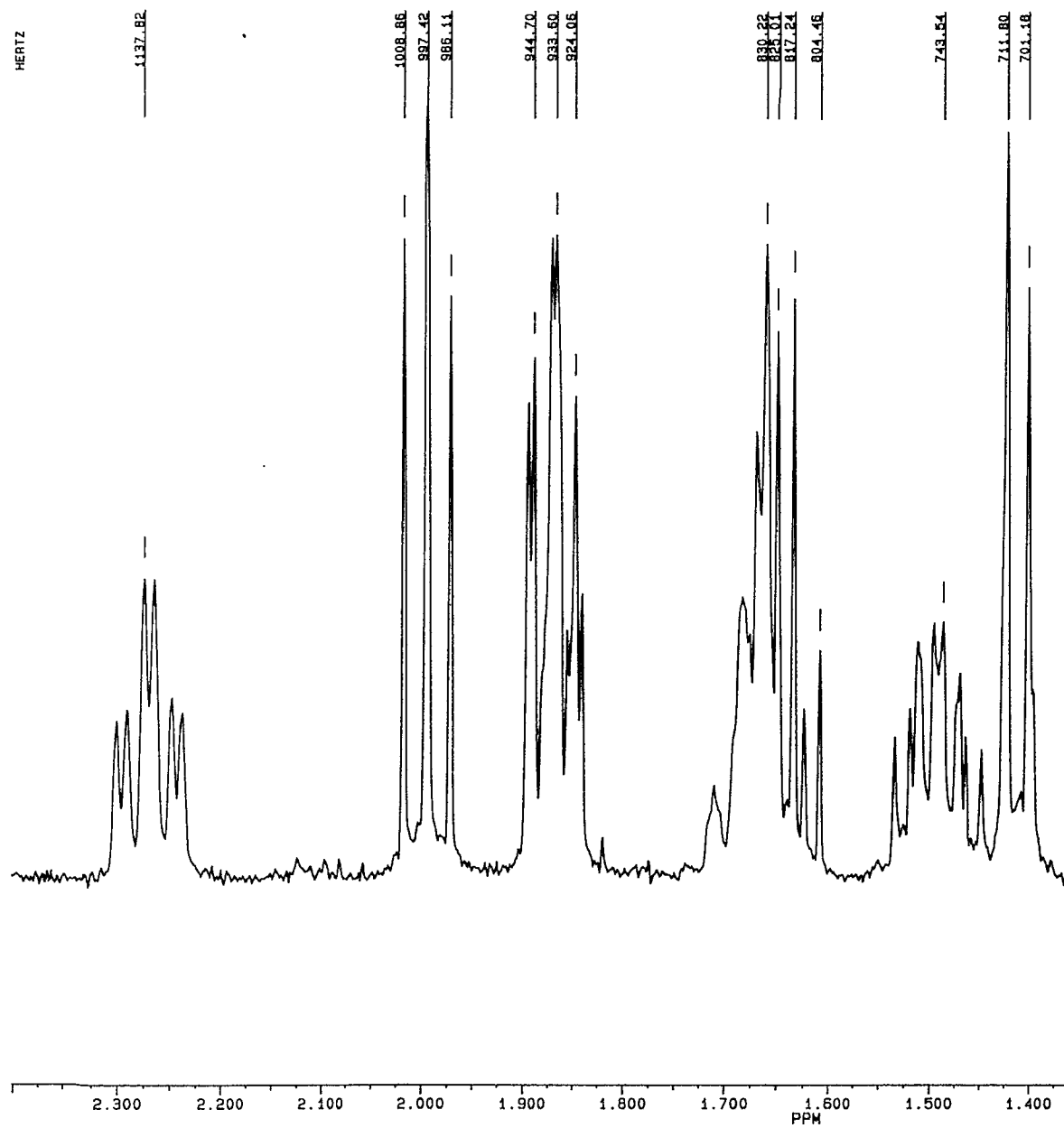
ATOM			ANGLE	ATOM			ANGLE
C2	-C1	-C20	111.1(2)	N10	-C11	-C16	109.3(2)
C1	-C2	-N3	111.6(2)	C12	-C11	-C16	122.5(2)
C2	-N3	-C4	112.5(2)	C11	-C12	-C13	117.8(2)
N3	-C4	-C5	109.2(2)	C12	-C13	-C14	120.8(2)
N3	-C4	-C19	108.5(2)	C13	-C14	-C15	121.2(2)
C5	-C4	-C19	114.9(2)	C14	-C15	-C16	119.5(2)
C4	-C5	-C6	112.7(2)	C11	-C16	-C15	118.3(2)
C5	-C6	-C7	116.2(2)	C11	-C16	-C17	108.7(2)
C5	-C6	-C18	106.3(2)	C15	-C16	-C17	133.0(2)
C7	-C6	-C18	107.2(2)	C16	-C17	-C18	104.9(2)
C6	-C7	-C8	110.2(2)	C16	-C17	-C19	134.2(2)
C7	-C8	-C9	118.4(2)	C18	-C17	-C19	120.6(2)
C8	-C9	-N10	116.8(2)	C6	-C18	-N10	113.5(2)
C8	-C9	-O21	120.4(2)	C6	-C18	-C17	113.3(2)
N10	-C9	-O21	122.7(2)	N10	-C18	-C17	104.4(2)
C9	-N10	-C11	127.6(2)	C4	-C19	-C17	121.3(2)
C9	-N10	-C18	123.5(2)	C4	-C19	-C20	112.3(2)
C11	-N10	-C18	108.7(1)	C17	-C19	-C20	126.3(2)
N10	-C11	-C12	128.2(2)	C1	-C20	-C19	110.5(2)

11 in CDCl₃ . 400MHz. 1.3-4.1 ppm.

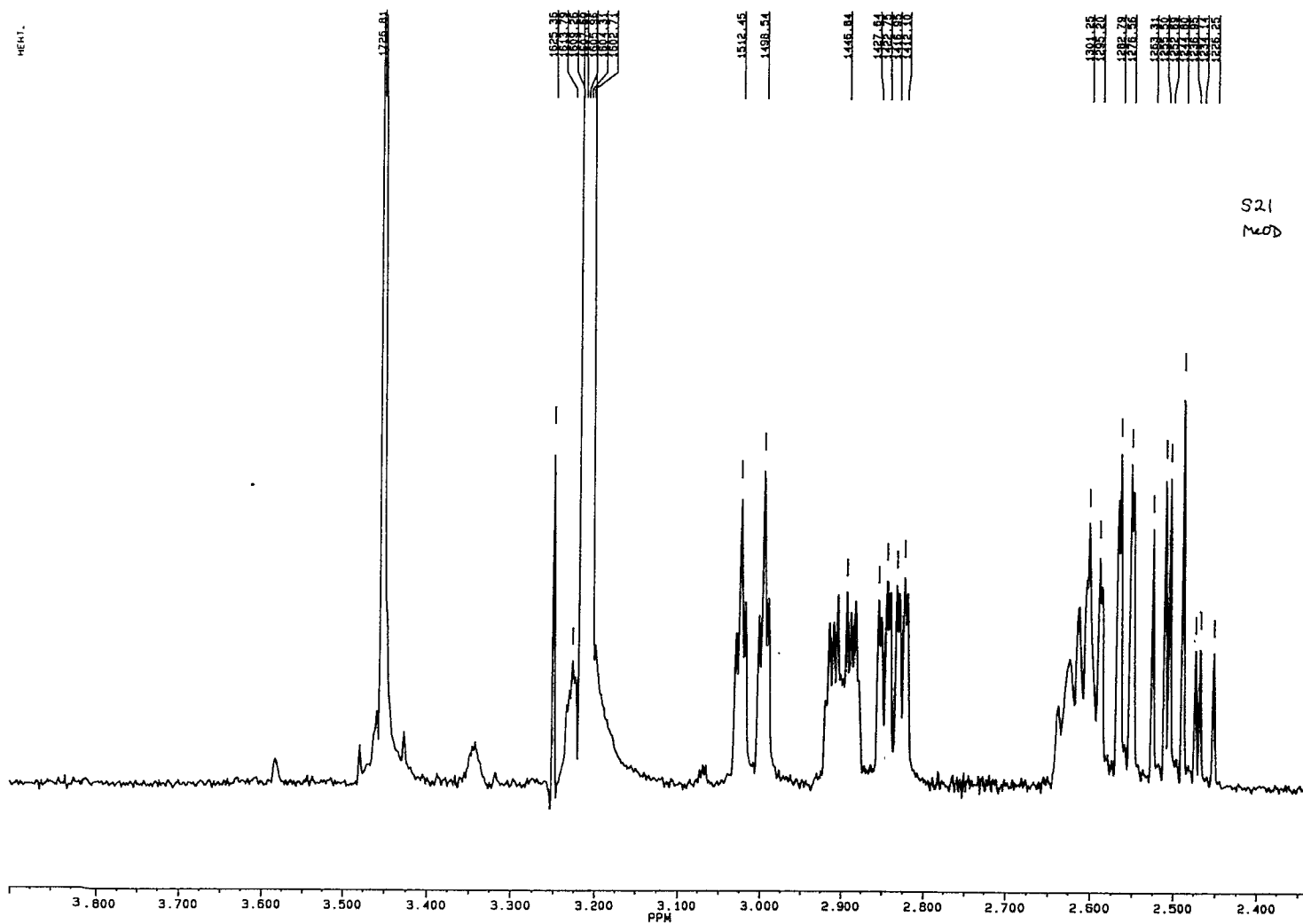




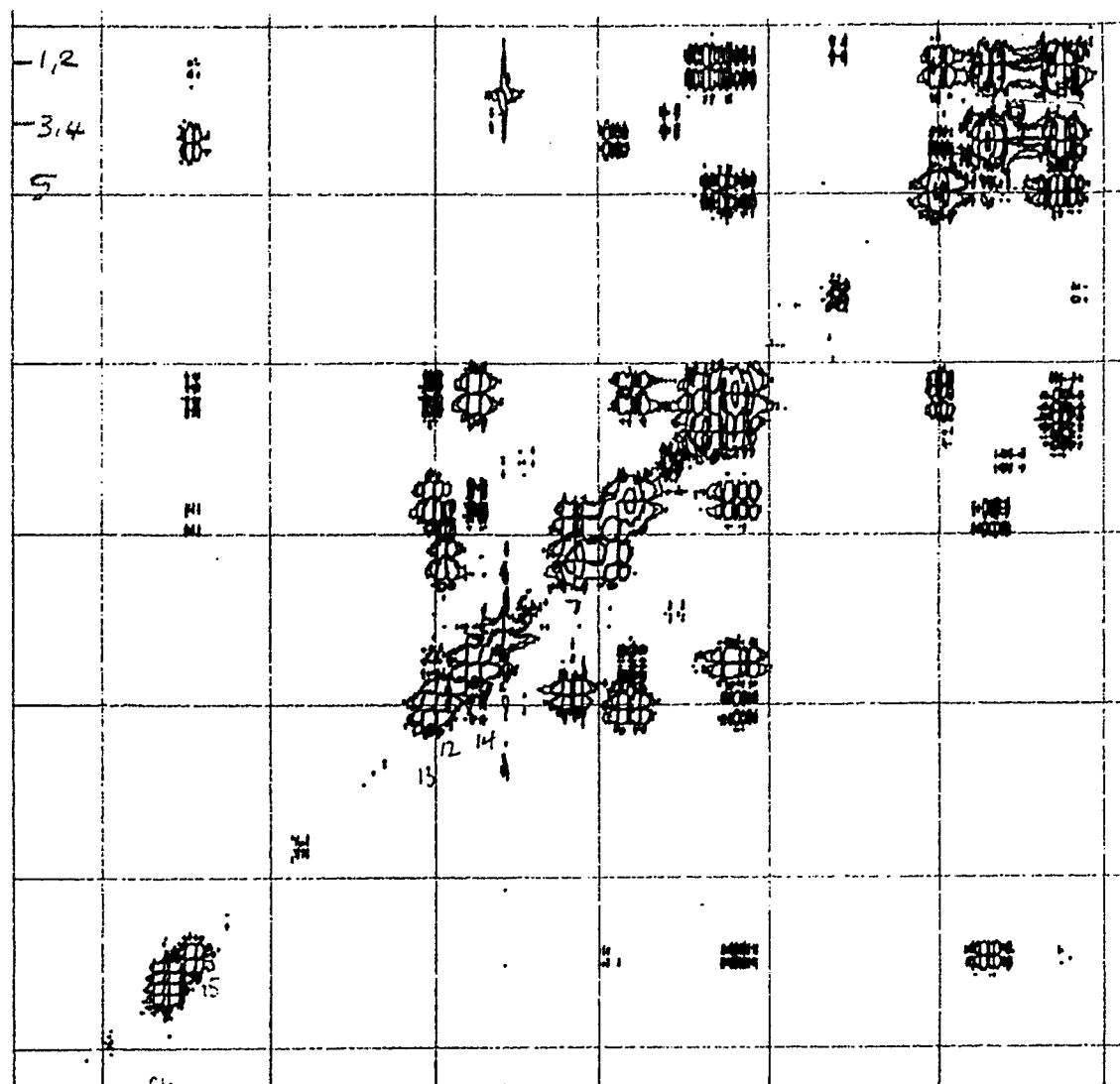
11 in CD₃OD. 400MHz. 1.4-2.4 ppm.



11 in CD₃OD. 400MHz. 2.4-3.6 ppm.

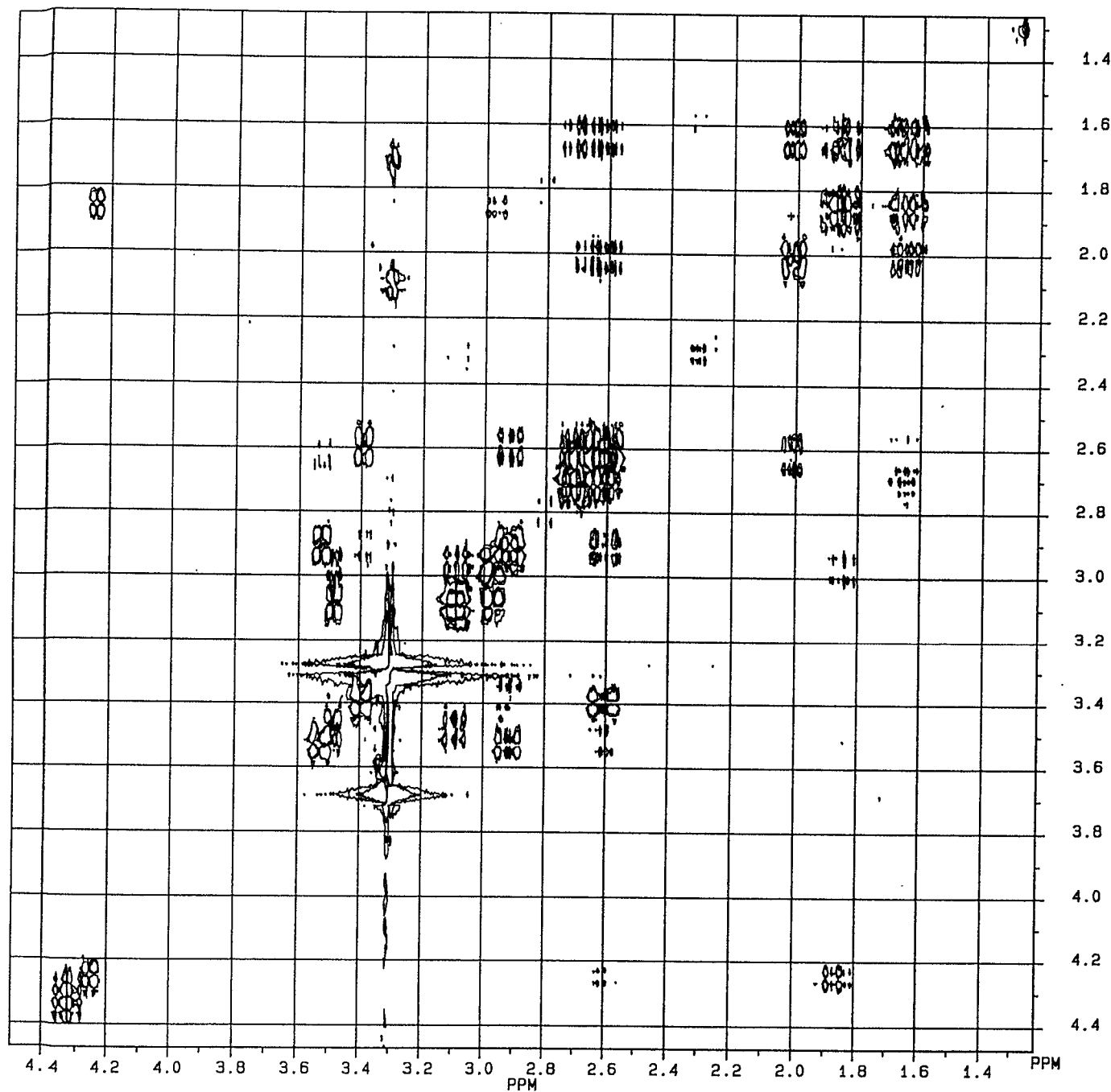


11 in CD₃OD. 400MHz COSY spectrum. 1.4-4.4 ppm



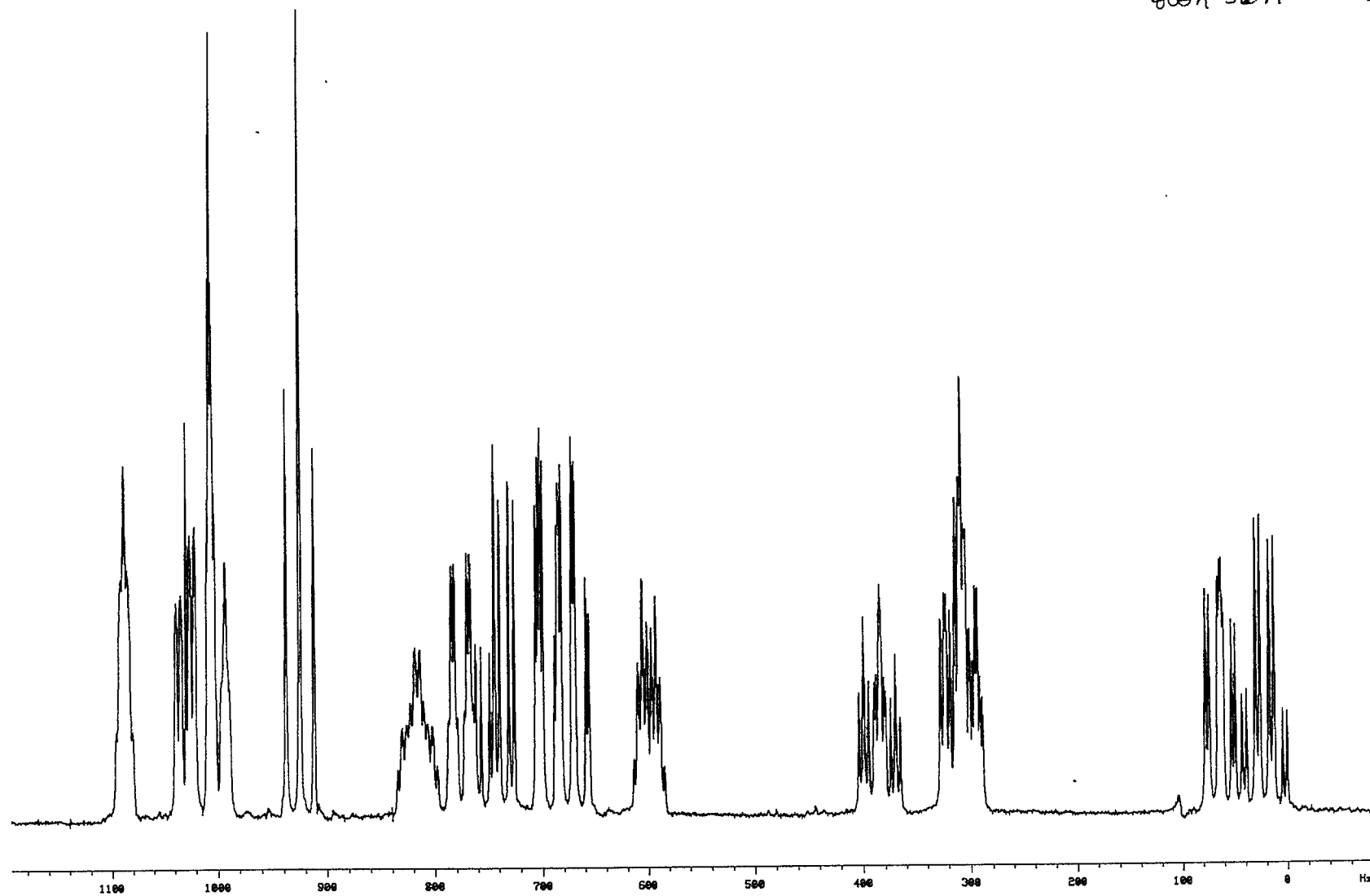
11 in CD₃OD + DCl. 400MHz COSY spectrum. 1.4-4.4 ppm

BB191 S21+DCL COSYNMR 400MHZ 298K

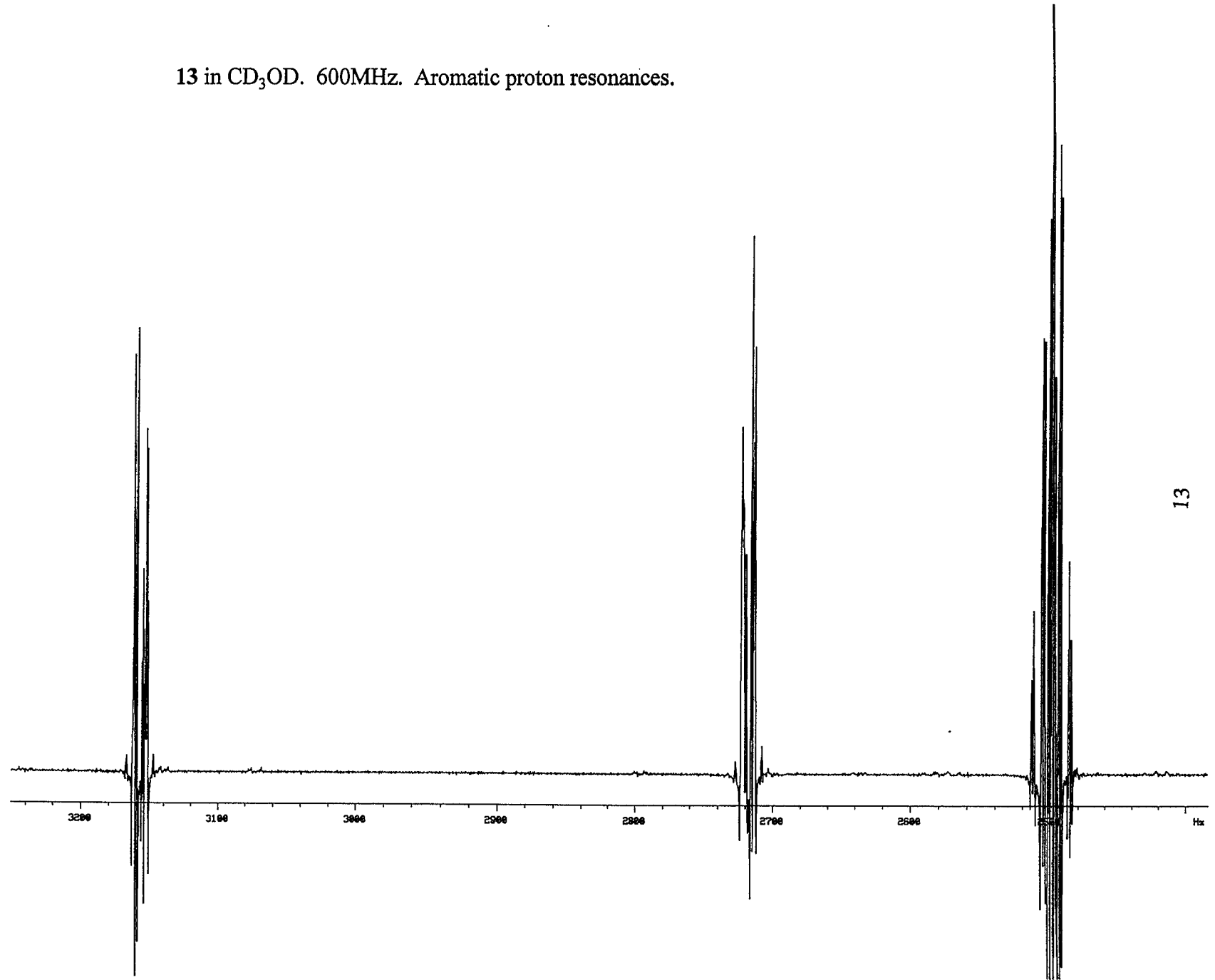


WD=Q
SSB = 6.6

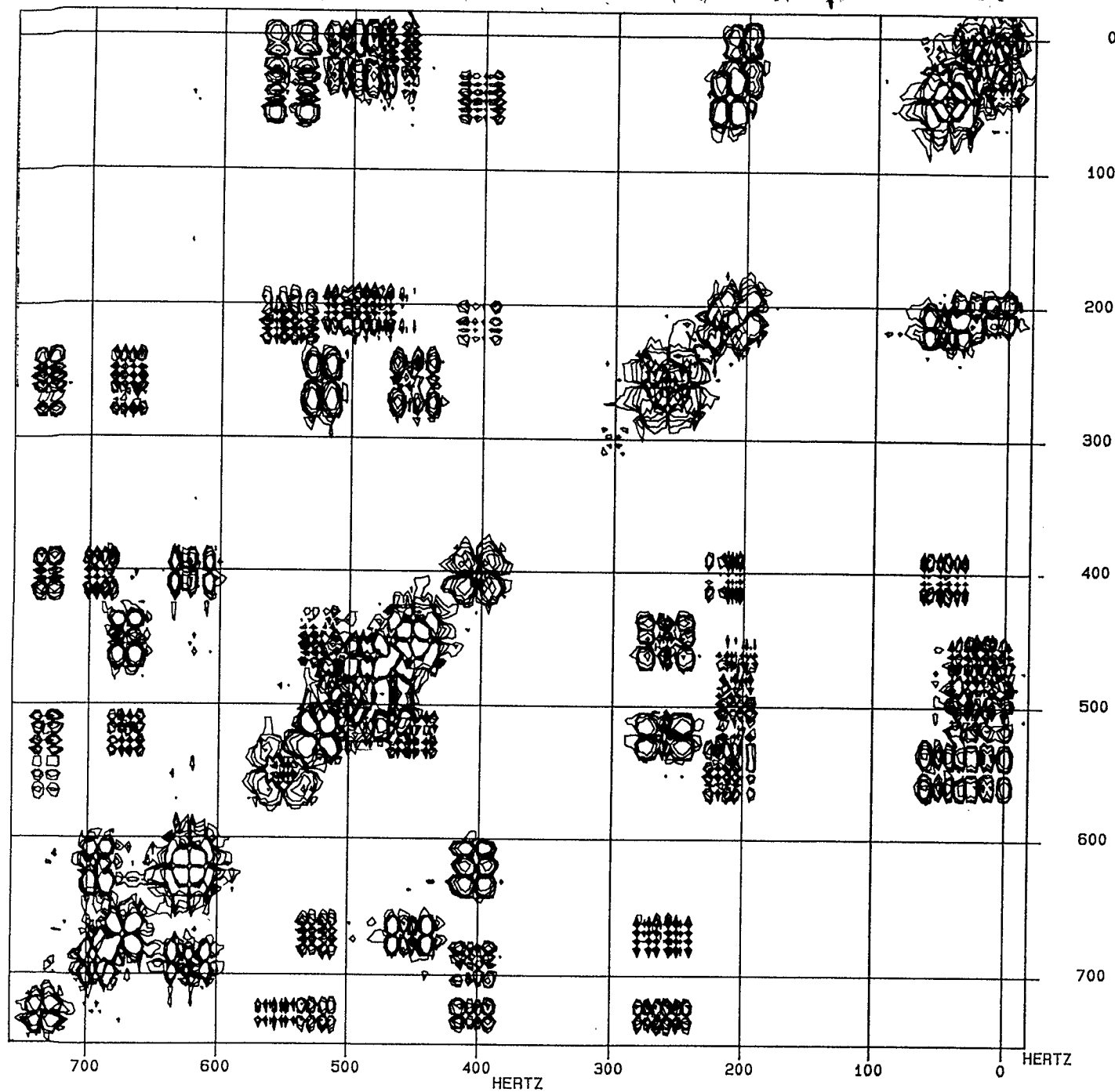
PC0 -23.6 , PC1 = 17.2 XFB
PC0 -90 , 0 XFIP



13 in CD₃OD. 600MHz. Aromatic proton resonances.

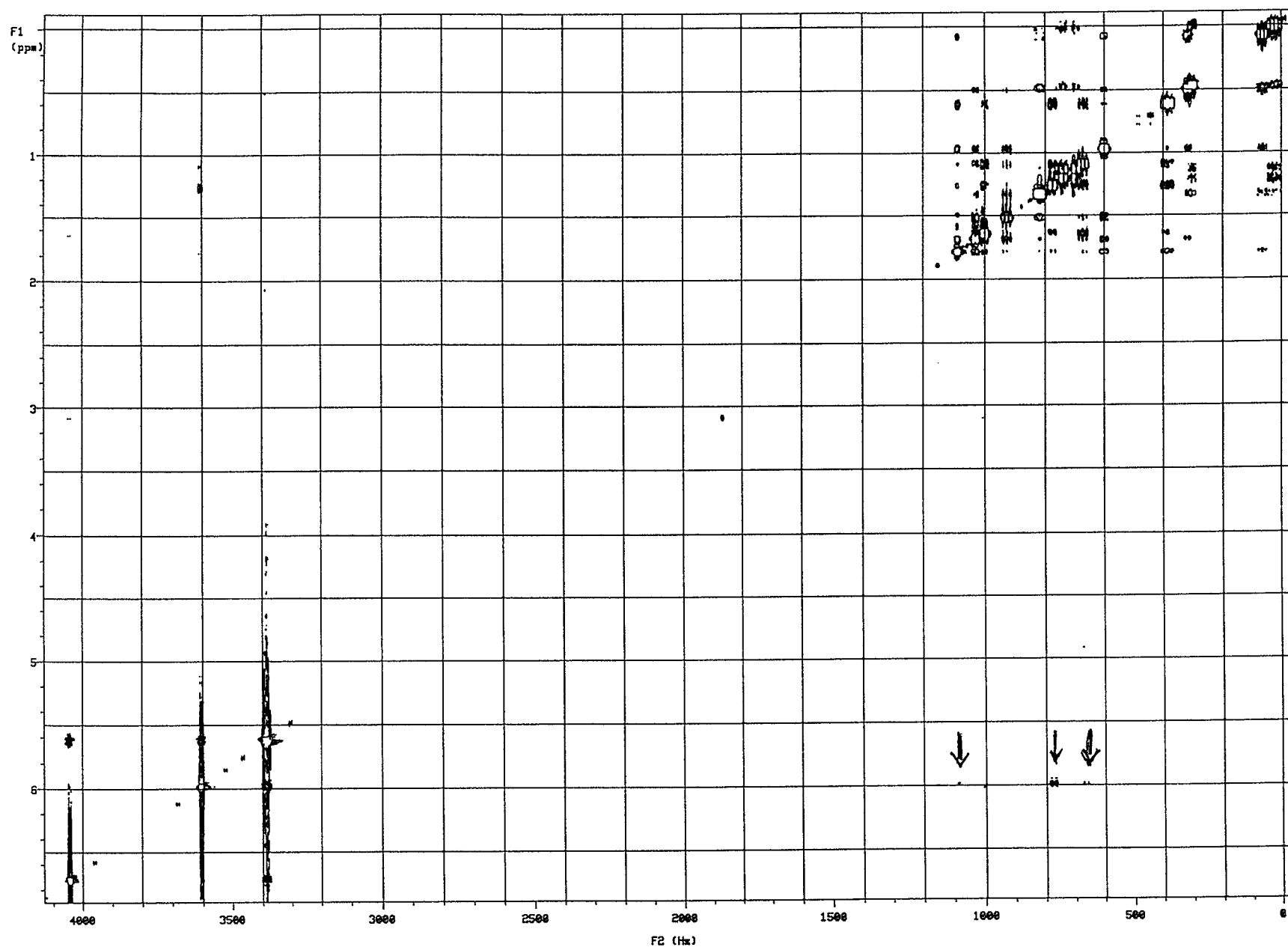


^{13}C in CD_3OD . 400MHz. COSY spectrum. Aliphatic resonances.

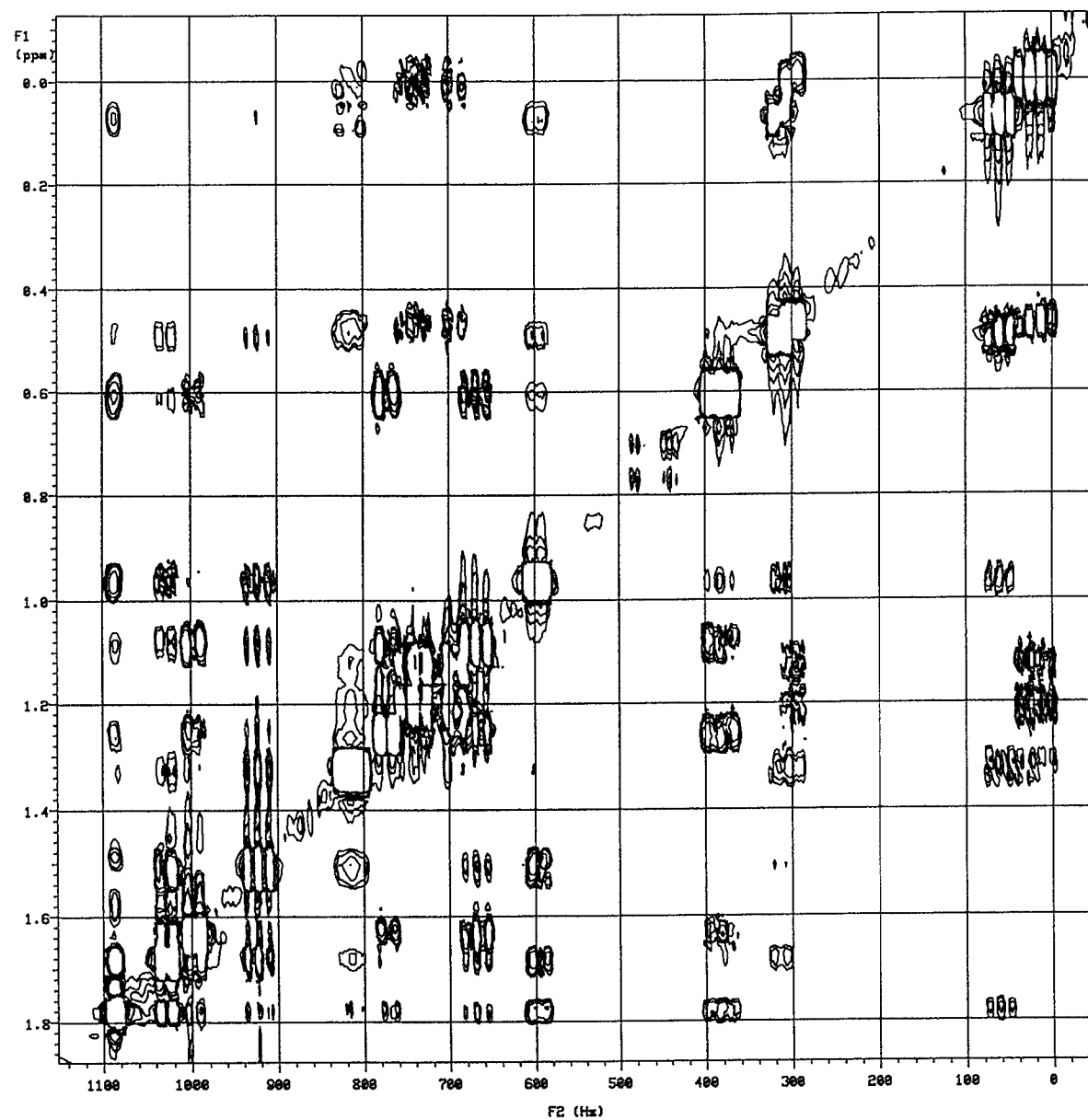


13 in CD₃OD. 600MHz. ROESY spectrum showing crosspeaks (arrowed) between the H₁₃ proton and three aliphatic resonances (H_{4b} and both 1-protons).

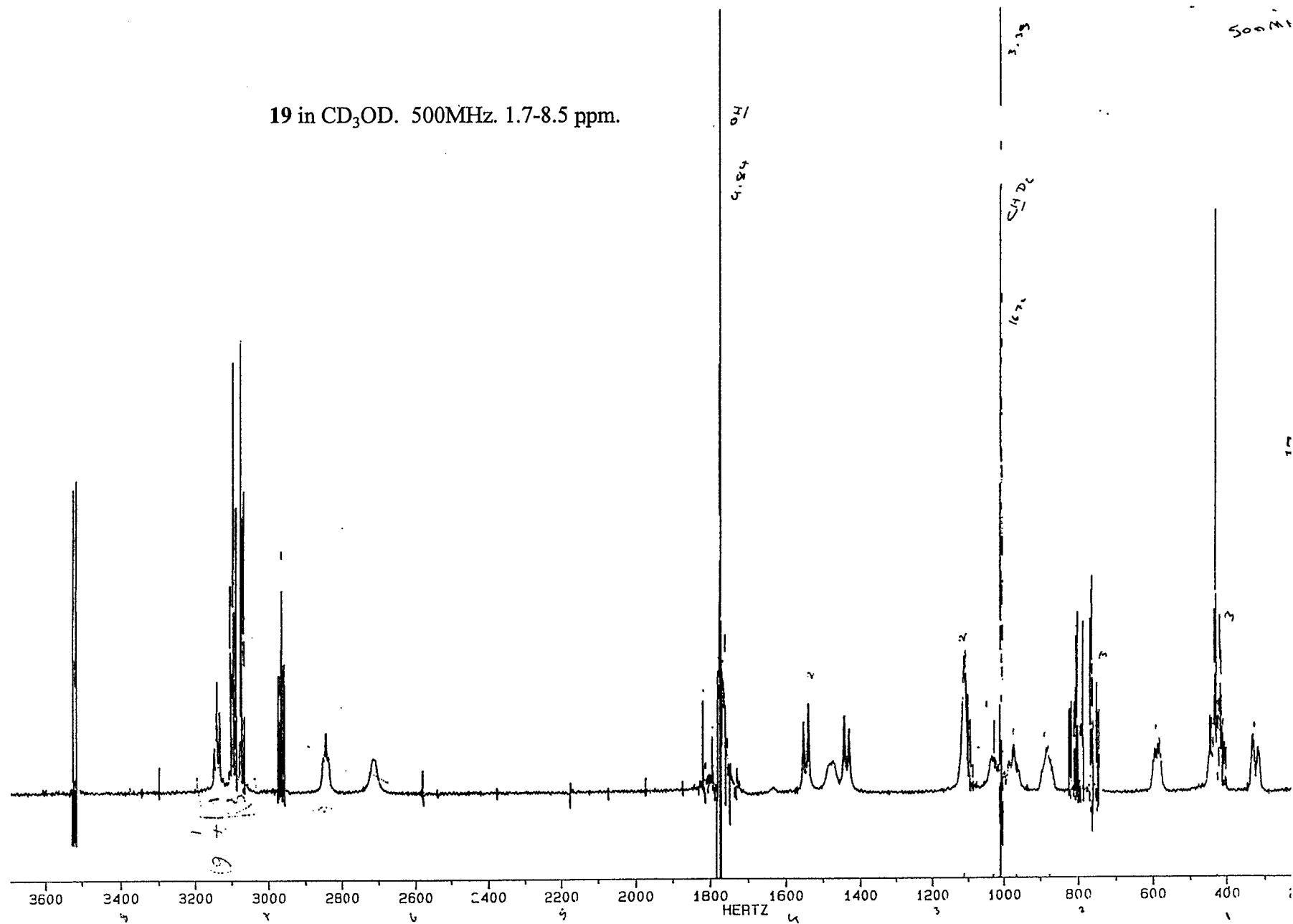
547



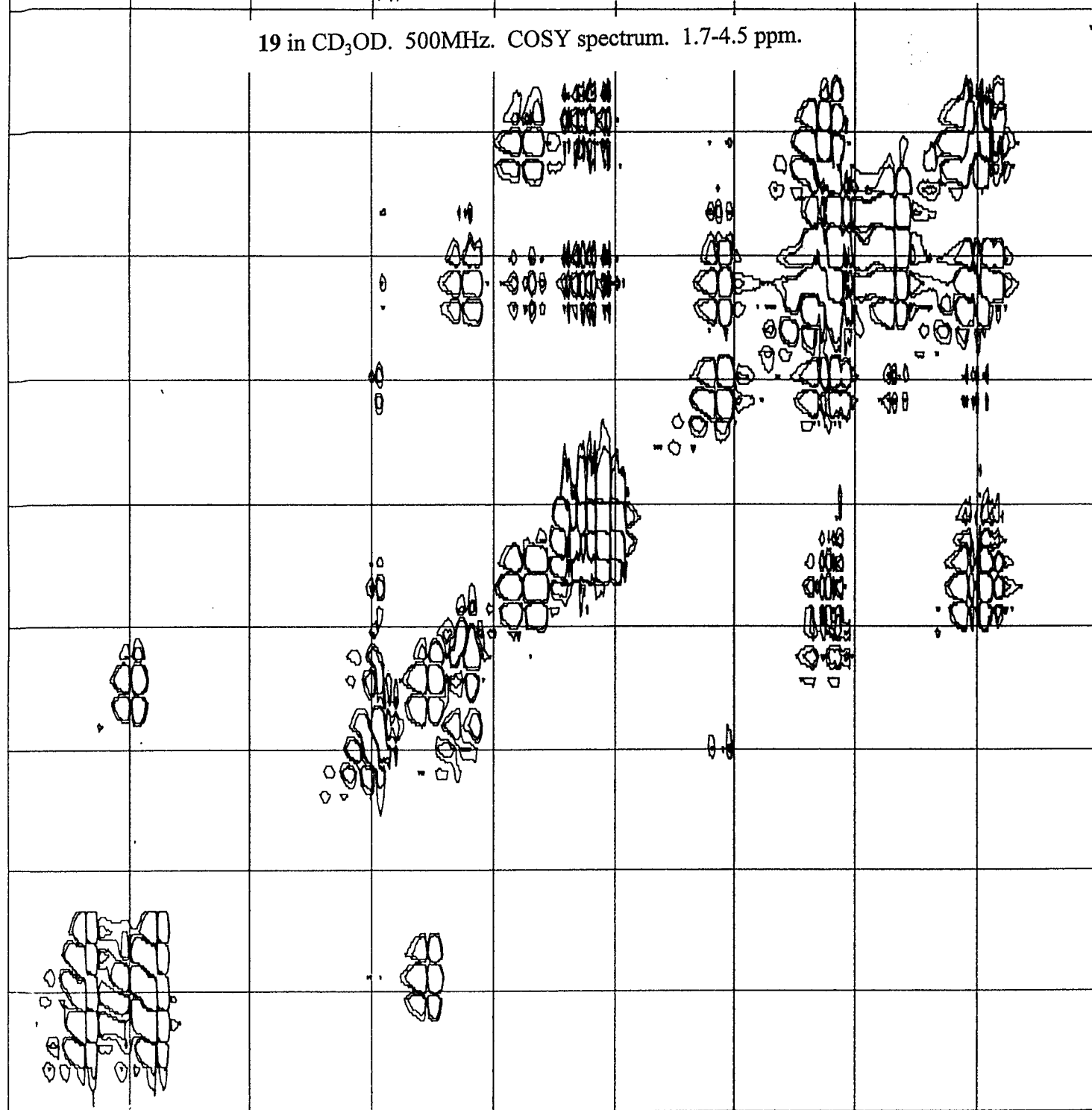
13 in CD₃OD. 600MHz. Expanded region of the ROESY spectrum showing the crosspeaks between the aliphatic proton resonances.



19 in CD₃OD. 500MHz. 1.7-8.5 ppm.



19 in CD₃OD. 500MHz. COSY spectrum. 1.7-4.5 ppm.



20 in CD₃OD. 500MHz. 1.4-8.6 ppm.

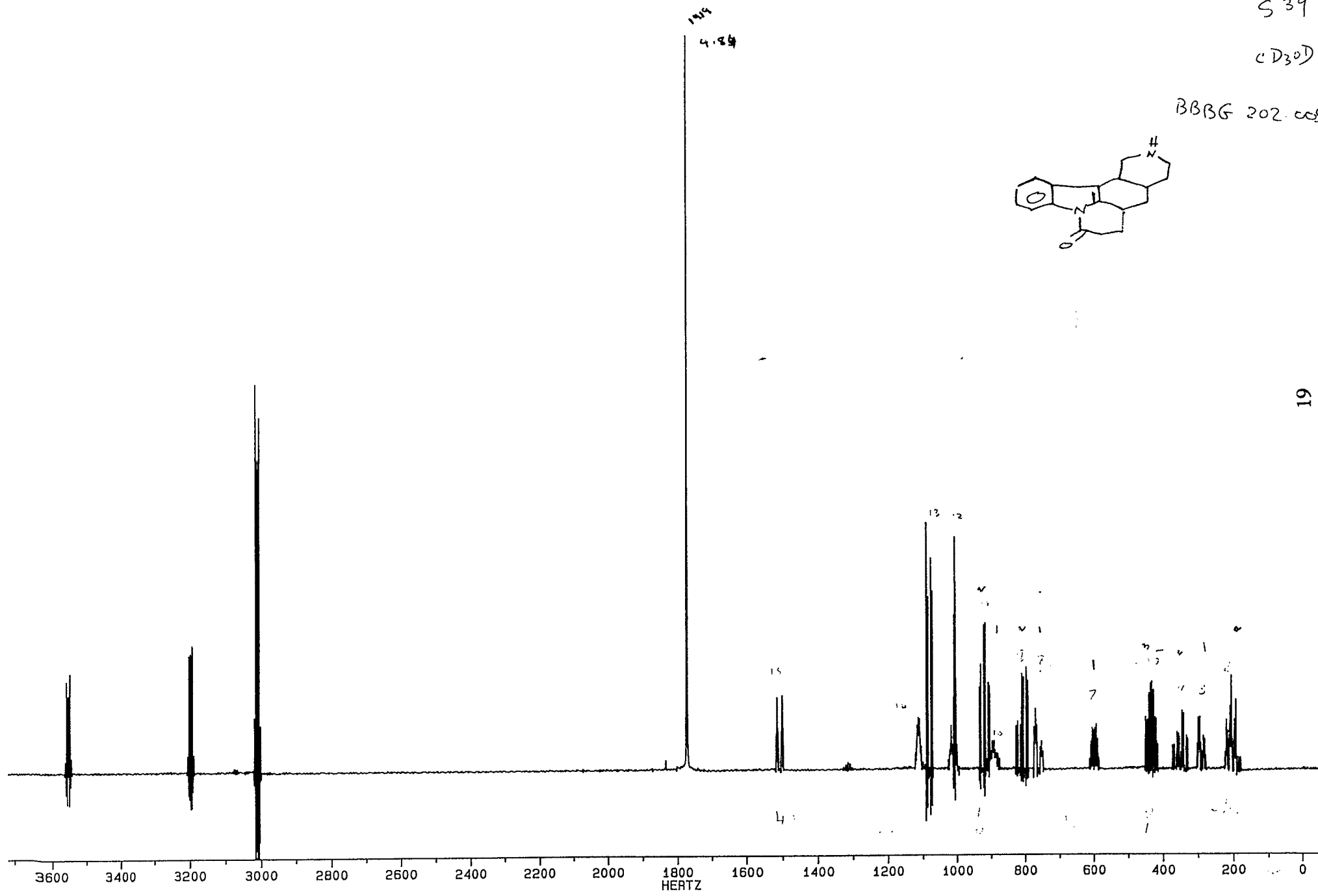
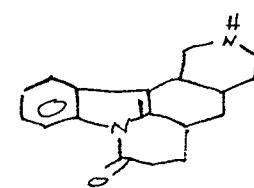
300 K

500MHz

S 39

CD₃OD

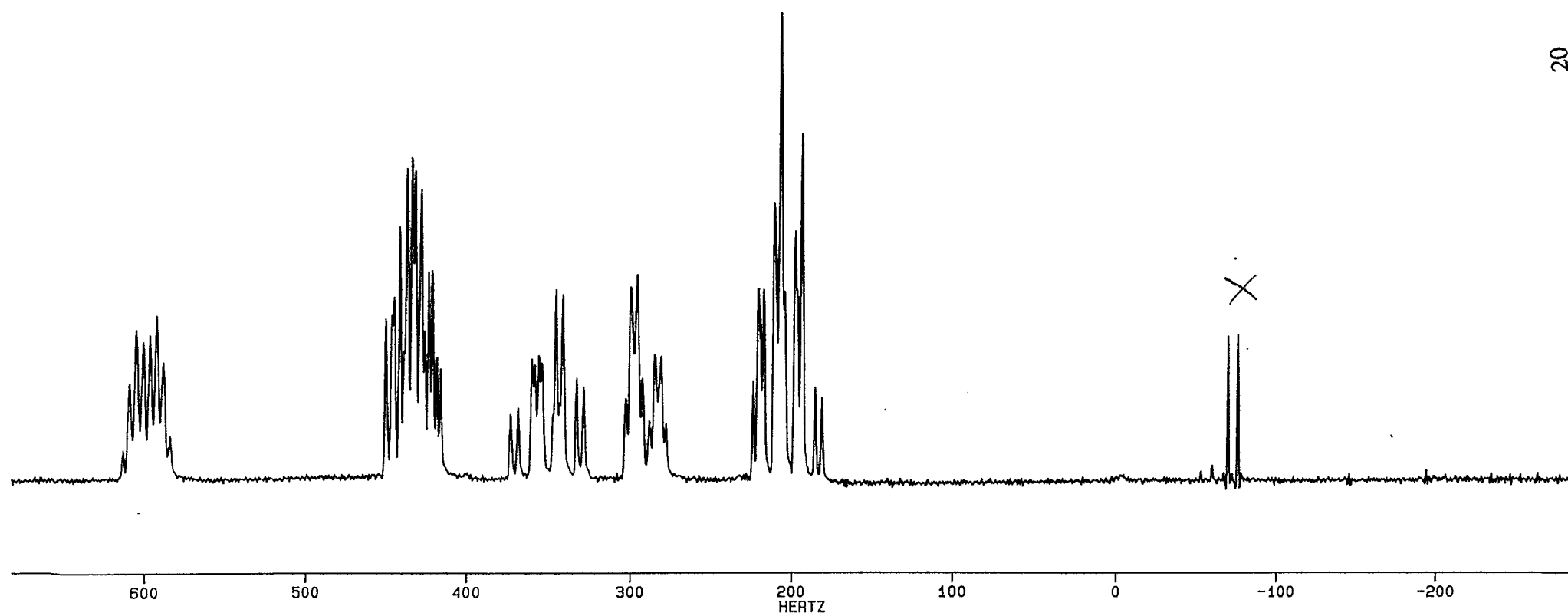
BBBG 202.ccs



20 in CD₃OD. 500MHz. 0.8-2.5 ppm.

SB9

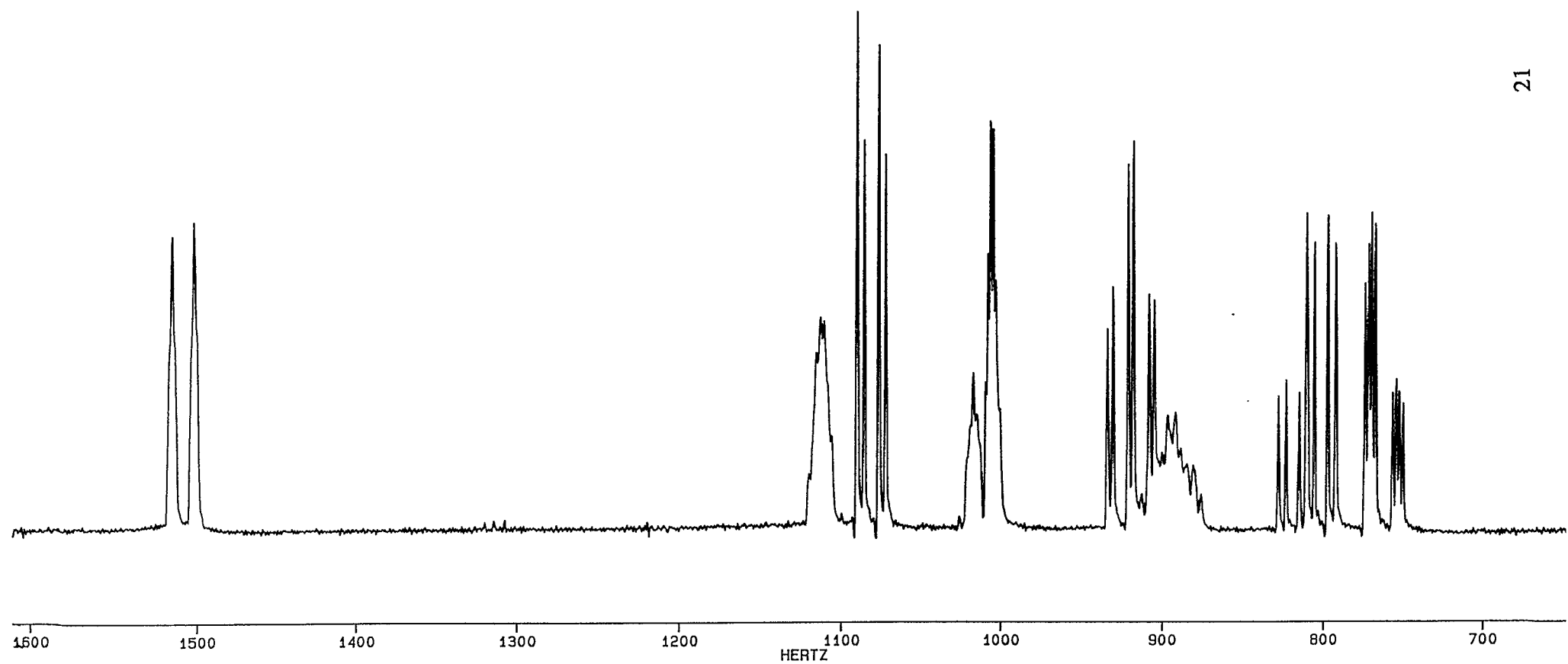
CONTIN



20

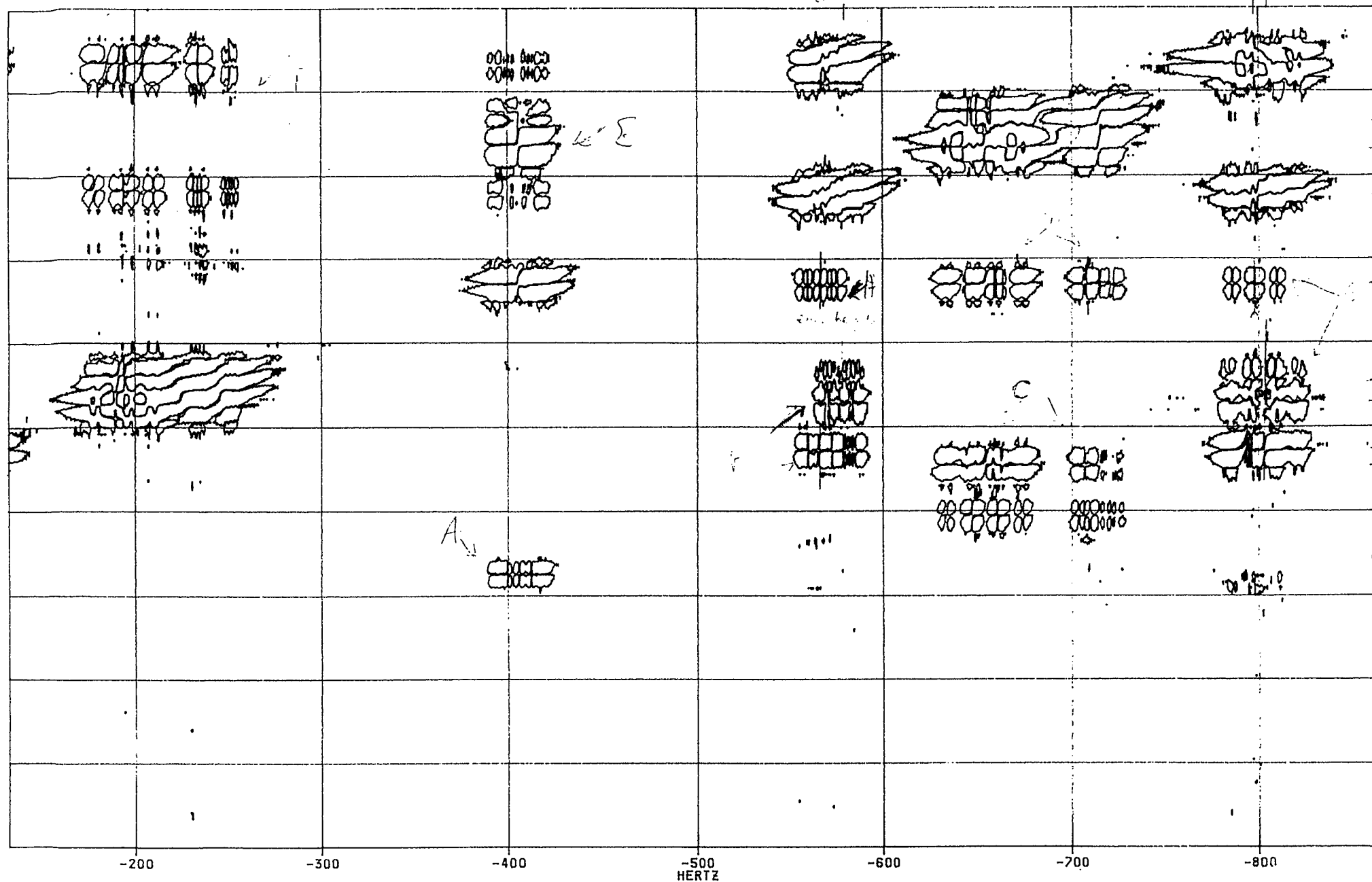
S39
Control 41

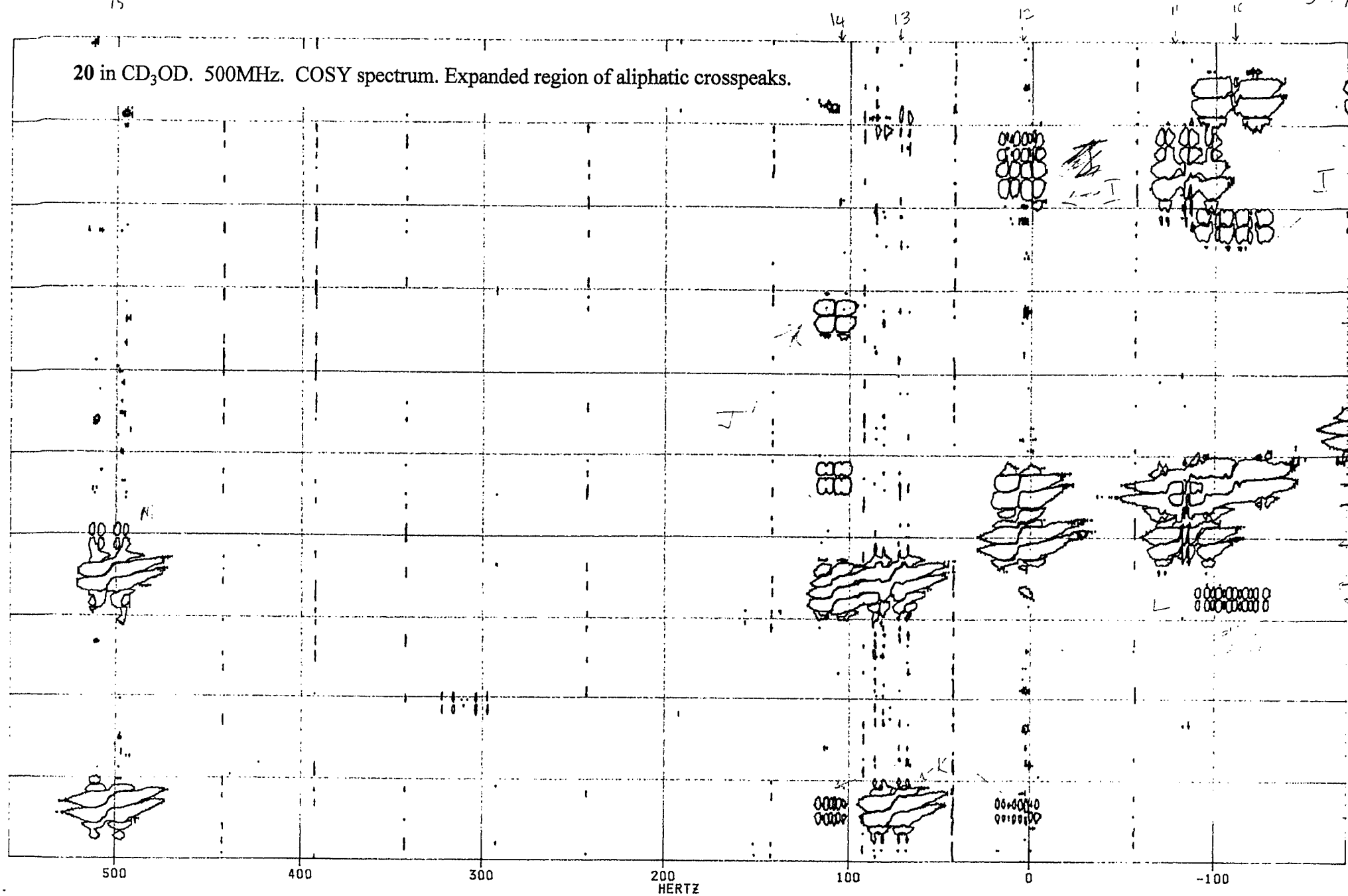
20 in CD₃OD. 500MHz. 2.5-4.2 ppm.



20 in CD₃OD. 500MHz. COSY spectrum. Expanded region of aliphatic crosspeaks.

529





292

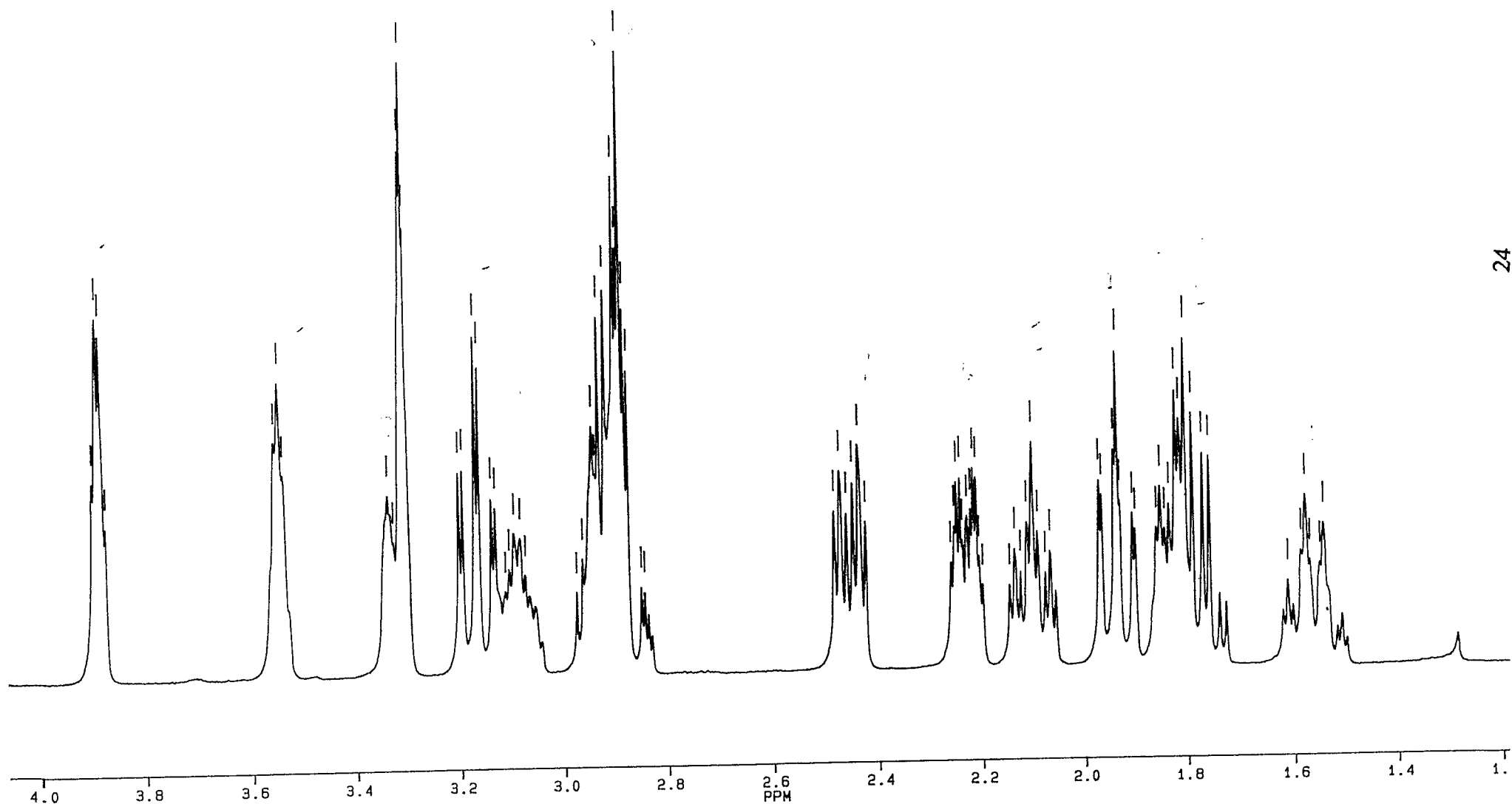
3.90133
3.89188
3.88587
3.87622

3.555603
3.54719
3.53843

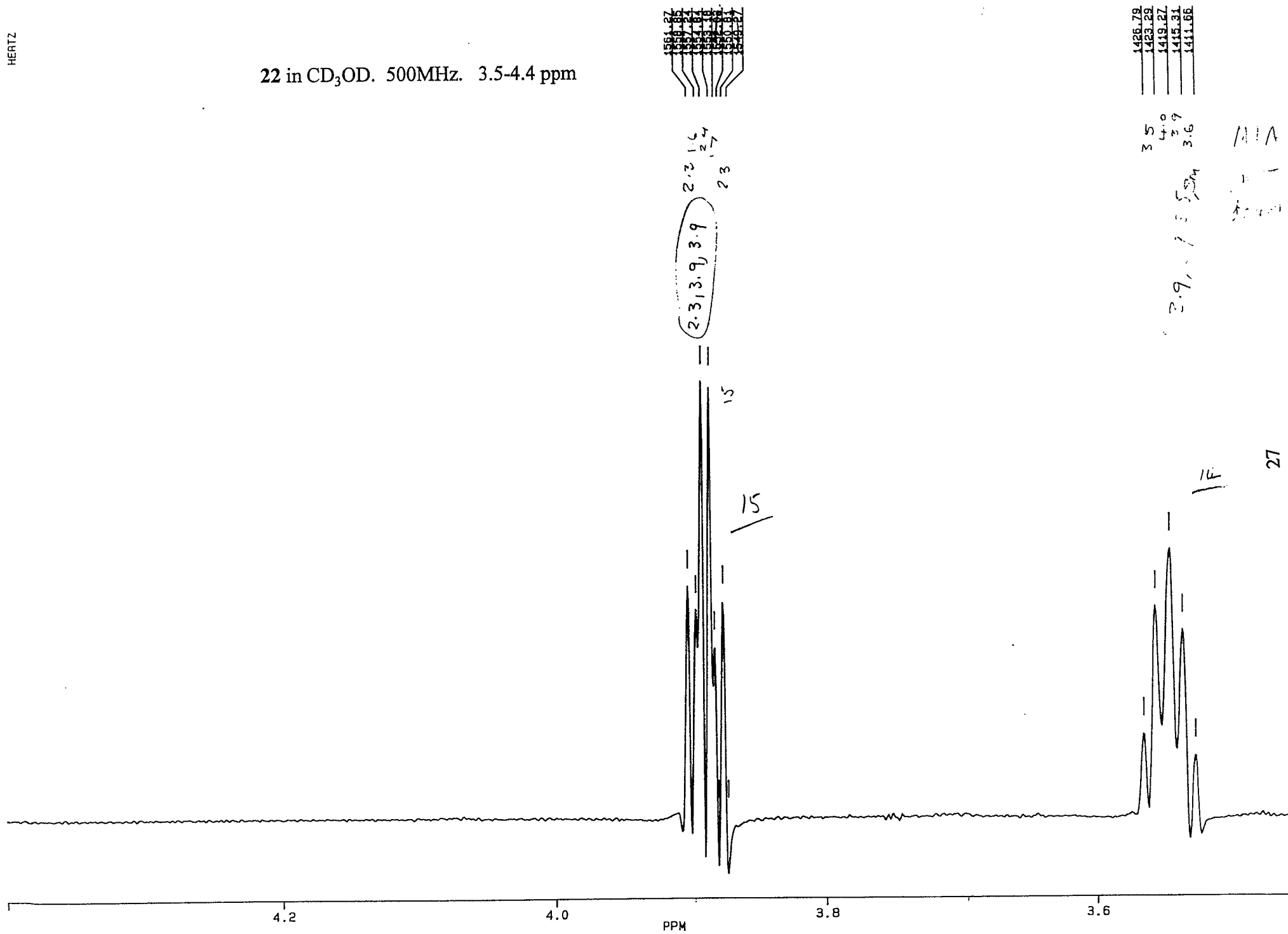
MIA
(R114)
in CD300

22 in CD₃OD. 500MHz. 1.2-4.0 ppm.

5211.

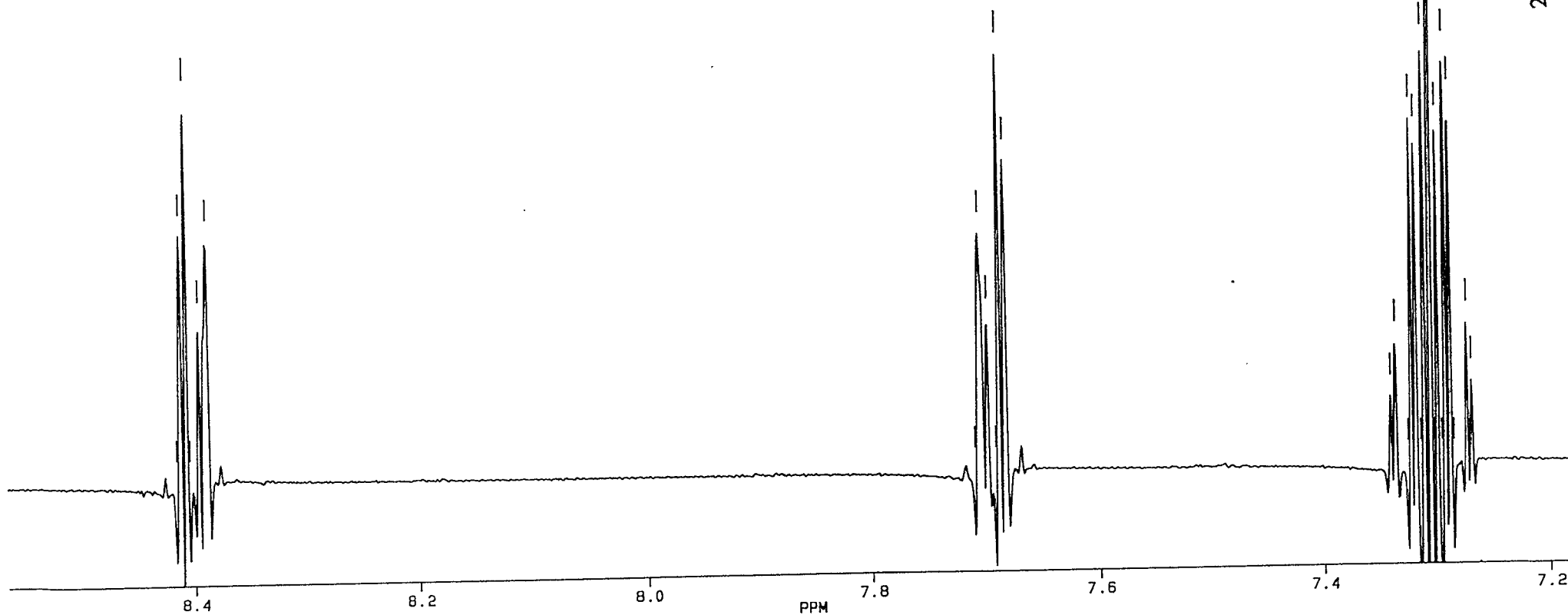


22 in CD₃OD. 500MHz. 3.5-4.4 ppm

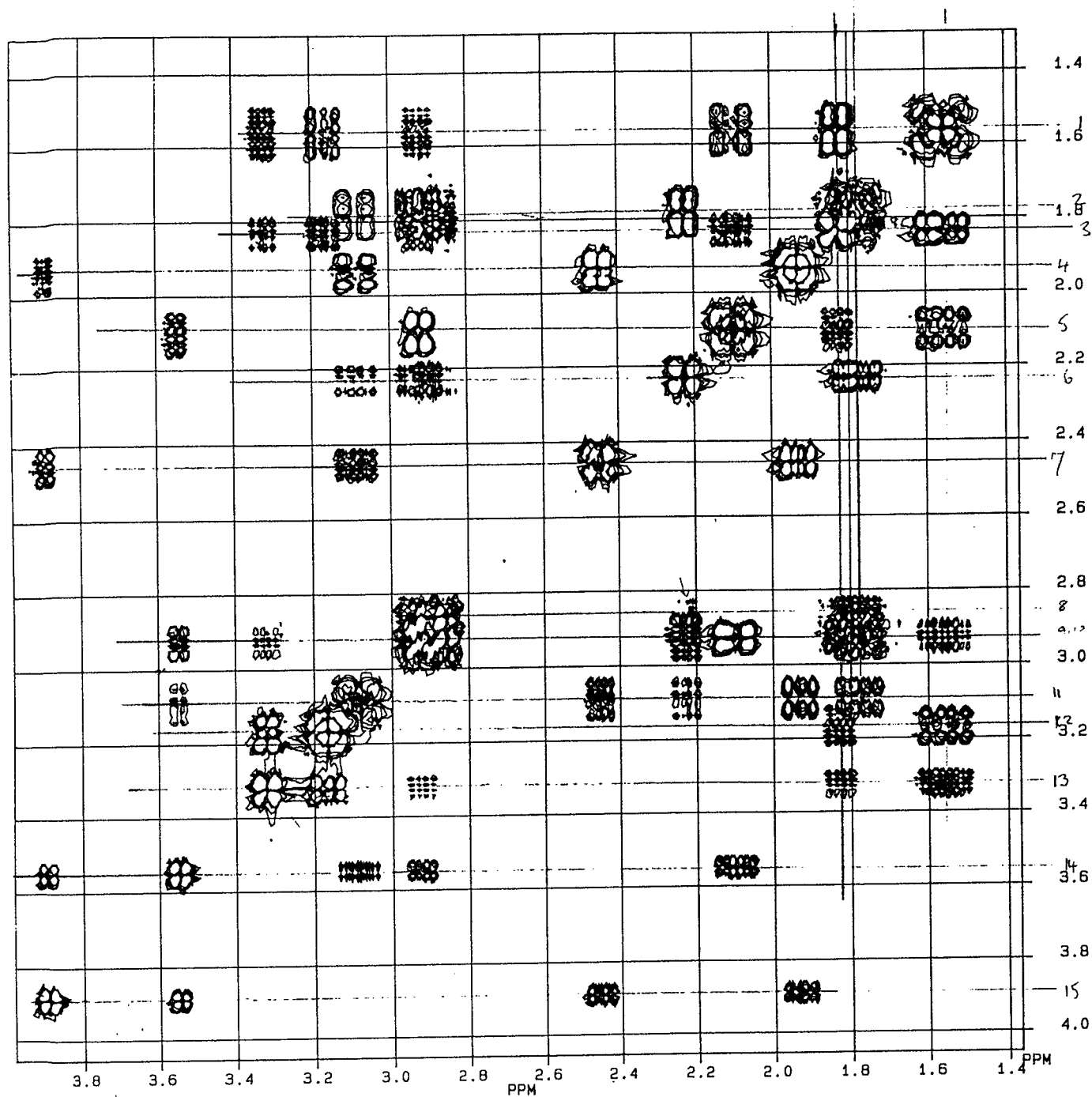


22 in CD₃OD. 500MHz. 7.2-8.5 ppm.

MIA
(R114)
DECONV



22 in CD₃OD. 500MHz. COSY spectrum. 1.4-4.0 ppm region.



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2113

9/10 2

rd 8,

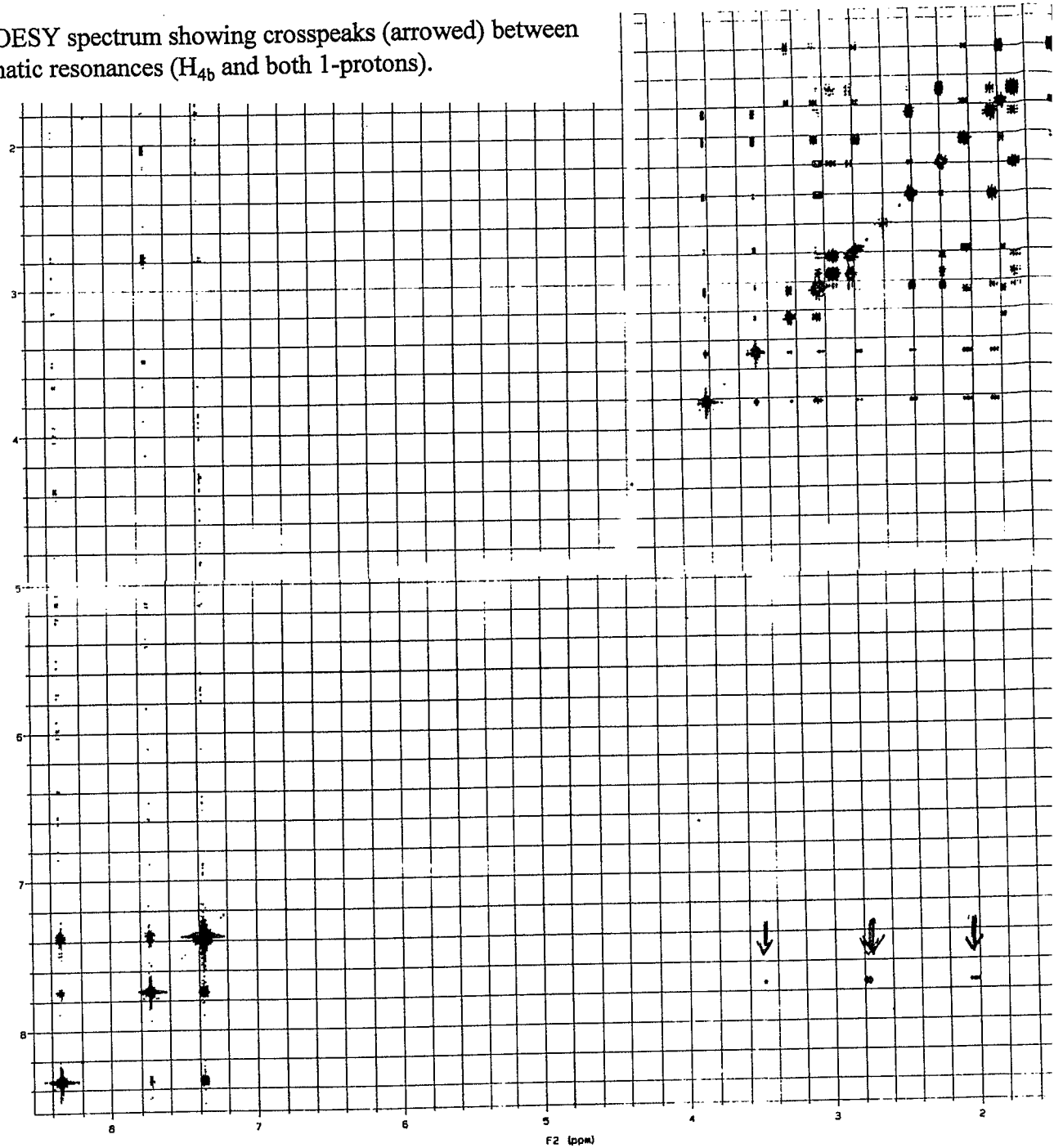
14, 7, 6, 1, 4, 2
13 3, 1

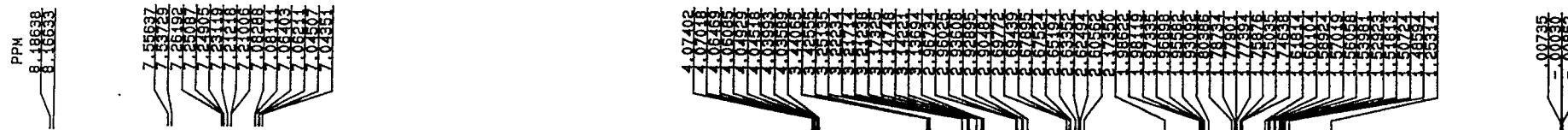
12 9/10 3, 1
long range

15, 11 9/10, 5

→ 14, 7, 4

22 in CD₃OD. 500MHz. ROESY spectrum showing crosspeaks (arrowed) between the H₁₃ proton and three aliphatic resonances (H_{4b} and both 1-protons).





11.

$1/4 + 4/5$

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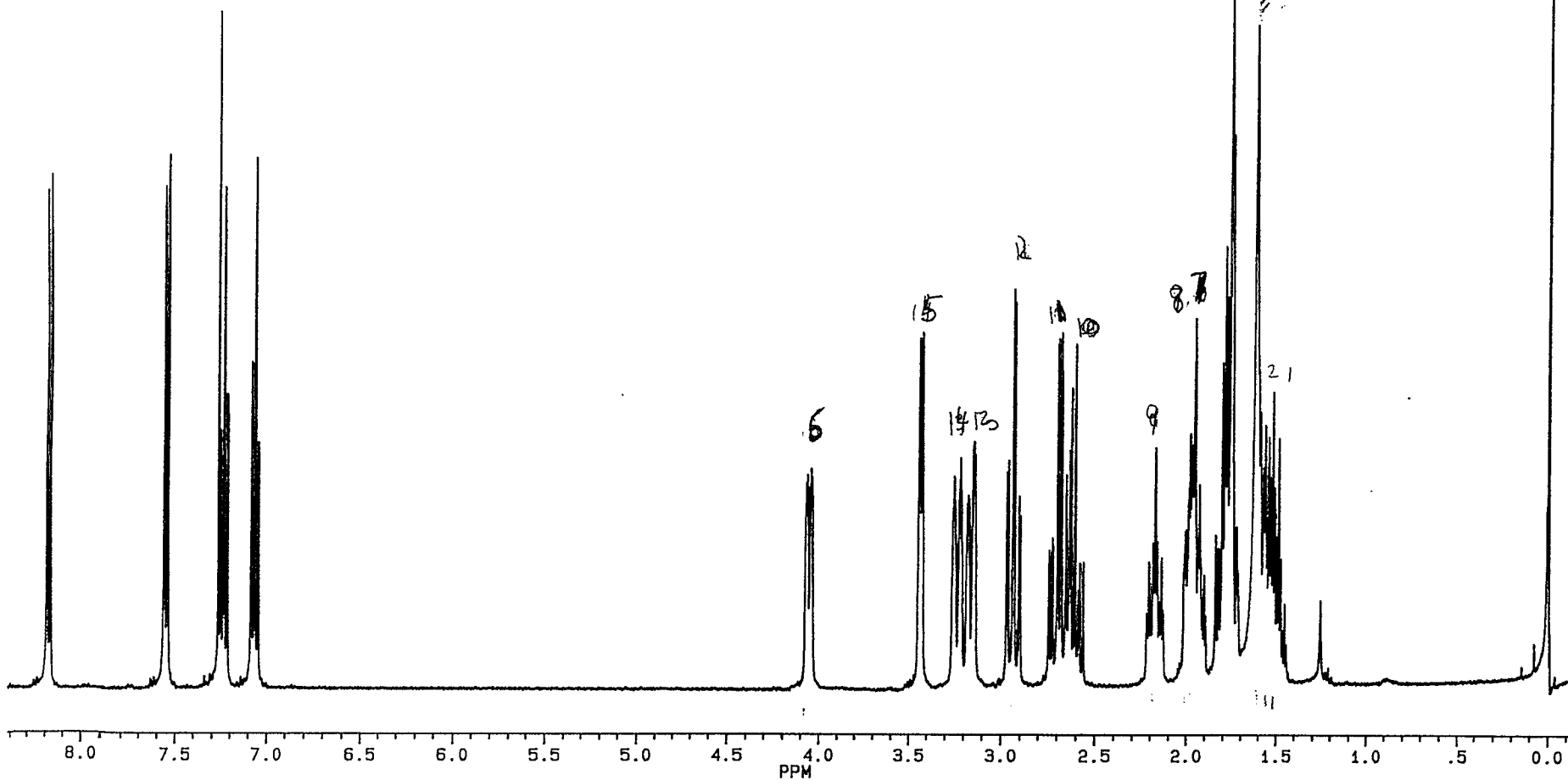
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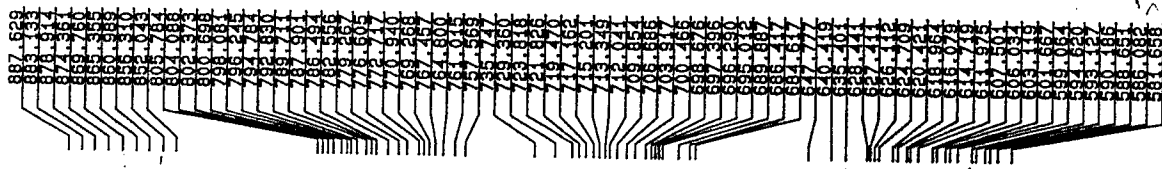
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531</

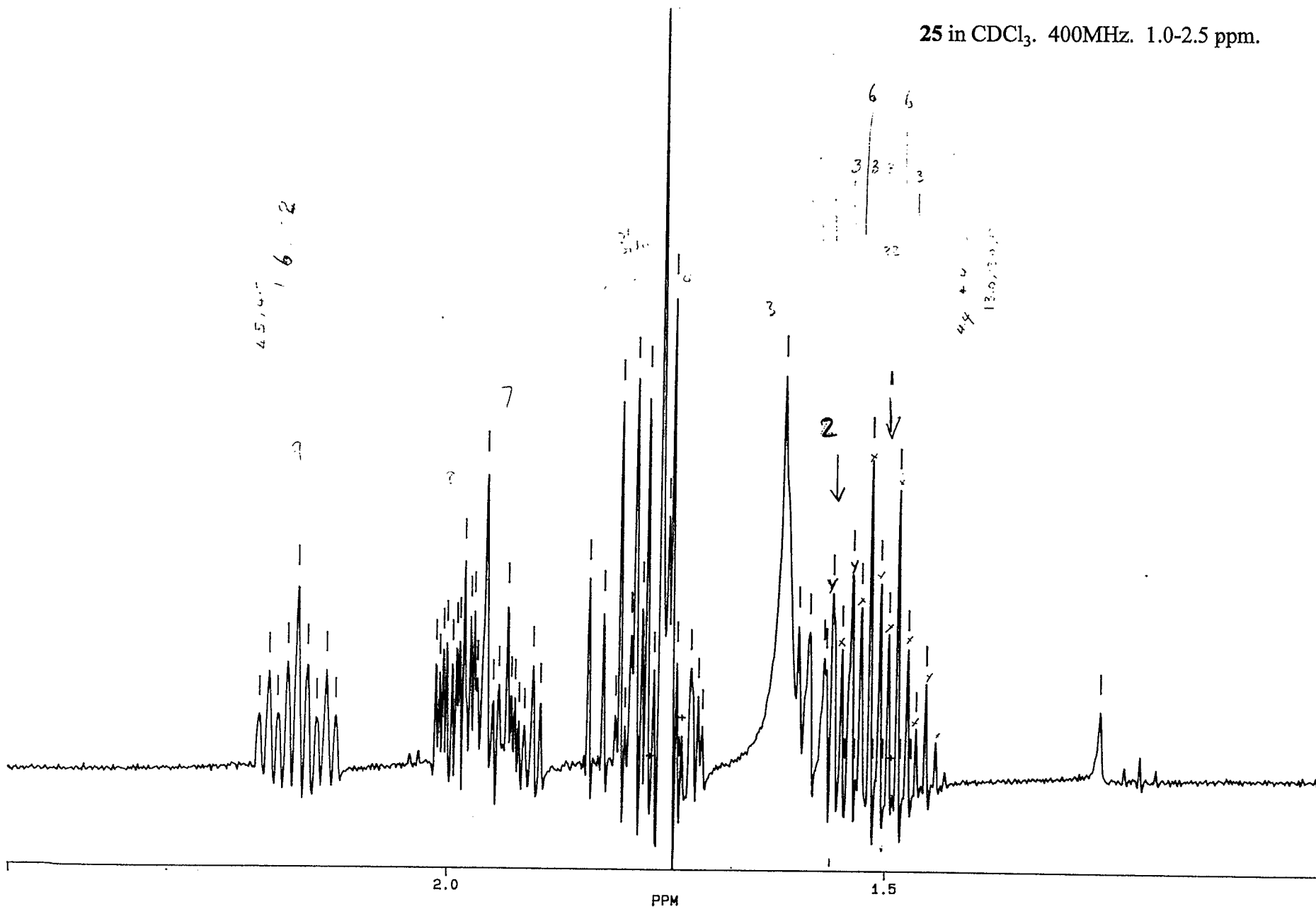


S99A
DATE 17-2-94
SF 400.134
SI 32768

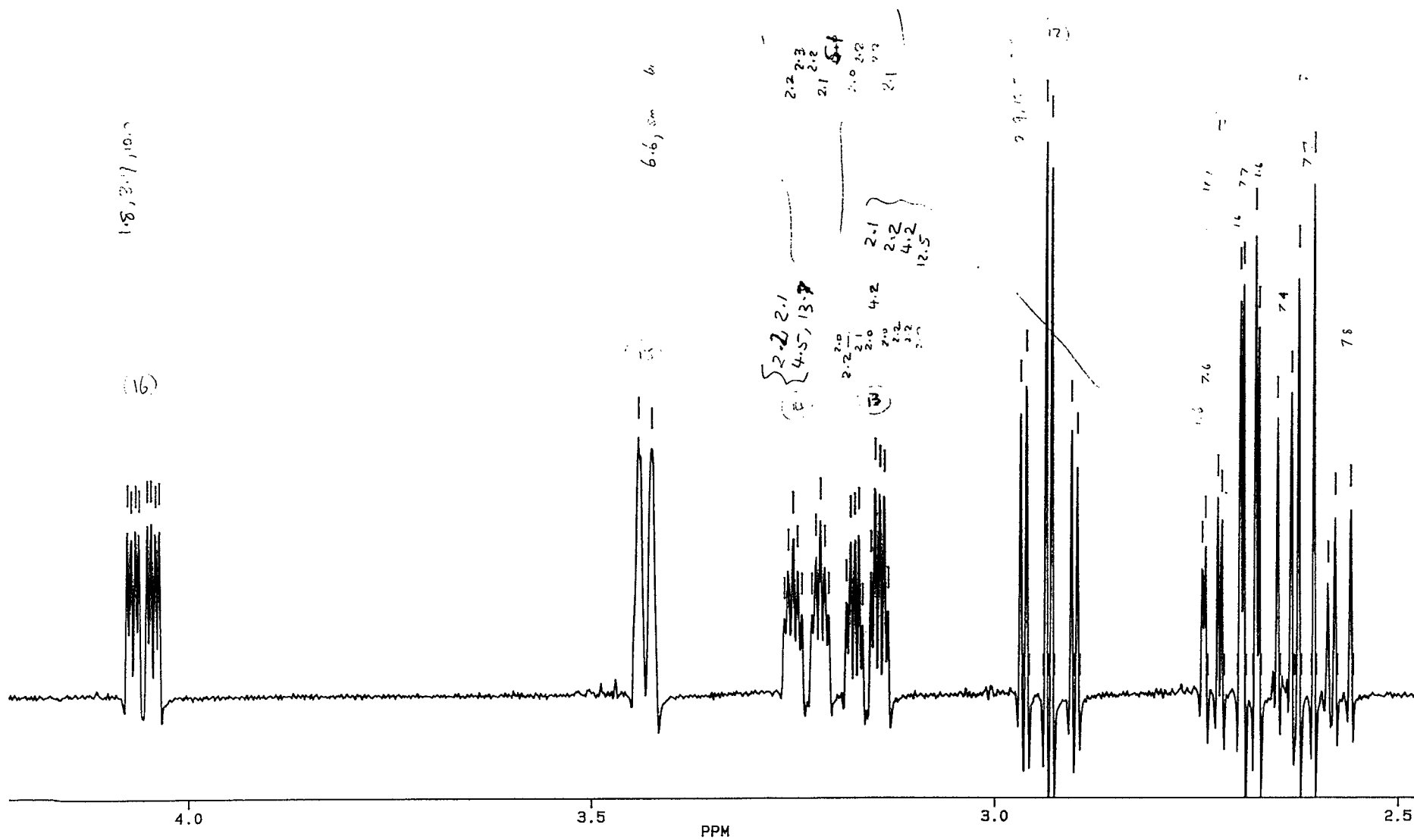




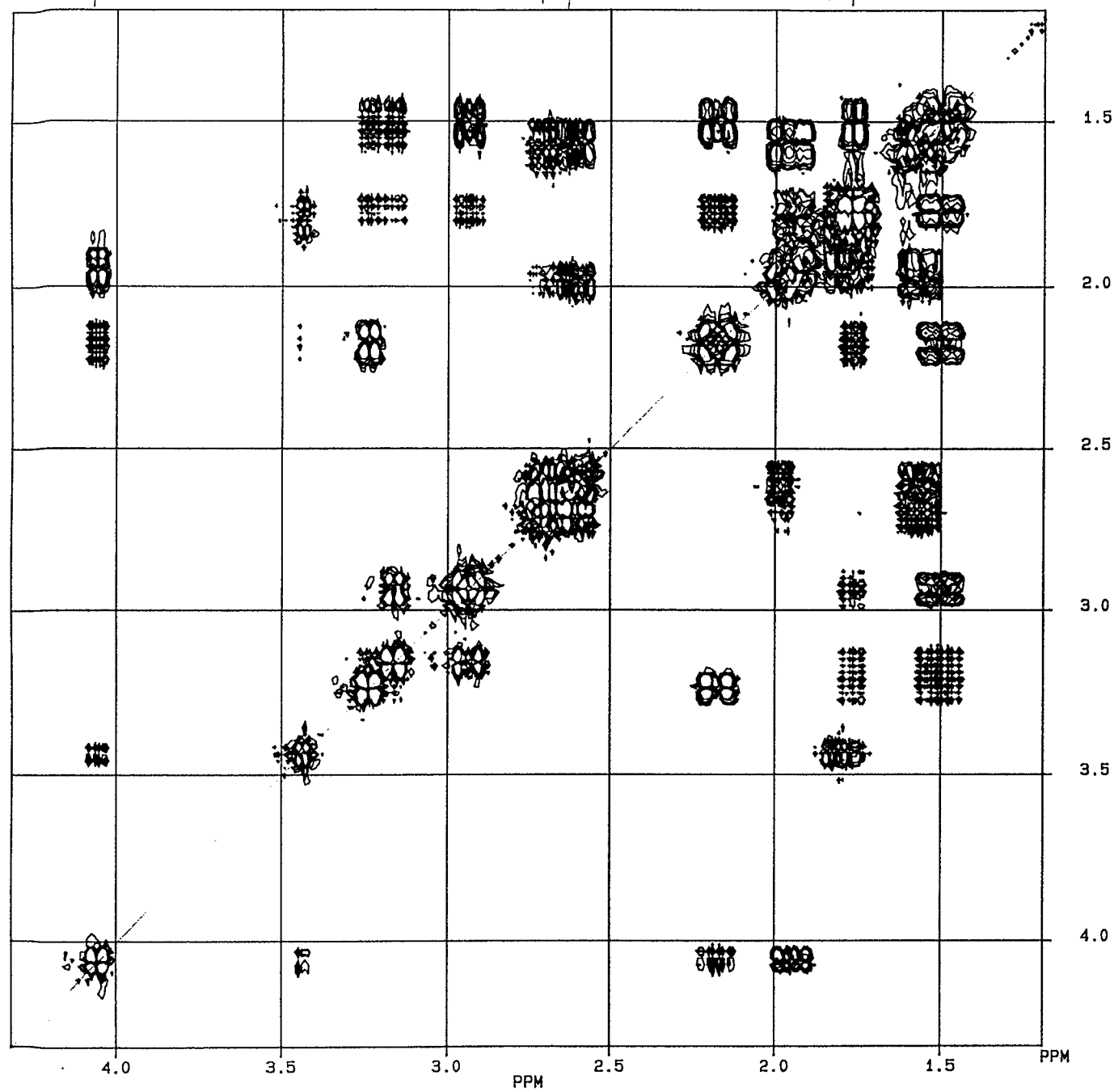
501.423

25 in CDCl_3 . 400MHz. 1.0-2.5 ppm.

1377.31
1370.70



25 in CDCl₃, 400MHz. COSY spectrum. Aliphatic region.



25 in CDCl₃, 400MHz. COSY spectrum. Aromatic region.

