JOC The Journal of Organic Chemistry

J. Org. Chem., 1998, 63(17), 6039-6042, DOI:10.1021/jo9803378

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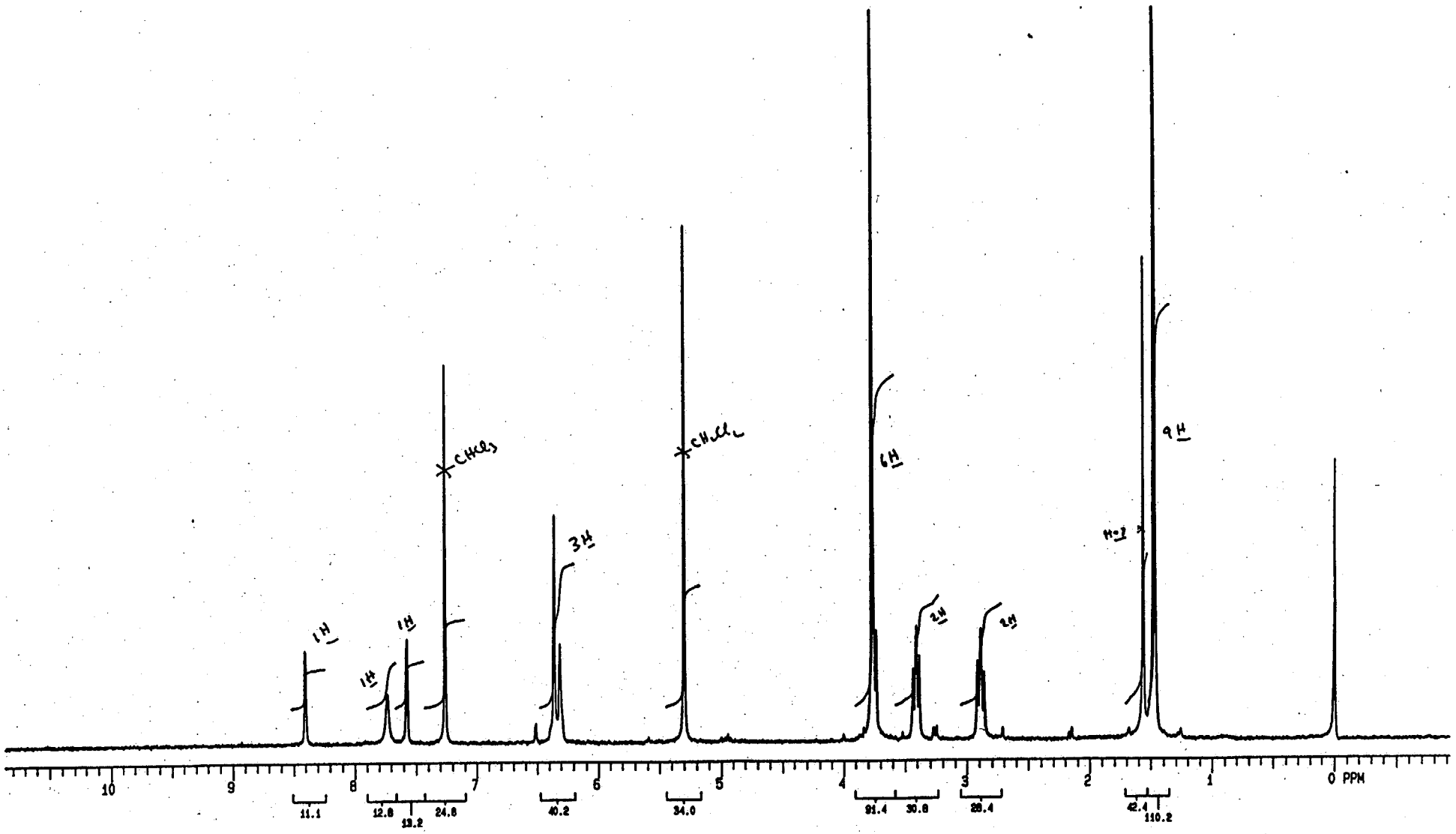

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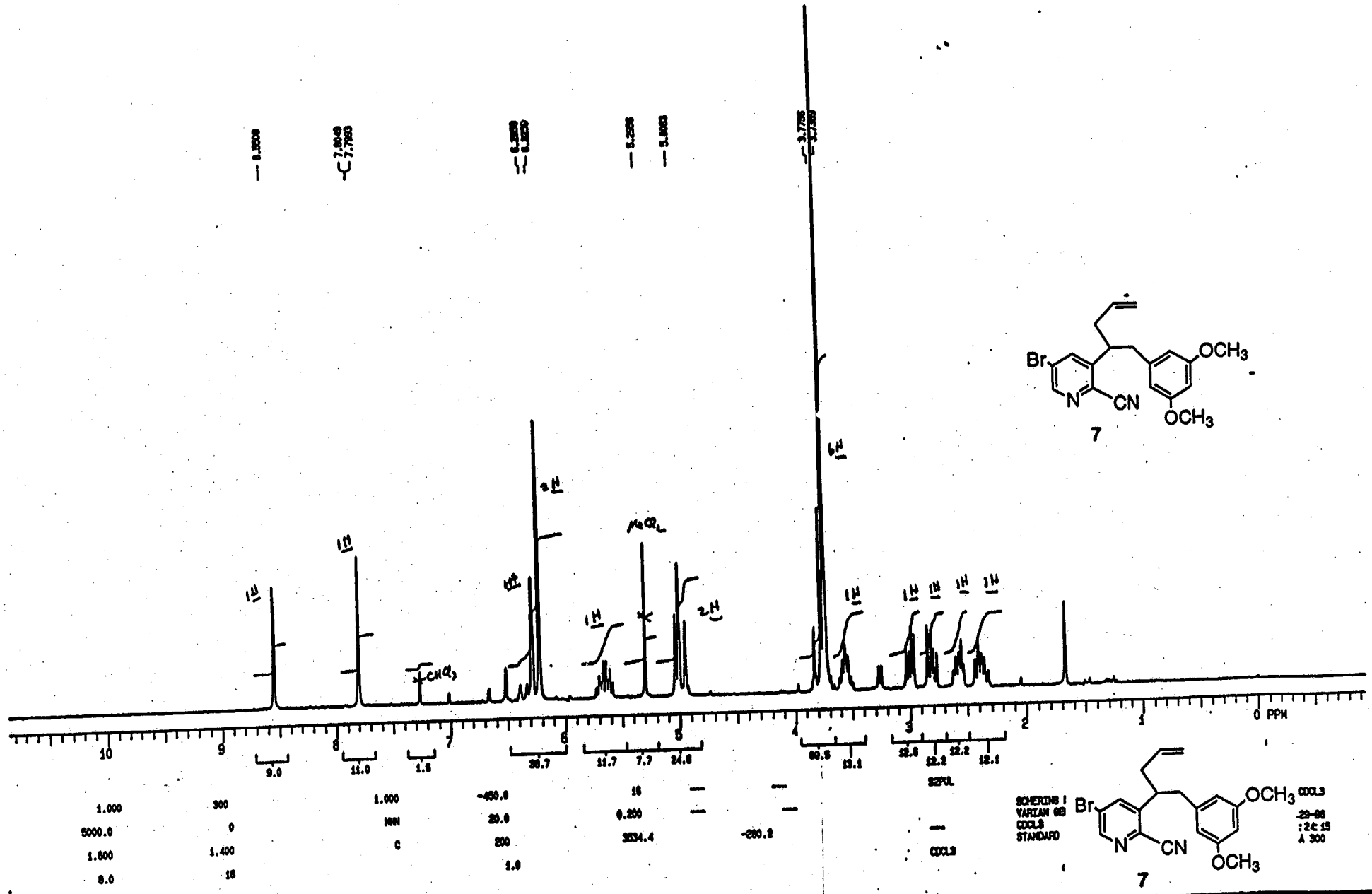

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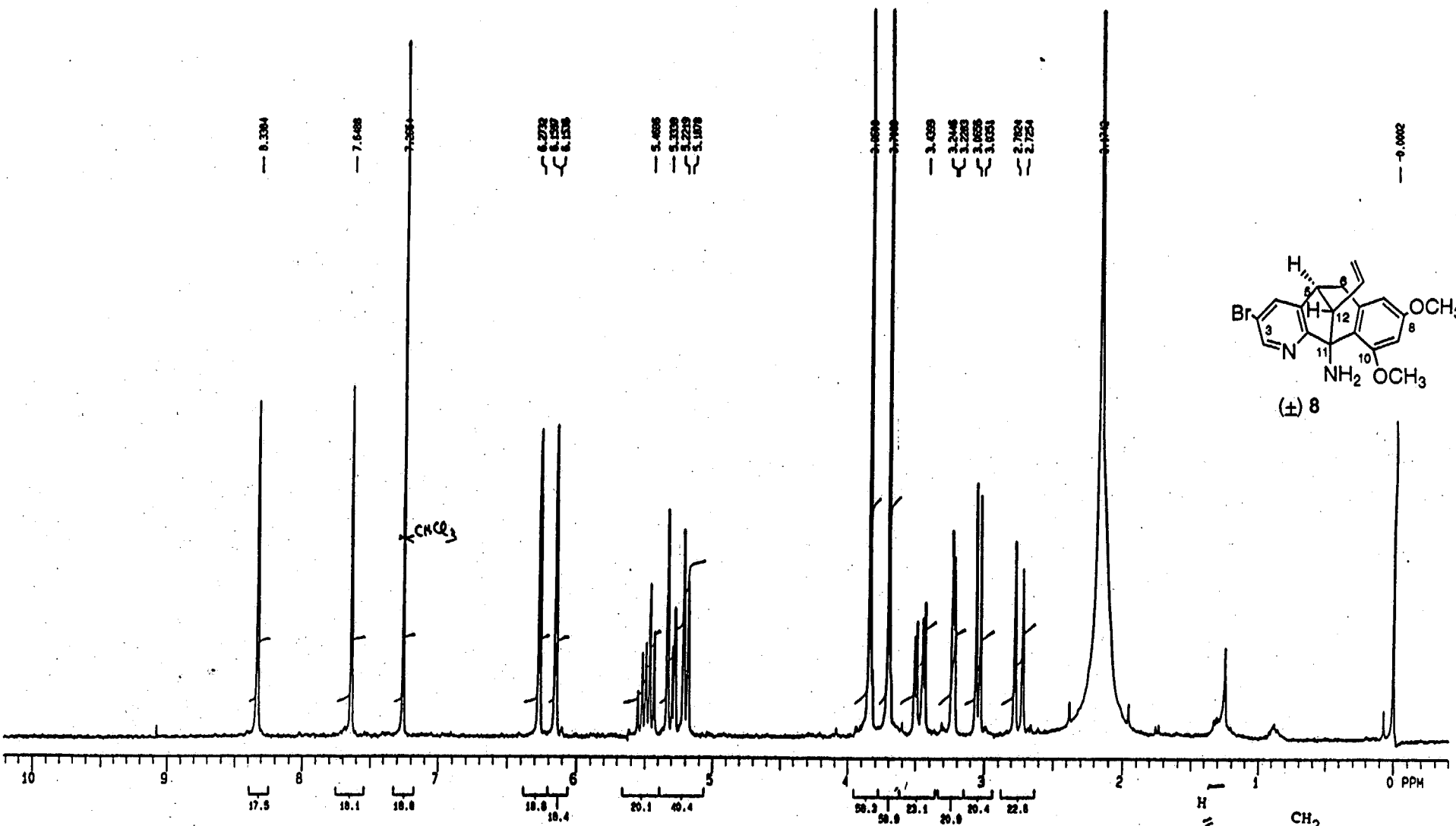

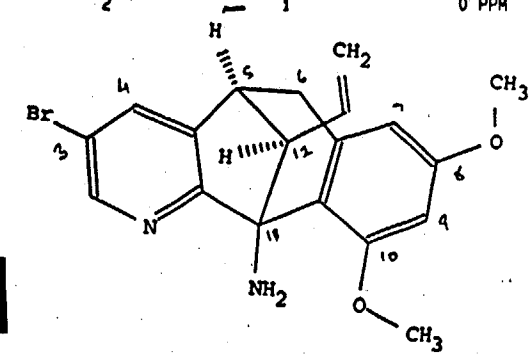

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J.KELLY
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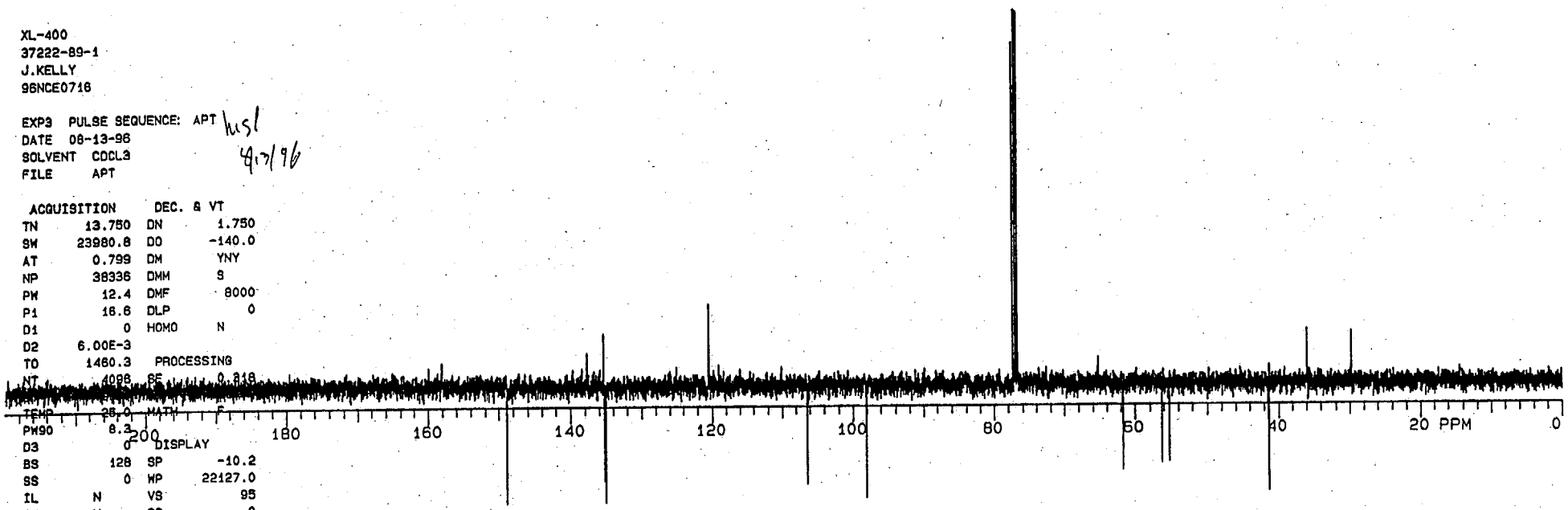

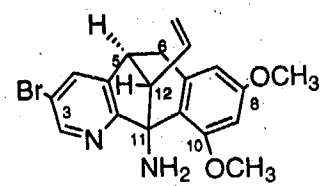

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kas P 8/13/96

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17	7720.7	76.764	327.929
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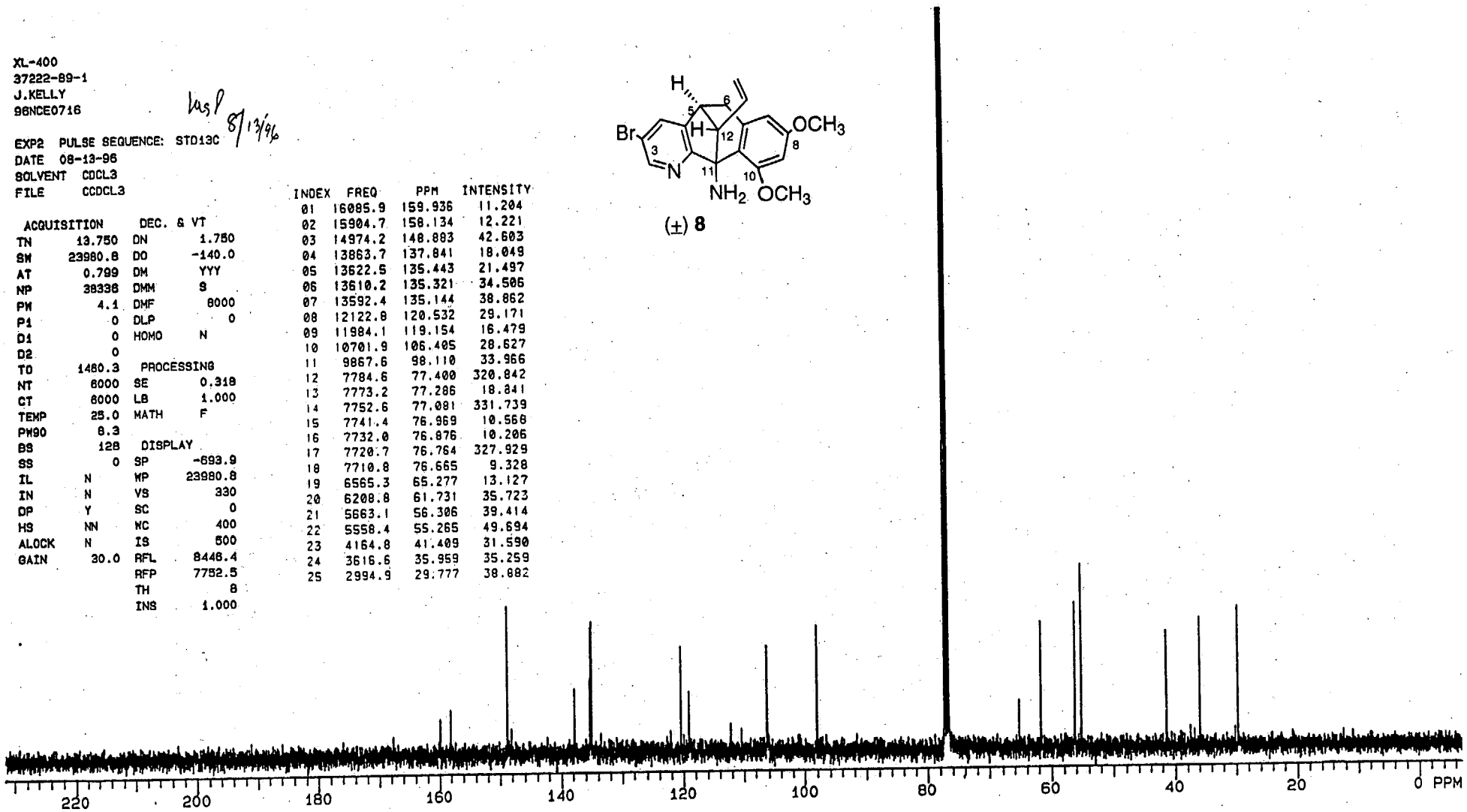

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ms ? 8/13/96

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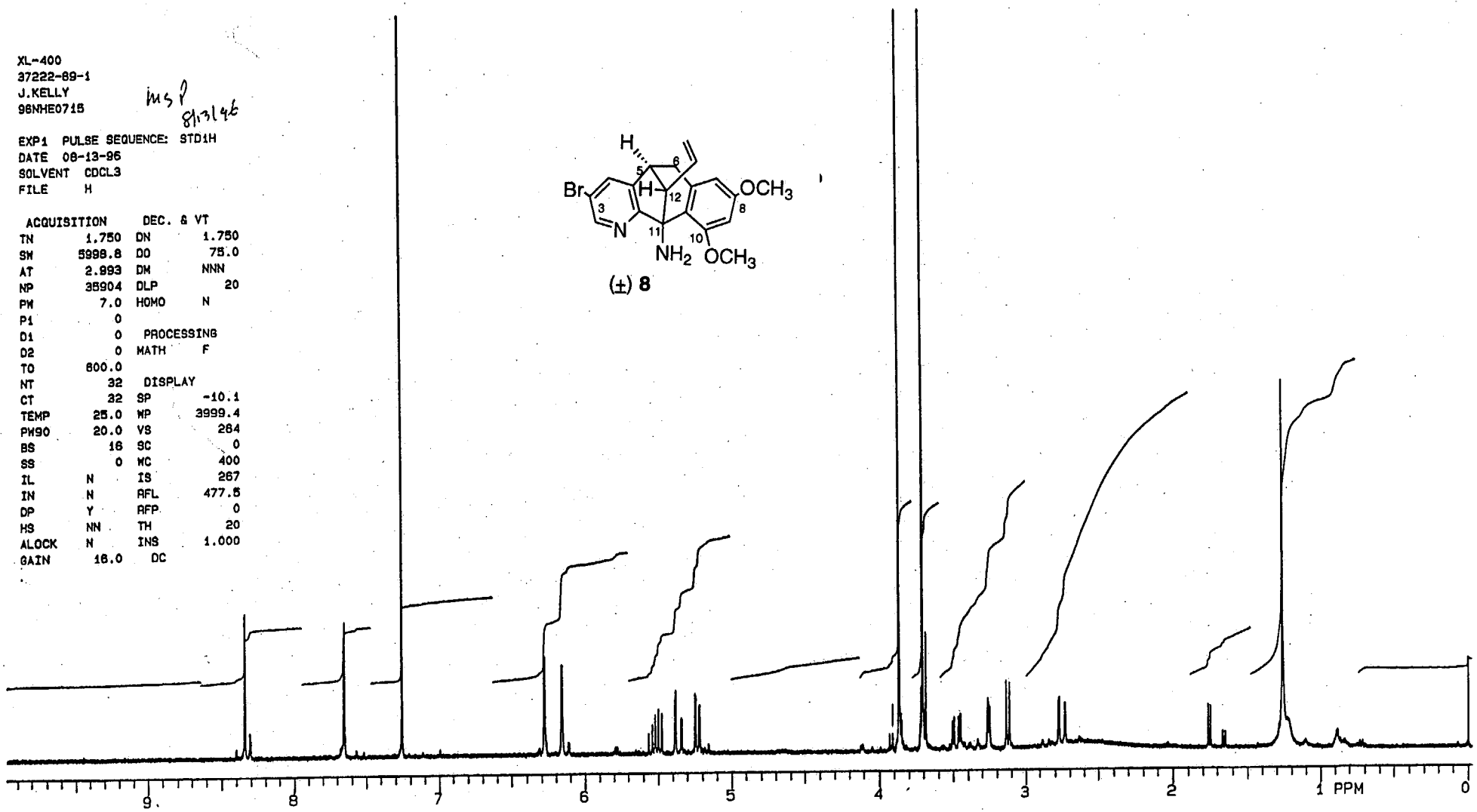

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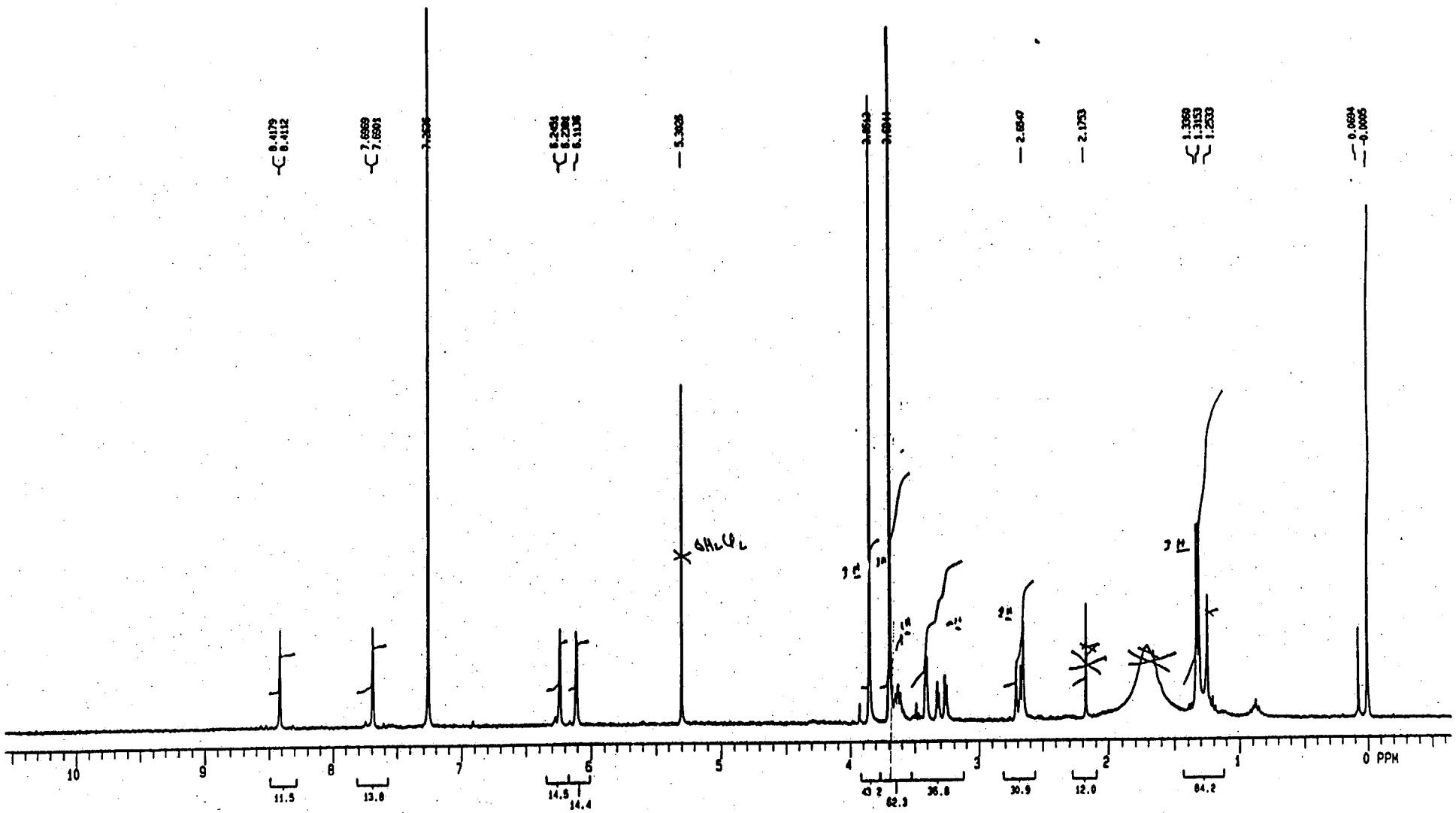

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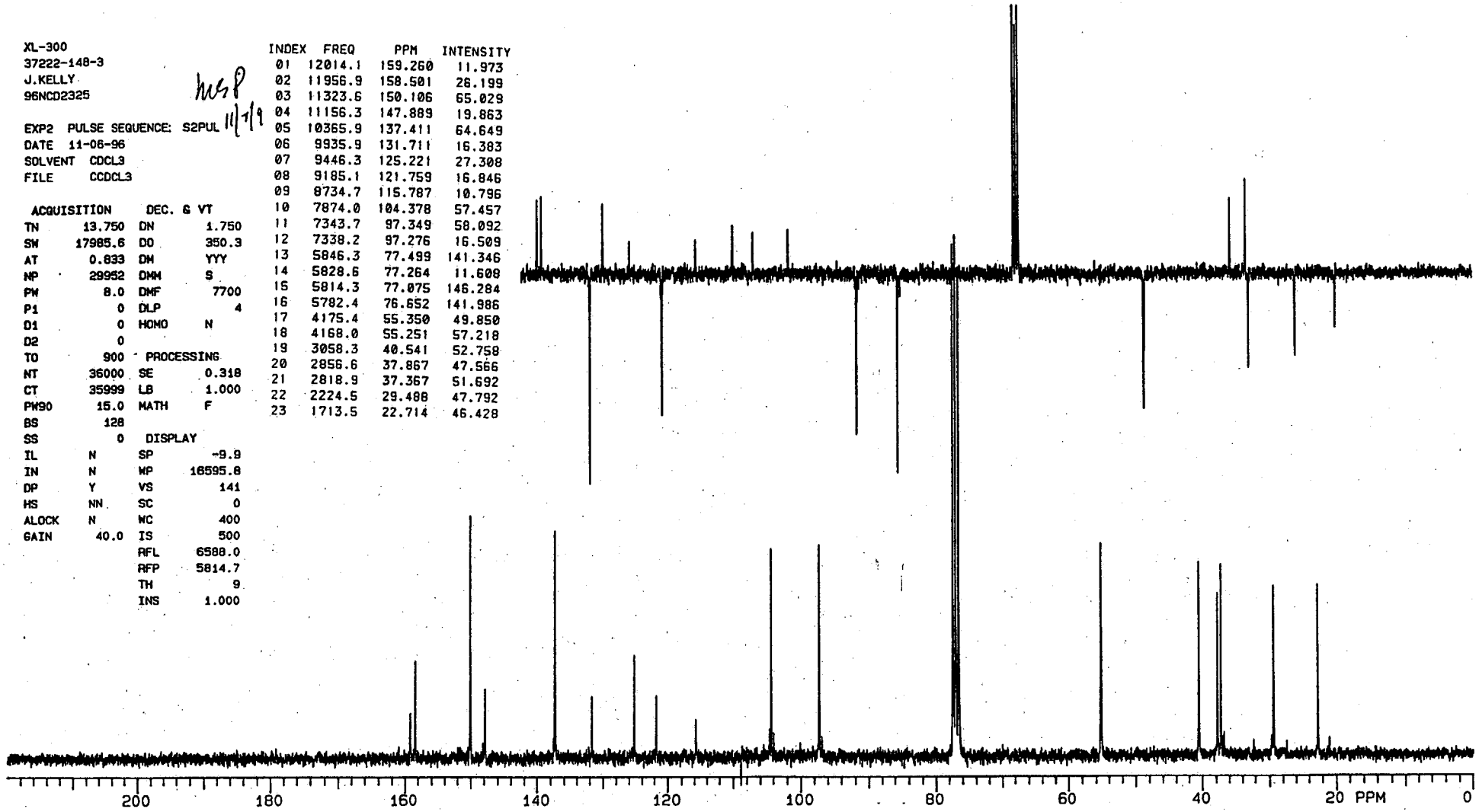

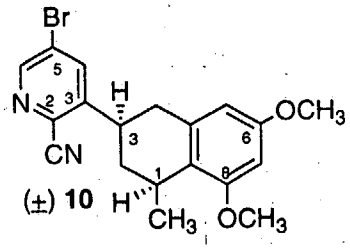

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11/22/96
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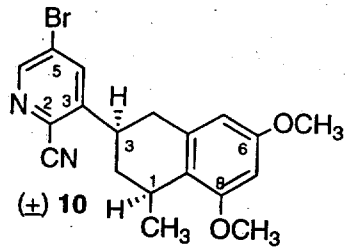
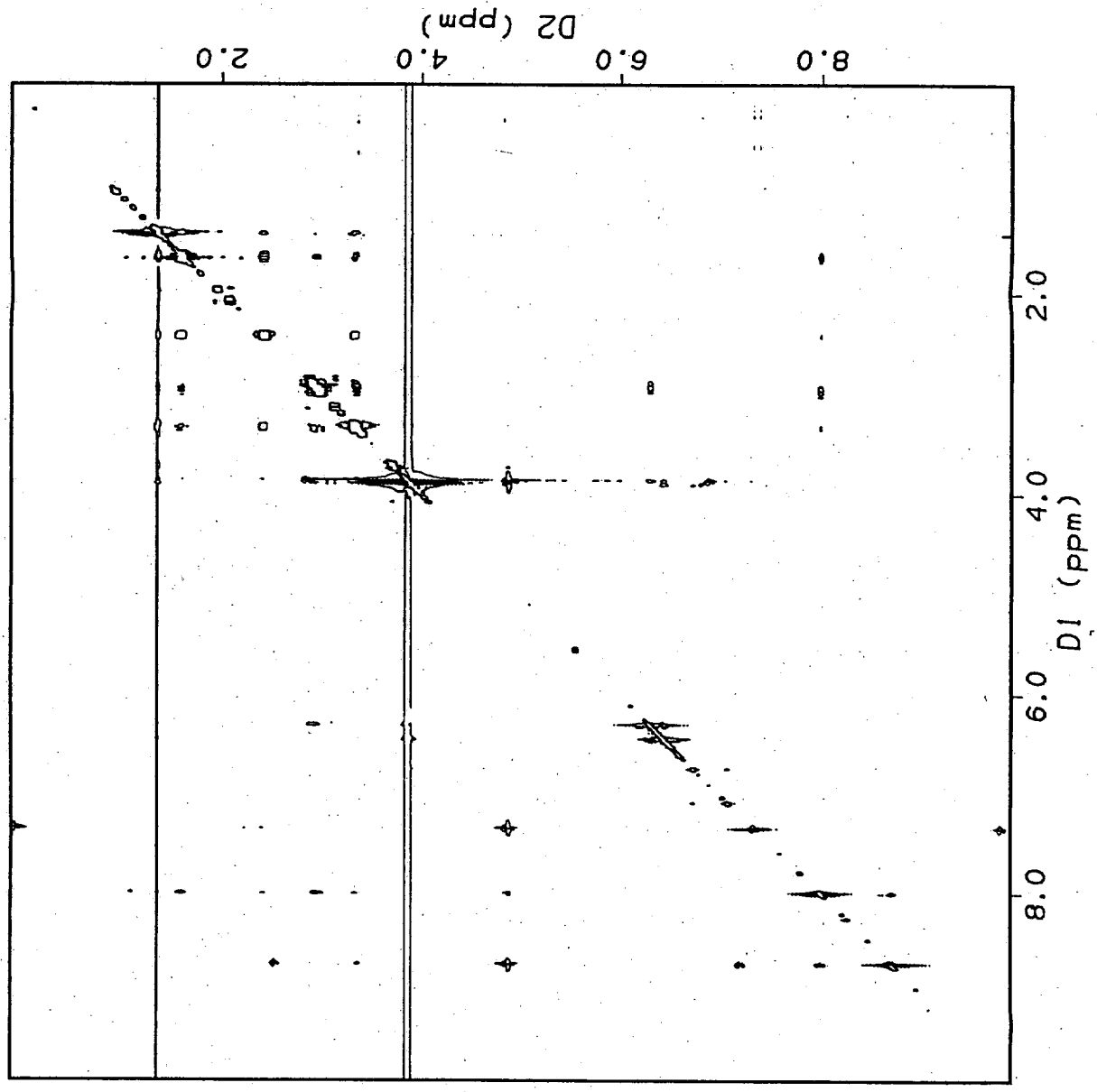
NOESY





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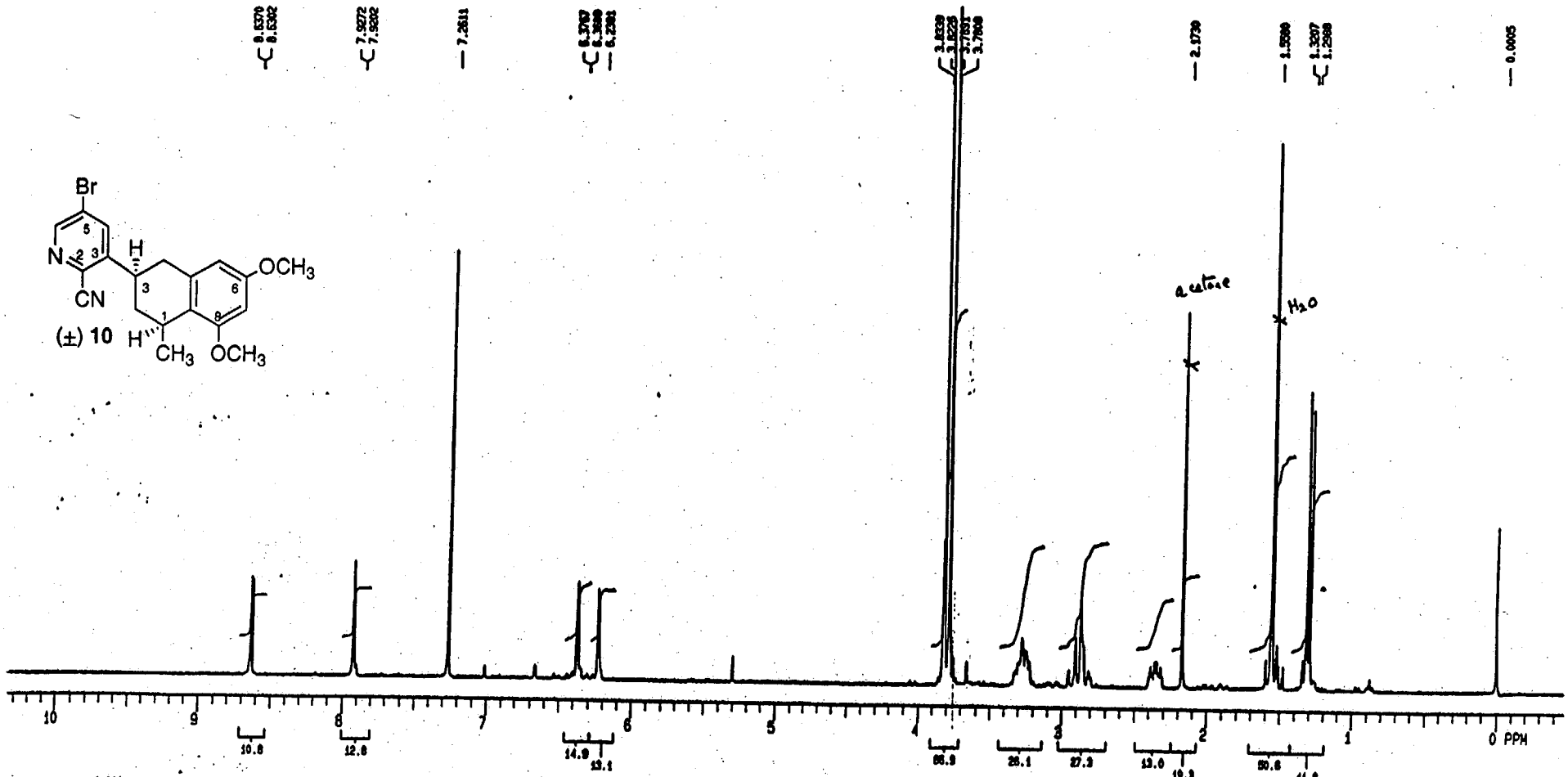

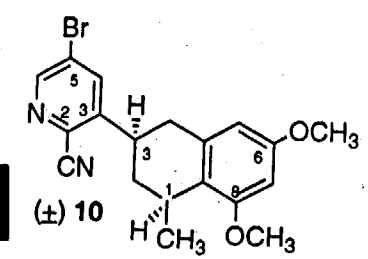

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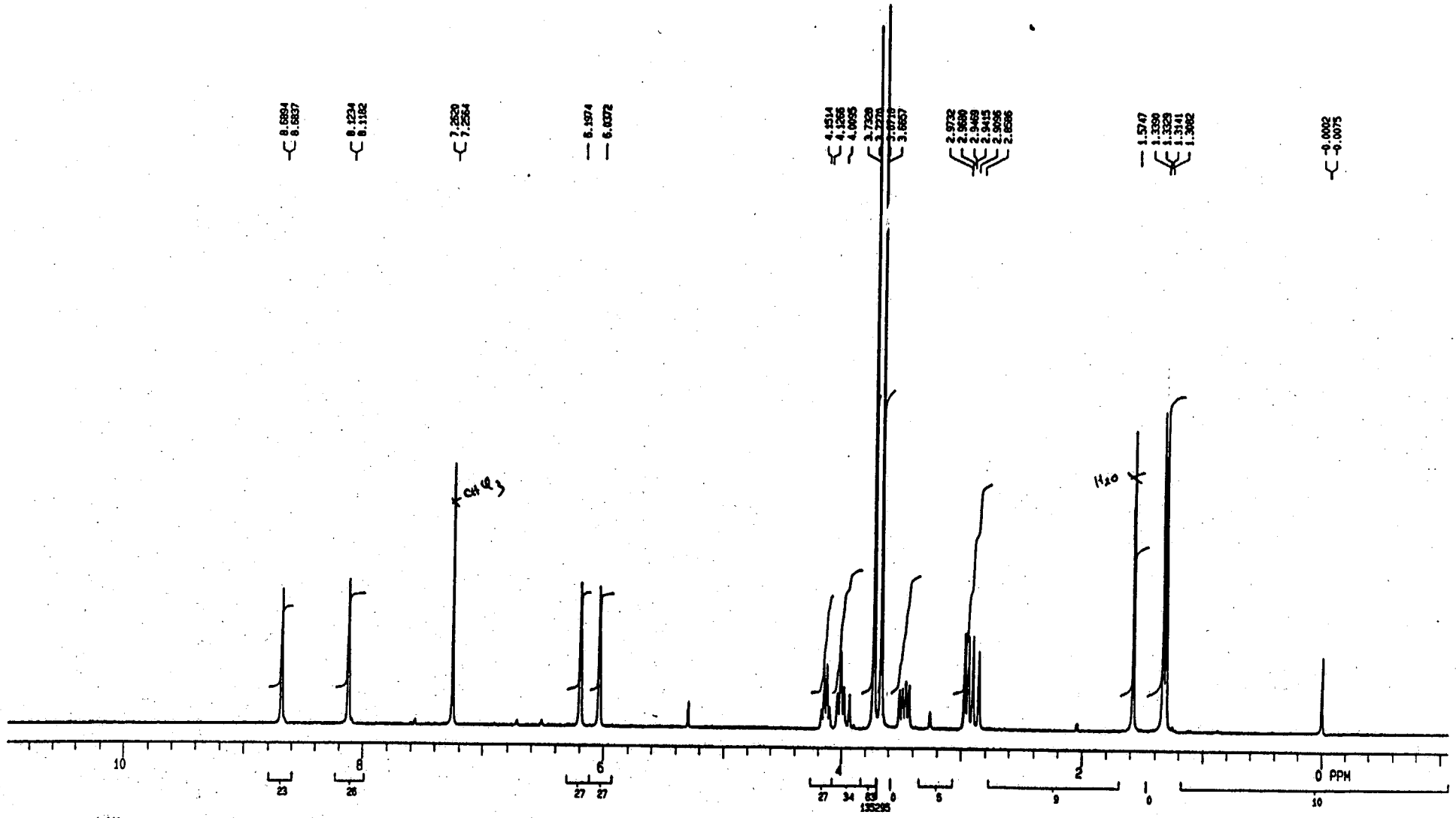

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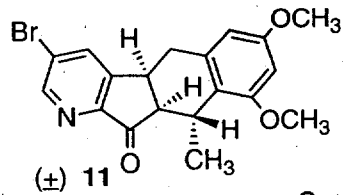
37222-88-1 J. Kelly 96 nhg 3465



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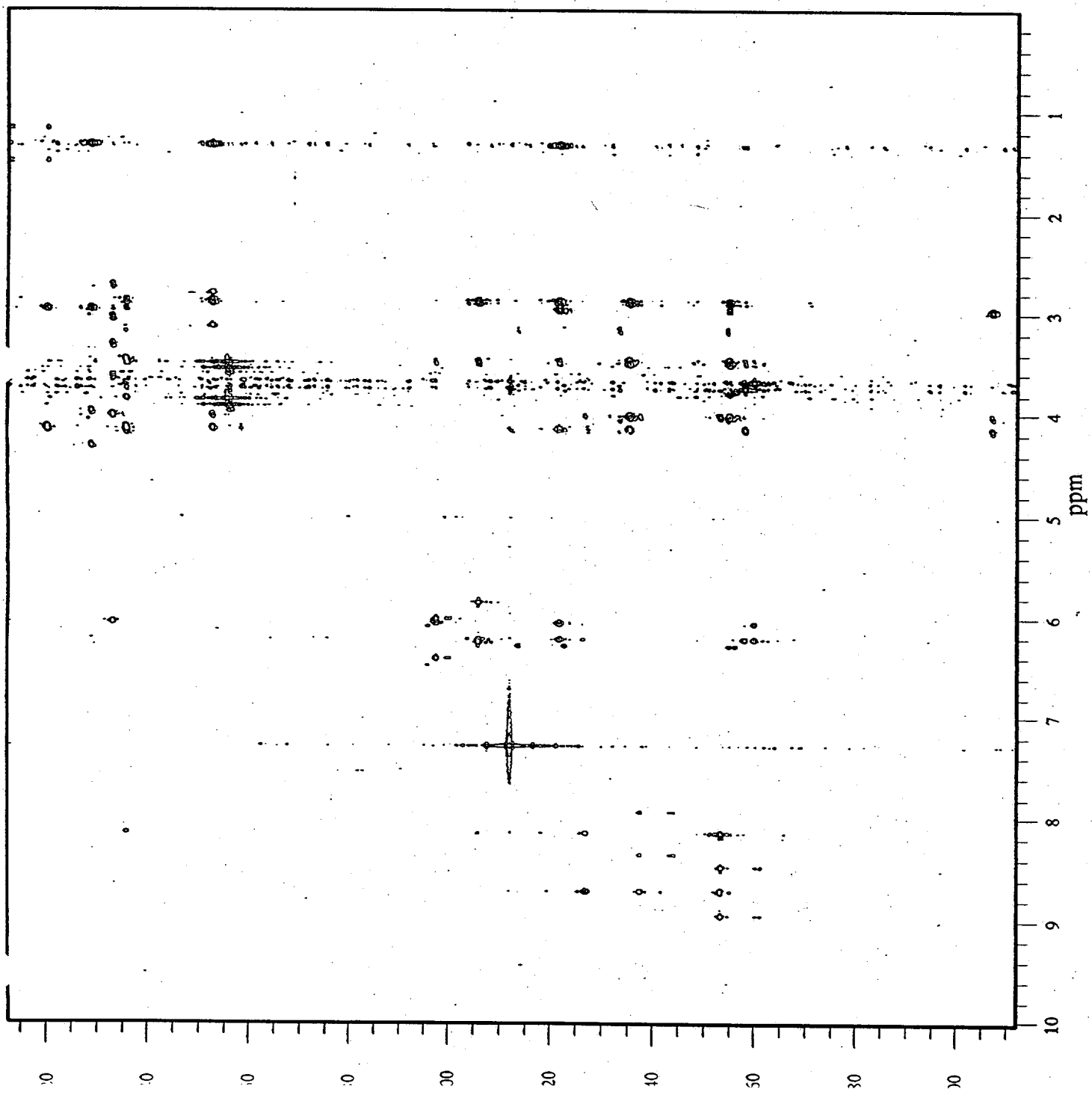

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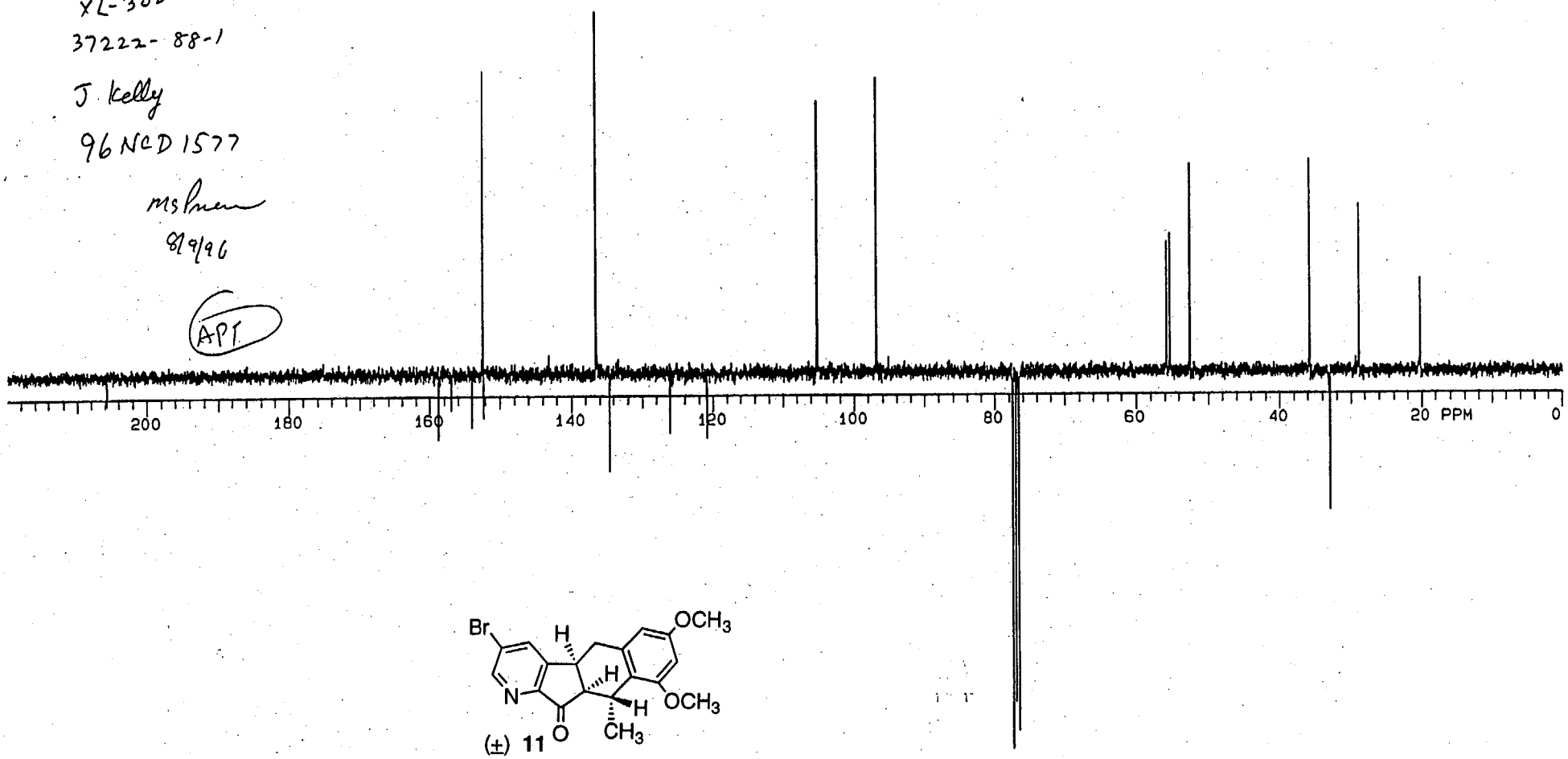
MS Pneu
8/9/96
APT



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17	4200.7	55.684	62.833
18	4165.8	55.222	70.838
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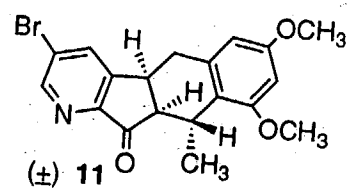

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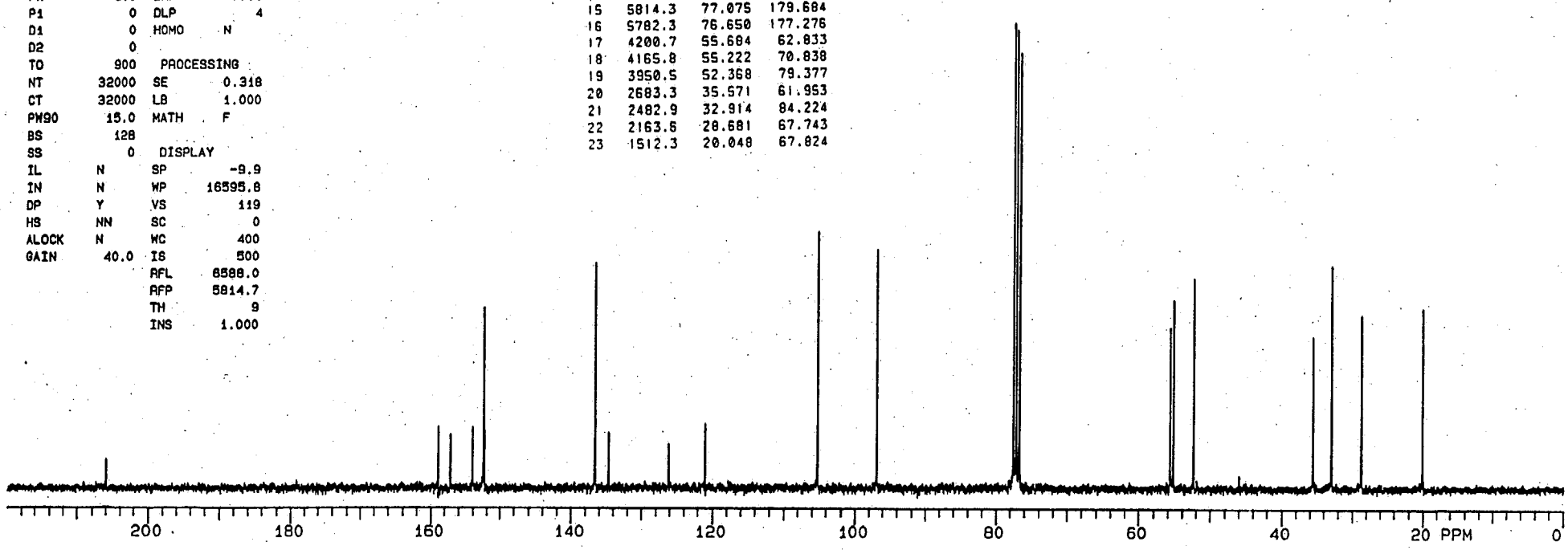

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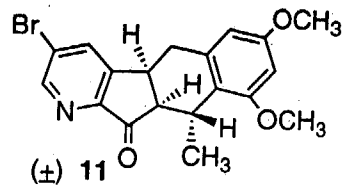

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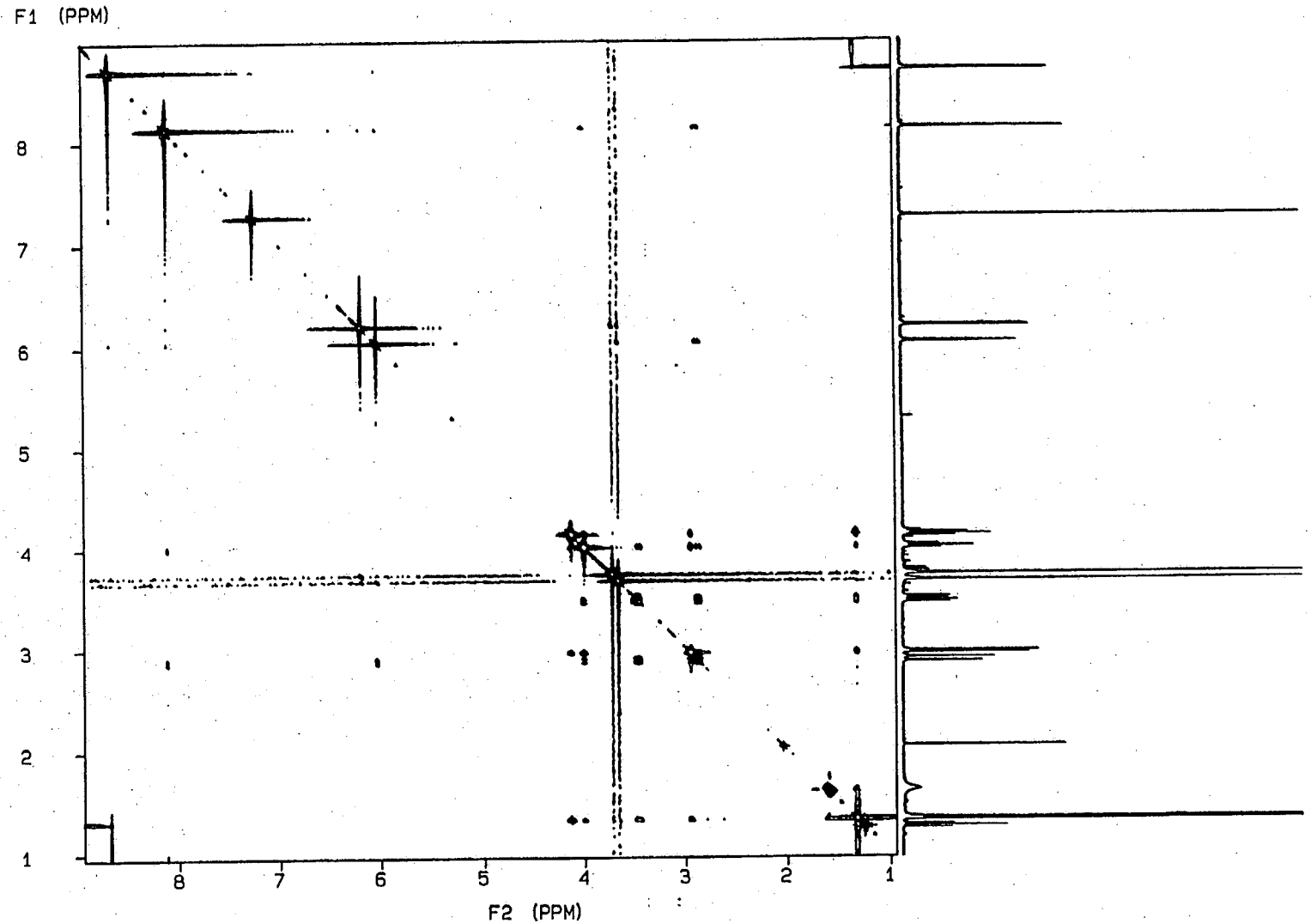

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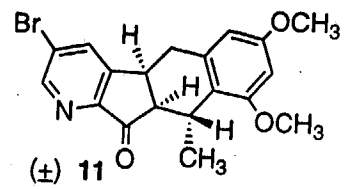

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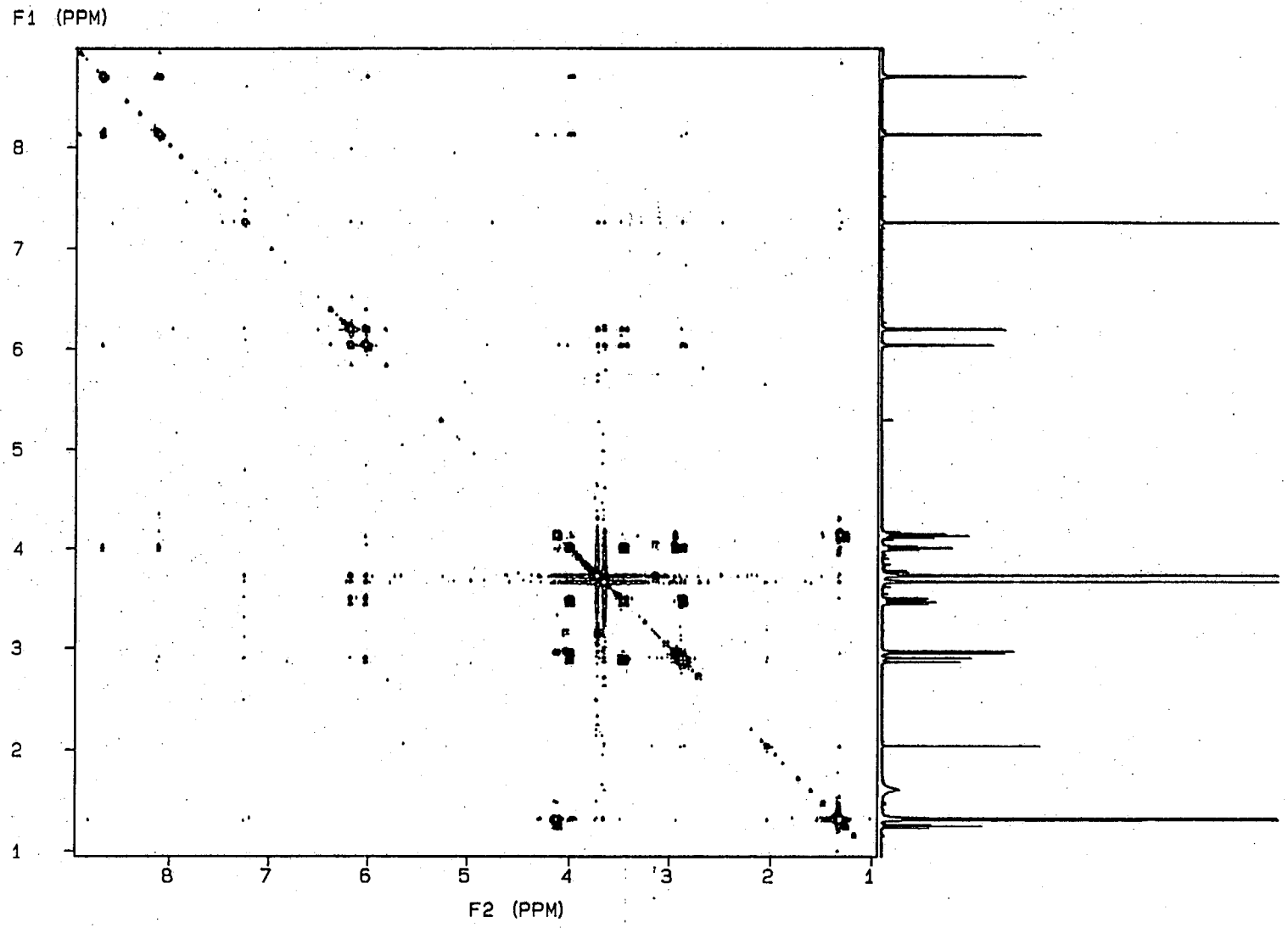

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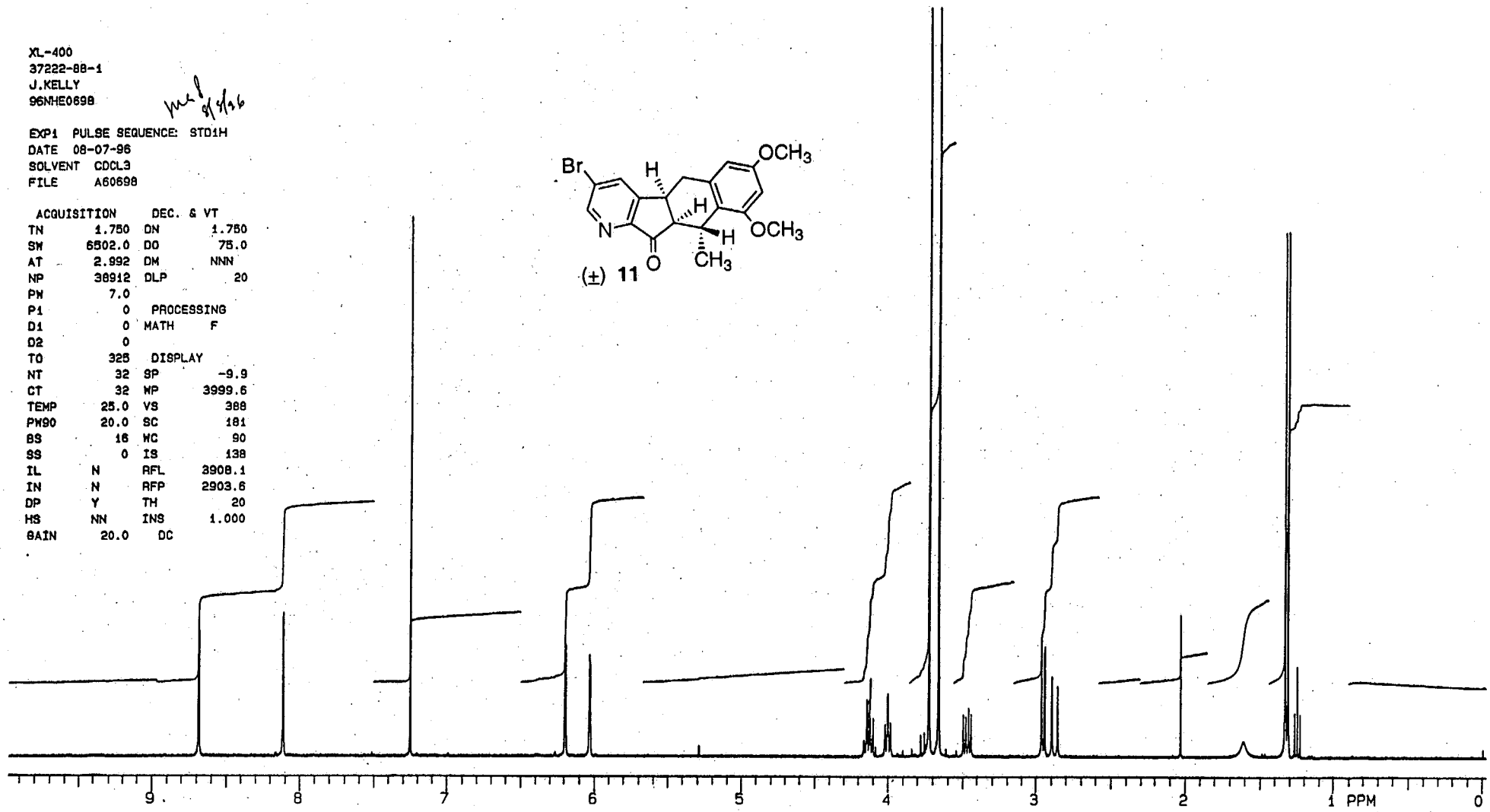
EXP1 PULSE SEQUENCE: STD1H
DATE 08-07-96
SOLVENT CDCL3
FILE A60698

ACQUISITION		DEC. & VT	
TN	1.750	DN	1.750
SW	6502.0	DO	75.0
AT	2.992	DM	NNN
NP	38912	DLP	20
PW	7.0		
P1	0	PROCESSING	
D1	0	MATH	F
D2	0		
TO	325	DISPLAY	
NT	32	SP	-9.9
CT	32	WP	3999.6
TEMP	25.0	VS	388
PW90	20.0	SC	181
BS	16	WC	90
SS	0	IS	138
IL	N	RFL	3908.1
IN	N	RFP	2903.6
DP	Y	TH	20
HS	NN	INS	1.000
GAIN	20.0	DC	

(±) **11**



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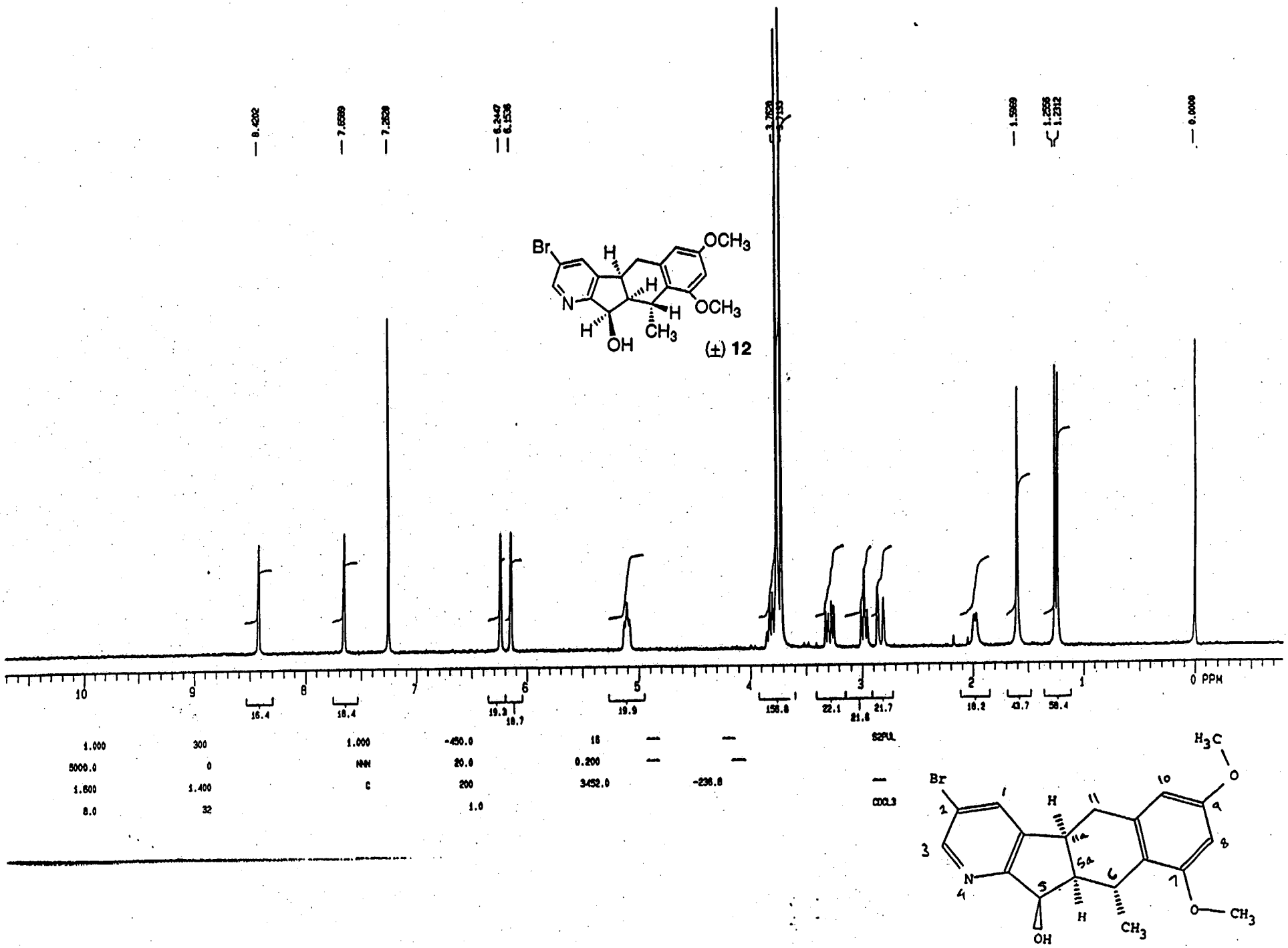

(+) 12

(46)

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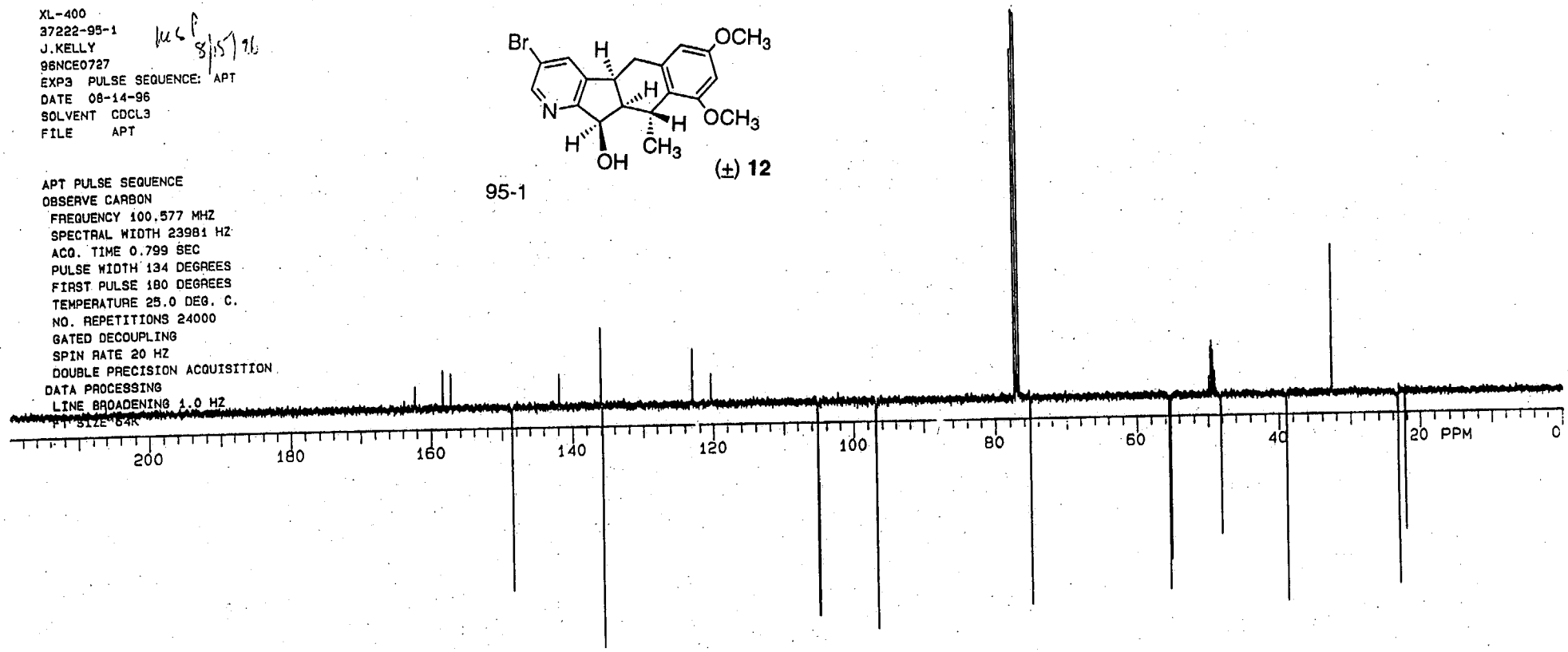
XL-400
37222-95-1
J.KELLY
96NCE0727
EXP3 PULSE SEQUENCE: APT
DATE 08-14-96
SOLVENT CDCL3
FILE APT

MCP 8/15/96

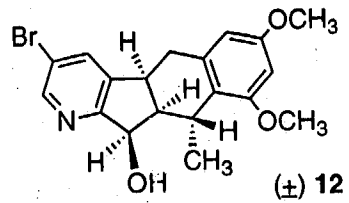
(±) **12**

95-1

APT PULSE SEQUENCE
OBSERVE CARBON
FREQUENCY 100.577 MHZ
SPECTRAL WIDTH 23981 HZ
ACQ. TIME 0.799 SEC
PULSE WIDTH 134 DEGREES
FIRST PULSE 180 DEGREES
TEMPERATURE 25.0 DEG. C.
NO. REPETITIONS 24000
GATED DECOUPLING
SPIN RATE 20 HZ
DOUBLE PRECISION ACQUISITION
DATA PROCESSING
LINE BROADENING 1.0 HZ
FT SIZE 64K



(19)



(±) **12**

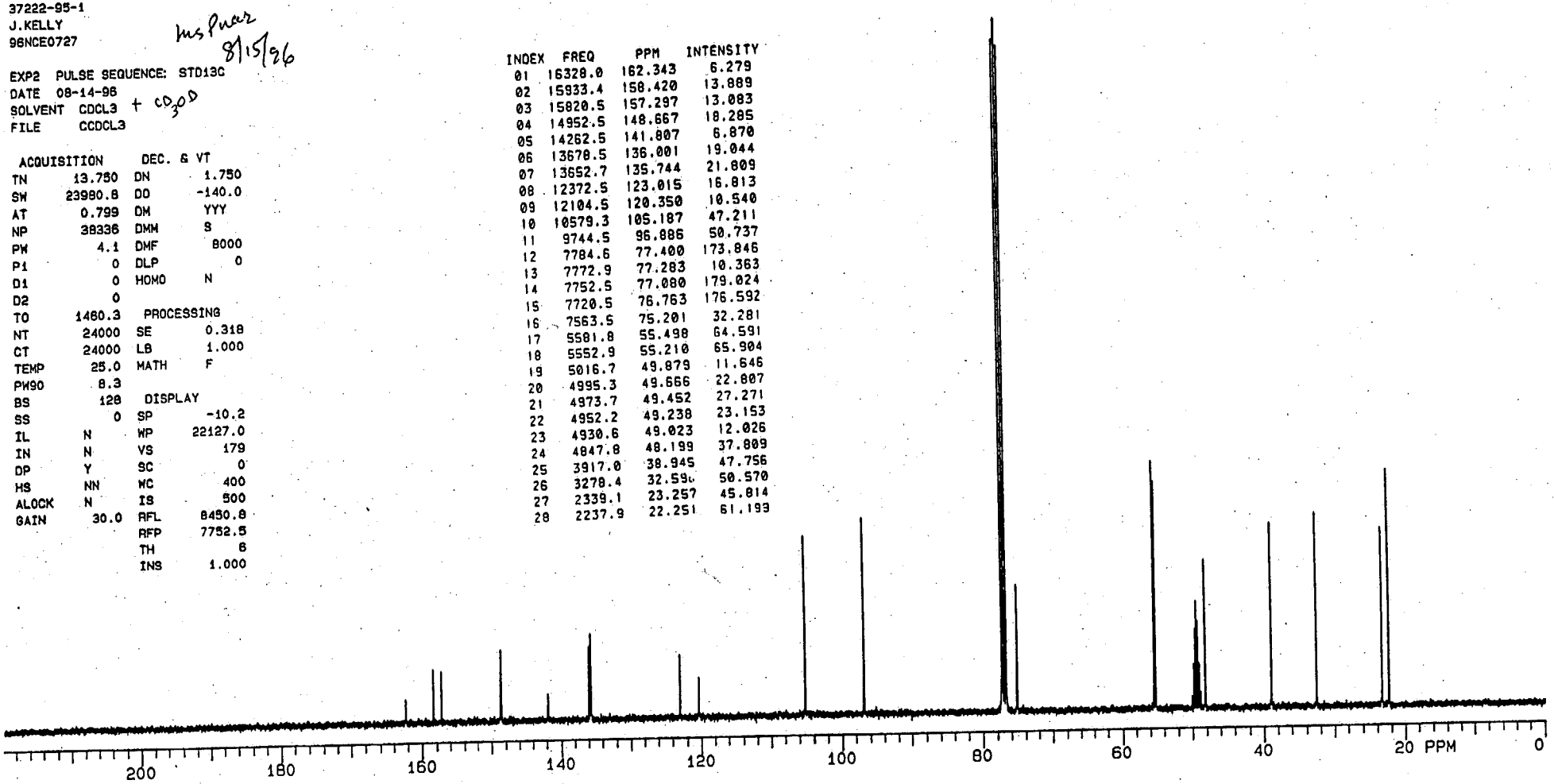
XL-400
37222-95-1
J.KELLY
96NCE0727

Ms Pnar 8/15/96

EXP2 PULSE SEQUENCE: STD13C
DATE 08-14-96
SOLVENT CDCL3 + CD_3OD
FILE CCDCL3

ACQUISITION		DEC. & VT	
TN	13.750	DN	1.750
SW	23980.8	DO	-140.0
AT	0.799	DM	YYY
NP	38336	DMM	S
PW	4.1	DMF	8000
P1	0	DLP	0
D1	0	HOMO	N
D2	0		
TO	1460.3	PROCESSING	
NT	24000	SE	0.318
CT	24000	LB	1.000
TEMP	25.0	MATH	F
PW90	8.3		
BS	128	DISPLAY	
SS	0	SP	-10.2
IL	N	WP	22127.0
IN	N	VS	179
DP	Y	SC	0
HS	NN	WC	400
ALOCK	N	IS	500
GAIN	30.0	RFL	8450.8
		RFP	7752.5
		TH	6
		INS	1.000

INDEX	FREQ	PPM	INTENSITY
01	16328.0	162.343	6.279
02	15933.4	158.420	13.889
03	15820.5	157.297	13.083
04	14952.5	148.667	18.285
05	14262.5	141.807	6.870
06	13678.5	136.001	19.044
07	13652.7	135.744	21.809
08	12372.5	123.015	16.813
09	12104.5	120.350	10.540
10	10579.3	105.187	47.211
11	9744.5	96.886	50.737
12	7784.6	77.400	173.846
13	7772.9	77.283	10.363
14	7752.5	77.080	179.024
15	7720.5	76.763	176.592
16	7563.5	75.201	32.281
17	5581.8	55.498	64.591
18	5552.9	55.210	65.904
19	5016.7	49.879	11.646
20	4995.3	49.666	22.807
21	4973.7	49.452	27.271
22	4952.2	49.238	23.153
23	4930.6	49.023	12.026
24	4847.8	48.199	37.809
25	3917.0	38.945	47.756
26	3278.4	32.59[illegible]	50.570
27	2339.1	23.257	45.814
28	2237.9	22.251	61.193



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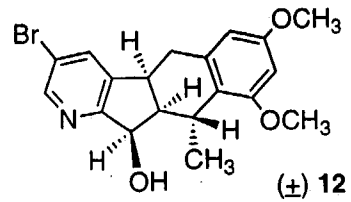


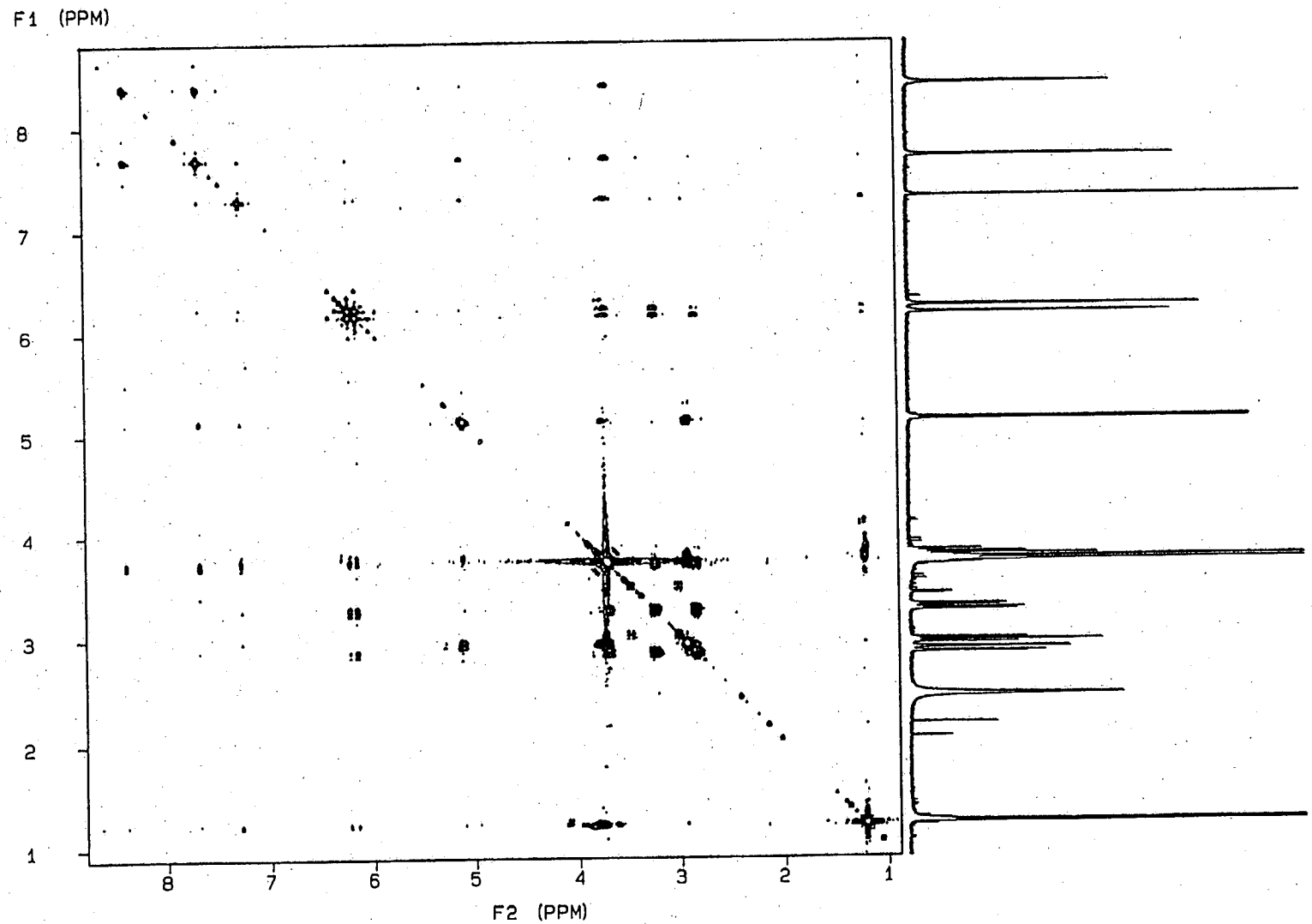
(±) 12

XL-400
37222-95-1
J.KELLY
96NHE0726

hsp 8/15/96

EXP7 PULSE SEQUENCE: COSY
DATE 08-14-96
SOLVENT CDCL3
FILE B60726

ACQUISITION		DEC. & VT	
TN	1.750	DN	1.750
SW	3157.6	DO	75.0
AT	0.324	DM	NNN
NP	2048	DLP	20
PW	25.6		
P1	25.6	PROCESSING	
D1	1.000	RE	0.020
D2	0	FN	2048
TO	3.00E-1	AF	0.081
NT	4	MATH	F
CT	4	FN2	2048
TEMP	25.0	RE2	0.010
PW90	20.0	AF2	0.041
SW2	3157.6		
NI	500	DISPLAY	
D3	0	SP	358.0
SS	2	WP	3157.6
IL	N	VS	50
IN	N	SP2	358.0
DP	N	WP2	3157.6
HS	NN	SC	15
GAIN	12.0	WC	200
		IS	500
		RFL	-358.6
		RFP	0
		TH	7
		SC2	65
		WC2	200
		INS	1.000
		RFL2	-358.6
		RFP2	0
		AI	AV



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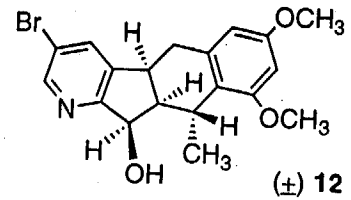


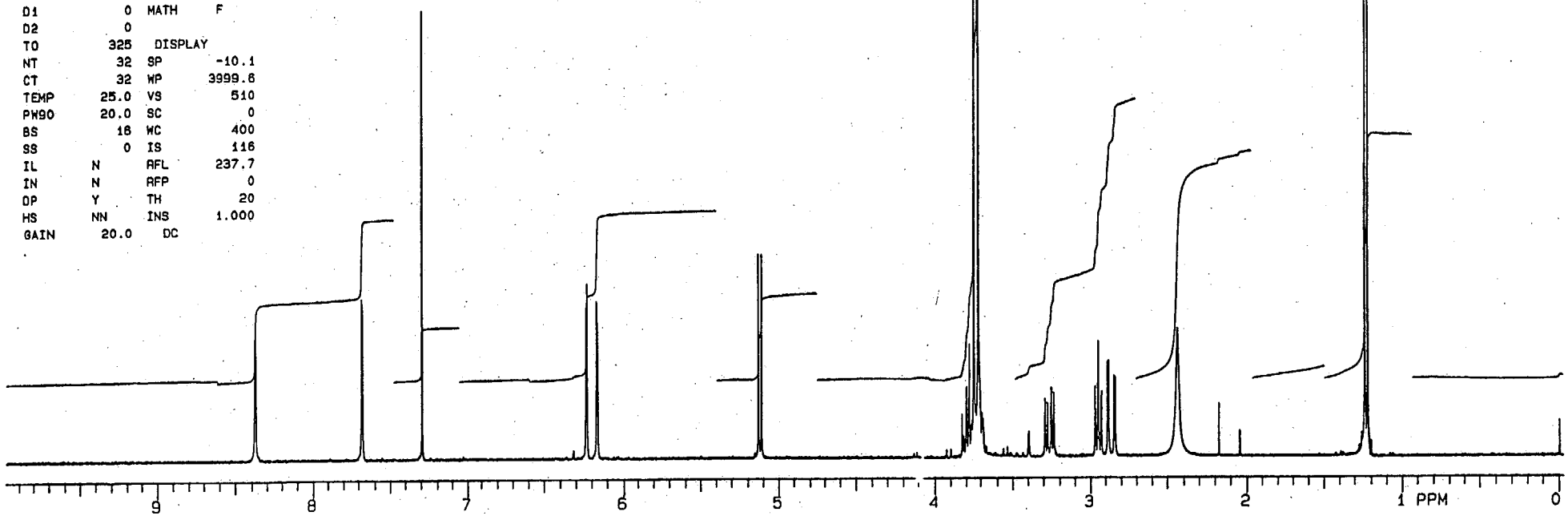
(±) **12**

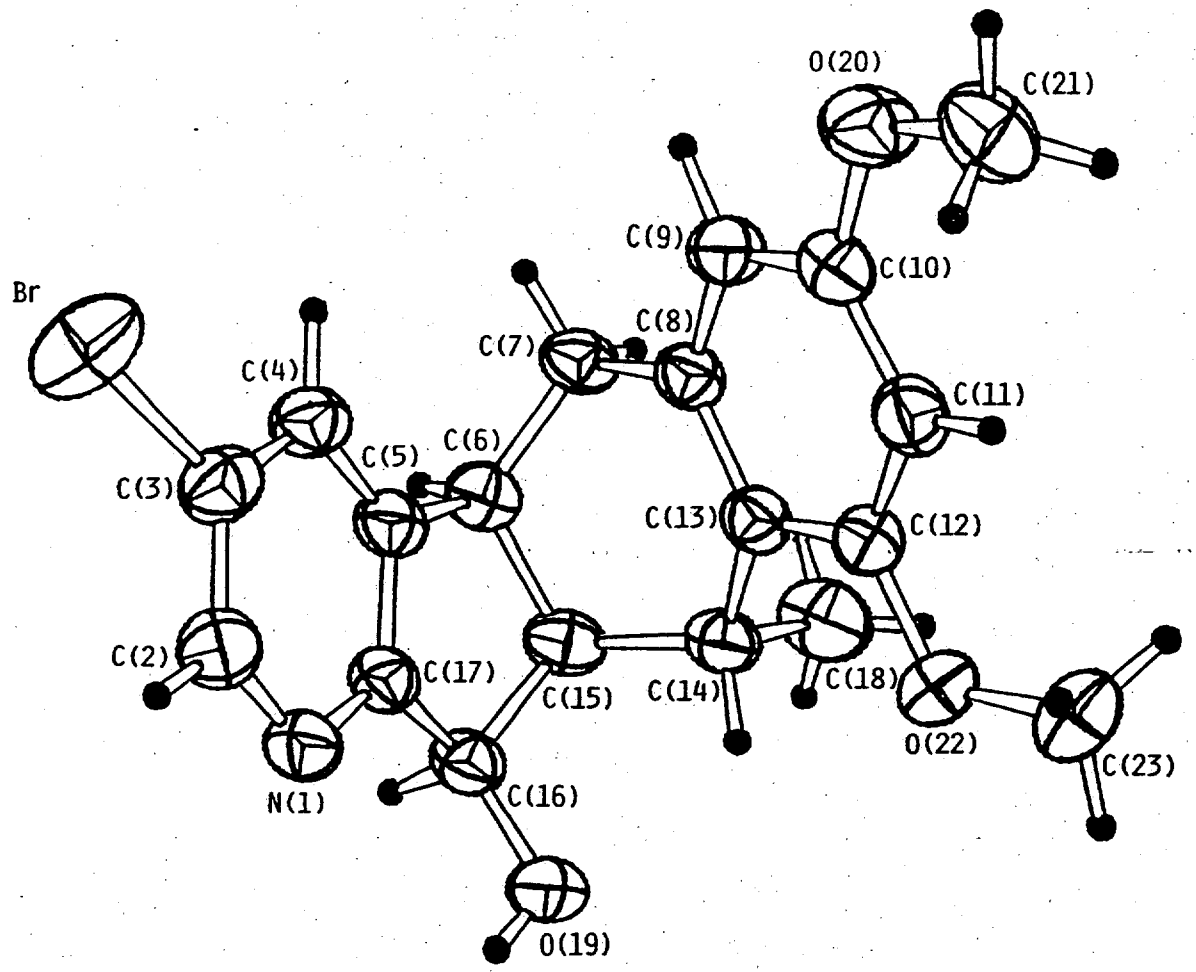
XL-400
37222-95-1
J.KELLY
96NHE0726

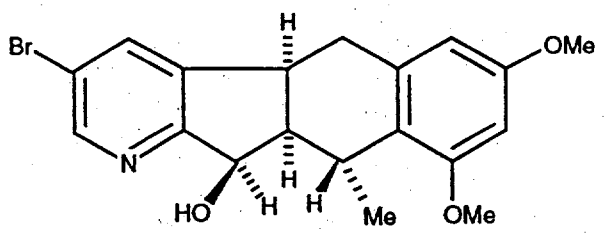
MSP
8/15/96

EXP1 PULSE SEQUENCE: STD1H
DATE 08-14-96
SOLVENT CDCL3
FILE A60726

ACQUISITION		DEC. & VT	
TN	1.750	DN	1.750
SW	5000.0	DO	75.0
AT	2.995	DM	NNN
NP	29952	DLP	20
PW	7.0		
P1	0	PROCESSING	
D1	0	MATH	F
D2	0		
TO	325	DISPLAY	
NT	32	SP	-10.1
CT	32	WP	3999.6
TEMP	25.0	VS	510
PW90	20.0	SC	0
BS	16	WC	400
SS	0	IS	116
IL	N	RFL	237.7
IN	N	RFP	0
DP	Y	TH	20
HS	NN	INS	1.000
GAIN	20.0	DC	







Sample: 37222-95-1

(Relative stereochemistry)

Figure 1. ORTEP diagram (50% probability ellipsoids) showing the crystallographic atom numbering scheme and sold-state conformation of one enantiomer; small filled circles represent hydrogen atoms.

Table 1. Crystallographic Data[a]

Molecular formula	$C_{19}H_{20}BrNO_3$
Formula weight	390.29
Crystal system	triclinic
Space group	$P1(C_i^1)$ - No.2
a(Å)	9.104(2)
b(Å)	11.474(2)
c(Å)	8.836(2)
α(°)	97.88(1)
β(°)	109.69(1)
γ(°)	85.61(1)
V(Å^3)	860.4(6)
Z	2
$D_{calcd.}$ (g cm^{-3})	1.506
Radiation (λ, Å)	Cu-$K\alpha$ (1.5418)
Absorption coefficient, μ(cm^{-1})	34.0
Temp. (°C)	25
Crystal dimensions (mm)	0.10 x 0.16 x 0.36
$T_{max.}$:$T_{min.}$(relative)	1.00:0.83
Scan type	ω-2θ
Scanwidth (°)	0.80 + 0.14tanθ
$\theta_{max.}$(°)	75
Intensity control refls.;	1 -1 -3, 1 -1 2, 2 2 1, 1 2 -3;
variation; repeat time (hr)	<1.0% ; 2
Total no. of refls. (+h,±k,±l) recorded	3707
No. of unique refls. recorded	3524
R_{merge} (on I)	0.022
No. of refls. retained [$I > 3.0\sigma(I)$]	3012
No. of parameters refined	298
Extinction correction	5.8(3) x 10^{-6}
R (R_w)[b]	0.036 (0.050)
Goodness-of-fit[c]	2.06
Max. shift:esd in final least-squares cycle	0.02
Final Δρ(e/Å^3) max.;min.	0.40; -0.56

Table 1 (continued)

[a]Oscillation and Weissenberg photographs yielded preliminary unit-cell and space group information. An Enraf-Nonius CAD-4 diffractometer (Cu-$K\alpha$ radiation, graphite monochromator) was used for all other measurements. Refined unit-cell parameters were derived from the diffractometer setting angles for 25 reflections ($36° < \theta < 40°$) widely separated in reciprocal space. Intensity data were corrected for the usual Lorentz and polarization effects. An empirical absorption correction, based on the ϕ-dependency of the intensities of several reflections with χ ca. 90°, was also applied. Laue symmetry indicated that the crystals were triclinic, space group $P1$ or $P\bar{1}$; the latter was assumed at the outset and shown to be correct by the structure solution and refinement.

The crystal structure was solved routinely by direct methods (MULTAN11/82). Initial co-ordinates for all non-hydrogen atoms were derived from an E-map. Positional and thermal parameters of these atoms (first isotropicand then anisotropic) were adjusted by means of several rounds of full-matrix least-squares calculations. Hydrogen atoms were located in a difference Fourier synthesis and their positional and isotropic thermal paramers were also refined in the subsequent iterations. An extinction correction was included as a variable during the later cycles. A final difference Fourier synthesis contained no unusual features.

Crystallographic calculations were performed on PDP11/44 and MicroVAX computers by use of the Enraf-Nonius Structure Determination Package (SDP). For all structure-factor calculations, neutral atom scattering factors and their anomalous dispersion corrections were taken from *International Tables for X-Ray Crystallography*, vol. IV, The Kynoch Press, Birmingham, England, 1974.

[b]$R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2}$; $\Sigma w\Delta^2[w = 1/\sigma^2(|F_o|), \Delta = (|F_o| - |F_c|)]$ was minimized.

[c]Goodness-of-fit = $[\Sigma w\Delta^2/(N_{\text{observations}} - N_{\text{parameters}})]^{1/2}$.

Table 2. Non-hydrogen Atom Fractional Coordinates and Equivalent Isotropic Thermal Parameters, with Estimated Standard Deviations in Parentheses

Atom	*x*	*y*	*z*	B_{eq}(Å^2)
N(1)	-0.0883(2)	0.0651(2)	0.2909(2)	3.60(3)
C(2)	-0.2207(3)	0.1035(2)	0.1852(3)	3.88(4)
C(3)	-0.2217(3)	0.1623(2)	0.0582(2)	3.70(4)
C(4)	-0.0837(3)	0.1852(2)	0.0352(2)	3.53(4)
C(5)	0.0526(2)	0.1449(2)	0.1429(2)	3.11(4)
C(6)	0.2198(2)	0.1516(2)	0.1519(2)	3.32(4)
C(7)	0.2595(2)	0.2711(2)	0.1181(2)	3.37(4)
C(8)	0.2319(2)	0.3669(2)	0.2407(2)	2.86(3)
C(9)	0.1579(2)	0.4734(2)	0.1945(2)	3.18(4)
C(10)	0.1288(2)	0.5578(2)	0.3097(2)	3.21(4)
C(11)	0.1744(2)	0.5384(2)	0.4707(2)	3.35(4)
C(12)	0.2507(2)	0.4320(2)	0.5149(2)	3.19(4)
C(13)	0.2783(2)	0.3438(2)	0.4014(2)	2.89(3)
C(14)	0.3699(2)	0.2310(2)	0.4508(2)	3.28(4)
C(15)	0.3143(2)	0.1214(2)	0.3252(2)	3.43(4)
C(16)	0.2029(3)	0.0443(2)	0.3664(2)	3.44(4)
C(17)	0.0438(2)	0.0859(2)	0.2679(2)	3.11(4)
C(18)	0.5433(3)	0.2496(2)	0.4847(4)	5.06(6)
O(19)	0.2326(2)	0.0512(1)	0.5345(2)	4.37(3)
O(20)	0.0528(2)	0.6593(1)	0.2515(2)	4.36(4)
C(21)	0.0148(3)	0.7478(2)	0.3624(3)	4.60(5)
O(22)	0.3045(2)	0.4071(1)	0.6724(2)	4.32(4)
C(23)	0.3002(4)	0.4993(3)	0.7966(3)	5.47(7)
Br	-0.41535(3)	0.20792(3)	-0.08918(3)	5.79(1)

Table 3. Anisotropic Temperature Factor Parameters,[a] with Estimated Standard Deviations in Parentheses

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	0.056(1)	0.045(1)	0.039(1)	-0.010(1)	0.018(1)	0.006(1)
C(2)	0.053(1)	0.054(1)	0.043(1)	-0.007(1)	0.020(1)	0.003(1)
C(3)	0.054(1)	0.048(1)	0.036(1)	0.002(1)	0.014(1)	0.003(1)
C(4)	0.058(1)	0.042(1)	0.036(1)	0.000(1)	0.019(1)	0.006(1)
C(5)	0.055(1)	0.031(1)	0.036(1)	-0.002(1)	0.020(1)	0.002(1)
C(6)	0.053(1)	0.037(1)	0.043(1)	0.003(1)	0.025(1)	0.005(1)
C(7)	0.055(1)	0.040(1)	0.043(1)	0.000(1)	0.027(1)	0.009(1)
C(8)	0.040(1)	0.035(1)	0.037(1)	-0.004(1)	0.016(1)	0.007(1)
C(9)	0.046(1)	0.039(1)	0.038(1)	0.000(1)	0.015(1)	0.009(1)
C(10)	0.043(1)	0.034(1)	0.047(1)	-0.002(1)	0.015(1)	0.008(1)
C(11)	0.049(1)	0.038(1)	0.043(1)	-0.007(1)	0.019(1)	0.000(1)
C(12)	0.044(1)	0.043(1)	0.035(1)	-0.012(1)	0.011(1)	0.006(1)
C(13)	0.035(1)	0.037(1)	0.039(1)	-0.005(1)	0.012(1)	0.008(1)
C(14)	0.039(1)	0.042(1)	0.046(1)	-0.001(1)	0.011(1)	0.016(1)
C(15)	0.048(1)	0.039(1)	0.051(1)	0.009(1)	0.024(1)	0.014(1)
C(16)	0.054(1)	0.033(1)	0.045(1)	0.000(1)	0.017(1)	0.011(1)
C(17)	0.052(1)	0.031(1)	0.037(1)	-0.005(1)	0.016(1)	0.003(1)
C(18)	0.039(1)	0.071(1)	0.082(1)	0.000(1)	0.012(1)	0.029(1)
O(19)	0.060(1)	0.063(1)	0.045(1)	-0.015(1)	0.012(1)	0.022(1)
O(20)	0.073(1)	0.039(1)	0.055(1)	0.014(1)	0.023(1)	0.011(1)
C(21)	0.068(1)	0.038(1)	0.074(1)	0.005(1)	0.034(1)	0.004(1)
O(22)	0.074(1)	0.054(1)	0.033(1)	-0.011(1)	0.011(1)	0.006(1)
C(23)	0.096(2)	0.069(1)	0.038(1)	-0.014(1)	0.015(1)	-0.001(1)
Br	0.0580(1)	0.1056(2)	0.0517(1)	0.0168(2)	0.0133(1)	0.0163(1)

[a]In the form: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$

Table 4. Hydrogen Atom Fractional Coordinates,[a] Isotropic Thermal Parameters, and Bonded Distances, with Estimated Standard Deviations in Parentheses

Atom	*x*	*y*	*z*	*B*(Å^2)	*d*(Å)
H(2)	-0.314(3)	0.090(2)	0.201(3)	2.6(6)	0.93(3)
H(4)	-0.084(3)	0.229(2)	-0.051(3)	2.0(5)	0.97(3)
H(6)	0.235(3)	0.093(2)	0.076(3)	1.1(4)	0.92(2)
H(7A)	0.200(3)	0.287(2)	0.009(3)	2.0(5)	0.97(2)
H(7B)	0.380(3)	0.270(2)	0.123(3)	2.1(5)	1.08(3)
H(9)	0.126(3)	0.492(2)	0.084(3)	2.0(5)	0.97(3)
H(11)	0.160(3)	0.594(2)	0.546(3)	2.0(5)	0.89(3)
H(14)	0.355(3)	0.210(2)	0.549(3)	2.2(5)	0.99(3)
H(15)	0.413(3)	0.073(2)	0.323(3)	1.7(5)	1.02(3)
H(16)	0.212(3)	-0.042(2)	0.320(3)	2.1(5)	1.03(2)
H(18A)	0.576(3)	0.312(3)	0.565(3)	3.7(7)	0.93(3)
H(18B)	0.597(4)	0.178(3)	0.522(4)	5.0(8)	0.96(3)
H(18C)	0.558(4)	0.258(3)	0.383(4)	4.6(8)	0.97(4)
H(19)	0.172(3)	0.016(2)	0.551(3)	1.9(5)	0.77(3)
H(21A)	-0.046(4)	0.804(3)	0.300(4)	5.0(9)	0.93(3)
H(21B)	-0.052(3)	0.717(2)	0.414(3)	2.2(5)	0.98(3)
H(21C)	0.107(3)	0.781(3)	0.449(3)	3.3(6)	0.99(2)
H(23A)	0.340(4)	0.461(3)	0.891(4)	4.2(7)	0.94(3)
H(23B)	0.356(4)	0.569(3)	0.796(4)	4.5(8)	0.98(4)
H(23C)	0.181(5)	0.534(3)	0.787(5)	6.3(10)	1.11(4)

[a]Hydrogen atoms bear the same labels as the atoms to which they are bonded.

Table 5. Interatomic Distances (Å) and Angles (deg.), with Estimated Standard Deviations in Parentheses

(a) Bond Lengths

N(1)-C(2)	1.339(3)	C(10)-C(11)	1.386(3)
N(1)-C(17)	1.329(3)	C(10)-O(20)	1.379(2)
C(2)-C(3)	1.384(4)	C(11)-C(12)	1.394(3)
C(3)-C(4)	1.388(4)	C(12)-C(13)	1.395(3)
C(3)-Br	1.891(2)	C(12)-O(22)	1.374(2)
C(4)-C(5)	1.377(3)	C(13)-C(14)	1.518(3)
C(5)-C(6)	1.505(3)	C(14)-C(15)	1.551(3)
C(5)-C(17)	1.399(3)	C(14)-C(18)	1.531(3)
C(6)-C(7)	1.536(3)	C(15)-C(16)	1.555(4)
C(6)-C(15)	1.555(2)	C(16)-C(17)	1.497(3)
C(7)-C(8)	1.503(3)	C(16)-O(19)	1.409(3)
C(8)-C(9)	1.393(3)	O(20)-C(21)	1.419(3)
C(8)-C(13)	1.395(3)	O(22)-C(23)	1.425(3)
C(9)-C(10)	1.384(3)		

(b) Bond Angles

C(2)-N(1)-C(17)	116.6(2)	C(11)-C(12)-C(13)	121.9(2)
N(1)-C(2)-C(3)	122.3(3)	C(11)-C(12)-O(22)	122.5(2)
C(2)-C(3)-C(4)	121.1(2)	C(13)-C(12)-O(22)	115.5(2)
C(2)-C(3)-Br	119.0(2)	C(8)-C(13)-C(12)	117.7(2)
C(4)-C(3)-Br	119.8(2)	C(8)-C(13)-C(14)	120.1(2)
C(3)-C(4)-C(5)	116.7(2)	C(12)-C(13)-C(14)	121.9(2)
C(4)-C(5)-C(6)	130.9(2)	C(13)-C(14)-C(15)	113.9(1)
C(4)-C(5)-C(17)	118.7(2)	C(13)-C(13)-C(18)	108.9(2)
C(6)-C(5)-C(17)	110.4(1)	C(15)-C(14)-C(18)	110.6(2)
C(5)-C(6)-C(7)	113.6(2)	C(6)-C(15)-C(14)	113.8(2)
C(5)-C(6)-C(15)	103.6(2)	C(6)-C(15)-C(16)	103.9(1)
C(7)-C(6)-C(15)	113.2(1)	C(14)-C(15)-C(16)	113.6(2)
C(6)-C(7)-C(8)	109.8(2)	C(15)-C(16)-C(17)	103.5(2)
C(7)-C(8)-C(9)	121.1(2)	C(15)-C(16)-O(19)	112.0(2)
C(7)-C(8)-C(13)	117.6(2)	C(17)-C(16)-O(19)	116.1(2)
C(9)-C(8)-C(13)	121.2(2)	N(1)-C(17)-C(5)	124.5(2)

Table 5 (continued)

C(8)-C(9)-C(10)	119.5(2)	N(1)-C(17)-C(16)	124.9(2)
C(9)-C(10)-C(11)	120.9(2)	C(5)-C(17)-C(16)	110.5(2)
C(9)-C(10)-O(20)	115.1(2)	C(10)-O(20)-C(21)	118.0(2)
C(11)-C(10)-O(20)	124.0(2)	C(12)-O(22)-C(23)	118.2(2)
C(10)-C(11)-C(12)	118.7(2)		

(c) Torsion Angles[a]

C(17)-N(1)-C(2)-C(3)	-0.2(3)	C(8)-C(9)-C(10)-C(11)	1.0(3)
C(2)-N(1)-C(17)-C(5)	0.4(3)	C(8)-C(9)-C(10)-O(20)	-179.0(2)
C(2)-N(1)-C(17)-C(16)	-176.3(2)	C(9)-C(10)-C(11)-C(12)	0.1(3)
N(1)-C(2)-C(3)-C(4)	-0.5(4)	O(20)-C(10)-C(11)-C(12)	180.0(3)
N(1)-C(2)-C(3)-Br	177.7(2)	C(9)-C(10)-O(20)-C(21)	178.0(2)
C(2)-C(3)-C(4)-C(5)	1.1(3)	C(11)-C(10)-O(20)-C(21)	-2.0(3)
Br-C(3)-C(4)-C(5)	-177.2(2)	C(10)-C(11)-C(12)-C(13)	-1.8(3)
C(3)-C(4)-C(5)-C(6)	178.5(2)	C(10)-C(11)-C(12)-O(22)	177.8(2)
C(3)-C(4)-C(5)-C(17)	-0.9(3)	C(11)-C(12)-C(13)-C(8)	2.3(3)
C(4)-C(5)-C(6)-C(7)	41.7(3)	C(11)-C(12)-C(13)-C(14)	176.3(2)
C(4)-C(5)-C(6)-C(15)	164.9(2)	O(22)-C(12)-C(13)-C(8)	-177.3(2)
C(17)-C(5)-C(6)-C(7)	-138.9(2)	O(22)-C(12)-C(13)-C(14)	-3.4(3)
C(17)-C(5)-C(6)-C(15)	-15.7(2)	C(11)-C(12)-O(22)-C(23)	-8.5(3)
C(4)-C(5)-C(17)-N(1)	0.2(3)	C(13)-C(12)-O(22)-C(23)	171.2(2)
C(4)-C(5)-C(17)-C(16)	177.3(2)	C(8)-C(13)-C(14)-C(15)	-39.4(3)
C(6)-C(5)-C(17)-N(1)	-179.3(2)	C(8)-C(13)-C(14)-C(18)	84.6(2)
C(6)-C(5)-C(17)-C(16)	-2.2(2)	C(12)-C(13)-C(14)-C(15)	146.9(2)
C(5)-C(6)-C(7)-C(8)	60.4(2)	C(12)-C(13)-C(14)-C(18)	-89.2(2)
C(15)-C(6)-C(7)-C(8)	-57.4(2)	C(13)-C(14)-C(15)-C(6)	20.4(2)
C(5)-C(6)-C(15)-C(14)	-97.6(2)	C(13)-C(14)-C(15)-C(16)	-98.2(2)
C(5)-C(6)-C(15)-C(16)	26.4(2)	C(18)-C(14)-C(15)-C(6)	-102.6(2)
C(7)-C(6)-C(15)-C(14)	25.9(2)	C(18)-C(14)-C(15)-C(16)	138.7(2)
C(7)-C(6)-C(15)-C(16)	149.9(2)	C(6)-C(15)-C(16)-C(17)	-27.7(2)
C(6)-C(7)-C(8)-C(9)	-136.6(2)	C(6)-C(15)-C(16)-O(19)	-153.5(2)
C(6)-C(7)-C(8)-C(13)	41.3(2)	C(14)-C(15)-C(16)-C(17)	96.6(2)
C(7)-C(8)-C(9)-C(10)	177.4(2)	C(14)-C(15)-C(16)-O(19)	-29.3(3)

Table 5 (continued)

C(13)-C(8)-C(9)-C(10)	-0.4(3)	C(15)-C(16)-C(17)-N(1)	-163.8(2)
C(7)-C(8)-C(13)-C(12)	-179.1(2)	C(15)-C(16)-C(17)-C(5)	19.1(2)
C(7)-C(8)-C(13)-C(14)	6.9(3)	O(19)-C(16)-C(17)-N(1)	-40.6(3)
C(9)-C(8)-C(13)-C(12)	-1.2(3)	O(19)-C(16)-C(17)-C(5)	142.3(2)
C(9)-C(8)-C(13)-C(14)	-175.2(2)		

(d) Hydrogen-bonded Distance[b] (Donor...Acceptor)

O(19)...N(1)[I]	2.844(3)

[a]The torsion angle A-B-C-D is defined as positive if, when viewed along the B-C bond, atom A must be rotated clockwise to eclipse atom D.

[b]The Roman numeral superscript refers to the following transformation of the fractional atomic coordinates listed in Table 2:

I $-x, -y, 1-z$.