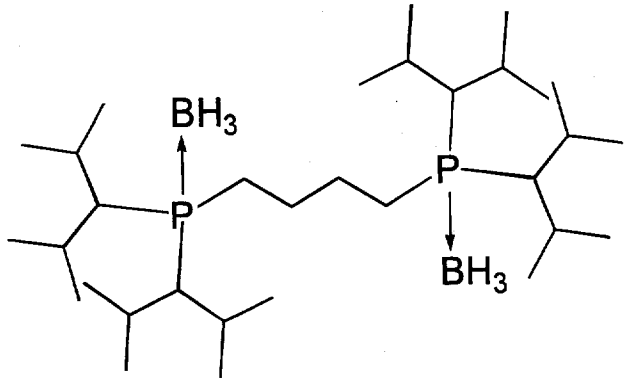
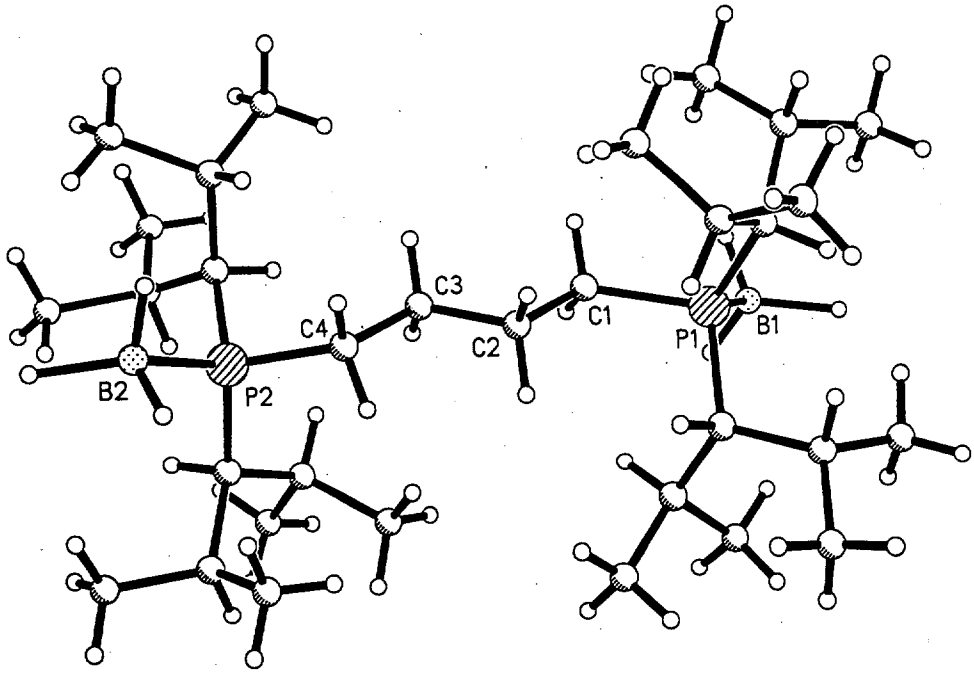
X-ray crystallographic data for phosphine-borane-complex **23**·2BH_3





ıtal data of the crystal structure determination of *graf1*.

Data:

colour	Block, colourless
size	0.3 x 0.3 x 0.25 mm
system	Orthorhombic
oup	Pbca, Z = 8
l dimensions	a = 1800.3(1) pm $\alpha = 90^{o}$
	b = 1447.4(1) pm $\beta = 90^{o}$
	c = 2838.4(1) pm $\gamma = 90^{o}$
	$7396.2(7)*10^{-30}$ m^3
ermination	25 reflections 25 to 35^{o} θ
formula	$C_{32}H_{74}B_2P_2$
weight	542.47 g/mol
	2448
(calculated)	0.974 Mg/m^3
ion coefficient	1.165 mm^{-1}

llection:

ometer type	Enraf Nonius CAD4
gth	CuKα (154.178pm)
ture	213(2) K
for data collection	3.11 to 59.90^{o}
anges	-20<=h<=0, 0<=k<=16, -31<=l<=0
thod	ω-scans
gle	$(0.8+0.15tg\theta)^{o}$
me	max 30 sec.
reflections and decay	3 refl. all 60 min., 0%
llection software	CAD4 EXPRESS
finement software	CAD4 EXPRESS
duction software	XCAD4 (Harms, 1997)

Solution and refinement:

Reflections collected	5470
Independent reflections	5470 [R_{int} = 0.0000]
Observed reflections	4136 [I>2σ(I)]
Reflections used for refinement	5470
Extinction coefficient	X = 0.00022(4)
Extinction correction formula	$F_c^* = F_c * k * [(1 + 0.001 * X * F_c^2 * \lambda^3)/\sin 2\theta]^{-1/4}$
k = overall scale factor	
Absorption correction	Empirical
Max. and min. transmission	0.6008 und 0.5303
Largest diff. peak and hole	0.316 and $-0.207*10^{30}$ e/m^{-3}
Solution	Direct methods / difference fourier
Refinement	Full-matrix refinement at F^2
Treatment of hydrogen atoms	C-H Calculated positions, B-H refined
Programs used	SHELXS-97 (Sheldrick, 1990)
	SHELXL-97 (Sheldrick, 1997)
	SHELXTL
Data / restraints / parameters	5470 / 0 / 360
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0635P)^2+4.3538P]$; $P=(F_o^2+2F_c^2)/3$
Goodness-of-fit on F^2	1.025
R index (all data)	wR_2 = 0.1341
R index conventional [I>2σ(I)]	R = 0.0491
Completeness to 2θ = 59.90	90.5%

Table 1. Atomic coordinates and equivalent isotropic displacement parameters [$Å^2$] for GRAF1. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U(eq)
P(1)	0.21432(4)	0.30237(4)	0.17262(2)	0.03342(19)
P(2)	0.23123(4)	0.00116(4)	-0.02357(2)	0.03271(19)
B(1)	0.2287(2)	0.4342(2)	0.16262(13)	0.0513(9)
B(2)	0.2065(2)	-0.1285(2)	-0.02786(13)	0.0458(8)
C(1)	0.19557(16)	0.25494(18)	0.11379(8)	0.0389(6)
C(2)	0.21290(15)	0.15397(17)	0.10246(8)	0.0378(6)
C(3)	0.19820(15)	0.13374(17)	0.05066(8)	0.0349(6)
C(4)	0.21649(15)	0.03350(18)	0.03799(8)	0.0359(6)
C(5)	0.29855(14)	0.23936(18)	0.19272(9)	0.0375(6)
C(6)	0.36423(16)	0.2589(2)	0.15860(11)	0.0485(7)
C(7)	0.41349(17)	0.1742(2)	0.15141(12)	0.0619(9)
C(8)	0.4134(2)	0.3422(3)	0.17220(15)	0.0819(12)
C(9)	0.31206(10)	0.24410(12)	0.24717(6)	0.0715(10)
C(10)	0.31692(10)	0.34202(12)	0.26834(6)	0.0690(10)
C(11)	0.36872(10)	0.17560(12)	0.26587(6)	0.0570(18)
C(11A)	0.30963(10)	0.17839(12)	0.27407(6)	0.092(5)
C(12)	0.13919(10)	0.26797(12)	0.21354(6)	0.0383(6)
C(13)	0.12396(16)	0.16157(19)	0.21585(10)	0.0470(7)
C(14)	0.1045(2)	0.1328(2)	0.26644(12)	0.0686(10)
C(15)	0.06387(18)	0.1260(2)	0.18228(13)	0.0677(10)
C(16)	0.06714(16)	0.3270(2)	0.21304(10)	0.0503(7)
C(17)	0.03265(19)	0.3474(3)	0.16490(12)	0.0719(11)
C(18)	0.07702(19)	0.4186(2)	0.23958(11)	0.0618(9)
C(19)	0.32694(15)	0.03359(17)	-0.04168(9)	0.0377(6)
C(20)	0.39057(17)	-0.0360(2)	-0.02934(11)	0.0519(8)
C(21)	0.3961(2)	-0.1119(2)	-0.06551(14)	0.0775(11)
C(22)	0.39131(17)	-0.0755(2)	0.02057(11)	0.0585(9)
C(23)	0.34895(15)	0.13543(18)	-0.03207(9)	0.0404(6)
C(24)	0.40130(17)	0.1704(2)	-0.07080(11)	0.0557(8)
C(25)	0.38442(16)	0.1531(2)	0.01601(11)	0.0493(7)
C(26)	0.16785(15)	0.07838(17)	-0.05734(8)	0.0345(6)
C(27)	0.19090(18)	0.09221(19)	-0.10950(9)	0.0464(7)
C(28)	0.1459(2)	0.1664(2)	-0.13437(11)	0.0625(9)
C(29)	0.1973(2)	0.0050(2)	-0.13979(10)	0.0593(9)
C(30)	0.08548(15)	0.05282(19)	-0.04504(10)	0.0434(7)
C(31)	0.03612(17)	0.1385(2)	-0.03992(11)	0.0537(8)
C(32)	0.04798(18)	-0.0170(2)	-0.07809(12)	0.0612(9)

Table 2. Bond lengths [Å] and angles [°] for GRAF1.

P(1)-C(1)	1.837(2)	P(1)-C(12)	1.8512(19)
P(1)-C(5)	1.859(3)	P(1)-B(1)	1.946(3)
P(2)-C(4)	1.828(2)	P(2)-C(19)	1.858(3)
P(2)-C(26)	1.863(3)	P(2)-B(2)	1.932(3)
B(1)-H(1)	1.43(3)	B(1)-H(2)	1.08(3)
B(1)-H(3)	1.10(3)	B(2)-H(4)	1.48(2)
B(2)-H(5)	1.08(3)	B(2)-H(6)	1.09(3)
C(1)-C(2)	1.529(3)	C(1)-H(1A)	0.9800
C(1)-H(1B)	0.9800	C(2)-C(3)	1.522(3)
C(2)-H(2A)	0.9800	C(2)-H(2B)	0.9800
C(3)-C(4)	1.531(3)	C(3)-H(3A)	0.9800
C(3)-H(3B)	0.9800	C(4)-H(4A)	0.9800
C(4)-H(4B)	0.9800	C(5)-C(6)	1.555(4)
C(5)-C(9)	1.566(3)	C(5)-H(5A)	0.9900
C(6)-C(7)	1.527(4)	C(6)-C(8)	1.545(4)
C(6)-H(6A)	0.9900	C(7)-H(7A)	0.9700
C(7)-H(7B)	0.9700	C(7)-H(7C)	0.9700
C(8)-H(8A)	0.9700	C(8)-H(8B)	0.9700
C(8)-H(8C)	0.9700	C(9)-C(11A)	1.2204
C(9)-C(11)	1.5183	C(9)-C(10)	1.5419
C(9)-H(9A)	0.9900	C(9)-H(9B)	0.9900
C(10)-H(10A)	0.9700	C(10)-H(10B)	0.9700
C(10)-H(10C)	0.9700	C(11)-H(11A)	0.9700
C(11)-H(11B)	0.9700	C(11)-H(11C)	0.9700
C(11A)-H(11D)	0.9700	C(11A)-H(11E)	0.9700
C(11A)-H(11F)	0.9700	C(12)-C(16)	1.553(3)
C(12)-C(13)	1.566(3)	C(12)-H(12A)	0.9900
C(13)-C(15)	1.530(4)	C(13)-C(14)	1.536(4)
C(13)-H(13A)	0.9900	C(14)-H(14A)	0.9700
C(14)-H(14B)	0.9700	C(14)-H(14C)	0.9700
C(15)-H(15A)	0.9700	C(15)-H(15B)	0.9700
C(15)-H(15C)	0.9700	C(16)-C(17)	1.529(4)
C(16)-C(18)	1.535(4)	C(16)-H(16A)	0.9900
C(17)-H(17A)	0.9700	C(17)-H(17B)	0.9700
C(17)-H(17C)	0.9700	C(18)-H(18A)	0.9700
C(18)-H(18B)	0.9700	C(18)-H(18C)	0.9700
C(19)-C(23)	1.550(4)	C(19)-C(20)	1.565(4)
C(19)-H(19A)	0.9900	C(20)-C(21)	1.507(4)
C(20)-C(22)	1.528(4)	C(20)-H(20A)	0.9900
C(21)-H(21A)	0.9700	C(21)-H(21B)	0.9700
C(21)-H(21C)	0.9700	C(22)-H(22A)	0.9700
C(22)-H(22B)	0.9700	C(22)-H(22C)	0.9700
C(23)-C(25)	1.528(4)	C(23)-C(24)	1.534(4)
C(23)-H(23A)	0.9900	C(24)-H(24A)	0.9700
C(24)-H(24B)	0.9700	C(24)-H(24C)	0.9700
C(25)-H(25A)	0.9700	C(25)-H(25B)	0.9700
C(25)-H(25C)	0.9700	C(26)-C(27)	1.551(3)
C(26)-C(30)	1.568(4)	C(26)-H(26A)	0.9900
C(27)-C(28)	1.519(4)	C(27)-C(29)	1.532(4)
C(27)-H(27A)	0.9900	C(28)-H(28A)	0.9700
C(28)-H(28B)	0.9700	C(28)-H(28C)	0.9700
C(29)-H(29A)	0.9700	C(29)-H(29B)	0.9700
C(29)-H(29C)	0.9700	C(30)-C(31)	1.533(4)
C(30)-C(32)	1.535(4)	C(30)-H(30A)	0.9900
C(31)-H(31A)	0.9700	C(31)-H(31B)	0.9700
C(31)-H(31C)	0.9700	C(32)-H(32A)	0.9700

B)	0.9700	C(32)-H(32C)	0.9700

C(12)	109.62(11)	C(1)-P(1)-C(5)	104.21(12)
-C(5)	105.74(10)	C(1)-P(1)-B(1)	104.97(14)
-B(1)	116.88(13)	C(5)-P(1)-B(1)	114.67(15)
C(19)	109.52(12)	C(4)-P(2)-C(26)	104.44(11)
-C(26)	105.90(11)	C(4)-P(2)-B(2)	105.99(14)
-B(2)	116.18(14)	C(26)-P(2)-B(2)	114.17(14)
H(1)	114.3(10)	P(1)-B(1)-H(2)	100.4(14)
H(2)	114.8(17)	P(1)-B(1)-H(3)	104.8(14)
H(3)	110.6(17)	H(2)-B(1)-H(3)	111.2(19)
H(4)	115.7(10)	P(2)-B(2)-H(5)	102.7(14)
H(5)	111.4(17)	P(2)-B(2)-H(6)	101.2(14)
H(6)	115.4(17)	H(5)-B(2)-H(6)	109(2)
P(1)	120.74(18)	C(2)-C(1)-H(1A)	107.1
H(1A)	107.1	C(2)-C(1)-H(1B)	107.1
H(1B)	107.1	H(1A)-C(1)-H(1B)	106.8
C(1)	110.6(2)	C(3)-C(2)-H(2A)	109.5
H(2A)	109.5	C(3)-C(2)-H(2B)	109.5
H(2B)	109.5	H(2A)-C(2)-H(2B)	108.1
C(4)	111.8(2)	C(2)-C(3)-H(3A)	109.3
H(3A)	109.3	C(2)-C(3)-H(3B)	109.3
H(3B)	109.3	H(3A)-C(3)-H(3B)	107.9
P(2)	119.89(17)	C(3)-C(4)-H(4A)	107.3
H(4A)	107.3	C(3)-C(4)-H(4B)	107.3
H(4B)	107.3	H(4A)-C(4)-H(4B)	106.9
C(9)	119.3(2)	C(6)-C(5)-P(1)	109.86(18)
P(1)	114.09(16)	C(6)-C(5)-H(5A)	103.9
H(5A)	103.9	P(1)-C(5)-H(5A)	103.9
C(8)	109.1(3)	C(7)-C(6)-C(5)	112.3(2)
C(5)	115.0(3)	C(7)-C(6)-H(6A)	106.7
H(6A)	106.7	C(5)-C(6)-H(6A)	106.7
H(7A)	109.5	C(6)-C(7)-H(7B)	109.5
-H(7B)	109.5	C(6)-C(7)-H(7C)	109.5
-H(7C)	109.5	H(7B)-C(7)-H(7C)	109.5
H(8A)	109.5	C(6)-C(8)-H(8B)	109.5
-H(8B)	109.5	C(6)-C(8)-H(8C)	109.5
-H(8C)	109.5	H(8B)-C(8)-H(8C)	109.5
)-C(11)	45.3	C(11A)-C(9)-C(10)	118.3
-C(10)	115.2	C(11A)-C(9)-C(5)	125.29(12)
-C(5)	114.88(11)	C(10)-C(9)-C(5)	115.70(12)
)-H(9A)	57.7	C(11)-C(9)-H(9A)	102.8
-H(9A)	102.8	C(5)-C(9)-H(9A)	102.8
)-H(9B)	92.7	C(11)-C(9)-H(9B)	47.6
-H(9B)	92.7	C(5)-C(9)-H(9B)	92.7
-H(9B)	150.3	C(9)-C(10)-H(10A)	109.5
-H(10B)	109.5	H(10A)-C(10)-H(10B)	109.5
-H(10C)	109.5	H(10A)-C(10)-H(10C)	109.5
10)-H(10C)	109.5	C(9)-C(11)-H(11A)	109.5
-H(11B)	109.5	C(9)-C(11)-H(11C)	109.5
A)-H(11D)	109.5	C(9)-C(11A)-H(11E)	109.5
11A)-H(11E)	109.5	C(9)-C(11A)-H(11F)	109.5
11A)-H(11F)	109.5	H(11E)-C(11A)-H(11F)	109.5
2)-C(13)	113.29(19)	C(16)-C(12)-P(1)	117.14(16)
2)-P(1)	114.76(14)	C(16)-C(12)-H(12A)	103.0
2)-H(12A)	103.0	P(1)-C(12)-H(12A)	103.0
3)-C(14)	109.2(3)	C(15)-C(13)-C(12)	115.3(2)
3)-C(12)	110.3(2)	C(15)-C(13)-H(13A)	107.2
3)-H(13A)	107.2	C(12)-C(13)-H(13A)	107.2
4)-H(14A)	109.5	C(13)-C(14)-H(14B)	109.5
14)-H(14B)	109.5	C(13)-C(14)-H(14C)	109.5
14)-H(14C)	109.5	H(14B)-C(14)-H(14C)	109.5

C(13)-C(15)-H(15A)	109.5	C(13)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5	C(13)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5	H(15B)-C(15)-H(15C)	109.5
C(17)-C(16)-C(18)	108.6(3)	C(17)-C(16)-C(12)	117.0(2)
C(18)-C(16)-C(12)	111.9(2)	C(17)-C(16)-H(16A)	106.2
C(18)-C(16)-H(16A)	106.2	C(12)-C(16)-H(16A)	106.2
C(16)-C(17)-H(17A)	109.5	C(16)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5	C(16)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5	H(17B)-C(17)-H(17C)	109.5
C(16)-C(18)-H(18A)	109.5	C(16)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5	C(16)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5	H(18B)-C(18)-H(18C)	109.5
C(23)-C(19)-C(20)	112.7(2)	C(23)-C(19)-P(2)	115.37(18)
C(20)-C(19)-P(2)	117.01(19)	C(23)-C(19)-H(19A)	103.1
C(20)-C(19)-H(19A)	103.1	P(2)-C(19)-H(19A)	103.1
C(21)-C(20)-C(22)	111.0(3)	C(21)-C(20)-C(19)	111.4(3)
C(22)-C(20)-C(19)	117.1(2)	C(21)-C(20)-H(20A)	105.5
C(22)-C(20)-H(20A)	105.5	C(19)-C(20)-H(20A)	105.5
C(20)-C(21)-H(21A)	109.5	C(20)-C(21)-H(21B)	109.5
H(21A)-C(21)-H(21B)	109.5	C(20)-C(21)-H(21C)	109.5
H(21A)-C(21)-H(21C)	109.5	H(21B)-C(21)-H(21C)	109.5
C(20)-C(22)-H(22A)	109.5	C(20)-C(22)-H(22B)	109.5
H(22A)-C(22)-H(22B)	109.5	C(20)-C(22)-H(22C)	109.5
H(22A)-C(22)-H(22C)	109.5	H(22B)-C(22)-H(22C)	109.5
C(25)-C(23)-C(24)	109.1(2)	C(25)-C(23)-C(19)	115.0(2)
C(24)-C(23)-C(19)	110.2(2)	C(25)-C(23)-H(23A)	107.4
C(24)-C(23)-H(23A)	107.4	C(19)-C(23)-H(23A)	107.4
C(23)-C(24)-H(24A)	109.5	C(23)-C(24)-H(24B)	109.5
H(24A)-C(24)-H(24B)	109.5	C(23)-C(24)-H(24C)	109.5
H(24A)-C(24)-H(24C)	109.5	H(24B)-C(24)-H(24C)	109.5
C(23)-C(25)-H(25A)	109.5	C(23)-C(25)-H(25B)	109.5
H(25A)-C(25)-H(25B)	109.5	C(23)-C(25)-H(25C)	109.5
H(25A)-C(25)-H(25C)	109.5	H(25B)-C(25)-H(25C)	109.5
C(27)-C(26)-C(30)	119.8(2)	C(27)-C(26)-P(2)	113.89(18)
C(30)-C(26)-P(2)	108.85(17)	C(27)-C(26)-H(26A)	104.2
C(30)-C(26)-H(26A)	104.2	P(2)-C(26)-H(26A)	104.2
C(28)-C(27)-C(29)	111.2(2)	C(28)-C(27)-C(26)	113.1(2)
C(29)-C(27)-C(26)	116.7(2)	C(28)-C(27)-H(27A)	104.8
C(29)-C(27)-H(27A)	104.8	C(26)-C(27)-H(27A)	104.8
C(27)-C(28)-H(28A)	109.5	C(27)-C(28)-H(28B)	109.5
H(28A)-C(28)-H(28B)	109.5	C(27)-C(28)-H(28C)	109.5
H(28A)-C(28)-H(28C)	109.5	H(28B)-C(28)-H(28C)	109.5
C(27)-C(29)-H(29A)	109.5	C(27)-C(29)-H(29B)	109.5
H(29A)-C(29)-H(29B)	109.5	C(27)-C(29)-H(29C)	109.5
H(29A)-C(29)-H(29C)	109.5	H(29B)-C(29)-H(29C)	109.5
C(31)-C(30)-C(32)	109.6(2)	C(31)-C(30)-C(26)	112.2(2)
C(32)-C(30)-C(26)	115.8(2)	C(31)-C(30)-H(30A)	106.2
C(32)-C(30)-H(30A)	106.2	C(26)-C(30)-H(30A)	106.2
C(30)-C(31)-H(31A)	109.5	C(30)-C(31)-H(31B)	109.5
H(31A)-C(31)-H(31B)	109.5	C(30)-C(31)-H(31C)	109.5
H(31A)-C(31)-H(31C)	109.5	H(31B)-C(31)-H(31C)	109.5
C(30)-C(32)-H(32A)	109.5	C(30)-C(32)-H(32B)	109.5
H(32A)-C(32)-H(32B)	109.5	C(30)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5	H(32B)-C(32)-H(32C)	109.5

Symmetry transformations used to generate equivalent atoms:

3. Torsion angles [°] for GRAF1.

)-C(1)-C(2)	-79.5(2)	C(5)-P(1)-C(1)-C(2)	33.3(2)
-C(1)-C(2)	154.2(2)	P(1)-C(1)-C(2)-C(3)	-175.53(19)
-C(3)-C(4)	178.9(2)	C(2)-C(3)-C(4)-P(2)	-161.18(19)
)-C(4)-C(3)	80.0(2)	C(26)-P(2)-C(4)-C(3)	-33.0(2)
-C(4)-C(3)	-153.9(2)	C(1)-P(1)-C(5)-C(6)	61.0(2)
)-C(5)-C(6)	176.54(17)	B(1)-P(1)-C(5)-C(6)	-53.2(2)
-C(5)-C(9)	-162.05(17)	C(12)-P(1)-C(5)-C(9)	-46.51(17)
-C(5)-C(9)	83.8(2)	C(9)-C(5)-C(6)-C(7)	82.3(3)
-C(6)-C(7)	-143.3(2)	C(9)-C(5)-C(6)-C(8)	-43.2(4)
-C(6)-C(8)	91.2(3)	C(6)-C(5)-C(9)-C(11A)	-112.9(2)
-C(9)-C(11A)	114.53(13)	C(6)-C(5)-C(9)-C(11)	-61.3(2)
-C(9)-C(11)	166.08(9)	C(6)-C(5)-C(9)-C(10)	76.8(2)
-C(9)-C(10)	-55.83(17)	C(1)-P(1)-C(12)-C(16)	-82.50(18)
-C(12)-C(16)	165.70(16)	B(1)-P(1)-C(12)-C(16)	36.7(2)
-C(12)-C(13)	54.03(18)	C(5)-P(1)-C(12)-C(13)	-57.77(18)
-C(12)-C(13)	173.25(19)	C(16)-C(12)-C(13)-C(15)	45.6(3)
)-C(13)-C(15)	-92.6(2)	C(16)-C(12)-C(13)-C(14)	-78.7(3)
)-C(13)-C(14)	143.1(2)	C(13)-C(12)-C(16)-C(17)	-88.4(3)
)-C(16)-C(17)	48.8(3)	C(13)-C(12)-C(16)-C(18)	145.4(2)
)-C(16)-C(18)	-77.5(2)	C(4)-P(2)-C(19)-C(23)	-52.6(2)
)-C(19)-C(23)	59.5(2)	B(2)-P(2)-C(19)-C(23)	-172.6(2)
-C(19)-C(20)	83.5(2)	C(26)-P(2)-C(19)-C(20)	-164.4(2)
-C(19)-C(20)	-36.5(3)	C(23)-C(19)-C(20)-C(21)	-139.7(3)
)-C(20)-C(21)	83.1(3)	C(23)-C(19)-C(20)-C(22)	91.1(3)
)-C(20)-C(22)	-46.2(3)	C(20)-C(19)-C(23)-C(25)	-47.9(3)
)-C(23)-C(25)	90.1(2)	C(20)-C(19)-C(23)-C(24)	76.0(3)
)-C(23)-C(24)	-146.0(2)	C(4)-P(2)-C(26)-C(27)	157.38(19)
)-C(26)-C(27)	41.8(2)	B(2)-P(2)-C(26)-C(27)	-87.3(2)
-C(26)-C(30)	-66.17(19)	C(19)-P(2)-C(26)-C(30)	178.23(17)
-C(26)-C(30)	49.1(2)	C(30)-C(26)-C(27)-C(28)	57.5(3)
)-C(27)-C(28)	-171.2(2)	C(30)-C(26)-C(27)-C(29)	-73.4(3)
)-C(27)-C(29)	57.9(3)	C(27)-C(26)-C(30)-C(31)	-86.7(3)
)-C(30)-C(31)	139.9(2)	C(27)-C(26)-C(30)-C(32)	40.2(3)
)-C(30)-C(32)	-93.2(2)		

y transformations used to generate equivalent atoms:

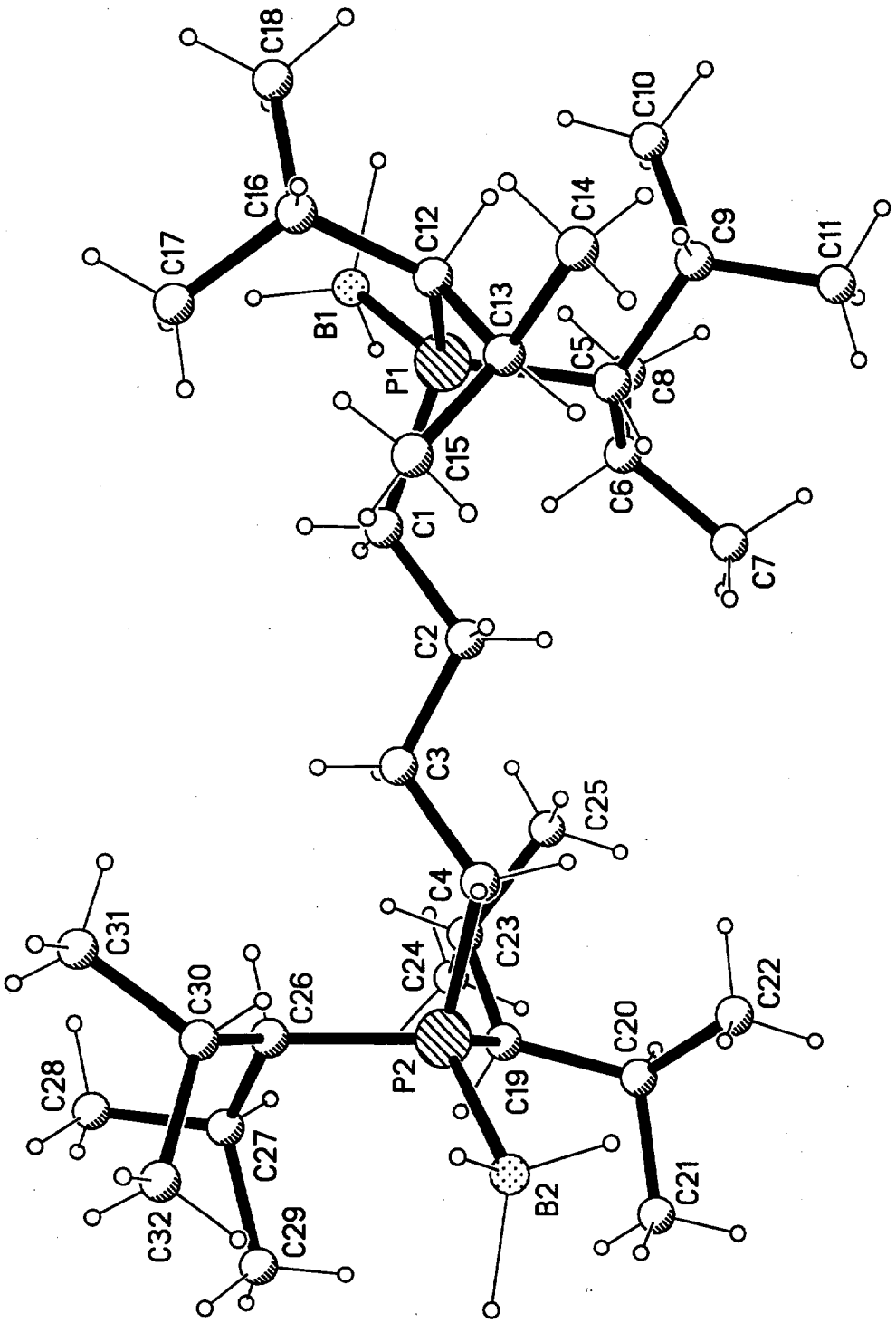
Table 4. Anisotropic displacement parameters [Å^2] for GRAF1.

The anisotropic displacement factor exponent takes the form:
$-2\pi^2 [(ha^*)^2 U_{11} + ... + 2hka^* b^* U_{12}]$

Atom	U11	U22	U33	U23	U13	U12
P(1)	0.0428(4)	0.0342(4)	0.0233(3)	-0.0008(3)	0.0004(3)	0.0006(3)
P(2)	0.0440(4)	0.0282(3)	0.0259(3)	-0.0002(3)	0.0003(3)	0.0003(3)
B(1)	0.066(2)	0.0356(18)	0.052(2)	0.0074(16)	0.0073(19)	0.0031(17)
B(2)	0.055(2)	0.0315(16)	0.051(2)	0.0020(15)	-0.0052(17)	-0.0034(15)
C(1)	0.0508(16)	0.0422(15)	0.0238(13)	-0.0018(11)	-0.0027(12)	0.0069(13)
C(2)	0.0477(16)	0.0391(14)	0.0267(13)	-0.0024(12)	-0.0005(12)	0.0060(12)
C(3)	0.0432(15)	0.0371(14)	0.0245(13)	-0.0021(11)	-0.0018(11)	0.0012(12)
C(4)	0.0442(15)	0.0387(14)	0.0247(13)	0.0018(11)	0.0011(11)	-0.0010(12)
C(5)	0.0401(15)	0.0392(14)	0.0333(14)	-0.0011(12)	-0.0028(12)	-0.0002(12)
C(6)	0.0405(15)	0.0539(18)	0.0511(17)	-0.0009(15)	0.0011(14)	-0.0044(14)
C(7)	0.0448(18)	0.080(2)	0.061(2)	-0.0102(18)	0.0058(15)	0.0075(17)
C(8)	0.059(2)	0.076(3)	0.111(3)	-0.010(2)	0.012(2)	-0.0247(19)
C(9)	0.096(3)	0.071(2)	0.0473(19)	-0.0079(19)	-0.0310(19)	0.023(2)
C(10)	0.076(2)	0.084(2)	0.0476(19)	-0.0258(18)	-0.0139(17)	0.000(2)
C(11)	0.042(3)	0.073(4)	0.056(3)	0.013(3)	-0.015(2)	0.002(3)
C(11A)	0.140(13)	0.081(8)	0.056(7)	0.026(6)	-0.045(7)	0.014(8)
C(12)	0.0387(14)	0.0461(16)	0.0301(14)	-0.0065(12)	0.0010(12)	-0.0011(12)
C(13)	0.0446(16)	0.0446(16)	0.0518(18)	-0.0049(14)	0.0105(14)	-0.0084(13)
C(14)	0.070(2)	0.065(2)	0.071(2)	0.0130(18)	0.0226(19)	-0.0119(18)
C(15)	0.054(2)	0.068(2)	0.081(2)	-0.0215(19)	0.0020(18)	-0.0152(17)
C(16)	0.0458(17)	0.0581(18)	0.0469(17)	-0.0107(15)	0.0010(14)	0.0062(14)
C(17)	0.057(2)	0.093(3)	0.066(2)	-0.021(2)	-0.0181(17)	0.0292(19)
C(18)	0.067(2)	0.063(2)	0.055(2)	-0.0111(17)	0.0076(17)	0.0152(17)
C(19)	0.0456(15)	0.0350(14)	0.0324(14)	0.0030(12)	0.0044(12)	0.0049(12)
C(20)	0.0493(18)	0.0478(17)	0.0587(19)	0.0027(15)	0.0088(15)	0.0115(14)
C(21)	0.081(3)	0.061(2)	0.090(3)	-0.017(2)	0.006(2)	0.025(2)
C(22)	0.0503(18)	0.0562(19)	0.069(2)	0.0175(17)	-0.0062(16)	0.0106(15)
C(23)	0.0381(15)	0.0391(15)	0.0439(16)	0.0052(13)	0.0076(12)	0.0027(12)
C(24)	0.0540(19)	0.0510(18)	0.062(2)	0.0121(16)	0.0174(16)	0.0003(15)
C(25)	0.0442(17)	0.0450(17)	0.0585(19)	0.0003(15)	-0.0010(14)	-0.0050(13)
C(26)	0.0469(15)	0.0312(13)	0.0253(13)	-0.0003(11)	-0.0032(11)	-0.0013(12)
C(27)	0.0647(19)	0.0439(16)	0.0305(14)	0.0011(13)	-0.0003(13)	0.0061(14)
C(28)	0.085(2)	0.064(2)	0.0381(17)	0.0064(15)	-0.0042(16)	0.0086(19)
C(29)	0.088(2)	0.0583(19)	0.0319(16)	-0.0063(15)	0.0000(16)	0.0071(18)
C(30)	0.0466(16)	0.0437(16)	0.0398(16)	0.0022(13)	-0.0038(13)	-0.0026(13)
C(31)	0.0496(18)	0.0574(19)	0.0541(19)	0.0059(15)	-0.0016(15)	0.0075(15)
C(32)	0.0566(19)	0.058(2)	0.068(2)	-0.0024(17)	-0.0180(17)	-0.0115(16)

Table 5. Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for GRAF1.

Atom	x	y	z	U(iso)
H(1A)	0.1426	0.2645	0.1074	0.047
H(1B)	0.2231	0.2929	0.0911	0.047
H(2A)	0.2651	0.1410	0.1098	0.045
H(2B)	0.1819	0.1136	0.1220	0.045
H(3A)	0.2284	0.1753	0.0312	0.042
H(3B)	0.1458	0.1459	0.0436	0.042
H(4A)	0.1761	-0.0051	0.0501	0.043
H(4B)	0.2614	0.0164	0.0554	0.043
H(5A)	0.2864	0.1736	0.1869	0.045
H(6A)	0.3421	0.2735	0.1276	0.058
H(7A)	0.3828	0.1215	0.1433	0.093
H(7B)	0.4404	0.1612	0.1803	0.093
H(7C)	0.4485	0.1862	0.1262	0.093
H(8A)	0.4462	0.3572	0.1462	0.123
H(8B)	0.4428	0.3266	0.1997	0.123
H(8C)	0.3822	0.3950	0.1792	0.123
H(9A)	0.2648	0.2204	0.2602	0.086
H(9B)	0.3666	0.2394	0.2435	0.086
H(10A)	0.2709	0.3748	0.2624	0.103
H(10B)	0.3578	0.3752	0.2539	0.103
H(10C)	0.3252	0.3377	0.3020	0.103
H(11A)	0.3640	0.1709	0.2998	0.086
H(11B)	0.4183	0.1966	0.2580	0.086
H(11C)	0.3601	0.1155	0.2518	0.086
H(11D)	0.3329	0.1253	0.2594	0.139
H(11E)	0.2582	0.1640	0.2813	0.139
H(11F)	0.3358	0.1936	0.3029	0.139
H(12A)	0.1607	0.2815	0.2449	0.046
H(13A)	0.1708	0.1299	0.2074	0.056
H(14A)	0.0953	0.0667	0.2674	0.103
H(14B)	0.0603	0.1655	0.2766	0.103
H(14C)	0.1455	0.1478	0.2872	0.103
H(15A)	0.0624	0.0591	0.1834	0.102
H(15B)	0.0751	0.1459	0.1504	0.102
H(15C)	0.0160	0.1506	0.1917	0.102
H(16A)	0.0295	0.2913	0.2308	0.060
H(17A)	-0.0144	0.3790	0.1692	0.108
H(17B)	0.0245	0.2898	0.1482	0.108
H(17C)	0.0660	0.3862	0.1468	0.108
H(18A)	0.0298	0.4507	0.2411	0.093
H(18B)	0.1130	0.4567	0.2232	0.093
H(18C)	0.0945	0.4062	0.2713	0.093
H(19A)	0.3246	0.0307	-0.0765	0.045
H(20A)	0.4373	-0.0003	-0.0323	0.062
H(21A)	0.4392	-0.1498	-0.0589	0.116
H(21B)	0.4008	-0.0849	-0.0966	0.116
H(21C)	0.3517	-0.1498	-0.0643	0.116
H(22A)	0.4349	-0.1140	0.0247	0.088
H(22B)	0.3470	-0.1124	0.0255	0.088
H(22C)	0.3925	-0.0253	0.0432	0.088
H(23A)	0.3030	0.1729	-0.0337	0.048
H(24A)	0.4147	0.2342	-0.0644	0.084
H(24B)	0.3765	0.1666	-0.1011	0.084
H(24C)	0.4458	0.1327	-0.0714	0.084
H(25A)	0.3523	0.1291	0.0406	0.074
H(25B)	0.3913	0.2190	0.0205	0.074
H(25C)	0.4322	0.1223	0.0175	0.074
H(26A)	0.1751	0.1399	-0.0429	0.041
H(27A)	0.2420	0.1173	-0.1080	0.056
H(28A)	0.1435	0.2211	-0.1147	0.094
H(28B)	0.0961	0.1437	-0.1402	0.094
H(28C)	0.1695	0.1818	-0.1641	0.094
H(29A)	0.2242	-0.0422	-0.1225	0.089
H(29B)	0.2238	0.0194	-0.1686	0.089
H(29C)	0.1480	-0.0175	-0.1474	0.089
H(30A)	0.0869	0.0233	-0.0136	0.052
H(31A)	0.0579	0.1802	-0.0170	0.081
H(31B)	-0.0129	0.1200	-0.0294	0.081
H(31C)	0.0322	0.1696	-0.0701	0.081
H(32A)	-0.0001	-0.0341	-0.0654	0.092
H(32B)	0.0789	-0.0716	-0.0808	0.092
H(32C)	0.0414	0.0107	-0.1089	0.092
H(1)	0.2407(14)	0.4859(17)	0.2048(9)	0.043(3)
H(2)	0.2754(15)	0.4331(18)	0.1385(9)	0.043(3)
H(3)	0.1779(15)	0.4573(18)	0.1449(9)	0.043(3)
H(4)	0.2152(14)	-0.1708(17)	-0.0750(9)	0.043(3)
H(5)	0.1492(15)	-0.1300(17)	-0.0164(9)	0.043(3)
H(6)	0.2415(15)	-0.1574(18)	-0.0004(9)	0.043(3)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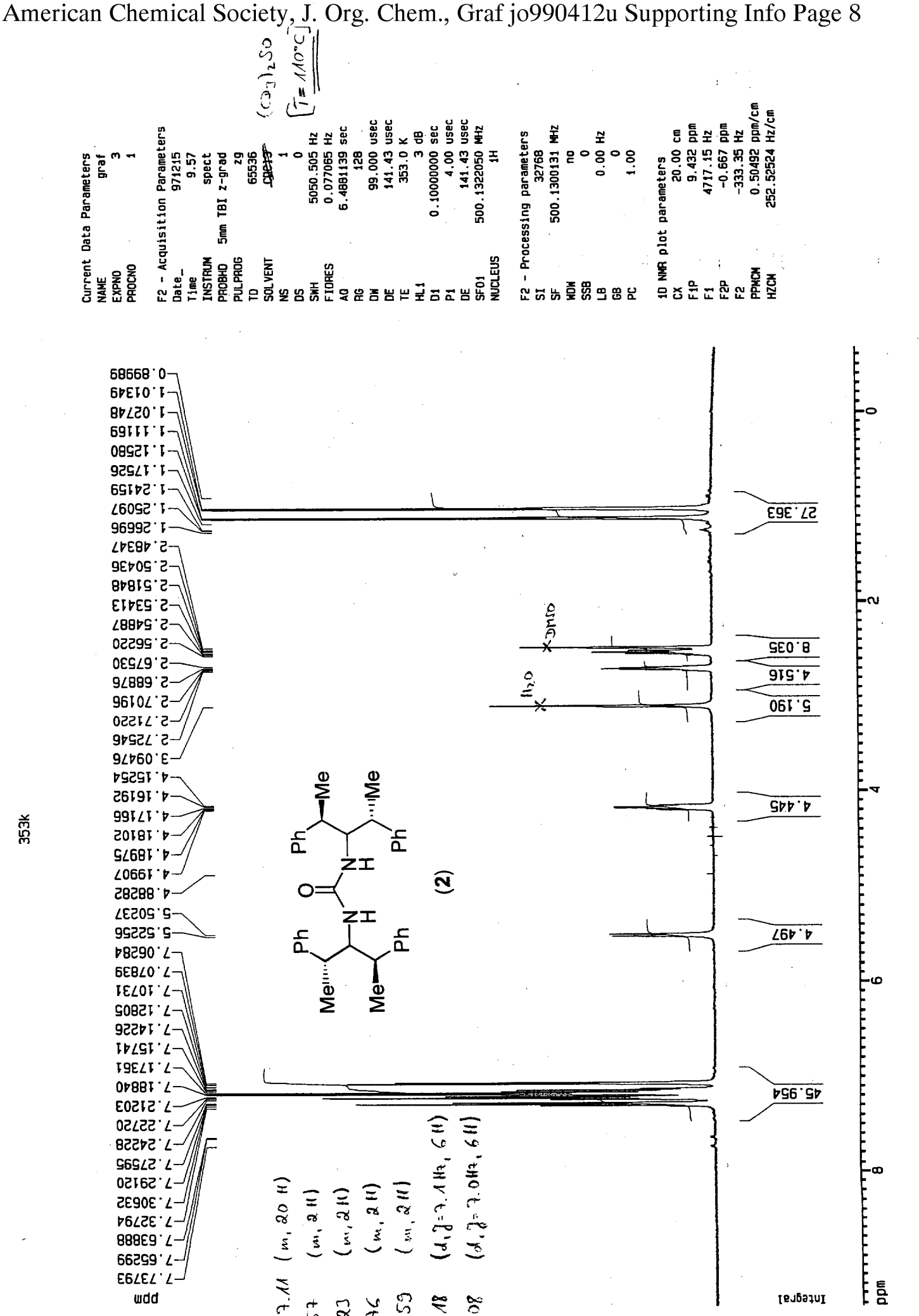
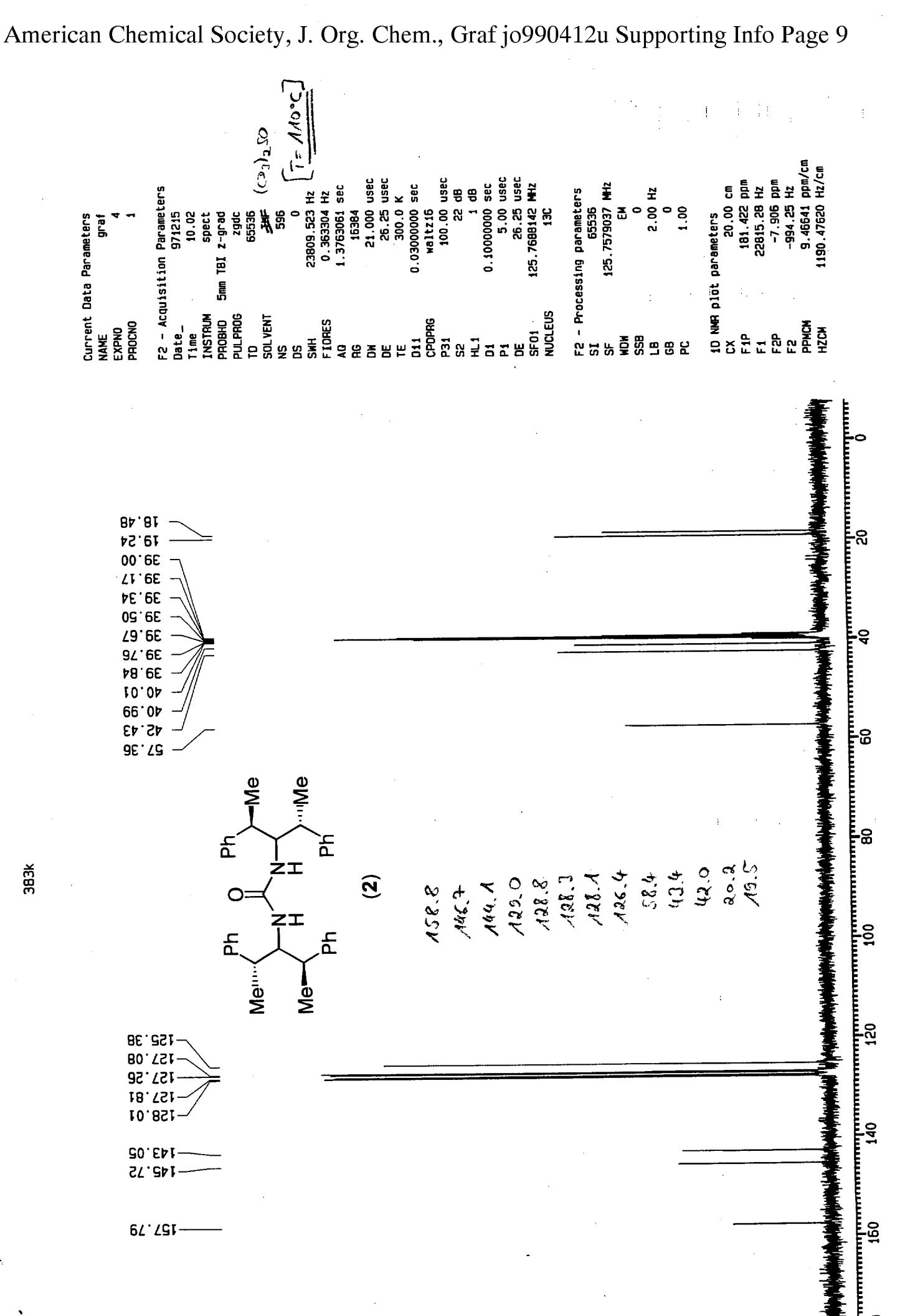
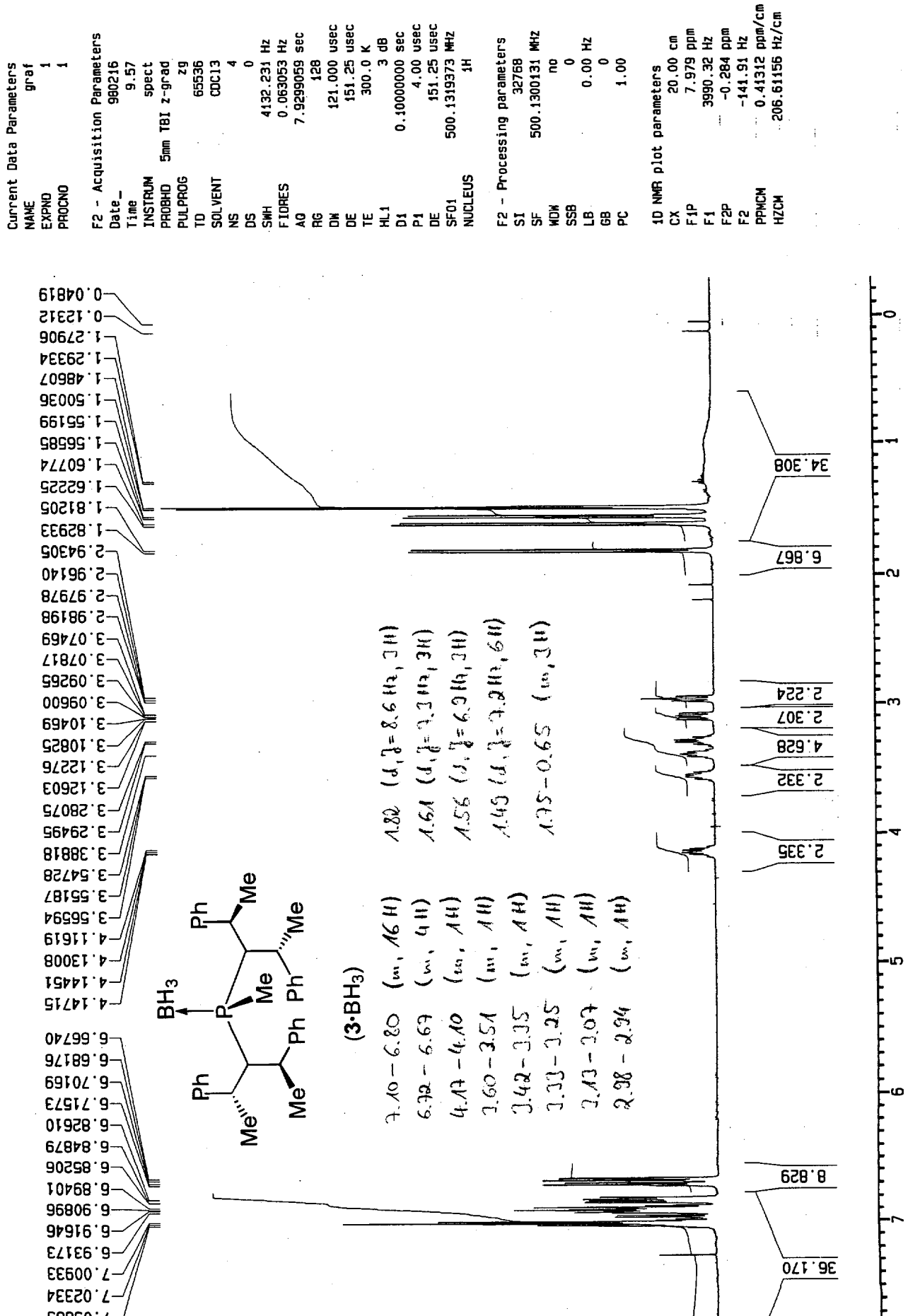
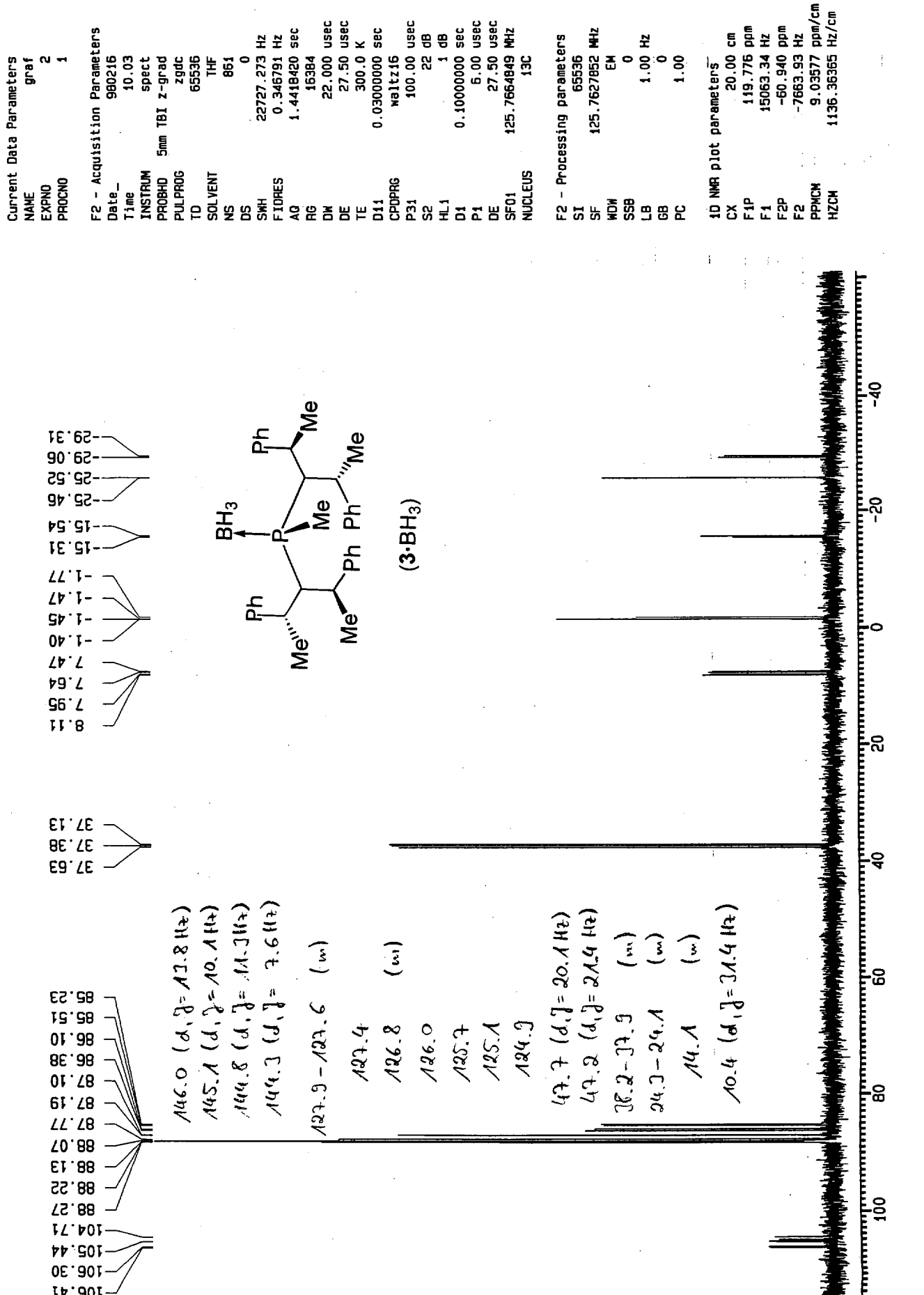
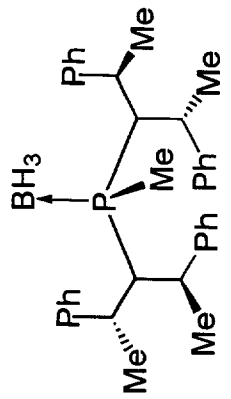








MePhos



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1D NMR plot parameters
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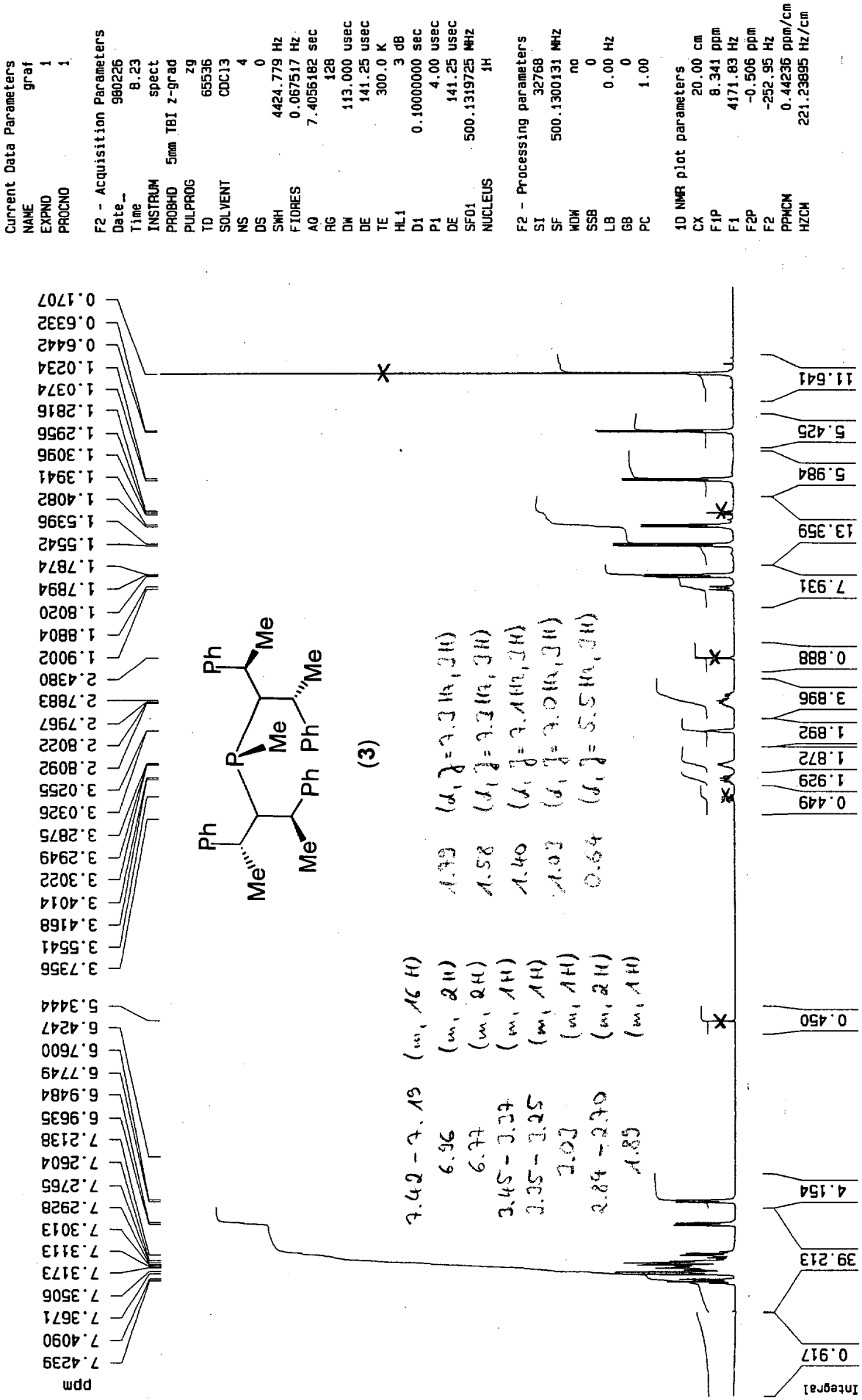

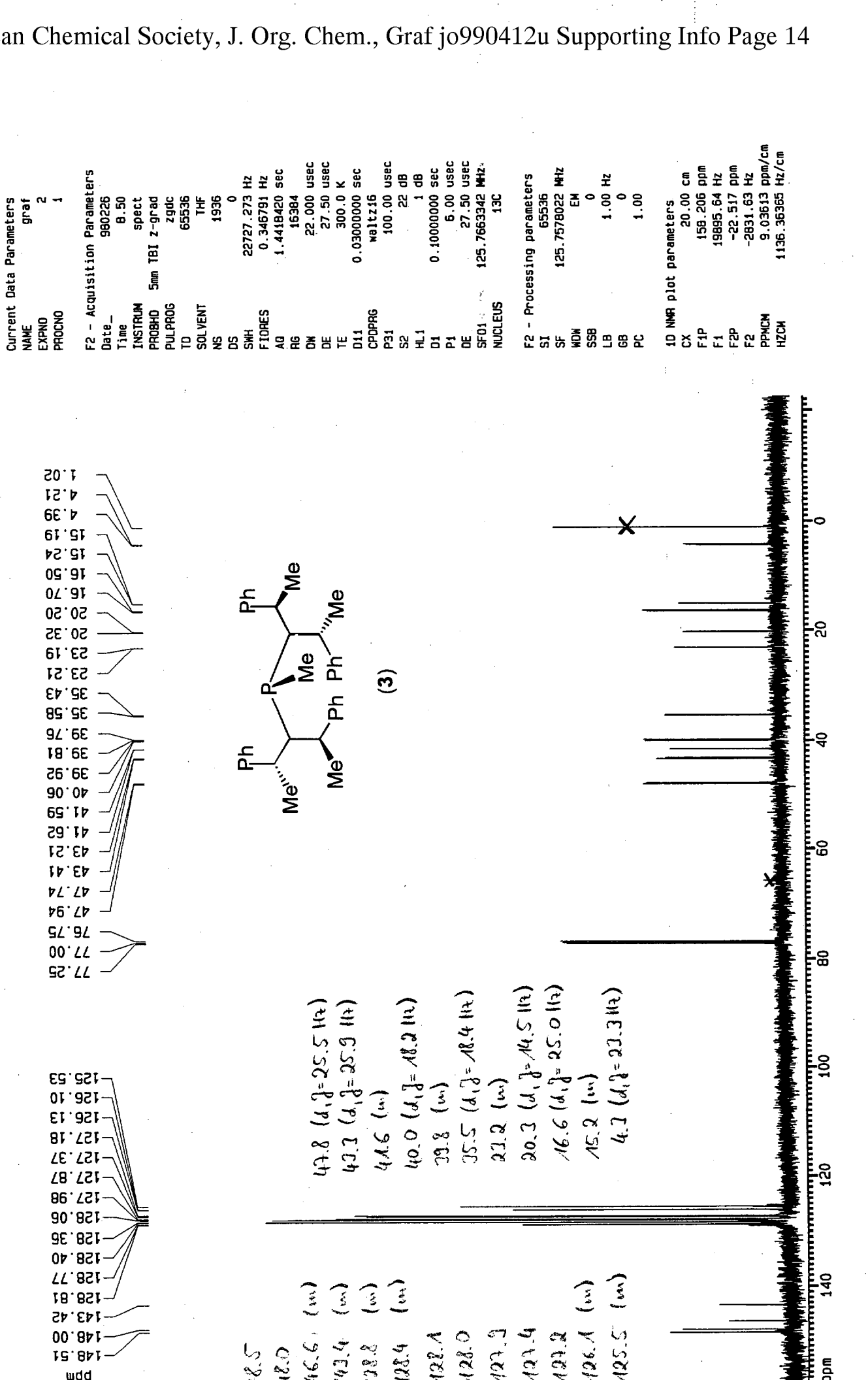

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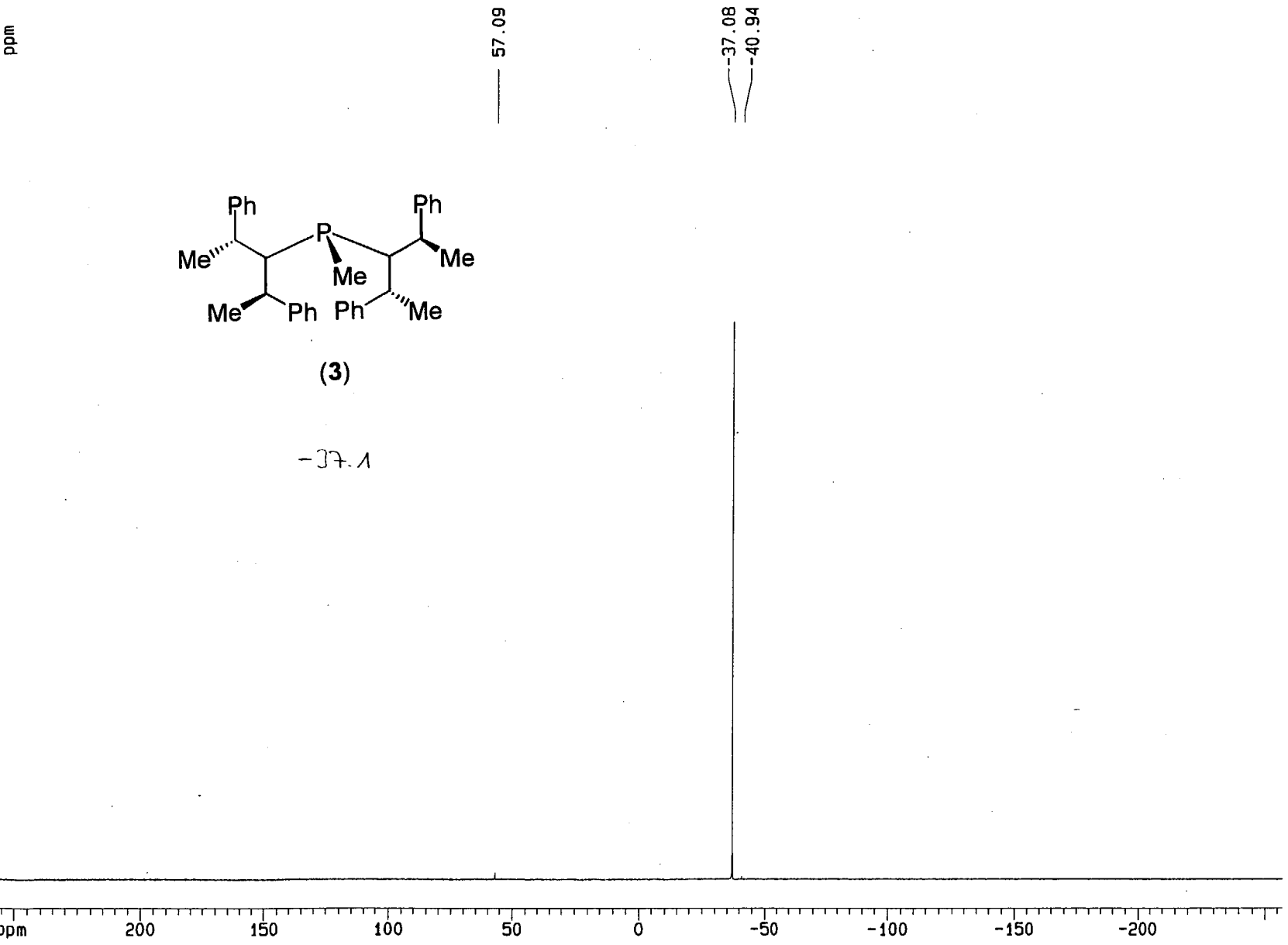

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1D NMR plot parameters

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DATE 5-2-97

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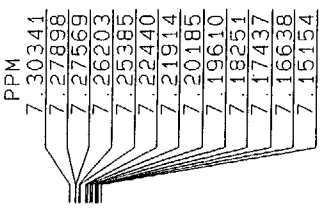
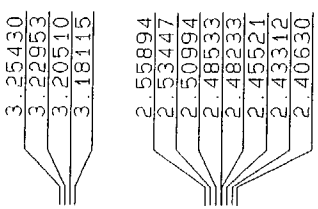
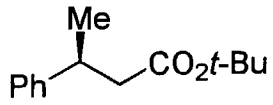
F2 -.799P
HZ/CM 107.192
PPM/CM .357
SR 3372.99

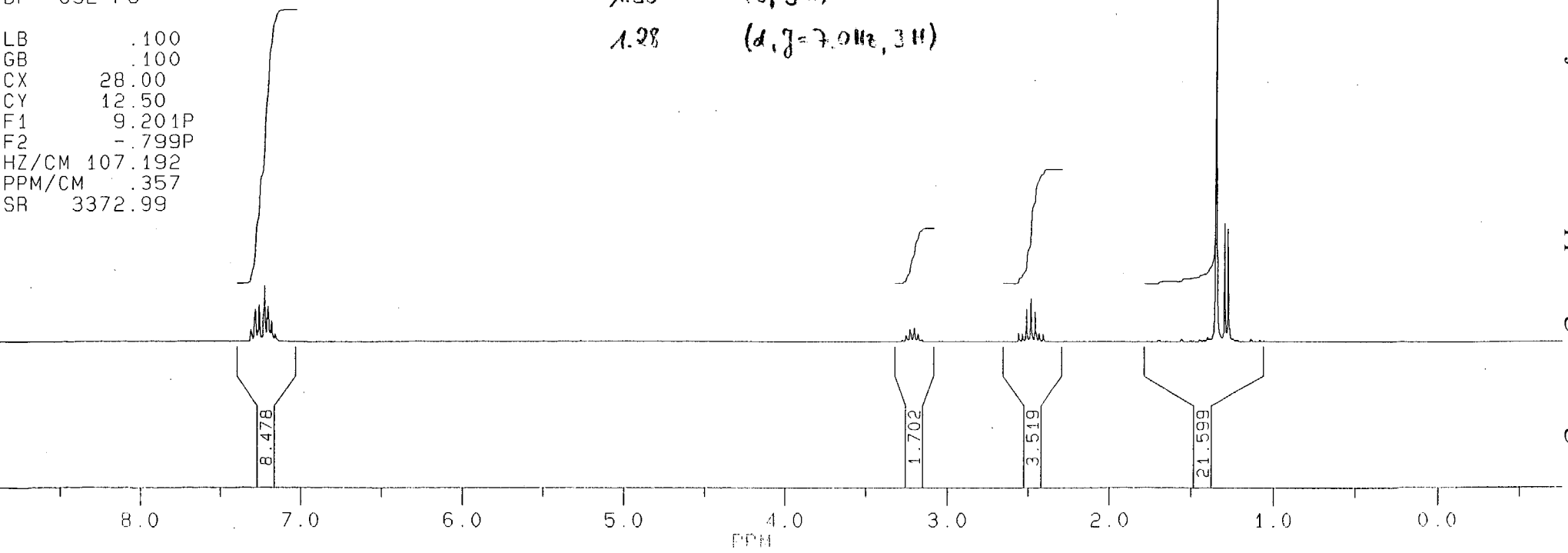


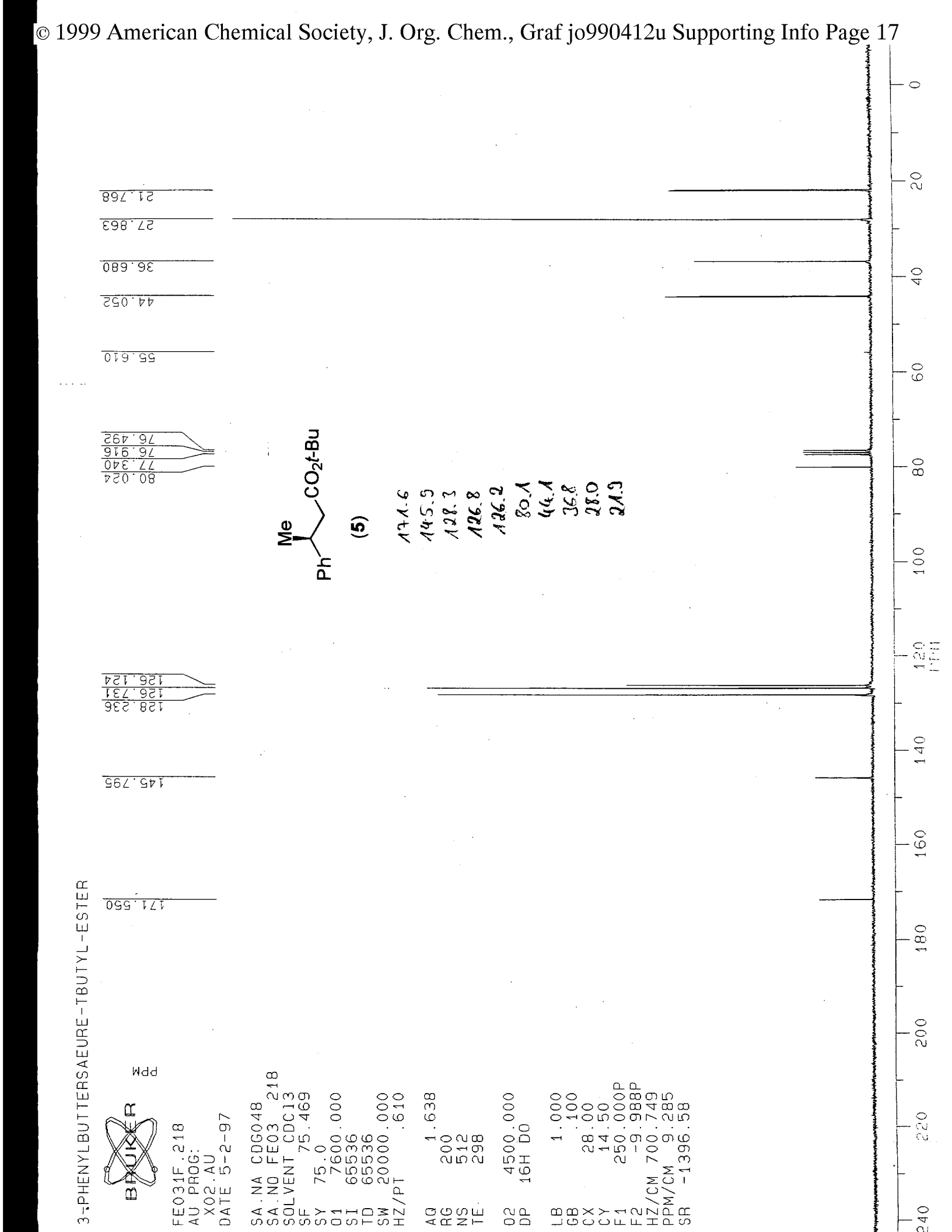




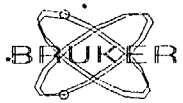
(5)

7.32 – 7.14	(m, 5H)
3.28 – 3.14	(m, 1H)
2.52	(dd, J = 7.3 and 14.7 Hz, 1H)
2.44	(dd, J = 8.1 and 14.7 Hz, 1H)
1.35	(s, 9H)
1.28	(d, J = 7.0 Hz, 3H)





TBU-ESTER - EE



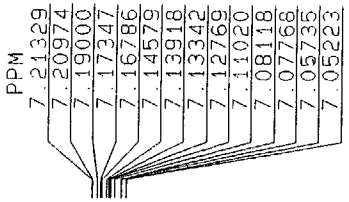
SE030F.142
AU PROG:
X00.AU
DATE 4-9-97

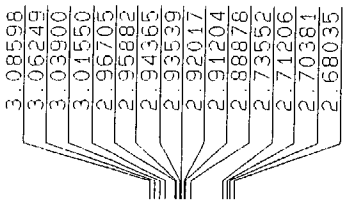
SA.NA CDG112
SA.NO SE03 142
SOLVENT CDC13
SF 300.133
SY 210.0
01 5200.000
SI 32768
TD 32768
SW 6024.096
HZ/PT .368

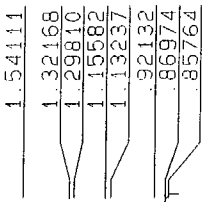
AQ 2.720
RG 2
NS 16
TE 298

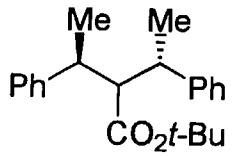
02 3200.000
DP 63L PO

LB .100
GB .100
CX 28.00
CY 12.50
F1 9.201P
F2 -.799P
HZ/CM 107.192
PPM/CM .357
SR 3401.67



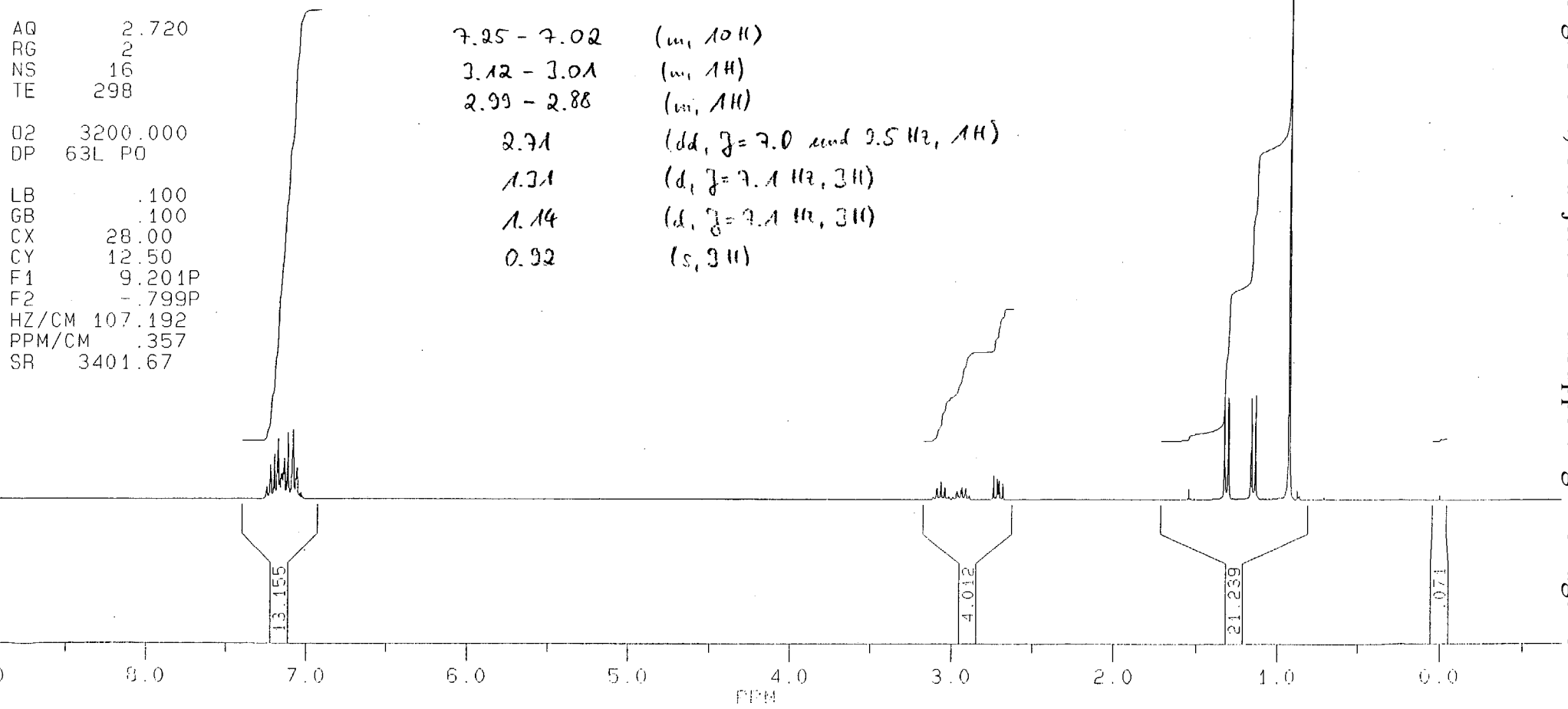






(6)

7.25 - 7.02 (m, 10H)
3.12 - 3.01 (m, 1H)
2.99 - 2.88 (m, 1H)
2.71 (dd, J = 7.0 and 9.5 Hz, 1H)
1.31 (d, J = 7.1 Hz, 3H)
1.14 (d, J = 7.1 Hz, 3H)
0.92 (s, 9H)



TBU-ESTER - EE

BRUKER

PPM

SE031F.142
AU PROG:
X02.AU
DATE 4-9-97

SA.NA CDG112
SA.NO SE03 142
SOLVENT CDCl3
SF 75.469
SY 75.0
O1 7600.000
SI 65536
TD 65536
SW 20000.000
HZ/PT .610

AQ 1.638
RG 200
NS 512
TE 298

O2 4500.000
DP 16H DO

LB 1.000
GB .100
CX 28.00
CY 14.50
F1 250.001P
F2 -9.988P
HZ/CM 700.749
PPM/CM 9.285
SR -1417.94

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144.983
128.478
128.290
128.074
127.901
126.660
126.420
126.399
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77.640
77.217
76.793
59.507
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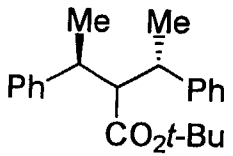
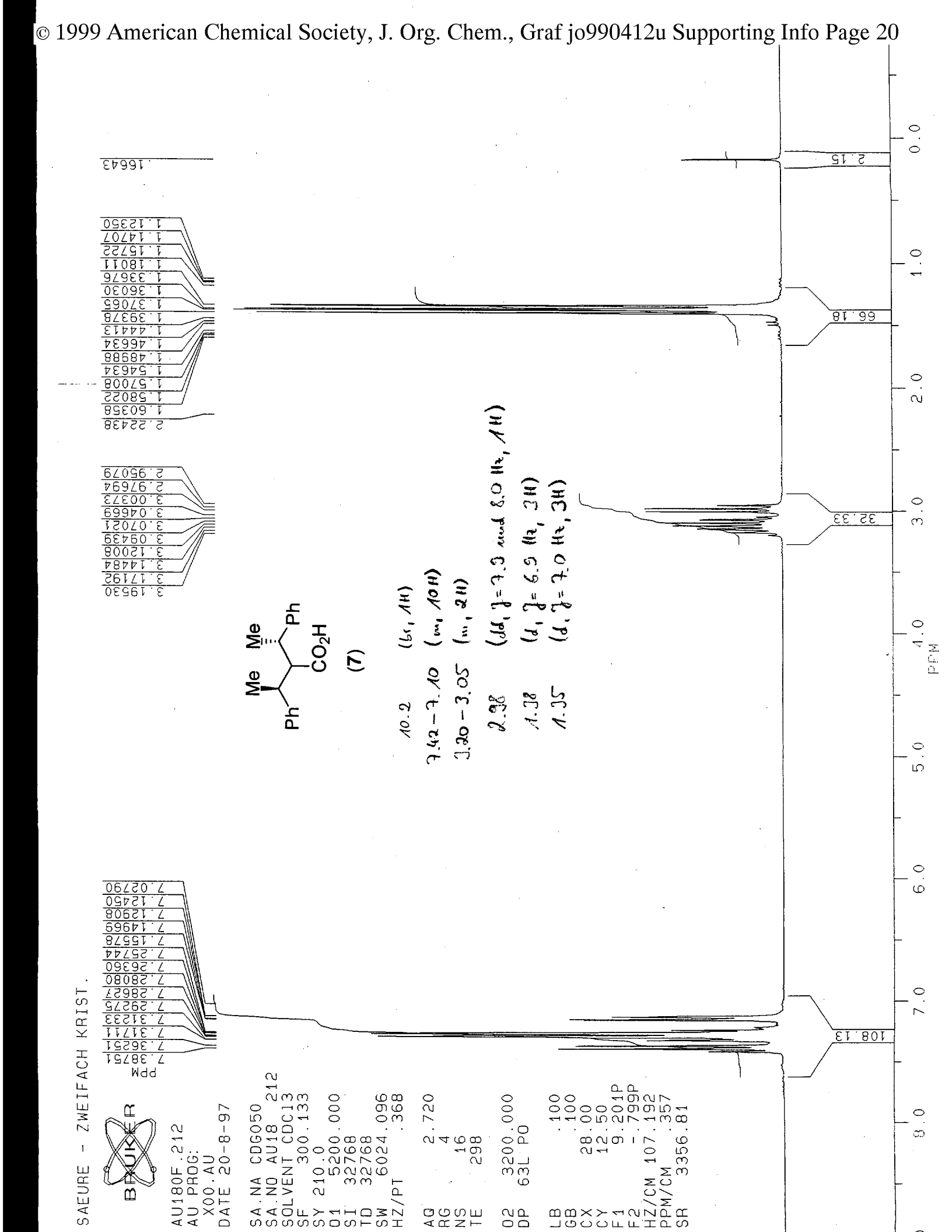
40.021
27.733
20.542
16.933

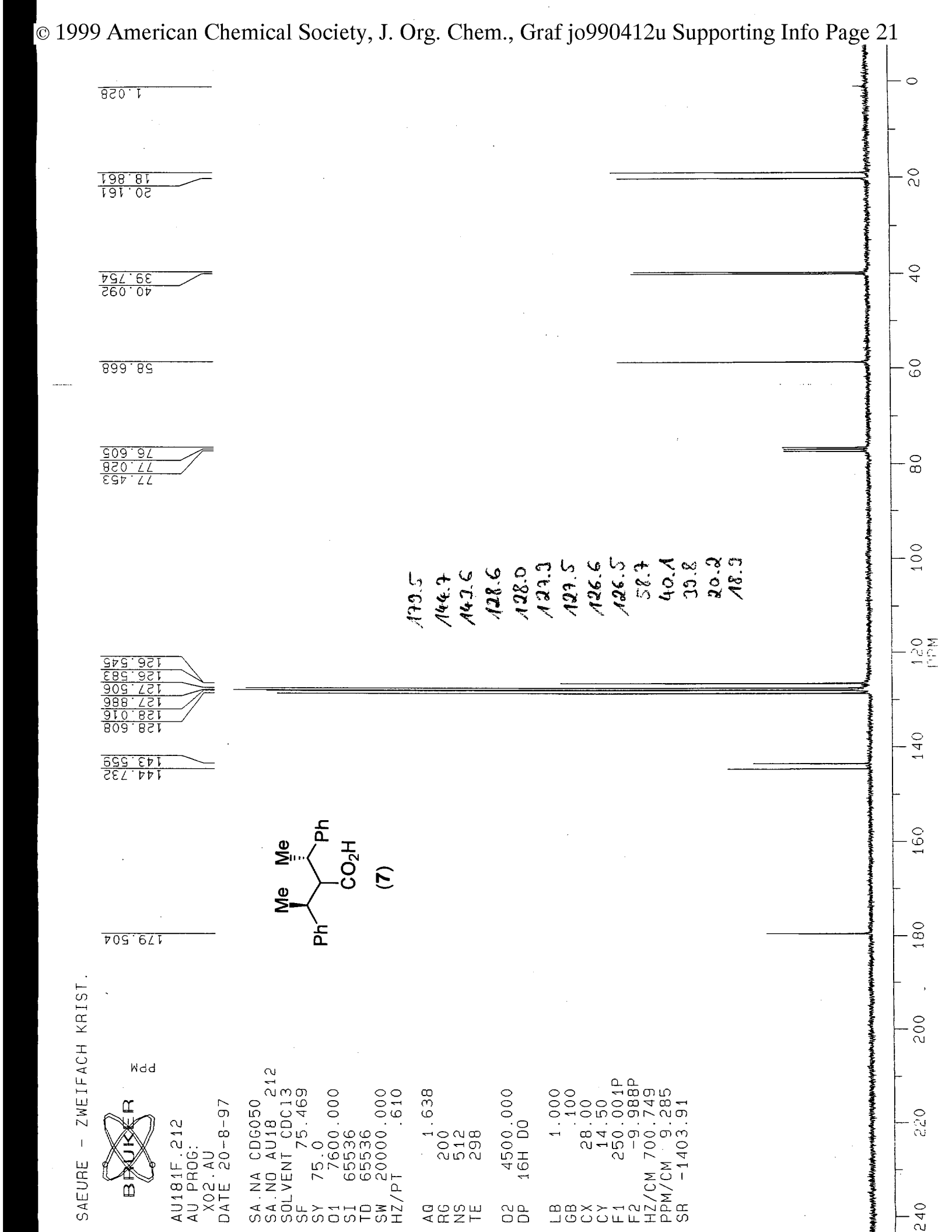


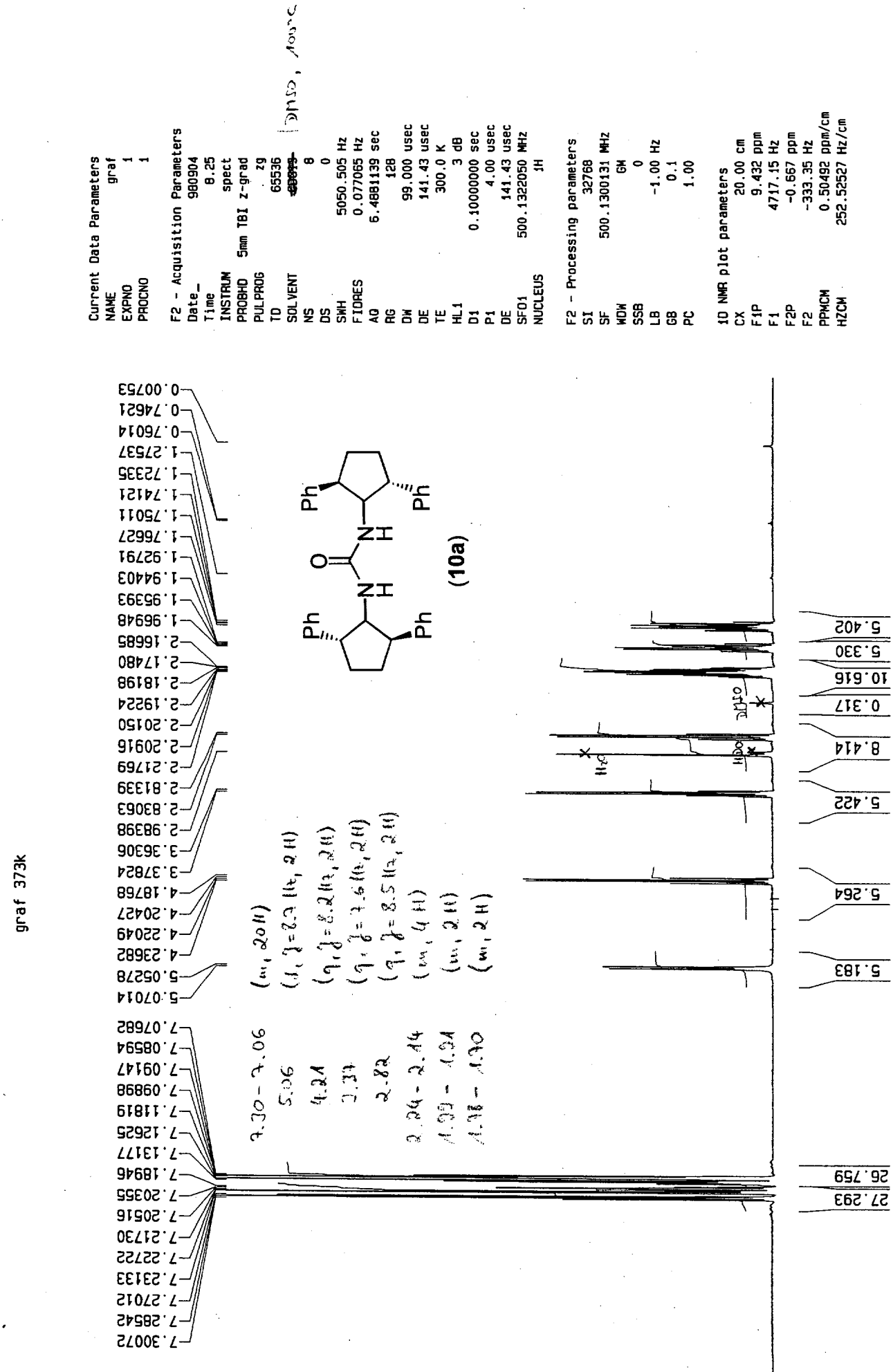
(6)

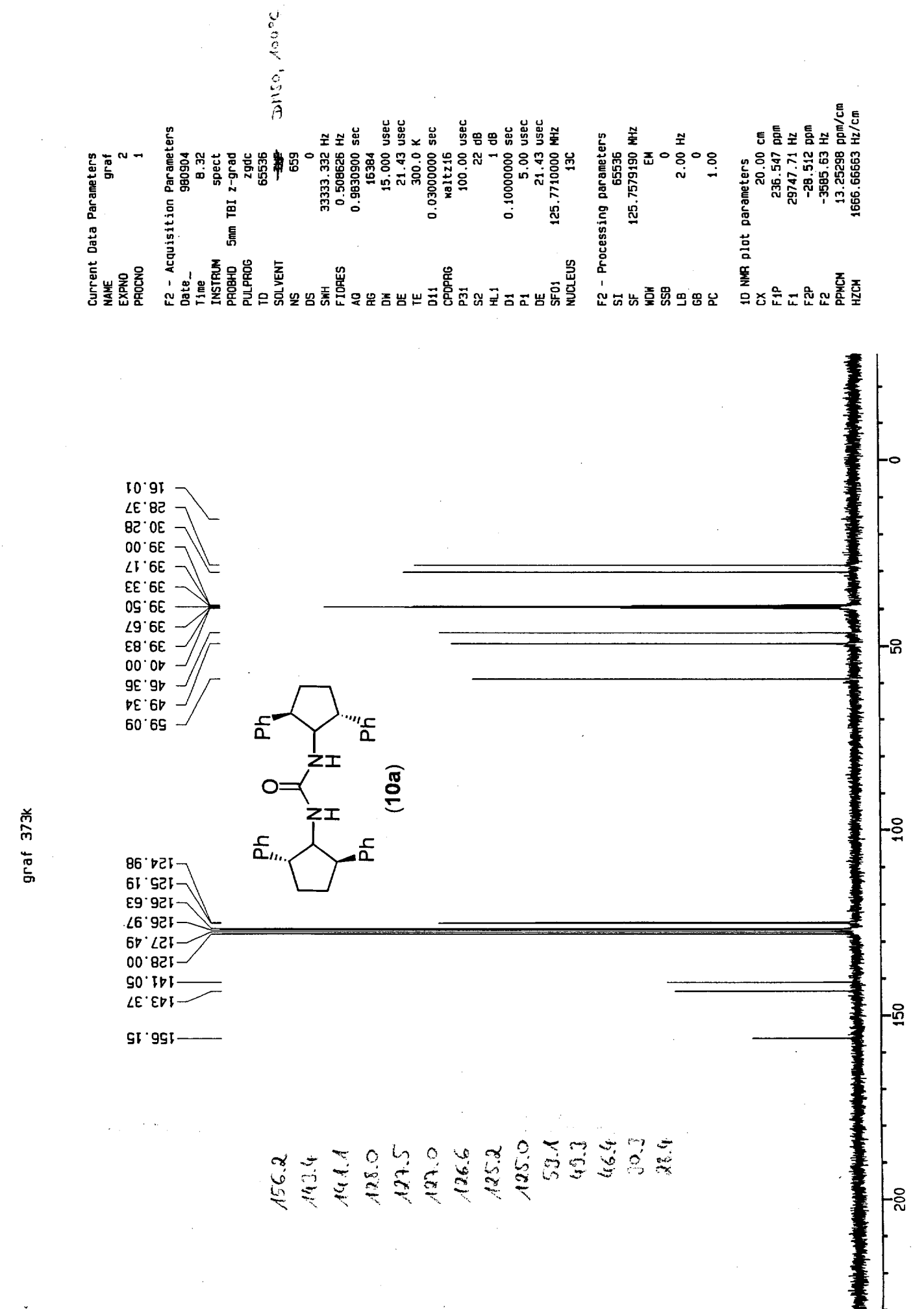
172.5
145.2
144.8
128.3
128.1
127.9
127.7
126.2
126.1
79.7
59.3
40.7
39.8
27.5
20.3
16.7

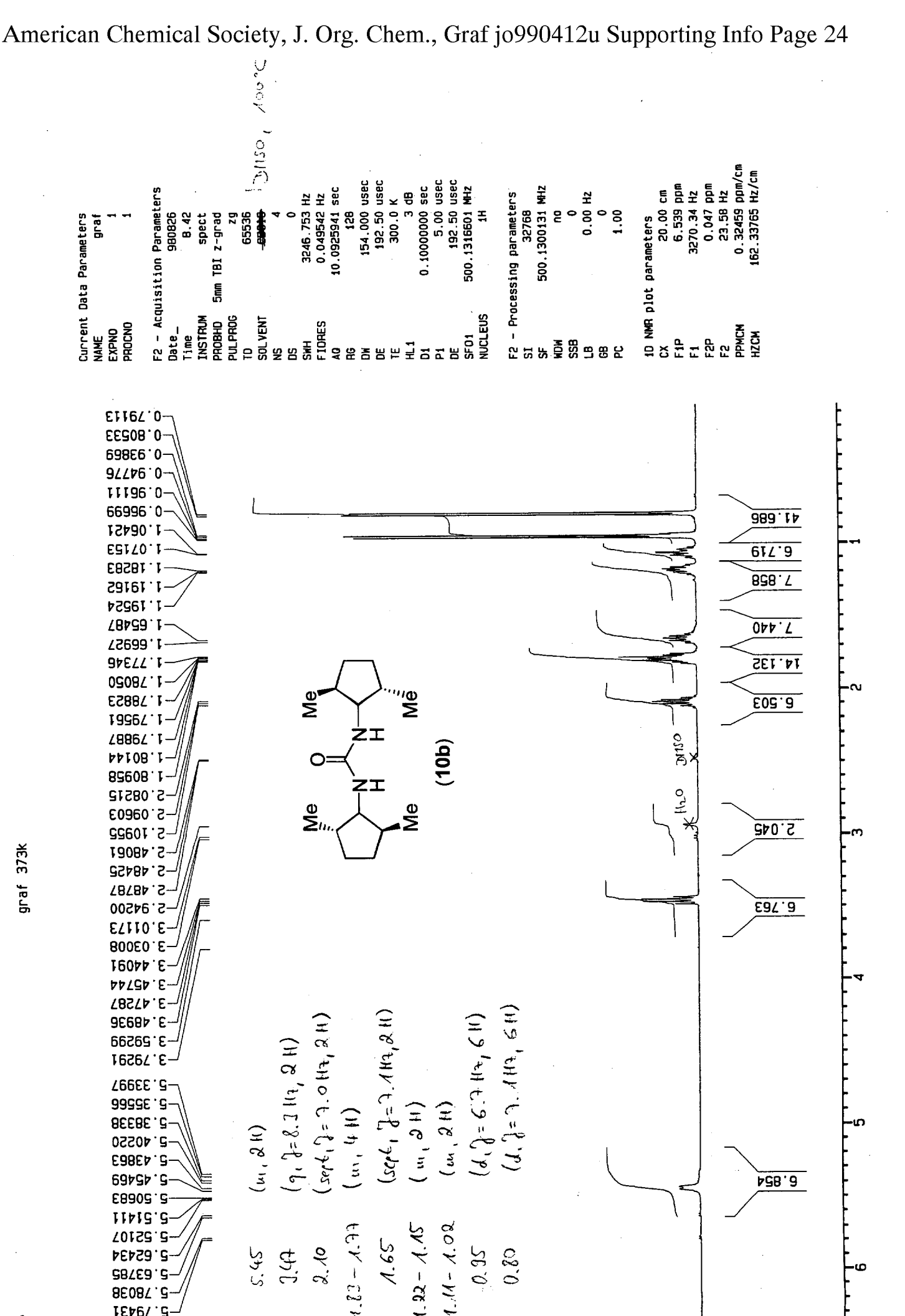
240 220 200 180 160 140 120 100 80 60 40 20 0
PPM

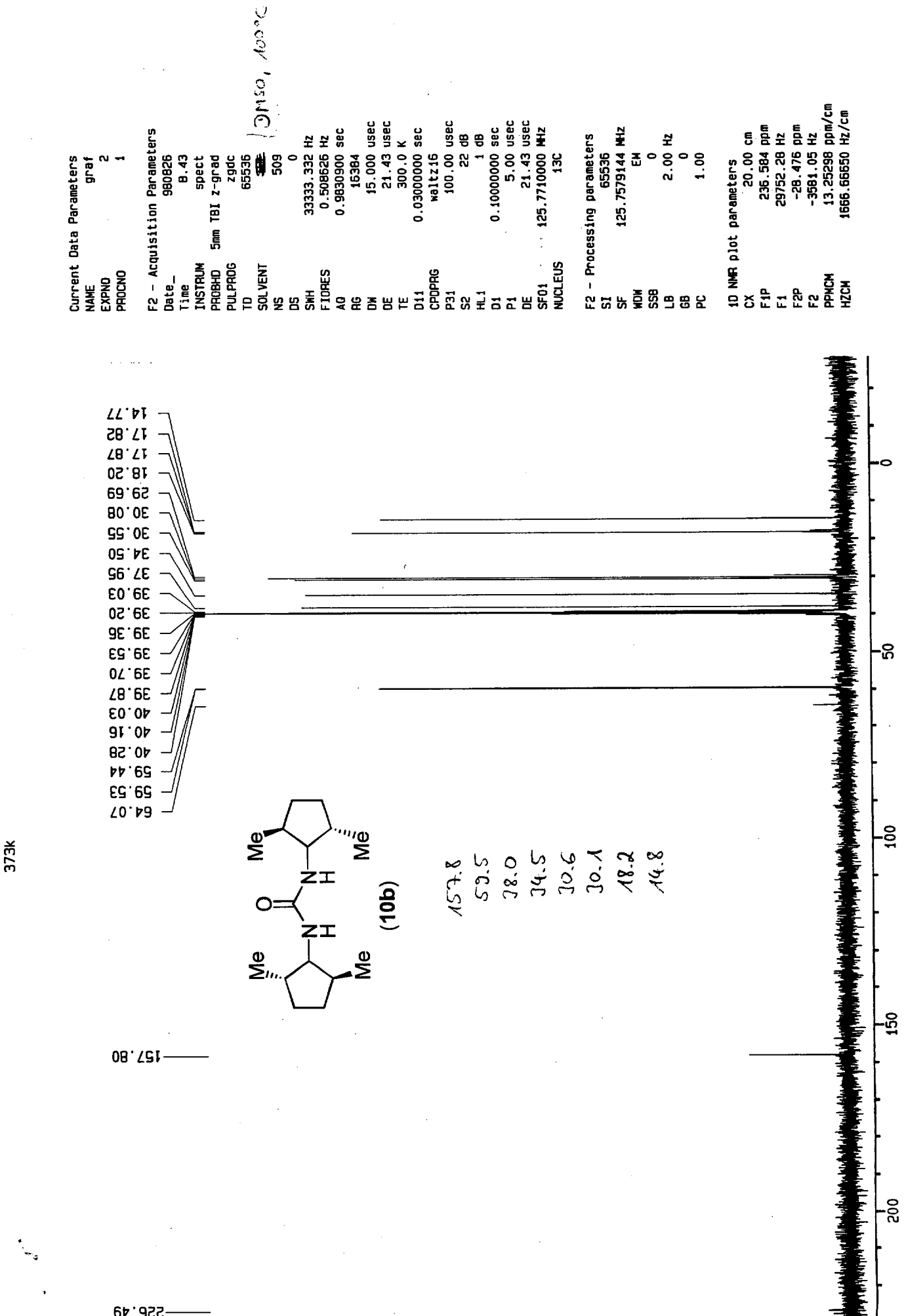


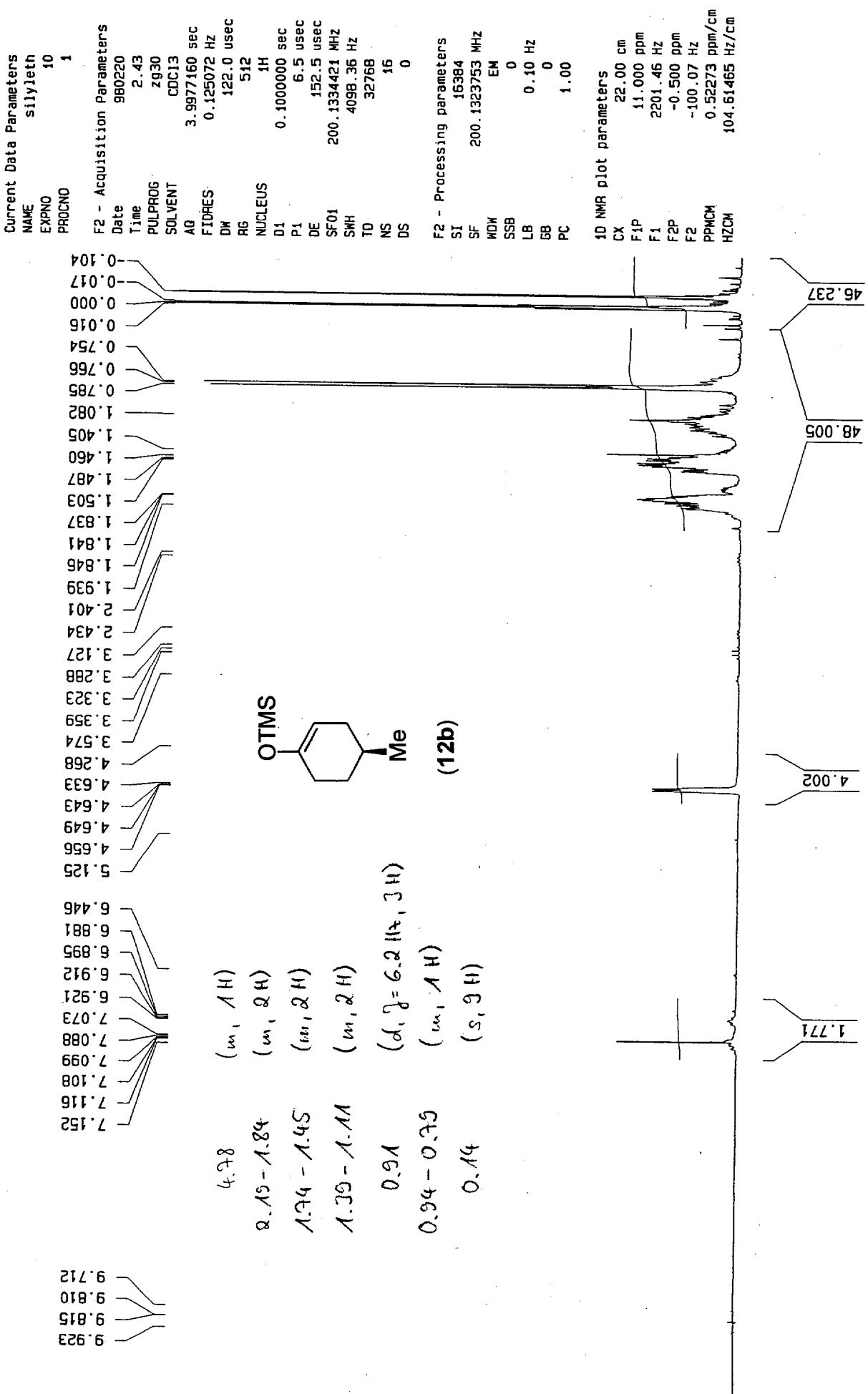


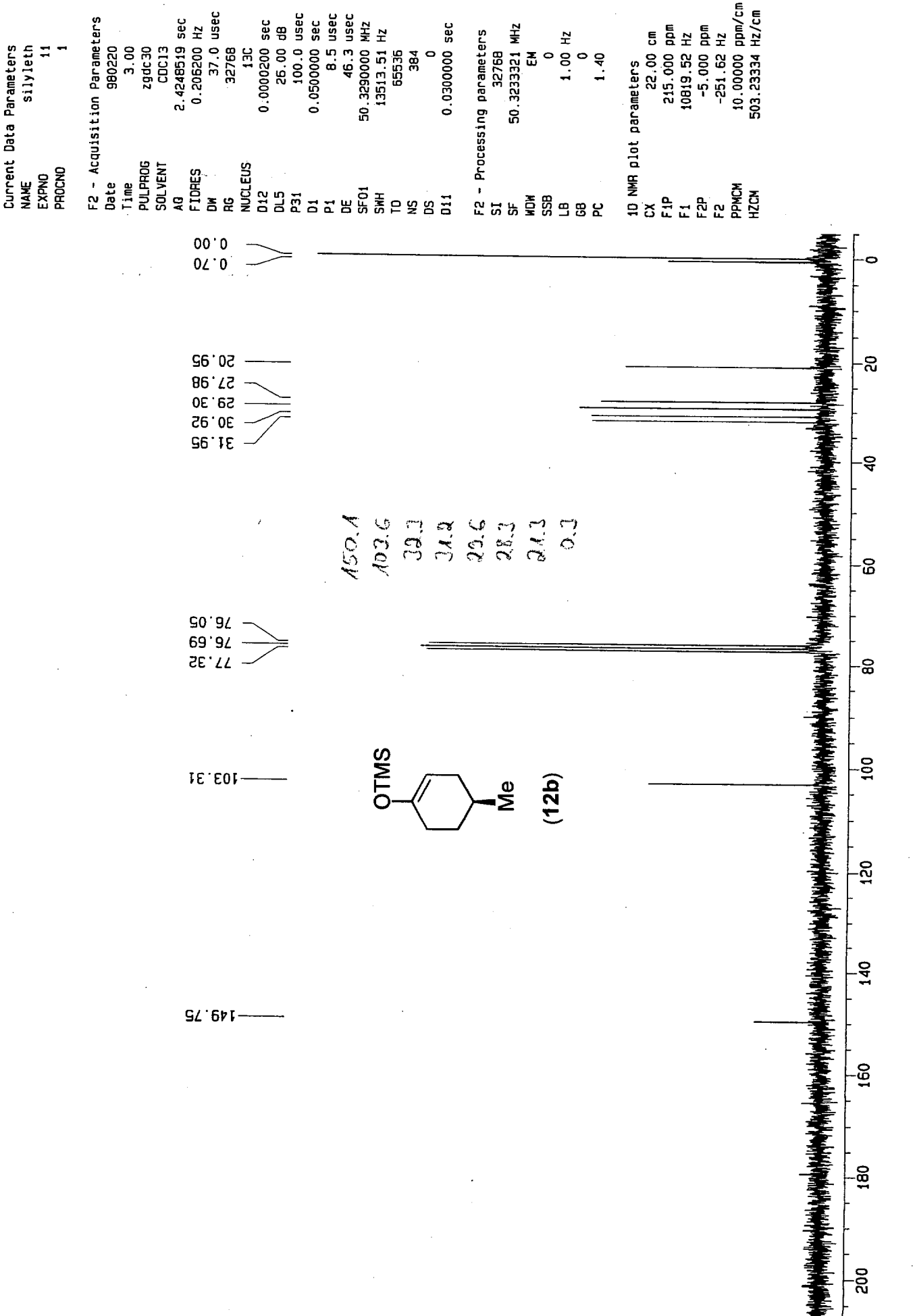


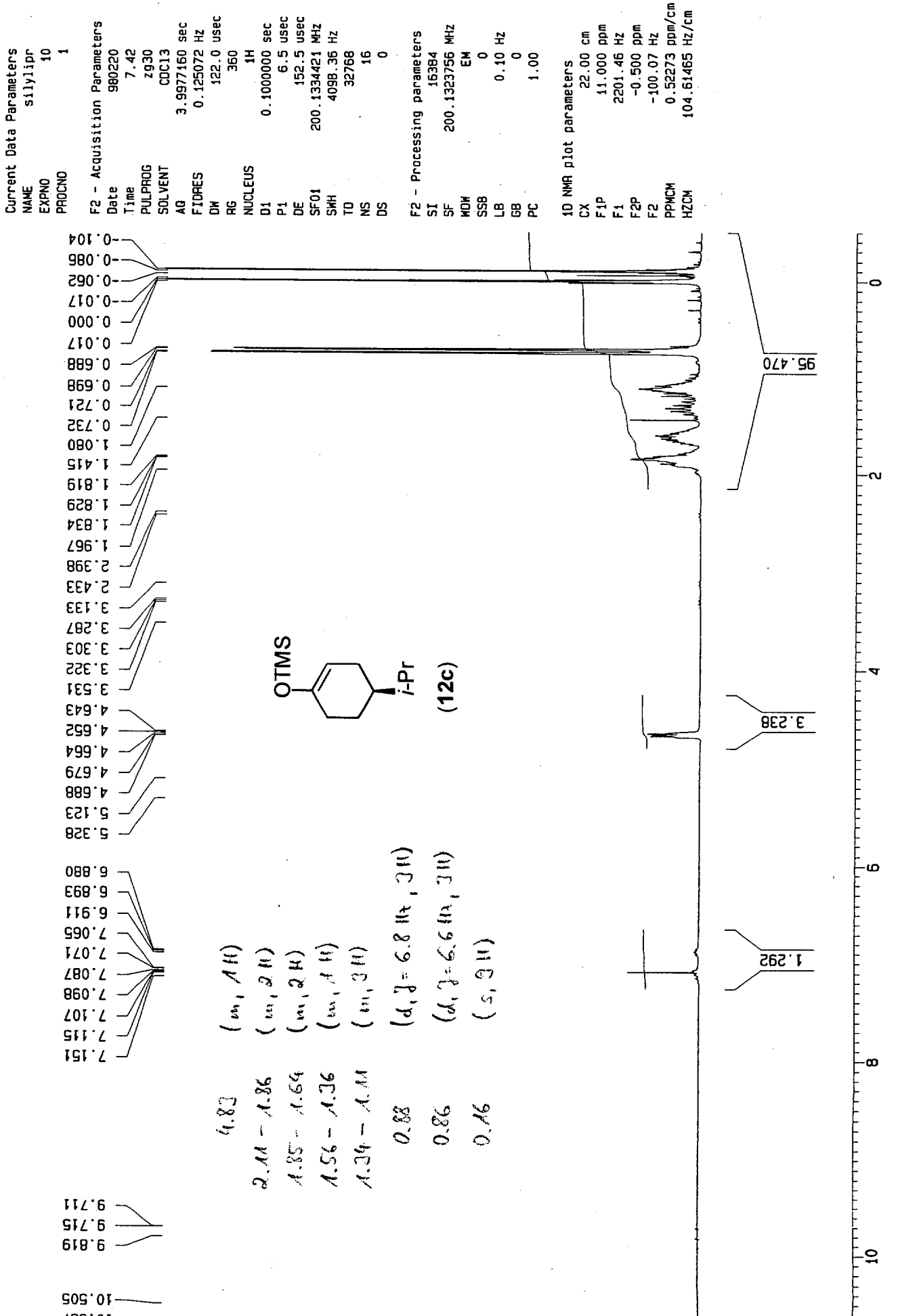


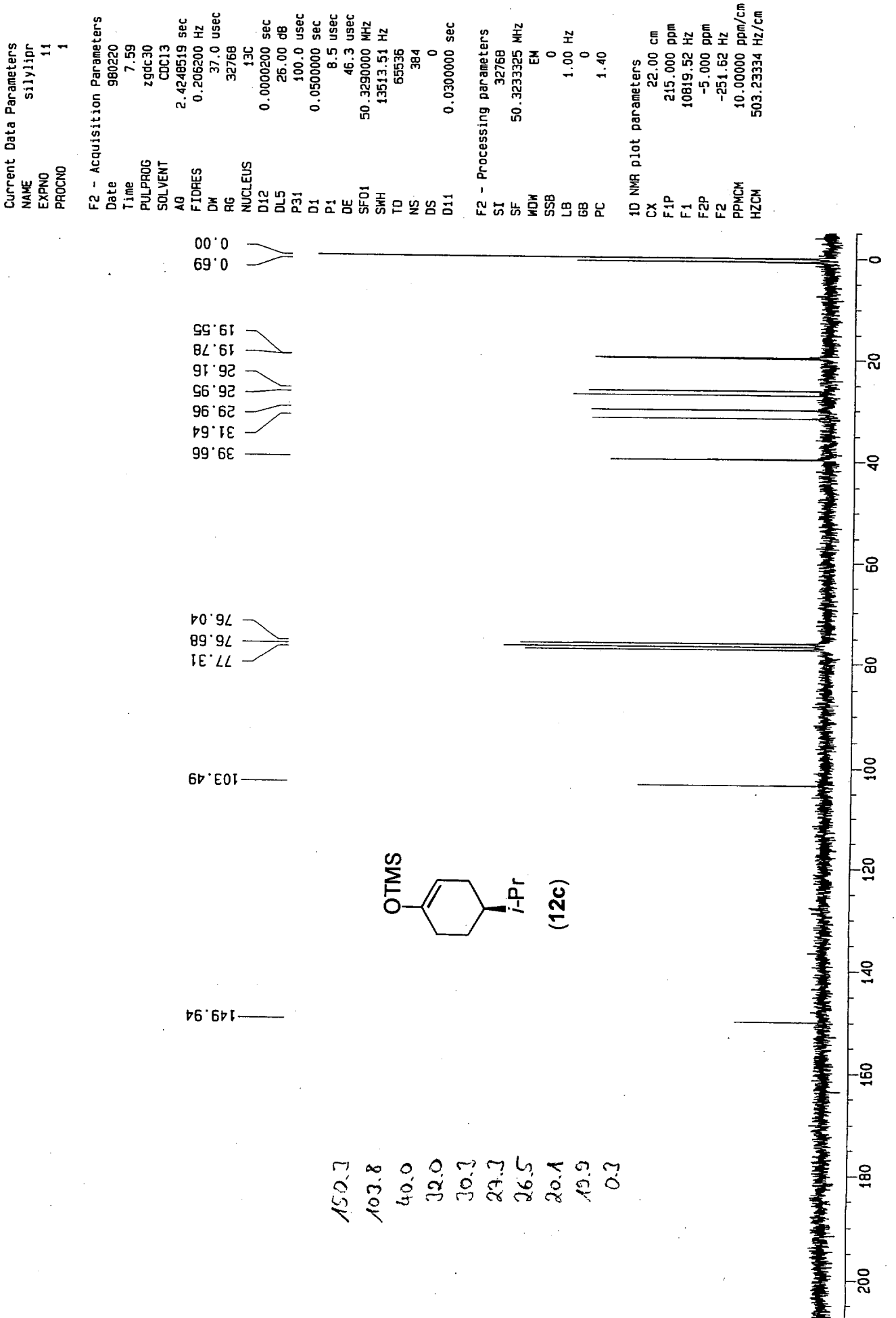












SILYLENOLETHER

FE120F.119
AU PROG:
XOO.AU
DATE 12-2-98

SA.NA MALAN
SA.NO FE12 119
SOLVENT CDCl3
SF 300.133
SY 210.0
O1 5200.000
SI 32768
TD 32768
SW 6024.096
HZ/PT .368

AQ 2.720
RG 2
NS 16
TE 298

O2 3200.000
DP 63L PO

LB .100
GB .100
CX 28.00
CY 12.50
F1 9.201P
F2 -.799P
HZ/CM 107.192
PPM/CM .357
SR 3405.35

PPM
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7.12351
7.10770
7.09989

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4.85853
4.85374

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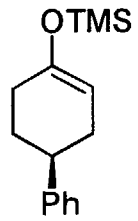
-.02111

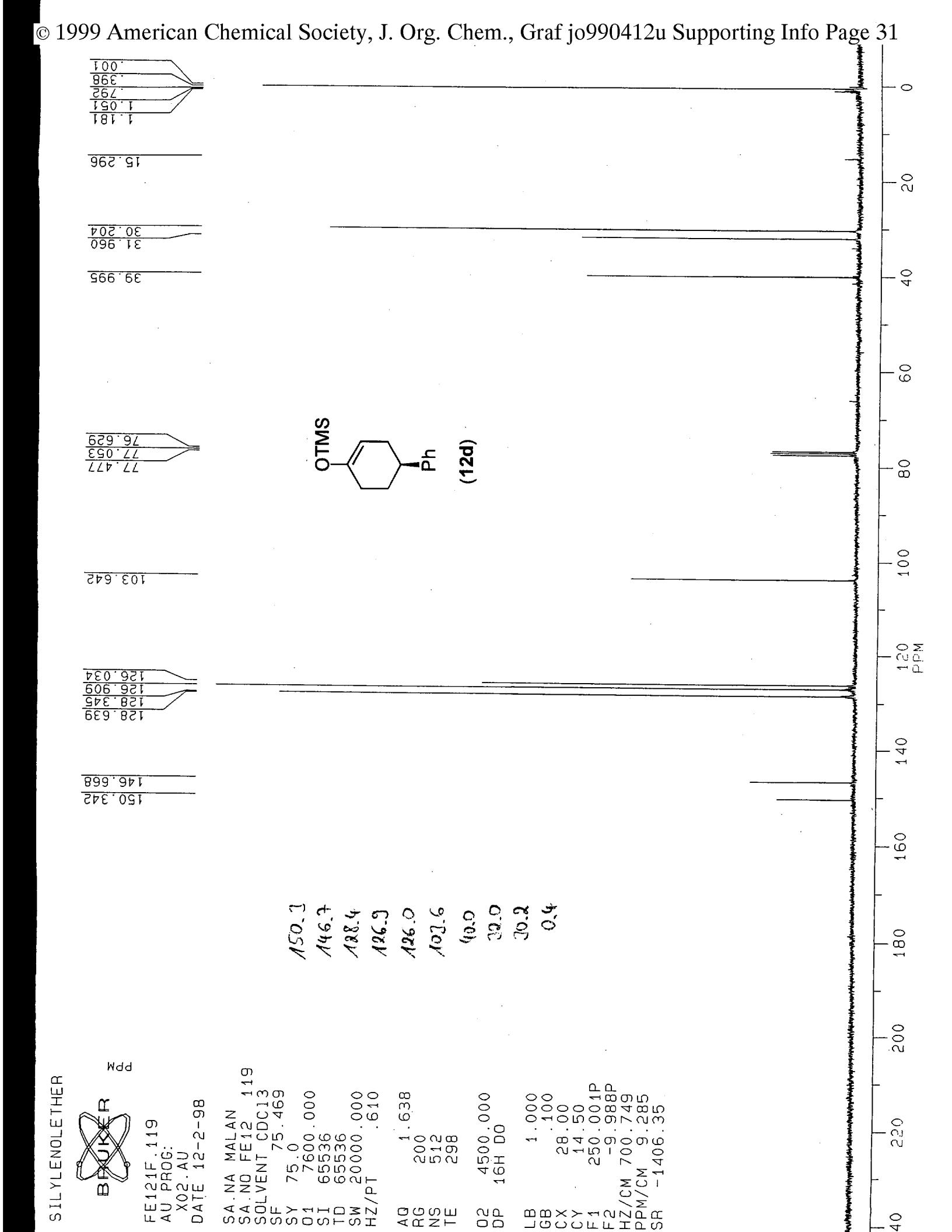
7.25 - 7.06 (m, 5H)
4.86 (m, 1H)
2.74 - 2.58 (m, 1H)
2.27 - 2.04 (m, 3H)
2.03 - 1.69 (m, 3H)
0.13 (s, 9H)



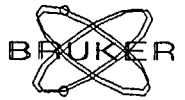
(12d)

8.637
1.604
.278
1.658
10.825
.880
.440
17.558

8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
PPM



ENOLETHER



PPM

7.20703

AP020F.145
AU PROG:
X00.AU
DATE 3-4-98

SA.NA MALAN2
SA.NO AP02 145
SOLVENT CDC13
SF 300.133
SY 210.0
O1 5200.000
SI 32768
TD 32768
SW 6024.096
HZ/PT .368

AQ 2.720
RG 2
NS 16
TE 298

O2 3200.000
DP 63L PO

LB .100
GB .100
CX 28.00
CY 12.50
F1 9.201P
F2 -.799P
HZ/CM 107.192
PPM/CM .357
SR 3383.28

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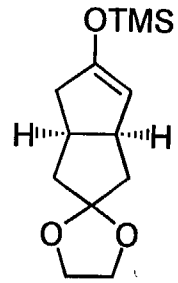
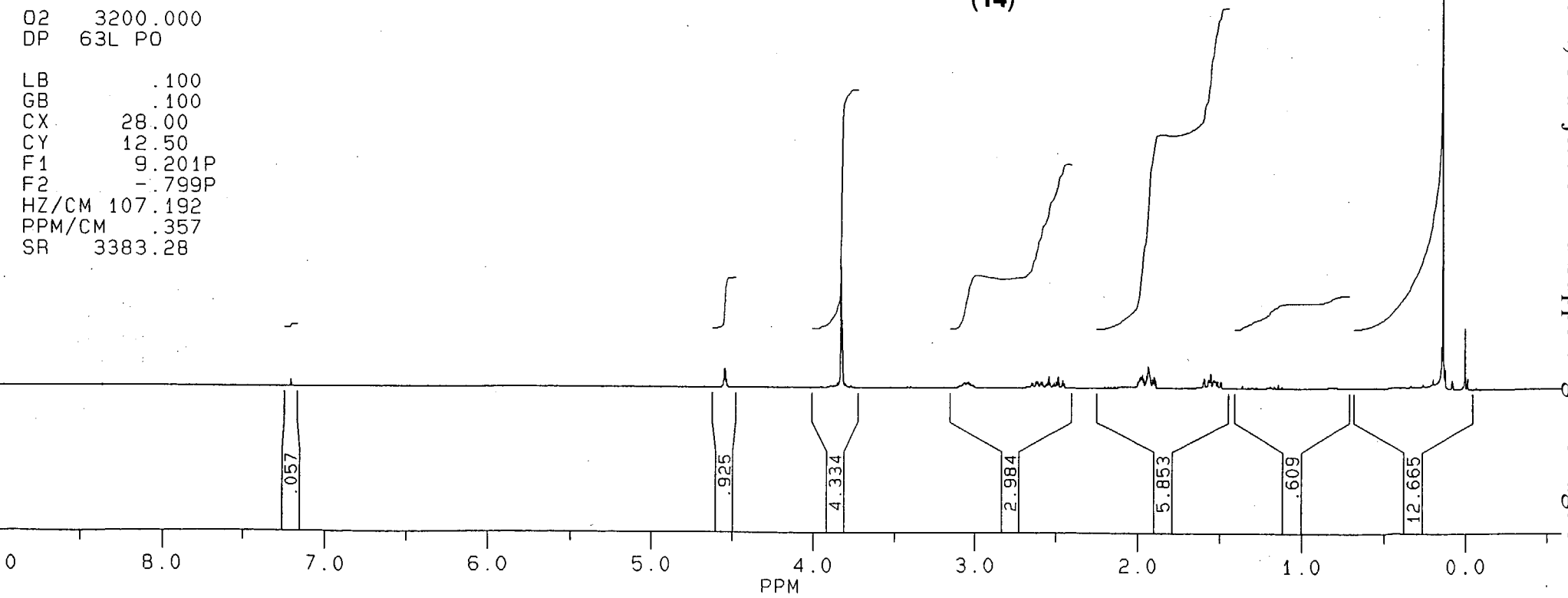
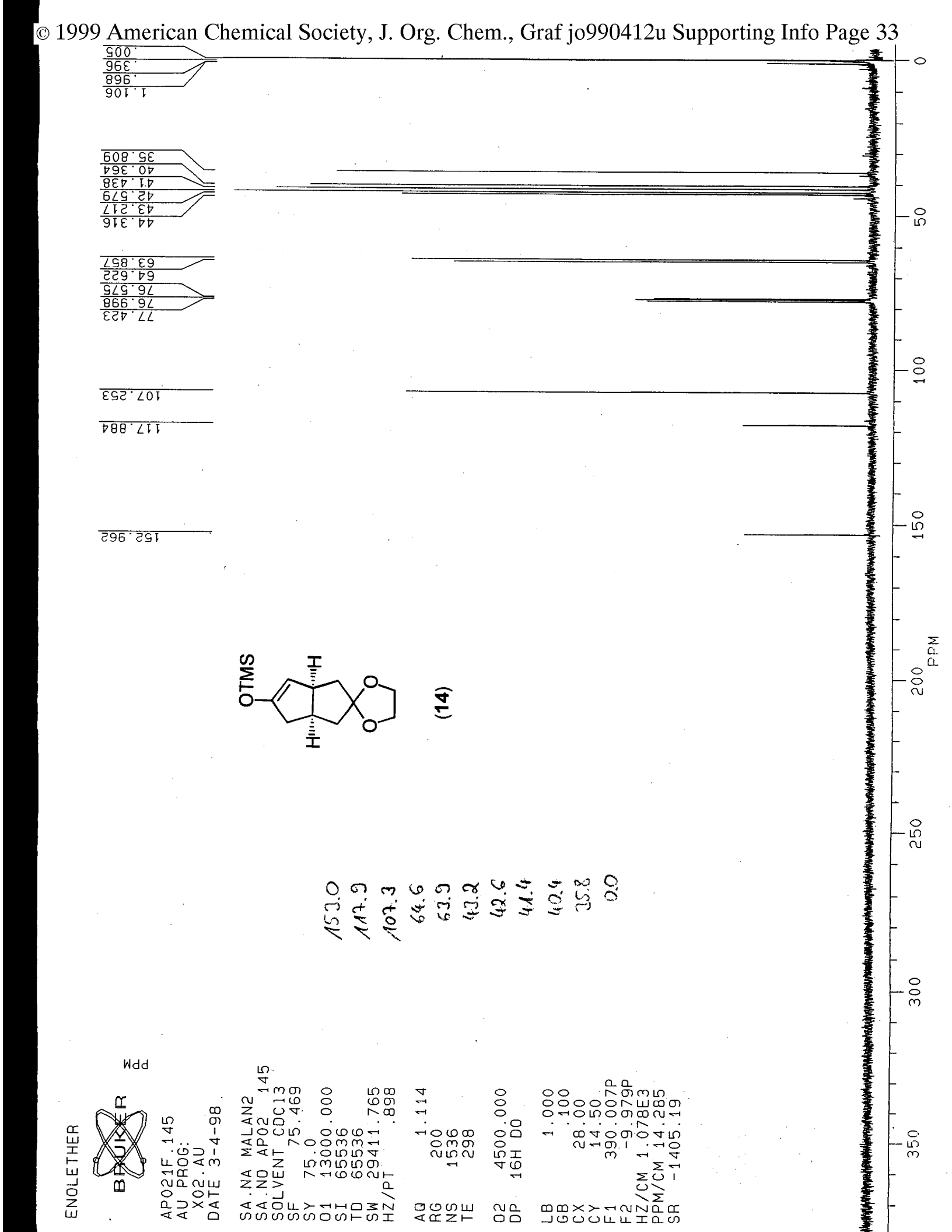
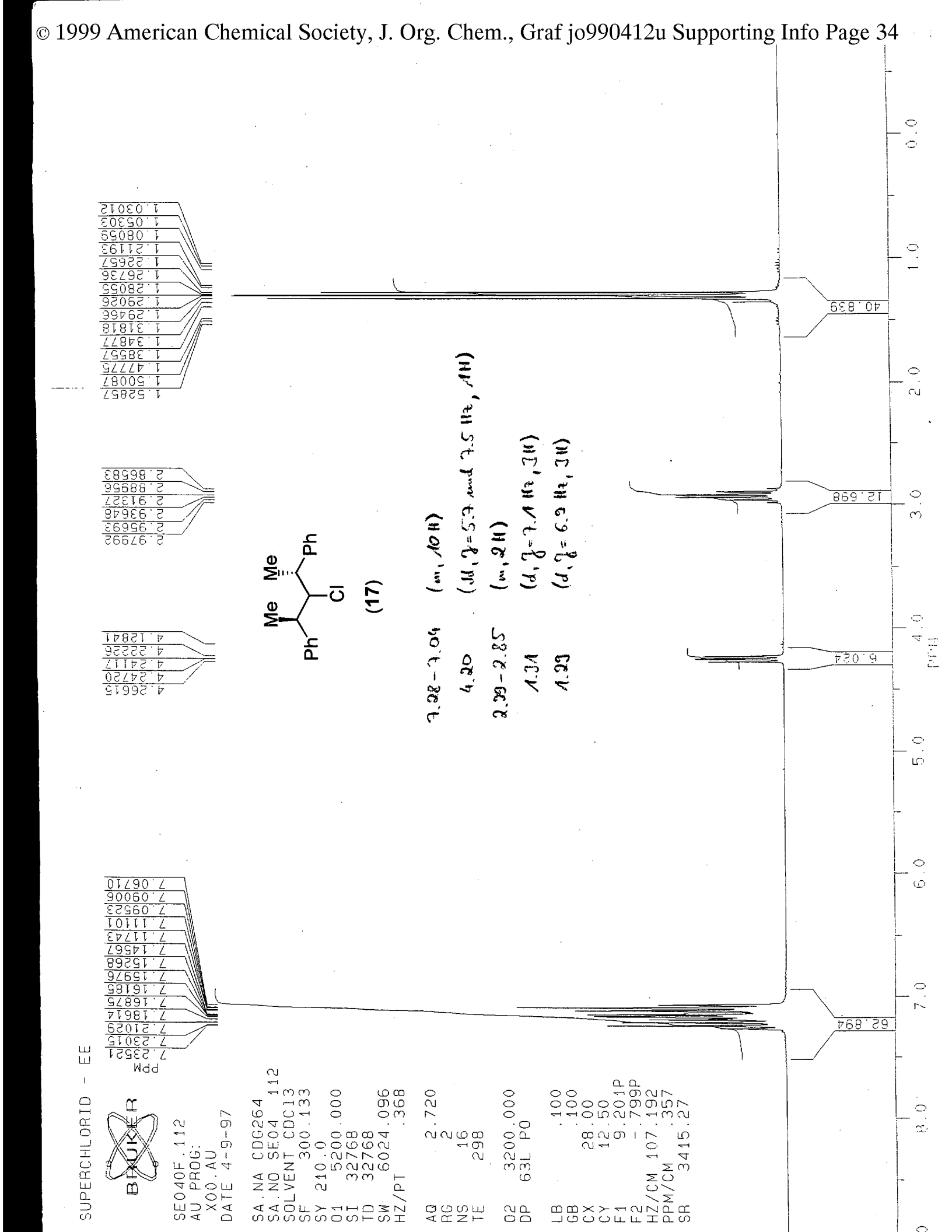
4.54 (m, 1H)
3.82 (m, 4H)
3.22 - 2.98 (m, 1H)
2.63 - 2.43 (m, 2H)
2.02 - 1.87 (m, 3H)
1.62 - 1.47 (m, 2H)
0.13 (s, 9H)

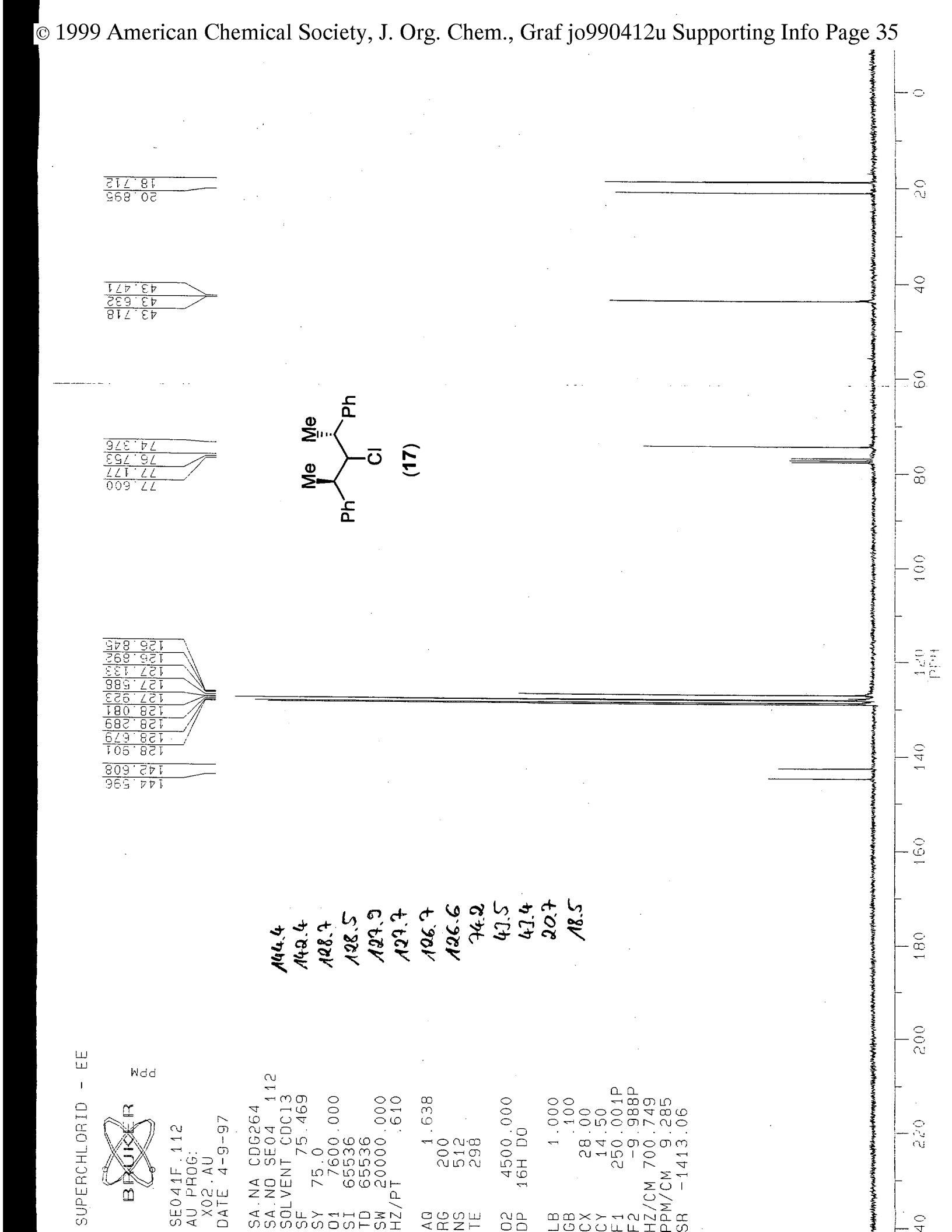


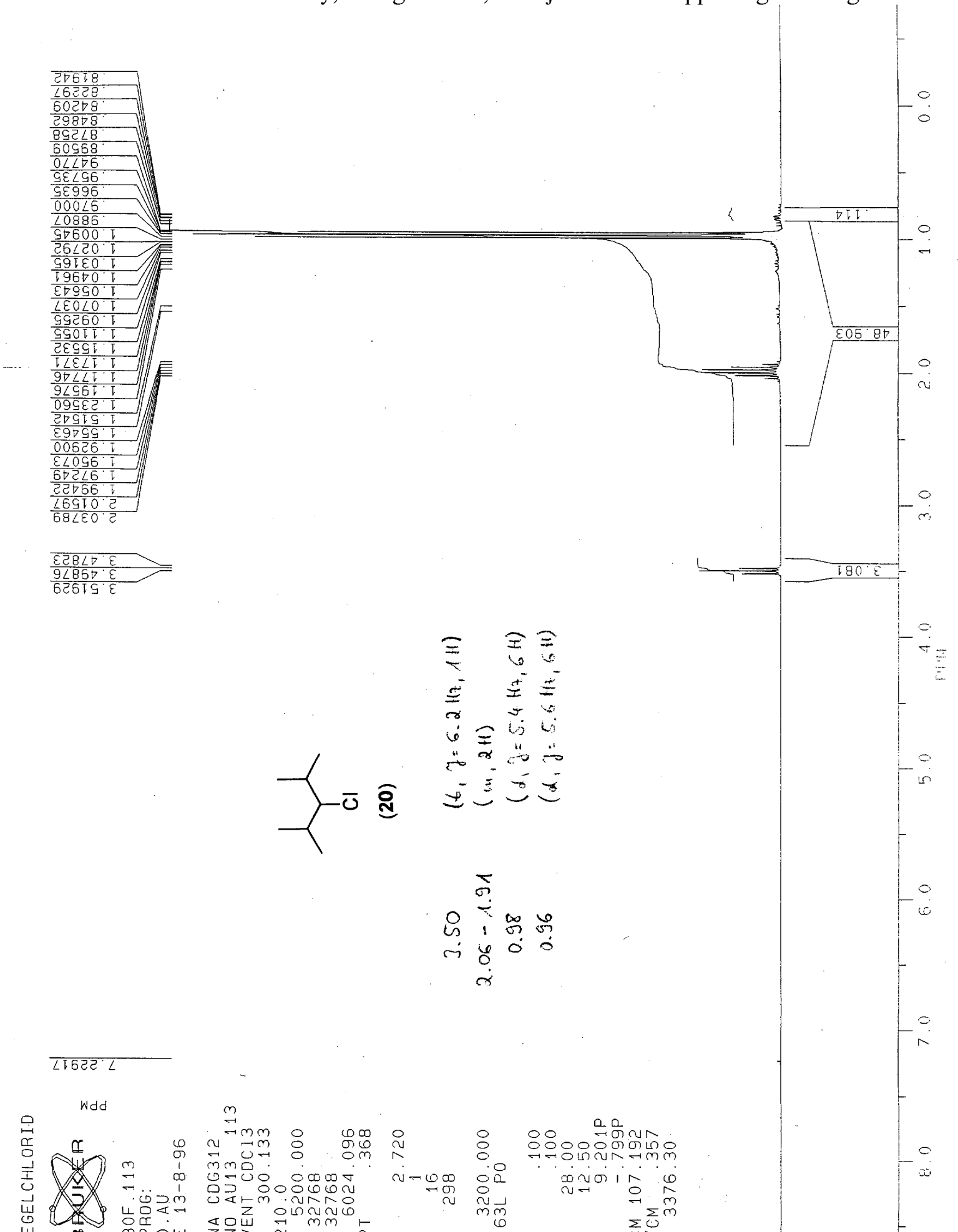
(14)

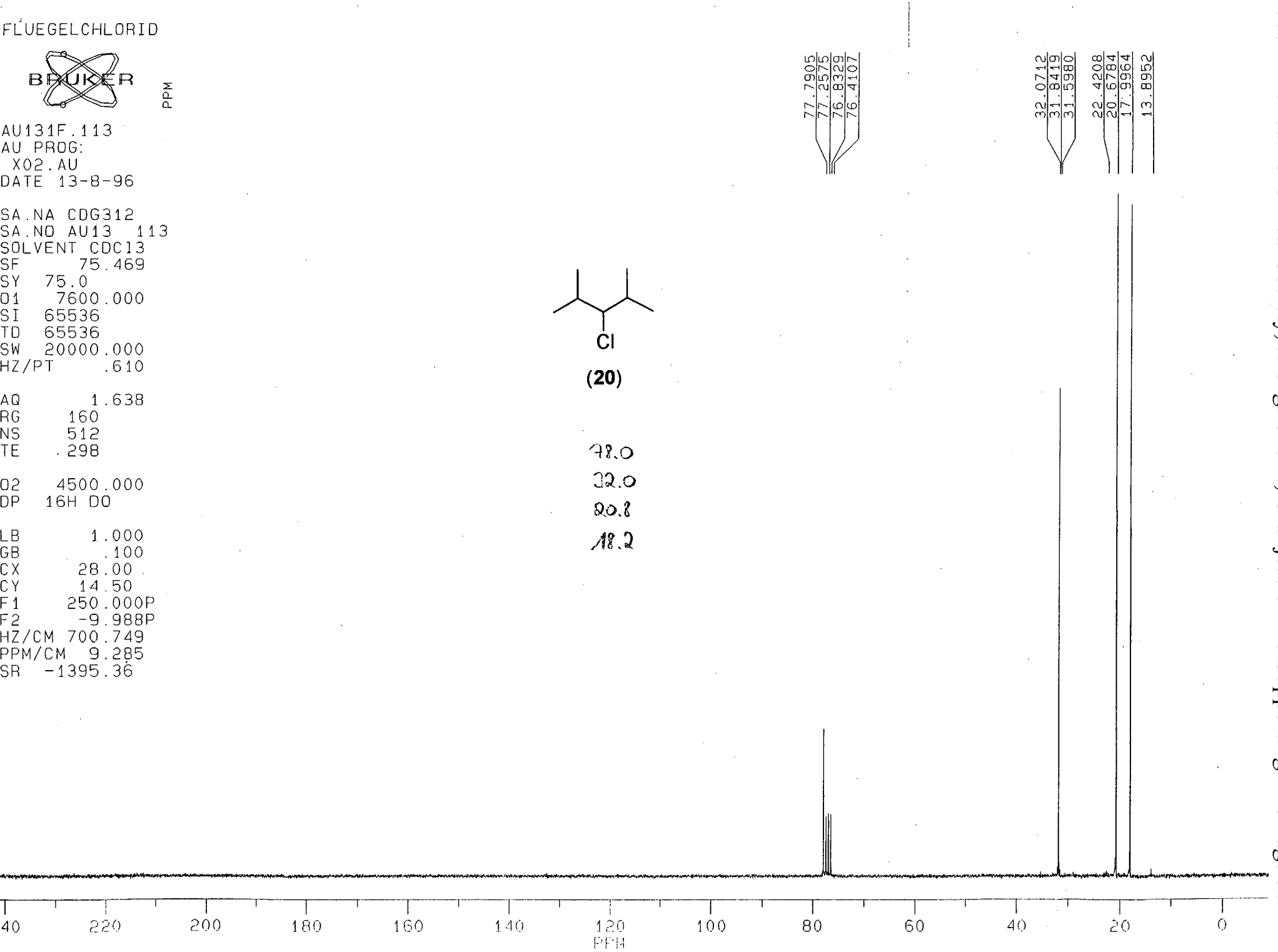


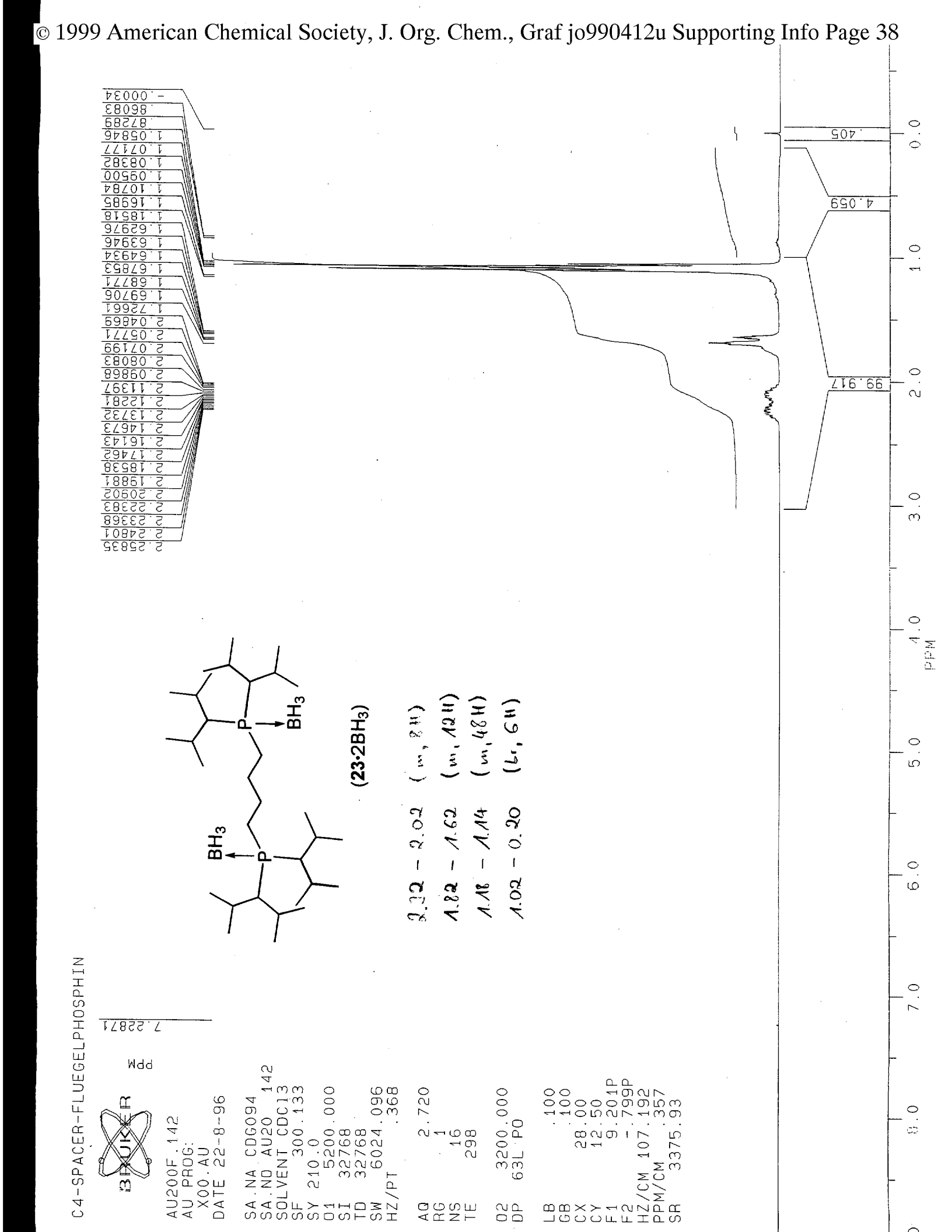


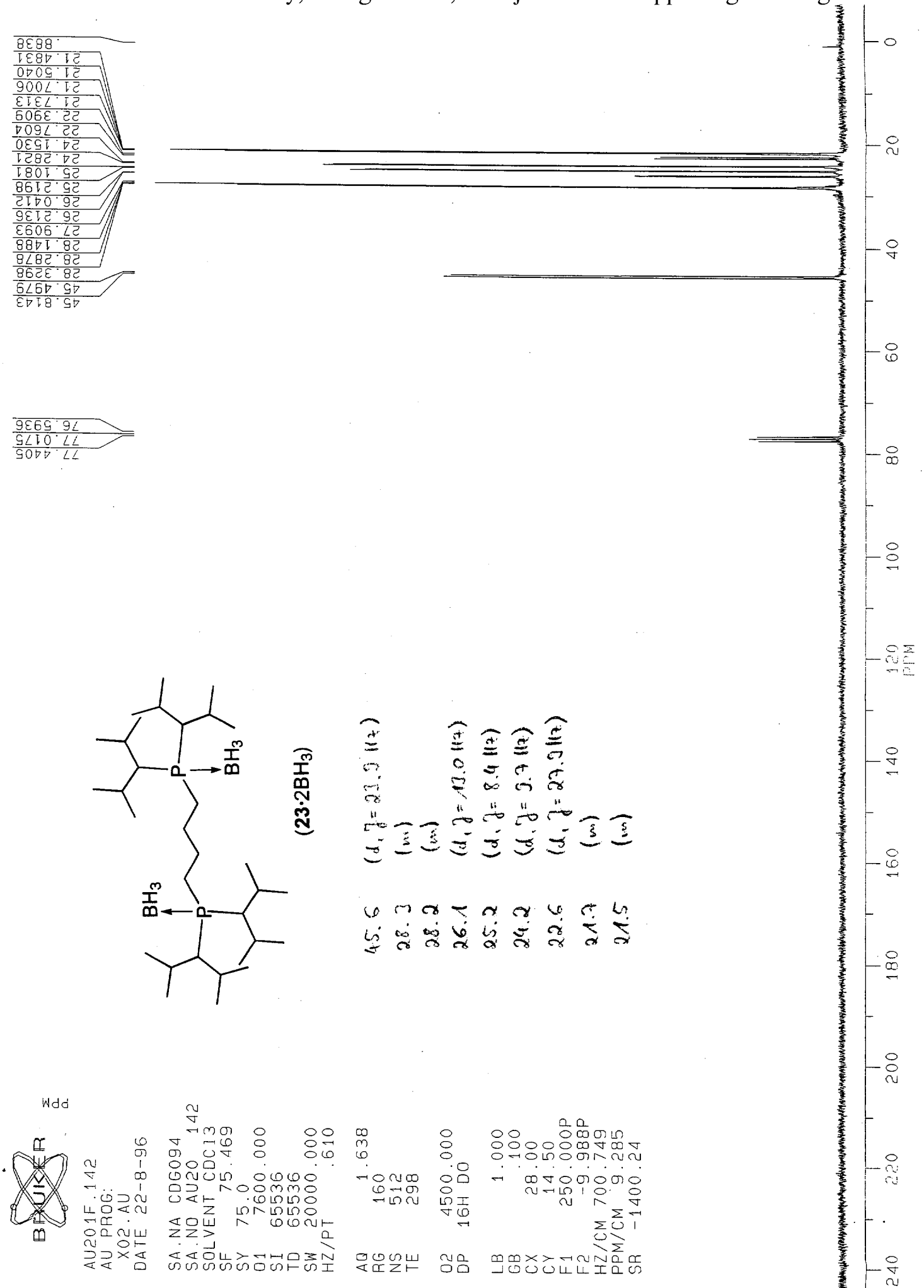




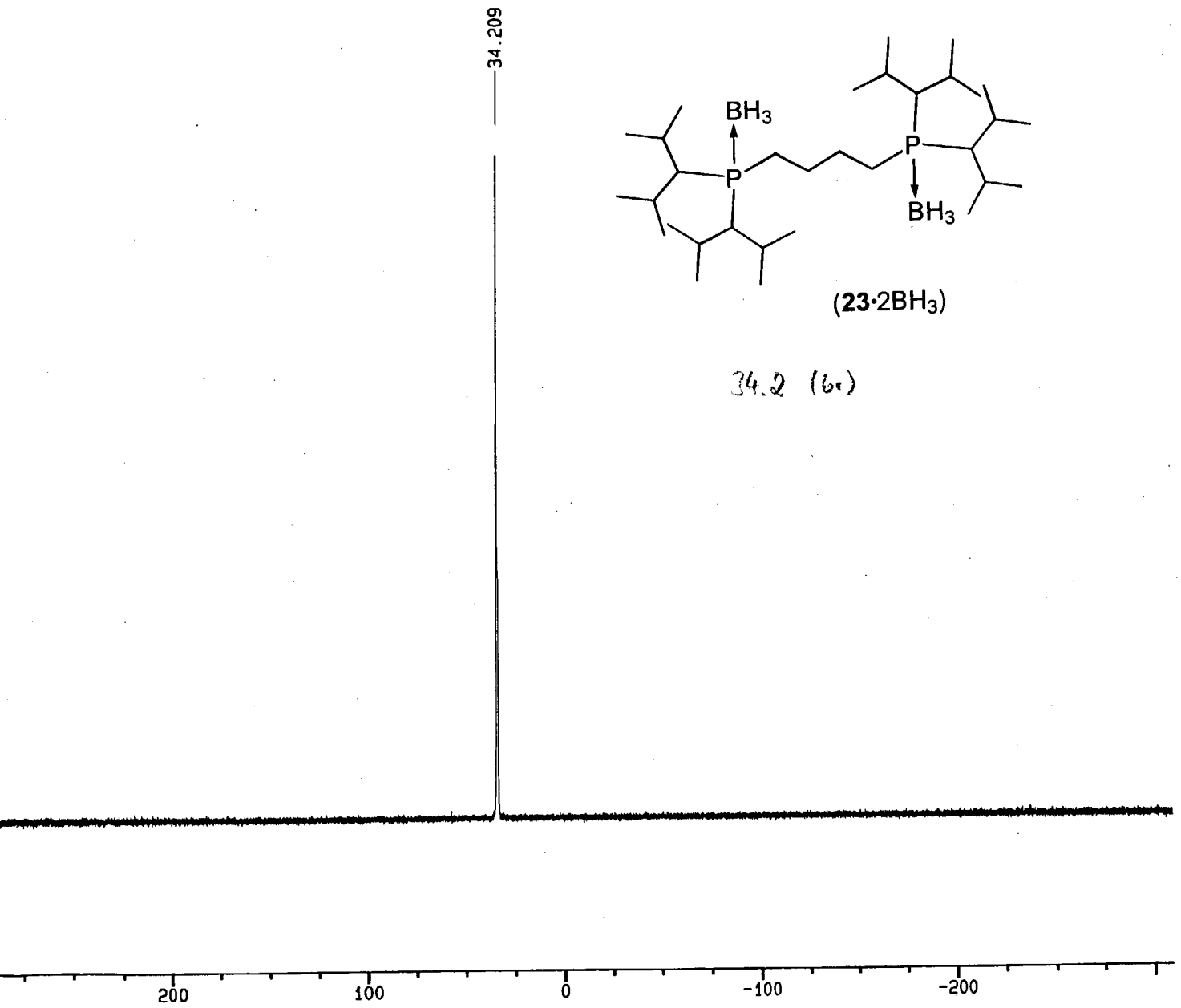








31p 048



(**23**·2BH_3)

34.2 (br)

Current Data Parameters

NAME	graf /Pü
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	960829
Time	9.40
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zgdc
TD	65536
SOLVENT	~~Aceton~~ $CDCl_3$
NS	70
DS	2
SWH	125000.000 Hz
FIDRES	1.907349 Hz
AQ	0.2621940 sec
RG	1024
DW	4.000 usec
DE	5.71 usec
TE	300.0 K
D11	0.0300000 sec
CPDPRG	waltz16
P31	100.00 usec
S2	18 dB
HL1	3 dB
D1	0.10000000 sec
P1	8.00 usec
DE	5.71 usec
SFO1	202.4561167 MHz
NUCLEUS	31P

F2 - Processing parameters

SI	32768
SF	202.4560977 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	308.803 ppm
F1	62519.10 Hz
F2P	-308.615 ppm
F2	-62480.91 Hz
PPMCM	30.87089 ppm/cm
HZCM	6250.00049 Hz/cm