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Total Synthesis of Himandravine

Samuel Chackalamannil* and Robert Davies

Schering-Plough Research Institute,

2015 Galloping Hill Road, Kenilworth, New Jersey 07033

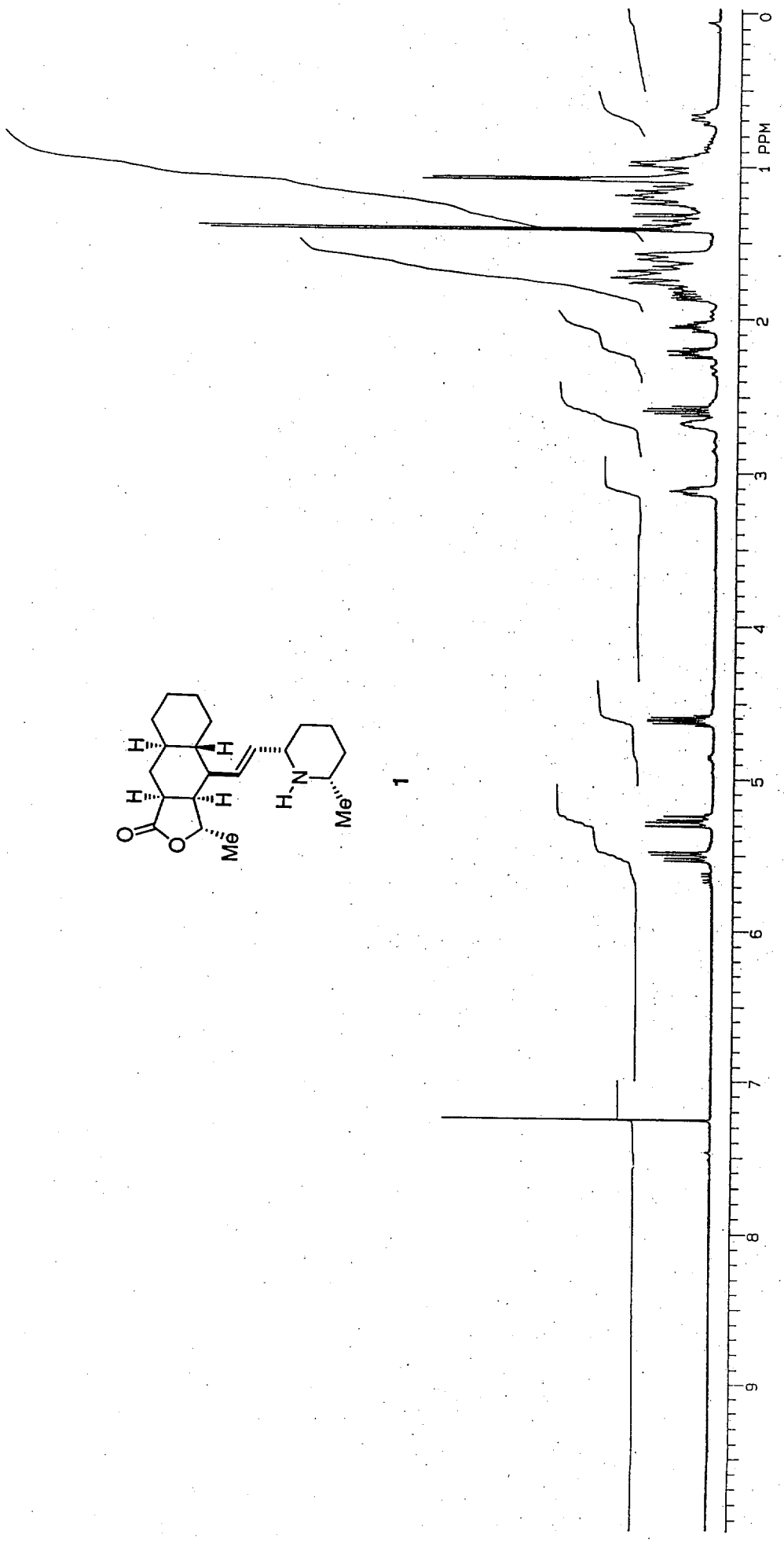
samuel.chackalamannil@spcorp.com

Andrew T. McPhail

P. M. Gross Chemistry Laboratory, Duke University, Durham , NC 27708-0346

Supporting Information

^{1}H and ^{13}C NMR spectra for compounds **1**, **5**, **8**, **9**, **11**, **14**, and **15**. Tables of crystal data, fractional coordinates and thermal parameters, and interatomic distances with standard deviation for the hydrochloride of himandravine (**1a**).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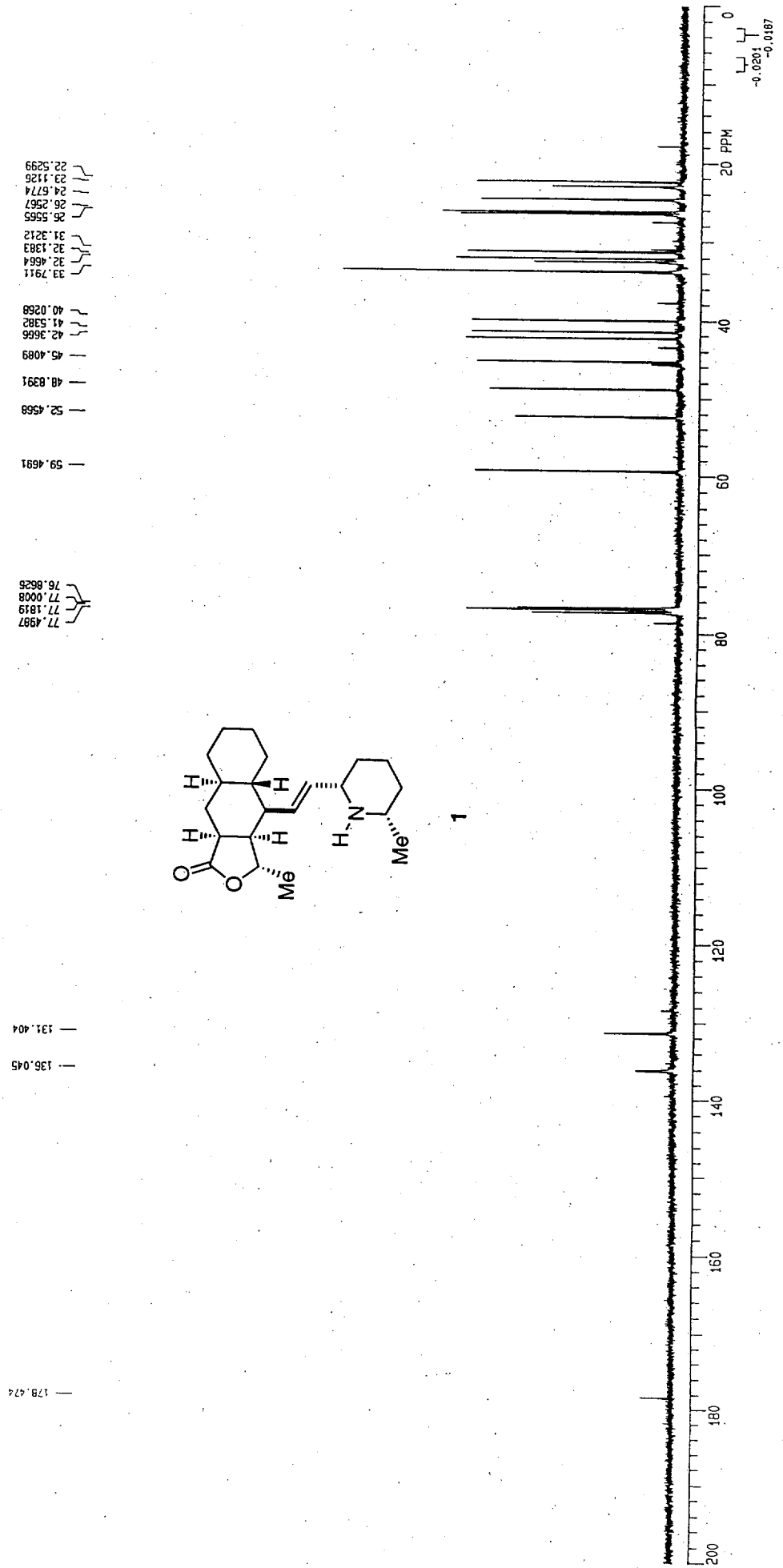

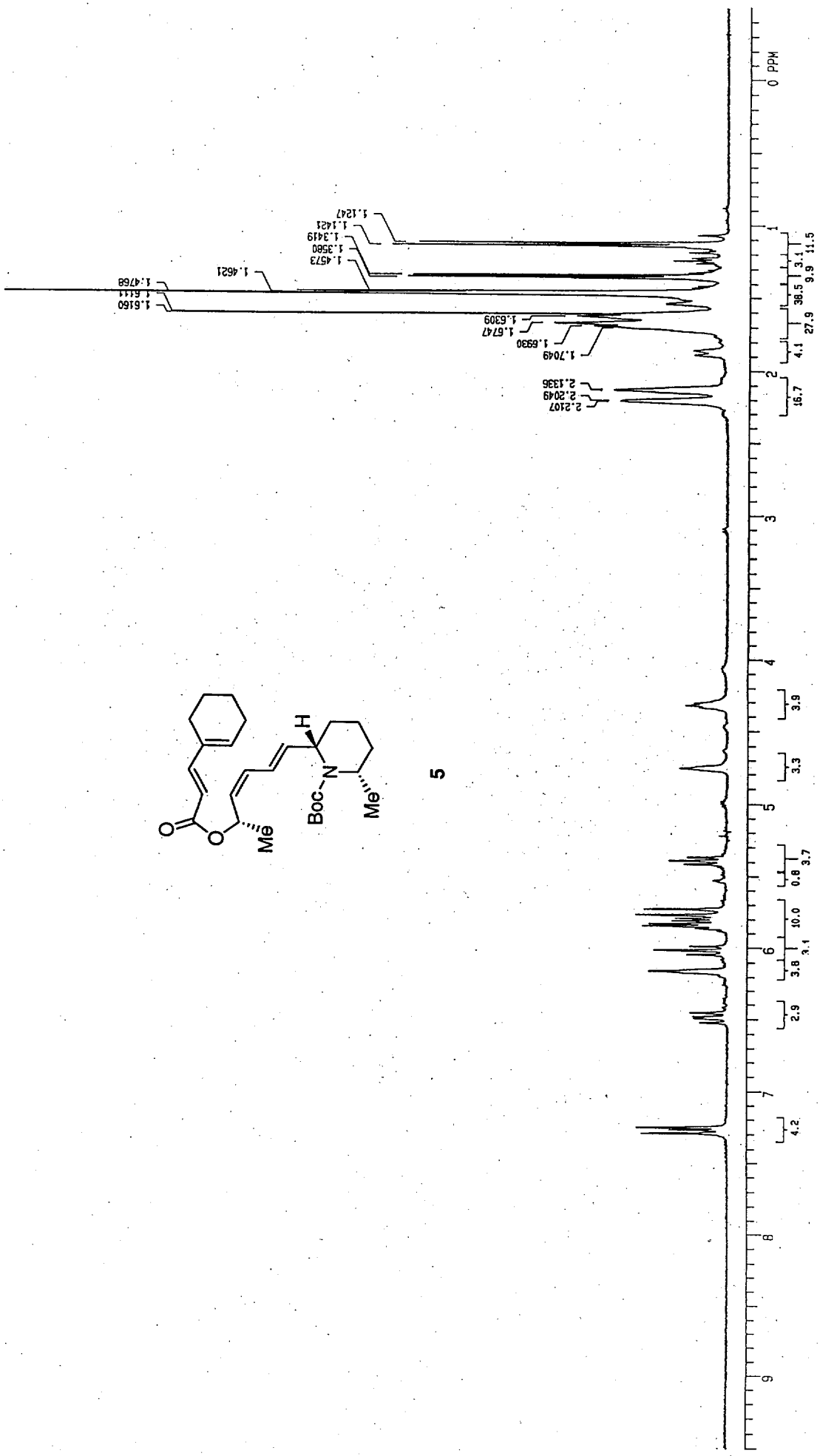
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O
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Boc
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Me

5


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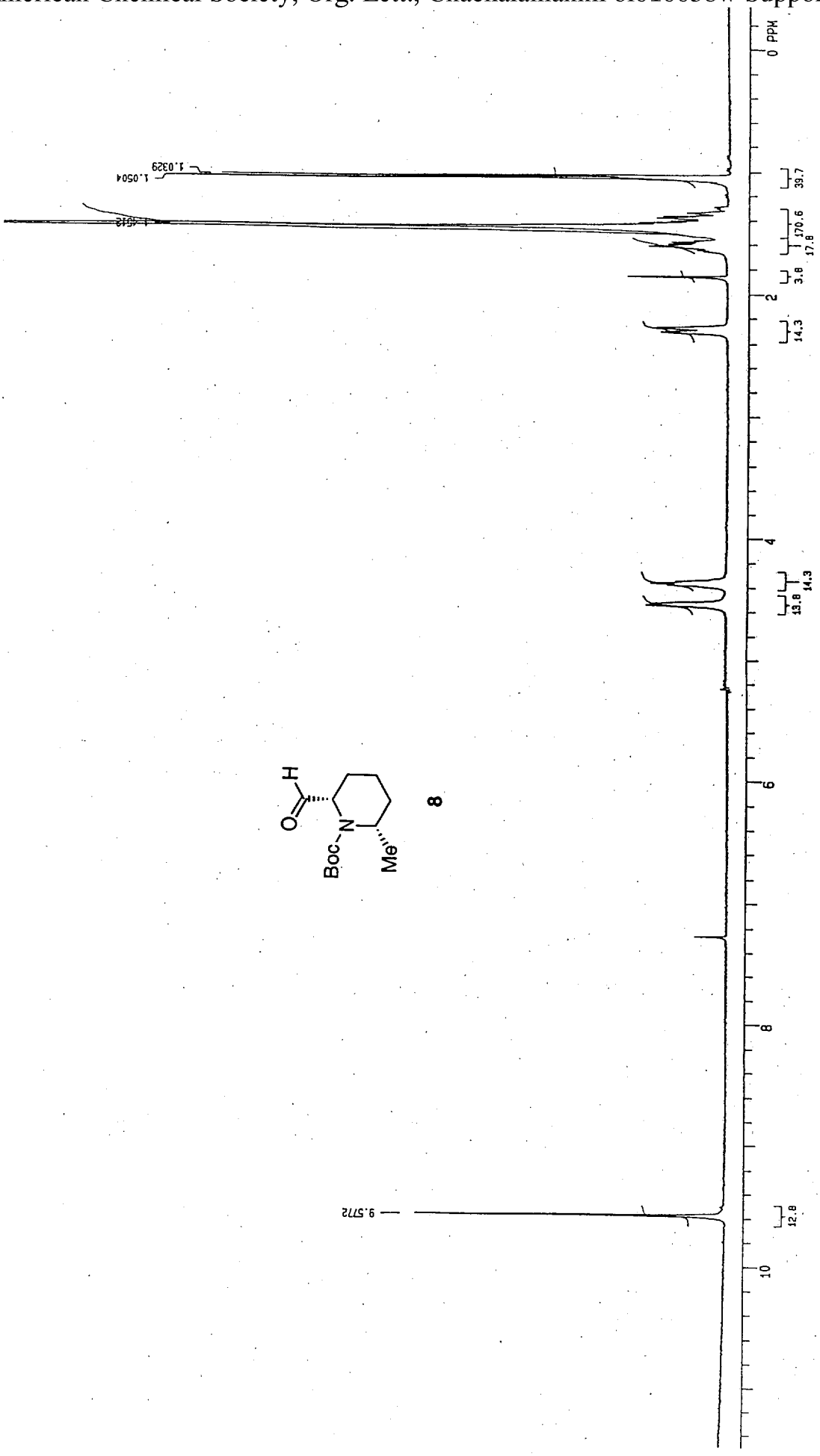

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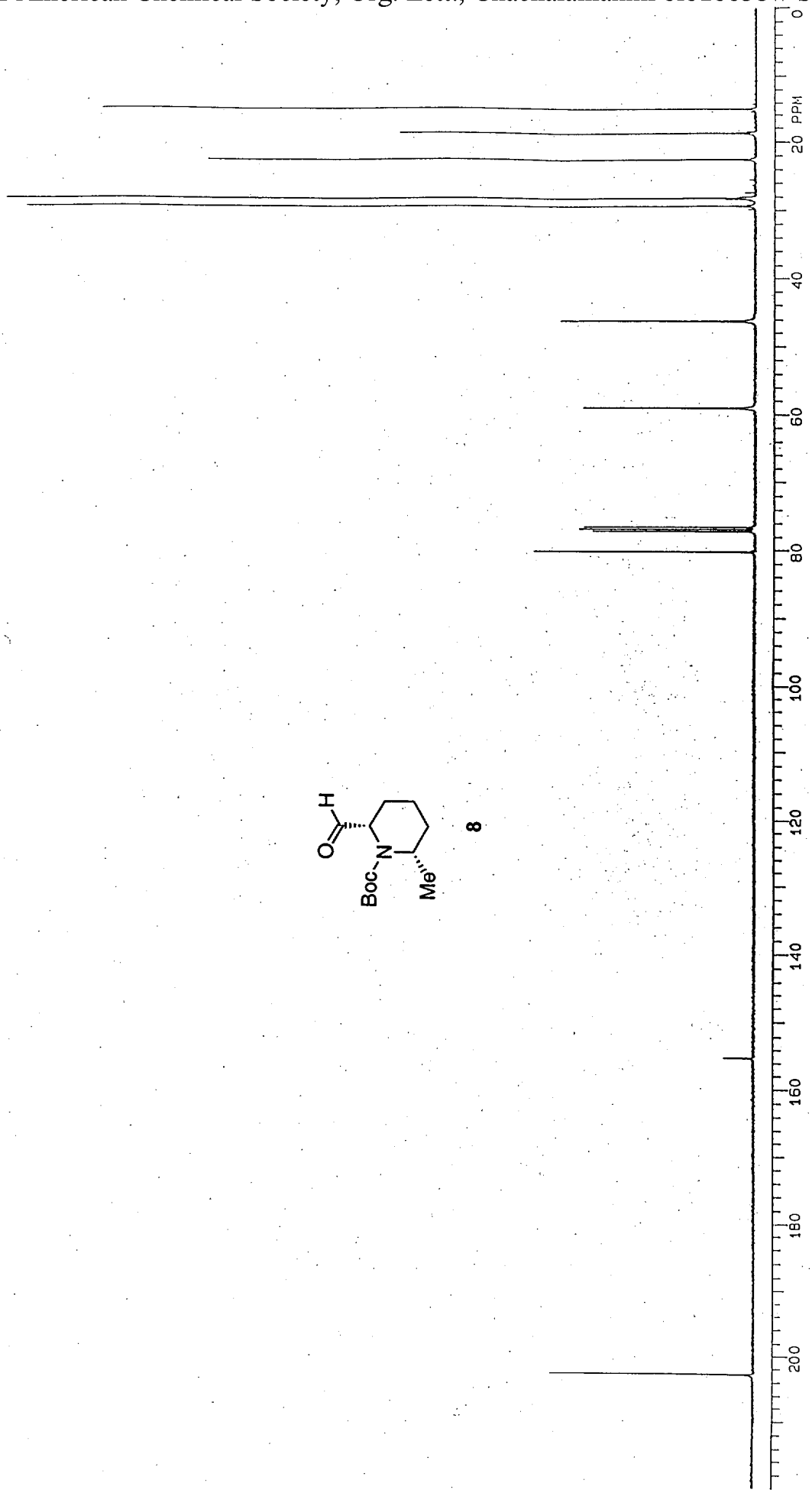

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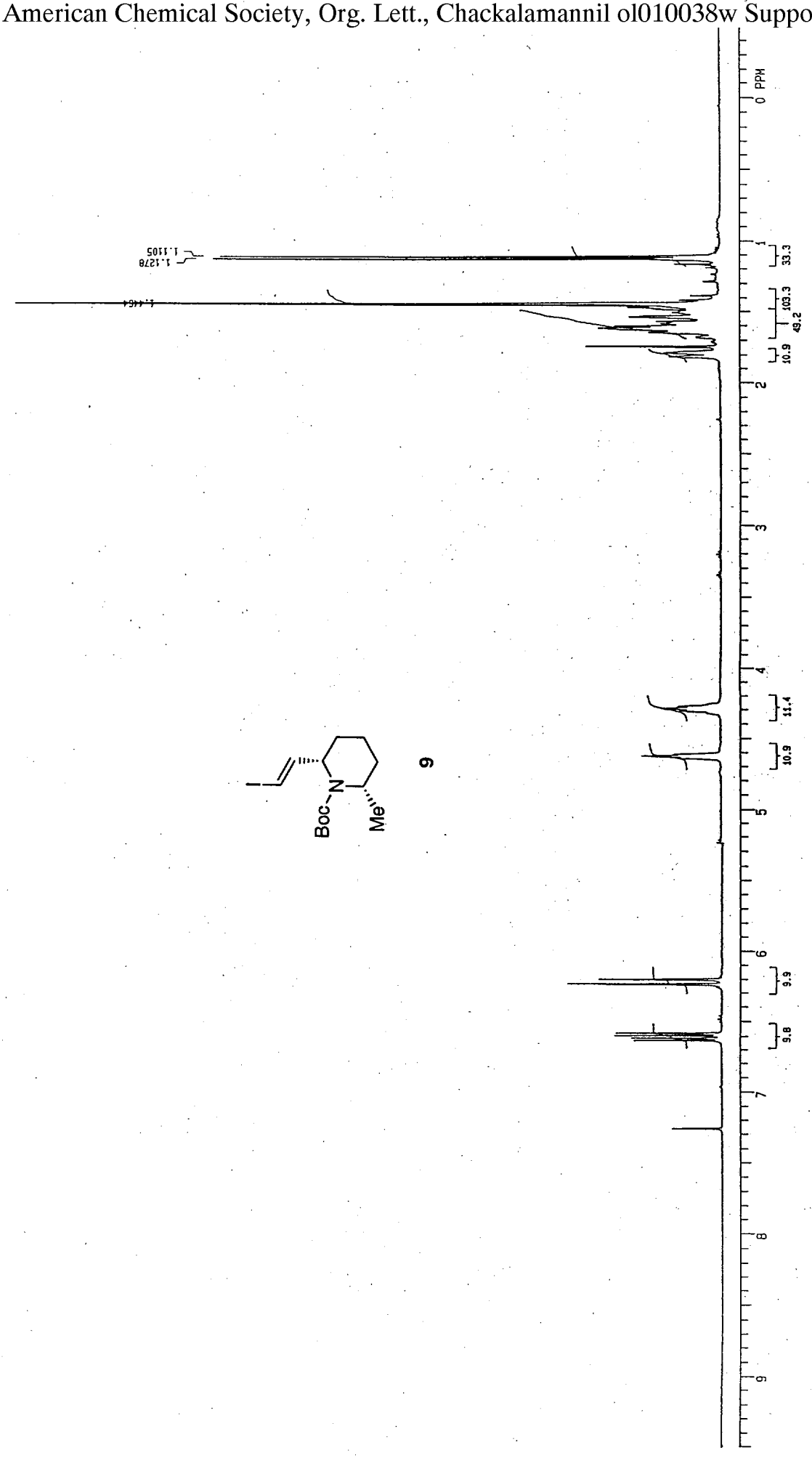
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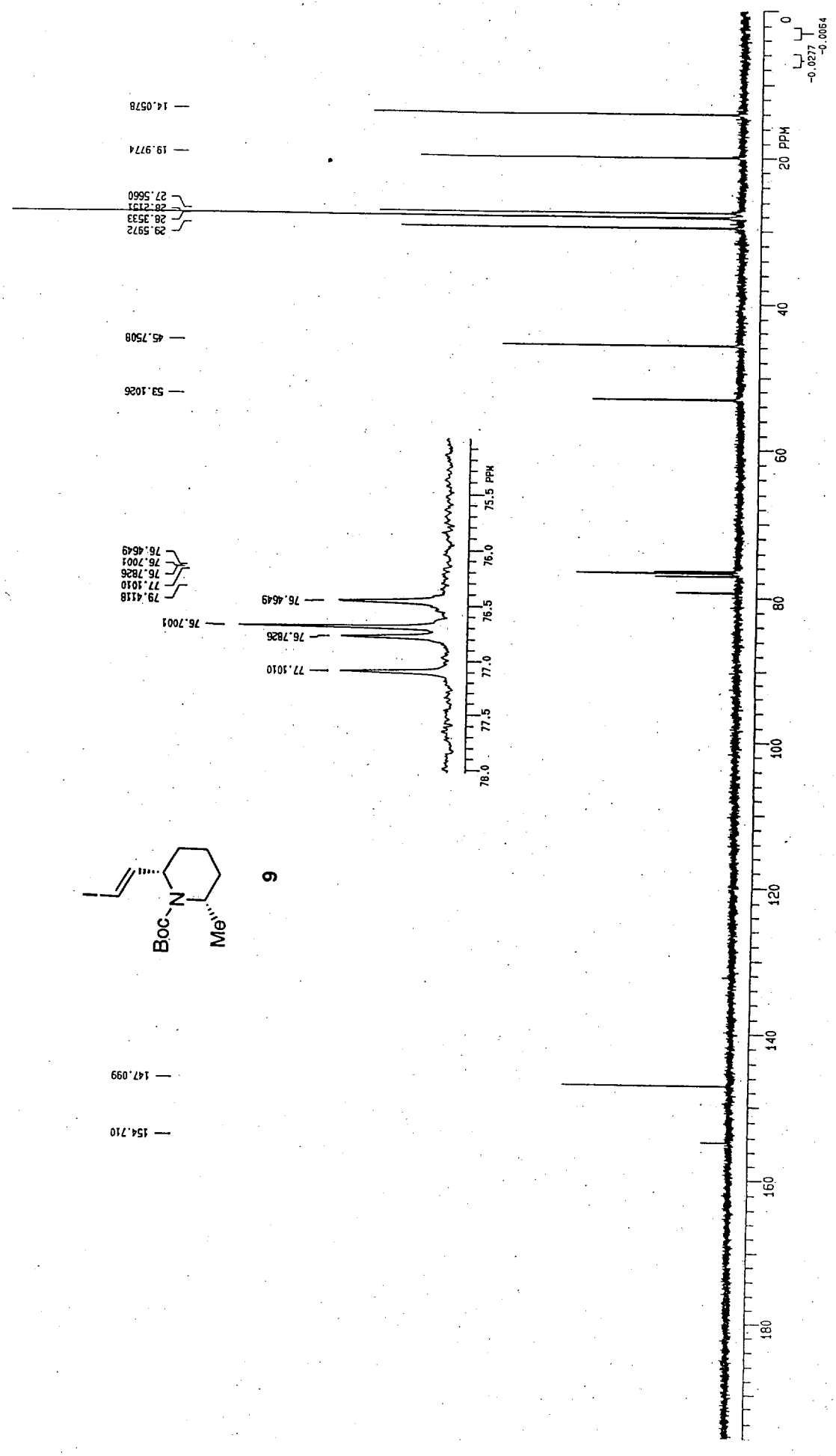

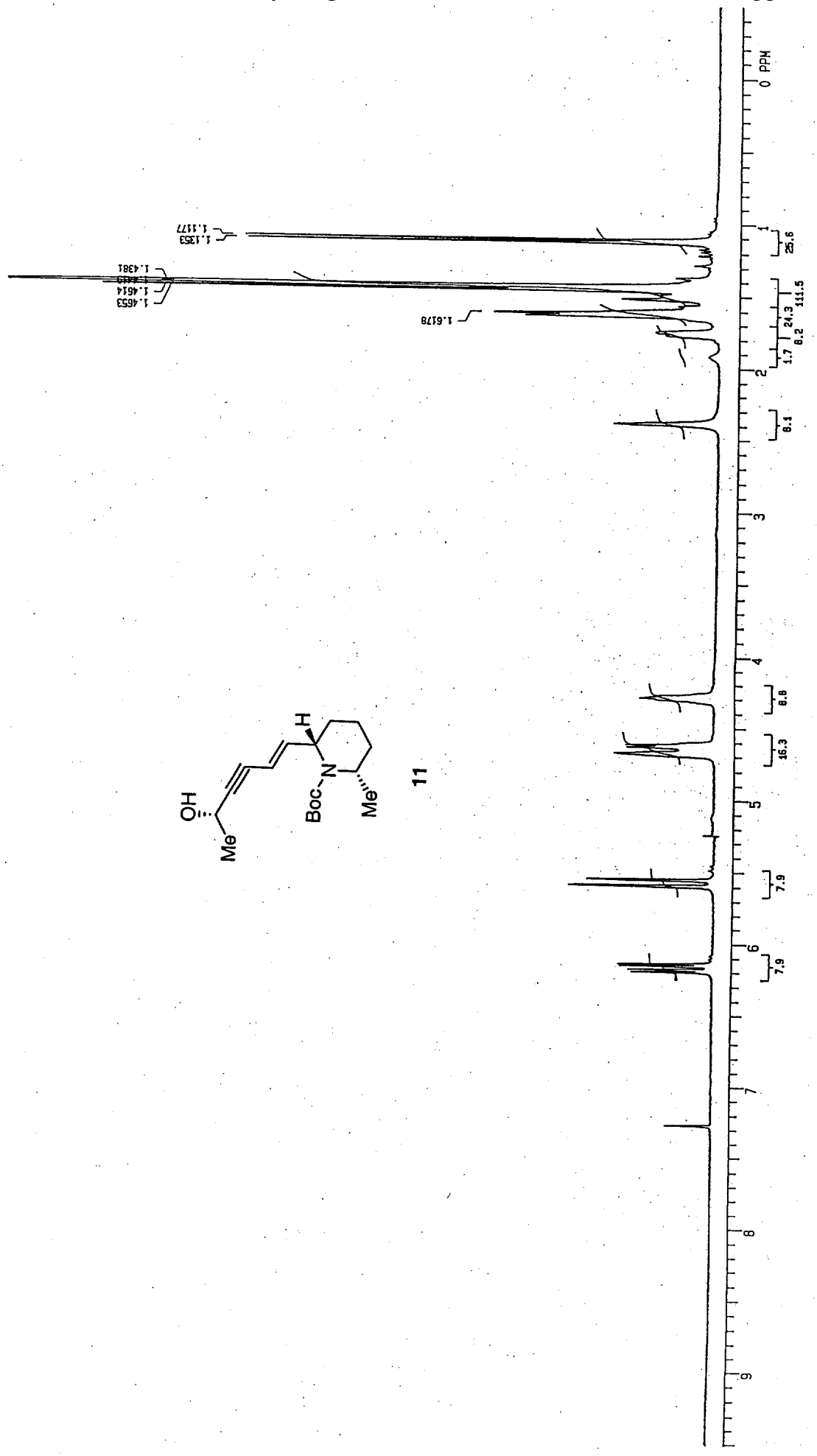

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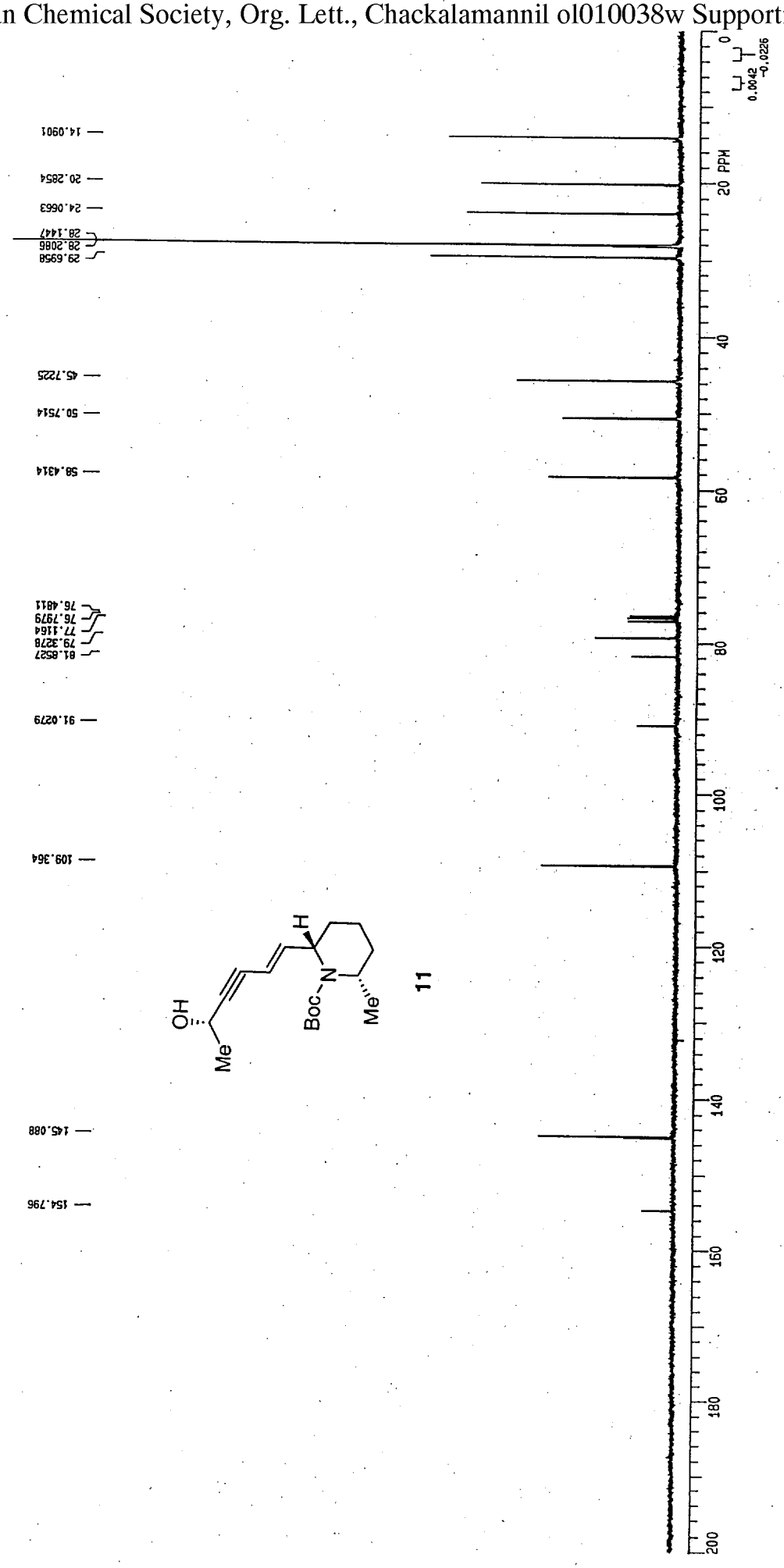

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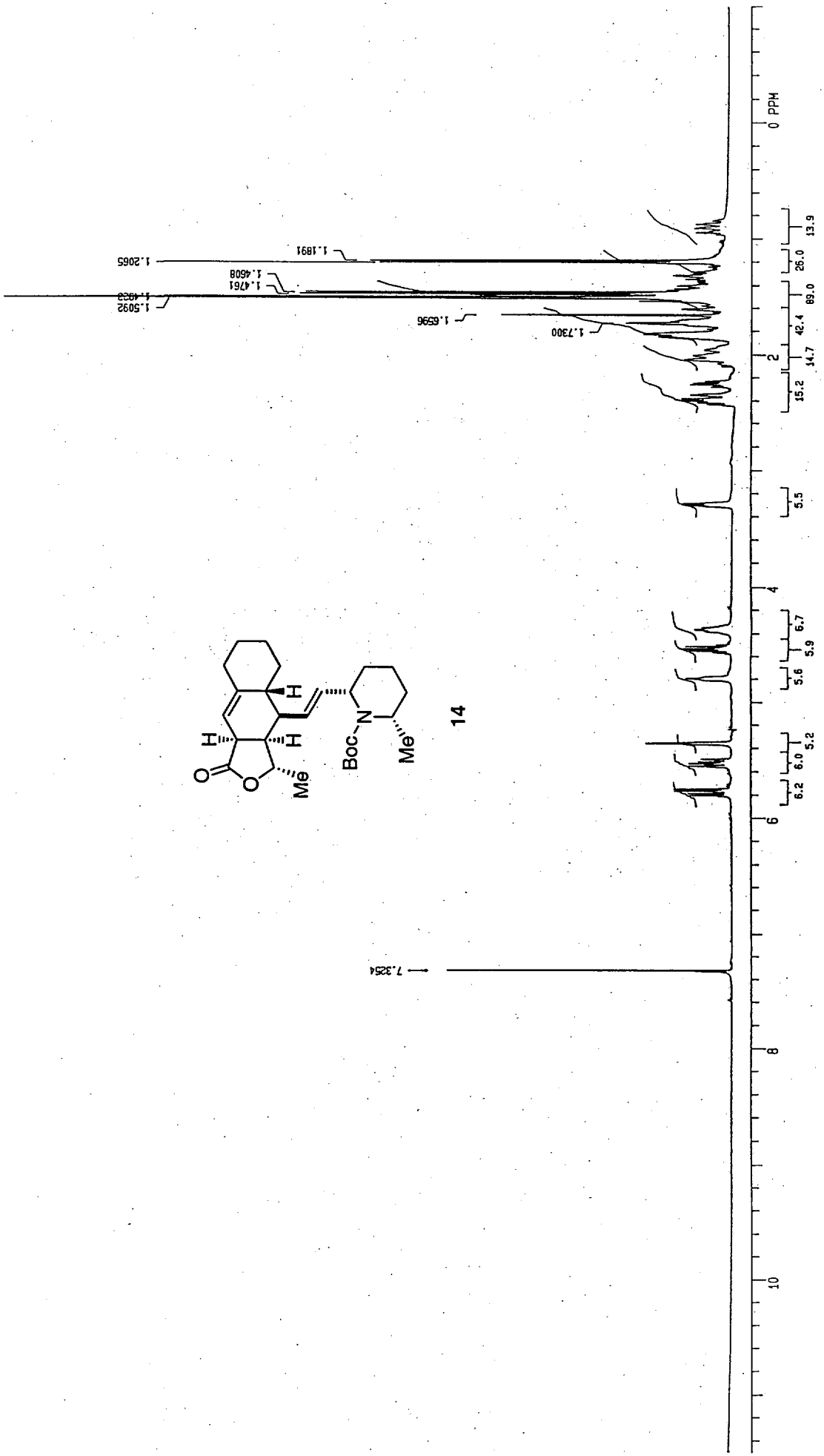
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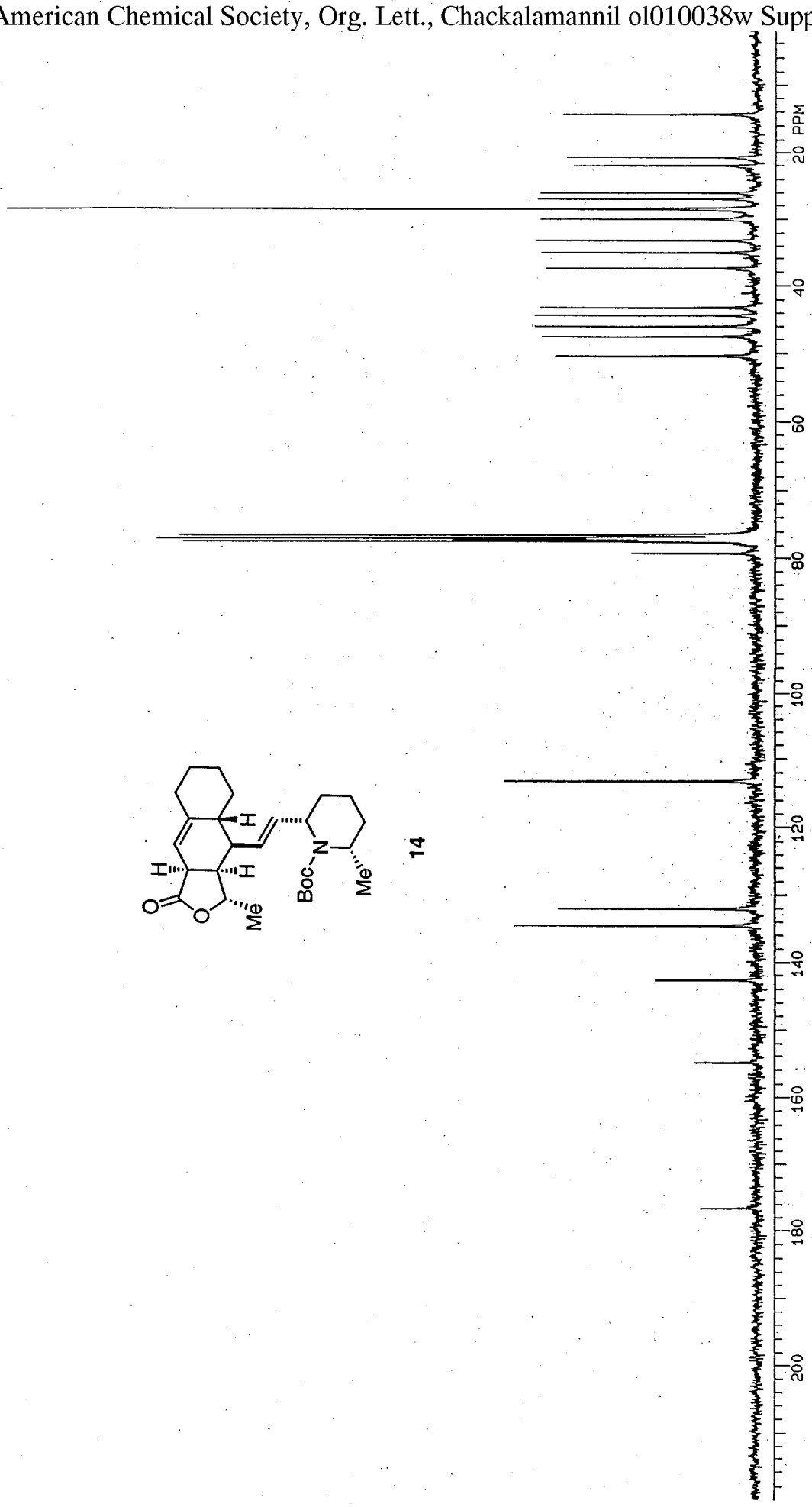

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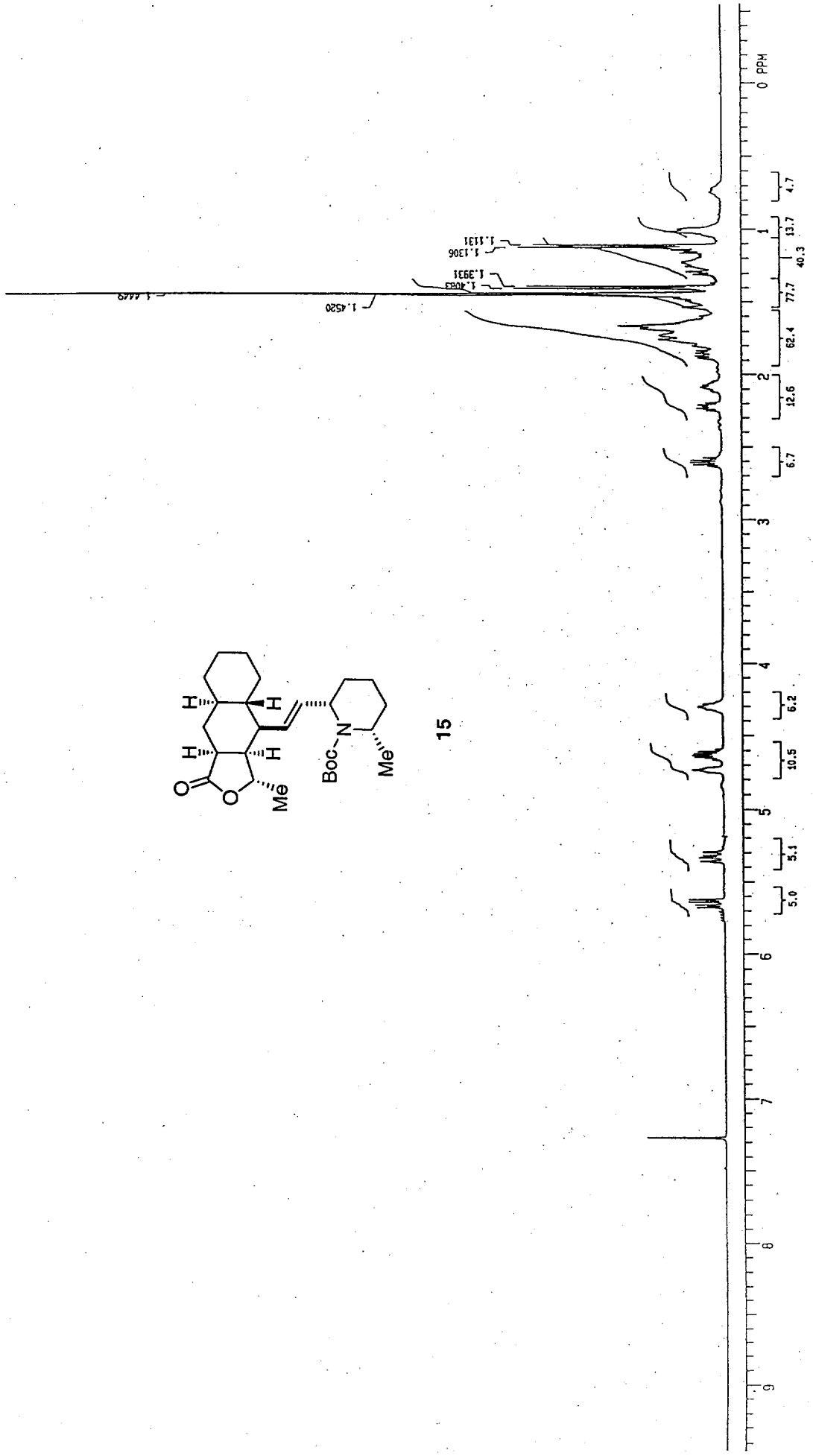
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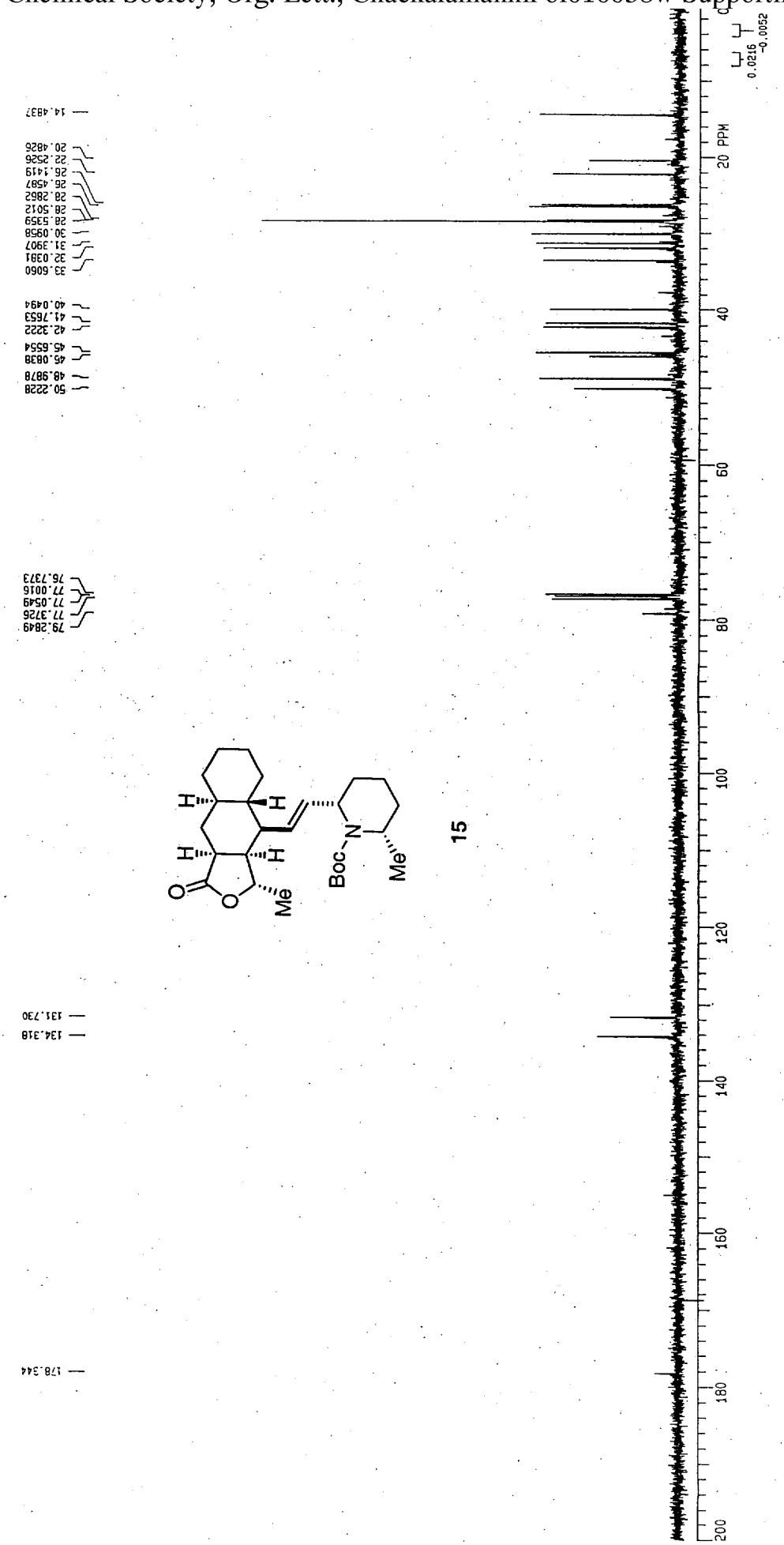


X-Ray Crystal Structure Analysis of Himandravine Hydrochloride (1a)

Figure 1. ORTEP diagram (50% probability ellipsoids) showing the crystallographic atom numbering scheme and solid-state conformation; the chloride ions lie on crystallographic C_2 symmetry axes. Small filled circles represent hydrogen atoms.

Table 1. Crystallographic Data

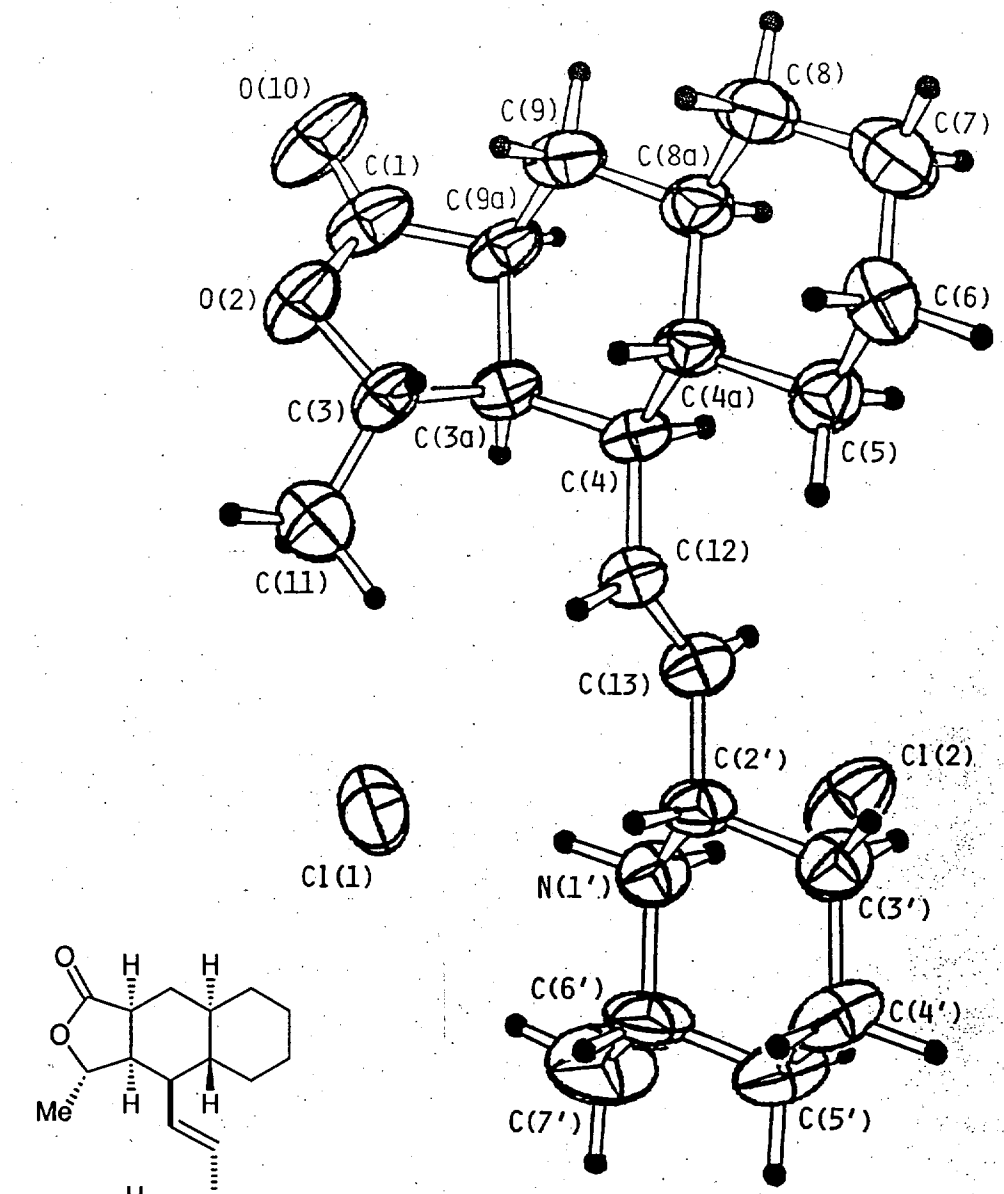
Table 2. Non-hydrogen Atom Fractional Coordinates and Equivalent Isotropic Thermal Parameters, with Estimated Standard Deviations

Table 3. Anisotropic Temperature Factor Parameters, with Estimated Standard Deviations

Table 4. Hydrogen Atom Fractional Coordinates and Isotropic Thermal Parameters

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

Table 6. Relative Intensities for Friedel Pairs of Enantiomer-sensitive Reflections with Intensity Ratios >1.33



1a (+)-Himandravine•HCl
(Absolute stereochemistry)

Figure 1. ORTEP diagram (50% probability ellipsoids) showing the crystallographic atom numbering scheme and solid-state conformation; the chloride ions lie on crystallographic C_2 symmetry axes. Small filled circles represent hydrogen atoms.

Table 1. Crystallographic Data[a]

Molecular formula	$[C_{21}H_{34}NO_2]Cl$
Formula weight	367.96
Crystal system	monoclinic
Space group	$C2(C_2^3)$ - No.5
a(Å)	27.556(6)
b(Å)	7.125(2)
c(Å)	10.989(2)
b(°)	94.50(1)
V(Å^3)	2151(2)
Z	4
$D_{calcd.}$ (g cm^{-3})	1.136
Radiation (λ, Å)	Cu-$K\alpha$ (1.5418)
Absorption coefficient, μ(cm^{-1})	16.7
Temp. (°C)	25
Crystal dimensions (mm)	0.03 x 0.09 x 0.50
$T_{max.}$:$T_{min.}$	1.00:0.89
Scan type	ω-2θ
Scanwidth (°)	0.90 + 0.14tanθ
$\theta_{max.}$(°)	75
Intensity control refls.;	3 -1 -3, 5 -3 2, 2 -2 3, 4 -2 1;
variation; repeat time (hr)	<1.0% ; 2
Total no. of non-equiv. refls. ($+h,-k,\pm l$) recorded	2394
No. of refls. retained [$I > 3.0\sigma(I)$]	1248
No. of parameters refined	227
Extinction correction	5.2(6) x 10^{-6}
R (R_w)[b]	0.048 (0.061)
Goodness-of-fit[c]	1.30
Max. shift:esd in final least-squares cycle	0.03
Final Δρ(e/Å^3) max.;min.	0.23; -0.20

[a]Preliminary unit-cell parameters and space group information were obtained from oscillation and Weissenberg photographs. An Enraf-Nonius CAD-4 diffractometer (Cu-$K\alpha$ radiation, graphite monochromator) was used for all other measurements. Intensity data were corrected for the usual Lorentz and polarization effects. The space group was deter-

Table 1 (continnued)

mined from the systematic absences (*hkl* when $h + k \neq 2n$) and the fact that the cation is chiral. Refined unit-cell parameters were derived from the diffractometer setting angles for 25 reflections ($32° < \theta < 40°$) widely separated in reciprocal space.

The crystal structure was solved by direct methods (MULTAN11/82). Initial coordinates for the the non-hydrogen atoms were obtained from an *E*-map. Positional and thermal parameters of these atoms (first isotropic and then anisotropic) were adjusted by means of several rounds of full-matrix least-squares calculations. Hydrogen atoms were incorporated at their calculated positions in the later iterations during which an extinction correction was included as a variable. The parameter refinement converged at $R = 0.0484$ ($R_w = 0.0607$). The absolute stereochemistry was established at this stage by introduction of the imaginary contributions to the anomalous dispersion corrections into the structure-factor calculations. For parameters corresponding to the absolute stereochemistry shown, R was 0.0501 while R_w was 0.0631; for parameters of the mirror image the values were 0.0515 and 0.0651, respectively. The differences indicate that the structure shown correctly represents the absolute stereochemistry. This determination is significant at the 0.005 level if R'_w/R_w (= 0.0651/0.0631 = 1.032) equals to or exceeds 1.004 (see: W. C. Hamilton, *Acta Cryst.*, **1965**, *18*, 502). Support for this assignment of absolute stereo-chemistry was obtained by measuring the relative intensities of 23 Friedel pairs of enantiomer-sensitive reflections for which the calculated intensity ratios were >1.33. In all cases, the sense of the difference between the measured values was in accord with that calculated (see Table 6). Continuation of the least-squares refine-ment of parameters for the correct enantiomer led to convergence at $R = 0.048$ ($R_w = 0.061$). No unusual features were present in a final difference Fourier synthesis.

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 correc-tions were taken from *International Tables for X-Ray Crystallography*, vol. IV, The Kynoch Press, Birmingham, U.K., 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 = $[\Sigma w\Delta^2/(N_{observations} - N_{parameters})]^{1/2}$.

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)
Cl(1)	0.0000(-)[a]	0.0000(-)[b]	0.0000(-)[a]	6.28(6)
Cl(2)	0.0000(-)[a]	0.0645(4)	0.5000(-)[a]	6.60(5)
C(1)	0.1883(2)	-0.5205(10)	0.0447(5)	5.2(1)
O(2)	0.1756(2)	-0.3981(6)	-0.0438(3)	5.1(1)
C(3)	0.1683(2)	-0.2115(9)	0.0093(5)	4.0(1)
C(3a)	0.1586(2)	-0.2548(9)	0.1428(5)	3.6(1)
C(4)	0.1680(2)	-0.0985(8)	0.2393(4)	3.4(1)
C(4a)	0.2224(2)	-0.0483(8)	0.2583(4)	3.3(1)
C(5)	0.2326(2)	0.1083(9)	0.3510(5)	4.1(1)
C(6)	0.2870(2)	0.1525(10)	0.3687(5)	4.7(1)
C(7)	0.3156(2)	-0.0221(13)	0.4115(5)	6.0(2)
C(8)	0.3057(2)	-0.1820(11)	0.3218(5)	4.9(1)
C(8a)	0.2519(2)	-0.2263(9)	0.2969(5)	4.0(1)
C(9)	0.2447(2)	-0.3819(9)	0.2009(5)	4.4(1)
C(9a)	0.1912(2)	-0.4281(9)	0.1668(4)	4.1(1)
O(10)	0.1960(2)	-0.6845(7)	0.0203(4)	7.3(1)
C(11)	0.1287(3)	-0.1200(12)	-0.0697(6)	6.5(2)
C(12)	0.1355(2)	0.0679(9)	0.2114(4)	3.4(1)
C(13)	0.1001(2)	0.1211(8)	0.2783(4)	3.6(1)
N(1')	0.0165(2)	0.2355(7)	0.2398(4)	3.9(1)
C(2')	0.0695(2)	0.2926(9)	0.2513(5)	3.5(1)
C(3')	0.0760(2)	0.4438(9)	0.3492(5)	4.2(1)
C(4')	0.0424(2)	0.6122(10)	0.3247(6)	5.2(1)
C(5')	-0.0097(2)	0.5471(10)	0.3084(6)	5.4(1)
C(6')	-0.0176(2)	0.3984(10)	0.2115(6)	5.3(1)
C(7')	-0.0697(2)	0.3195(14)	0.2004(8)	8.1(2)

[a]Fixed by symmetry; occupancy factor = 0.50.

[b]The y-coordinate of Cl(1) was held constant throughout the least-squares parameter refinement to define the space group origin in this direction.

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}
Cl(1)	0.067(1)	0.113(2)	0.058(1)	0.000(-)[b]	0.001(1)	0.000(-)[b]
Cl(2)	0.112(1)	0.056(1)	0.089(1)	0.000(-)[b]	0.053(1)	0.000(-)[b]
C(1)	0.079(3)	0.041(3)	0.082(3)	0.009(3)	0.041(2)	0.007(3)
O(2)	0.086(2)	0.045(2)	0.067(2)	0.000(2)	0.024(2)	-0.013(2)
C(3)	0.063(3)	0.031(3)	0.058(3)	0.003(3)	0.013(2)	-0.009(3)
C(3a)	0.040(2)	0.041(3)	0.056(3)	0.003(3)	0.013(2)	0.000(3)
C(4)	0.042(2)	0.034(3)	0.052(3)	0.009(2)	0.010(2)	0.002(3)
C(4a)	0.045(2)	0.038(3)	0.041(2)	0.010(2)	0.007(2)	0.008(2)
C(5)	0.062(3)	0.043(3)	0.051(3)	0.003(3)	0.008(2)	0.001(3)
C(6)	0.057(3)	0.067(5)	0.054(3)	-0.007(3)	-0.001(3)	0.003(3)
C(7)	0.058(3)	0.104(6)	0.064(3)	0.011(4)	-0.002(3)	0.014(4)
C(8)	0.051(3)	0.070(4)	0.063(3)	0.016(3)	0.001(3)	0.009(4)
C(8a)	0.054(3)	0.053(3)	0.045(2)	0.014(3)	0.008(2)	0.009(3)
C(9)	0.056(3)	0.052(4)	0.061(3)	0.019(3)	0.018(2)	0.012(3)
C(9a)	0.066(3)	0.032(3)	0.059(2)	0.010(3)	0.026(2)	0.005(3)
O(10)	0.143(4)	0.037(3)	0.108(3)	0.004(3)	0.066(2)	-0.009(2)
C(11)	0.097(5)	0.078(5)	0.067(4)	0.020(4)	-0.014(3)	-0.005(4)
C(12)	0.042(2)	0.038(3)	0.049(2)	0.001(3)	0.008(2)	0.001(3)
C(13)	0.045(3)	0.040(3)	0.054(2)	0.001(3)	0.008(2)	0.000(3)
N(1')	0.038(2)	0.047(3)	0.065(2)	-0.003(2)	0.004(2)	-0.004(2)
C(2')	0.032(2)	0.043(3)	0.057(3)	0.004(2)	0.003(2)	-0.001(3)
C(3')	0.044(3)	0.052(4)	0.064(3)	-0.002(3)	0.008(2)	-0.007(3)
C(4')	0.058(3)	0.042(3)	0.102(4)	0.004(3)	0.020(3)	-0.016(3)
C(5')	0.054(3)	0.050(4)	0.101(4)	0.011(3)	0.017(3)	-0.005(4)
C(6')	0.039(3)	0.064(4)	0.094(4)	0.023(3)	-0.009(3)	0.003(4)
C(7')	0.037(3)	0.101(7)	0.168(7)	0.001(4)	-0.014(4)	-0.006(7)

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

[b] Fixed by symmetry.

Table 4. Hydrogen Atom Fractional Coordinates[a] and Isotropic Thermal Parameters

Atom	*x*	*y*	*z*	*B*(Å2)
H(3)	0.1971	-0.1149	0.0119	4.0
H(3a)	0.1213	-0.2731	0.1522	3.6
H(4)	0.1586	-0.1504	0.3237	3.4
H(4a)	0.2333	0.0024	0.1749	3.3
H(5A)	0.2204	0.0666	0.4350	4.1
H(5B)	0.2138	0.2295	0.3200	4.1
H(6A)	0.2927	0.2588	0.4344	4.7
H(6B)	0.2991	0.1983	0.2855	4.7
H(7A)	0.3051	-0.0627	0.4974	6.0
H(7B)	0.3530	0.0090	0.4182	6.0
H(8A)	0.3235	-0.3026	0.3575	4.9
H(8B)	0.3198	-0.1455	0.2390	4.9
H(8a)	0.2386	-0.2749	0.3781	4.0
H(9A)	0.2622	-0.5038	0.2356	4.4
H(9B)	0.2606	-0.3386	0.1218	4.4
H(9a)	0.1797	-0.5069	0.2400	4.1
H(11A)	0.1404	-0.0992	-0.1574	6.5
H(11B)	0.0977	-0.2063	-0.0754	6.5
H(11C)	0.1201	0.0101	-0.0319	6.5
H(12)	0.1410	0.1436	0.1371	3.4
H(13)	0.0934	0.0430	0.3507	3.6
H(1'A)	0.0082	0.1763	0.3201	3.9
H(1'B)	0.0114	0.1391	0.1713	3.9
H(2')	0.0808	0.3499	0.1702	3.5
H(3'A)	0.1122	0.4910	0.3536	4.2
H(3'B)	0.0688	0.3845	0.4334	4.2
H(4'A)	0.0515	0.6809	0.2451	5.2
H(4'B)	0.0466	0.7053	0.3988	5.2
H(5'A)	-0.0319	0.6628	0.2837	5.4
H(5'B)	-0.0195	0.4921	0.3916	5.4
H(6')	-0.0110	0.4642	0.1289	5.3
H(7'A)	-0.0728	0.2170	0.1316	8.1
H(7'B)	-0.0775	0.2591	0.2838	8.1

Table 4 (continued)

Atom	*x*	*y*	*z*	*B*(Å2)
H(7'C)	-0.0945	0.4286	0.1782	8.1

[a]Hydrogen atoms bear the same labels as the atoms to which they are bonded and were assigned the equivalent isotropic thermal parameters of these atoms.

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

(a) Bond Lengths

C(1)-O(2)	1.332(7)	C(7)-C(8)	1.517(10)
C(1)-C(9a)	1.491(8)	C(8)-C(8a)	1.519(8)
C(1)-O(10)	1.221(9)	C(8a)-C(9)	1.532(8)
O(2)-C(3)	1.472(8)	C(9)-C(9a)	1.528(8)
C(3)-C(3a)	1.543(8)	C(12)-C(13)	1.322(8)
C(3)-C(11)	1.490(9)	C(13)-C(2')	1.501(8)
C(3a)-C(4)	1.546(8)	N(1')-C(2')	1.512(8)
C(3a)-C(9a)	1.538(9)	N(1')-C(6')	1.510(8)
C(4)-C(4a)	1.539(8)	C(2')-C(3')	1.523(8)
C(4)-C(12)	1.503(8)	C(3')-C(4')	1.527(9)
C(4a)-C(5)	1.522(8)	C(4')-C(5')	1.506(8)
C(4a)-C(8a)	1.548(8)	C(5')-C(6')	1.506(10)
C(5)-C(6)	1.529(8)	C(6')-C(7')	1.538(9)
C(6)-C(7)	1.527(11)		

(b) Bond Angles

O(2)-C(1)-C(9a)	111.1(6)	C(4a)-C(8a)-C(8)	111.3(5)
O(2)-C(1)-O(10)	120.4(5)	C(4a)-C(8a)-C(9)	111.6(4)
C(9a)-C(1)-O(10)	128.5(6)	C(8)-C(8a)-C(9)	110.2(5)
C(1)-O(2)-C(3)	109.8(4)	C(8a)-C(9)-C(9a)	113.3(5)
O(2)-C(3)-C(3a)	103.6(5)	C(1)-C(9a)-C(3a)	102.2(4)
O(2)-C(3)-C(11)	106.3(5)	C(1)-C(9a)-C(9)	107.4(4)
C(3a)-C(3)-C(11)	117.9(5)	C(3a)-C(9a)-C(9)	114.1(5)
C(3)-C(3a)-C(4)	118.4(5)	C(4)-C(12)-C(13)	124.5(5)
C(3)-C(3a)-C(9a)	100.4(4)	C(12)-C(13)-C(2')	123.4(5)
C(4)-C(3a)-C(9a)	113.4(4)	C(2')-N(1')-C(6')	113.1(5)
C(3a)-C(4)-C(4a)	111.7(4)	C(13)-C(2')-N(1')	108.8(5)
C(3a)-C(4)-C(12)	111.3(4)	C(13)-C(2')-C(3')	113.8(4)
C(4a)-C(4)-C(12)	113.8(5)	N(1')-C(2')-C(3')	108.0(5)
C(4)-C(4a)-C(5)	112.8(4)	C(2')-C(3')-C(4')	113.3(5)
C(4)-C(4a)-C(8a)	109.4(5)	C(3')-C(4')-C(5')	109.8(5)
C(5)-C(4a)-C(8a)	110.6(4)	C(4')-C(5')-C(6')	112.6(5)

Table 5 (continued)

C(4a)-C(5)-C(6)	111.3(5)	N(1')-C(6')-C(5')	110.0(5)
C(5)-C(6)-C(7)	110.4(5)	N(1')-C(6')-C(7')	107.3(6)
C(6)-C(7)-C(8)	110.5(5)	C(5')-C(6')-C(7')	113.3(6)
C(7)-C(8)-C(8a)	113.3(5)		

(c) Torsion Angles[a]

C(9a)-C(1)-O(2)-C(3)	-0.4(7)	C(8a)-C(4a)-C(5)-C(6)	56.4(6)
O(10)-C(1)-O(2)-C(3)	179.8(6)	C(4)-C(4a)-C(8a)-C(8)	-177.7(4)
O(2)-C(1)-C(9a)-C(3a)	22.4(6)	C(4)-C(4a)-C(8a)-C(9)	58.7(5)
O(2)-C(1)-C(9a)-C(9)	-98.0(6)	C(5)-C(4a)-C(8a)-C(8)	-52.8(6)
O(10)-C(1)-C(9a)-C(3a)	-157.9(6)	C(5)-C(4a)-C(8a)-C(9)	-176.4(5)
O(10)-C(1)-C(9a)-C(9)	81.8(8)	C(4a)-C(5)-C(6)-C(7)	-58.7(6)
C(1)-O(2)-C(3)-C(3a)	-21.8(6)	C(5)-C(6)-C(7)-C(8)	56.6(6)
C(1)-O(2)-C(3)-C(11)	-146.7(5)	C(6)-C(7)-C(8)-C(8a)	-54.7(7)
O(2)-C(3)-C(3a)-C(4)	157.5(5)	C(7)-C(8)-C(8a)-C(4a)	52.8(7)
O(2)-C(3)-C(3a)-C(9a)	33.6(5)	C(7)-C(8)-C(8a)-C(9)	177.1(5)
C(11)-C(3)-C(3a)-C(4)	-85.4(7)	C(4a)-C(8a)-C(9)-C(9a)	-53.2(6)
C(11)-C(3)-C(3a)-C(9a)	150.7(6)	C(8)-C(8a)-C(9)-C(9a)	-177.4(5)
C(3)-C(3a)-C(4)-C(4a)	-66.0(6)	C(8a)-C(9)-C(9a)-C(1)	158.5(5)
C(3)-C(3a)-C(4)-C(12)	62.4(6)	C(8a)-C(9)-C(9a)-C(3a)	46.1(6)
C(9a)-C(3a)-C(4)-C(4a)	51.3(6)	C(4)-C(12)-C(13)-C(2')	177.4(5)
C(9a)-C(3a)-C(4)-C(12)	179.7(4)	C(12)-C(13)-C(2')-N(1')	124.7(6)
C(3)-C(3a)-C(9a)-C(1)	-33.2(5)	C(12)-C(13)-C(2')-C(3')	-115.0(6)
C(3)-C(3a)-C(9a)-C(9)	82.3(5)	C(6')-N(1')-C(2')-C(13)	-179.5(4)
C(4)-C(3a)-C(9a)-C(1)	-160.6(5)	C(6')-N(1')-C(2')-C(3')	56.6(6)
C(4)-C(3a)-C(9a)-C(9)	-45.0(6)	C(2')-N(1')-C(6')-C(5')	-57.3(6)
C(3a)-C(4)-C(4a)-C(5)	178.8(4)	C(2')-N(1')-C(6')-C(7')	179.1(5)
C(3a)-C(4)-C(4a)-C(8a)	-57.7(5)	C(13)-C(2')-C(3')-C(4')	-176.4(5)
C(12)-C(4)-C(4a)-C(5)	51.8(5)	N(1')-C(2')-C(3')-C(4')	-55.6(6)
C(12)-C(4)-C(4a)-C(8a)	175.3(4)	C(2')-C(3')-C(4')-C(5')	55.2(7)
C(3a)-C(4)-C(12)-C(13)	112.3(6)	C(3')-C(4')-C(5')-C(6')	-54.4(7)
C(4a)-C(4)-C(12)-C(13)	-120.5(6)	C(4')-C(5')-C(6')-N(1')	55.5(7)
C(4)-C(4a)-C(5)-C(6)	179.3(5)	C(4')-C(5')-C(6')-C(7')	175.6(6)

Table 5 (continued)

(d) Hydrogen-bonded Distances (Donor...Acceptor)

N(1')...Cl(1)	3.127(5)	N(1')...Cl(2)	3.174(5)

[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.

Table 6. Relative Intensities for Friedel Pairs of Enantiomer-sensitive Reflections with Intensity Ratios >1.33

hkl	$I(hkl)$ vs. $I(\bar{h}\bar{k}\bar{l})$	*hkl*	$I(hkl)$ vs. $I(\bar{h}\bar{k}\bar{l})$
4 -2 -1	>	12 -2 -6	>
8 -2 2	>	4 -4 -5	>
6 -2 3	>	3 -1 8	<
1 -1 -6	<	3 -5 3	>
14 -2 -2	<	9 -5 -1	<
3 -3 -5	<	21 -1 -2	<
12 -2 4	<	6 -2 -8	>
10 -4 -1	<	2 -6 -1	>
9 -1 6	<	15 -3 -6	>
13 -1 5	>	15 -1 8	<
17 -1 -4	>	9 -3 -10	>
15 -3 2	<		