Total Synthesis of (–)-7-Epicylindrospermopsin, a Toxic Metabolite of the Freshwater Cyanobacterium *Aphanizomenon ovalisporum*, and Assignment of Its Absolute Configuration.

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SUPPORTING INFORMATION

1. X-Ray Crystallographic Data for **30**.
2. X-Ray Crystallographic Data for **51**.
3. ^{1}H and ^{13}C NMR Spectra for Synthetic Intermediates.

General Methods. All moisture-sensitive reactions were performed in flame-dried glassware under a dry argon atmosphere. Tetrahydrofuran (THF), diethyl ether (Et_2O), and toluene (PhMe) were distilled from sodium-benzophenone ketyl. Dichloromethane (CH_2Cl_2), triethylamine (Et_3N), dimethyl sulfoxide (DMSO) and *N*,*N*-dimethylformamide (DMF) were distilled from calcium hydride. All other commercial reagents were purchased and used as received unless otherwise specified. Melting points were recorded using open capillary tubes and are uncorrected. Specific optical rotations were measured at ambient temperature (24 °C) from $CHCl_3$ solutions (unless otherwise noted) using a 1 mL cell with 1 dm path length. Infrared spectra (IR) are reported in wavenumbers (cm^{-1}). High resolution mass spectra were obtained using chemical ionization (CI) or fast atom bombardment (FAB). ^1H and ^{13}C NMR spectra were recorded in Fourier transform mode at the field strength specified either on a 300 MHz or 400 MHz spectrometer. Analytical thin layer chromatography (TLC) was performed using TLC plates pre-coated with silica gel 60 F-254 (0.25 mm layer thickness). TLC visualization was accomplished using either a UV lamp, potassium permangenate solution, or phosphomolybdic acid (PMA) solution. Flash chromatography was performed on 320-400-mesh silica gel.

X-Ray Crystallographic Date for **30** (JH060401)

Experimental details for jh060401.

The crystalline sample used was that supplied by Mr. Joshua Hansen. Determination of the crystallographic parameters, data collection and structure solution and refinement was done as described elsewhere,[1] with the following details.

From a mass of crystals, a well shaped crystal of dimensions 0.30 x 0.20 x 0.20 mm^3 was selected and mounted on the tip of a thin glass fiber using epoxy glue. An automated routine was used to find and center 12 reflections with $7° < 2\theta < 25°$, with which the crystal was indexed. The reflection list was then expanded to include 96 reflections with $8.713° < 2\theta < 61.630°$, and the lattice parameters refined against this list. All unique data, including a small set of redundant reflections were collected (-10 – 12, -11 – 13, -25 – 25). Monitoring of three standards during data collection showed no intensity decay. Correction for the effects of absorption anisotropy was carried out by means of psi-scans[2] using the program XEMP v4.3.[3]

The structure was solved using direct methods as programmed in SHELXS-90,[4] which revealed the positions of all atoms in the molecule. The solution was refined using the program SHELXL-97,[5] followed by Fourier synthesis, which revealed the positions of the remaining atoms. Though all hydrogen atoms could be clearly identified from the Fourier map, in order to preserve a favorable data-to-parameter ratio the hydrogen atoms were placed in geometrically idealized positions. The hydrogen atoms were given an isotropic displacement parameter equal to 1.5 (methyl group) or 1.2 (all other hydrogens) times the equivalent isotropic displacement parameter of the atom it is bonded to. During the final cycle of least squares refinement, all non-hydrogen atoms were refined with anisotropic displacement parameters.

An ORTEP[6] of the final model is given in Figure 1, with displacement ellipsoids drawn at the 30% probability level.

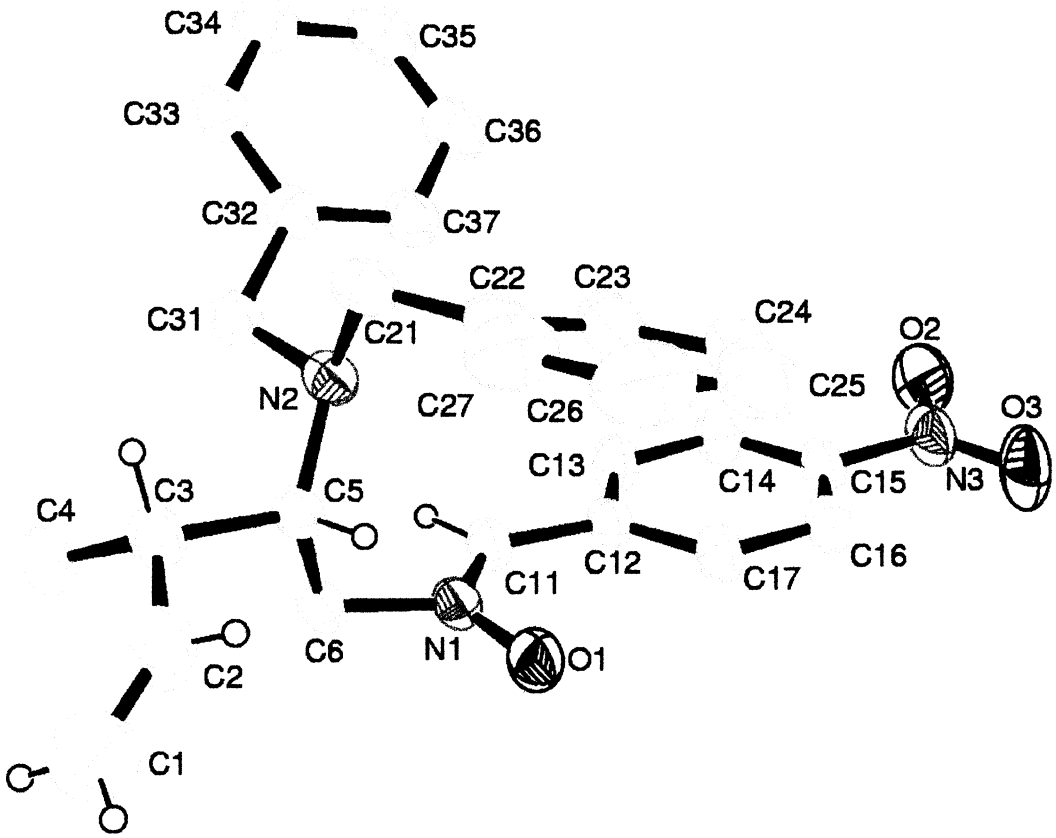


Figure 1: ORTEP with 30% displacement ellipsoids.

Table 1. Crystal data and structure refinement for jh060401.

Identification code	jh060401	
Empirical formula	$C_{27}H_{29}N_3O_3$	
Formula weight	443.53	
Temperature	290(2) K	
Wavelength	1.54184 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 10.277(4) Å	α= 90°.
	b = 11.329(2) Å	β= 90°.
	c = 21.0730(10) Å	γ = 90°.
Volume	2453.5(11) $Å^3$	
Z	4	
Density (calculated)	1.201 Mg/m^3	
Absorption coefficient	0.632 mm^{-1}	
F(000)	944	
Crystal size	0.30 x 0.20 x 0.20 mm^3	
Theta range for data collection	4.20 to 67.43°.	
Index ranges	-10<=h<=12, -11<=k<=13, -25<=l<=25	
Reflections collected	4234	
Independent reflections	3704 [R(int) = 0.0381]	
Completeness to theta = 67.43°	93.7 %	
Absorption correction	Empirical	
Max. and min. transmission	0.8840 and 0.8330	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3704 / 0 / 299	
Goodness-of-fit on F^2	1.054	
Final R indices [I>2sigma(I)]	R1 = 0.0433, wR2 = 0.1159	
R indices (all data)	R1 = 0.0456, wR2 = 0.1194	
Absolute structure parameter	0.0(3)	
Largest diff. peak and hole	0.208 and -0.195 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for jh060401. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2818(4)	-2051(4)	1622(3)	154(2)
C(2)	2279(3)	-1122(3)	1760(2)	106(1)
C(3)	997(2)	-599(2)	1589(1)	67(1)
C(4)	197(3)	-1376(2)	1158(1)	79(1)
C(5)	1180(2)	706(2)	1361(1)	56(1)
C(6)	1463(2)	775(2)	647(1)	61(1)
C(11)	1230(2)	2717(2)	201(1)	57(1)
C(12)	1519(2)	3947(2)	83(1)	56(1)
C(13)	576(2)	4575(2)	-248(1)	65(1)
C(14)	722(2)	5770(2)	-370(1)	69(1)
C(15)	1844(2)	6314(2)	-161(1)	64(1)
C(16)	2801(2)	5724(2)	165(1)	67(1)
C(17)	2646(2)	4528(2)	290(1)	63(1)
C(21)	235(2)	1898(2)	2205(1)	68(1)
C(22)	1417(2)	2610(2)	2354(1)	73(1)
C(23)	1641(3)	3675(2)	2061(1)	91(1)
C(24)	2724(4)	4358(3)	2222(2)	116(1)
C(25)	3570(4)	3976(5)	2666(2)	134(2)
C(26)	3369(4)	2914(5)	2957(2)	134(2)
C(27)	2307(3)	2235(4)	2808(1)	102(1)
C(31)	-1163(2)	1232(2)	1358(1)	63(1)
C(32)	-2082(2)	2279(2)	1338(1)	59(1)
C(33)	-3376(2)	2146(2)	1505(1)	69(1)
C(34)	-4242(2)	3061(3)	1458(1)	83(1)
C(35)	-3826(3)	4149(3)	1239(1)	86(1)
C(36)	-2548(3)	4300(2)	1075(1)	80(1)
C(37)	-1687(2)	3370(2)	1120(1)	69(1)
N(1)	1973(2)	1956(1)	492(1)	58(1)
N(2)	160(2)	1556(1)	1533(1)	57(1)
N(3)	1999(3)	7576(2)	-287(1)	79(1)
O(1)	3132(2)	2162(1)	689(1)	73(1)

O(2)	1096(2)	8113(2)	-530(1)	102(1)
O(3)	3024(2)	8047(2)	-141(1)	115(1)

Table 3. Bond lengths [Å] and angles [°] for jh060401.

C(1)-C(2)	1.224(5)
C(2)-C(3)	1.489(4)
C(3)-C(4)	1.508(3)
C(3)-C(5)	1.566(3)
C(5)-N(2)	1.468(2)
C(5)-C(6)	1.534(3)
C(6)-N(1)	1.474(3)
C(11)-N(1)	1.303(3)
C(11)-C(12)	1.447(3)
C(12)-C(13)	1.389(3)
C(12)-C(17)	1.402(3)
C(13)-C(14)	1.386(3)
C(14)-C(15)	1.380(3)
C(15)-C(16)	1.373(3)
C(15)-N(3)	1.462(3)
C(16)-C(17)	1.389(3)
C(21)-N(2)	1.470(2)
C(21)-C(22)	1.492(3)
C(22)-C(23)	1.376(4)
C(22)-C(27)	1.390(4)
C(23)-C(24)	1.397(5)
C(24)-C(25)	1.349(6)
C(25)-C(26)	1.366(7)
C(26)-C(27)	1.372(6)
C(31)-N(2)	1.455(3)
C(31)-C(32)	1.516(3)
C(32)-C(37)	1.381(3)
C(32)-C(33)	1.384(3)
C(33)-C(34)	1.369(3)
C(34)-C(35)	1.384(4)
C(35)-C(36)	1.368(4)
C(36)-C(37)	1.379(3)
N(1)-O(1)	1.284(2)
N(3)-O(3)	1.221(3)

N(3)-O(2)	1.222(3)
C(1)-C(2)-C(3)	133.2(4)
C(2)-C(3)-C(4)	113.3(2)
C(2)-C(3)-C(5)	110.14(19)
C(4)-C(3)-C(5)	115.59(18)
N(2)-C(5)-C(6)	110.14(15)
N(2)-C(5)-C(3)	117.25(16)
C(6)-C(5)-C(3)	111.80(16)
N(1)-C(6)-C(5)	109.35(15)
N(1)-C(11)-C(12)	126.7(2)
C(13)-C(12)-C(17)	119.47(18)
C(13)-C(12)-C(11)	115.90(19)
C(17)-C(12)-C(11)	124.61(19)
C(14)-C(13)-C(12)	121.2(2)
C(15)-C(14)-C(13)	117.9(2)
C(16)-C(15)-C(14)	122.70(19)
C(16)-C(15)-N(3)	119.3(2)
C(14)-C(15)-N(3)	118.0(2)
C(15)-C(16)-C(17)	119.2(2)
C(16)-C(17)-C(12)	119.6(2)
N(2)-C(21)-C(22)	112.88(17)
C(23)-C(22)-C(27)	117.9(3)
C(23)-C(22)-C(21)	121.1(2)
C(27)-C(22)-C(21)	121.0(3)
C(22)-C(23)-C(24)	120.6(3)
C(25)-C(24)-C(23)	120.3(4)
C(24)-C(25)-C(26)	119.7(4)
C(25)-C(26)-C(27)	120.8(4)
C(26)-C(27)-C(22)	120.6(4)
N(2)-C(31)-C(32)	113.06(17)
C(37)-C(32)-C(33)	117.7(2)
C(37)-C(32)-C(31)	121.71(19)
C(33)-C(32)-C(31)	120.49(19)
C(34)-C(33)-C(32)	121.6(2)
C(33)-C(34)-C(35)	119.8(2)

C(36)-C(35)-C(34)	119.5(2)
C(35)-C(36)-C(37)	120.2(3)
C(36)-C(37)-C(32)	121.2(2)
O(1)-N(1)-C(11)	125.12(17)
O(1)-N(1)-C(6)	115.03(17)
C(11)-N(1)-C(6)	119.77(18)
C(31)-N(2)-C(5)	116.02(15)
C(31)-N(2)-C(21)	111.09(16)
C(5)-N(2)-C(21)	111.91(16)
O(3)-N(3)-O(2)	122.9(2)
O(3)-N(3)-C(15)	118.4(2)
O(2)-N(3)-C(15)	118.7(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for jh060401. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	123(3)	115(3)	222(5)	66(3)	18(3)	42(3)
C(2)	97(2)	58(2)	164(3)	25(2)	-13(2)	7(2)
C(3)	81(1)	49(1)	73(1)	12(1)	6(1)	7(1)
C(4)	99(2)	43(1)	94(2)	8(1)	7(1)	1(1)
C(5)	66(1)	41(1)	61(1)	2(1)	5(1)	5(1)
C(6)	78(1)	39(1)	65(1)	-2(1)	13(1)	2(1)
C(11)	67(1)	46(1)	58(1)	0(1)	6(1)	-4(1)
C(12)	68(1)	43(1)	56(1)	-1(1)	10(1)	-5(1)
C(13)	70(1)	51(1)	74(1)	7(1)	-1(1)	-8(1)
C(14)	79(1)	51(1)	76(1)	10(1)	2(1)	2(1)
C(15)	83(1)	41(1)	68(1)	-1(1)	11(1)	-7(1)
C(16)	78(1)	49(1)	75(1)	-3(1)	-1(1)	-11(1)
C(17)	73(1)	46(1)	70(1)	4(1)	0(1)	-4(1)
C(21)	83(1)	68(1)	52(1)	-3(1)	5(1)	12(1)
C(22)	81(1)	79(2)	59(1)	-22(1)	2(1)	11(1)
C(23)	98(2)	74(2)	102(2)	-24(1)	-4(2)	5(2)
C(24)	113(2)	90(2)	145(3)	-50(2)	11(2)	-8(2)
C(25)	93(2)	173(4)	135(3)	-95(3)	5(2)	-13(3)
C(26)	99(2)	212(5)	91(2)	-44(3)	-18(2)	7(3)
C(27)	91(2)	150(3)	66(1)	-13(2)	-8(1)	11(2)
C(31)	72(1)	48(1)	69(1)	1(1)	4(1)	5(1)
C(32)	70(1)	50(1)	56(1)	-2(1)	0(1)	7(1)
C(33)	73(1)	62(1)	72(1)	1(1)	3(1)	4(1)
C(34)	73(1)	87(2)	89(2)	-9(1)	0(1)	17(2)
C(35)	96(2)	70(2)	90(2)	-6(1)	-11(1)	34(2)
C(36)	103(2)	55(1)	81(1)	6(1)	-6(1)	16(1)
C(37)	82(1)	53(1)	71(1)	5(1)	4(1)	4(1)
N(1)	69(1)	42(1)	62(1)	-3(1)	11(1)	-1(1)
N(2)	67(1)	49(1)	54(1)	-1(1)	2(1)	6(1)
N(3)	106(2)	45(1)	85(1)	5(1)	11(1)	-10(1)
O(1)	66(1)	53(1)	100(1)	5(1)	-2(1)	2(1)

O(2)	125(2)	53(1)	127(2)	25(1)	4(1)	2(1)
O(3)	130(2)	56(1)	159(2)	15(1)	-14(2)	-30(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for jh060401.

	x	y	z	U(eq)
H(1A)	2404	-2590	1357	184
H(1B)	3641	-2218	1782	184
H(2)	2773	-645	2027	128
H(3)	508	-549	1987	81
H(4A)	-617	-996	1067	118
H(4B)	37	-2118	1364	118
H(4C)	662	-1509	770	118
H(5)	1972	988	1571	67
H(6A)	2096	178	531	73
H(6B)	671	629	409	73
H(11)	433	2440	56	68
H(13)	-165	4186	-390	78
H(14)	83	6191	-585	82
H(16)	3543	6120	301	81
H(17)	3286	4115	509	76
H(21A)	237	1191	2464	81
H(21B)	-534	2352	2314	81
H(23)	1066	3944	1752	109
H(24)	2862	5079	2022	139
H(25)	4287	4435	2775	161
H(26)	3960	2648	3259	161
H(27)	2181	1517	3013	123
H(31A)	-1489	659	1660	76
H(31B)	-1149	860	943	76
H(33)	-3665	1419	1653	83
H(34)	-5107	2951	1572	99
H(35)	-4410	4772	1203	103
H(36)	-2259	5033	934	95
H(37)	-824	3481	1001	83

Table 6. Torsion angles [°] for jh060401.

C(1)-C(2)-C(3)-C(4)	0.2(5)
C(1)-C(2)-C(3)-C(5)	-131.0(4)
C(2)-C(3)-C(5)-N(2)	-144.0(2)
C(4)-C(3)-C(5)-N(2)	86.0(2)
C(2)-C(3)-C(5)-C(6)	87.5(3)
C(4)-C(3)-C(5)-C(6)	-42.5(3)
N(2)-C(5)-C(6)-N(1)	63.1(2)
C(3)-C(5)-C(6)-N(1)	-164.66(17)
N(1)-C(11)-C(12)-C(13)	178.84(19)
N(1)-C(11)-C(12)-C(17)	-2.7(3)
C(17)-C(12)-C(13)-C(14)	-0.9(3)
C(11)-C(12)-C(13)-C(14)	177.65(19)
C(12)-C(13)-C(14)-C(15)	1.0(3)
C(13)-C(14)-C(15)-C(16)	-0.8(3)
C(13)-C(14)-C(15)-N(3)	-179.8(2)
C(14)-C(15)-C(16)-C(17)	0.3(3)
N(3)-C(15)-C(16)-C(17)	179.37(19)
C(15)-C(16)-C(17)-C(12)	-0.2(3)
C(13)-C(12)-C(17)-C(16)	0.4(3)
C(11)-C(12)-C(17)-C(16)	-177.97(19)
N(2)-C(21)-C(22)-C(23)	60.1(3)
N(2)-C(21)-C(22)-C(27)	-121.8(2)
C(27)-C(22)-C(23)-C(24)	-0.7(4)
C(21)-C(22)-C(23)-C(24)	177.5(2)
C(22)-C(23)-C(24)-C(25)	0.4(5)
C(23)-C(24)-C(25)-C(26)	0.4(5)
C(24)-C(25)-C(26)-C(27)	-0.9(6)
C(25)-C(26)-C(27)-C(22)	0.5(5)
C(23)-C(22)-C(27)-C(26)	0.2(4)
C(21)-C(22)-C(27)-C(26)	-177.9(3)
N(2)-C(31)-C(32)-C(37)	37.4(3)
N(2)-C(31)-C(32)-C(33)	-146.55(19)
C(37)-C(32)-C(33)-C(34)	0.0(3)
C(31)-C(32)-C(33)-C(34)	-176.3(2)

C(32)-C(33)-C(34)-C(35)	-0.1(4)
C(33)-C(34)-C(35)-C(36)	-0.4(4)
C(34)-C(35)-C(36)-C(37)	0.9(4)
C(35)-C(36)-C(37)-C(32)	-1.1(4)
C(33)-C(32)-C(37)-C(36)	0.6(3)
C(31)-C(32)-C(37)-C(36)	176.8(2)
C(12)-C(11)-N(1)-O(1)	-4.5(3)
C(12)-C(11)-N(1)-C(6)	172.02(17)
C(5)-C(6)-N(1)-O(1)	71.4(2)
C(5)-C(6)-N(1)-C(11)	-105.4(2)
C(32)-C(31)-N(2)-C(5)	-160.35(15)
C(32)-C(31)-N(2)-C(21)	70.4(2)
C(6)-C(5)-N(2)-C(31)	75.6(2)
C(3)-C(5)-N(2)-C(31)	-53.7(2)
C(6)-C(5)-N(2)-C(21)	-155.52(17)
C(3)-C(5)-N(2)-C(21)	75.2(2)
C(22)-C(21)-N(2)-C(31)	-160.50(18)
C(22)-C(21)-N(2)-C(5)	68.1(2)
C(16)-C(15)-N(3)-O(3)	5.6(3)
C(14)-C(15)-N(3)-O(3)	-175.3(2)
C(16)-C(15)-N(3)-O(2)	-173.9(2)
C(14)-C(15)-N(3)-O(2)	5.1(3)

Symmetry transformations used to generate equivalent atoms:

[1] Blakemore, P. R., Kim, S.-K., Schulze, V. K., White, J. D., Yokochi, A. F. T. *J. Chem. Soc. Perkin Trans. 1*, In Press.
[2] North, A. C. T.; Phillips, D. C.; Mathews, F. S. *Acta. Crystallogr.* **1968**, *A24*, 351-359.
[3] Siemens (1990). XEMP. Version 4.3. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
[4] Sheldrick, G. M. *Acta Crystallogr.* **1990**, *A46*, 467.
[5] Sheldrick, G. M. In *Crystallographic Computing 6*; Flack, H. D., Parkanyi, L., Simon, K., Eds.; Oxford University Press: Oxford, 1993.
[6]ORTEP-III - Burnett, M. N.; Johnson, C.K., Report ORNL-6895. Oak Ridge National Laboratory, Oak Ridge, Tennessee, 1996. ORTEP3 for Windows - Farrugia, L. J. *J. Appl. Crystallogr.* **1997**, *30*, 565.

X-Ray Crystallographic Date for **51** (JH092601)

Experimental details for JH092601.

The crystalline sample used was that supplied by Mr. Joshua Hansen. Determination of the crystallographic parameters, data collection and structure solution and refinement was done as described elsewhere,[1] with the following details. From a mass of crystals, a well shaped crystal of dimensions 0.40 x 0.20 x 0.05 mm^3 was selected and mounted on the tip of a thin glass fiber using epoxy glue.

An automated routine was used to find and center 15 reflections with $7° < 2\theta < 25°$, with which the crystal was indexed. The reflection list was then expanded to include 87 reflections with $7° < 2\theta < 49.7°$, and the lattice parameters refined against this list. All unique data, including a small set of redundant reflections were collected (-1 – 27, -8 – 1, -19 – 1). Monitoring of three standards during data collection showed a total decay of 17.1%. This decay was undoubtedly due to the loss of solvent of crystallization during the course of the data collection, and, at the end of data collection, the crystal that had formerly been a perfectly clear specimen had become completely opaque. Correction for the effects of absorption anisotropy was carried out by means of psi-scans[2] using the program XEMP v4.3.[3]

The structure was solved using direct methods as programmed in SHELXS-90,[4] which revealed the position of the bicyclic core of the molecule. The solution was refined using the program SHELXL-97,[5] followed by Fourier synthesis, which revealed the positions of the remaining atoms. In addition to the main molecule, there is a highly disordered molecule of solvent located about a twofold rotational axis, which was modeled as a half occupied molecule of diethyl ether. Hydrogen atoms were placed in geometrically idealized positions, with the C-H distances fixed at tabulated values. All hydrogen atoms were given an isotropic displacement parameter equal to 1.5 (methyl group) or 1.2 (all other hydrogen atoms) times that of the atom to which it was attached. During the final cycle of least squares refinement, all non-hydrogen atoms were refined with anisotropic displacement parameters, including the atoms of the disordered solvent molecule.

The molecule contains short contacts between the hydrogen atoms of the hydroxo groups and acceptor groups that may be interpreted as hydrogen bonds, though these are rather long, and at the extreme range of a classical hydrogen bond. These short contacts are given in Table 7.

An ORTEP[6] of the final model is given in Figure 1, with displacement ellipsoids drawn at the 20% probability level.

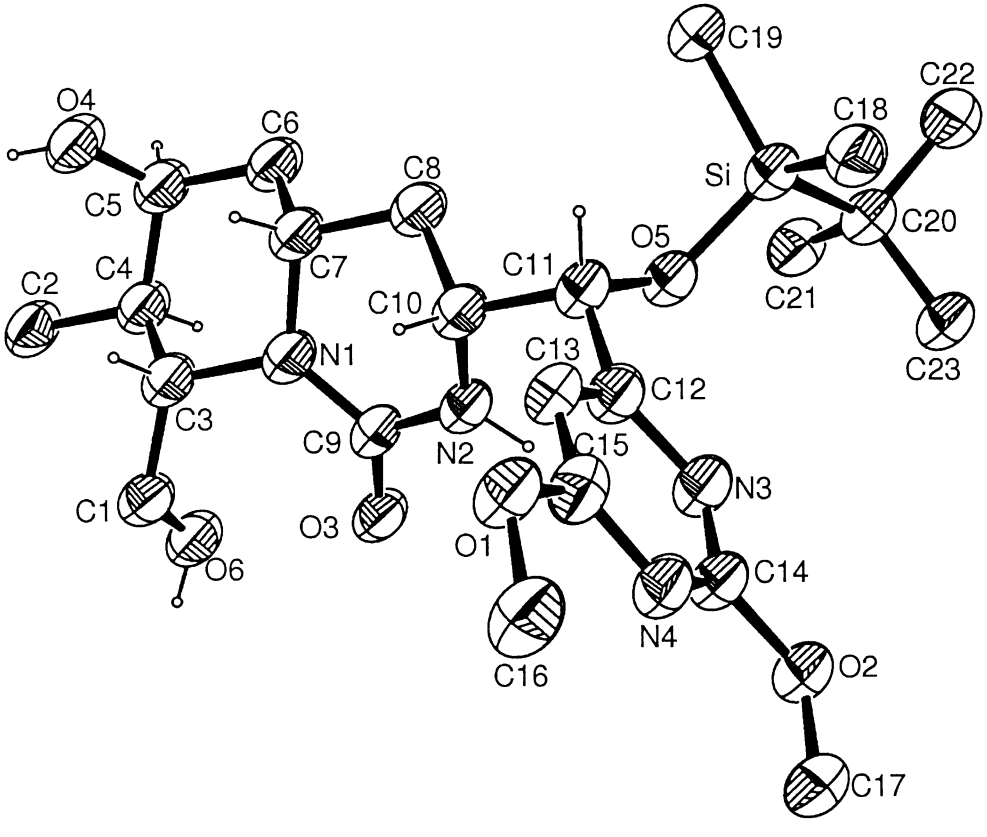


Figure 1. ORTEP of **51** with 20% displacement ellipsoids.

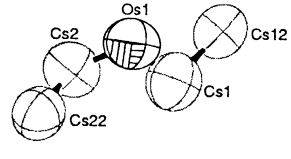


Figure 2. ORTEP of the molecule of Ethyl Ether trapped in the lattice, with 10% displacement ellipsoids.

Table 1. Crystal data and structure refinement for JH092601.

Identification code	JH092601	
Empirical formula	$C_{23}H_{38}N_4O_6Si$ $0.5Et_2O$	
Formula weight	531.72	
Temperature	290(2) K	
Wavelength	1.54173 Å	
Crystal system	Orthorhombic	
Space group	$P\ 2_12_12$	
Unit cell dimensions	a = 23.105(3) Å	α= 90°.
	b = 8.107(1) Å	β= 90°.
	c = 15.917(4) Å	γ = 90°.
Volume	2981.4(9) $Å^3$	
Z	4	
Density (calculated)	1.182 Mg/m^3	
Absorption coefficient	1.062 mm^{-1}	
F(000)	1148	
Crystal size	0.40 x 0.20 x 0.05 mm^3	
Theta range for data collection	2.78 to 67.68°.	
Index ranges	-1<=h<=27, -8<=k<=1, -19<=l<=1	
Reflections collected	3630	
Independent reflections	3430 [R(int) = 0.0653]	
Completeness to theta = 67.68°	91.3 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9488 and 0.6761	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3430 / 48 / 352	
Goodness-of-fit on F^2	0.907	
Final R indices [I>2sigma(I)]	R1 = 0.0560, wR2 = 0.1849	
R indices (all data)	R1 = 0.0652, wR2 = 0.2053	
Absolute structure parameter	-0.05(9)	
Largest diff. peak and hole	0.072 and -0.115 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for jh092601. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	3085(5)	7252(12)	-544(6)	364(4)
C(2)	3743(5)	10324(11)	-581(6)	381(4)
C(3)	3212(4)	8166(9)	243(5)	329(3)
C(4)	3749(4)	9247(10)	186(5)	330(3)
C(5)	3837(5)	10207(11)	996(6)	348(3)
C(6)	3801(4)	9139(9)	1760(6)	336(3)
C(7)	3249(4)	8150(8)	1794(5)	320(2)
C(8)	3218(4)	7075(9)	2588(5)	329(3)
C(9)	3212(4)	5426(7)	1037(7)	315(2)
C(10)	2791(4)	5688(8)	2455(5)	313(2)
C(11)	2709(3)	4647(8)	3241(5)	305(2)
C(12)	2206(4)	3436(10)	3106(5)	311(2)
C(13)	1711(4)	4105(7)	3102(4)	289(2)
C(14)	1768(6)	883(8)	2835(5)	323(3)
C(15)	1260(4)	3124(12)	2988(5)	319(3)
C(16)	242(4)	2756(14)	2771(9)	392(5)
C(17)	1341(4)	-1676(9)	2464(6)	350(3)
C(18)	2884(4)	3133(12)	5176(5)	347(3)
C(19)	3748(4)	5787(9)	4728(6)	350(3)
C(20)	4085(4)	2171(10)	4395(6)	335(3)
C(21)	4491(4)	2549(14)	3669(7)	366(4)
C(22)	4434(5)	2200(15)	5234(7)	386(4)
C(23)	3829(5)	416(9)	4298(7)	369(4)
N(1)	3229(3)	7078(7)	1023(4)	324(2)
N(2)	3032(3)	4726(7)	1783(4)	322(2)
N(3)	2292(4)	1735(9)	2963(5)	351(3)
N(4)	1271(3)	1520(8)	2857(4)	313(2)
O(1)	740(3)	3833(7)	2983(5)	348(2)
O(2)	1852(3)	-777(6)	2682(4)	328(2)
O(3)	3353(3)	4548(6)	437(4)	344(2)
O(4)	3379(3)	11448(7)	1094(4)	379(3)

O(5)	3219(2)	3799(6)	3460(3)	320(2)
O(6)	3534(4)	6353(8)	-886(4)	391(3)
Si	3487(1)	3720(3)	4434(2)	323(1)
OS1	4906(13)	5920(50)	465(13)	511(11)
CS1	4698(17)	4430(60)	90(20)	541(13)
CS12	5010(20)	4330(60)	-752(17)	510(15)
CS2	4800(20)	4750(70)	1130(20)	541(13)
CS22	5191(10)	5360(60)	1842(18)	466(13)

Table 3. Bond lengths [Å] and angles [°] for jh092601.

C(1)-O(6)	1.379(11)
C(1)-C(3)	1.484(11)
C(2)-C(4)	1.502(11)
C(3)-N(1)	1.523(10)
C(3)-C(4)	1.522(11)
C(4)-C(5)	1.520(12)
C(5)-O(4)	1.468(11)
C(5)-C(6)	1.495(12)
C(6)-C(7)	1.507(10)
C(7)-N(1)	1.505(9)
C(7)-C(8)	1.538(10)
C(8)-C(10)	1.510(10)
C(9)-O(3)	1.236(10)
C(9)-N(1)	1.340(8)
C(9)-N(2)	1.381(10)
C(10)-N(2)	1.436(9)
C(10)-C(11)	1.522(10)
C(11)-O(5)	1.408(8)
C(11)-C(12)	1.535(10)
C(12)-C(13)	1.267(9)
C(12)-N(3)	1.411(11)
C(13)-C(15)	1.323(10)
C(14)-N(4)	1.259(10)
C(14)-O(2)	1.381(9)
C(14)-N(3)	1.409(11)
C(15)-N(4)	1.317(11)
C(15)-O(1)	1.331(9)
C(16)-O(1)	1.483(11)
C(17)-O(2)	1.430(9)
C(18)-Si	1.888(8)
C(19)-Si	1.841(7)
C(20)-C(21)	1.520(12)
C(20)-C(23)	1.549(11)
C(20)-C(22)	1.559(13)

C(20)-Si	1.868(9)
O(5)-Si	1.671(5)
OS1-CS1	1.431(10)
OS1-CS2	1.439(10)
CS1-CS12	1.525(10)
CS2-CS22	1.528(10)

O(6)-C(1)-C(3)	116.6(9)
C(1)-C(3)-N(1)	113.8(6)
C(1)-C(3)-C(4)	113.5(8)
N(1)-C(3)-C(4)	111.1(7)
C(2)-C(4)-C(5)	113.2(7)
C(2)-C(4)-C(3)	112.1(8)
C(5)-C(4)-C(3)	110.7(7)
O(4)-C(5)-C(6)	105.7(7)
O(4)-C(5)-C(4)	110.2(8)
C(6)-C(5)-C(4)	112.7(7)
C(5)-C(6)-C(7)	112.6(8)
N(1)-C(7)-C(6)	107.6(6)
N(1)-C(7)-C(8)	110.0(5)
C(6)-C(7)-C(8)	111.7(7)
C(10)-C(8)-C(7)	109.7(7)
O(3)-C(9)-N(1)	123.7(9)
O(3)-C(9)-N(2)	120.5(5)
N(1)-C(9)-N(2)	115.8(9)
N(2)-C(10)-C(8)	104.9(6)
N(2)-C(10)-C(11)	111.1(6)
C(8)-C(10)-C(11)	112.4(6)
O(5)-C(11)-C(10)	111.6(6)
O(5)-C(11)-C(12)	110.9(6)
C(10)-C(11)-C(12)	109.6(6)
C(13)-C(12)-N(3)	123.0(8)
C(13)-C(12)-C(11)	114.2(7)
N(3)-C(12)-C(11)	122.7(9)
C(12)-C(13)-C(15)	117.1(6)
N(4)-C(14)-O(2)	122.2(9)

N(4)-C(14)-N(3)	125.3(6)
O(2)-C(14)-N(3)	112.5(10)
N(4)-C(15)-C(13)	126.8(7)
N(4)-C(15)-O(1)	116.3(9)
C(13)-C(15)-O(1)	116.9(8)
C(21)-C(20)-C(23)	110.2(9)
C(21)-C(20)-C(22)	109.2(8)
C(23)-C(20)-C(22)	107.3(9)
C(21)-C(20)-Si	110.2(7)
C(23)-C(20)-Si	109.7(7)
C(22)-C(20)-Si	110.1(7)
C(9)-N(1)-C(7)	124.3(8)
C(9)-N(1)-C(3)	126.3(8)
C(7)-N(1)-C(3)	109.3(5)
C(9)-N(2)-C(10)	122.3(6)
C(14)-N(3)-C(12)	112.5(8)
C(14)-N(4)-C(15)	115.3(7)
C(15)-O(1)-C(16)	116.5(7)
C(14)-O(2)-C(17)	115.1(7)
C(11)-O(5)-Si	124.0(5)
O(5)-Si-C(19)	108.8(3)
O(5)-Si-C(20)	105.6(4)
C(19)-Si-C(20)	112.2(4)
O(5)-Si-C(18)	108.5(4)
C(19)-Si-C(18)	108.2(5)
C(20)-Si-C(18)	113.4(4)
CS1-OS1-CS2	72(3)
OS1-CS1-CS12	104.5(19)
OS1-CS2-CS22	103.6(19)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3)for jh092601. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	527(12)	236(6)	328(7)	-19(6)	4(8)	5(7)
C(2)	554(12)	233(5)	357(7)	25(6)	46(9)	-28(8)
C(3)	433(7)	220(4)	335(7)	3(5)	5(6)	10(6)
C(4)	438(7)	220(4)	331(6)	7(5)	23(6)	-7(6)
C(5)	461(9)	229(5)	354(7)	-6(6)	24(7)	-11(6)
C(6)	440(7)	211(4)	358(7)	-1(5)	19(6)	-31(5)
C(7)	440(7)	200(4)	320(5)	-1(4)	12(5)	-9(5)
C(8)	450(7)	220(4)	317(5)	-14(4)	18(6)	-14(5)
C(9)	418(7)	184(4)	344(7)	4(5)	-10(6)	0(4)
C(10)	417(6)	209(4)	314(5)	-3(4)	5(5)	-5(5)
C(11)	368(5)	202(4)	343(6)	9(4)	-1(5)	10(5)
C(12)	389(7)	230(6)	313(5)	3(5)	10(5)	16(6)
C(13)	336(5)	189(3)	341(5)	13(3)	4(4)	0(4)
C(14)	447(9)	194(5)	327(6)	-6(4)	-7(7)	-6(6)
C(15)	360(7)	245(7)	354(7)	25(6)	34(6)	34(7)
C(16)	363(6)	293(9)	520(16)	27(11)	18(7)	-22(6)
C(17)	434(8)	214(5)	402(9)	-22(5)	-37(7)	-3(5)
C(18)	427(7)	281(7)	333(6)	10(6)	23(5)	-38(6)
C(19)	478(8)	210(4)	364(7)	-6(5)	-9(7)	-5(6)
C(20)	420(7)	237(5)	349(7)	-8(6)	-19(7)	16(5)
C(21)	409(7)	276(8)	412(11)	-12(8)	7(8)	7(7)
C(22)	457(9)	305(10)	395(10)	-15(9)	-34(8)	55(8)
C(23)	503(10)	211(4)	393(9)	-11(5)	0(8)	6(6)
N(1)	435(6)	211(4)	325(4)	8(4)	6(5)	-13(4)
N(2)	444(6)	193(3)	327(5)	-3(4)	-2(5)	14(4)
N(3)	469(8)	228(5)	356(6)	11(4)	2(6)	-4(6)
N(4)	386(5)	208(4)	345(5)	6(4)	1(4)	25(4)
O(1)	381(4)	220(3)	444(7)	0(4)	2(4)	6(4)
O(2)	411(4)	216(3)	358(4)	-8(3)	-9(4)	4(3)
O(3)	502(6)	204(3)	326(4)	-17(3)	31(4)	0(4)
O(4)	541(8)	218(3)	377(5)	1(4)	65(6)	11(5)

O(5)	427(4)	206(3)	327(3)	-1(3)	2(3)	-3(4)
O(6)	606(10)	239(4)	329(4)	2(4)	41(5)	-5(6)
Si	419(2)	213(1)	337(2)	2(1)	1(2)	-6(1)
OS1	471(18)	520(20)	548(16)	31(13)	-42(18)	25(17)
CS1	550(20)	530(20)	535(17)	24(15)	-31(14)	-7(18)
CS12	472(17)	520(30)	540(20)	30(20)	-50(20)	0(30)
CS2	550(20)	530(20)	535(17)	24(15)	-31(14)	-7(18)
CS22	450(20)	460(30)	498(18)	20(20)	40(14)	0(20)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for jh092601.

	x	y	z	U(eq)
H(1A)	2766	6505	-435	436
H(1B)	2955	8040	-962	436
H(2A)	3696	9652	-1073	572
H(2B)	4102	10918	-618	572
H(2C)	3428	11092	-544	572
H(3)	2887	8923	325	395
H(4)	4081	8503	129	396
H(5)	4214	10757	982	418
H(6A)	3826	9827	2257	404
H(6B)	4128	8389	1765	404
H(7)	2917	8906	1786	384
H(8A)	3597	6624	2711	395
H(8B)	3097	7743	3063	395
H(10)	2418	6145	2280	376
H(11)	2606	5383	3706	366
H(13)	1666	5235	3177	346
H(16A)	-109	3387	2792	588
H(16B)	221	1867	3169	588
H(16C)	293	2315	2216	588
H(17A)	1437	-2815	2379	525
H(17B)	1180	-1231	1956	525
H(17C)	1063	-1584	2909	525
H(18A)	2590	3968	5165	521
H(18B)	3035	3035	5736	521
H(18C)	2721	2097	5005	521
H(19A)	4053	6113	4354	526
H(19B)	3891	5764	5294	526
H(19C)	3435	6563	4689	526
H(21A)	4639	3650	3727	548
H(21B)	4284	2455	3148	548

H(21C)	4807	1780	3673	548
H(22A)	4178	1970	5695	578
H(22B)	4605	3269	5310	578
H(22C)	4733	1379	5214	578
H(23A)	3573	191	4759	554
H(23B)	4136	-379	4294	554
H(23C)	3617	348	3780	554
H(2)	3064	3676	1848	386
H(4A)	3377	12059	683	568
H(6)	3424	5902	-1319	587
HS1A	4282	4470	16	649
HS1B	4794	3485	440	649
HS1C	4925	3294	-1014	764
HS1D	5418	4437	-667	764
HS1E	4876	5214	-1108	764
HS2A	4914	3644	951	649
HS2B	4401	4741	1293	649
HS2C	5341	4427	2146	699
HS2D	4968	6037	2214	699
HS2E	5506	5985	1614	699

Table 6. Torsion angles [°] for jh092601.

O(6)-C(1)-C(3)-N(1)	-77.0(10)
O(6)-C(1)-C(3)-C(4)	51.5(10)
C(1)-C(3)-C(4)-C(2)	49.9(9)
N(1)-C(3)-C(4)-C(2)	179.7(7)
C(1)-C(3)-C(4)-C(5)	177.2(8)
N(1)-C(3)-C(4)-C(5)	-52.9(9)
C(2)-C(4)-C(5)-O(4)	58.2(10)
C(3)-C(4)-C(5)-O(4)	-68.6(8)
C(2)-C(4)-C(5)-C(6)	176.0(9)
C(3)-C(4)-C(5)-C(6)	49.3(11)
O(4)-C(5)-C(6)-C(7)	66.6(9)
C(4)-C(5)-C(6)-C(7)	-53.8(11)
C(5)-C(6)-C(7)-N(1)	59.8(8)
C(5)-C(6)-C(7)-C(8)	-179.4(7)
N(1)-C(7)-C(8)-C(10)	-39.8(9)
C(6)-C(7)-C(8)-C(10)	-159.3(6)
C(7)-C(8)-C(10)-N(2)	63.3(8)
C(7)-C(8)-C(10)-C(11)	-175.9(7)
N(2)-C(10)-C(11)-O(5)	51.7(8)
C(8)-C(10)-C(11)-O(5)	-65.4(8)
N(2)-C(10)-C(11)-C(12)	-71.4(8)
C(8)-C(10)-C(11)-C(12)	171.4(7)
O(5)-C(11)-C(12)-C(13)	164.4(6)
C(10)-C(11)-C(12)-C(13)	-72.0(8)
O(5)-C(11)-C(12)-N(3)	-18.2(10)
C(10)-C(11)-C(12)-N(3)	105.5(8)
N(3)-C(12)-C(13)-C(15)	2.5(11)
C(11)-C(12)-C(13)-C(15)	180.0(7)
C(12)-C(13)-C(15)-N(4)	-1.7(13)
C(12)-C(13)-C(15)-O(1)	-179.9(7)
O(3)-C(9)-N(1)-C(7)	-159.4(8)
N(2)-C(9)-N(1)-C(7)	19.7(13)
O(3)-C(9)-N(1)-C(3)	22.7(15)
N(2)-C(9)-N(1)-C(3)	-158.2(8)

C(6)-C(7)-N(1)-C(9)	119.3(9)
C(8)-C(7)-N(1)-C(9)	-2.6(12)
C(6)-C(7)-N(1)-C(3)	-62.5(8)
C(8)-C(7)-N(1)-C(3)	175.6(8)
C(1)-C(3)-N(1)-C(9)	8.7(13)
C(4)-C(3)-N(1)-C(9)	-121.0(9)
C(1)-C(3)-N(1)-C(7)	-169.5(8)
C(4)-C(3)-N(1)-C(7)	60.9(8)
O(3)-C(9)-N(2)-C(10)	-171.5(8)
N(1)-C(9)-N(2)-C(10)	9.4(12)
C(8)-C(10)-N(2)-C(9)	-50.5(9)
C(11)-C(10)-N(2)-C(9)	-172.1(7)
N(4)-C(14)-N(3)-C(12)	-1.3(12)
O(2)-C(14)-N(3)-C(12)	179.1(7)
C(13)-C(12)-N(3)-C(14)	-1.1(12)
C(11)-C(12)-N(3)-C(14)	-178.4(7)
O(2)-C(14)-N(4)-C(15)	-178.3(8)
N(3)-C(14)-N(4)-C(15)	2.1(12)
C(13)-C(15)-N(4)-C(14)	-0.6(13)
O(1)-C(15)-N(4)-C(14)	177.6(7)
N(4)-C(15)-O(1)-C(16)	-4.2(12)
C(13)-C(15)-O(1)-C(16)	174.2(8)
N(4)-C(14)-O(2)-C(17)	6.4(12)
N(3)-C(14)-O(2)-C(17)	-174.0(7)
C(10)-C(11)-O(5)-Si	133.6(5)
C(12)-C(11)-O(5)-Si	-104.0(6)
C(11)-O(5)-Si-C(19)	-68.6(6)
C(11)-O(5)-Si-C(20)	170.8(5)
C(11)-O(5)-Si-C(18)	48.9(6)
C(21)-C(20)-Si-O(5)	50.3(7)
C(23)-C(20)-Si-O(5)	-71.3(8)
C(22)-C(20)-Si-O(5)	170.8(7)
C(21)-C(20)-Si-C(19)	-68.2(7)
C(23)-C(20)-Si-C(19)	170.3(7)
C(22)-C(20)-Si-C(19)	52.4(9)
C(21)-C(20)-Si-C(18)	168.9(6)

C(23)-C(20)-Si-C(18)	47.3(9)
C(22)-C(20)-Si-C(18)	-70.5(8)
CS2-OS1-CS1-CS12	-146(5)
CS1-OS1-CS2-CS22	157(4)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for jh092601 [Å and °].

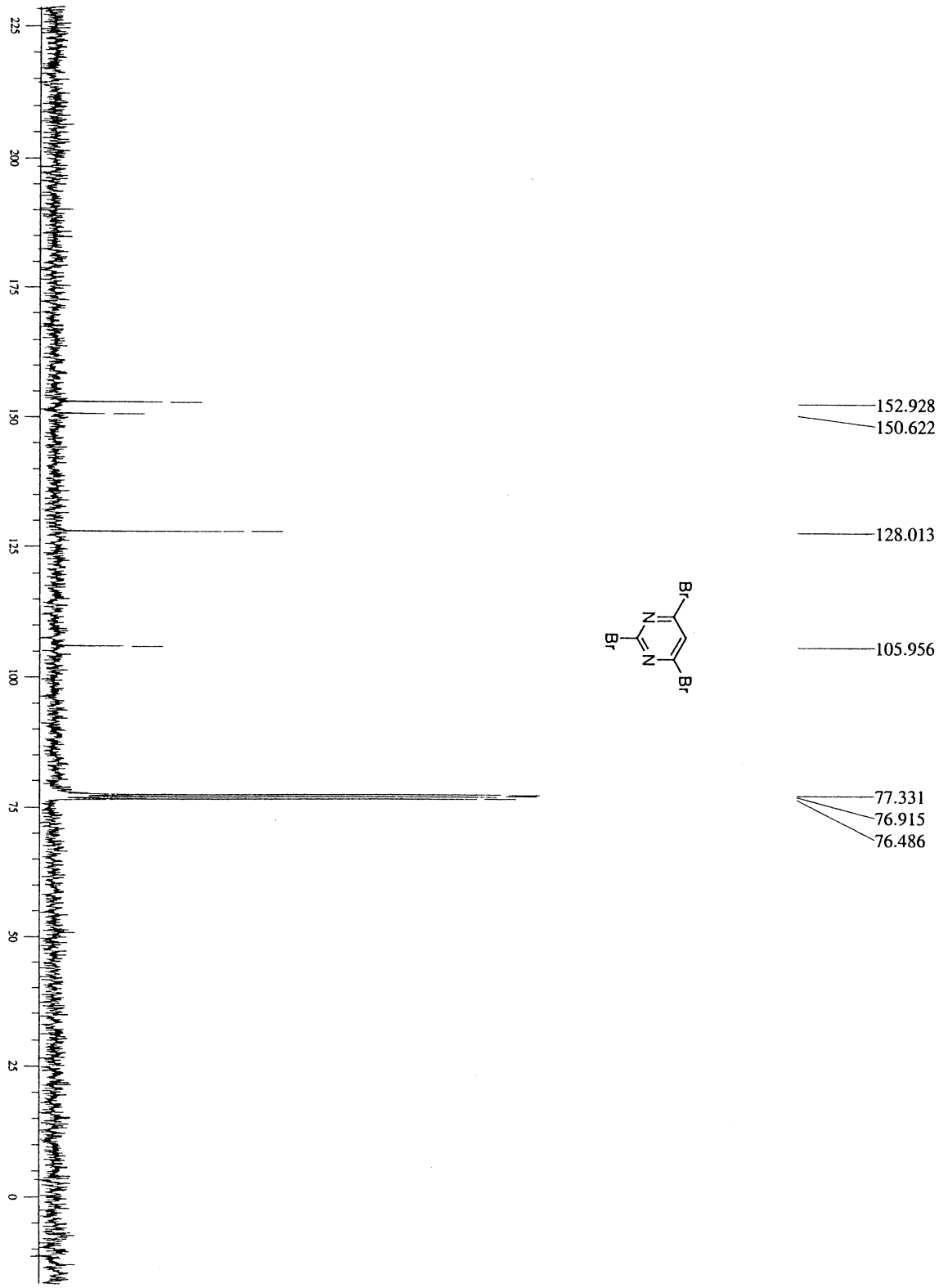
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(4)-H(4A)...O(3)#1	0.82	2.06	2.722(7)	138.1
O(6)-H(6)...N(4)#2	0.82	2.60	3.172(9)	128.4
O(6)-H(6)...O(2)#2	0.82	2.64	3.456(9)	174.2
N(2)-H(2)...O(4)#3	0.86	2.29	2.984(8)	138.3

Symmetry transformations used to generate equivalent atoms:

#1 x,y+1,z #2 -x+1/2,y+1/2,-z #3 x,y-1,z

[1] Blakemore, P. R., Kim, S.-K., Schulze, V. K., White, J. D., Yokochi, A. F. T. *J. Chem. Soc. Perkin Trans. 1*, In Press.
[2] North, A. C. T.; Phillips, D. C.; Mathews, F. S. *Acta. Crystallogr.* **1968**, *A24*, 351-359.
[3] Siemens (1990). XEMP. Version 4.3. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
[4] Sheldrick, G. M. *Acta Crystallogr.* **1990**, *A46*, 467.
[5] Sheldrick, G. M. In *Crystallographic Computing 6*; Flack, H. D., Parkanyi, L., Simon, K., Eds.; Oxford University Press: Oxford, 1993.
[6] ORTEP-III - Burnett, M. N.; Johnson, C.K., Report ORNL-6895. Oak Ridge National Laboratory, Oak Ridge, Tennessee, 1996. ORTEP3 for Windows - Farrugia, L. J. *J. Appl. Crystallogr.* **1997**, *30*, 565.

^{1}H and ^{13}C NMR Spectra for Synthetic Intermediates.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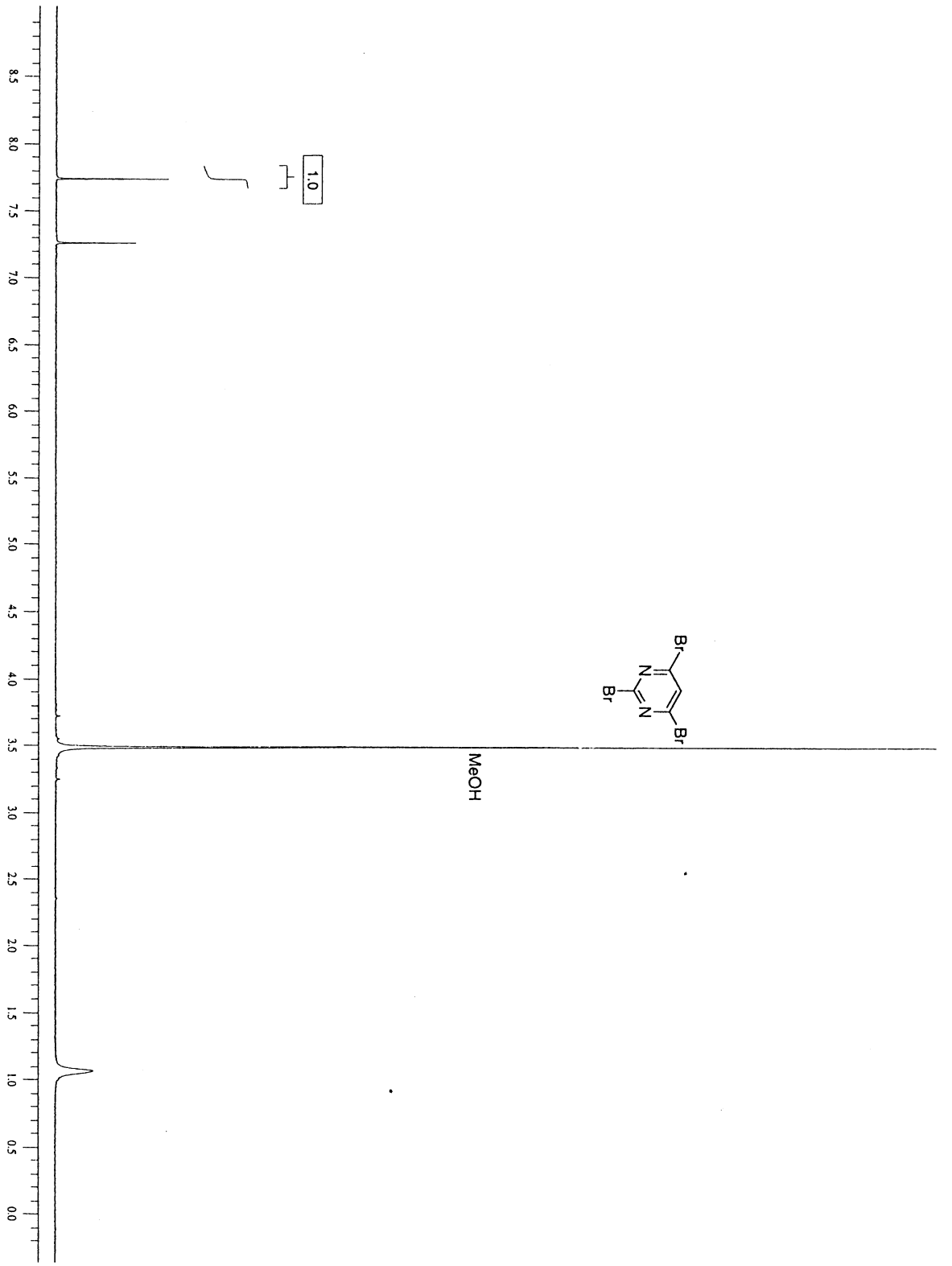
N
Br
Br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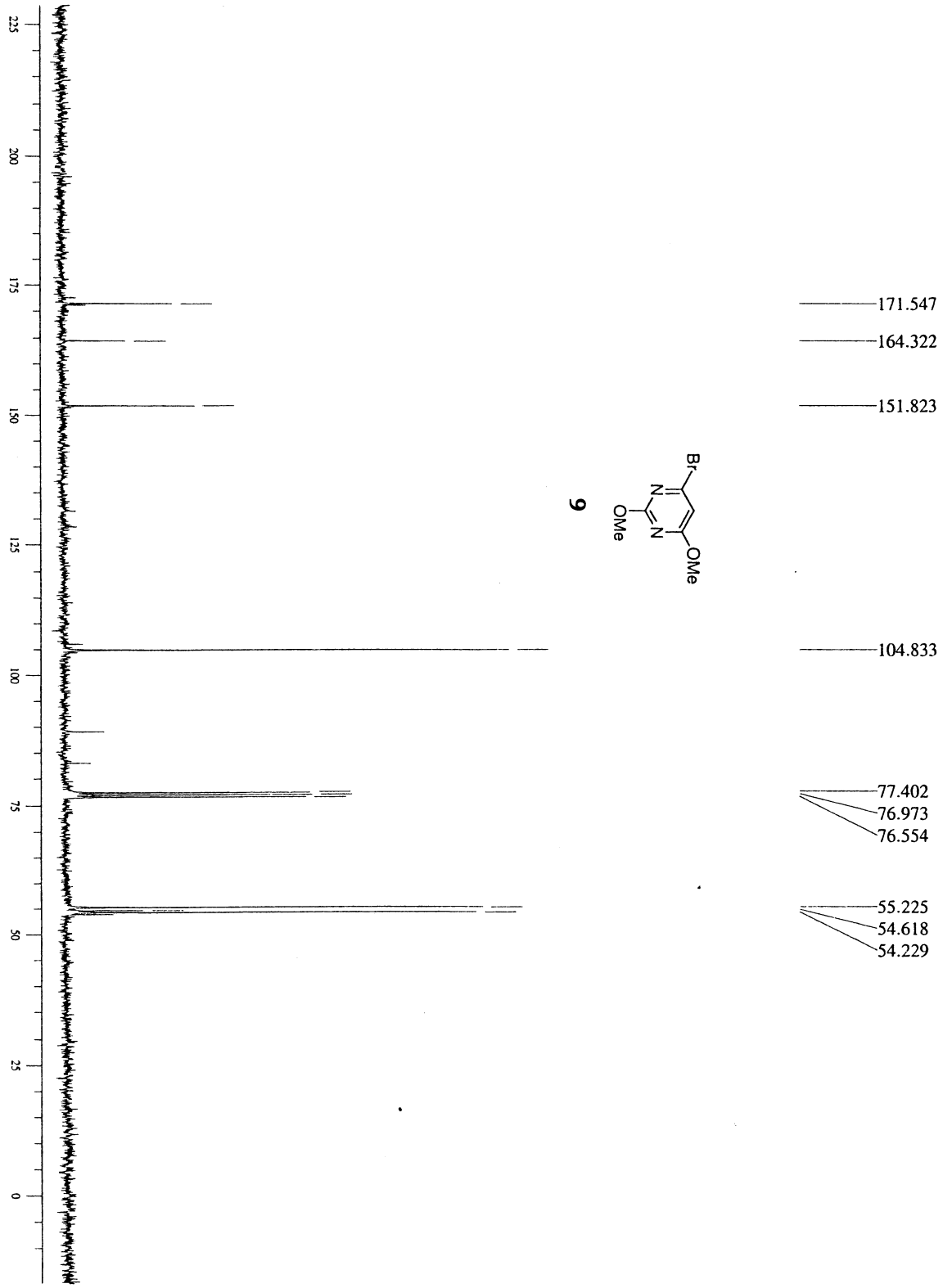
Br
N
Br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75
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54.229
9
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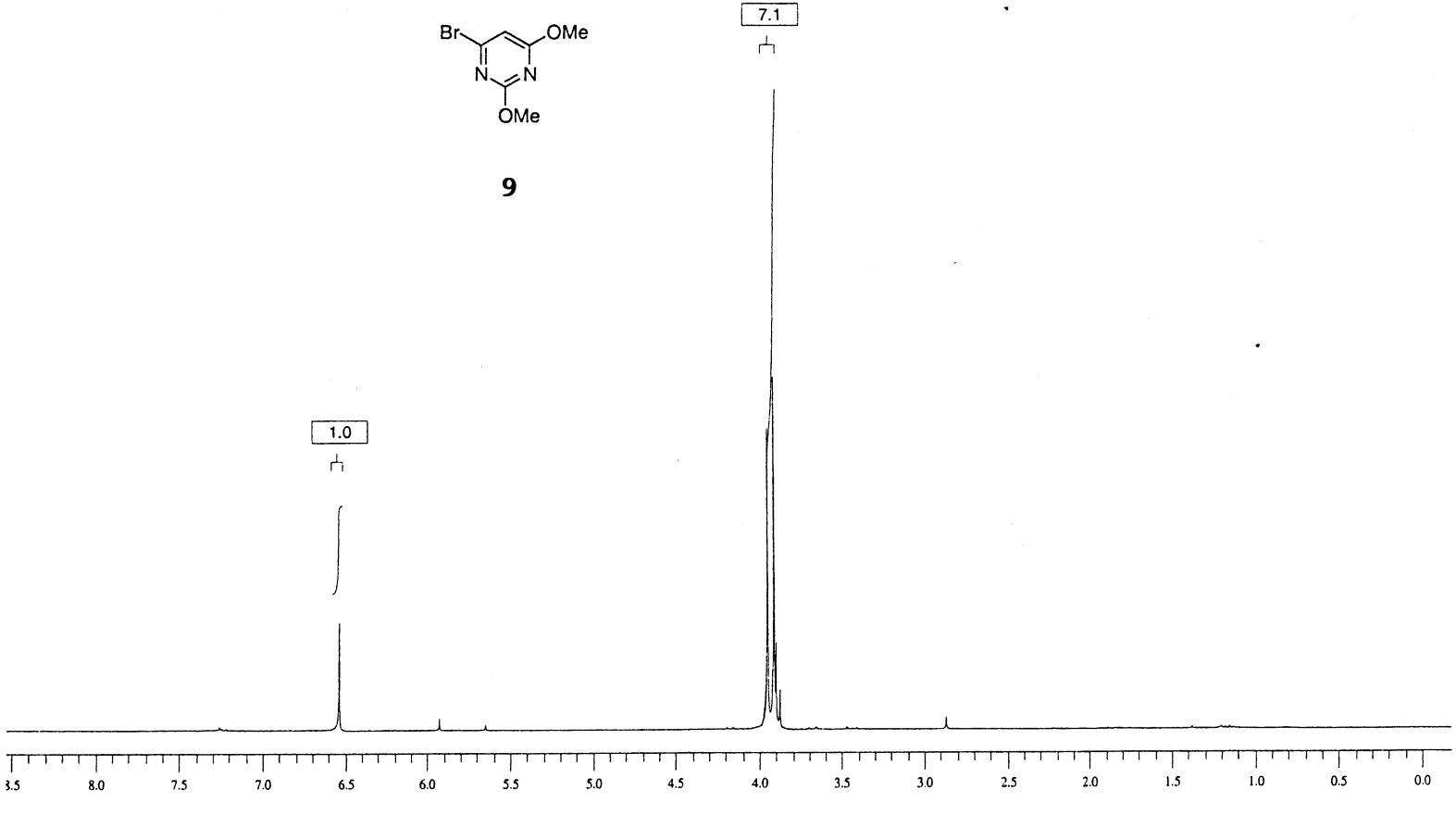
N
OMe
OMe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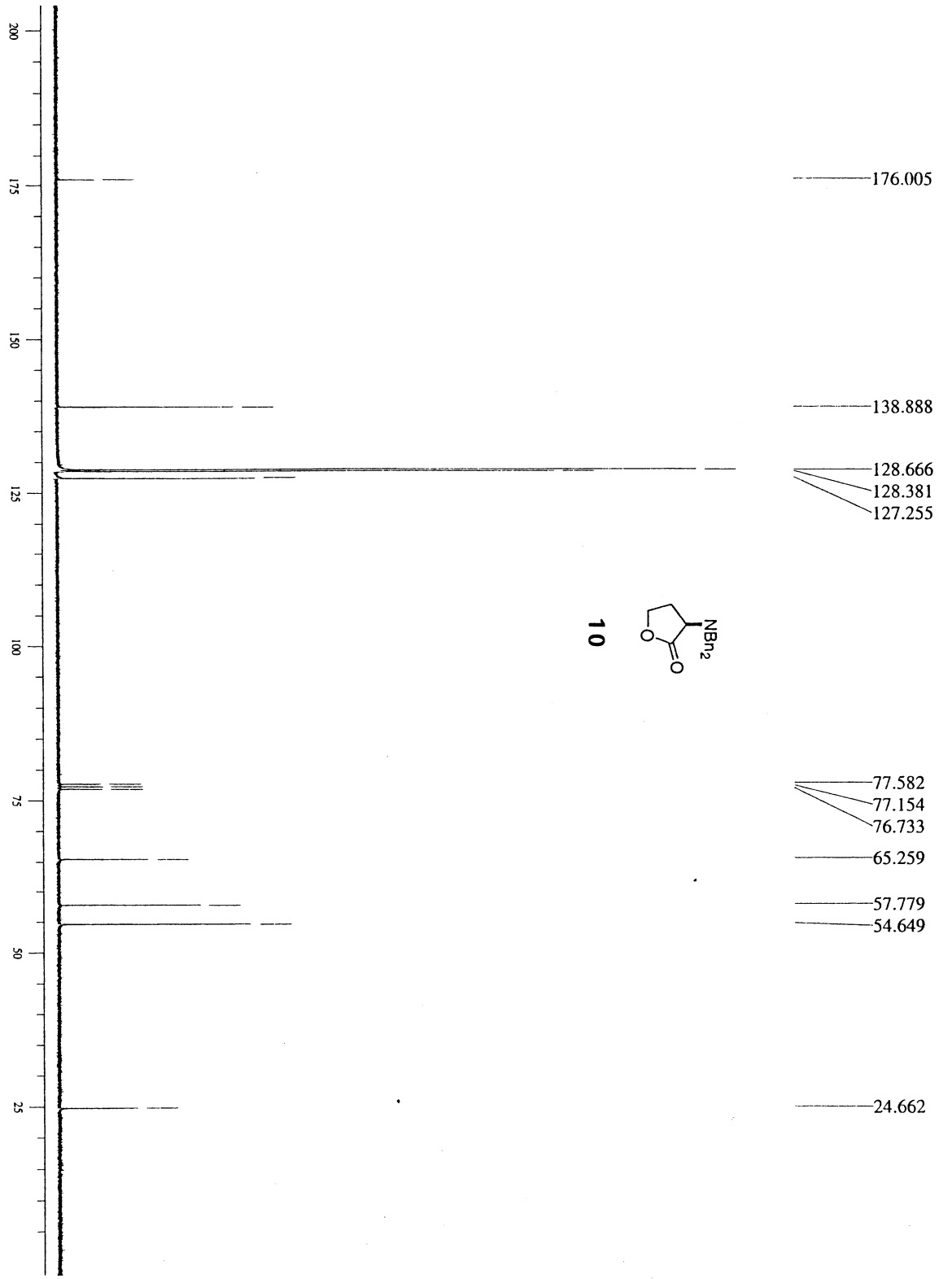
0.0

9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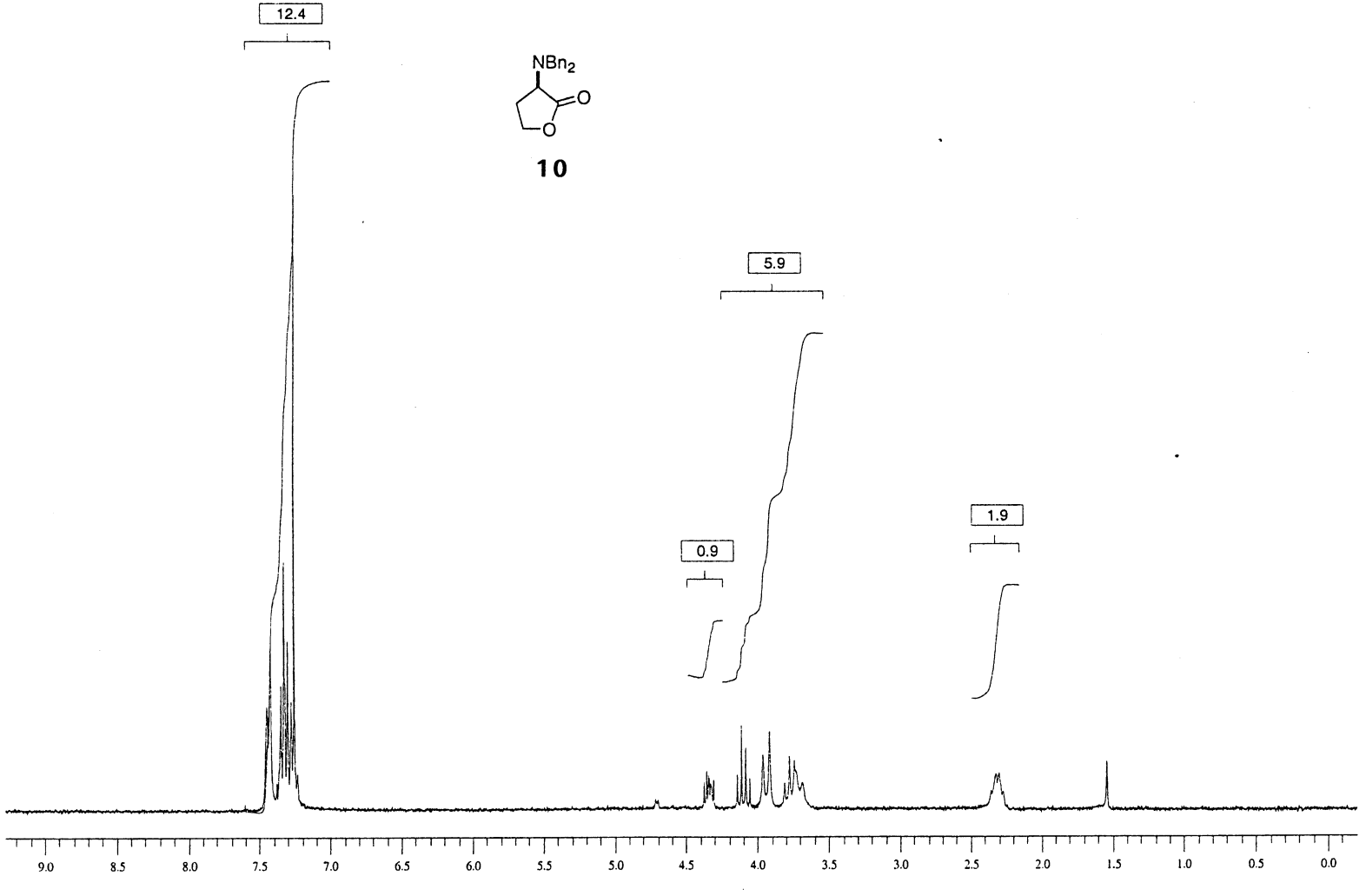
O
200
175
150
125
100
75
50
25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O
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7.0
6.5
6.0
5.5
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4.0
3.5
3.0
2.5
2.0
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1.0
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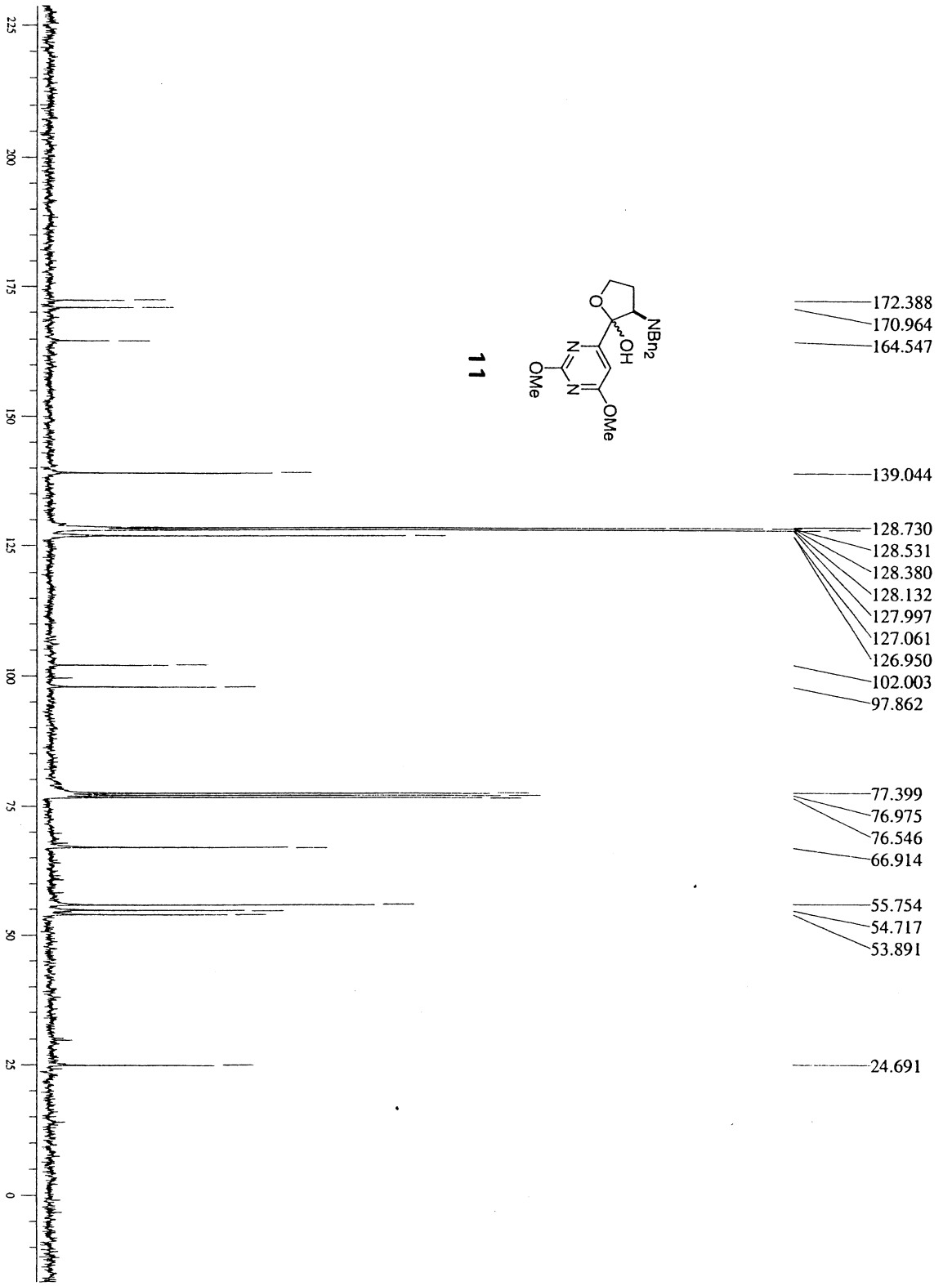
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N
O
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150
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75
50
25
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170.964
164.547
139.044
128.730
128.531
128.380
128.132
127.997
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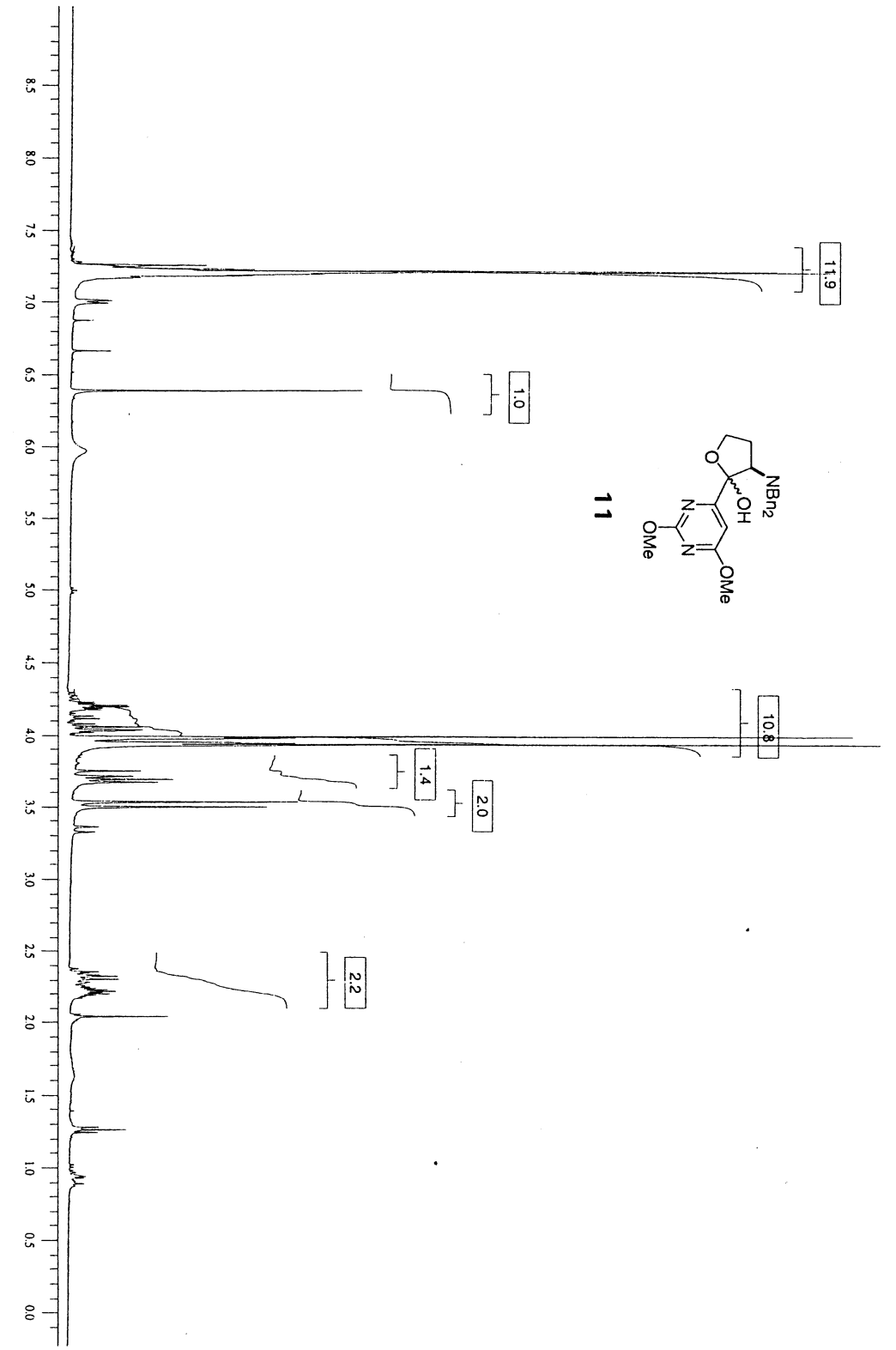
53.891
24.691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OH
OMe
OMe
N
N
O
11
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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4.0
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3.0
2.5
2.0
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1.0
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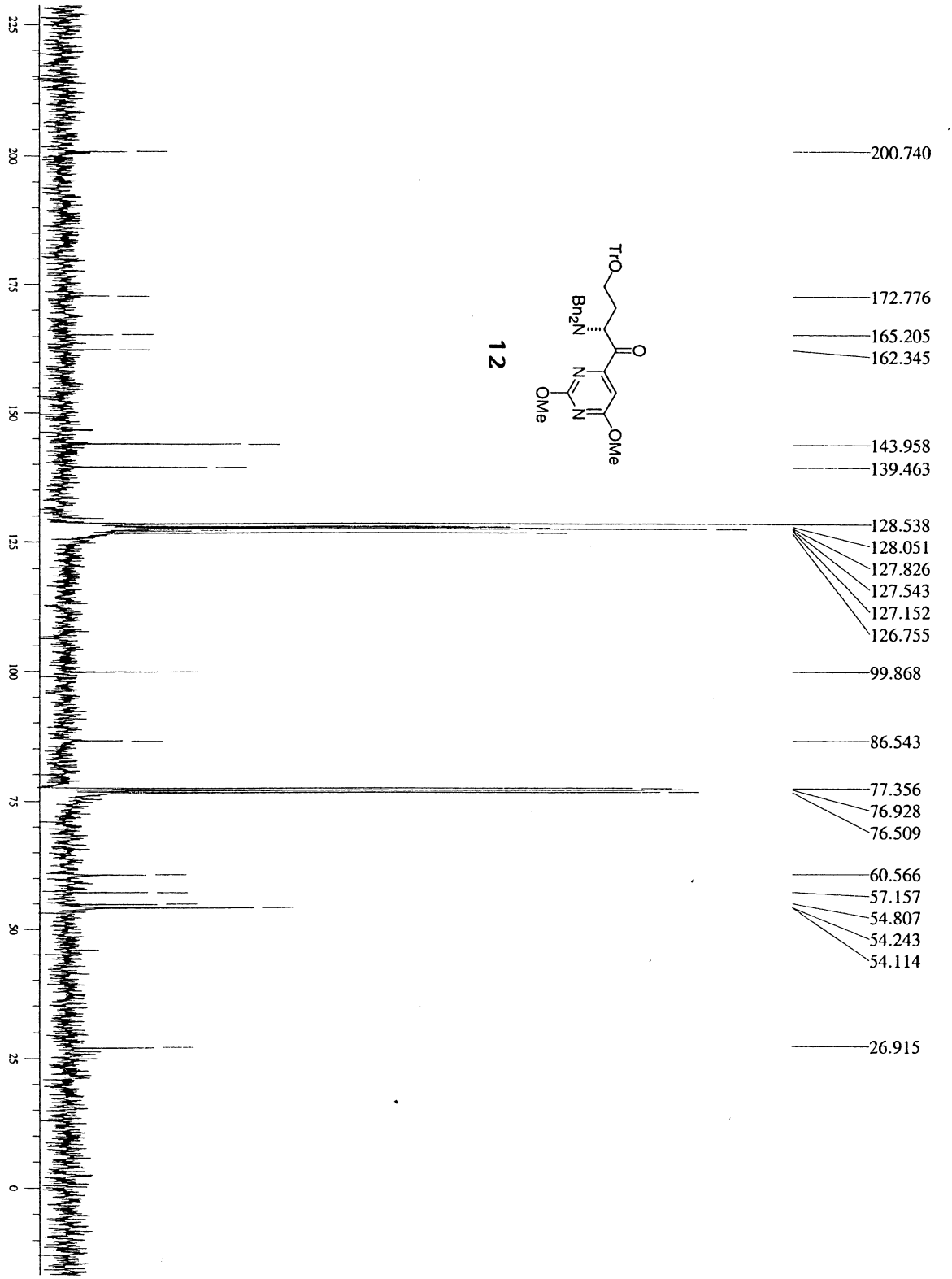
0.0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99.868
86.543
77.356
76.928
76.509
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54.243
54.114
26.915
12
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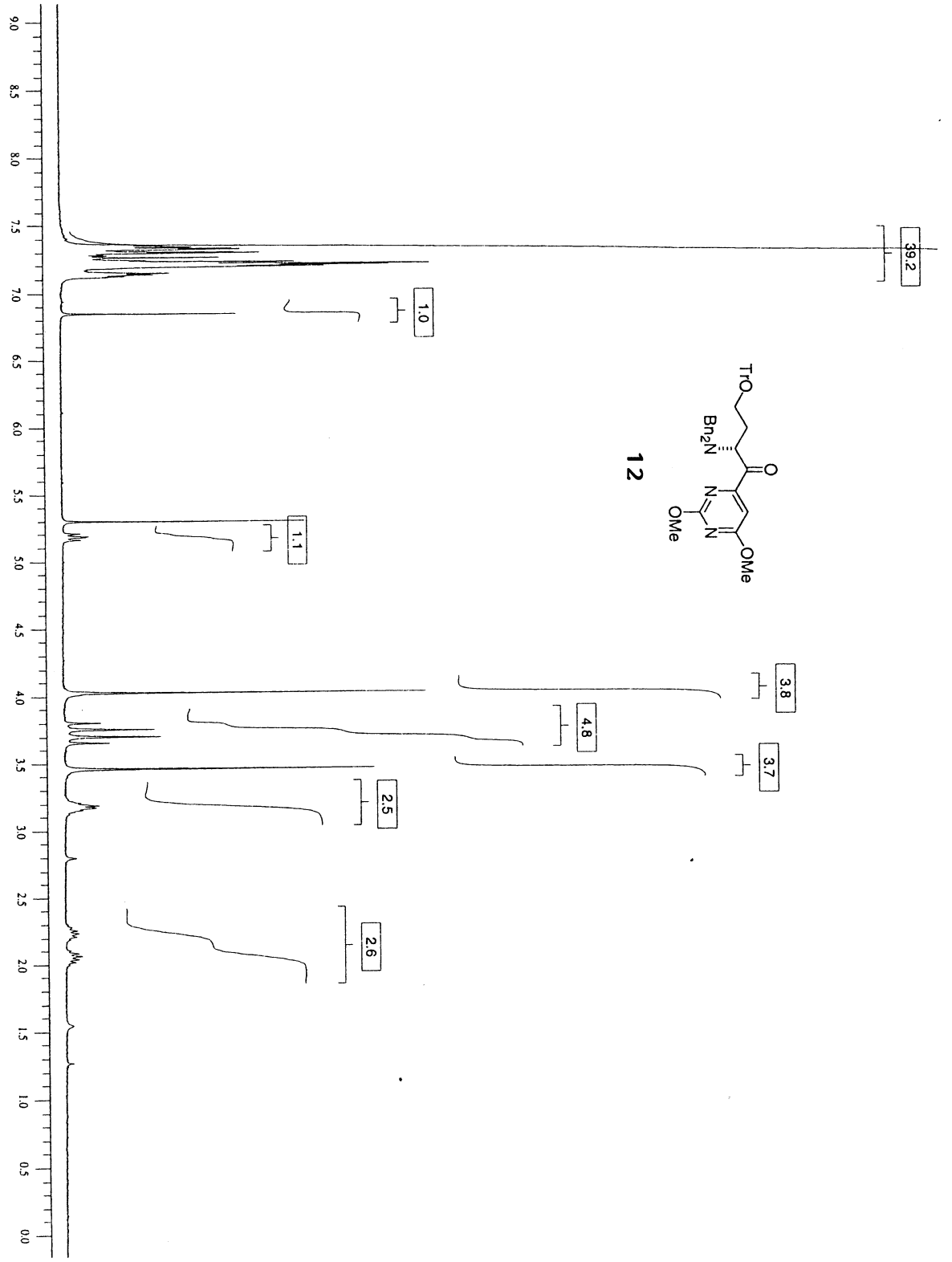
Bn2N
O
N
N
OMe
OMe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Bn2N
O
N
N
OMe
OMe
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4.8
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2.5
2.6
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8.0
7.5
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6.0
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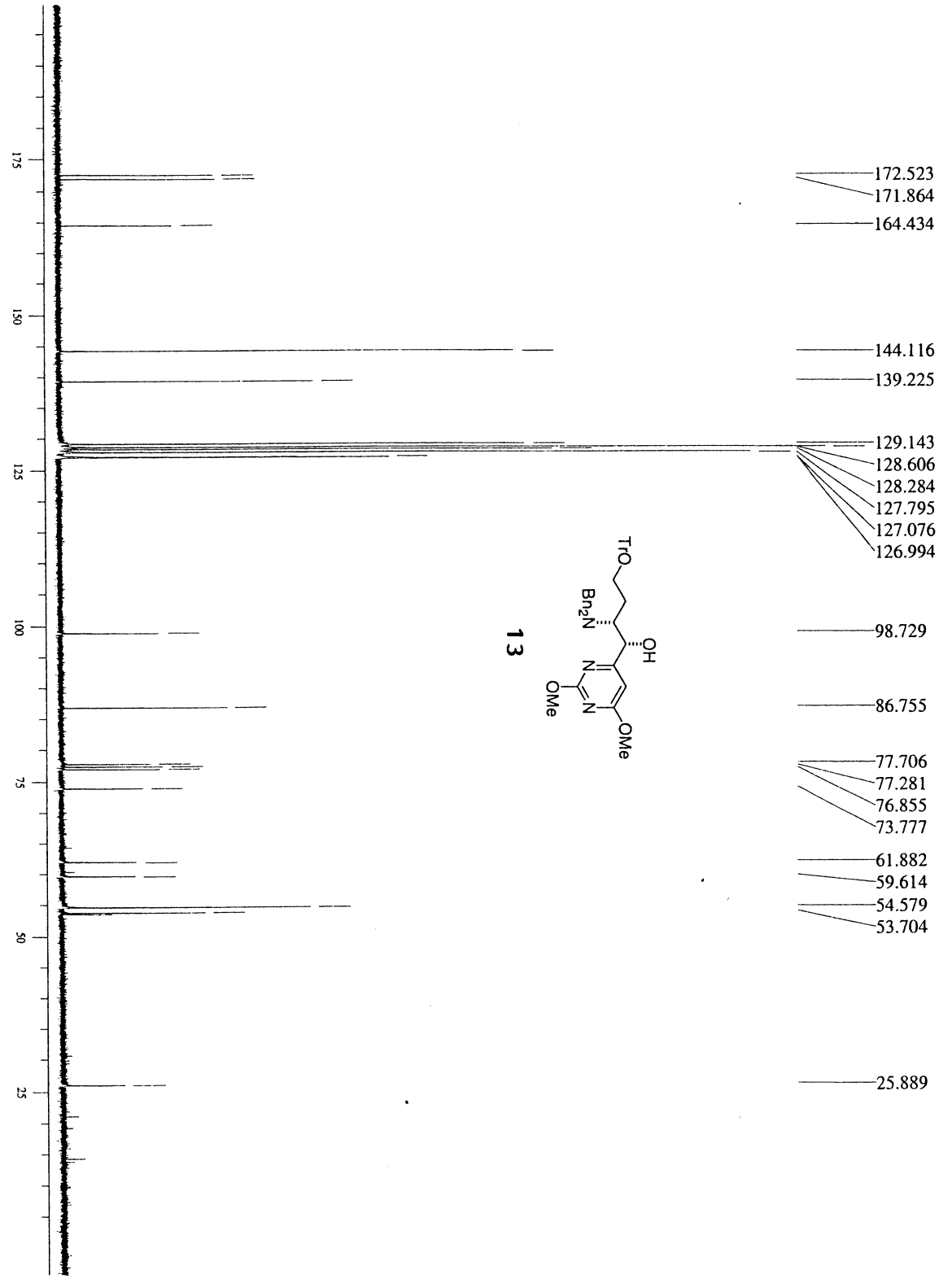
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75
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128.284
127.795
127.076
126.994
98.729
86.755
77.706
77.281
76.855
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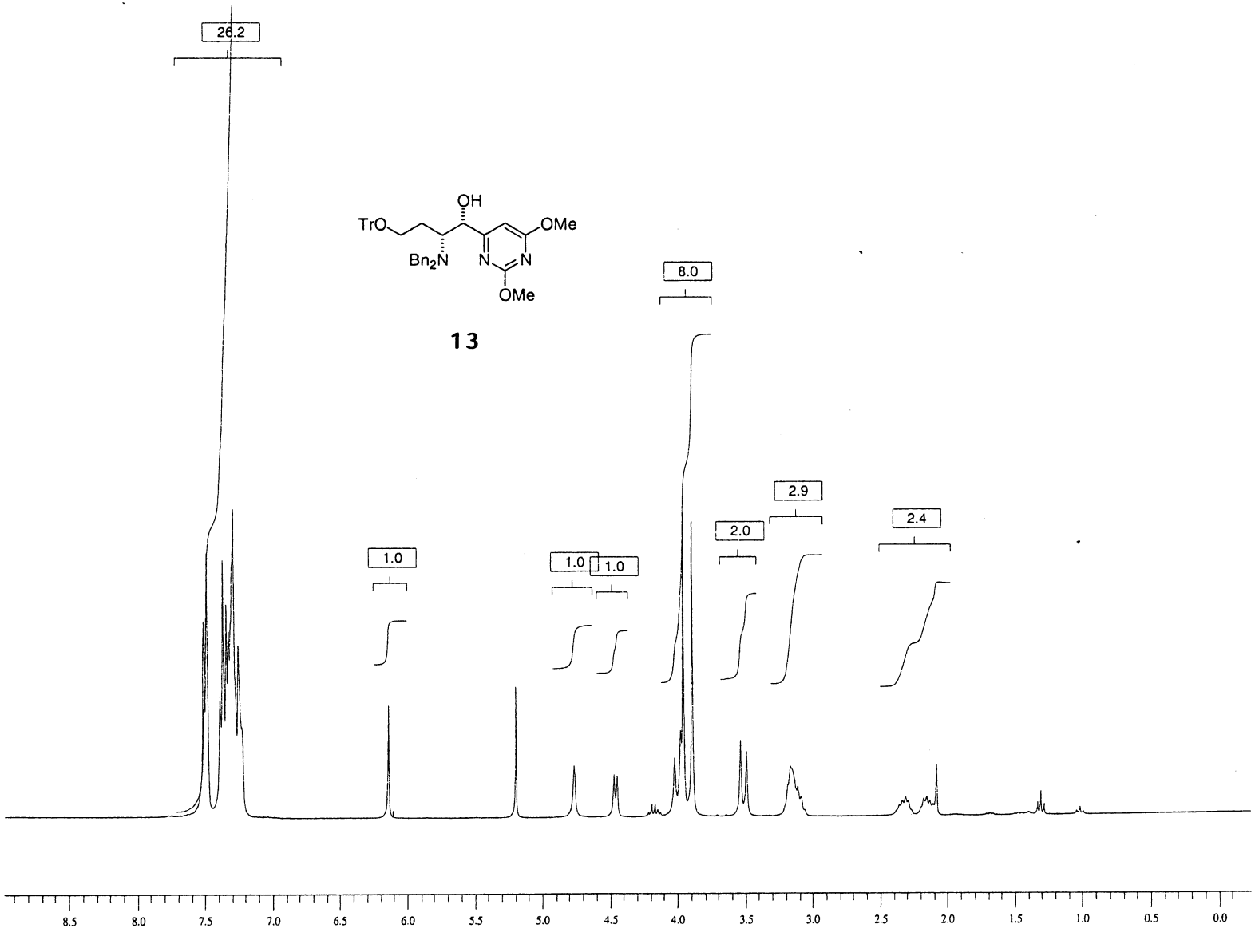
54.579
53.704
25.889
TrO
OH
Bn2N
N
OMe
N
OMe

13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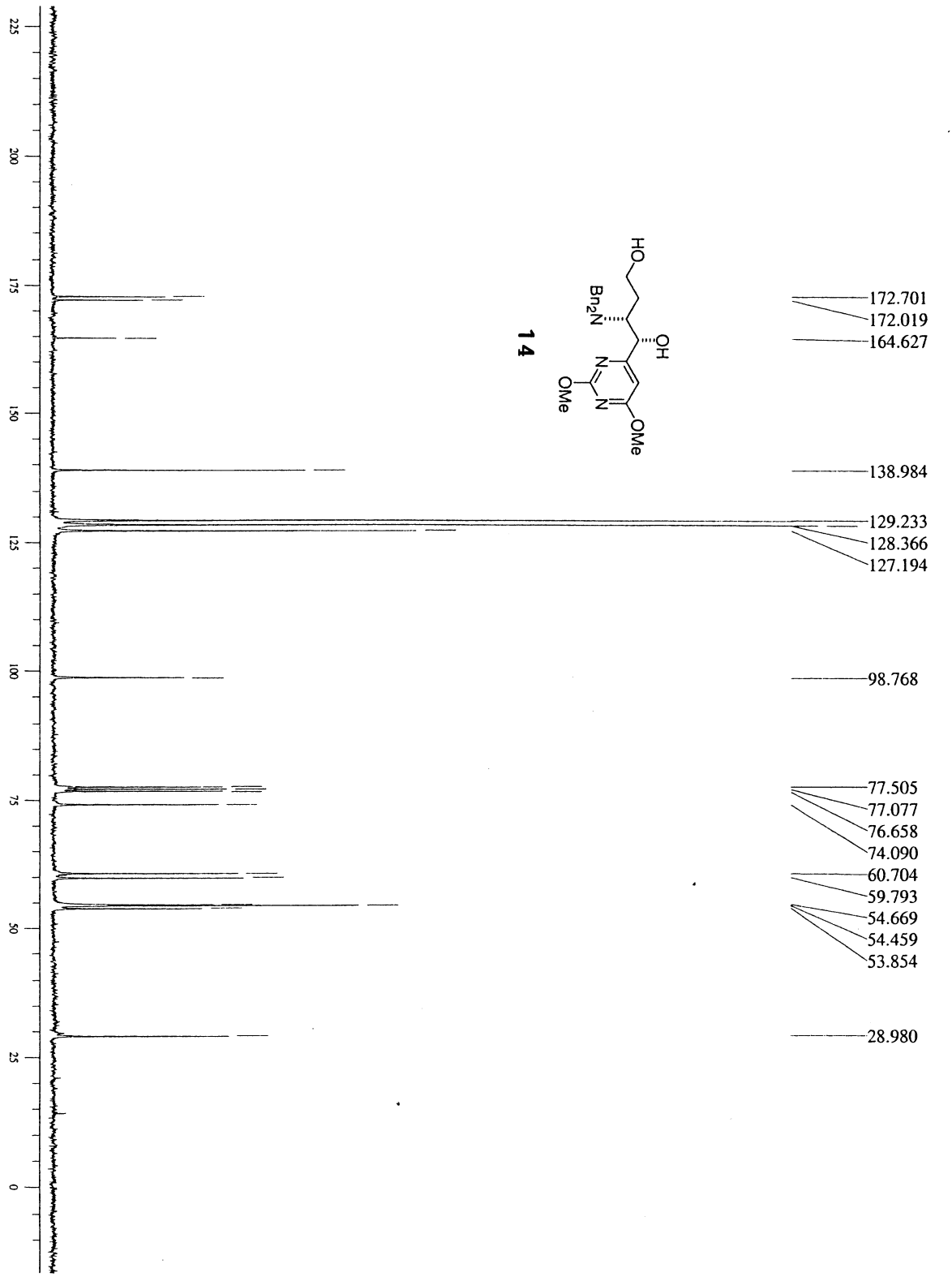
13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25
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164.627
138.984
129.233
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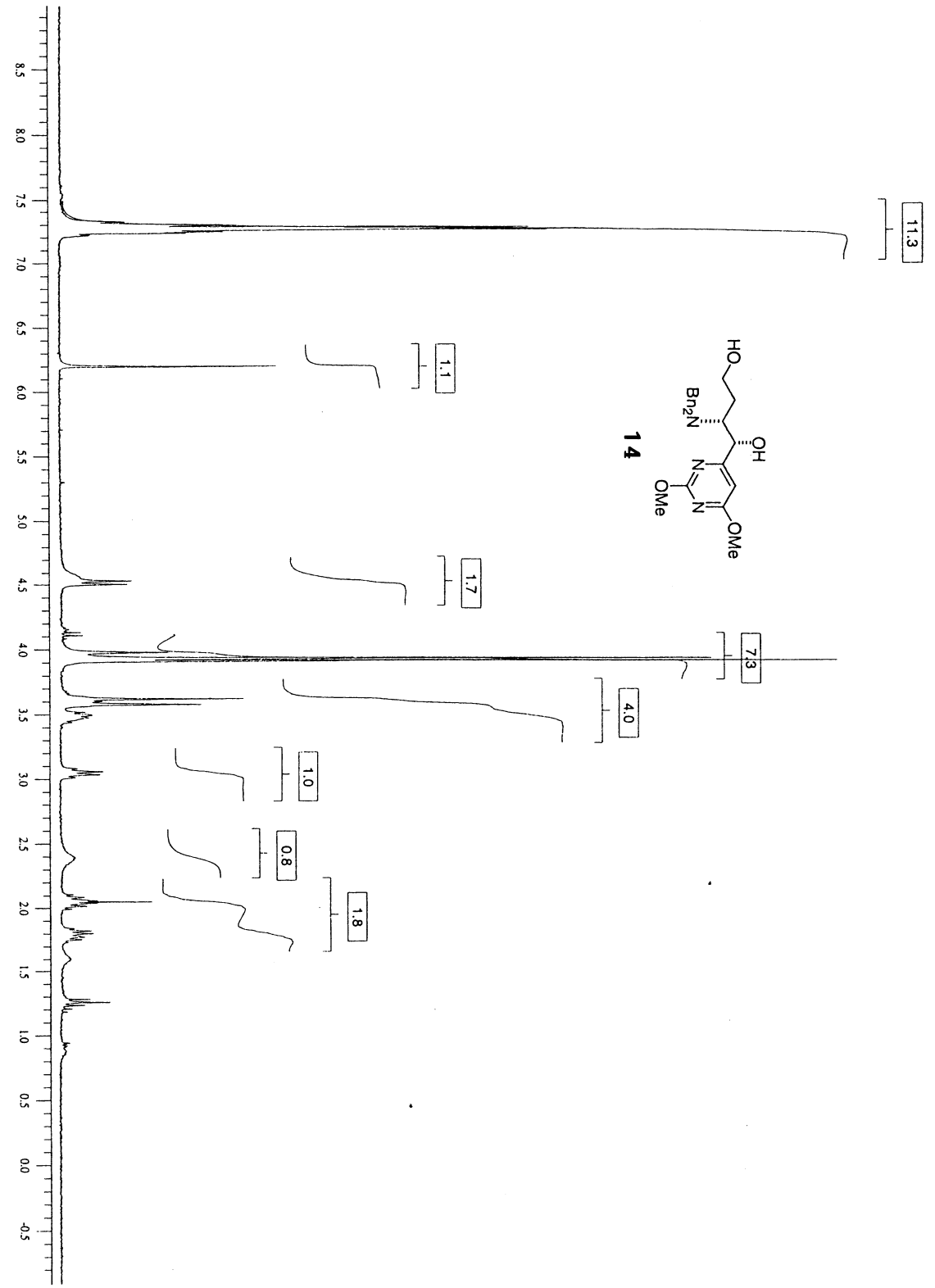
28.980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1.8
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Bn2N
OH
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N
OMe
OMe
14
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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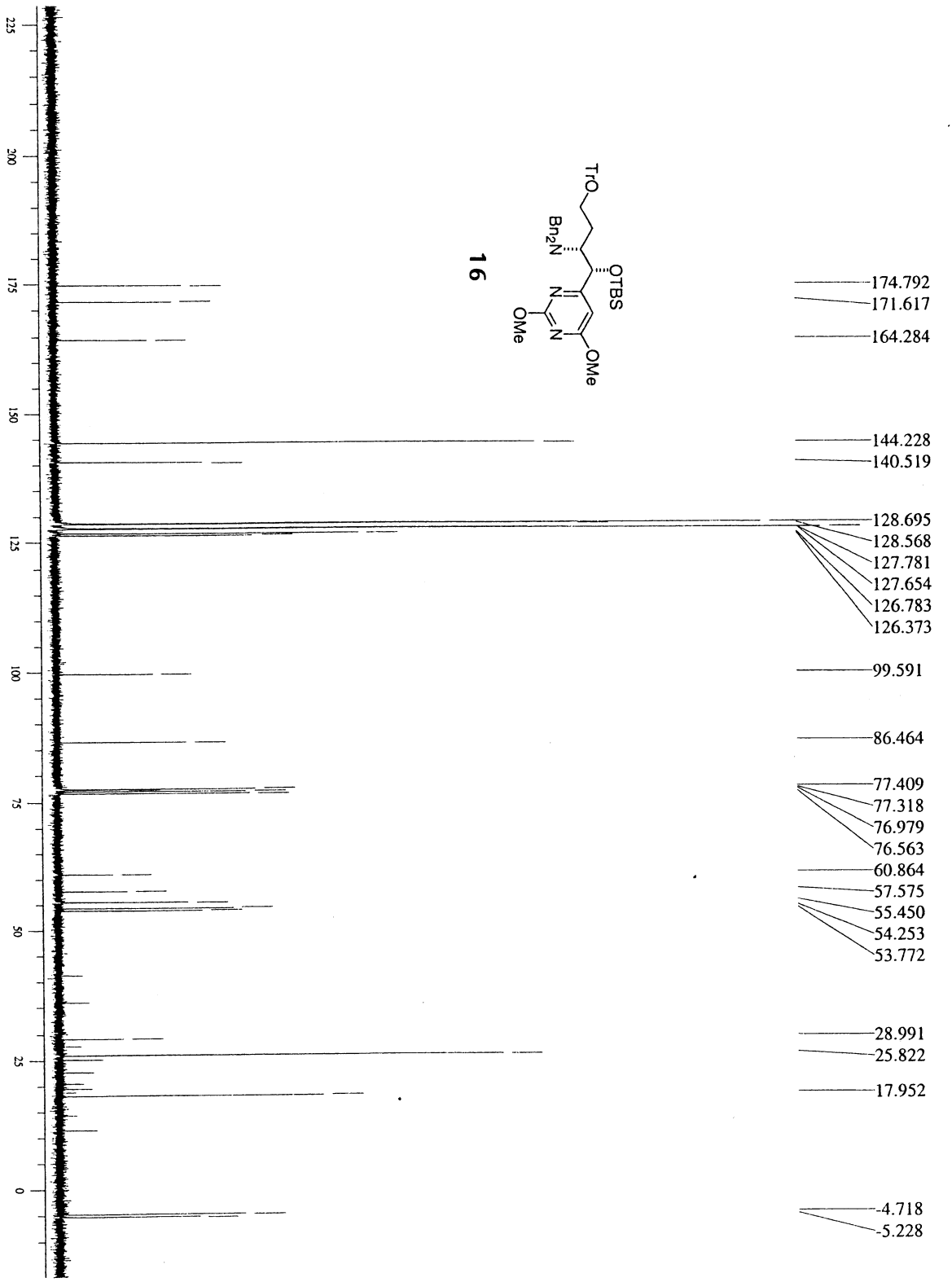
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Bn2N
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OMe
OMe
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127.781
127.654
126.783
126.373
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86.464
77.409
77.318
76.979
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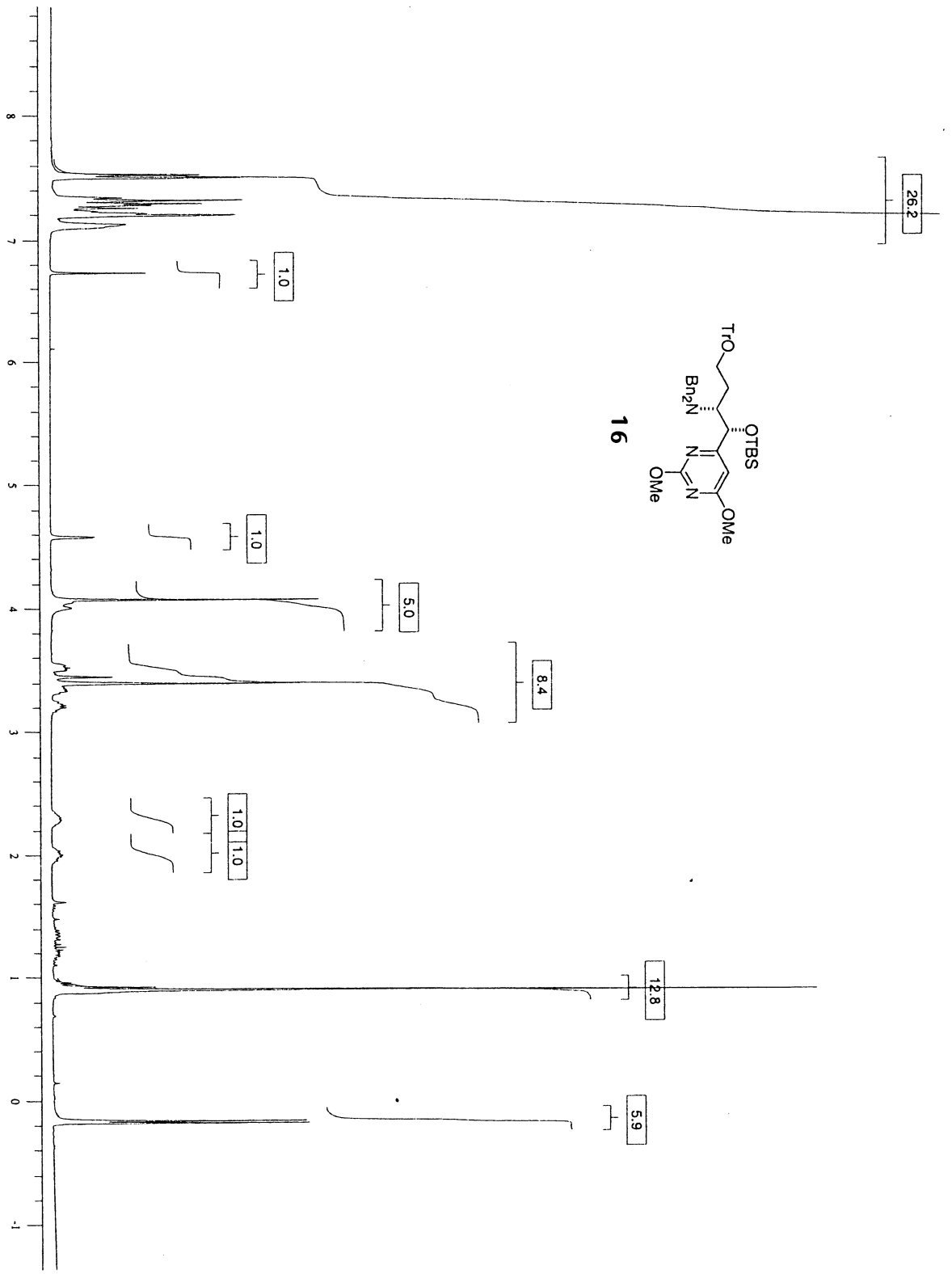
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Bn2N
OTBS
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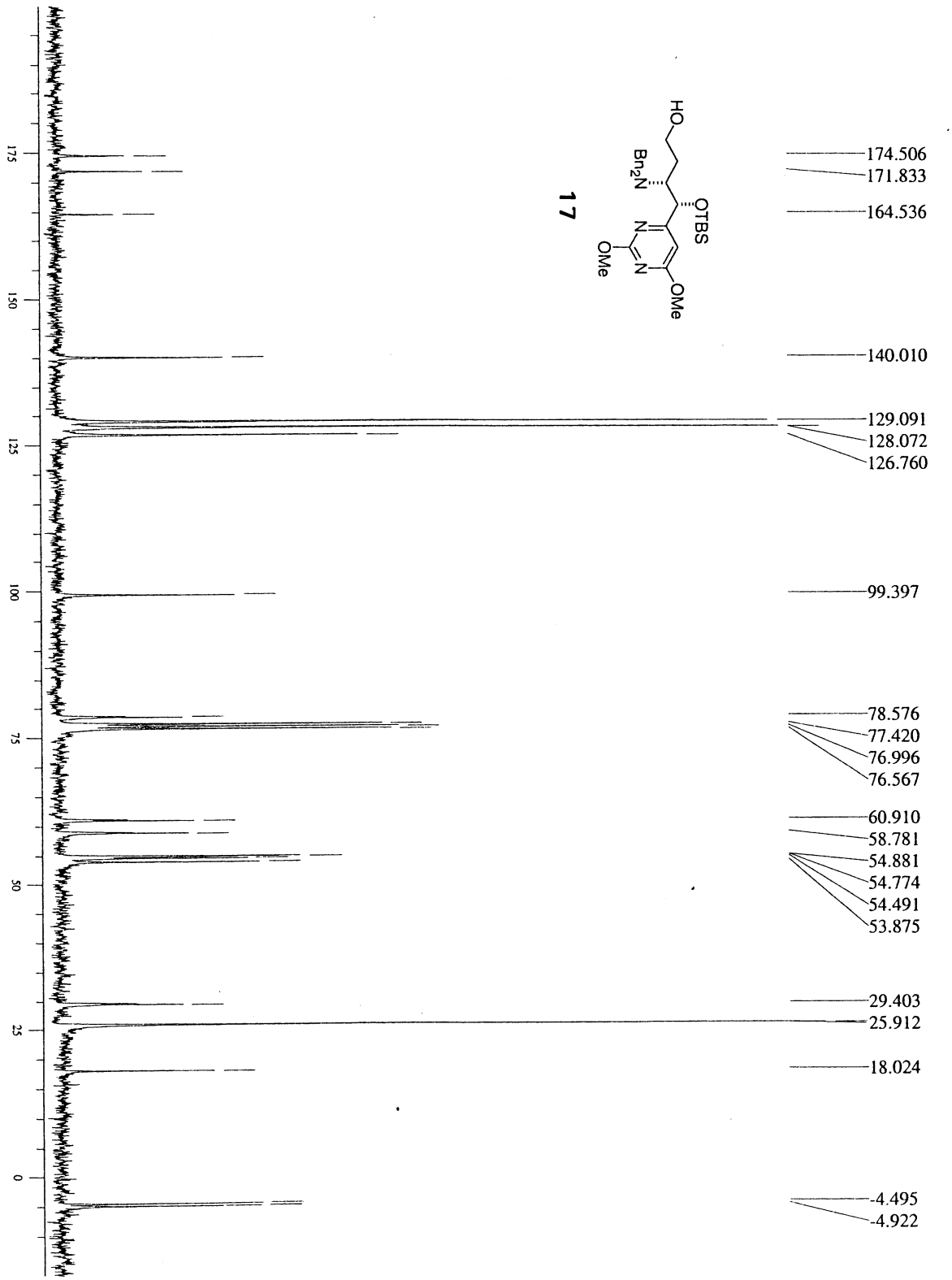
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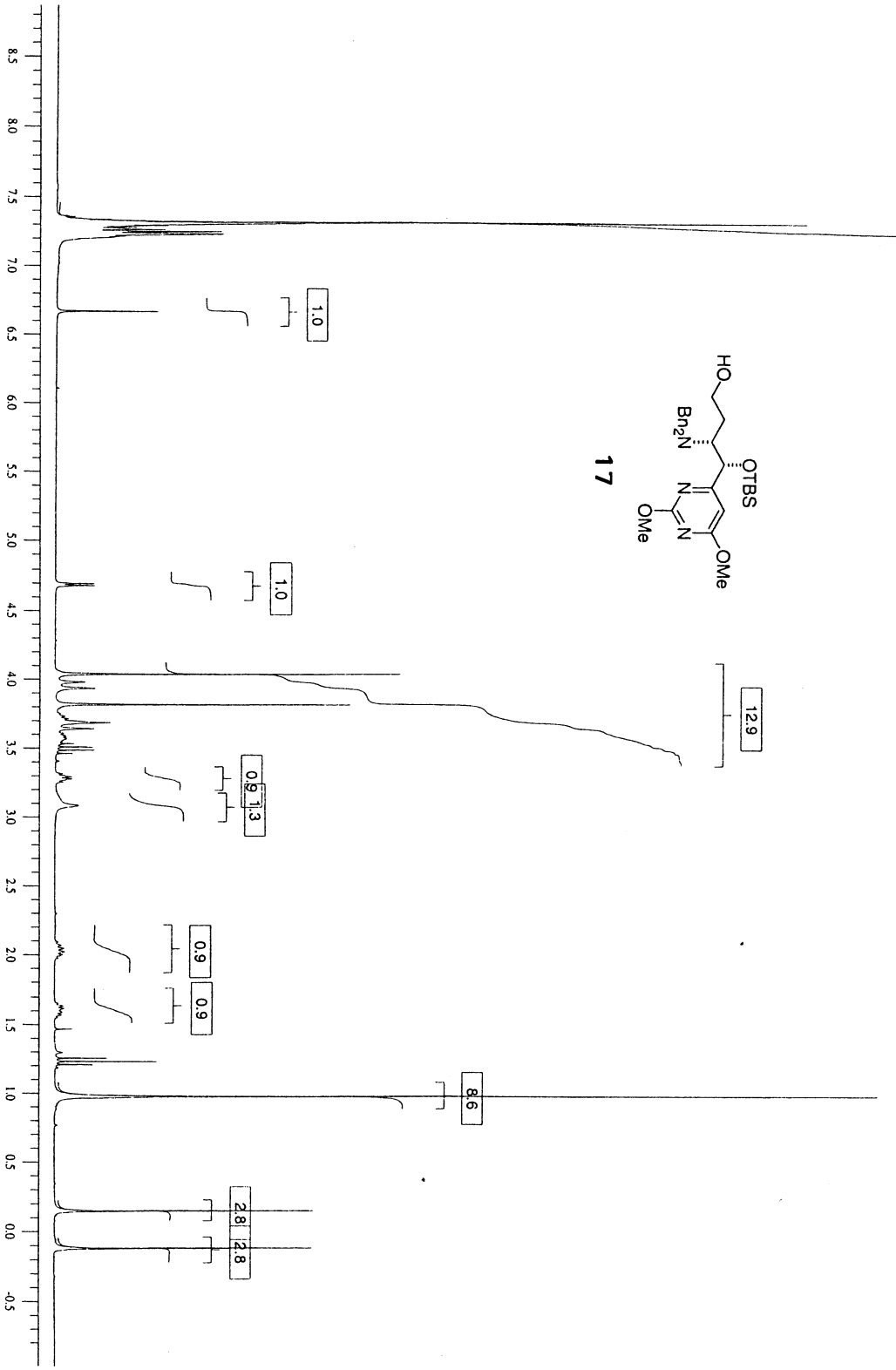
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OTBS
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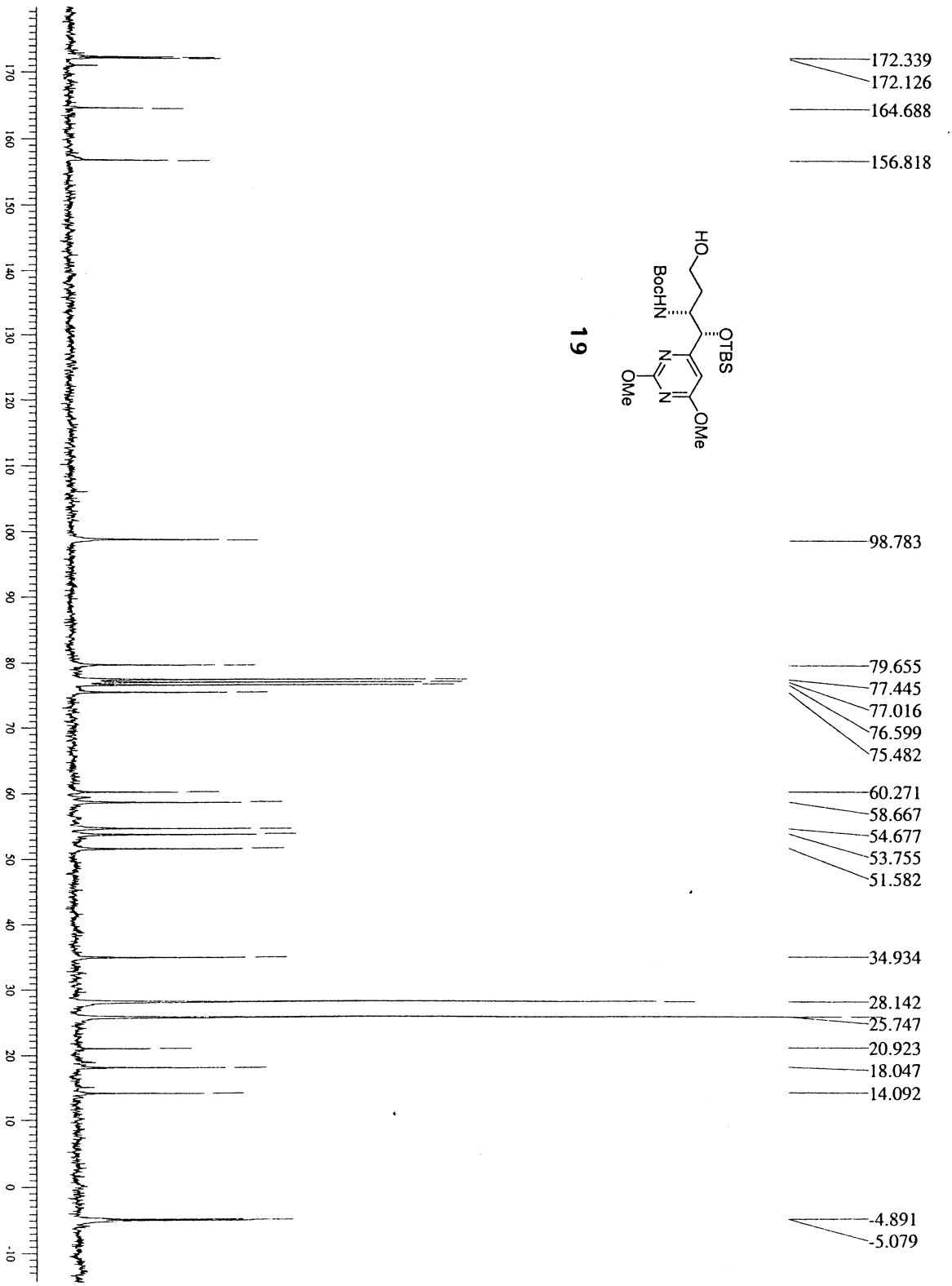
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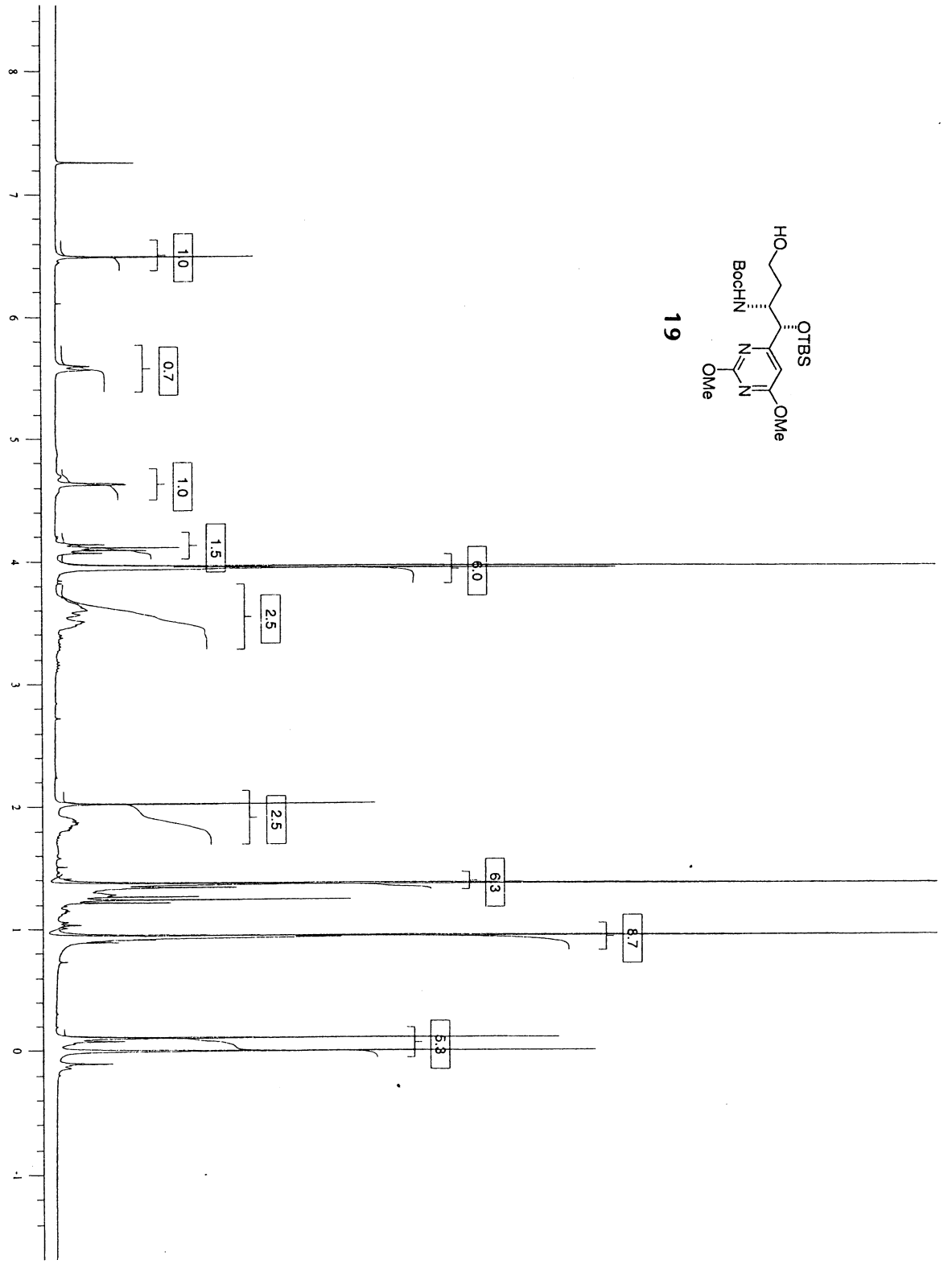
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OTBS
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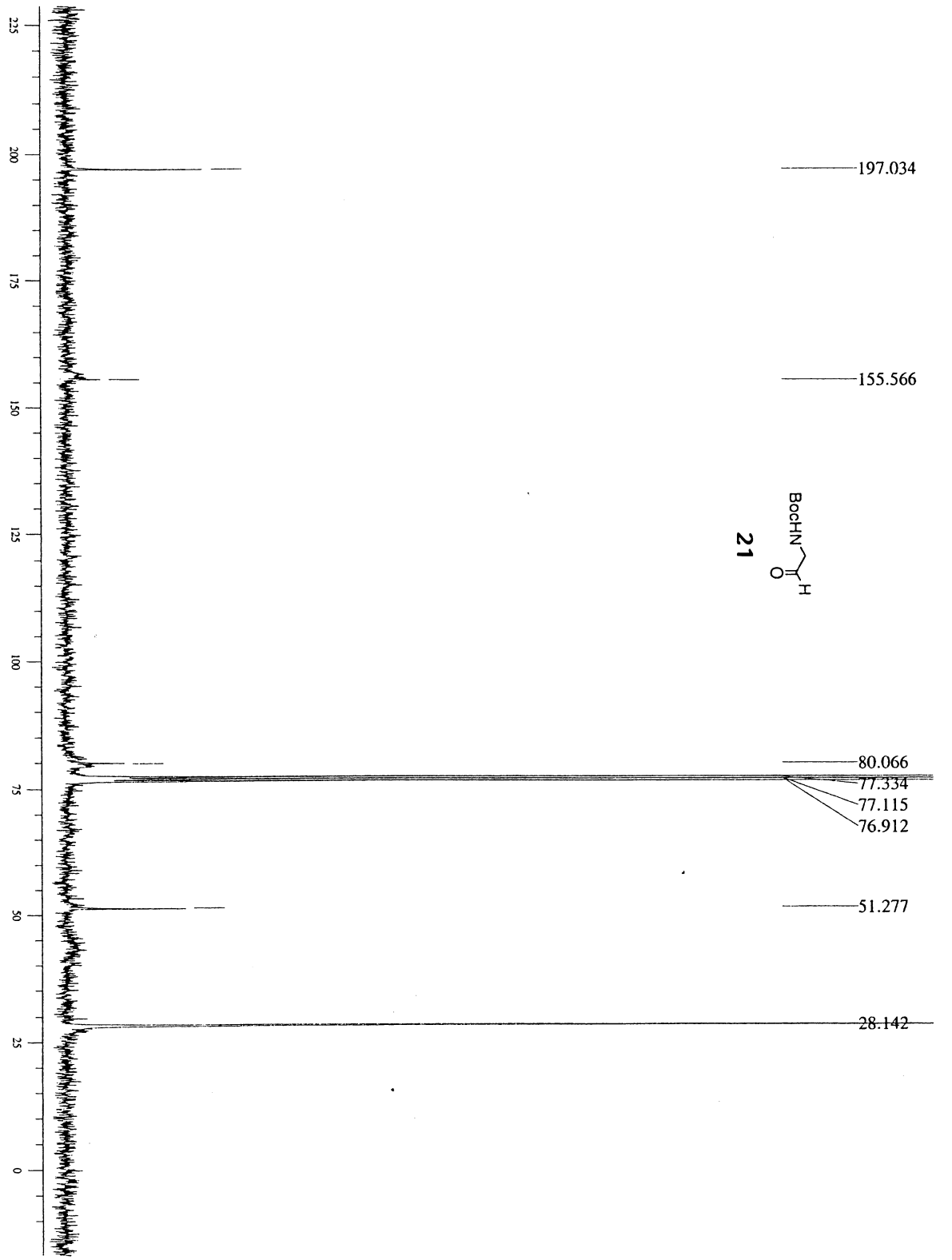
8.7
5.8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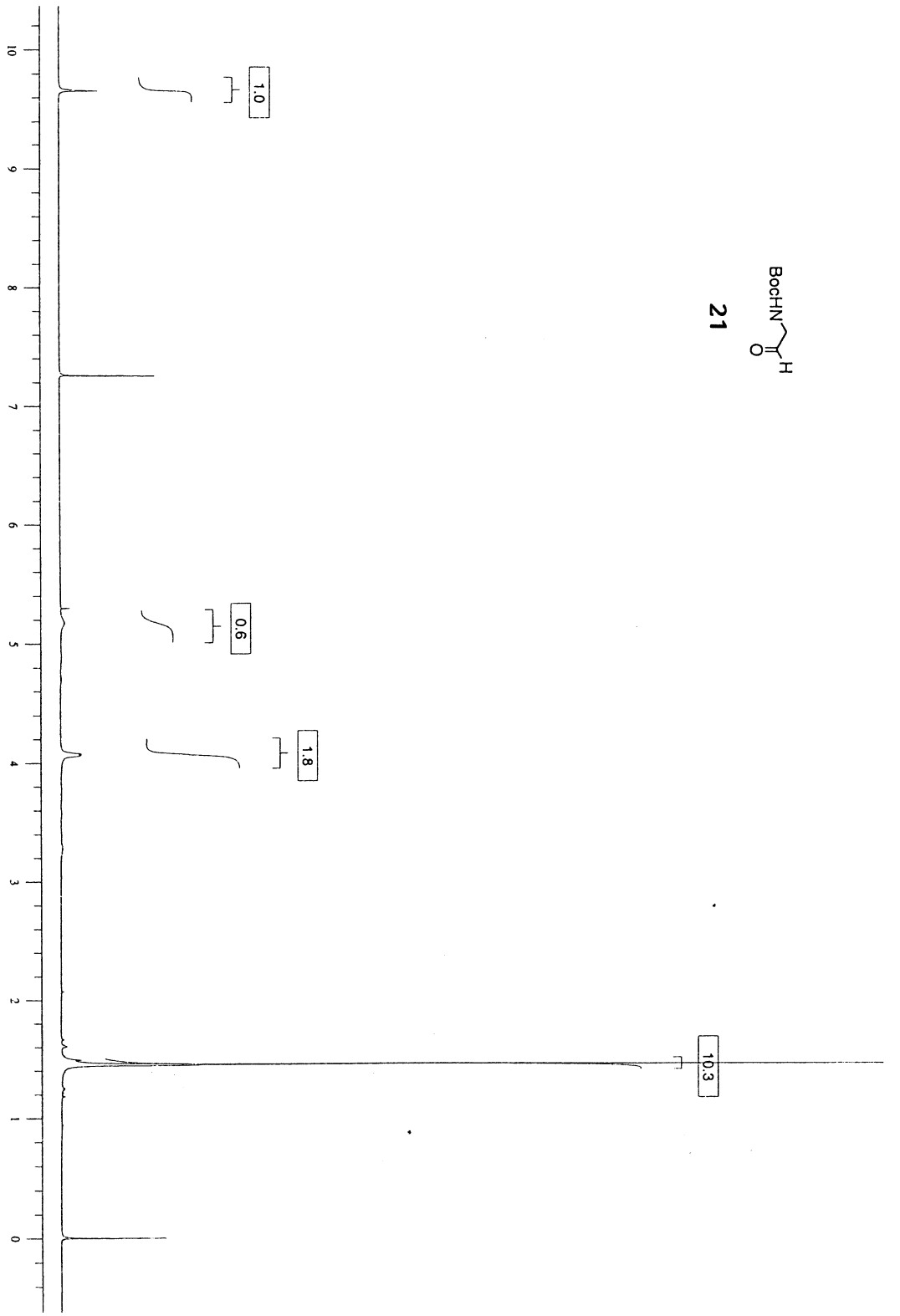
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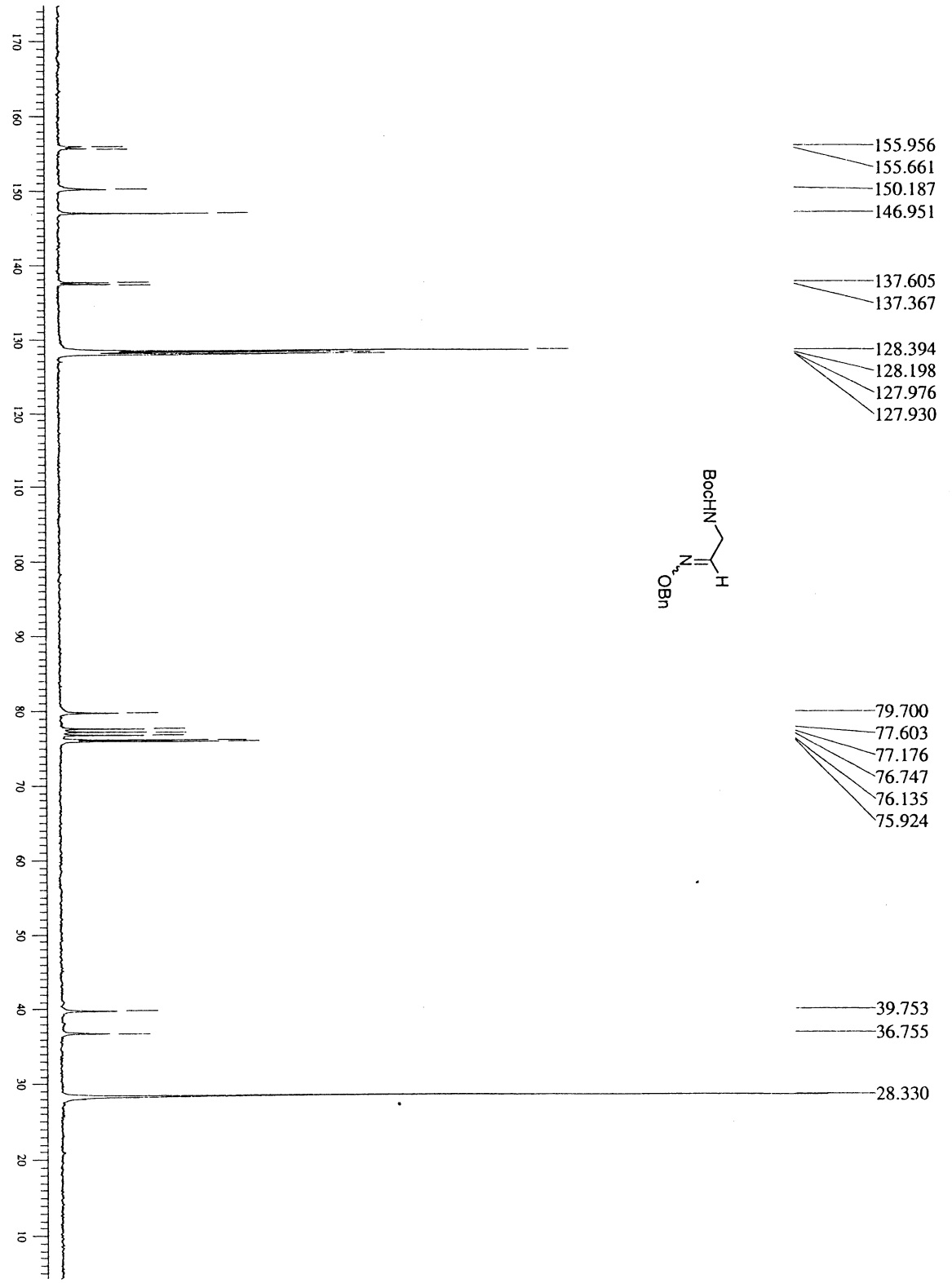
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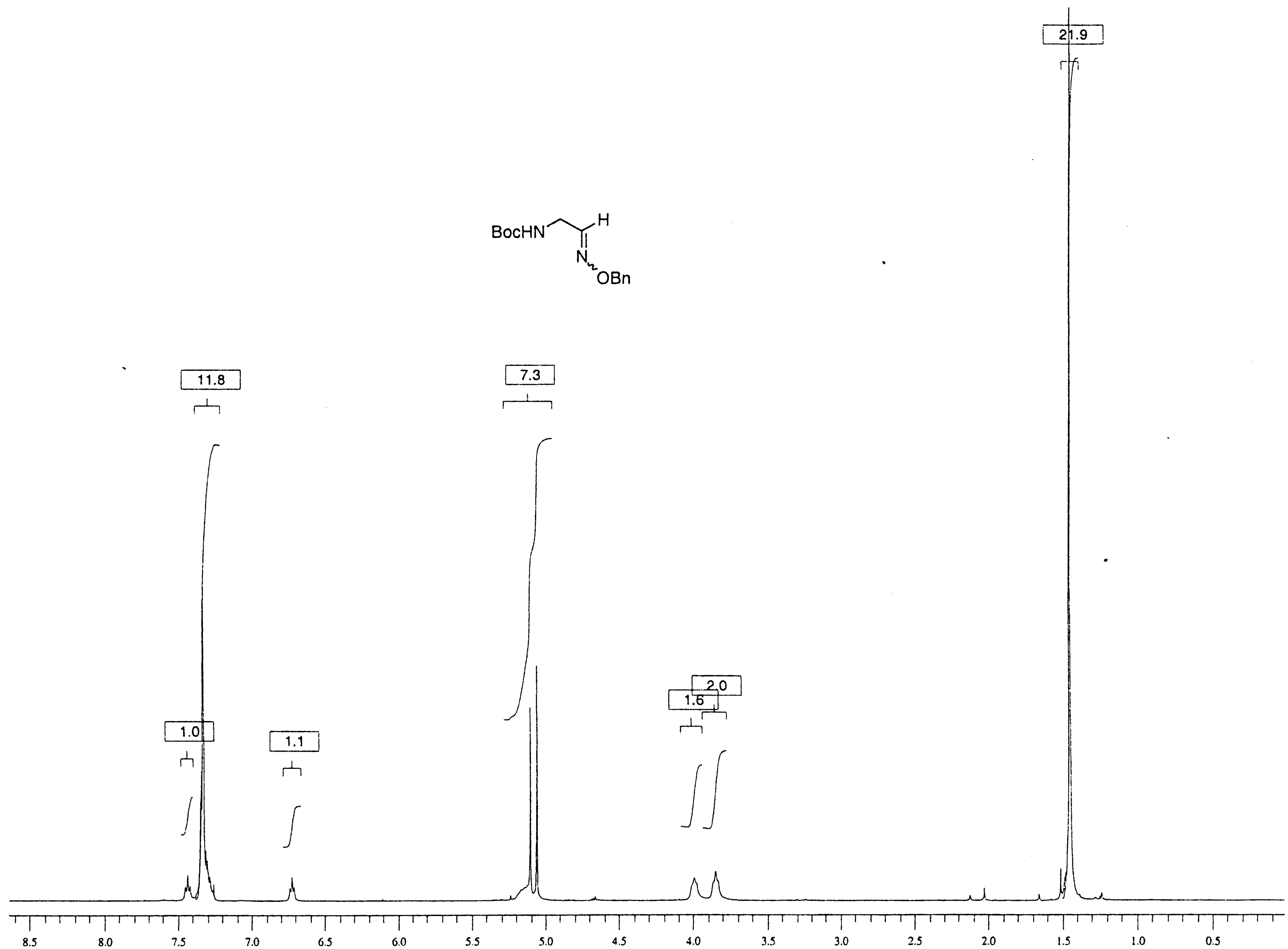
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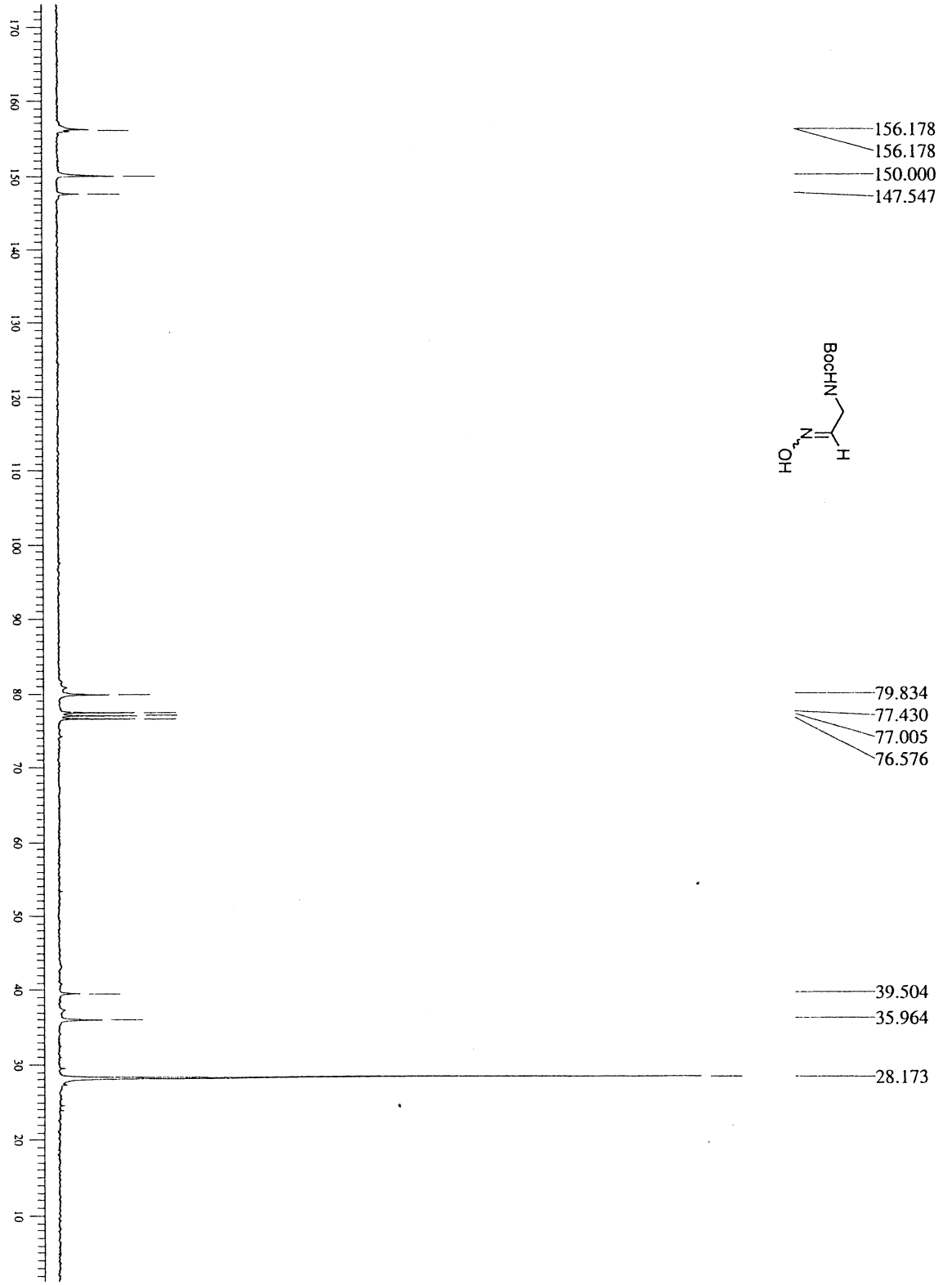
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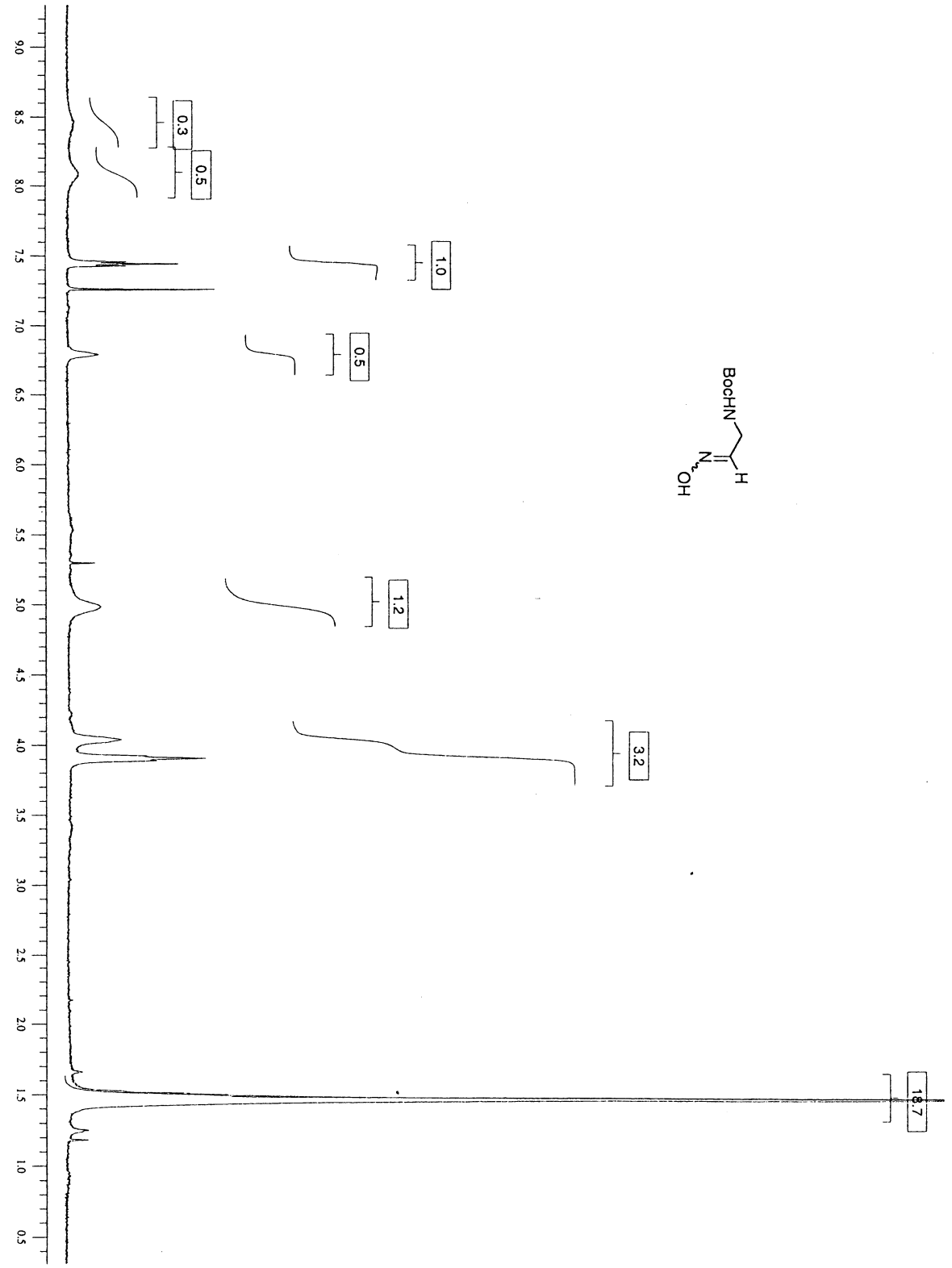
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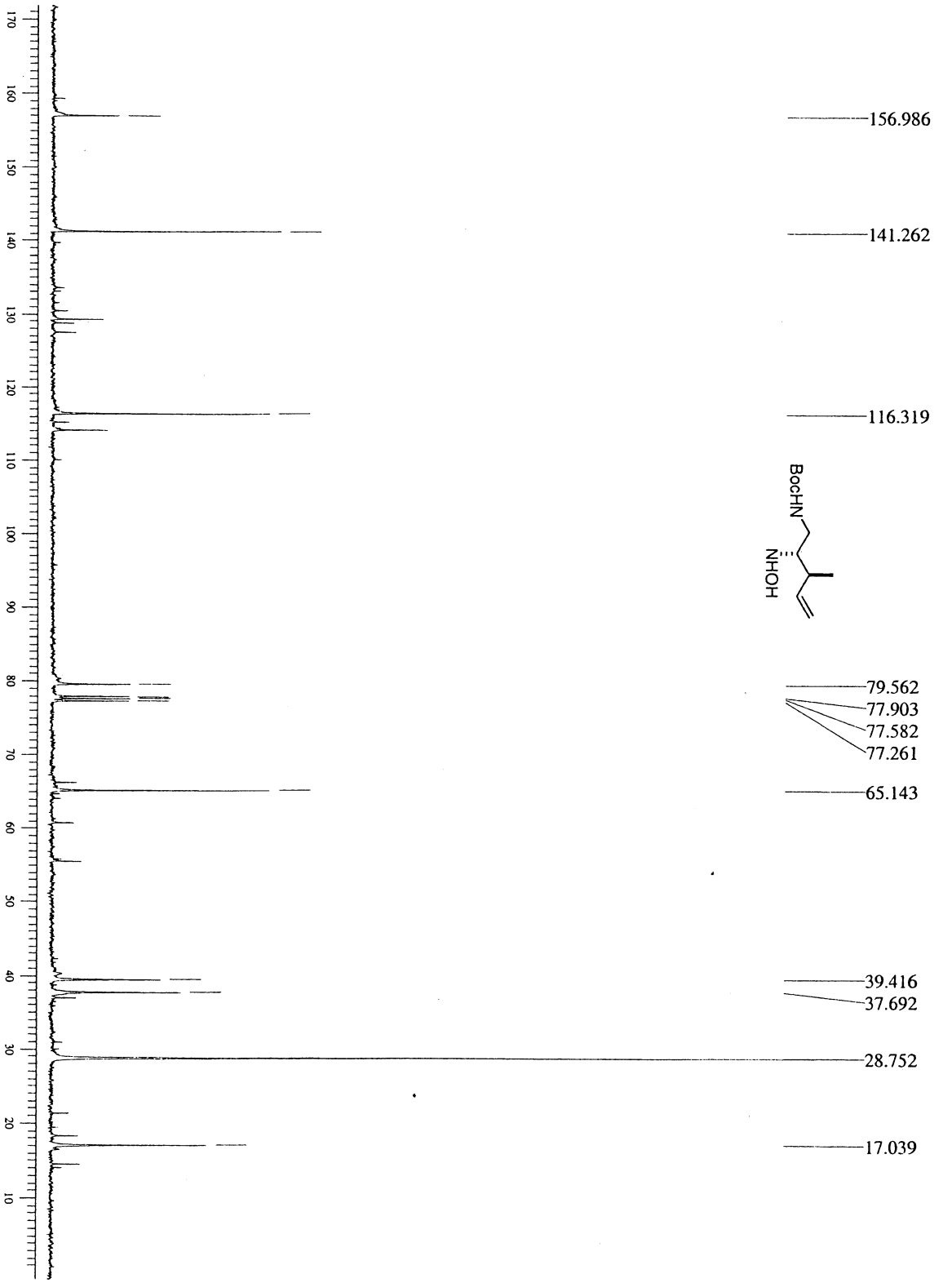
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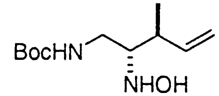
17.039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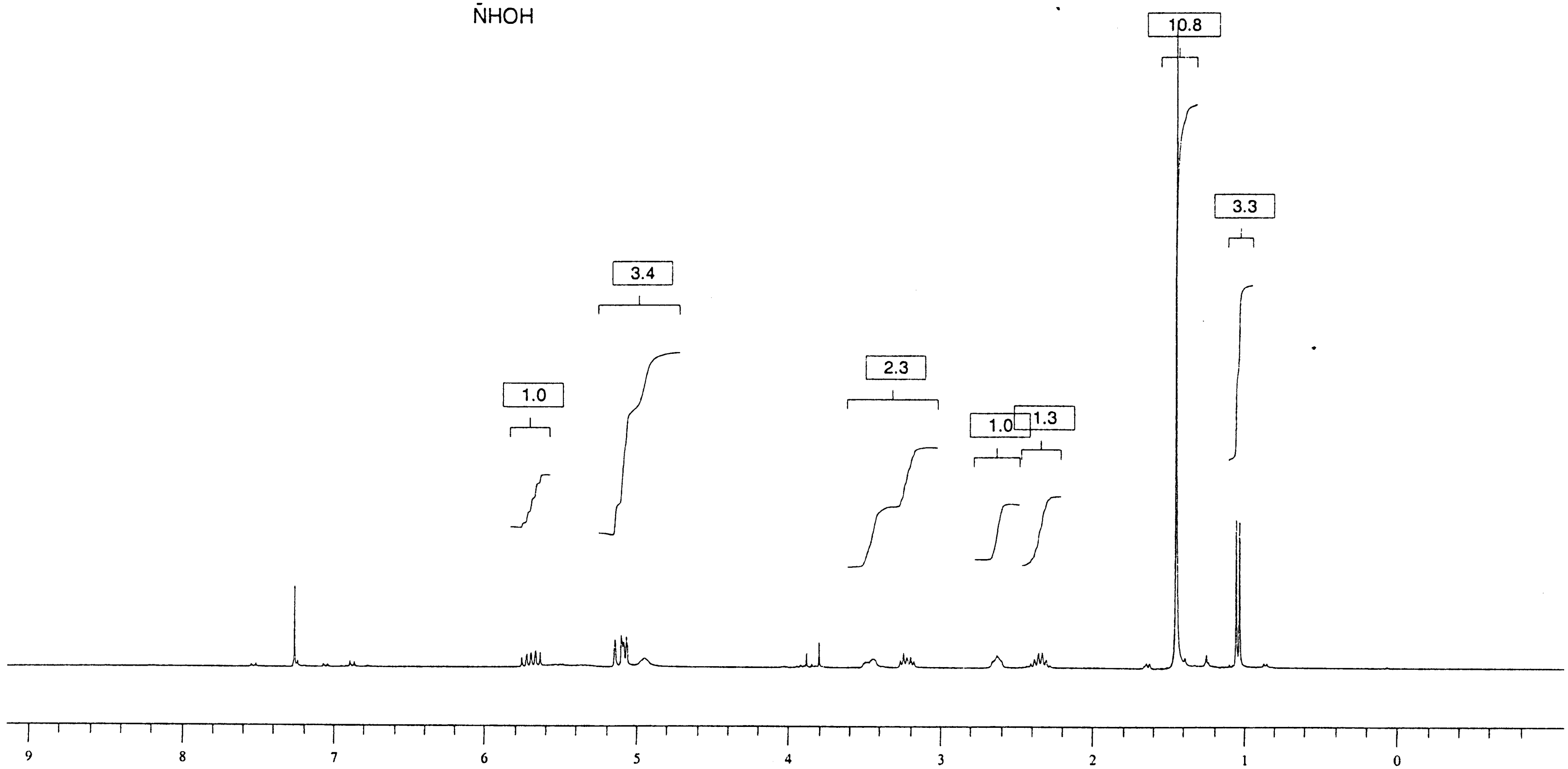
NHOH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1.0
1.3
9
8
7
6
5
4
3
2
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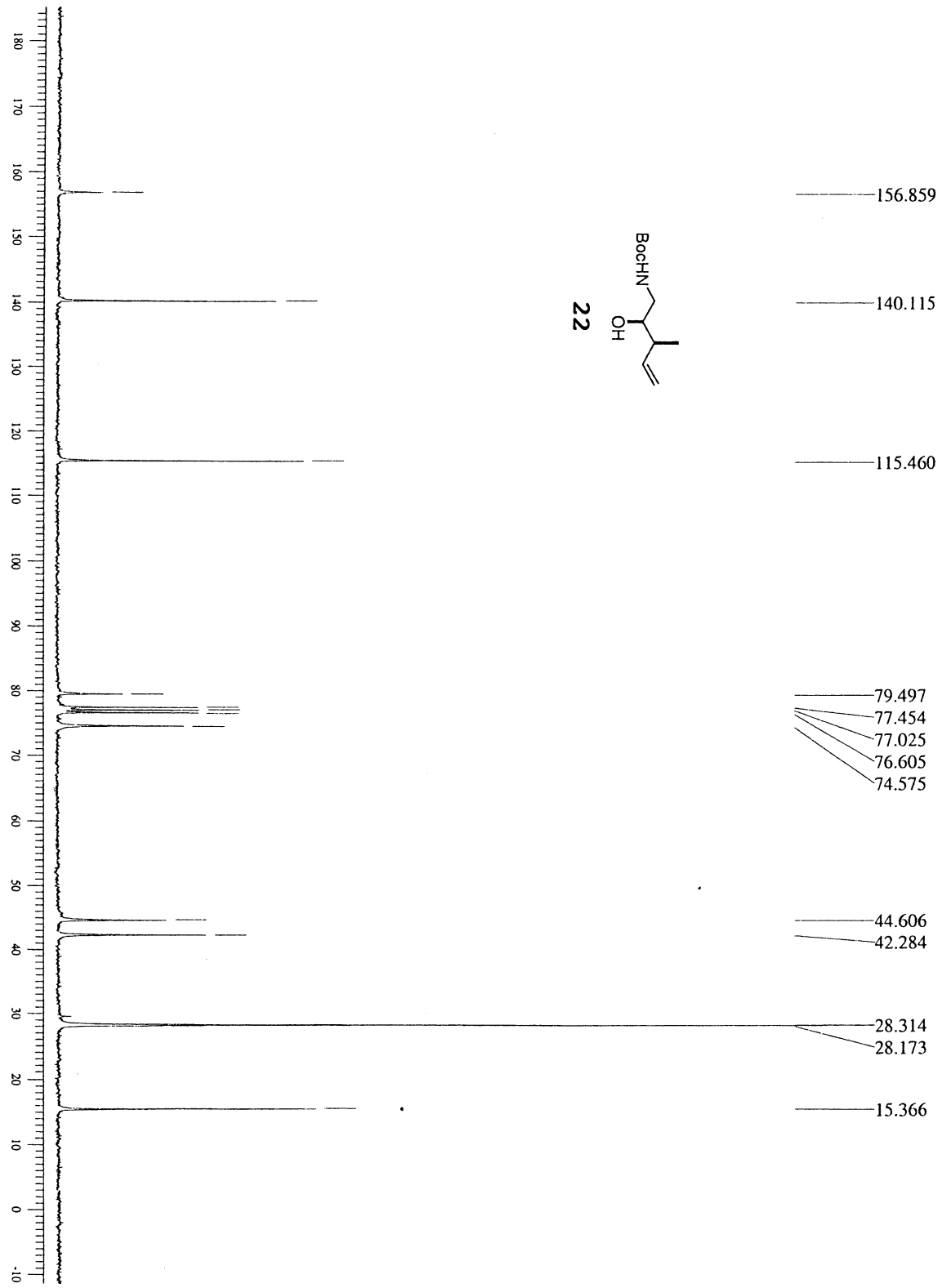
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115.460
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74.575
44.606
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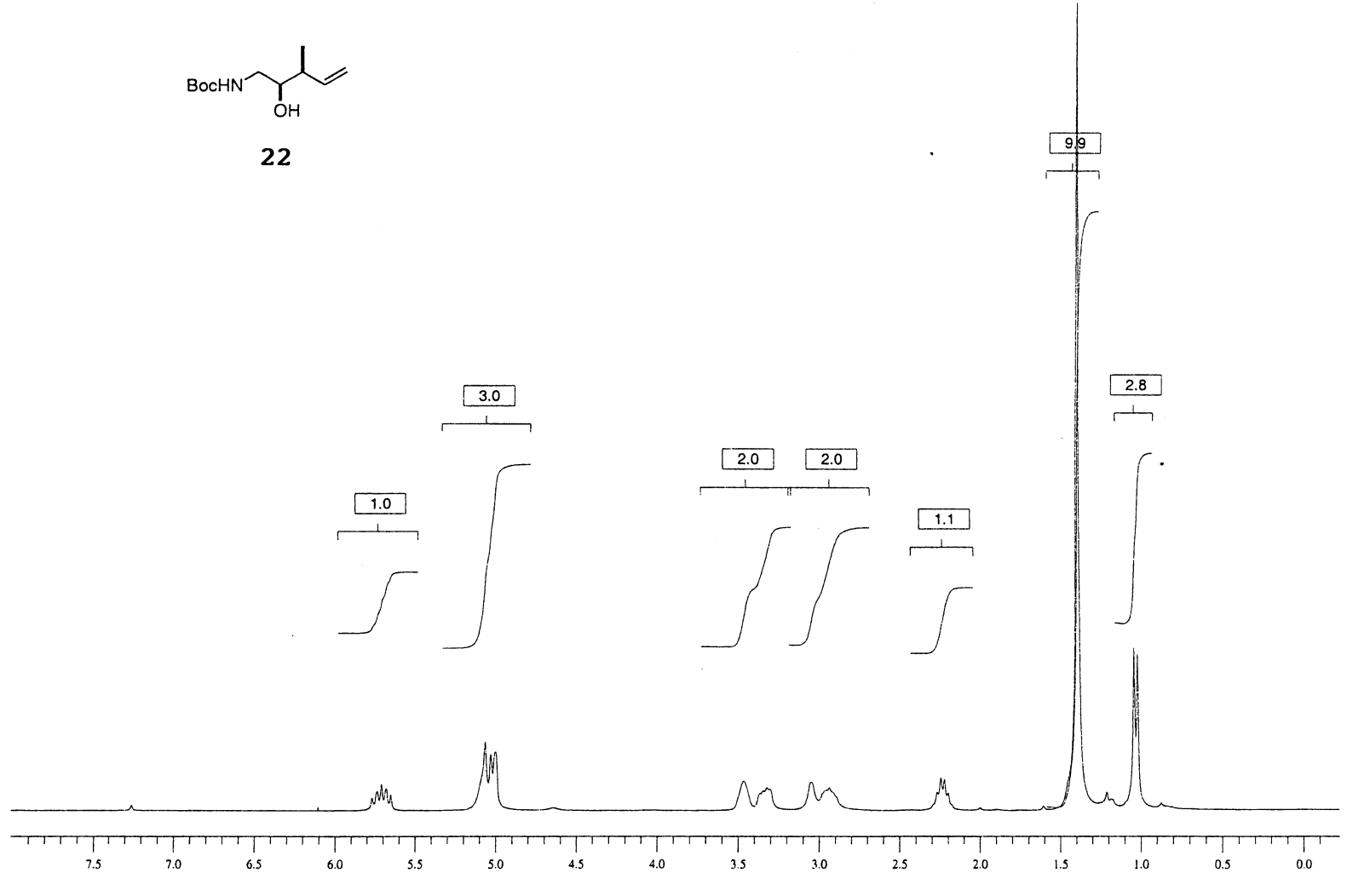
28.314
28.173
15.366
BocHN
OH
22


BocHN
OH
22
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3.0
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2.0
1.1
9.9
2.8
7.5
7.0
6.5
6.0
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4.0
3.5
3.0
2.5
2.0
1.5
1.0
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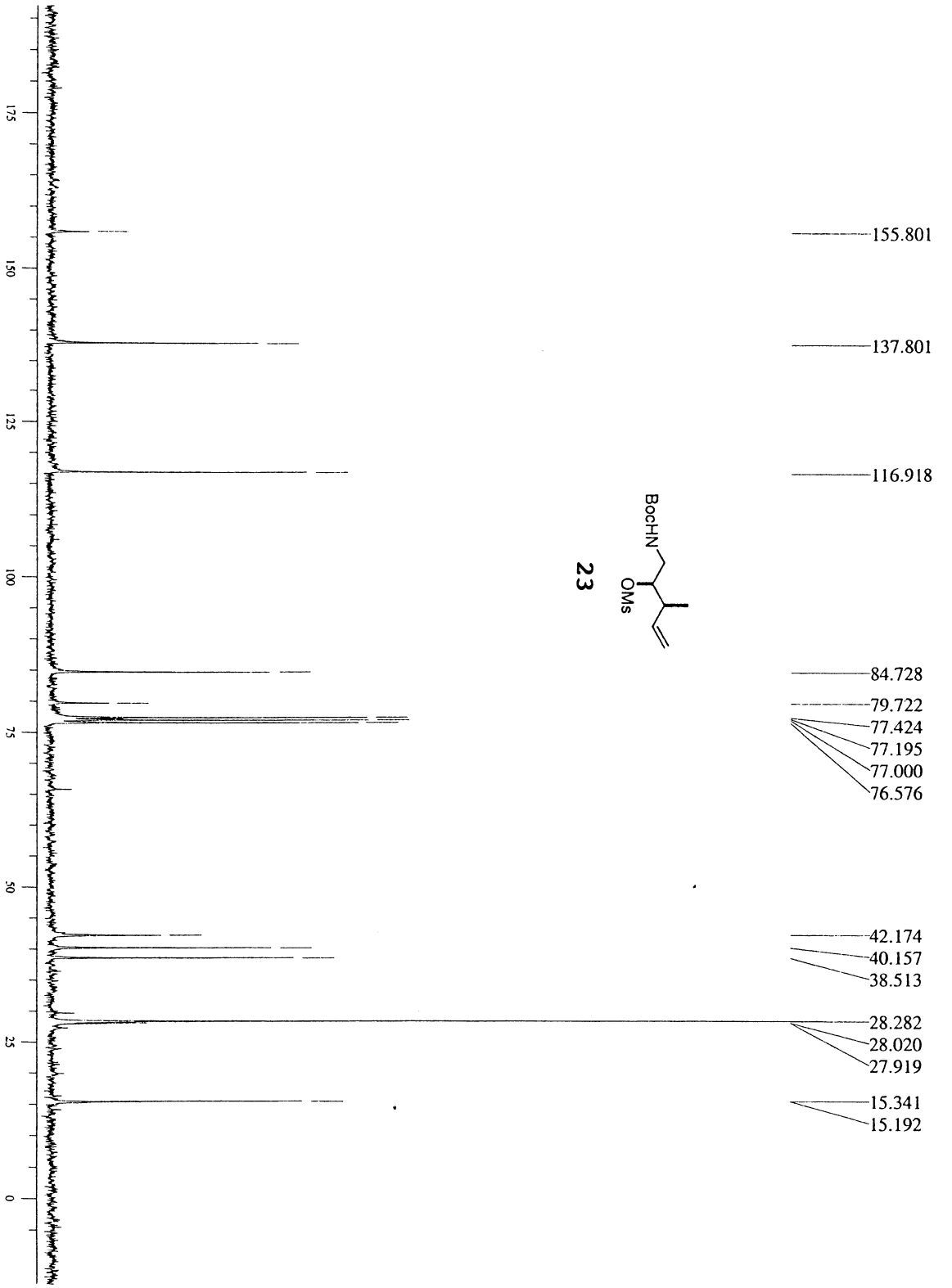
0.0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77.000
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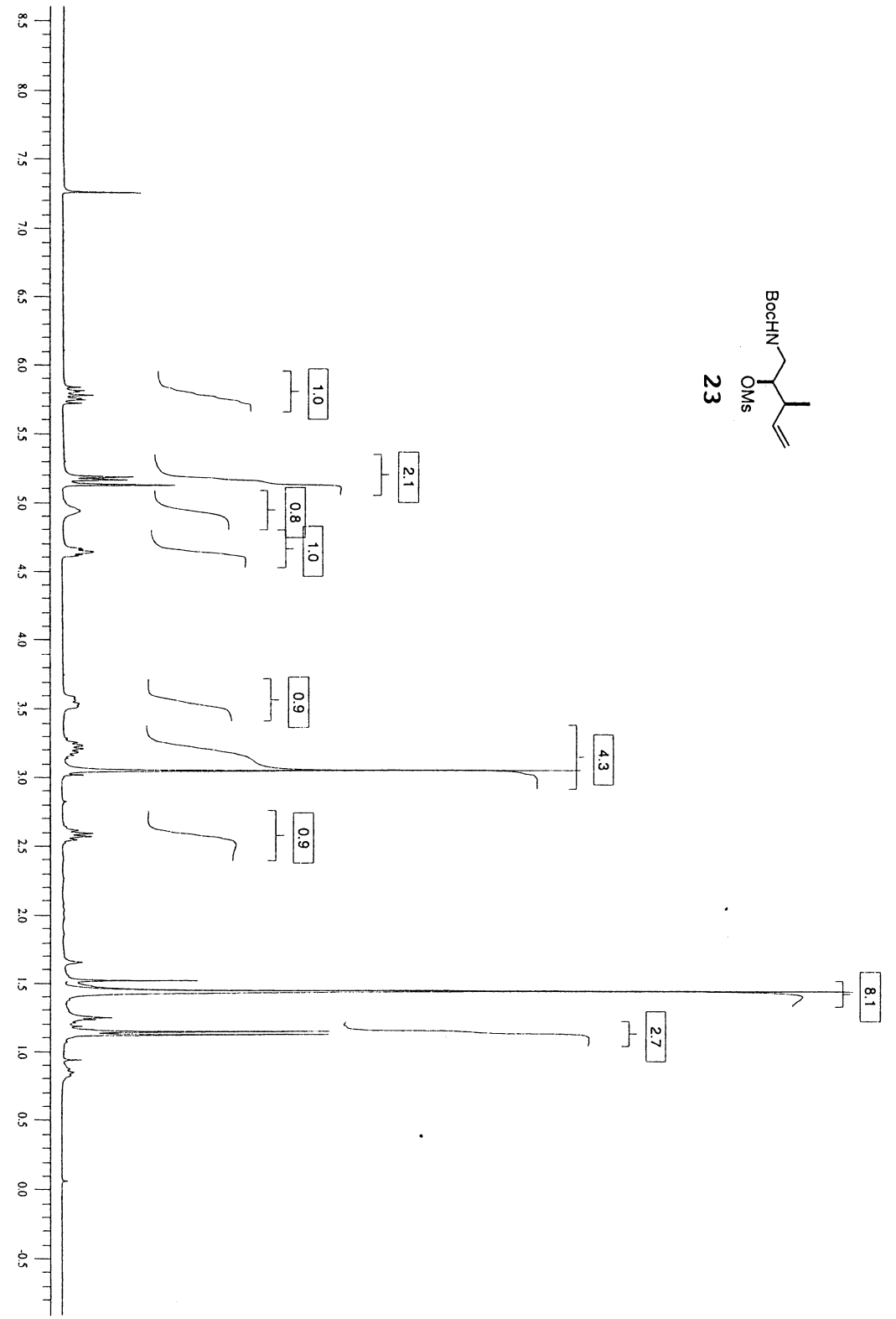
28.282
28.020
27.919
15.341
15.192
BocHN
OMs
23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OMs
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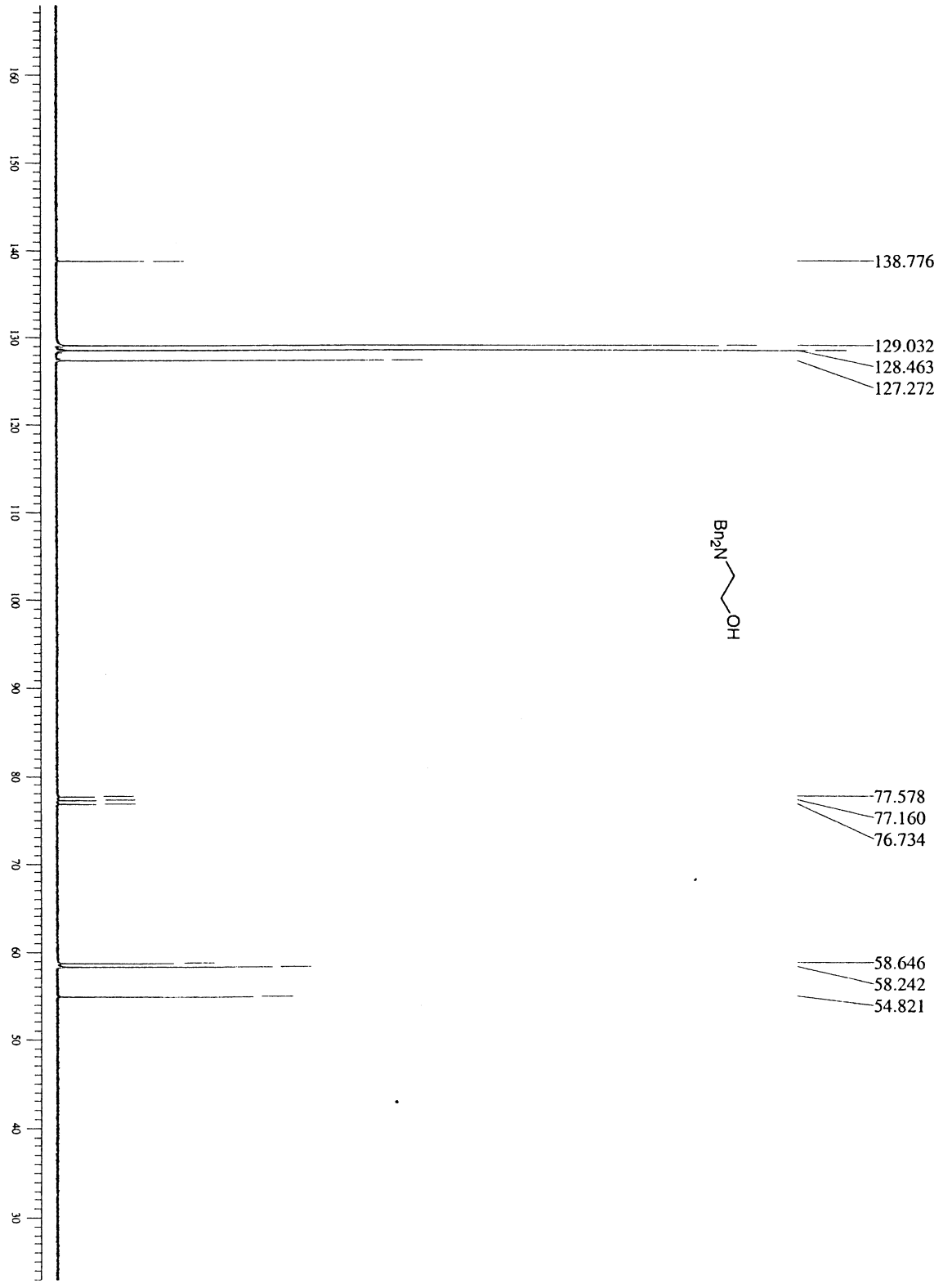
2.1
0.8
1.0
1.0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OH
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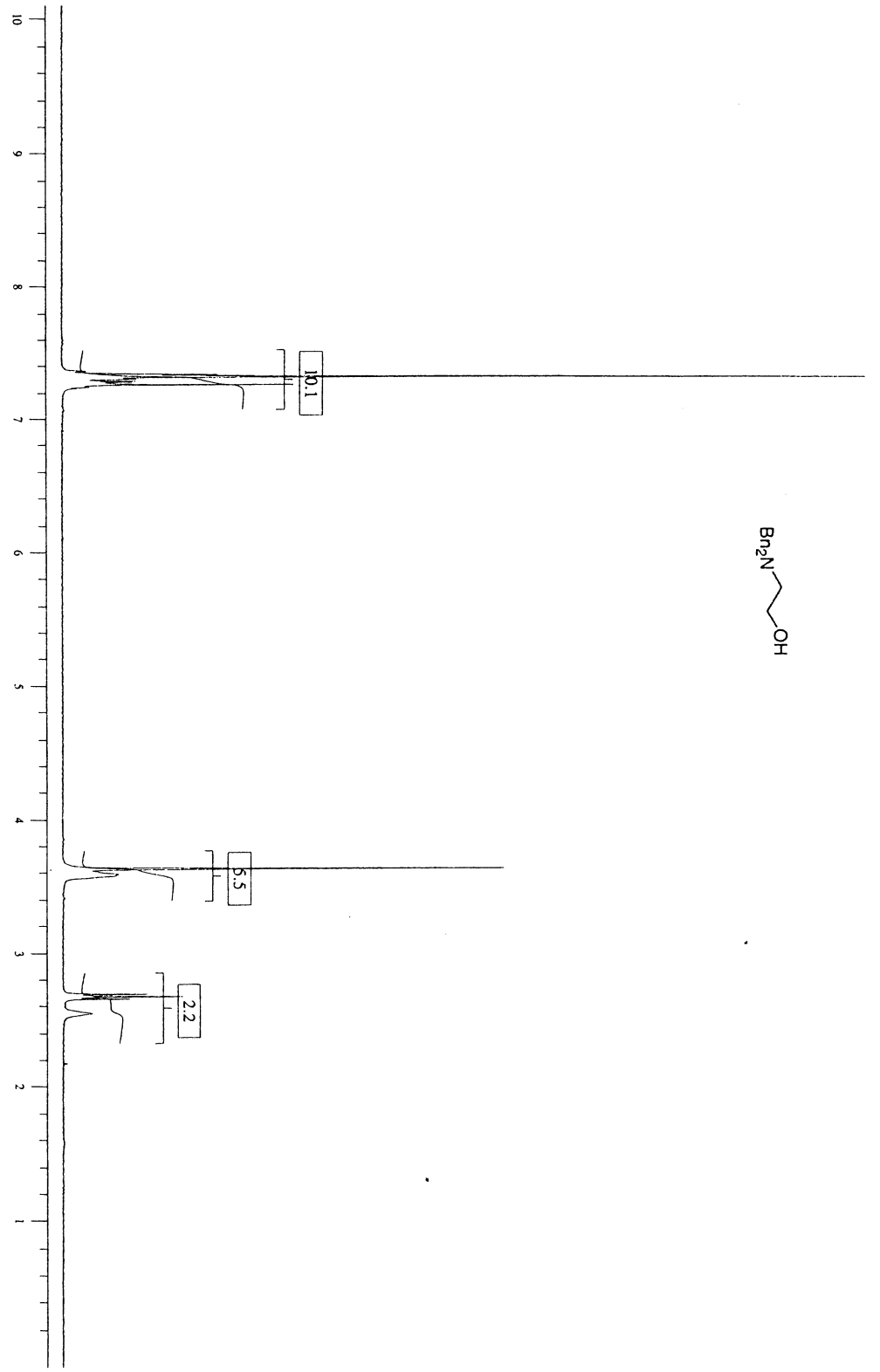
77.160
76.734
58.646
58.242
54.821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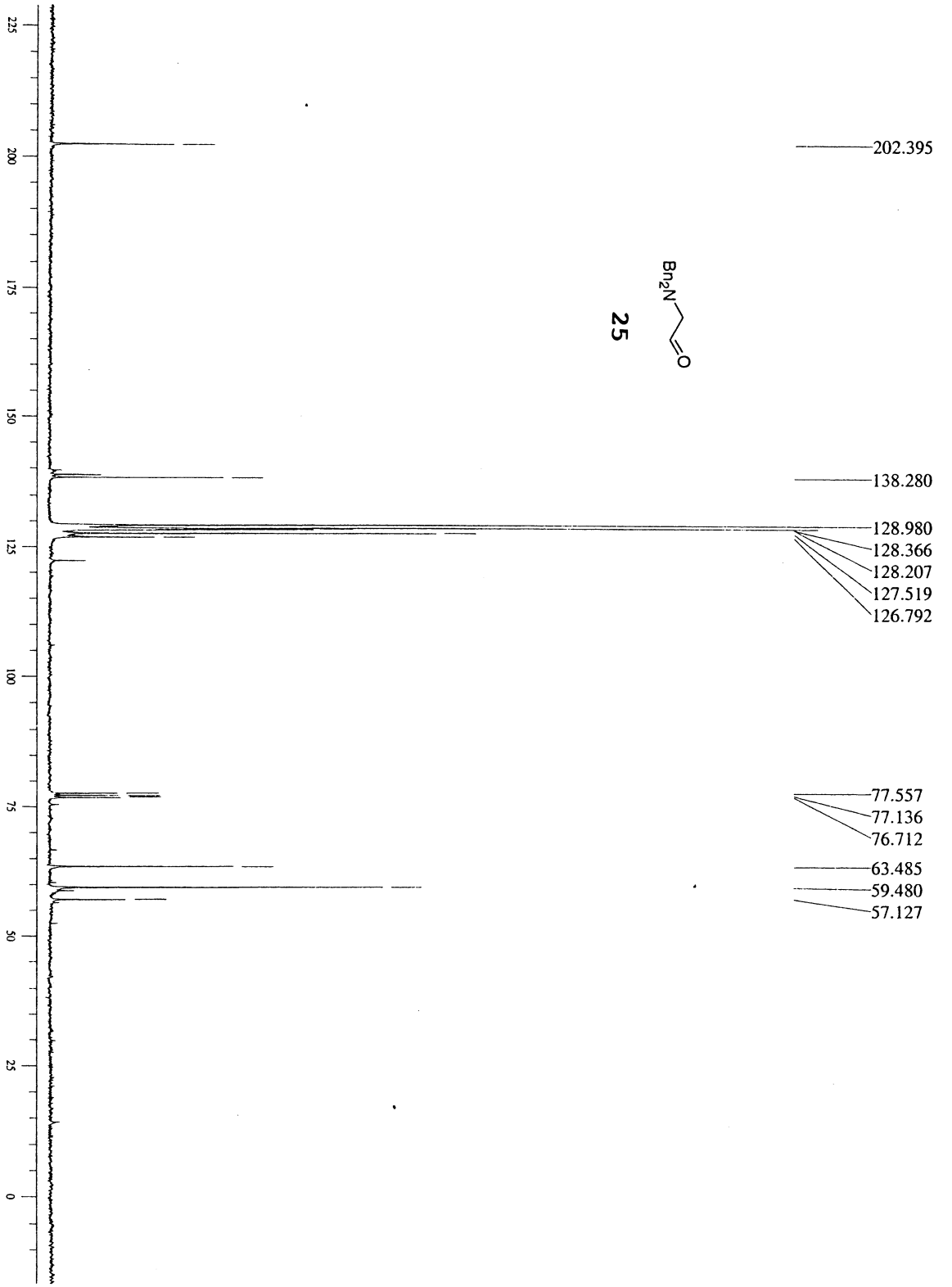
5.5
2.2
Bn2N
OH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200
175
150
125
100
75
50
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126.792
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77.136
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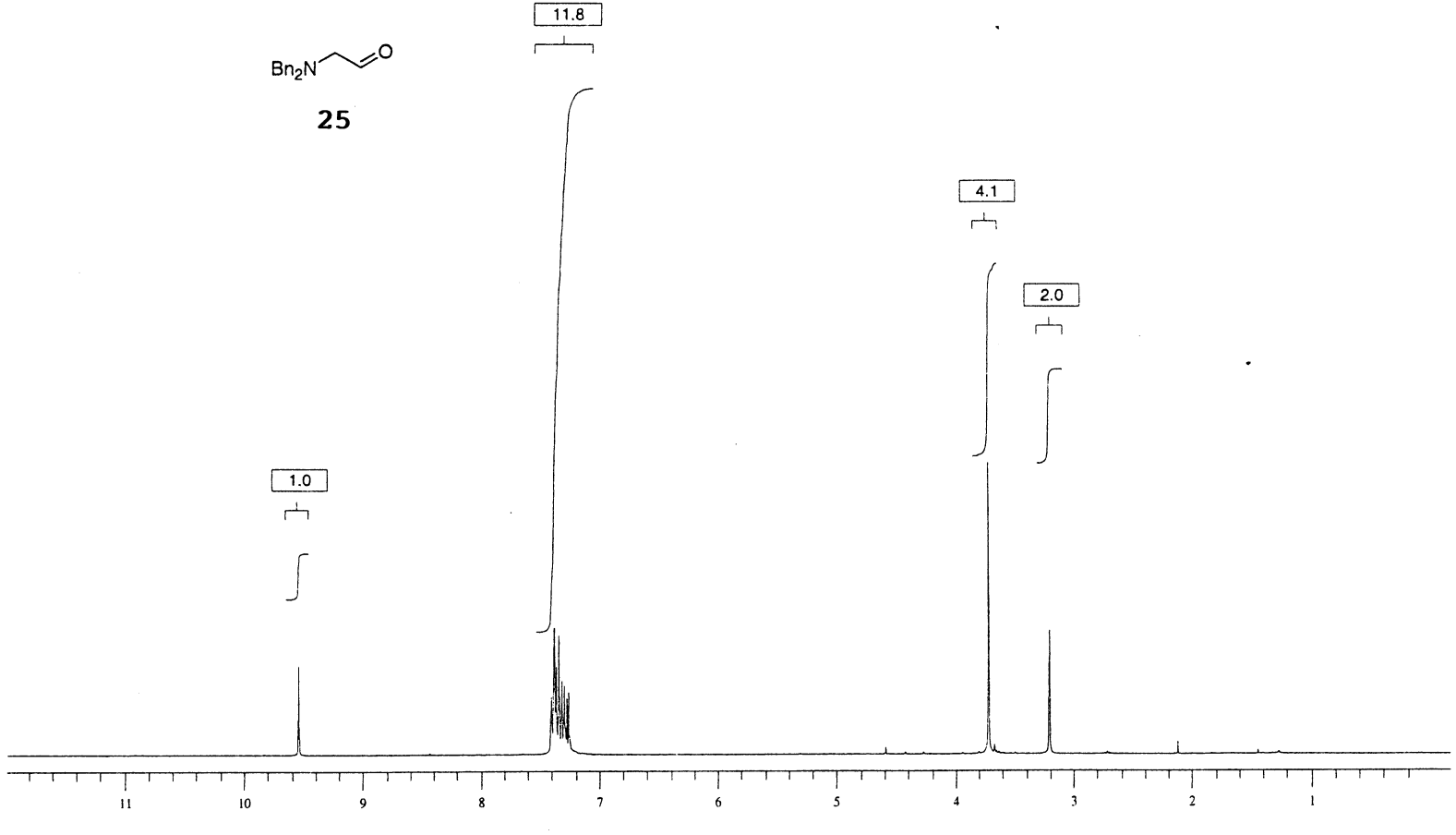
59.480
57.127
Bn2N
O
25


Bn2N
O
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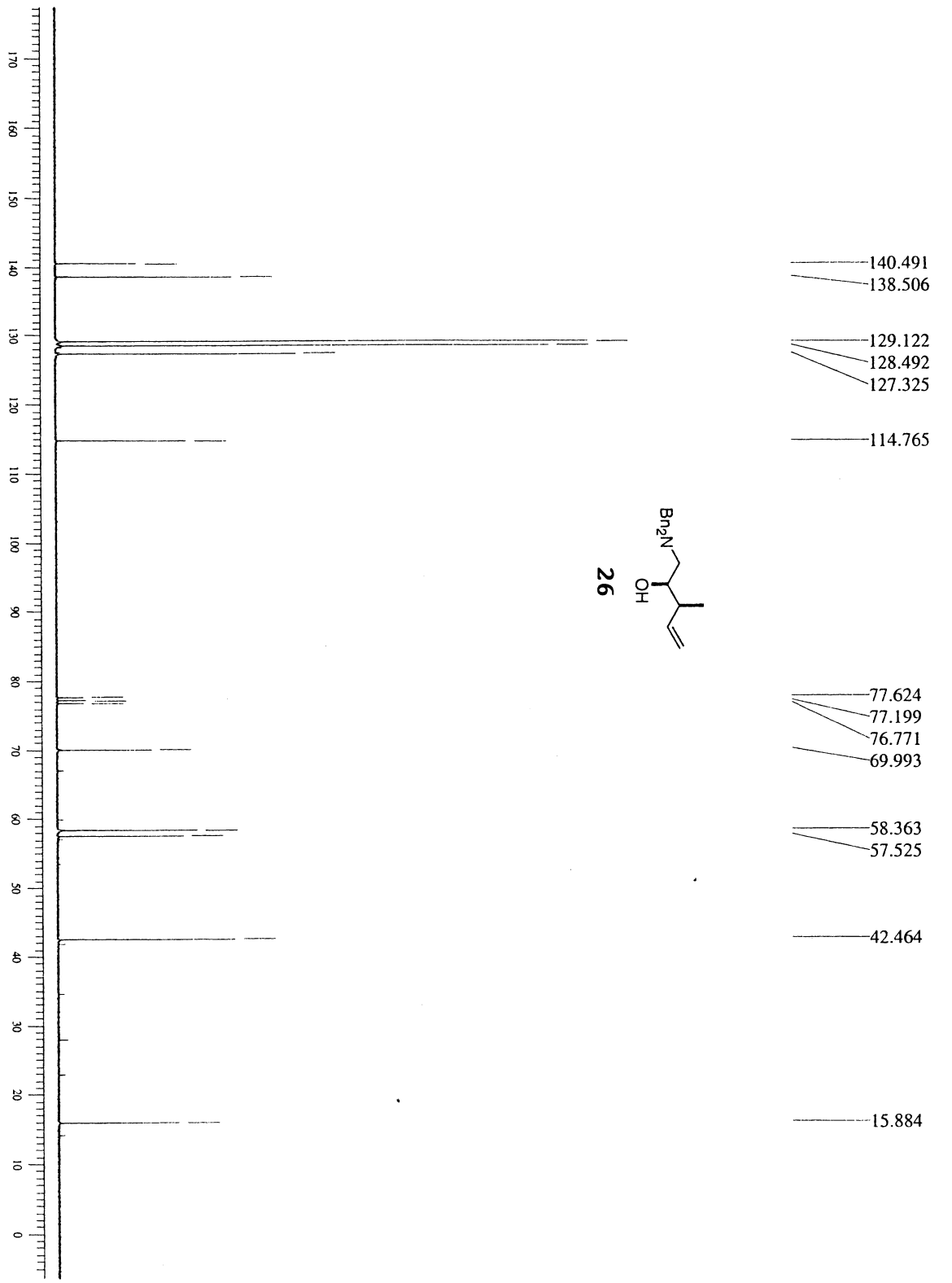
4.1
2.0
1.0
11
10
9
8
7
6
5
4
3
2
1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138.506
129.122
128.492
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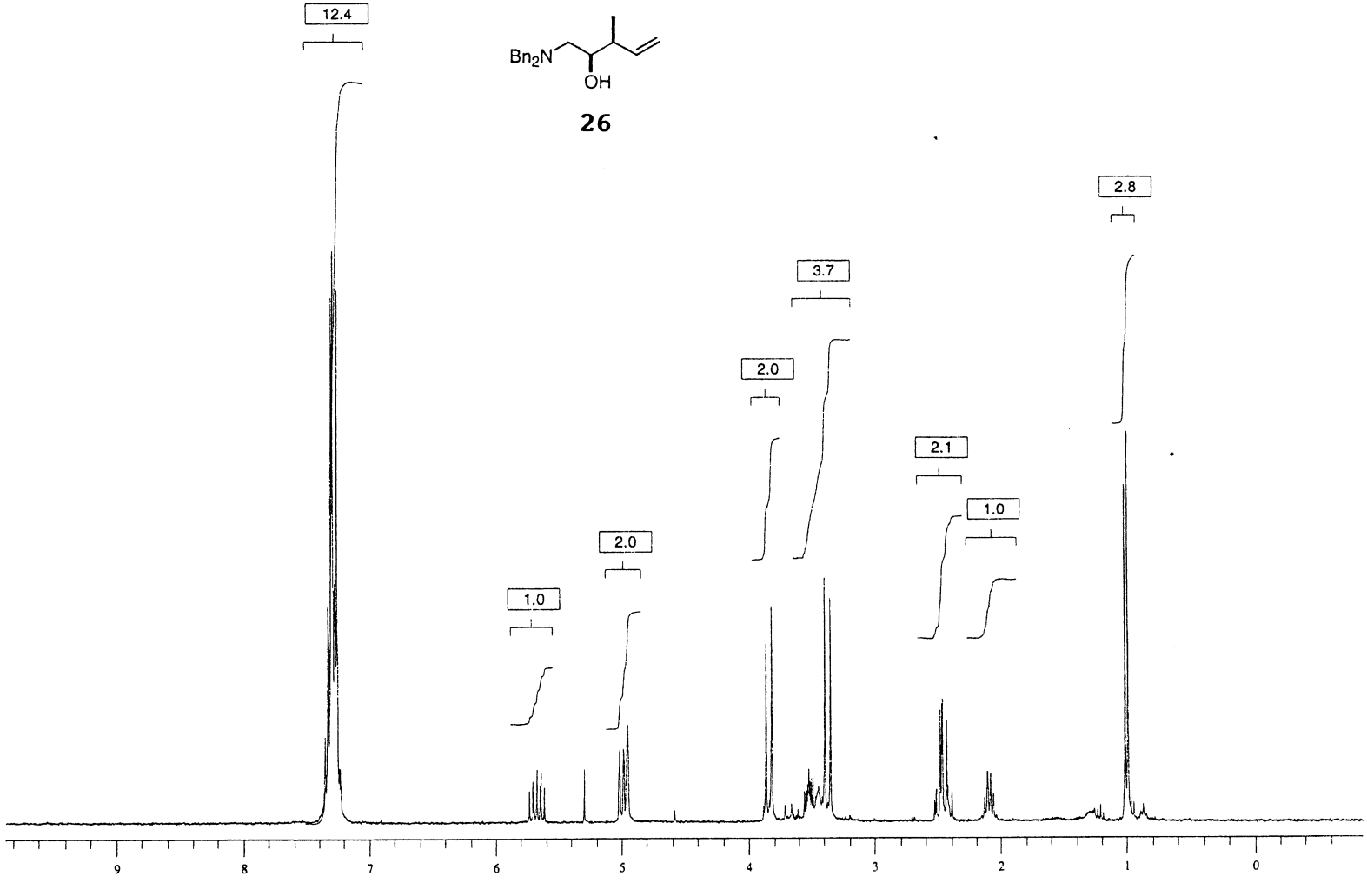
77.624
77.199
76.771
69.993
58.363
57.525
42.464
15.884
26
Bn2N
OH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Bn2N
OH
26
2.8
3.7
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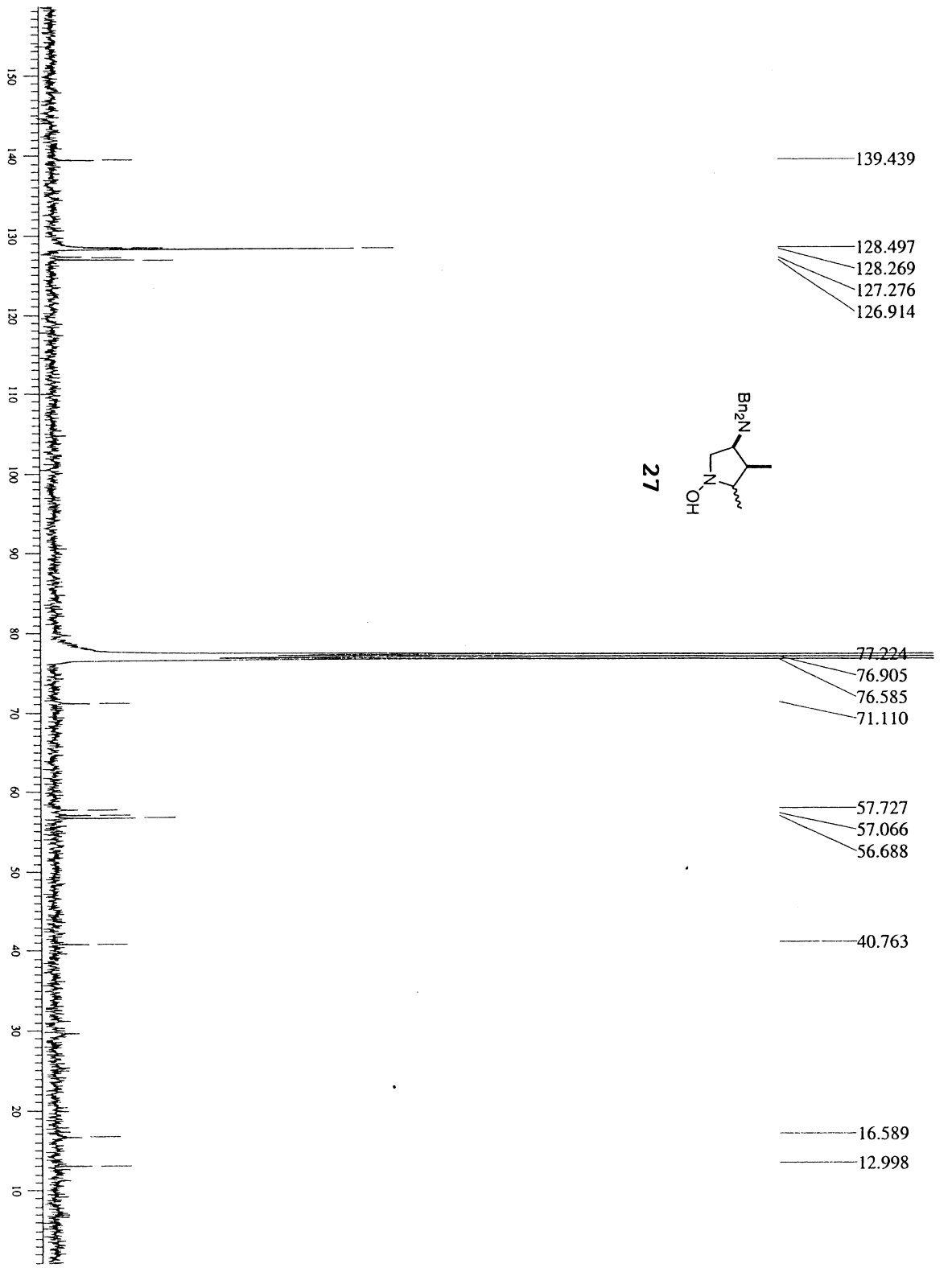
2.1
1.0
2.0
1.0
9
8
7
6
5
4
3
2
1
0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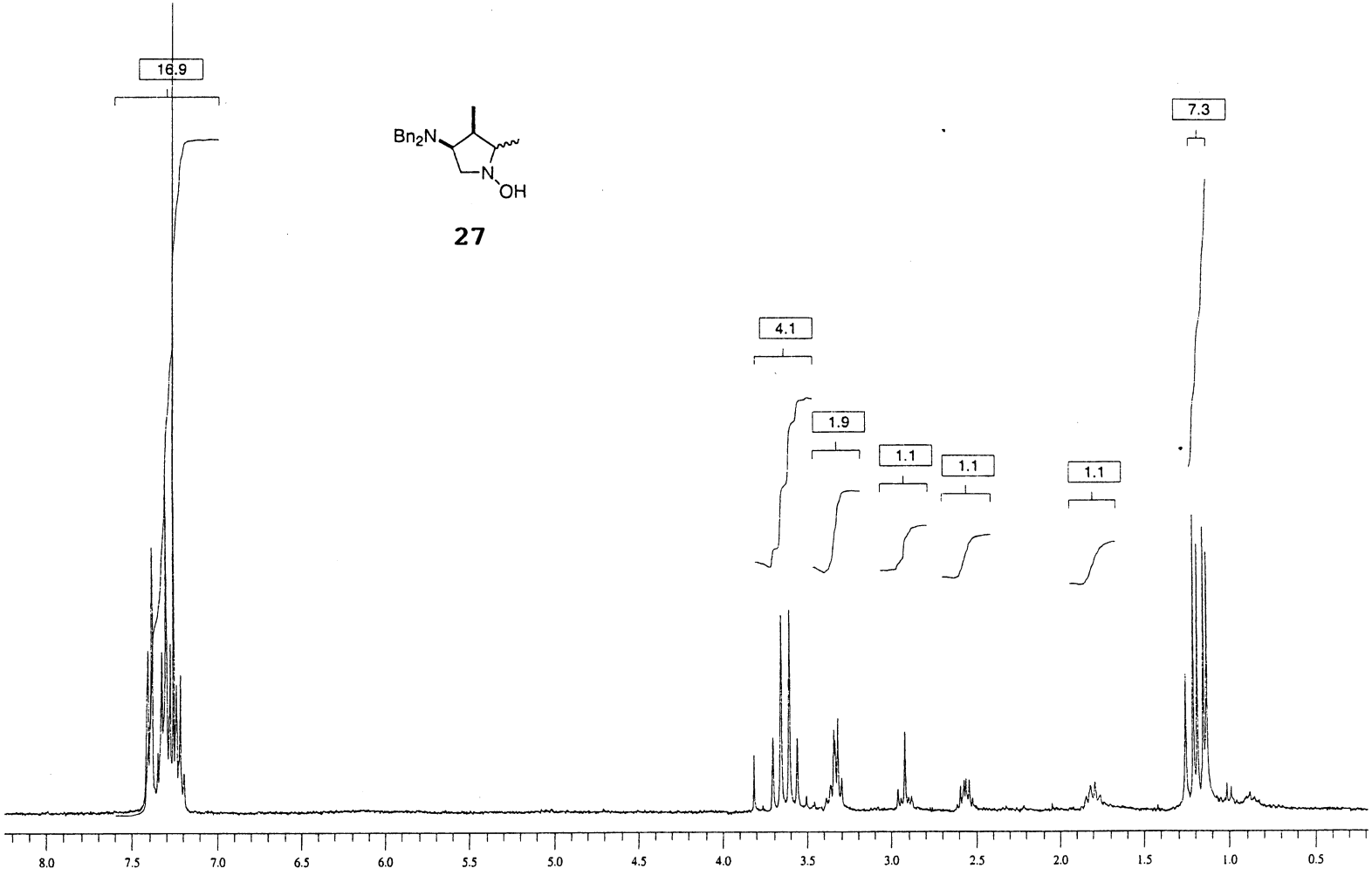
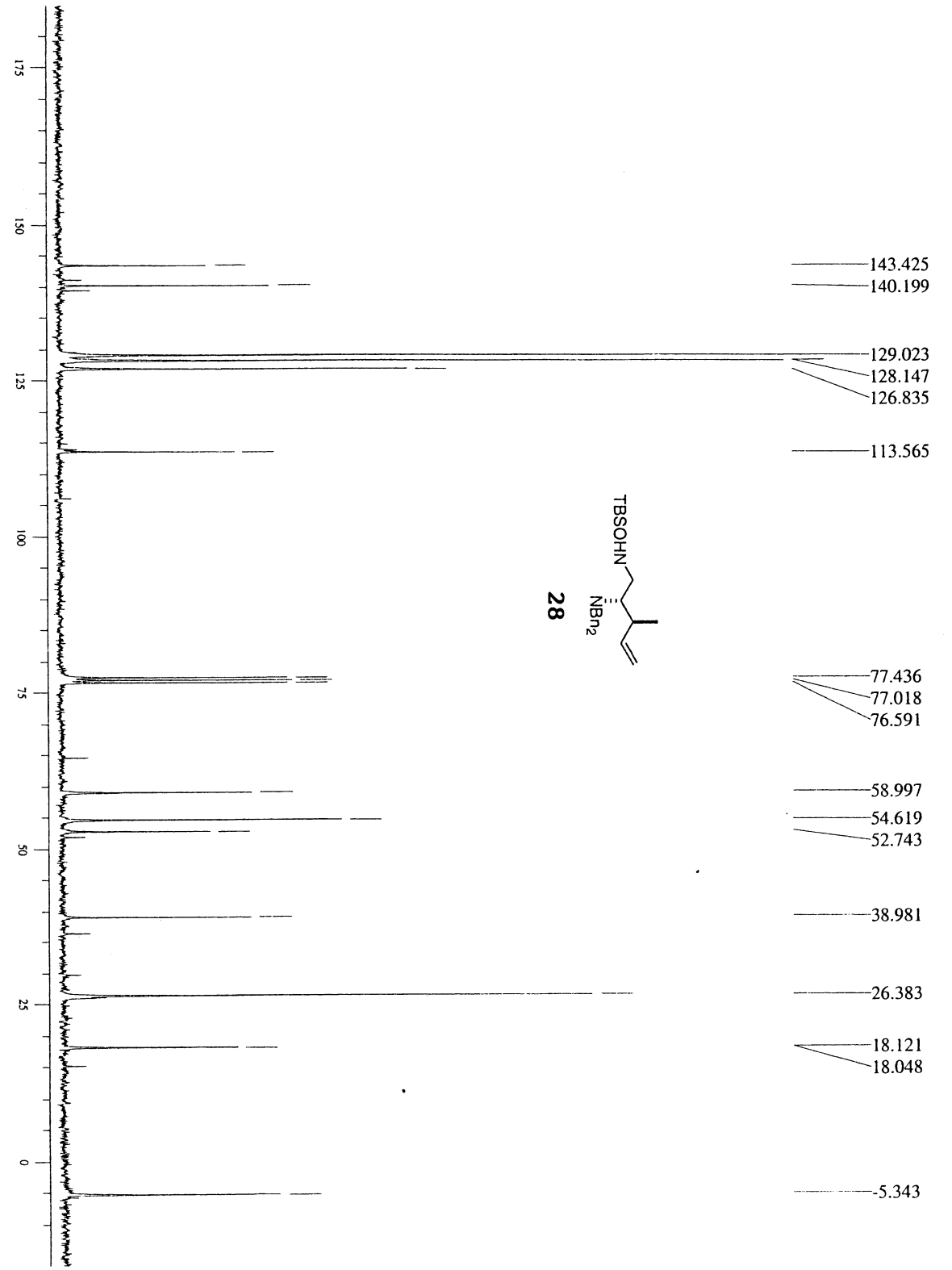
128.269
127.276
126.914
77.224
76.905
76.585
71.110
57.727
57.066
56.688
40.763
16.589
12.998
27
Bn2N
N
OH


16.9
Bn2N
N
OH
27
4.1
1.9
1.1
1.1
1.1
7.3
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5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128.147
126.835
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77.436
77.018
76.591
58.997
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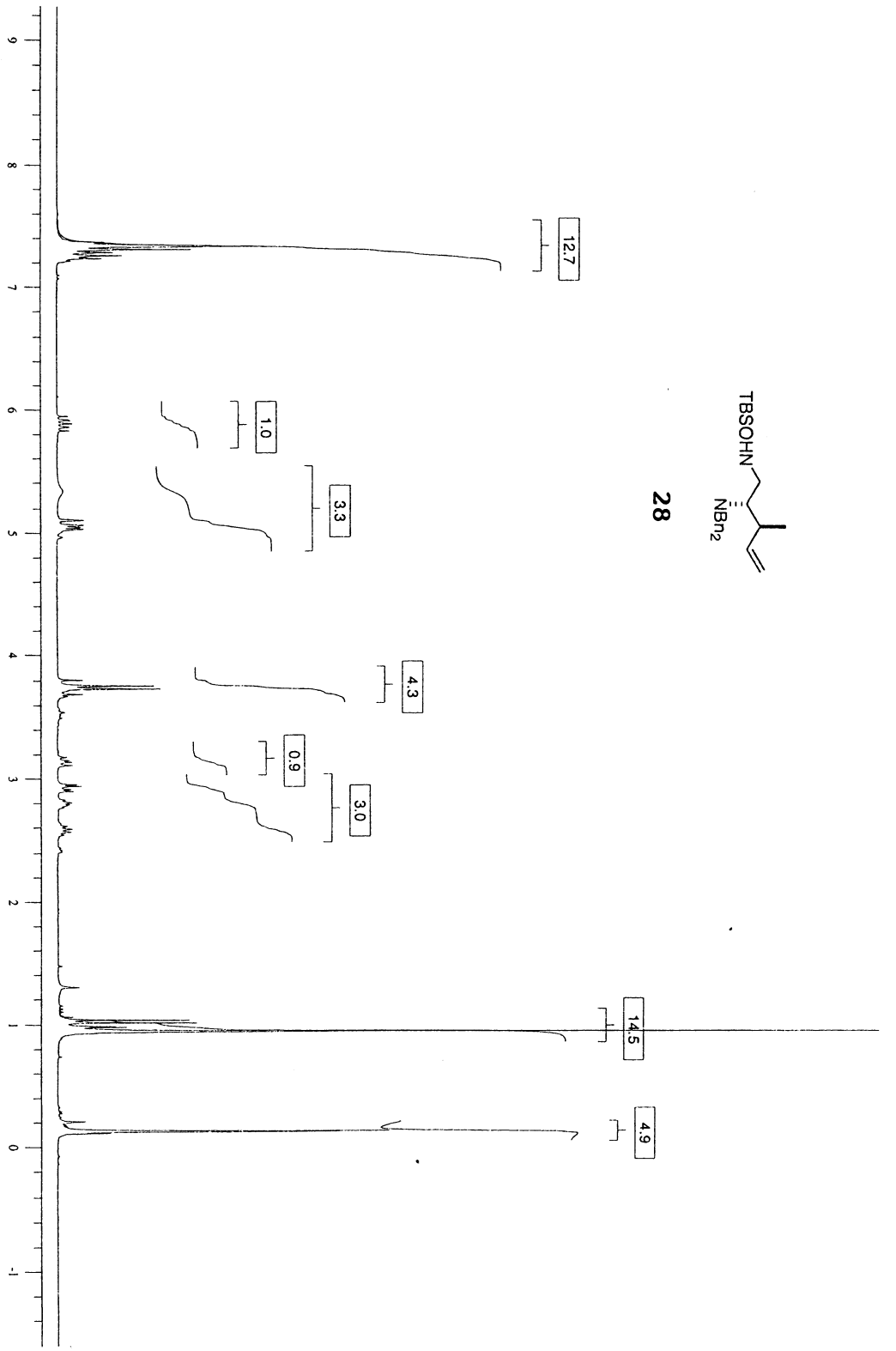
52.743
38.981
26.383
18.121
18.048
-5.343
TBSOHN
NBn2
28


TBSOHN
NBn2
28
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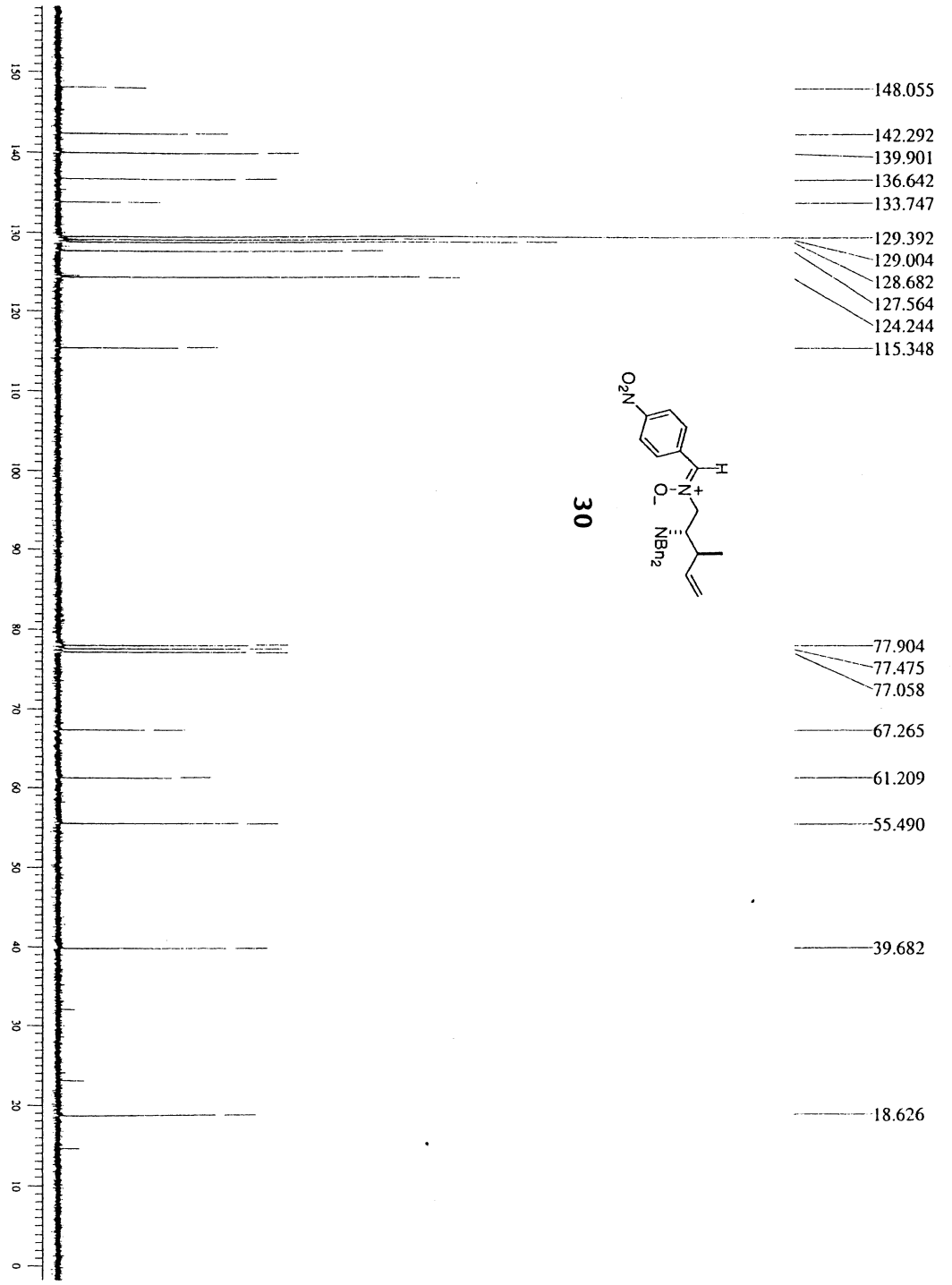
1.0
3.3
4.3
0.9
3.0
14.5
4.9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142.292
139.901
136.642
133.747
129.392
129.004
128.682
127.564
124.244
115.348
77.904
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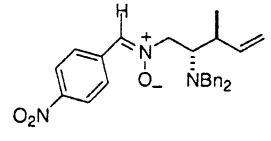
67.265
61.209
55.490
39.682
18.626
O2N
H
N+
O-
NBn2
30



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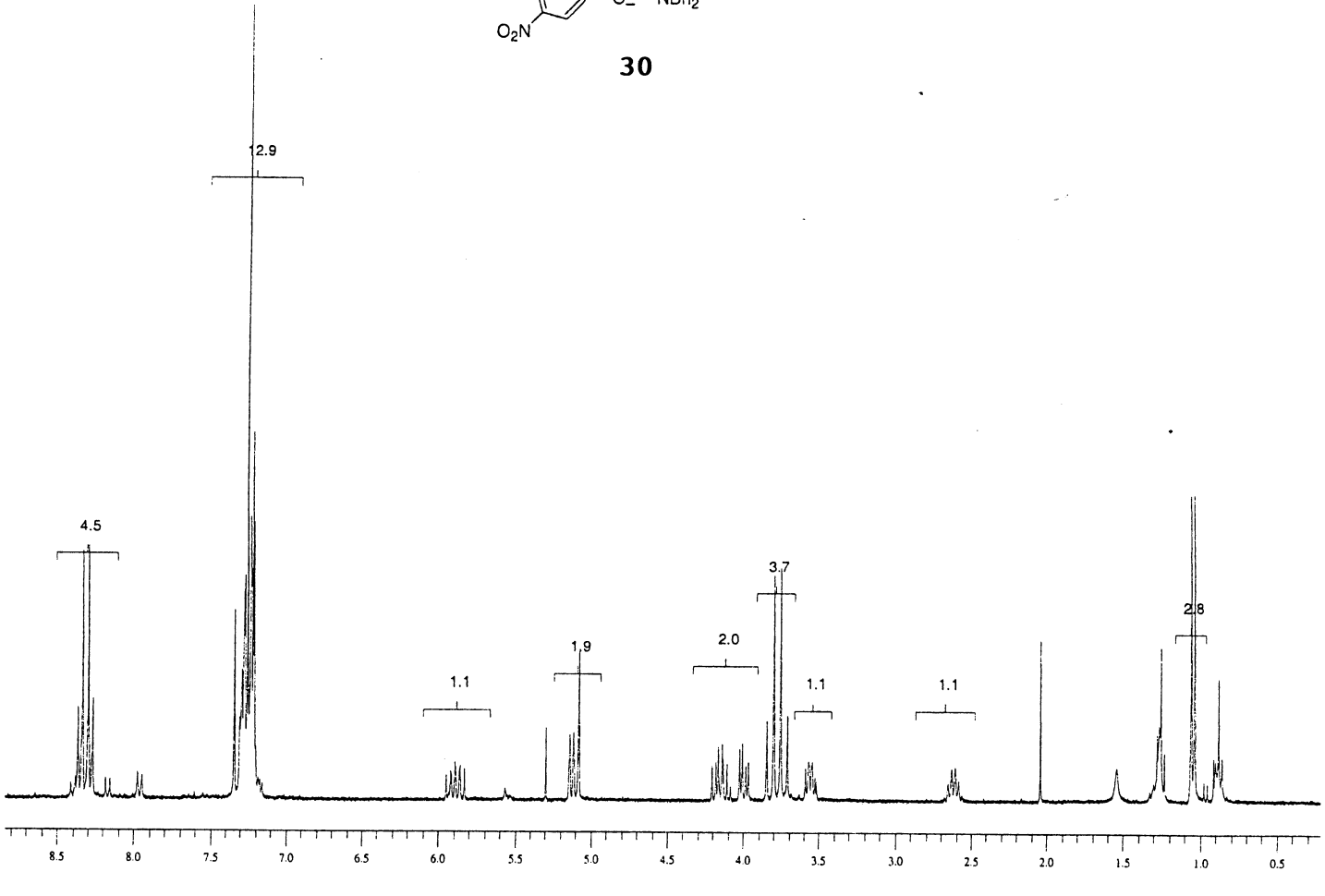
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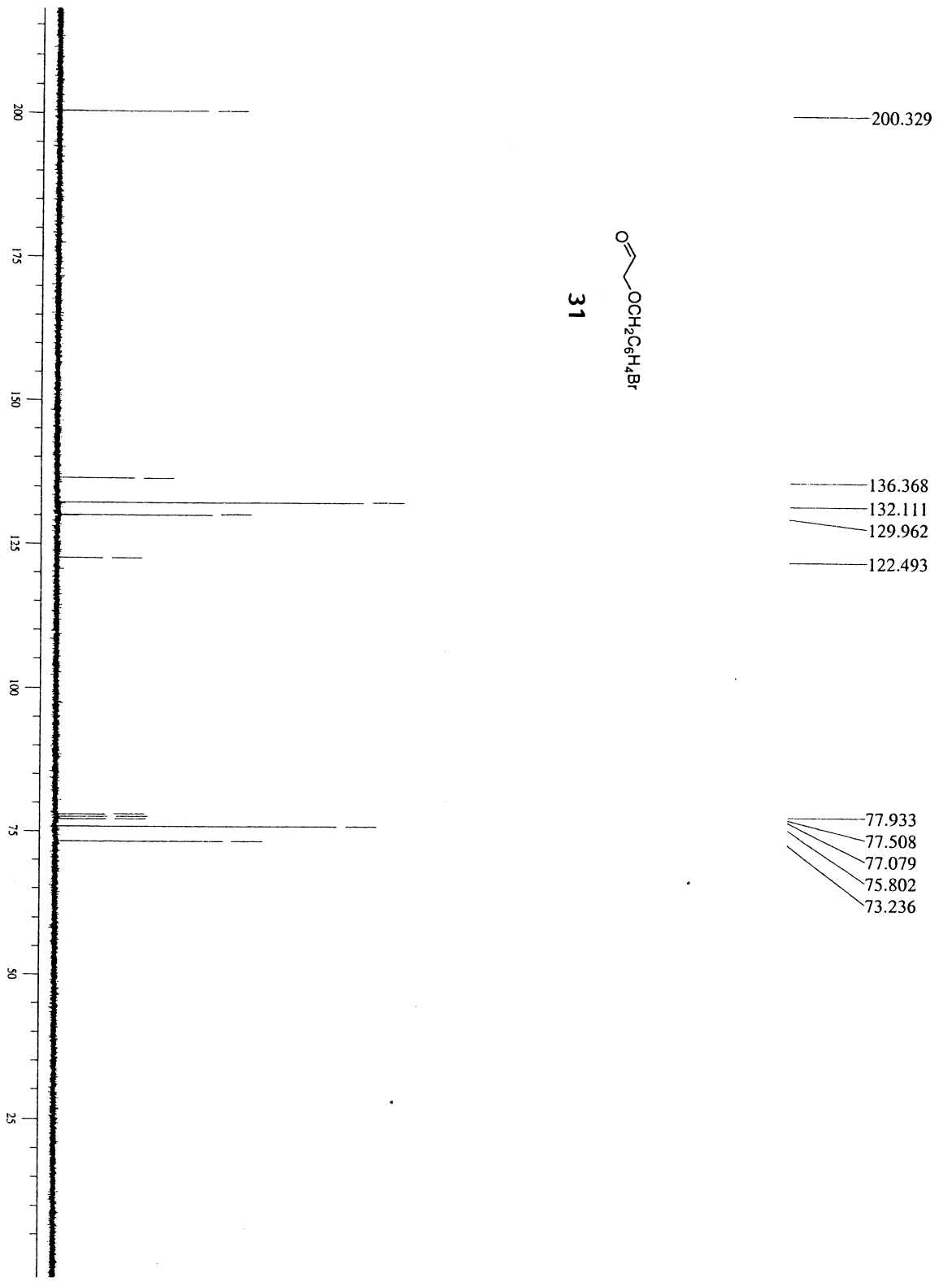
4.5
1.1
1.9
2.0
3.7
1.1
1.1
2.8
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5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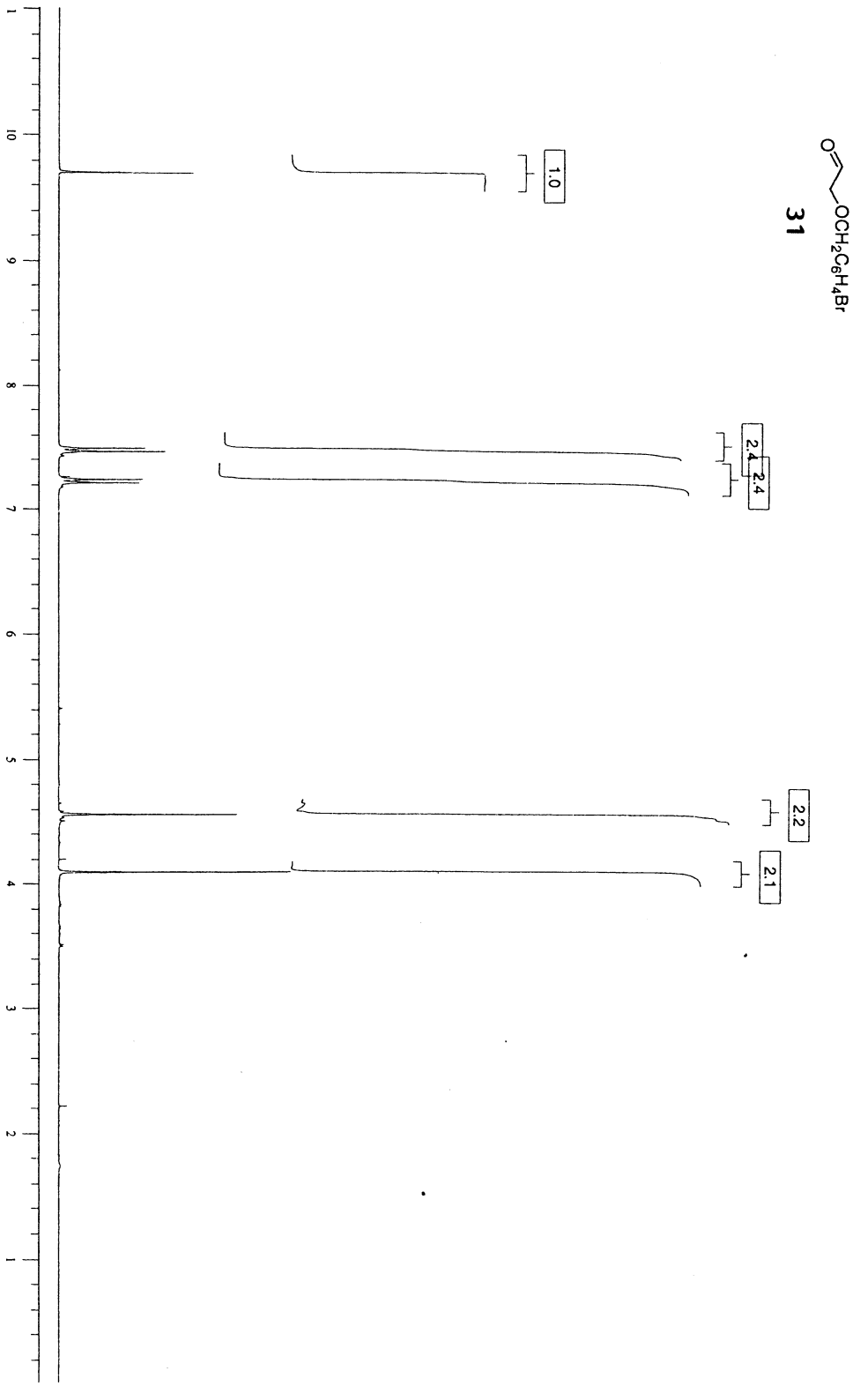
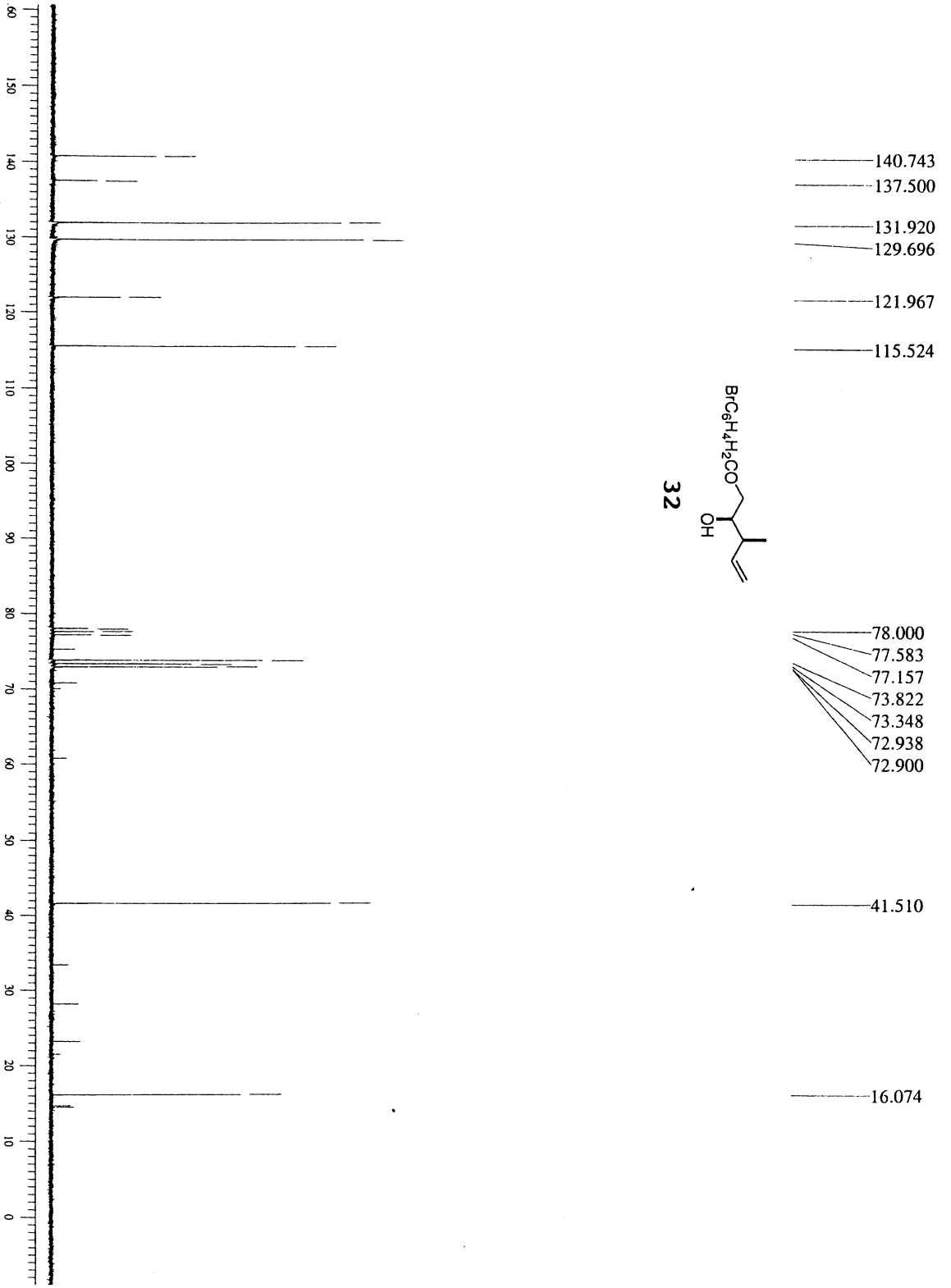
136.368
132.111
129.962
122.493
77.933
77.508
77.079
75.802
73.236
31
O
OCH2C6H4Br
200
175
150
125
100
75
50
25


OCH2C6H4Br
31
1.0
2.4
2.4
2.2
2.1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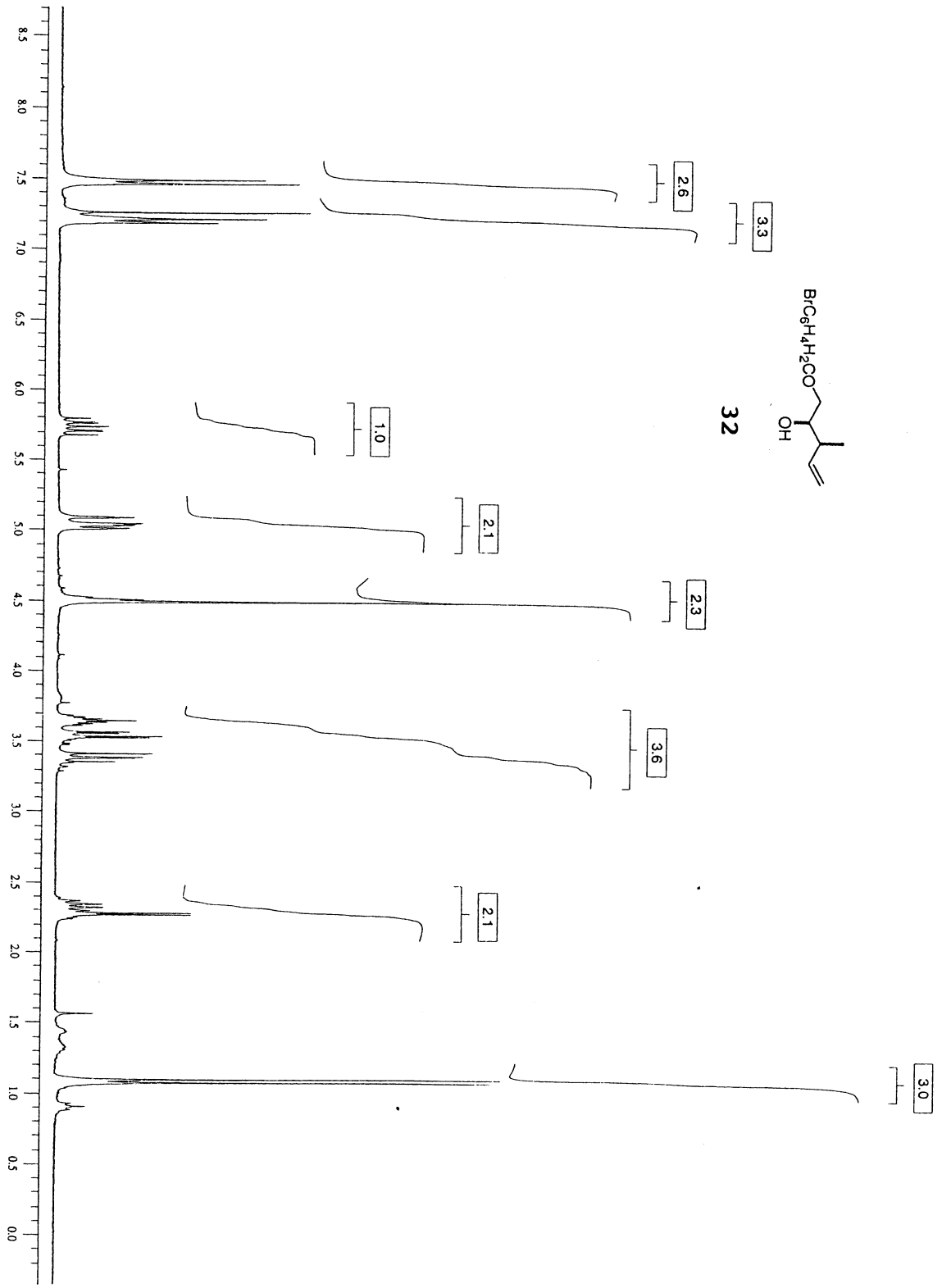
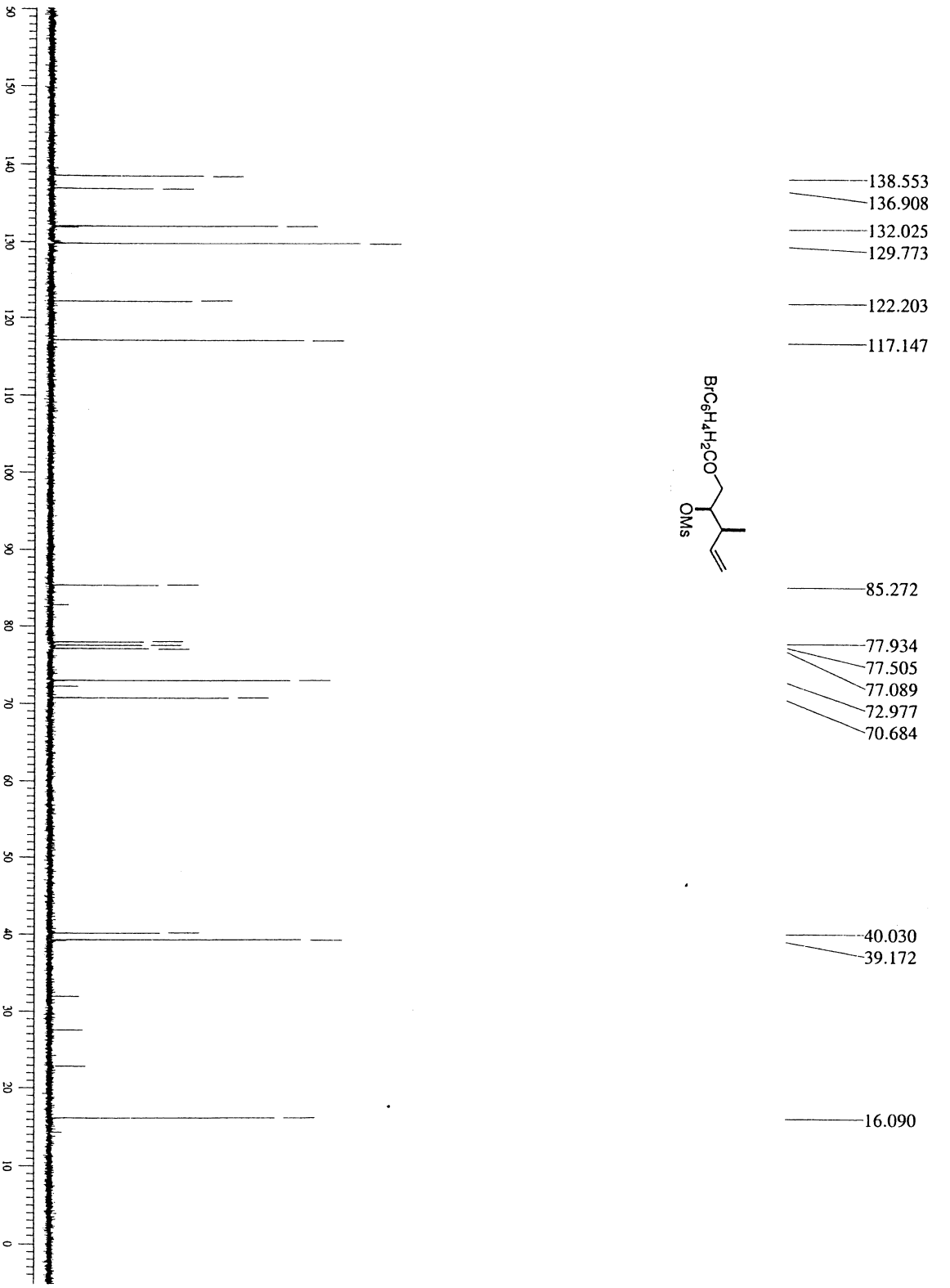
129.696
121.967
115.524
78.000
77.583
77.157
73.822
73.348
72.938
72.900
41.510
16.074
32
BrC6H4H2CO
OH


BrC6H4H2CO
OH
32
2.6
3.3
1.0
2.1
2.3
3.6
2.1
3.0


138.553
136.908
132.025
129.773
122.203
117.147
BrC6H4H2CO
OMs
85.272
77.934
77.505
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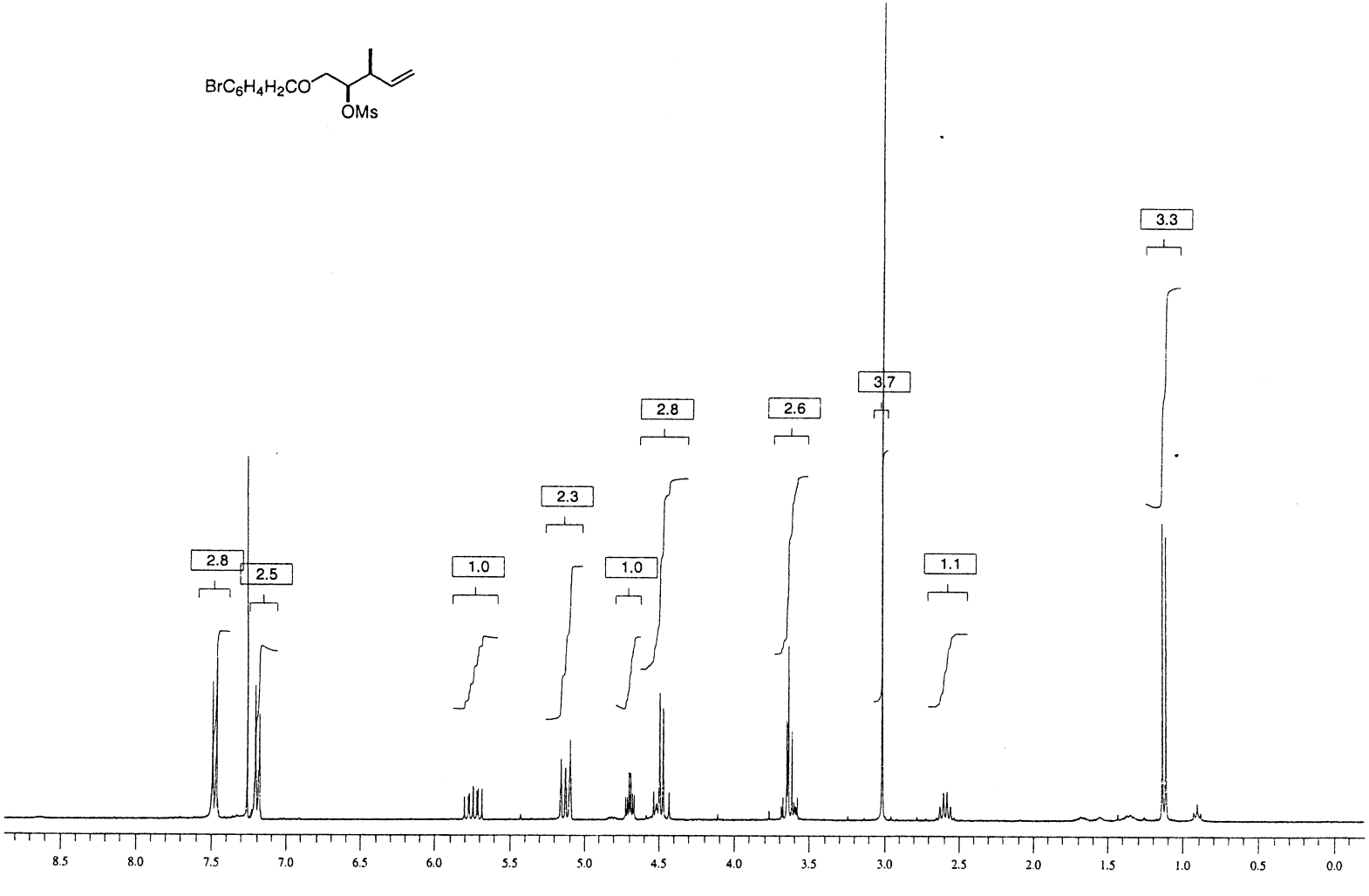
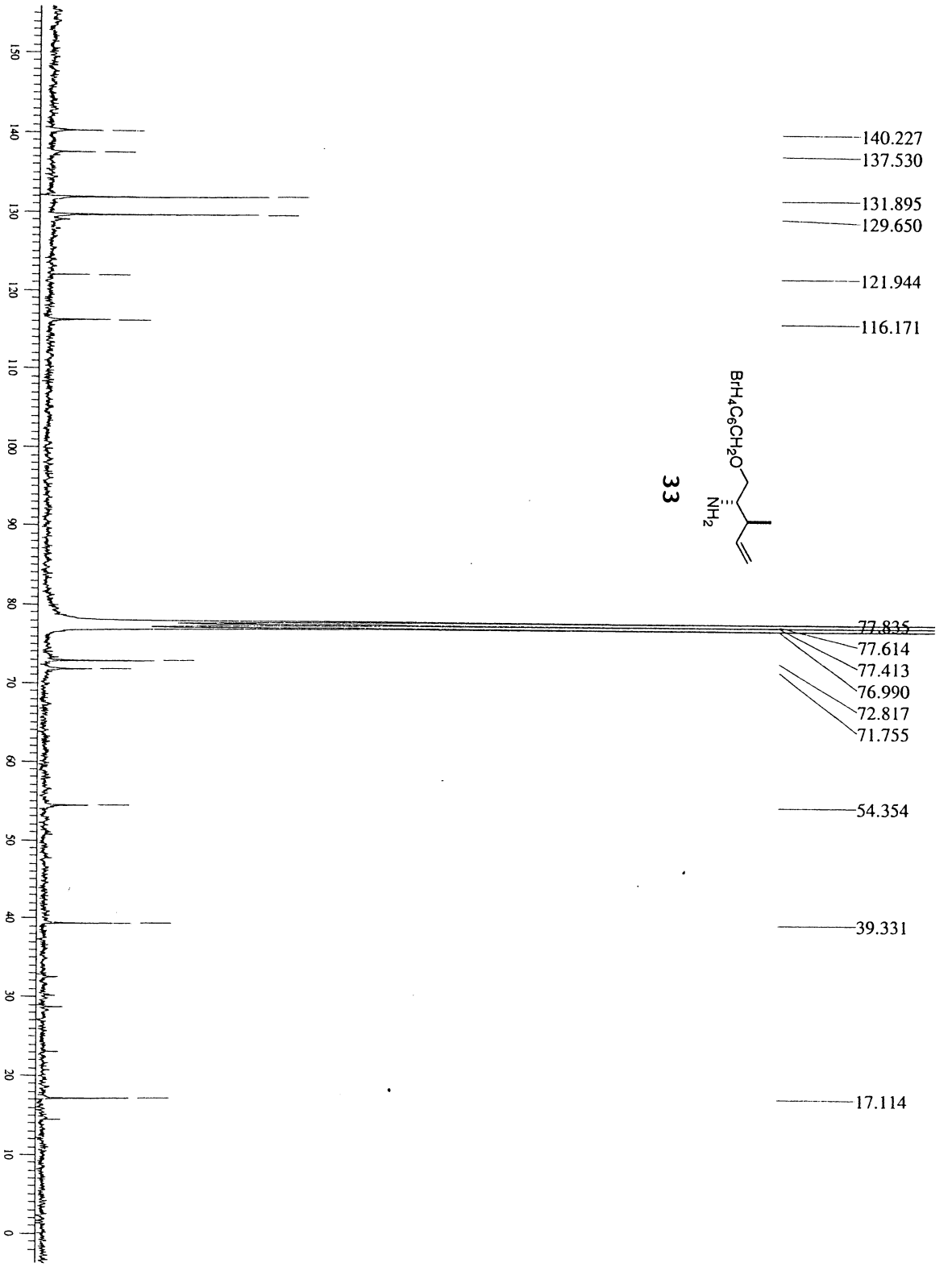
72.977
70.684
40.030
39.172
16.090


BrC6H4H2CO
OMs
2.8
2.5
1.0
2.3
1.0
2.8
2.6
3.7
1.1
3.3
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0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129.650
121.944
116.171
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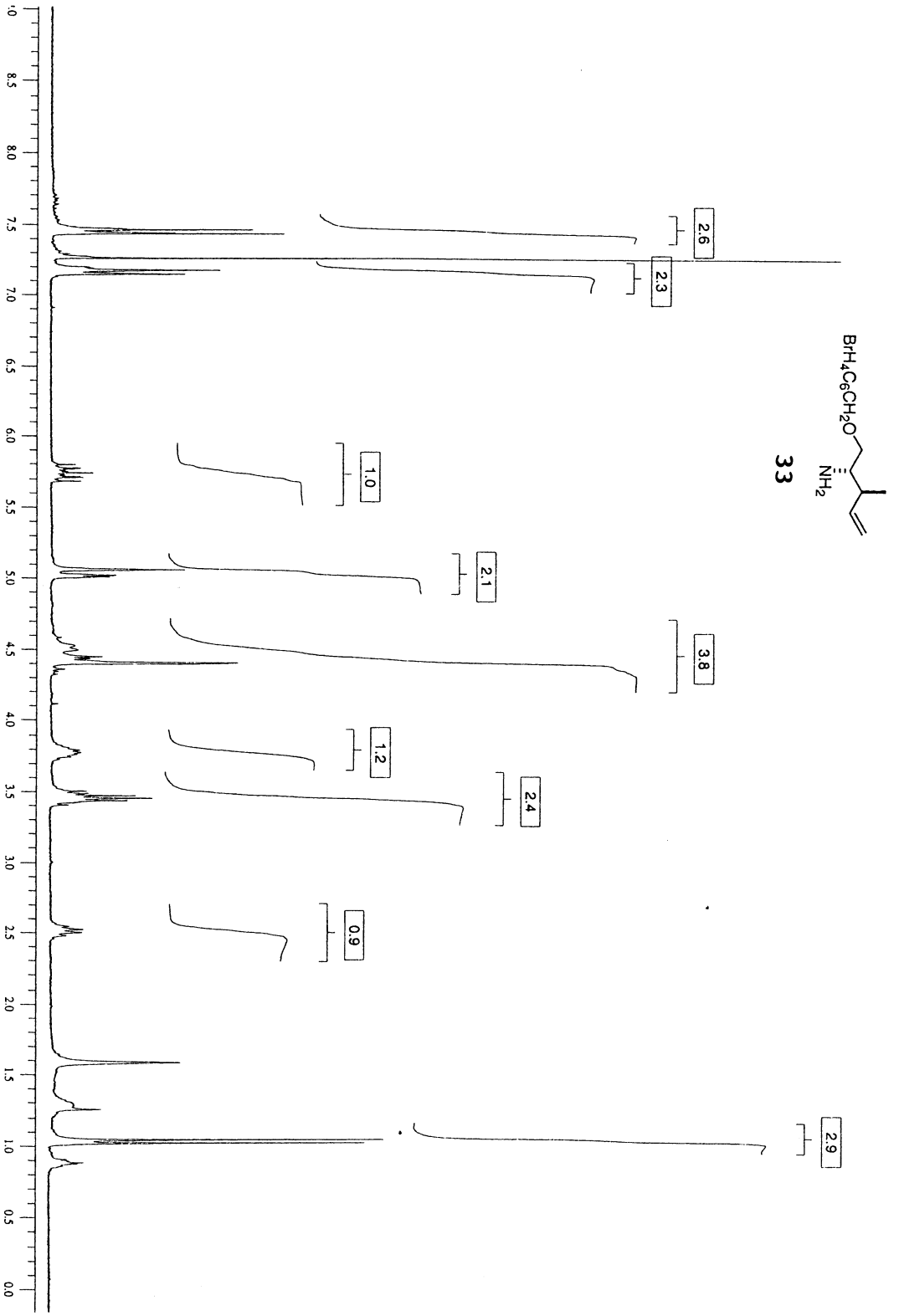
77.614
77.413
76.990
72.817
71.755
54.354
39.331
17.114
33
BrH4C6CH2O
NH2


BrH4C6CH2O
NH2
33
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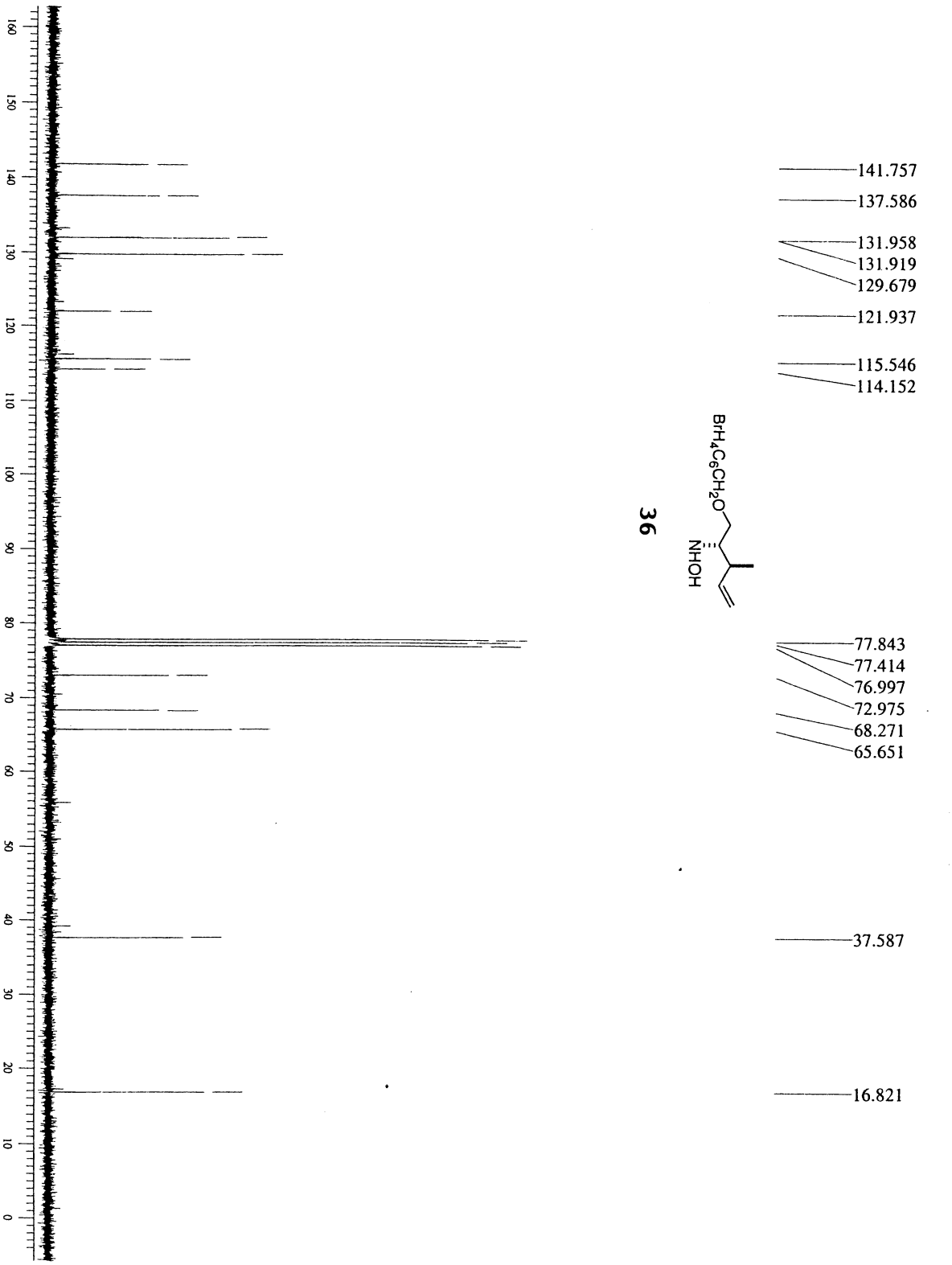
1.0
2.1
3.8
1.2
2.4
0.9
2.9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131.958
131.919
129.679
121.937
115.546
114.152
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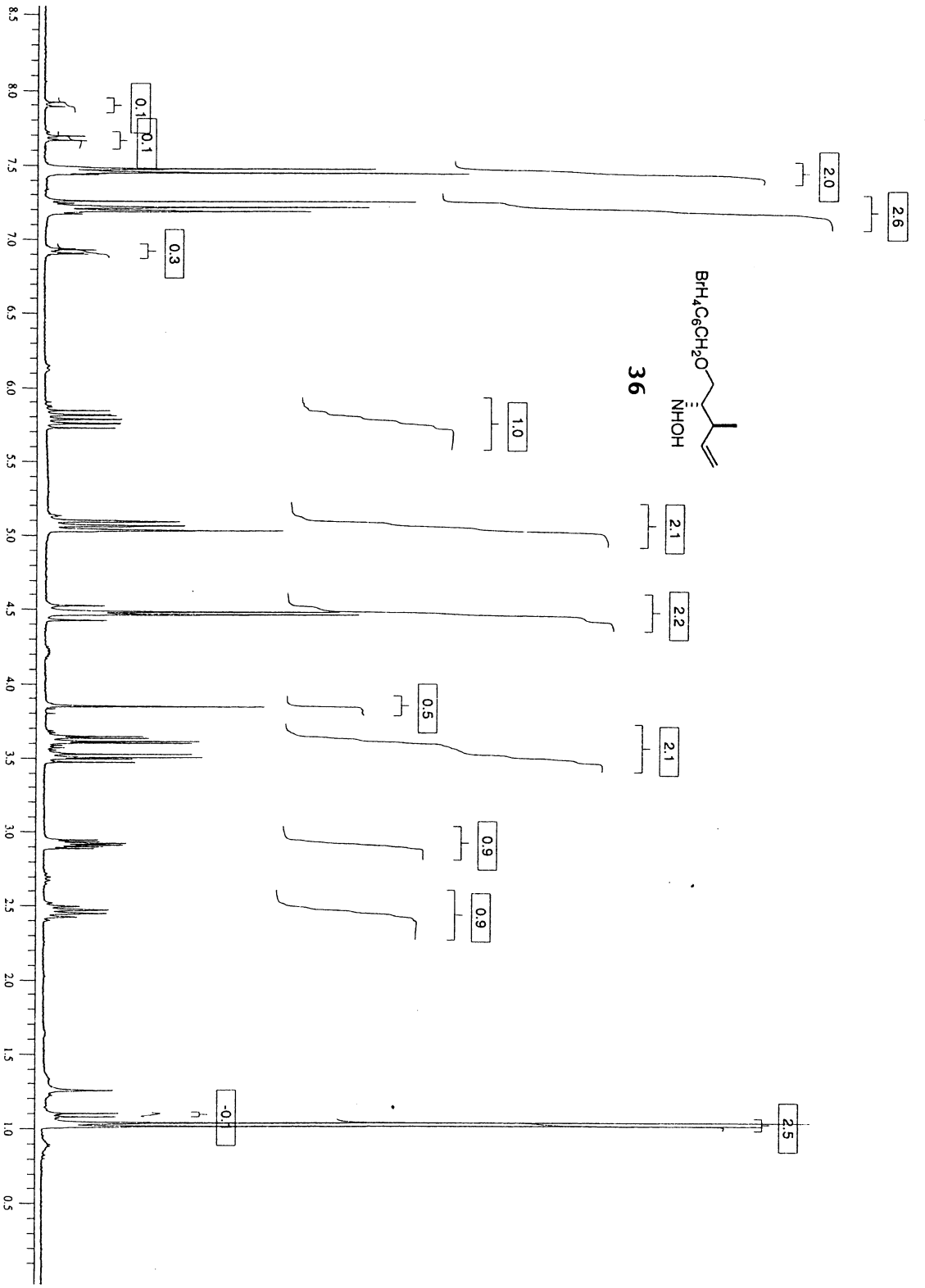
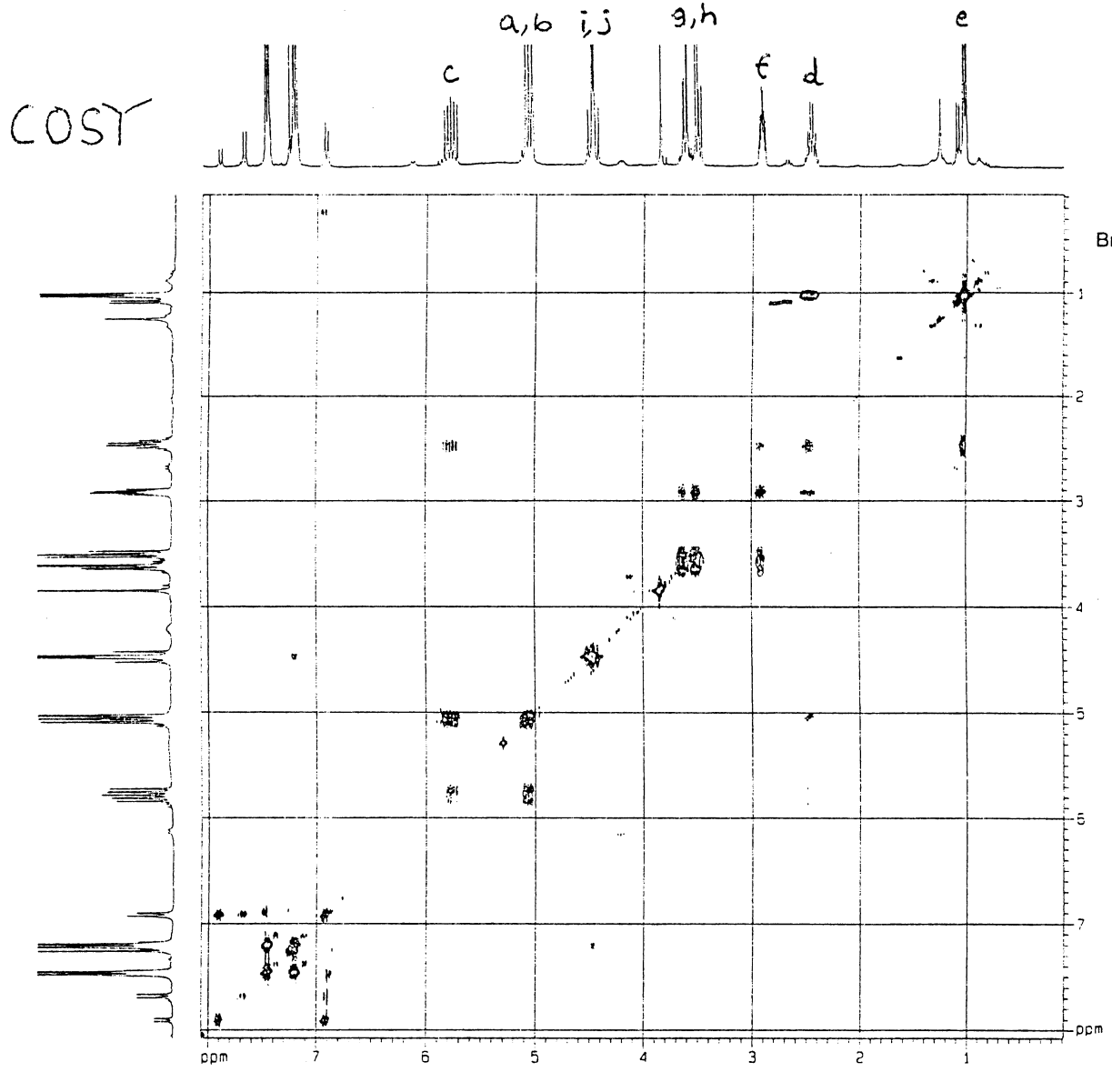
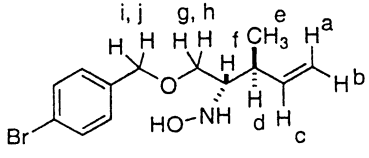
76.997
72.975
68.271
65.651
37.587
16.821
36
BrH4C6CH2O
NHOH


BrH4C6CH2O
NHOH
36

COSY





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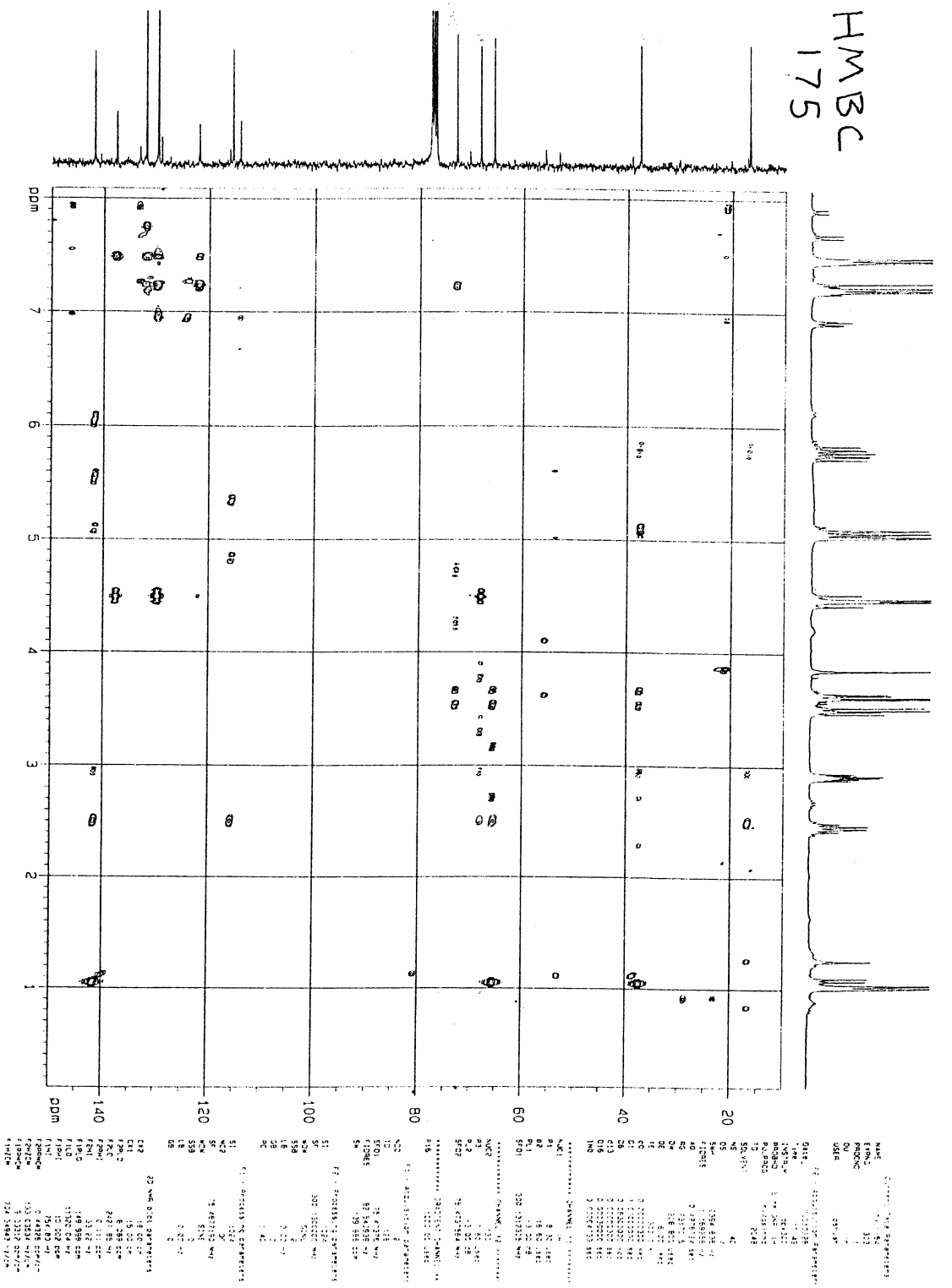
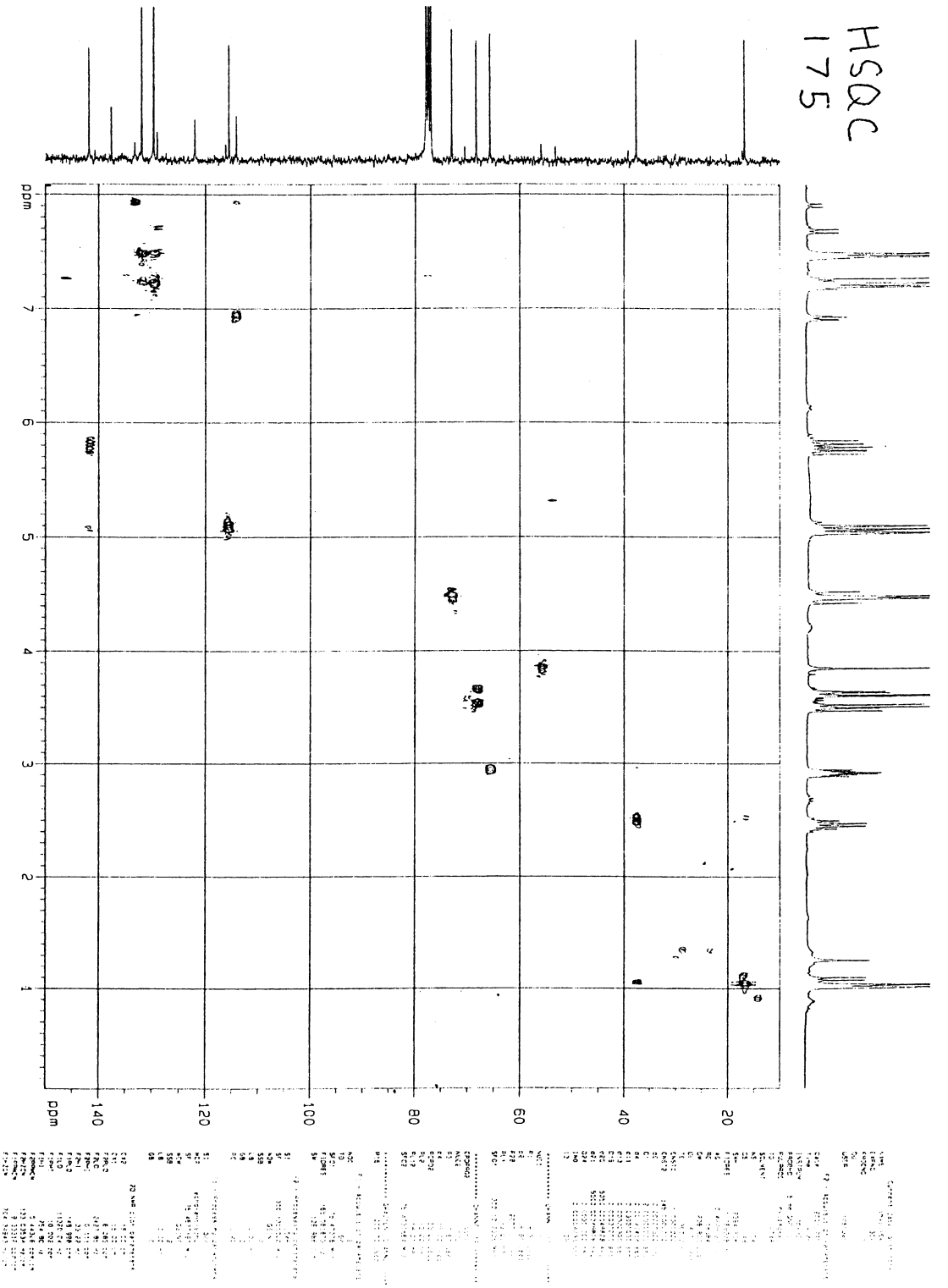
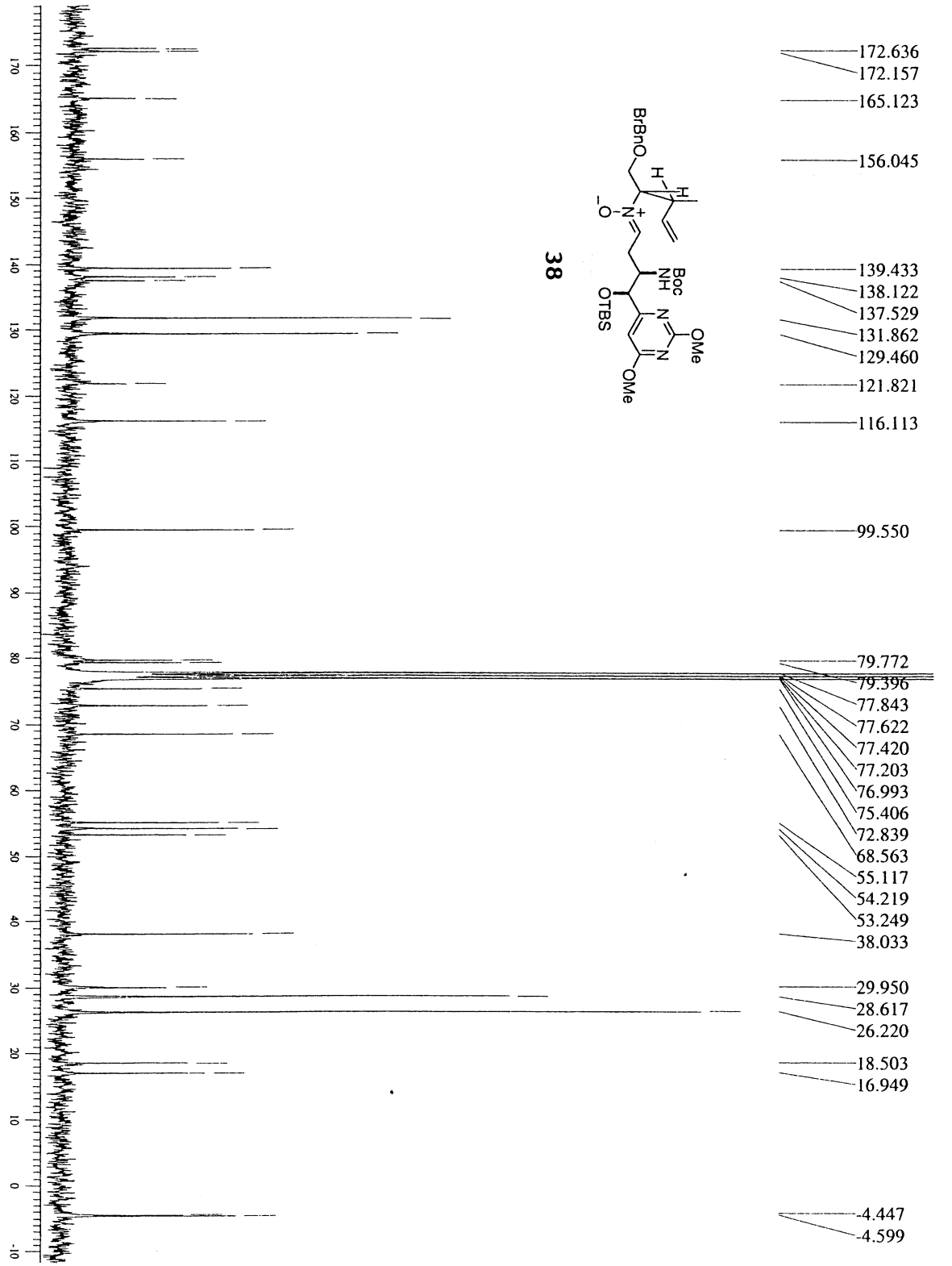

HMBC
175
ppm
ppm


HSQC
175
ppm
1
2
3
4
5
6
7
ppm
140
120
100
80
60
40
20


38
BrBnO
H
H
N+
O-
Boc
NH
OTBS
N
N
OMe
OMe
172.636
172.157
165.123
156.045
139.433
138.122
137.529
131.862
129.460
121.821
116.113
99.550
79.772
79.396
77.843
77.622
77.420
77.203
76.993
75.406
72.839
68.563
55.117
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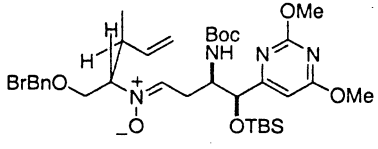
18.503
16.949
-4.447
-4.599


OMe
Boc
NH
N
N
H
H
BrBnO
N
+
O
OMe
OTBS

38

12.7
10.3
7.3
6.5
3.1
2.1
2.1
1.0
1.8
2.2
1.7
2.1
0.7
0.9
1.1
7.5
7.0
6.5
6.0
5.5
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4.5
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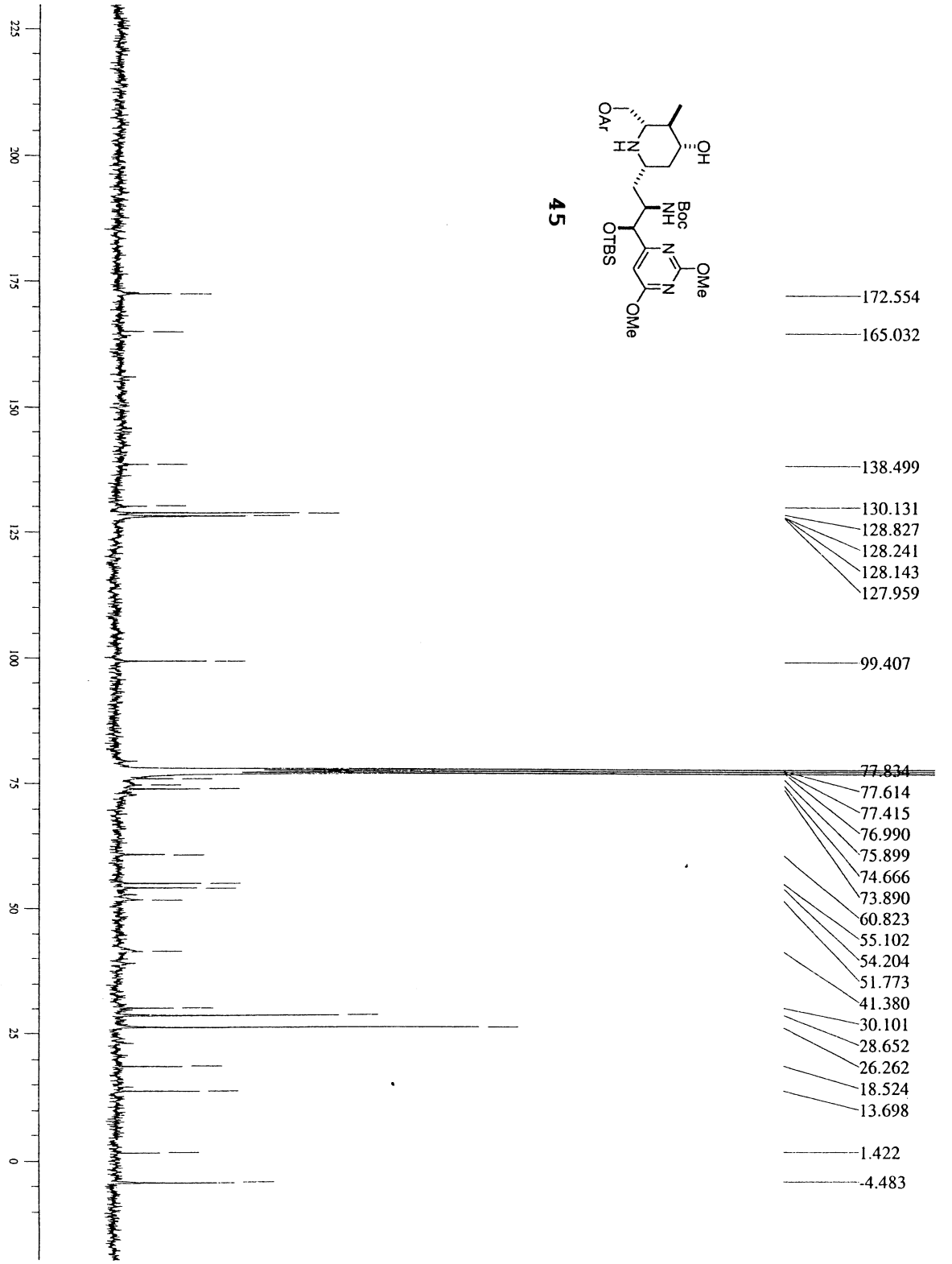
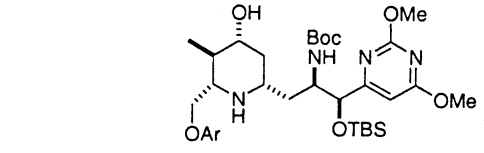
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5


45
OAr
N
H
OH
Boc
NH
OTBS
N
N
OMe
OMe
172.554
165.032
138.499
130.131
128.827
128.241
128.143
127.959
99.407
77.834
77.614
77.415
76.990
75.899
74.666
73.890
60.823
55.102
54.204
51.773
41.380
30.101
28.652
26.262
18.524
13.698
1.422
-4.483
225
200
175
150
125
100
75
50
25
0


OH
OMe
Boc
NH
N
N
N
H
OMe
OAr
OTBS

45

7.5
1.0
0.7
3.0
6.8
0.8
1.6
2.4
10.5
11.8
6.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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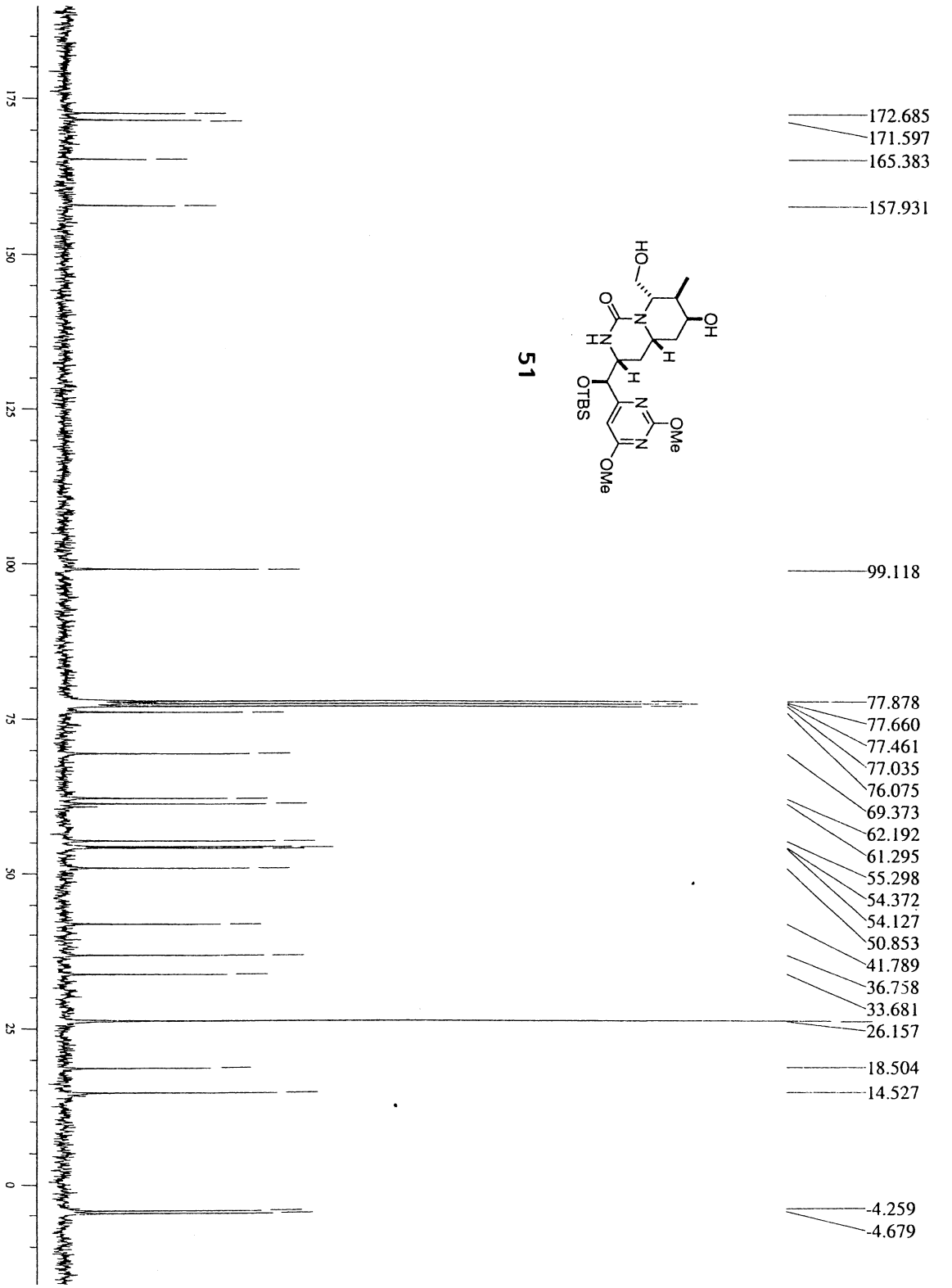
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5


175
150
125
100
75
50
25
0
172.685
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165.383
157.931
99.118
77.878
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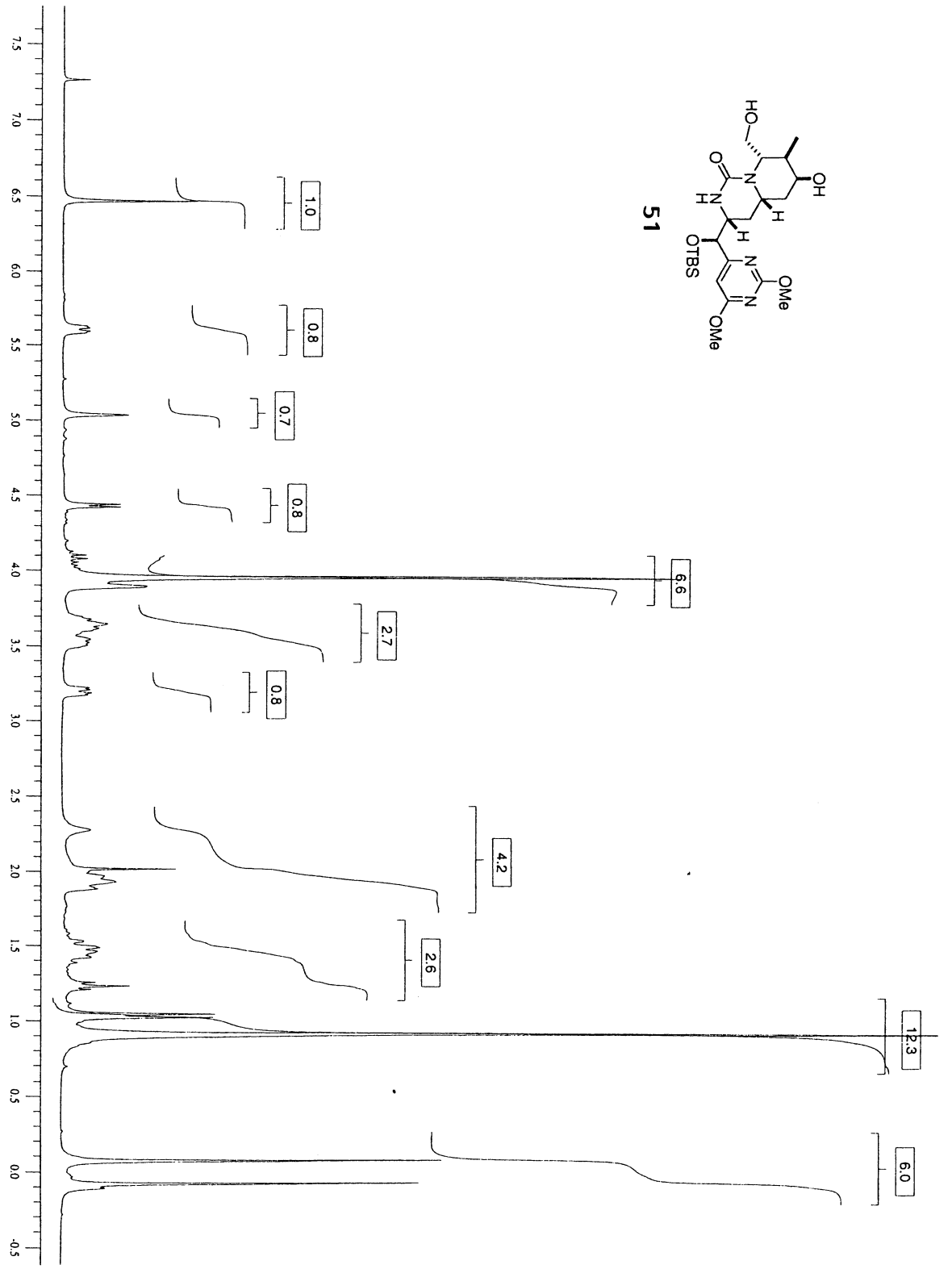
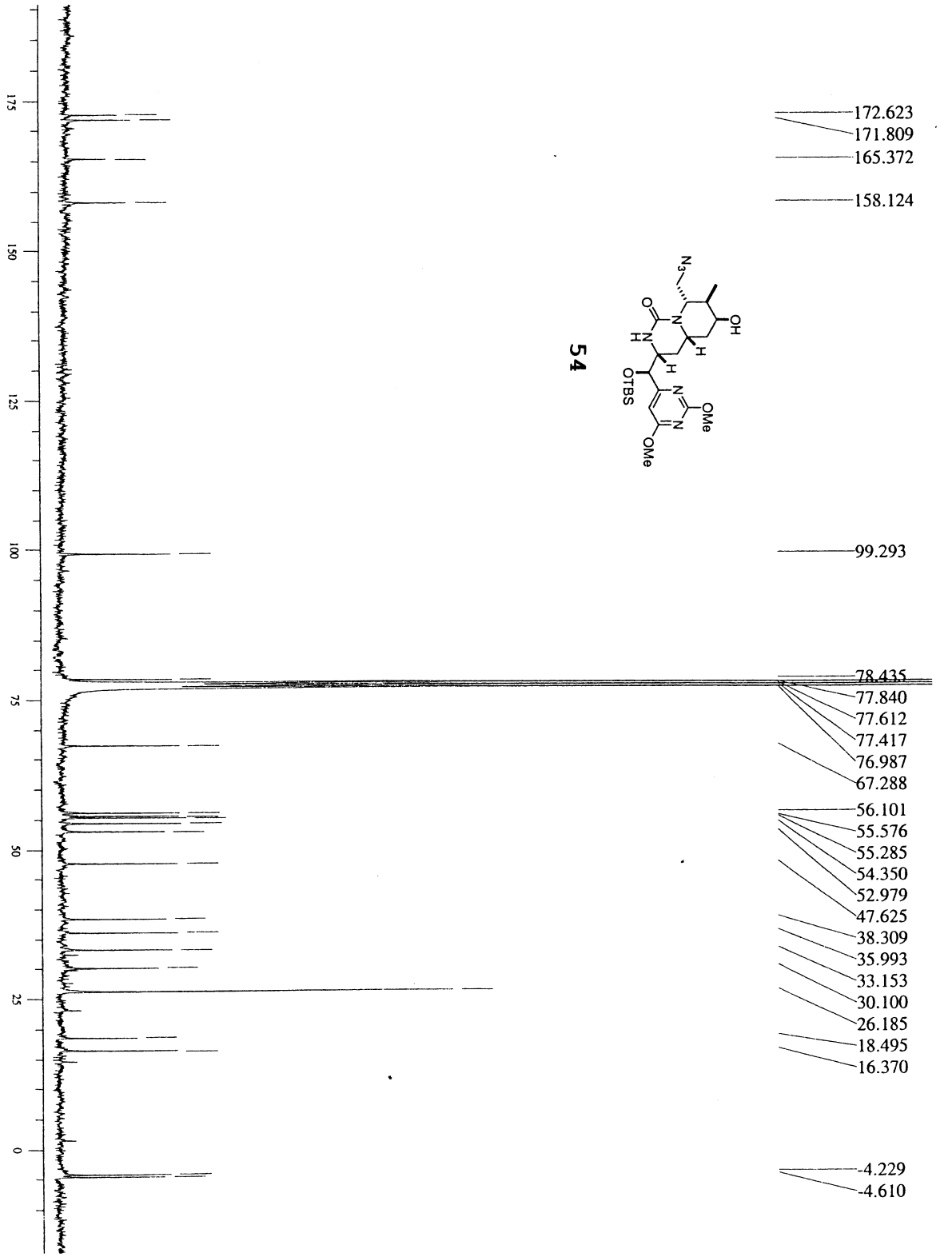
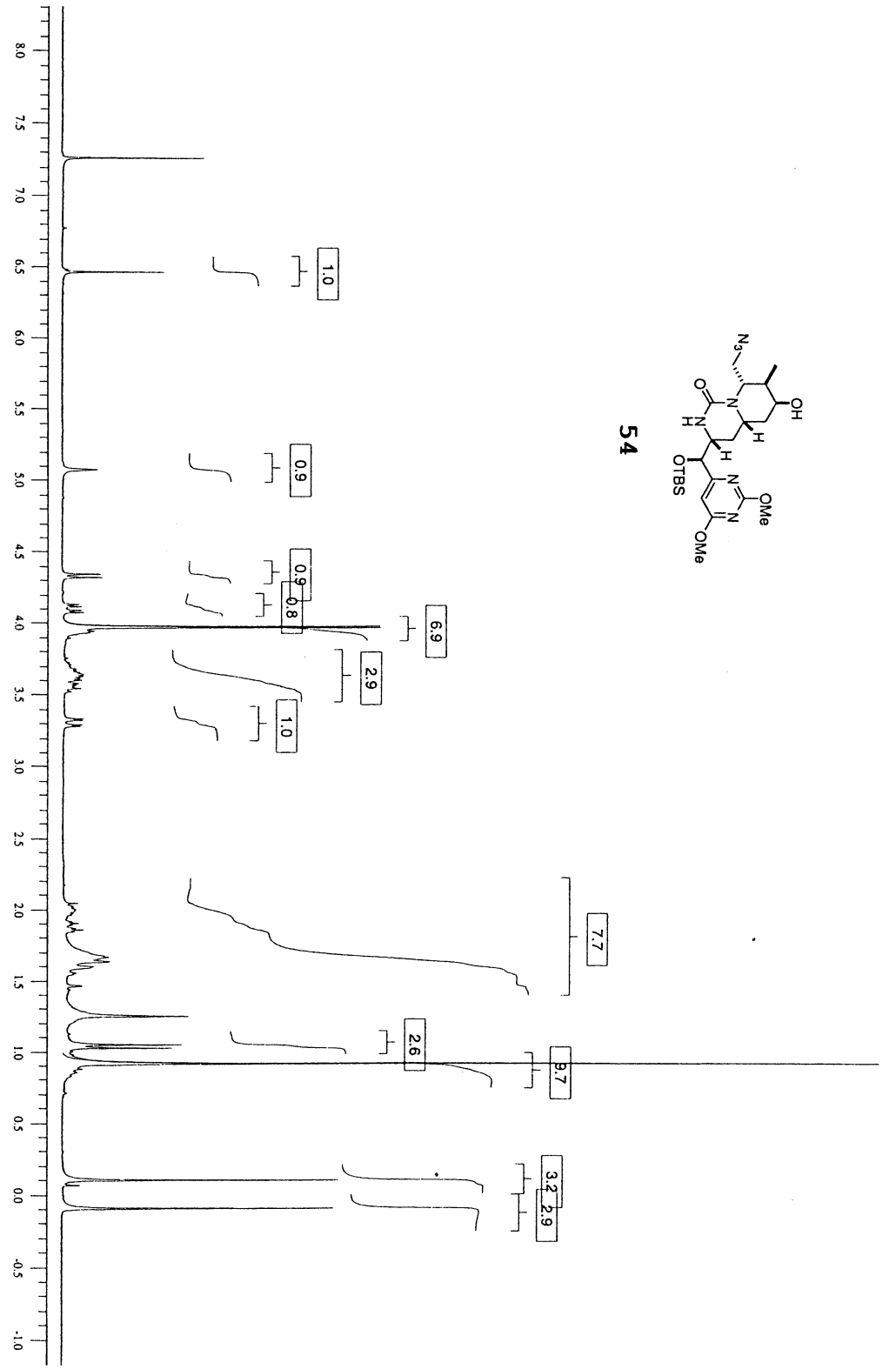
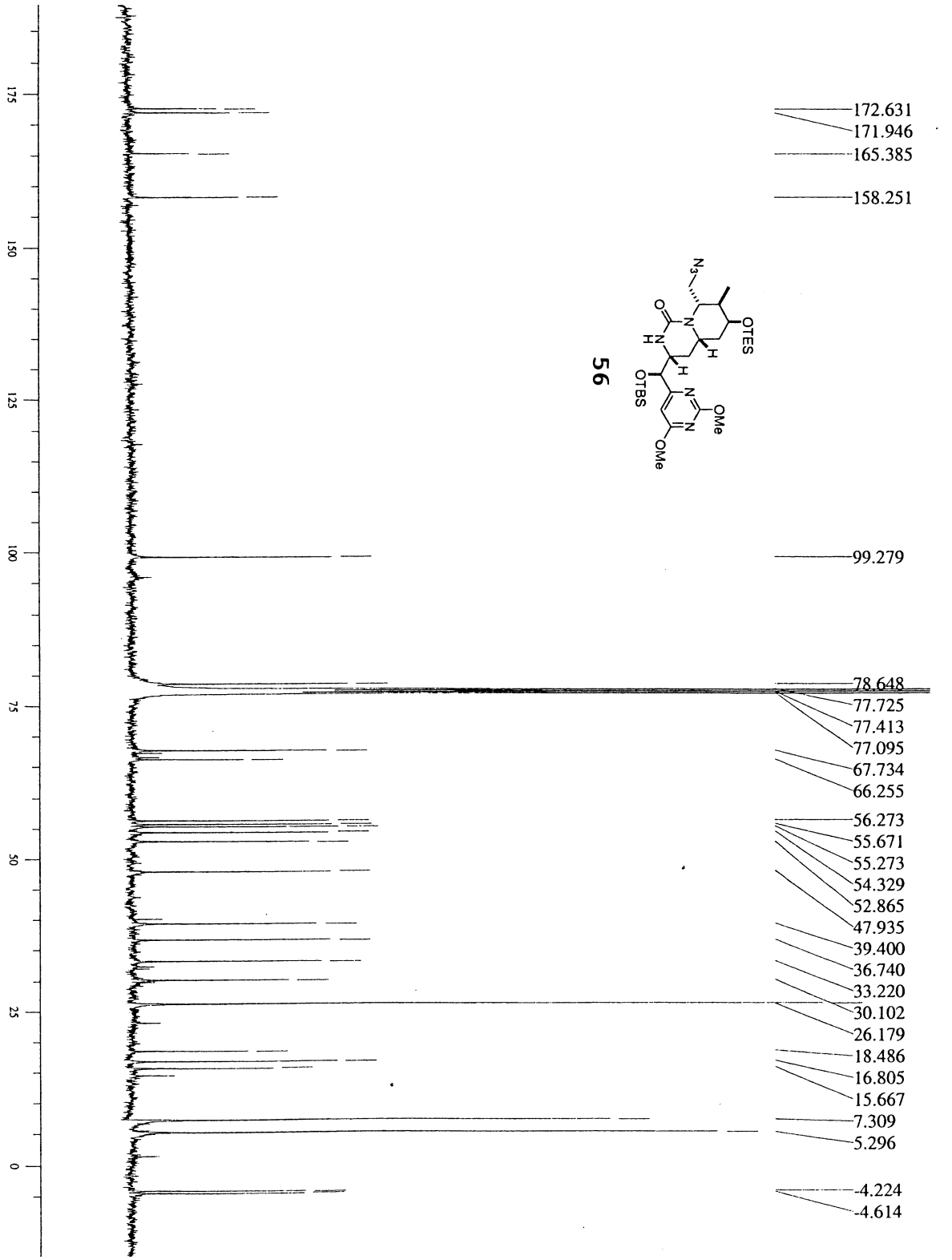
77.461
77.035
76.075
69.373
62.192
61.295
55.298
54.372
54.127
50.853
41.789
36.758
33.681
26.157
18.504
14.527
-4.259
-4.679
HO
OH
OTBS
OMe
OMe

51


HO
OH
OTBS
OMe
OMe
51
1.0
0.8
0.7
0.8
6.6
2.7
0.8
4.2
2.6
12.3
6.0


172.623
171.809
165.372
158.124
99.293
78.435
77.840
77.612
77.417
76.987
67.288
56.101
55.576
55.285
54.350
52.979
47.625
38.309
35.993
33.153
30.100
26.185
18.495
16.370
-4.229
-4.610
175
150
125
100
75
50
25
0
54
N3
OH
OTBS
OMe
OMe


54
N3
OH
H
N
O
N
H
H
OTBS
N
OMe
N
OMe
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
1.0
0.9
0.9
0.8
6.9
2.9
1.0
7.7
2.6
9.7
3.2
2.9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99.279
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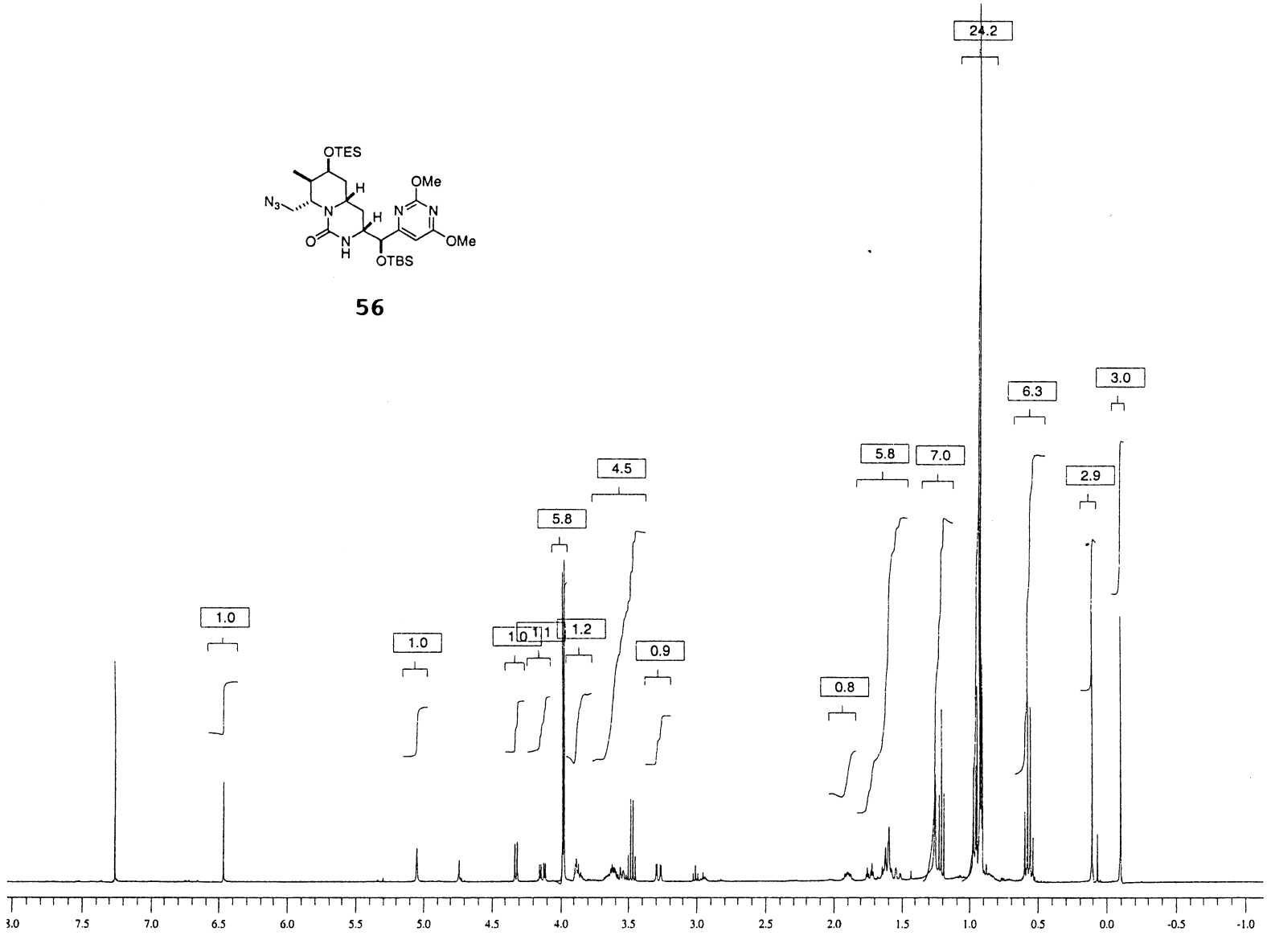
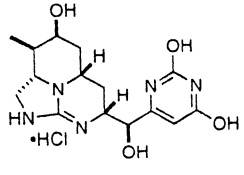
56.273
55.671
55.273
54.329
52.865
47.935
39.400
36.740
33.220
30.102
26.179
18.486
16.805
15.667
7.309
5.296
-4.224
-4.614
175
150
125
100
75
50
25
0
56
N3
O
N
H
OTES
H
H
OTBS
N
N
OMe
OMe


OTES
OMe
H
N3
N
N
H
O
N
H
OMe
OTBS
24.2
6.3
3.0
5.8
7.0
4.5
5.8
2.9
1.0
1.0
1.2
0.9
0.8
3.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0

56



60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Current Data Parameters
NAME                  jmix70
EXPNO                    208
PROCNO                     1
DU                        /n
USER                joshuam

F2 - Acquisition Parameters
Date_              20011201
Time                  22.56
INSTRUM               dpx400
PROBHD      5 mm BBO Z-G
PULPROG     zgpg30rep.rx
TD                     65536
SOLVENT
NS                     36399
DS                         4
SWH               25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                    3649.1
DW                   19.900 usec
DE                    6.00 usec
TE                   300.0 K
D1                 0.00000300 sec
D11                0.03000000 sec
D12                0.00002000 sec
L2                        60

=========== CHANNEL f1 ============
NUC1                     13C
P1                     10.00 usec
PL1 *                   0.00 dB
SFO1              100.6237959 MHz

=========== CHANNEL f2 ============
CPDPRG2              waltz16
NUC2                      1H
PCPD2                  80.00 usec
PL2                     0.00 dB
PL12                   17.40 dB
PL13                   17.40 dB
SFO2              400.1316005 MHz

F2 - Processing parameters
SI                       32768
SF                100.6127290 MHz
WDW                         EM
SSB                          0
LB                      3.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                      20.00 cm
F1P                   200.000 ppm
F1                  20122.55 Hz
F2P                   -10.000 ppm
F2                  -1006.13 Hz
PPMCM                10.50000 ppm/cm
HZCM              1056.43372 Hz/cm
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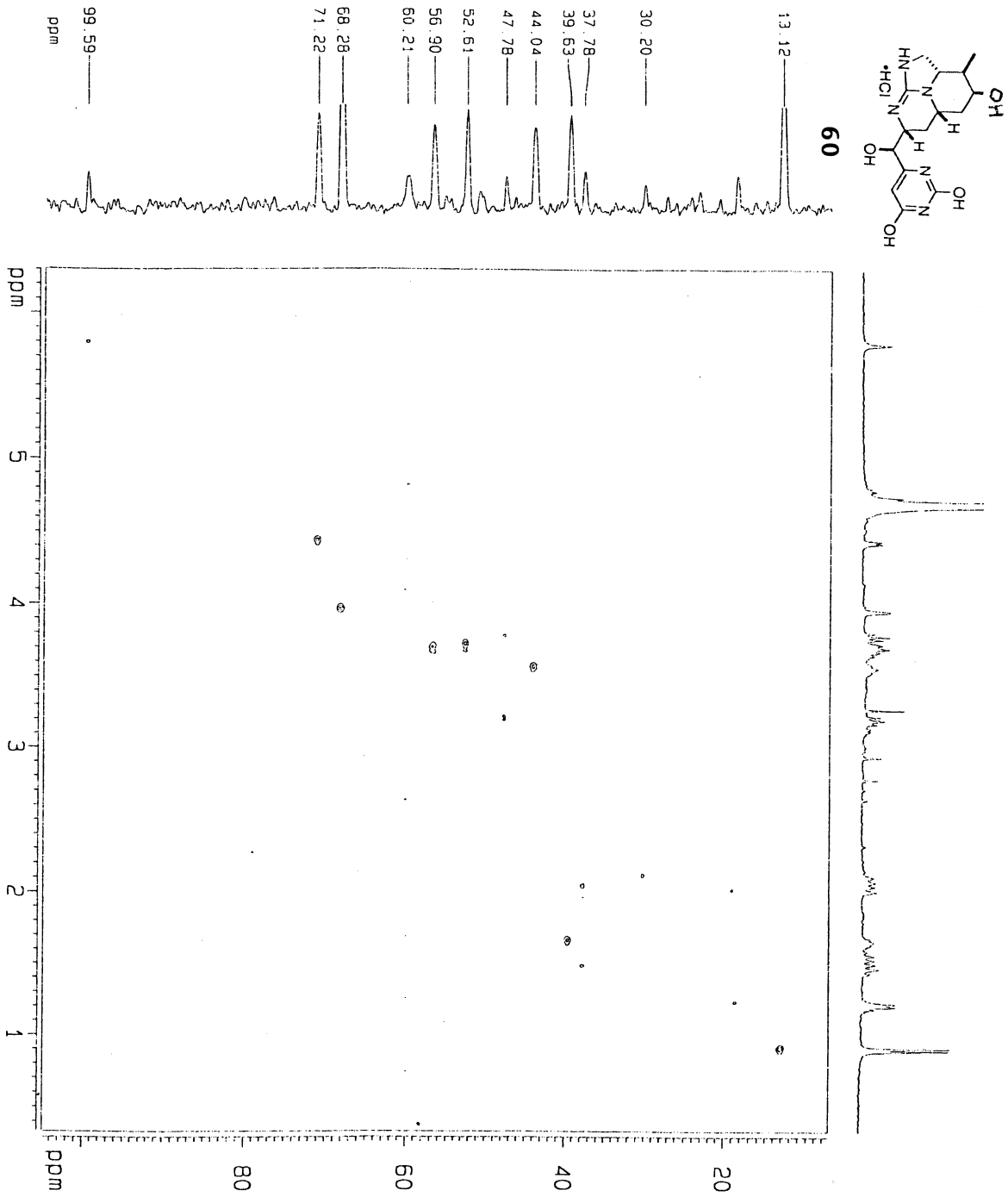
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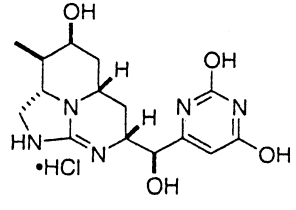
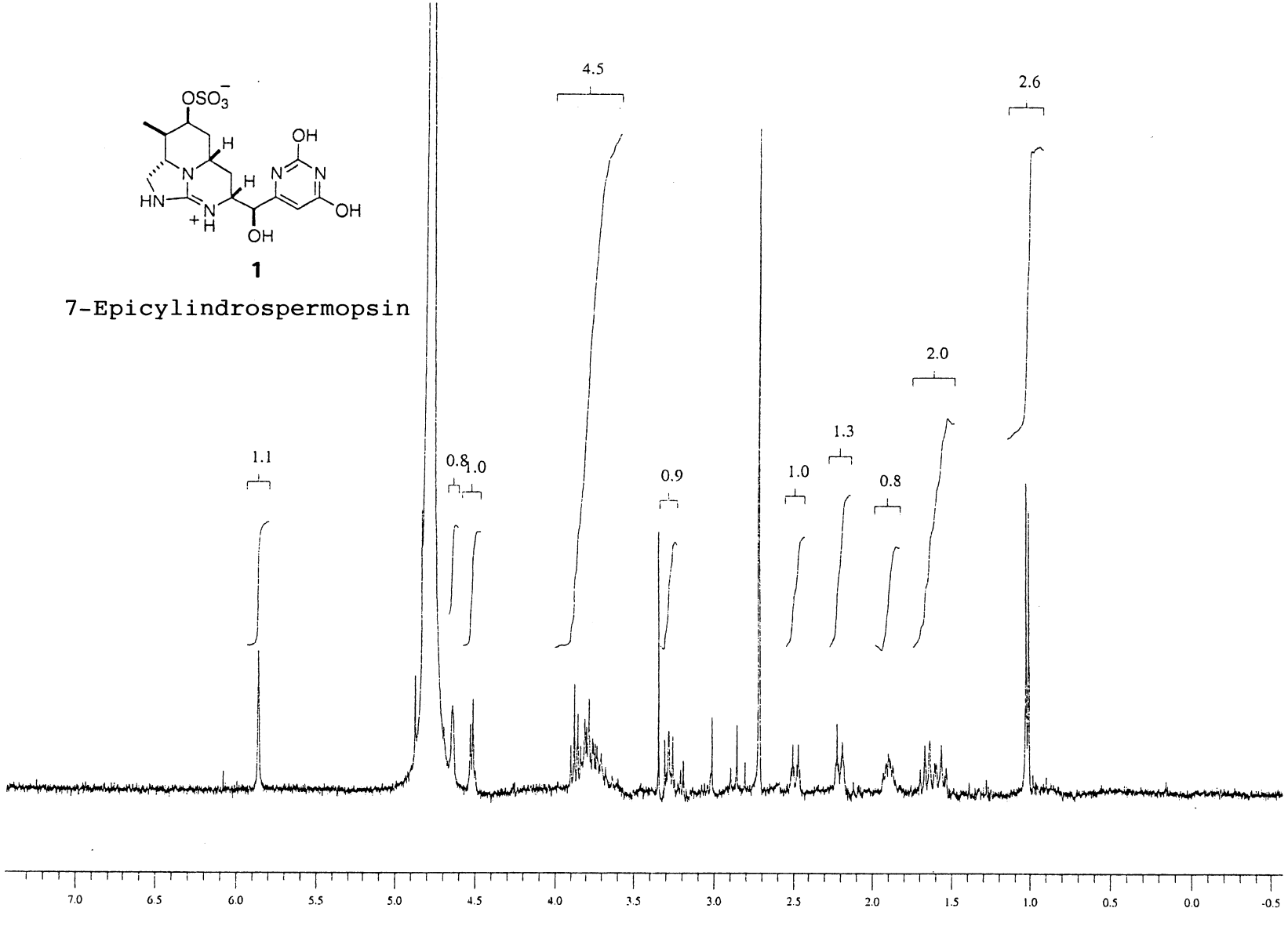
71.22
68.28
60.21
56.90
52.61
47.78
44.04
39.63
37.78
30.20
13.12
ppm
60
•HCl
OH
OH
OH
OH
ppm
1
2
3
4
5
ppm
20
40
60
80


OH
H
OH
N
N
N
H
HN
N
OH
•HCl
OH

60

0.9
0.9
1.1
3.5
1.1
1.5
1.9
3.2
3.4
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5


OSO3−
OH
H
N
N
N
HN
+H
H
OH
OH
1
7-Epicylindrospermopsin
4.5
2.6
2.0
1.3
1.1
0.8
1.0
0.9
1.0
0.8
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5