Supporting Information

Synthesis and Evaluation of Eight- and Four-membered Iminosugar Analogues as Inhibitors of Testicular Ceramide-specific Glucosyltransferase, Testicular β-Glucosidase 2, and other Glycosidases

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REFERENCE NUMBER: 08001

CRYSTAL STRUCTURE REPORT

$2(C_{16}\ H_{33}\ N\ O_5);\ H_2O$

$C_{32}\ H_{68}\ N_2\ O_{11}$

Report prepared for:

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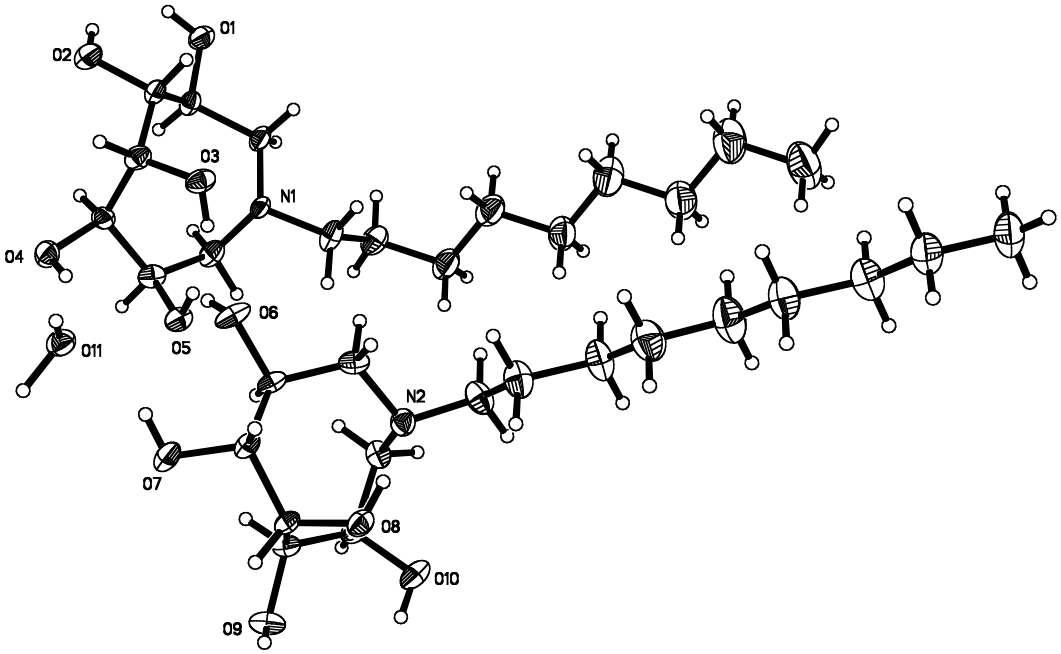

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Data collection

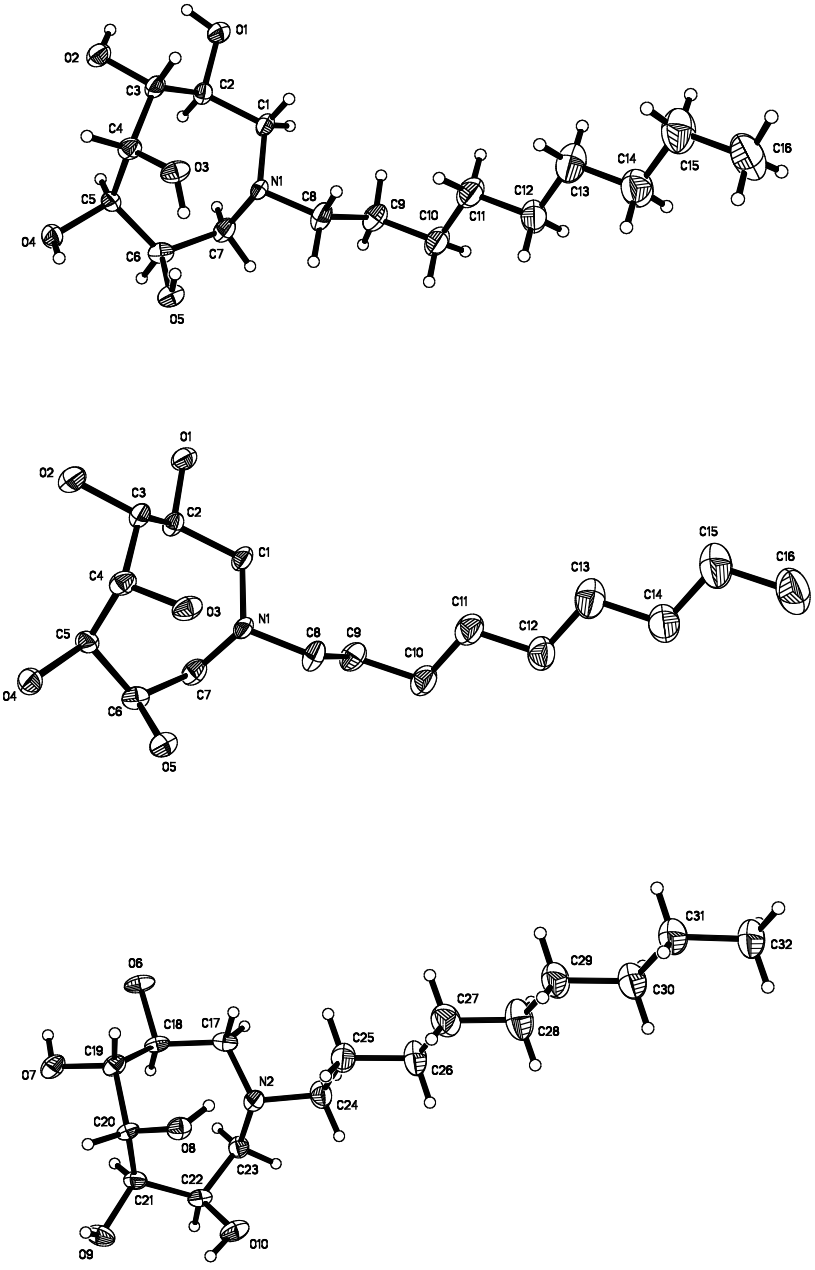
A crystal (approximate dimensions 0.35 x 0.30 x 0.02 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary and mounted on a Bruker SMART Platform CCD diffractometer for a data collection at 173(2) K.[1] A preliminary set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames were oriented such that orthogonal wedges of reciprocal space were surveyed. This produced initial orientation matrices determined from 93 reflections. The data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 120 seconds and a detector distance of 4.86 cm. A randomly oriented region of reciprocal space was surveyed to the extent of one sphere and to a resolution of 0.84 Å. Four major sections of frames were collected with 0.30º steps in ω at four different ϕ settings and a detector position of -28º in 2θ. The intensity data were corrected for absorption and decay (SADABS).[2] Final cell constants were calculated from the xyz centroids of 2524 strong reflections from the actual data collection after integration (SAINT).[3] Please refer to Table 1 for additional crystal and refinement information.

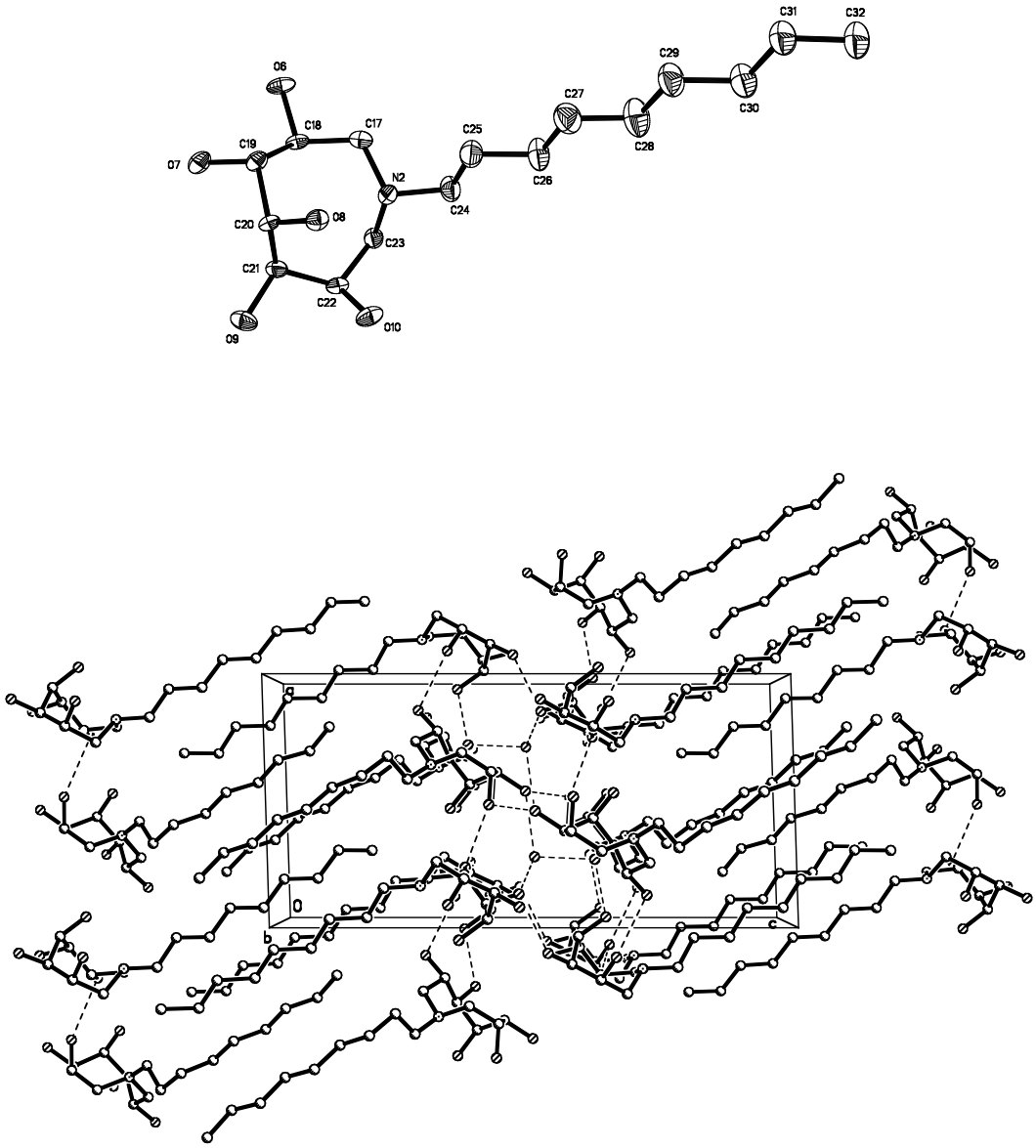
Structure solution and refinement

The structure was solved using SHELXS-97 [4] and refined using SHELXL-97.[4] The space group $P2_1$ was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0527$ and $wR2 = 0.1700$ (F^2, all data).

Structure description

The structure is the one suggested. There are two identical molecules in the asymmetric unit, as well as one water molecule. The absolute stereochemistry cannot be assigned from the diffraction data due to the lack of a heavy atom (> Si); however, the chemist has assigned the stereochemistry based on a known chiral center. The stereochemistry of C2, C3, C5 and C6 are S, R, R, and R respectively; and the stereochemistry of C18, C19, C21 and C22 are S, R, R, and R respectively. The two molecules have several hydrogen bond interactions to each other, as well as to the water molecule.





Matrix Packing Plot (looking down the b-axis, hydrogen atoms omitted for clarity)

Data collection and structure solution were conducted at the X-Ray Crystallographic Laboratory, S146 Kolthoff Hall, Department of Chemistry, University of Minnesota. All calculations were performed using Pentium computers using the current SHELXTL suite of programs. All publications arising from this report MUST either 1)include Benjamin E. Kucera as a coauthor or 2)acknowledge Benjamin E. Kucera, Victor G. Young, Jr., and the X-Ray Crystallographic Laboratory.

References:

1. SMART V5.054, Bruker Analytical X-ray Systems, Madison, WI (2001).
2. An empirical correction for absorption anisotropy, R. Blessing, *Acta Cryst.* **A51**, 33-38 (1995).
3. SAINT+ V6.45, Bruker Analytical X-Ray Systems, Madison, WI (2003).
4. SHELXTL V6.14, Bruker Analytical X-Ray Systems, Madison, WI (2000).

Some equations of interest:

$$R_{int} = \Sigma\ |F_o^2 - < F_o^2 >| \ / \ \Sigma|F_o\ ^2|$$

$$R_1 = \Sigma||F_o|-|F_c|| \ / \ \Sigma|F_o|$$

$$wR2 = [\Sigma[w(F_o^2-F_c\ ^2)^2] \ / \ \Sigma\ [w(F_o^2\ )^2]]^{1/2}$$

$$\text{where } w = q \ / \ [\sigma^2\ (F_o^2) + (a*P)^2 + b*P + d + e*\sin(\theta)]$$

$$\text{GooF} = S = [\Sigma[w(F_o^2-F_c\ ^2)^2] \ / \ (n-p)]^{1/2}$$

Table 1. Crystal data and structure refinement for 08001.

Identification code	08001	
Empirical formula	$C_{32}\ H_{68}\ N_2\ O_{11}$	
Formula weight	656.88	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1$	
Unit cell dimensions	a = 10.3632(13) Å	α = 90°
	b = 8.1773(10) Å	β = 91.751(2)°
	c = 21.547(3) Å	γ = 90°
Volume	1825.1(4) Å^3	
Z	2	
Density (calculated)	1.195 Mg/m^3	
Absorption coefficient	0.088 mm^{-1}	
F(000)	724	
Crystal color, morphology	colorless, plate	
Crystal size	0.35 x 0.30 x 0.02 mm^3	
Theta range for data collection	0.95 to 25.08°	
Index ranges	$-12 \le h \le 12$, $-9 \le k \le 9$, $-25 \le l \le 23$	
Reflections collected	13985	
Independent reflections	3487 [R(int) = 0.0746]	
Observed reflections	2408	
Completeness to theta = 25.08°	99.6%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9982 and 0.9697	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3487 / 1 / 408	
Goodness-of-fit on F^2	1.016	
Final R indices [I>2sigma(I)]	R1 = 0.0527, wR2 = 0.1310	
R indices (all data)	R1 = 0.1022, wR2 = 0.1700	
Absolute structure parameter	0(10)	
Largest diff. peak and hole	0.314 and -0.460 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 08001. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O1	1311(4)	707(5)	7139(2)	29(1)
O2	2682(4)	-818(5)	6198(2)	27(1)
O3	5308(3)	1909(5)	6554(2)	25(1)
O4	4608(4)	1994(5)	5087(2)	26(1)
O5	5152(3)	4703(5)	5785(2)	24(1)
N1	3562(4)	4120(6)	6867(2)	21(1)
C1	2623(6)	3056(7)	7163(3)	25(1)
C2	2174(5)	1622(7)	6764(3)	20(1)
C3	3249(5)	474(7)	6560(3)	23(1)
C4	4322(5)	1164(7)	6169(3)	22(1)
C5	3844(6)	2274(7)	5627(3)	21(1)
C6	3847(5)	4148(7)	5728(3)	22(1)
C7	3102(5)	4796(8)	6270(3)	27(1)
C8	4153(6)	5330(8)	7303(3)	30(1)
C9	3272(6)	6510(7)	7633(3)	31(2)
C10	4040(6)	7555(9)	8102(3)	38(2)
C11	4481(7)	6607(8)	8680(3)	41(2)
C12	5336(7)	7555(10)	9136(3)	41(2)
C13	5735(7)	6591(10)	9719(3)	49(2)
C14	6641(7)	7494(11)	10157(3)	51(2)
C15	7016(8)	6530(11)	10740(4)	58(2)
C16	7977(8)	7383(14)	11162(3)	65(3)
O6	7638(4)	3831(5)	6277(2)	28(1)
O7	8657(4)	5383(5)	5264(2)	27(1)
O8	10348(3)	7959(5)	6390(2)	23(1)
O9	8998(4)	10016(5)	5252(2)	29(1)
O10	8888(4)	10945(5)	6529(2)	30(1)
N2	8263(4)	7804(6)	7073(2)	24(1)
C17	8018(5)	6049(7)	6959(3)	24(1)
C18	7888(5)	5563(7)	6280(3)	22(1)
C19	9029(5)	5854(7)	5881(3)	22(1)

C20	9552(5)	7630(7)	5854(2)	19(1)
C21	8501(5)	8903(7)	5705(3)	23(1)
C22	7942(5)	9882(7)	6240(3)	22(1)
C23	7326(5)	8878(7)	6744(3)	23(1)
C24	8344(6)	8176(8)	7742(3)	32(2)
C25	9493(6)	7443(9)	8084(3)	36(2)
C26	9543(7)	8005(10)	8759(3)	43(2)
C27	10682(7)	7341(10)	9137(3)	46(2)
C28	10719(6)	7865(12)	9809(3)	50(2)
C29	11839(7)	7229(10)	10196(3)	44(2)
C30	11895(6)	7800(11)	10869(3)	45(2)
C31	13007(7)	7106(10)	11260(3)	49(2)
C32	13006(8)	7702(12)	11926(3)	61(2)
O11	7251(3)	2615(5)	4946(2)	26(1)

Table 3. Bond lengths [Å] and angles [°] for 08001.

O(1)-C(2)	1.435(7)	C(10)-C(11)	1.525(9)
O(1)-H(1C)	0.8400	C(10)-H(10A)	0.9900
O(2)-C(3)	1.430(7)	C(10)-H(10B)	0.9900
O(2)-H(2B)	0.8400	C(11)-C(12)	1.516(9)
O(3)-C(4)	1.432(6)	C(11)-H(11A)	0.9900
O(3)-H(3B)	0.8400	C(11)-H(11B)	0.9900
O(4)-C(5)	1.445(6)	C(12)-C(13)	1.530(10)
O(4)-H(4B)	0.8400	C(12)-H(12A)	0.9900
O(5)-C(6)	1.429(7)	C(12)-H(12B)	0.9900
O(5)-H(5B)	0.8400	C(13)-C(14)	1.504(10)
N(1)-C(1)	1.466(7)	C(13)-H(13A)	0.9900
N(1)-C(7)	1.467(7)	C(13)-H(13B)	0.9900
N(1)-C(8)	1.483(7)	C(14)-C(15)	1.524(10)
C(1)-C(2)	1.520(8)	C(14)-H(14A)	0.9900
C(1)-H(1A)	0.9900	C(14)-H(14B)	0.9900
C(1)-H(1B)	0.9900	C(15)-C(16)	1.499(11)
C(2)-C(3)	1.531(8)	C(15)-H(15A)	0.9900
C(2)-H(2A)	1.0000	C(15)-H(15B)	0.9900
C(3)-C(4)	1.525(8)	C(16)-H(16A)	0.9800
C(3)-H(3A)	1.0000	C(16)-H(16B)	0.9800
C(4)-C(5)	1.548(8)	C(16)-H(16C)	0.9800
C(4)-H(4A)	1.0000	O(6)-C(18)	1.439(7)
C(5)-C(6)	1.547(8)	O(6)-H(6B)	0.8400
C(5)-H(5A)	1.0000	O(7)-C(19)	1.426(7)
C(6)-C(7)	1.515(8)	O(7)-H(7C)	0.8400
C(6)-H(6A)	1.0000	O(8)-C(20)	1.424(6)
C(7)-H(7A)	0.9900	O(8)-H(8C)	0.8400
C(7)-H(7B)	0.9900	O(9)-C(21)	1.441(7)
C(8)-C(9)	1.520(8)	O(9)-H(9C)	0.8400
C(8)-H(8A)	0.9900	O(10)-C(22)	1.438(7)
C(8)-H(8B)	0.9900	O(10)-H(10C)	0.8400
C(9)-C(10)	1.529(8)	N(2)-C(24)	1.473(7)
C(9)-H(9A)	0.9900	N(2)-C(23)	1.475(7)
C(9)-H(9B)	0.9900	N(2)-C(17)	1.476(8)

C(17)-C(18)	1.518(8)	C(31)-C(32)	1.514(10)
C(17)-H(17A)	0.9900	C(31)-H(31A)	0.9900
C(17)-H(17B)	0.9900	C(31)-H(31B)	0.9900
C(18)-C(19)	1.502(8)	C(32)-H(32A)	0.9800
C(18)-H(18A)	1.0000	C(32)-H(32B)	0.9800
C(19)-C(20)	1.552(8)	C(32)-H(32C)	0.9800
C(19)-H(19A)	1.0000	O(11)-H(11C)	1.0679
C(20)-C(21)	1.535(8)	O(11)-H(11D)	0.8495
C(20)-H(20A)	1.0000		
C(21)-C(22)	1.532(8)	C(2)-O(1)-H(1C)	109.5
C(21)-H(21A)	1.0000	C(3)-O(2)-H(2B)	109.5
C(22)-C(23)	1.516(8)	C(4)-O(3)-H(3B)	109.5
C(22)-H(22A)	1.0000	C(5)-O(4)-H(4B)	109.5
C(23)-H(23A)	0.9900	C(6)-O(5)-H(5B)	109.5
C(23)-H(23B)	0.9900	C(1)-N(1)-C(7)	113.8(4)
C(24)-C(25)	1.505(8)	C(1)-N(1)-C(8)	112.8(4)
C(24)-H(24A)	0.9900	C(7)-N(1)-C(8)	114.9(5)
C(24)-H(24B)	0.9900	N(1)-C(1)-C(2)	114.0(4)
C(25)-C(26)	1.524(8)	N(1)-C(1)-H(1A)	108.7
C(25)-H(25A)	0.9900	C(2)-C(1)-H(1A)	108.7
C(25)-H(25B)	0.9900	N(1)-C(1)-H(1B)	108.7
C(26)-C(27)	1.515(9)	C(2)-C(1)-H(1B)	108.7
C(26)-H(26A)	0.9900	H(1A)-C(1)-H(1B)	107.6
C(26)-H(26B)	0.9900	O(1)-C(2)-C(1)	105.6(4)
C(27)-C(28)	1.509(9)	O(1)-C(2)-C(3)	108.3(5)
C(27)-H(27A)	0.9900	C(1)-C(2)-C(3)	115.0(4)
C(27)-H(27B)	0.9900	O(1)-C(2)-H(2A)	109.3
C(28)-C(29)	1.502(9)	C(1)-C(2)-H(2A)	109.3
C(28)-H(28A)	0.9900	C(3)-C(2)-H(2A)	109.3
C(28)-H(28B)	0.9900	O(2)-C(3)-C(4)	105.4(4)
C(29)-C(30)	1.523(9)	O(2)-C(3)-C(2)	108.6(4)
C(29)-H(29A)	0.9900	C(4)-C(3)-C(2)	118.8(5)
C(29)-H(29B)	0.9900	O(2)-C(3)-H(3A)	107.9
C(30)-C(31)	1.517(9)	C(4)-C(3)-H(3A)	107.9
C(30)-H(30A)	0.9900	C(2)-C(3)-H(3A)	107.9
C(30)-H(30B)	0.9900	O(3)-C(4)-C(3)	110.9(4)

O(3)-C(4)-C(5)	113.2(4)	C(11)-C(10)-H(10A)	109.0
C(3)-C(4)-C(5)	114.3(4)	C(9)-C(10)-H(10A)	109.0
O(3)-C(4)-H(4A)	105.9	C(11)-C(10)-H(10B)	109.0
C(3)-C(4)-H(4A)	105.9	C(9)-C(10)-H(10B)	109.0
C(5)-C(4)-H(4A)	105.9	H(10A)-C(10)-H(10B)	107.8
O(4)-C(5)-C(6)	105.8(4)	C(12)-C(11)-C(10)	115.1(6)
O(4)-C(5)-C(4)	110.1(4)	C(12)-C(11)-H(11A)	108.5
C(6)-C(5)-C(4)	118.4(5)	C(10)-C(11)-H(11A)	108.5
O(4)-C(5)-H(5A)	107.4	C(12)-C(11)-H(11B)	108.5
C(6)-C(5)-H(5A)	107.4	C(10)-C(11)-H(11B)	108.5
C(4)-C(5)-H(5A)	107.4	H(11A)-C(11)-H(11B)	107.5
O(5)-C(6)-C(7)	109.0(5)	C(11)-C(12)-C(13)	114.0(6)
O(5)-C(6)-C(5)	108.9(5)	C(11)-C(12)-H(12A)	108.7
C(7)-C(6)-C(5)	117.1(5)	C(13)-C(12)-H(12A)	108.7
O(5)-C(6)-H(6A)	107.1	C(11)-C(12)-H(12B)	108.7
C(7)-C(6)-H(6A)	107.1	C(13)-C(12)-H(12B)	108.7
C(5)-C(6)-H(6A)	107.1	H(12A)-C(12)-H(12B)	107.6
N(1)-C(7)-C(6)	112.7(5)	C(14)-C(13)-C(12)	114.2(7)
N(1)-C(7)-H(7A)	109.1	C(14)-C(13)-H(13A)	108.7
C(6)-C(7)-H(7A)	109.1	C(12)-C(13)-H(13A)	108.7
N(1)-C(7)-H(7B)	109.1	C(14)-C(13)-H(13B)	108.7
C(6)-C(7)-H(7B)	109.1	C(12)-C(13)-H(13B)	108.7
H(7A)-C(7)-H(7B)	107.8	H(13A)-C(13)-H(13B)	107.6
N(1)-C(8)-C(9)	118.5(5)	C(13)-C(14)-C(15)	113.7(7)
N(1)-C(8)-H(8A)	107.7	C(13)-C(14)-H(14A)	108.8
C(9)-C(8)-H(8A)	107.7	C(15)-C(14)-H(14A)	108.8
N(1)-C(8)-H(8B)	107.7	C(13)-C(14)-H(14B)	108.8
C(9)-C(8)-H(8B)	107.7	C(15)-C(14)-H(14B)	108.8
H(8A)-C(8)-H(8B)	107.1	H(14A)-C(14)-H(14B)	107.7
C(8)-C(9)-C(10)	110.9(5)	C(16)-C(15)-C(14)	114.0(8)
C(8)-C(9)-H(9A)	109.5	C(16)-C(15)-H(15A)	108.7
C(10)-C(9)-H(9A)	109.5	C(14)-C(15)-H(15A)	108.7
C(8)-C(9)-H(9B)	109.5	C(16)-C(15)-H(15B)	108.7
C(10)-C(9)-H(9B)	109.5	C(14)-C(15)-H(15B)	108.7
H(9A)-C(9)-H(9B)	108.0	H(15A)-C(15)-H(15B)	107.6
C(11)-C(10)-C(9)	113.1(6)	C(15)-C(16)-H(16A)	109.5

C(15)-C(16)-H(16B)	109.5	C(19)-C(20)-H(20A)	105.7
H(16A)-C(16)-H(16B)	109.5	O(9)-C(21)-C(22)	109.4(4)
C(15)-C(16)-H(16C)	109.5	O(9)-C(21)-C(20)	107.7(4)
H(16A)-C(16)-H(16C)	109.5	C(22)-C(21)-C(20)	118.6(5)
H(16B)-C(16)-H(16C)	109.5	O(9)-C(21)-H(21A)	106.9
C(18)-O(6)-H(6B)	109.5	C(22)-C(21)-H(21A)	106.9
C(19)-O(7)-H(7C)	109.5	C(20)-C(21)-H(21A)	106.9
C(20)-O(8)-H(8C)	109.5	O(10)-C(22)-C(23)	108.3(5)
C(21)-O(9)-H(9C)	109.5	O(10)-C(22)-C(21)	111.9(4)
C(22)-O(10)-H(10C)	109.5	C(23)-C(22)-C(21)	115.7(5)
C(24)-N(2)-C(23)	111.4(4)	O(10)-C(22)-H(22A)	106.8
C(24)-N(2)-C(17)	111.6(5)	C(23)-C(22)-H(22A)	106.8
C(23)-N(2)-C(17)	113.0(5)	C(21)-C(22)-H(22A)	106.8
N(2)-C(17)-C(18)	115.1(5)	N(2)-C(23)-C(22)	112.4(5)
N(2)-C(17)-H(17A)	108.5	N(2)-C(23)-H(23A)	109.1
C(18)-C(17)-H(17A)	108.5	C(22)-C(23)-H(23A)	109.1
N(2)-C(17)-H(17B)	108.5	N(2)-C(23)-H(23B)	109.1
C(18)-C(17)-H(17B)	108.5	C(22)-C(23)-H(23B)	109.1
H(17A)-C(17)-H(17B)	107.5	H(23A)-C(23)-H(23B)	107.9
O(6)-C(18)-C(19)	107.3(5)	N(2)-C(24)-C(25)	114.7(5)
O(6)-C(18)-C(17)	105.9(5)	N(2)-C(24)-H(24A)	108.6
C(19)-C(18)-C(17)	117.5(5)	C(25)-C(24)-H(24A)	108.6
O(6)-C(18)-H(18A)	108.6	N(2)-C(24)-H(24B)	108.6
C(19)-C(18)-H(18A)	108.6	C(25)-C(24)-H(24B)	108.6
C(17)-C(18)-H(18A)	108.6	H(24A)-C(24)-H(24B)	107.6
O(7)-C(19)-C(18)	107.2(4)	C(24)-C(25)-C(26)	110.5(5)
O(7)-C(19)-C(20)	107.6(4)	C(24)-C(25)-H(25A)	109.6
C(18)-C(19)-C(20)	116.9(5)	C(26)-C(25)-H(25A)	109.6
O(7)-C(19)-H(19A)	108.3	C(24)-C(25)-H(25B)	109.6
C(18)-C(19)-H(19A)	108.3	C(26)-C(25)-H(25B)	109.6
C(20)-C(19)-H(19A)	108.3	H(25A)-C(25)-H(25B)	108.1
O(8)-C(20)-C(21)	115.6(4)	C(27)-C(26)-C(25)	114.1(6)
O(8)-C(20)-C(19)	109.9(4)	C(27)-C(26)-H(26A)	108.7
C(21)-C(20)-C(19)	113.3(4)	C(25)-C(26)-H(26A)	108.7
O(8)-C(20)-H(20A)	105.7	C(27)-C(26)-H(26B)	108.7
C(21)-C(20)-H(20A)	105.7	C(25)-C(26)-H(26B)	108.7

H(26A)-C(26)-H(26B)	107.6	C(31)-C(30)-H(30A)	108.6
C(28)-C(27)-C(26)	114.3(6)	C(29)-C(30)-H(30A)	108.6
C(28)-C(27)-H(27A)	108.7	C(31)-C(30)-H(30B)	108.6
C(26)-C(27)-H(27A)	108.7	C(29)-C(30)-H(30B)	108.6
C(28)-C(27)-H(27B)	108.7	H(30A)-C(30)-H(30B)	107.5
C(26)-C(27)-H(27B)	108.7	C(32)-C(31)-C(30)	112.6(7)
H(27A)-C(27)-H(27B)	107.6	C(32)-C(31)-H(31A)	109.1
C(29)-C(28)-C(27)	115.5(6)	C(30)-C(31)-H(31A)	109.1
C(29)-C(28)-H(28A)	108.4	C(32)-C(31)-H(31B)	109.1
C(27)-C(28)-H(28A)	108.4	C(30)-C(31)-H(31B)	109.1
C(29)-C(28)-H(28B)	108.4	H(31A)-C(31)-H(31B)	107.8
C(27)-C(28)-H(28B)	108.4	C(31)-C(32)-H(32A)	109.5
H(28A)-C(28)-H(28B)	107.5	C(31)-C(32)-H(32B)	109.5
C(28)-C(29)-C(30)	115.4(6)	H(32A)-C(32)-H(32B)	109.5
C(28)-C(29)-H(29A)	108.4	C(31)-C(32)-H(32C)	109.5
C(30)-C(29)-H(29A)	108.4	H(32A)-C(32)-H(32C)	109.5
C(28)-C(29)-H(29B)	108.4	H(32B)-C(32)-H(32C)	109.5
C(30)-C(29)-H(29B)	108.4	H(11C)-O(11)-H(11D)	102.2
H(29A)-C(29)-H(29B)	107.5		
C(31)-C(30)-C(29)	114.9(6)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 08001. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	35(2)	22(2)	31(2)	-3(2)	8(2)	-8(2)
O2	26(2)	22(2)	34(2)	-3(2)	5(2)	-3(2)
O3	20(2)	22(2)	33(2)	5(2)	-6(2)	-4(2)
O4	26(2)	28(2)	26(2)	-6(2)	8(2)	-5(2)
O5	21(2)	20(2)	30(2)	3(2)	-5(2)	-1(2)
N1	27(3)	15(2)	21(2)	-6(2)	4(2)	-5(2)
C1	30(3)	20(3)	25(3)	-8(3)	5(3)	-6(3)
C2	15(3)	22(3)	24(3)	-6(2)	4(2)	-5(2)
C3	28(3)	18(3)	21(3)	-4(2)	-3(2)	-4(2)
C4	18(3)	21(3)	28(3)	-1(3)	4(2)	-3(2)
C5	26(3)	19(3)	19(3)	0(2)	7(2)	0(2)
C6	15(3)	23(3)	28(3)	4(3)	-2(2)	0(2)
C7	27(3)	19(3)	34(4)	-6(3)	2(3)	0(3)
C8	29(3)	27(3)	33(3)	-12(3)	-2(3)	-4(3)
C9	31(3)	23(3)	39(4)	-6(3)	-1(3)	3(3)
C10	46(4)	27(3)	40(4)	-9(3)	-3(3)	-3(3)
C11	51(4)	28(4)	43(4)	-6(3)	-5(3)	-2(3)
C12	46(4)	45(4)	32(4)	-10(4)	2(3)	-4(4)
C13	56(5)	42(4)	49(4)	-10(4)	-14(4)	7(4)
C14	51(4)	62(5)	39(4)	-3(4)	-3(3)	-6(4)
C15	61(5)	62(6)	49(5)	-7(4)	-16(4)	10(4)
C16	57(5)	103(8)	35(4)	-3(5)	2(4)	-8(5)
O6	19(2)	14(2)	49(3)	2(2)	-3(2)	-4(2)
O7	26(2)	22(2)	33(2)	-8(2)	0(2)	-6(2)
O8	21(2)	22(2)	27(2)	-1(2)	-3(2)	-4(2)
O9	24(2)	27(2)	37(3)	11(2)	8(2)	1(2)
O10	27(2)	18(2)	43(3)	-3(2)	-6(2)	-4(2)
N2	29(3)	21(3)	22(3)	-3(2)	0(2)	2(2)
C17	21(3)	20(3)	30(3)	6(3)	2(2)	1(2)
C18	22(3)	12(3)	32(3)	1(3)	0(3)	-3(2)
C19	20(3)	19(3)	28(3)	-4(3)	0(2)	-5(2)

C20	21(3)	11(3)	25(3)	-1(3)	0(2)	-1(2)
C21	24(3)	17(3)	26(3)	3(3)	3(2)	-2(3)
C22	20(3)	15(3)	29(3)	2(3)	-3(3)	0(2)
C23	24(3)	24(3)	21(3)	-1(3)	0(2)	6(3)
C24	35(3)	38(4)	23(3)	-1(3)	4(3)	7(3)
C25	40(4)	39(4)	29(3)	-5(3)	-3(3)	8(3)
C26	47(4)	57(5)	25(3)	-6(4)	-3(3)	3(4)
C27	49(4)	50(5)	37(4)	1(3)	-7(3)	8(4)
C28	39(4)	76(6)	35(4)	-5(4)	-5(3)	5(4)
C29	45(4)	55(5)	33(4)	-2(3)	-10(3)	3(4)
C30	48(4)	56(5)	32(4)	-1(4)	-3(3)	3(4)
C31	60(5)	50(5)	35(4)	-2(4)	-12(4)	-6(4)
C32	87(6)	57(5)	37(4)	-5(4)	-21(4)	-12(5)
O11	29(2)	19(2)	30(2)	0(2)	2(2)	0(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 08001.

	x	y	z	U(eq)
H1C	1209	-234	6989	44
H2B	1947	-1036	6329	41
H3B	5470	2847	6418	37
H4B	5374	2282	5164	39
H5B	5578	4068	6019	36
H1A	1861	3718	7270	30
H1B	3014	2628	7556	30
H2A	1691	2043	6388	24
H3A	3658	-22	6942	27
H4A	4737	199	5972	27
H5A	2937	1943	5516	25
H6A	3460	4654	5343	26
H7A	3181	6002	6283	32
H7B	2177	4525	6205	32
H8A	4776	5987	7069	36
H8B	4655	4718	7625	36
H9A	2603	5886	7850	38
H9B	2832	7229	7323	38
H10A	4807	8006	7900	45
H10B	3498	8487	8229	45
H11A	3707	6232	8898	49
H11B	4955	5622	8547	49
H12A	4873	8555	9262	49
H12B	6124	7902	8924	49
H13A	4948	6297	9944	59
H13B	6153	5561	9592	59
H14A	6229	8532	10281	61
H14B	7434	7773	9935	61
H15A	6227	6305	10974	69
H15B	7381	5465	10616	69

H16A	8256	6640	11497	98
H16B	7576	8354	11340	98
H16C	8726	7711	10925	98
H6B	6884	3658	6139	41
H7C	8242	4501	5274	41
H8C	9982	7617	6708	35
H9C	9777	9797	5193	44
H10C	9350	11355	6256	44
H17A	7215	5739	7167	28
H17B	8733	5413	7156	28
H18A	7122	6137	6090	27
H19A	9747	5123	6030	26
H20A	10142	7655	5496	23
H21A	7764	8306	5498	27
H22A	7250	10599	6055	26
H23A	6934	9625	7046	28
H23B	6627	8201	6554	28
H24A	7549	7776	7935	38
H24B	8372	9378	7795	38
H25A	10294	7778	7880	43
H25B	9436	6235	8068	43
H26A	9576	9215	8768	52
H26B	8737	7662	8956	52
H27A	10660	6132	9119	55
H27B	11488	7705	8944	55
H28A	10735	9074	9824	60
H28B	9910	7501	9999	60
H29A	12648	7564	9999	53
H29B	11808	6019	10191	53
H30A	11957	9008	10874	54
H30B	11075	7497	11063	54
H31A	12952	5897	11257	59
H31B	13831	7421	11073	59
H32A	13740	7222	12158	91
H32B	12200	7373	12116	91
H32C	13079	8897	11933	91

H11C	7564	2984	4500	39
H11D	7769	1840	5037	39

Table 6. Torsion angles [°] for 08001.

C7-N1-C1-C2	56.9(6)	C23-N2-C17-C18	52.1(6)
C8-N1-C1-C2	-169.9(5)	N2-C17-C18-O6	-179.2(4)
N1-C1-C2-O1	177.9(5)	N2-C17-C18-C19	61.0(7)
N1-C1-C2-C3	58.6(7)	O6-C18-C19-O7	63.8(6)
O1-C2-C3-O2	61.4(5)	C17-C18-C19-O7	-177.1(5)
C1-C2-C3-O2	179.2(5)	O6-C18-C19-C20	-175.3(5)
O1-C2-C3-C4	-178.2(4)	C17-C18-C19-C20	-56.3(7)
C1-C2-C3-C4	-60.5(7)	O7-C19-C20-O8	-159.1(4)
O2-C3-C4-O3	-154.1(4)	C18-C19-C20-O8	80.2(6)
C2-C3-C4-O3	84.0(6)	O7-C19-C20-C21	69.8(5)
O2-C3-C4-C5	76.5(6)	C18-C19-C20-C21	-50.8(7)
C2-C3-C4-C5	-45.5(7)	O8-C20-C21-O9	95.9(5)
O3-C4-C5-O4	90.0(5)	C19-C20-C21-O9	-135.9(5)
C3-C4-C5-O4	-141.7(5)	O8-C20-C21-C22	-28.9(7)
O3-C4-C5-C6	-31.8(7)	C19-C20-C21-C22	99.3(6)
C3-C4-C5-C6	96.5(6)	O9-C21-C22-O10	-57.8(6)
O4-C5-C6-O5	-56.0(6)	C20-C21-C22-O10	66.1(6)
C4-C5-C6-O5	67.9(6)	O9-C21-C22-C23	177.5(4)
O4-C5-C6-C7	179.7(4)	C20-C21-C22-C23	-58.6(7)
C4-C5-C6-C7	-56.3(7)	C24-N2-C23-C22	120.4(5)
C1-N1-C7-C6	-113.2(5)	C17-N2-C23-C22	-113.0(5)
C8-N1-C7-C6	114.6(5)	O10-C22-C23-N2	-63.0(6)
O5-C6-C7-N1	-65.5(6)	C21-C22-C23-N2	63.5(7)
C5-C6-C7-N1	58.7(7)	C23-N2-C24-C25	-165.6(5)
C1-N1-C8-C9	-58.1(7)	C17-N2-C24-C25	67.0(7)
C7-N1-C8-C9	74.5(7)	N2-C24-C25-C26	175.7(6)
N1-C8-C9-C10	175.0(5)	C24-C25-C26-C27	-179.2(6)
C8-C9-C10-C11	-73.0(7)	C25-C26-C27-C28	-178.7(7)
C9-C10-C11-C12	175.3(6)	C26-C27-C28-C29	-179.9(7)
C10-C11-C12-C13	178.4(6)	C27-C28-C29-C30	178.3(7)
C11-C12-C13-C14	176.9(6)	C28-C29-C30-C31	178.1(7)
C12-C13-C14-C15	179.2(6)	C29-C30-C31-C32	-179.5(7)
C13-C14-C15-C16	176.8(7)		
C24-N2-C17-C18	178.6(4)		

Table 7. Hydrogen bonds for 08001 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O1-H1C...O8#1	0.84	2.14	2.924(5)	155.5
O1-H1C...O2	0.84	2.37	2.805(5)	112.7
O2-H2B...O8#1	0.84	1.86	2.662(5)	159.6
O2-H2B...O1	0.84	2.36	2.805(5)	113.5
O3-H3B...O5	0.84	2.06	2.825(5)	150.9
O3-H3B...O6	0.84	2.41	2.958(5)	123.0
O4-H4B...O11	0.84	2.03	2.811(5)	153.8
O5-H5B...O3	0.84	2.13	2.825(5)	139.6
O5-H5B...O6	0.84	2.20	2.847(5)	134.0
O6-H6B...O5	0.84	2.11	2.847(5)	146.3
O6-H6B...O3	0.84	2.37	2.958(5)	127.8
O7-H7C...O11	0.84	1.97	2.767(5)	157.6
O7-H7C...O6	0.84	2.33	2.762(6)	112.1
O8-H8C...N2	0.84	1.98	2.654(6)	137.2
O9-H9C...O7#2	0.84	1.98	2.719(5)	145.9
O10-H10C...O9	0.84	2.44	2.861(6)	111.7
O11-H11C...O2#3	1.07	1.81	2.781(5)	149.8
O11-H11D...O9#4	0.85	2.01	2.856(6)	179.8

Symmetry transformations used to generate equivalent atoms:

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

#4 x,y-1,z

checkCIF/PLATON report

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 08001

Bond precision: C-C = 0.0092 A Wavelength=0.71073

Cell: a=10.3632(13) b=8.1773(10) c=21.547(3)

alpha=90 beta=91.751(2) gamma=90

	Calculated	Reported
Volume	1825.1(4)	1825.1(4)
Space group	P 21	P2(1)
Hall group	P 2yb	?
Moiety formula	$2(C_{16}H_{33}NO_5)$, H_2O	$2(C_{16}H_{33}NO_5)$, H_2O
Sum formula	$C_{32}H_{68}N_2O_{11}$	$C_{32}H_{68}N_2O_{11}$
Mr	656.88	656.88
Dx,g cm^{-3}	1.195	1.195
Z	2	2
Mu (mm^{-1})	0.088	0.088
F000	724.0	724.0
F000’	724.38	
h,k,lmax	12,9,25	12,9,25
Nref	3501(6500)	3487
Tmin,Tmax	0.970,0.998	0.970,0.998
Tmin’	0.970	

Correction method= AbsCorr=MULTI-SCAN
Data completeness= 1.00(0.54) Theta(max)= 25.080
R(reflections)= 0.0527(2408) wR2(reflections)= 0.1700(3487)
S = 1.016 Npar= 408

The following ALERTS were generated. Each ALERT has the format test-name_ALERT_alert-type_alert-level.

Alert level B

PLAT222_ALERT_3_B Large Non-Solvent H Ueq(max)/Ueq(min) ... 4.08 Ratio
PLAT416_ALERT_2_B Short Intra D-H..H-D H1C .. H2B .. 1.76 Ang.
PLAT416_ALERT_2_B Short Intra D-H..H-D H3B .. H5B .. 1.32 Ang.
PLAT417_ALERT_2_B Short Inter D-H..H-D H2B .. H11C .. 2.04 Ang.
PLAT417_ALERT_2_B Short Inter D-H..H-D H3B .. H6B .. 1.73 Ang.
PLAT417_ALERT_2_B Short Inter D-H..H-D H5B .. H6B .. 1.41 Ang.

Alert level C

STRVA01_ALERT_4_C Flack test results are meaningless.
From the CIF: _refine_ls_abs_structure_Flack 0.000
From the CIF: _refine_ls_abs_structure_Flack_su 10.000
PLAT032_ALERT_4_C Std. Uncertainty in Flack Parameter too High ... 10.00
PLAT066_ALERT_1_C Predicted and Reported Transmissions Identical . ?
PLAT220_ALERT_2_C Large Non-Solvent C Ueq(max)/Ueq(min) ... 3.20 Ratio
PLAT220_ALERT_2_C Large Non-Solvent C Ueq(max)/Ueq(min) ... 3.21 Ratio
PLAT222_ALERT_3_C Large Non-Solvent H Ueq(max)/Ueq(min) ... 3.96 Ratio
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds (x 1000) Ang ... 9
PLAT355_ALERT_3_C Long O-H Bond (0.82A) O11 - H11C ... 1.07 Ang.
PLAT414_ALERT_2_C Short Intra D-H..H-X H9C .. H20A .. 1.90 Ang.
PLAT850_ALERT_2_C Check Flack Parameter Exact Value 0.00 and su .. 10.00

Alert level G

REFLT03_ALERT_4_G Please check that the estimate of the number of Friedel pairs is correct. If it is not, please give the correct count in the _publ_section_exptl_refinement section of the submitted CIF.

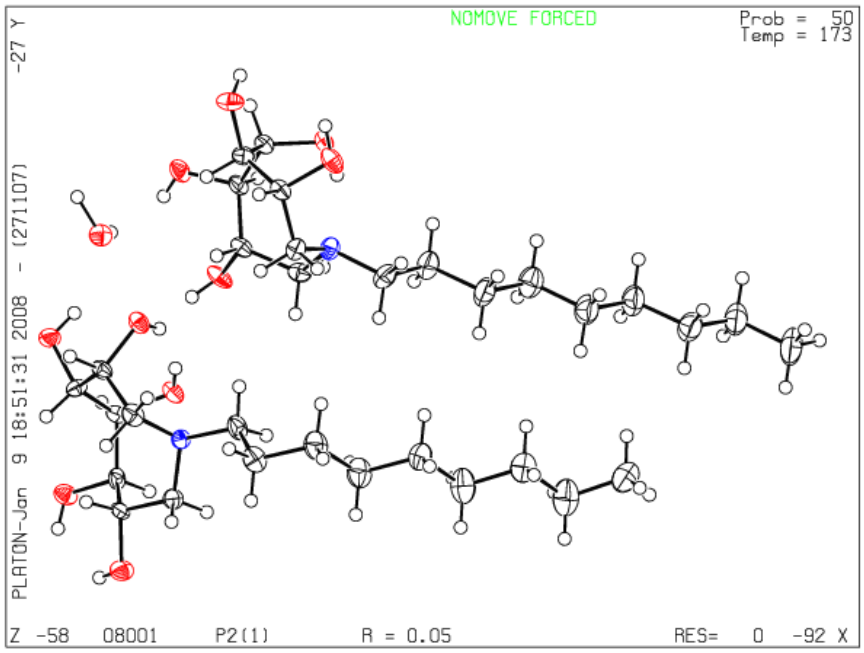
From the CIF: _diffrn_reflns_theta_max 25.08
From the CIF: _reflns_number_total 3487
Count of symmetry unique reflns 3501
Completeness (_total/calc) 99.60%
TEST3: Check Friedels for noncentro structure
Estimate of Friedel pairs measured 0
Fraction of Friedel pairs measured 0.000
Are heavy atom types Z>Si present no
PLAT791_ALERT_1_G Confirm the Absolute Configuration of C2 ... S
PLAT791_ALERT_1_G Confirm the Absolute Configuration of C3 ... R
PLAT791_ALERT_1_G Confirm the Absolute Configuration of C5 ... R
PLAT791_ALERT_1_G Confirm the Absolute Configuration of C6 ... R
PLAT791_ALERT_1_G Confirm the Absolute Configuration of C18 ... S
PLAT791_ALERT_1_G Confirm the Absolute Configuration of C19 ... R
PLAT791_ALERT_1_G Confirm the Absolute Configuration of C21 ... R
PLAT791_ALERT_1_G Confirm the Absolute Configuration of C22 ... R

PLAT860_ALERT_3_G Note: Number of Least-Squares Restraints 1

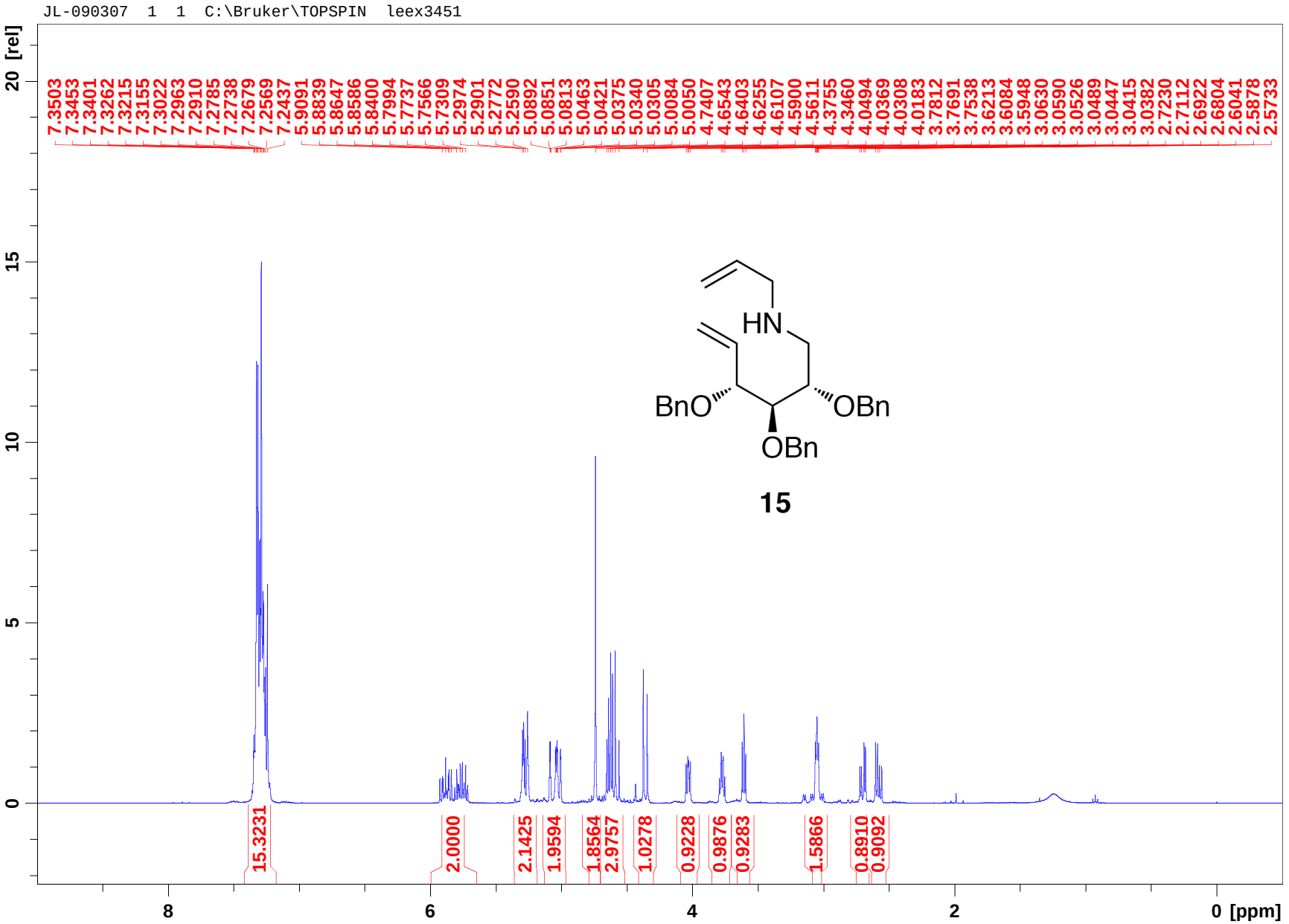
0 ALERT level A = In general: serious problem

6 ALERT level B = Potentially serious problem
10 ALERT level C = Check and explain
10 ALERT level G = General alerts; check

9 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
9 ALERT type 2 Indicator that the structure model may be wrong or deficient
5 ALERT type 3 Indicator that the structure quality may be low
3 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check



1H-NMR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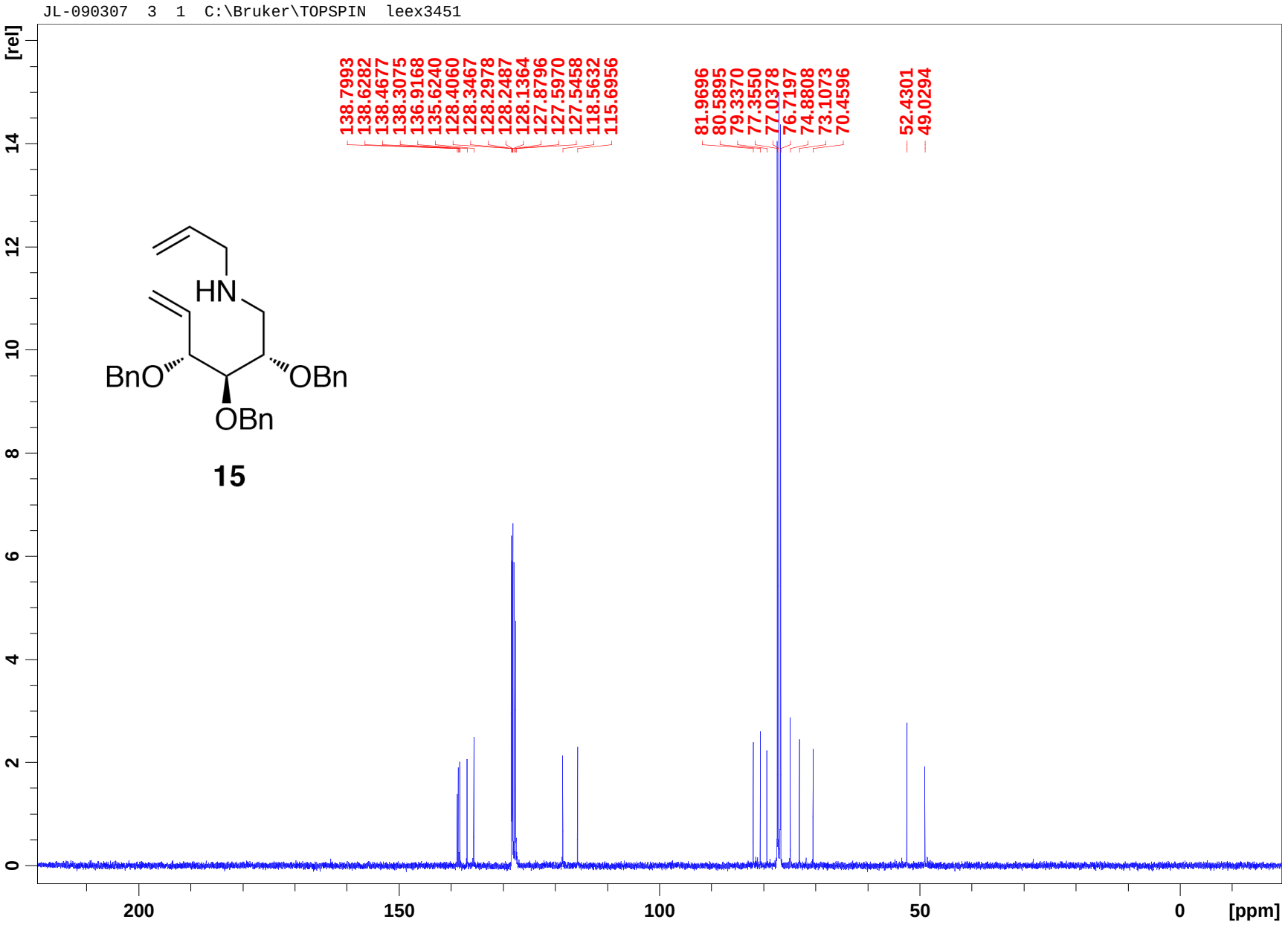
5
0
7.3503 7.3453 7.3401 7.3262 7.3215 7.3155 7.3022 7.2963 7.2910 7.2785 7.2738 7.2679 7.2569 7.2437 5.9091 5.8839 5.8647 5.8586 5.8400 5.7994 5.7737 5.7566 5.7309 5.2974 5.2901 5.2772 5.2590 5.0892 5.0851 5.0813 5.0463 5.0421 5.0375 5.0340 5.0305 5.0084 5.0050 4.7407 4.6543 4.6403 4.6255 4.6107 4.5900 4.5611 4.3755 4.3460 4.0494 4.0369 4.0308 4.0183 3.7812 3.7691 3.7538 3.6213 3.6084 3.5948 3.0630 3.0590 3.0526 3.0489 3.0447 3.0415 3.0382 2.7230 2.7112 2.6922 2.6804 2.6041 2.5878 2.5733
HN
BnO
OBn
OBn
15
15.3231
2.0000
2.1425
1.9594
1.8564
2.9757
1.0278
0.9228
0.9876
0.9283
1.5866
0.8910
0.9092
8
6
4
2
0 [ppm]

13C-NMR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135.6240
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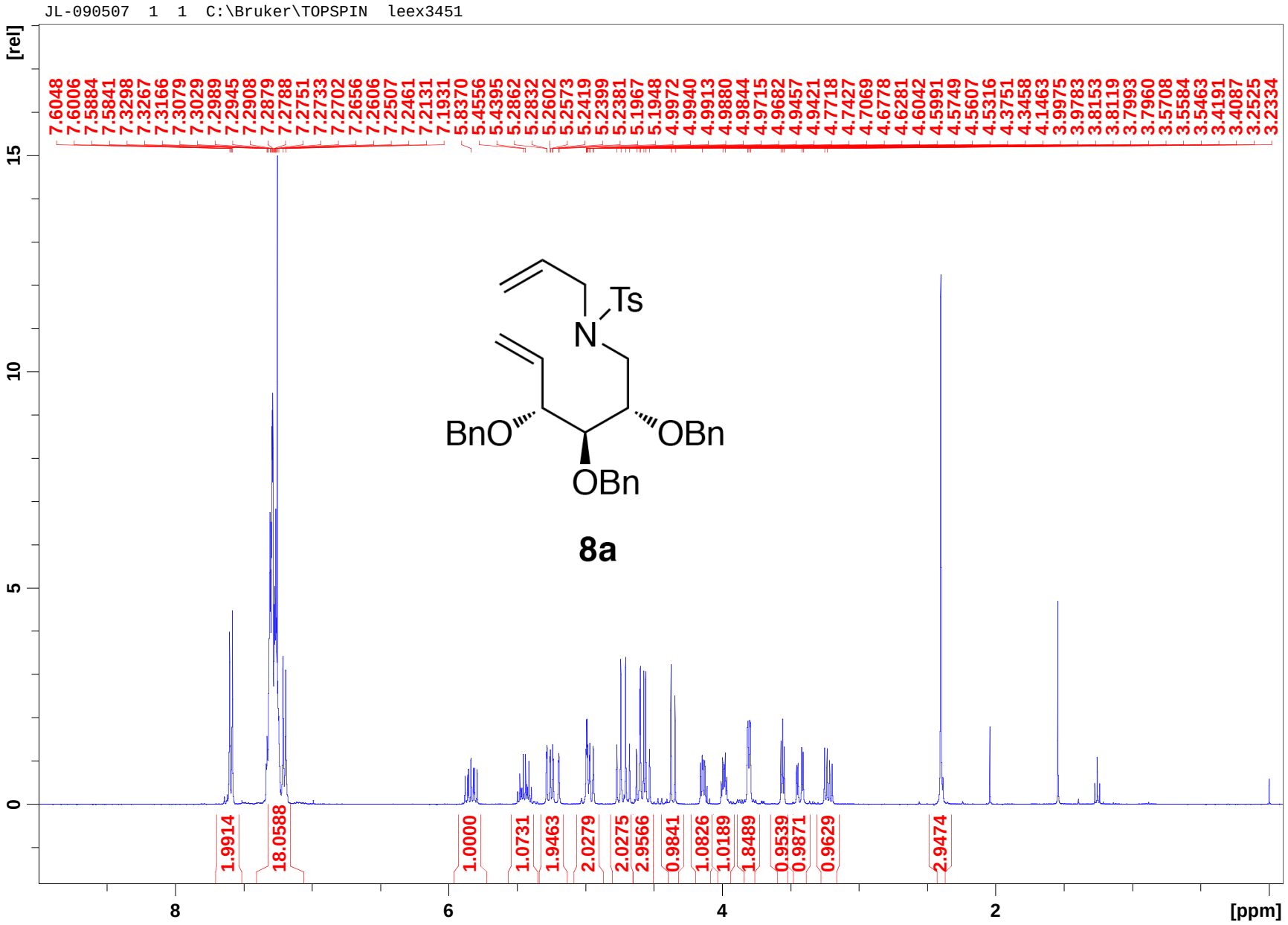
128.3467
128.2978
128.2487
128.1364
127.8796
127.5970
127.5458
118.5632
115.6956
81.9696
80.5895
79.3370
77.3550
77.0378
76.7197
74.8808
73.1073
70.4596
52.4301
49.0294
HN
BnO
OBn
OBn
15
200
150
100
50
0
[ppm]

1H-NMR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[rel]
15
10
5
0
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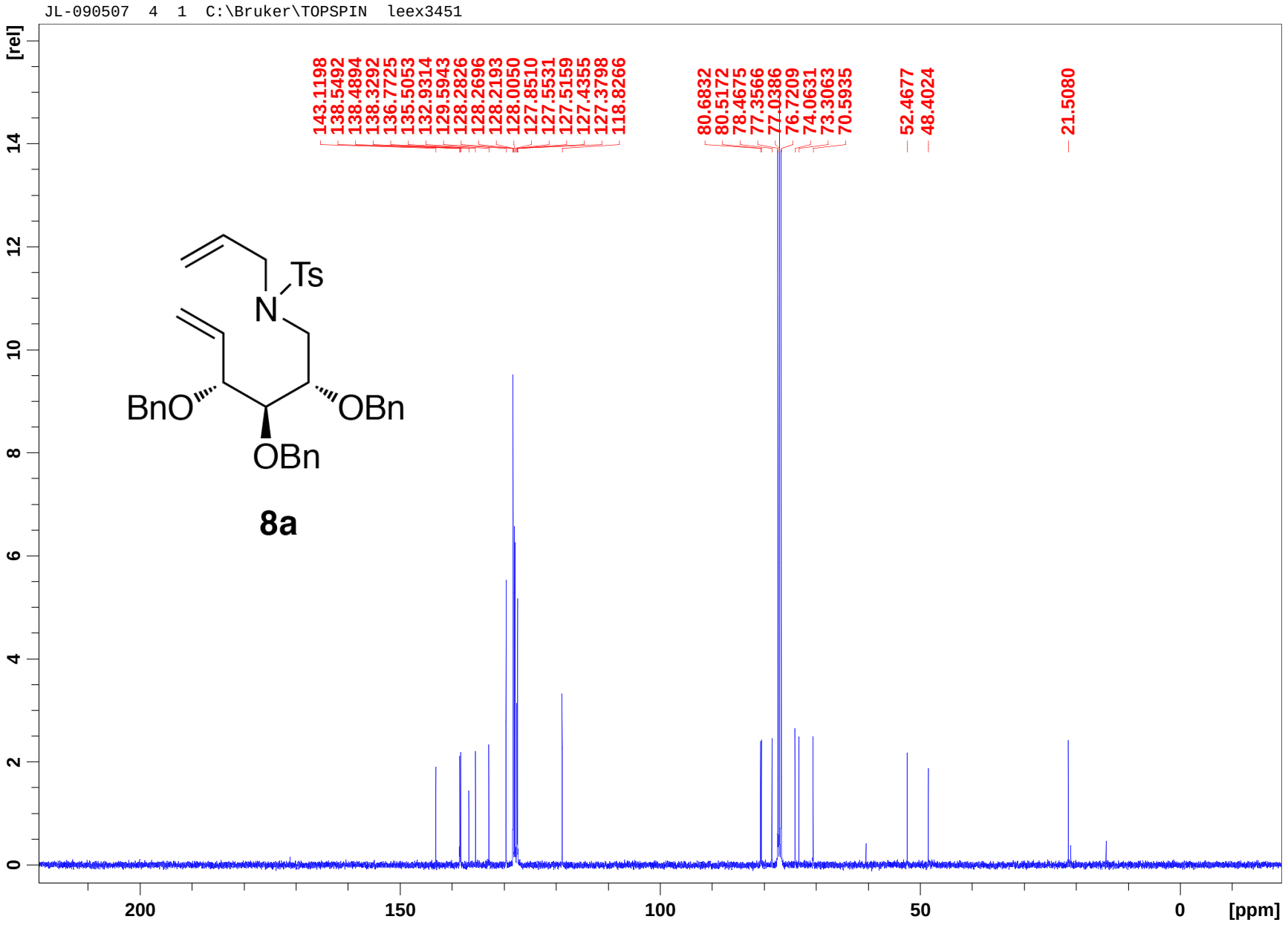
Ts
N
BnO
OBn
OBn
8a
1.9914 18.0588 1.0000 1.0731 1.9463 2.0279 2.0275 2.9566 0.9841 1.0826 1.0189 1.8489 0.9539 0.9871 0.9629 2.9474
8
6
4
2
[ppm]

13C-NMR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138.3292
136.7725
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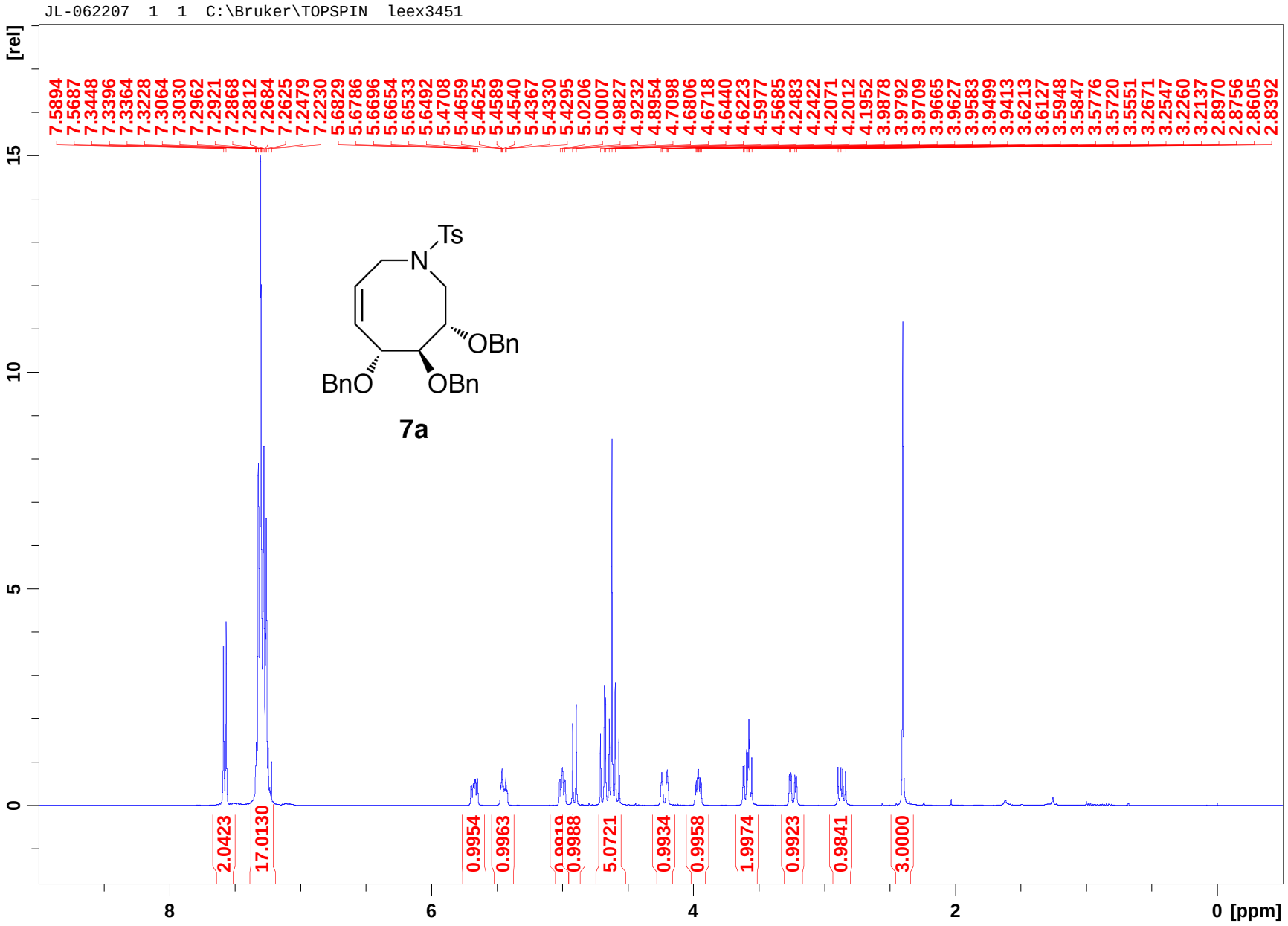
129.5943
128.2826
128.2696
128.2193
128.0050
127.8510
127.5531
127.5159
127.4355
127.3798
118.8266
80.6832
80.5172
78.4675
77.3566
77.0386
76.7209
74.0631
73.3063
70.5935
52.4677
48.4024
21.5080
Ts
N
BnO
OBn
OBn
8a
200
150
100
50
0
[ppm]

1H-NMR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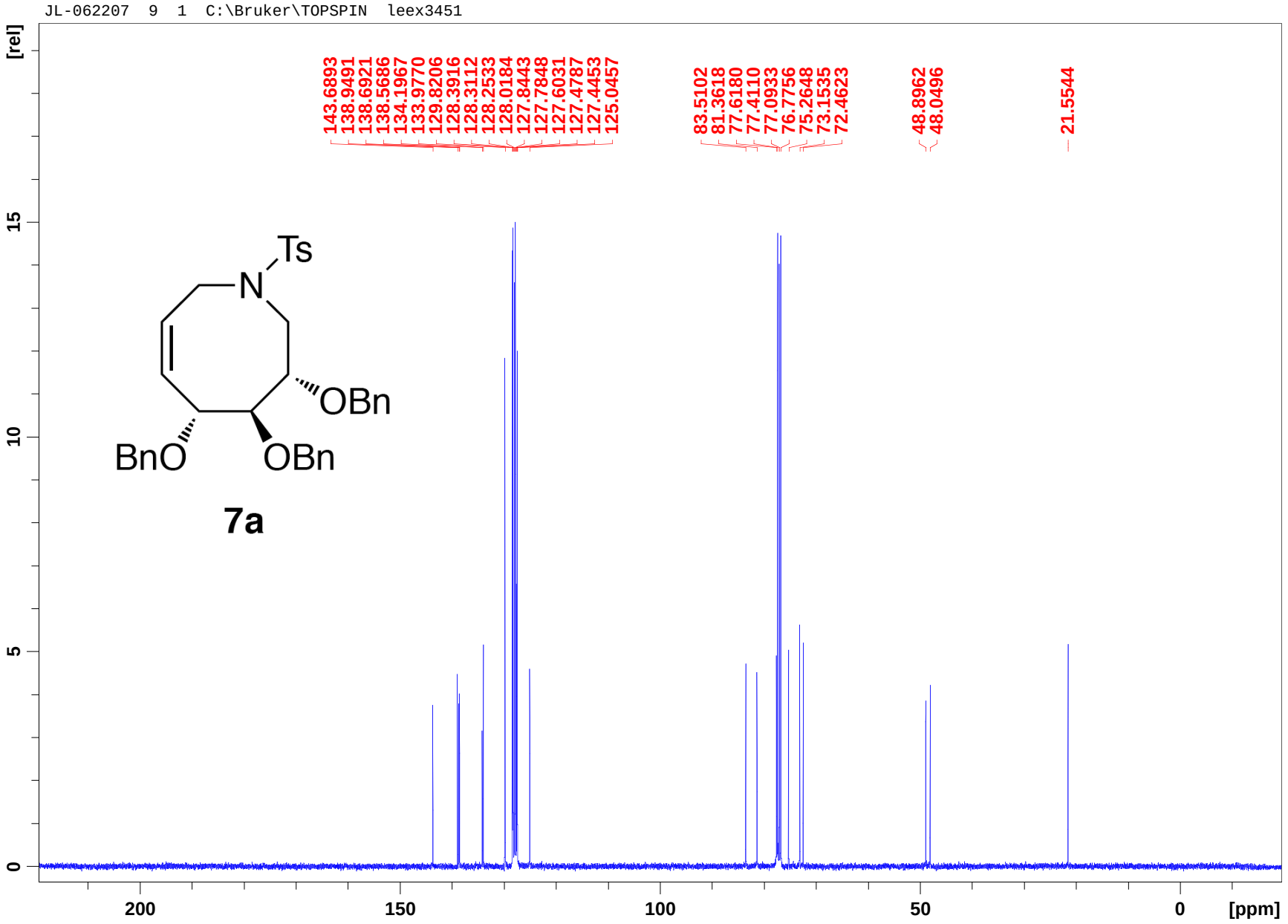
Ts
N
OBn
BnO
OBn
7a
2.0423
17.0130
0.9954
0.9963
0.9988
5.0721
0.9934
0.9958
1.9974
0.9923
0.9841
3.0000
8
6
4
2
0 [ppm]

13C-NMR


JL-062207 9 1 C:\Bruker\TOPSPIN leex3451
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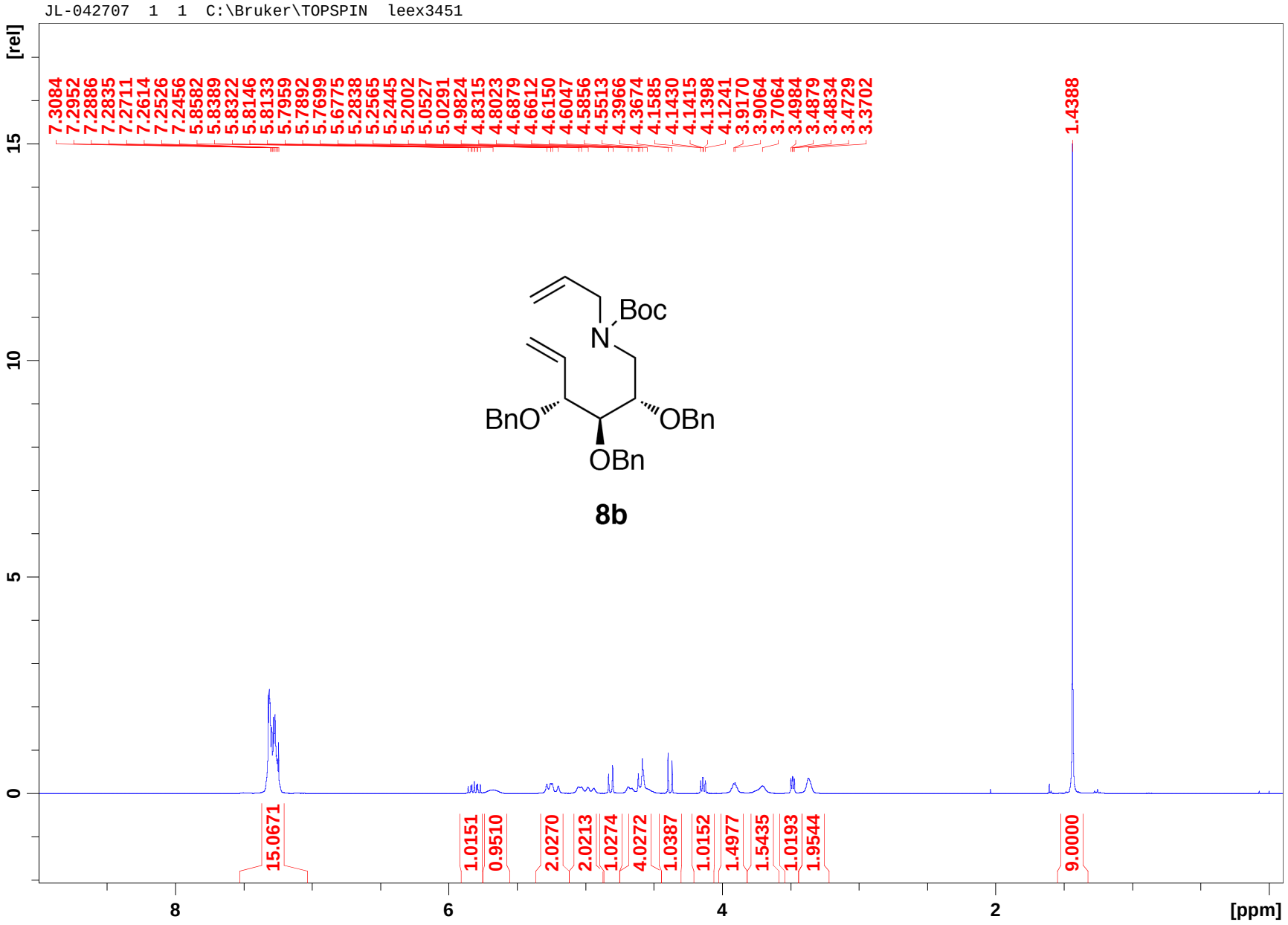
133.9770
129.8206
128.3916
128.3112
128.2533
128.0184
127.8443
127.7848
127.6031
127.4787
127.4453
125.0457
83.5102
81.3618
77.6180
77.4110
77.0933
76.7756
75.2648
73.1535
72.4623
48.8962
48.0496
21.5544
[rel]
0
5
10
15
200
150
100
50
0
[ppm]
Ts
N
OBn
BnO
OBn
7a

1H-NMR


JL-042707 1 1 C:\Bruker\TOPSPIN leex3451
[rel]
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5
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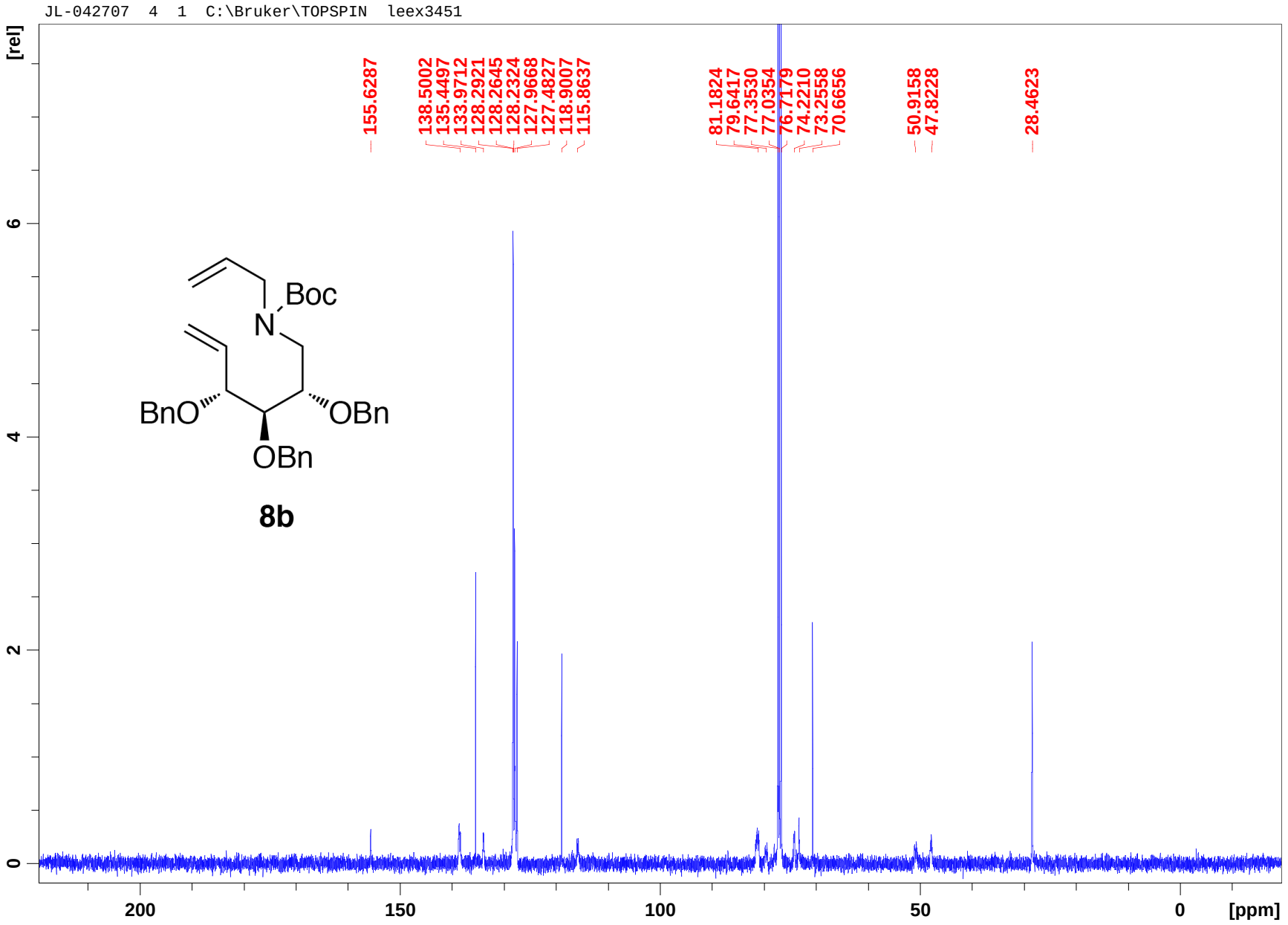
7.2952
7.2886
7.2835
7.2711
7.2614
7.2526
7.2456
5.8582
5.8389
5.8322
5.8146
5.8133
5.7959
5.7892
5.7699
5.6775
5.2838
5.2565
5.2445
5.2002
5.0527
5.0291
4.9824
4.8315
4.8023
4.6879
4.6612
4.6150
4.6047
4.5856
4.5513
4.3966
4.3674
4.1585
4.1430
4.1415
4.1398
4.1241
3.9170
3.9064
3.7064
3.4984
3.4879
3.4834
3.4729
3.3702
1.4388
Boc
N
BnO
OBn
OBn
8b
15.0671
1.0151
0.9510
2.0270
2.0213
1.0274
4.0272
1.0387
1.0152
1.4977
1.5435
1.0193
1.9544
9.0000
8
6
4
2
[ppm]

13C-NMR


JL-042707 4 1 C:\Bruker\TOPSPIN leex3451
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128.2921
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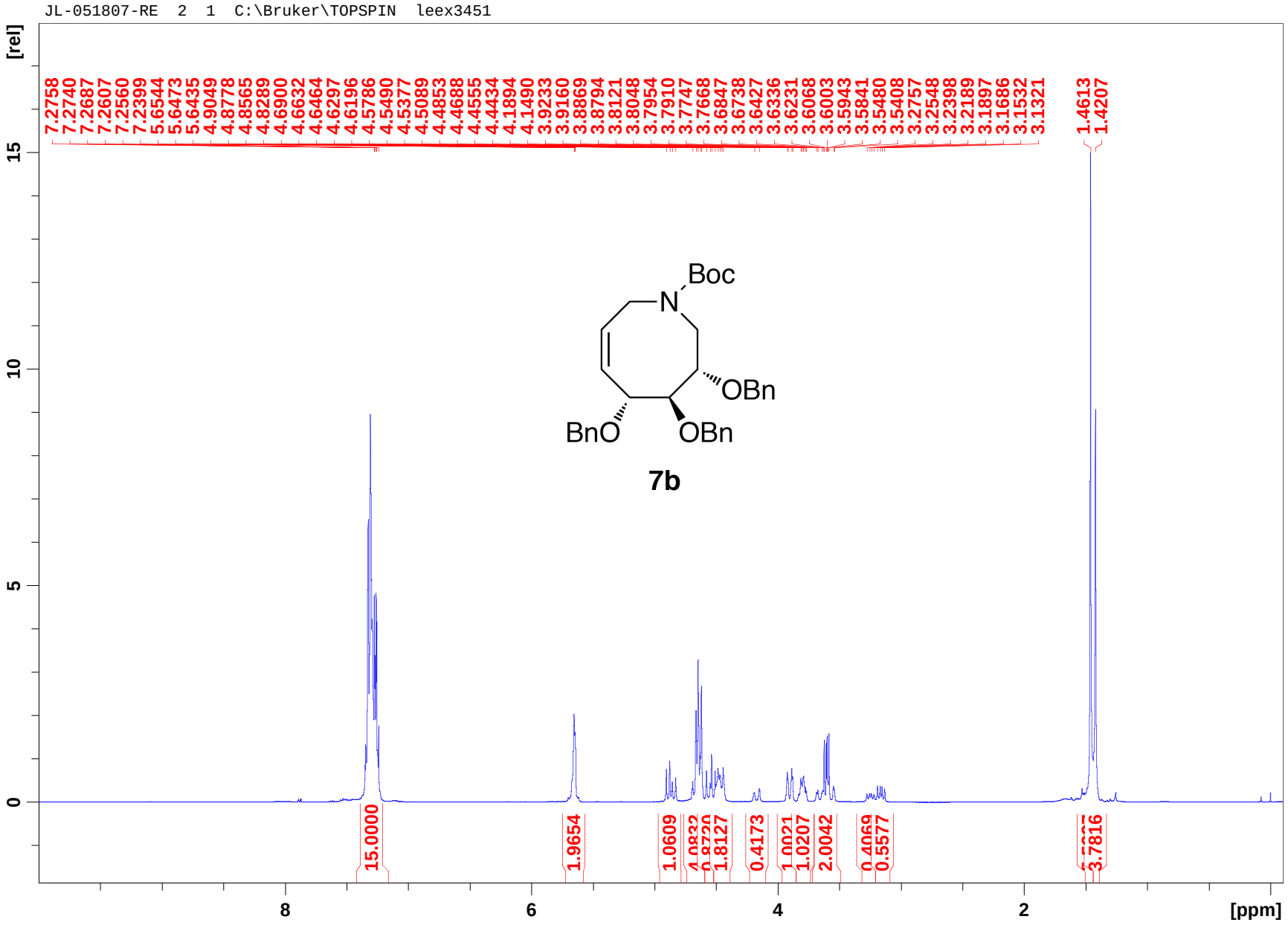
128.2324
127.9668
127.4827
118.9007
115.8637
81.1824
79.6417
77.3530
77.0354
76.7179
74.2210
73.2558
70.6656
50.9158
47.8228
28.4623
Boc
N
BnO
OBn
OBn
8b
0
2
4
6
200
150
100
50
0
[ppm]

1H-NMR


JL-051807-RE 2 1 C:\Bruker\TOPSPIN leex3451
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7.2607
7.2560
7.2399
5.6544
5.6473
5.6435
4.9049
4.8778
4.8565
4.8289
4.6900
4.6632
4.6464
4.6297
4.6196
4.5786
4.5490
4.5377
4.5089
4.4853
4.4688
4.4555
4.4434
4.1894
4.1490
3.9233
3.9160
3.8869
3.8794
3.8121
3.8048
3.7954
3.7910
3.7747
3.7668
3.6847
3.6738
3.6427
3.6336
3.6231
3.6068
3.6003
3.5943
3.5841
3.5480
3.5408
3.2757
3.2548
3.2398
3.2189
3.1897
3.1686
3.1532
3.1321
1.4613
1.4207
Boc
N
OBn
BnO
OBn
7b
15.0000
1.9654
1.0609
1.8127
0.4173
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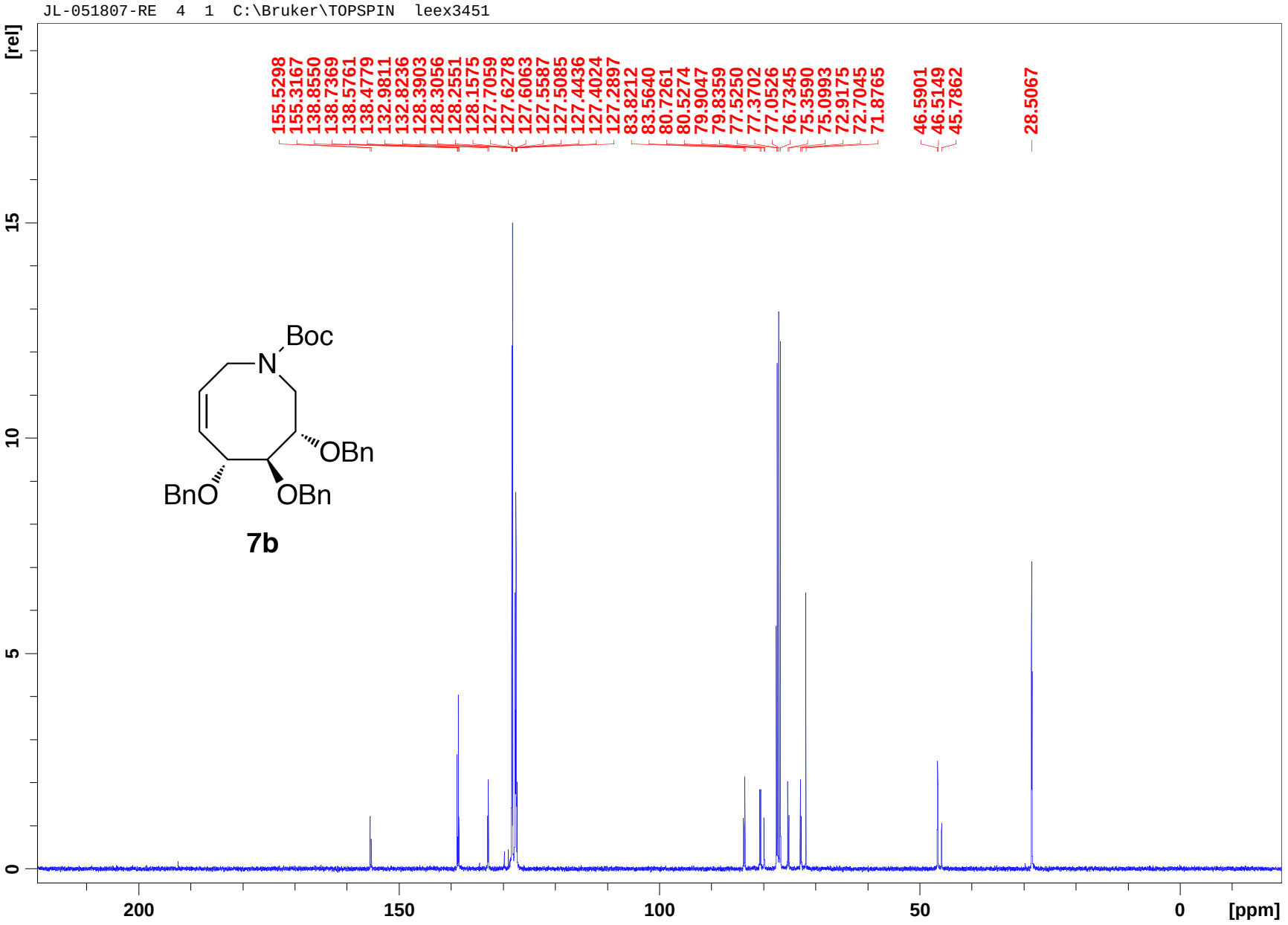
1.0207
2.0042
0.5577
3.7816
8
6
4
2
[ppm]

13C-NMR


JL-051807-RE 4 1 C:\Bruker\TOPSPIN leex3451
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132.8236
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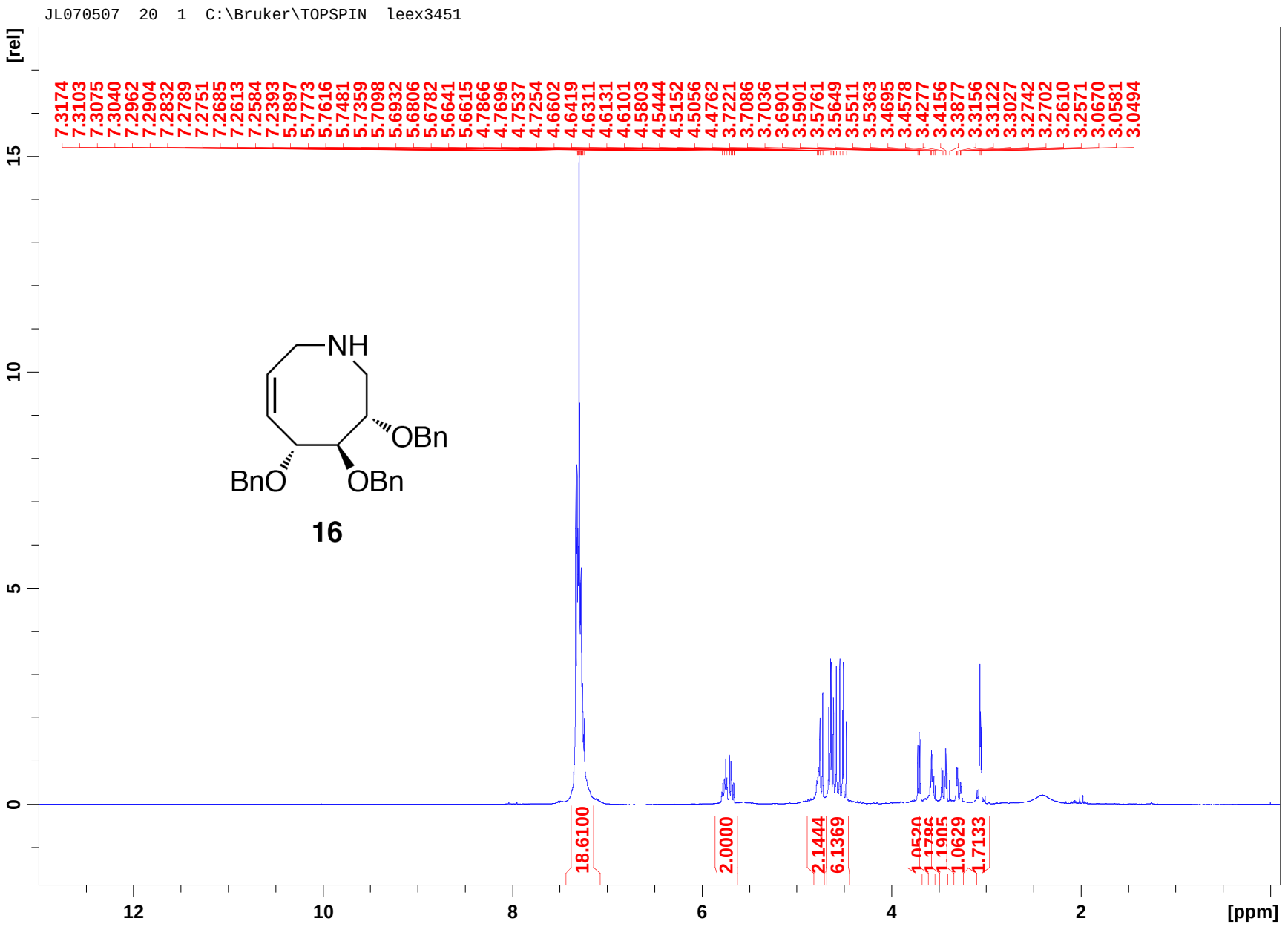
128.3056
128.2551
128.1575
127.7059
127.6278
127.6063
127.5587
127.5085
127.4436
127.4024
127.2897
83.8212
83.5640
80.7261
80.5274
79.9047
79.8359
77.5250
77.3702
77.0526
76.7345
75.3590
75.0993
72.9175
72.7045
71.8765
46.5901
46.5149
45.7862
28.5067
Boc
N
OBn
BnO
OBn
7b
0
5
10
15
200
150
100
50
0
[ppm]

1H-NMR


JL070507 20 1 C:\Bruker\TOPSPIN leex3451
[rel]
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10
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7.3075
7.3040
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7.2904
7.2832
7.2789
7.2751
7.2685
7.2613
7.2584
7.2393
5.7897
5.7773
5.7616
5.7481
5.7359
5.7098
5.6932
5.6806
5.6782
5.6641
5.6615
4.7866
4.7696
4.7537
4.7254
4.6602
4.6419
4.6311
4.6131
4.6101
4.5803
4.5444
4.5152
4.5056
4.4762
3.7221
3.7086
3.7036
3.6901
3.5901
3.5761
3.5649
3.5511
3.5363
3.4695
3.4578
3.4277
3.4156
3.3877
3.3156
3.3122
3.3027
3.2742
3.2702
3.2610
3.2571
3.0670
3.0581
3.0494
NH
OBn
BnO
OBn
16
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2.0000
2.1444
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1.1905
1.0629
1.7133
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8
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4
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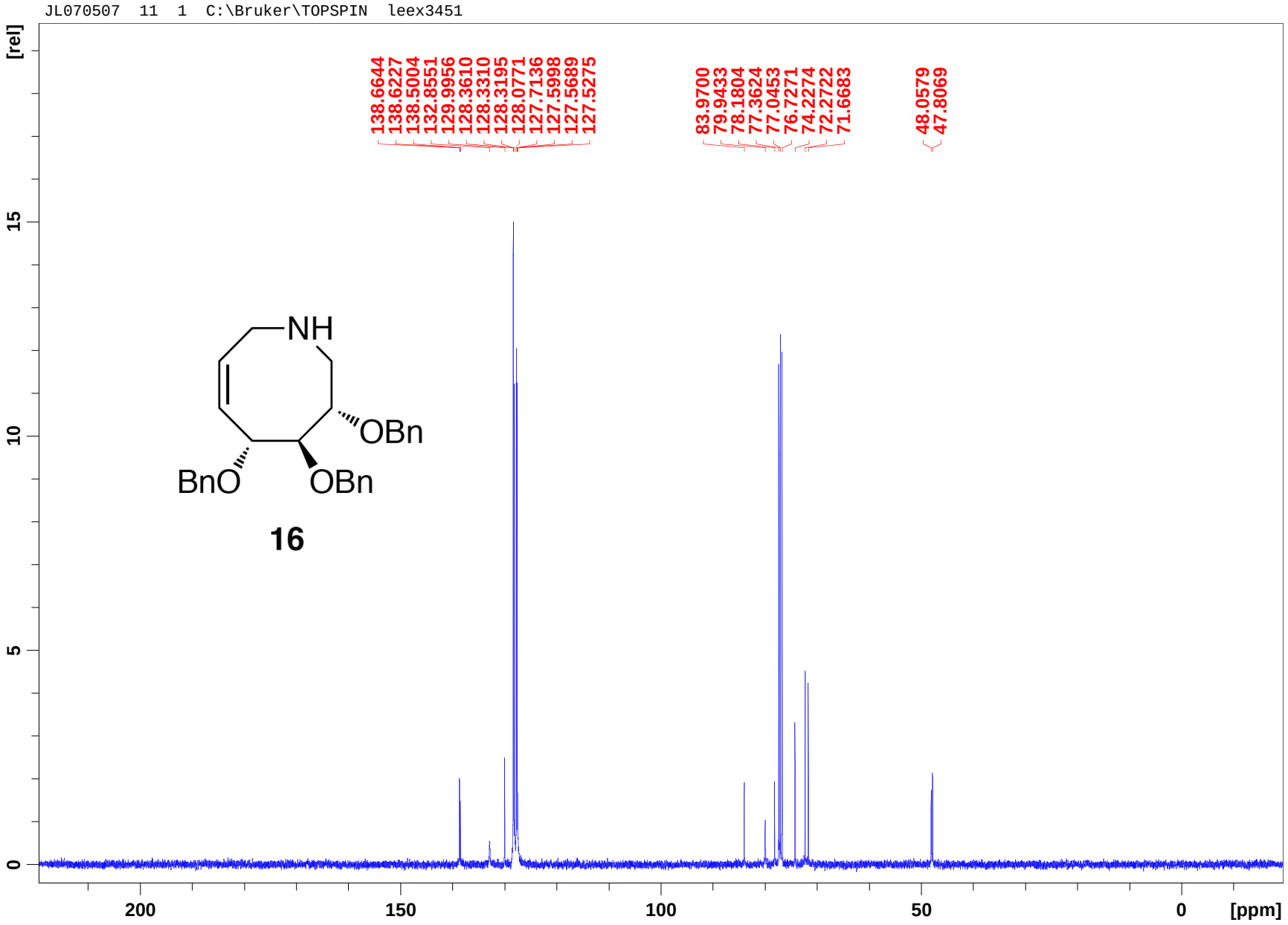
[ppm]

13C-NMR


JL070507 11 1 C:\Bruker\TOPSPIN leex3451
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127.5689
127.5275
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79.9433
78.1804
77.3624
77.0453
76.7271
74.2274
72.2722
71.6683
48.0579
47.8069
NH
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BnO
OBn
16
0
5
10
15
200
150
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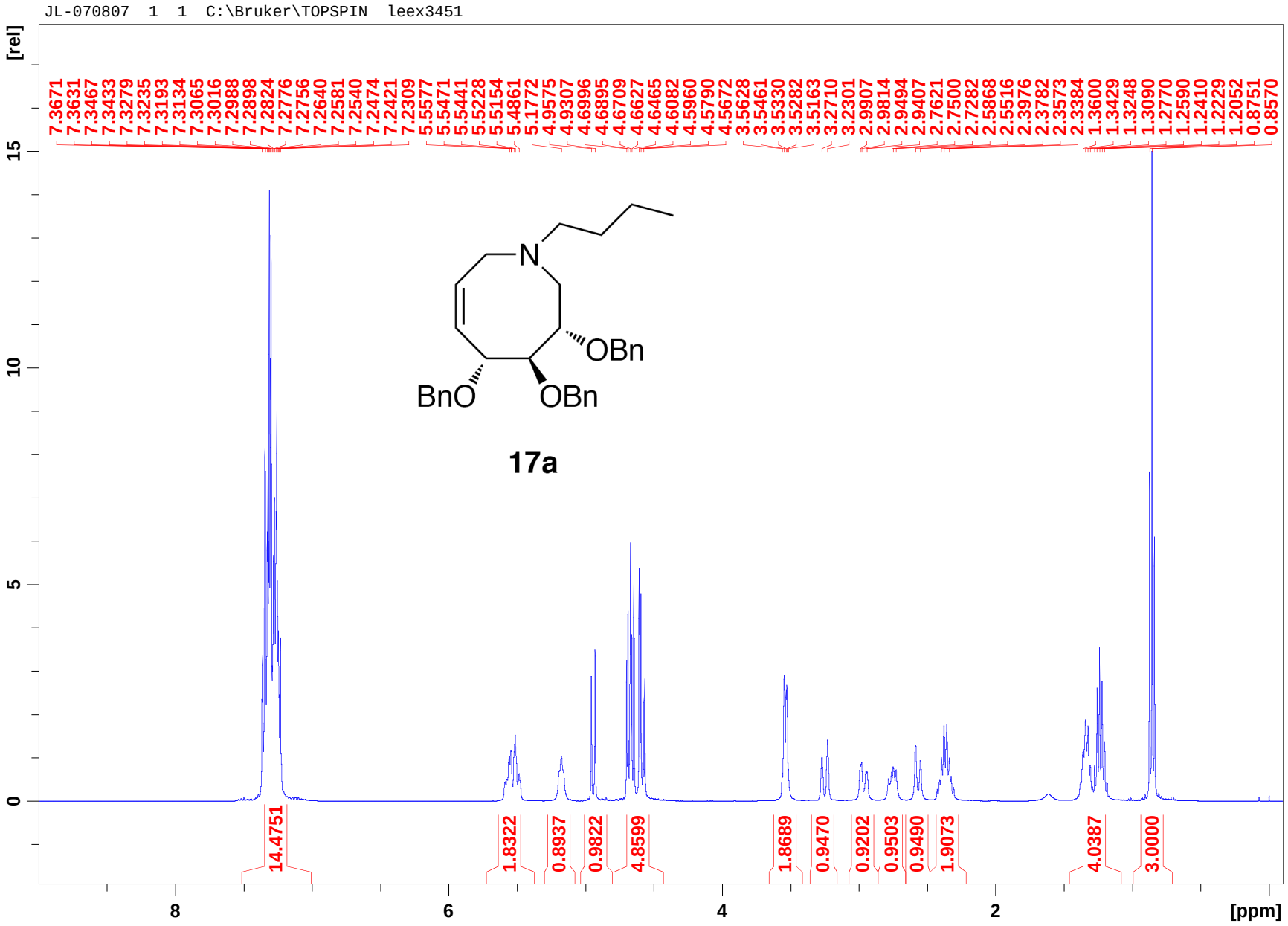
0
[ppm]

1H-NMR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N
OBn
BnO
OBn
17a
14.4751
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0.9822
4.8599
1.8689
0.9470
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8
6
4
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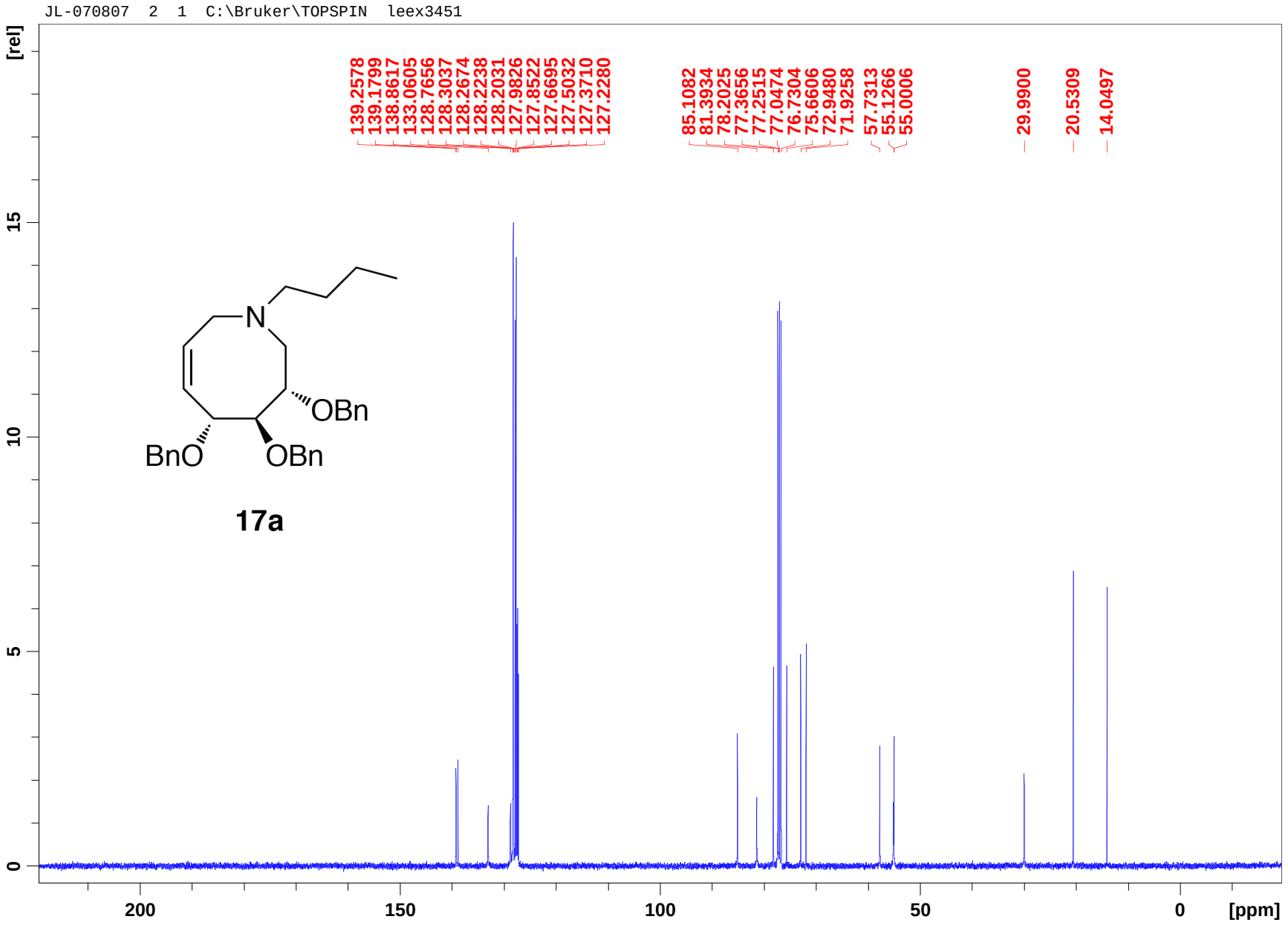
[ppm]

13C-NMR


JL-070807 2 1 C:\Bruker\TOPSPIN leex3451
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128.2674
128.2238
128.2031
127.9826
127.8522
127.6695
127.5032
127.3710
127.2280
85.1082
81.3934
78.2025
77.3656
77.2515
77.0474
76.7304
75.6606
72.9480
71.9258
57.7313
55.1266
55.0006
29.9900
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OBn
17a
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150
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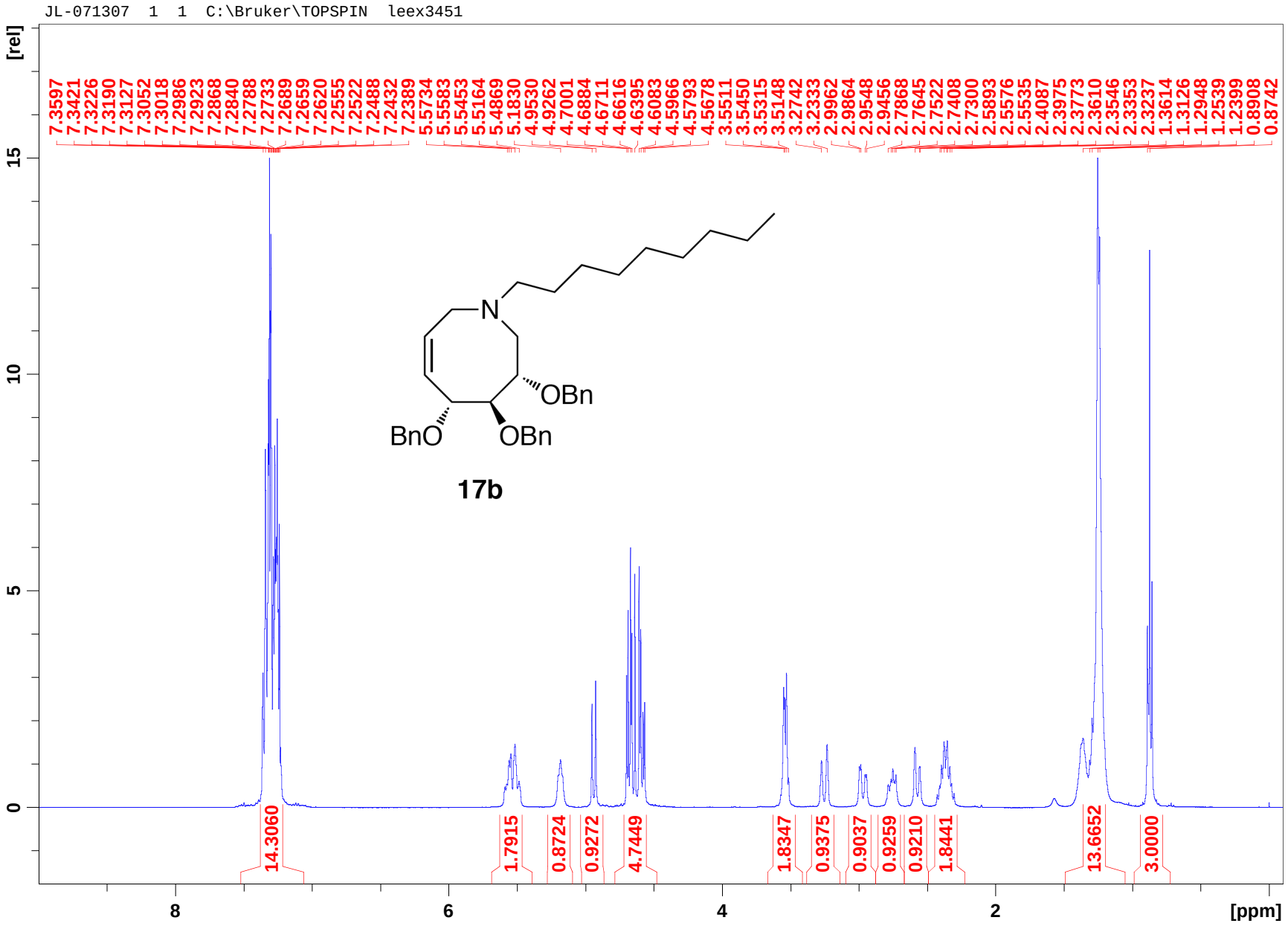
[ppm]

1H-NMR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14.3060
1.7915
0.8724
0.9272
4.7449
1.8347
0.9375
0.9037
0.9259
0.9210
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3.0000
8
6
4
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[ppm]
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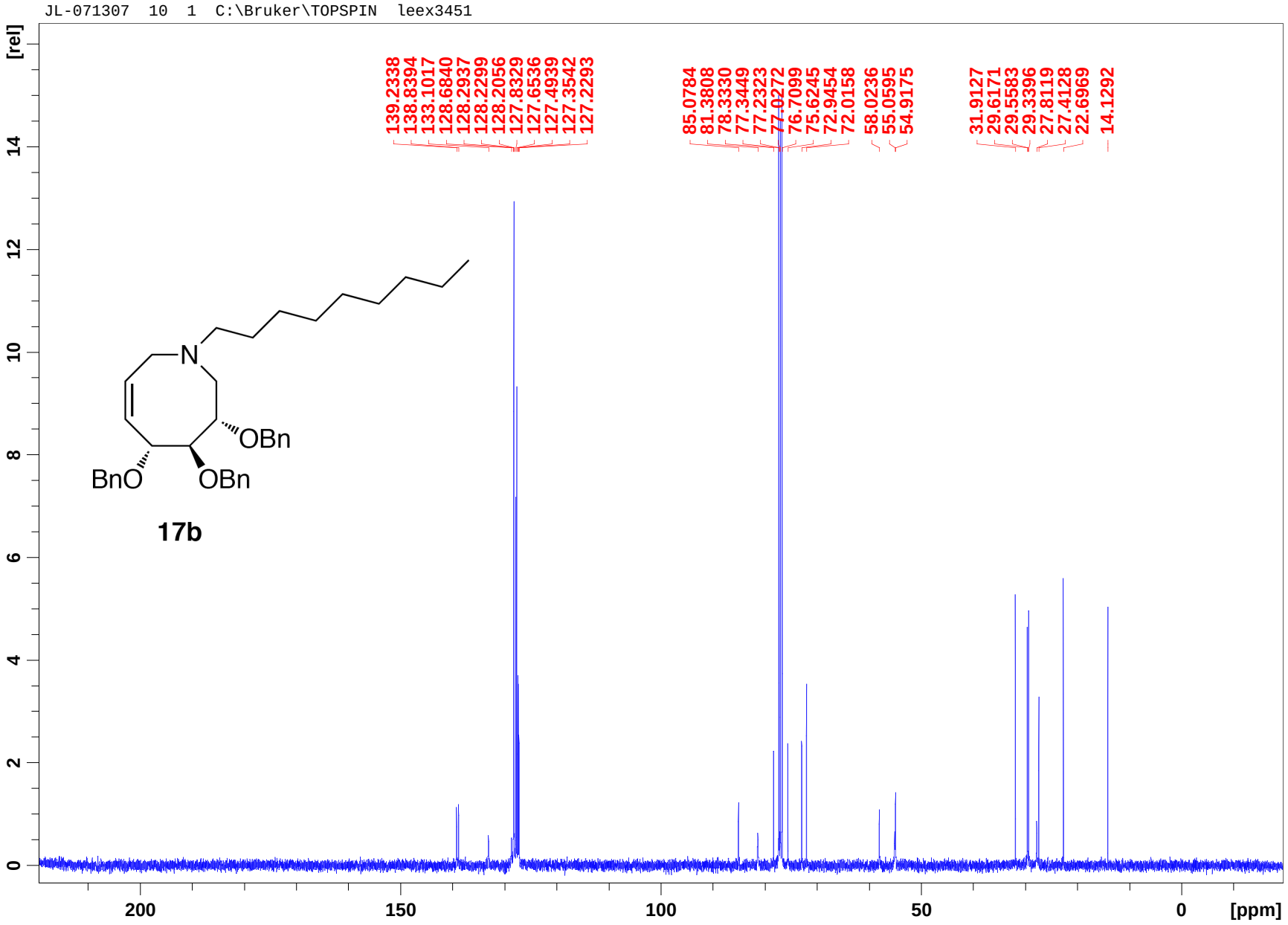
BnO
OBn
17b

13C-NMR


JL-071307 10 1 C:\Bruker\TOPSPIN leex3451
[rel]
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4
6
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10
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128.2056
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127.4939
127.3542
127.2293
85.0784
81.3808
78.3330
77.3449
77.2323
77.0272
76.7099
75.6245
72.9454
72.0158
58.0236
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31.9127
29.6171
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29.3396
27.8119
27.4128
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N
OBn
BnO
OBn
17b
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150
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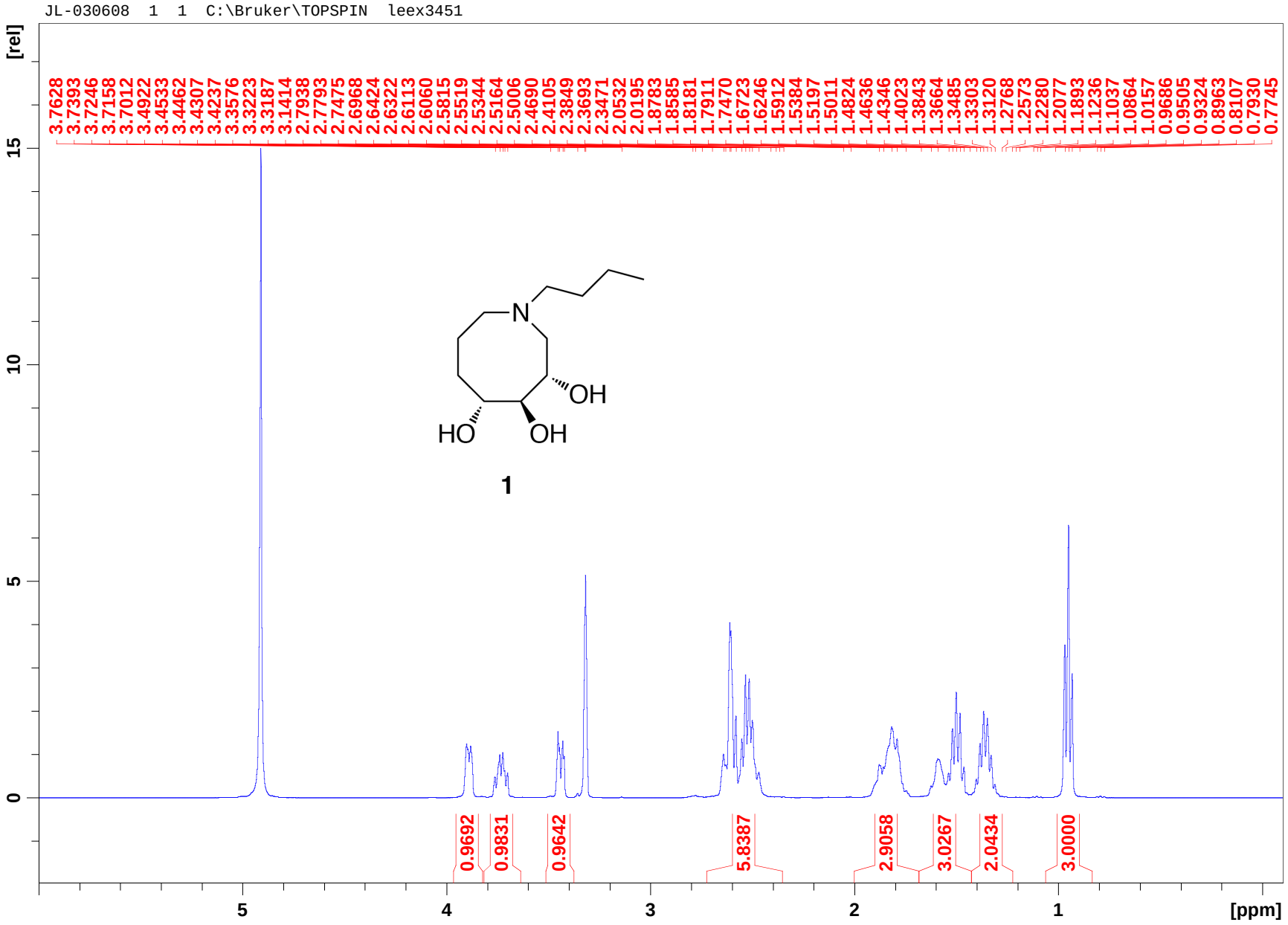
50
0
[ppm]

1H-NMR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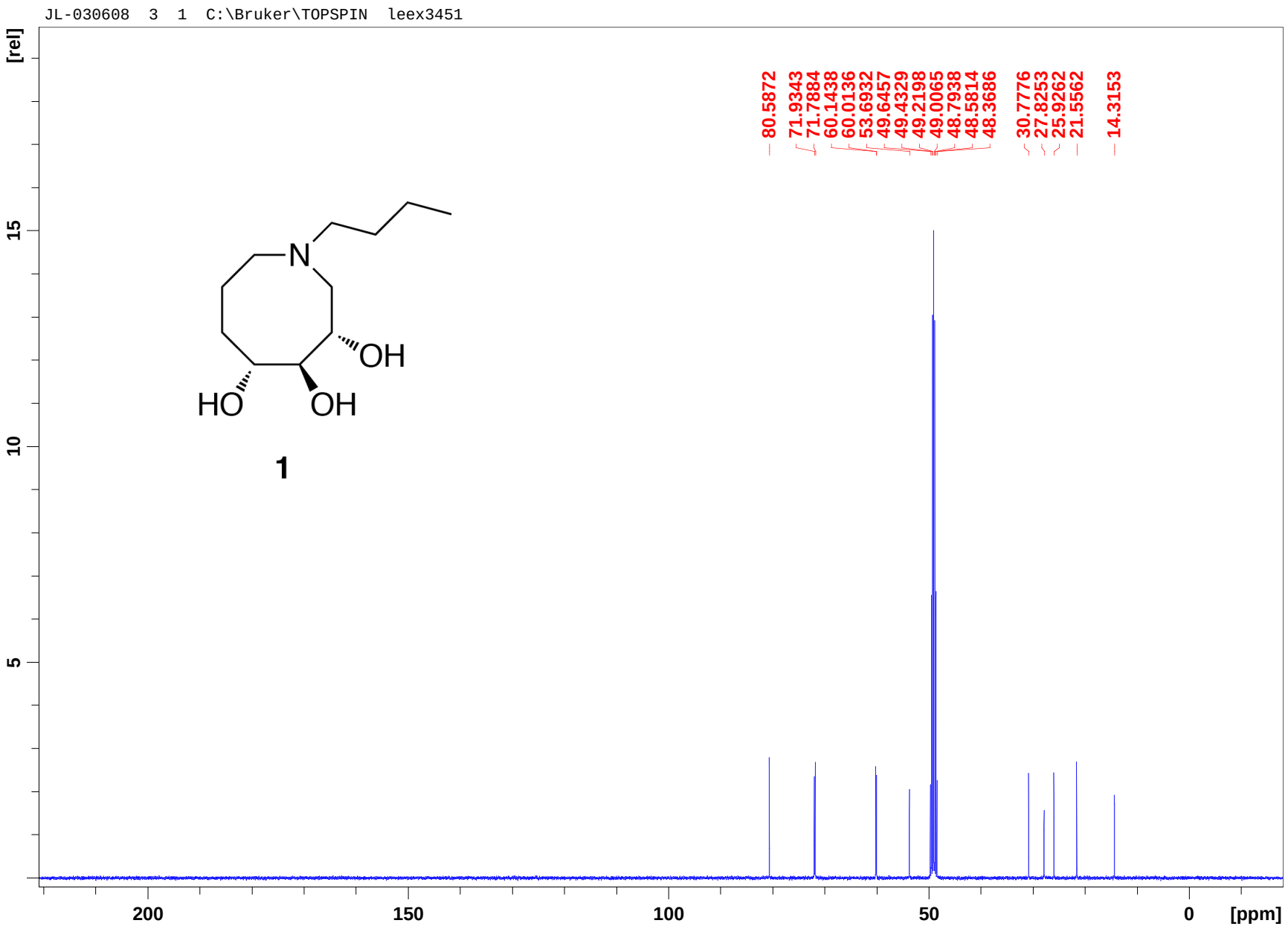
N
OH
HO
OH
1
0.9692
0.9831
0.9642
5.8387
2.9058
3.0267
2.0434
3.0000
5
4
3
2
1
[ppm]

13C-NMR


JL-030608 3 1 C:\Bruker\TOPSPIN leex3451
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15
10
5
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60.0136
53.6932
49.6457
49.4329
49.2198
49.0065
48.7938
48.5814
48.3686
30.7776
27.8253
25.9262
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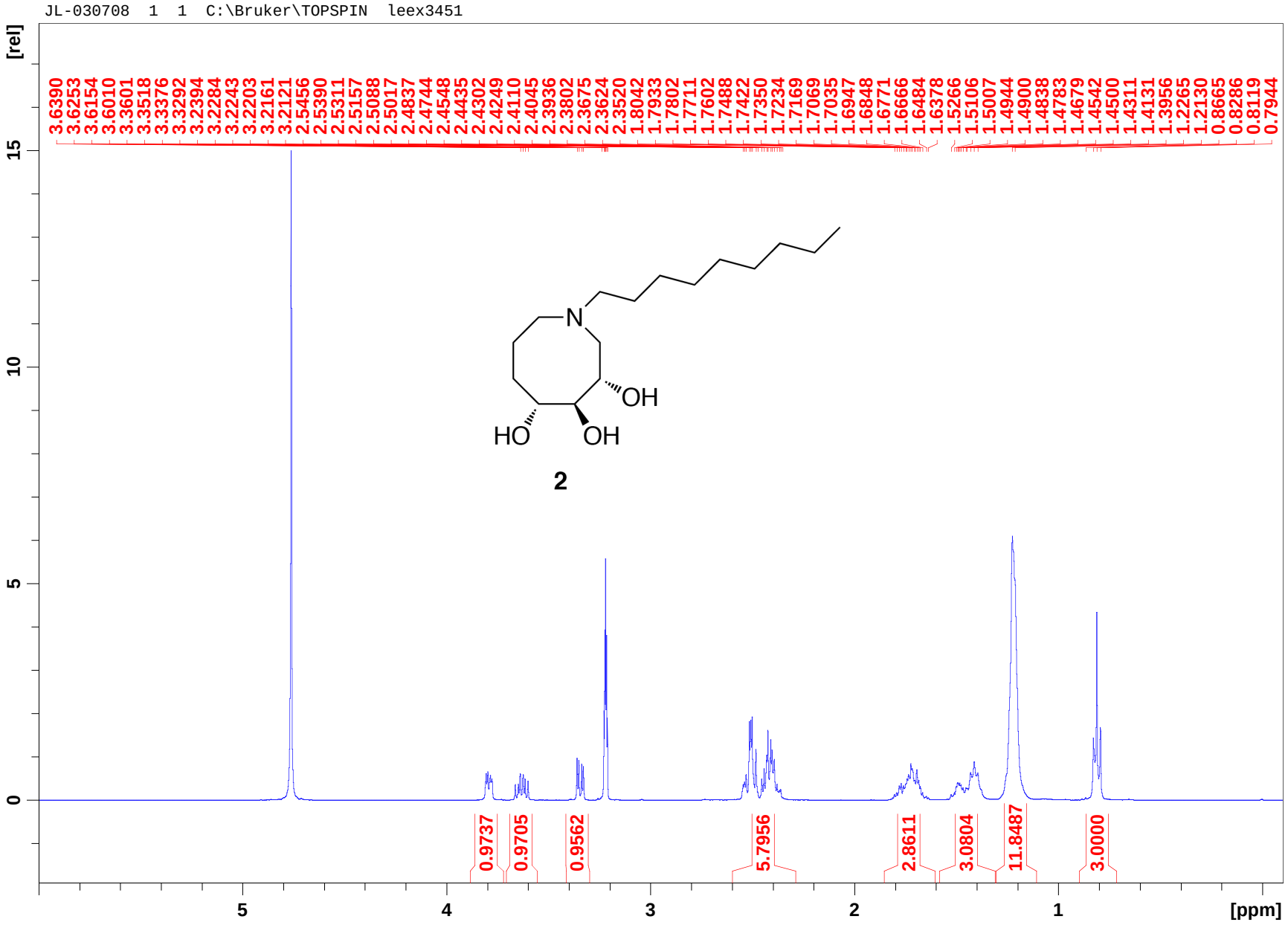
14.3153
N
OH
HO
OH
1
200
150
100
50
0
[ppm]

1H-NMR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N
OH
HO
OH
2
0.9737
0.9705
0.9562
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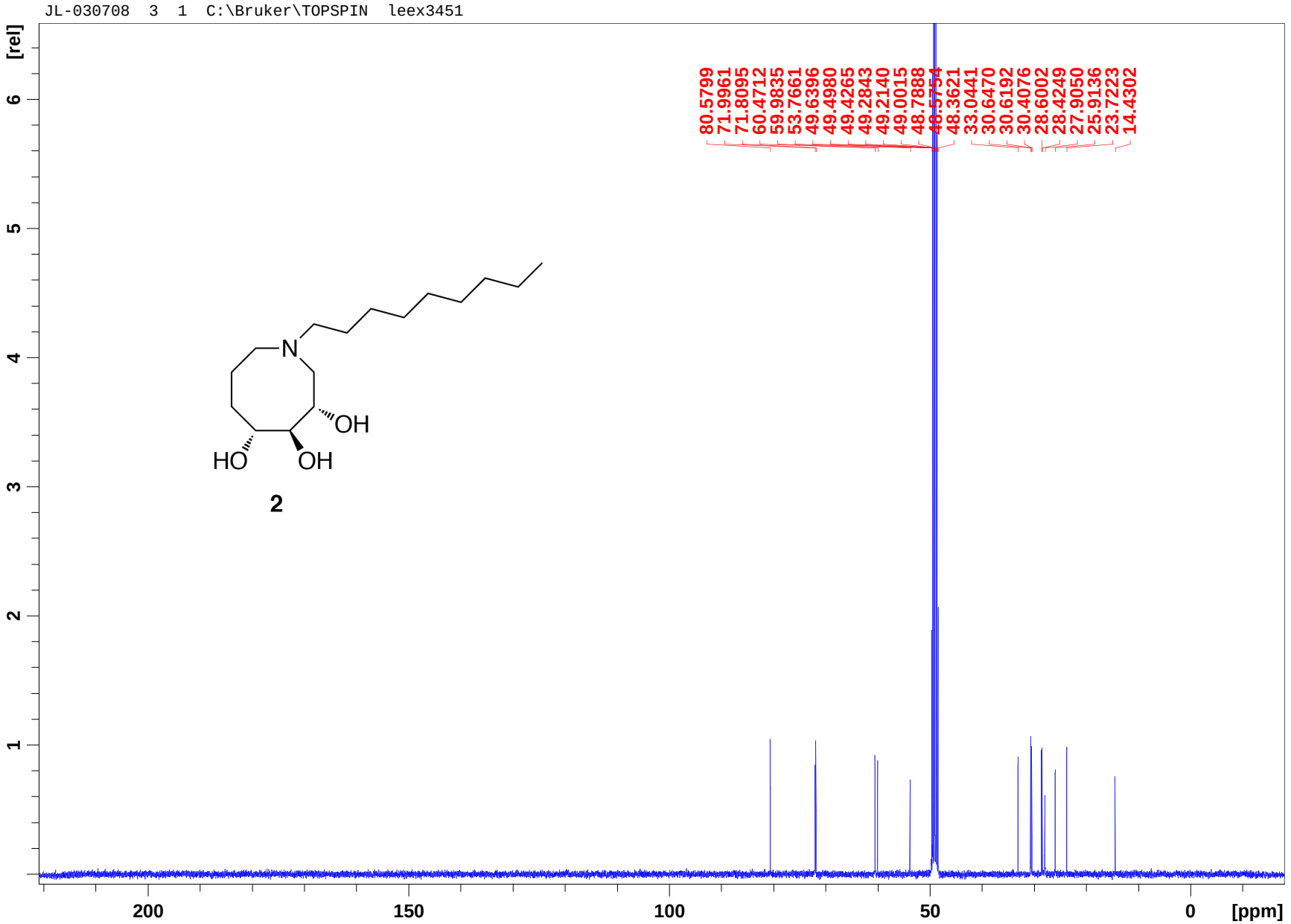
2.8611
3.0804
11.8487
3.0000
5
4
3
2
1
[ppm]

13C-NMR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49.6396
49.4980
49.4265
49.2843
49.2140
49.0015
48.7888
48.5754
48.3621
33.0441
30.6470
30.6192
30.4076
28.6002
28.4249
27.9050
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N
OH
HO
OH
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150
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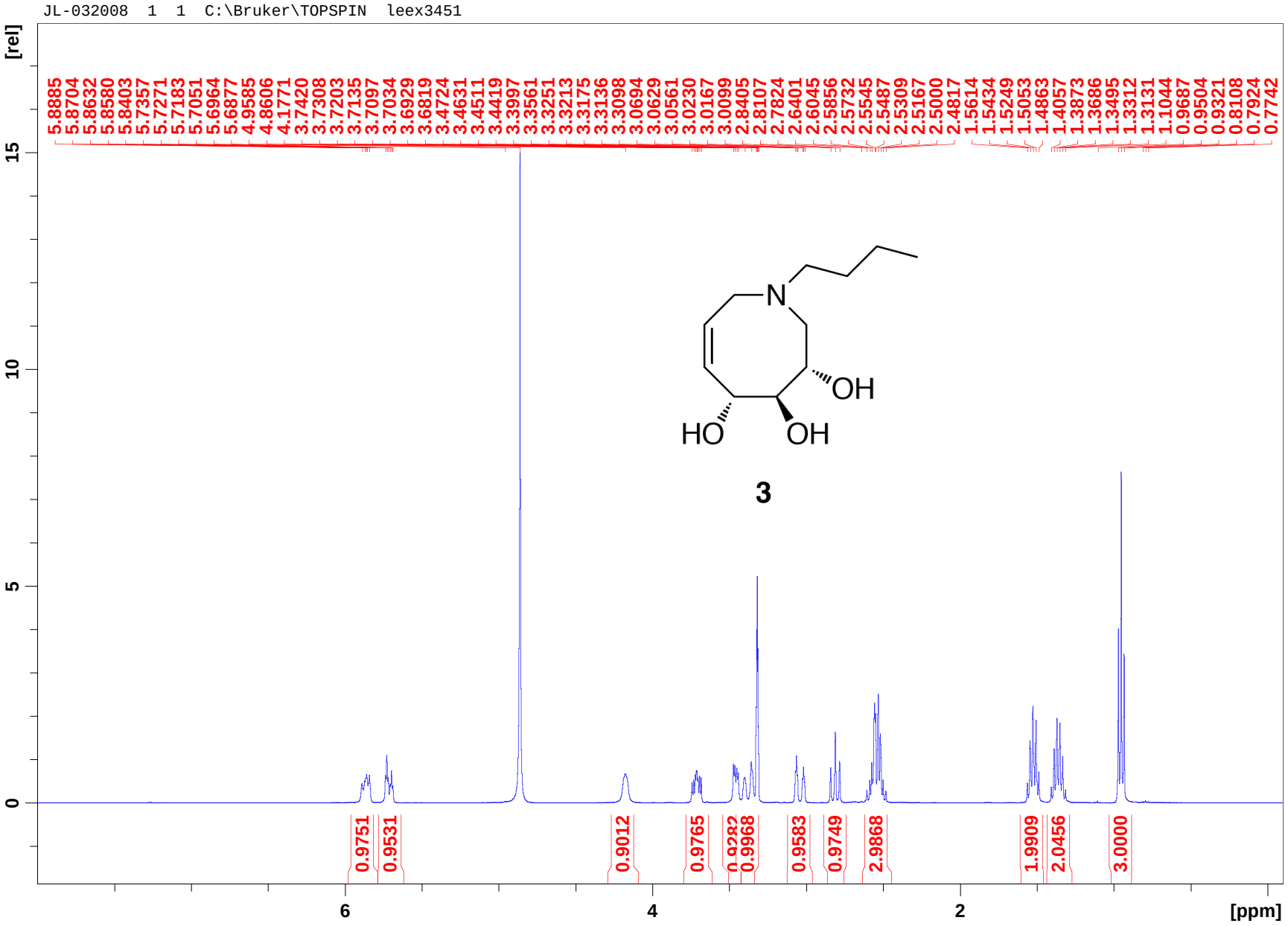
0
[ppm]

1H-NMR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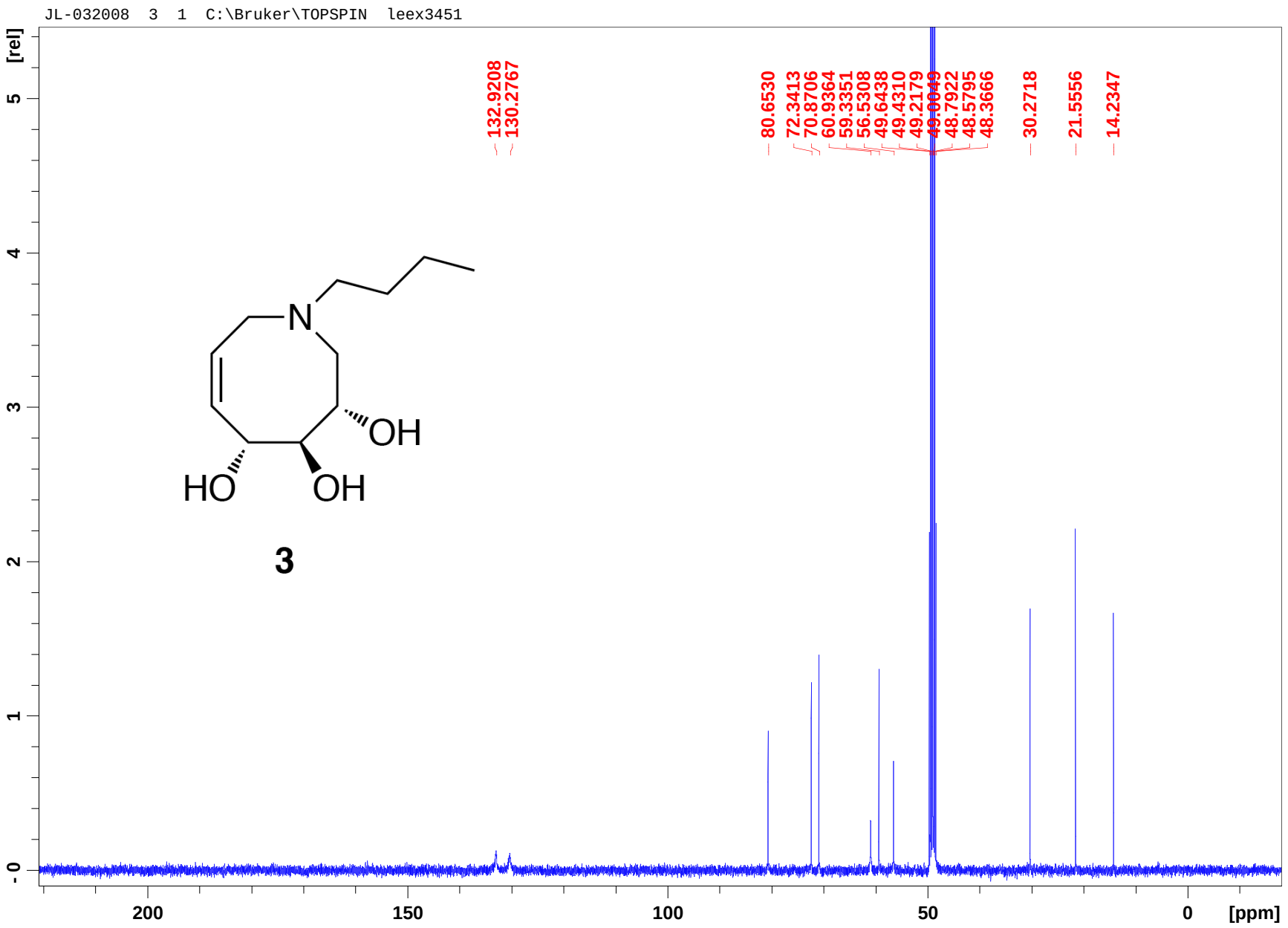
N
OH
HO
OH
3
0.9751
0.9531
0.9012
0.9765
0.9968
0.9583
0.9749
2.9868
1.9909
2.0456
3.0000
6
4
2
[ppm]

13C-NMR


JL-032008 3 1 C:\Bruker\TOPSPIN leex3451
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3
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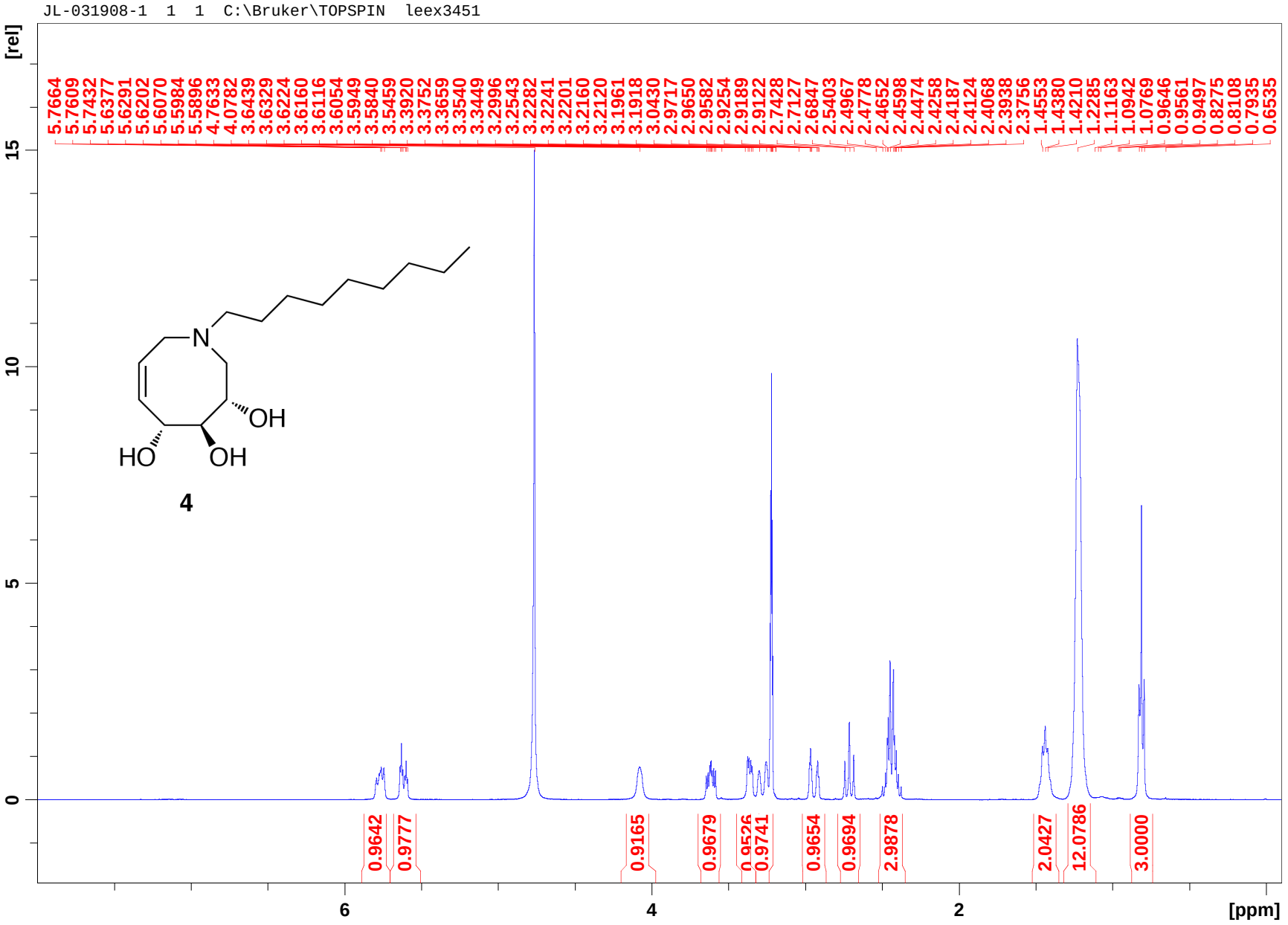
72.3413
70.8706
60.9364
59.3351
56.5308
49.6438
49.4310
49.2179
49.0049
48.7922
48.5795
48.3666
30.2718
21.5556
14.2347
N
OH
HO
OH
3
200
150
100
50
0
[ppm]

1H-NMR


JL-031908-1 1 1 C:\Bruker\TOPSPIN leex3451
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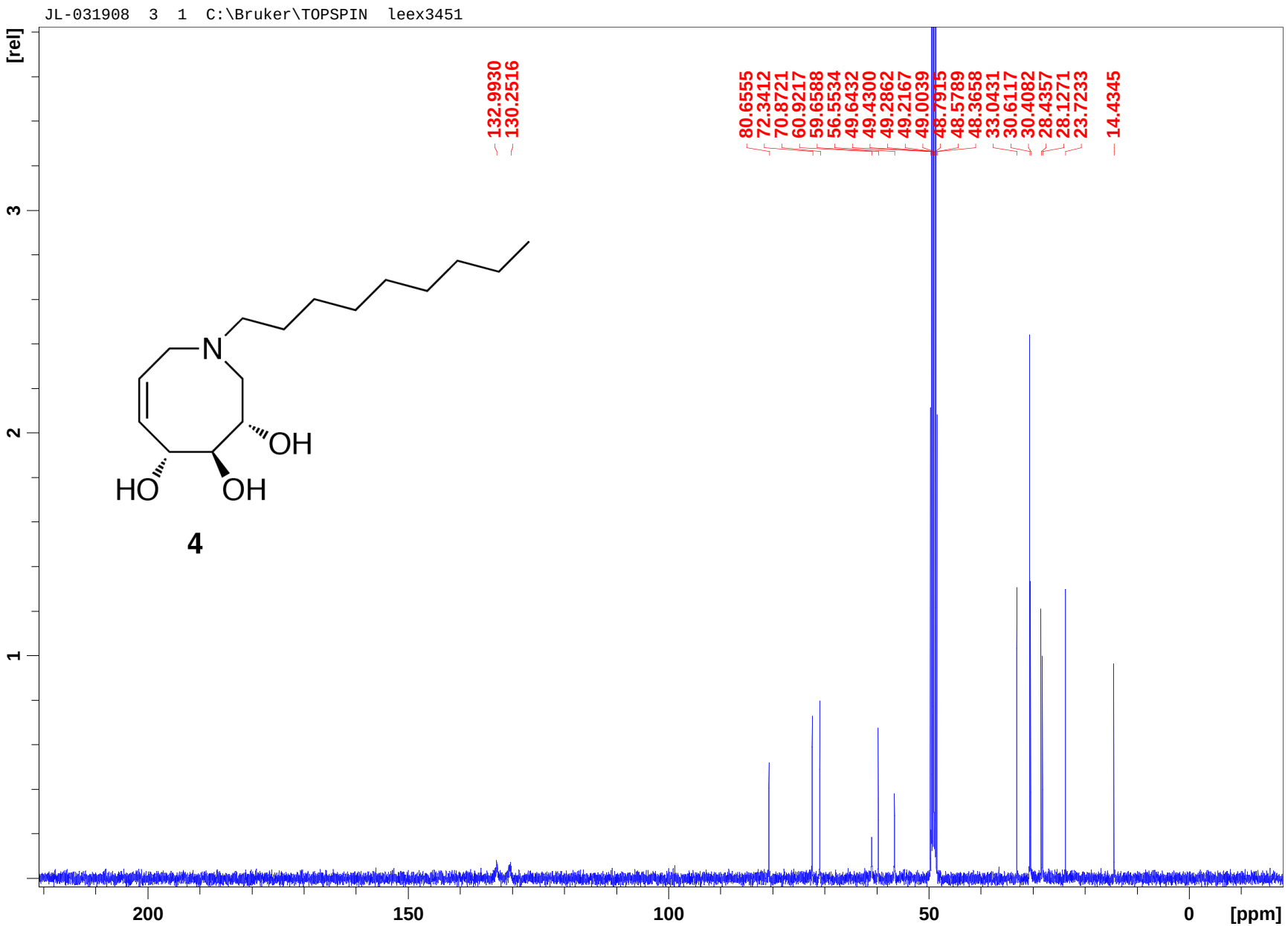
N
OH
HO
OH
4
0.9642 0.9777 0.9165 0.9679 0.9526 0.9741 0.9654 0.9694 2.9878 2.0427 12.0786 3.0000
6
4
2
[ppm]

13C-NMR


JL-031908 3 1 C:\Bruker\TOPSPIN leex3451
[rel]
3
2
1
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80.6555
72.3412
70.8721
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59.6588
56.5534
49.6432
49.4300
49.2862
49.2167
49.0039
48.7915
48.5789
48.3658
33.0431
30.6117
30.4082
28.4357
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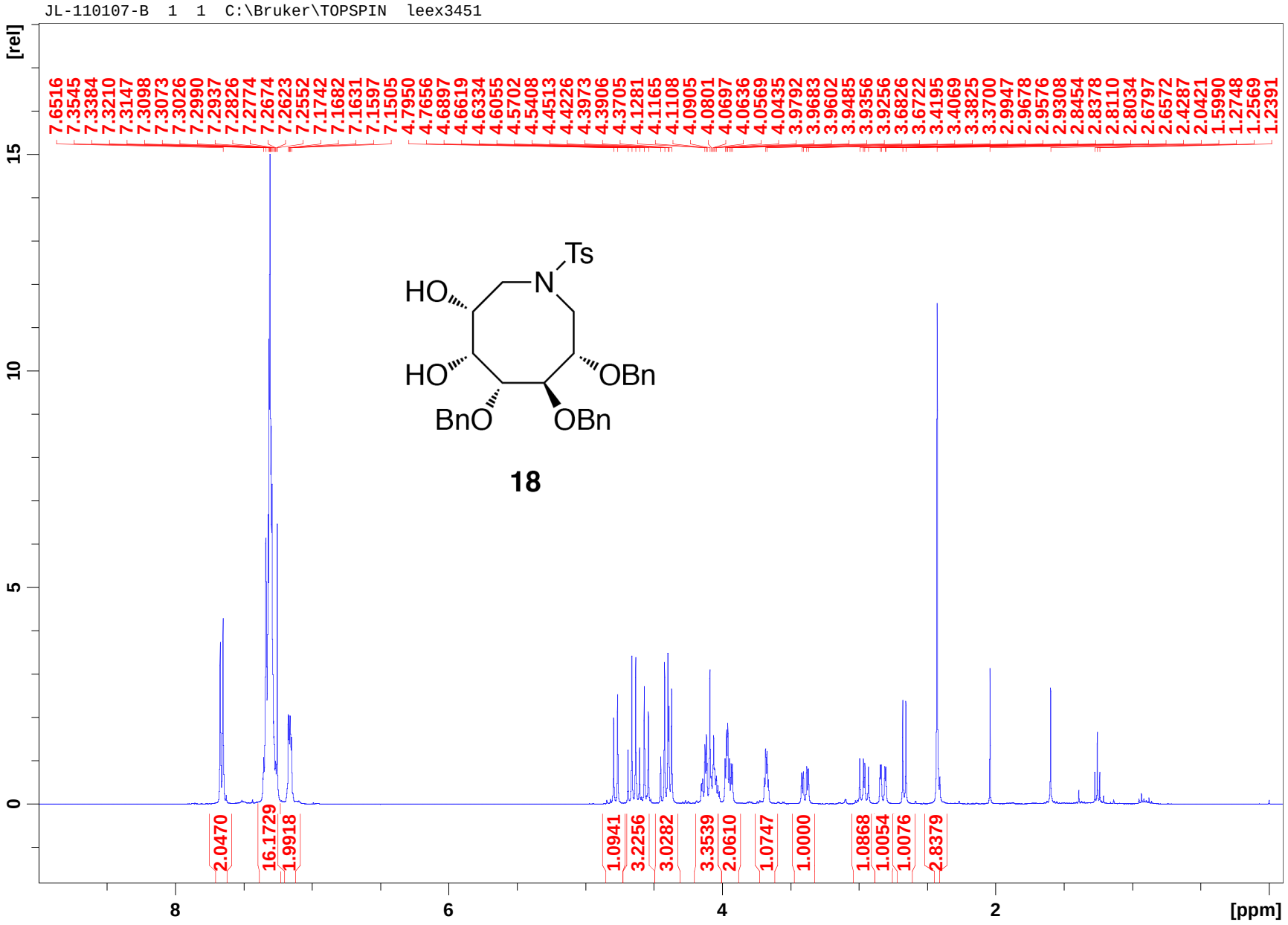
14.4345
N
OH
HO
OH
4
200
150
100
50
0
[ppm]

1H-NMR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[rel]
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0
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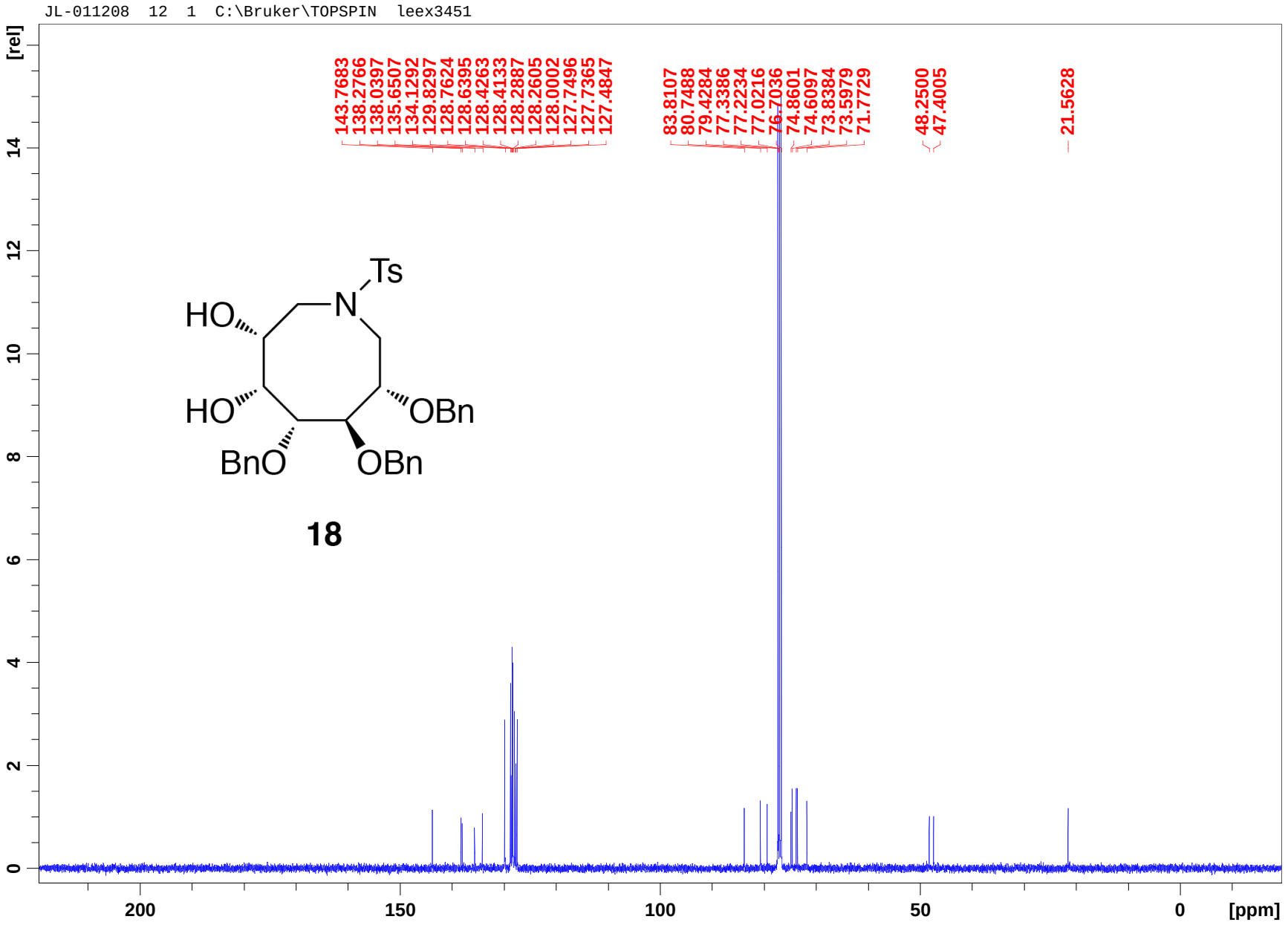
Ts
N
HO
HO
OBn
BnO
OBn
18
2.0470 16.1729 1.9918 1.0941 3.2256 3.0282 3.3539 2.0610 1.0747 1.0000 1.0868 1.0054 1.0076 2.8379
8
6
4
2
[ppm]

13C-NMR


JL-011208 12 1 C:\Bruker\TOPSPIN leex3451
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138.0397
135.6507
134.1292
129.8297
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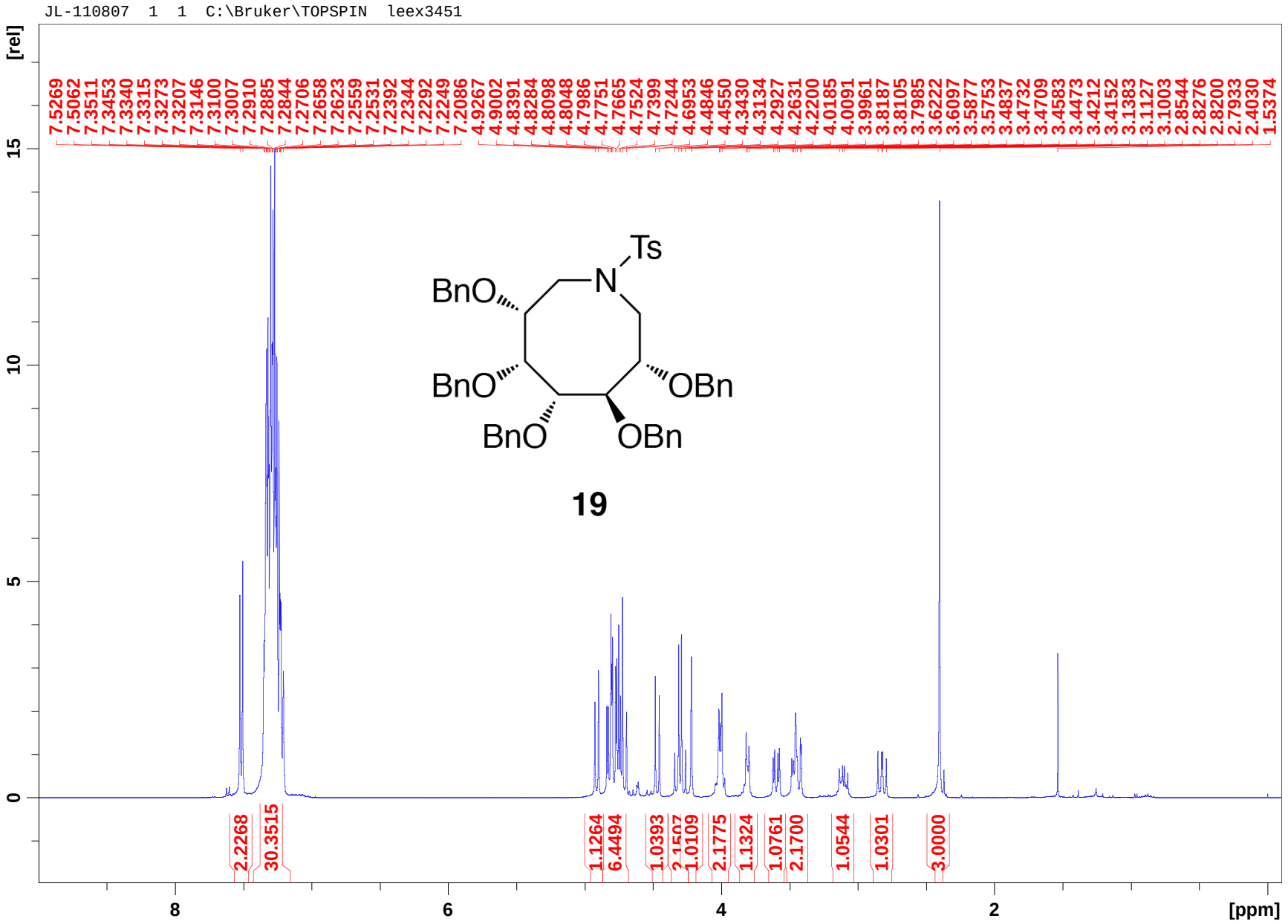
128.6395
128.4263
128.4133
128.2887
128.2605
128.0002
127.7496
127.7365
127.4847
83.8107
80.7488
79.4284
77.3386
77.2234
77.0216
76.7036
74.8601
74.6097
73.8384
73.5979
71.7729
48.2500
47.4005
21.5628
Ts
N
HO
HO
OBn
BnO
OBn
18
200
150
100
50
0
[ppm]

1H-NMR


JL-110807 1 1 C:\Bruker\TOPSPIN leex3451
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10
5
0
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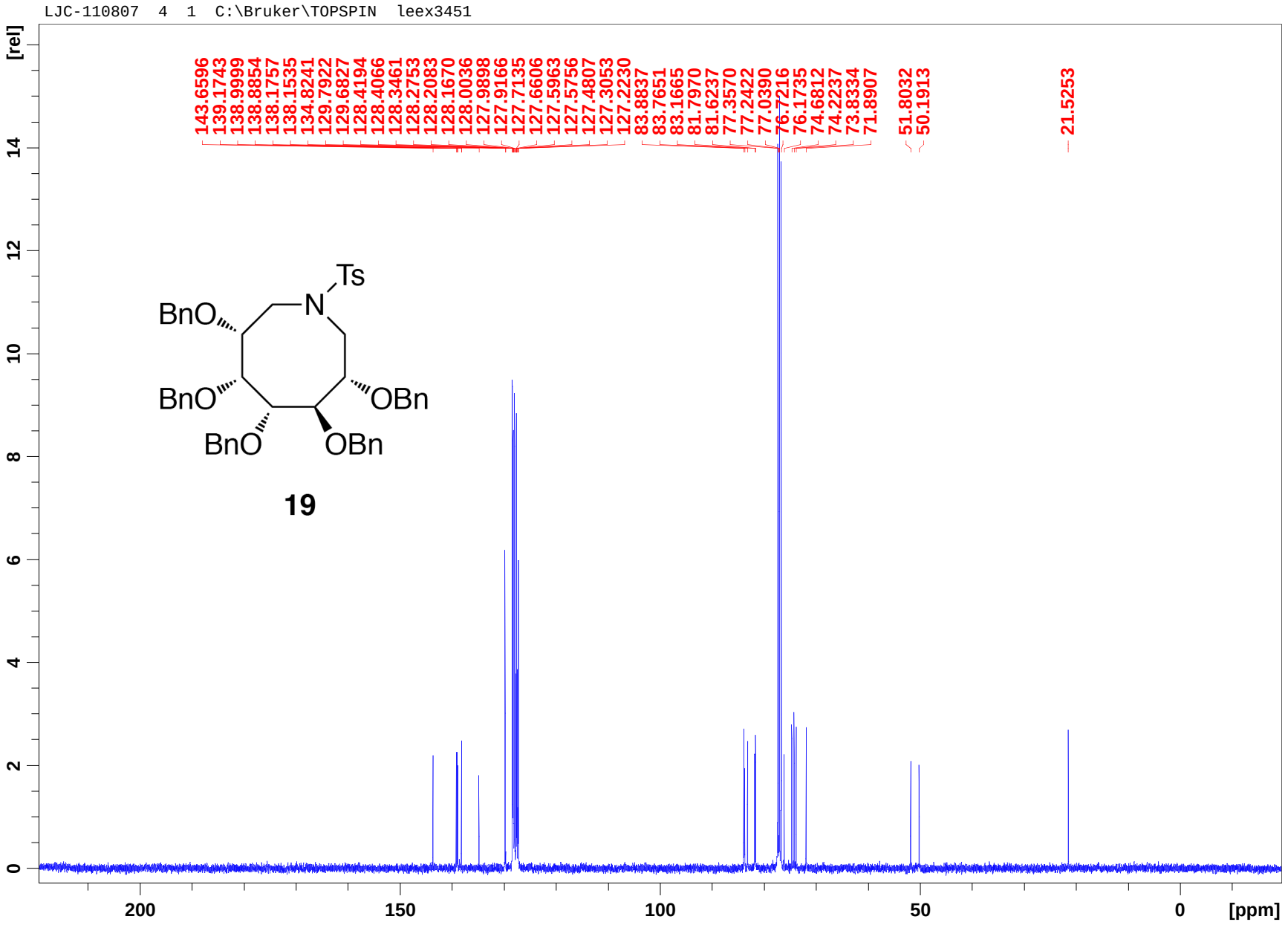
Ts
N
BnO
BnO
BnO
OBn
OBn
19
2.2268 30.3515 1.1264 6.4494 1.0393 2.1507 1.0109 2.1775 1.1324 1.0761 2.1700 1.0544 1.0301 3.0000
8
6
4
2
[ppm]

13C-NMR


LJC-110807 4 1 C:\Bruker\TOPSPIN leex3451
[rel]
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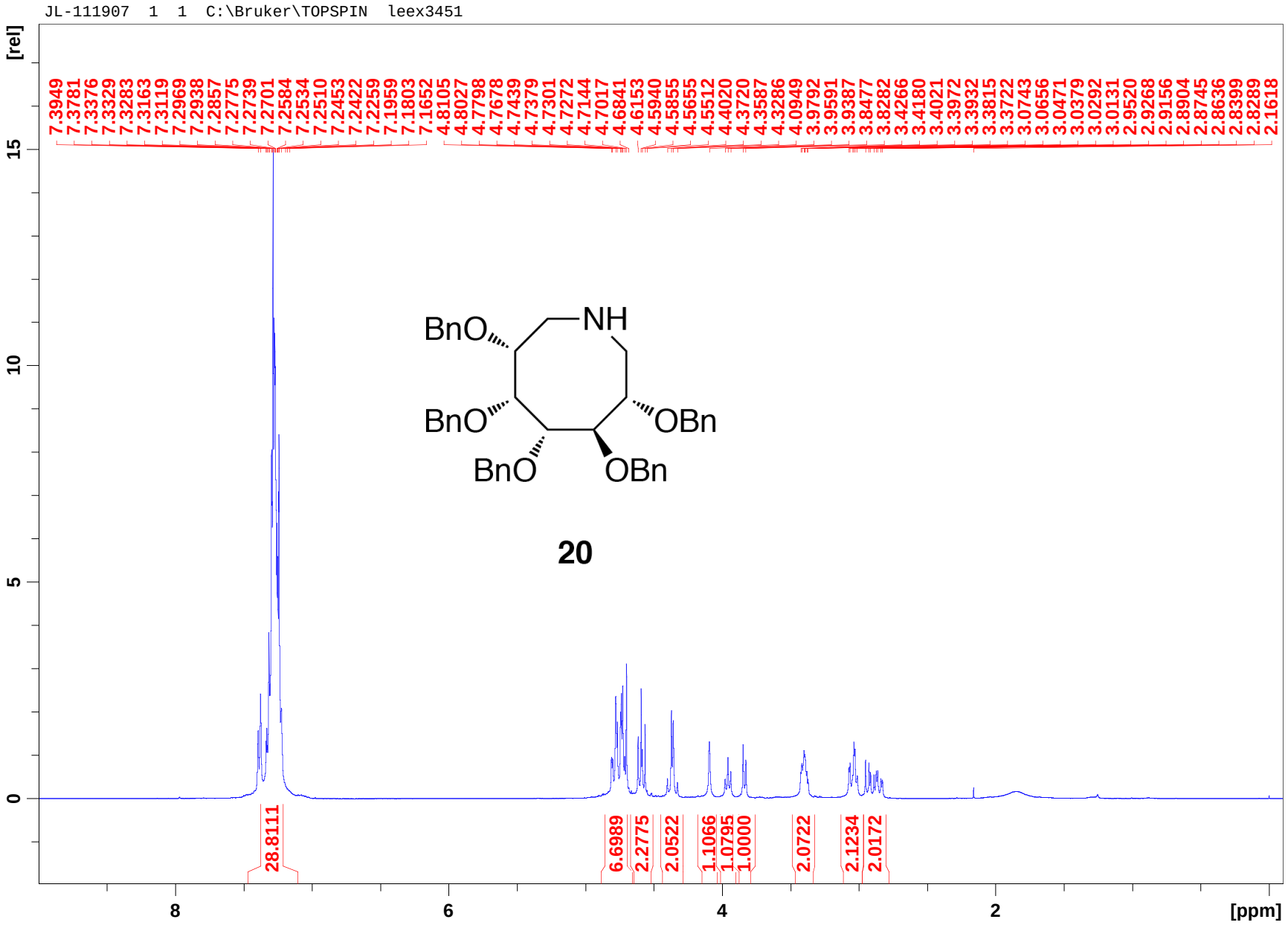
138.8854
138.1757
138.1535
134.8241
129.7922
129.6827
128.4194
128.4066
128.3461
128.2753
128.2083
128.1670
128.0036
127.9898
127.9166
127.7135
127.6606
127.5963
127.5756
127.4807
127.3053
127.2230
83.8837
83.7651
83.1665
81.7970
81.6237
77.3570
77.2422
77.0390
76.7216
76.1735
74.6812
74.2237
73.8334
71.8907
51.8032
50.1913
21.5253
Ts
N
BnO
BnO
BnO
OBn
OBn
19
0
2
4
6
8
10
12
14
200
150
100
50
0
[ppm]

1H-NMR


JL-111907 1 1 C:\Bruker\TOPSPIN leex3451
[rel]
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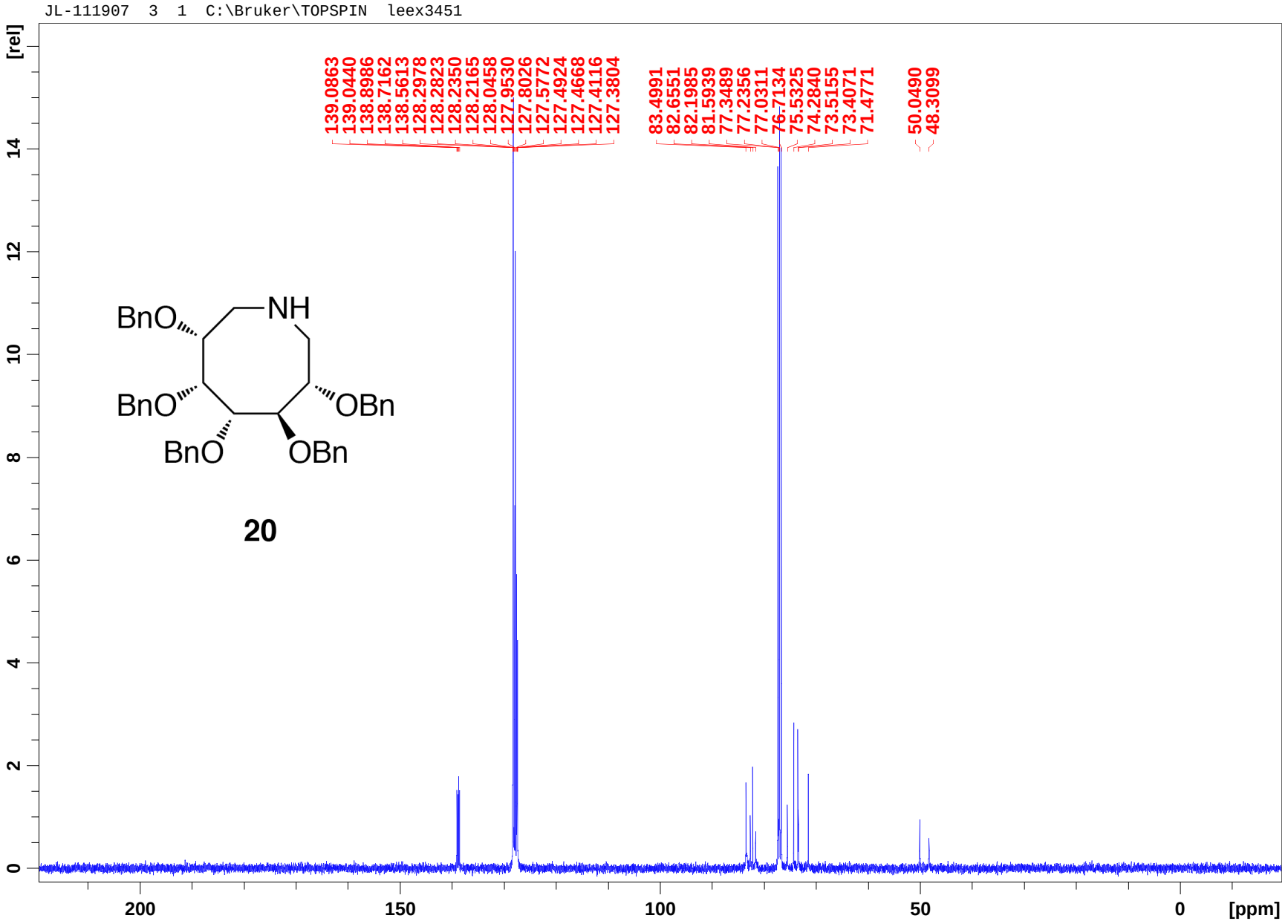
BnO
NH
BnO
OBn
BnO
OBn
20
28.8111
6.6989
2.2775
2.0522
1.1066
1.0795
1.0000
2.0722
2.1234
2.0172
8
6
4
2
[ppm]

13C-NMR


JL-111907 3 1 C:\Bruker\TOPSPIN leex3451
[rel]
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2
4
6
8
10
12
14
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138.7162
138.5613
128.2978
128.2823
128.2350
128.2165
128.0458
127.9530
127.8026
127.5772
127.4924
127.4668
127.4116
127.3804
83.4991
82.6551
82.1985
81.5939
77.3489
77.2356
77.0311
76.7134
75.5325
74.2840
73.5155
73.4071
71.4771
50.0490
48.3099
NH
BnO
BnO
BnO
OBn
OBn
20
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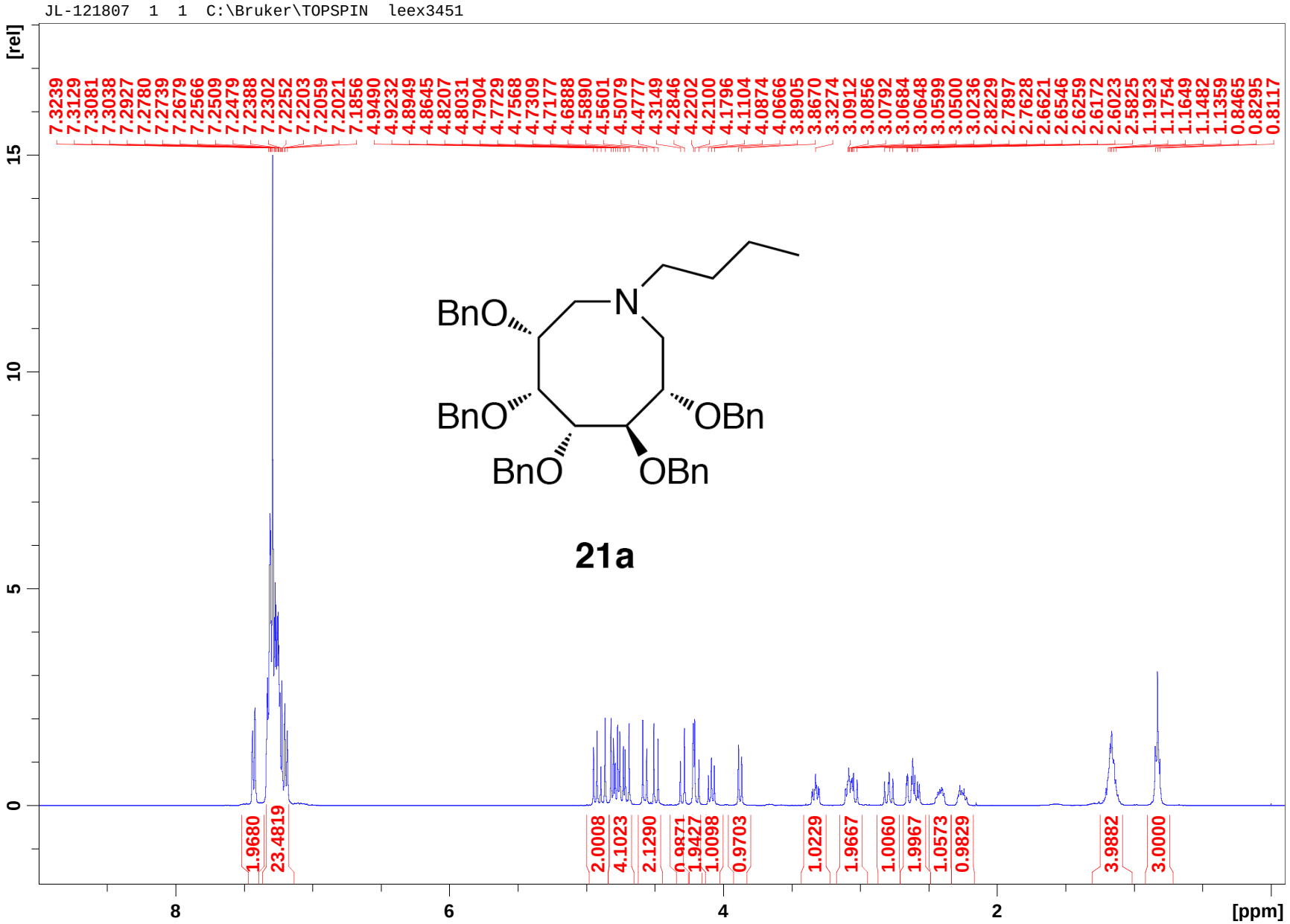
150
100
50
0
[ppm]

1H-NMR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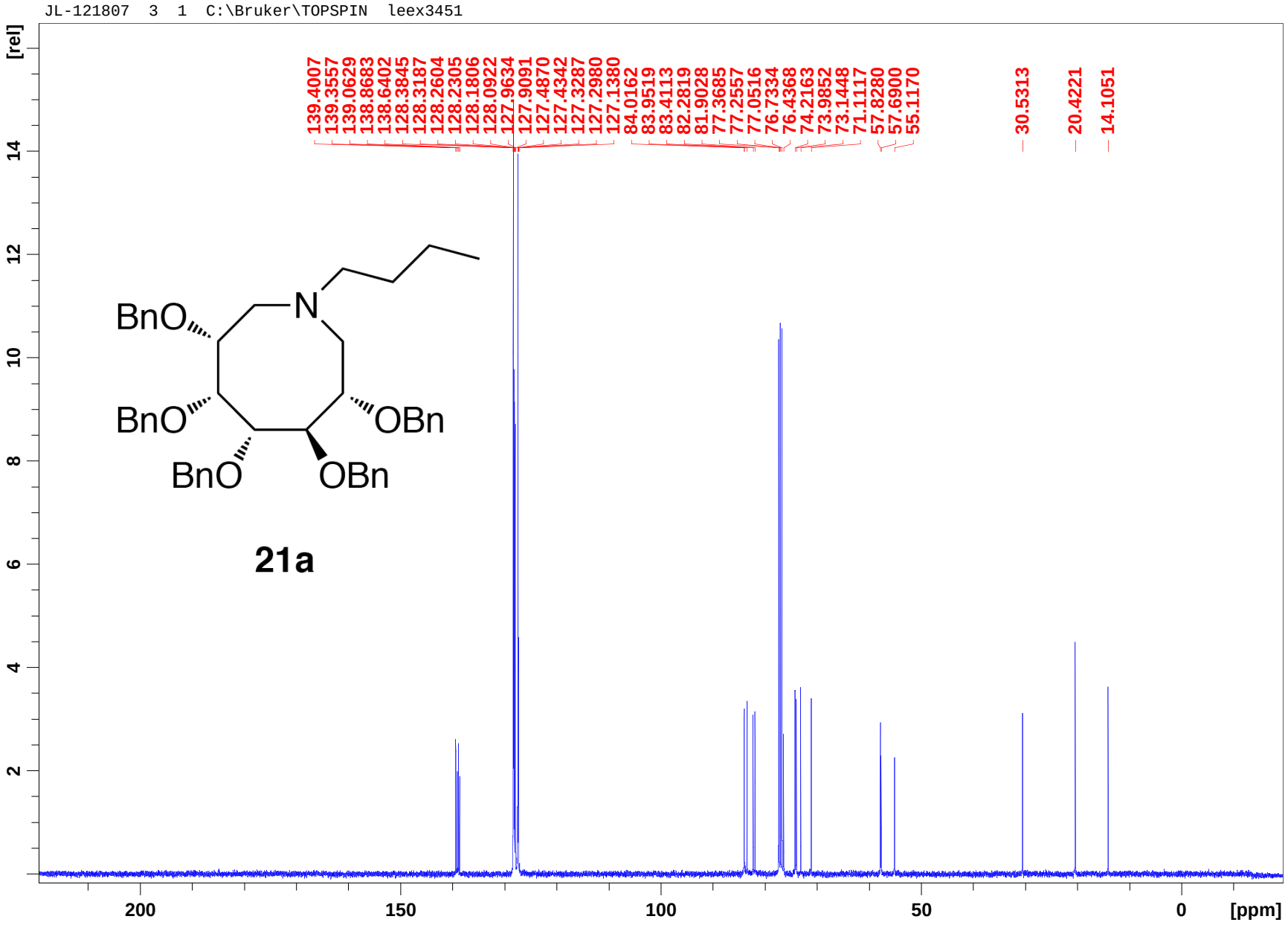
4.9490 4.9232 4.8949 4.8645 4.8207 4.8031 4.7904 4.7729 4.7568 4.7309 4.7177 4.6888 4.5890 4.5601 4.5079 4.4777 4.3149 4.2846 4.2202 4.2100 4.1796 4.1104 4.0874 4.0666 3.8905 3.8670 3.3274
3.0912 3.0856 3.0792 3.0684 3.0648 3.0599 3.0500 3.0236 2.8229 2.7897 2.7628 2.6621 2.6546 2.6259 2.6172 2.6023 2.5825 1.1923 1.1754 1.1649 1.1482 1.1359 0.8465 0.8295 0.8117
BnO
BnO
BnO
N
OBn
OBn
21a
1.9680 23.4819
2.0008 4.1023 2.1290 0.9871 1.9427 1.0098 0.9703 1.0229 1.9667 1.0060 1.9967 1.0573 0.9829 3.9882 3.0000
8
6
4
2
[ppm]

13C-NMR


JL-121807 3 1 C:\Bruker\TOPSPIN leex3451
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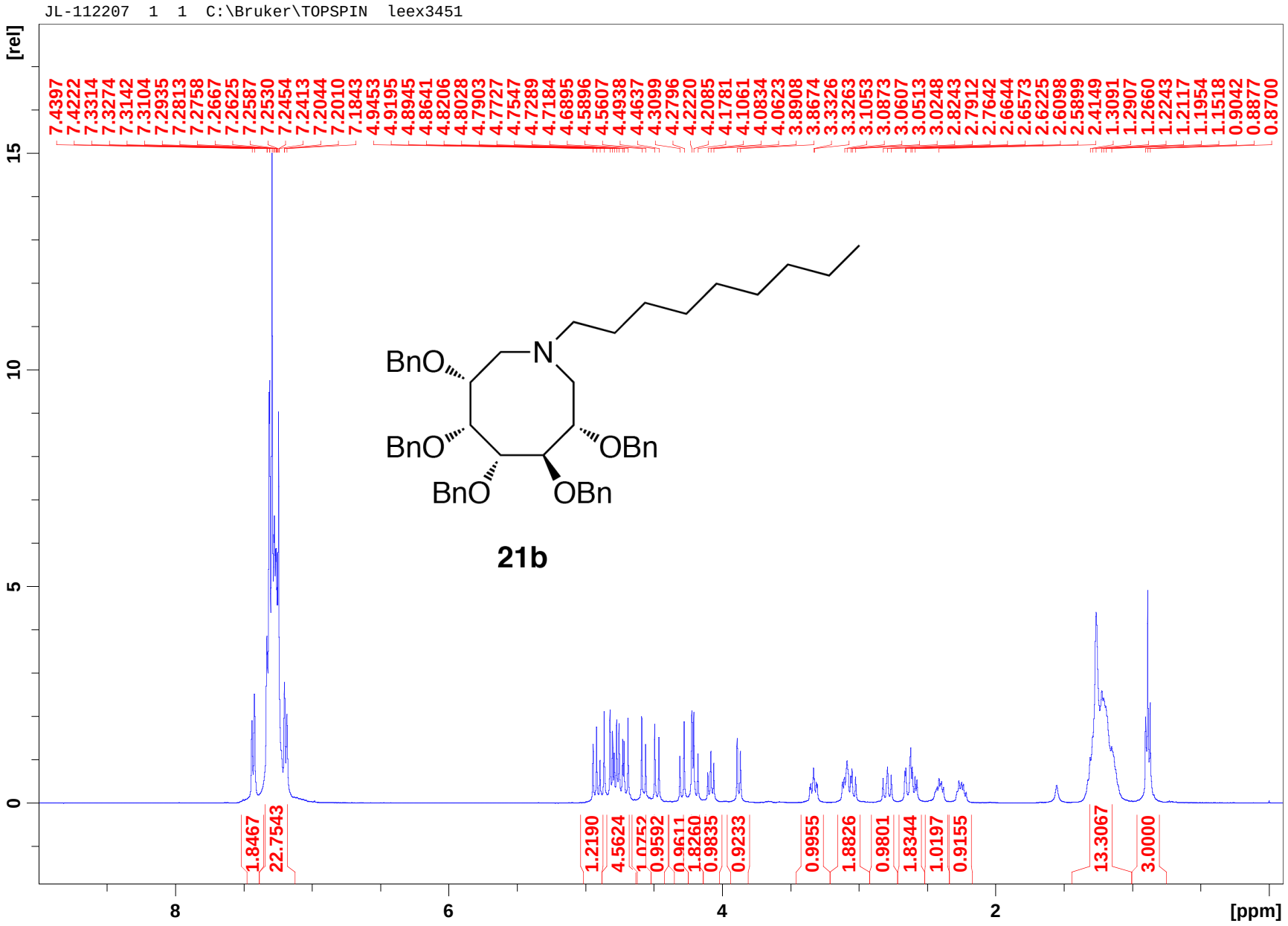
128.3187
128.2604
128.2305
128.1806
128.0922
127.9634
127.9091
127.4870
127.4342
127.3287
127.2980
127.1380
84.0162
83.9519
83.4113
82.2819
81.9028
77.3685
77.2557
77.0516
76.7334
76.4368
74.2163
73.9852
73.1448
71.1117
57.8280
57.6900
55.1170
30.5313
20.4221
14.1051
BnO
BnO
BnO
OBn
OBn
N
21a
200
150
100
50
0
[ppm]

1H-NMR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[rel]
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10
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4.9453 4.9195 4.8945 4.8641 4.8206 4.8028 4.7903 4.7727 4.7547 4.7289 4.7184 4.6895 4.5896 4.5607 4.4938 4.4637 4.3099 4.2796 4.2220 4.2085 4.1781 4.1061 4.0834 4.0623 3.8908 3.8674 3.3326 3.3263 3.1053 3.0873 3.0607 3.0513 3.0248 2.8243 2.7912 2.7642 2.6644 2.6573 2.6225 2.6098 2.5899 2.4149 1.3091 1.2907 1.2660 1.2243 1.2117 1.1954 1.1518 0.9042 0.8877 0.8700
BnO
BnO
BnO
N
OBn
OBn
21b
1.8467 22.7543
1.2190 4.5624 1.0752 0.9592 0.9611 1.8260 0.9835 0.9233
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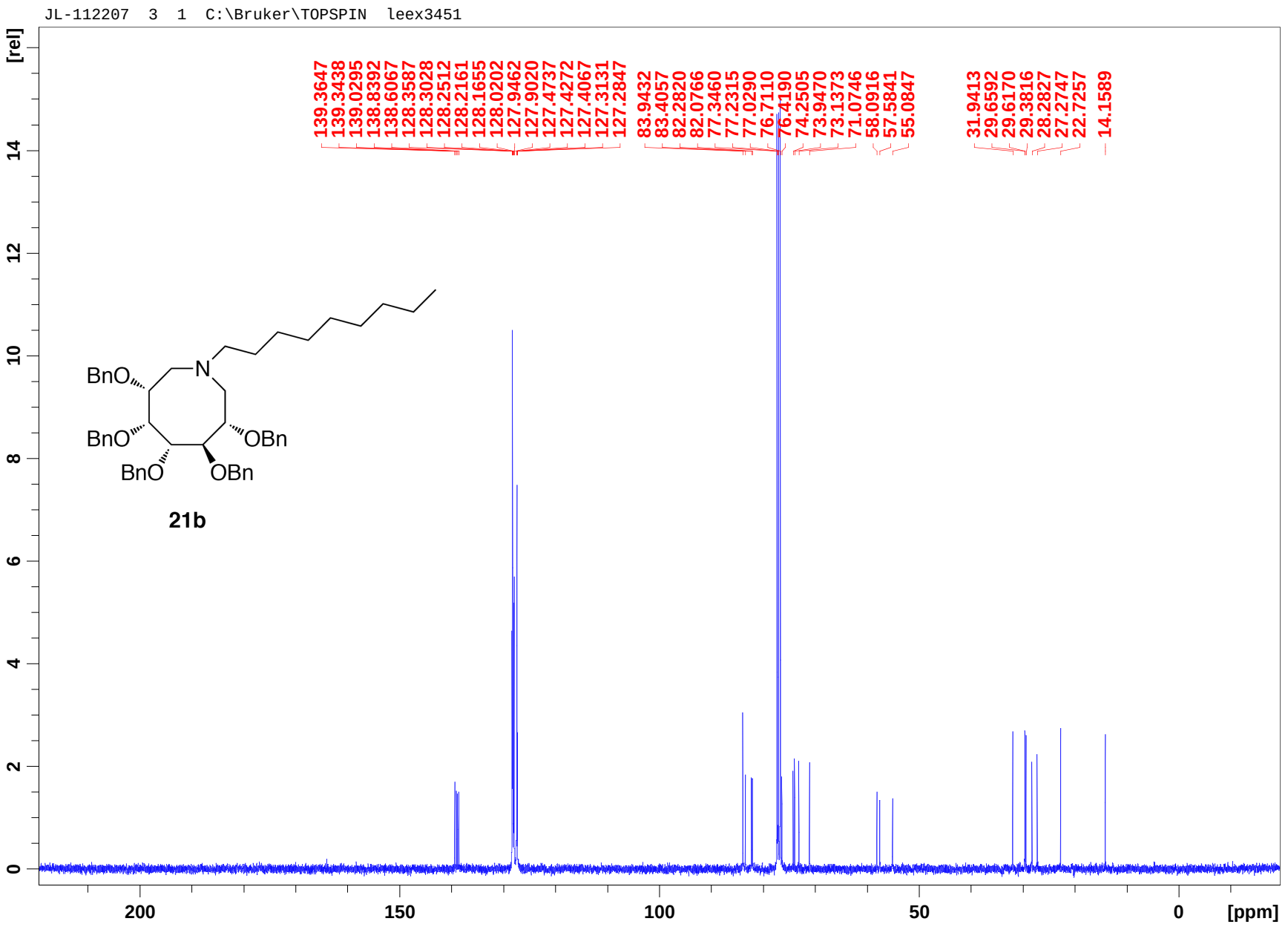
8
6
4
2
[ppm]

13C-NMR


JL-112207 3 1 C:\Bruker\TOPSPIN leex3451
[rel]
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2
4
6
8
10
12
14
139.3647
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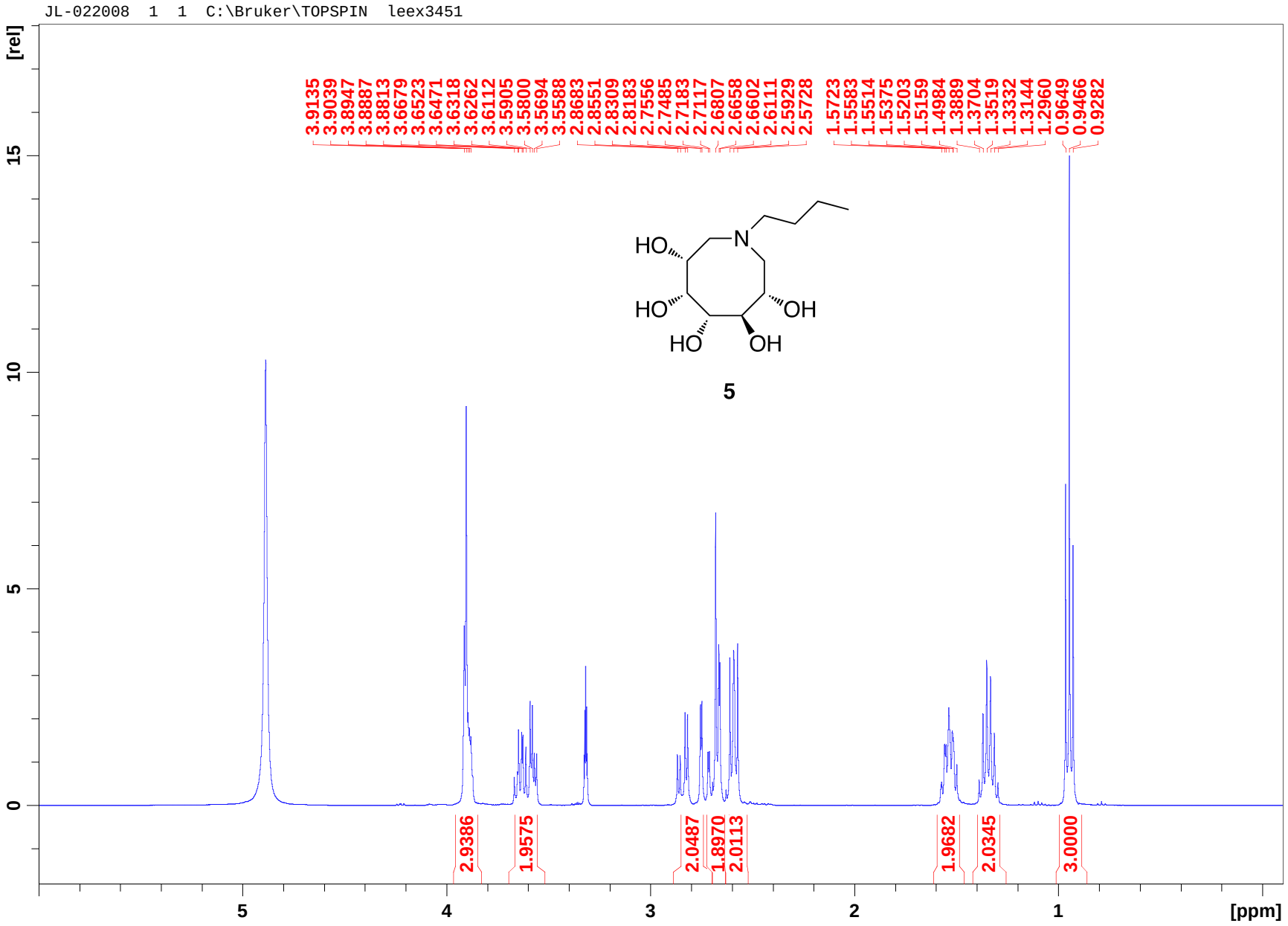
139.0295
138.8392
138.6067
128.3587
128.3028
128.2512
128.2161
128.1655
128.0202
127.9462
127.9020
127.4737
127.4272
127.4067
127.3131
127.2847
83.9432
83.4057
82.2820
82.0766
77.3460
77.2315
77.0290
76.7110
76.4190
74.2505
73.9470
73.1373
71.0746
58.0916
57.5841
55.0847
31.9413
29.6592
29.6170
29.3816
28.2827
27.2747
22.7257
14.1589
BnO
BnO
BnO
N
OBn
OBn
21b
200
150
100
50
0
[ppm]

1H-NMR


JL-022008 1 1 C:\Bruker\TOPSPIN leex3451
[rel]
15
10
5
0
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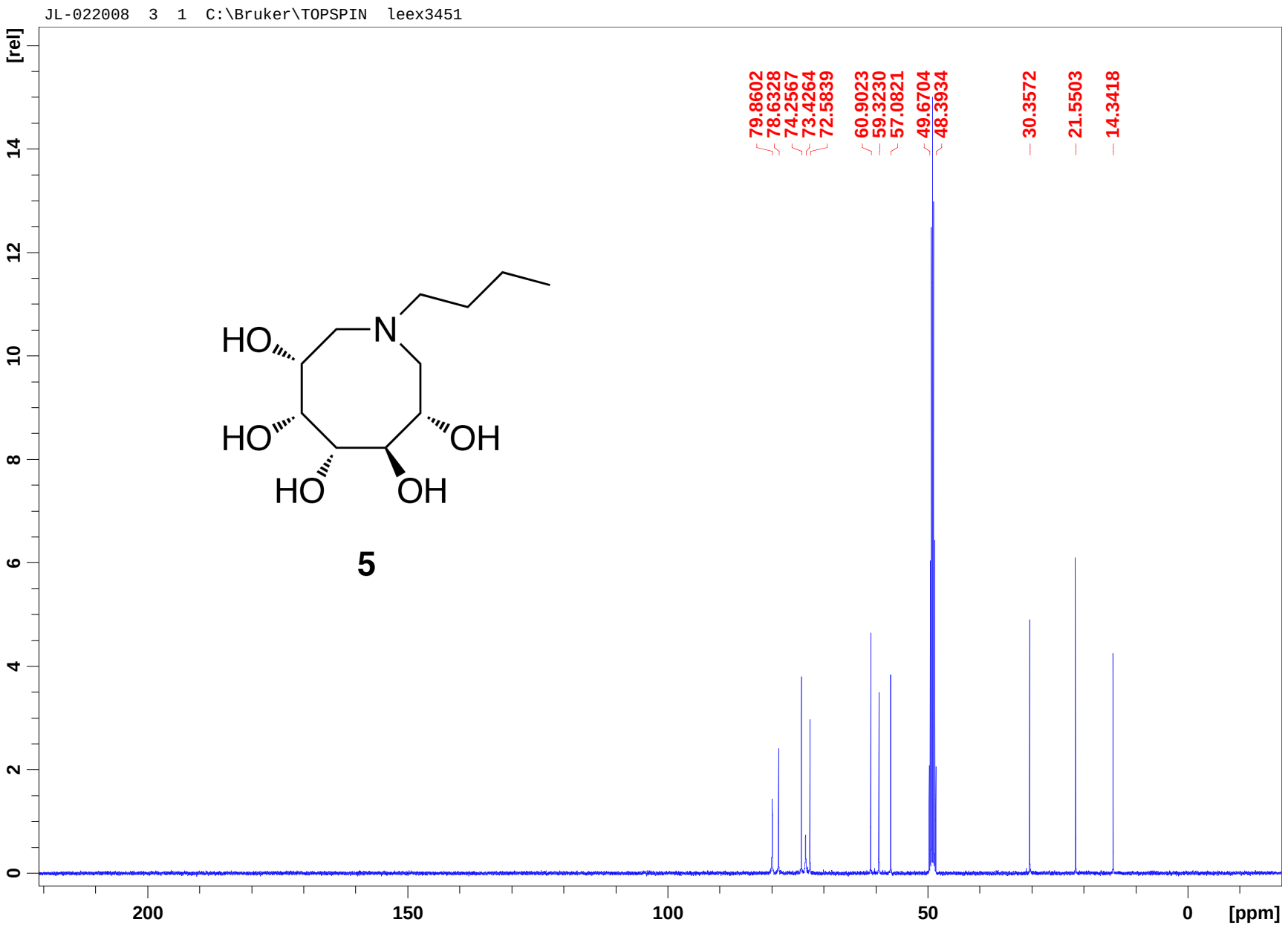
3.9039
3.8947
3.8887
3.8813
3.6679
3.6523
3.6471
3.6318
3.6262
3.6112
3.5905
3.5800
3.5694
3.5588
2.8683
2.8551
2.8309
2.8183
2.7556
2.7485
2.7183
2.7117
2.6807
2.6658
2.6602
2.6111
2.5929
2.5728
1.5723
1.5583
1.5514
1.5375
1.5203
1.5159
1.4984
1.3889
1.3704
1.3519
1.3332
1.3144
1.2960
0.9649
0.9466
0.9282
HO
HO
HO
N
OH
OH
5
2.9386
1.9575
2.0487
1.8970
2.0113
1.9682
2.0345
3.0000
5
4
3
2
1
[ppm]

13C-NMR


JL-022008 3 1 C:\Bruker\TOPSPIN leex3451
[rel]
0
2
4
6
8
10
12
14
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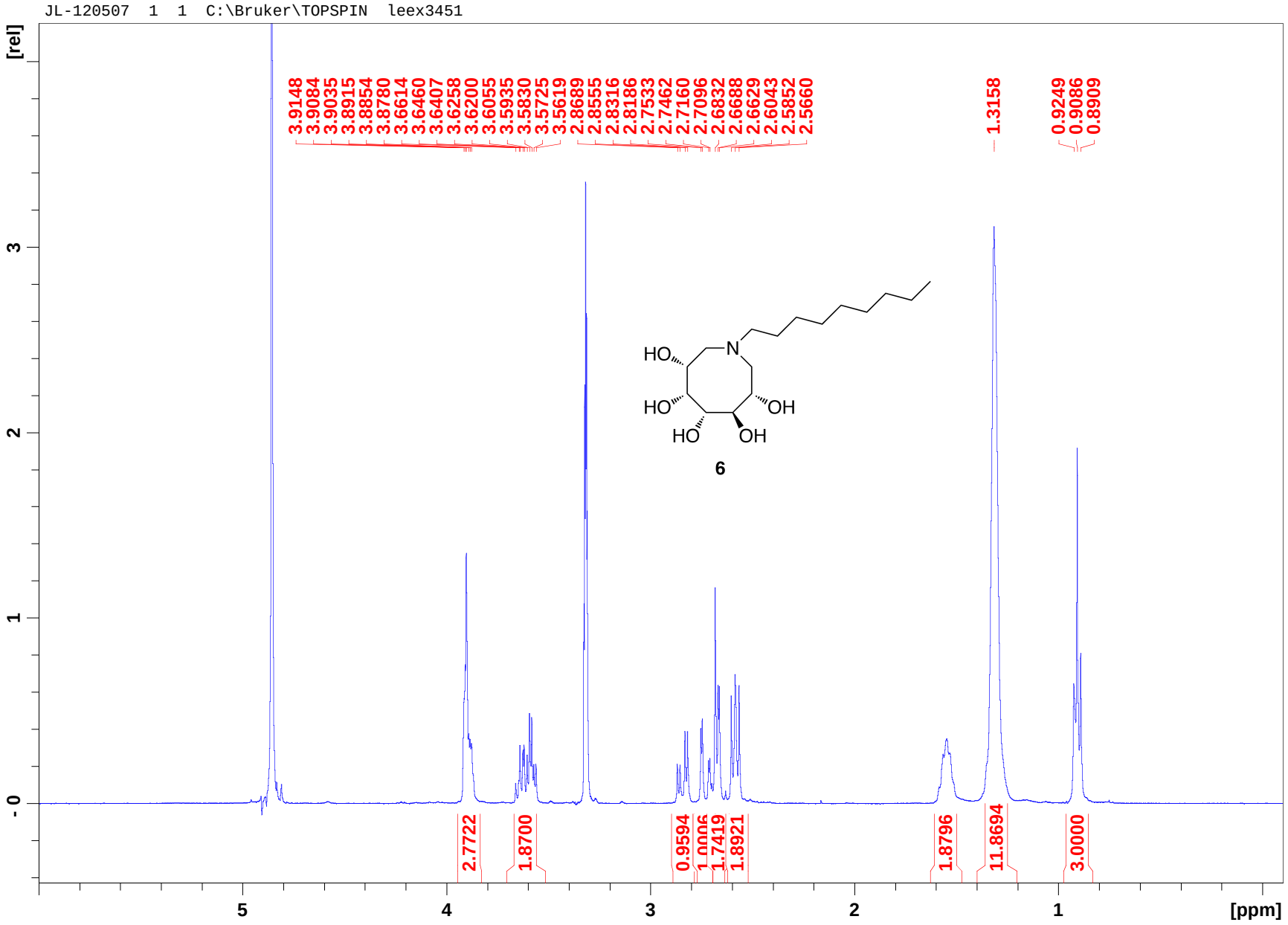
78.6328
74.2567
73.4264
72.5839
60.9023
59.3230
57.0821
49.6704
48.3934
30.3572
21.5503
14.3418
HO
HO
HO
N
OH
OH
200
150
100
50
0
[ppm]

5

1H-NMR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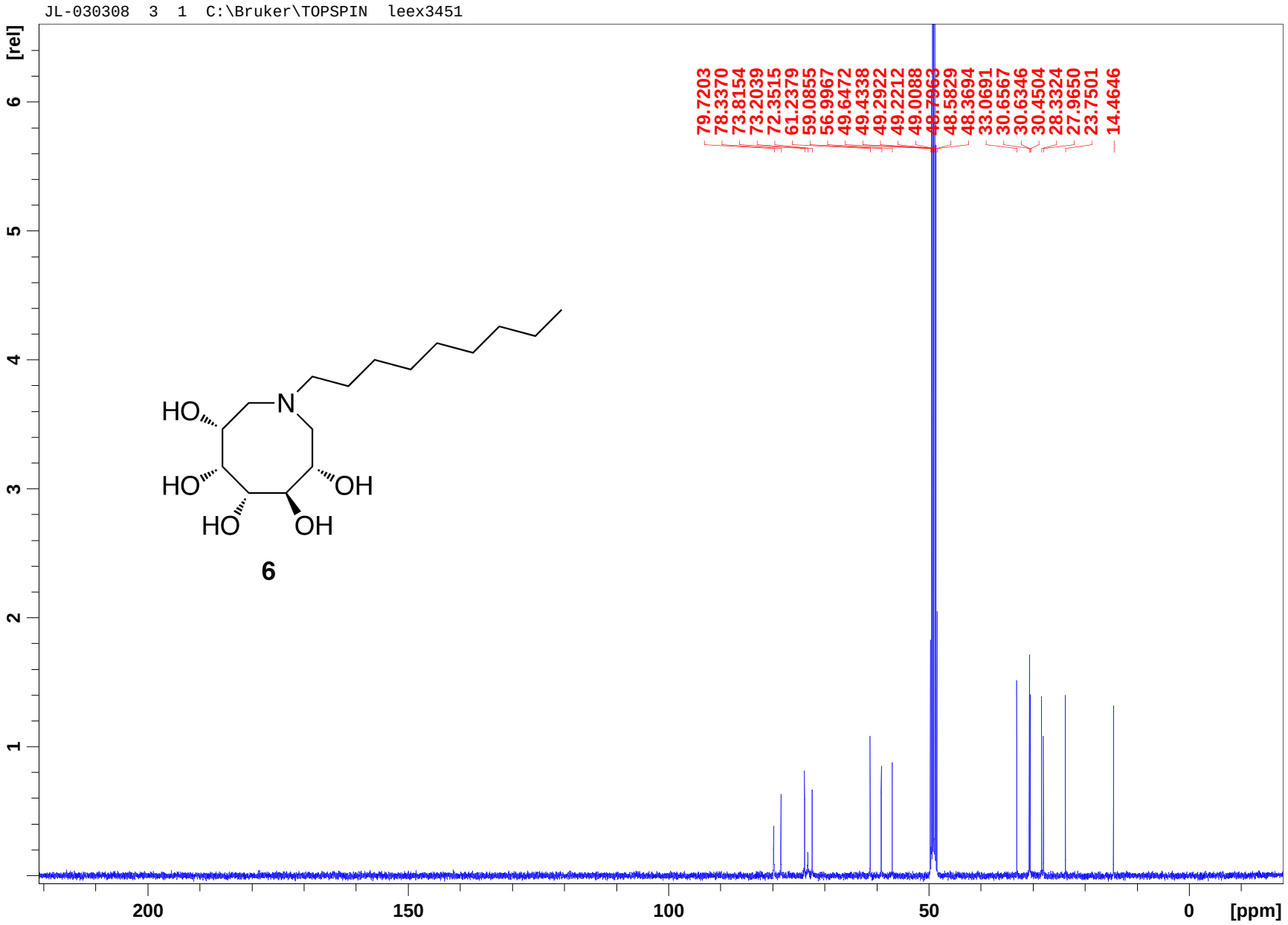
3.9035
3.8915
3.8854
3.8780
3.6614
3.6460
3.6407
3.6258
3.6200
3.6055
3.5935
3.5830
3.5725
3.5619
2.8689
2.8555
2.8316
2.8186
2.7533
2.7462
2.7160
2.7096
2.6832
2.6688
2.6629
2.6043
2.5852
2.5660
1.3158
0.9249
0.9086
0.8909
HO
N
HO
OH
HO
OH
6
2.7722
1.8700
0.9594
1.7419
1.8921
1.8796
11.8694
3.0000
[ppm]

13C-NMR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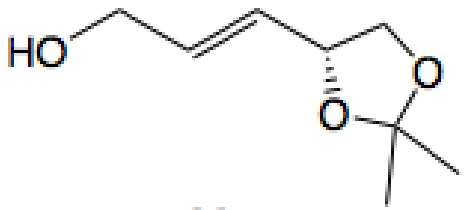
78.3370
73.8154
73.2039
72.3515
61.2379
59.0855
56.9967
49.6472
49.4338
49.2922
49.2212
49.0088
48.7963
48.5829
48.3694
33.0691
30.6567
30.6346
30.4504
28.3324
27.9650
23.7501
14.4646
HO
N
HO
OH
HO
OH
6
200
150
100
50
0
[ppm]

Jul18-2007-ranga
r,r alcohol

1H-NMR

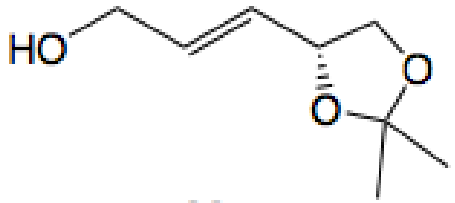


29

Jul18-2007-ranga
r,r alcohol

13C-NMR

133.71
133.41
128.88
127.81
109.30
109.25
77.78
77.46
77.14
76.52 CDCl3
71.87
69.36
69.26
61.92
58.03
26.63
25.87
25.83
-0.00



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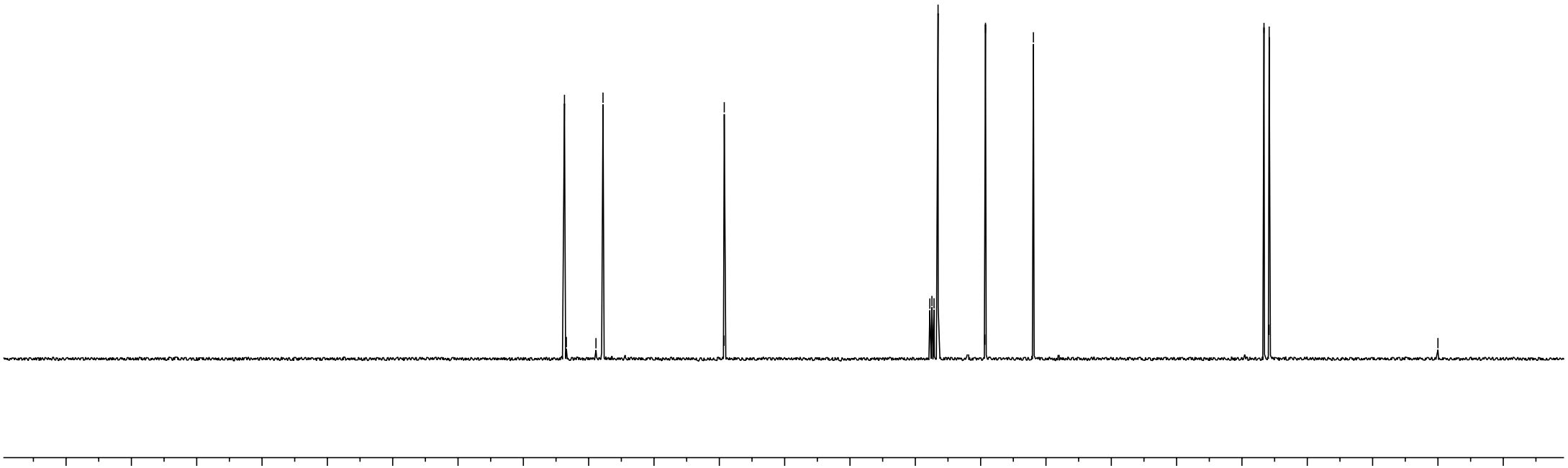
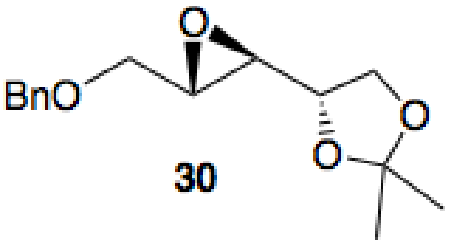


Jun01-2007-ranga
monobenz

1H-NMR

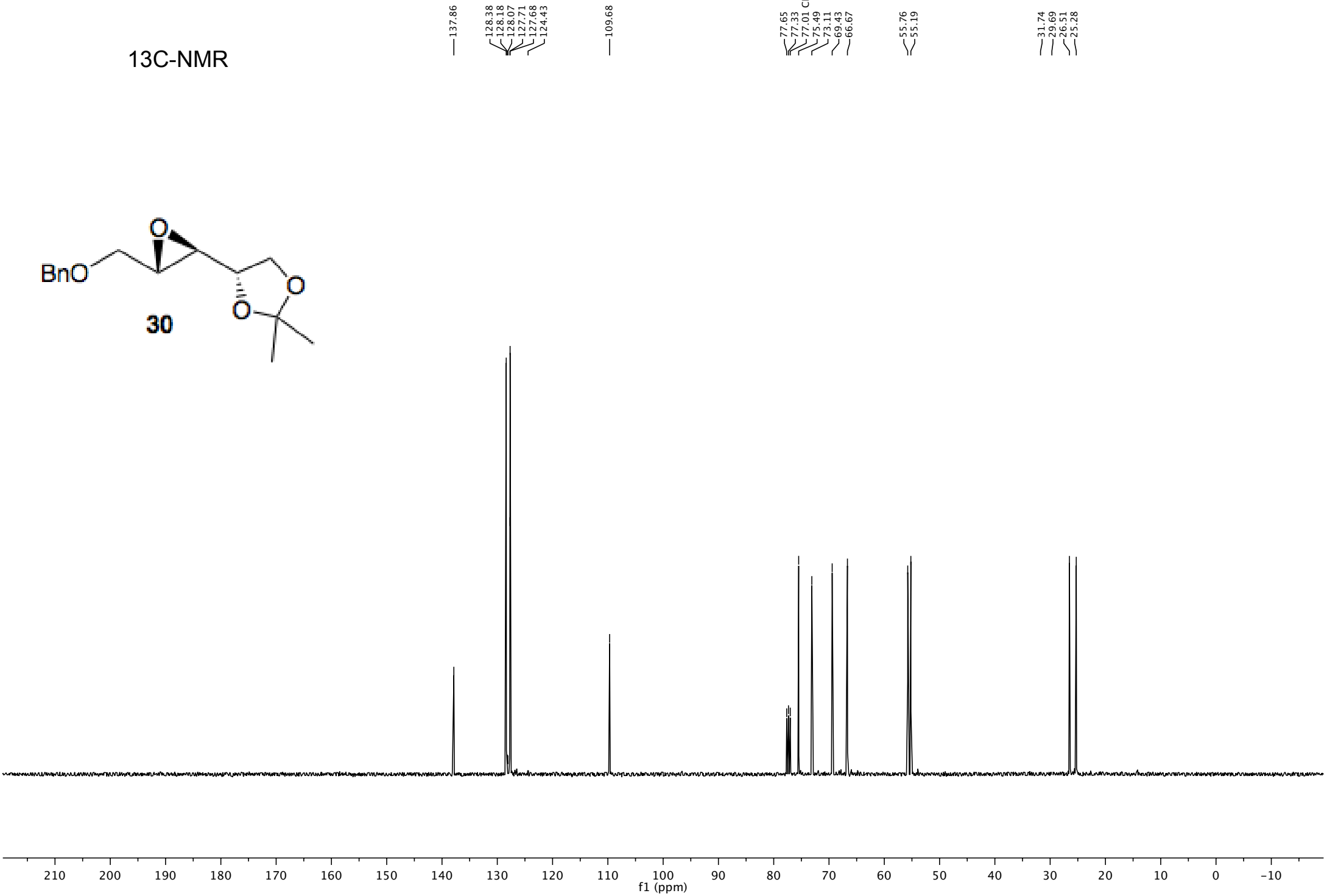


30

Jun01-2007-ranga
monobenz

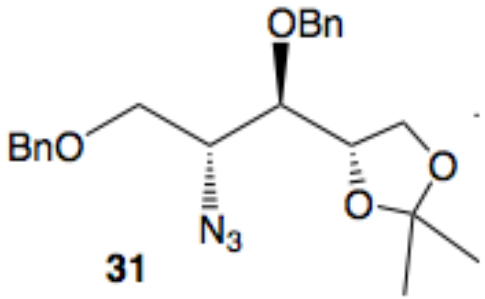
13C-NMR

30



Aug10-2007-ranga/10
azide-obn

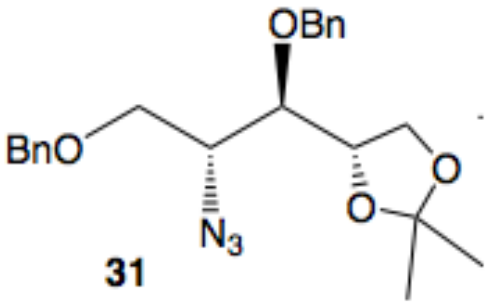
1H-NMR



31

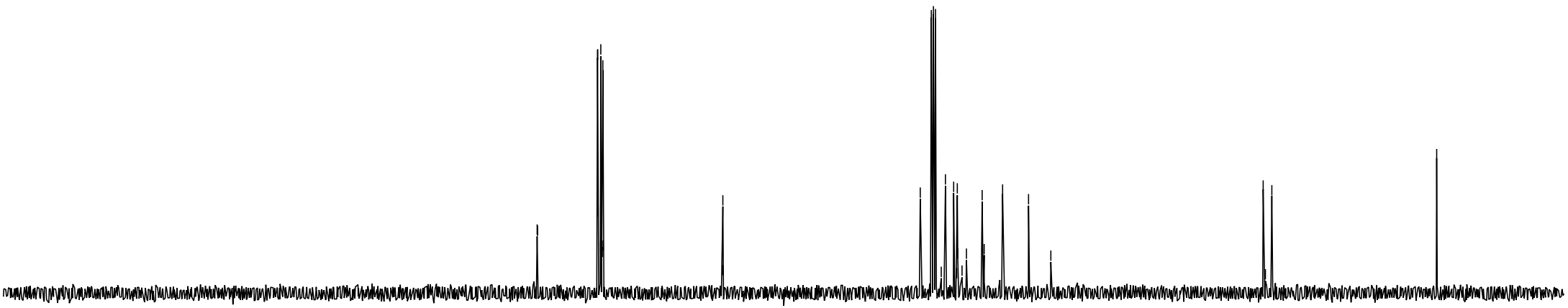
Aug10-2007-ranga/11
azide-obn

13C-NMR



31

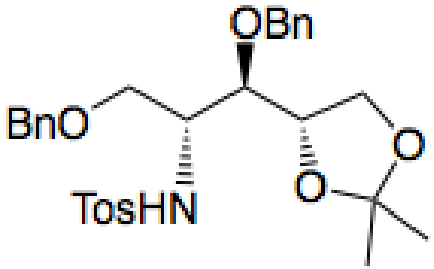
137.72
137.65
128.48
128.46
128.38
128.01
127.98
127.97
127.93
127.79
127.75
127.67
127.62
109.33
109.30
79.08
77.38
77.06 CDCl3
76.75 CDCl3
75.87
75.23
73.97
73.50
73.41
73.34
72.68
72.01
69.60
69.30
66.47
62.50
59.09
36.65
26.59
26.25
25.26
0.03



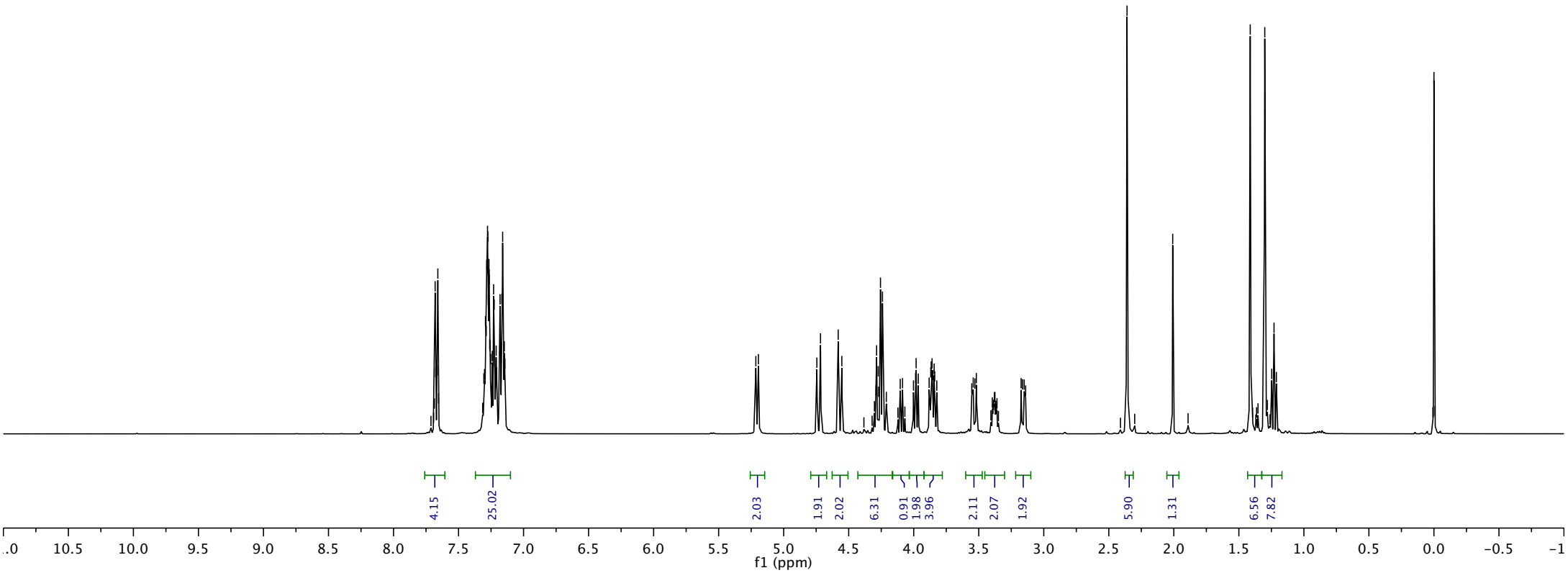
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Aug15-2007-ranga
r,r N-Ts prdt

1H-NMR



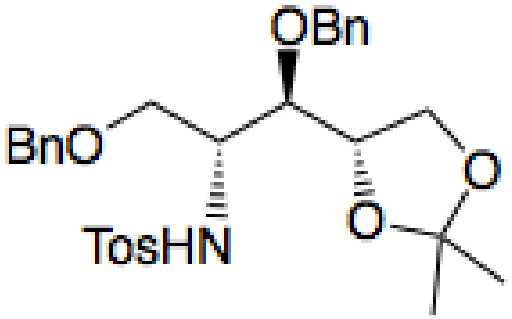
32



Aug15-2007-ranga
r,r N-Ts prdt

13C-NMR

171.04
143.38
138.35
138.07
137.49
137.23
129.57
129.45
128.34
128.32
128.29
127.86
127.83
127.76
127.70
127.68
127.13
108.99
78.09
77.46
77.14 CDCl3
76.82
75.93
74.65
72.96
67.35
65.47
60.32
54.58
36.62
26.41
24.95
21.47
20.98
14.17
0.00



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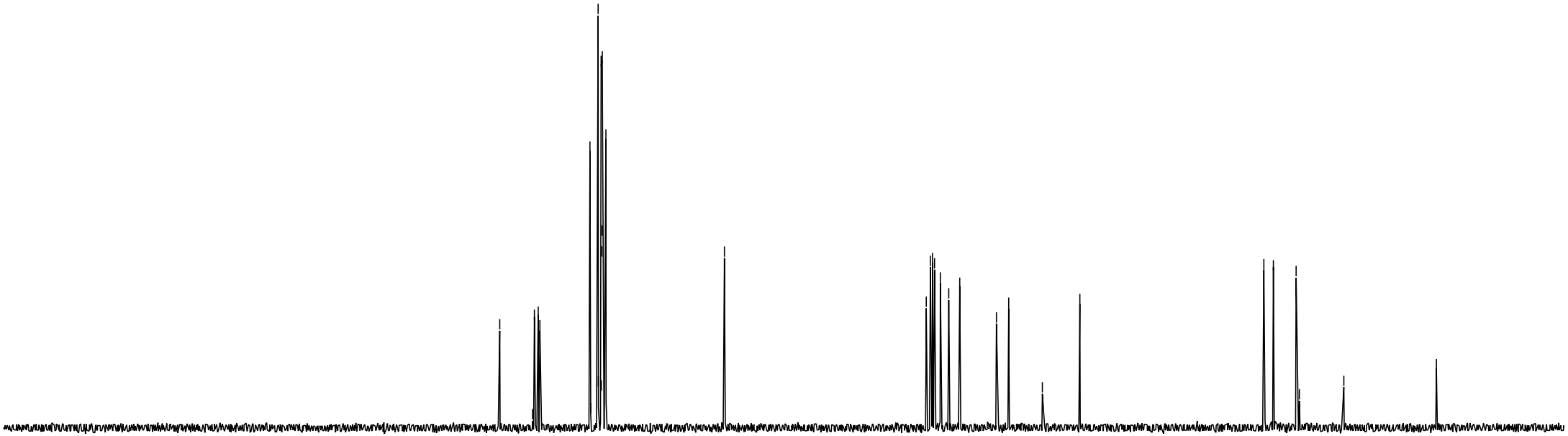
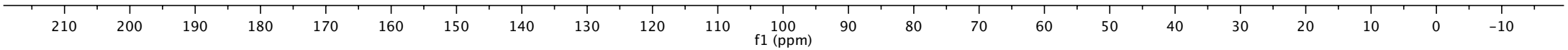
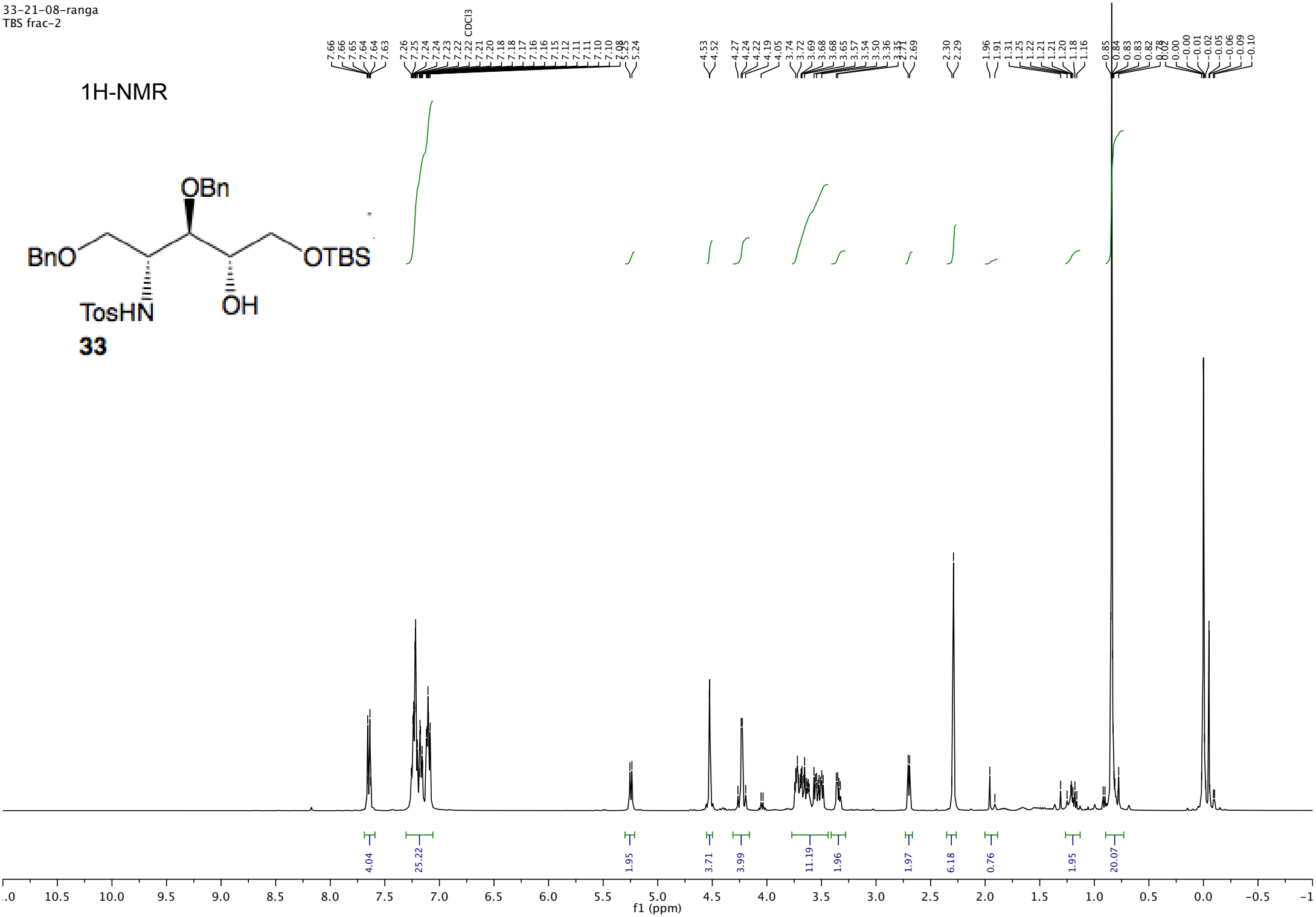




33-21-08-ranga
TBS frac-2

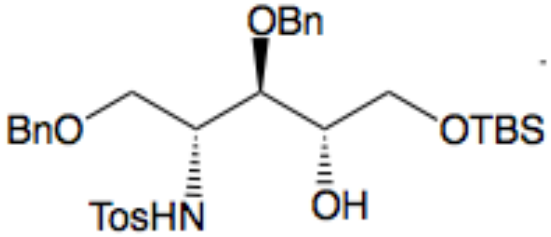
1H-NMR

33



Jun22-2007-ranga
fully prot compd - large scale

13C-NMR



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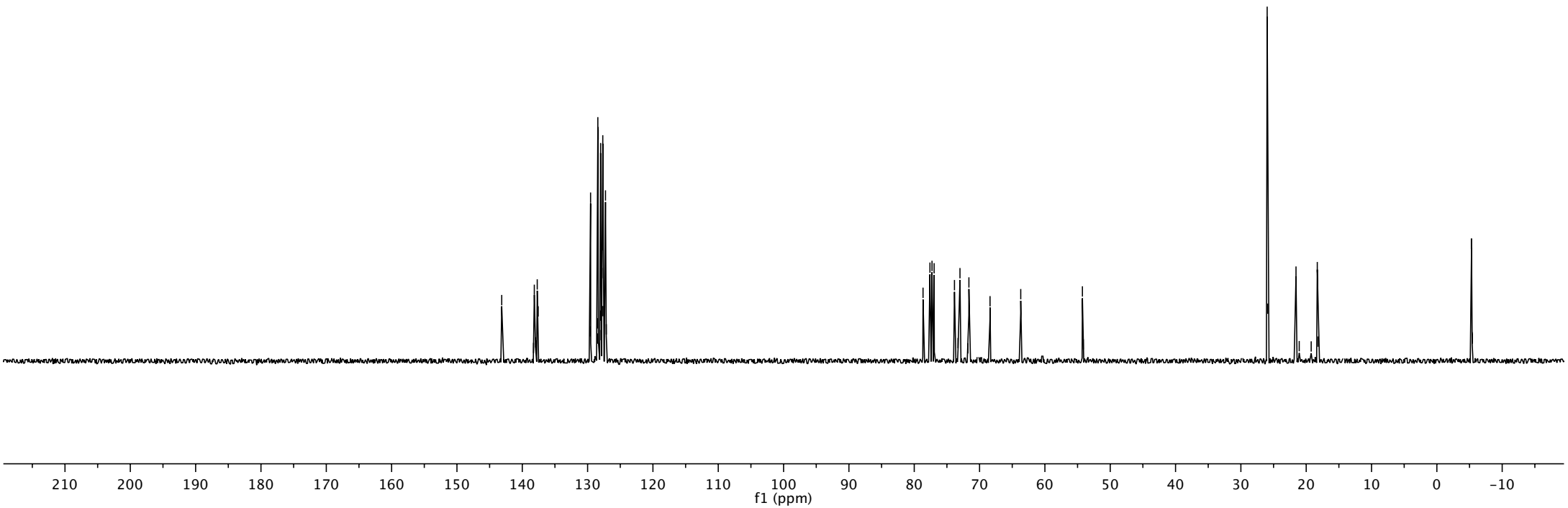
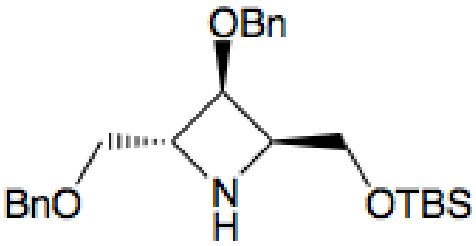


Jan14-2008-ranga
r,r nanaph smp J

1H-NMR

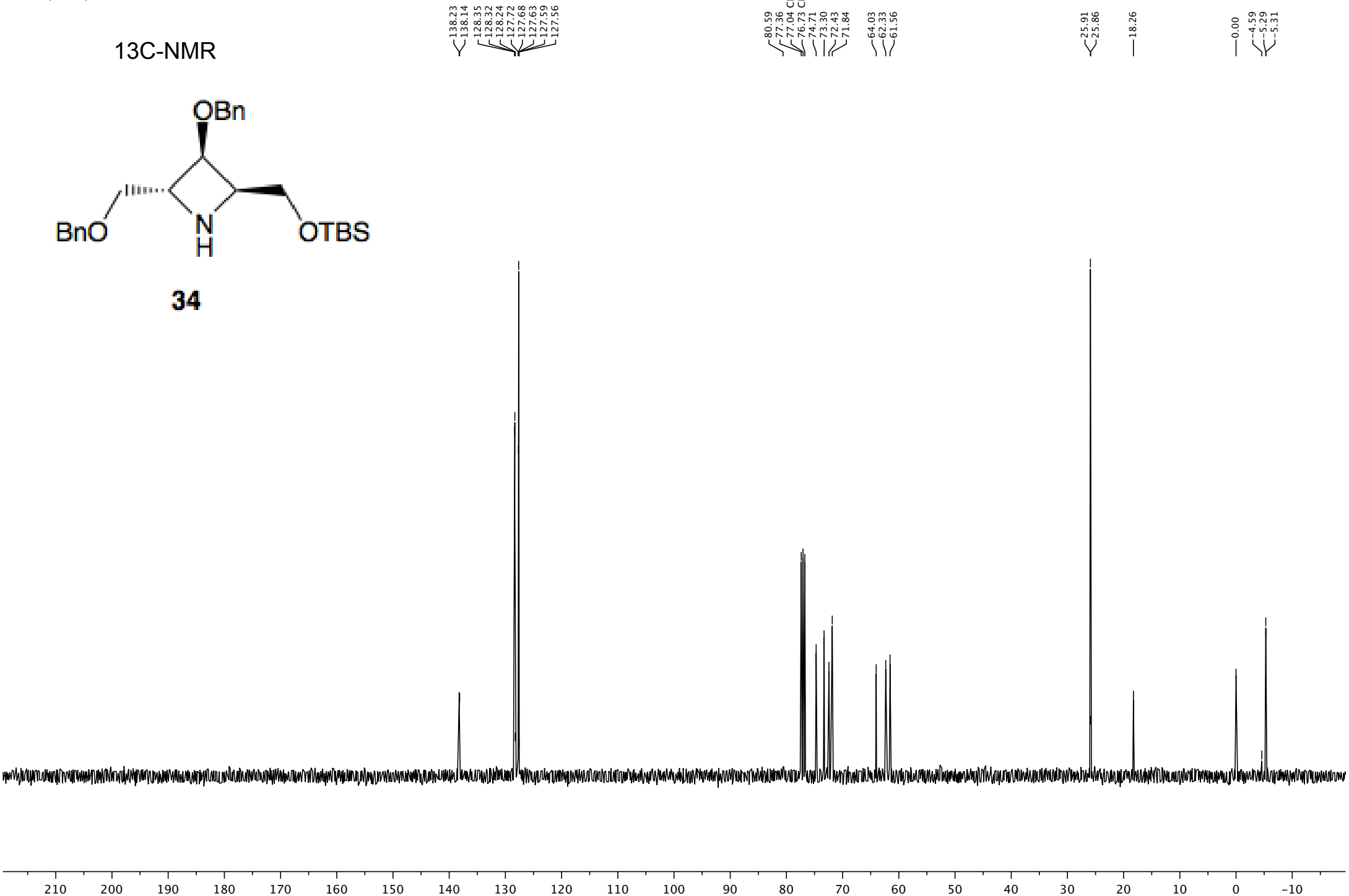


34

Jan14-2008-ranga
r,r nanaph smp J

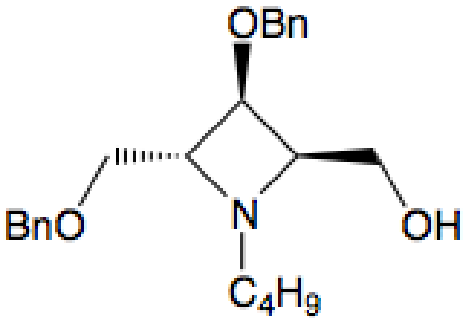
13C-NMR

34



26-07-11-ranga

1H-NMR

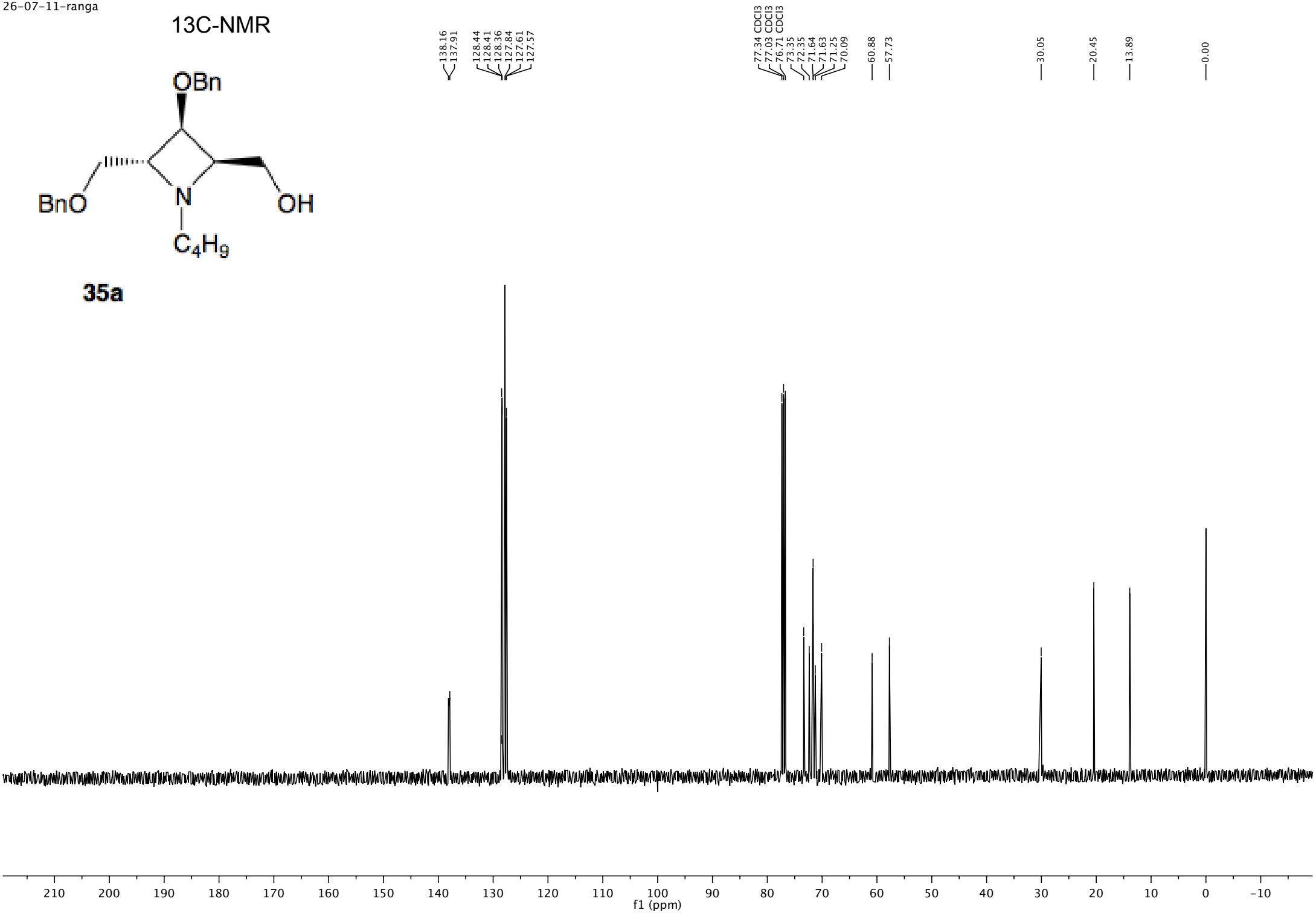


35a

26-07-11-ranga

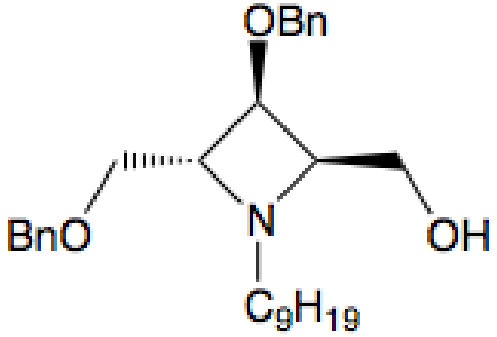
13C-NMR

35a



Feb01-2008-ranga
r,r n-non f3

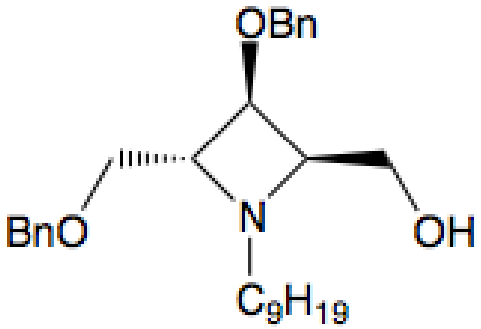
1H-NMR



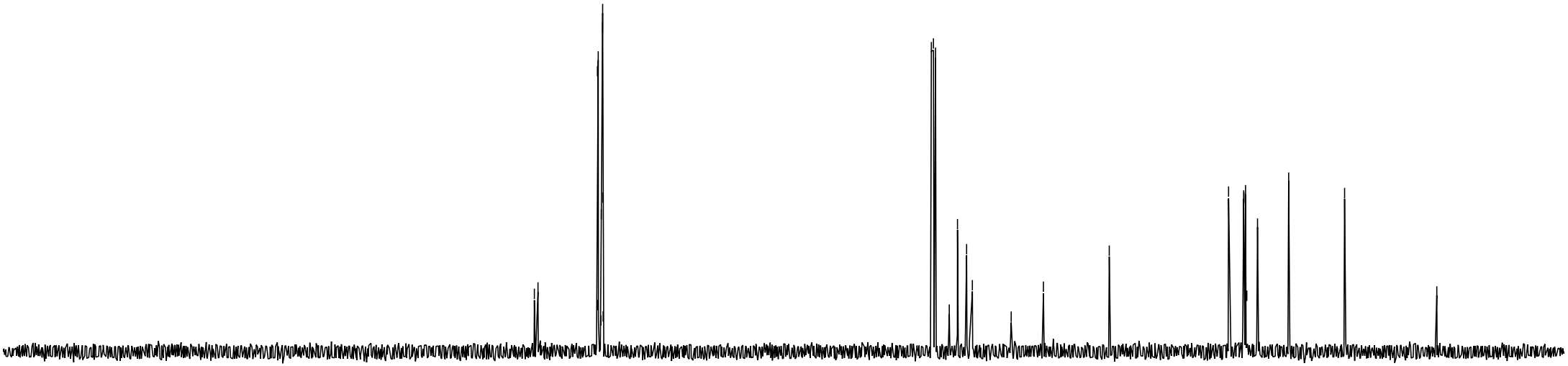
35b

Feb01-2008-ranga
r,r n-non f3

13C-NMR

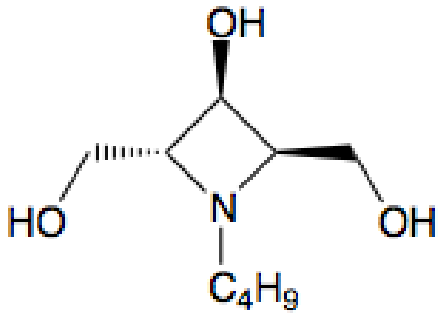


35b

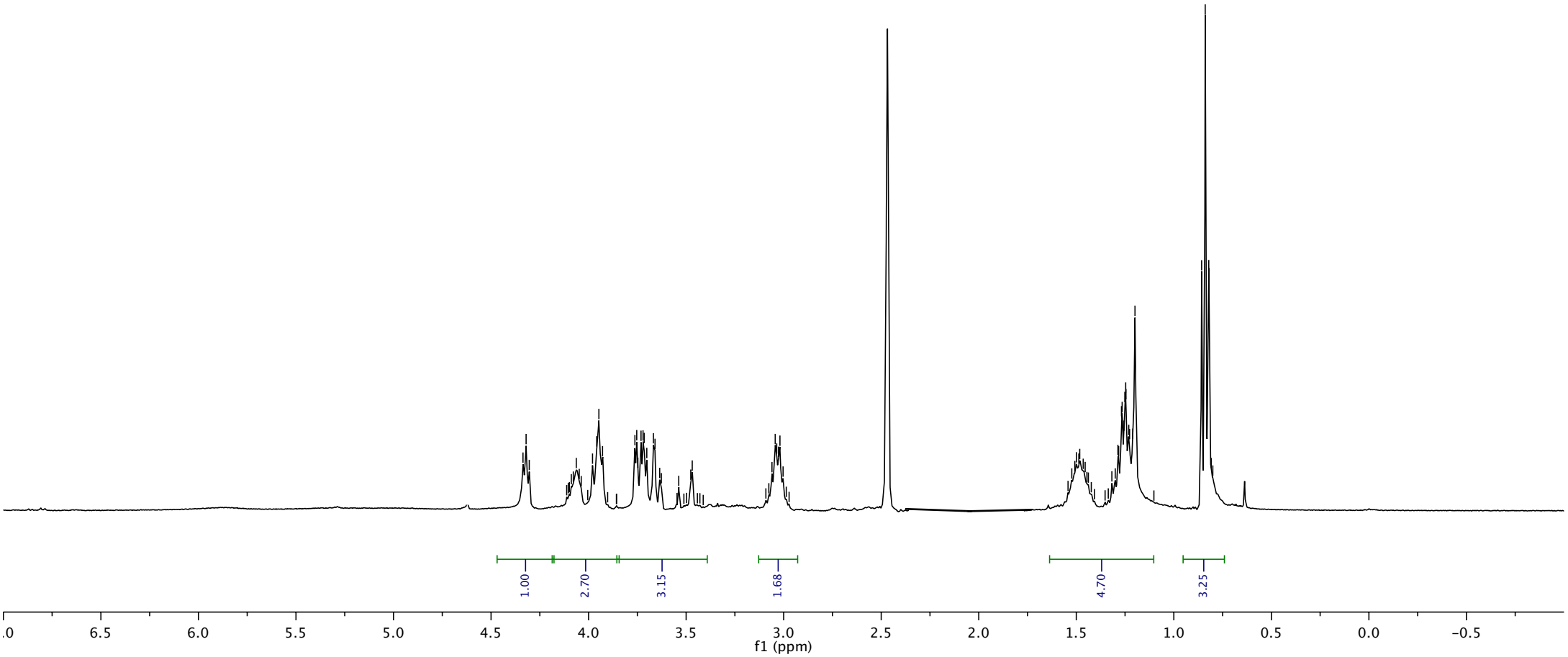


Apr07-2008-ranga
rrnbu

1H-NMR



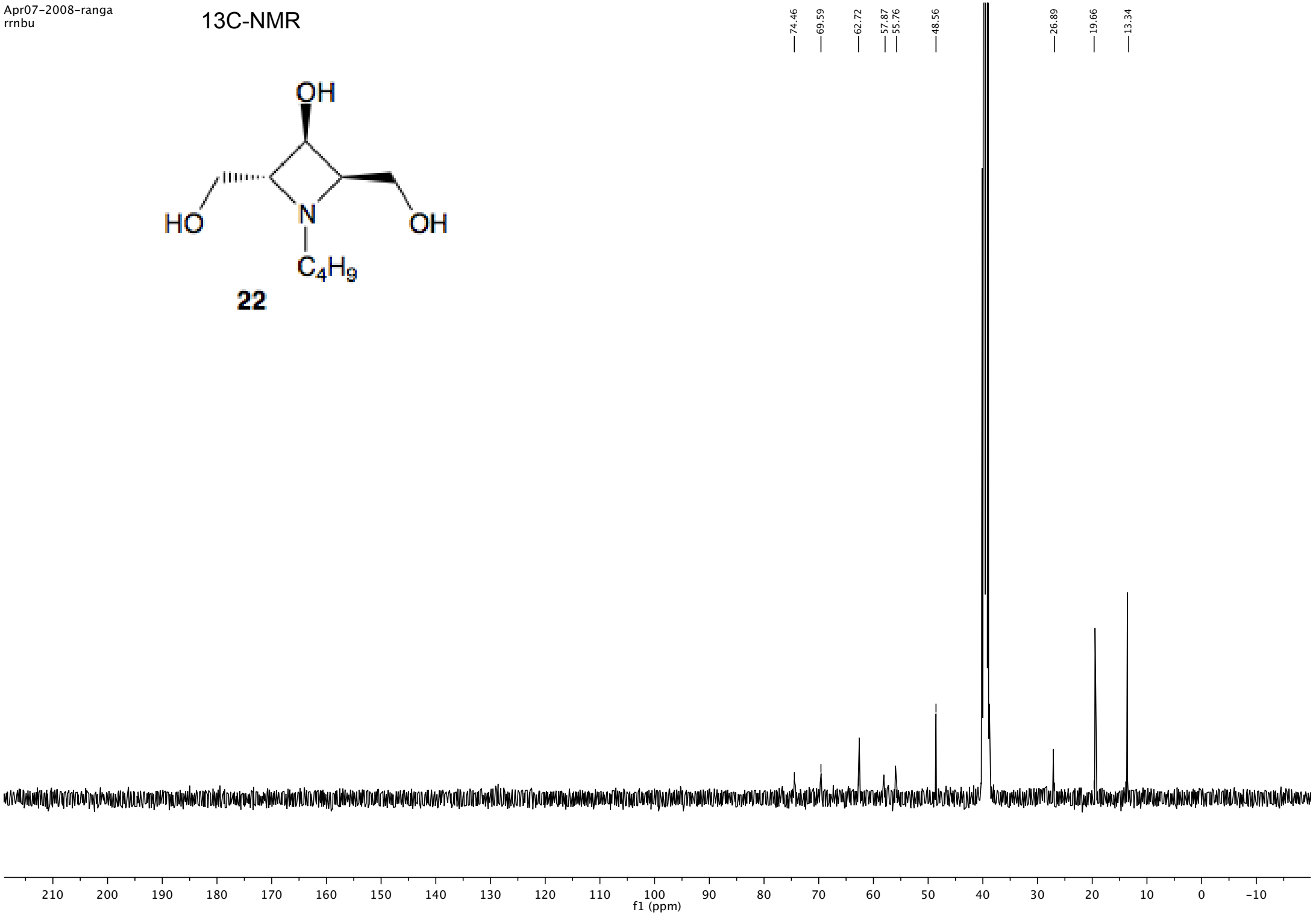
22



Apr07-2008-ranga
rrnbu

13C-NMR

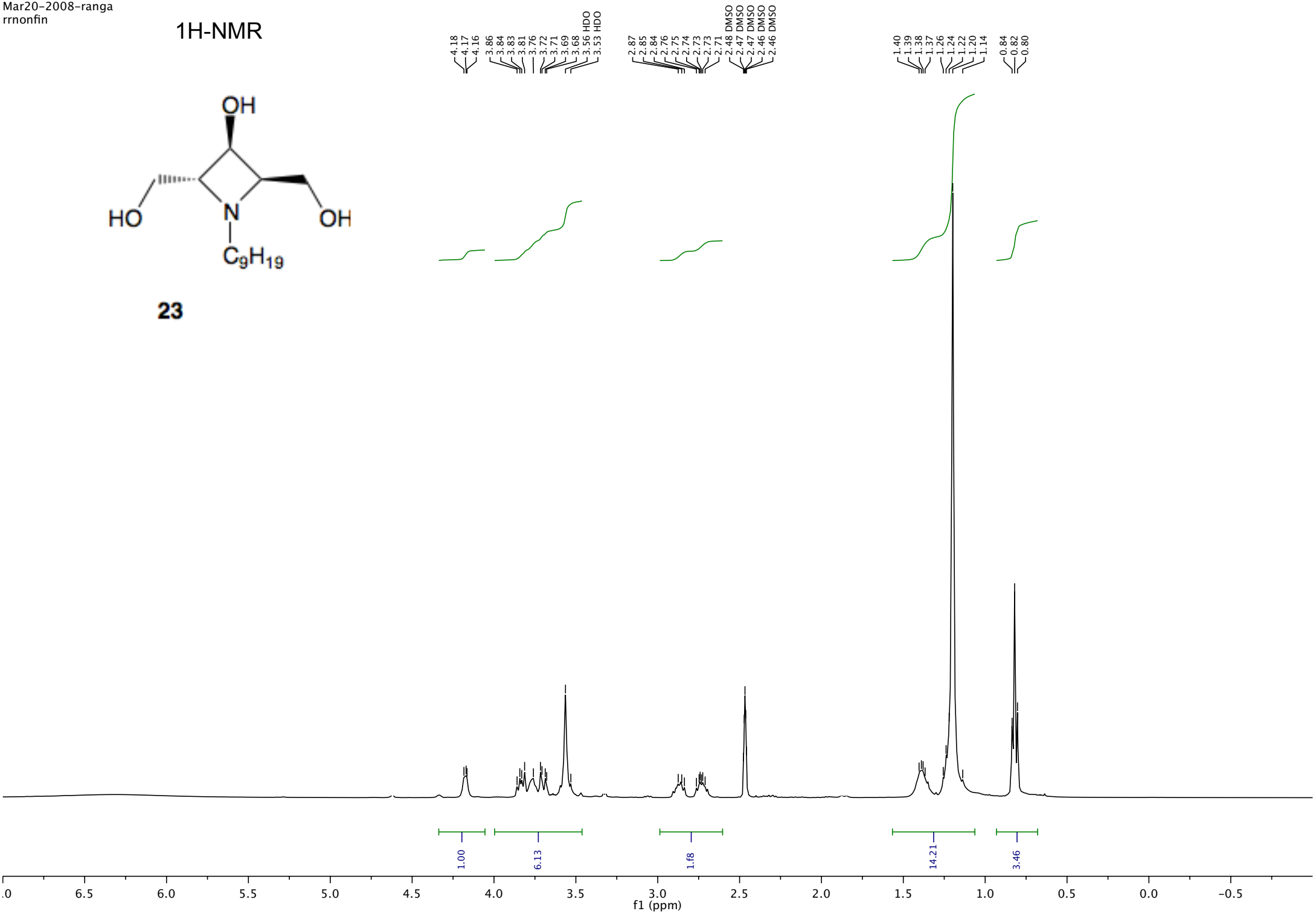
22



Mar20-2008-ranga
rrnonfin

1H-NMR

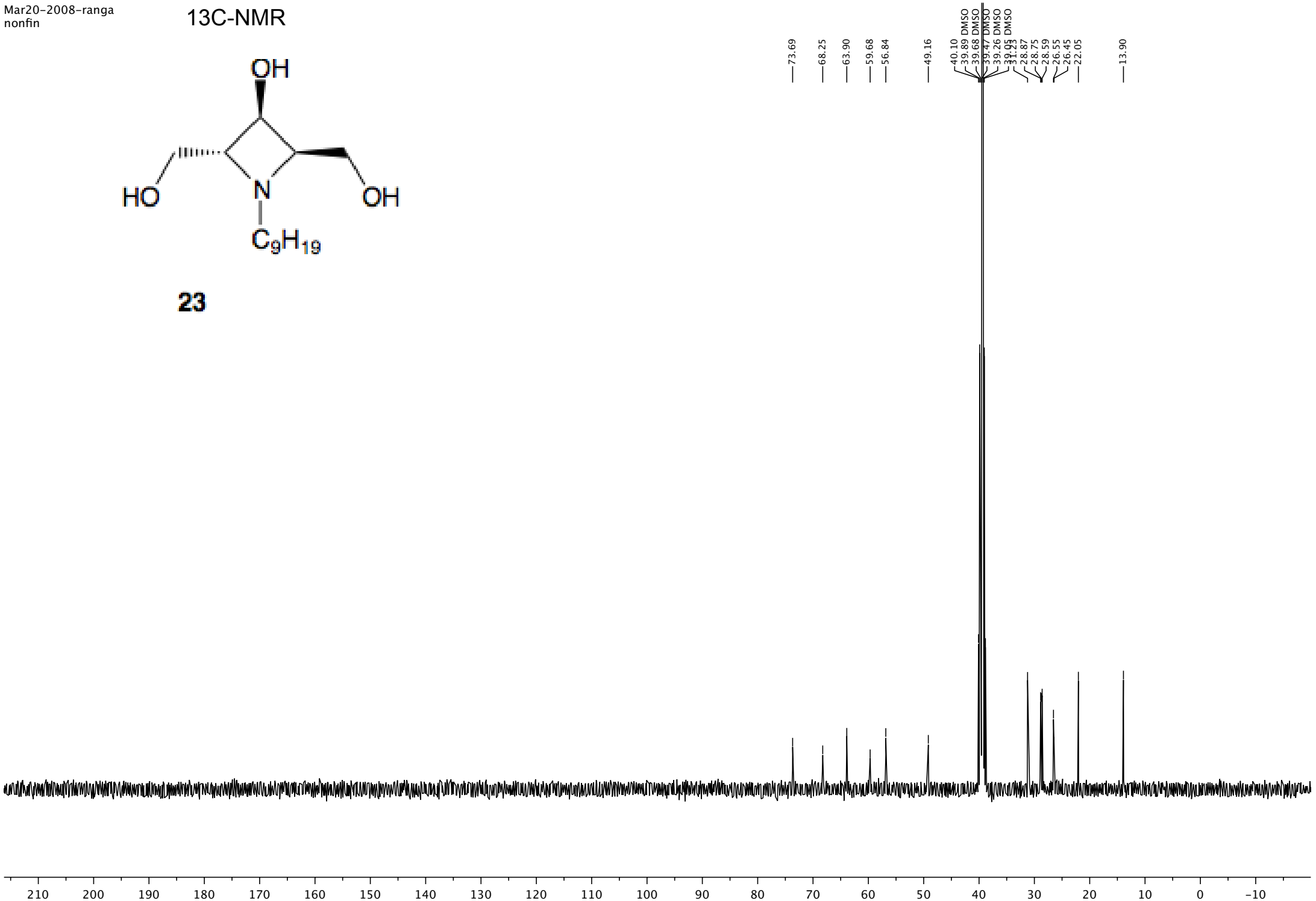
23



Mar20-2008-ranga
nonfin

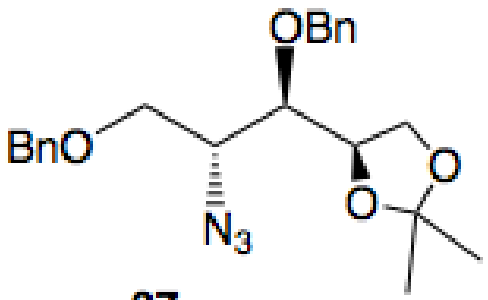
13C-NMR

23



Aug16-2007-ranga
meso-azide obn

1H-NMR

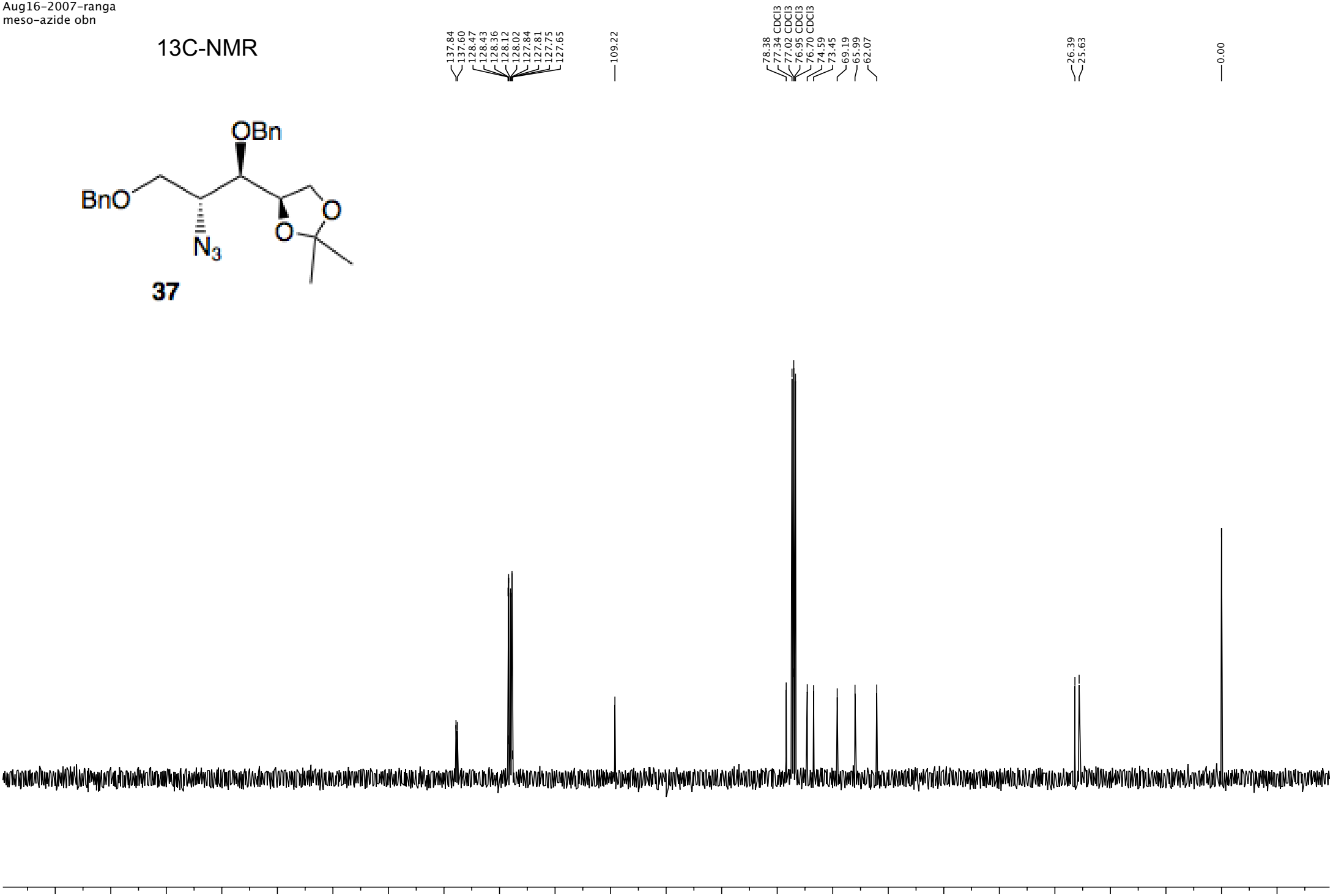


37

Aug16-2007-ranga
meso-azide obn

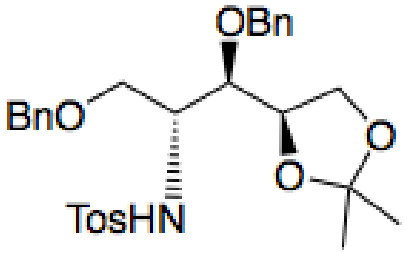
13C-NMR

37

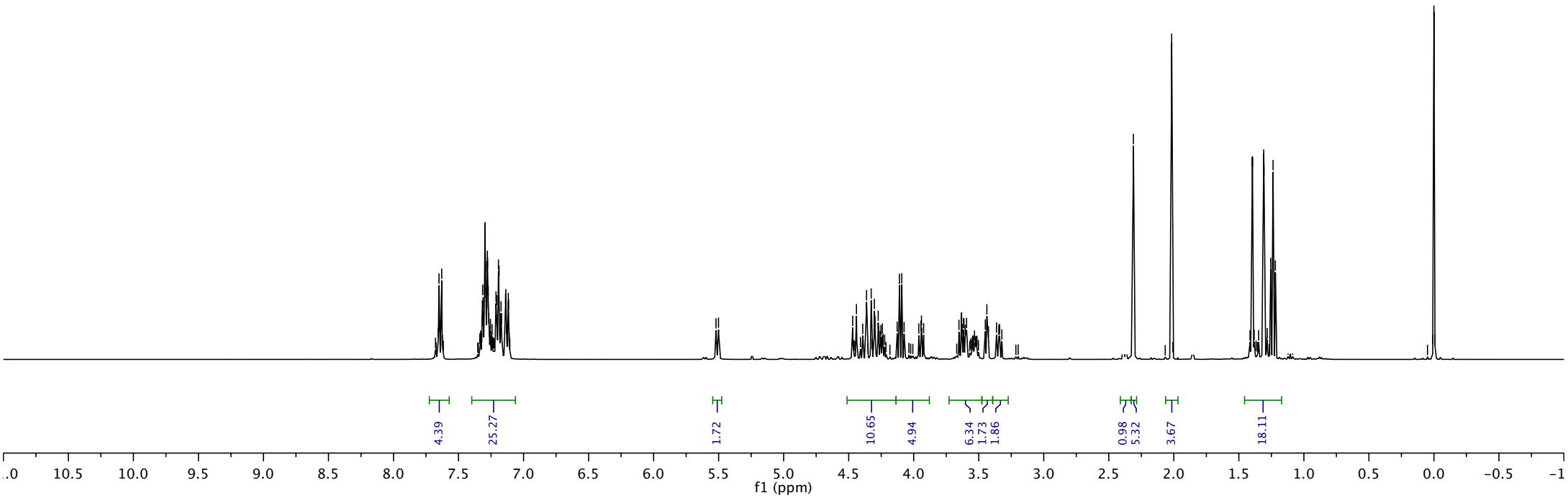


Aug29-2007
meso-tosyl

1H-NMR

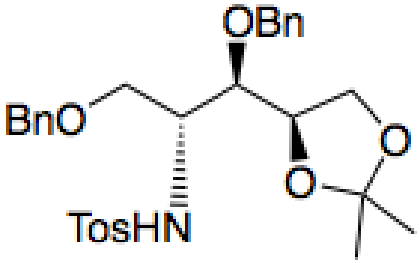


38



Aug29-2007
meso-tosyl

13C-NMR



38

143.19
137.93
137.55
137.47
129.49
128.40
128.36
128.34
128.31
127.84
127.78
127.72
127.68
127.65
127.06
109.43
109.00
99.95
77.45 CDCl3
77.13 CDCl3
77.07 CDCl3
76.82
76.46
75.10
73.23
73.01
68.80
67.57
66.29
60.33
54.59
53.91
26.59
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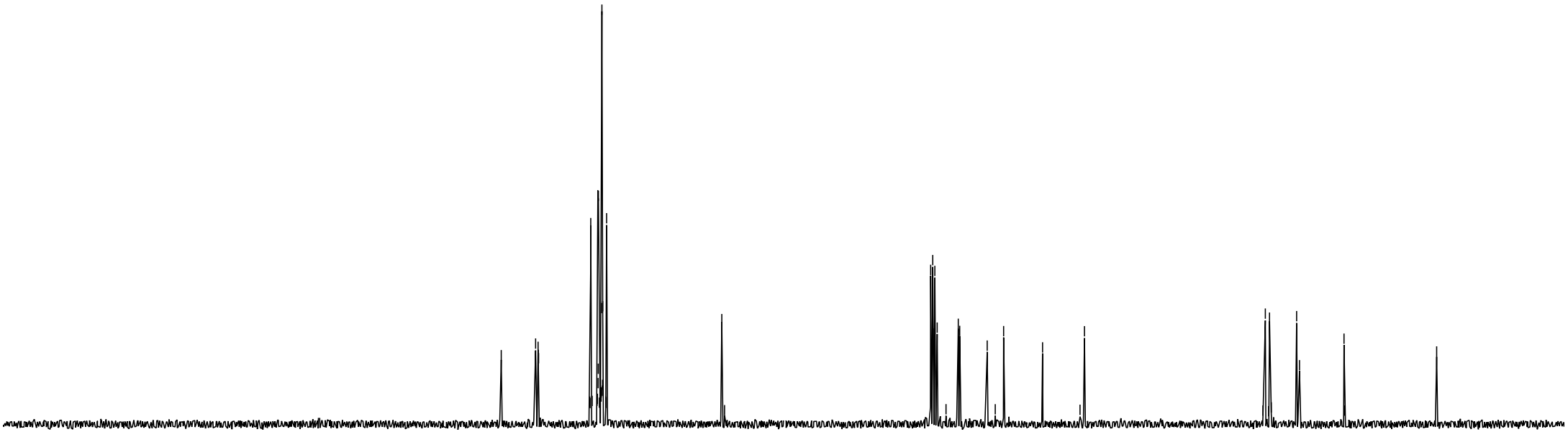
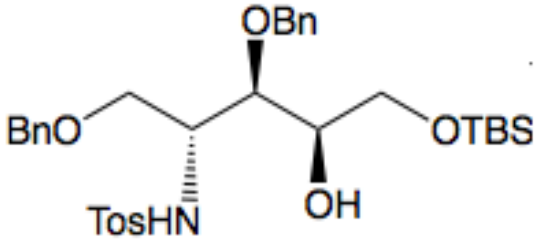
14.18
14.13
-0.00



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Sep04-2007-ranga
meso - fully protected

1H-NMR

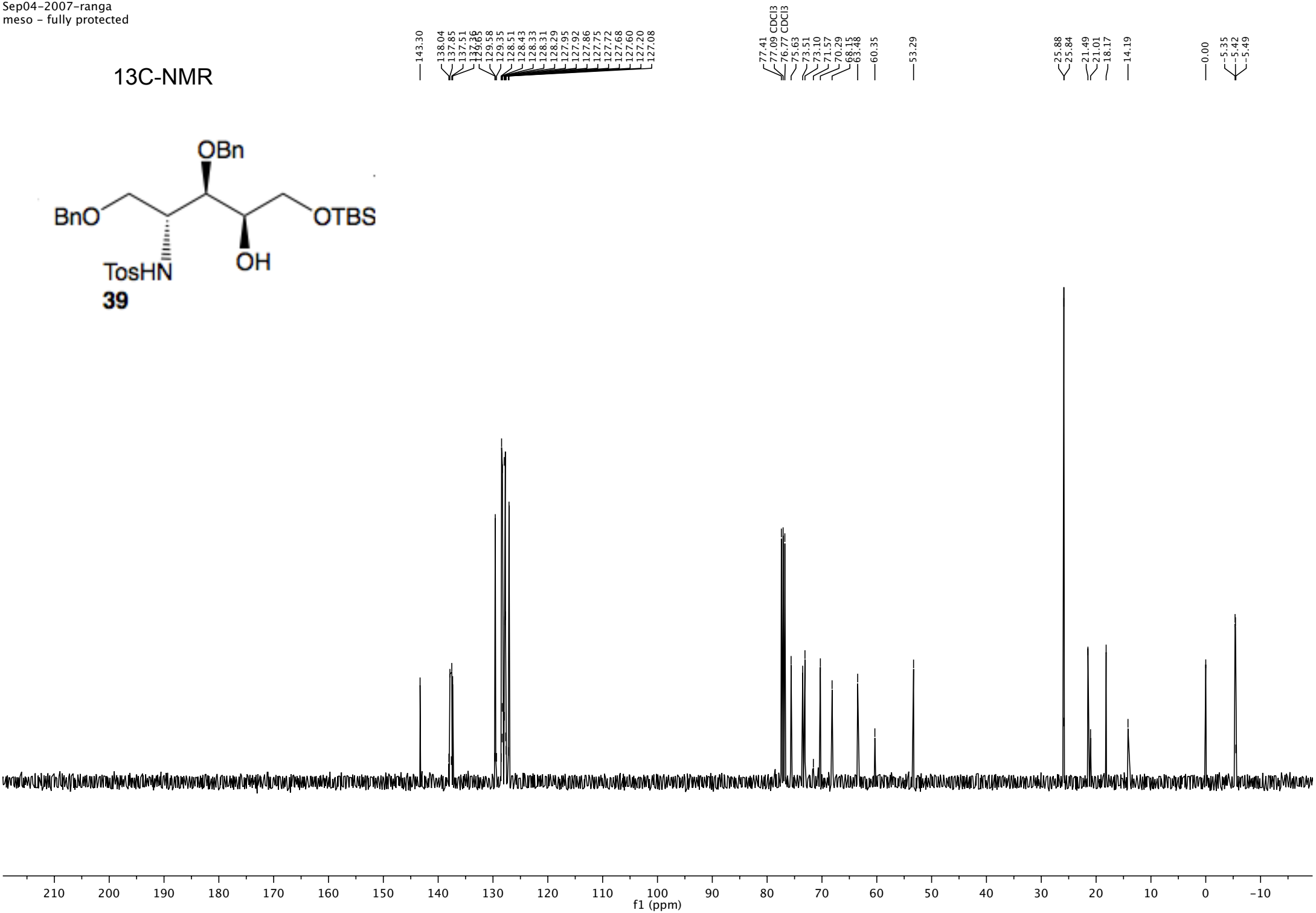


39

Sep04-2007-ranga
meso - fully protected

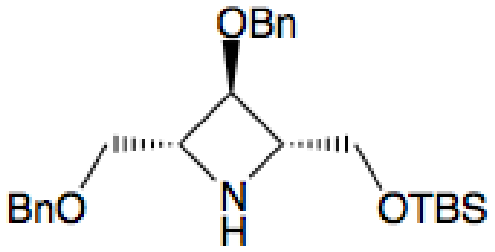
13C-NMR

39



Sep06-2007-ranga
ring aft N-ts removal

1H-NMR



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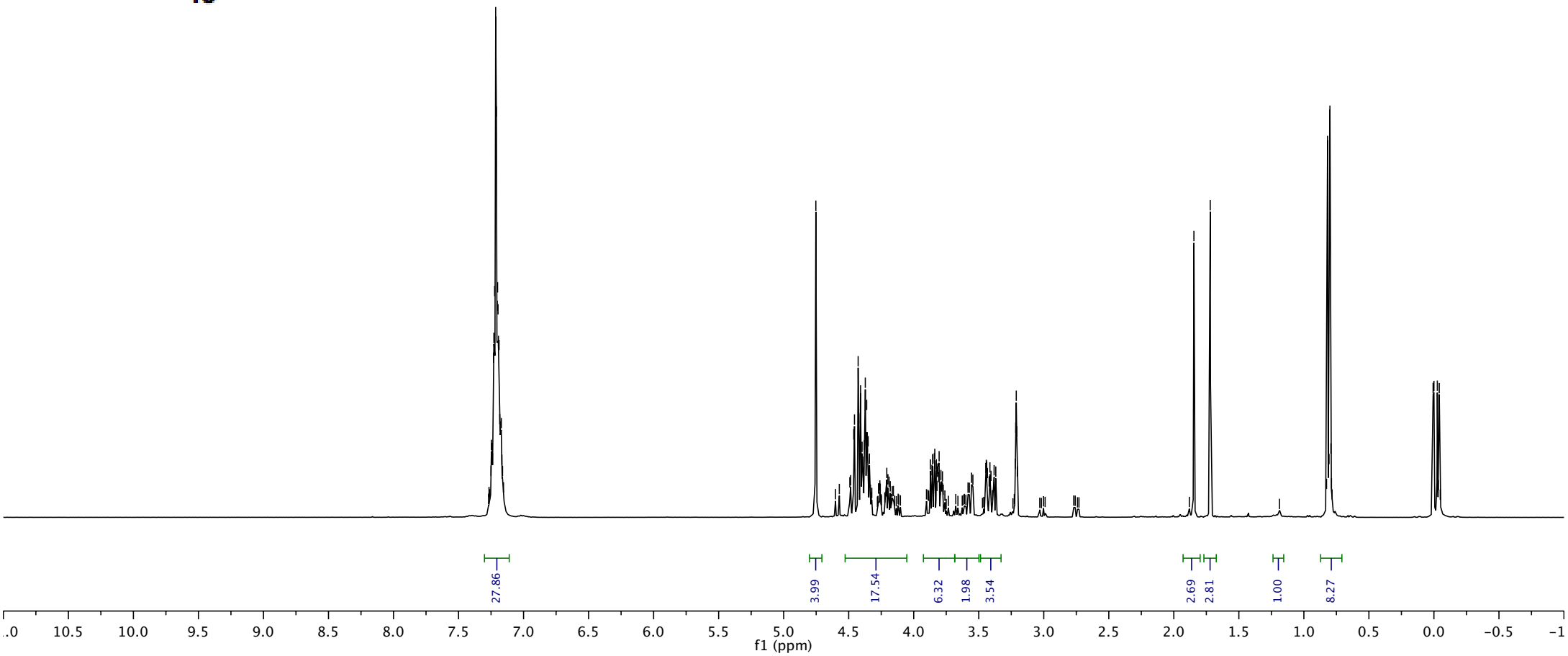
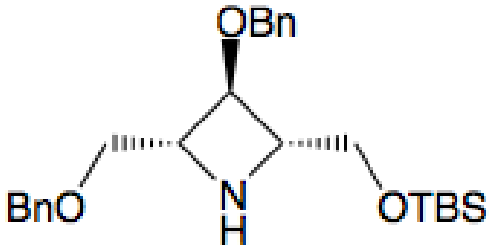


Sep11-2007-ranga
meso-NH

13C-NMR

138.26
138.16
137.78
128.54
128.42
128.35
128.34
128.31
127.82
127.71
127.68
127.65
127.60
127.53
77.40
77.08 CDCl3
76.98 CDCl3
76.76 CDCl3
76.43
73.34
73.22
72.45
72.30
71.32
71.10
66.17
64.93
63.93
62.12
61.80
60.71
25.92
25.87
20.82
18.31
-0.00
-5.36
-5.38



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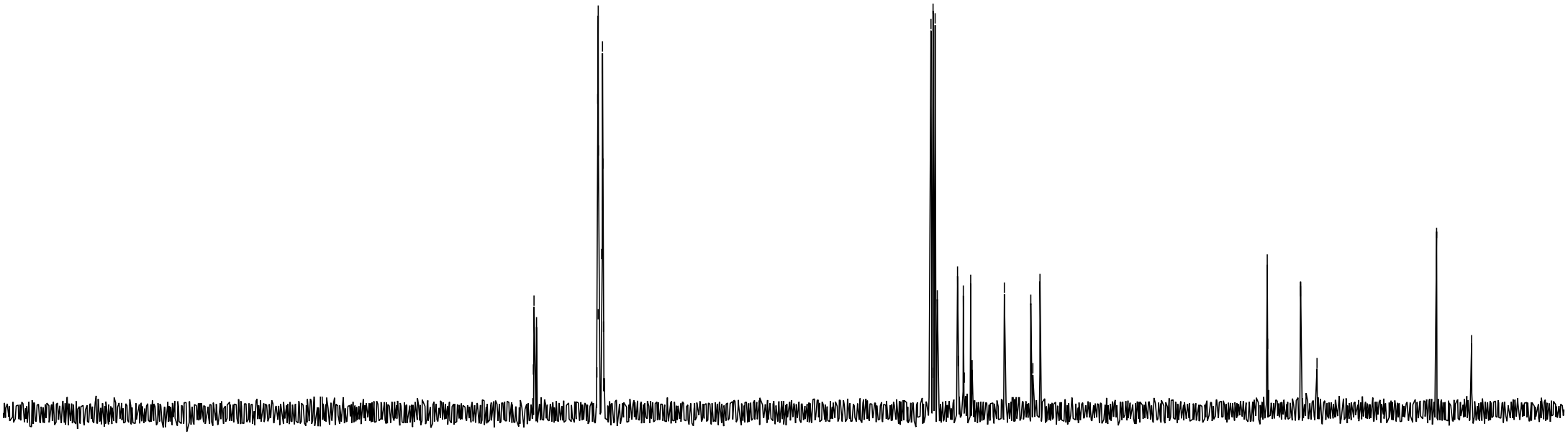
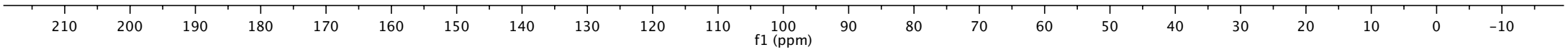
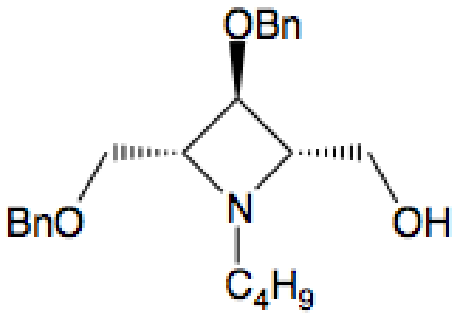


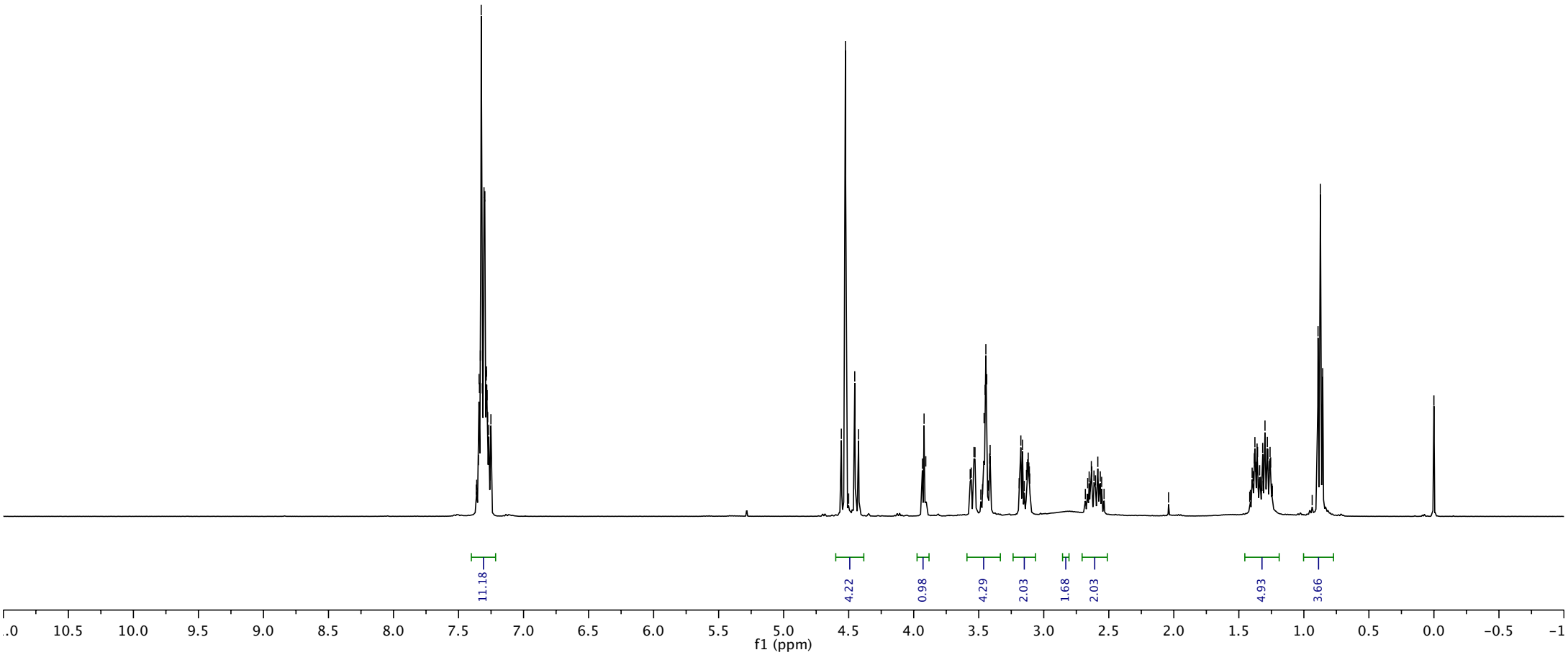


Sep04-2008
ms nbu tbaf

1H-NMR



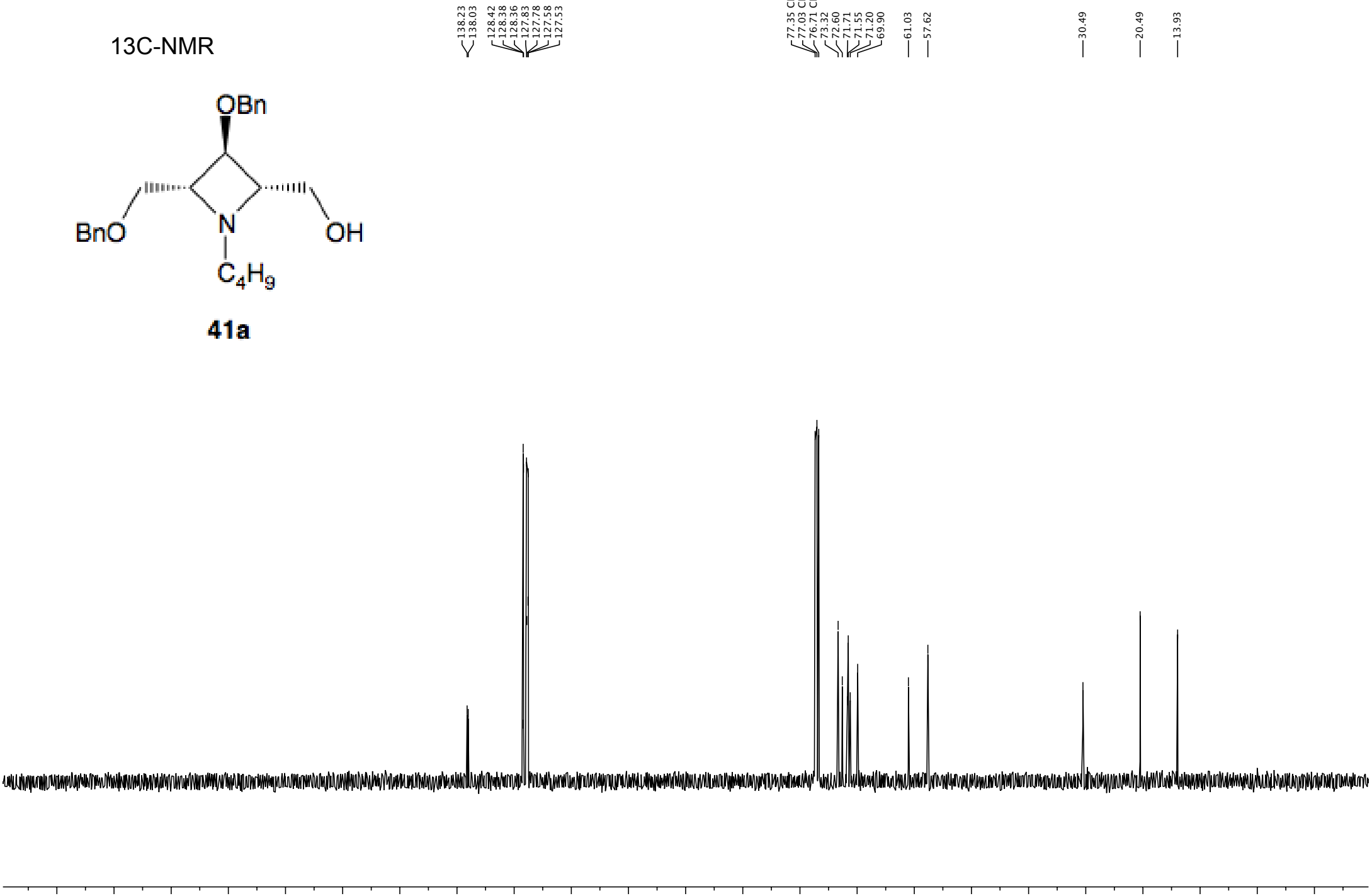
41a



Sep04-2008
ms nbu tbaf

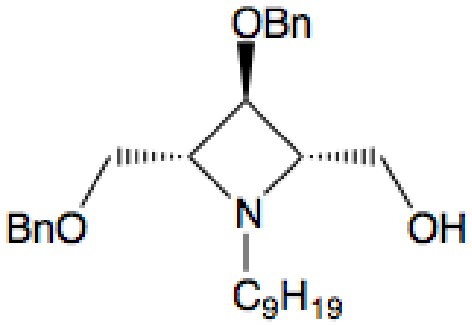
13C-NMR

41a



Aug22-2008-ranga
ms non frac

1H-NMR

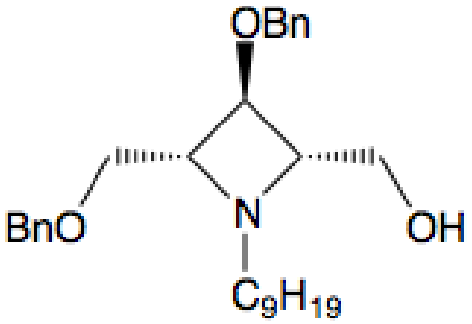


41b

Sep11-2008-ranga
ms non

13C-NMR

137.91
137.52
128.51
128.43
128.38
128.32
128.02
127.89
127.82
127.69
96.43
77.37 CDCl3
77.05 CDCl3
76.73 CDCl3
73.42
72.74
71.91
71.60
70.64
69.90
60.36
58.19
31.83
29.46
29.30
29.22
27.12
26.81
22.66
22.49
21.04
14.19
14.11



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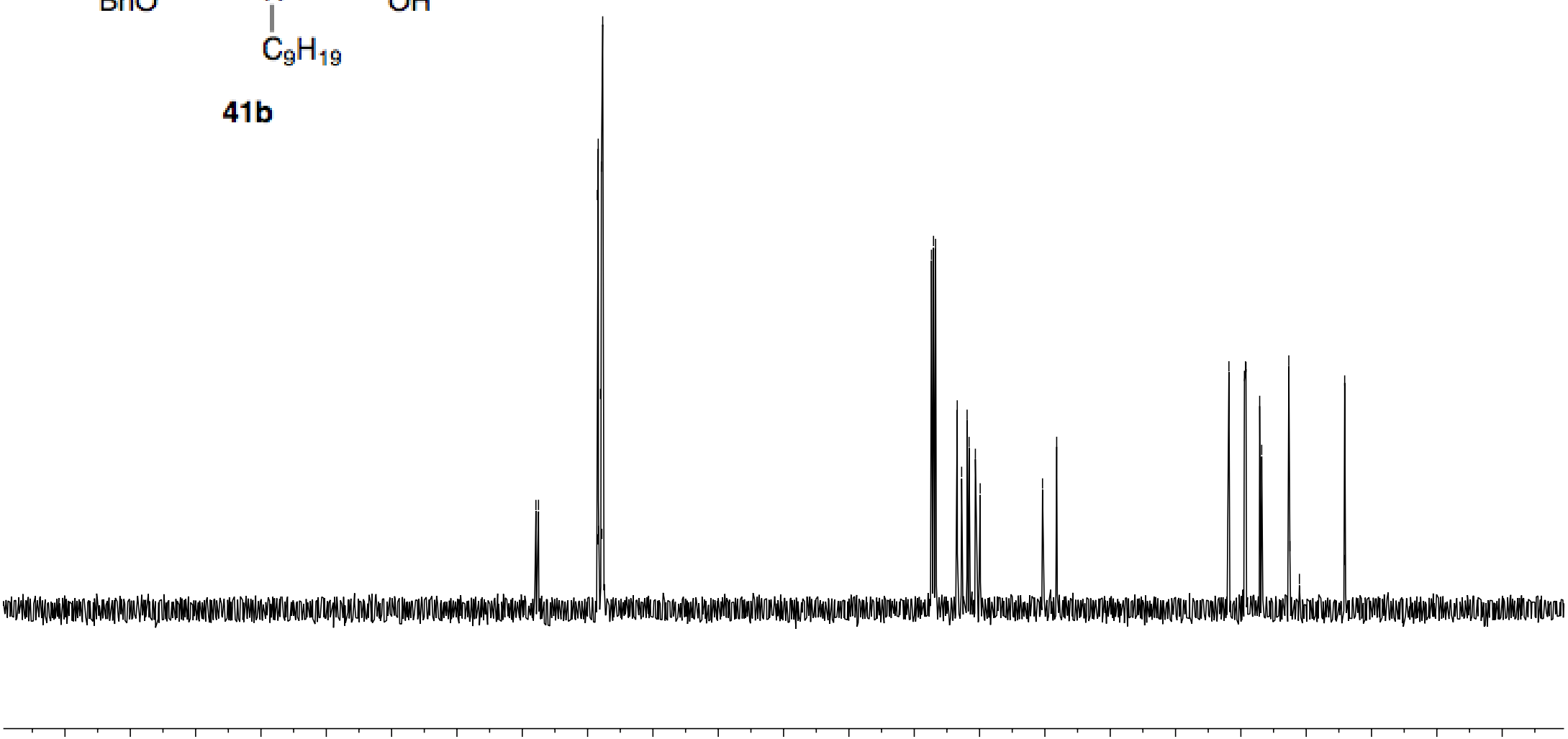
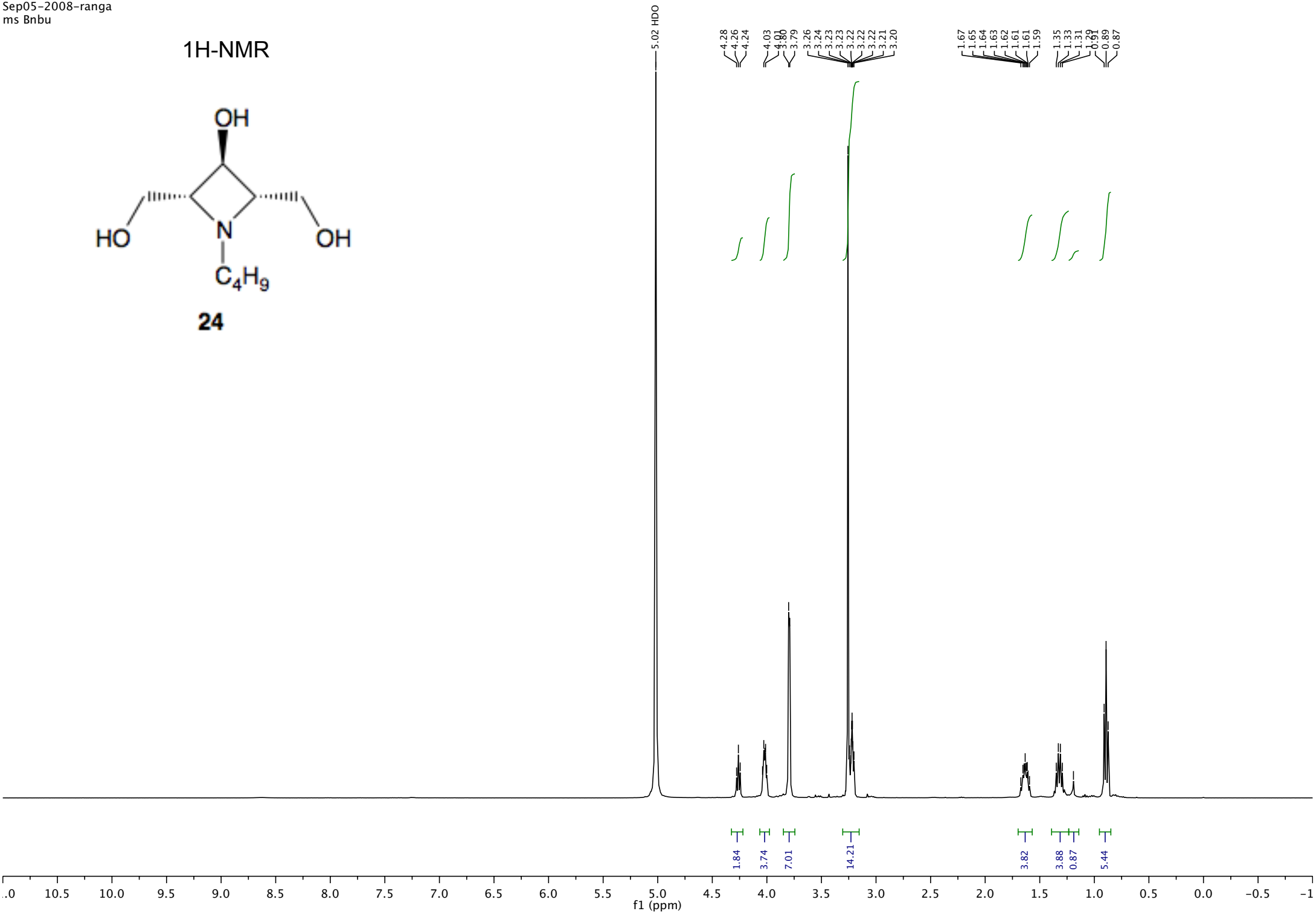


Sep05-2008-ranga
ms Bnbu

1H-NMR

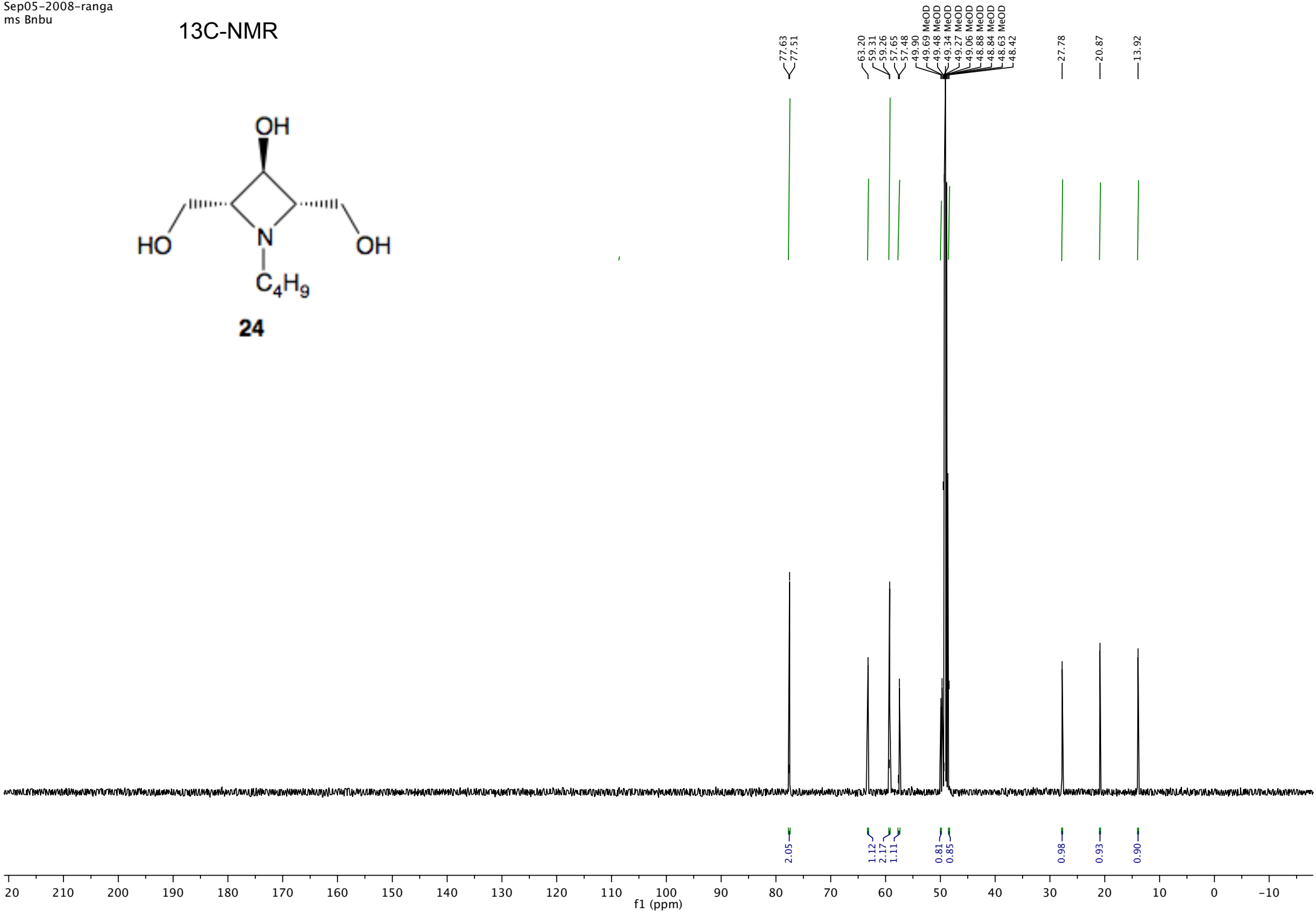
24



Sep05-2008-ranga
ms Bnbu

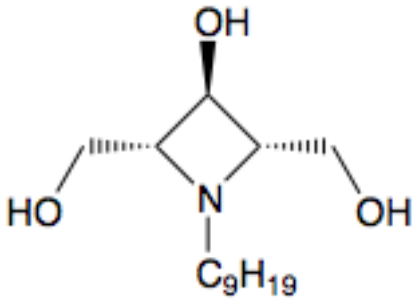
13C-NMR

24



Sep12-2008-ranga
ms non

1H-NMR

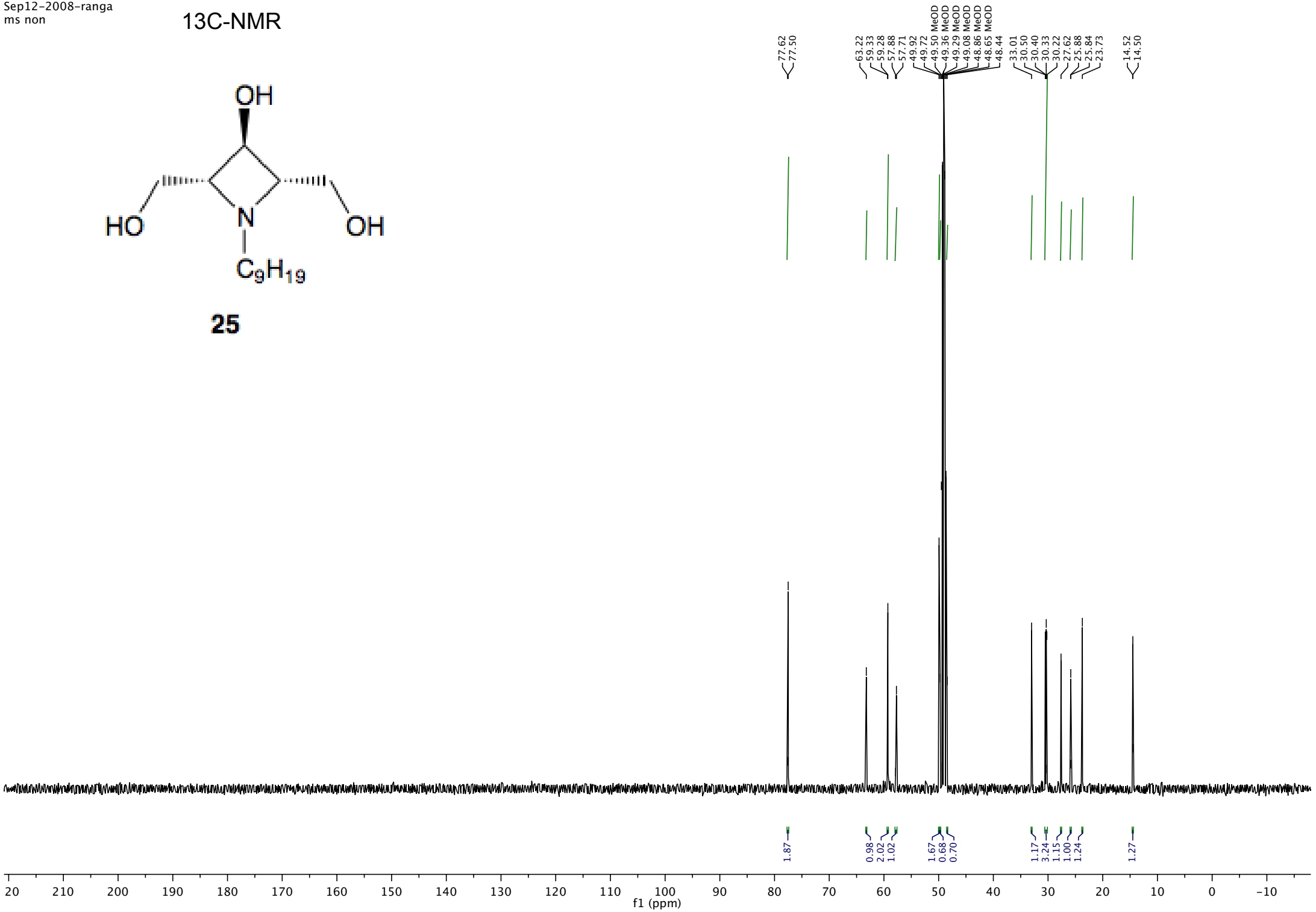


25

Sep12-2008-ranga
ms non

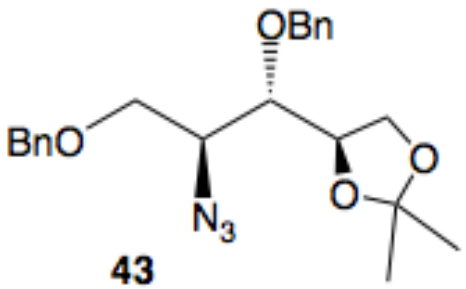
13C-NMR

25



Jun12-2008-ranga
SS azide obn

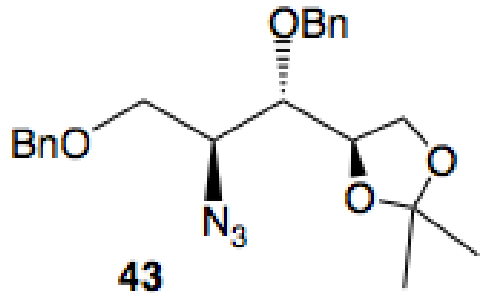
1H-NMR



43

Jun12-2008-ranga
SS azide obn

13C-NMR



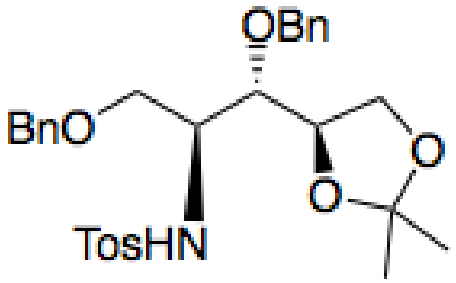
43

137.72
137.65
128.46
127.97
127.67
109.29
79.13
77.35 CDCl3
77.04 CDCl3
76.72 CDCl3
75.27
73.97
73.41
69.60
66.48
62.52
26.61
25.25
0.01

f1 (ppm)

Jun16-2008-ranga
ss N-ts

1H-NMR

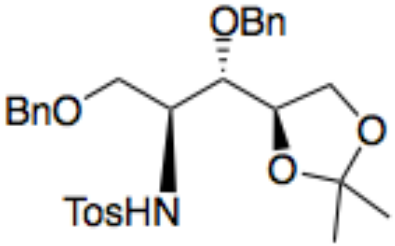


44

f1 (ppm)

Jun16-2008-ranga
ss N-ts

13C-NMR



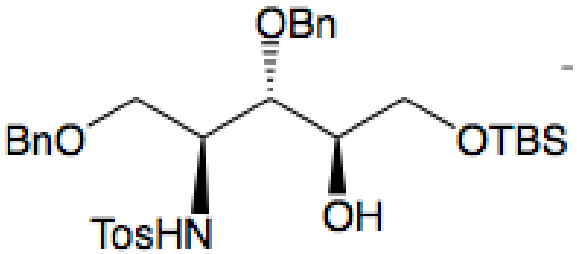
44

143.50
138.06
137.46
137.15
129.63
128.42
128.38
128.36
127.90
127.87
127.78
127.72
127.18
109.06
77.99
77.34 CDCl3
77.02 CDCl3
76.70 CDCl3
75.98
74.73
73.05
67.33
65.48
54.60
26.43
24.96
21.54
-0.00

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Jun25-2008-ranga
ss fully protected

1H-NMR



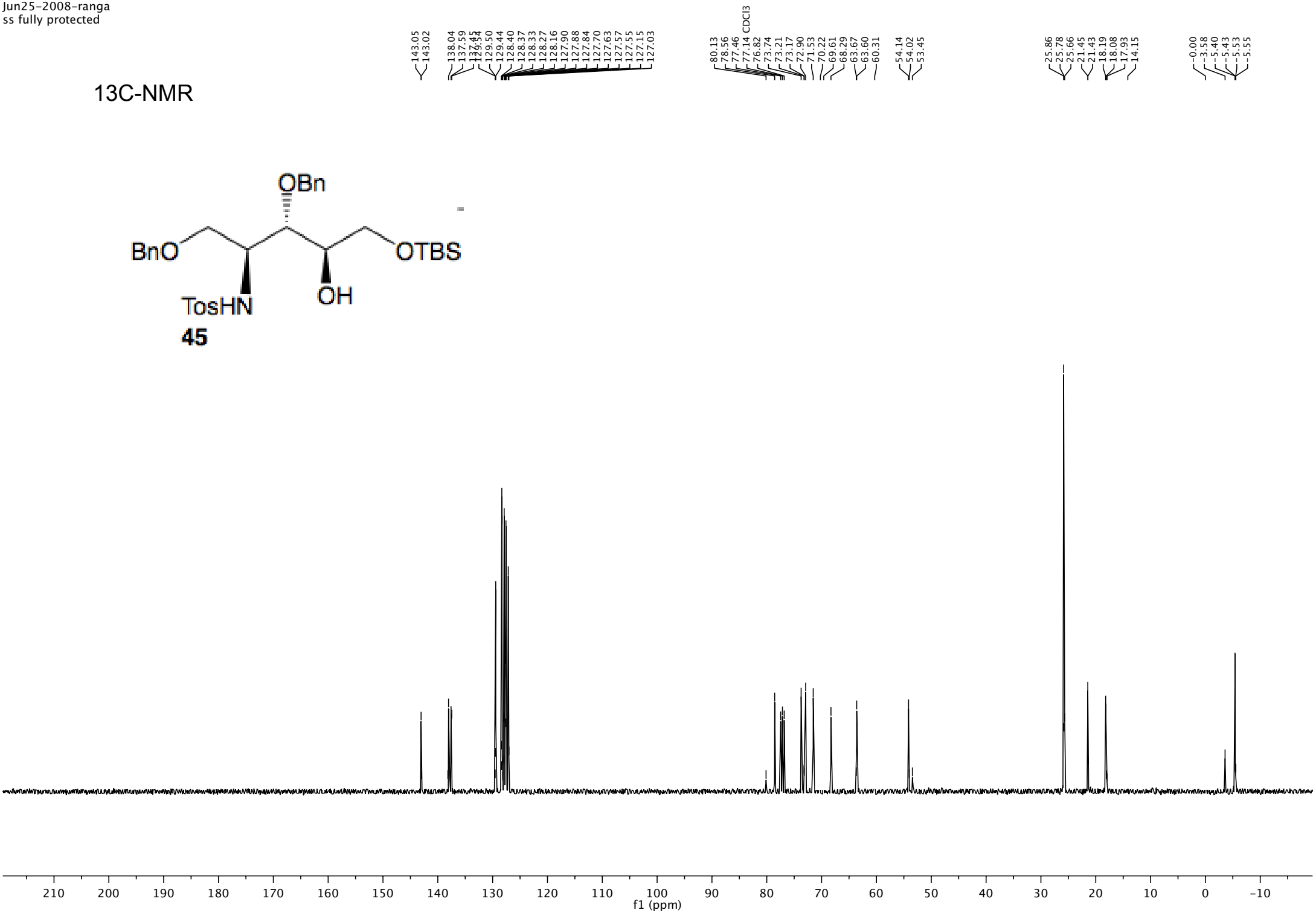
45

f1 (ppm)

Jun25-2008-ranga
ss fully protected

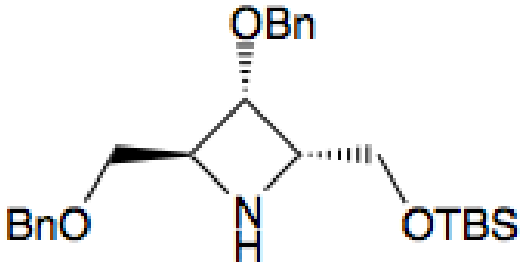
13C-NMR

45



Aug06-2008-ranga
ss ring nanaph f2

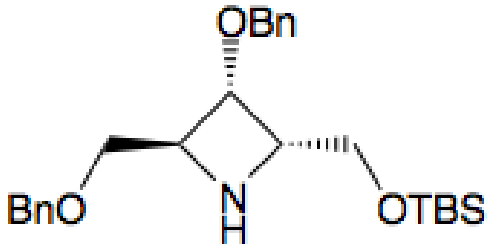
1H-NMR



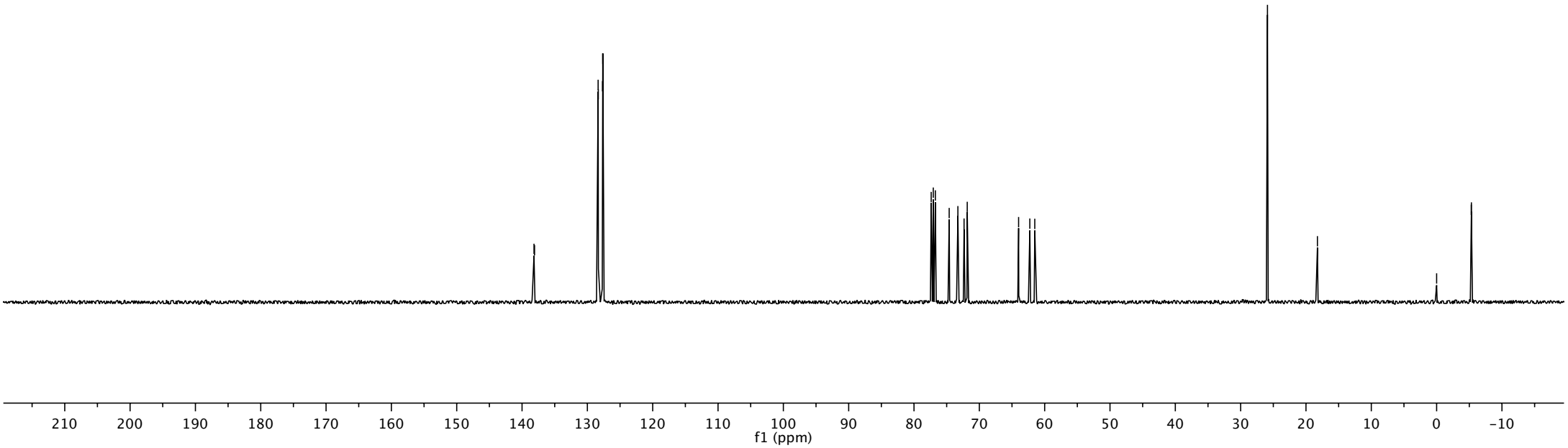
46

Aug06-2008-ranga
ss ring nanaph f2

13C-NMR

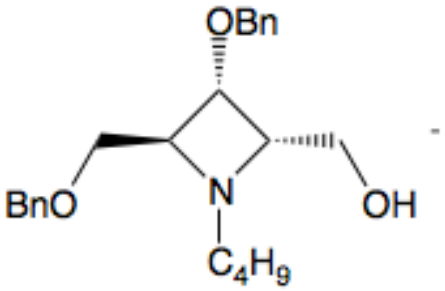


46



Sep04-2008-ranga
ssnbu tbaf

1H-NMR



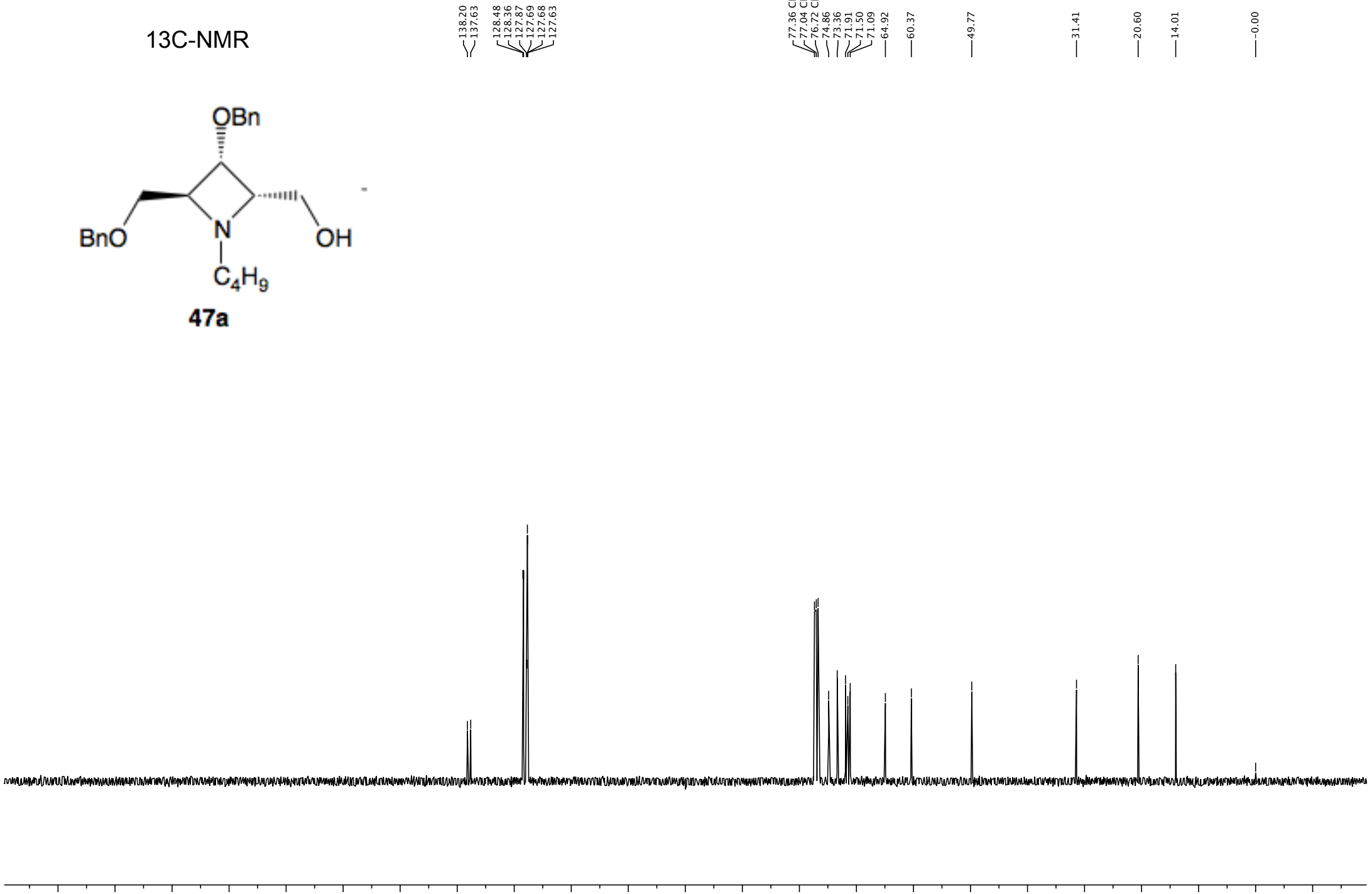
47a

f1 (ppm)

Sep04-2008-ranga
ssnbu tbaf

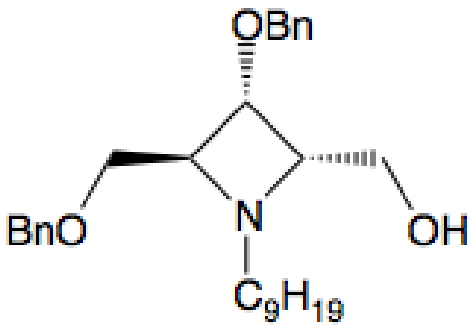
13C-NMR

47a



Sep02-2008-ranga
ss nonyl tbaf

1H-NMR

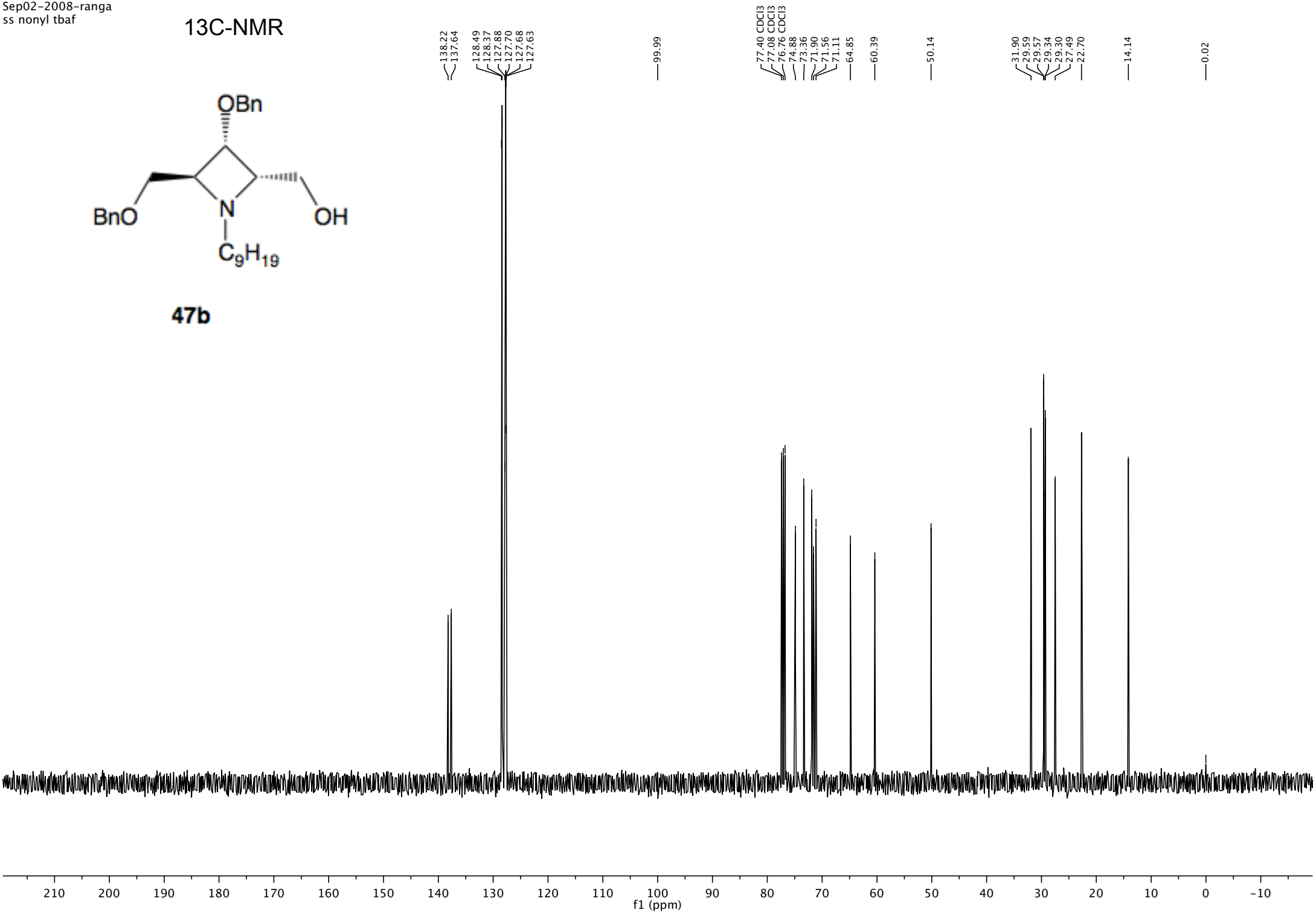


47b

Sep02-2008-ranga
ss nonyl tbaf

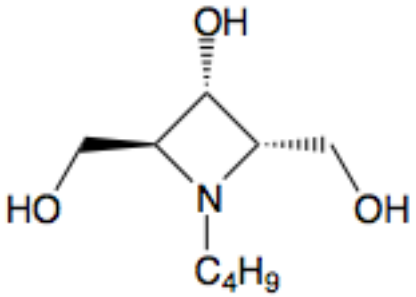
13C-NMR

47b

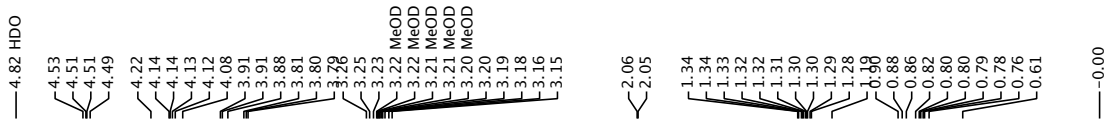


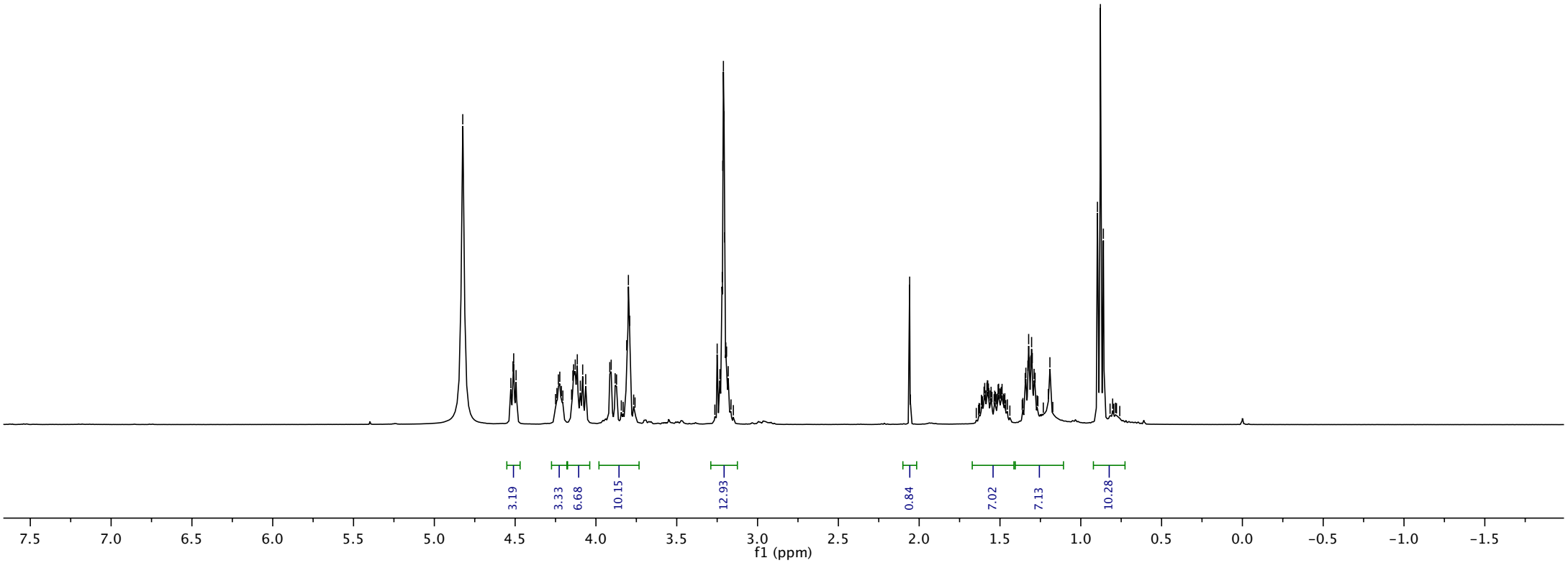
Feb22-2008-ranga
nbuaftpurif

1H-NMR



26

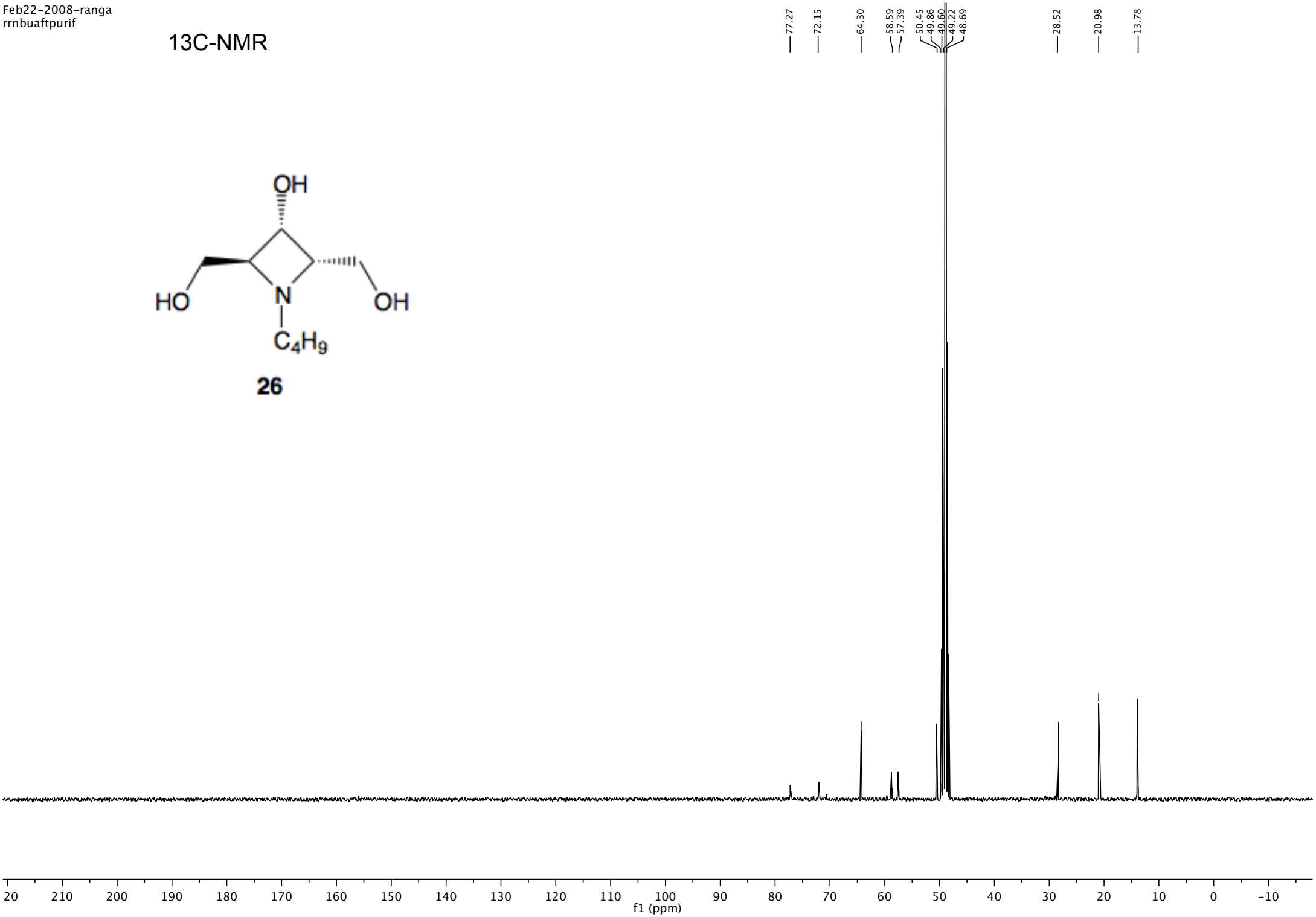




Feb22-2008-ranga
rrnbuaftpurif

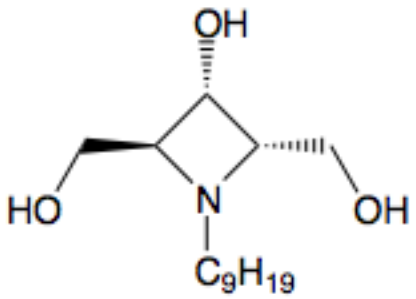
13C-NMR

26



Mar14-2008-ranga
non aftpur

1H-NMR



27

Mar14-2008-ranga
r,r non aftpur

13C-NMR

27