Nucleophilic addition to silyl-protected five-membered ring oxocarbenium ions governed by stereoelectronic effects

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Supporting Information

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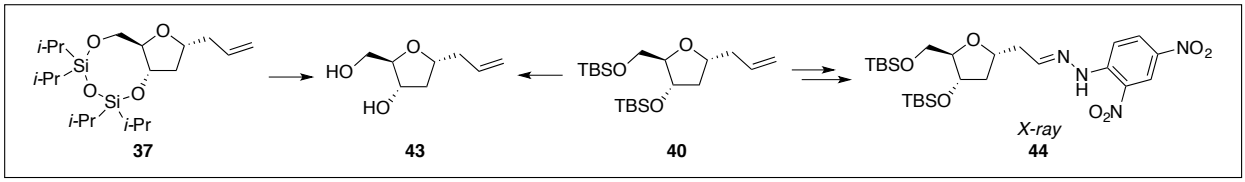
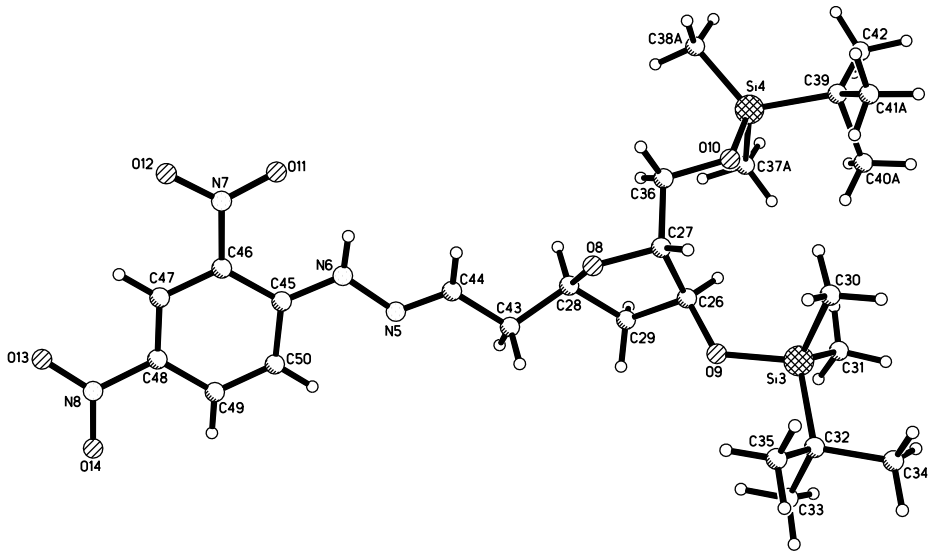
I. General Procedures.

Liquid chromatography was performed using forced flow (flash chromatography) of the indicated solvent system on silica gel (SiO_2) 60 (230-400 mesh). ^{1}H NMR and ^{13}C NMR spectra were recorded at ambient temperature using 400 (400 and 100 MHz, respectively), 500 (500 and 125 MHz, respectively) or 600 (600 and 150 MHz, respectively) spectrometers, as indicated. The data are reported as follows: chemical shift in ppm from internal tetramethylsilane on the δ scale, multiplicity (app = apparent, br = broad, s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sext = sextet, m = multiplet), coupling constants (Hz), and integration. Due to difficulties with purification of certain products, only distinctive peaks are listed in tabulated ^{1}H NMR spectral data as indicated, and the structures were assigned using a combination of COSY, HMQC, and nOe experiments. Proton count at each carbon was confirmed by HSQC. Gas chromatography–mass spectrometry (GC–MS) was performed with a quadrupole system with a fused silica capillary column (30 m x 0.32 mm x 0.25 μm) wall-coated with DB-5 using electron ionization (70 eV). High resolution mass spectra (HRMS) were acquired on a quadrupole time-of-flight spectrometer or an orthogonal acceleration time-of-flight spectrometer and were obtained by peak matching. Optical rotations were obtained using a digital polarimeter and concentrations were reported in g/100 mL. Specific rotations were assumed to be constant over a small range of concentrations. Analytical thin layer chromatography was performed on silica gel 60 F_{254} plates. THF, DMF, and CH_2Cl_2 were dried by filtration through alumina according to the method of Grubbs.[1] All reactions using Et_2O, THF, DMF, and CH_2Cl_2 as solvents were run under an atmosphere of nitrogen in glassware that was flame-dried under a stream of nitrogen.

II. X-ray Crystallographic Data

Chemical Correlation:





X-ray Data Collection, Structure Solution and Refinement for **44**.

A yellow crystal of approximate dimensions 0.060 x 0.259 x 0.315 mm was mounted on a glass fiber and transferred to a Bruker SMART APEX II diffractometer. The APEX2[1] program package was used to determine the unit-cell parameters and for data collection (45 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT[2] and SADABS[3] to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL[4] program. There were no systematic absences. The noncentrosymmetric triclinic space group *P*1 was assigned and later determined to be correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques[5]. The analytical scattering factors[6] for neutral atoms were used throughout the analysis. Hydrogen atoms were included using a riding model. There were two molecules of the formula-unit present. Carbon atoms C(37), C(38), C(40) and C(41) were disordered and included using multiple components with partial site-occupancy-factors.

At convergence, wR2 = 0.1251 and Goof = 1.018 for 747 variables (3 restraints) refined against 14227 data (0.74Å), R1 = 0.0464 for those 13387 data with $I > 2.0\sigma(I)$. The absolute structure was assigned by refinement of the Flack parameter[6]

References.

1. APEX2 Version 2010.3-0, Bruker AXS, Inc.; Madison, WI 2010.

2. SAINT Version 7.68a, Bruker AXS, Inc.; Madison, WI 2009.

3. Sheldrick, G. M. SADABS, Version 2008/1, Bruker AXS, Inc.; Madison, WI 2008.

4. Sheldrick, G. M. SHELXTL, Version 2008/4, Bruker AXS, Inc.; Madison, WI 2008.

5. Sheldrick, G. M. SHELXL-2012/9

6. International Tables for X-Ray Crystallography 1992, Vol. C., Dordrecht: Kluwer Academic Publishers

7. Flack, H. D. Acta. Cryst., A39, 876-881, 1983.

Definitions:

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

$R1 = \Sigma||F_o|-|F_c|| / \Sigma|F_o|$

$Goof = S = [\Sigma[w(F_o^2-F_c^2)^2] / (n-p)]^{1/2}$ where n is the number of reflections and p is the total number of parameters refined.

The thermal ellipsoid plot is shown at the 50% probability level.

Table 1. Crystal data and structure refinement for **44**.

Identification code	kaw159 (Vi Tran)	
Empirical formula	$C_{25}H_{44}N_4O_7Si_2$	
Formula weight	568.82	
Temperature	93(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*1	
Unit cell dimensions	a = 7.0843(5) Å	α= 88.0590(8)°.
	b = 8.9087(6) Å	β= 89.2760(9)°.
	c = 24.7678(18) Å	γ = 88.0016(9)°.
Volume	1561.19(19) $Å^3$	
Z	2	
Density (calculated)	1.210 Mg/m^3	
Absorption coefficient	0.159 mm^{-1}	

F(000)	612
Crystal color	yellow
Crystal size	0.315 x 0.259 x 0.060 mm^3
Theta range for data collection	2.289 to 28.923°
Index ranges	$-9 \leq h \leq 9$, $-12 \leq k \leq 11$, $-33 \leq l \leq 32$
Reflections collected	18757
Independent reflections	14227 [R(int) = 0.0103]
Completeness to theta = 25.500°	99.7 %
Absorption correction	Numerical
Max. and min. transmission	1.0000 and 0.9245
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	14227 / 3 / 747
Goodness-of-fit on F^2	1.018
Final R indices [I>2sigma(I) = 13387 data]	R1 = 0.0464, wR2 = 0.1216
R indices (all data, 0.74Å)	R1 = 0.0498, wR2 = 0.1251
Absolute structure parameter	0.00(3)
Largest diff. peak and hole	0.681 and -0.416 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **44**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Si(1)	1738(1)	3241(1)	8570(1)	30(1)
Si(2)	-1915(1)	-1329(1)	10220(1)	31(1)
O(1)	261(3)	2769(3)	10564(1)	32(1)
O(2)	925(3)	3708(3)	9173(1)	33(1)
O(3)	-920(3)	209(3)	9983(1)	34(1)
O(4)	-152(3)	803(3)	13005(1)	32(1)
O(5)	124(4)	-951(4)	13617(1)	53(1)
O(6)	5753(4)	-3875(3)	13875(1)	42(1)
O(7)	7969(4)	-3839(3)	13269(1)	42(1)
N(1)	3380(3)	1830(3)	11828(1)	23(1)
N(2)	2509(3)	1202(3)	12282(1)	23(1)
N(3)	762(4)	-231(3)	13236(1)	30(1)
N(4)	6452(4)	-3361(3)	13459(1)	31(1)
C(1)	1264(4)	2774(3)	9645(1)	27(1)
C(2)	-451(4)	2862(3)	10024(1)	26(1)
C(3)	2289(4)	2629(3)	10542(1)	25(1)
C(4)	2829(4)	3309(3)	9994(1)	27(1)
C(5)	621(7)	1489(5)	8369(2)	50(1)
C(6)	4344(6)	2914(5)	8592(2)	45(1)
C(7)	1073(6)	4894(5)	8116(2)	43(1)
C(8)	1965(8)	6304(6)	8308(2)	64(1)
C(9)	-1097(7)	5124(6)	8116(2)	60(1)
C(10)	1741(9)	4574(8)	7537(2)	71(2)

C(11)	-1859(4)	1651(4)	9946(2)	31(1)
C(12)	25(6)	-2776(5)	10269(2)	48(1)
C(13)	-2949(9)	-997(7)	10898(2)	72(2)
C(14)	-3745(5)	-1859(5)	9708(2)	52(1)
C(15)	-2881(9)	-1700(11)	9130(3)	91(2)
C(16)	-4405(9)	-3442(6)	9838(5)	136(5)
C(17)	-5484(6)	-834(5)	9728(2)	48(1)
C(18)	3058(5)	3404(3)	11027(1)	27(1)
C(19)	2319(4)	2706(3)	11540(1)	26(1)
C(20)	3444(4)	133(3)	12577(1)	21(1)
C(21)	2655(4)	-611(3)	13039(1)	24(1)
C(22)	3649(5)	-1743(3)	13328(1)	26(1)
C(23)	5444(5)	-2145(3)	13165(1)	26(1)
C(24)	6296(4)	-1447(4)	12714(1)	27(1)
C(25)	5321(4)	-336(3)	12427(1)	24(1)
Si(3)	-3099(2)	-1549(1)	16550(1)	38(1)
Si(4)	287(1)	-5358(1)	15059(1)	35(1)
O(8)	-2304(4)	-51(2)	14793(1)	34(1)
O(9)	-3798(4)	-922(3)	15944(1)	40(1)
O(10)	32(5)	-3602(3)	15222(1)	45(1)
O(11)	-1575(3)	3290(3)	12386(1)	33(1)
O(12)	-1826(4)	4954(3)	11735(1)	41(1)
O(13)	-7408(4)	7908(3)	11435(1)	46(1)
O(14)	-9647(3)	7922(3)	12035(1)	37(1)
N(5)	-5257(4)	2295(3)	13532(1)	24(1)
N(6)	-4305(3)	2920(3)	13091(1)	21(1)
N(7)	-2489(4)	4287(3)	12132(1)	26(1)
N(8)	-8142(4)	7428(3)	11851(1)	30(1)
C(26)	-3468(6)	-1737(4)	15462(1)	35(1)
C(27)	-1685(6)	-1239(4)	15164(1)	35(1)
C(28)	-4052(6)	-511(4)	14580(1)	36(1)
C(29)	-5052(6)	-1333(4)	15058(2)	39(1)
C(30)	-520(9)	-1715(10)	16565(2)	87(2)
C(31)	-4071(14)	-3412(7)	16706(2)	100(3)
C(32)	-4004(8)	-138(6)	17035(2)	60(1)
C(33)	-6153(12)	0(15)	17018(3)	137(5)
C(34)	-3445(12)	-627(7)	17616(2)	86(2)
C(35)	-3218(17)	1403(7)	16892(3)	124(4)
C(36)	-690(6)	-2476(4)	14852(2)	42(1)
C(37A)	-2309(10)	-6008(9)	15128(5)	50(3)
C(38A)	1069(14)	-5673(11)	14384(3)	52(3)
C(37B)	-1483(17)	-6246(11)	14683(5)	63(4)
C(38B)	2422(16)	-5171(10)	14532(3)	45(3)
C(39)	1420(7)	-6370(4)	15639(2)	47(1)
C(40A)	-513(13)	-6545(12)	16053(4)	64(3)
C(41A)	2702(10)	-5505(10)	15956(3)	45(2)

C(40B)	1145(16)	-5909(10)	16175(3)	47(3)
C(41B)	3764(12)	-5987(9)	15488(4)	43(2)
C(42)	1729(10)	-8015(5)	15550(2)	64(1)
C(43)	-5119(6)	859(4)	14358(1)	35(1)
C(44)	-4218(5)	1548(4)	13869(1)	30(1)
C(45)	-5215(4)	3974(3)	12775(1)	19(1)
C(46)	-4382(4)	4682(3)	12311(1)	20(1)
C(47)	-5351(4)	5789(3)	12006(1)	22(1)
C(48)	-7160(4)	6210(3)	12154(1)	23(1)
C(49)	-8053(4)	5535(3)	12603(1)	24(1)
C(50)	-7101(4)	4441(3)	12906(1)	22(1)

Table 3. Bond lengths [Å] and angles [°] for **44**.

Si(1)-O(2)	1.655(3)
Si(1)-C(5)	1.859(4)
Si(1)-C(6)	1.861(4)
Si(1)-C(7)	1.872(4)
Si(2)-O(3)	1.650(2)
Si(2)-C(13)	1.852(5)
Si(2)-C(12)	1.853(4)
Si(2)-C(14)	1.907(5)
O(1)-C(2)	1.435(4)
O(1)-C(3)	1.438(4)
O(2)-C(1)	1.431(4)
O(3)-C(11)	1.426(4)
O(4)-N(3)	1.236(4)
O(5)-N(3)	1.214(4)
O(6)-N(4)	1.220(4)
O(7)-N(4)	1.236(4)
N(1)-C(19)	1.271(4)
N(1)-N(2)	1.388(3)
N(2)-C(20)	1.343(4)
N(3)-C(21)	1.454(4)
N(4)-C(23)	1.456(4)
C(1)-C(4)	1.514(4)
C(1)-C(2)	1.527(4)
C(2)-C(11)	1.514(4)
C(3)-C(4)	1.517(4)
C(3)-C(18)	1.523(4)
C(7)-C(8)	1.519(7)
C(7)-C(10)	1.538(6)
C(7)-C(9)	1.543(6)
C(14)-C(17)	1.509(6)
C(14)-C(16)	1.523(7)
C(14)-C(15)	1.556(9)

C(18)-C(19)	1.493(4)
C(20)-C(21)	1.420(4)
C(20)-C(25)	1.428(4)
C(21)-C(22)	1.394(4)
C(22)-C(23)	1.369(5)
C(23)-C(24)	1.400(5)
C(24)-C(25)	1.370(4)
Si(3)-O(9)	1.658(3)
Si(3)-C(30)	1.829(6)
Si(3)-C(31)	1.846(5)
Si(3)-C(32)	1.861(5)
Si(4)-O(10)	1.632(3)
Si(4)-C(38A)	1.782(8)
Si(4)-C(37B)	1.792(9)
Si(4)-C(39)	1.847(4)
Si(4)-C(37A)	1.952(8)
Si(4)-C(38B)	1.993(9)
O(8)-C(28)	1.430(5)
O(8)-C(27)	1.439(4)
O(9)-C(26)	1.430(4)
O(10)-C(36)	1.422(4)
O(11)-N(7)	1.240(3)
O(12)-N(7)	1.228(4)
O(13)-N(8)	1.221(4)
O(14)-N(8)	1.228(4)
N(5)-C(44)	1.275(4)
N(5)-N(6)	1.386(3)
N(6)-C(45)	1.354(4)
N(7)-C(46)	1.443(4)
N(8)-C(48)	1.460(4)
C(26)-C(27)	1.527(5)
C(26)-C(29)	1.539(5)
C(27)-C(36)	1.516(5)
C(28)-C(43)	1.505(5)
C(28)-C(29)	1.547(5)
C(32)-C(33)	1.525(10)
C(32)-C(35)	1.528(10)
C(32)-C(34)	1.541(6)
C(39)-C(40B)	1.413(9)
C(39)-C(41A)	1.465(9)
C(39)-C(42)	1.497(6)
C(39)-C(40A)	1.709(9)
C(39)-C(41B)	1.740(10)
C(43)-C(44)	1.486(5)
C(45)-C(50)	1.423(4)
C(45)-C(46)	1.424(4)

C(46)-C(47)	1.389(4)
C(47)-C(48)	1.371(4)
C(48)-C(49)	1.401(4)
C(49)-C(50)	1.372(4)

O(2)-Si(1)-C(5)	109.21(16)
O(2)-Si(1)-C(6)	109.87(16)
C(5)-Si(1)-C(6)	109.2(2)
O(2)-Si(1)-C(7)	104.44(16)
C(5)-Si(1)-C(7)	112.5(2)
C(6)-Si(1)-C(7)	111.49(19)
O(3)-Si(2)-C(13)	109.8(3)
O(3)-Si(2)-C(12)	105.41(16)
C(13)-Si(2)-C(12)	110.2(2)
O(3)-Si(2)-C(14)	107.37(18)
C(13)-Si(2)-C(14)	112.9(3)
C(12)-Si(2)-C(14)	110.9(2)
C(2)-O(1)-C(3)	109.0(2)
C(1)-O(2)-Si(1)	122.0(2)
C(11)-O(3)-Si(2)	124.1(2)
C(19)-N(1)-N(2)	114.7(3)
C(20)-N(2)-N(1)	119.2(2)
O(5)-N(3)-O(4)	122.3(3)
O(5)-N(3)-C(21)	119.0(3)
O(4)-N(3)-C(21)	118.7(3)
O(6)-N(4)-O(7)	123.1(3)
O(6)-N(4)-C(23)	118.9(3)
O(7)-N(4)-C(23)	118.1(3)
O(2)-C(1)-C(4)	113.6(3)
O(2)-C(1)-C(2)	109.8(2)
C(4)-C(1)-C(2)	102.4(2)
O(1)-C(2)-C(11)	110.4(3)
O(1)-C(2)-C(1)	106.7(2)
C(11)-C(2)-C(1)	114.1(3)
O(1)-C(3)-C(4)	104.8(2)
O(1)-C(3)-C(18)	108.0(2)
C(4)-C(3)-C(18)	115.5(3)
C(1)-C(4)-C(3)	101.2(2)
C(8)-C(7)-C(10)	110.1(4)
C(8)-C(7)-C(9)	109.5(4)
C(10)-C(7)-C(9)	108.4(4)
C(8)-C(7)-Si(1)	110.4(3)
C(10)-C(7)-Si(1)	109.0(3)
C(9)-C(7)-Si(1)	109.3(3)
O(3)-C(11)-C(2)	109.7(3)
C(17)-C(14)-C(16)	106.4(4)

C(17)-C(14)-C(15)	107.5(5)
C(16)-C(14)-C(15)	112.2(6)
C(17)-C(14)-Si(2)	111.6(3)
C(16)-C(14)-Si(2)	110.0(5)
C(15)-C(14)-Si(2)	109.1(3)
C(19)-C(18)-C(3)	110.5(3)
N(1)-C(19)-C(18)	120.1(3)
N(2)-C(20)-C(21)	123.5(3)
N(2)-C(20)-C(25)	120.2(3)
C(21)-C(20)-C(25)	116.3(3)
C(22)-C(21)-C(20)	122.0(3)
C(22)-C(21)-N(3)	116.2(3)
C(20)-C(21)-N(3)	121.8(3)
C(23)-C(22)-C(21)	119.0(3)
C(22)-C(23)-C(24)	121.5(3)
C(22)-C(23)-N(4)	118.7(3)
C(24)-C(23)-N(4)	119.8(3)
C(25)-C(24)-C(23)	119.6(3)
C(24)-C(25)-C(20)	121.6(3)
O(9)-Si(3)-C(30)	109.9(2)
O(9)-Si(3)-C(31)	110.0(2)
C(30)-Si(3)-C(31)	108.9(4)
O(9)-Si(3)-C(32)	106.09(18)
C(30)-Si(3)-C(32)	110.8(3)
C(31)-Si(3)-C(32)	111.2(3)
O(10)-Si(4)-C(38A)	115.9(3)
O(10)-Si(4)-C(37B)	121.2(4)
O(10)-Si(4)-C(39)	106.57(16)
C(38A)-Si(4)-C(39)	121.1(3)
C(37B)-Si(4)-C(39)	120.0(3)
O(10)-Si(4)-C(37A)	101.1(3)
C(38A)-Si(4)-C(37A)	107.8(5)
C(39)-Si(4)-C(37A)	101.7(4)
O(10)-Si(4)-C(38B)	99.1(3)
C(37B)-Si(4)-C(38B)	103.5(6)
C(39)-Si(4)-C(38B)	102.4(3)
C(28)-O(8)-C(27)	105.9(3)
C(26)-O(9)-Si(3)	123.4(2)
C(36)-O(10)-Si(4)	121.7(2)
C(44)-N(5)-N(6)	115.1(3)
C(45)-N(6)-N(5)	118.8(2)
O(12)-N(7)-O(11)	122.0(3)
O(12)-N(7)-C(46)	119.1(3)
O(11)-N(7)-C(46)	118.9(2)
O(13)-N(8)-O(14)	123.9(3)
O(13)-N(8)-C(48)	118.2(3)

O(14)-N(8)-C(48)	117.9(3)
O(9)-C(26)-C(27)	111.7(3)
O(9)-C(26)-C(29)	109.3(3)
C(27)-C(26)-C(29)	103.5(3)
O(8)-C(27)-C(36)	109.2(3)
O(8)-C(27)-C(26)	105.4(3)
C(36)-C(27)-C(26)	113.8(3)
O(8)-C(28)-C(43)	108.5(3)
O(8)-C(28)-C(29)	105.2(3)
C(43)-C(28)-C(29)	114.5(3)
C(26)-C(29)-C(28)	104.7(3)
C(33)-C(32)-C(35)	108.3(7)
C(33)-C(32)-C(34)	107.7(5)
C(35)-C(32)-C(34)	109.8(5)
C(33)-C(32)-Si(3)	110.4(5)
C(35)-C(32)-Si(3)	110.3(5)
C(34)-C(32)-Si(3)	110.2(4)
O(10)-C(36)-C(27)	109.2(3)
C(40B)-C(39)-C(42)	117.9(5)
C(41A)-C(39)-C(42)	122.8(5)
C(41A)-C(39)-C(40A)	103.3(6)
C(42)-C(39)-C(40A)	96.1(5)
C(40B)-C(39)-C(41B)	104.5(6)
C(42)-C(39)-C(41B)	92.8(5)
C(40B)-C(39)-Si(4)	122.1(4)
C(41A)-C(39)-Si(4)	116.2(4)
C(42)-C(39)-Si(4)	112.8(3)
C(40A)-C(39)-Si(4)	99.2(4)
C(41B)-C(39)-Si(4)	99.0(4)
C(44)-C(43)-C(28)	113.5(3)
N(5)-C(44)-C(43)	118.7(3)
N(6)-C(45)-C(50)	119.7(3)
N(6)-C(45)-C(46)	123.6(2)
C(50)-C(45)-C(46)	116.6(2)
C(47)-C(46)-C(45)	121.7(3)
C(47)-C(46)-N(7)	116.5(3)
C(45)-C(46)-N(7)	121.8(2)
C(48)-C(47)-C(46)	119.3(3)
C(47)-C(48)-C(49)	121.3(3)
C(47)-C(48)-N(8)	119.2(3)
C(49)-C(48)-N(8)	119.4(3)
C(50)-C(49)-C(48)	119.7(3)
C(49)-C(50)-C(45)	121.4(3)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **44**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Si(1)	31(1)	34(1)	26(1)	4(1)	-2(1)	-5(1)
Si(2)	30(1)	25(1)	36(1)	6(1)	7(1)	2(1)
O(1)	28(1)	42(1)	27(1)	-3(1)	2(1)	-4(1)
O(2)	39(1)	29(1)	29(1)	6(1)	0(1)	1(1)
O(3)	30(1)	23(1)	50(2)	-1(1)	3(1)	-3(1)
O(4)	25(1)	31(1)	38(1)	6(1)	3(1)	9(1)
O(5)	49(2)	53(2)	53(2)	27(1)	21(1)	18(1)
O(6)	62(2)	33(1)	28(1)	-1(1)	-10(1)	16(1)
O(7)	35(1)	42(1)	49(2)	-1(1)	-11(1)	17(1)
N(1)	23(1)	21(1)	24(1)	-1(1)	1(1)	-3(1)
N(2)	21(1)	21(1)	25(1)	1(1)	1(1)	1(1)
N(3)	30(1)	27(1)	33(1)	4(1)	5(1)	4(1)
N(4)	38(2)	25(1)	31(1)	-8(1)	-16(1)	9(1)
C(1)	30(2)	23(1)	27(1)	1(1)	-1(1)	4(1)
C(2)	28(1)	20(1)	28(1)	0(1)	-2(1)	2(1)
C(3)	30(2)	21(1)	26(1)	0(1)	0(1)	0(1)
C(4)	29(1)	26(1)	26(1)	1(1)	1(1)	-1(1)
C(5)	59(3)	50(2)	42(2)	-10(2)	8(2)	-19(2)
C(6)	37(2)	60(3)	37(2)	7(2)	-1(2)	3(2)
C(7)	45(2)	50(2)	33(2)	15(2)	-3(2)	4(2)
C(8)	67(3)	47(2)	77(3)	31(2)	-11(3)	-15(2)
C(9)	52(3)	66(3)	63(3)	6(2)	-16(2)	11(2)
C(10)	84(4)	94(4)	32(2)	26(2)	6(2)	21(3)
C(11)	26(2)	27(2)	39(2)	2(1)	-6(1)	1(1)
C(12)	40(2)	31(2)	71(3)	11(2)	4(2)	7(2)
C(13)	76(3)	92(4)	44(2)	26(3)	23(2)	37(3)
C(14)	29(2)	35(2)	93(4)	-20(2)	-3(2)	-5(1)
C(15)	64(3)	146(7)	68(4)	-56(4)	-8(3)	-5(4)
C(16)	61(4)	25(2)	325(14)	9(4)	-68(6)	-8(2)
C(17)	35(2)	37(2)	72(3)	-5(2)	-10(2)	-5(2)
C(18)	31(2)	24(1)	27(2)	-1(1)	-4(1)	0(1)
C(19)	27(1)	26(1)	25(1)	-4(1)	-1(1)	2(1)
C(20)	22(1)	16(1)	25(1)	-5(1)	-2(1)	-1(1)
C(21)	25(1)	21(1)	25(1)	-3(1)	-3(1)	1(1)
C(22)	34(2)	22(1)	23(1)	-3(1)	-4(1)	3(1)
C(23)	30(2)	21(1)	27(1)	-5(1)	-12(1)	5(1)
C(24)	22(1)	26(1)	34(2)	-6(1)	-5(1)	5(1)
C(25)	22(1)	22(1)	30(2)	-3(1)	-1(1)	0(1)
Si(3)	55(1)	32(1)	29(1)	5(1)	-9(1)	-10(1)
Si(4)	49(1)	24(1)	33(1)	4(1)	-17(1)	0(1)
O(8)	52(1)	21(1)	30(1)	7(1)	-11(1)	-1(1)

O(9)	59(2)	35(1)	27(1)	1(1)	-8(1)	-2(1)
O(10)	77(2)	26(1)	31(1)	7(1)	-15(1)	9(1)
O(11)	26(1)	29(1)	42(1)	6(1)	5(1)	9(1)
O(12)	33(1)	42(1)	47(2)	16(1)	16(1)	5(1)
O(13)	47(2)	49(2)	40(1)	20(1)	-6(1)	10(1)
O(14)	29(1)	30(1)	52(2)	5(1)	-10(1)	10(1)
N(5)	32(1)	19(1)	20(1)	0(1)	-3(1)	2(1)
N(6)	20(1)	17(1)	26(1)	1(1)	0(1)	3(1)
N(7)	24(1)	23(1)	30(1)	0(1)	0(1)	1(1)
N(8)	30(1)	24(1)	35(1)	3(1)	-13(1)	2(1)
C(26)	57(2)	22(1)	27(2)	4(1)	-11(1)	-5(1)
C(27)	57(2)	21(1)	27(2)	9(1)	-12(2)	-2(1)
C(28)	58(2)	24(2)	27(2)	2(1)	-7(2)	0(1)
C(29)	54(2)	28(2)	35(2)	9(1)	-14(2)	-9(2)
C(30)	66(3)	136(6)	54(3)	40(4)	-3(2)	30(4)
C(31)	208(8)	60(3)	37(2)	16(2)	-35(4)	-73(4)
C(32)	84(3)	67(3)	28(2)	-3(2)	-19(2)	21(3)
C(33)	97(5)	255(13)	49(3)	35(5)	6(3)	87(7)
C(34)	147(6)	74(4)	35(2)	-17(2)	-37(3)	35(4)
C(35)	256(12)	34(3)	83(5)	-8(3)	-59(6)	4(4)
C(36)	65(2)	28(2)	32(2)	11(1)	-7(2)	10(2)
C(37A)	29(4)	35(4)	88(7)	-13(4)	-7(4)	-2(3)
C(38A)	58(6)	63(6)	32(4)	11(4)	4(3)	28(4)
C(37B)	71(7)	36(5)	83(9)	24(5)	-50(7)	-19(5)
C(38B)	70(7)	34(4)	28(4)	7(3)	9(4)	5(4)
C(39)	78(3)	26(2)	37(2)	12(1)	-23(2)	4(2)
C(40A)	51(5)	73(6)	63(6)	39(5)	23(4)	13(4)
C(41A)	37(4)	61(5)	36(4)	12(3)	-10(3)	4(3)
C(40B)	86(7)	33(4)	23(4)	6(3)	-9(4)	10(4)
C(41B)	39(4)	33(4)	57(5)	-3(3)	-21(4)	3(3)
C(42)	113(4)	36(2)	40(2)	6(2)	-11(2)	23(2)
C(43)	47(2)	34(2)	23(1)	5(1)	-1(1)	6(1)
C(44)	35(2)	30(2)	23(1)	1(1)	-3(1)	5(1)
C(45)	20(1)	16(1)	22(1)	-2(1)	-4(1)	-1(1)
C(46)	19(1)	18(1)	25(1)	-3(1)	-1(1)	1(1)
C(47)	25(1)	18(1)	23(1)	1(1)	-3(1)	-1(1)
C(48)	24(1)	20(1)	26(1)	-2(1)	-8(1)	0(1)
C(49)	18(1)	22(1)	31(2)	-3(1)	-4(1)	1(1)
C(50)	23(1)	20(1)	24(1)	-2(1)	0(1)	0(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **44**.

	x	y	z	U(eq)
H(2A)	1358	1502	12376	27
H(1B)	1534	1711	9543	32
H(2B)	-1109	3864	9965	31
H(3A)	2689	1541	10560	31
H(4A)	2829	4420	9999	32
H(4B)	4084	2922	9871	32
H(5A)	1036	654	8612	75
H(5B)	995	1267	7997	75
H(5C)	-756	1623	8393	75
H(6A)	4925	3784	8746	67
H(6B)	4838	2771	8226	67
H(6C)	4642	2014	8818	67
H(8A)	1530	7171	8083	96
H(8B)	3343	6189	8280	96
H(8C)	1598	6462	8686	96
H(9A)	-1443	6033	7901	91
H(9B)	-1559	5231	8488	91
H(9C)	-1666	4253	7960	91
H(10A)	1308	5399	7292	107
H(10B)	1216	3632	7424	107
H(10C)	3123	4486	7525	107
H(11A)	-2865	1716	10226	37
H(11B)	-2453	1798	9587	37
H(12A)	1069	-2389	10473	72
H(12B)	471	-3020	9905	72
H(12C)	-439	-3683	10454	72
H(13A)	-1983	-616	11132	109
H(13B)	-3411	-1943	11055	109
H(13C)	-4002	-257	10866	109
H(15A)	-3781	-2041	8867	137
H(15B)	-1709	-2314	9109	137
H(15C)	-2607	-645	9049	137
H(16A)	-5649	-3561	9679	205
H(16B)	-4492	-3605	10230	205
H(16C)	-3499	-4176	9687	205
H(17A)	-6405	-1152	9467	72
H(17B)	-5135	201	9639	72
H(17C)	-6041	-884	10093	72
H(18A)	4455	3319	11022	33
H(18B)	2680	4485	11007	33
H(19A)	1053	2918	11653	31

H(22A)	3090	-2229	13635	31
H(24A)	7543	-1743	12607	33
H(25A)	5907	133	12122	29
H(6D)	-3128	2638	13019	25
H(26A)	-3399	-2844	15546	43
H(27A)	-791	-835	15428	42
H(28A)	-3794	-1229	14284	44
H(29A)	-6033	-669	15223	47
H(29B)	-5649	-2250	14937	47
H(30A)	-69	-2417	16292	130
H(30B)	-117	-2090	16924	130
H(30C)	7	-727	16488	130
H(31A)	-3494	-4150	16464	151
H(31B)	-5442	-3361	16655	151
H(31C)	-3794	-3715	17081	151
H(33A)	-6602	808	17251	205
H(33B)	-6687	-950	17145	205
H(33C)	-6553	230	16646	205
H(34A)	-4090	37	17872	128
H(34B)	-2075	-564	17655	128
H(34C)	-3814	-1664	17690	128
H(35A)	-3710	2135	17149	186
H(35B)	-3605	1720	16526	186
H(35C)	-1836	1341	16909	186
H(36A)	-1587	-2920	14605	51
H(36B)	357	-2056	14633	51
H(37A)	-2796	-5802	15491	76
H(37B)	-2342	-7089	15068	76
H(37C)	-3095	-5461	14859	76
H(38A)	134	-5237	14130	78
H(38B)	1221	-6755	14331	78
H(38C)	2283	-5198	14320	78
H(37D)	-2664	-6259	14893	95
H(37E)	-1059	-7280	14610	95
H(37F)	-1693	-5686	14341	95
H(38D)	3561	-4901	14722	67
H(38E)	2102	-4388	14258	67
H(38F)	2654	-6131	14357	67
H(40A)	-120	-6985	16404	95
H(40B)	-1414	-7198	15889	95
H(40C)	-1113	-5551	16105	95
H(41A)	3020	-6071	16291	67
H(41B)	2087	-4540	16044	67
H(41C)	3859	-5326	15746	67
H(40D)	2106	-6404	16407	71
H(40E)	-114	-6186	16304	71

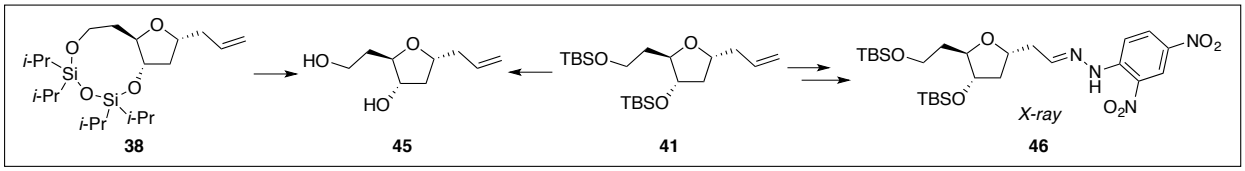
H(40F)	1253	-4817	16186	71
H(41D)	4594	-6598	15728	64
H(41E)	3970	-4919	15540	64
H(41F)	4046	-6226	15112	64
H(42A)	2503	-8468	15841	96
H(42B)	2380	-8155	15203	96
H(42C)	508	-8498	15547	96
H(43A)	-6414	577	14268	42
H(43B)	-5223	1616	14641	42
H(44A)	-2895	1433	13809	35
H(47A)	-4768	6249	11699	27
H(49A)	-9311	5834	12698	28
H(50A)	-7715	3987	13210	26

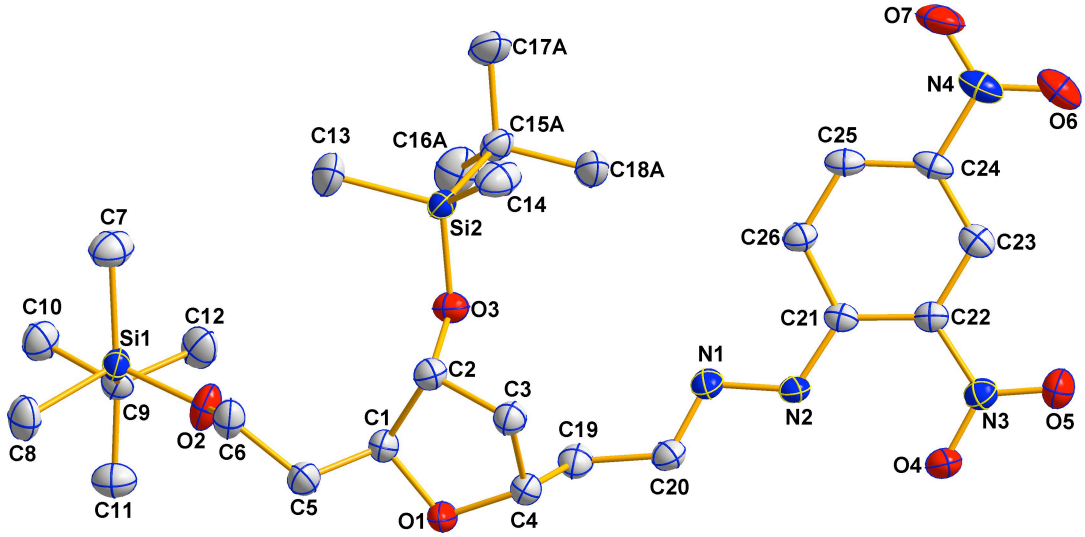
Table 6. Hydrogen bonds for **44** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(2)-H(2A)...O(4)	0.88	1.97	2.608(3)	127.8
N(2)-H(2A)...O(11)	0.88	2.57	3.397(3)	155.9
C(19)-H(19A)...O(11)	0.95	2.61	3.479(4)	152.8
C(24)-H(24A)...O(14)#1	0.95	2.44	3.351(4)	159.7
N(6)-H(6D)...O(4)	0.88	2.62	3.447(3)	156.3
N(6)-H(6D)...O(11)	0.88	1.99	2.611(3)	126.9
C(28)-H(28A)...O(6)#2	1.00	2.63	3.527(4)	149.5
C(49)-H(49A)...O(7)#3	0.95	2.39	3.287(4)	157.1

Symmetry transformations used to generate equivalent atoms:
#1 x+2,y-1,z #2 x-1,y,z #3 x-2,y+1,z

Chemical Correlation:





X-ray crystallographic data for **46**.

A yellow block-like crystal with the size of 0.24 × 0.32 × 0.52 mm^3 was selected for geometry and intensity data collection with a Bruker SMART APEXII CCD area detector on a D8 goniometer at 100 K. The temperature during the data collection was controlled with an Oxford Cryosystems Series 700+ instrument. Preliminary lattice parameters and orientation matrices were obtained from three sets of frames. Data were collected using graphite-monochromated and 0.5 mm-MonoCap-collimated Mo-K_α radiation (λ = 0.71073 Å) with the ω and ϕ scan method [1]. Data were processed with the INTEGRATE program of the APEX2 software [1] for reduction and cell refinement. Multi-scan absorption corrections were applied by using the SCALE program for the area detector. The structure was solved by the direct method and refined on F^2 (SHELXTL) [2]. Non-hydrogen atoms were refined with anisotropic displacement parameters, and hydrogen atoms were placed in idealized positions (C-H = 0.95-1.00 and N-H = 0.88 Å) and included as riding with *U*iso(H) = 1.2 or 1.5 *U*eq(non-H).

[1] APEX2 (version 2012.4). *Program for Bruker CCD X-ray Diffractometer Control*, Bruker AXS Inc., Madison, WI, 2012.

[2] G. M. Sheldrick, SHELXTL, version 6.14. *Program for solution and refinement of crystal structures*, Universität Göttingen, Germany, 2009.

Table 7. Crystal data and structure refinement for **46**.

Identification code	12kaw11h
Empirical formula	$C_{26}H_{46}N_4O_7Si_2$
Formula weight	582.85
Temperature	100(2) K

Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, $P2_1/c$
Unit cell dimensions	a = 24.0157(13) Å α = 90°
	b = 8.1309(4) Å β = 100.0940(10)°
	c = 16.5638(9) Å γ = 90°
Volume	3184.3(3) Å^3
Z, Calculated density	4, 1.216 Mg/m^3
Absorption coefficient	0.157 mm^{-1}
F(000)	1256
Crystal size	0.52 x 0.32 x 0.24 mm^3
Theta range for data collection	1.72 to 28.29°
Limiting indices	$-31 \leq h \leq 31, -10 \leq k \leq 10, -22 \leq l \leq 21$
Reflections collected / unique	64142 / 7864 [R(int) = 0.0295]
Completeness to theta = 28.29	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9632 and 0.9226
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7864 / 40 / 406
Goodness-of-fit on F^2	1.067
Final R indices [I>2sigma(I)]	R1 = 0.0436, wR2 = 0.1115
R indices (all data)	R1 = 0.0511, wR2 = 0.1167
Largest diff. peak and hole	0.423 and -0.243 e.Å^{-3}

Table 8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for **46**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Si(1)	4105(1)	12980(1)	3894(1)	26(1)
Si(2)	2598(1)	9399(1)	5741(1)	25(1)
Si(2')	2892(3)	8990(10)	5457(5)	25(1)
O(1)	1857(1)	10352(2)	2822(1)	37(1)
O(2)	3437(1)	12439(2)	3630(1)	38(1)
O(3)	2372(1)	9381(1)	4735(1)	30(1)
O(4)	-307(1)	3264(2)	3400(1)	34(1)
O(5)	-714(1)	1657(2)	4160(1)	41(1)
O(6)	65(1)	455(2)	6901(1)	44(1)
O(7)	828(1)	1697(2)	7494(1)	50(1)
N(1)	1106(1)	5768(2)	3996(1)	28(1)
N(2)	626(1)	4820(2)	4009(1)	24(1)
N(3)	-325(1)	2567(2)	4060(1)	28(1)
N(4)	462(1)	1416(2)	6893(1)	36(1)
C(1)	2298(1)	10929(2)	3471(1)	29(1)
C(2)	2076(1)	10703(2)	4285(1)	28(1)
C(3)	1449(1)	10297(2)	4003(1)	30(1)
C(4)	1437(1)	9482(2)	3170(1)	30(1)

C(5)	2429(1)	12691(2)	3270(1)	34(1)
C(6)	2963(1)	13359(2)	3781(1)	34(1)
C(7)	4339(1)	12918(3)	5024(1)	45(1)
C(8)	4204(1)	15107(2)	3526(1)	40(1)
C(9)	4504(1)	11428(2)	3374(1)	29(1)
C(10)	5143(1)	11695(2)	3624(1)	38(1)
C(11)	4340(1)	11594(2)	2438(1)	41(1)
C(12)	4356(1)	9684(2)	3622(1)	40(1)
C(13)	3169(1)	10946(3)	6015(2)	51(1)
C(14)	2011(1)	9932(3)	6284(1)	46(1)
C(15A)	2833(2)	7233(4)	6043(2)	30(1)
C(16A)	3286(2)	6700(5)	5552(3)	48(1)
C(17A)	3073(2)	7106(6)	6963(3)	45(1)
C(18A)	2326(2)	6055(6)	5846(3)	39(1)
C(15B)	2943(4)	7295(9)	5850(5)	30(1)
C(16B)	3356(4)	7093(12)	5253(6)	48(1)
C(17B)	3259(4)	7155(13)	6738(5)	45(1)
C(18B)	2476(4)	5965(16)	5712(8)	39(1)
C(13')	3110(20)	10870(30)	6060(30)	51(1)
C(14')	3535(10)	7990(40)	5220(20)	46(1)
C(15')	2575(8)	7550(30)	6143(12)	30(1)
C(16')	1964(10)	8100(50)	6150(20)	48(1)
C(17')	2937(17)	7490(60)	7000(20)	45(1)
C(18')	2609(15)	5970(40)	5650(20)	39(1)
C(19)	1576(1)	7645(2)	3213(1)	32(1)
C(20)	1082(1)	6637(2)	3348(1)	30(1)
C(21)	583(1)	3950(2)	4686(1)	22(1)
C(22)	132(1)	2852(2)	4739(1)	23(1)
C(23)	96(1)	2018(2)	5461(1)	27(1)
C(24)	507(1)	2271(2)	6135(1)	28(1)
C(25)	961(1)	3327(2)	6119(1)	29(1)
C(26)	998(1)	4150(2)	5408(1)	27(1)

Table 9. Bond lengths [Å] and angles [°] for **46**.

Si(1)-O(2)	1.6479(12)
Si(1)-C(7)	1.8591(19)
Si(1)-C(8)	1.8621(18)
Si(1)-C(9)	1.8808(17)
Si(2)-O(3)	1.6600(11)
Si(2)-C(14)	1.852(2)
Si(2)-C(13)	1.858(2)
Si(2)-C(15A)	1.890(3)
Si(2)-C(15B)	1.895(7)
Si(2')-O(3)	1.603(6)

Si(2')-C(14')	1.849(10)
Si(2')-C(13')	1.853(10)
Si(2')-C(15')	1.884(9)
O(1)-C(4)	1.4343(18)
O(1)-C(1)	1.4485(18)
O(2)-C(6)	1.420(2)
O(3)-C(2)	1.4251(19)
O(4)-N(3)	1.2388(17)
O(5)-N(3)	1.2260(18)
O(6)-N(4)	1.234(2)
O(7)-N(4)	1.229(2)
N(1)-C(20)	1.278(2)
N(1)-N(2)	1.3902(17)
N(2)-C(21)	1.3447(18)
N(2)-H(2N)	0.8800
N(3)-C(22)	1.4459(18)
N(4)-C(24)	1.4544(19)
C(1)-C(5)	1.516(2)
C(1)-C(2)	1.546(2)
C(1)-H(1)	1.0000
C(2)-C(3)	1.531(2)
C(2)-H(2)	1.0000
C(3)-C(4)	1.527(2)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-C(19)	1.530(2)
C(4)-H(4)	1.0000
C(5)-C(6)	1.509(2)
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-H(7A)	0.9800
C(7)-H(7B)	0.9800
C(7)-H(7C)	0.9800
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(9)-C(10)	1.534(2)
C(9)-C(12)	1.535(2)
C(9)-C(11)	1.537(2)
C(10)-H(10A)	0.9800
C(10)-H(10B)	0.9800
C(10)-H(10C)	0.9800
C(11)-H(11A)	0.9800
C(11)-H(11B)	0.9800

C(11)-H(11C)	0.9800
C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(12)-H(12C)	0.9800
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800
C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
C(15A)-C(16A)	1.530(4)
C(15A)-C(17A)	1.535(4)
C(15A)-C(18A)	1.540(4)
C(16A)-H(16A)	0.9800
C(16A)-H(16B)	0.9800
C(16A)-H(16C)	0.9800
C(17A)-H(17A)	0.9800
C(17A)-H(17B)	0.9800
C(17A)-H(17C)	0.9800
C(18A)-H(18A)	0.9800
C(18A)-H(18B)	0.9800
C(18A)-H(18C)	0.9800
C(15B)-C(16B)	1.528(8)
C(15B)-C(17B)	1.536(8)
C(15B)-C(18B)	1.546(8)
C(16B)-H(16D)	0.9800
C(16B)-H(16E)	0.9800
C(16B)-H(16F)	0.9800
C(17B)-H(17D)	0.9800
C(17B)-H(17E)	0.9800
C(17B)-H(17F)	0.9800
C(18B)-H(18D)	0.9800
C(18B)-H(18E)	0.9800
C(18B)-H(18F)	0.9800
C(13')-H(13D)	0.9800
C(13')-H(13E)	0.9800
C(13')-H(13F)	0.9800
C(14')-H(14D)	0.9800
C(14')-H(14E)	0.9800
C(14')-H(14F)	0.9800
C(15')-C(18')	1.529(10)
C(15')-C(17')	1.535(10)
C(15')-C(16')	1.538(10)
C(16')-H(16G)	0.9800
C(16')-H(16H)	0.9800
C(16')-H(16I)	0.9800

C(17')-H(17G)	0.9800
C(17')-H(17H)	0.9800
C(17')-H(17I)	0.9800
C(18')-H(18G)	0.9800
C(18')-H(18H)	0.9800
C(18')-H(18I)	0.9800
C(19)-C(20)	1.490(2)
C(19)-H(19A)	0.9900
C(19)-H(19B)	0.9900
C(20)-H(20)	0.9500
C(21)-C(22)	1.418(2)
C(21)-C(26)	1.425(2)
C(22)-C(23)	1.392(2)
C(23)-C(24)	1.370(2)
C(23)-H(23)	0.9500
C(24)-C(25)	1.392(2)
C(25)-C(26)	1.372(2)
C(25)-H(25)	0.9500
C(26)-H(26)	0.9500

O(2)-Si(1)-C(7)	111.59(8)
O(2)-Si(1)-C(8)	109.59(8)
C(7)-Si(1)-C(8)	108.73(10)
O(2)-Si(1)-C(9)	104.48(7)
C(7)-Si(1)-C(9)	110.76(8)
C(8)-Si(1)-C(9)	111.67(8)
O(3)-Si(2)-C(14)	110.25(8)
O(3)-Si(2)-C(13)	110.45(11)
C(14)-Si(2)-C(13)	108.36(12)
O(3)-Si(2)-C(15A)	106.73(12)
C(14)-Si(2)-C(15A)	107.88(15)
C(13)-Si(2)-C(15A)	113.13(16)
O(3)-Si(2)-C(15B)	98.6(3)
C(14)-Si(2)-C(15B)	121.5(3)
C(13)-Si(2)-C(15B)	107.2(3)
O(3)-Si(2')-C(14')	119.9(13)
O(3)-Si(2')-C(13')	110.4(18)
C(14')-Si(2')-C(13')	108.3(8)
O(3)-Si(2')-C(15')	103.3(8)
C(14')-Si(2')-C(15')	107.2(7)
C(13')-Si(2')-C(15')	106.9(8)
C(4)-O(1)-C(1)	109.57(11)
C(6)-O(2)-Si(1)	125.88(11)
C(2)-O(3)-Si(2')	142.4(3)
C(2)-O(3)-Si(2)	124.35(10)
C(20)-N(1)-N(2)	113.87(13)

C(21)-N(2)-N(1)	119.32(12)
C(21)-N(2)-H(2N)	120.3
N(1)-N(2)-H(2N)	120.3
O(5)-N(3)-O(4)	122.73(13)
O(5)-N(3)-C(22)	118.99(13)
O(4)-N(3)-C(22)	118.28(13)
O(7)-N(4)-O(6)	123.49(14)
O(7)-N(4)-C(24)	117.42(15)
O(6)-N(4)-C(24)	119.09(15)
O(1)-C(1)-C(5)	107.21(13)
O(1)-C(1)-C(2)	106.88(12)
C(5)-C(1)-C(2)	114.91(14)
O(1)-C(1)-H(1)	109.2
C(5)-C(1)-H(1)	109.2
C(2)-C(1)-H(1)	109.2
O(3)-C(2)-C(3)	111.78(13)
O(3)-C(2)-C(1)	109.21(13)
C(3)-C(2)-C(1)	103.35(12)
O(3)-C(2)-H(2)	110.8
C(3)-C(2)-H(2)	110.8
C(1)-C(2)-H(2)	110.8
C(4)-C(3)-C(2)	103.38(12)
C(4)-C(3)-H(3A)	111.1
C(2)-C(3)-H(3A)	111.1
C(4)-C(3)-H(3B)	111.1
C(2)-C(3)-H(3B)	111.1
H(3A)-C(3)-H(3B)	109.1
O(1)-C(4)-C(3)	104.27(12)
O(1)-C(4)-C(19)	109.68(13)
C(3)-C(4)-C(19)	114.34(13)
O(1)-C(4)-H(4)	109.5
C(3)-C(4)-H(4)	109.5
C(19)-C(4)-H(4)	109.5
C(6)-C(5)-C(1)	113.92(13)
C(6)-C(5)-H(5A)	108.8
C(1)-C(5)-H(5A)	108.8
C(6)-C(5)-H(5B)	108.8
C(1)-C(5)-H(5B)	108.8
H(5A)-C(5)-H(5B)	107.7
O(2)-C(6)-C(5)	109.87(14)
O(2)-C(6)-H(6A)	109.7
C(5)-C(6)-H(6A)	109.7
O(2)-C(6)-H(6B)	109.7
C(5)-C(6)-H(6B)	109.7
H(6A)-C(6)-H(6B)	108.2
Si(1)-C(7)-H(7A)	109.5

Si(1)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
Si(1)-C(7)-H(7C)	109.5
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
Si(1)-C(8)-H(8A)	109.5
Si(1)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
Si(1)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(10)-C(9)-C(12)	109.00(14)
C(10)-C(9)-C(11)	109.07(14)
C(12)-C(9)-C(11)	108.68(14)
C(10)-C(9)-Si(1)	110.32(11)
C(12)-C(9)-Si(1)	109.73(12)
C(11)-C(9)-Si(1)	110.00(11)
C(9)-C(10)-H(10A)	109.5
C(9)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(9)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
C(9)-C(11)-H(11A)	109.5
C(9)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
C(9)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
C(9)-C(12)-H(12A)	109.5
C(9)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(9)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(16A)-C(15A)-C(17A)	109.4(3)
C(16A)-C(15A)-C(18A)	108.7(3)
C(17A)-C(15A)-C(18A)	108.3(3)
C(16A)-C(15A)-Si(2)	109.3(2)
C(17A)-C(15A)-Si(2)	111.6(3)
C(18A)-C(15A)-Si(2)	109.6(3)
C(16B)-C(15B)-C(17B)	110.2(7)
C(16B)-C(15B)-C(18B)	111.4(7)
C(17B)-C(15B)-C(18B)	108.3(8)
C(16B)-C(15B)-Si(2)	110.9(5)
C(17B)-C(15B)-Si(2)	107.0(6)

C(18B)-C(15B)-Si(2)	108.9(7)
Si(2')-C(13')-H(13D)	109.5
Si(2')-C(13')-H(13E)	109.5
H(13D)-C(13')-H(13E)	109.5
Si(2')-C(13')-H(13F)	109.5
H(13D)-C(13')-H(13F)	109.5
H(13E)-C(13')-H(13F)	109.5
Si(2')-C(14')-H(14D)	109.5
Si(2')-C(14')-H(14E)	109.5
H(14D)-C(14')-H(14E)	109.5
Si(2')-C(14')-H(14F)	109.5
H(14D)-C(14')-H(14F)	109.5
H(14E)-C(14')-H(14F)	109.5
C(18')-C(15')-C(17')	113.4(10)
C(18')-C(15')-C(16')	113.0(10)
C(17')-C(15')-C(16')	112.7(10)
C(18')-C(15')-Si(2')	97.8(19)
C(17')-C(15')-Si(2')	111(2)
C(16')-C(15')-Si(2')	108(2)
C(15')-C(16')-H(16G)	109.5
C(15')-C(16')-H(16H)	109.5
H(16G)-C(16')-H(16H)	109.5
C(15')-C(16')-H(16I)	109.5
H(16G)-C(16')-H(16I)	109.5
H(16H)-C(16')-H(16I)	109.5
C(15')-C(17')-H(17G)	109.5
C(15')-C(17')-H(17H)	109.5
H(17G)-C(17')-H(17H)	109.5
C(15')-C(17')-H(17I)	109.5
H(17G)-C(17')-H(17I)	109.5
H(17H)-C(17')-H(17I)	109.5
C(15')-C(18')-H(18G)	109.5
C(15')-C(18')-H(18H)	109.5
H(18G)-C(18')-H(18H)	109.5
C(15')-C(18')-H(18I)	109.5
H(18G)-C(18')-H(18I)	109.5
H(18H)-C(18')-H(18I)	109.5
C(20)-C(19)-C(4)	111.80(14)
C(20)-C(19)-H(19A)	109.3
C(4)-C(19)-H(19A)	109.3
C(20)-C(19)-H(19B)	109.3
C(4)-C(19)-H(19B)	109.3
H(19A)-C(19)-H(19B)	107.9
N(1)-C(20)-C(19)	120.70(14)
N(1)-C(20)-H(20)	119.7
C(19)-C(20)-H(20)	119.7

N(2)-C(21)-C(22)	123.58(13)
N(2)-C(21)-C(26)	119.64(13)
C(22)-C(21)-C(26)	116.76(13)
C(23)-C(22)-C(21)	121.57(13)
C(23)-C(22)-N(3)	115.91(13)
C(21)-C(22)-N(3)	122.50(13)
C(24)-C(23)-C(22)	118.88(15)
C(24)-C(23)-H(23)	120.6
C(22)-C(23)-H(23)	120.6
C(23)-C(24)-C(25)	122.9(14)
C(23)-C(24)-N(4)	118.68(15)
C(25)-C(24)-N(4)	119.23(14)
C(26)-C(25)-C(24)	119.20(14)
C(26)-C(25)-H(25)	120.4
C(24)-C(25)-H(25)	120.4
C(25)-C(26)-C(21)	121.51(14)
C(25)-C(26)-H(26)	119.2
C(21)-C(26)-H(26)	119.2

Symmetry transformations used to generate equivalent atoms:

Table 10. Anisotropic displacement parameters ($Å^2$ x 10^3) for **46**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Si(1)	23(1)	24(1)	30(1)	0(1)	2(1)	-5(1)
Si(2)	23(1)	26(1)	25(1)	0(1)	-1(1)	-3(1)
Si(2')	23(1)	26(1)	25(1)	0(1)	-1(1)	-3(1)
O(1)	35(1)	51(1)	24(1)	4(1)	2(1)	-19(1)
O(2)	23(1)	31(1)	57(1)	-8(1)	2(1)	-4(1)
O(3)	35(1)	30(1)	24(1)	0(1)	2(1)	2(1)
O(4)	37(1)	38(1)	24(1)	1(1)	-2(1)	-4(1)
O(5)	34(1)	47(1)	41(1)	0(1)	3(1)	-13(1)
O(6)	56(1)	43(1)	38(1)	12(1)	22(1)	4(1)
O(7)	73(1)	52(1)	22(1)	9(1)	4(1)	1(1)
N(1)	31(1)	31(1)	22(1)	-4(1)	4(1)	-5(1)
N(2)	28(1)	26(1)	18(1)	-1(1)	1(1)	-2(1)
N(3)	30(1)	28(1)	27(1)	-2(1)	3(1)	0(1)
N(4)	52(1)	32(1)	26(1)	6(1)	14(1)	10(1)
C(1)	26(1)	34(1)	27(1)	2(1)	3(1)	-7(1)
C(2)	30(1)	30(1)	25(1)	0(1)	3(1)	-1(1)
C(3)	28(1)	32(1)	30(1)	-2(1)	5(1)	-3(1)
C(4)	28(1)	36(1)	26(1)	2(1)	2(1)	-7(1)

C(5)	27(1)	37(1)	36(1)	8(1)	1(1)	-3(1)
C(6)	28(1)	28(1)	46(1)	2(1)	4(1)	-3(1)
C(7)	41(1)	61(1)	33(1)	-4(1)	7(1)	1(1)
C(8)	40(1)	26(1)	55(1)	1(1)	7(1)	-5(1)
C(9)	28(1)	26(1)	30(1)	3(1)	2(1)	0(1)
C(10)	28(1)	42(1)	44(1)	2(1)	6(1)	2(1)
C(11)	46(1)	43(1)	32(1)	-1(1)	4(1)	5(1)
C(12)	43(1)	25(1)	51(1)	3(1)	1(1)	0(1)
C(13)	51(1)	45(1)	54(1)	2(1)	-5(1)	-22(1)
C(14)	41(1)	66(1)	31(1)	-7(1)	8(1)	1(1)
C(15A)	24(2)	32(1)	30(2)	0(1)	-2(1)	1(1)
C(16A)	36(1)	45(2)	63(2)	-3(2)	6(2)	9(1)
C(17A)	44(2)	43(2)	40(2)	9(1)	-12(1)	1(2)
C(18A)	40(2)	33(1)	40(2)	6(1)	-4(1)	-9(2)
C(15B)	24(2)	32(1)	30(2)	0(1)	-2(1)	1(1)
C(16B)	36(1)	45(2)	63(2)	-3(2)	6(2)	9(1)
C(17B)	44(2)	43(2)	40(2)	9(1)	-12(1)	1(2)
C(18B)	40(2)	33(1)	40(2)	6(1)	-4(1)	-9(2)
C(13')	51(1)	45(1)	54(1)	2(1)	-5(1)	-22(1)
C(14')	41(1)	66(1)	31(1)	-7(1)	8(1)	1(1)
C(15')	24(2)	32(1)	30(2)	0(1)	-2(1)	1(1)
C(16')	36(1)	45(2)	63(2)	-3(2)	6(2)	9(1)
C(17')	44(2)	43(2)	40(2)	9(1)	-12(1)	1(2)
C(18')	40(2)	33(1)	40(2)	6(1)	-4(1)	-9(2)
C(19)	33(1)	36(1)	28(1)	-4(1)	8(1)	-4(1)
C(20)	34(1)	30(1)	23(1)	-3(1)	2(1)	-3(1)
C(21)	27(1)	21(1)	19(1)	-1(1)	6(1)	5(1)
C(22)	26(1)	22(1)	22(1)	-2(1)	4(1)	4(1)
C(23)	32(1)	24(1)	26(1)	-1(1)	11(1)	4(1)
C(24)	39(1)	27(1)	20(1)	3(1)	9(1)	9(1)
C(25)	34(1)	32(1)	19(1)	-1(1)	3(1)	7(1)
C(26)	29(1)	30(1)	21(1)	-2(1)	4(1)	0(1)

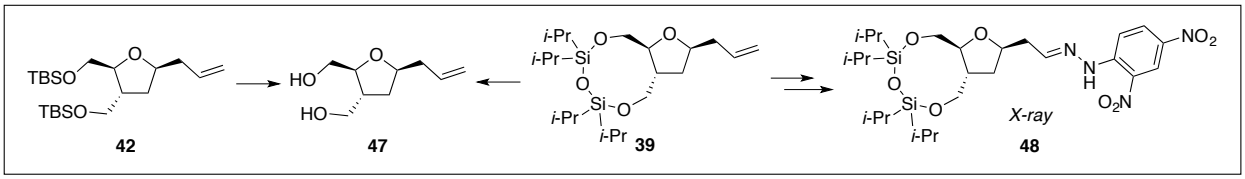
Table 11. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **46**.

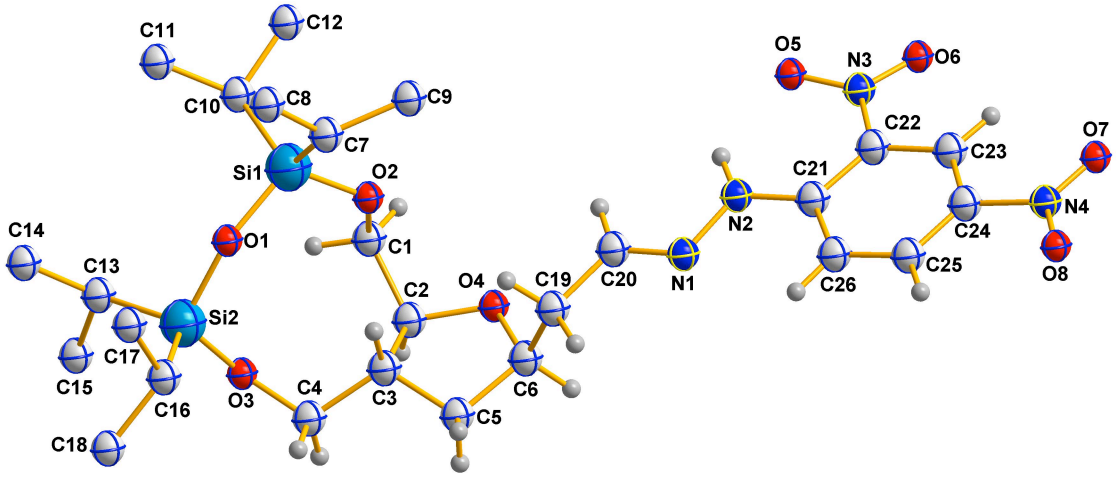
	x	y	z	U(eq)
H(2N)	353	4790	3579	29
H(1)	2644	10241	3483	35
H(2)	2121	11740	4614	34
H(3A)	1311	9537	4391	36
H(3B)	1215	11308	3951	36
H(4)	1057	9655	2821	36
H(5A)	2464	12755	2684	41
H(5B)	2107	13397	3350	41

H(6A)	3011	14529	3643	41
H(6B)	2936	13290	4370	41
H(7A)	4083	13586	5289	67
H(7B)	4724	13356	5163	67
H(7C)	4334	11779	5216	67
H(8A)	4057	15174	2937	61
H(8B)	4608	15382	3630	61
H(8C)	3999	15884	3819	61
H(10A)	5251	11572	4219	57
H(10B)	5241	12804	3464	57
H(10C)	5346	10881	3349	57
H(11A)	4539	10755	2172	61
H(11B)	4446	12689	2270	61
H(11C)	3931	11444	2275	61
H(12A)	4563	8880	3347	61
H(12B)	3949	9501	3458	61
H(12C)	4463	9559	4217	61
H(13A)	3486	10676	5738	77
H(13B)	3299	10938	6610	77
H(13C)	3024	12040	5843	77
H(14A)	1883	11055	6140	69
H(14B)	2143	9858	6877	69
H(14C)	1697	9164	6122	69
H(16A)	3388	5549	5676	72
H(16B)	3622	7394	5701	72
H(16C)	3138	6815	4965	72
H(17A)	3173	5961	7104	67
H(17B)	2787	7480	7278	67
H(17C)	3411	7798	7095	67
H(18A)	2173	6097	5258	59
H(18B)	2032	6386	6156	59
H(18C)	2449	4931	5999	59
H(16D)	3548	6028	5345	72
H(16E)	3638	7978	5341	72
H(16F)	3149	7145	4689	72
H(17D)	3394	6026	6844	67
H(17E)	3001	7438	7116	67
H(17F)	3581	7913	6822	67
H(18D)	2248	6097	5164	59
H(18E)	2234	6081	6127	59
H(18F)	2651	4872	5758	59
H(13D)	2785	11294	6291	77
H(13E)	3234	11706	5710	77
H(13F)	3418	10605	6513	77
H(14D)	3801	8832	5100	69
H(14E)	3432	7275	4738	69

H(14F)	3712	7329	5689	69
H(16G)	1743	7167	6298	72
H(16H)	1794	8507	5607	72
H(16I)	1964	8982	6556	72
H(17G)	2807	8342	7347	67
H(17H)	3334	7688	6966	67
H(17I)	2900	6408	7249	67
H(18G)	2399	5092	5871	59
H(18H)	3005	5641	5691	59
H(18I)	2443	6165	5075	59
H(19A)	1694	7299	2695	39
H(19B)	1897	7446	3667	39
H(20)	746	6641	2949	36
H(23)	-209	1286	5486	32
H(25)	1241	3475	6594	34
H(26)	1308	4870	5396	32

Chemical Correlation:





X-ray Data Collection, Structure Solution and Refinement for **48**.

A colorless prismatic crystal with the size of 0.04 × 0.09 × 0.56 mm^3 was selected for geometry and intensity data collection with a Bruker SMART APEXII CCD area detector on a D8 goniometer at 100 K. The temperature during the data collection was controlled with an Oxford Cryosystems Series 700+ instrument. Preliminary lattice parameters and orientation matrices were obtained from three sets of frames. Data were collected using graphite-monochromated and 0.5 mm-MonoCap-collimated Mo-K_α radiation (λ = 0.71073 Å) with the ω scan method [1]. Data

were processed with the INTEGRATE program of the APEX2 software [1] for reduction and cell refinement. Multi-scan absorption corrections were applied by using the SCALE program for area detector. The structure was solved by the direct method and refined on F^2 (SHELXTL) [2]. Non-hydrogen atoms were refined with anisotropic displacement parameters, and hydrogen atoms on carbons and nitrogen were placed in idealized positions (C-H = 0.95-1.00 Å and N-H = 0.88 Å) and included as riding with *U*iso(H) = 1.2 or 1.5 *U*eq(non-H).

[1] APEX2 (version 2011.4). *Program for Bruker CCD X-ray Diffractometer Control*, Bruker AXS Inc., Madison, WI, 2011.
[2] G. M. Sheldrick, SHELXTL, version 6.14. *Program for solution and refinement of crystal structures*, Universität Göttingen, Germany, 2009.

Table 12. Crystal data and structure refinement for **48**.

Identification code	12kaw3h2	
Empirical formula	$C_{26}H_{44}N_4O_8Si_2$	
Formula weight	596.83	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	Monoclinic, $P2_1/c$	
Unit cell dimensions	a = 22.418(6) Å	α = 90°
	b = 7.6590(19) Å	β = 100.870(4)°
	c = 18.298(5) Å	γ = 90°
Volume	3085.4(13) $Å^3$	
Z, Calculated density	4, 1.285 Mg/m^3	
Absorption coefficient	0.167 mm^{-1}	
F(000)	1280	
Crystal size	0.56 x 0.09 x 0.04 mm^3	
Theta range for data collection	1.85 to 21.99°	
Limiting indices	$-23 \le h \le 23$, $-8 \le k \le 8$, $-19 \le l \le 19$	
Reflections collected / unique	31572 / 3768 [R(int) = 0.1028]	
Completeness to theta = 21.99°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9934 and 0.9125	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3768 / 122 / 369	
Goodness-of-fit on F^2	1.033	
Final R indices [I>2sigma(I)]	R1 = 0.0933, wR2 = 0.2181	
R indices (all data)	R1 = 0.1617, wR2 = 0.2357	
Largest diff. peak and hole	0.527 and -0.403 e.$Å^{-3}$	

Table 13. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **48**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Si(1)	1989(1)	843(4)	190(2)	57(1)

Si(2)	1416(1)	-737(4)	1508(2)	61(1)
O(1)	1554(3)	300(9)	770(4)	65(2)
O(2)	2688(3)	1143(8)	626(3)	47(2)
O(3)	2040(3)	-894(9)	2117(4)	53(2)
O(4)	3688(3)	1996(8)	1848(3)	44(2)
O(5)	5348(3)	6373(8)	54(3)	37(2)
O(6)	6163(3)	7916(8)	67(3)	52(2)
O(7)	6323(3)	13722(8)	976(4)	55(2)
O(8)	5695(3)	14662(8)	1642(4)	46(2)
N(1)	4199(3)	6859(10)	1443(4)	35(2)
N(2)	4636(3)	7125(9)	1000(4)	32(2)
N(3)	5669(4)	7657(11)	257(4)	37(2)
N(4)	5898(4)	13512(10)	1292(5)	46(2)
C(1)	3032(4)	-81(13)	1095(6)	50(3)
C(2)	3225(4)	696(13)	1881(5)	43(3)
C(3)	2743(4)	1592(12)	2207(5)	41(3)
C(4)	2356(5)	363(14)	2591(6)	60(3)
C(5)	3100(4)	2912(12)	2710(5)	43(3)
C(6)	3599(4)	3482(12)	2296(5)	40(3)
C(7)	1764(5)	2999(17)	-182(7)	90(4)
C(8)	1132(5)	3218(19)	-606(8)	120(4)
C(9)	2266(6)	4099(18)	-410(7)	105(5)
C(10)	1976(5)	-965(16)	-467(6)	72(3)
C(11)	1337(5)	-1491(16)	-910(6)	88(4)
C(12)	2369(5)	-695(16)	-1043(6)	81(4)
C(13)	1219(6)	-3044(16)	1225(7)	87(4)
C(14)	562(5)	-3306(17)	780(7)	98(4)
C(15)	1382(6)	-4418(17)	1831(7)	106(5)
C(16)	859(6)	657(18)	1865(7)	97(4)
C(17)	342(5)	1301(17)	1258(7)	95(4)
C(18)	586(6)	-300(20)	2466(8)	115(4)
C(19)	3434(4)	5039(11)	1786(5)	40(3)
C(20)	3908(4)	5426(12)	1344(5)	36(2)
C(21)	4971(4)	8612(11)	1098(4)	26(2)
C(22)	5461(4)	8930(11)	730(4)	26(2)
C(23)	5774(4)	10541(13)	818(5)	38(2)
C(24)	5588(4)	11781(12)	1252(5)	31(2)
C(25)	5117(4)	11507(12)	1625(5)	35(2)
C(26)	4824(4)	9953(12)	561(5)	32(2)

Table 14. Bond lengths [Å] and angles [°] for **48**.

Si(1)-O(1)	1.626(7)
Si(1)-O(2)	1.634(7)
Si(1)-C(7)	1.821(13)
Si(1)-C(10)	1.830(12)

Si(2)-O(3)	1.620(7)
Si(2)-O(1)	1.646(7)
Si(2)-C(16)	1.851(13)
Si(2)-C(13)	1.871(12)
O(2)-C(1)	1.400(11)
O(3)-C(4)	1.396(11)
O(4)-C(6)	1.439(10)
O(4)-C(2)	1.448(10)
O(5)-N(3)	1.234(9)
O(6)-N(3)	1.236(8)
O(7)-N(4)	1.214(9)
O(8)-N(4)	1.227(9)
N(1)-C(20)	1.273(1)
N(1)-N(2)	1.399(9)
N(2)-C(21)	1.357(10)
N(2)-H(2N)	0.8800
N(3)-C(22)	1.439(10)
N(4)-C(24)	1.493(11)
C(1)-C(2)	1.542(12)
C(1)-H(1A)	0.9900
C(1)-H(1B)	0.9900
C(2)-C(3)	1.496(11)
C(2)-H(2)	1.0000
C(3)-C(5)	1.493(12)
C(3)-C(4)	1.536(12)
C(3)-H(3)	1.0000
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-C(6)	1.527(11)
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
C(6)-C(19)	1.517(12)
C(6)-H(6)	1.0000
C(7)-C(8)	1.492(9)
C(7)-C(9)	1.525(15)
C(7)-H(7)	1.0000
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(9)-H(9A)	0.9800
C(9)-H(9B)	0.9800
C(9)-H(9C)	0.9800
C(10)-C(12)	1.510(13)
C(10)-C(11)	1.561(15)
C(10)-H(10)	1.0000
C(11)-H(11A)	0.9800

C(11)-H(11B)	0.9800
C(11)-H(11C)	0.9800
C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(12)-H(12C)	0.9800
C(13)-C(15)	1.522(15)
C(13)-C(14)	1.556(16)
C(13)-H(13)	1.0000
C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
C(15)-H(15A)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800
C(16)-C(17)	1.528(16)
C(16)-C(18)	1.543(16)
C(16)-H(16)	1.0000
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800
C(19)-C(20)	1.481(11)
C(19)-H(19A)	0.9900
C(19)-H(19B)	0.9900
C(20)-H(20)	0.9500
C(21)-C(26)	1.410(11)
C(21)-C(22)	1.414(11)
C(22)-C(23)	1.413(12)
C(23)-C(24)	1.355(11)
C(23)-H(23)	0.9500
C(24)-C(25)	1.376(11)
C(25)-C(26)	1.354(11)
C(25)-H(25)	0.9500
C(26)-H(26)	0.9500

O(1)-Si(1)-O(2)	110.8(4)
O(1)-Si(1)-C(7)	108.5(5)
O(2)-Si(1)-C(7)	103.2(5)
O(1)-Si(1)-C(10)	107.3(4)
O(2)-Si(1)-C(10)	108.8(4)
C(7)-Si(1)-C(10)	118.2(6)
O(3)-Si(2)-O(1)	109.2(4)
O(3)-Si(2)-C(16)	110.7(5)
O(1)-Si(2)-C(16)	104.7(5)

O(3)-Si(2)-C(13)	104.4(5)
O(1)-Si(2)-C(13)	107.4(5)
C(16)-Si(2)-C(13)	120.2(6)
Si(1)-O(1)-Si(2)	152.6(5)
C(1)-O(2)-Si(1)	124.9(6)
C(4)-O(3)-Si(2)	130.4(7)
C(6)-O(4)-C(2)	110.0(6)
C(20)-N(1)-N(2)	116.1(7)
C(21)-N(2)-N(1)	118.4(7)
C(21)-N(2)-H(2N)	120.8
N(1)-N(2)-H(2N)	120.8
O(5)-N(3)-O(6)	123.1(8)
O(5)-N(3)-C(22)	119.0(8)
O(6)-N(3)-C(22)	117.9(8)
O(7)-N(4)-O(8)	123.8(8)
O(7)-N(4)-C(24)	119.6(8)
O(8)-N(4)-C(24)	116.5(8)
O(2)-C(1)-C(2)	109.7(8)
O(2)-C(1)-H(1A)	109.7
C(2)-C(1)-H(1A)	109.7
O(2)-C(1)-H(1B)	109.7
C(2)-C(1)-H(1B)	109.7
H(1A)-C(1)-H(1B)	108.2
O(4)-C(2)-C(3)	106.2(7)
O(4)-C(2)-C(1)	107.6(7)
C(3)-C(2)-C(1)	117.0(8)
O(4)-C(2)-H(2)	108.6
C(3)-C(2)-H(2)	108.6
C(1)-C(2)-H(2)	108.6
C(5)-C(3)-C(2)	102.3(7)
C(5)-C(3)-C(4)	114.6(8)
C(2)-C(3)-C(4)	114.4(8)
C(5)-C(3)-H(3)	108.4
C(2)-C(3)-H(3)	108.4
C(4)-C(3)-H(3)	108.4
O(3)-C(4)-C(3)	113.8(8)
O(3)-C(4)-H(4A)	108.8
C(3)-C(4)-H(4A)	108.8
O(3)-C(4)-H(4B)	108.8
C(3)-C(4)-H(4B)	108.8
H(4A)-C(4)-H(4B)	107.7
C(3)-C(5)-C(6)	104.3(7)
C(3)-C(5)-H(5A)	110.9
C(6)-C(5)-H(5A)	110.9
C(3)-C(5)-H(5B)	110.9
C(6)-C(5)-H(5B)	110.9

H(5A)-C(5)-H(5B)	108.9
O(4)-C(6)-C(19)	108.3(7)
O(4)-C(6)-C(5)	104.5(7)
C(19)-C(6)-C(5)	114.4(8)
O(4)-C(6)-H(6)	109.8
C(19)-C(6)-H(6)	109.8
C(5)-C(6)-H(6)	109.8
C(8)-C(7)-C(9)	118.2(11)
C(8)-C(7)-Si(1)	117.7(10)
C(9)-C(7)-Si(1)	115.9(9)
C(8)-C(7)-H(7)	99.6
C(9)-C(7)-H(7)	99.6
Si(1)-C(7)-H(7)	99.6
C(7)-C(8)-H(8A)	109.5
C(7)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(7)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(7)-C(9)-H(9A)	109.5
C(7)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
C(7)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
C(12)-C(10)-C(11)	105.8(9)
C(12)-C(10)-Si(1)	114.6(8)
C(11)-C(10)-Si(1)	115.8(8)
C(12)-C(10)-H(10)	106.7
C(11)-C(10)-H(10)	106.7
Si(1)-C(10)-H(10)	106.7
C(10)-C(11)-H(11A)	109.5
C(10)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
C(10)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
C(10)-C(12)-H(12A)	109.5
C(10)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(10)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(15)-C(13)-C(14)	111.4(11)
C(15)-C(13)-Si(2)	116.0(9)
C(14)-C(13)-Si(2)	114.7(9)

C(15)-C(13)-H(13)	104.4
C(14)-C(13)-H(13)	104.4
Si(2)-C(13)-H(13)	104.4
C(13)-C(14)-H(14A)	109.5
C(13)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(13)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(13)-C(15)-H(15A)	109.5
C(13)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(13)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(17)-C(16)-C(18)	108.8(11)
C(17)-C(16)-Si(2)	113.6(9)
C(18)-C(16)-Si(2)	111.3(10)
C(17)-C(16)-H(16)	107.7
C(18)-C(16)-H(16)	107.7
Si(2)-C(16)-H(16)	107.7
C(16)-C(17)-H(17A)	109.5
C(16)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(16)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(16)-C(18)-H(18A)	109.5
C(16)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(16)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
C(20)-C(19)-C(6)	112.1(8)
C(20)-C(19)-H(19A)	109.2
C(6)-C(19)-H(19A)	109.2
C(20)-C(19)-H(19B)	109.2
C(6)-C(19)-H(19B)	109.2
H(19A)-C(19)-H(19B)	107.9
N(1)-C(20)-C(19)	119.8(8)
N(1)-C(20)-H(20)	120.1
C(19)-C(20)-H(20)	120.1
N(2)-C(21)-C(26)	120.3(8)
N(2)-C(21)-C(22)	122.9(8)
C(26)-C(21)-C(22)	116.8(8)
C(23)-C(22)-C(21)	120.7(8)

C(23)-C(22)-N(3)	116.7(8)
C(21)-C(22)-N(3)	122.6(8)
C(24)-C(23)-C(22)	118.5(8)
C(24)-C(23)-H(23)	120.8
C(22)-C(23)-H(23)	120.8
C(23)-C(24)-C(25)	122.3(8)
C(23)-C(24)-N(4)	117.3(8)
C(25)-C(24)-N(4)	120.4(9)
C(26)-C(25)-C(24)	119.8(8)
C(26)-C(25)-H(25)	120.1
C(24)-C(25)-H(25)	120.1
C(25)-C(26)-C(21)	121.8(8)
C(25)-C(26)-H(26)	119.1
C(21)-C(26)-H(26)	119.1

Symmetry transformations used to generate equivalent atoms:

Table 15. Anisotropic displacement parameters ($Å^2$ x 10^3) for **48**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U11	U22	U33	U23	U13	U12
Si(1)	53(2)	62(2)	60(2)	2(2)	18(2)	0(2)
Si(2)	45(2)	70(2)	67(2)	16(2)	10(2)	-6(2)
O(1)	56(4)	81(4)	61(4)	7(3)	15(3)	-6(3)
O(2)	40(4)	47(4)	56(5)	5(4)	12(4)	2(4)
O(3)	51(4)	52(4)	57(4)	7(3)	13(3)	-12(3)
O(4)	42(4)	27(4)	65(5)	-4(3)	19(4)	1(3)
O(5)	49(4)	25(4)	40(4)	-3(3)	17(3)	-6(3)
O(6)	54(4)	47(4)	62(4)	-12(3)	32(4)	-10(3)
O(7)	58(4)	34(4)	76(4)	3(3)	24(4)	-4(3)
O(8)	57(5)	25(4)	56(5)	-7(4)	11(4)	1(3)
N(1)	43(5)	29(5)	35(5)	-5(4)	14(4)	11(4)
N(2)	37(5)	29(5)	34(5)	0(4)	17(4)	11(4)
N(3)	46(6)	37(6)	32(5)	1(4)	17(4)	2(5)
N(4)	50(6)	26(6)	59(6)	-6(5)	2(5)	4(5)
C(1)	42(7)	38(7)	72(8)	-8(6)	13(6)	11(5)
C(2)	37(6)	36(6)	56(7)	-3(6)	12(5)	-2(5)
C(3)	45(6)	35(6)	44(6)	4(5)	11(5)	-3(5)
C(4)	58(5)	62(5)	63(5)	7(4)	19(4)	-3(4)
C(5)	45(4)	44(4)	44(4)	4(4)	15(4)	2(4)
C(6)	45(4)	37(4)	40(4)	-3(4)	10(4)	5(4)
C(7)	84(5)	89(5)	97(5)	14(4)	14(4)	4(4)
C(8)	106(7)	111(7)	134(7)	22(6)	3(6)	-3(6)
C(9)	103(8)	102(8)	114(8)	24(7)	30(7)	-4(7)

C(10)	61(8)	75(9)	85(9)	-3(7)	23(7)	-9(7)
C(11)	96(8)	96(8)	75(7)	-10(6)	27(6)	-28(6)
C(12)	89(7)	80(7)	81(7)	-20(6)	38(6)	-12(6)
C(13)	85(6)	80(6)	91(6)	8(4)	5(4)	-9(4)
C(14)	82(8)	98(8)	108(8)	4(7)	1(7)	-22(6)
C(15)	128(9)	88(8)	96(8)	9(7)	1(7)	-16(7)
C(16)	97(5)	101(6)	95(5)	12(4)	26(4)	8(4)
C(17)	81(7)	100(8)	106(8)	15(7)	18(7)	12(6)
C(18)	117(7)	125(7)	114(7)	10(6)	46(6)	15(6)
C(19)	57(7)	29(6)	42(6)	-8(5)	31(6)	-2(5)
C(20)	56(7)	22(6)	34(6)	-7(5)	21(5)	-4(5)
C(21)	31(5)	21(6)	24(5)	6(5)	3(5)	1(5)
C(22)	35(5)	27(6)	20(5)	1(4)	16(4)	6(5)
C(23)	30(6)	45(7)	37(6)	5(5)	3(5)	-2(5)
C(24)	32(6)	22(6)	37(6)	1(5)	0(5)	-1(5)
C(25)	35(6)	29(6)	44(6)	-11(5)	11(5)	10(5)
C(26)	37(6)	26(6)	39(6)	-5(5)	18(5)	4(5)

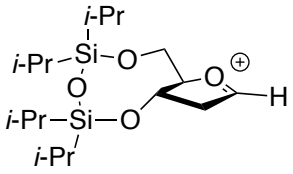
Table 16. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **48**.

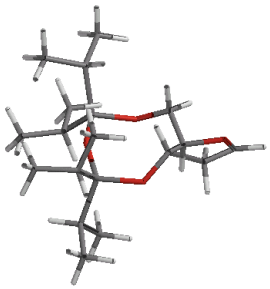
	x	y	z	U(eq)
H(2N)	4692	6347	666	38
H(1A)	3398	-400	893	60
H(1B)	2790	-1152	1119	60
H(2)	3406	-255	2227	51
H(3)	2467	2225	1799	49
H(4A)	2625	-236	3007	72
H(4B)	2061	1069	2805	72
H(5A)	3276	2387	3197	52
H(5B)	2841	3914	2791	52
H(6)	3981	3733	2661	49
H(7)	1725	3612	290	109
H(8A)	1067	4438	-761	179
H(8B)	843	2895	-289	179
H(8C)	1071	2464	-1047	179
H(9A)	2435	3479	-793	157
H(9B)	2587	4306	24	157
H(9C)	2097	5220	-610	157
H(10)	2142	-2009	-168	87
H(11A)	1185	-576	-1272	131
H(11B)	1055	-1633	-564	131
H(11C)	1368	-2594	-1171	131
H(12A)	2390	-1785	-1318	121
H(12B)	2778	-349	-796	121

H(12C)	2192	225	-1390	121
H(13)	1485	-3323	859	104
H(14A)	544	-4375	482	147
H(14B)	447	-2304	451	147
H(14C)	280	-3406	1128	147
H(15A)	1149	-4206	2225	159
H(15B)	1817	-4352	2040	159
H(15C)	1284	-5580	1618	159
H(16)	1081	1706	2102	116
H(17A)	61	335	1095	143
H(17B)	508	1735	835	143
H(17C)	123	2245	1456	143
H(18A)	165	83	2441	173
H(18B)	825	-28	2959	173
H(18C)	593	-1562	2381	173
H(19A)	3045	4801	1443	48
H(19B)	3375	6075	2088	48
H(20)	3994	4606	989	43
H(23)	6108	10749	577	45
H(25)	4999	12404	1927	42
H(26)	4510	9762	1836	39

III. Details of Computation

Low-energy conformations of the oxocarbenium ions of interest were found using the conformational search package on Spartan 10 applying semi-empirical methods (PM3). A sparse systematic was performed with three-fold rotational freedom for all endocyclic bonds and six-fold rotational freedom for exocyclic bonds. Spartan 10 keeps 100 conformers while searching for the lowest energy ones. Duplicates were removed and the remaining low energy conformers were subjected to energy optimization at higher levels of theory, beginning with the Hartree–Fock method (HF/3-21G). Resulting conformers were further optimized at the HF/6-31G* level of theory and with density functional theory (B3LYP/6-31G*).





49-equatorial

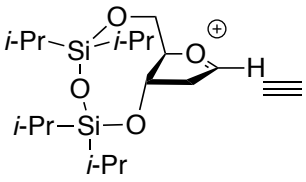
E_{rel} = 0 kcal/mol

Energy = -1548.81245 a.u.

Cartesian Coordinates (Angstroms)

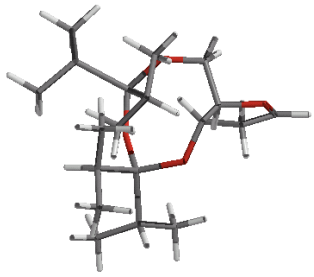
ATOM	X	Y	Z
C	-1.88827	1.494464	-0.281843
H	-1.366777	1.432022	-1.241831
C	-3.215077	2.26456	-0.46591
H	-4.015696	1.846106	0.167911
H	-3.618738	2.296144	-1.483846
C	-1.075132	2.396966	0.684152
H	-1.181302	2.072256	1.721053
C	-2.910046	3.60287	0.032536
H	-3.529088	4.49804	-0.031648
O	-1.798692	3.720282	0.62755
C	0.391097	2.685551	0.363992
H	0.526772	2.838333	-0.713703
H	0.692461	3.604389	0.875801
O	1.189935	1.62671	0.864235
O	-2.115019	0.213281	0.264735
Si	1.701208	0.226892	0.101629
Si	-1.210412	-1.172002	-0.041031
O	0.370708	-0.711263	-0.234252
C	2.758644	-0.611243	1.437457
H	2.069927	-0.669158	2.295304

C	2.502106	0.656767	-1.570212
H	1.669326	1.04845	-2.17628
C	-1.474235	-2.274469	1.481591
H	-0.74123	-3.090995	1.390298
C	-1.790412	-2.013685	-1.640486
H	-2.840727	-2.286649	-1.456367
C	3.564443	1.771041	-1.487413
H	3.881692	2.082522	-2.491806
H	4.465732	1.438436	-0.958771
H	3.198996	2.663483	-0.968279
C	3.046104	-0.572222	-2.325375
H	2.302595	-1.37175	-2.413524
H	3.92876	-0.99833	-1.832454
H	3.354211	-0.300947	-3.344206
C	3.188957	-2.052377	1.10403
H	3.911456	-2.087494	0.279788
H	2.342374	-2.686962	0.821265
H	3.672938	-2.524987	1.969376
C	3.959935	0.241285	1.88935
H	3.663915	1.264112	2.143272
H	4.735738	0.301556	1.115994
H	4.43503	-0.193469	2.779331
C	-1.198192	-1.536829	2.805039
H	-0.185565	-1.12081	2.847394
H	-1.304231	-2.214314	3.663359
H	-1.905673	-0.713572	2.953456
C	-2.88074	-2.907204	1.502792
H	-3.082591	-3.518371	0.616198
H	-3.666058	-2.144024	1.563841
H	-3.002165	-3.560048	2.377828
C	-1.765953	-1.097108	-2.878372
H	-2.118351	-1.633343	-3.770316
H	-0.755271	-0.736146	-3.102314
H	-2.420756	-0.226996	-2.758641
C	-1.013044	-3.31779	-1.909639
H	0.050618	-3.124322	-2.094191
H	-1.405517	-3.831515	-2.797337
H	-1.074768	-4.026445	-1.075174



49-axial

E_{rel} = +0.27 kcal/mol

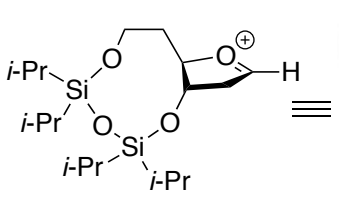


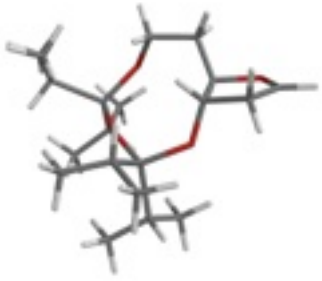
Energy = -1548.81200 a.u.

Cartesian Coordinates (Angstroms)

ATOM	X	Y	Z
C	-1.69201	1.545547	-0.552137
H	-1.303907	1.289931	-1.540605
C	-2.86922	2.537161	-0.662952
H	-3.818147	2.125148	-0.293799
H	-3.076121	2.886675	-1.685845
C	-0.62014	2.327226	0.244097
H	-0.403228	1.849597	1.200074
C	-2.458603	3.681308	0.145392
H	-3.033296	4.58017	0.369489
O	-1.291902	3.614522	0.634135
C	0.657157	2.701313	-0.511708
H	0.410814	3.328634	-1.374412
H	1.319593	3.274796	0.145635
O	1.296115	1.536197	-1.006793
O	-2.105396	0.386881	0.152204
Si	1.747281	0.138305	-0.199924
Si	-1.258093	-1.067834	0.203414
O	0.360314	-0.706829	0.145597
C	2.614288	0.604273	1.42753
H	1.872948	1.166522	2.017849
C	2.856759	-0.828449	-1.40158
H	3.883931	-0.526605	-1.142482
C	-1.709733	-1.840177	1.871209
H	-2.791964	-2.034724	1.819373
C	-1.74838	-2.15199	-1.278776
H	-1.007029	-2.965574	-1.316721
C	2.757968	-2.352001	-1.18731
H	3.473807	-2.884999	-1.827523
H	1.758846	-2.727216	-1.435957
H	2.966052	-2.650453	-0.15333
C	2.636633	-0.462132	-2.881193

H	2.750278	0.611263	-3.056906
H	1.635933	-0.746955	-3.228595
H	3.357665	-0.98612	-3.523265
C	3.032121	-0.61304	2.275553
H	3.806423	-1.20854	1.77502
H	2.192664	-1.28199	2.493795
H	3.453773	-0.294234	3.238384
C	3.817153	1.541288	1.192673
H	4.630255	1.03214	0.660006
H	4.23334	1.891097	2.146729
H	3.55387	2.427346	0.605303
C	-1.00588	-3.191989	2.103023
H	-1.221264	-3.921354	1.313177
H	-1.325872	-3.641831	3.052112
H	0.083984	-3.079892	2.152403
C	-1.472294	-0.879246	3.050671
H	-2.024273	0.057683	2.924171
H	-0.409313	-0.634532	3.166966
H	-1.80168	-1.327457	3.99807
C	-3.140874	-2.788177	-1.091798
H	-3.411304	-3.401608	-1.961679
H	-3.922653	-2.026274	-0.98293
H	-3.192547	-3.437045	-0.210754
C	-1.685652	-1.396172	-2.620279
H	-0.706685	-0.938799	-2.801605
H	-2.444415	-0.60612	-2.665661
H	-1.885072	-2.07223	-3.46289





50-axial

E_{rel} = 0 kcal/mol

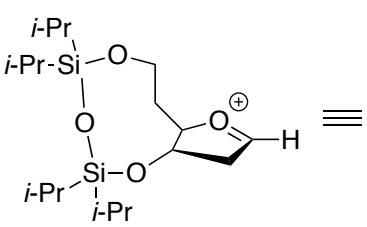
Energy = -1588.13364 a.u.

Cartesian Coordinates (Angstroms)

ATOM	X	Y	Z
C	-2.255818	1.134369	-0.450881
H	-1.868952	1.002229	-1.464217
C	-3.732259	1.580961	-0.4854

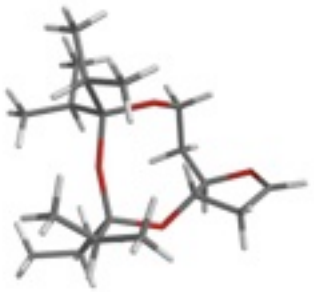
H	-4.43943	0.794504	-0.198671
H	-4.061677	1.941857	-1.47295
C	-1.530235	2.302611	0.257063
H	-0.982595	1.963701	1.134289
C	-3.781357	2.712389	0.439697
H	-4.663129	3.244331	0.796954
O	-2.659952	3.123216	0.851895
C	-0.695623	3.242387	-0.596701
H	-1.2858	3.633232	-1.43363
H	-0.377965	4.090465	0.017173
C	0.532891	2.528204	-1.167662
H	1.158558	3.263596	-1.684064
H	0.232036	1.78008	-1.909654
O	1.297762	1.93172	-0.115162
O	-2.134913	-0.05656	0.309324
Si	1.805192	0.345654	-0.026409
Si	-1.033464	-1.299454	0.038555
O	0.478358	-0.650335	-0.162279
C	2.553195	0.170299	1.71248
H	1.677284	0.031912	2.36586
C	2.999242	-0.10903	-1.438018
H	3.269322	-1.160171	-1.248697
C	-1.202843	-2.368973	1.598687
H	-2.195603	-2.836624	1.501044
C	-1.507854	-2.168327	-1.585734
H	-1.528432	-1.364455	-2.340069
C	3.432079	-1.087278	1.860661
H	4.350917	-1.017185	1.265529
H	2.914533	-2.002583	1.550444
H	3.74068	-1.231849	2.904672
C	3.286029	1.428744	2.215469
H	4.184514	1.652433	1.627223
H	3.611488	1.300883	3.257349
H	2.643835	2.313226	2.173619
C	4.294114	0.725027	-1.396558
H	4.983101	0.423315	-2.197277
H	4.836283	0.619762	-0.450362
H	4.090365	1.793937	-1.535609
C	2.366754	-0.04314	-2.841495
H	3.046125	-0.458178	-3.598899
H	2.160964	0.991194	-3.140027
H	1.429721	-0.606455	-2.902418
C	-2.92132	-2.781194	-1.531142

H	-3.224395	-3.163854	-2.514932
H	-3.678347	-2.055049	-1.216861
H	-2.971262	-3.625561	-0.831737
C	-0.476397	-3.203861	-2.074672
H	-0.723289	-3.557373	-3.084801
H	-0.4525	-4.09026	-1.429047
H	0.539701	-2.797935	-2.108408
C	-1.220294	-1.535325	2.894363
H	-0.253425	-1.049639	3.078719
H	-1.429015	-2.169428	3.767038
H	-1.983989	-0.753118	2.863486
C	-0.166299	-3.504047	1.686063
H	0.856995	-3.116485	1.75254
H	-0.203694	-4.177831	0.823601
H	-0.333993	-4.118786	2.580643



50-equatorial

E_{rel} = + 5.16 kcal/mol



Energy = -1588.12540 a.u.

Cartesian Coordinates (Angstroms)

ATOM	X	Y	Z
C	-2.190252	0.866384	-0.184157
H	-1.358161	1.052729	-0.866964
C	-3.512001	1.273715	-0.870813
H	-4.373387	0.754142	-0.414755
H	-3.576236	1.107629	-1.949605
C	-2.117021	1.856038	0.99939
H	-2.766524	1.505659	1.806094
C	-3.618305	2.690702	-0.512807
H	-4.257573	3.451632	-0.961541
O	-2.875411	3.058759	0.444646
C	-0.787888	2.320855	1.55653
H	-0.988926	3.085881	2.315528
H	-0.366495	1.466455	2.099539
C	0.244543	2.891828	0.579905

H	1.057447	3.331397	1.167789
H	-0.193062	3.705486	-0.007935
O	0.77608	1.959618	-0.36283
O	-2.194092	-0.460178	0.287714
Si	1.593603	0.520111	-0.197763
Si	-0.923598	-1.559579	0.177392
O	0.474991	-0.695612	0.022097
C	2.714244	0.498766	1.340051
H	2.008489	0.52302	2.186627
C	2.434747	0.230963	-1.875998
H	2.674931	-0.842906	-1.902343
C	-0.990968	-2.538047	1.802411
H	-2.012526	-2.950047	1.831143
C	-1.199423	-2.693746	-1.331544
H	-1.80174	-3.533931	-0.950073
C	3.504271	-0.820536	1.459539
H	4.24879	-0.925094	0.659892
H	2.853706	-1.700619	1.416972
H	4.052295	-0.866522	2.410327
C	3.652986	1.710838	1.498784
H	4.428181	1.738303	0.724634
H	4.171621	1.674386	2.466474
H	3.122764	2.66748	1.454801
C	3.750172	1.009176	-2.074339
H	4.165463	0.817007	-3.073086
H	4.522125	0.728321	-1.349413
H	3.599132	2.092682	-1.996701
C	1.469149	0.525892	-3.041244
H	1.935221	0.292409	-4.00859
H	1.184711	1.583537	-3.058542
H	0.550029	-0.066318	-2.977019
C	0.110587	-3.282045	-1.891503
H	-0.097669	-4.015758	-2.682146
H	0.709302	-3.791723	-1.128863
H	0.743683	-2.503884	-2.332133
C	-2.017073	-2.02252	-2.452864
H	-2.150343	-2.702551	-3.305397
H	-1.516216	-1.126716	-2.840821
H	-3.01587	-1.733975	-2.110394
C	-0.818833	-1.647788	3.046257
H	0.171125	-1.176115	3.07294
H	-0.919489	-2.233784	3.970172
H	-1.574824	-0.856831	3.083848

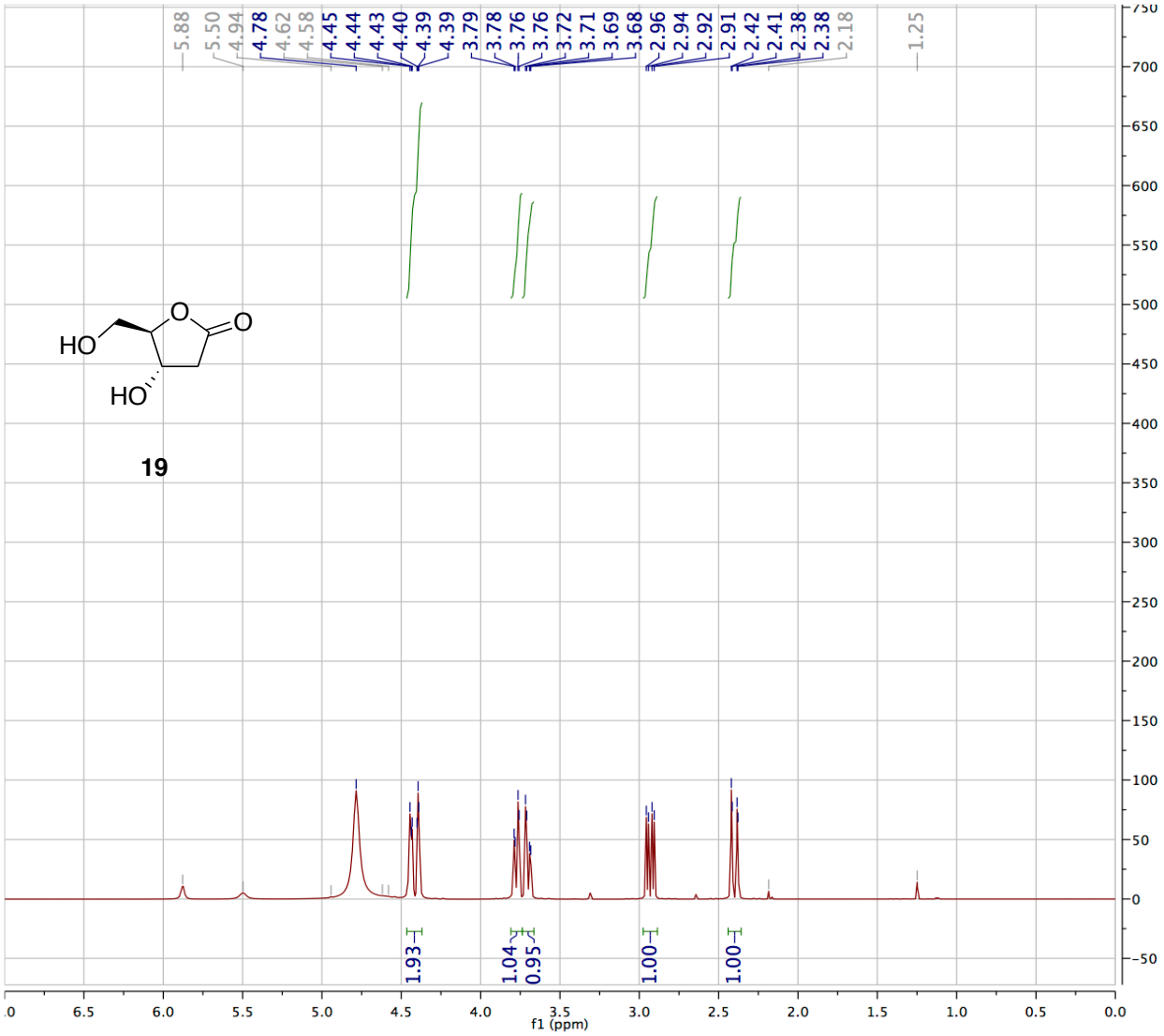
C	-0.017408	-3.731146	1.828497
H	1.029933	-3.407806	1.784081
H	-0.184315	-4.42168	0.994905
H	-0.131605	-4.312232	2.753746

IV. References

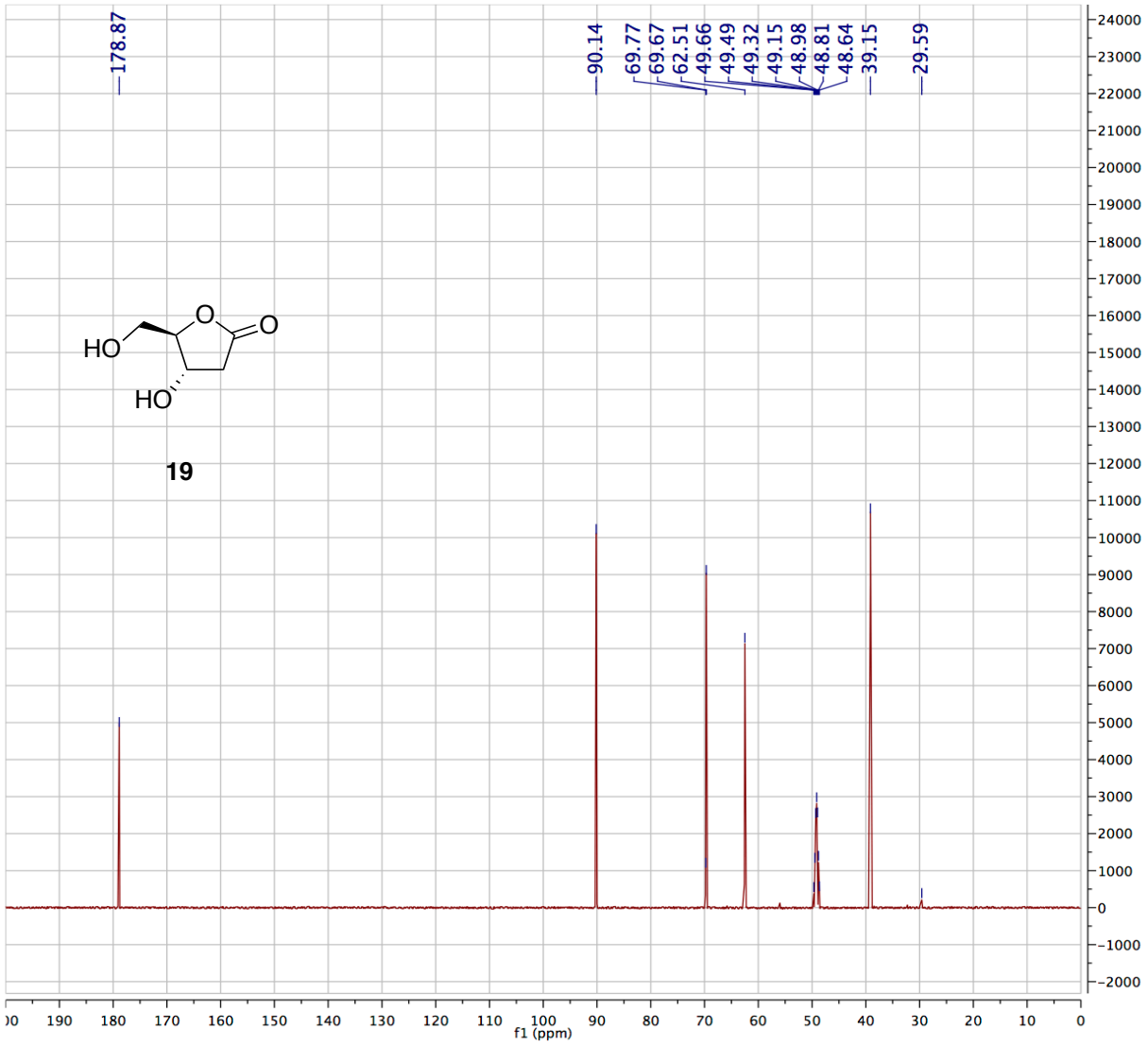
(1) Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996,** *15*, 1518–1520.

V. 1H and 13C NMR Spectra

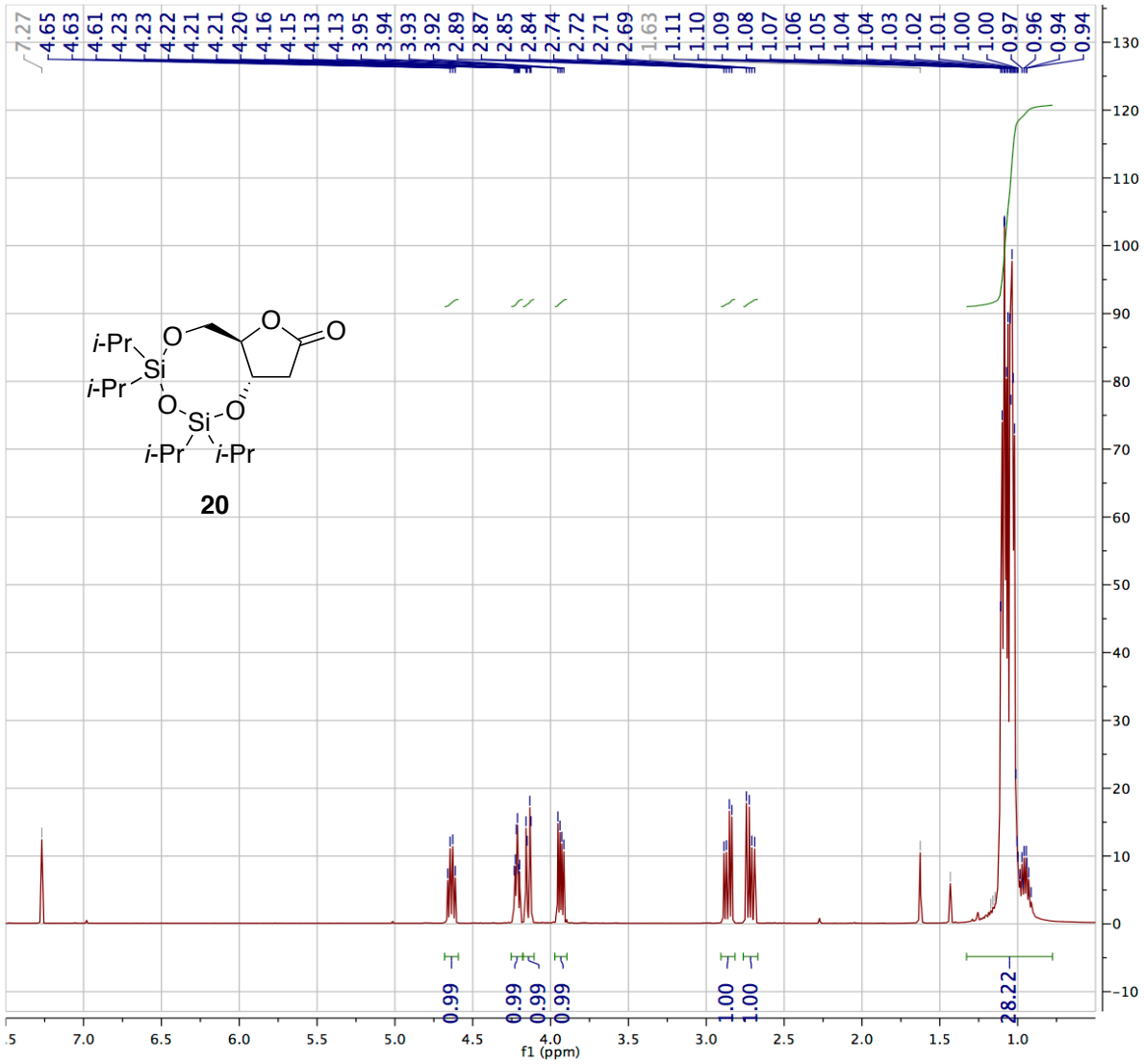
Note: There are no peaks beyond the window provided.



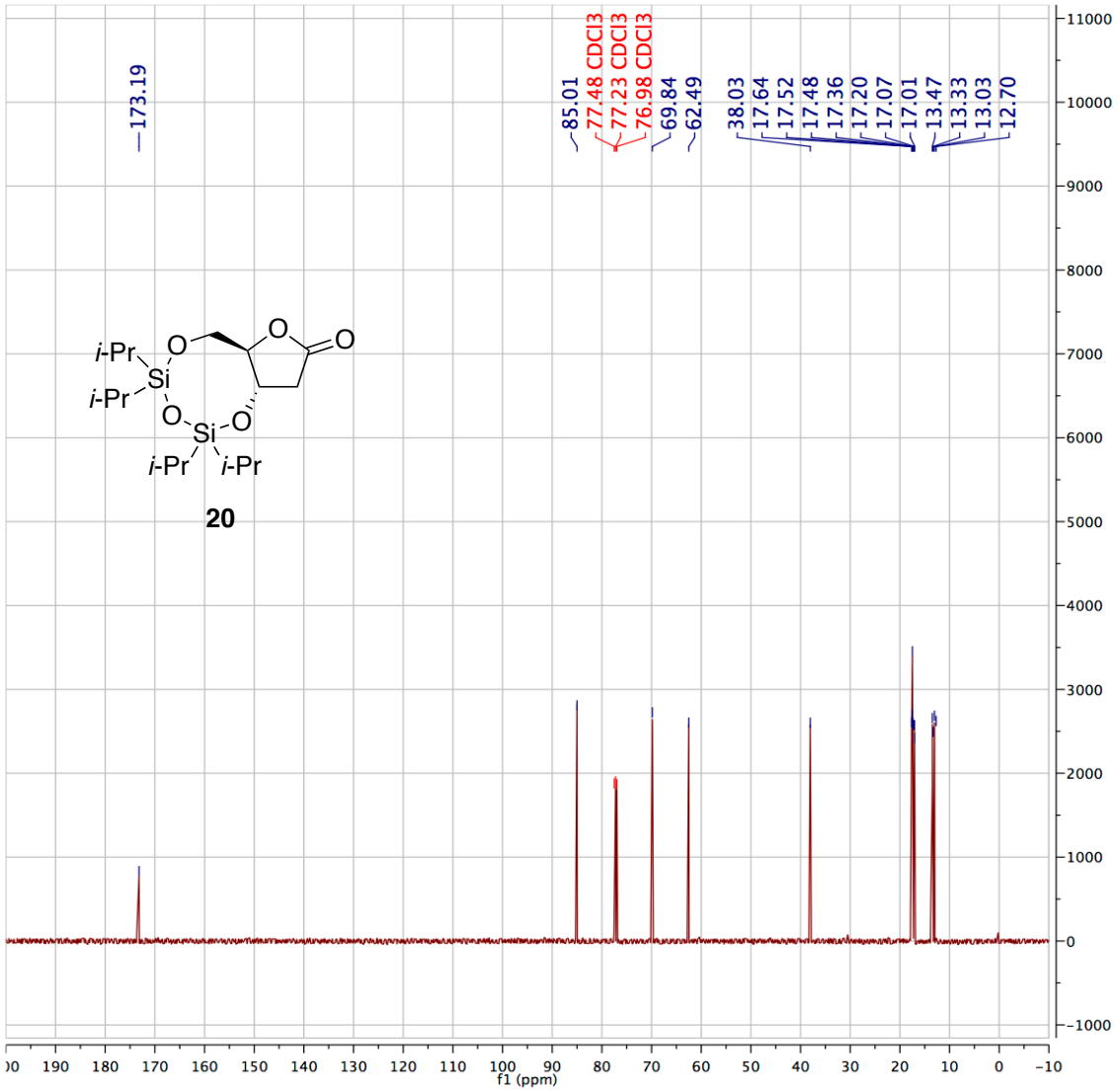
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	8
7	Receiver Gain	4
8	Relaxation Delay	0.1000
9	Pulse Width	7.5000
10	Spectrometer Frequency	500.22
11	Spectral Width	8012.8
12	Lowest Frequency	-523.8
13	Nucleus	1H
14	Acquired Size	40864
15	Spectral Size	131072



	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	SpinEchopg30gp.prd
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	84
7	Receiver Gain	7298
8	Relaxation Delay	0.2500
9	Pulse Width	14.8500
10	Spectrometer Frequency	125.79
11	Spectral Width	30303.0
12	Lowest Frequency	-1147.8
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



	Parameter	Value
1	Solvent	CD3OD
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	8
7	Receiver Gain	4
8	Relaxation Delay	0.1000
9	Pulse Width	7.5000
10	Spectrometer Frequency	500.22
11	Spectral Width	8012.8
12	Lowest Frequency	-532.1
13	Nucleus	1H
14	Acquired Size	40864
15	Spectral Size	131072



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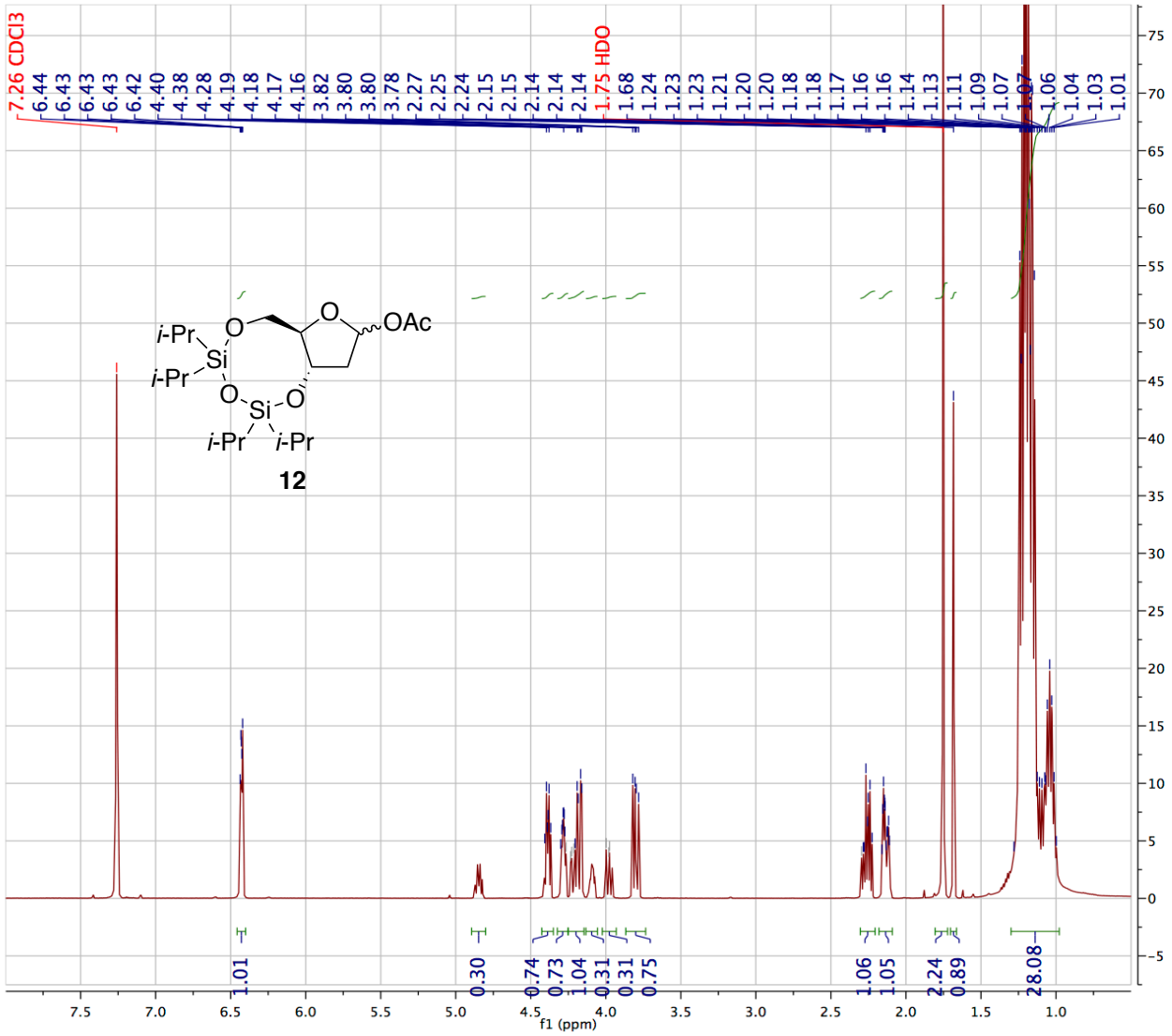
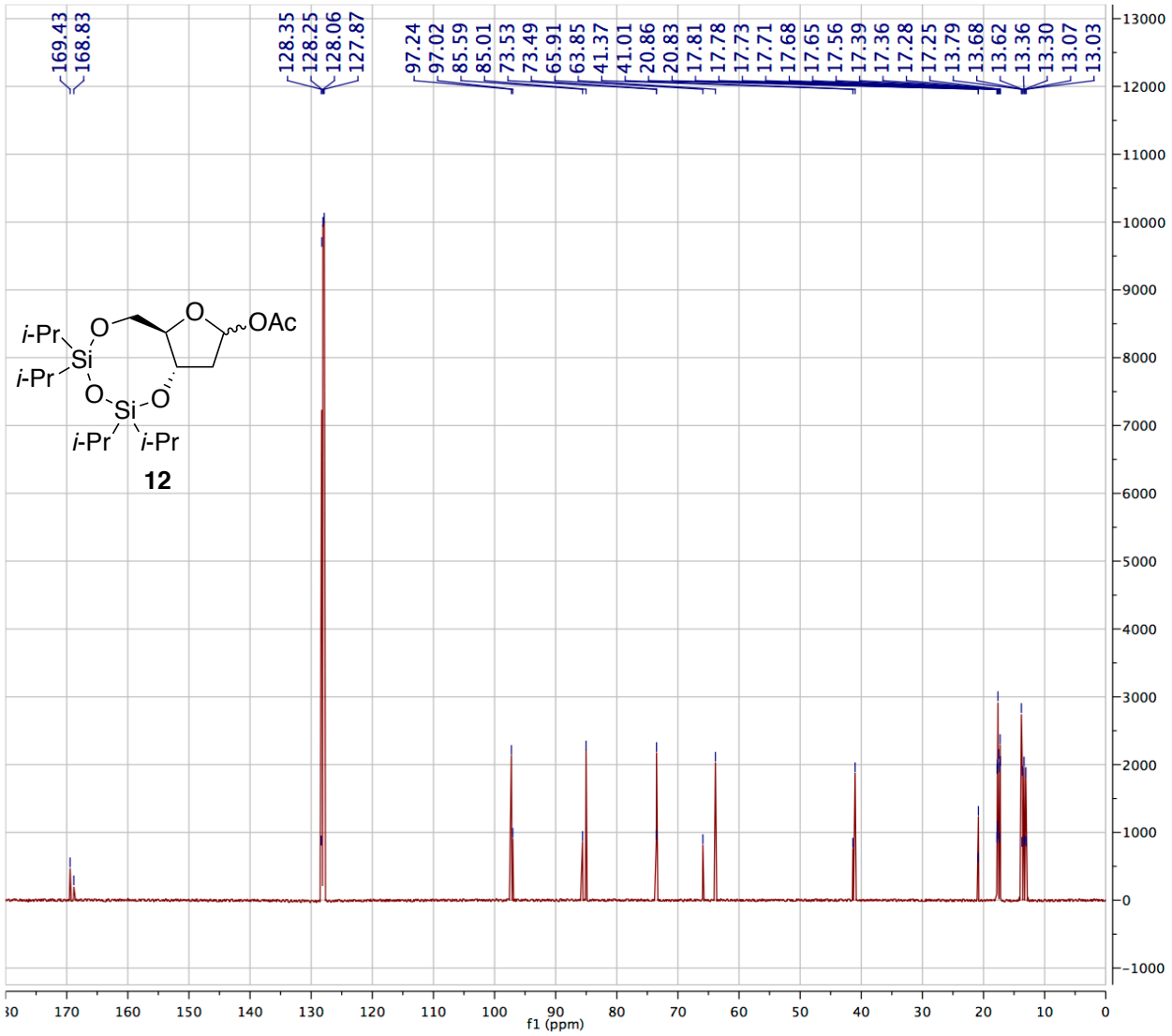
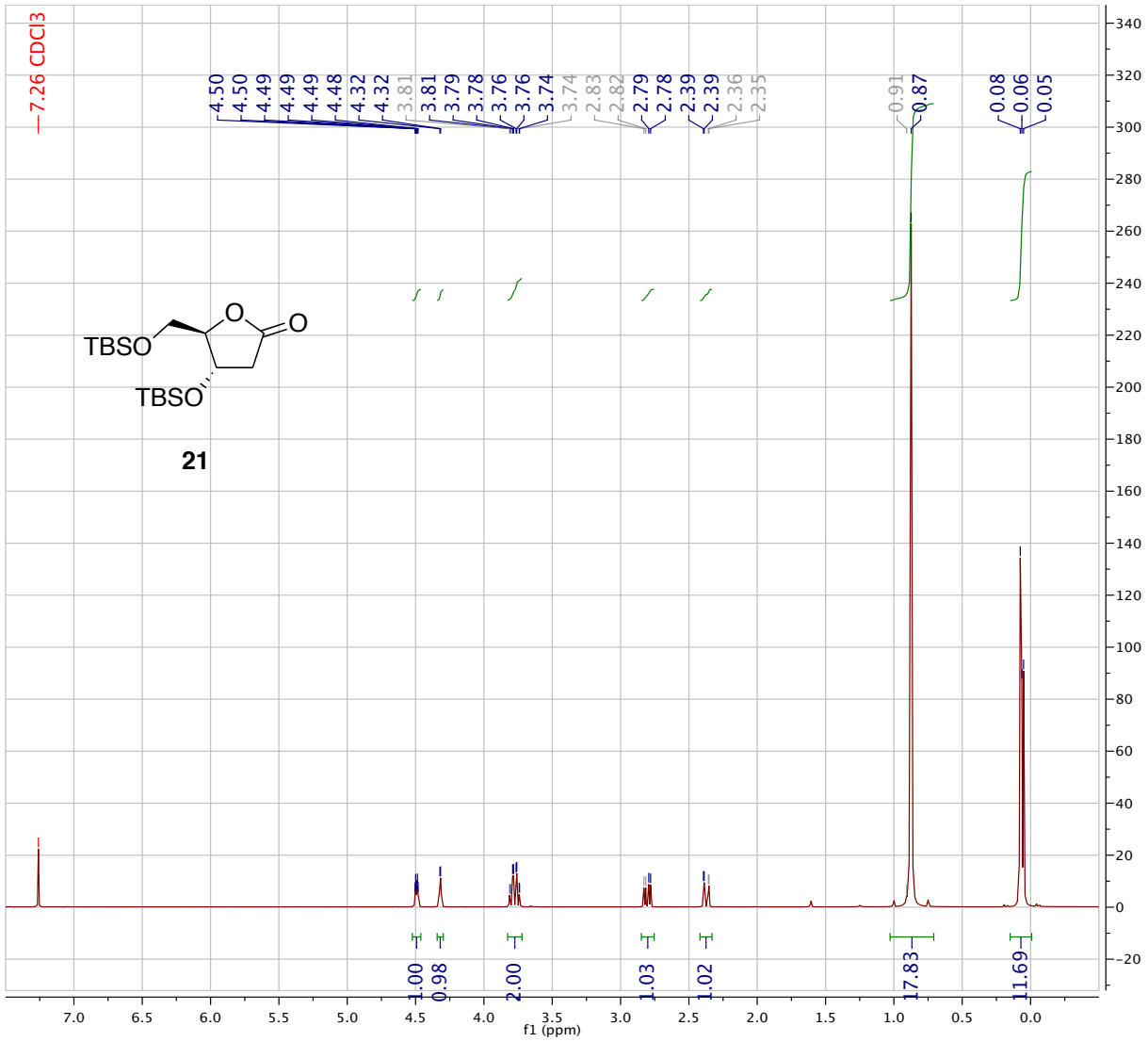
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	SpinEchopg30gp.prd
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	112
7	Receiver Gain	7298
8	Relaxation Delay	0.2500
9	Pulse Width	15.5000
10	Spectrometer Frequency	125.79
11	Spectral Width	30303.0
12	Lowest Frequency	-1295.7
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



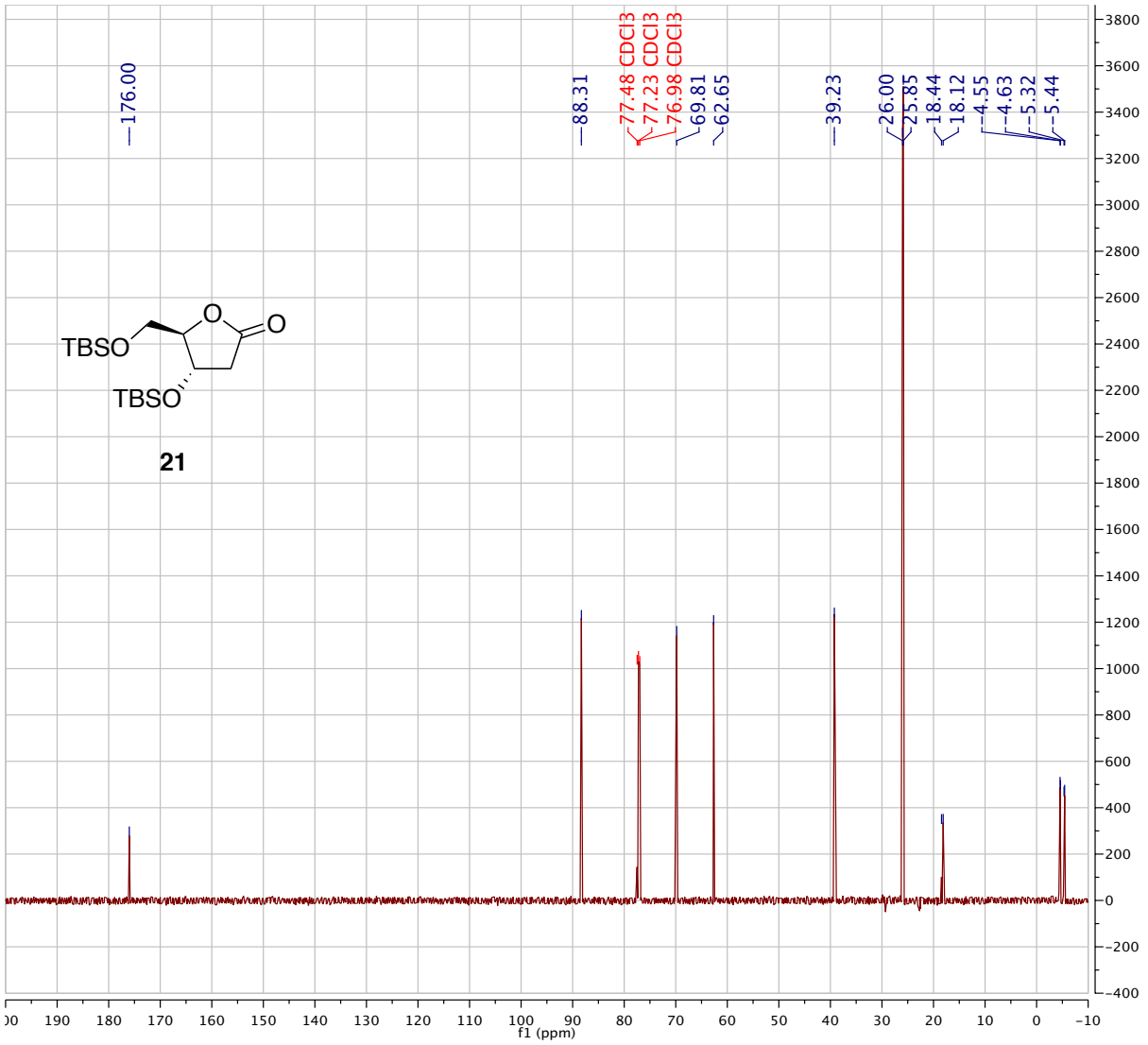
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	8
7	Receiver Gain	2
8	Relaxation Delay	0.1000
9	Pulse Width	7.5000
10	Spectrometer Frequency	500.22
11	Spectral Width	8012.8
12	Lowest Frequency	-455.0
13	Nucleus	1H
14	Acquired Size	40864
15	Spectral Size	131072



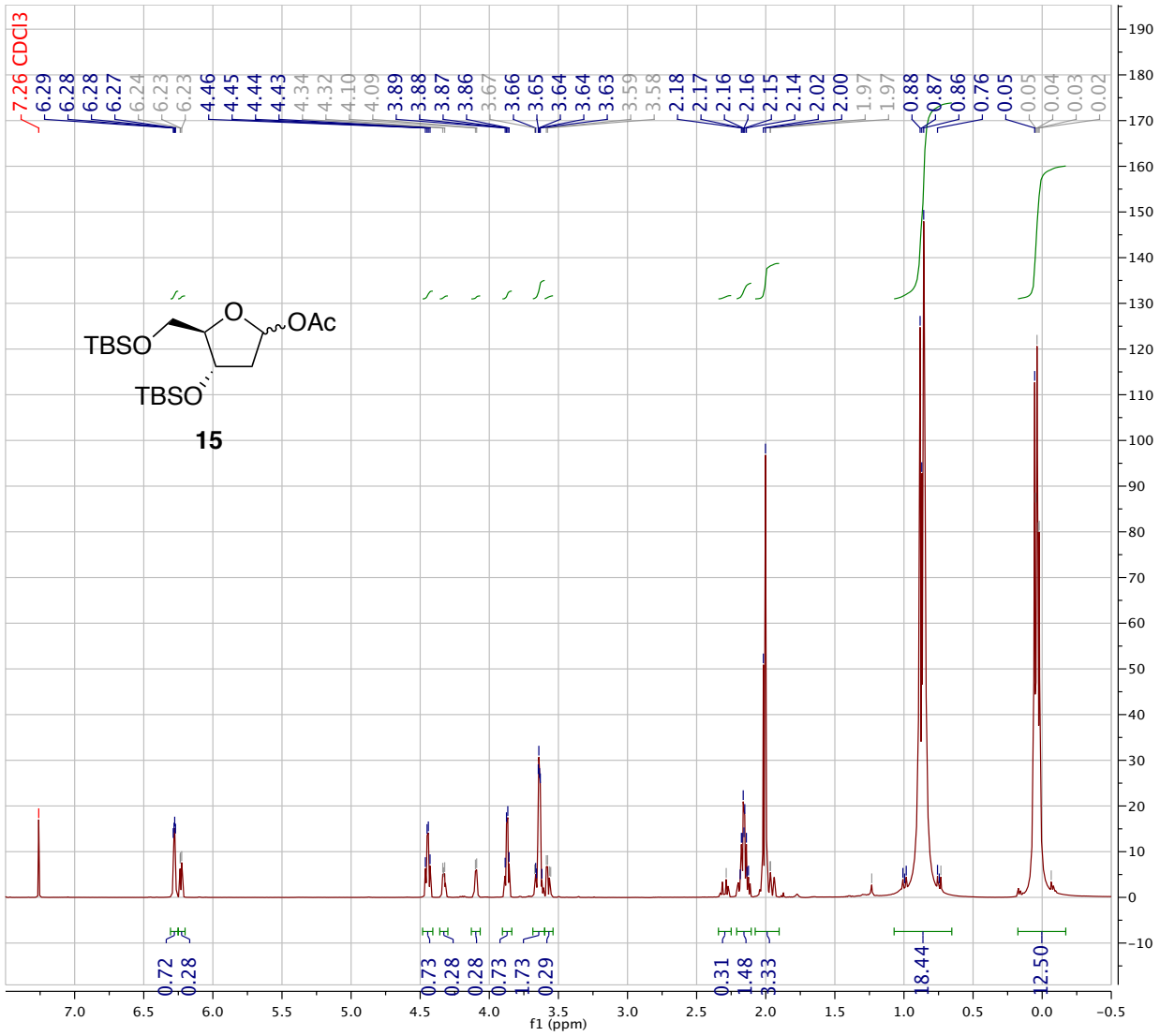
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	SpinEchopg30gp.prd
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	122
7	Receiver Gain	4598
8	Relaxation Delay	0.2500
9	Pulse Width	15.5000
10	Spectrometer Frequency	125.79
11	Spectral Width	30303.0
12	Lowest Frequency	-1267.2
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



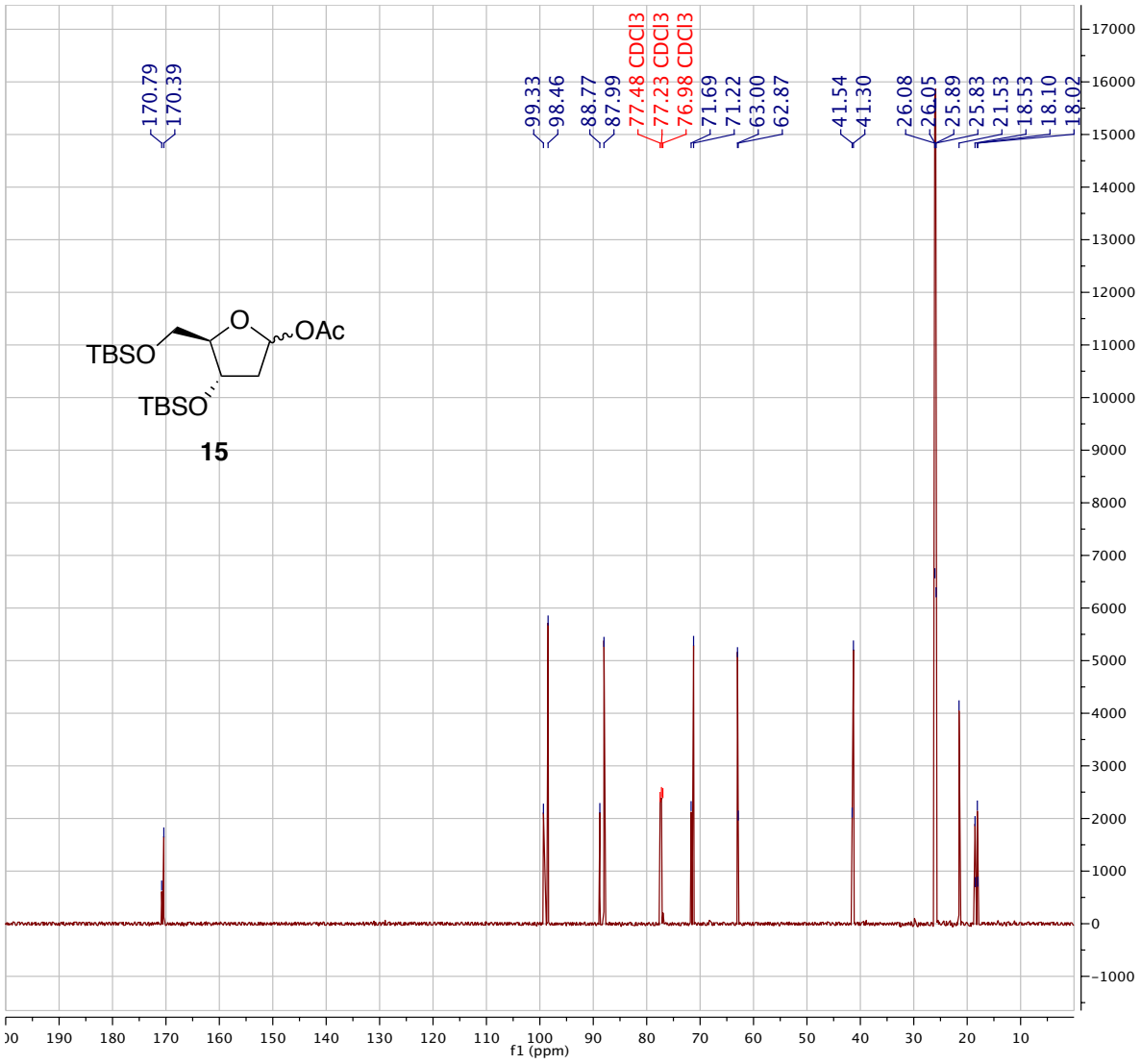
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	8
7	Receiver Gain	6
8	Relaxation Delay	0.1000
9	Pulse Width	7.5000
10	Spectrometer Frequency	500.22
11	Spectral Width	8012.8
12	Lowest Frequency	-536.6
13	Nucleus	1H
14	Acquired Size	40864
15	Spectral Size	131072



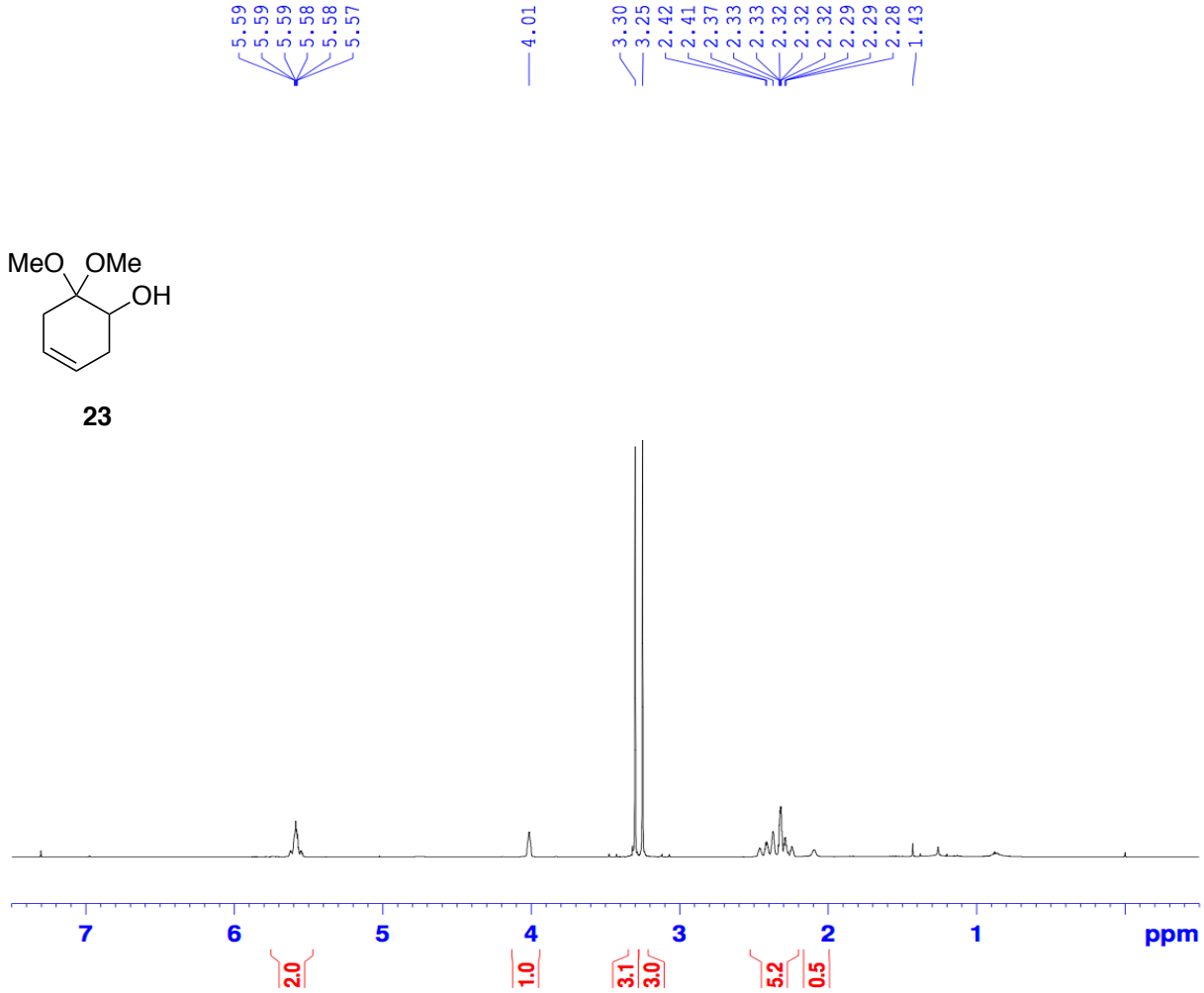
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	SpinEchopg30gp.prd
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	159
7	Receiver Gain	3251
8	Relaxation Delay	0.2500
9	Pulse Width	15.5000
10	Spectrometer Frequency	125.79
11	Spectral Width	30303.0
12	Lowest Frequency	-1295.2
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	8
7	Receiver Gain	2
8	Relaxation Delay	0.1000
9	Pulse Width	7.5000
10	Spectrometer Frequency	500.22
11	Spectral Width	8012.8
12	Lowest Frequency	-536.2
13	Nucleus	1H
14	Acquired Size	40864
15	Spectral Size	131072



	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	SpinEchopg30gp.prd
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	114
7	Receiver Gain	7298
8	Relaxation Delay	0.2500
9	Pulse Width	15.5000
10	Spectrometer Frequency	125.79
11	Spectral Width	30303.0
12	Lowest Frequency	-1298.1
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



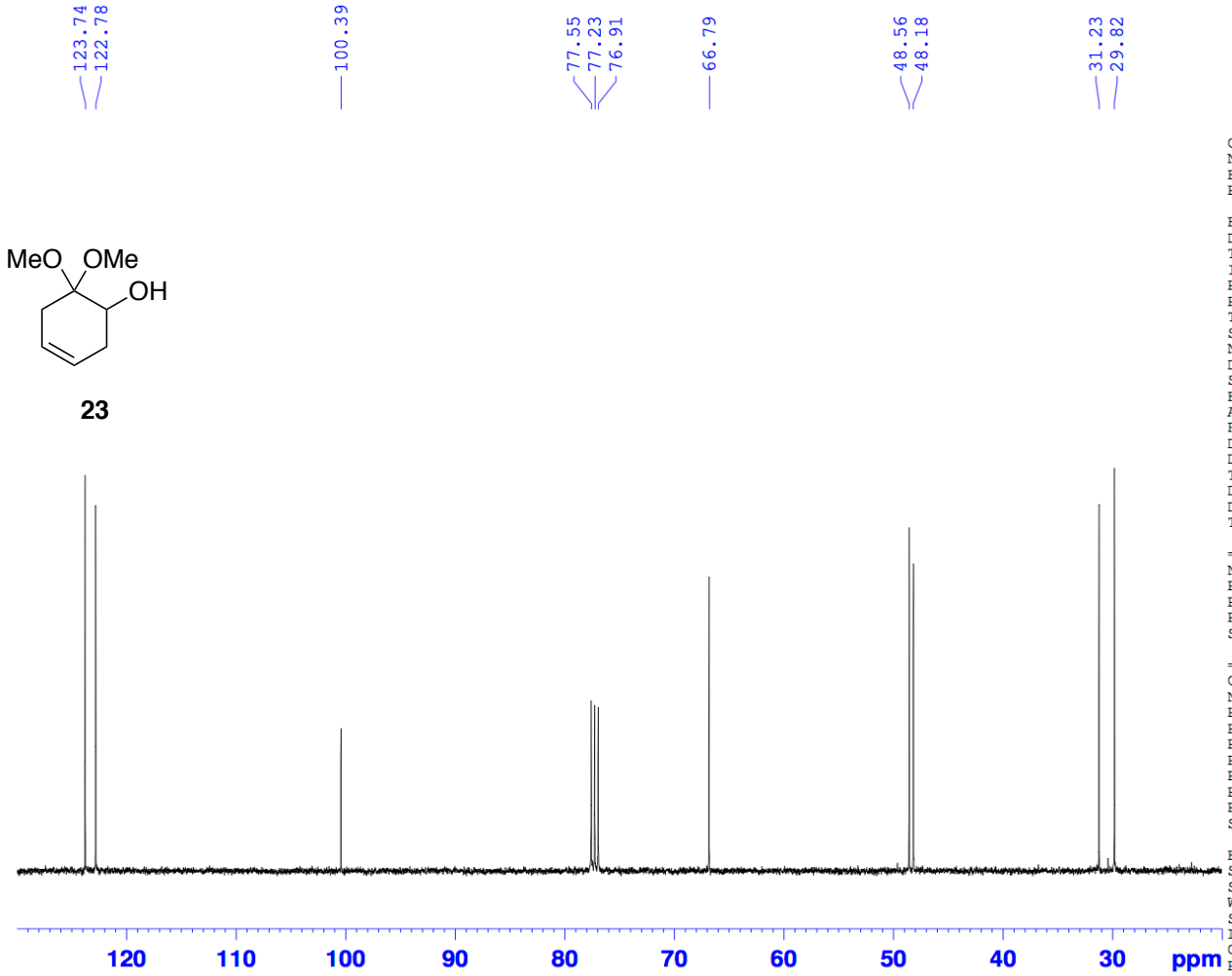
23

```
Current Data Parameters
NAME        VTT-III-78-A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120322
Time                0.15
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  35.9
DW                60.400 usec
DE                 6.50 usec
TE                298.1 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.75 usec
PL1               -5.00 dB
PL1W         31.77312851 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1299929 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



23

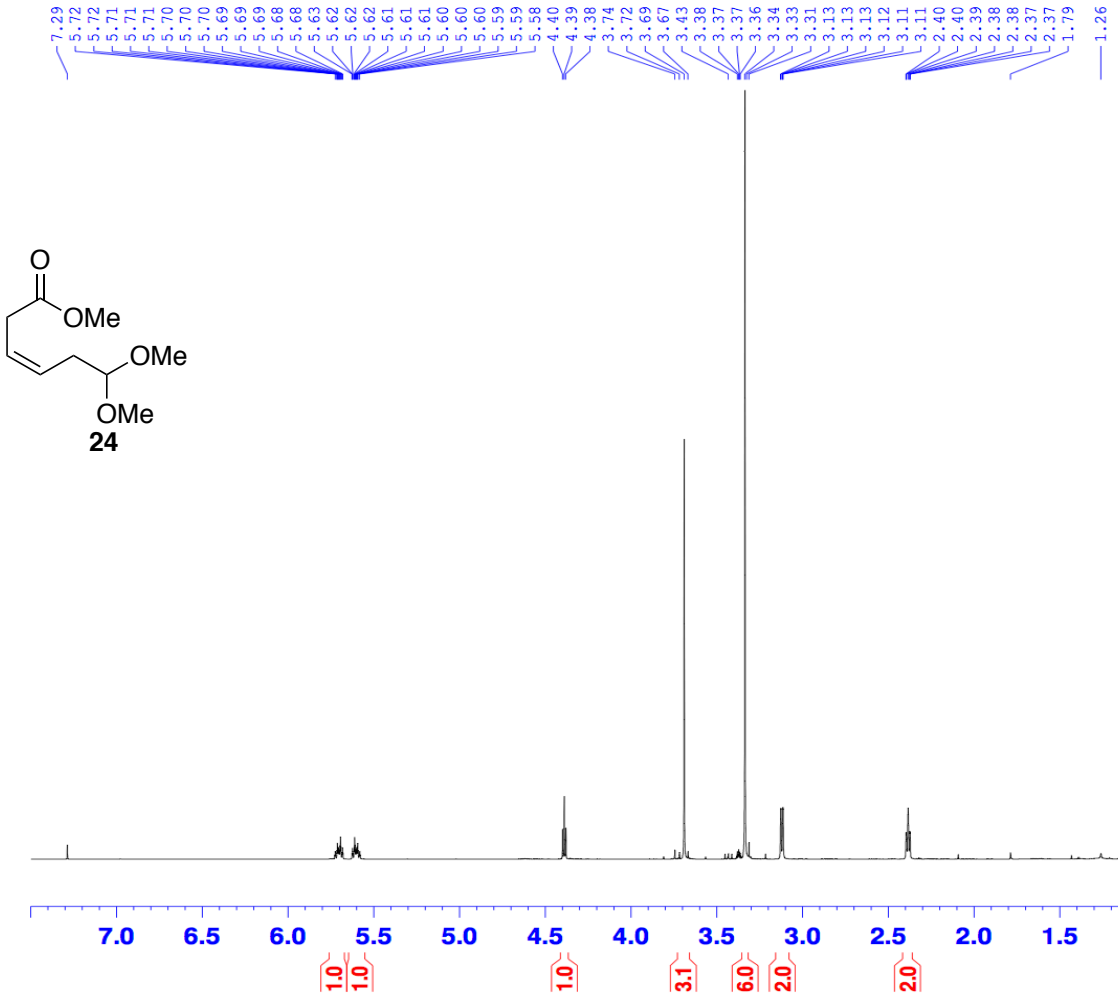
```
Current Data Parameters
NAME        VTT-III-78-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120322
Time                0.20
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   79
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG                32768
DW               20.850 usec
DE                6.50 usec
TE                298.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  6.60 usec
PL1                -4.00 dB
PL1W        25.23829460 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              70.00 usec
PL2                -5.00 dB
PL12               13.84 dB
PL13               13.84 dB
PL2W        31.77312851 W
PL12W        0.41501135 W
PL13W        0.41501135 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127555 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



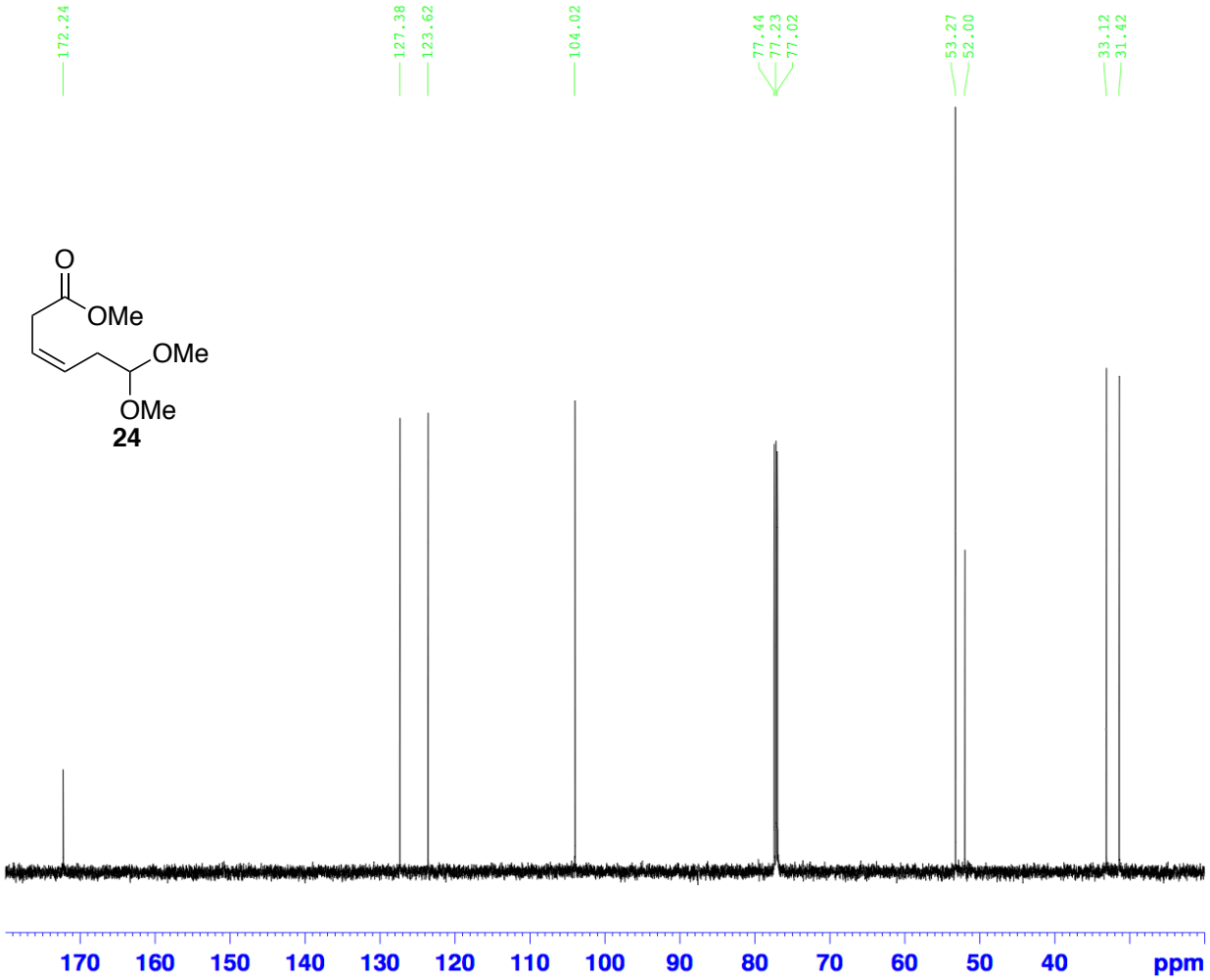
BRUKER

```
Current Data Parameters
NAME        VTT-III-79-A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120324
Time               21.49
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6564426 sec
RG                33.59
DW               40.533 usec
DE                6.50 usec
TE               298.1 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.79 usec
PLW1         9.30000019 W
SFO1        600.1937064 MHz

F2 - Processing parameters
SI                65536
SF          600.1899985 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



BRUKER

```
Current Data Parameters
NAME        VTT-III-79-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120324
Time               21.54
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   89
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG               184.65
DW               13.867 usec
DE                6.50 usec
TE                298.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.00 usec
PLW1       106.00000000 W
SFO1        150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PLW2         9.30000019 W
PLW12        0.18190999 W
PLW13        0.08913500 W
SFO2        600.1924008 MHz

F2 - Processing parameters
SI                32768
SF          150.9178689 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
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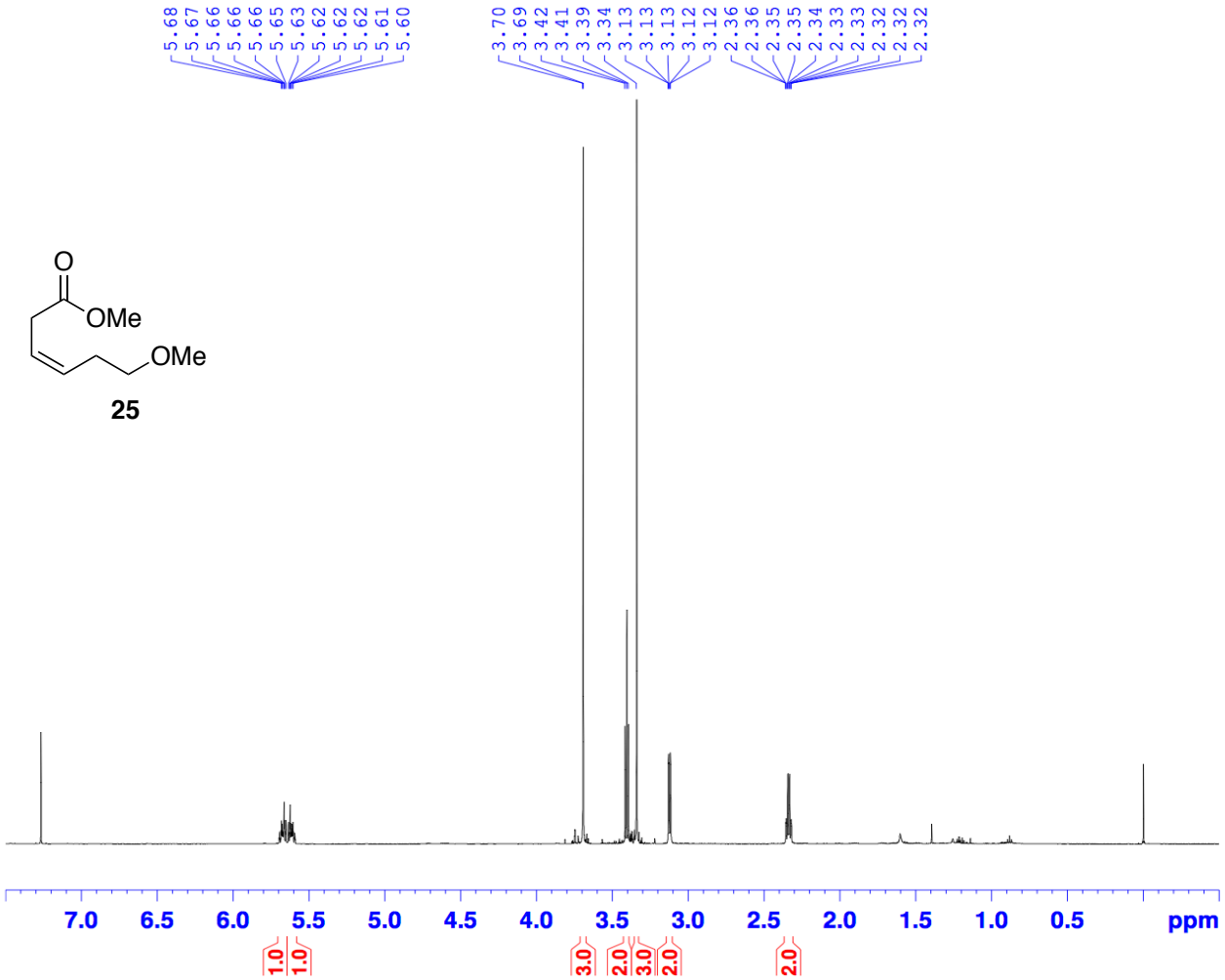

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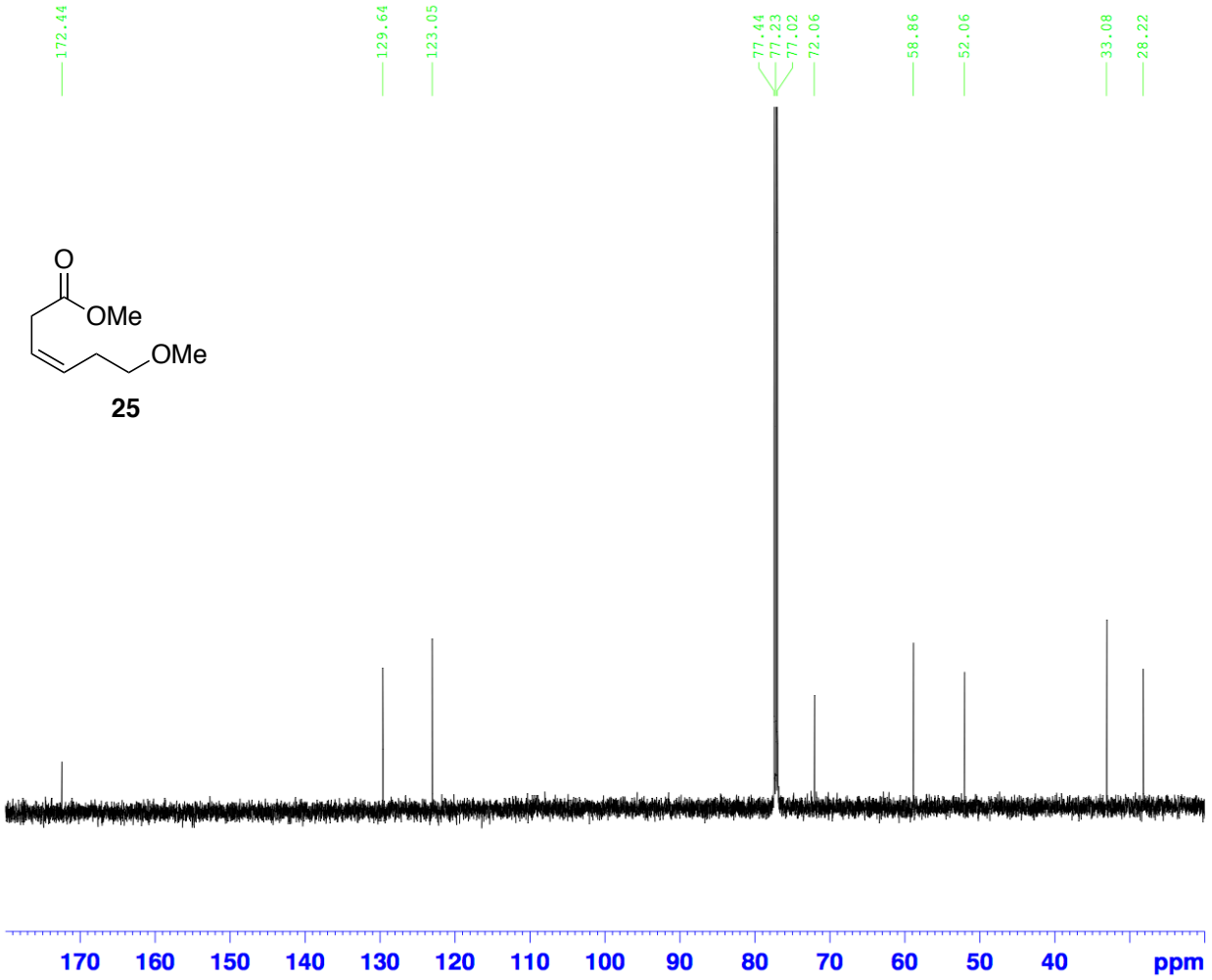
BRUKER

```
Current Data Parameters
NAME        VTT-III-82-B
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120330
Time              23.48
INSTRUM           spect
PROBHD   5 mm PAQXI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG               104.51
DW               40.533 usec
DE                6.50 usec
TE                298.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.79 usec
PLW1         9.30000019 W
SFO1        600.1937064 MHz

F2 - Processing parameters
SI                65536
SF          600.1900111 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



25

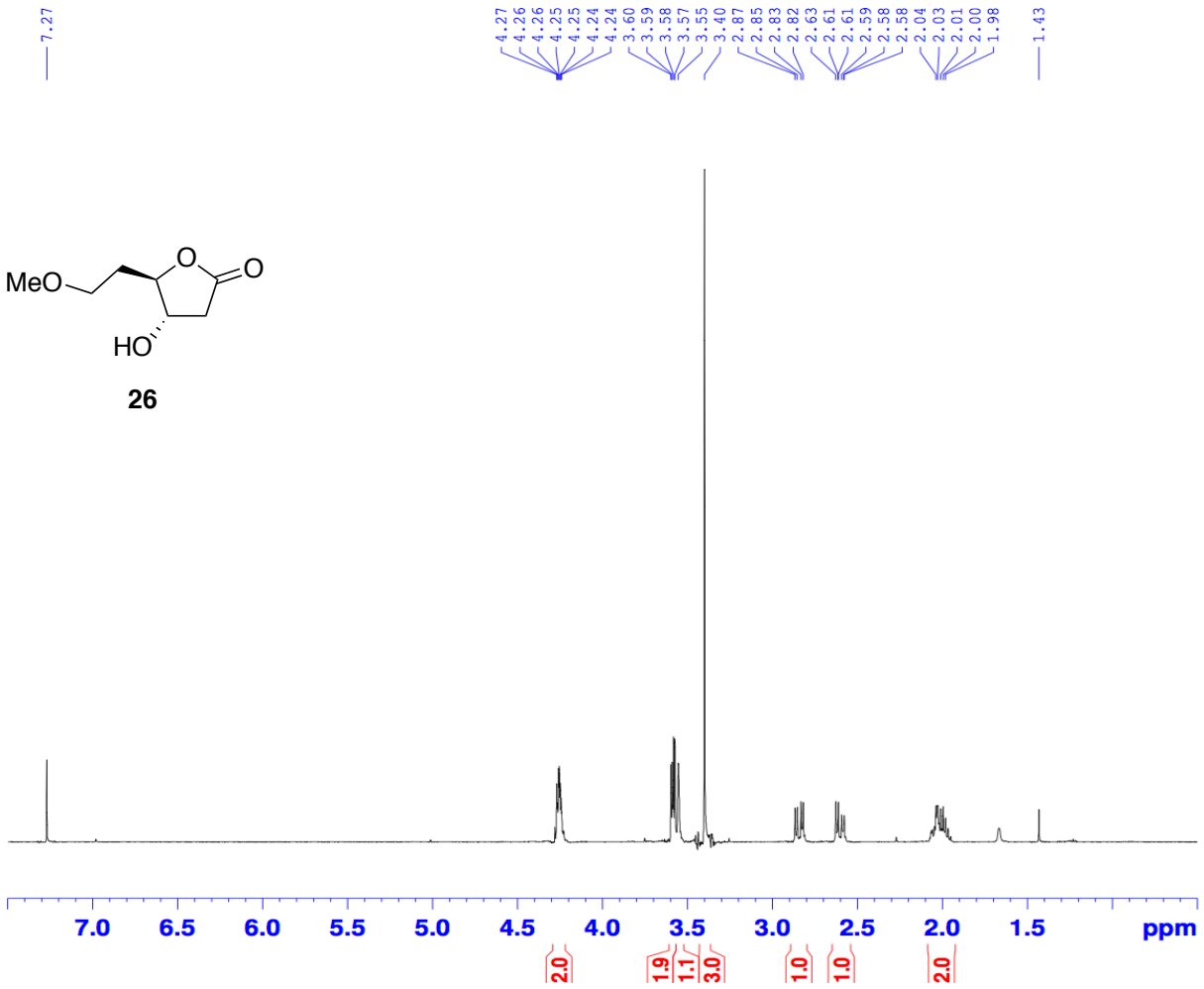
```
Current Data Parameters
NAME        VTT-III-82-B
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120330
Time               23.50
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG               184.65
DW               13.867 usec
DE                6.50 usec
TE               298.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.00 usec
PLW1       106.00000000 W
SFO1        150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PLW2         9.30000019 W
PLW12        0.18190999 W
PLW13        0.08913500 W
SFO2        600.1924008 MHz

F2 - Processing parameters
SI                32768
SF          150.9178644 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



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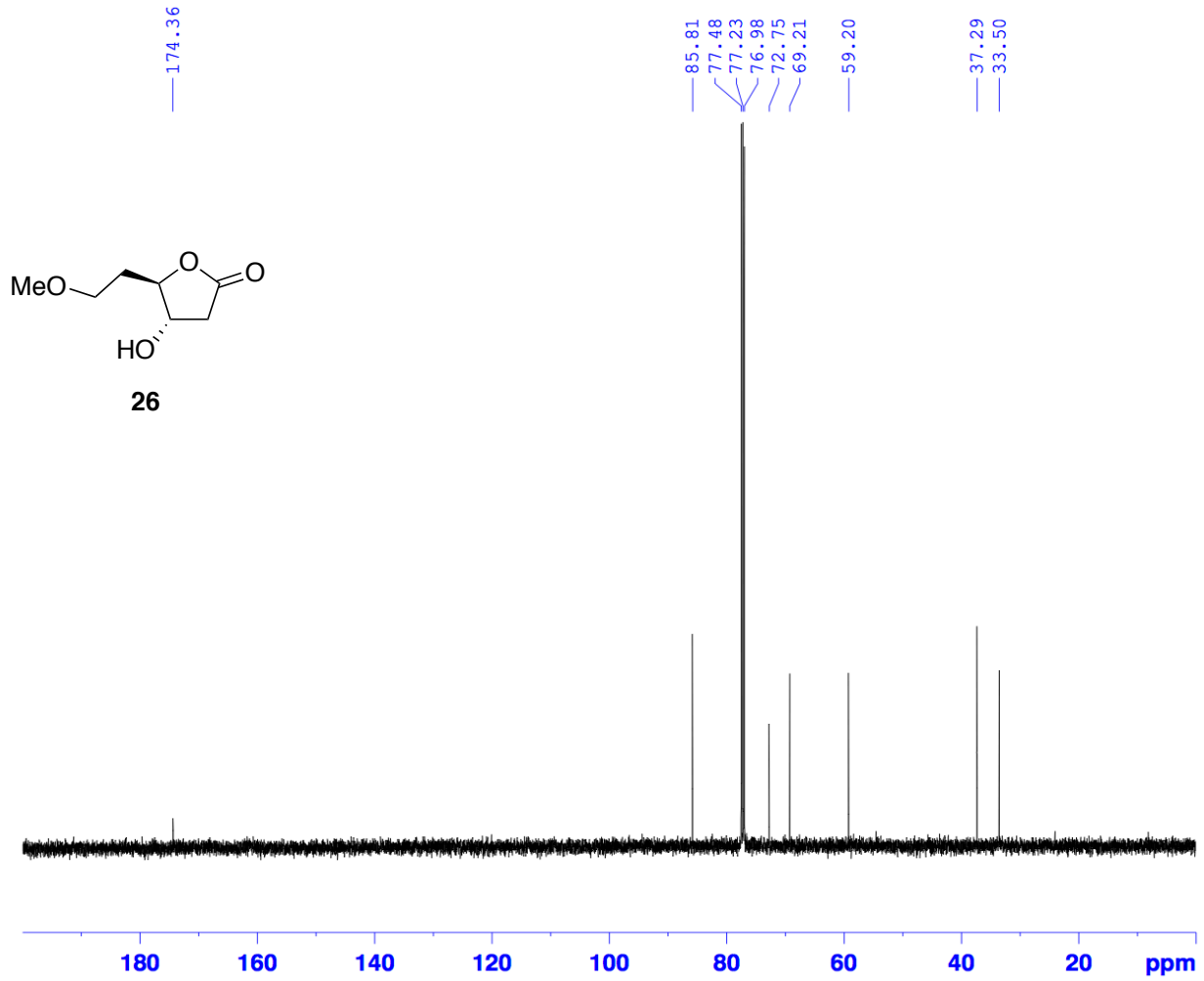
```
Current Data Parameters
NAME        VTT-III-87-A
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120403
Time               16.11
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            10000.000 Hz
FIDRES         0.152588 Hz
AQ             3.2768500 sec
RG                  90.5
DW                50.000 usec
DE                 6.50 usec
TE                298.1 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.20 usec
PL1                -3.00 dB
PL1W         37.58904266 W
SFO1         500.2020889 MHz

F2 - Processing parameters
SI                 32768
SF           500.1990105 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



26

```
Current Data Parameters
NAME        VTT-III-87-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120403
Time               15.46
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ            1.0912244 sec
RG               5792.6
DW               16.650 usec
DE                6.50 usec
TE                298.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.75 usec
PL1                 2.00 dB
PL1W        49.29017639 W
SFO1       125.7877161 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              70.00 usec
PL2                -3.00 dB
PL12               13.50 dB
PL13               13.50 dB
PL2W        37.58904266 W
PL12W        0.84151381 W
PL13W        0.84151381 W
SFO2       500.2010008 MHz

F2 - Processing parameters
SI                 32768
SF          125.7751143 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
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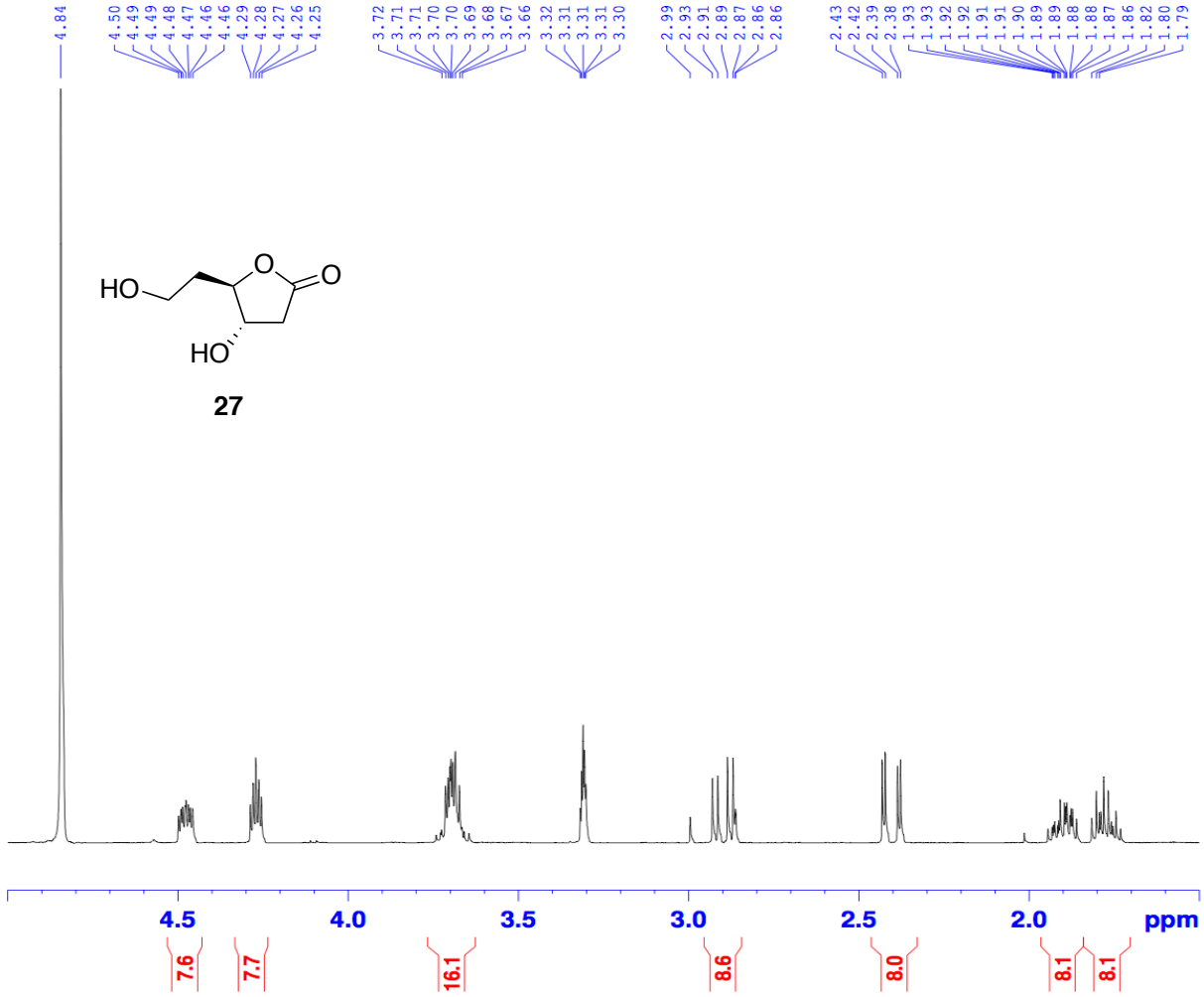

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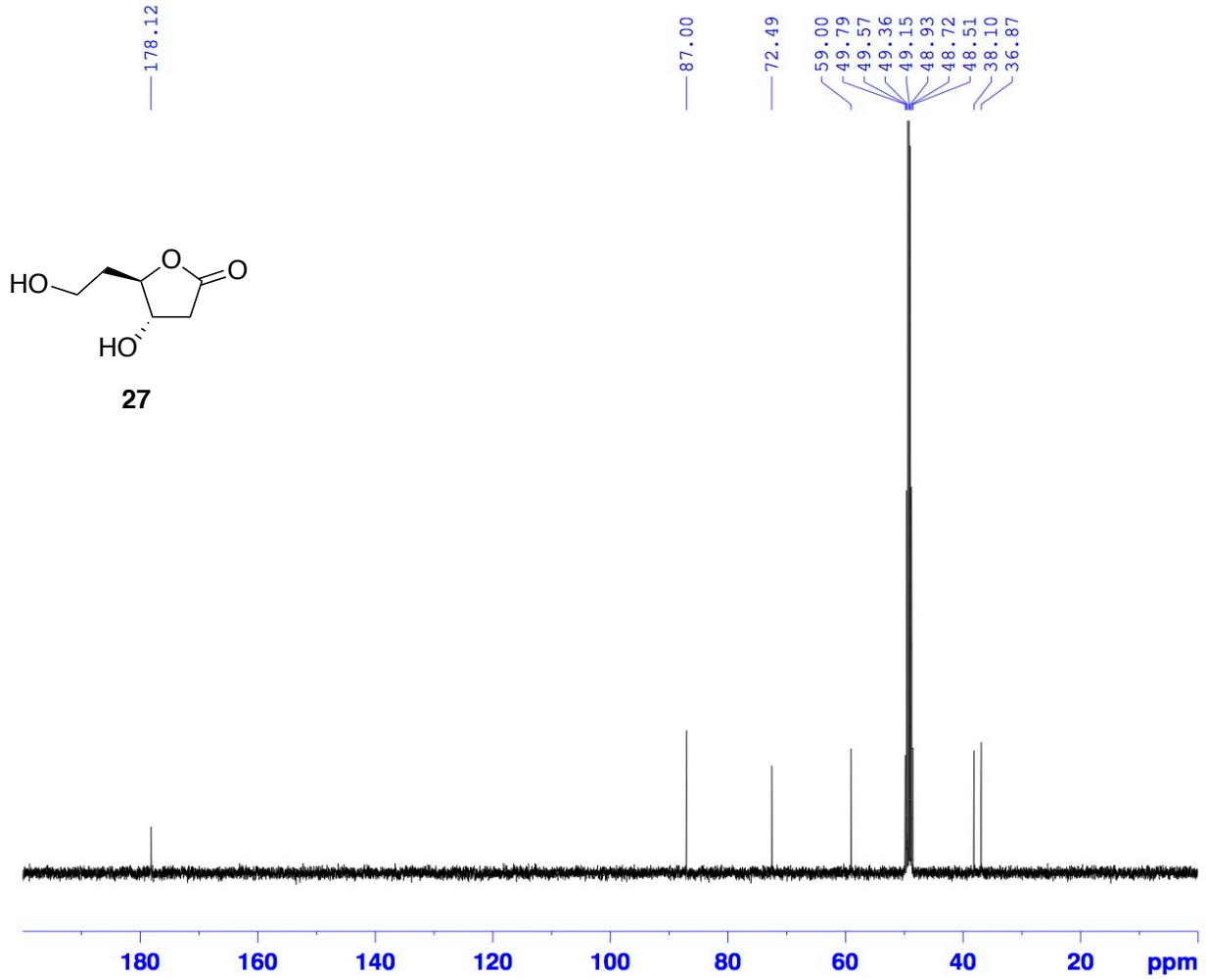
```
Current Data Parameters
NAME       VTT-III-99-A2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120421
Time               22.53
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   181
DW                60.400 usec
DE                 6.50 usec
TE                298.3 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.75 usec
PL1               -5.00 dB
PL1W        31.77312851 W
SFO1       400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF         400.1300076 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```



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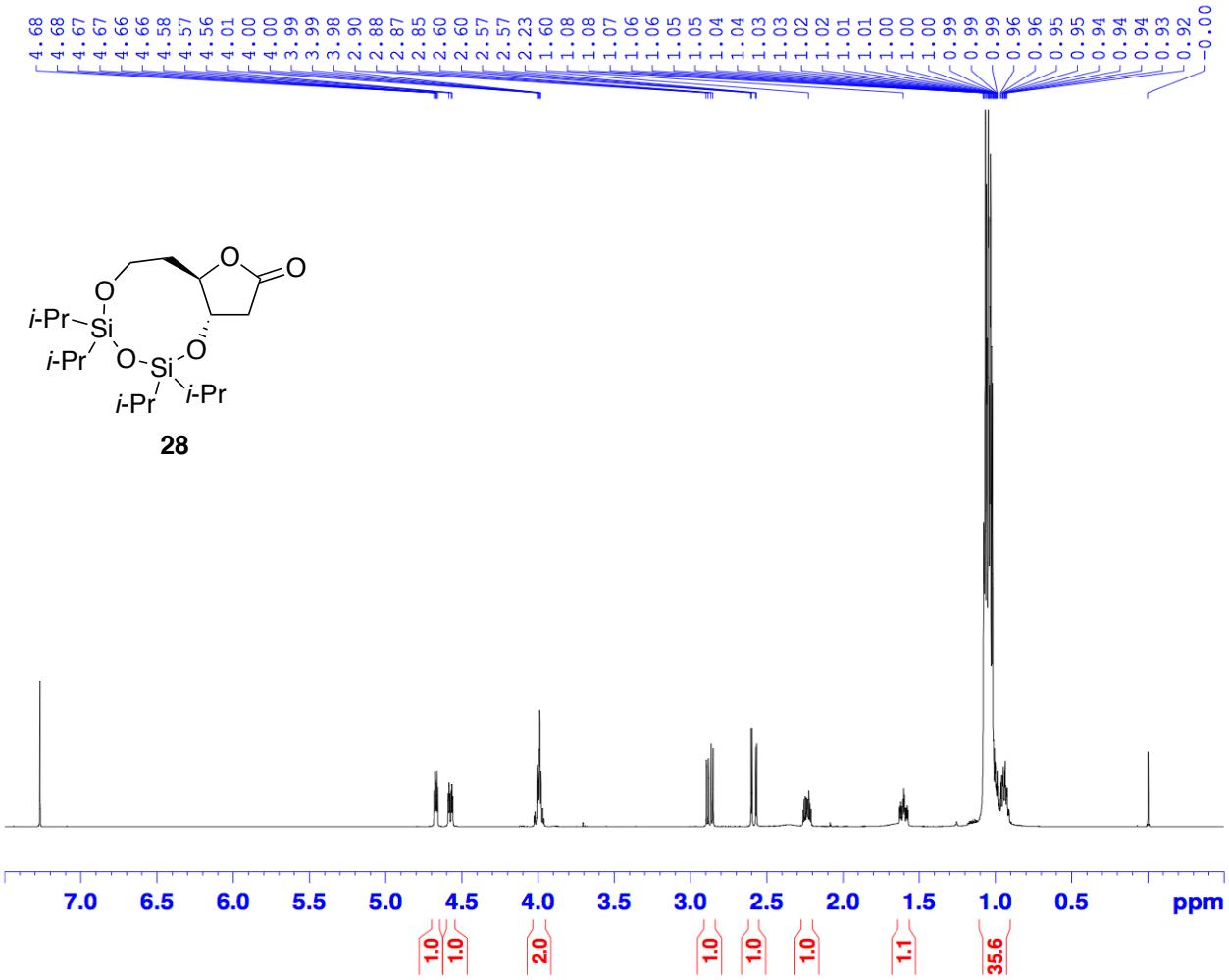
```
Current Data Parameters
NAME       VTT-III-99-A2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120421
Time               22.56
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT             MeOD
NS                   129
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                 32768
DW                20.850 usec
DE                 6.50 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  6.60 usec
PL1                -4.00 dB
PL1W        25.23829460 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              70.00 usec
PL2                -5.00 dB
PL12               13.84 dB
PL13               13.84 dB
PL2W        31.77312851 W
PL12W        0.41501135 W
PL13W        0.41501135 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6126128 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



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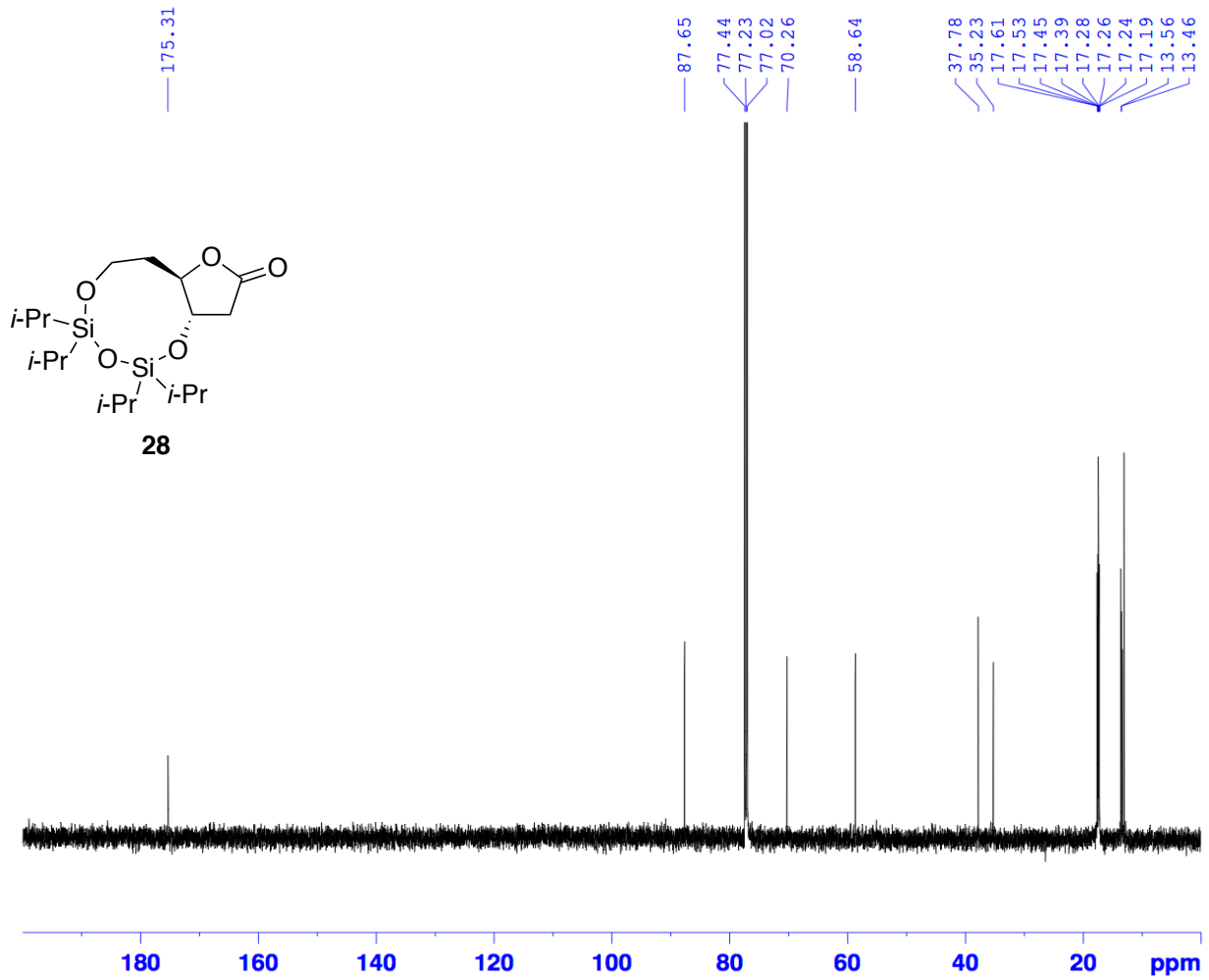
```
Current Data Parameters
NAME      VTT-III-121-A
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20120521
Time              13.53
INSTRUM           spect
PROBHD   5 mm PAQXI 1H/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ             2.6564426 sec
RG                33.59
DW               40.533 usec
DE                6.50 usec
TE                298.1 K
D1           2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                9.79 usec
PLW1         9.30000019 W
SFO1        600.1937064 MHz

F2 - Processing parameters
SI               65536
SF          600.1900099 MHz
WDW                 EM
SSB       0
LB             0.30 Hz
GB        0
PC             1.00
```



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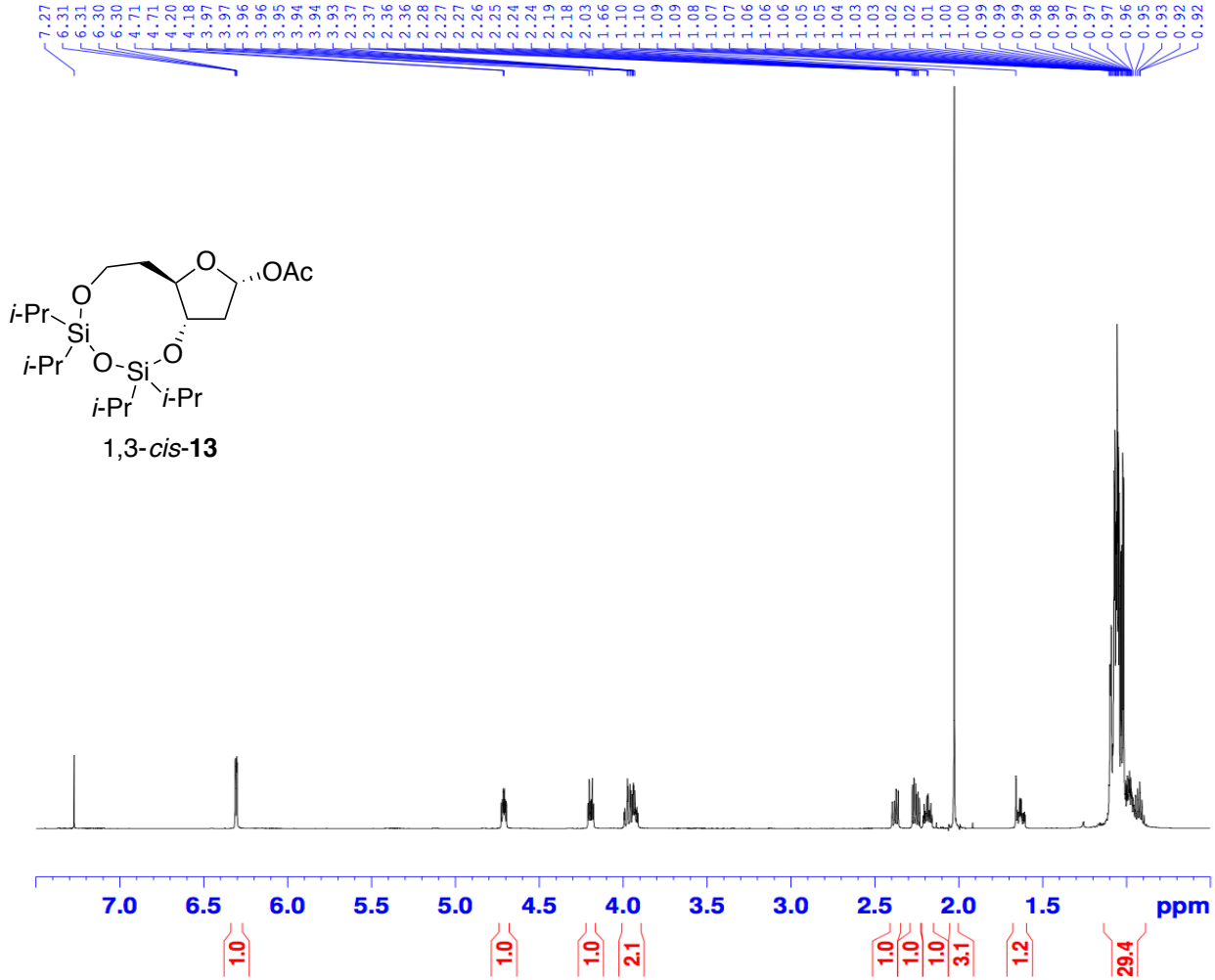
```
Current Data Parameters
NAME       VTT-III-121-A
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120521
Time              14.00
INSTRUM           spect
PROBHD   5 mm PAQXI 1H/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9088159 sec
RG                184.65
DW              13.867 usec
DE                6.50 usec
TE                298.1 K
D1           2.50000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.00 usec
PLW1       106.00000000 W
SFO1        150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PLW2          9.30000019 W
PLW12         0.18190999 W
PLW13         0.08913500 W
SFO2        600.1924008 MHz

F2 - Processing parameters
SI                32768
SF          150.9178647 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



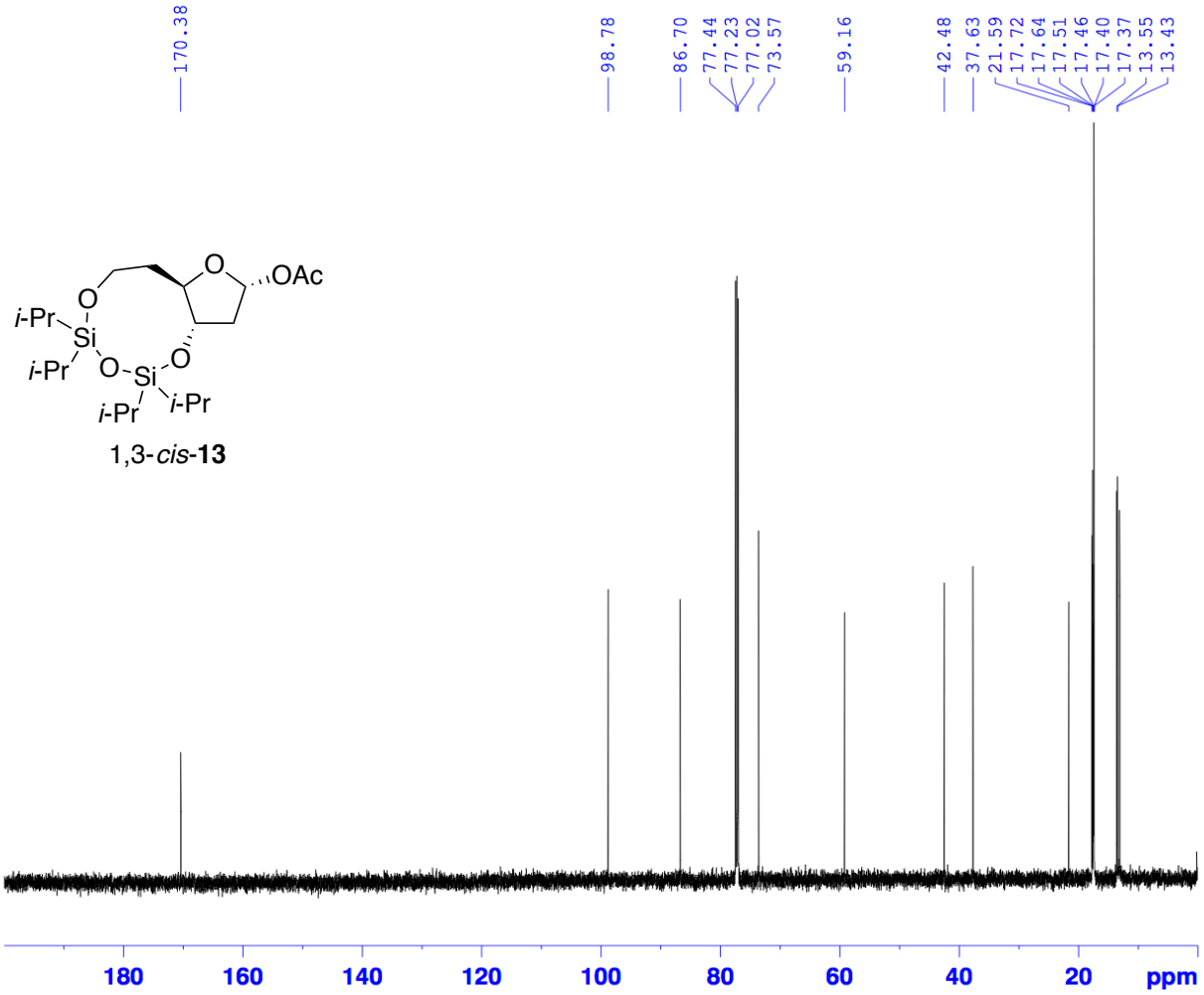
1,3-*cis*-**13**

```
Current Data Parameters
NAME       VTT-III-147-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120620
Time               13.38
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                33.59
DW               40.533 usec
DE                6.50 usec
TE                298.1 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.79 usec
PLW1         9.30000019 W
SFO1        600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF          600.1900075 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



1,3-*cis*-**13**

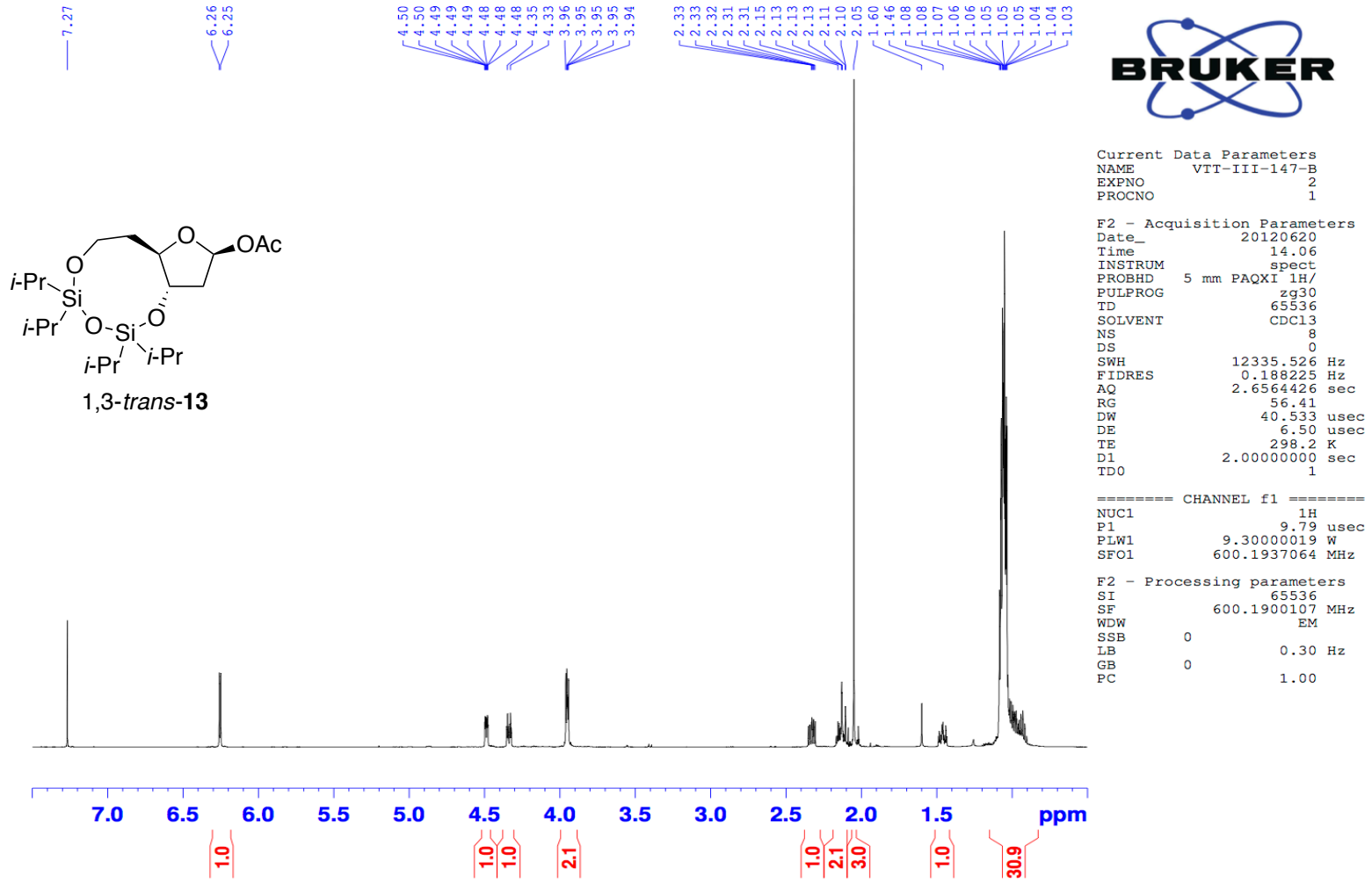
```
Current Data Parameters
NAME      VTT-III-147-A
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20120620
Time              13.38
INSTRUM           spect
PROBHD   5 mm PAQXI 1H/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9088159 sec
RG                184.65
DW                13.867 usec
DE                 6.50 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.00 usec
PLW1       106.00000000 W
SFO1        150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PLW2          9.30000019 W
PLW12         0.18190999 W
PLW13         0.08913500 W
SFO2         600.1924008 MHz

F2 - Processing parameters
SI                32768
SF          150.9178656 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



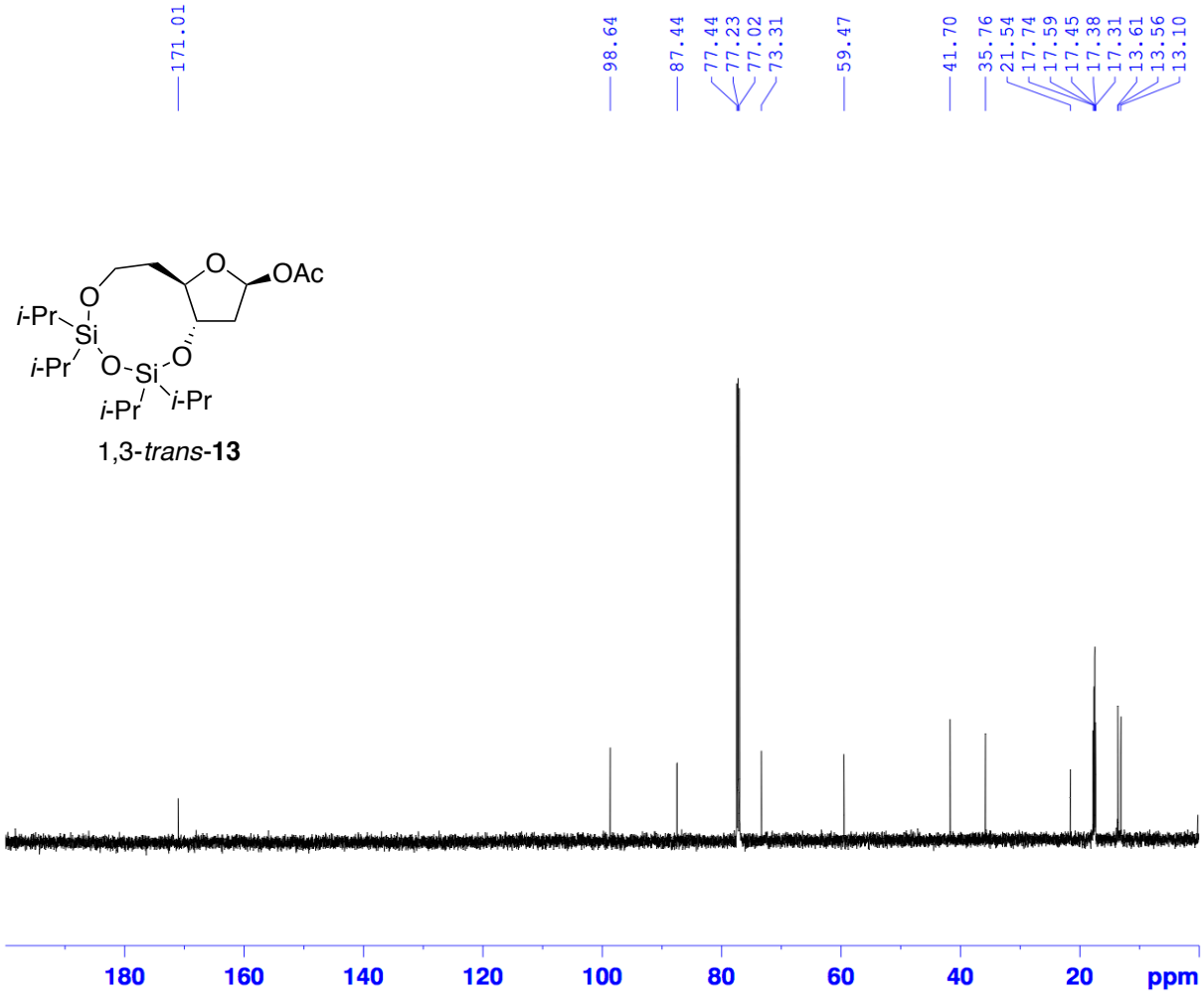
1,3-*trans*-**13**

```
Current Data Parameters
NAME       VTT-III-147-B
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120620
Time               14.06
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            12335.526 Hz
FIDRES         0.188225 Hz
AQ             2.6564426 sec
RG                 56.41
DW                40.533 usec
DE                 6.50 usec
TE                298.2 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.79 usec
PLW1          9.30000019 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF           600.1900107 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



1,3-*trans*-**13**

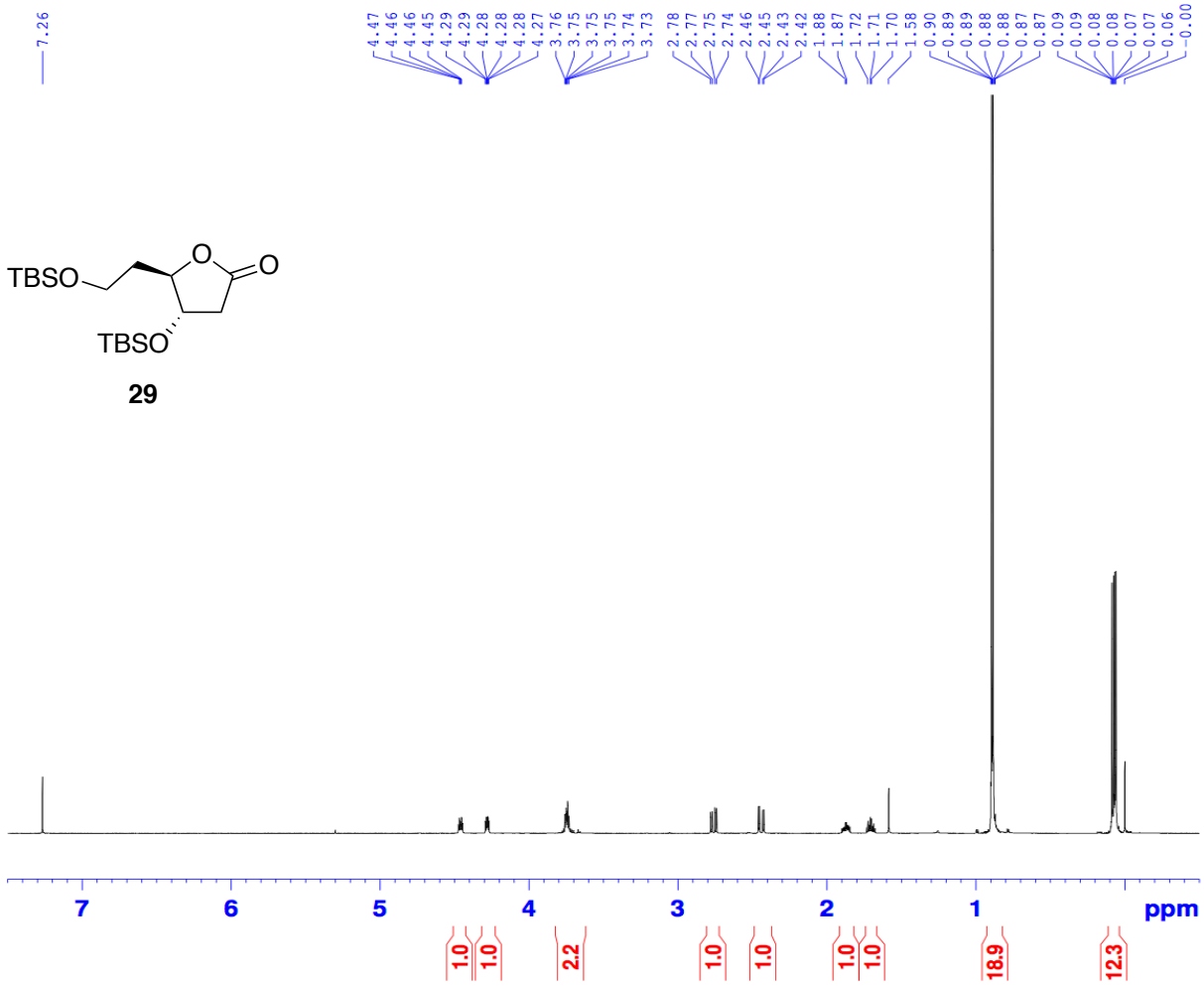
```
Current Data Parameters
NAME       VTT-III-147-B
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20120620
Time             14.10
INSTRUM          spect
PROBHD   5 mm PAQXI 1H/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 128
DS                   0
SWH          36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9088159 sec
RG               184.65
DW               13.867 usec
DE                6.50 usec
TE               298.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                15.00 usec
PLW1       106.00000000 W
SFO1        150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            70.00 usec
PLW2          9.30000019 W
PLW12         0.18190999 W
PLW13         0.08913500 W
SFO2        600.1924008 MHz

F2 - Processing parameters
SI                32768
SF          150.9178640 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



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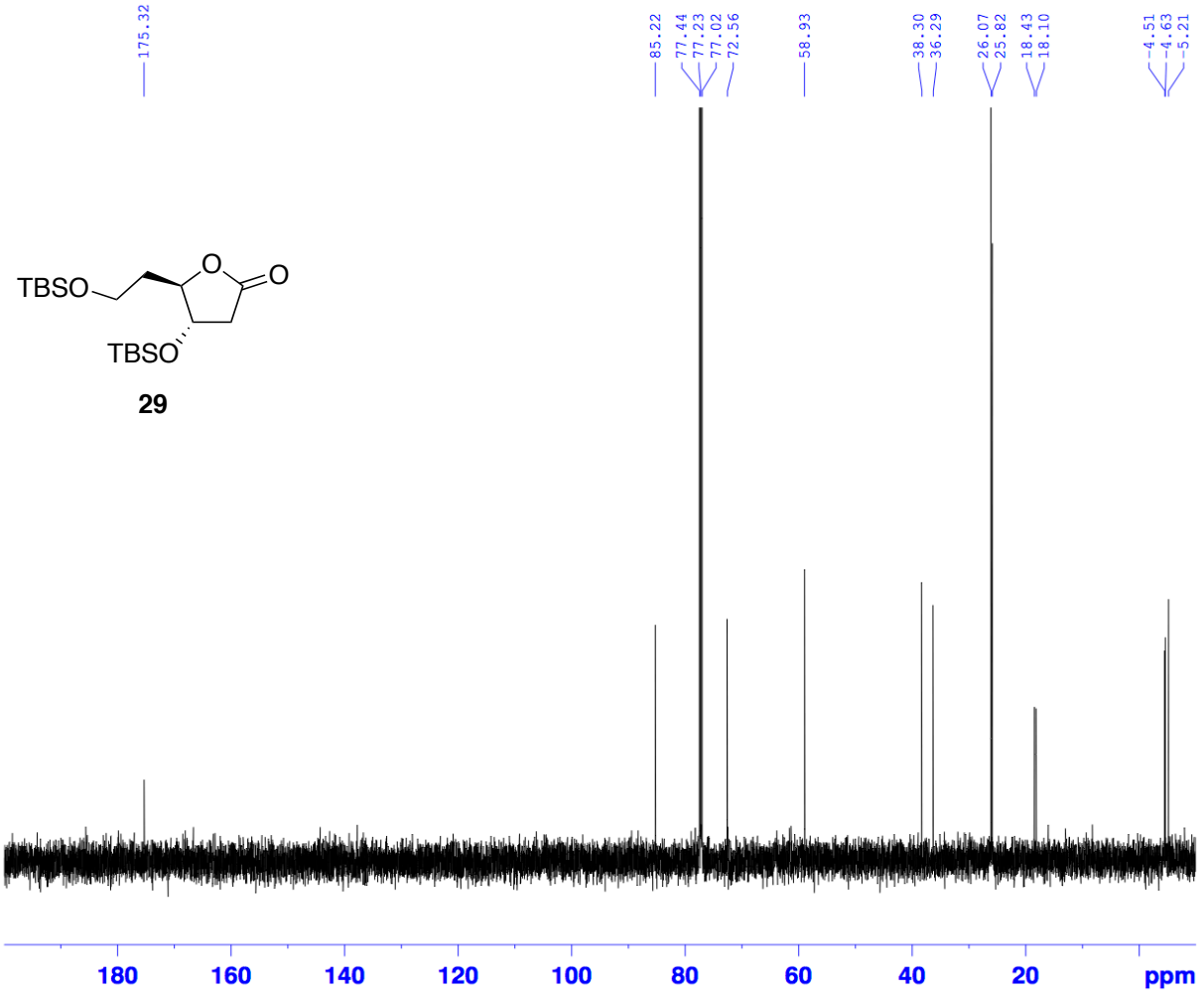
```
Current Data Parameters
NAME       VTT-III-185-A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120728
Time               12.31
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            12335.526 Hz
FIDRES         0.188225 Hz
AQ             2.6564426 sec
RG                 56.41
DW                40.533 usec
DE                 6.50 usec
TE                298.2 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.00 usec
PLW1         26.50000000 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF           600.1900126 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



29

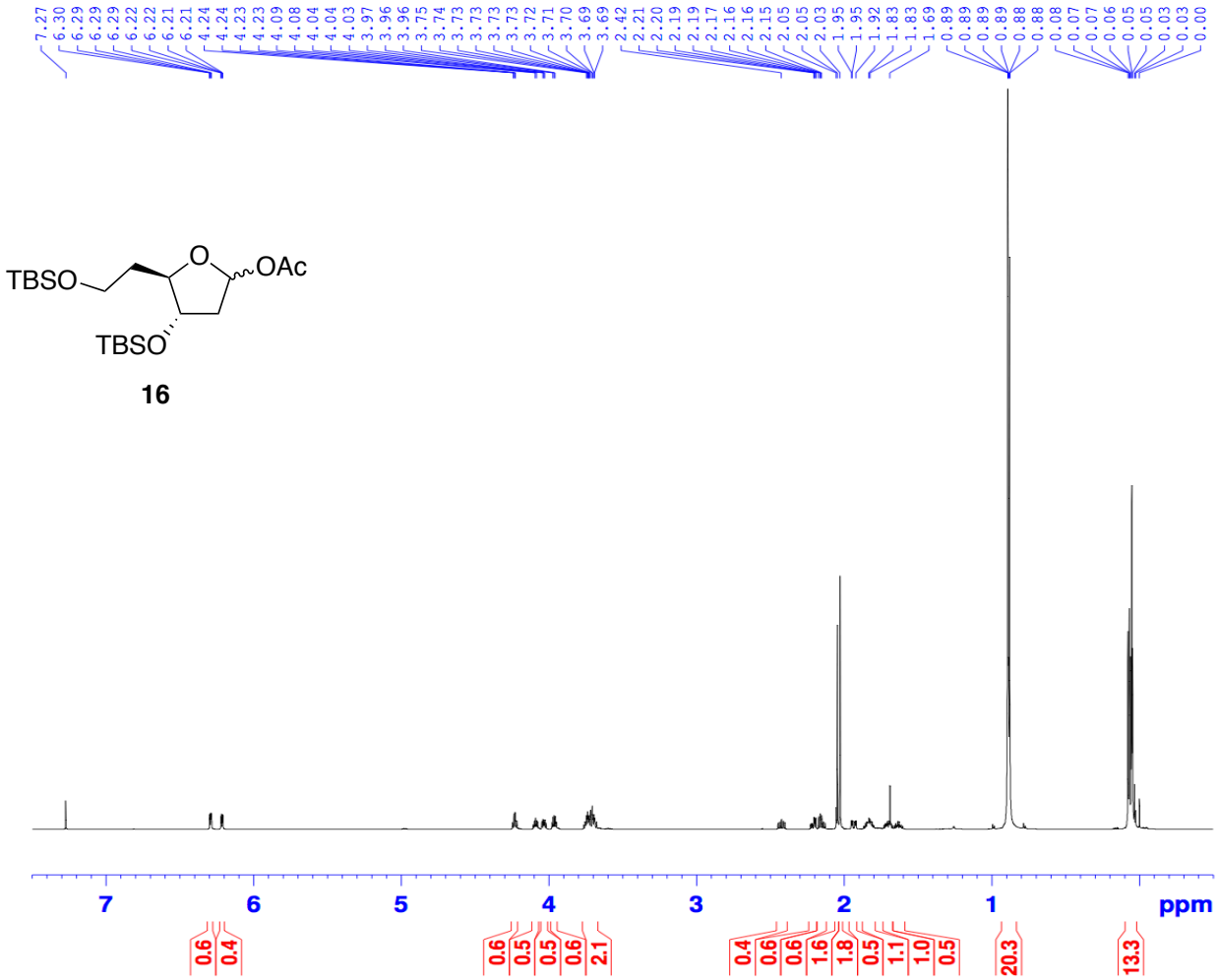
```
Current Data Parameters
NAME       VTT-III-185-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120728
Time               12.32
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                   128
DS                     0
SWH            36057.691 Hz
FIDRES          0.550197 Hz
AQ             0.9088159 sec
RG                184.65
DW                13.867 usec
DE                 6.50 usec
TE                298.3 K
D1            2.50000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.65 usec
PLW1        104.00000000 W
SFO1         150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              70.00 usec
PLW2         26.50000000 W
PLW12         0.65438998 W
PLW13         0.32065001 W
SFO2         600.1924008 MHz

F2 - Processing parameters
SI                 32768
SF          150.9178645 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

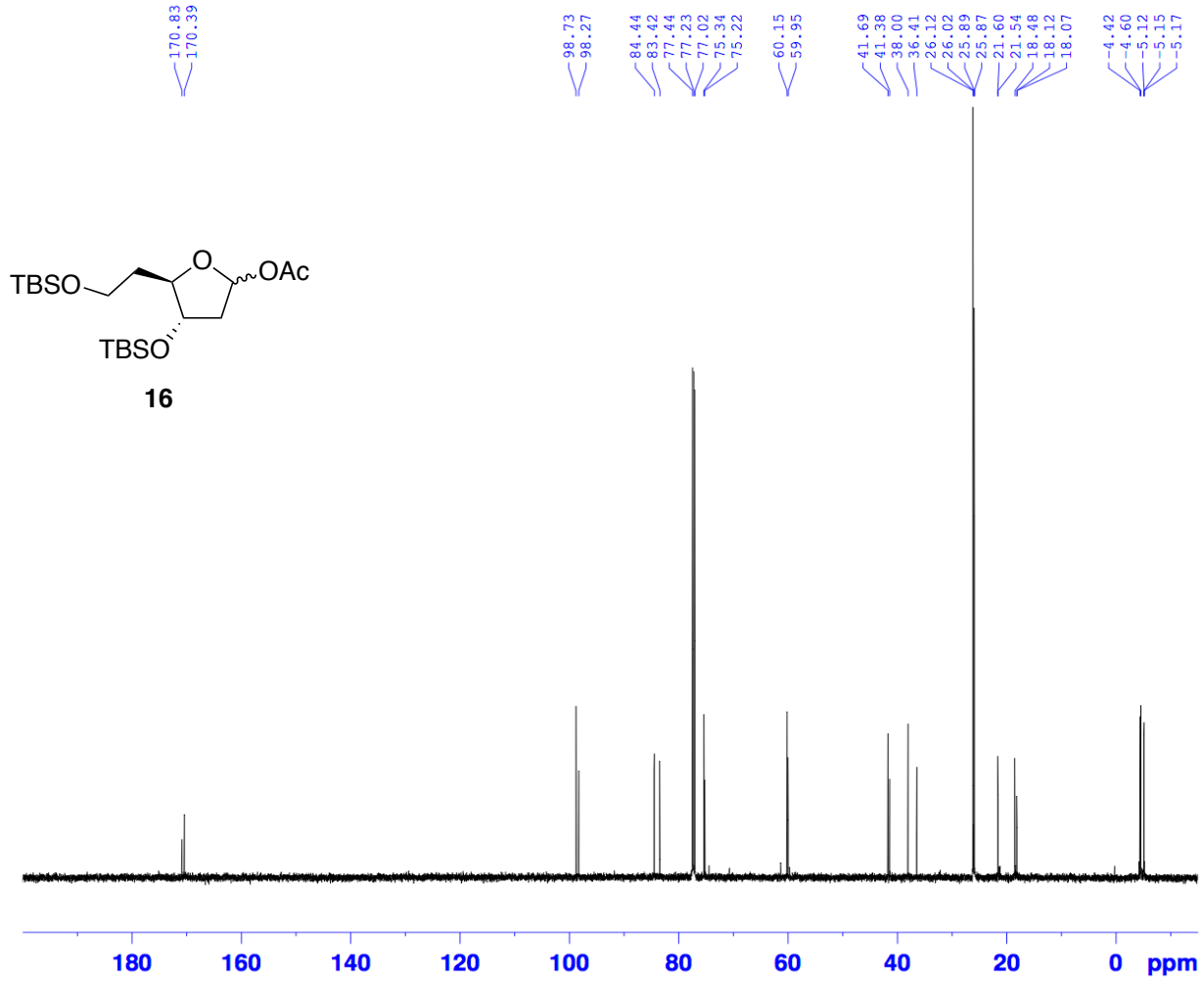



```
Current Data Parameters
NAME       VTT-III-190-A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120801
Time               22.30
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            12335.526 Hz
FIDRES         0.188225 Hz
AQ             2.6564426 sec
RG                 33.59
DW                40.533 usec
DE                 6.50 usec
TE                298.2 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.00 usec
PLW1         26.50000000 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF           600.1900084 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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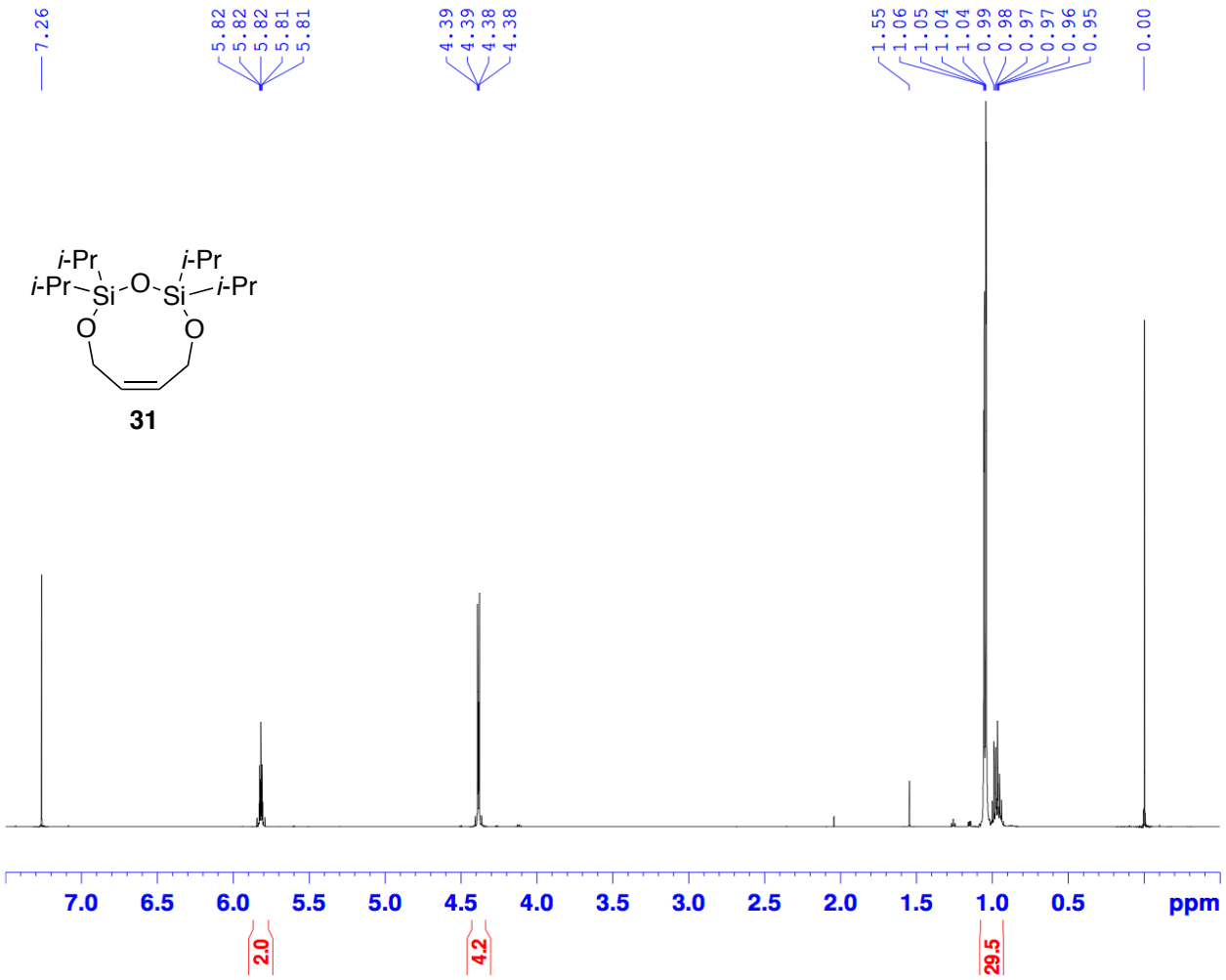
```
Current Data Parameters
NAME       VTT-III-190-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120801
Time               22.38
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                   128
DS                     0
SWH            36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9088159 sec
RG                184.65
DW                13.867 usec
DE                 6.50 usec
TE                298.1 K
D1            2.50000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.65 usec
PLW1        104.00000000 W
SFO1         150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              70.00 usec
PLW2         26.50000000 W
PLW12         0.65438998 W
PLW13         0.32065001 W
SFO2         600.1924008 MHz

F2 - Processing parameters
SI                 32768
SF          150.9178651 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



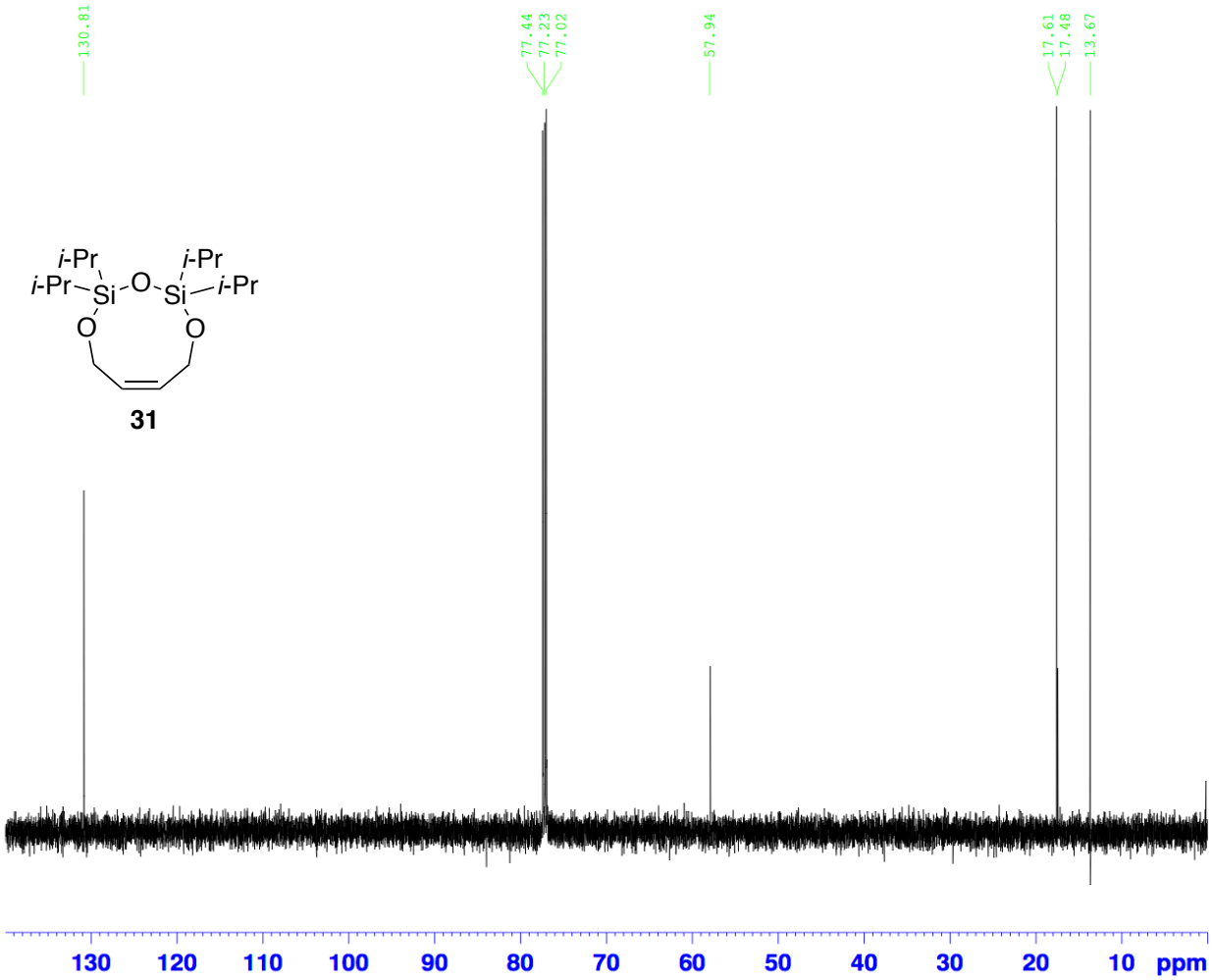
31

```
Current Data Parameters
NAME         VTT-IV-26-A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121005
Time              16.08
INSTRUM           spect
PROBHD   5 mm PAQXI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                92.46
DW               40.533 usec
DE                6.50 usec
TE                298.2 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.79 usec
PLW1         9.30000019 W
SFO1        600.1937064 MHz

F2 - Processing parameters
SI                65536
SF          600.1900134 MHz
WDW                  no
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```



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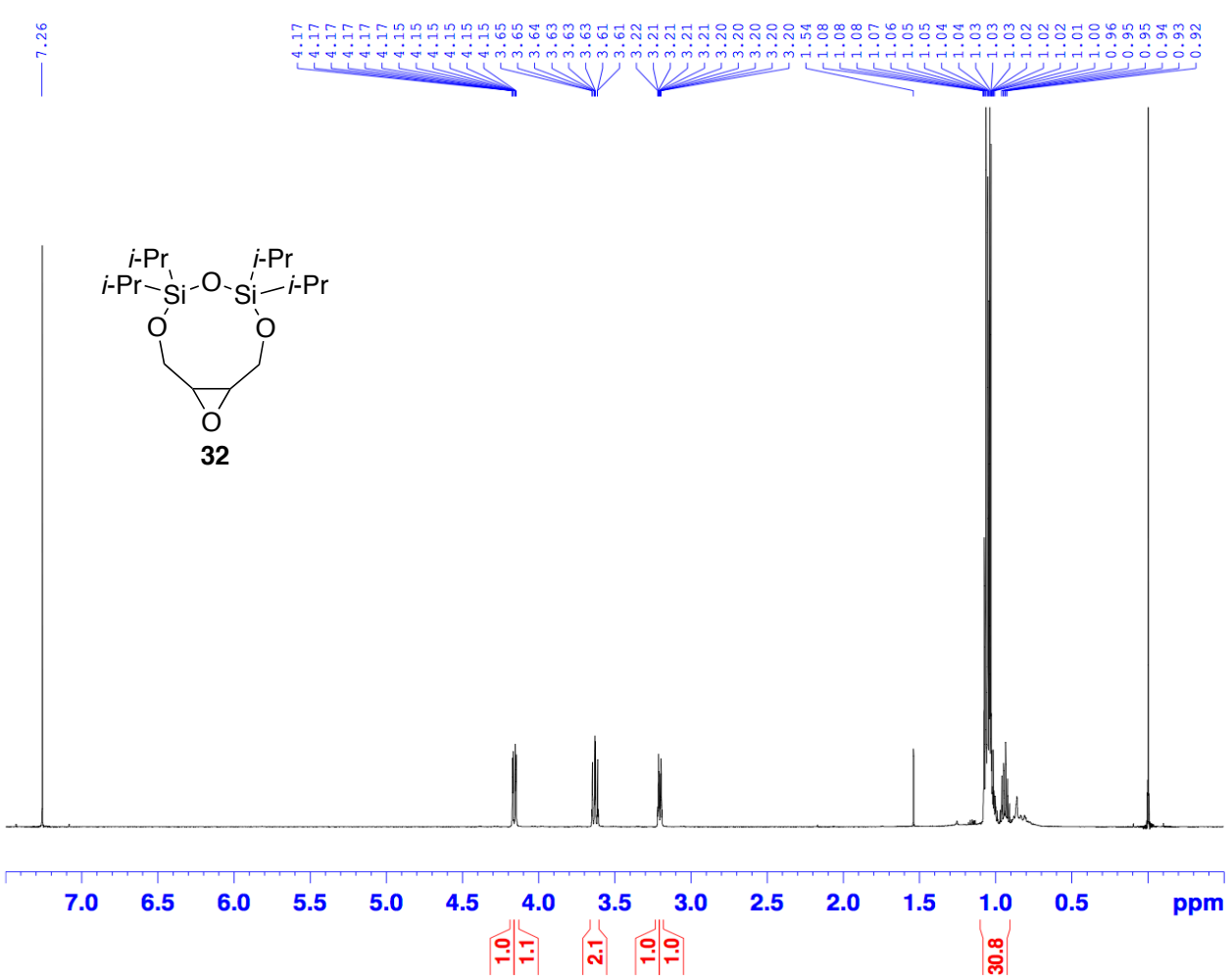
```
Current Data Parameters
NAME          VTT-IV-26-A
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20121005
Time              16.09
INSTRUM           spect
PROBHD   5 mm PAQXI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG               184.65
DW               13.867 usec
DE                6.50 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.00 usec
PLW1       106.00000000 W
SFO1        150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PLW2          9.30000019 W
PLW12         0.18190999 W
PLW13         0.08913500 W
SFO2        600.1924008 MHz

F2 - Processing parameters
SI                32768
SF          150.9178624 MHz
WDW                  no
SSB       0
LB        0 Hz
GB        0
PC                1.40
```



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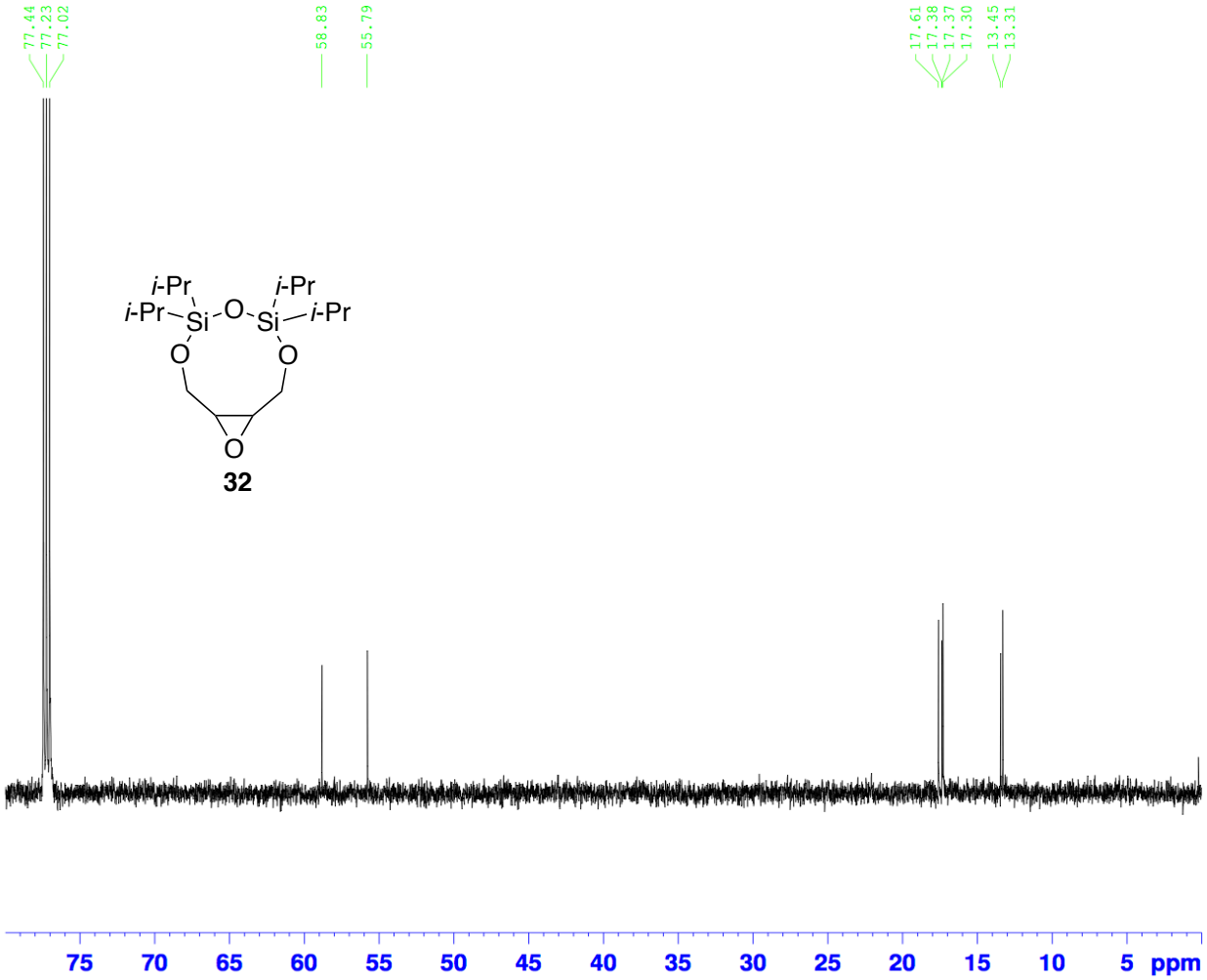
```
Current Data Parameters
NAME         VTT-IV-28-A
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121007
Time               13.26
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            12335.526 Hz
FIDRES         0.188225 Hz
AQ             2.6564426 sec
RG                113.19
DW                40.533 usec
DE                 6.50 usec
TE                298.1 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.79 usec
PLW1          9.30000019 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF           600.1900134 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```



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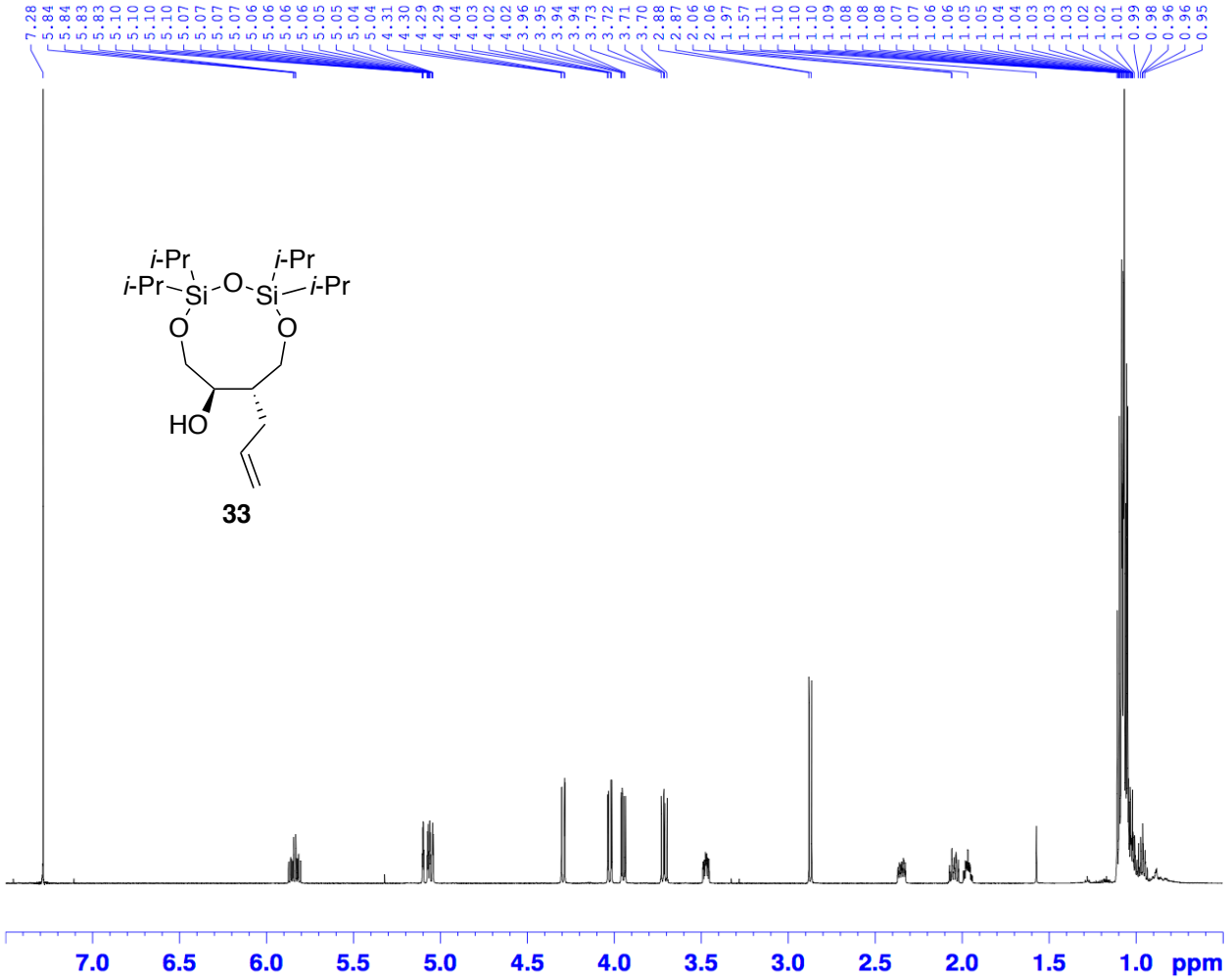
```
Current Data Parameters
NAME          VTT-IV-28-A
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20121007
Time               13.27
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9088159 sec
RG               184.65
DW                13.867 usec
DE                 6.50 usec
TE                298.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.00 usec
PLW1        106.00000000 W
SFO1         150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PLW2           9.30000019 W
PLW12          0.18190999 W
PLW13          0.08913500 W
SFO2         600.1924008 MHz

F2 - Processing parameters
SI                  32768
SF           150.9178623 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

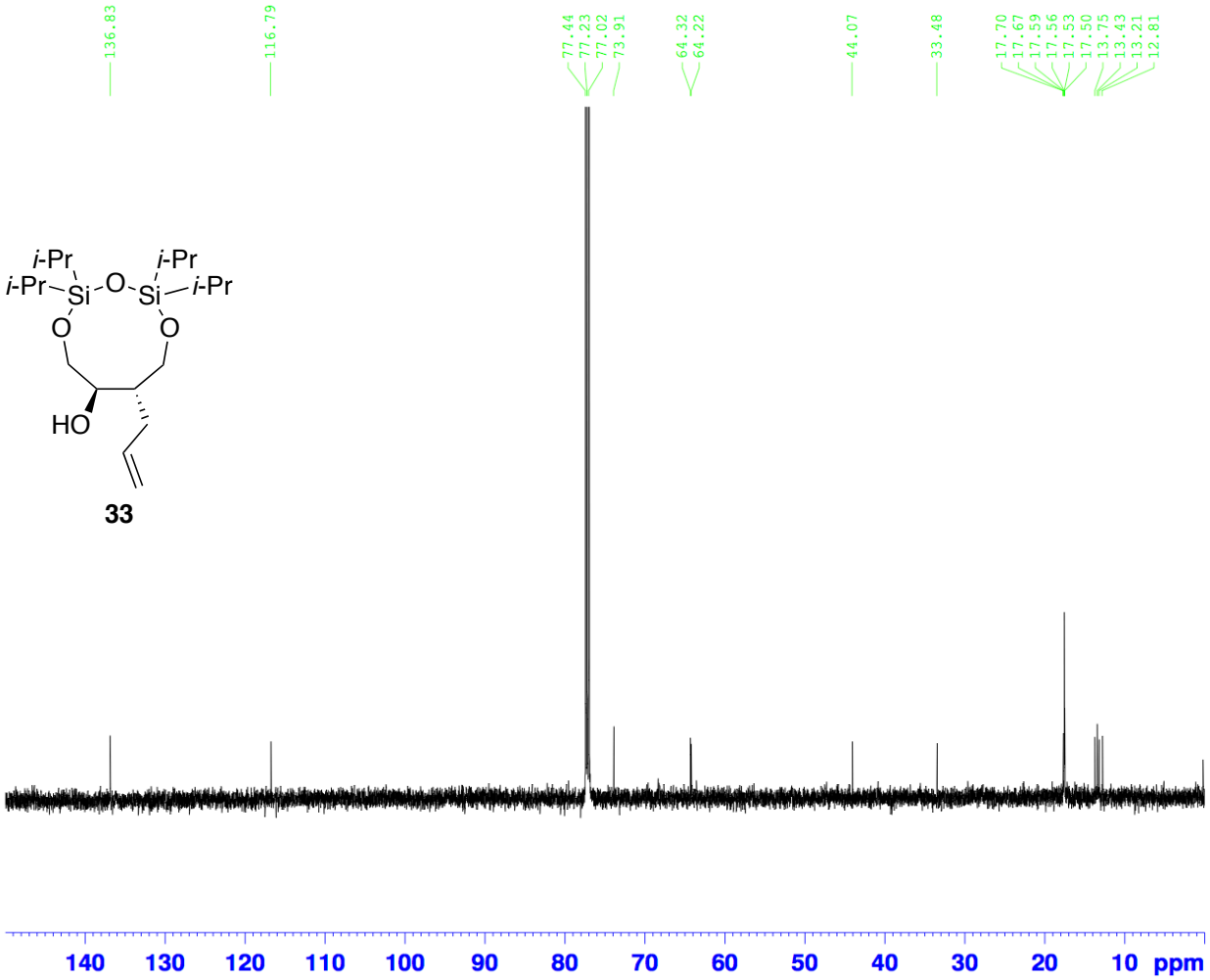



```
Current Data Parameters
NAME         VTT-IV-29-A
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121008
Time               17.09
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                 104.51
DW                 40.533 usec
DE                   6.50 usec
TE                  298.1 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.79 usec
PLW1          9.30000019 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF           600.1900000 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```



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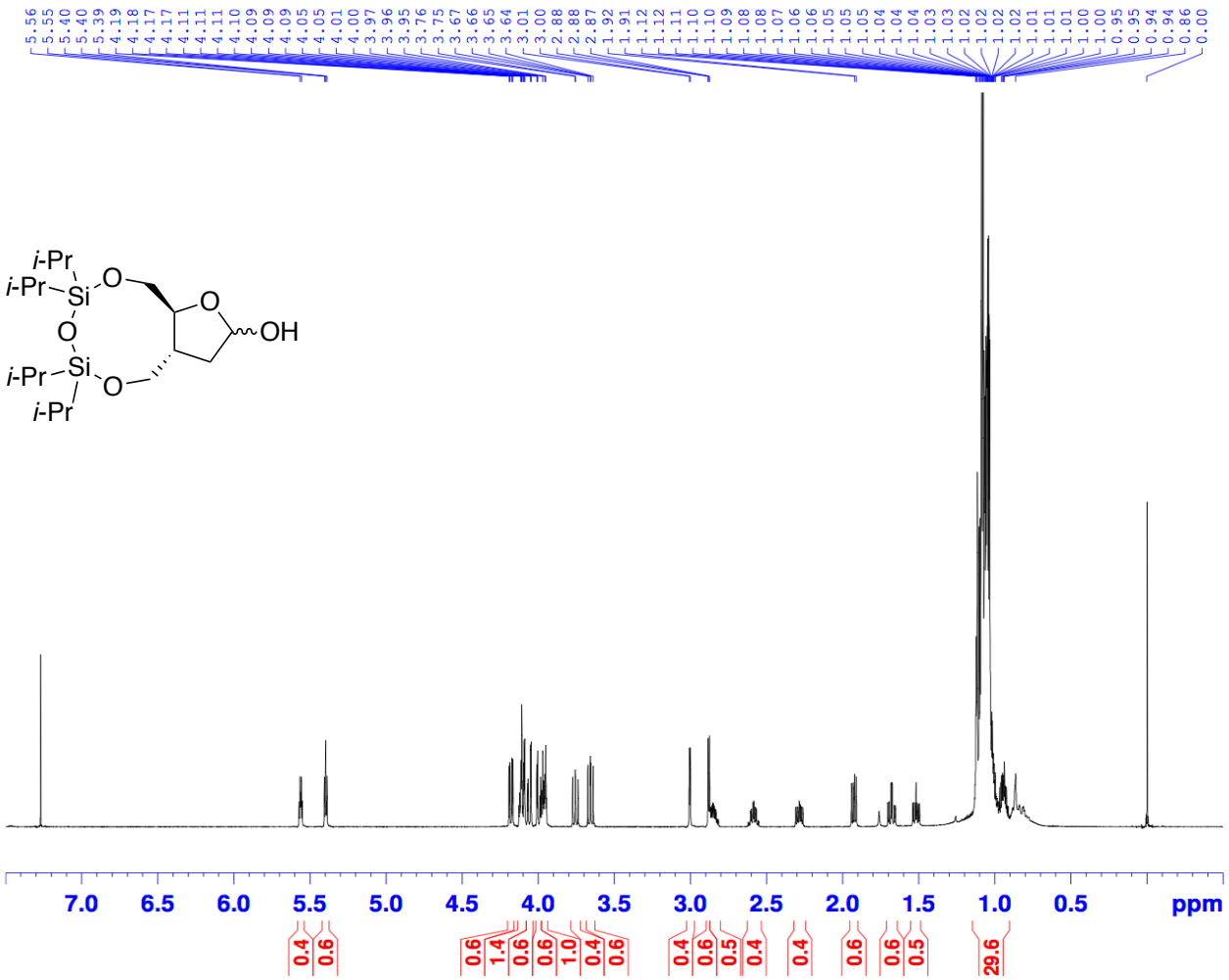
```
Current Data Parameters
NAME          VTT-IV-29-A
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20121008
Time               17.13
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9088159 sec
RG               184.65
DW              13.867 usec
DE                6.50 usec
TE               298.1 K
D1            2.50000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.00 usec
PLW1        106.00000000 W
SFO1         150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PLW2           9.30000019 W
PLW12          0.18190999 W
PLW13          0.08913500 W
SFO2         600.1924008 MHz

F2 - Processing parameters
SI                  32768
SF           150.9178629 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



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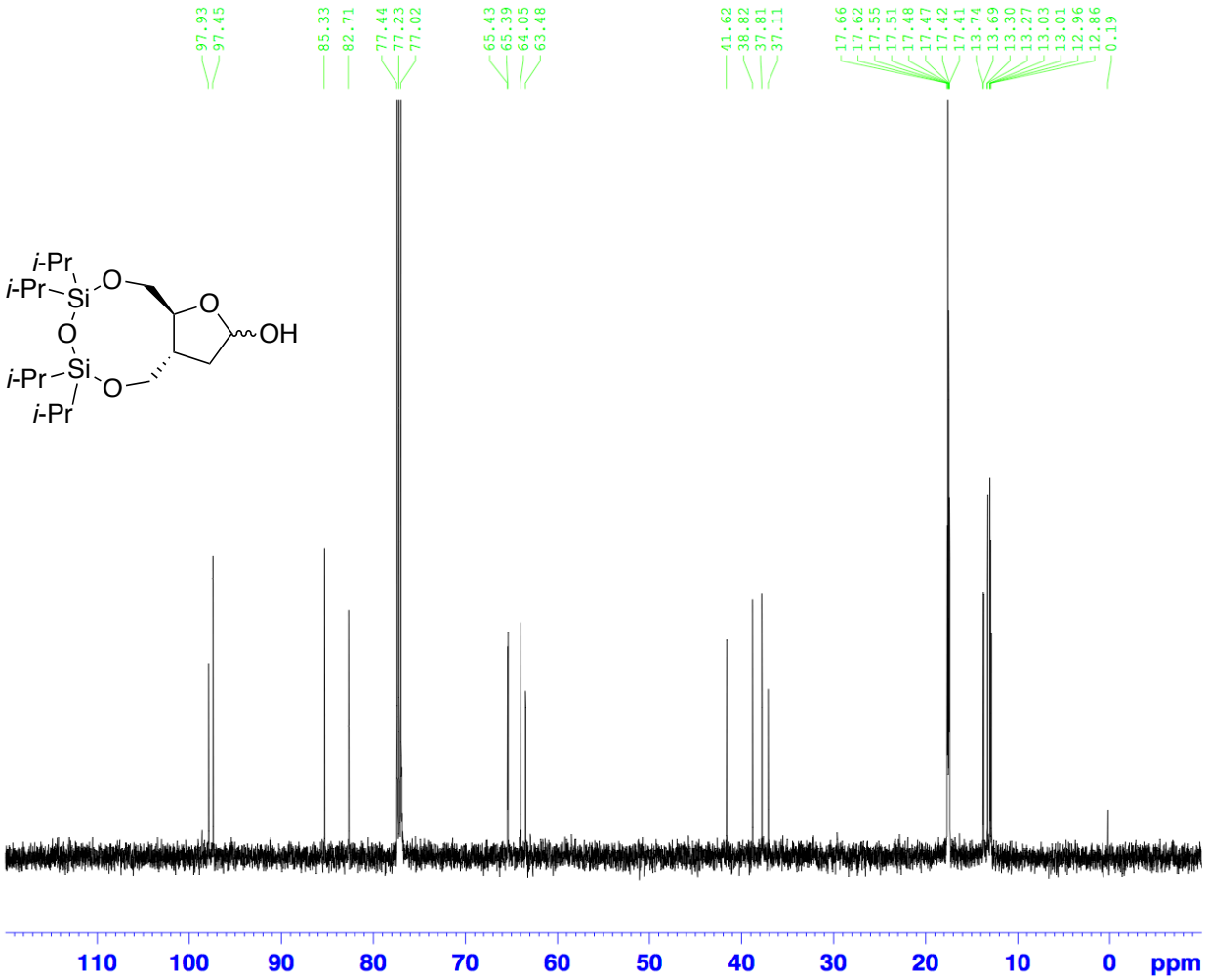
```
Current Data Parameters
NAME        VTT-IV-30-A2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121010
Time              13.03
INSTRUM           spect
PROBHD   5 mm PAQXI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                 18.8
DW               40.533 usec
DE                6.50 usec
TE                298.1 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.79 usec
PLW1         9.30000019 W
SFO1        600.1937064 MHz

F2 - Processing parameters
SI                65536
SF          600.1900085 MHz
WDW                  no
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```



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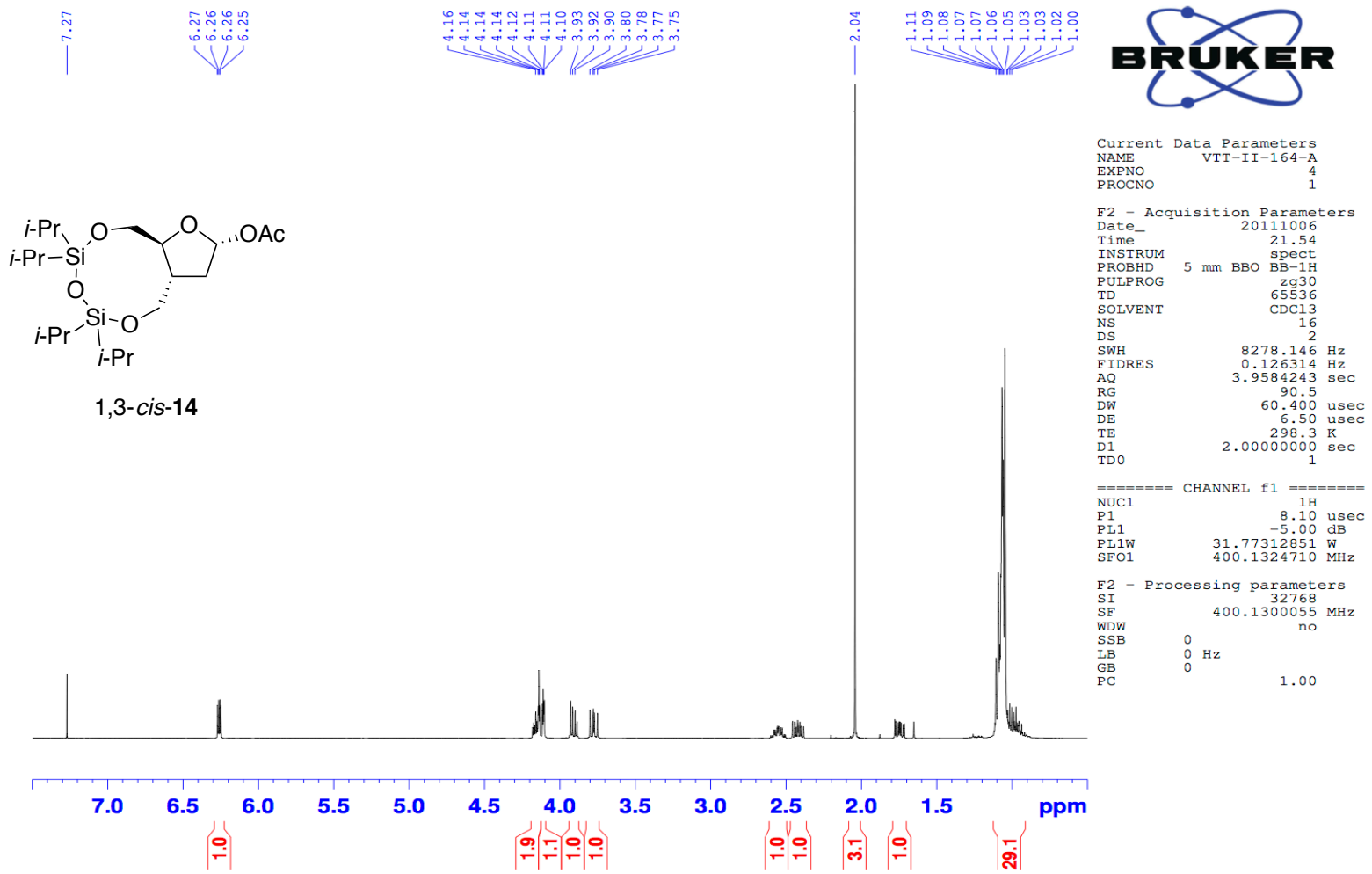
```
Current Data Parameters
NAME        VTT-IV-30-A2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121010
Time               13.07
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG               184.65
DW               13.867 usec
DE                6.50 usec
TE                298.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.00 usec
PLW1       106.00000000 W
SFO1        150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             70.00 usec
PLW2          9.30000019 W
PLW12         0.18190999 W
PLW13         0.08913500 W
SFO2        600.1924008 MHz

F2 - Processing parameters
SI                32768
SF          150.9178637 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



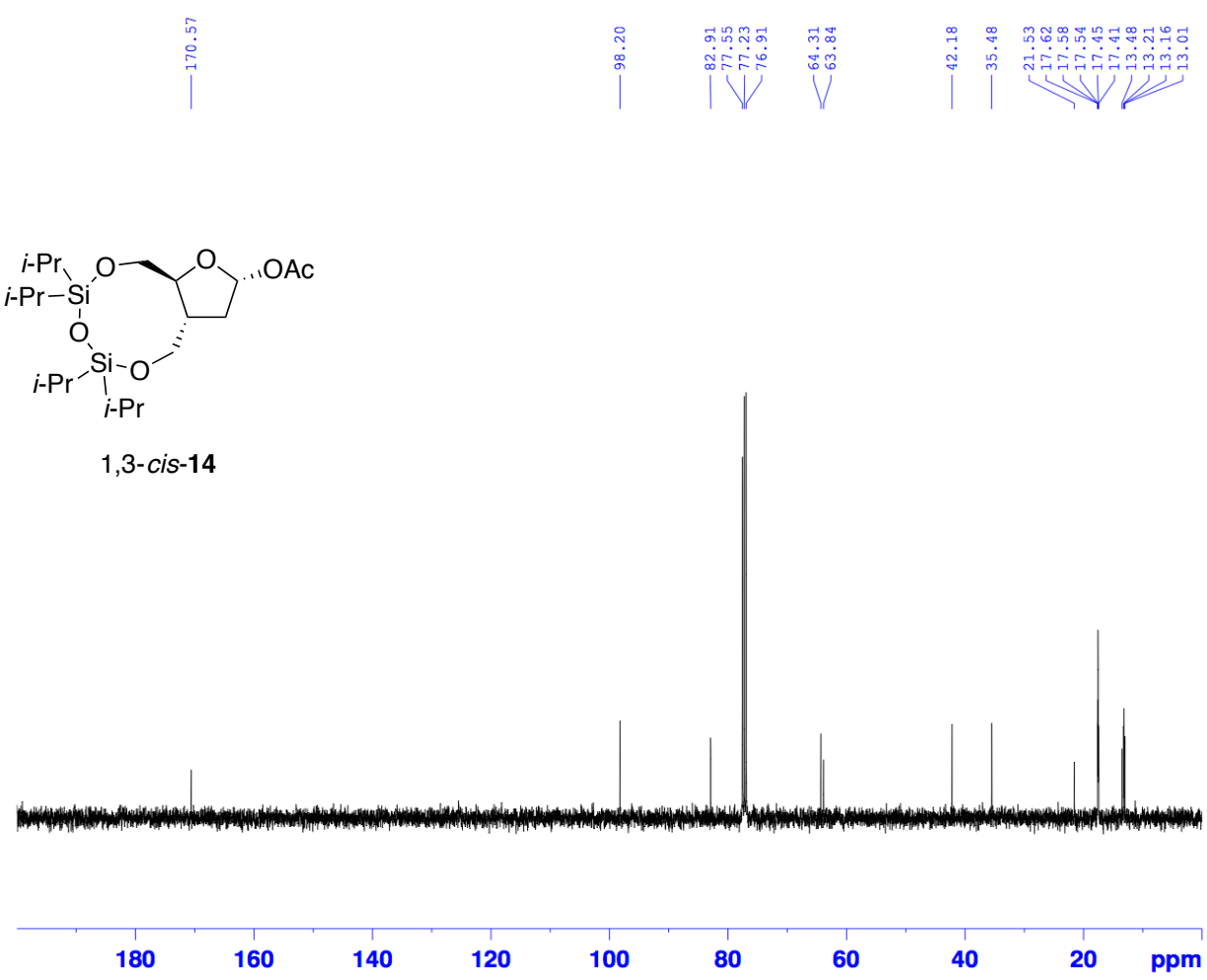
1,3-*cis*-**14**

```
Current Data Parameters
NAME         VTT-II-164-A
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111006
Time                21.54
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                    90.5
DW                  60.400 usec
DE                   6.50 usec
TE                  298.3 K
D1              2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.10 usec
PL1                 -5.00 dB
PL1W          31.77312851 W
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF            400.1300055 MHz
WDW                    no
SSB          0
LB           0 Hz
GB           0
PC                   1.00
```



1,3-*cis*-**14**

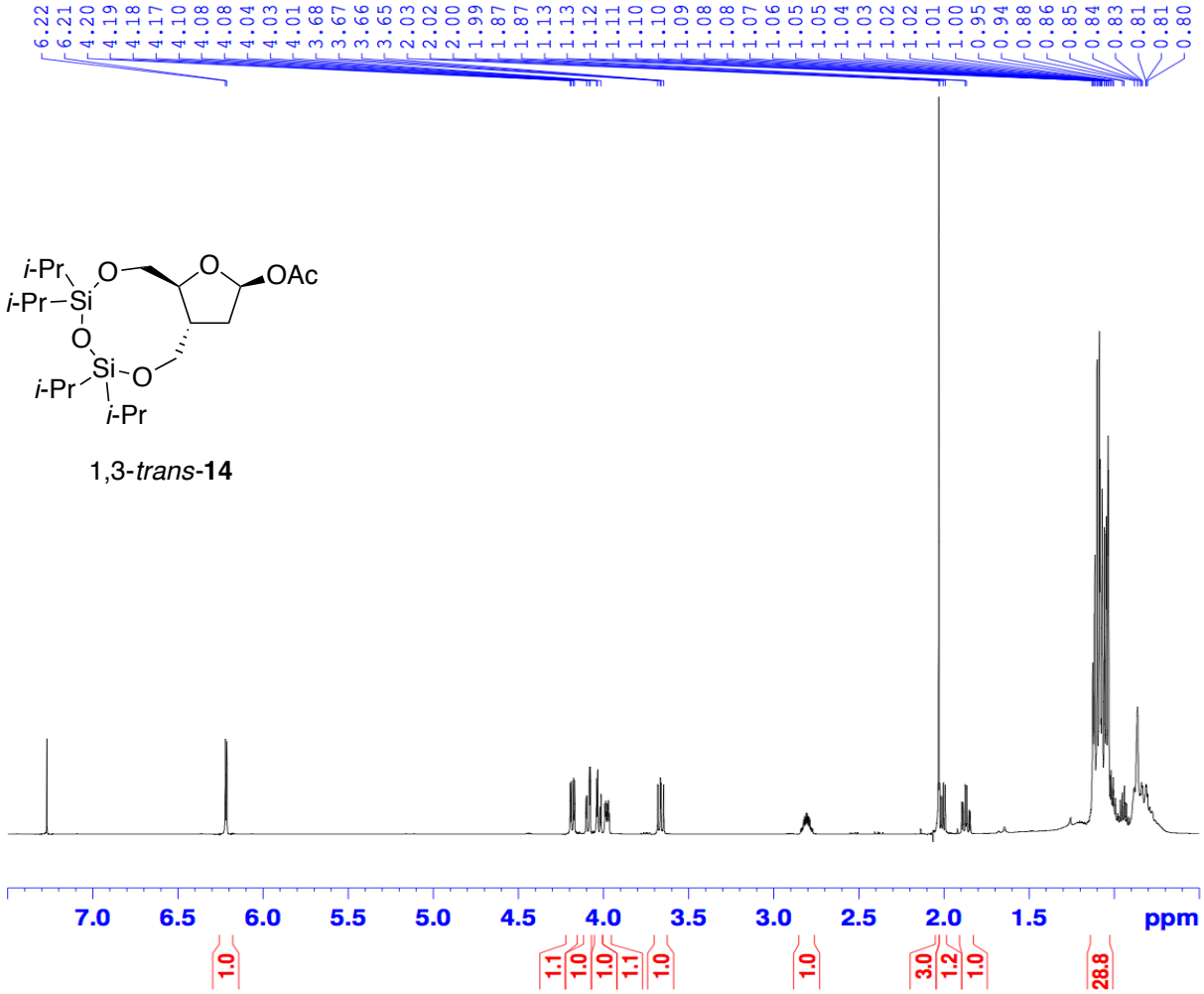
```
Current Data Parameters
NAME        VTT-II-164-A
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111006
Time               21.55
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    0
SWH           39920.160 Hz
FIDRES         0.609133 Hz
AQ             0.8208884 sec
RG               20642.5
DW                12.525 usec
DE                 6.50 usec
TE                298.3 K
D1            2.50000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  6.50 usec
PL1                -4.00 dB
PL1W        25.23829460 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              70.00 usec
PL2                -5.00 dB
PL12               13.73 dB
PL13               13.73 dB
PL2W        31.77312851 W
PL12W        0.42565724 W
PL13W        0.42565724 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127479 MHz
WDW                   EM
SSB       0
LB                 1.20 Hz
GB        0
PC                 1.40
```



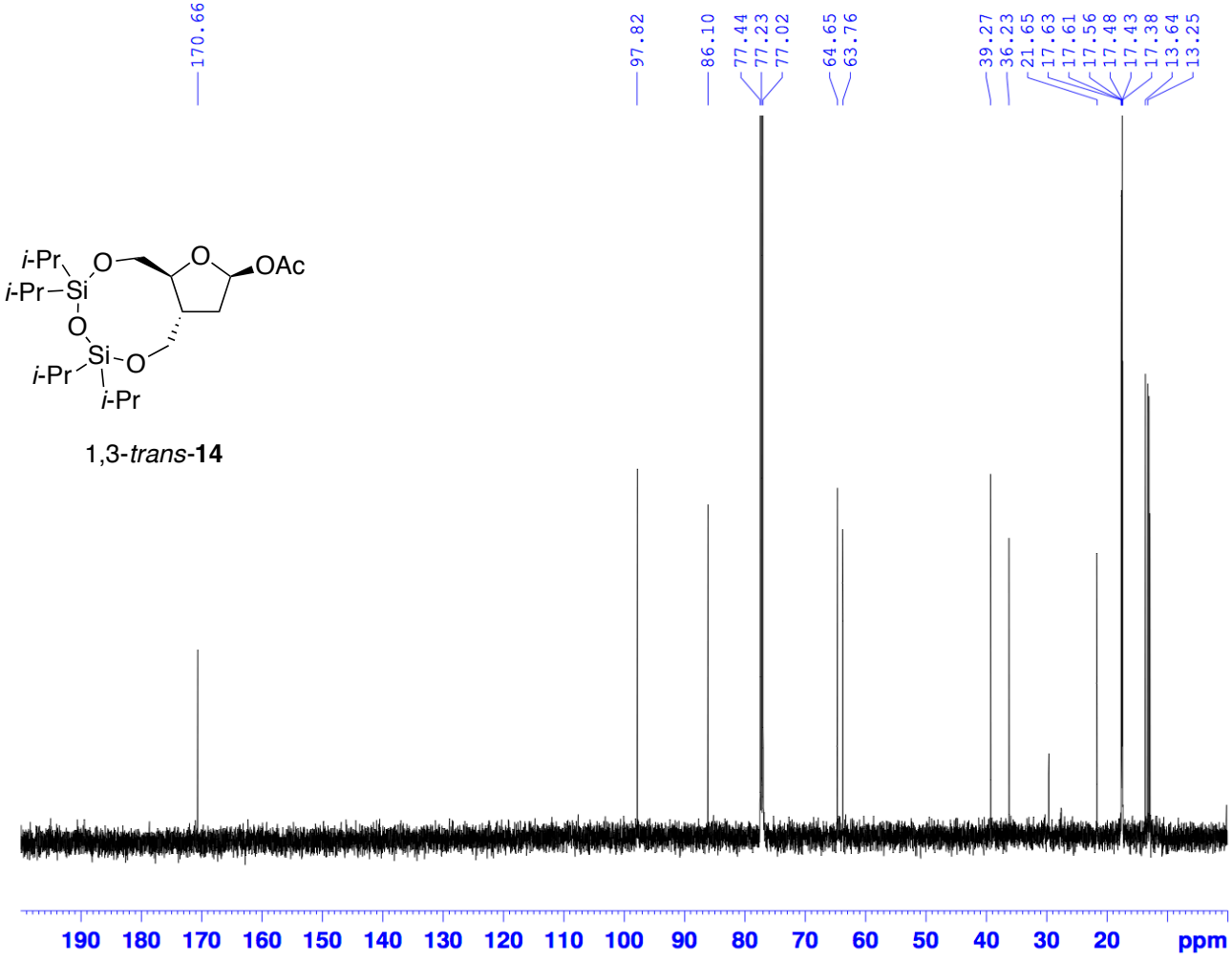
1,3-*trans*-**14**

```
Current Data Parameters
NAME          VTT-IV-31-B
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20121012
Time                11.08
INSTRUM             spect
PROBHD   5 mm PAQXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                  33.59
DW                 40.533 usec
DE                  6.50 usec
TE                 298.2 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.79 usec
PLW1          9.30000019 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF           600.1900090 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



1,3-*trans*-**14**

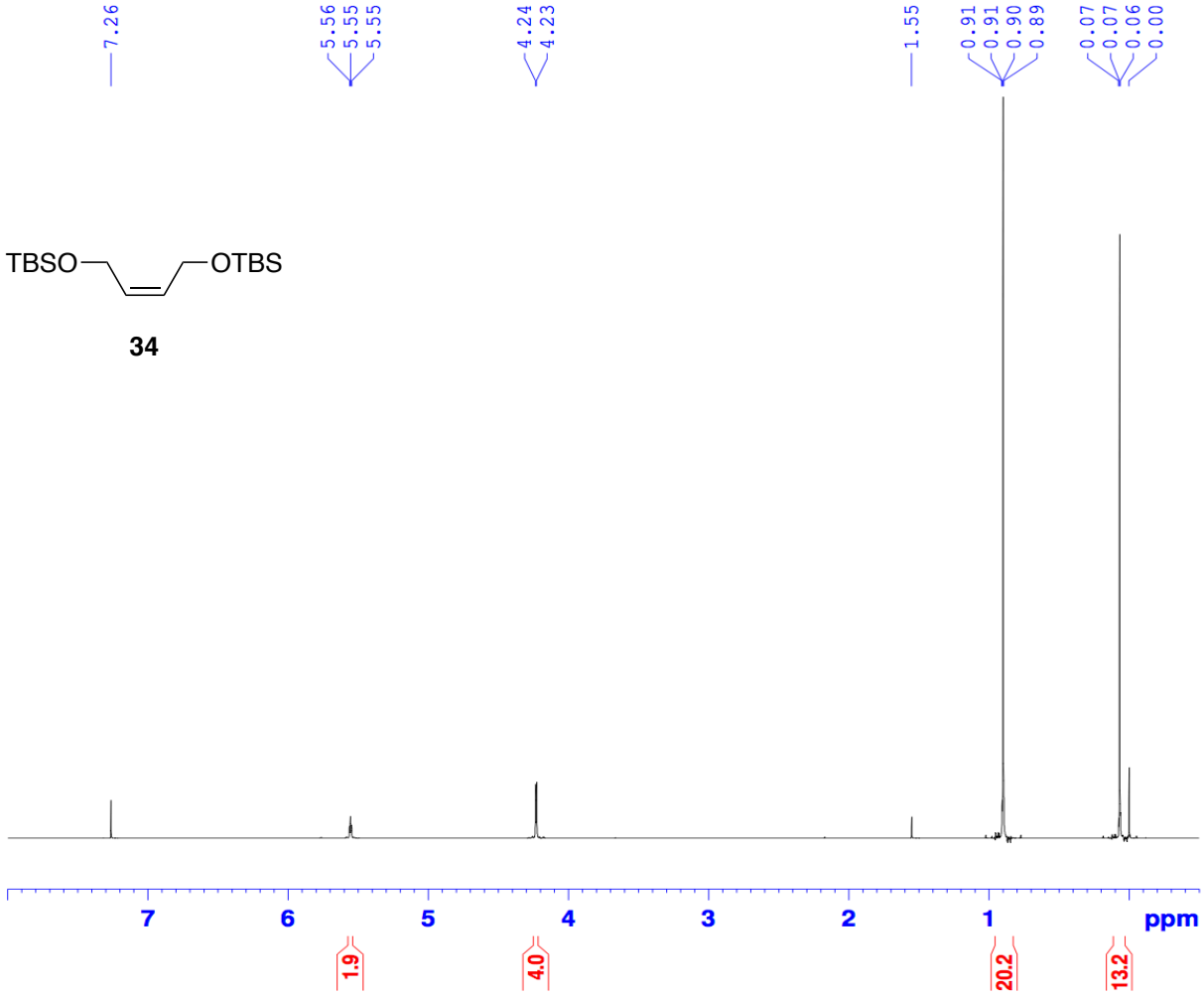
```
Current Data Parameters
NAME          VTT-IV-31-B
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20121012
Time                13.07
INSTRUM             spect
PROBHD   5 mm PAQXI 1H/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   128
DS                     0
SWH            36057.691 Hz
FIDRES          0.550197 Hz
AQ             0.9088159 sec
RG                 184.65
DW                 13.867 usec
DE                  6.50 usec
TE                 298.2 K
D1           2.50000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 15.00 usec
PLW1       106.00000000 W
SFO1        150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              70.00 usec
PLW2         9.30000019 W
PLW12        0.18190999 W
PLW13        0.08913500 W
SFO2        600.1924008 MHz

F2 - Processing parameters
SI                 32768
SF          150.9178644 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



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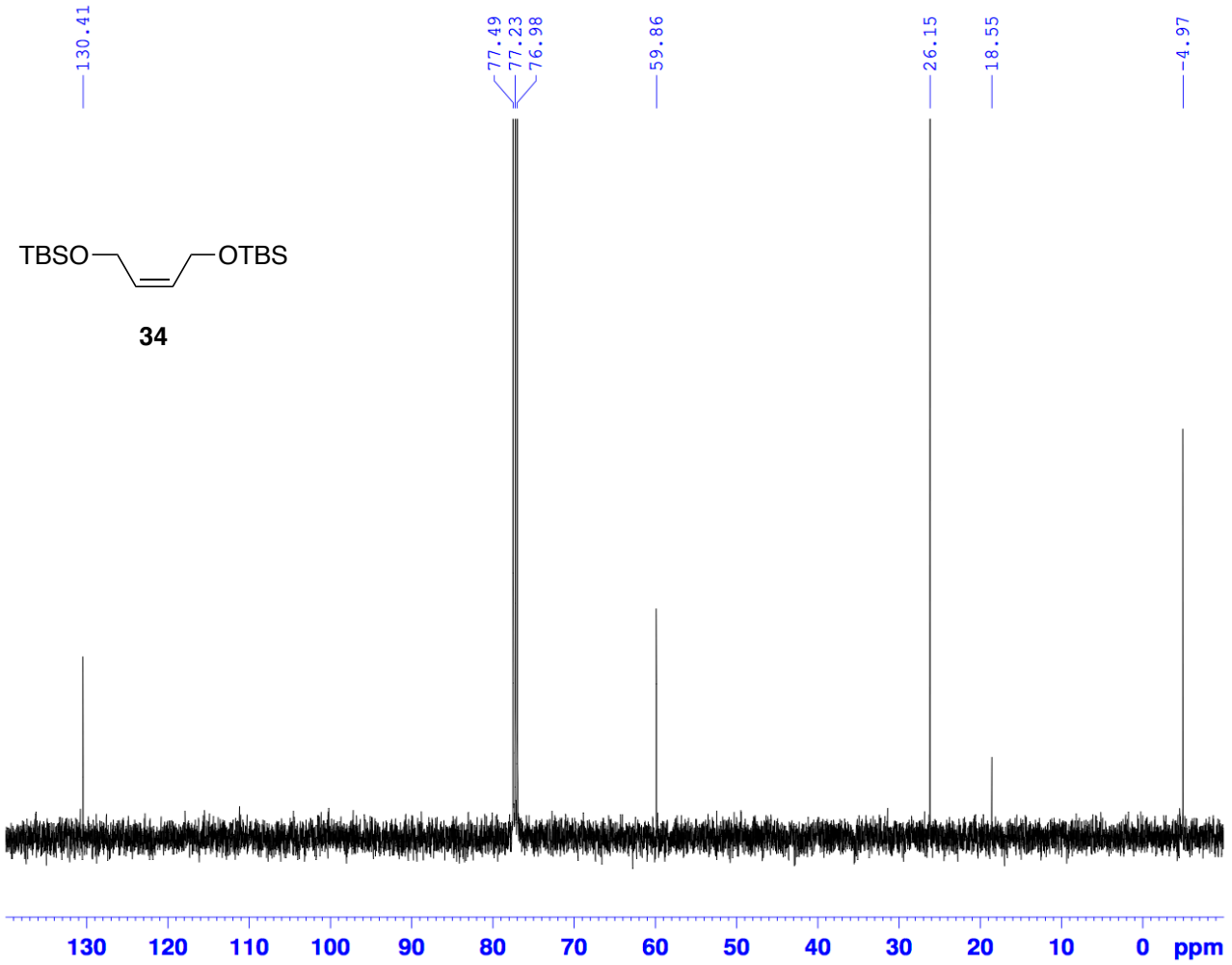
```
Current Data Parameters
NAME          VTT-IV-34-A
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20121107
Time                13.54
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719923 sec
RG                  228.1
DW                 48.400 usec
DE                  6.50 usec
TE                 298.2 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.20 usec
PL1                -3.00 dB
PL1W        37.58904266 W
SFO1        500.2020889 MHz

F2 - Processing parameters
SI                  32768
SF          500.1990143 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



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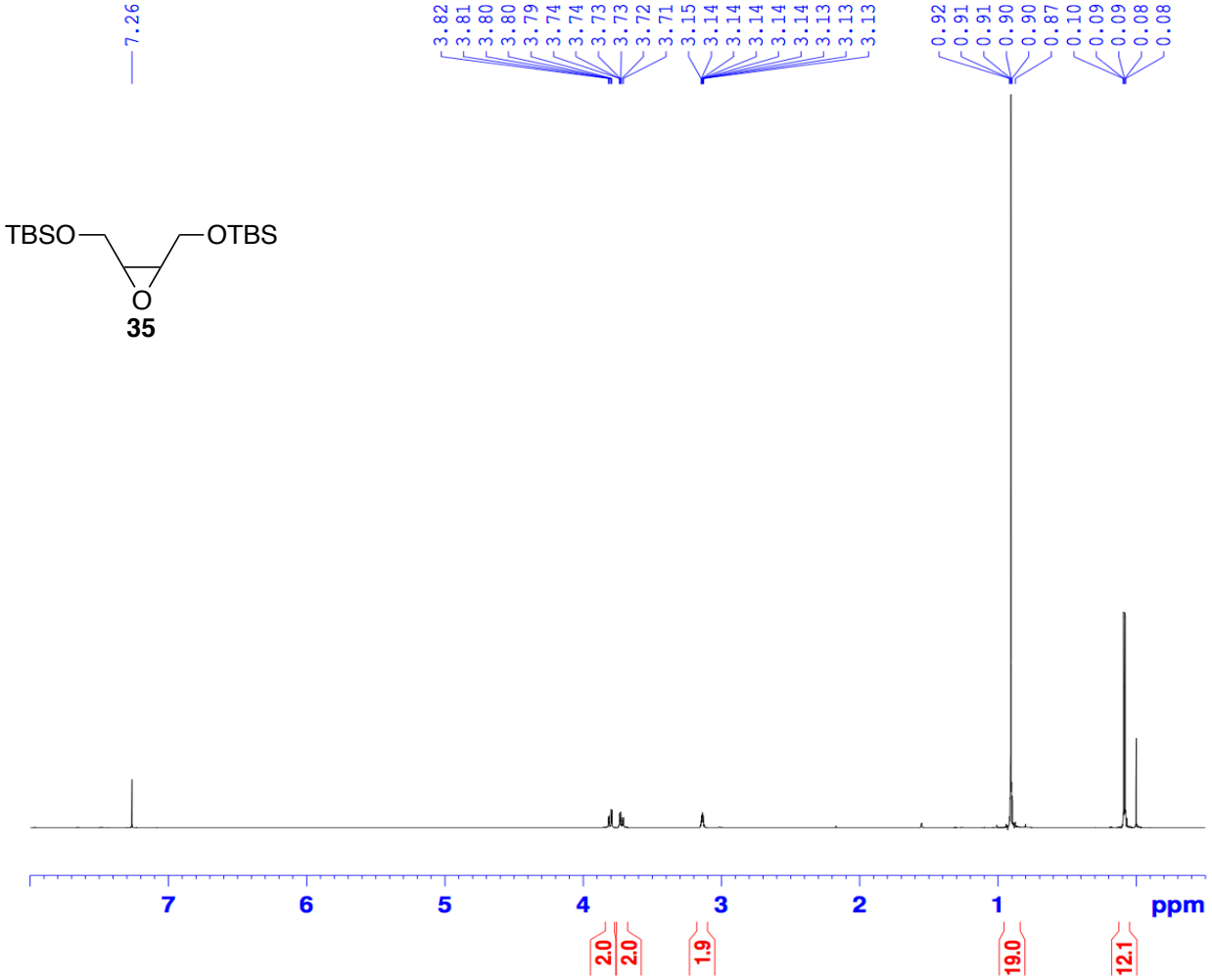
```
Current Data Parameters
NAME         VTT-IV-34-A
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121107
Time               13.57
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   98
DS                    0
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ            1.0912244 sec
RG                32768
DW               16.650 usec
DE                6.50 usec
TE               298.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.75 usec
PL1                2.00 dB
PL1W       49.29017639 W
SFO1       125.7877161 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PL2               -3.00 dB
PL12              13.50 dB
PL13              13.50 dB
PL2W       37.58904266 W
PL12W       0.84151381 W
PL13W       0.84151381 W
SFO2       500.2010008 MHz

F2 - Processing parameters
SI                32768
SF         125.7751116 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

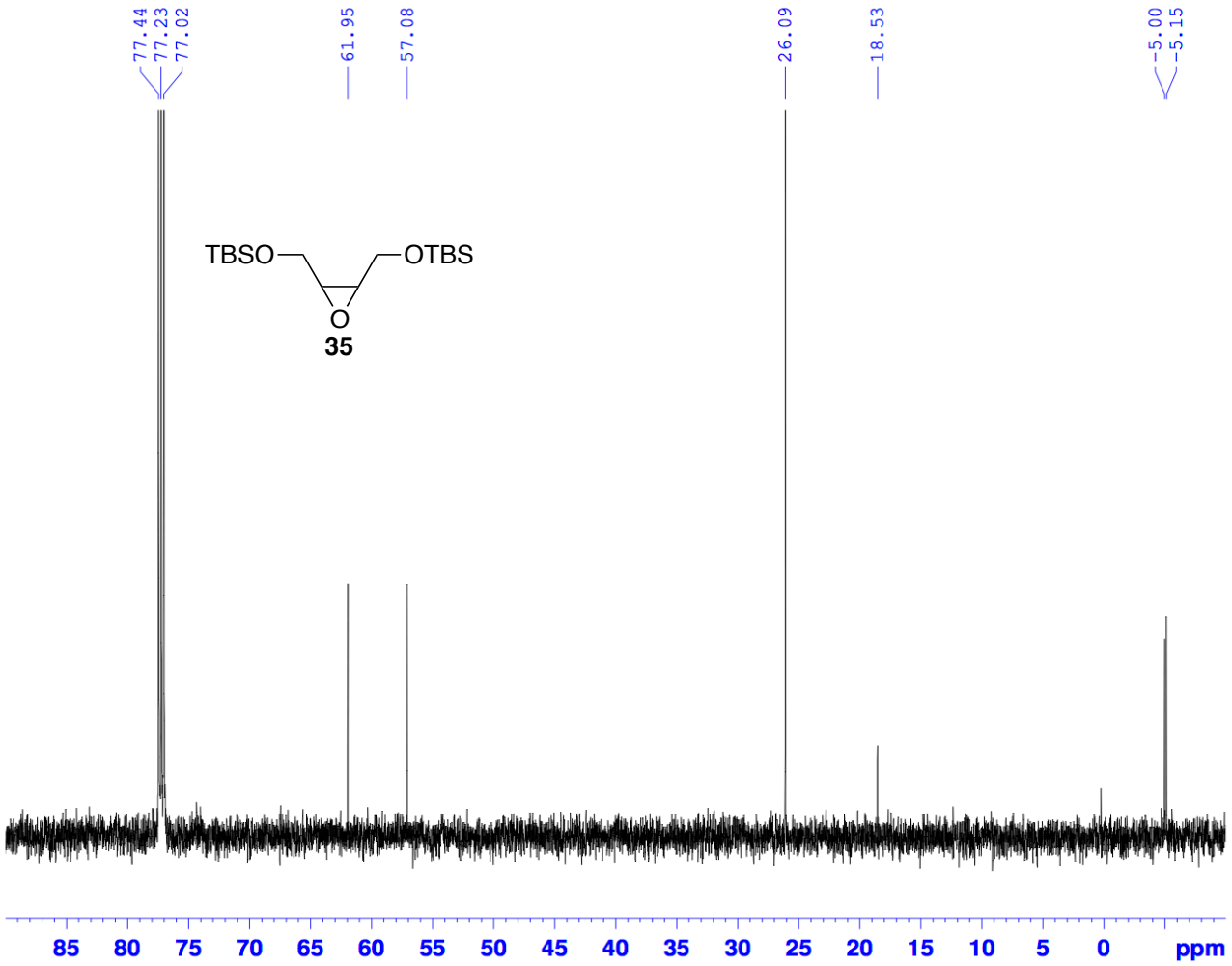



```
Current Data Parameters
NAME          VTT-IV-36-A
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20121109
Time                11.36
INSTRUM             spect
PROBHD   5 mm PAQXI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                 184.65
DW                 40.533 usec
DE                   6.50 usec
TE                 298.2 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.79 usec
PLW1          9.30000019 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                  65536
SF           600.1900132 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



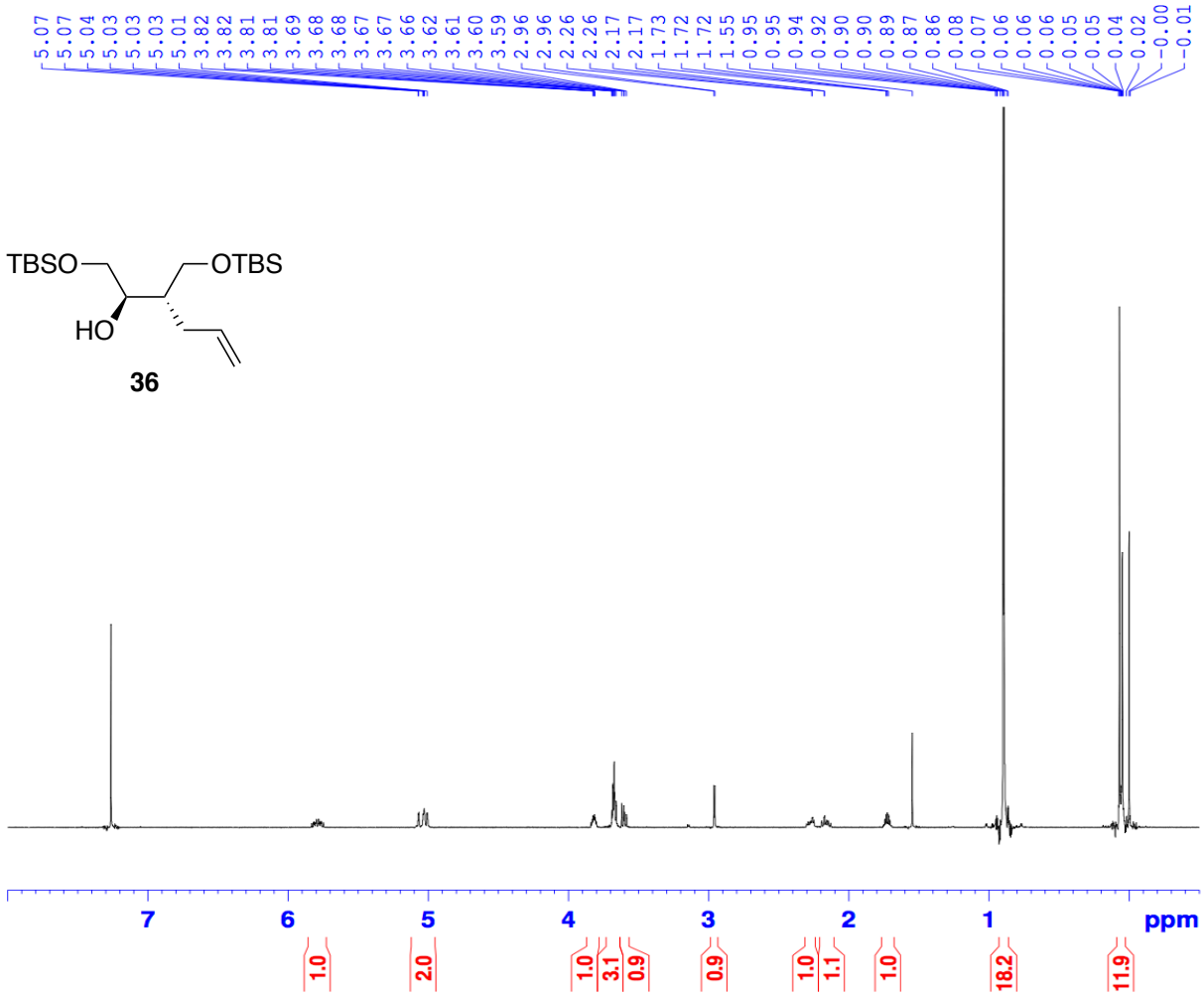

```
Current Data Parameters
NAME         VTT-IV-36-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121109
Time               11.38
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG                184.65
DW                13.867 usec
DE                 6.50 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.00 usec
PLW1       106.00000000 W
SFO1        150.9216024 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PLW2         9.30000019 W
PLW12        0.18190999 W
PLW13        0.08913500 W
SFO2        600.1924008 MHz

F2 - Processing parameters
SI                32768
SF          150.9178628 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

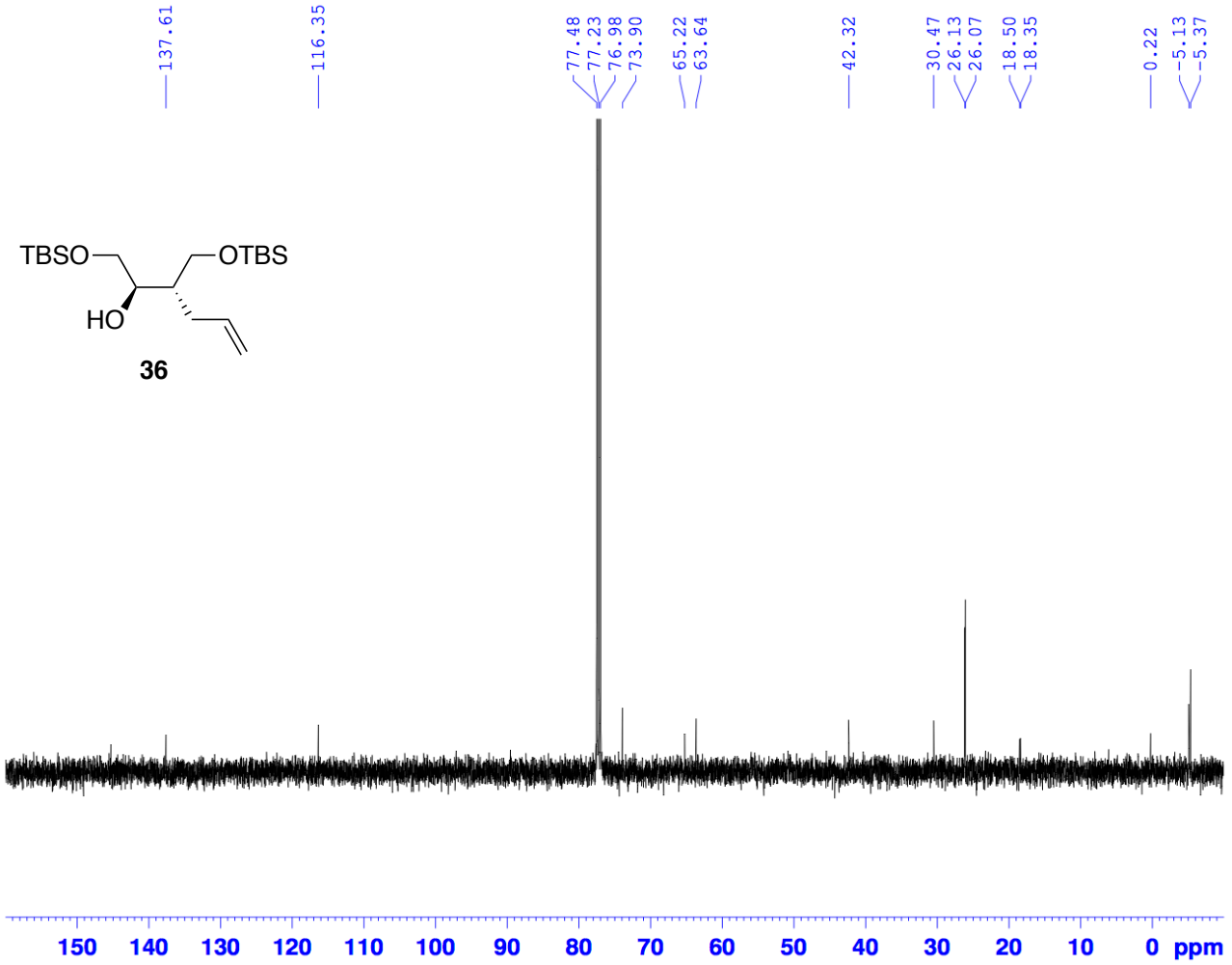



```
Current Data Parameters
NAME          VTT-IV-40-A
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20121115
Time                11.47
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1719923 sec
RG                    512
DW               48.400 usec
DE                6.50 usec
TE                298.1 K
D1            1.50000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.20 usec
PL1                -3.00 dB
PL1W        37.58904266 W
SFO1        500.2020889 MHz

F2 - Processing parameters
SI                  32768
SF          500.1990141 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



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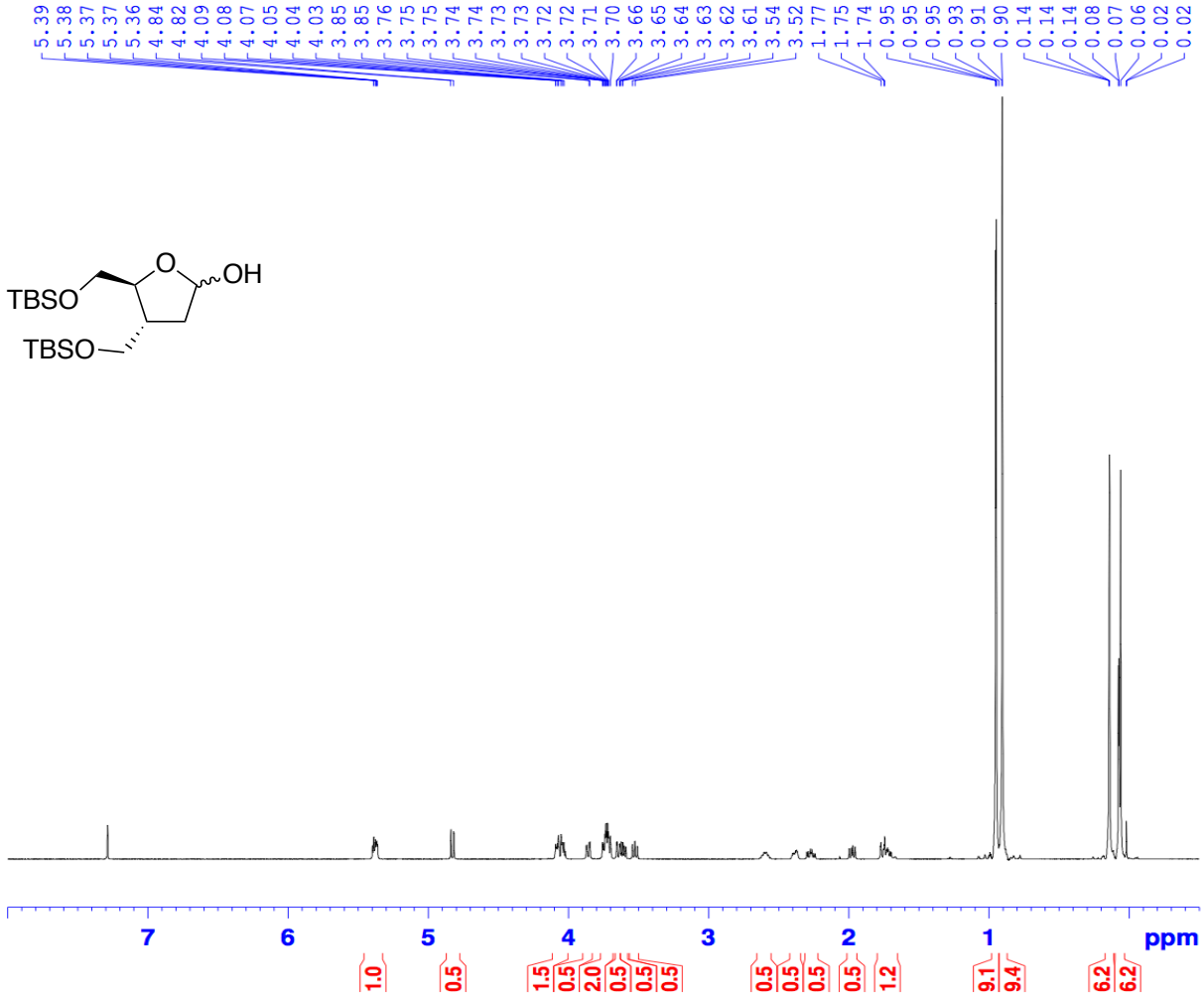
```
Current Data Parameters
NAME         VTT-IV-40-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121115
Time               11.53
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    0
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ            1.0912244 sec
RG                32768
DW               16.650 usec
DE                6.50 usec
TE               298.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.75 usec
PL1                 2.00 dB
PL1W        49.29017639 W
SFO1        125.7877161 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              70.00 usec
PL2                -3.00 dB
PL12               13.50 dB
PL13               13.50 dB
PL2W        37.58904266 W
PL12W        0.84151381 W
PL13W        0.84151381 W
SFO2        500.2010008 MHz

F2 - Processing parameters
SI                32768
SF          125.7751122 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

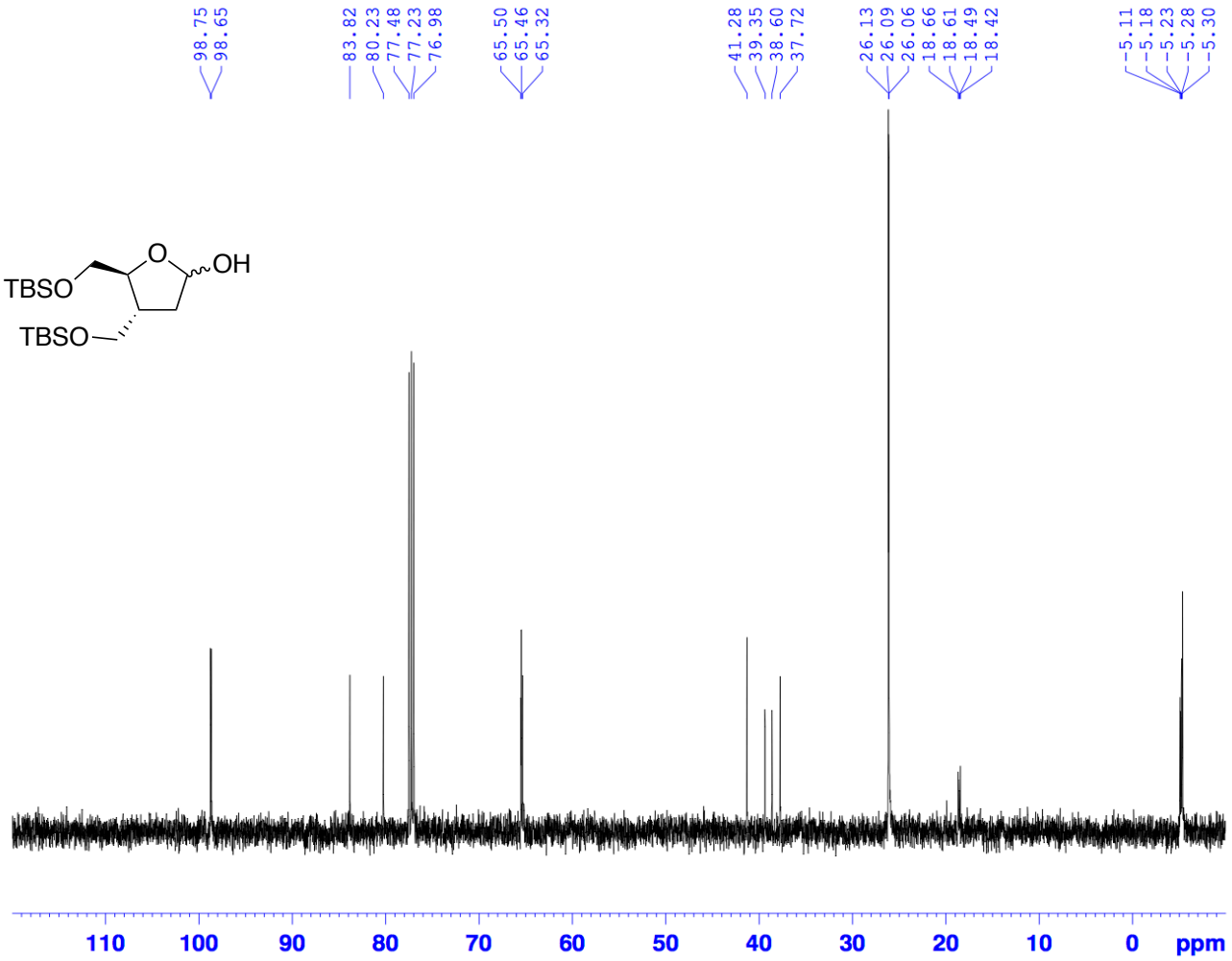



```
Current Data Parameters
NAME        VTT-II-177-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111019
Time               13.05
INSTRUM            spect
PROBHD   5 mm TXI 1H-13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1719923 sec
RG                  90.5
DW                48.400 usec
DE                 6.50 usec
TE                297.9 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.55 usec
PL1       0 dB
PL1W        18.83914757 W
SFO1        500.2020889 MHz

F2 - Processing parameters
SI                 32768
SF          500.1990000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



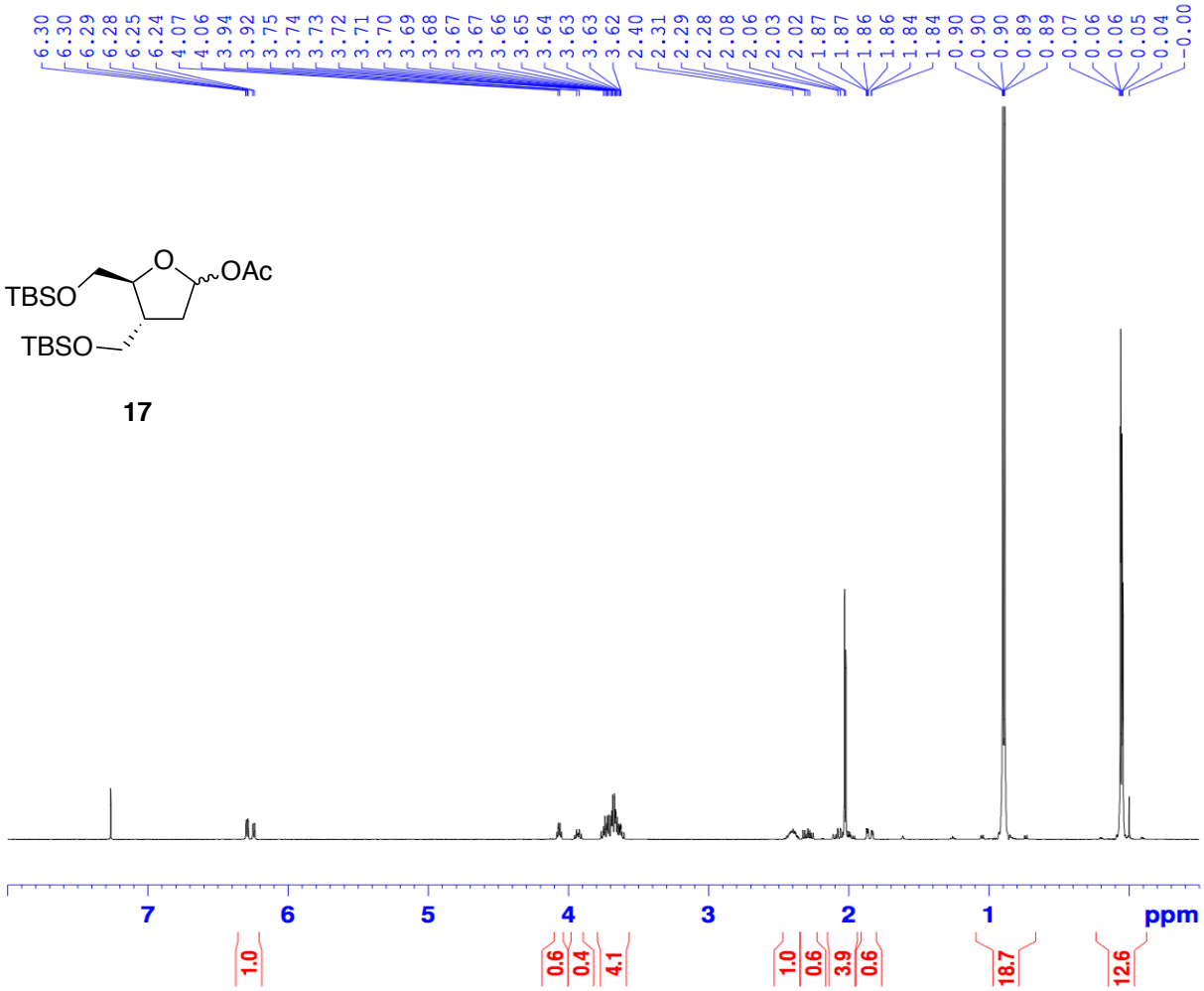

```
Current Data Parameters
NAME       VTT-II-177-A
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20111019
Time              13.07
INSTRUM           spect
PROBHD   5 mm TXI 1H-13
PULPROG         zgpg30
TD              65536
SOLVENT          CDCl3
NS                 104
DS                   4
SWH          30030.029 Hz
FIDRES         0.458222 Hz
AQ           1.0912244 sec
RG               32768
DW              16.650 usec
DE               6.50 usec
TE              298.1 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                13.00 usec
PL1               -4.00 dB
PL1W       196.22773743 W
SFO1        125.7877161 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             70.00 usec
PL2       0 dB
PL12              19.34 dB
PL13             120.00 dB
PL2W       18.83914757 W
PL12W       0.21931142 W
PL13W     0 W
SFO2        500.2010008 MHz

F2 - Processing parameters
SI               32768
SF          125.7751134 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



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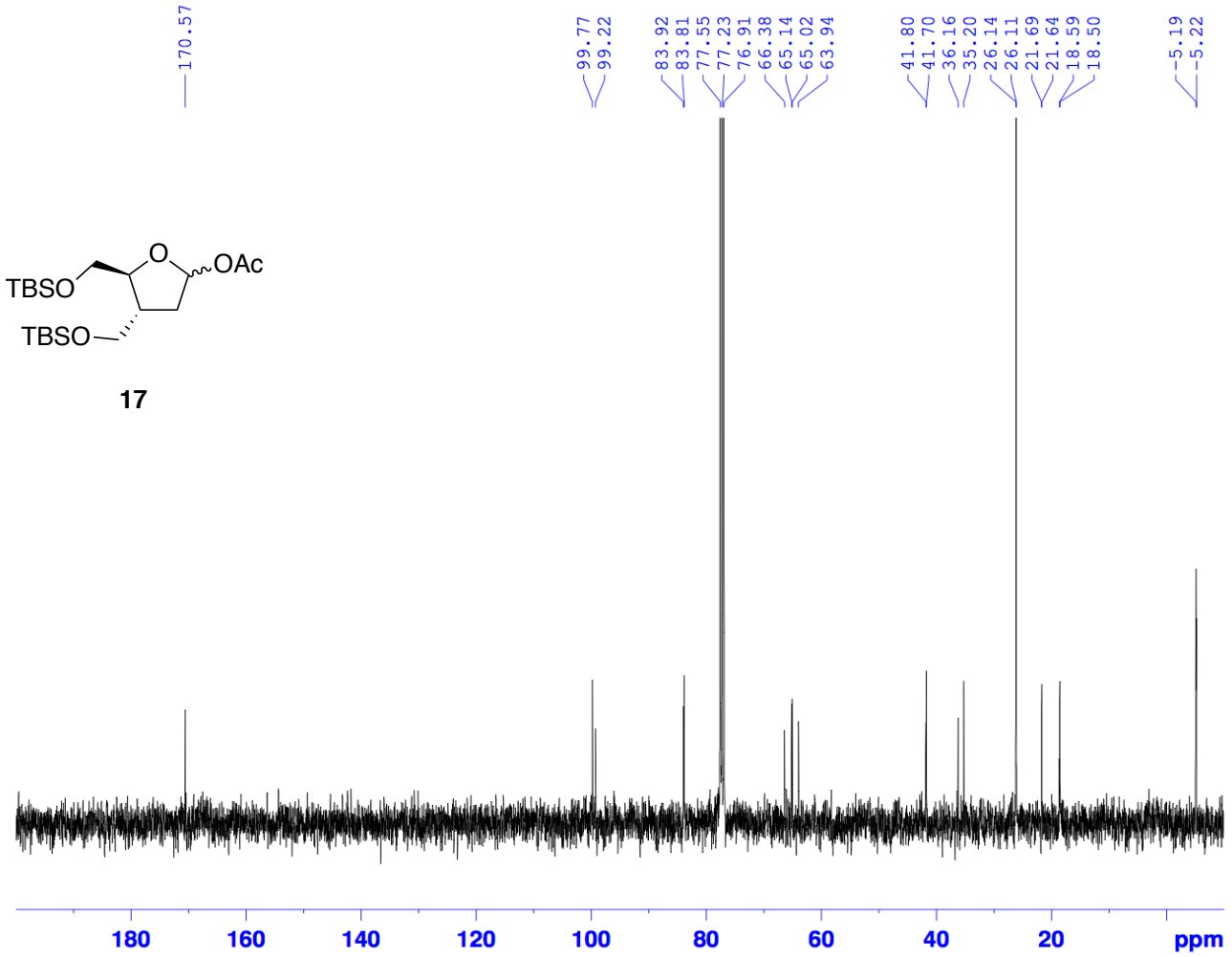
```
Current Data Parameters
NAME        VTT-II-180-A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111022
Time               23.24
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  90.5
DW                60.400 usec
DE                 6.50 usec
TE                298.2 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.10 usec
PL1               -5.00 dB
PL1W        31.77312851 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300073 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



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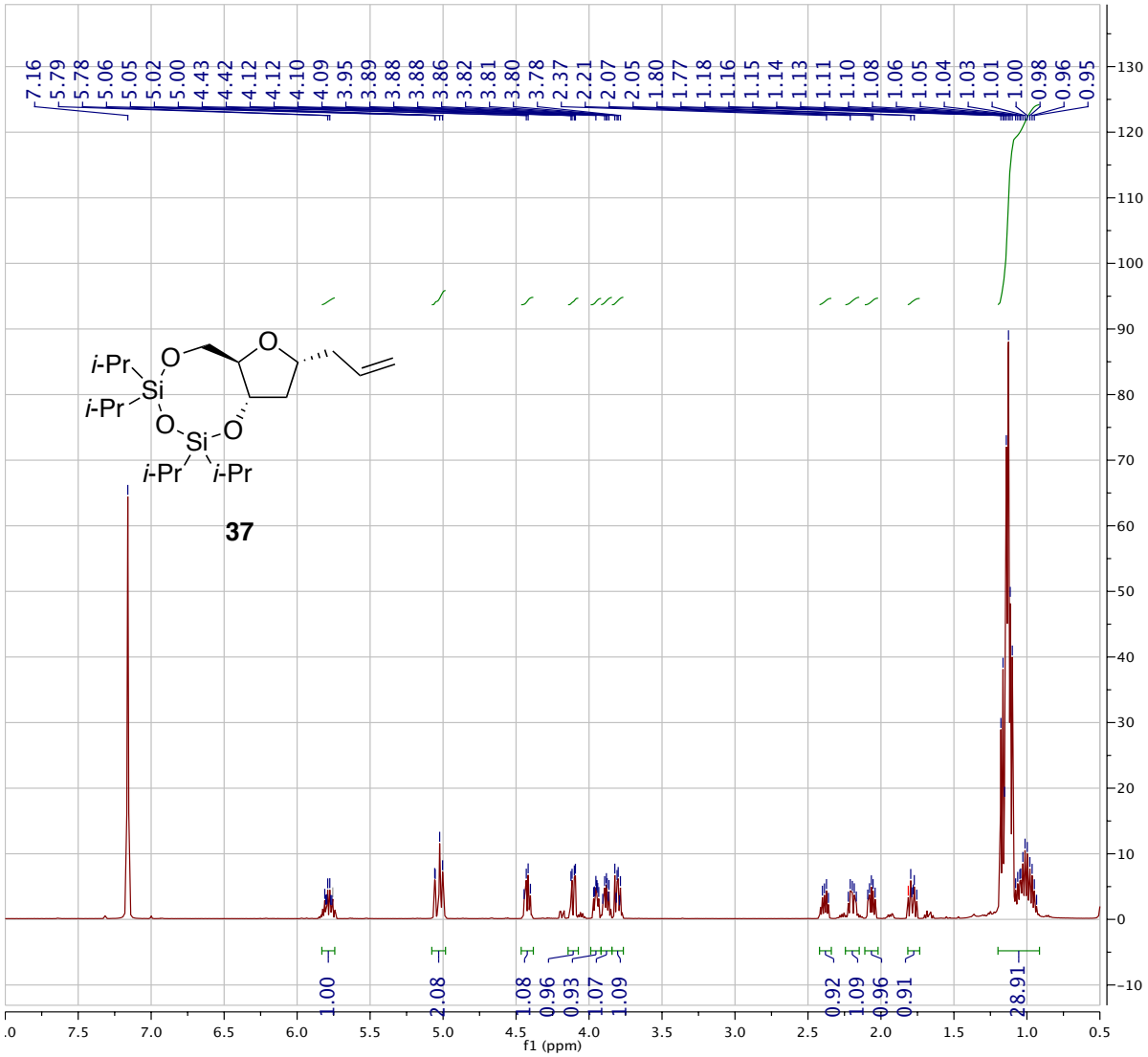
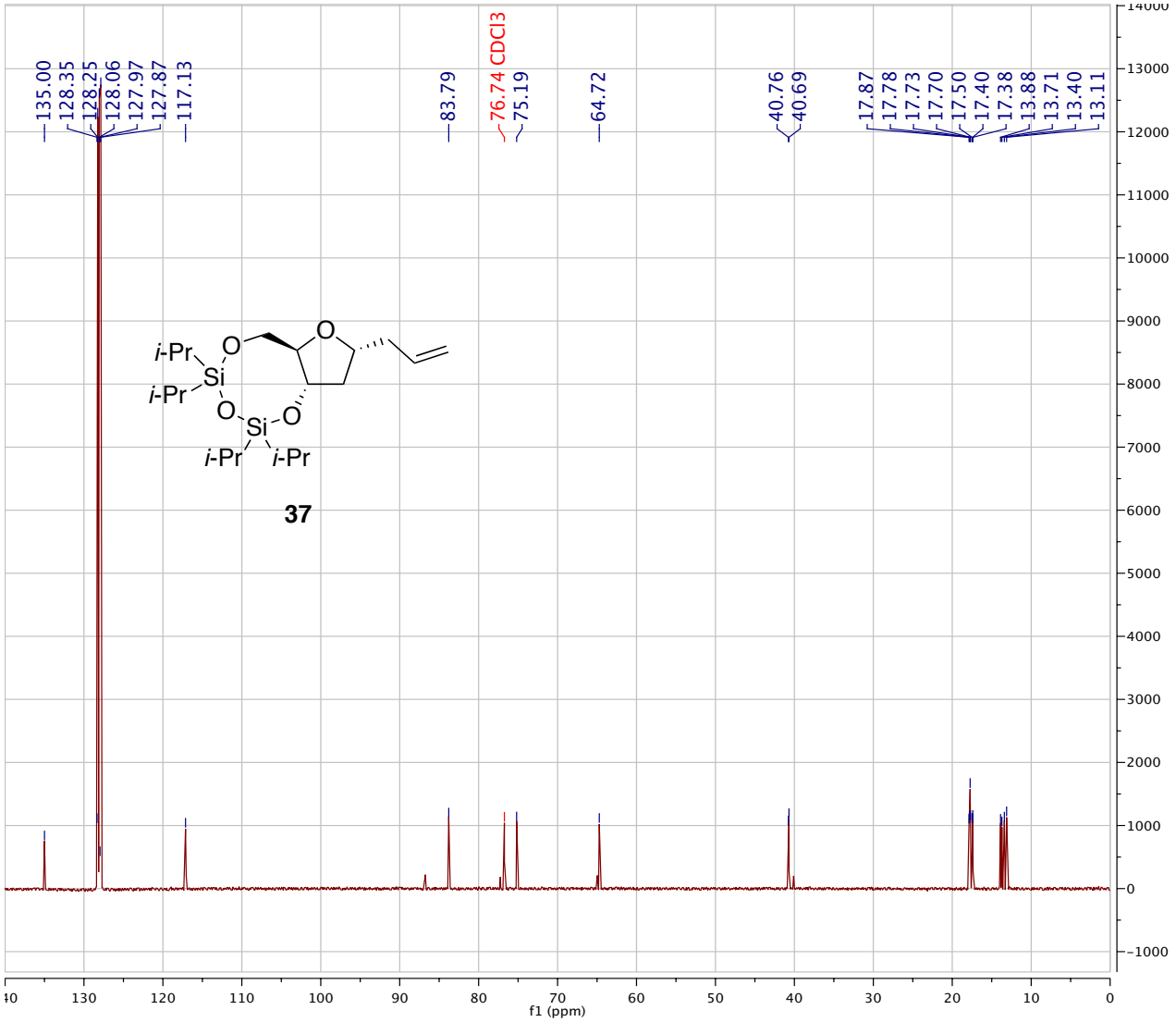
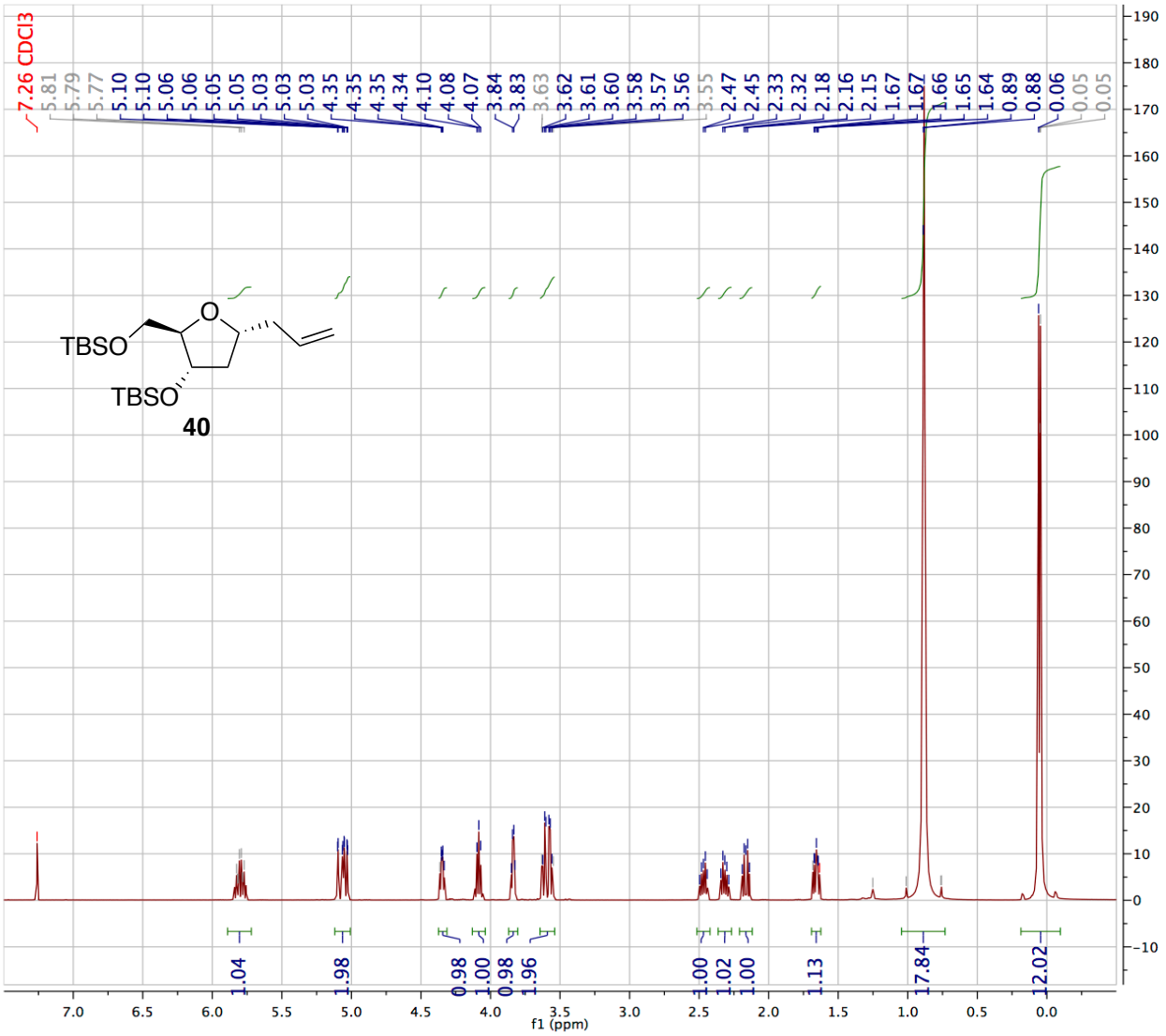
```
Current Data Parameters
NAME        VTT-II-180-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111022
Time               23.27
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG              20642.5
DW                20.850 usec
DE                 6.50 usec
TE                298.2 K
D1           2.50000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  6.50 usec
PL1                -4.00 dB
PL1W        25.23829460 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              70.00 usec
PL2                -5.00 dB
PL12               13.73 dB
PL13               13.73 dB
PL2W        31.77312851 W
PL12W        0.42565724 W
PL13W        0.42565724 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127472 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```



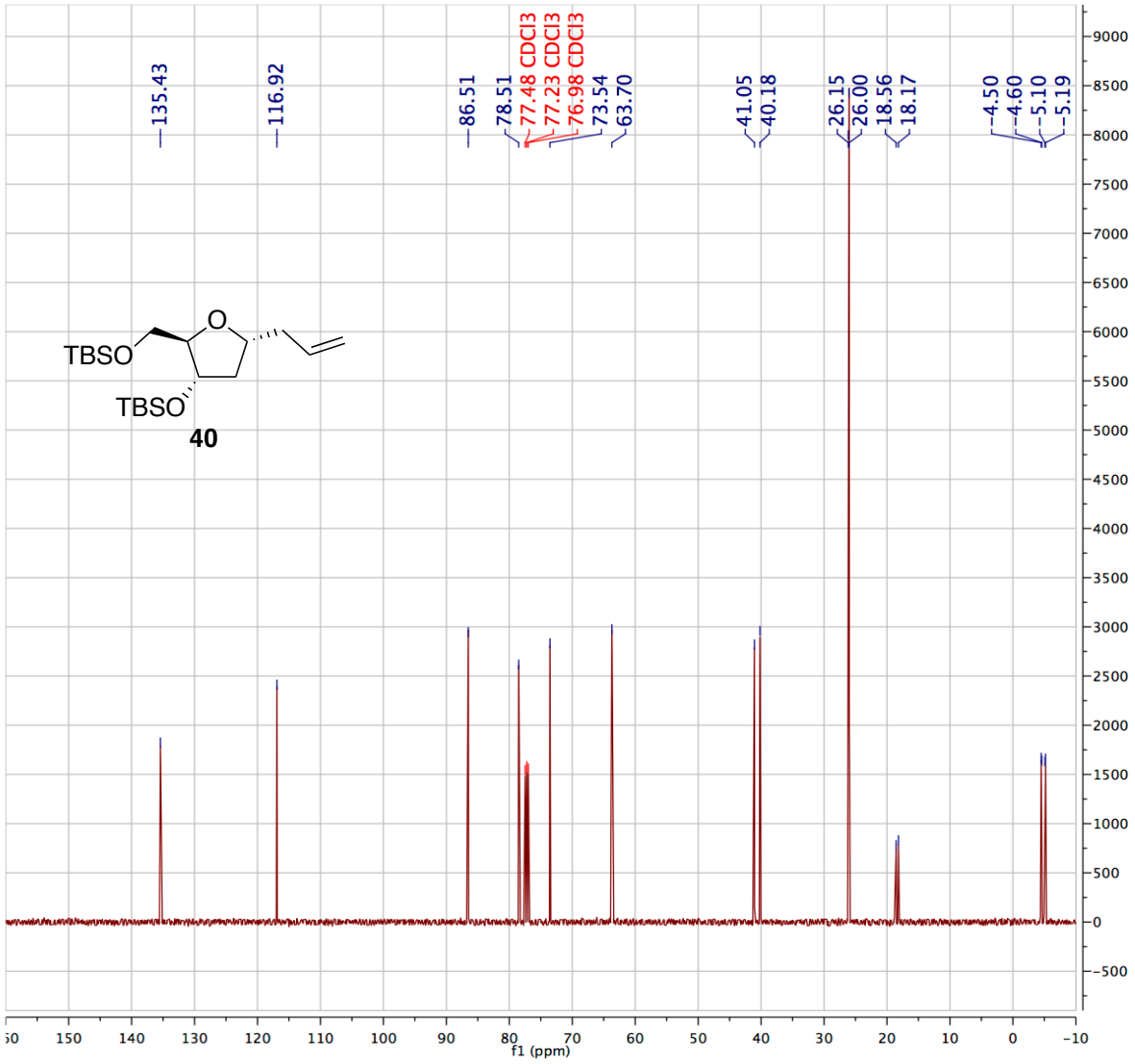
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	8
7	Receiver Gain	4
8	Relaxation Delay	0.1000
9	Pulse Width	7.5000
10	Spectrometer Frequency	500.22
11	Spectral Width	8012.8
12	Lowest Frequency	-505.0
13	Nucleus	1H
14	Acquired Size	40864
15	Spectral Size	131072



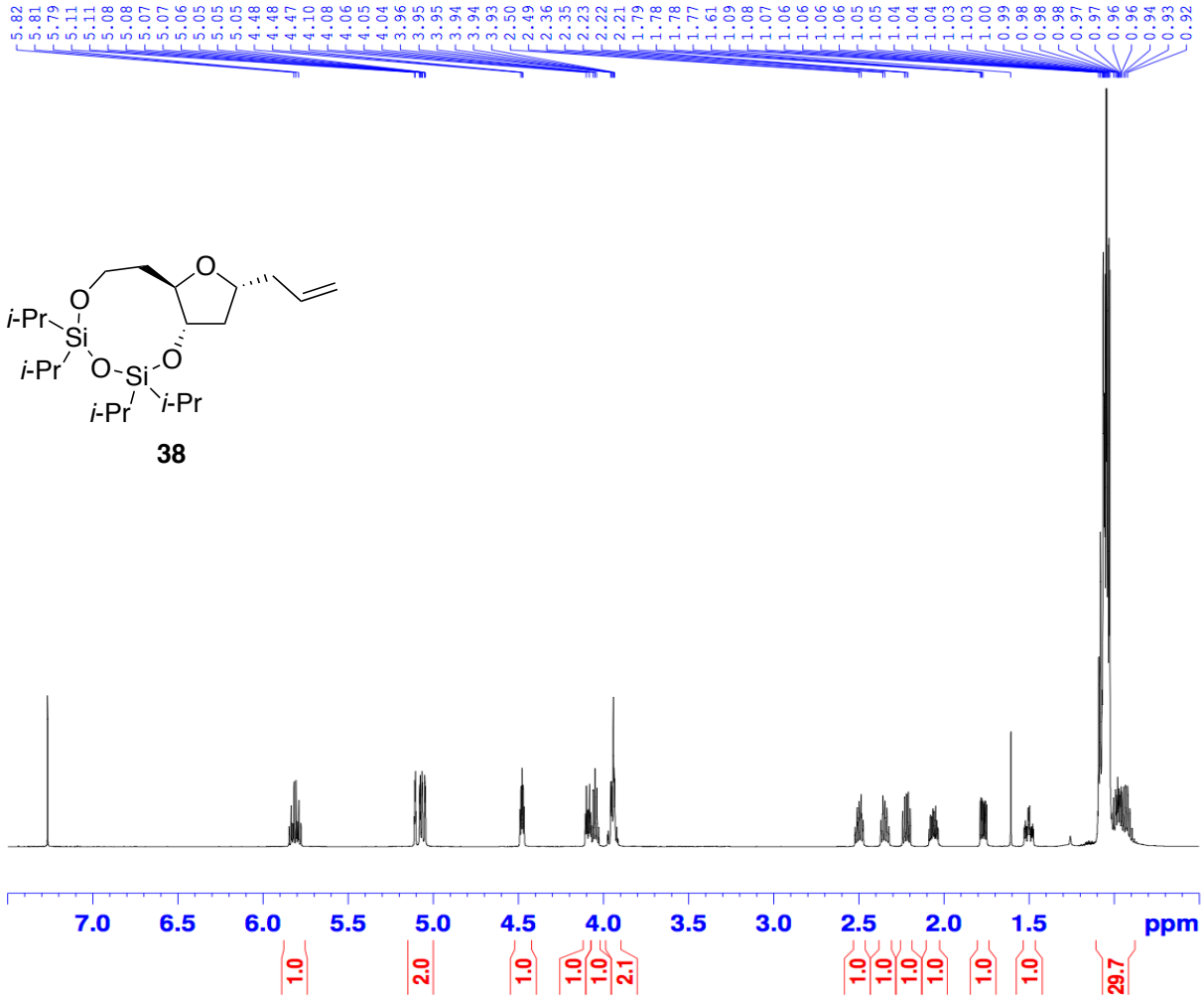
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	SpinEchopg30 gp.prd
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	138
7	Receiver Gain	5161
8	Relaxation Delay	0.2500
9	Pulse Width	15.5000
10	Spectrometer Frequency	125.79
11	Spectral Width	30303.0
12	Lowest Frequency	-1268.5
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	8
7	Receiver Gain	3
8	Relaxation Delay	0.1000
9	Pulse Width	7.5000
10	Spectrometer Frequency	500.22
11	Spectral Width	8012.8
12	Lowest Frequency	-536.3
13	Nucleus	1H
14	Acquired Size	40864
15	Spectral Size	131072

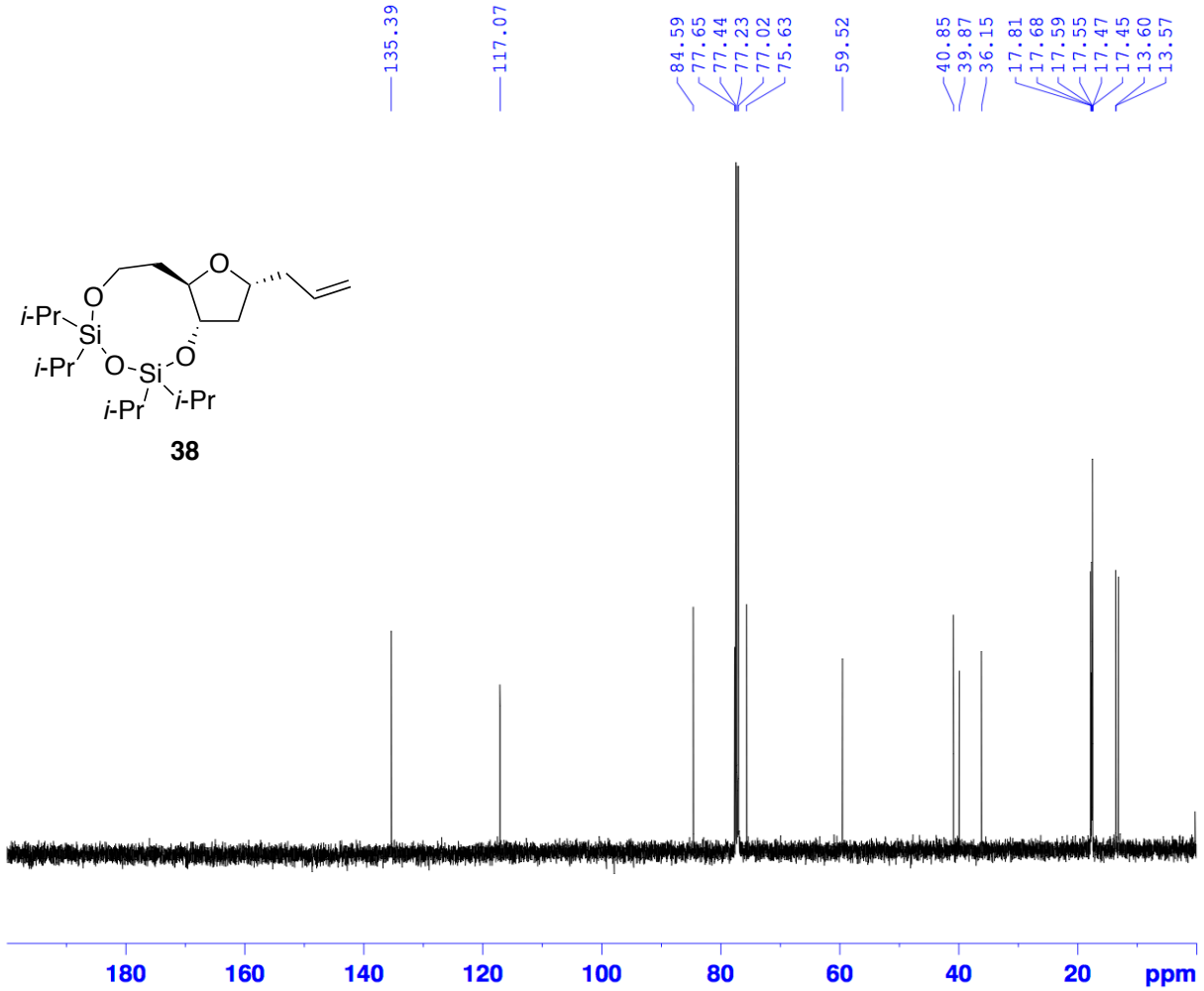


	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	SpinEchopg30gp.prd
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	92
7	Receiver Gain	8192
8	Relaxation Delay	0.2500
9	Pulse Width	15.5000
10	Spectrometer Frequency	125.79
11	Spectral Width	30303.0
12	Lowest Frequency	-1294.0
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



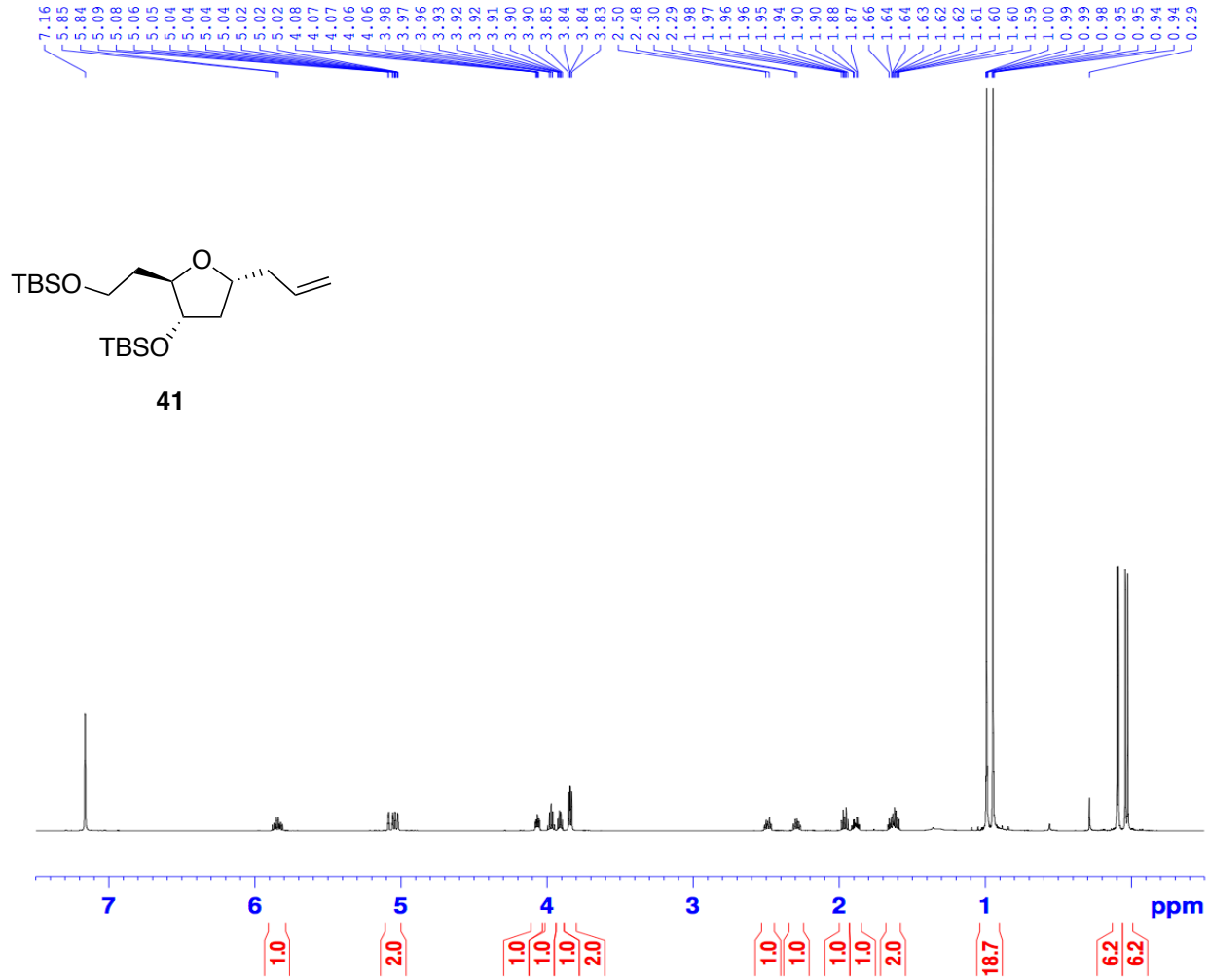
38

```
Current Data Parameters
NAME       VTT-III-153-A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120622
Time               20.19
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                68.89
DW               40.533 usec
DE                6.50 usec
TE                298.2 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.79 usec
PLW1          9.30000019 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF          600.1900118 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



38

```
Current Data Parameters
NAME       VTT-III-153-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120622
Time               20.20
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9088159 sec
RG                184.65
DW                13.867 usec
DE                 6.50 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.00 usec
PLW1       106.00000000 W
SFO1        150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PLW2          9.30000019 W
PLW12         0.18190999 W
PLW13         0.08913500 W
SFO2        600.1924008 MHz

F2 - Processing parameters
SI                32768
SF          150.9178633 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



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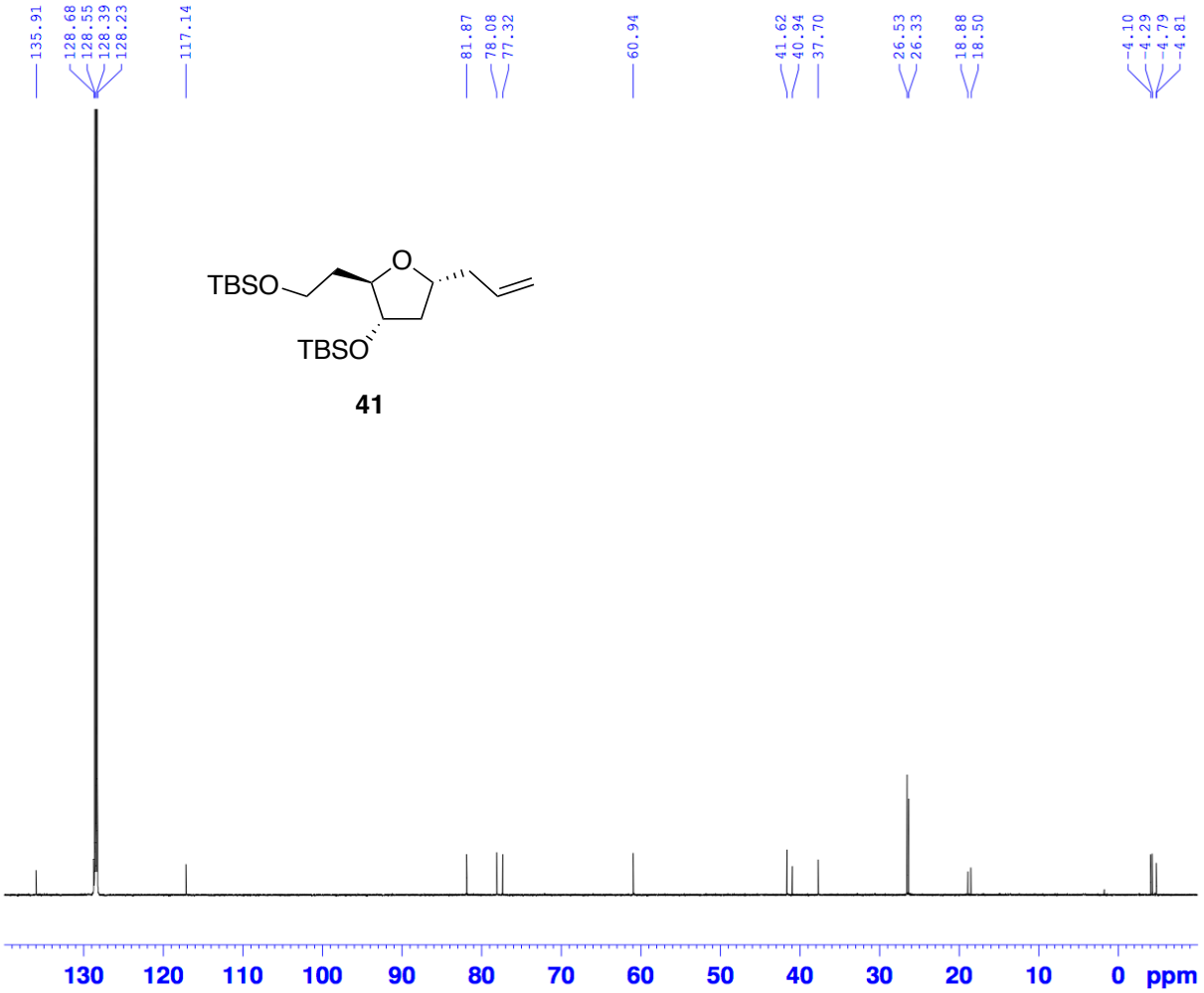
```
Current Data Parameters
NAME       VTT-III-202-A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120817
Time               12.58
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             C6D6
NS                     8
DS                     0
SWH            12335.526 Hz
FIDRES         0.188225 Hz
AQ             2.6564426 sec
RG                 33.59
DW                40.533 usec
DE                 6.50 usec
TE                298.1 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.00 usec
PLW1         26.50000000 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF           600.1899954 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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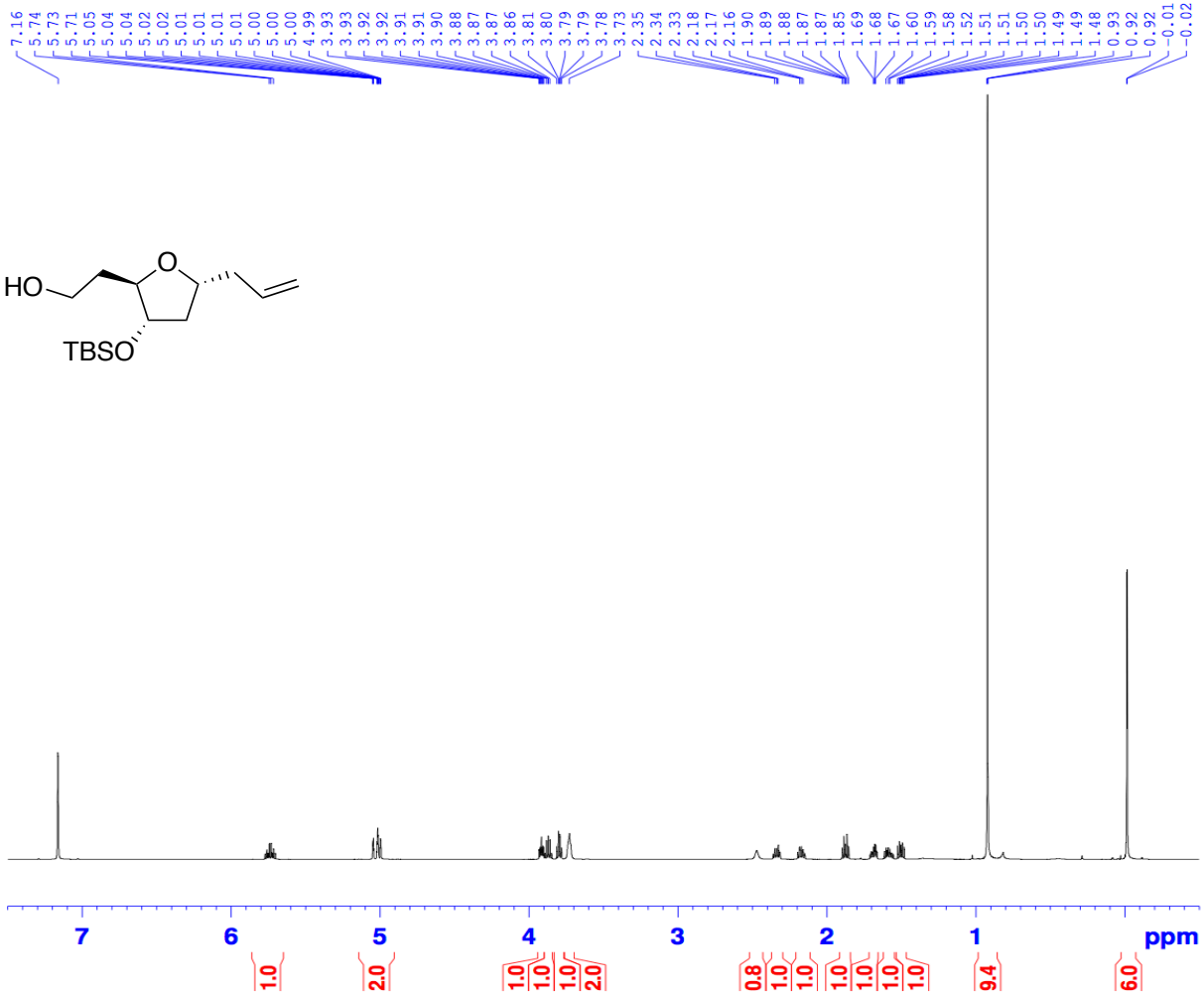
```
Current Data Parameters
NAME       VTT-III-202-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120817
Time               13.03
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT             C6D6
NS                   128
DS                     0
SWH            36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9088159 sec
RG                184.65
DW                13.867 usec
DE                 6.50 usec
TE                298.3 K
D1            2.50000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.65 usec
PLW1        104.00000000 W
SFO1         150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              70.00 usec
PLW2         26.50000000 W
PLW12         0.65438998 W
PLW13         0.32065001 W
SFO2         600.1924008 MHz

F2 - Processing parameters
SI                 32768
SF          150.9177886 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

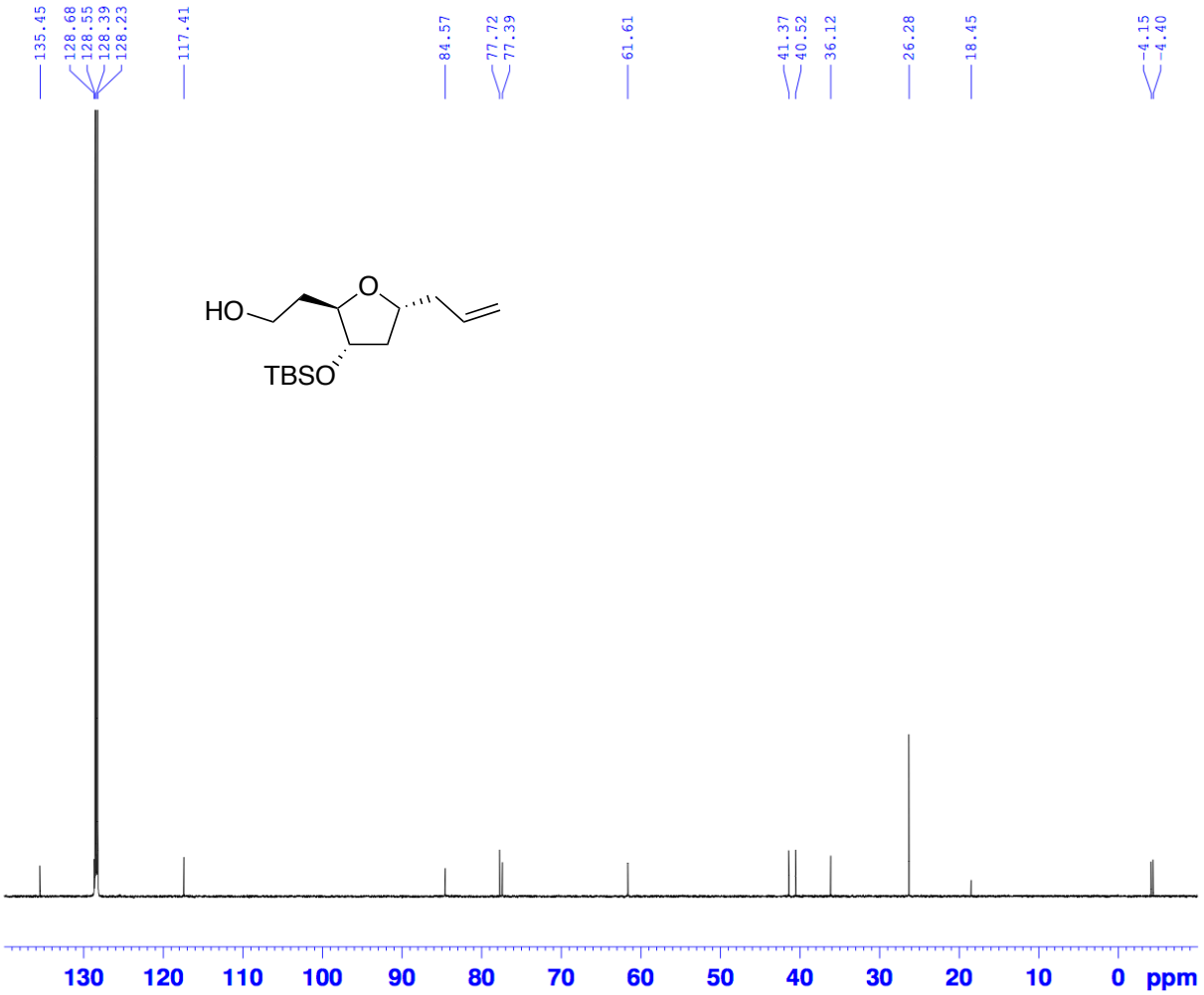



```
Current Data Parameters
NAME       VTT-III-203-B
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120817
Time               13.35
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             C6D6
NS                     8
DS                     0
SWH            12335.526 Hz
FIDRES         0.188225 Hz
AQ             2.6564426 sec
RG                 33.59
DW                40.533 usec
DE                 6.50 usec
TE                298.1 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.00 usec
PLW1         26.50000000 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF          600.1899953 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



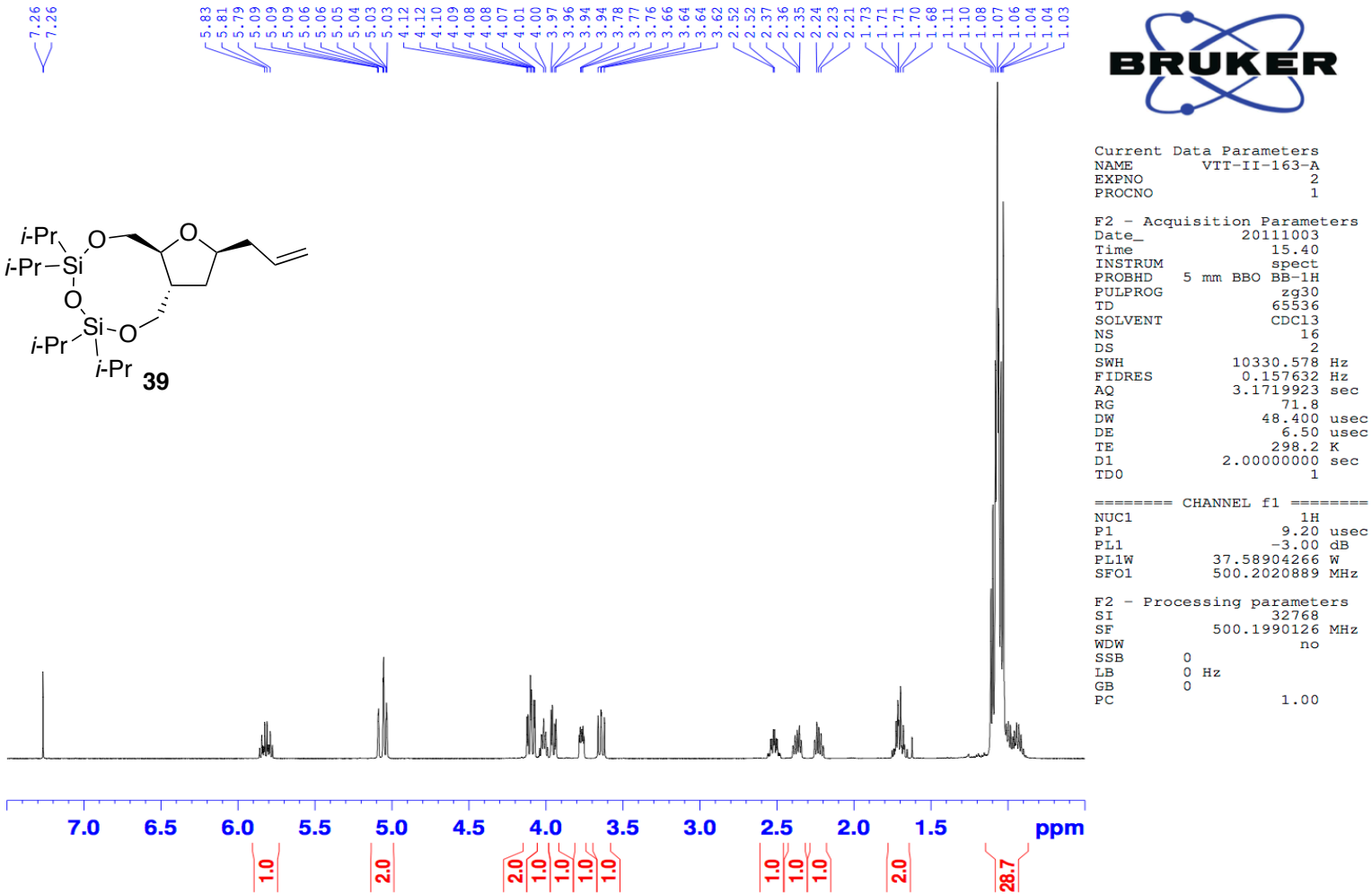

```
Current Data Parameters
NAME      VTT-III-203-B
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20120817
Time              13.41
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT             C6D6
NS                  128
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG               184.65
DW               13.867 usec
DE                6.50 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.65 usec
PLW1       104.00000000 W
SFO1        150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PLW2        26.50000000 W
PLW12        0.65438998 W
PLW13        0.32065001 W
SFO2        600.1924008 MHz

F2 - Processing parameters
SI                32768
SF          150.9177890 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

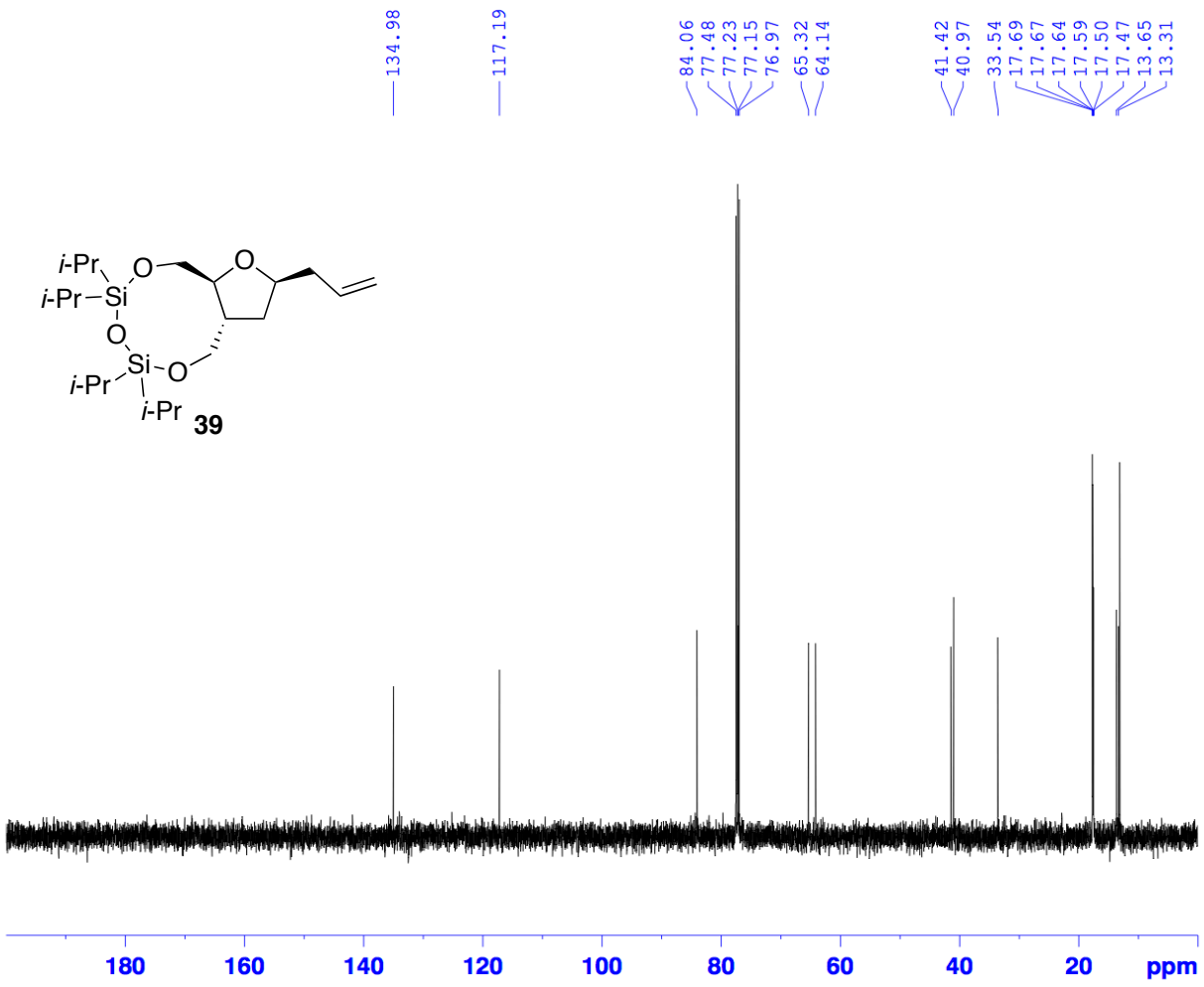



```
Current Data Parameters
NAME        VTT-II-163-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111003
Time               15.40
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719923 sec
RG                 71.8
DW                48.400 usec
DE                 6.50 usec
TE                298.2 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.20 usec
PL1                -3.00 dB
PL1W        37.58904266 W
SFO1        500.2020889 MHz

F2 - Processing parameters
SI                 32768
SF          500.1990126 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```



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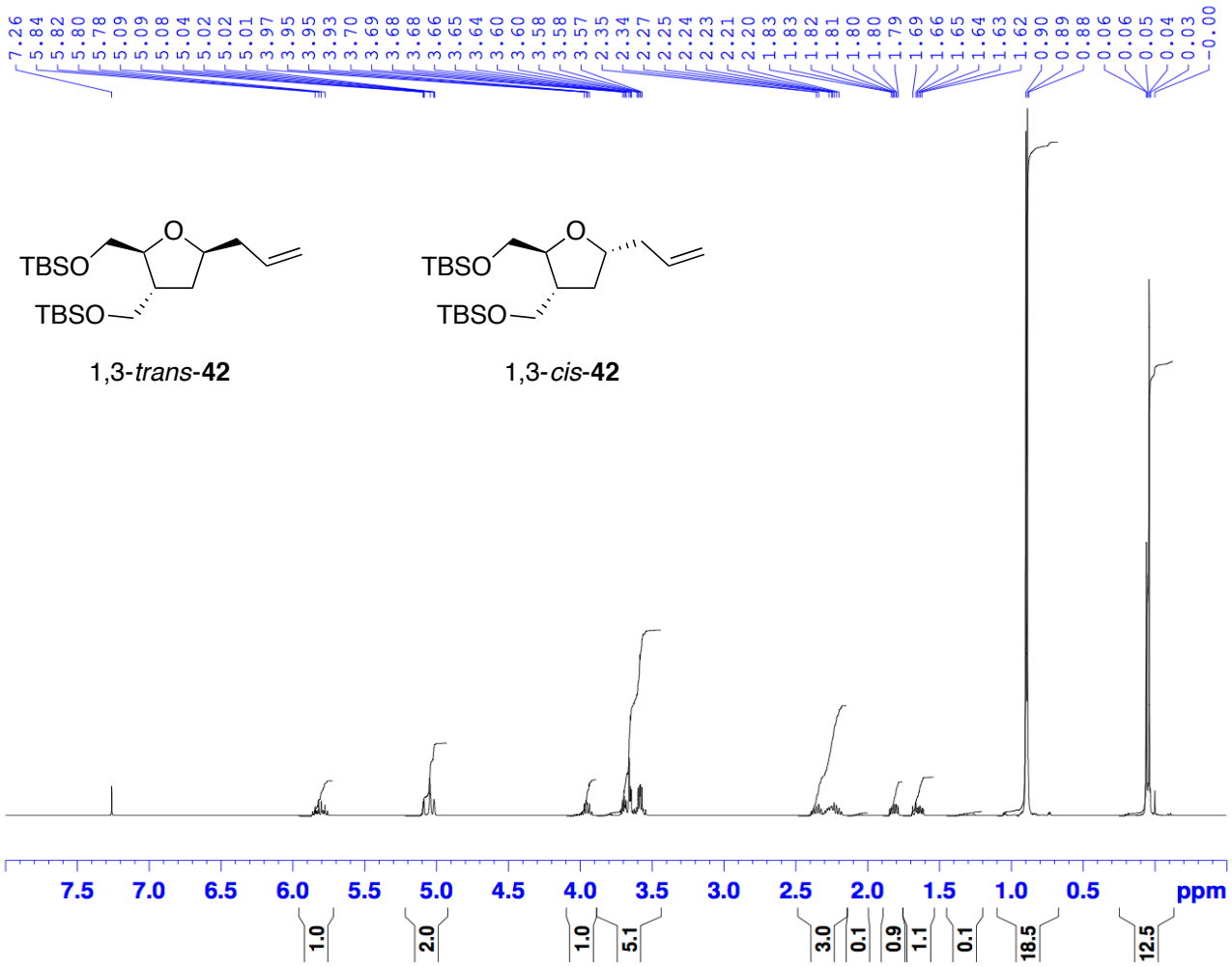
BRUKER

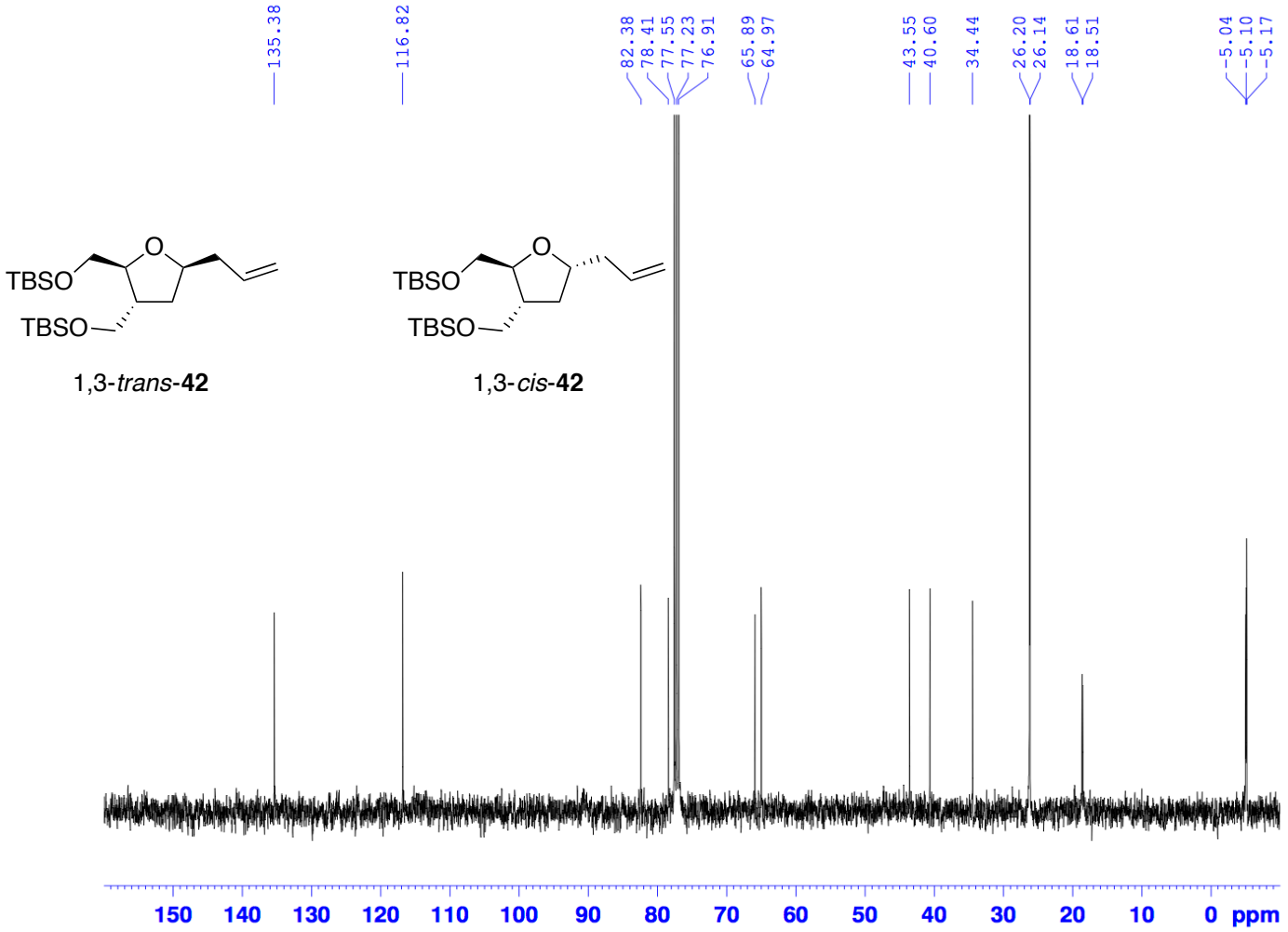
```
Current Data Parameters
NAME        VTT-II-163-A
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111003
Time               15.44
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   30
DS                    4
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ             1.0912244 sec
RG                9195.2
DW                16.650 usec
DE                 6.50 usec
TE                298.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.25 usec
PL1                 2.00 dB
PL1W        49.29017639 W
SFO1        125.7877161 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              70.00 usec
PL2                -3.00 dB
PL12               14.63 dB
PL13               14.91 dB
PL2W        37.58904266 W
PL12W        0.64872593 W
PL13W        0.60822082 W
SFO2        500.2010008 MHz

F2 - Processing parameters
SI                 32768
SF          125.7751125 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



1,3-*trans*-**42**

1,3-*cis*-**42**

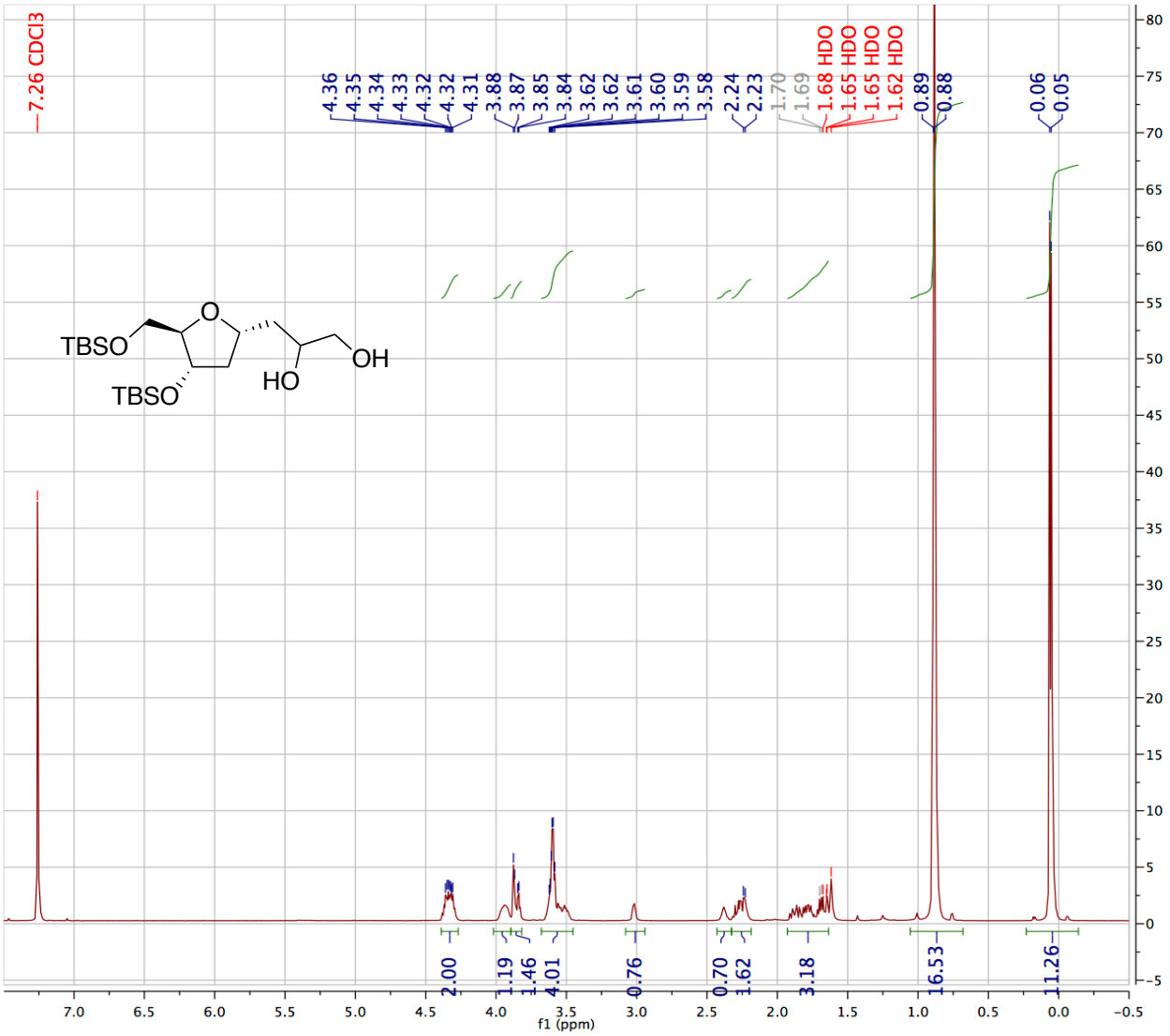
```
Current Data Parameters
NAME        VTT-II-188-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111101
Time               23.52
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                90.5
DW               60.400 usec
DE                6.50 usec
TE               298.2 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.10 usec
PL1               -5.00 dB
PL1W        31.77312851 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300093 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



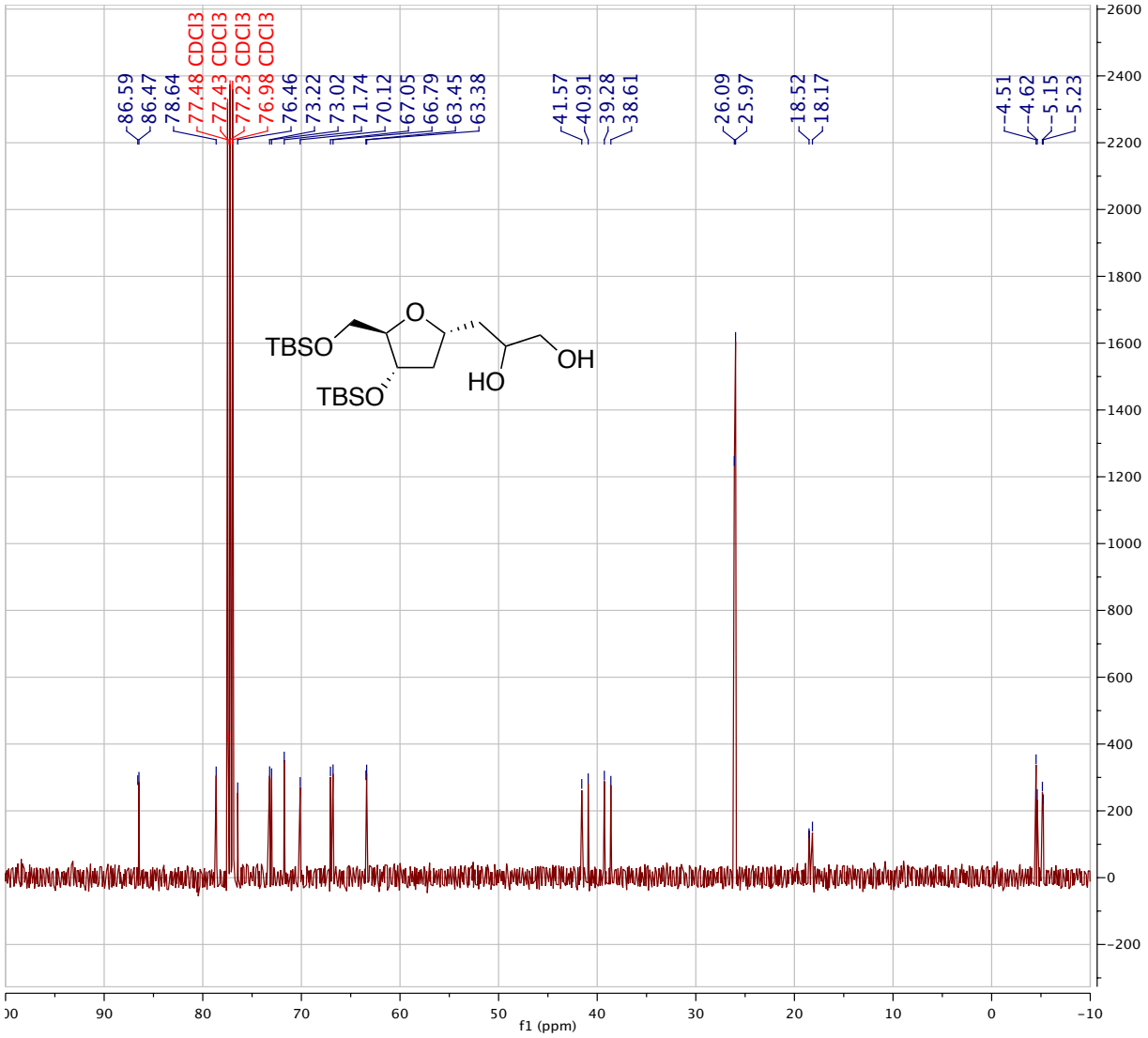
1,3-*trans*-**42**

1,3-*cis*-**42**

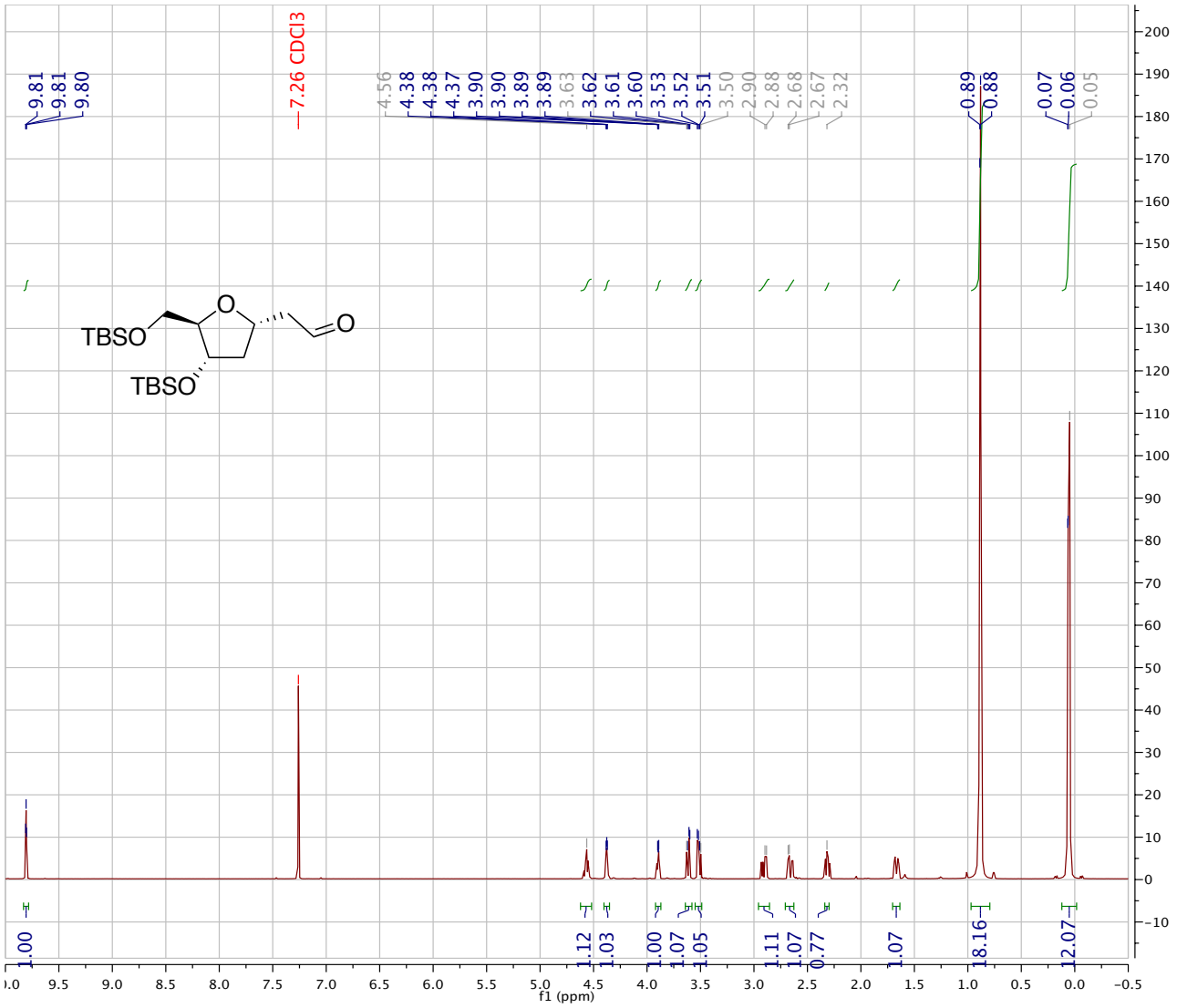
```
Current Data Parameters
NAME        VTT-II-188-A
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111101
Time               23.54
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   68
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG              20642.5
DW                20.850 usec
DE                 6.50 usec
TE                298.2 K
D1           2.50000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  6.50 usec
PL1                -4.00 dB
PL1W        25.23829460 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              70.00 usec
PL2                -5.00 dB
PL12               13.73 dB
PL13               13.73 dB
PL2W        31.77312851 W
PL12W        0.42565724 W
PL13W        0.42565724 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF         100.6127465 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```



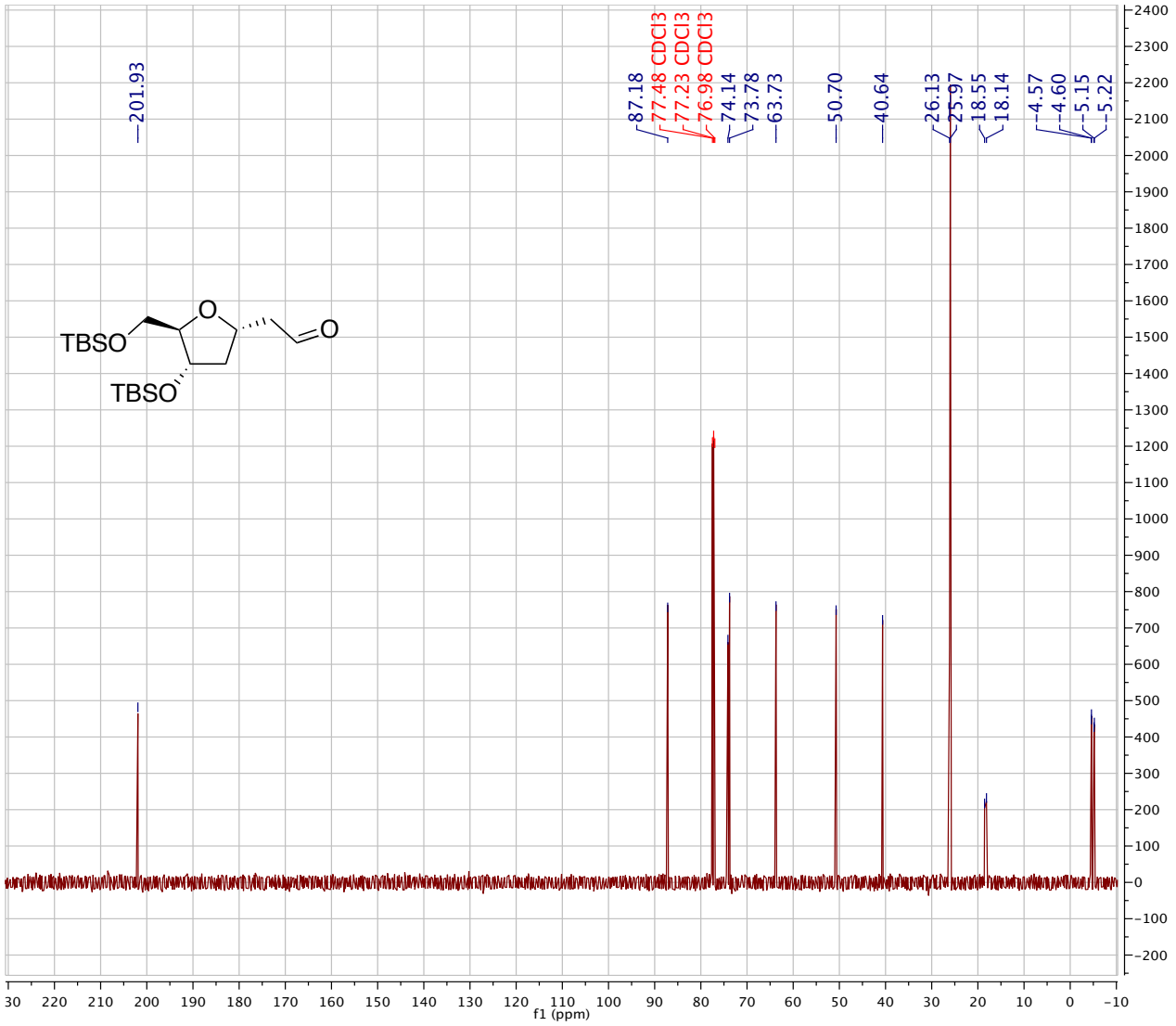
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	8
7	Receiver Gain	7
8	Relaxation Delay	0.1000
9	Pulse Width	7.5000
10	Spectrometer Frequency	500.22
11	Spectral Width	8012.8
12	Lowest Frequency	-536.9
13	Nucleus	1H
14	Acquired Size	40864
15	Spectral Size	131072



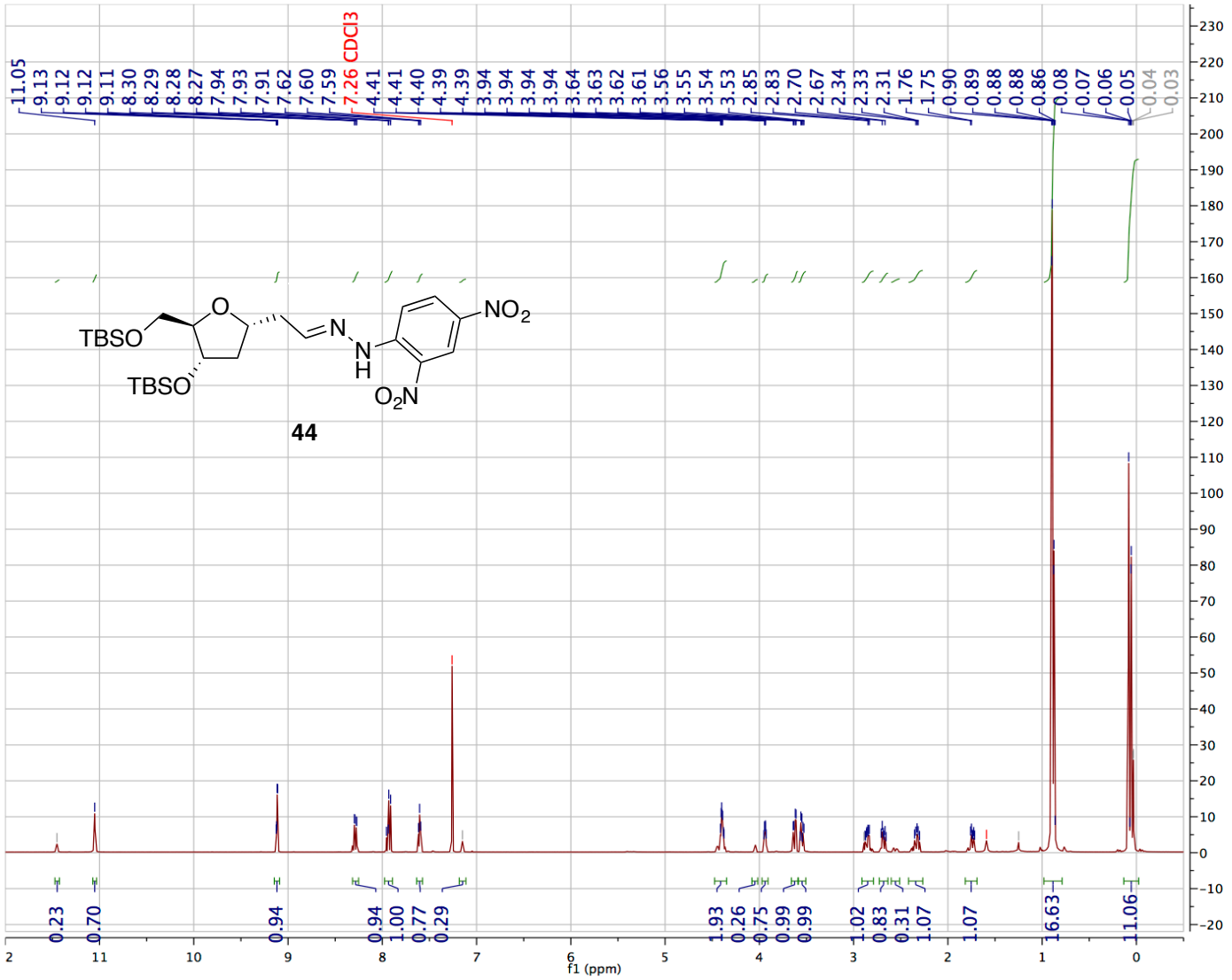
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	SpinEchopg30gp.prd
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	152
7	Receiver Gain	7298
8	Relaxation Delay	0.2500
9	Pulse Width	15.5000
10	Spectrometer Frequency	125.79
11	Spectral Width	30303.0
12	Lowest Frequency	-1294.2
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



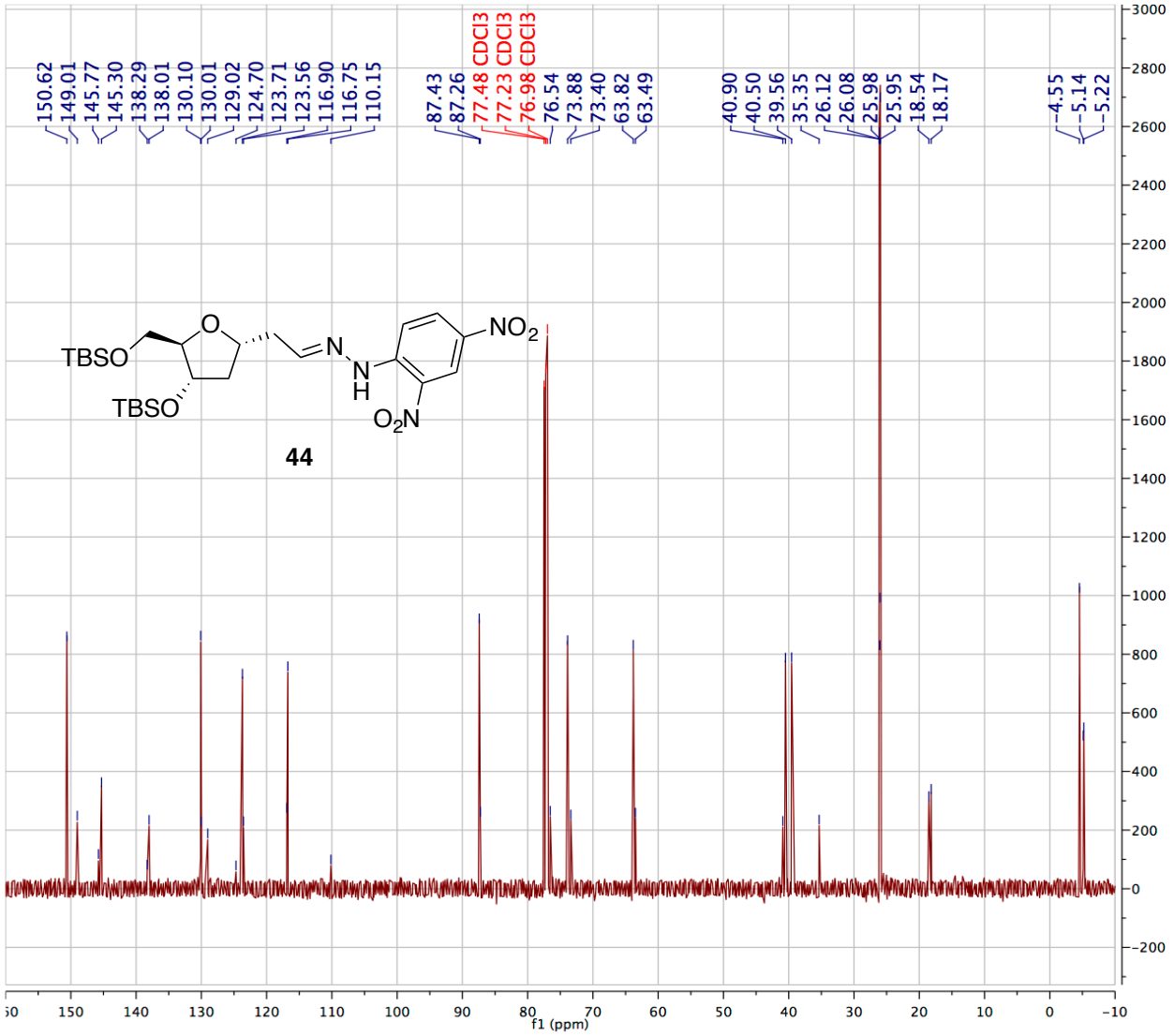
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	8
7	Receiver Gain	6
8	Relaxation Delay	0.1000
9	Pulse Width	7.5000
10	Spectrometer Frequency	500.22
11	Spectral Width	8012.8
12	Lowest Frequency	-536.5
13	Nucleus	1H
14	Acquired Size	40864
15	Spectral Size	131072



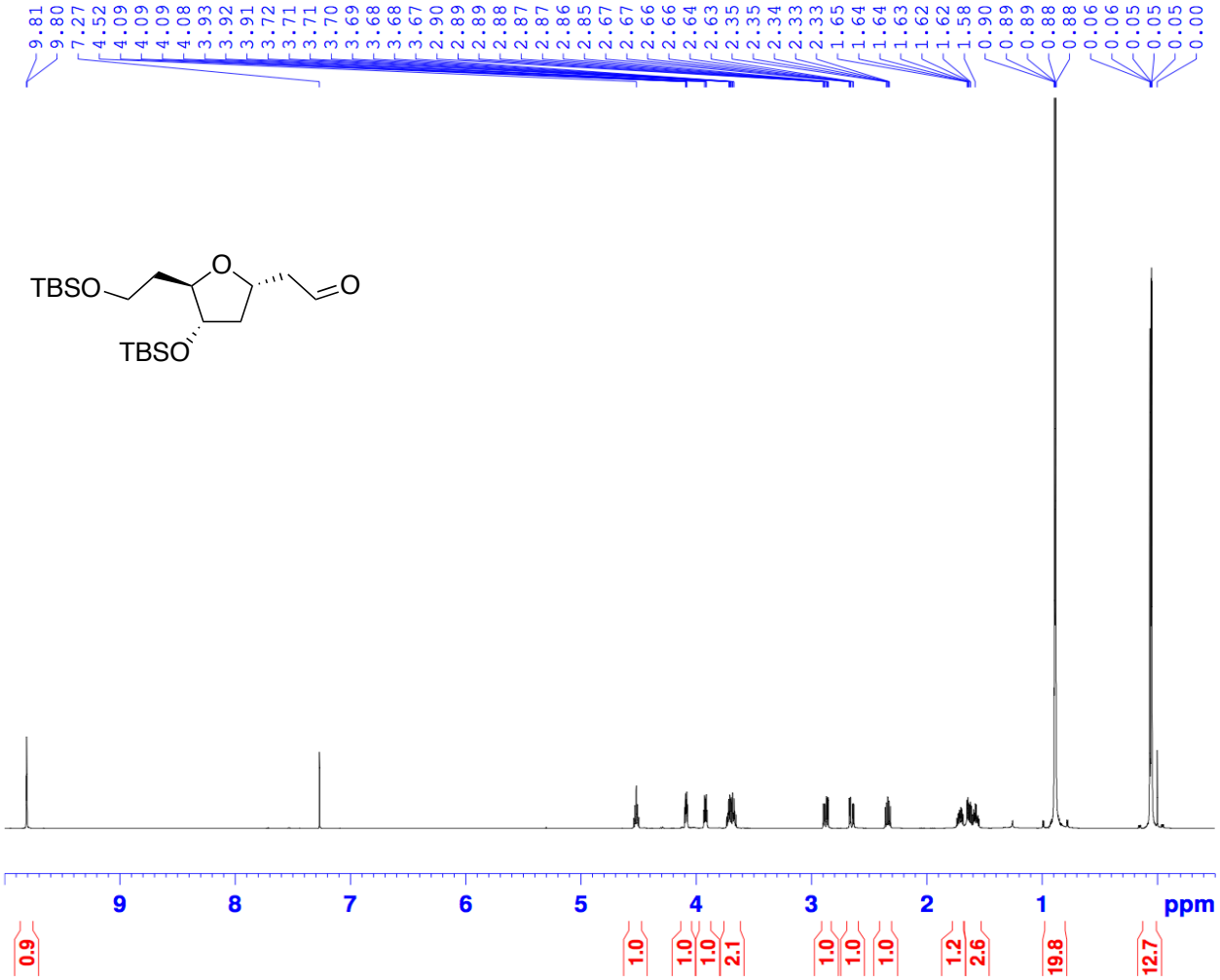
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	SpinEchopg30 gp.prd
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	129
7	Receiver Gain	4598
8	Relaxation Delay	0.2500
9	Pulse Width	15.5000
10	Spectrometer Frequency	125.79
11	Spectral Width	30303.0
12	Lowest Frequency	-1294.2
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



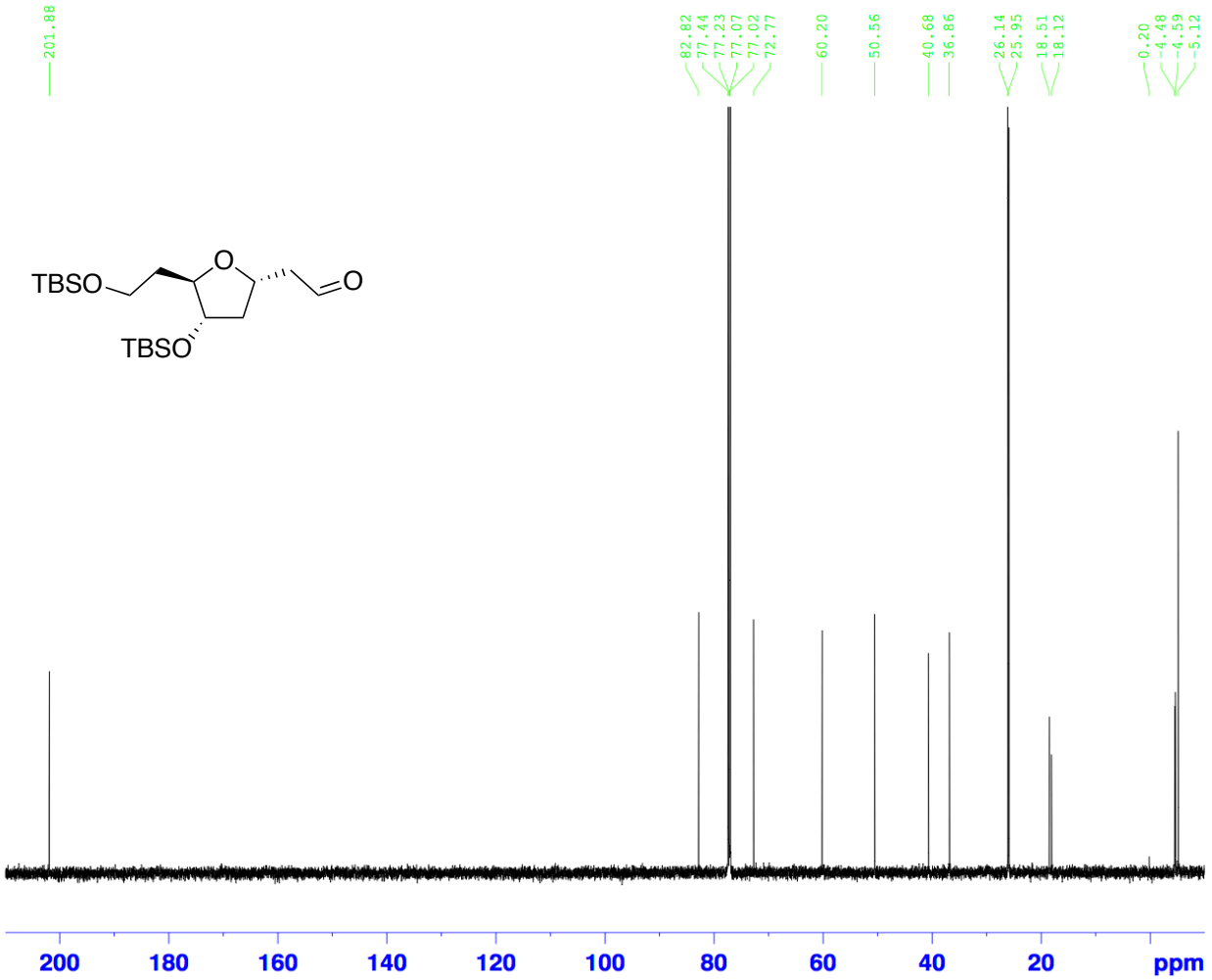
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/15N/ 2H Z-GRD Z44881/0020
6	Number of Scans	8
7	Receiver Gain	6
8	Relaxation Delay	0.1000
9	Pulse Width	7.5000
10	Spectrometer Frequency	500.22
11	Spectral Width	8012.8
12	Lowest Frequency	-536.5
13	Nucleus	1H
14	Acquired Size	40864
15	Spectral Size	131072



	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	SpinEchopg30gp.prd
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	114
7	Receiver Gain	7298
8	Relaxation Delay	0.2500
9	Pulse Width	15.5000
10	Spectrometer Frequency	125.79
11	Spectral Width	30303.0
12	Lowest Frequency	-1295.4
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536




```
Current Data Parameters
NAME         VTT-IV-12-A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120905
Time               22.06
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                 33.59
DW                40.533 usec
DE                 6.50 usec
TE                298.2 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.00 usec
PLW1         26.50000000 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF           600.1900109 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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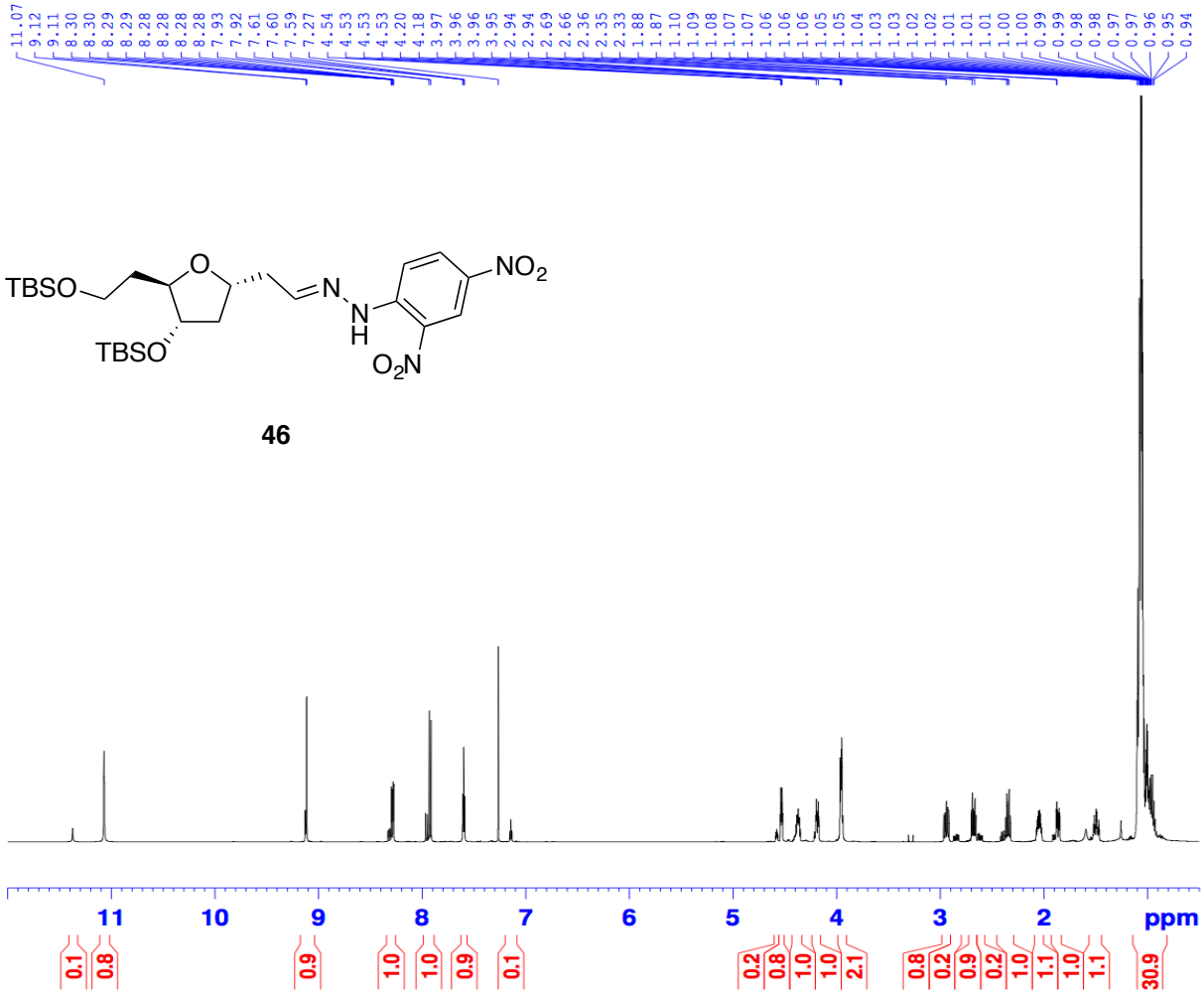
```
Current Data Parameters
NAME          VTT-IV-12-A
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20120905
Time               22.07
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG               184.65
DW               13.867 usec
DE                6.50 usec
TE                298.3 K
D1           2.50000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.65 usec
PLW1       104.00000000 W
SFO1        150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PLW2        26.50000000 W
PLW12        0.65438998 W
PLW13        0.32065001 W
SFO2        600.1924008 MHz

F2 - Processing parameters
SI                32768
SF          150.9178645 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



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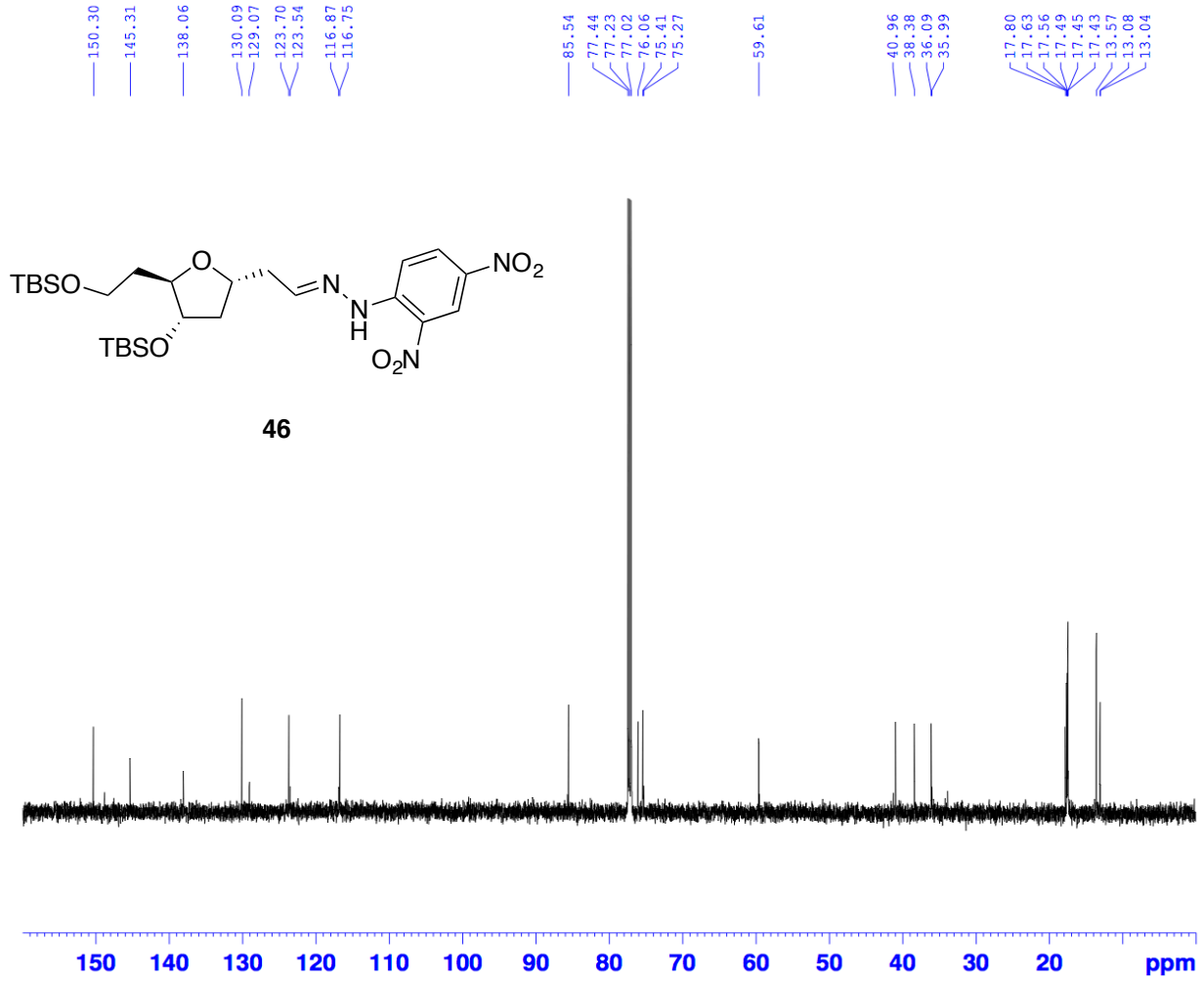
```
Current Data Parameters
NAME          VTT-IV-24-A
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20120926
Time                20.37
INSTRUM             spect
PROBHD   5 mm PAQXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                 33.59
DW                40.533 usec
DE                 6.50 usec
TE                298.2 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.79 usec
PLW1          9.30000019 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF           600.1900105 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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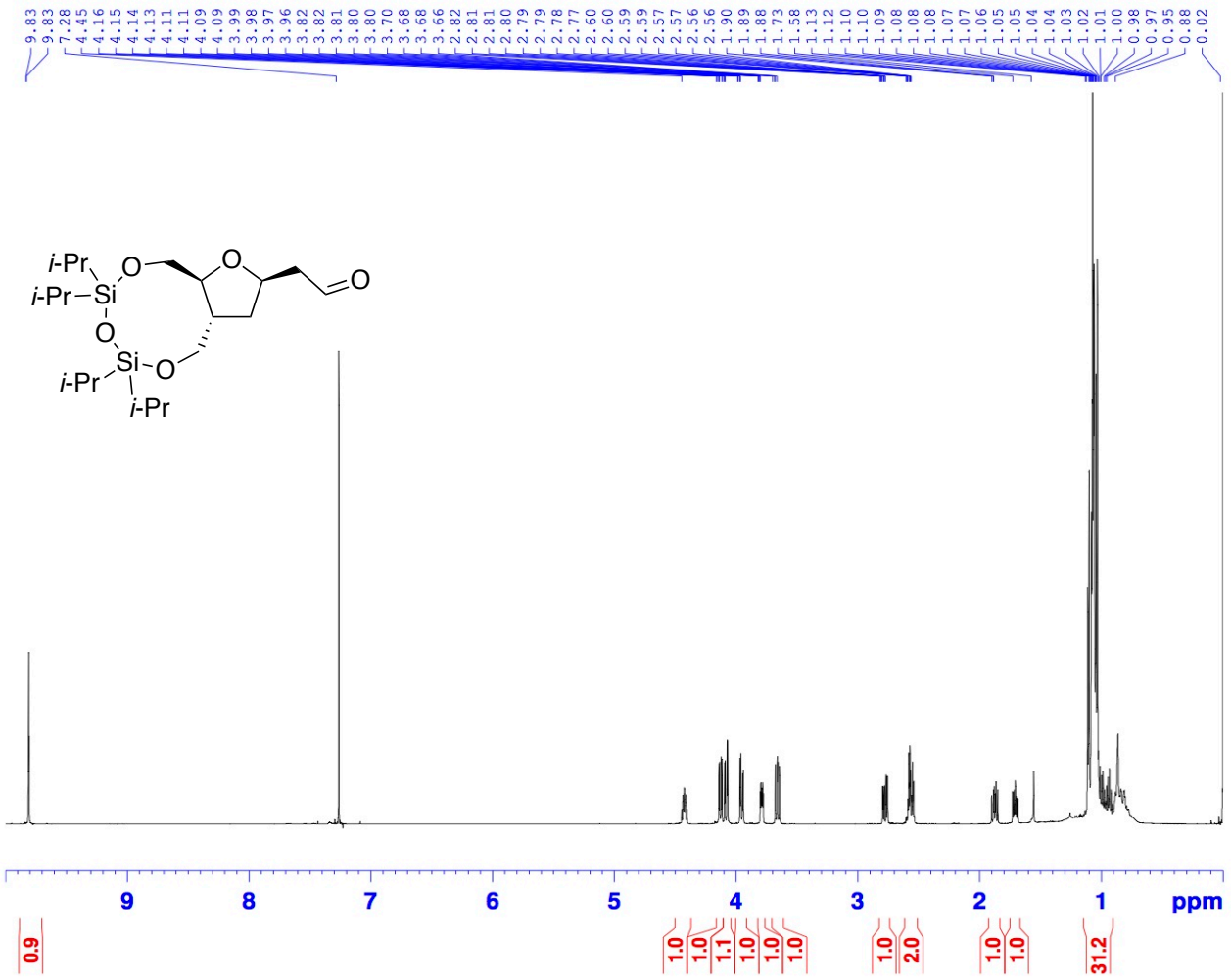
```
Current Data Parameters
NAME          VTT-IV-24-A
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20120926
Time                20.40
INSTRUM             spect
PROBHD   5 mm PAQXI 1H/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    128
DS                      0
SWH             36057.691 Hz
FIDRES          0.550197 Hz
AQ              0.9088159 sec
RG                 184.65
DW                 13.867 usec
DE                   6.50 usec
TE                  298.1 K
D1             2.50000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                   15.00 usec
PLW1        106.00000000 W
SFO1         150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2                70.00 usec
PLW2           9.30000019 W
PLW12          0.18190999 W
PLW13          0.08913500 W
SFO2          600.1924008 MHz

F2 - Processing parameters
SI                  32768
SF           150.9178644 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



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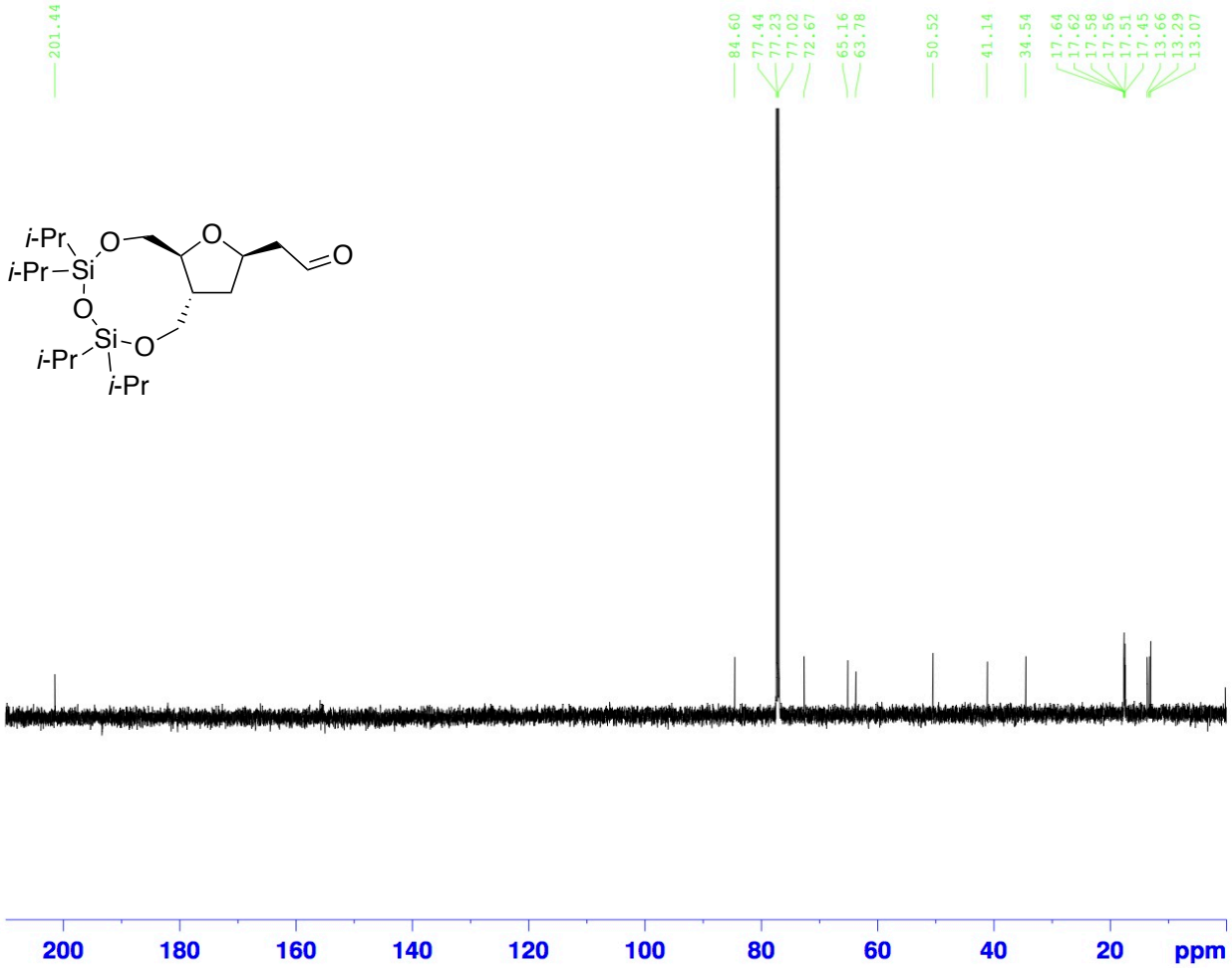
```
Current Data Parameters
NAME         VTT-IV-32-A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121022
Time               10.49
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                  92.46
DW                 40.533 usec
DE                  6.50 usec
TE                 298.2 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.79 usec
PLW1          9.30000019 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF           600.1900129 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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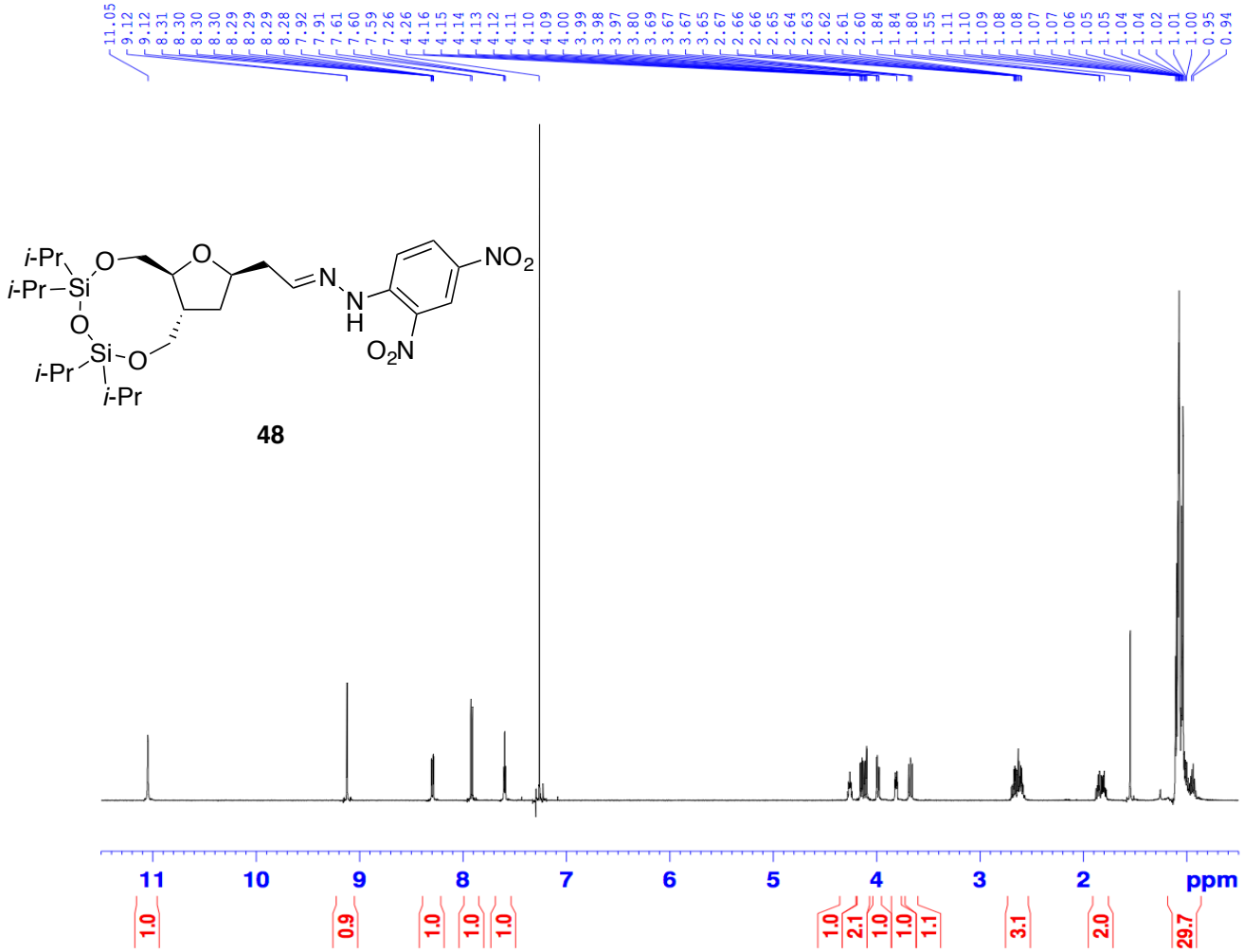
```
Current Data Parameters
NAME          VTT-IV-32-A
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20121022
Time               10.50
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9088159 sec
RG               184.65
DW              13.867 usec
DE                6.50 usec
TE              298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.00 usec
PLW1        106.00000000 W
SFO1         150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PLW2           9.30000019 W
PLW12          0.18190999 W
PLW13          0.08913500 W
SFO2          600.1924008 MHz

F2 - Processing parameters
SI                  32768
SF           150.9178634 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



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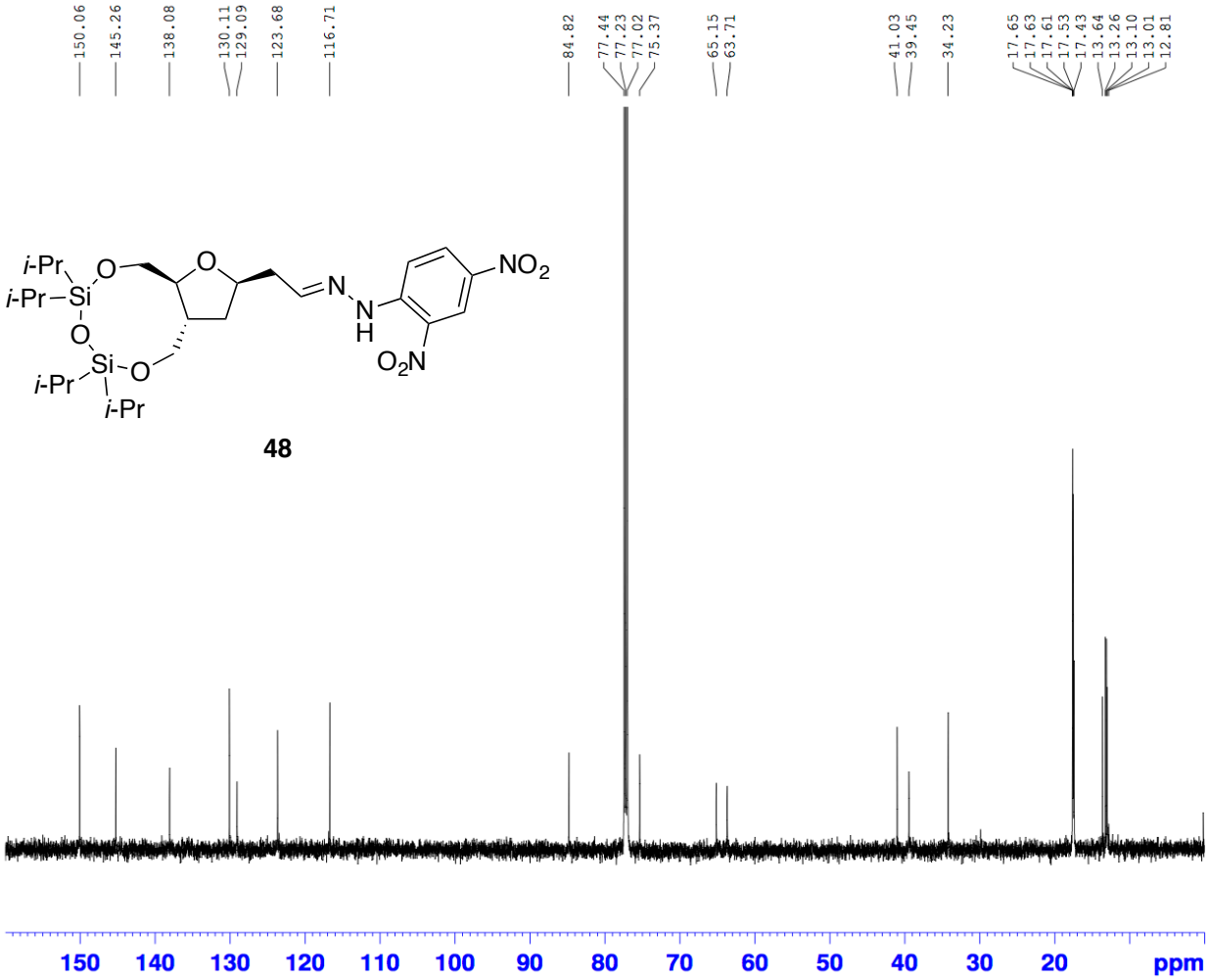
```
Current Data Parameters
NAME        VTT-III-12-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120331
Time               17.46
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            12335.526 Hz
FIDRES         0.188225 Hz
AQ             2.6564426 sec
RG                164.46
DW                40.533 usec
DE                 6.50 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.79 usec
PLW1          9.30000019 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF           600.1900136 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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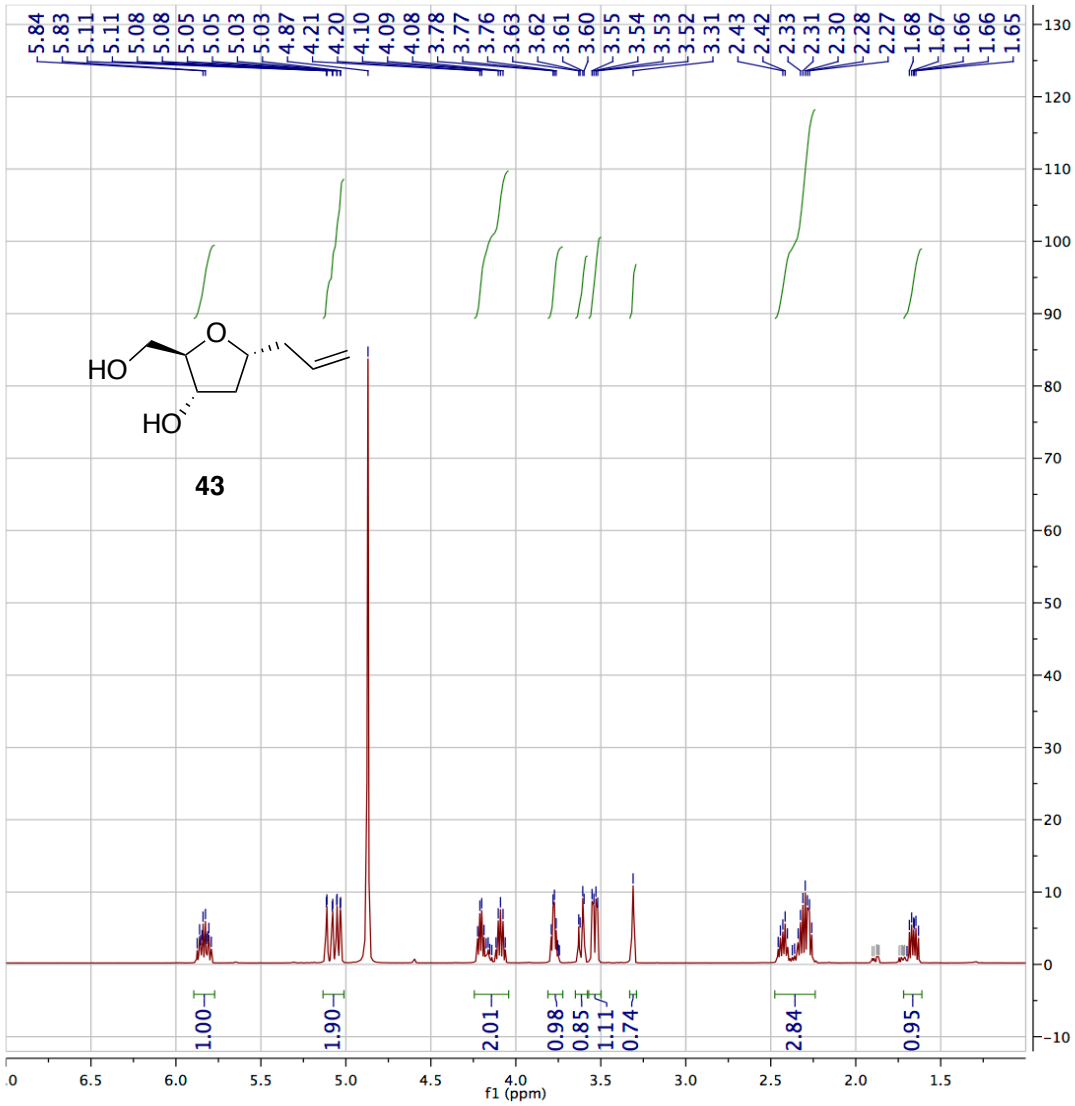
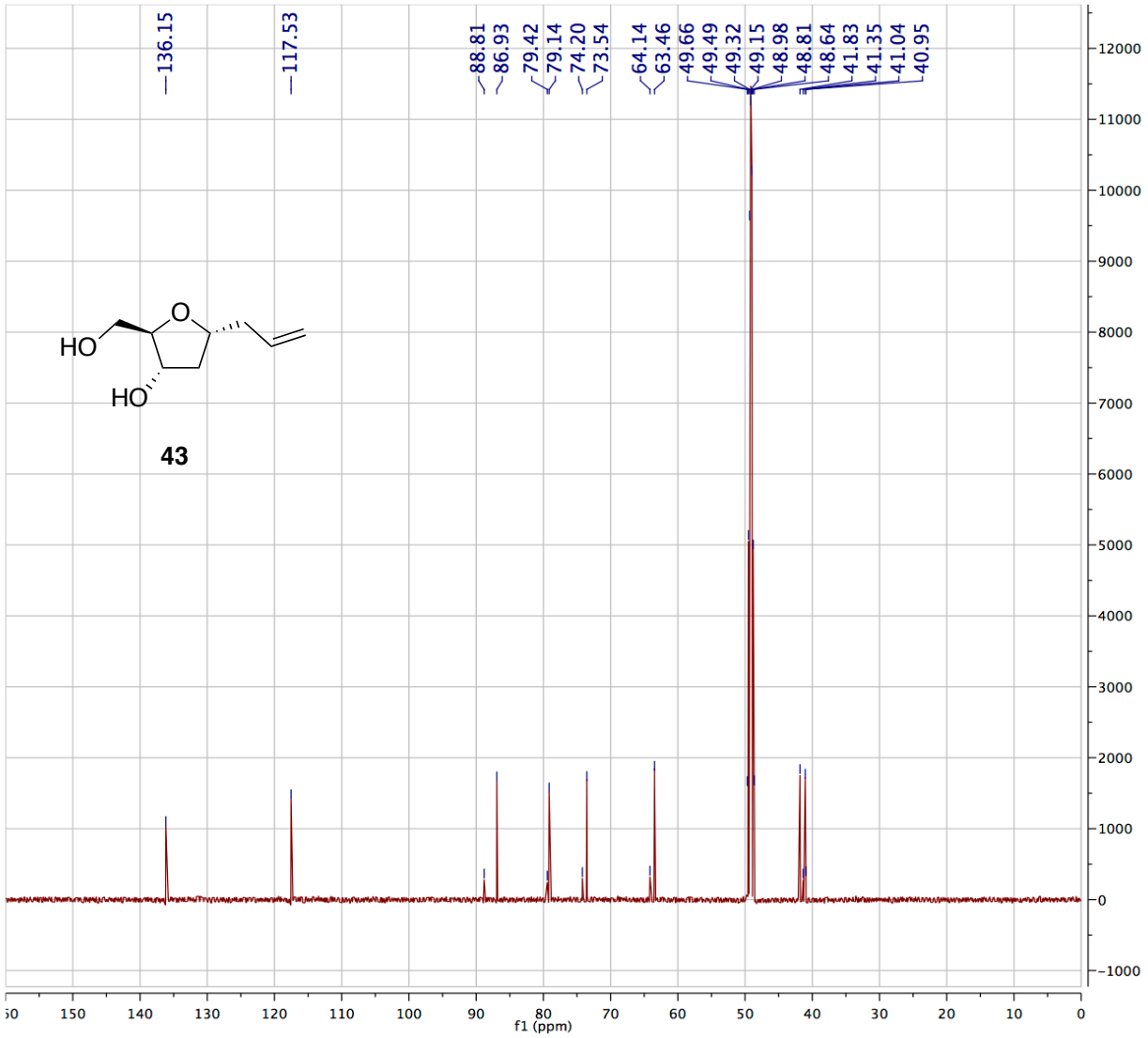
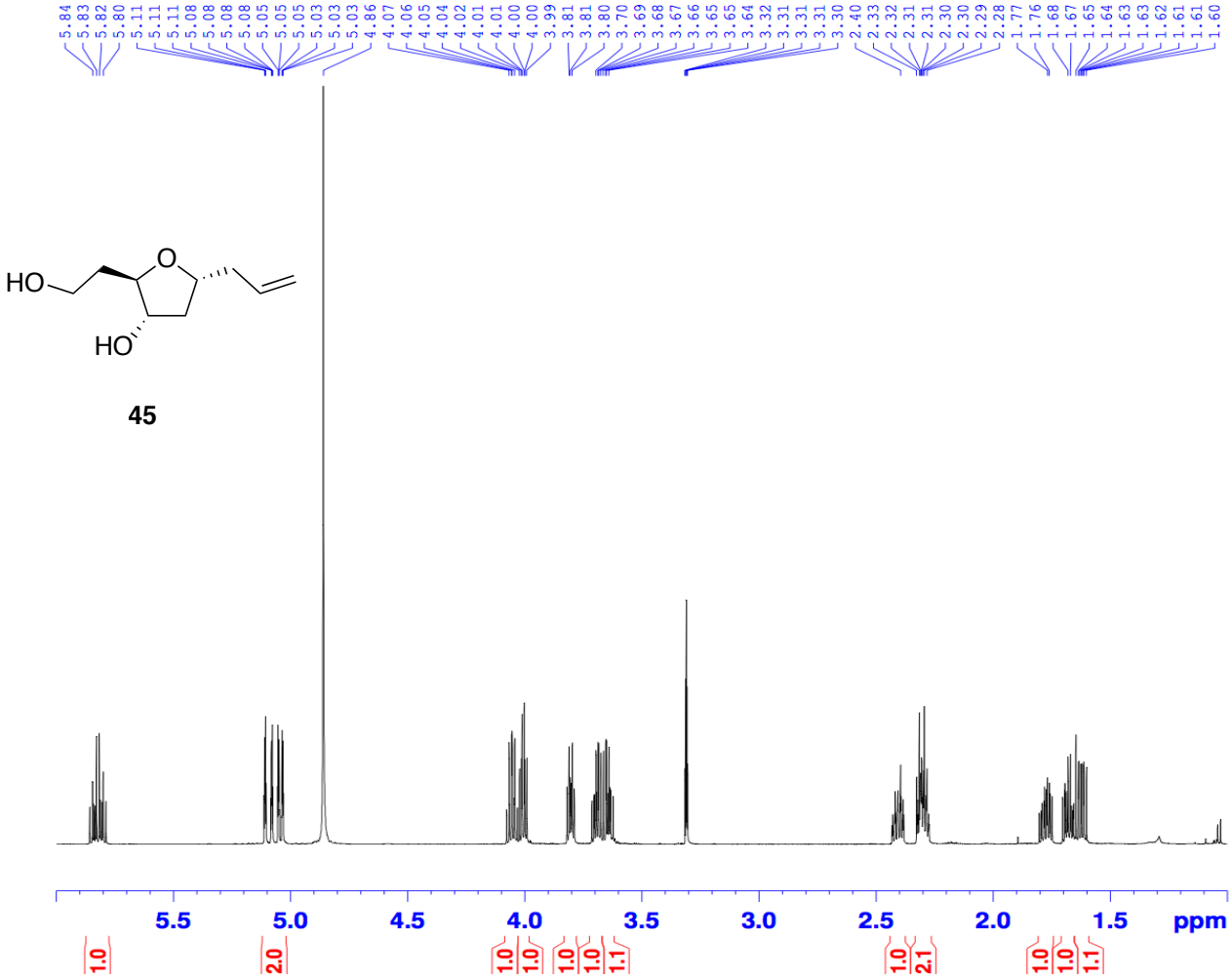
```
Current Data Parameters
NAME          VTT-III-12-A
EXPNO                    7
PROCNO                   1

F2 - Acquisition Parameters
Date_           20121023
Time               23.27
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9088159 sec
RG               184.65
DW                13.867 usec
DE                 6.50 usec
TE                298.1 K
D1            2.50000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.00 usec
PLW1        106.00000000 W
SFO1         150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PLW2           9.30000019 W
PLW12          0.18190999 W
PLW13          0.08913500 W
SFO2          600.1924008 MHz

F2 - Processing parameters
SI                32768
SF          150.9178660 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



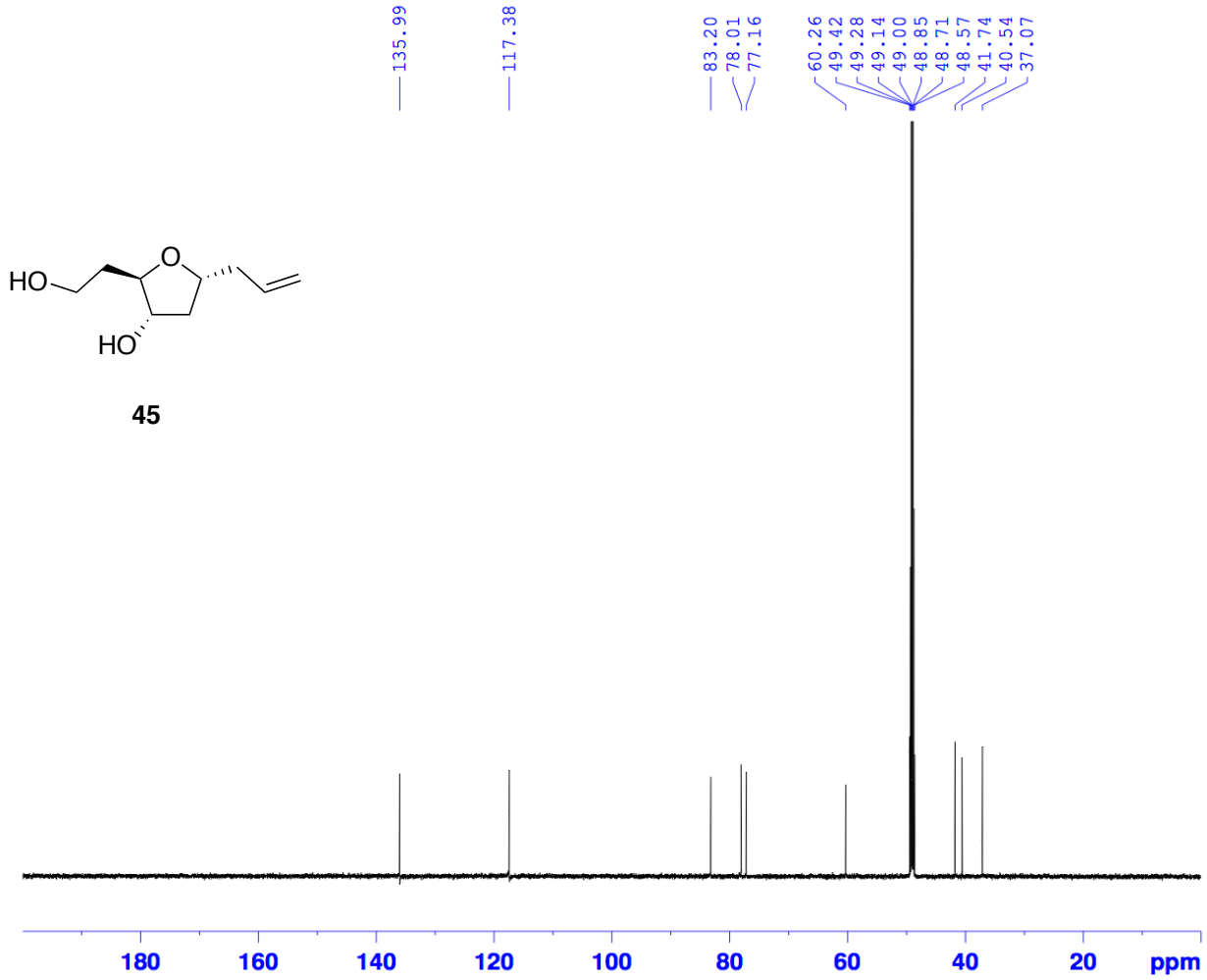
	Parameter	Value
1	Solvent	C6D6
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	8
7	Receiver Gain	5
8	Relaxation Delay	0.1000
9	Pulse Width	7.5000
10	Spectrometer Frequency	500.22
11	Spectral Width	8012.8
12	Lowest Frequency	-523.8
13	Nucleus	1H
14	Acquired Size	40864
15	Spectral Size	131072



	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	SpinEchopg30g p.prd
4	Experiment	1D
5	Probe	5 mm CPTCI 1H-13C/ 15N/ 2H Z-GRD Z44881/ 0020
6	Number of Scans	171
7	Receiver Gain	7298
8	Relaxation Delay	0.2500
9	Pulse Width	15.5000
10	Spectrometer Frequency	125.79
11	Spectral Width	30303.0
12	Lowest Frequency	-1122.7
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536




```
Current Data Parameters
NAME          VTT-IV-3-A
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120828
Time               16.16
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                     8
DS                     0
SWH            12335.526 Hz
FIDRES         0.188225 Hz
AQ             2.6564426 sec
RG                 92.46
DW                40.533 usec
DE                 6.50 usec
TE                298.2 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.00 usec
PLW1         26.50000000 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF           600.1900118 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```



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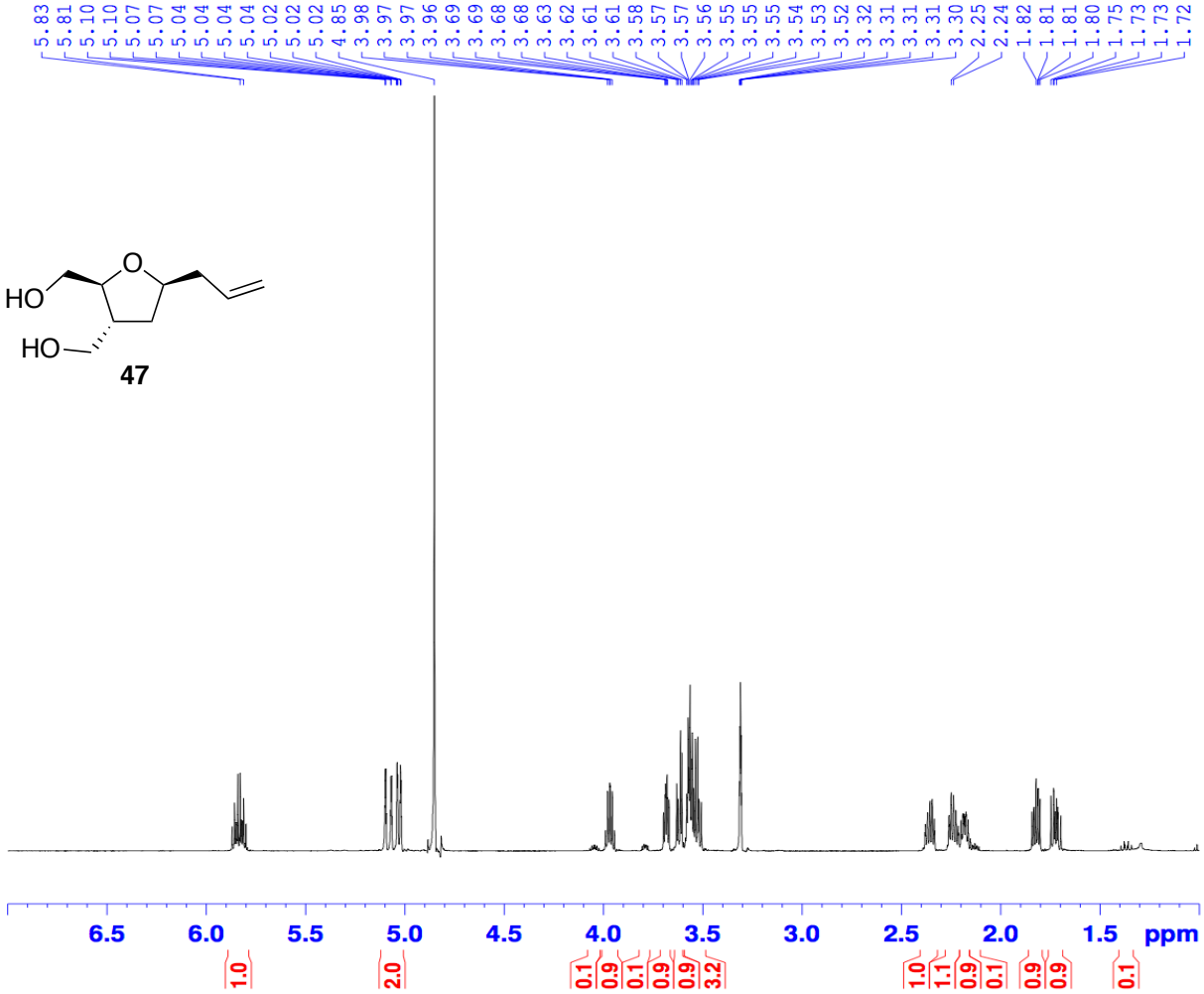
```
Current Data Parameters
NAME          VTT-IV-3-A
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120828
Time               16.18
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT             MeOD
NS                   128
DS                     0
SWH            36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9088159 sec
RG                184.65
DW                13.867 usec
DE                 6.50 usec
TE                298.2 K
D1            2.50000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.65 usec
PLW1        104.00000000 W
SFO1         150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              70.00 usec
PLW2         26.50000000 W
PLW12         0.65438998 W
PLW13         0.32065001 W
SFO2         600.1924008 MHz

F2 - Processing parameters
SI                 32768
SF          150.9176859 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



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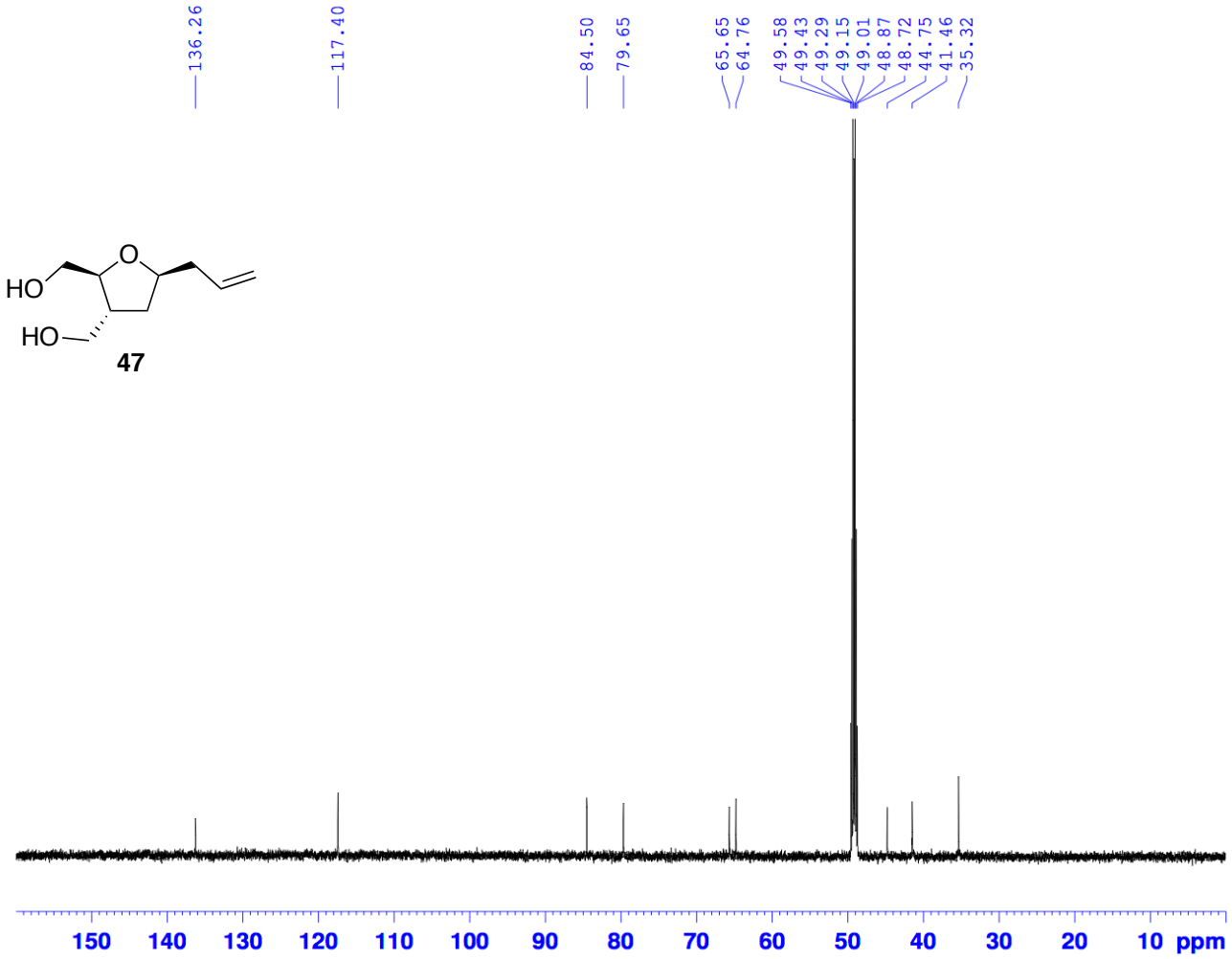
```
Current Data Parameters
NAME          VTT-IV-46-A
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20121123
Time                14.41
INSTRUM             spect
PROBHD   5 mm PAQXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                     8
DS                     0
SWH            12335.526 Hz
FIDRES          0.188225 Hz
AQ             2.6564426 sec
RG                113.19
DW                40.533 usec
DE                 6.50 usec
TE                298.2 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.79 usec
PLW1          9.30000019 W
SFO1         600.1937064 MHz

F2 - Processing parameters
SI                 65536
SF           600.1900114 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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```
Current Data Parameters
NAME         VTT-IV-46-A
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121124
Time               16.46
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG               184.65
DW               13.867 usec
DE                6.50 usec
TE                298.2 K
D1           2.50000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.00 usec
PLW1       106.00000000 W
SFO1        150.9329866 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             70.00 usec
PLW2         9.30000019 W
PLW12        0.18190999 W
PLW13        0.08913500 W
SFO2        600.1924008 MHz

F2 - Processing parameters
SI                32768
SF          150.9182567 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```