Strained Organosilacyclic Compounds: Synthesis of anti-Bredt Olefins and *trans*-Dioxasilacyclooctenes

Michel Prévost, Joseph W. Ziller, and K. A. Woerpel*[a]
Department of Chemistry, University of California, Irvine, California 92697-2025

Supplementary information

Contents

I. Stereochemical Proofs 2

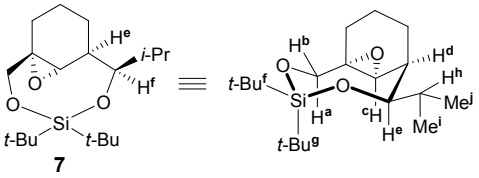
II. X-ray Crystallographic Data (Aziridine **8**) 4

III. Selected Spectra 13

I. Stereochemical Proofs

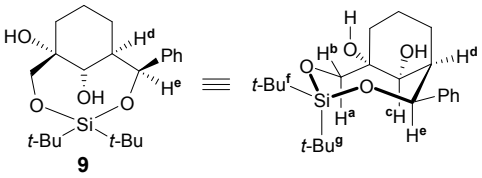
The peaks in the ^{1}H NMR spectra were assigned using ^{1}H/^{1}H COSY experiments, chemical shifts, and coupling constants. The selectivities were determined by ^{1}H NMR spectroscopy of the unpurified reaction mixture. The relative configurations of the products were determined by relevant nuclear Overhauser effect (nOe) enhancements (2D NOESY) and ^{1}H NMR coupling constant data. The configuration of aziridine **8** was confirmed by X-ray crystallographic analysis.

Figure S1. Relevant 2D NOESY Data (mixing time 0.8 s) for Epoxide **7**



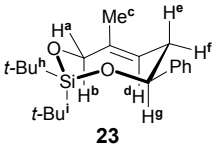
H^a and H^c (medium)
H^c and H^d (medium)
H^c and H^e (medium)
$H^d$and H^e (strong)
H^f and H^b (weak)
$H^g$and H^a (medium)
H^g and H^c (medium)
H^g and H^e (medium)
H^g and H^h (medium)

Figure S2. Relevant 2D NOESY Data (mixing time 0.8 s) for Diol **9**



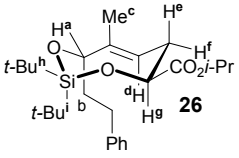
H^a and H^e (weak)
H^a and H^c (medium)
H^c and H^d (strong)
$H^c$and H^e (medium)
$H^d$and H^e (medium)
H^f and H^b (medium)
H^f and H^a (medium)
H^g and H^a (weak)
H^g and H^c(strong)
H^g and H^e (strong)

Figure S3. Relevant 2D NOESY Data (mixing time 0.8 s) for *trans*-Dioxasilacyclooctene **23**



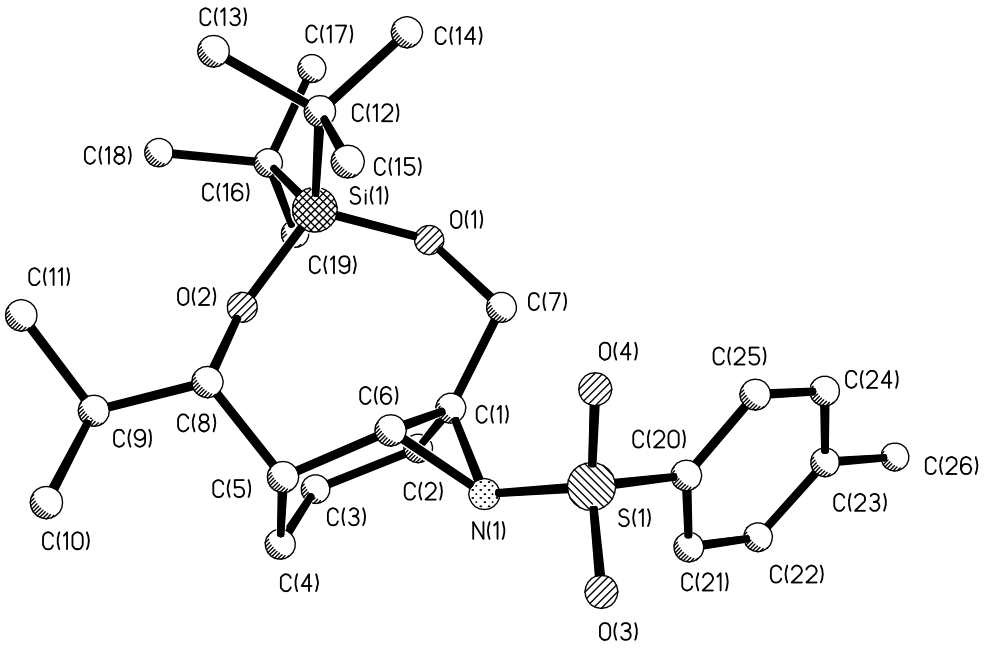
H^c and H^a (medium)
H^c and H^e (medium)
H^b and H^d (strong)
H^g and H^d (medium)
H^g and H^f (medium)
H^h and H^c (weak)
H^h and H^a (weak)
H^i and H^b (medium)
H^i and H^d (strong)
H^i and H^g (strong)

Figure S4. Relevant 2D NOESY Data (mixing time 0.8 s) for *trans*-Dioxasilacyclooctene **26**



H^c and H^a (medium)
H^c and H^e (medium)
H^b and H^d (medium)
H^g and H^d (medium)
H^g and H^f (medium)
H^h and H^c (medium)
H^h and H^a (weak)
H^i and H^b (medium)
H^i and H^d (medium)
H^i and H^g (Strong)

X-Ray Structure of aziridine **8**:



II. X-ray Crystallographic Data (Aziridine 8)

Table 1. Crystal data and structure refinement for aziridine **8**.

Identification code	kaw133 (Michel Prevost)	
Empirical formula	$C_{26}\ H_{43}\ N\ O_4\ S\ Si$	
Formula weight	493.76	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 9.7886(5) Å	α= 81.5630(10)°.
	b = 10.4633(6) Å	β= 79.6200(10)°.
	c = 13.3805(7) Å	γ = 82.6760(10)°.
Volume	1326.22(12) $Å^3$	
Z	2	
Density (calculated)	1.236 Mg/m^3	
Absorption coefficient	0.199 mm^{-1}	
F(000)	536	
Crystal color	colorless	
Crystal size	0.32 x 0.24 x 0.16 mm^3	
Theta range for data collection	1.56 to 28.40°	
Index ranges	$-12 \le h \le 13$, $-13 \le k \le 13$, $-16 \le l \le 17$	
Reflections collected	15338	
Independent reflections	6091 [R(int) = 0.0154]	
Completeness to theta = 28.40°	91.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9697 and 0.9390	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6091 / 0 / 470	
Goodness-of-fit on F^2	1.049	
Final R indices [I>2sigma(I) = 5589 data]	R1 = 0.0321, wR2 = 0.0901	
R indices (all data, 0.75Å)	R1 = 0.0348, wR2 = 0.0925	
Largest diff. peak and hole	0.450 and -0.326 $e.Å^{-3}$	

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

	x	y	z	U(eq)
S(1)	7518(1)	8547(1)	4670(1)	16(1)
Si(1)	7339(1)	12237(1)	1369(1)	13(1)
N(1)	6882(1)	8618(1)	3595(1)	15(1)
O(1)	8620(1)	11141(1)	1662(1)	16(1)
O(2)	5844(1)	11640(1)	1482(1)	15(1)
O(3)	6636(1)	7767(1)	5428(1)	23(1)
O(4)	7718(1)	9805(1)	4906(1)	20(1)
C(1)	7511(1)	9357(1)	2605(1)	14(1)
C(2)	7523(1)	8606(1)	1709(1)	18(1)
C(3)	6122(1)	8723(1)	1322(1)	19(1)
C(4)	4861(1)	8801(1)	2176(1)	18(1)
C(5)	4828(1)	9936(1)	2787(1)	15(1)
C(6)	6171(1)	9886(1)	3190(1)	14(1)
C(7)	8730(1)	10149(1)	2503(1)	15(1)
C(8)	4617(1)	11327(1)	2213(1)	14(1)
C(9)	3361(1)	11560(1)	1648(1)	17(1)
C(10)	2030(1)	11155(1)	2351(1)	23(1)
C(11)	3117(1)	12992(1)	1220(1)	23(1)
C(12)	7266(1)	13628(1)	2157(1)	18(1)
C(13)	6420(2)	14849(1)	1716(1)	27(1)
C(14)	8757(1)	13951(2)	2161(1)	29(1)
C(15)	6614(1)	13282(1)	3284(1)	23(1)
C(16)	7869(1)	12634(1)	-71(1)	17(1)
C(17)	9150(1)	13403(1)	-367(1)	26(1)
C(18)	6669(1)	13396(1)	-574(1)	24(1)
C(19)	8244(1)	11348(1)	-536(1)	22(1)
C(20)	9168(1)	7676(1)	4363(1)	17(1)
C(21)	9263(1)	6475(1)	4012(1)	25(1)
C(22)	10568(1)	5788(1)	3796(1)	28(1)
C(23)	11774(1)	6271(1)	3934(1)	23(1)
C(24)	11651(1)	7476(1)	4276(1)	25(1)
C(25)	10353(1)	8184(1)	4490(1)	24(1)
C(26)	13177(2)	5499(2)	3727(1)	31(1)

Table 3. Bond lengths [Å] and angles [°] for aziridine **8**.

S(1)-O(3)	1.4363(9)
S(1)-O(4)	1.4428(9)
S(1)-N(1)	1.6559(10)
S(1)-C(20)	1.7616(12)
Si(1)-O(2)	1.6372(8)
Si(1)-O(1)	1.6496(8)
Si(1)-C(16)	1.9011(11)
Si(1)-C(12)	1.9051(12)
N(1)-C(6)	1.4921(14)
N(1)-C(1)	1.5019(13)
O(1)-C(7)	1.4241(13)
O(2)-C(8)	1.4452(12)
C(1)-C(6)	1.4859(15)
C(1)-C(7)	1.5129(14)
C(1)-C(2)	1.5251(15)
C(2)-C(3)	1.5357(16)
C(3)-C(4)	1.5266(16)
C(4)-C(5)	1.5321(15)
C(5)-C(6)	1.5000(15)
C(5)-C(8)	1.5505(15)
C(8)-C(9)	1.5306(15)
C(9)-C(11)	1.5284(16)
C(9)-C(10)	1.5312(16)
C(12)-C(13)	1.5336(17)
C(12)-C(15)	1.5383(16)
C(12)-C(14)	1.5412(16)
C(16)-C(18)	1.5353(16)
C(16)-C(17)	1.5369(16)
C(16)-C(19)	1.5394(16)
C(20)-C(25)	1.3827(17)
C(20)-C(21)	1.3919(17)
C(21)-C(22)	1.3875(18)
C(22)-C(23)	1.3932(19)
C(23)-C(24)	1.3878(18)
C(23)-C(26)	1.5042(17)
C(24)-C(25)	1.3919(18)

O(3)-S(1)-O(4)	117.10(5)
O(3)-S(1)-N(1)	105.44(5)
O(4)-S(1)-N(1)	113.20(5)
O(3)-S(1)-C(20)	109.95(5)
O(4)-S(1)-C(20)	108.57(5)
N(1)-S(1)-C(20)	101.44(5)
O(2)-Si(1)-O(1)	113.50(4)
O(2)-Si(1)-C(16)	103.42(5)
O(1)-Si(1)-C(16)	101.80(5)
O(2)-Si(1)-C(12)	113.68(5)
O(1)-Si(1)-C(12)	107.85(5)
C(16)-Si(1)-C(12)	116.13(5)
C(6)-N(1)-C(1)	59.51(7)
C(6)-N(1)-S(1)	117.90(7)
C(1)-N(1)-S(1)	122.49(7)
C(7)-O(1)-Si(1)	132.84(7)
C(8)-O(2)-Si(1)	142.64(7)
C(6)-C(1)-N(1)	59.92(7)
C(6)-C(1)-C(7)	115.92(9)
N(1)-C(1)-C(7)	123.70(9)
C(6)-C(1)-C(2)	120.75(9)
N(1)-C(1)-C(2)	110.69(9)
C(7)-C(1)-C(2)	115.11(9)
C(1)-C(2)-C(3)	114.73(9)
C(4)-C(3)-C(2)	113.22(10)
C(3)-C(4)-C(5)	112.79(9)
C(6)-C(5)-C(4)	110.70(9)
C(6)-C(5)-C(8)	104.93(8)
C(4)-C(5)-C(8)	117.13(9)
C(1)-C(6)-N(1)	60.57(7)
C(1)-C(6)-C(5)	120.62(9)
N(1)-C(6)-C(5)	120.55(9)
O(1)-C(7)-C(1)	107.16(8)
O(2)-C(8)-C(9)	108.18(9)
O(2)-C(8)-C(5)	111.42(8)
C(9)-C(8)-C(5)	113.99(9)
C(11)-C(9)-C(8)	110.58(9)
C(11)-C(9)-C(10)	109.01(10)

C(8)-C(9)-C(10)	111.64(9)
C(13)-C(12)-C(15)	108.11(10)
C(13)-C(12)-C(14)	108.81(11)
C(15)-C(12)-C(14)	106.38(10)
C(13)-C(12)-Si(1)	111.13(8)
C(15)-C(12)-Si(1)	112.49(8)
C(14)-C(12)-Si(1)	109.75(8)
C(18)-C(16)-C(17)	108.61(10)
C(18)-C(16)-C(19)	107.21(10)
C(17)-C(16)-C(19)	107.60(10)
C(18)-C(16)-Si(1)	111.86(8)
C(17)-C(16)-Si(1)	113.04(8)
C(19)-C(16)-Si(1)	108.27(8)
C(25)-C(20)-C(21)	120.75(11)
C(25)-C(20)-S(1)	119.58(9)
C(21)-C(20)-S(1)	119.66(9)
C(22)-C(21)-C(20)	118.91(12)
C(21)-C(22)-C(23)	121.36(12)
C(24)-C(23)-C(22)	118.54(11)
C(24)-C(23)-C(26)	120.63(12)
C(22)-C(23)-C(26)	120.83(12)
C(23)-C(24)-C(25)	120.96(12)
C(20)-C(25)-C(24)	119.45(12)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for aziridine **8**. 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}
S(1)	17(1)	17(1)	14(1)	-2(1)	-3(1)	-1(1)
Si(1)	11(1)	14(1)	14(1)	-1(1)	-1(1)	-2(1)
N(1)	15(1)	16(1)	14(1)	0(1)	-3(1)	-2(1)
O(1)	12(1)	17(1)	18(1)	2(1)	-2(1)	-2(1)
O(2)	11(1)	18(1)	16(1)	-1(1)	-1(1)	-4(1)
O(3)	25(1)	26(1)	17(1)	2(1)	0(1)	-4(1)
O(4)	22(1)	20(1)	19(1)	-6(1)	-5(1)	0(1)
C(1)	14(1)	15(1)	14(1)	-1(1)	-2(1)	-2(1)
C(2)	18(1)	19(1)	16(1)	-5(1)	-1(1)	-1(1)
C(3)	22(1)	18(1)	19(1)	-5(1)	-4(1)	-3(1)
C(4)	17(1)	18(1)	23(1)	-3(1)	-5(1)	-5(1)
C(5)	12(1)	17(1)	15(1)	-1(1)	-1(1)	-3(1)
C(6)	14(1)	14(1)	14(1)	-1(1)	-2(1)	-2(1)
C(7)	12(1)	16(1)	17(1)	0(1)	-3(1)	-2(1)
C(8)	11(1)	17(1)	15(1)	-2(1)	0(1)	-2(1)
C(9)	13(1)	20(1)	18(1)	-1(1)	-4(1)	-2(1)
C(10)	12(1)	29(1)	27(1)	2(1)	-3(1)	-4(1)
C(11)	17(1)	23(1)	28(1)	2(1)	-6(1)	0(1)
C(12)	16(1)	17(1)	20(1)	-5(1)	-2(1)	-3(1)
C(13)	35(1)	18(1)	28(1)	-5(1)	-6(1)	3(1)
C(14)	21(1)	35(1)	37(1)	-17(1)	-1(1)	-11(1)
C(15)	28(1)	23(1)	19(1)	-7(1)	-2(1)	-6(1)
C(16)	16(1)	18(1)	16(1)	-1(1)	-1(1)	-3(1)
C(17)	24(1)	30(1)	24(1)	-2(1)	2(1)	-12(1)
C(18)	25(1)	26(1)	19(1)	1(1)	-5(1)	0(1)
C(19)	24(1)	24(1)	17(1)	-4(1)	0(1)	-1(1)
C(20)	18(1)	18(1)	16(1)	-2(1)	-4(1)	1(1)
C(21)	24(1)	22(1)	31(1)	-9(1)	-8(1)	-1(1)
C(22)	29(1)	21(1)	35(1)	-12(1)	-7(1)	3(1)
C(23)	23(1)	23(1)	20(1)	0(1)	-2(1)	3(1)
C(24)	20(1)	25(1)	32(1)	-4(1)	-8(1)	-1(1)
C(25)	23(1)	20(1)	30(1)	-7(1)	-8(1)	0(1)
C(26)	25(1)	29(1)	36(1)	-2(1)	-1(1)	7(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for aziridine **8**.

	x	y	z	U(eq)
H(2A)	8220(15)	8913(14)	1156(11)	20(3)
H(2B)	7820(15)	7691(15)	1935(11)	21(4)
H(3A)	6096(16)	7986(15)	974(12)	22(4)
H(3B)	6034(15)	9515(14)	828(11)	19(3)
H(4A)	4872(16)	7974(15)	2672(12)	25(4)
H(4B)	4005(16)	8850(15)	1899(12)	23(4)
H(5A)	4047(15)	9900(14)	3357(12)	20(3)
H(6A)	6232(14)	10589(14)	3564(11)	15(3)
H(7A)	8749(15)	10530(14)	3102(11)	16(3)
H(7B)	9603(15)	9653(14)	2330(11)	14(3)
H(8A)	4467(14)	11892(13)	2737(10)	11(3)
H(9A)	3574(15)	11033(14)	1077(12)	19(3)
H(10A)	1821(17)	11651(16)	2950(13)	30(4)
H(10B)	2083(16)	10244(16)	2595(12)	25(4)
H(10C)	1249(18)	11378(17)	1971(13)	33(4)
H(11A)	2327(18)	13174(16)	823(13)	35(4)
H(11B)	3936(18)	13311(16)	776(13)	33(4)
H(11C)	2854(17)	13524(16)	1790(13)	30(4)
H(13A)	6901(19)	15197(17)	1009(14)	38(5)
H(13B)	6383(18)	15530(17)	2147(13)	34(4)
H(13C)	5483(19)	14679(17)	1673(14)	37(5)
H(14A)	8703(18)	14690(17)	2532(13)	35(4)
H(14B)	9290(20)	14172(19)	1457(16)	48(5)
H(14C)	9300(20)	13190(19)	2536(15)	45(5)
H(15A)	5677(19)	13110(17)	3366(13)	35(4)
H(15B)	6651(17)	14021(16)	3646(13)	30(4)
H(15C)	7096(18)	12549(18)	3618(14)	34(4)
H(17A)	8937(18)	14265(18)	-175(13)	34(4)
H(17B)	9954(18)	12965(16)	-50(13)	30(4)
H(17C)	9439(17)	13486(16)	-1110(13)	29(4)
H(18A)	6345(17)	14220(17)	-315(13)	29(4)

H(18B)	6987(17)	13598(16)	-1305(13)	30(4)
H(18C)	5879(17)	12867(15)	-462(12)	25(4)
H(19A)	7464(17)	10788(16)	-387(12)	28(4)
H(19B)	9047(17)	10856(15)	-278(12)	26(4)
H(19C)	8475(17)	11542(15)	-1302(13)	29(4)
H(21A)	8431(17)	6116(16)	3913(13)	30(4)
H(22A)	10658(19)	4998(19)	3565(14)	40(5)
H(24A)	12447(18)	7817(17)	4380(13)	35(4)
H(25A)	10303(19)	9002(18)	4687(14)	39(5)
H(26A)	13800(30)	5750(30)	4060(20)	85(8)
H(26B)	13150(30)	4620(30)	3880(20)	77(8)
H(26C)	13560(30)	5600(30)	3050(20)	90(9)

Table 6. Torsion angles [°] for aziridine **8**.

O(3)-S(1)-N(1)-C(6)	109.81(8)
O(4)-S(1)-N(1)-C(6)	-19.42(9)
C(20)-S(1)-N(1)-C(6)	-135.55(8)
O(3)-S(1)-N(1)-C(1)	179.72(8)
O(4)-S(1)-N(1)-C(1)	50.49(10)
C(20)-S(1)-N(1)-C(1)	-65.63(9)
O(2)-Si(1)-O(1)-C(7)	-47.50(11)
C(16)-Si(1)-O(1)-C(7)	-157.95(10)
C(12)-Si(1)-O(1)-C(7)	79.37(10)
O(1)-Si(1)-O(2)-C(8)	86.35(12)
C(16)-Si(1)-O(2)-C(8)	-164.20(11)
C(12)-Si(1)-O(2)-C(8)	-37.40(13)
S(1)-N(1)-C(1)-C(6)	-105.59(9)
C(6)-N(1)-C(1)-C(7)	102.76(11)
S(1)-N(1)-C(1)-C(7)	-2.84(14)
C(6)-N(1)-C(1)-C(2)	-114.38(10)
S(1)-N(1)-C(1)-C(2)	140.03(8)
C(6)-C(1)-C(2)-C(3)	13.44(15)
N(1)-C(1)-C(2)-C(3)	79.94(12)
C(7)-C(1)-C(2)-C(3)	-133.74(10)
C(1)-C(2)-C(3)-C(4)	-35.19(14)
C(2)-C(3)-C(4)-C(5)	57.30(13)

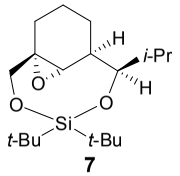
C(3)-C(4)-C(5)-C(6)	-54.20(13)
C(3)-C(4)-C(5)-C(8)	66.01(12)
C(7)-C(1)-C(6)-N(1)	-115.56(10)
C(2)-C(1)-C(6)-N(1)	97.51(11)
N(1)-C(1)-C(6)-C(5)	-110.14(11)
C(7)-C(1)-C(6)-C(5)	134.30(10)
C(2)-C(1)-C(6)-C(5)	-12.63(15)
S(1)-N(1)-C(6)-C(1)	113.18(8)
C(1)-N(1)-C(6)-C(5)	110.25(11)
S(1)-N(1)-C(6)-C(5)	-136.57(9)
C(4)-C(5)-C(6)-C(1)	32.09(14)
C(8)-C(5)-C(6)-C(1)	-95.16(11)
C(4)-C(5)-C(6)-N(1)	-39.63(13)
C(8)-C(5)-C(6)-N(1)	-166.88(9)
Si(1)-O(1)-C(7)-C(1)	52.06(13)
C(6)-C(1)-C(7)-O(1)	-83.54(11)
N(1)-C(1)-C(7)-O(1)	-153.32(9)
C(2)-C(1)-C(7)-O(1)	65.27(12)
Si(1)-O(2)-C(8)-C(9)	146.34(10)
Si(1)-O(2)-C(8)-C(5)	-87.60(13)
C(6)-C(5)-C(8)-O(2)	51.82(11)
C(4)-C(5)-C(8)-O(2)	-71.39(12)
C(6)-C(5)-C(8)-C(9)	174.61(9)
C(4)-C(5)-C(8)-C(9)	51.40(13)
O(2)-C(8)-C(9)-C(11)	-62.66(12)
C(5)-C(8)-C(9)-C(11)	172.80(9)
O(2)-C(8)-C(9)-C(10)	175.79(9)
C(5)-C(8)-C(9)-C(10)	51.25(13)
O(2)-Si(1)-C(12)-C(13)	-70.05(9)
O(1)-Si(1)-C(12)-C(13)	163.18(8)
C(16)-Si(1)-C(12)-C(13)	49.78(10)
O(2)-Si(1)-C(12)-C(15)	51.33(10)
O(1)-Si(1)-C(12)-C(15)	-75.44(9)
C(16)-Si(1)-C(12)-C(15)	171.16(8)
O(2)-Si(1)-C(12)-C(14)	169.55(8)
O(1)-Si(1)-C(12)-C(14)	42.78(10)
C(16)-Si(1)-C(12)-C(14)	-70.62(10)
O(2)-Si(1)-C(16)-C(18)	48.39(9)

O(1)-Si(1)-C(16)-C(18)	166.33(8)
C(12)-Si(1)-C(16)-C(18)	-76.85(9)
O(2)-Si(1)-C(16)-C(17)	171.36(8)
O(1)-Si(1)-C(16)-C(17)	-70.70(9)
C(12)-Si(1)-C(16)-C(17)	46.12(10)
O(2)-Si(1)-C(16)-C(19)	-69.53(8)
O(1)-Si(1)-C(16)-C(19)	48.41(9)
C(12)-Si(1)-C(16)-C(19)	165.23(8)
O(3)-S(1)-C(20)-C(25)	-122.15(10)
O(4)-S(1)-C(20)-C(25)	7.13(12)
N(1)-S(1)-C(20)-C(25)	126.61(10)
O(3)-S(1)-C(20)-C(21)	56.77(11)
O(4)-S(1)-C(20)-C(21)	-173.95(9)
N(1)-S(1)-C(20)-C(21)	-54.48(11)
C(25)-C(20)-C(21)-C(22)	0.39(19)
S(1)-C(20)-C(21)-C(22)	-178.51(10)
C(20)-C(21)-C(22)-C(23)	0.7(2)
C(21)-C(22)-C(23)-C(24)	-1.3(2)
C(21)-C(22)-C(23)-C(26)	178.20(13)
C(22)-C(23)-C(24)-C(25)	0.8(2)
C(26)-C(23)-C(24)-C(25)	-178.69(13)
C(21)-C(20)-C(25)-C(24)	-0.85(19)
S(1)-C(20)-C(25)-C(24)	178.04(10)
C(23)-C(24)-C(25)-C(20)	0.3(2)

III. Selected Spectra

(Next page)

^{1}H Spectrum:



7

Current Data Parameters
USER michel
NAME mp-II-p040-p
EXPNO 1
PROCNO 1

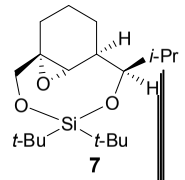
F2 - Acquisition Parameters
Date_ 20090126
Time 16.08
INSTRUM drx400
PROBHD 5 mm QNP H/ F/ P
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 143.7
DW 78.000 usec
DE 4.50 usec
TE 298.1 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.000 ppm
F1 3201.04 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/ cm
HZCM 140.39650 Hz/ cm

^{13}C Spectrum with ^{1}H decoupling:



7

Current Data Parameters
USER michel
NAME mp-II-p040-p-c13-c6d6
EXPNO 1
PROCNO 1

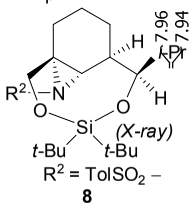
F2 - Acquisition Parameters
Date_ 20090127
Time 16.58
INSTRUM drx400
PROBHD 5 mm QNP H/ F/ P
PULPROG zgdc30
TD 65536
SOLVENT C6D6
NS 246
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 9195.2
DW 20.700 usec
DE 20.39 usec
TE 298.1 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 10.75 usec
PL1 0.00 dB
SFO1 100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 100.6126973 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

^{1}H Spectrum:



8

Current Data Parameters
USER michel
NAME mp-II-p044-f1
EXPNO 1
PROCNO 1

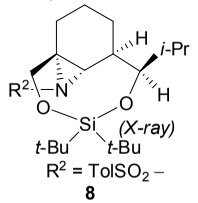
F2 - Acquisition Parameters
Date_ 20090130
Time 10.27
INSTRUM drx400
PROBHD 5 mm QNP H/ F/ P
PULPROG zg30
TD 65536
SOLVENT c6d6
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 181
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/ cm
HZCM 157.94606 Hz/ cm

^{13}C Spectrum with ^{1}H decoupling:



R^2 = $TolSO_2$ –

8

Current Data Parameters
USER michel
NAME mp-II-p044-f1-cryo-c6d6
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090202
Time 12.22
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT c6d6
NS 65
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0814105 sec
RG 5160.6
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

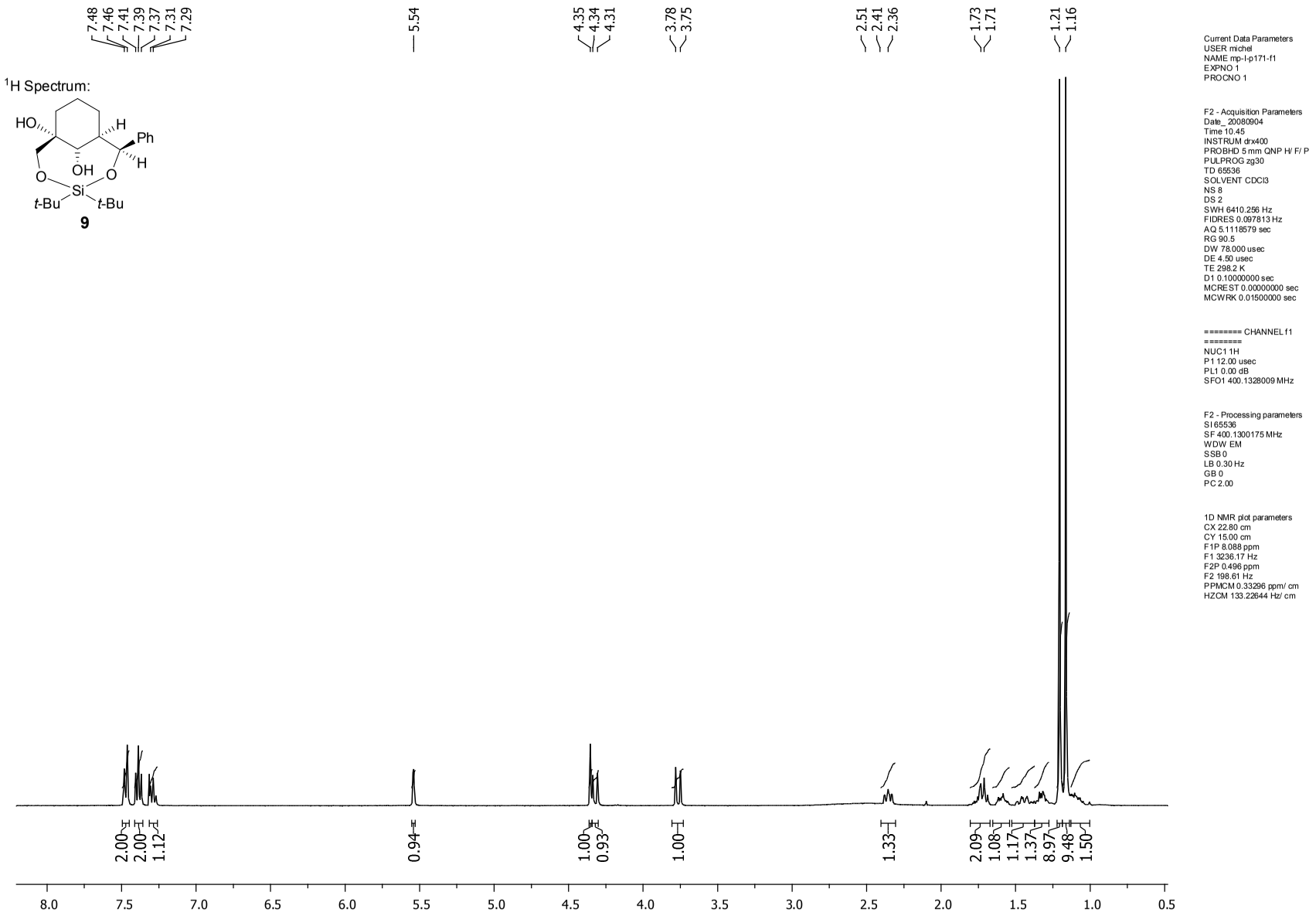
======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

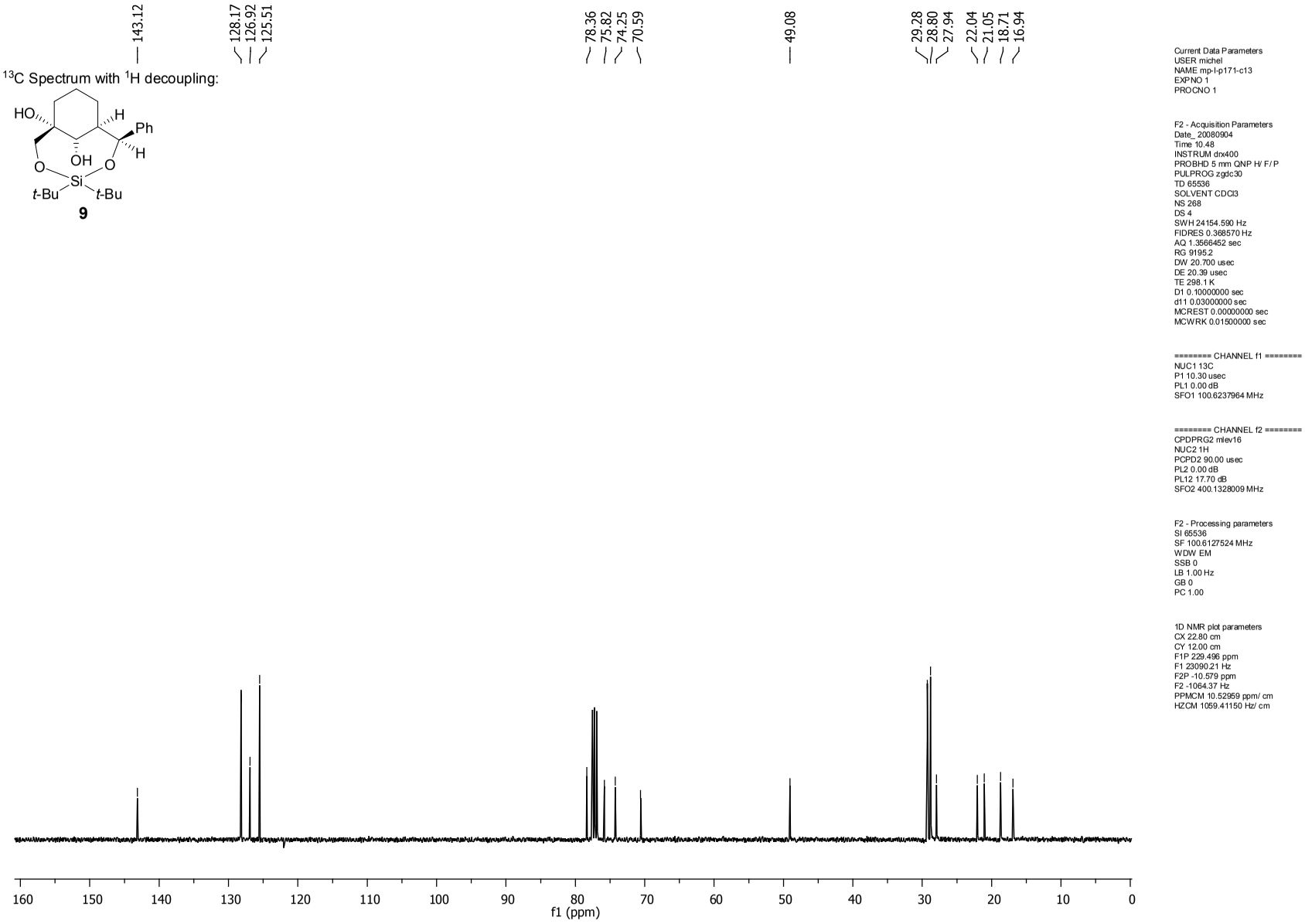
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

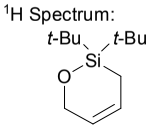
F2 - Processing parameters
SI 65536
SF 125.7803337 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 50.00 cm
F1P 230.637 ppm
F1 29009.66 Hz
F2P -10.287 ppm

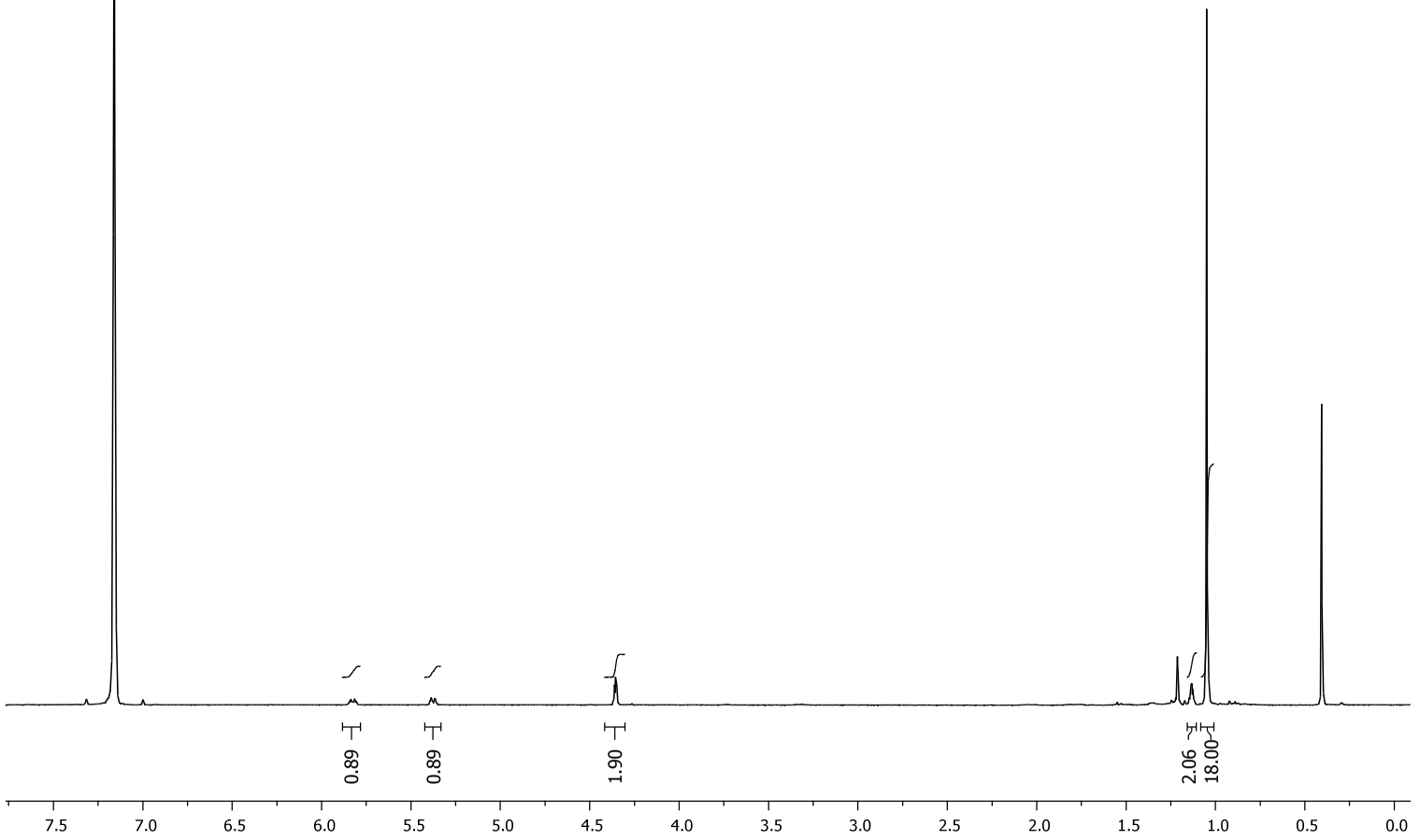




^{1}H Spectrum:



11b

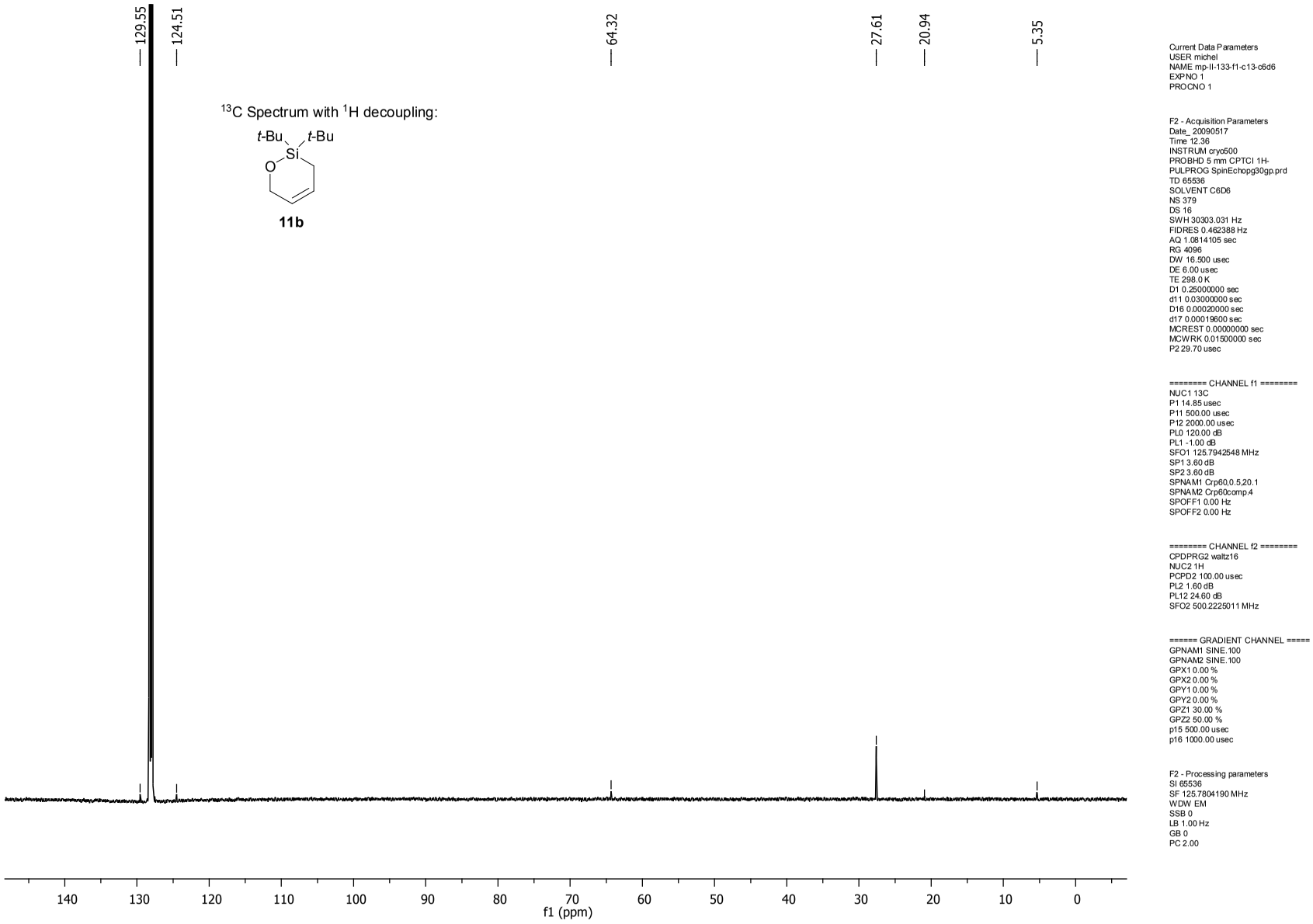


Current Data Parameters
USER michel
NAME mp-II-133-f1-c6d6
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090517
Time 12.32
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT C6D6
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0999398 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00



Current Data Parameters
USER michel
NAME mp-II-133-f1-c13-c6d6
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090517
Time 12.36
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT C6D6
NS 379
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0814105 sec
RG 4096
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

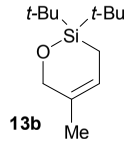
======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

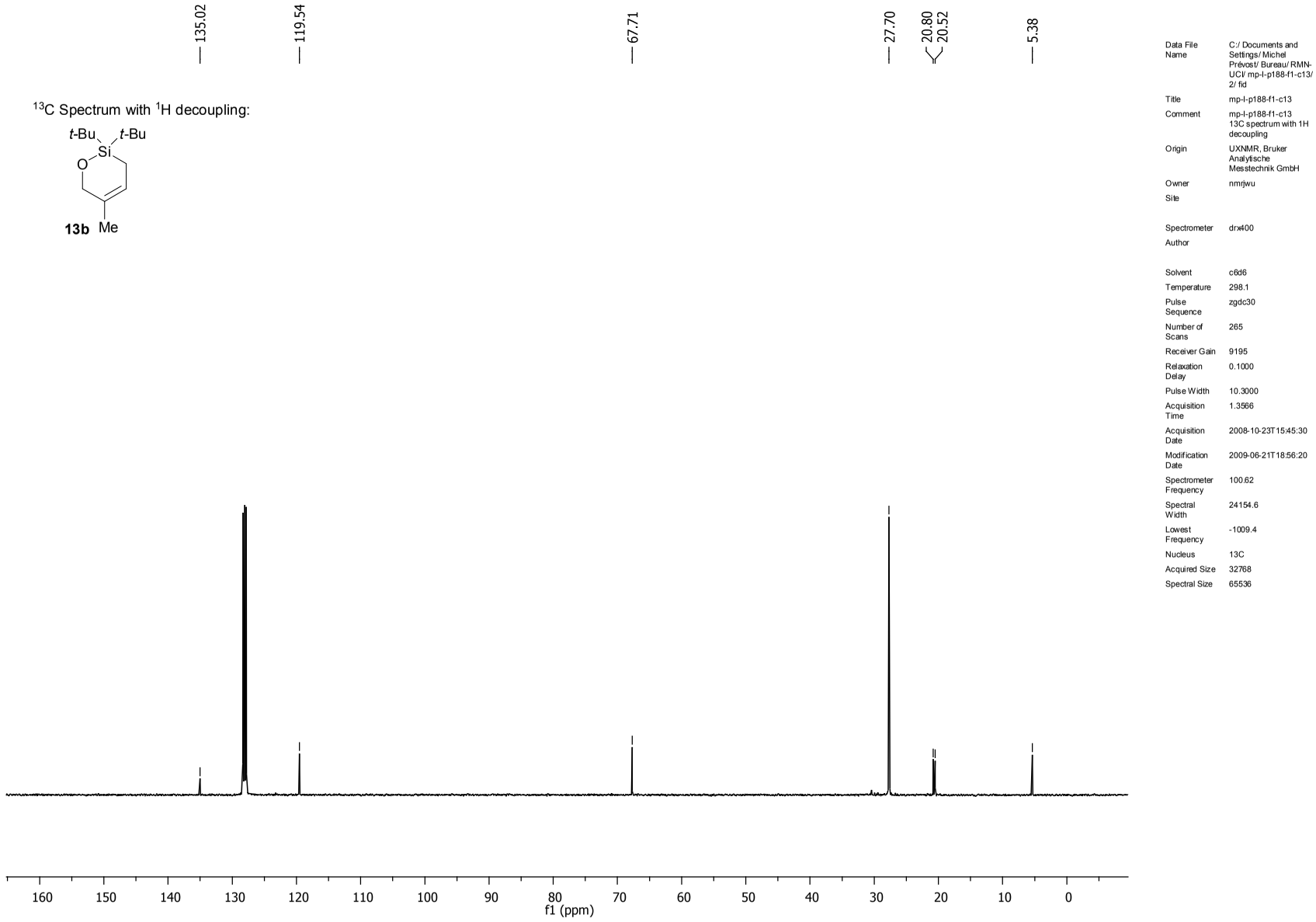
F2 - Processing parameters
SI 65536
SF 125.7804190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

^{1}H Spectrum:



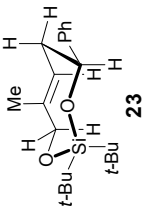
13b

Data File Name	C:/ Documents and Settings/ Michel Prévost/ Bureau/ RMN-UCI/ mp-l-p188-f1/ 1/ fid
Title	mp-l-p188-f1
Comment	mp-l-p188-f1 1H spectrum
Origin	UXNMR, Bruker Analytische Messtechnik GmbH
Owner	nmrjwu
Site	
Spectrometer	drx400
Author	
Solvent	C6D6
Temperature	298.0
Pulse Sequence	zg30
Number of Scans	8
Receiver Gain	256
Relaxation Delay	0.1000
Pulse Width	12.0000
Acquisition Time	5.1119
Acquisition Date	2008-10-23T13 :43:18
Modification Date	2009-06-21T18 :56:09
Spectrometer Frequency	400.13
Spectral Width	6410.3
Lowest Frequency	-401.0
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



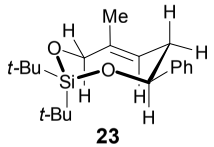
Data File Name	C:/ Documents and Settings/ Michel Prévost/ Bureau/ RMN-UCI/ mp-l-p188-f1-c13/ 2/ fid
Title	mp-l-p188-f1-c13
Comment	mp-l-p188-f1-c13 13C spectrum with 1H decoupling
Origin	UXNMR, Bruker Analytische Messtechnik GmbH
Owner	nmrjwu
Site	
Spectrometer	drx400
Author	
Solvent	c6d6
Temperature	298.1
Pulse Sequence	zgdc30
Number of Scans	265
Receiver Gain	9195
Relaxation Delay	0.1000
Pulse Width	10.3000
Acquisition Time	1.3566
Acquisition Date	2008-10-23T15:45:30
Modification Date	2009-06-21T18:56:20
Spectrometer Frequency	100.62
Spectral Width	24154.6
Lowest Frequency	-1009.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

^{1}H Spectrum:



23

^{13}C Spectrum with ^{1}H decoupling:



23

Current Data Parameters
USER michel
NAME mp-II-135-pur-c6d6-c13
EXPNO 1
PROCNO 1

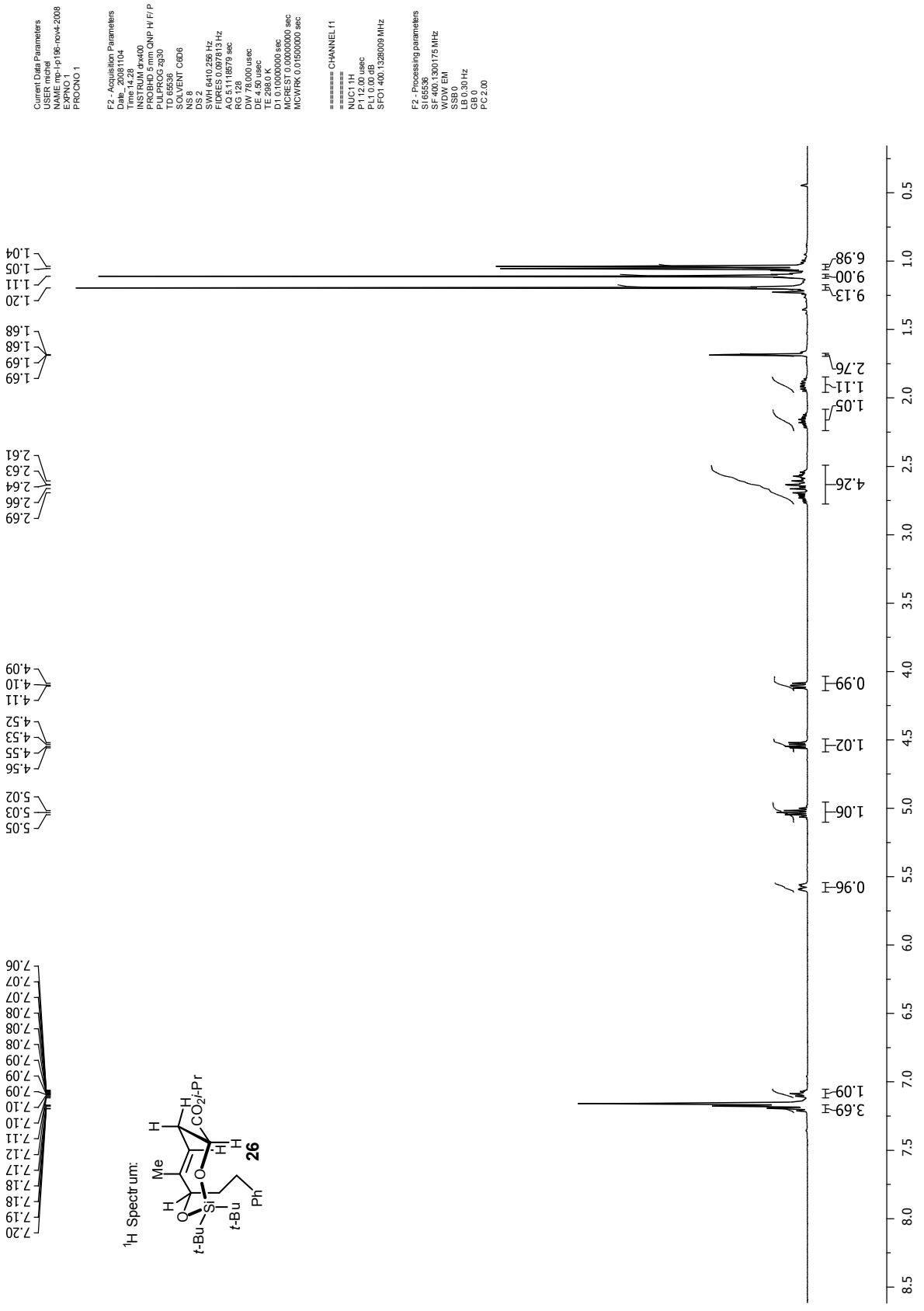
F2 - Acquisition Parameters
Date_ 20090521
Time 10.51
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT c6d6
NS 115
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0814105 sec
RG 3649.1
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

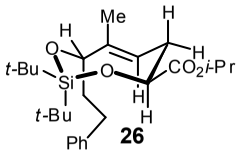
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7803314 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00



Z-restored spin-echo 13C spectrum with 1H decoupling

^{13}C Spectrum with ^{1}H decoupling:



26


```
Current Data Parameters
USER              michel
NAME      mp-I-p196-crys-c6d6
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081105
Time              15.42
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG   SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  91
DS                  16
SWH           30303.031 Hz
FIDRES        0.462388 Hz
AQ            1.0814105 sec
RG              7296.2
DW              16.500 usec
DE               6.00 usec
TE              298.0 K
D1            0.25000000 sec
d11           0.03000000 sec
D15           0.00020000 sec
d17           0.00019600 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
P2              29.70 usec

======== CHANNEL f1 ========
NUC1              13C
P1              14.85 usec
P11            500.00 usec
P12           2000.00 usec
PL0            120.00 dB
PL1             -1.00 dB
SFO1       125.7942548 MHz
SP1             3.69 dB
SP2             3.69 dB
SPNAM1   Crp60.0.5.20.1
SPNAM2     Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2           100.00 usec
PL2             1.60 dB
PL12            24.60 dB
SFO2       500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPX1               0.00 %
GPX2               0.00 %
GPY1               0.00 %
GPY2               0.00 %
GPZ1              30.00 %
GPZ2              50.00 %
p15             500.00 usec
p16            1000.00 usec

F2 - Processing parameters
SI                65536
SF          125.7803323 MHz
WDW                EM
SSB                0
LB              1.00 Hz
GB                 0
PC              2.00

1D NMR plot parameters
CX              22.00 cm
CY              15.65 cm
F1P           230.637 ppm
F1            29009.66 Hz
F2P           -10.287 ppm
F2            -1293.96 Hz
PPMCM         10.56688 ppm/cm
HZCM        1329.10626 Hz/cm
```