Synthesis of D-Galactosamine and D-Allosamine Derivatives via a Microwave-Assisted Preparation of 1,6-Anhydroglucosamine

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SUPPORTING INFORMATION

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X-ray Analysis

Single-crystal X-ray structure of 7 (CCDC 1498510)

Table 1. Crystal data and structure refinement for **7**.

Identification code	i14251	
Empirical formula	C14 H26 Cl3 N O5 Si2	
Formula weight	450.89	
Temperature	100.0(1) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 9.4564(2) Å	α= 90°.
	b = 11.1358(2) Å	β= 90°.
	c = 20.3440(5) Å	γ = 90°.
Volume	2142.32(8) $Å^3$	
Z	4	
Density (calculated)	1.398 Mg/m^3	
Absorption coefficient	0.563 mm^{-1}	
F(000)	944	
Crystal size	0.30 x 0.26 x 0.20 mm^3	
Theta range for data collection	2.00 to 27.10°.	
Index ranges	-12<=h<=10, -10<=k<=14, -26<=l<=26	
Reflections collected	19906	
Independent reflections	4547 [R(int) = 0.0213]	
Completeness to theta = 25.00°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8958 and 0.8494	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4547 / 0 / 236	

Goodness-of-fit on F^2	1.029
Final R indices [I>2sigma(I)]	R1 = 0.0231, wR2 = 0.0517
R indices (all data)	R1 = 0.0254, wR2 = 0.0529
Absolute structure parameter	-0.01(4)
Largest diff. peak and hole	0.298 and -0.234 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for i14251. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	1253(1)	4082(1)	-531(1)	29(1)
Cl(2)	-711(1)	2139(1)	-400(1)	33(1)
Cl(3)	-1478(1)	4471(1)	76(1)	41(1)
Si(1)	3431(1)	122(1)	2345(1)	15(1)
Si(2)	-842(1)	-1507(1)	587(1)	14(1)
O(1)	-1862(1)	1603(1)	1808(1)	17(1)
O(3)	1689(1)	316(1)	2298(1)	16(1)
O(4)	-866(1)	-220(1)	1011(1)	16(1)
O(6)	-523(1)	1942(1)	2710(1)	19(1)
O(7)	1241(1)	4056(1)	933(1)	24(1)
N(2)	307(2)	2182(1)	946(1)	16(1)
C(1)	-691(2)	2263(2)	2040(1)	16(1)
C(2)	621(2)	1921(1)	1637(1)	14(1)
C(3)	963(2)	567(1)	1701(1)	14(1)
C(4)	-407(2)	-165(1)	1684(1)	14(1)
C(5)	-1583(2)	435(1)	2083(1)	17(1)
C(6)	-1158(2)	765(1)	2786(1)	19(1)
C(7)	580(2)	3247(1)	679(1)	15(1)
C(8)	-48(2)	3453(2)	-22(1)	16(1)
C(9)	4001(2)	-1014(2)	1729(1)	22(1)

C(10)	4319(2)	1584(2)	2191(1)	25(1)
C(11)	3761(2)	-416(2)	3193(1)	22(1)
C(12)	-2293(2)	-2461(2)	911(1)	25(1)
C(13)	879(2)	-2276(2)	702(1)	23(1)
C(14)	-1040(2)	-1059(2)	-286(1)	18(1)

Table 3. Bond lengths [Å] and angles [°] for i14251.

Cl(1)-C(8)	1.7536(16)
Cl(2)-C(8)	1.7683(17)
Cl(3)-C(8)	1.7751(17)
Si(1)-O(3)	1.6641(11)
Si(1)-C(11)	1.8532(16)
Si(1)-C(10)	1.8579(18)
Si(1)-C(9)	1.8611(17)
Si(2)-O(4)	1.6727(11)
Si(2)-C(14)	1.8542(16)
Si(2)-C(13)	1.8542(18)
Si(2)-C(12)	1.8565(17)
O(1)-C(1)	1.4103(19)
O(1)-C(5)	1.4403(19)
O(3)-C(3)	1.4225(17)
O(4)-C(4)	1.4387(17)
O(6)-C(1)	1.4189(17)
O(6)-C(6)	1.4507(19)
O(7)-C(7)	1.2121(19)
N(2)-C(7)	1.329(2)
N(2)-C(2)	1.4666(18)
N(2)-H(2A)	0.751(19)
C(1)-C(2)	1.535(2)

C(1)-H(1)	1.0000
C(2)-C(3)	1.547(2)
C(2)-H(2)	1.0000
C(3)-C(4)	1.531(2)
C(3)-H(3)	1.0000
C(4)-C(5)	1.530(2)
C(4)-H(4)	1.0000
C(5)-C(6)	1.530(2)
C(5)-H(5)	1.0000
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.562(2)
C(9)-H(9A)	0.9800
C(9)-H(9B)	0.9800
C(9)-H(9C)	0.9800
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

O(3)-Si(1)-C(11)	105.19(7)
O(3)-Si(1)-C(10)	108.92(7)
C(11)-Si(1)-C(10)	111.37(8)
O(3)-Si(1)-C(9)	109.64(7)
C(11)-Si(1)-C(9)	111.00(8)
C(10)-Si(1)-C(9)	110.55(8)
O(4)-Si(2)-C(14)	105.18(7)
O(4)-Si(2)-C(13)	110.02(7)
C(14)-Si(2)-C(13)	109.53(7)
O(4)-Si(2)-C(12)	107.29(7)
C(14)-Si(2)-C(12)	114.82(8)
C(13)-Si(2)-C(12)	109.84(8)
C(1)-O(1)-C(5)	101.34(11)
C(3)-O(3)-Si(1)	123.52(9)
C(4)-O(4)-Si(2)	121.59(9)
C(1)-O(6)-C(6)	106.49(12)
C(7)-N(2)-C(2)	121.95(14)
C(7)-N(2)-H(2A)	121.4(14)
C(2)-N(2)-H(2A)	114.9(14)
O(1)-C(1)-O(6)	106.19(12)
O(1)-C(1)-C(2)	109.04(12)
O(6)-C(1)-C(2)	111.05(13)
O(1)-C(1)-H(1)	110.2
O(6)-C(1)-H(1)	110.2
C(2)-C(1)-H(1)	110.2
N(2)-C(2)-C(1)	107.36(12)
N(2)-C(2)-C(3)	108.42(12)
C(1)-C(2)-C(3)	111.47(12)
N(2)-C(2)-H(2)	109.8

C(1)-C(2)-H(2)	109.8
C(3)-C(2)-H(2)	109.8
O(3)-C(3)-C(4)	108.83(12)
O(3)-C(3)-C(2)	111.34(12)
C(4)-C(3)-C(2)	109.89(12)
O(3)-C(3)-H(3)	108.9
C(4)-C(3)-H(3)	108.9
C(2)-C(3)-H(3)	108.9
O(4)-C(4)-C(5)	107.76(12)
O(4)-C(4)-C(3)	107.41(11)
C(5)-C(4)-C(3)	111.75(12)
O(4)-C(4)-H(4)	110.0
C(5)-C(4)-H(4)	110.0
C(3)-C(4)-H(4)	110.0
O(1)-C(5)-C(4)	108.70(12)
O(1)-C(5)-C(6)	101.29(12)
C(4)-C(5)-C(6)	114.18(13)
O(1)-C(5)-H(5)	110.8
C(4)-C(5)-H(5)	110.8
C(6)-C(5)-H(5)	110.8
O(6)-C(6)-C(5)	103.04(12)
O(6)-C(6)-H(6A)	111.2
C(5)-C(6)-H(6A)	111.2
O(6)-C(6)-H(6B)	111.2
C(5)-C(6)-H(6B)	111.2
H(6A)-C(6)-H(6B)	109.1
O(7)-C(7)-N(2)	126.11(14)
O(7)-C(7)-C(8)	118.43(14)
N(2)-C(7)-C(8)	115.45(13)
C(7)-C(8)-Cl(1)	109.31(10)

C(7)-C(8)-Cl(2)	114.26(11)
Cl(1)-C(8)-Cl(2)	108.78(8)
C(7)-C(8)-Cl(3)	106.36(10)
Cl(1)-C(8)-Cl(3)	110.19(9)
Cl(2)-C(8)-Cl(3)	107.89(8)
Si(1)-C(9)-H(9A)	109.5
Si(1)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
Si(1)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
Si(1)-C(10)-H(10A)	109.5
Si(1)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
Si(1)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
Si(1)-C(11)-H(11A)	109.5
Si(1)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
Si(1)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
Si(2)-C(12)-H(12A)	109.5
Si(2)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
Si(2)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
Si(2)-C(13)-H(13A)	109.5

Si(2)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
Si(2)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
Si(2)-C(14)-H(14A)	109.5
Si(2)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
Si(2)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for i14251. 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}
Cl(1)	27(1)	42(1)	17(1)	10(1)	1(1)	-13(1)
Cl(2)	59(1)	24(1)	17(1)	4(1)	-11(1)	-17(1)
Cl(3)	41(1)	53(1)	29(1)	5(1)	2(1)	30(1)
Si(1)	16(1)	17(1)	14(1)	0(1)	-2(1)	1(1)
Si(2)	16(1)	13(1)	13(1)	-1(1)	1(1)	0(1)
O(1)	17(1)	15(1)	20(1)	1(1)	-1(1)	1(1)
O(3)	16(1)	20(1)	12(1)	2(1)	-2(1)	1(1)
O(4)	22(1)	12(1)	13(1)	-1(1)	-5(1)	0(1)
O(6)	28(1)	16(1)	13(1)	0(1)	1(1)	-1(1)
O(7)	37(1)	18(1)	17(1)	0(1)	-1(1)	-9(1)
N(2)	20(1)	14(1)	13(1)	1(1)	-4(1)	-4(1)
C(1)	22(1)	14(1)	14(1)	0(1)	-2(1)	2(1)
C(2)	17(1)	14(1)	11(1)	2(1)	-2(1)	-2(1)

C(3)	14(1)	17(1)	11(1)	0(1)	-2(1)	2(1)
C(4)	19(1)	13(1)	11(1)	0(1)	-1(1)	-1(1)
C(5)	18(1)	16(1)	17(1)	2(1)	1(1)	-2(1)
C(6)	24(1)	17(1)	15(1)	-1(1)	5(1)	-1(1)
C(7)	17(1)	16(1)	12(1)	0(1)	3(1)	1(1)
C(8)	19(1)	14(1)	15(1)	1(1)	2(1)	-1(1)
C(9)	21(1)	24(1)	22(1)	-2(1)	-2(1)	4(1)
C(10)	22(1)	24(1)	30(1)	-1(1)	-2(1)	-2(1)
C(11)	22(1)	26(1)	19(1)	1(1)	-5(1)	4(1)
C(12)	31(1)	23(1)	21(1)	-3(1)	4(1)	-8(1)
C(13)	26(1)	26(1)	18(1)	1(1)	2(1)	8(1)
C(14)	20(1)	19(1)	16(1)	-2(1)	0(1)	-2(1)

Single-crystal X-ray structure of 11 (CCDC 1498511)

Table 1. Crystal data and structure refinement for i14245.

Identification code	i14245	
Empirical formula	C17 H18 Cl3 N O6	
Formula weight	438.67	
Temperature	100.0(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 10.7676(4) Å	α= 90°.
	b = 6.1959(2) Å	β= 91.2550(10)°.
	c = 13.8977(5) Å	γ = 90°.
Volume	926.96(6) $Å^3$	
Z	2	
Density (calculated)	1.572 Mg/m^3	
Absorption coefficient	0.530 mm^{-1}	
F(000)	452	
Crystal size	0.36 x 0.14 x 0.04 mm^3	
Theta range for data collection	1.47 to 27.09°.	
Index ranges	-13<=h<=13, -7<=k<=7, -17<=l<=17	
Reflections collected	8208	
Independent reflections	3693 [R(int) = 0.0177]	
Completeness to theta = 25.00°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9791 and 0.8322	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3693 / 1 / 250	
Goodness-of-fit on F^2	1.037	
Final R indices [I>2sigma(I)]	R1 = 0.0287, wR2 = 0.0581	
R indices (all data)	R1 = 0.0324, wR2 = 0.0600	
Absolute structure parameter	-0.02(4)	
Extinction coefficient	0.0002(9)	
Largest diff. peak and hole	0.430 and -0.553 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)

for i14245. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	646(1)	5888(1)	5913(1)	28(1)
Cl(2)	1523(1)	7771(1)	7698(1)	31(1)
Cl(3)	1695(1)	10158(1)	5921(1)	43(1)
O(1)	5980(1)	10690(2)	5908(1)	18(1)
O(3)	6725(1)	6192(2)	7705(1)	15(1)
O(4)	5055(1)	11334(2)	7776(1)	16(1)
O(6)	7394(1)	7972(3)	5849(1)	20(1)
O(7)	3190(1)	4576(2)	6462(1)	21(1)
O(16)	4976(1)	10076(3)	9292(1)	23(1)
N(2)	4041(2)	7896(3)	6277(1)	16(1)
C(1)	6118(2)	8459(3)	5710(2)	18(1)
C(2)	5325(2)	7196(3)	6419(1)	15(1)
C(3)	5736(2)	7623(3)	7469(1)	13(1)
C(4)	6139(2)	10001(3)	7625(1)	14(1)
C(5)	6775(2)	10943(4)	6750(1)	16(1)
C(6)	7911(2)	9627(3)	6473(2)	18(1)
C(7)	3091(2)	6511(3)	6368(1)	16(1)
C(8)	1789(2)	7574(4)	6434(1)	19(1)
C(9)	6874(2)	5868(4)	8719(1)	18(1)
C(10)	7842(2)	4144(3)	8887(2)	15(1)
C(11)	7733(2)	2712(4)	9651(1)	16(1)
C(12)	8633(2)	1137(4)	9814(1)	20(1)
C(13)	9640(2)	983(4)	9214(2)	23(1)
C(14)	9744(2)	2395(4)	8446(2)	22(1)
C(15)	8857(2)	3981(4)	8286(2)	19(1)
C(16)	4559(2)	11221(3)	8662(1)	17(1)
C(17)	3461(2)	12674(4)	8746(2)	23(1)

Table 3. Bond lengths [Å] and angles [°] for i14245.

Cl(1)-C(8)	1.758(2)
Cl(2)-C(8)	1.790(2)

Cl(3)-C(8)	1.754(2)
O(1)-C(1)	1.418(3)
O(1)-C(5)	1.442(2)
O(3)-C(3)	1.418(2)
O(3)-C(9)	1.428(2)
O(4)-C(16)	1.356(2)
O(4)-C(4)	1.449(2)
O(6)-C(1)	1.416(3)
O(6)-C(6)	1.446(2)
O(7)-C(7)	1.211(3)
O(16)-C(16)	1.206(2)
N(2)-C(7)	1.343(3)
N(2)-C(2)	1.458(2)
N(2)-H(2A)	0.80(2)
C(1)-C(2)	1.532(3)
C(1)-H(1)	1.0000
C(2)-C(3)	1.539(3)
C(2)-H(2)	1.0000
C(3)-C(4)	1.549(3)
C(3)-H(3)	1.0000
C(4)-C(5)	1.524(3)
C(4)-H(4)	1.0000
C(5)-C(6)	1.526(3)
C(5)-H(5)	1.0000
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.554(3)
C(9)-C(10)	1.507(3)
C(9)-H(9A)	0.9900
C(9)-H(9B)	0.9900
C(10)-C(11)	1.391(3)
C(10)-C(15)	1.393(3)
C(11)-C(12)	1.391(3)
C(11)-H(11)	0.9500
C(12)-C(13)	1.386(3)
C(12)-H(12)	0.9500

C(13)-C(14)	1.386(3)
C(13)-H(13)	0.9500
C(14)-C(15)	1.385(3)
C(14)-H(14)	0.9500
C(15)-H(15)	0.9500
C(16)-C(17)	1.493(3)
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
C(1)-O(1)-C(5)	101.56(15)
C(3)-O(3)-C(9)	112.54(14)
C(16)-O(4)-C(4)	115.89(15)
C(1)-O(6)-C(6)	106.86(15)
C(7)-N(2)-C(2)	121.30(18)
C(7)-N(2)-H(2A)	119.8(17)
C(2)-N(2)-H(2A)	118.5(17)
O(6)-C(1)-O(1)	106.63(16)
O(6)-C(1)-C(2)	110.88(16)
O(1)-C(1)-C(2)	108.13(16)
O(6)-C(1)-H(1)	110.4
O(1)-C(1)-H(1)	110.4
C(2)-C(1)-H(1)	110.4
N(2)-C(2)-C(1)	107.53(16)
N(2)-C(2)-C(3)	109.21(16)
C(1)-C(2)-C(3)	111.69(16)
N(2)-C(2)-H(2)	109.5
C(1)-C(2)-H(2)	109.5
C(3)-C(2)-H(2)	109.5
O(3)-C(3)-C(2)	108.08(15)
O(3)-C(3)-C(4)	110.84(15)
C(2)-C(3)-C(4)	111.66(16)
O(3)-C(3)-H(3)	108.7
C(2)-C(3)-H(3)	108.7
C(4)-C(3)-H(3)	108.7
O(4)-C(4)-C(5)	105.93(16)
O(4)-C(4)-C(3)	109.78(15)

C(5)-C(4)-C(3)	112.47(16)
O(4)-C(4)-H(4)	109.5
C(5)-C(4)-H(4)	109.5
C(3)-C(4)-H(4)	109.5
O(1)-C(5)-C(4)	109.67(15)
O(1)-C(5)-C(6)	101.67(15)
C(4)-C(5)-C(6)	111.88(18)
O(1)-C(5)-H(5)	111.1
C(4)-C(5)-H(5)	111.1
C(6)-C(5)-H(5)	111.1
O(6)-C(6)-C(5)	103.29(15)
O(6)-C(6)-H(6A)	111.1
C(5)-C(6)-H(6A)	111.1
O(6)-C(6)-H(6B)	111.1
C(5)-C(6)-H(6B)	111.1
H(6A)-C(6)-H(6B)	109.1
O(7)-C(7)-N(2)	125.3(2)
O(7)-C(7)-C(8)	119.4(2)
N(2)-C(7)-C(8)	115.17(18)
C(7)-C(8)-Cl(3)	113.99(14)
C(7)-C(8)-Cl(1)	110.39(15)
Cl(3)-C(8)-Cl(1)	109.95(11)
C(7)-C(8)-Cl(2)	104.53(13)
Cl(3)-C(8)-Cl(2)	109.11(12)
Cl(1)-C(8)-Cl(2)	108.62(11)
O(3)-C(9)-C(10)	108.36(16)
O(3)-C(9)-H(9A)	110.0
C(10)-C(9)-H(9A)	110.0
O(3)-C(9)-H(9B)	110.0
C(10)-C(9)-H(9B)	110.0
H(9A)-C(9)-H(9B)	108.4
C(11)-C(10)-C(15)	119.46(19)
C(11)-C(10)-C(9)	120.04(19)
C(15)-C(10)-C(9)	120.50(18)
C(10)-C(11)-C(12)	120.15(19)
C(10)-C(11)-H(11)	119.9

C(12)-C(11)-H(11)	119.9
C(13)-C(12)-C(11)	120.1(2)
C(13)-C(12)-H(12)	120.0
C(11)-C(12)-H(12)	120.0
C(14)-C(13)-C(12)	119.8(2)
C(14)-C(13)-H(13)	120.1
C(12)-C(13)-H(13)	120.1
C(15)-C(14)-C(13)	120.3(2)
C(15)-C(14)-H(14)	119.9
C(13)-C(14)-H(14)	119.9
C(14)-C(15)-C(10)	120.2(2)
C(14)-C(15)-H(15)	119.9
C(10)-C(15)-H(15)	119.9
O(16)-C(16)-O(4)	122.88(18)
O(16)-C(16)-C(17)	125.51(19)
O(4)-C(16)-C(17)	111.61(17)
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

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3)for i14245. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	18(1)	38(1)	30(1)	-7(1)	-1(1)	-8(1)
Cl(2)	29(1)	38(1)	25(1)	-11(1)	5(1)	5(1)
Cl(3)	19(1)	32(1)	78(1)	32(1)	-5(1)	1(1)
O(1)	19(1)	20(1)	14(1)	2(1)	-1(1)	0(1)
O(3)	16(1)	16(1)	13(1)	0(1)	0(1)	4(1)
O(4)	19(1)	14(1)	15(1)	0(1)	4(1)	5(1)
O(6)	16(1)	25(1)	19(1)	-6(1)	2(1)	2(1)

O(7)	24(1)	17(1)	23(1)	1(1)	3(1)	0(1)
O(16)	24(1)	27(1)	17(1)	2(1)	2(1)	7(1)
N(2)	16(1)	14(1)	18(1)	0(1)	-2(1)	1(1)
C(1)	20(1)	23(1)	12(1)	-3(1)	0(1)	1(1)
C(2)	13(1)	16(1)	17(1)	-2(1)	-2(1)	1(1)
C(3)	14(1)	13(1)	13(1)	2(1)	1(1)	3(1)
C(4)	14(1)	14(1)	15(1)	-2(1)	0(1)	2(1)
C(5)	17(1)	17(1)	15(1)	-1(1)	0(1)	-2(1)
C(6)	15(1)	23(1)	17(1)	-2(1)	1(1)	-3(1)
C(7)	17(1)	22(1)	9(1)	0(1)	0(1)	-1(1)
C(8)	18(1)	19(1)	20(1)	4(1)	-2(1)	-4(1)
C(9)	21(1)	20(1)	14(1)	2(1)	1(1)	5(1)
C(10)	16(1)	15(1)	15(1)	-2(1)	-4(1)	0(1)
C(11)	17(1)	17(1)	16(1)	-2(1)	-1(1)	-3(1)
C(12)	25(1)	16(1)	18(1)	3(1)	-6(1)	-2(1)
C(13)	21(1)	19(1)	28(1)	-1(1)	-6(1)	9(1)
C(14)	17(1)	26(1)	24(1)	-1(1)	2(1)	1(1)
C(15)	19(1)	17(1)	20(1)	4(1)	-2(1)	-2(1)
C(16)	18(1)	17(1)	17(1)	-3(1)	2(1)	-2(1)
C(17)	24(1)	24(1)	20(1)	-2(1)	3(1)	6(1)

Single-crystal X-ray structure of 14 (CCDC 1498510)

Table 1. Crystal data and structure refinement for i14285.

Identification code	i14285	
Empirical formula	C19 H19 N3 O5	
Formula weight	369.37	
Temperature	100.0(1) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 6.8459(3) Å	α= 90°.
	b = 8.6718(3) Å	β= 90°.
	c = 29.3079(11) Å	γ = 90°.
Volume	1739.90(12) $Å^3$	
Z	4	
Density (calculated)	1.410 Mg/m^3	
Absorption coefficient	0.104 mm^{-1}	
F(000)	776	
Crystal size	0.30 x 0.28 x 0.14 mm^3	
Theta range for data collection	1.39 to 27.10°.	
Index ranges	-3<=h<=8, -9<=k<=11, -27<=l<=37	
Reflections collected	15946	
Independent reflections	3821 [R(int) = 0.0232]	
Completeness to theta = 25.00°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9856 and 0.9695	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3821 / 0 / 246	
Goodness-of-fit on F^2	1.043	
Final R indices [I>2sigma(I)]	R1 = 0.0339, wR2 = 0.0709	

R indices (all data)	R1 = 0.0421, wR2 = 0.0751
Absolute structure parameter	0.1(8)
Extinction coefficient	0.0045(9)
Largest diff. peak and hole	0.199 and -0.162 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for i14285. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	8239(2)	5223(1)	9001(1)	26(1)
O(3)	7015(2)	9375(1)	9296(1)	26(1)
O(4)	5436(1)	6826(1)	8431(1)	22(1)
O(6)	9824(1)	6805(1)	9489(1)	28(1)
O(7)	4152(2)	10304(2)	9551(1)	37(1)
N(1)	4588(2)	5713(2)	9539(1)	34(1)
N(2)	4376(2)	4736(2)	9236(1)	32(1)
N(3)	3952(2)	3767(2)	8995(1)	45(1)
C(1)	8177(2)	5834(2)	9447(1)	26(1)
C(2)	6293(2)	6753(2)	9510(1)	24(1)
C(3)	6052(2)	7994(2)	9138(1)	21(1)
C(4)	6960(2)	7579(2)	8678(1)	21(1)
C(5)	8756(2)	6560(2)	8737(1)	24(1)
C(6)	10324(2)	7297(2)	9036(1)	27(1)
C(7)	5893(2)	10422(2)	9515(1)	26(1)
C(8)	7112(3)	11695(2)	9697(1)	36(1)
C(9)	5960(2)	6408(2)	7975(1)	28(1)
C(10)	4126(2)	6051(2)	7713(1)	23(1)
C(11)	3797(2)	6665(2)	7293(1)	24(1)
C(12)	1668(2)	7017(2)	6617(1)	28(1)
C(13)	-69(2)	6746(2)	6400(1)	33(1)

C(14)	-1457(2)	5745(2)	6593(1)	33(1)
C(15)	-1107(2)	5041(2)	6999(1)	30(1)
C(16)	1060(2)	4674(2)	7671(1)	26(1)
C(17)	2727(2)	5030(2)	7903(1)	24(1)
C(18)	2068(2)	6332(2)	7041(1)	22(1)
C(19)	666(2)	5326(2)	7240(1)	23(1)

Table 3. Bond lengths [Å] and angles [°] for i14285.

O(1)-C(1)	1.4123(19)
O(1)-C(5)	1.438(2)
O(3)-C(7)	1.351(2)
O(3)-C(3)	1.4437(19)
O(4)-C(4)	1.4286(17)
O(4)-C(9)	1.4293(18)
O(6)-C(1)	1.4125(19)
O(6)-C(6)	1.4351(19)
O(7)-C(7)	1.201(2)
N(1)-N(2)	1.236(2)
N(1)-C(2)	1.477(2)
N(2)-N(3)	1.136(2)
C(1)-C(2)	1.527(2)
C(1)-H(1)	1.0000
C(2)-C(3)	1.541(2)
C(2)-H(2)	1.0000
C(3)-C(4)	1.527(2)
C(3)-H(3)	1.0000
C(4)-C(5)	1.524(2)
C(4)-H(4)	1.0000
C(5)-C(6)	1.527(2)

C(5)-H(5)	1.0000
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.483(2)
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(9)-C(10)	1.504(2)
C(9)-H(9A)	0.9900
C(9)-H(9B)	0.9900
C(10)-C(11)	1.359(2)
C(10)-C(17)	1.418(2)
C(11)-C(18)	1.425(2)
C(11)-H(11)	0.9500
C(12)-C(13)	1.368(2)
C(12)-C(18)	1.406(2)
C(12)-H(12)	0.9500
C(13)-C(14)	1.406(3)
C(13)-H(13)	0.9500
C(14)-C(15)	1.357(2)
C(14)-H(14)	0.9500
C(15)-C(19)	1.427(2)
C(15)-H(15)	0.9500
C(16)-C(17)	1.364(2)
C(16)-C(19)	1.411(2)
C(16)-H(16)	0.9500
C(17)-H(17)	0.9500
C(18)-C(19)	1.422(2)

C(1)-O(1)-C(5)	101.77(12)

C(7)-O(3)-C(3)	116.77(12)
C(4)-O(4)-C(9)	114.00(11)
C(1)-O(6)-C(6)	106.83(11)
N(2)-N(1)-C(2)	117.99(13)
N(3)-N(2)-N(1)	169.83(17)
O(1)-C(1)-O(6)	106.15(13)
O(1)-C(1)-C(2)	109.44(12)
O(6)-C(1)-C(2)	110.65(13)
O(1)-C(1)-H(1)	110.2
O(6)-C(1)-H(1)	110.2
C(2)-C(1)-H(1)	110.2
N(1)-C(2)-C(1)	110.83(13)
N(1)-C(2)-C(3)	112.47(12)
C(1)-C(2)-C(3)	111.71(13)
N(1)-C(2)-H(2)	107.2
C(1)-C(2)-H(2)	107.2
C(3)-C(2)-H(2)	107.2
O(3)-C(3)-C(4)	107.03(12)
O(3)-C(3)-C(2)	107.62(12)
C(4)-C(3)-C(2)	114.62(13)
O(3)-C(3)-H(3)	109.1
C(4)-C(3)-H(3)	109.1
C(2)-C(3)-H(3)	109.1
O(4)-C(4)-C(5)	112.43(13)
O(4)-C(4)-C(3)	104.96(11)
C(5)-C(4)-C(3)	111.44(13)
O(4)-C(4)-H(4)	109.3
C(5)-C(4)-H(4)	109.3
C(3)-C(4)-H(4)	109.3
O(1)-C(5)-C(4)	109.27(11)

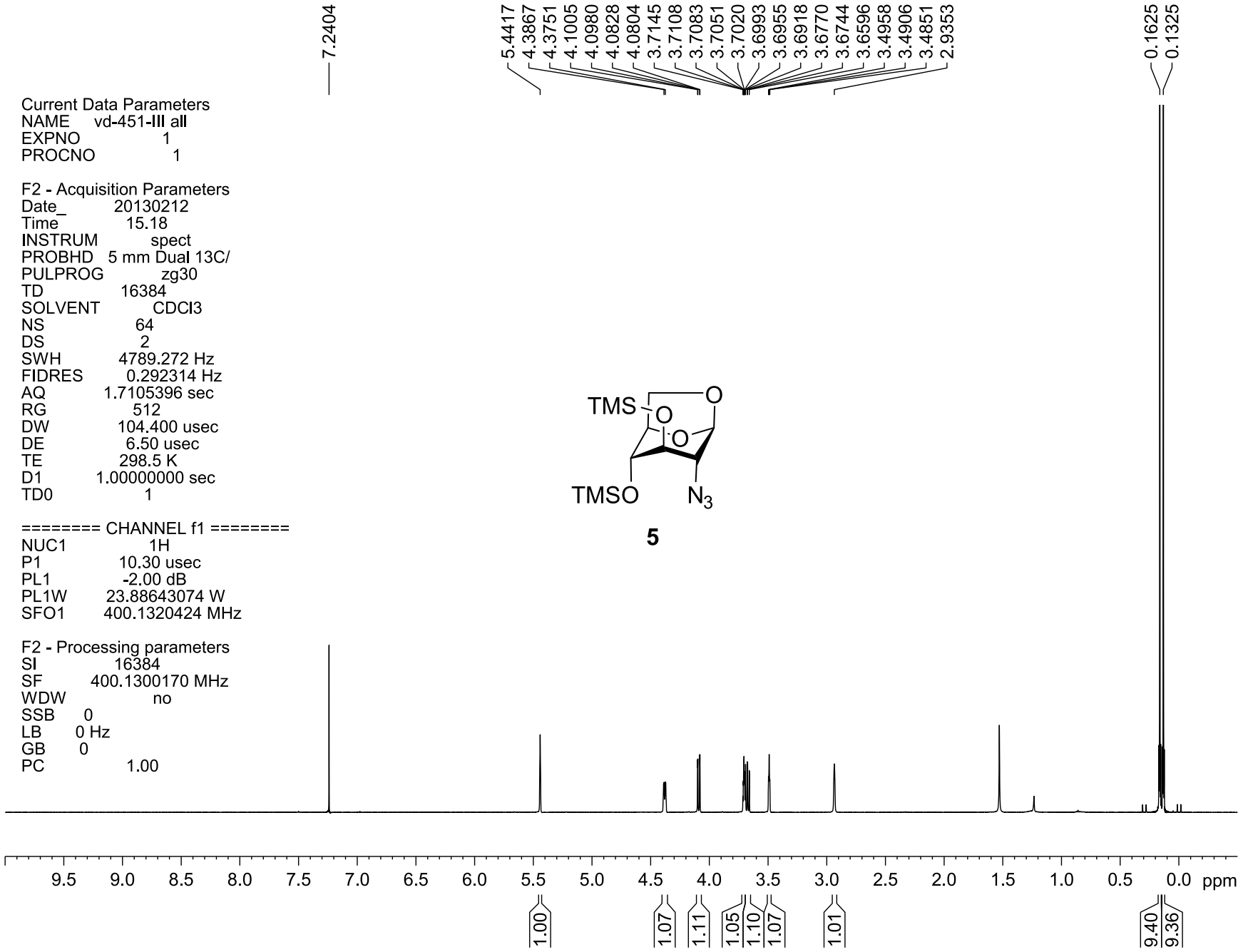
O(1)-C(5)-C(6)	101.64(13)
C(4)-C(5)-C(6)	112.92(13)
O(1)-C(5)-H(5)	110.9
C(4)-C(5)-H(5)	110.9
C(6)-C(5)-H(5)	110.9
O(6)-C(6)-C(5)	103.76(12)
O(6)-C(6)-H(6A)	111.0
C(5)-C(6)-H(6A)	111.0
O(6)-C(6)-H(6B)	111.0
C(5)-C(6)-H(6B)	111.0
H(6A)-C(6)-H(6B)	109.0
O(7)-C(7)-O(3)	123.25(17)
O(7)-C(7)-C(8)	126.19(17)
O(3)-C(7)-C(8)	110.56(14)
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
O(4)-C(9)-C(10)	108.64(11)
O(4)-C(9)-H(9A)	110.0
C(10)-C(9)-H(9A)	110.0
O(4)-C(9)-H(9B)	110.0
C(10)-C(9)-H(9B)	110.0
H(9A)-C(9)-H(9B)	108.3
C(11)-C(10)-C(17)	119.24(14)
C(11)-C(10)-C(9)	121.32(14)
C(17)-C(10)-C(9)	119.43(14)
C(10)-C(11)-C(18)	121.82(14)

C(10)-C(11)-H(11)	119.1
C(18)-C(11)-H(11)	119.1
C(13)-C(12)-C(18)	120.45(16)
C(13)-C(12)-H(12)	119.8
C(18)-C(12)-H(12)	119.8
C(12)-C(13)-C(14)	120.50(16)
C(12)-C(13)-H(13)	119.7
C(14)-C(13)-H(13)	119.7
C(15)-C(14)-C(13)	120.73(15)
C(15)-C(14)-H(14)	119.6
C(13)-C(14)-H(14)	119.6
C(14)-C(15)-C(19)	120.38(16)
C(14)-C(15)-H(15)	119.8
C(19)-C(15)-H(15)	119.8
C(17)-C(16)-C(19)	121.03(15)
C(17)-C(16)-H(16)	119.5
C(19)-C(16)-H(16)	119.5
C(16)-C(17)-C(10)	120.71(15)
C(16)-C(17)-H(17)	119.6
C(10)-C(17)-H(17)	119.6
C(12)-C(18)-C(19)	119.33(14)
C(12)-C(18)-C(11)	122.37(15)
C(19)-C(18)-C(11)	118.23(14)
C(16)-C(19)-C(18)	118.93(13)
C(16)-C(19)-C(15)	122.49(15)
C(18)-C(19)-C(15)	118.57(15)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for i14285. 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}
O(1)	25(1)	23(1)	31(1)	-5(1)	-7(1)	2(1)
O(3)	24(1)	22(1)	33(1)	-8(1)	0(1)	-1(1)
O(4)	17(1)	32(1)	19(1)	-1(1)	-1(1)	-3(1)
O(6)	23(1)	33(1)	27(1)	-2(1)	-7(1)	-6(1)
O(7)	37(1)	32(1)	43(1)	0(1)	13(1)	7(1)
N(1)	28(1)	42(1)	33(1)	6(1)	2(1)	-13(1)
N(2)	27(1)	37(1)	33(1)	16(1)	-10(1)	-15(1)
N(3)	49(1)	42(1)	44(1)	13(1)	-21(1)	-25(1)
C(1)	26(1)	23(1)	28(1)	1(1)	-9(1)	-4(1)
C(2)	22(1)	28(1)	21(1)	-1(1)	-1(1)	-7(1)
C(3)	18(1)	22(1)	24(1)	-4(1)	1(1)	-1(1)
C(4)	17(1)	25(1)	21(1)	0(1)	-1(1)	-3(1)
C(5)	17(1)	32(1)	25(1)	-5(1)	1(1)	-1(1)
C(6)	18(1)	35(1)	29(1)	-3(1)	-2(1)	-2(1)
C(7)	39(1)	21(1)	19(1)	4(1)	2(1)	5(1)
C(8)	51(1)	24(1)	33(1)	-3(1)	-7(1)	7(1)
C(9)	20(1)	45(1)	20(1)	-3(1)	0(1)	-2(1)
C(10)	21(1)	28(1)	22(1)	-3(1)	-1(1)	2(1)
C(11)	24(1)	25(1)	23(1)	-1(1)	2(1)	-1(1)
C(12)	31(1)	29(1)	23(1)	0(1)	2(1)	5(1)
C(13)	35(1)	41(1)	23(1)	-1(1)	-2(1)	16(1)
C(14)	25(1)	49(1)	25(1)	-12(1)	-7(1)	9(1)
C(15)	22(1)	41(1)	28(1)	-10(1)	0(1)	1(1)
C(16)	26(1)	28(1)	23(1)	-1(1)	4(1)	-3(1)
C(17)	25(1)	28(1)	20(1)	1(1)	0(1)	2(1)
C(18)	24(1)	21(1)	19(1)	-2(1)	2(1)	4(1)
C(19)	22(1)	24(1)	21(1)	-4(1)	1(1)	3(1)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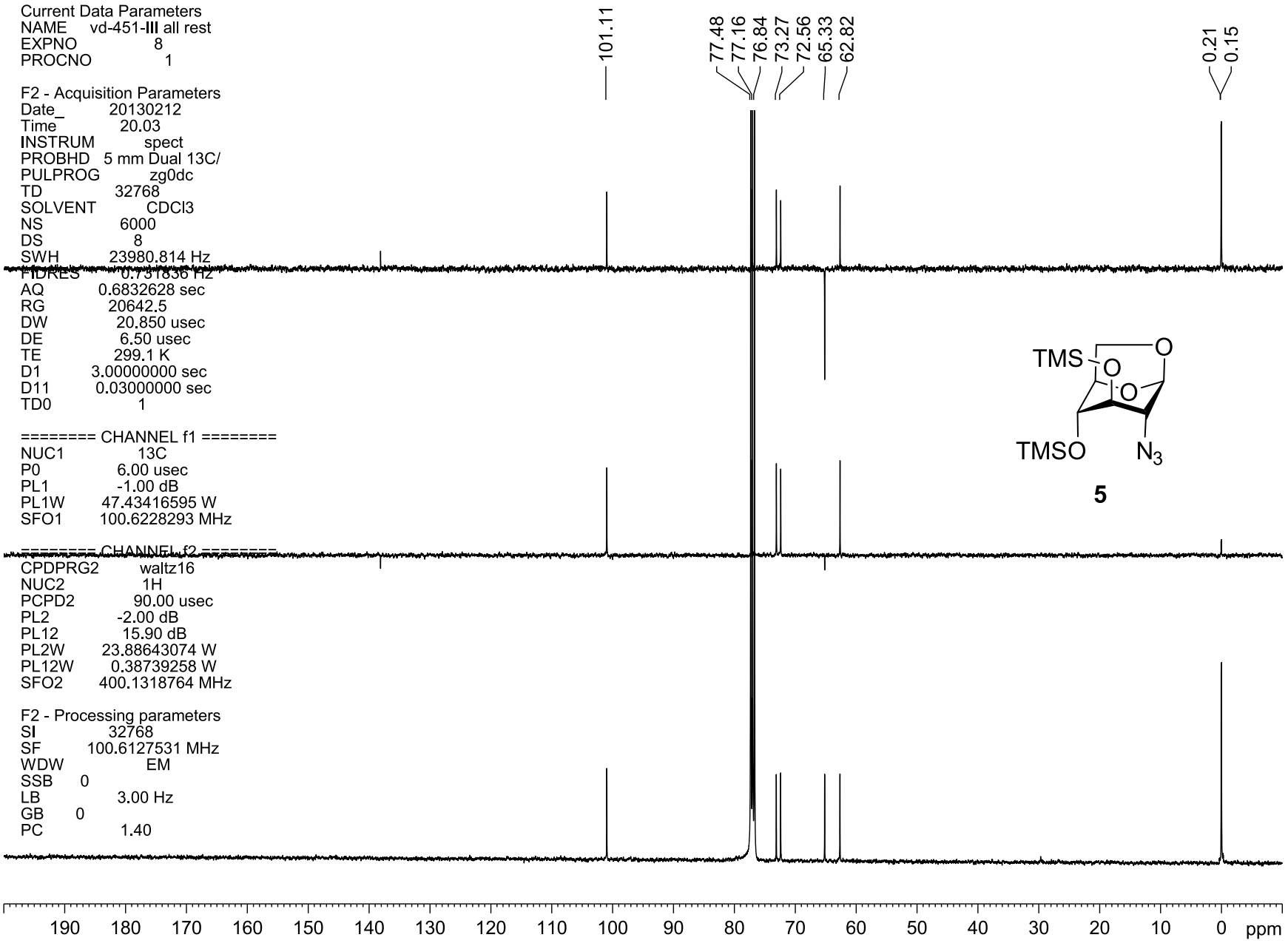
9.40
9.36


Current Data Parameters
NAME vd-451-III all rest
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130212
Time 20.03
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 6000
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 20642.5
DW 20.850 usec
DE 6.50 usec
TE 299.1 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127531 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
101.11
77.48
77.16
76.84
73.27
72.56
65.33
62.82
0.21
0.15
TMS
O
O
O
TMSO
N3
5
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

vd-457-III (HR-ESI)

Elemental Composition Report

Single Mass Analysis

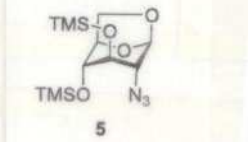
Tolerance = 100.0 PPM / DBE: min = -1.5, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
4 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 3-3 O: 4-4 Si: 2-2 Na: 1-1

vd-451-III

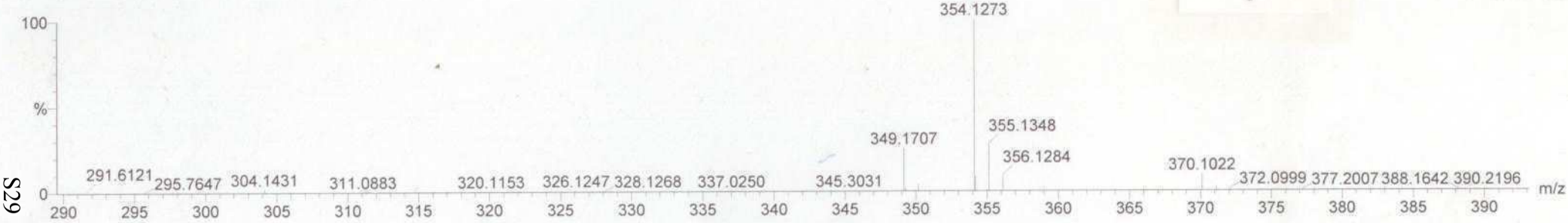
KE267

$[M+Na]^+$

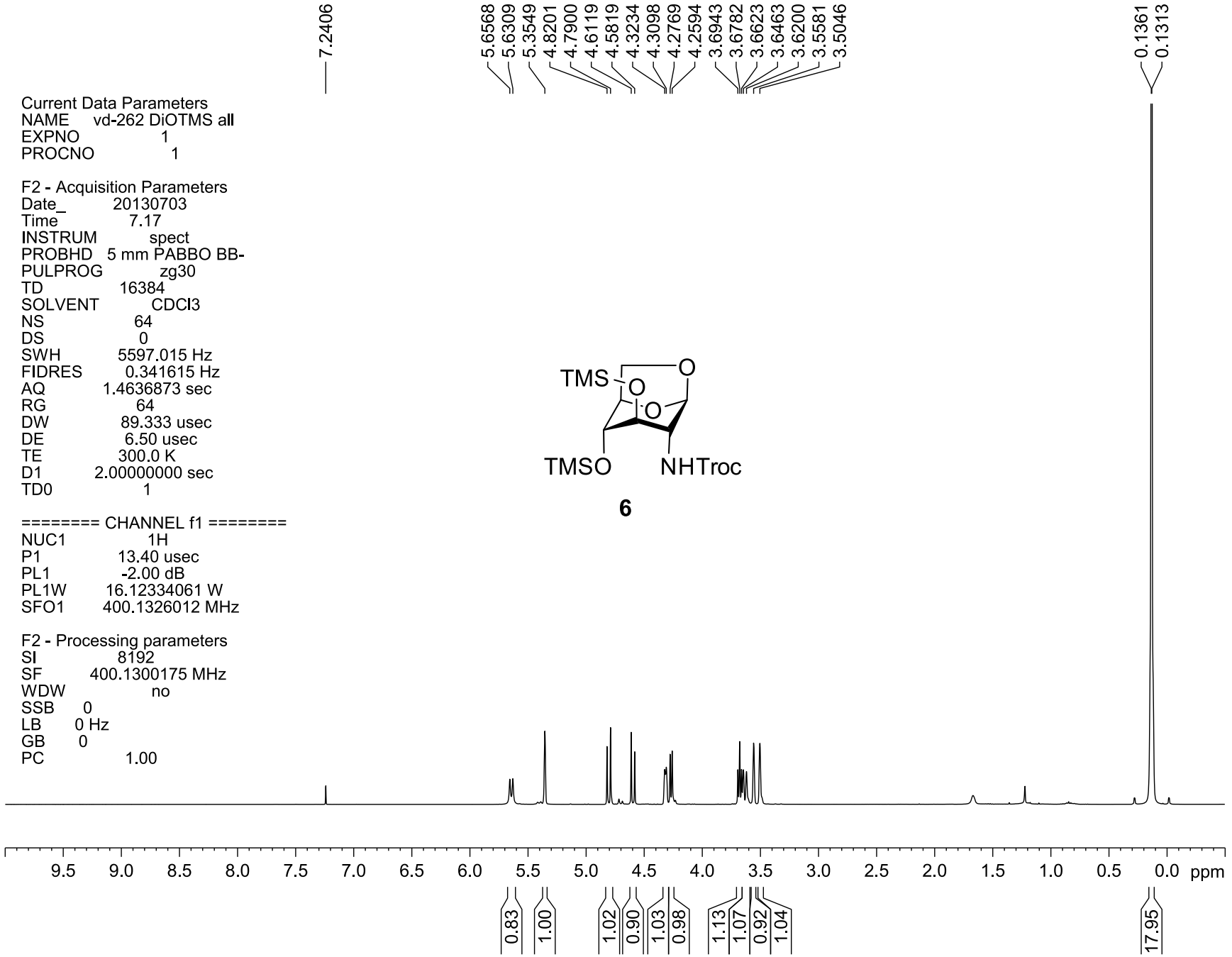


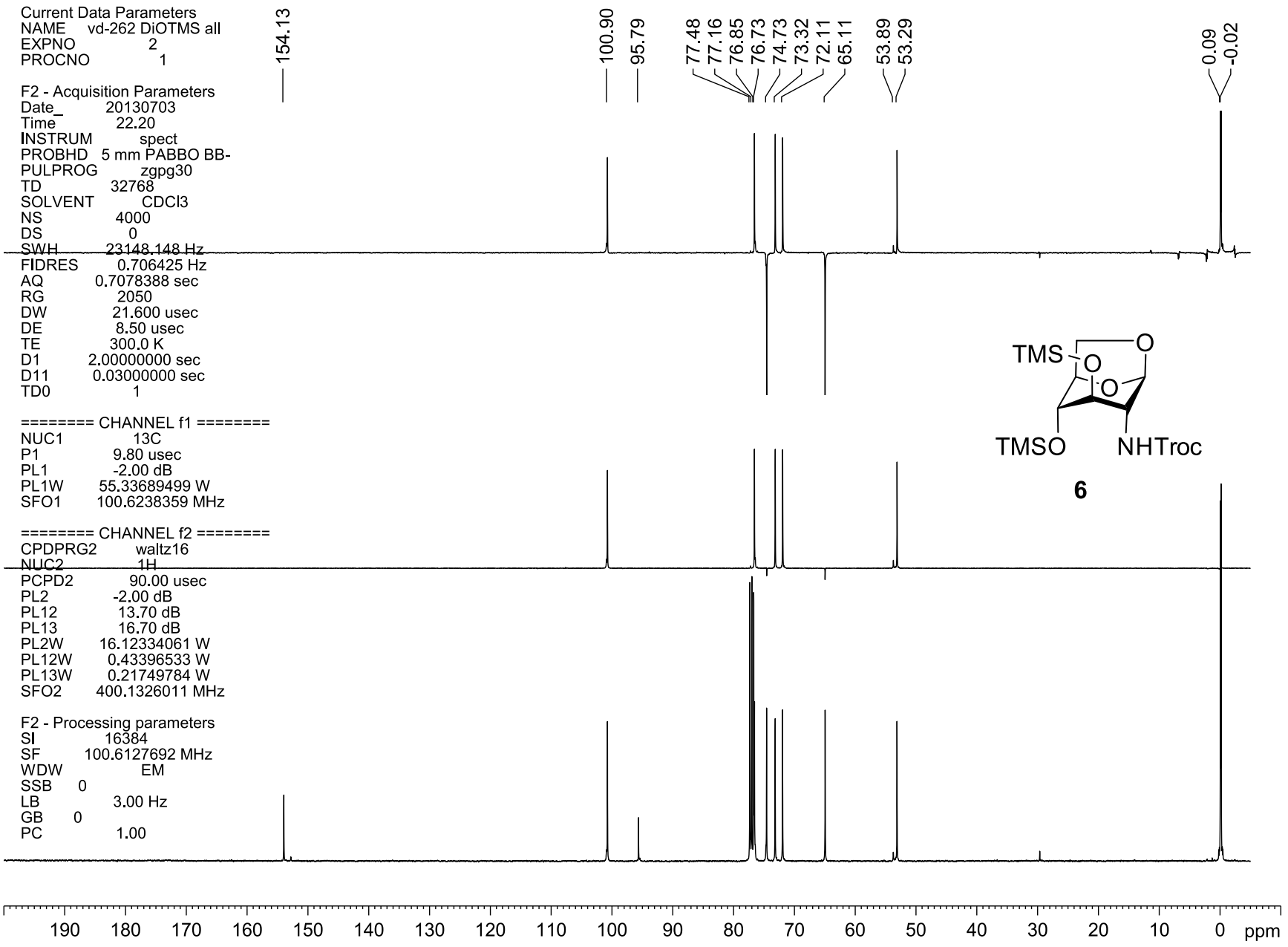
28-Jun-2013
10:57:46
1: TOF MS ES+
2.41e+003

0628_vd-451-III 81 (3.626) Cm (81-1x10.000)



Minimum:				-1.5			
Maximum:		5.0	100.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
354.1273	354.1281	-0.8	-2.3	3.5	22.6	0.0	C12 H25 N3 O4 Si2 Na


7.2406
5.6568
5.6309
5.3549
4.8201
4.7900
4.6119
4.5819
4.3234
4.3098
4.2769
4.2594
3.6943
3.6782
3.6623
3.6463
3.6200
3.5581
3.5046
0.1361
0.1313
Current Data Parameters
NAME vd-262 DiOTMS all
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130703
Time 7.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 64
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
TMS
O
O
O
TMSO
NHTroc
6
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.83
1.00
1.02
0.90
1.03
0.98
1.13
1.07
0.92
1.04
17.95


Current Data Parameters
NAME vd-262 DiOTMS all
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130703
Time 22.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4000
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238359 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127692 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
154.13
100.90
95.79
77.48
77.16
76.85
76.73
74.73
73.32
72.11
65.11
53.89
53.29
0.09
-0.02
TMS
O
O
O
TMSO
NHTroc
6
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

VD-494 (HR-ESI)

Elemental Composition Report

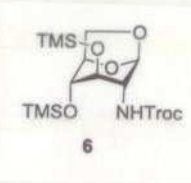
Multiple Mass Analysis: 3 mass(es) processed
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
47 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 15-15 H: 0-1000 N: 1-1 O: 6-6 Na: 1-1 Si: 2-2 35Cl: 0-3 37Cl: 0-3

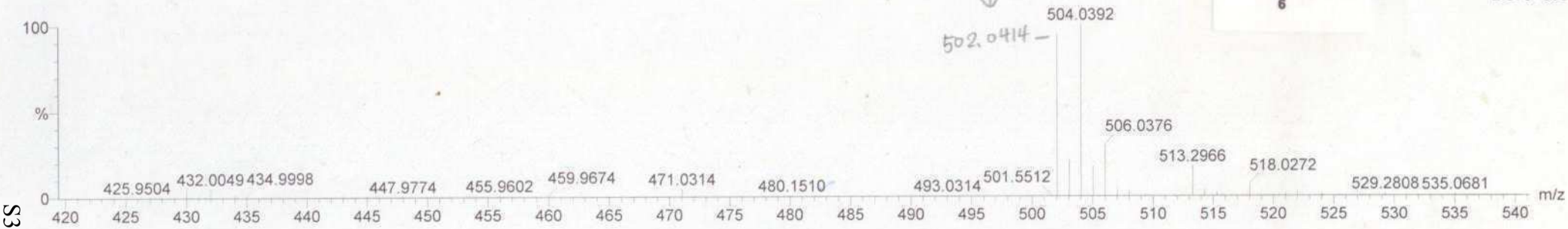
VD-494 KE267

0626_VD-494 21 (2.077) Cm (21-1x10.000)

$[M+Na]^+$

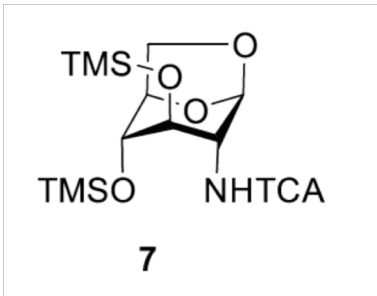


26-Jun-2013
12:08:56
1: TOF MS ES+
2.87e+003

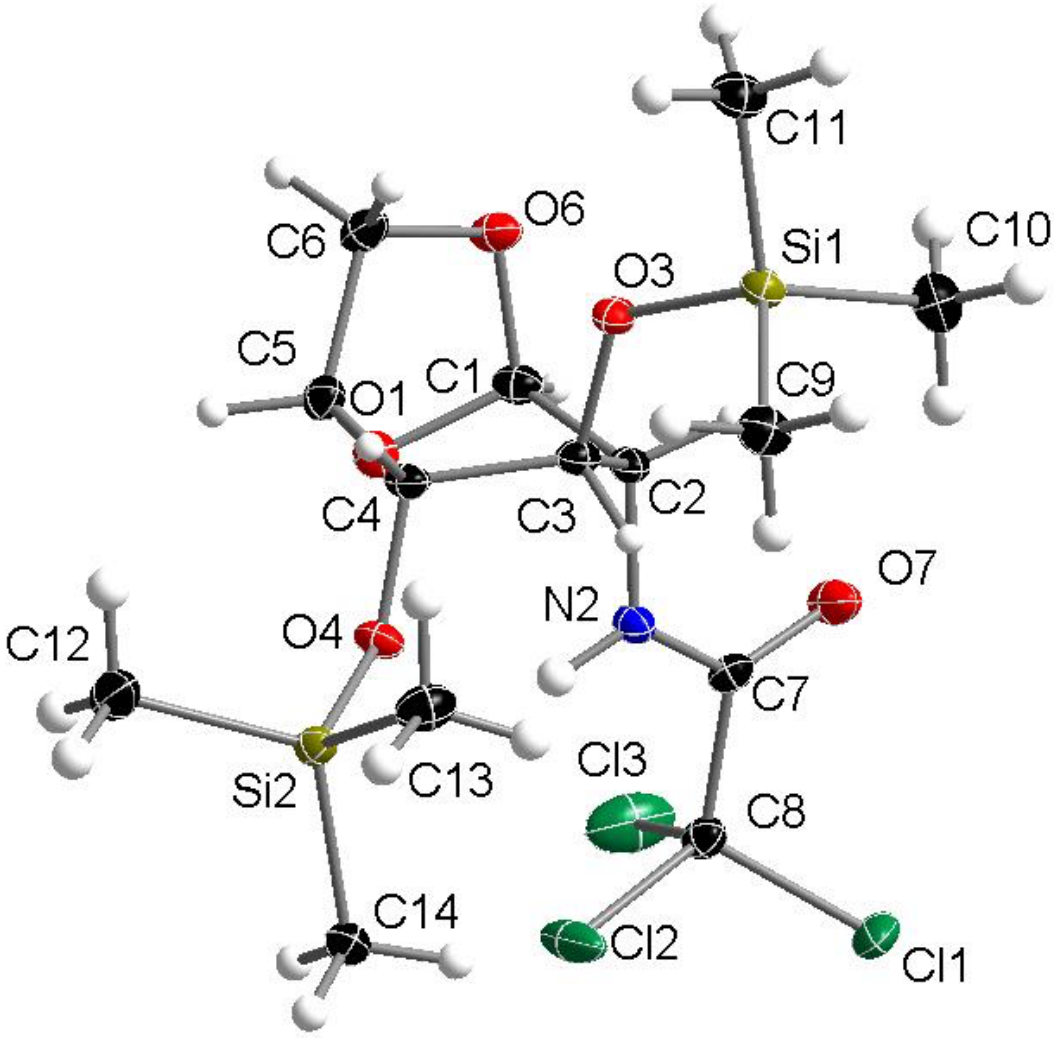


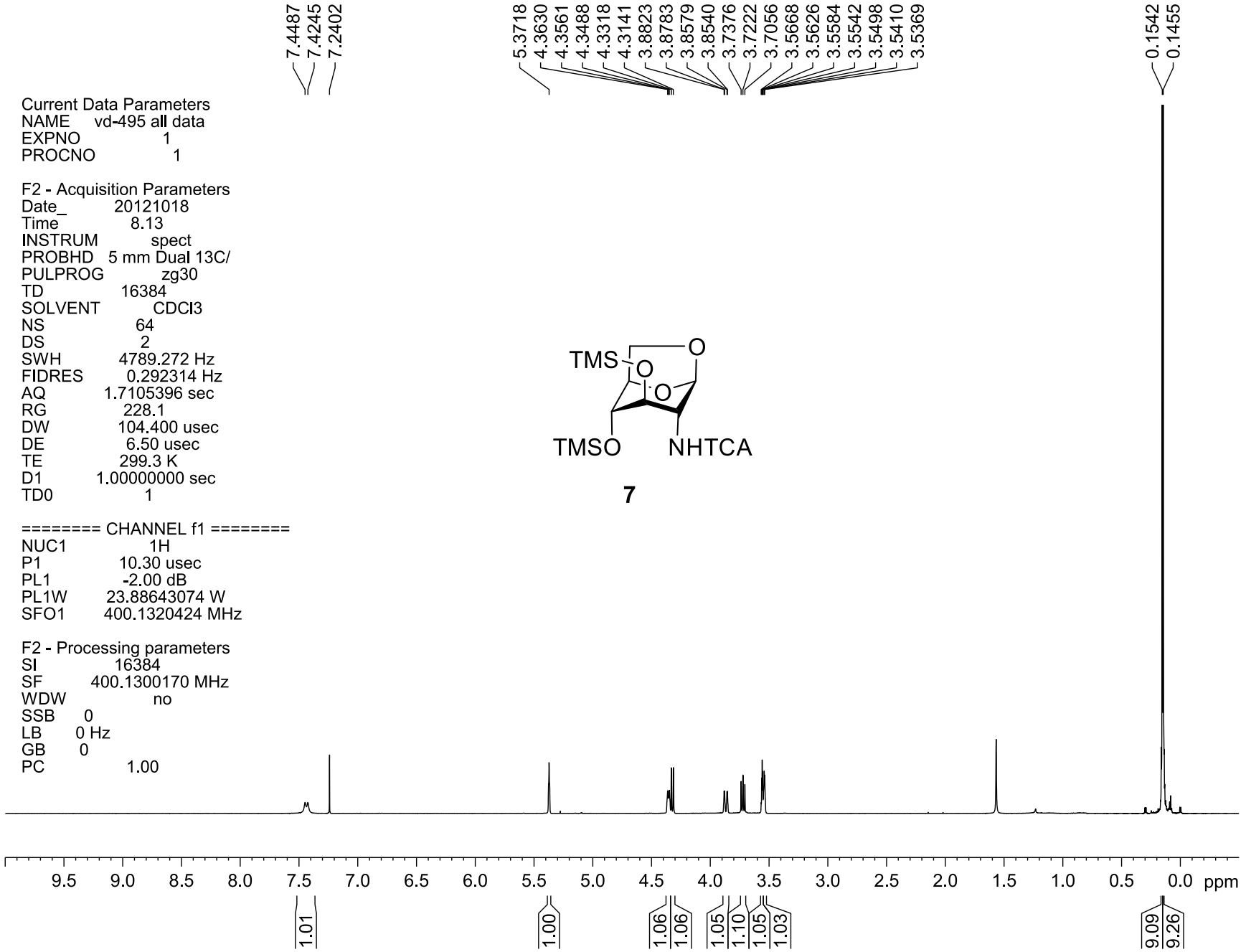
Minimum:	25.00				-1.5			
Maximum:	100.00		5.0	10.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
502.0414	94.59	502.0418	-0.4	-0.8	2.5	29.8	0.0	C15 H28 N O6 Na Si2 35Cl3
504.0392	100.00	504.0389	0.3	0.6	2.5	26.2	0.0	C15 H28 N O6 Na Si2 35Cl2 37Cl
506.0376	30.01	506.0359	1.7	3.4	2.5	16.3	0.0	C15 H28 N O6 Na Si2 35Cl 37Cl2

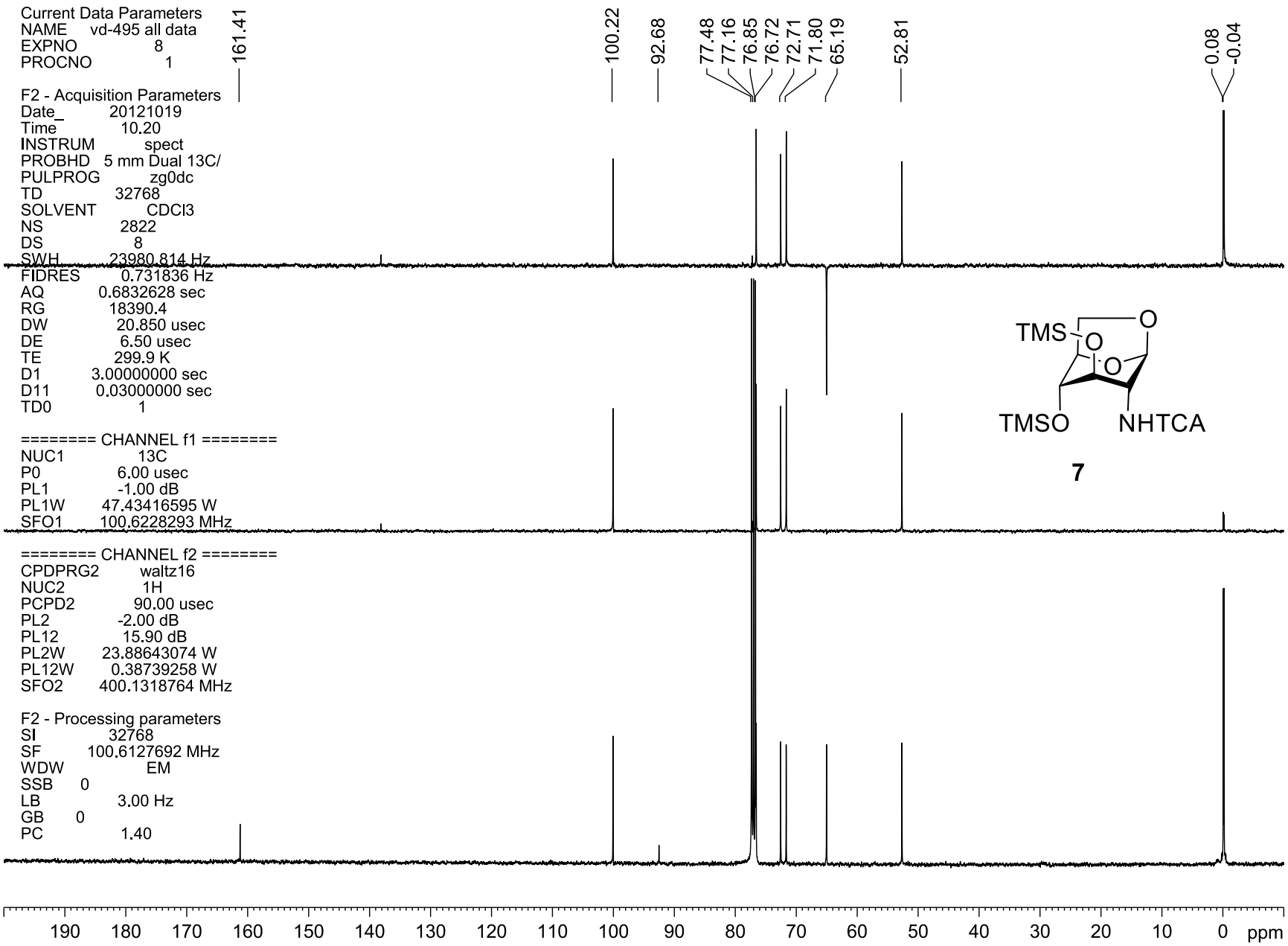
ORTEP diagram of **7** with the thermal ellipsoids drawn at 50% probability level.



7




7.4487
7.4245
7.2402
5.3718
4.3630
4.3561
4.3488
4.3318
4.3141
3.8823
3.8783
3.8579
3.8540
3.7376
3.7222
3.7056
3.5668
3.5626
3.5584
3.5542
3.5498
3.5410
3.5369
0.1542
0.1455
Current Data Parameters
NAME vd-495 all data
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121018
Time 8.13
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 228.1
DW 104.400 usec
DE 6.50 usec
TE 299.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300170 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
TMS
O
O
O
TMSO
NHTCA
7
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.01
1.00
1.06
1.06
1.05
1.10
1.05
1.03
9.09
9.26


Current Data Parameters
NAME vd-495 all data
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121019
Time 10.20
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 2822
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 18390.4
DW 20.850 usec
DE 6.50 usec
TE 299.9 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127692 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
161.41
100.22
92.68
77.48
77.16
76.85
76.72
72.71
71.80
65.19
52.81
0.08
-0.04
TMS
O
O
O
TMSO
NHTCA
7
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

VD-495 (HR-ESI)

Elemental Composition Report

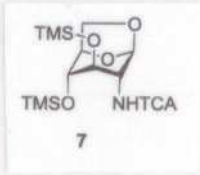
Multiple Mass Analysis: 3 mass(es) processed
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
87 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-14 H: 0-1000 N: 1-1 O: 5-5 Na: 1-1 Si: 2-2 35Cl: 0-3 37Cl: 0-3

VD-495

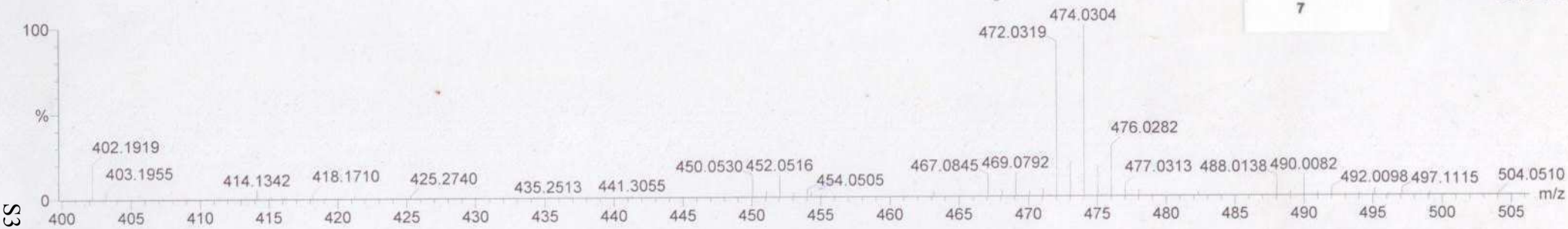
KE267

$[M+Na]^+$

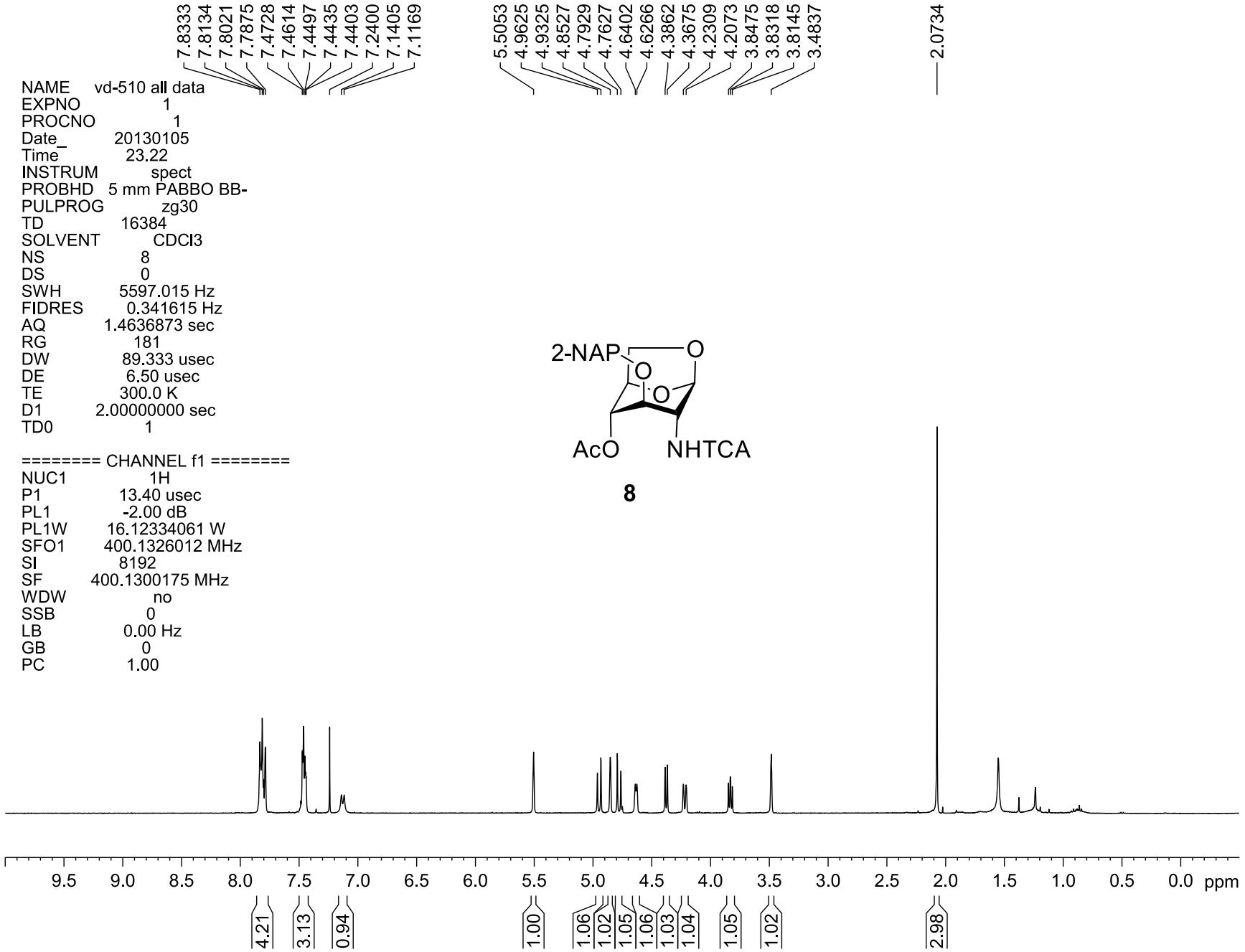


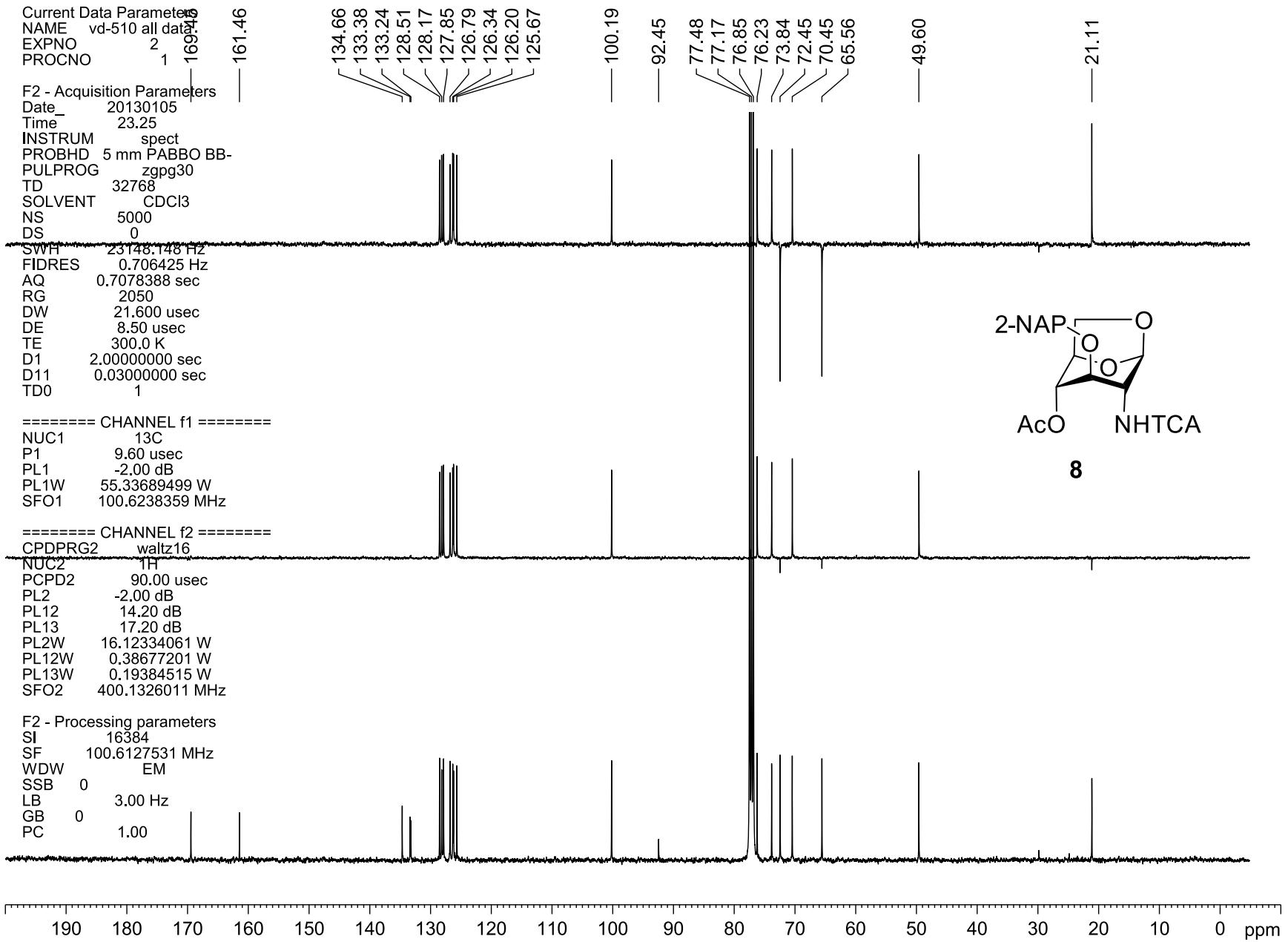
26-Jun-2013
11:46:24
1: TOF MS ES+
4.04e+003

0626_VD-495 79 (7.722)



Minimum:	25.00				-1.5			
Maximum:	100.00		5.0	10.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
472.0319	90.18	472.0313	0.6	1.3	2.5	40.2	0.0	C14 H26 N O5 Na Si2 35Cl3
474.0304	100.00	474.0283	2.1	4.4	2.5	40.7	0.0	C14 H26 N O5 Na Si2 35Cl2 37Cl
476.0282	29.07	476.0254	2.8	5.9	2.5	38.6	0.0	C14 H26 N O5 Na Si2 35Cl 37Cl2


7.8333
7.8134
7.8021
7.7875
7.4728
7.4614
7.4497
7.4435
7.4403
7.2400
7.1405
7.1169
5.5053
4.9625
4.9325
4.8527
4.7929
4.7627
4.6402
4.6266
4.3862
4.3675
4.2309
4.2073
3.8475
3.8318
3.8145
3.4837
2.0734
NAME vd-510 all data
EXPNO 1
PROCNO 1
Date_ 20130105
Time 23.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 8
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 181
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2-NAP
O
O
O
AcO
NHTCA
8
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.21
3.13
0.94
1.00
1.06
1.02
1.05
1.06
1.03
1.04
1.05
1.02
2.98


Current Data Parameters
NAME vd-510 all data
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130105
Time 23.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 5000
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238359 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.20 dB
PL13 17.20 dB
PL2W 16.12334061 W
PL12W 0.38677201 W
PL13W 0.19384515 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127531 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
169.45
161.46
134.66
133.38
133.24
128.51
128.17
127.85
126.79
126.34
126.20
125.67
100.19
92.45
77.48
77.17
76.85
76.23
73.84
72.45
70.45
65.56
49.60
21.11
2-NAP
O
O
O
AcO
NHTCA
8
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

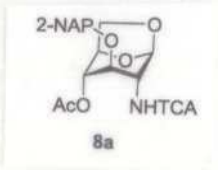
VD-510 (HR-ESI neg)

Elemental Composition Report

Multiple Mass Analysis: 3 mass(es) processed
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

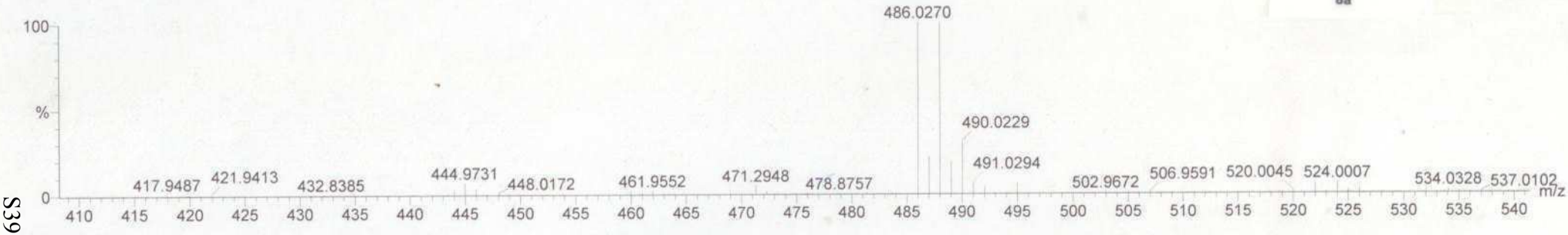
Monoisotopic Mass, Even Electron Ions
48 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 21-21 H: 0-1000 N: 1-1 O: 6-6 35Cl: 0-3 37Cl: 0-3
VD-510

KE267 $[M-H]^-$

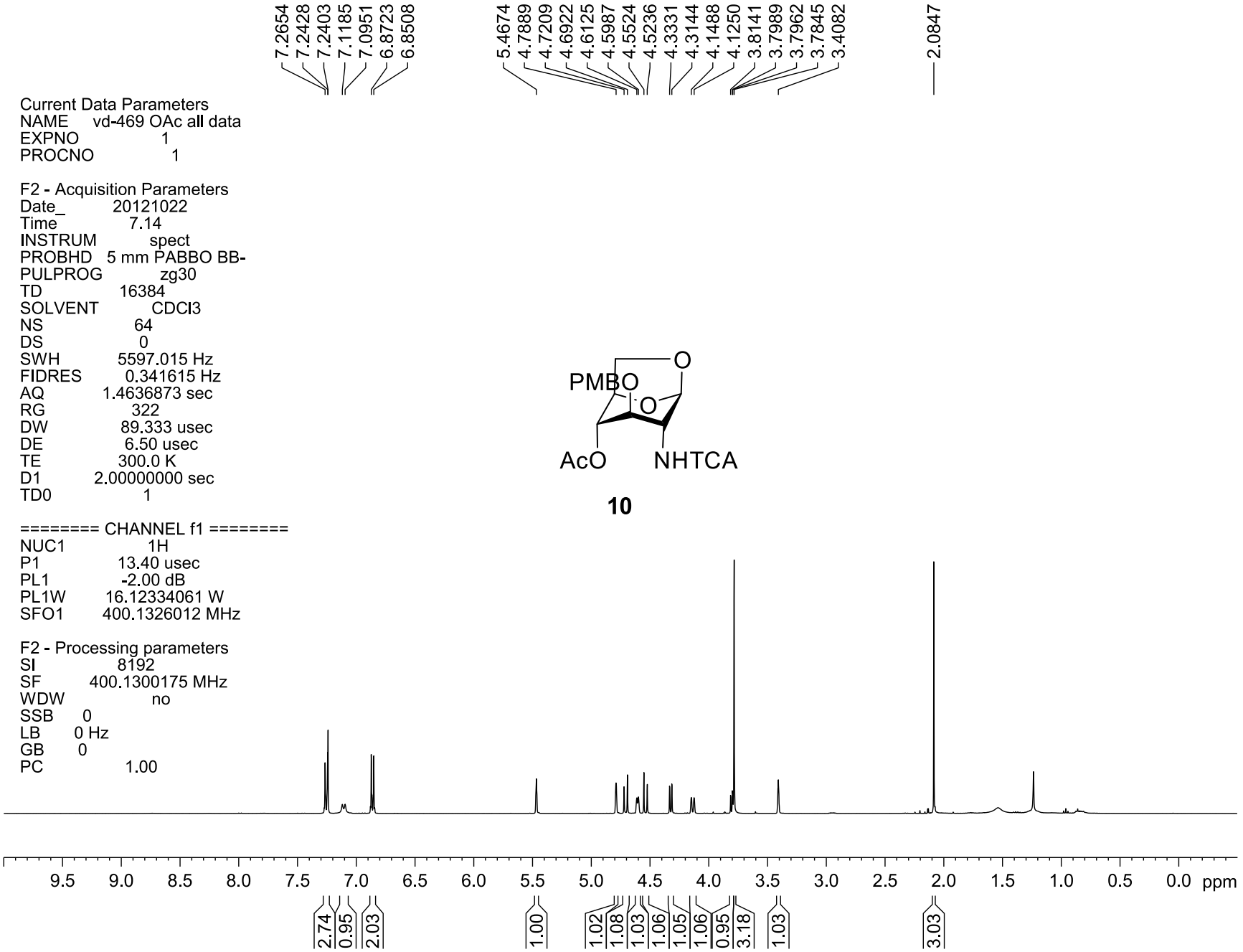


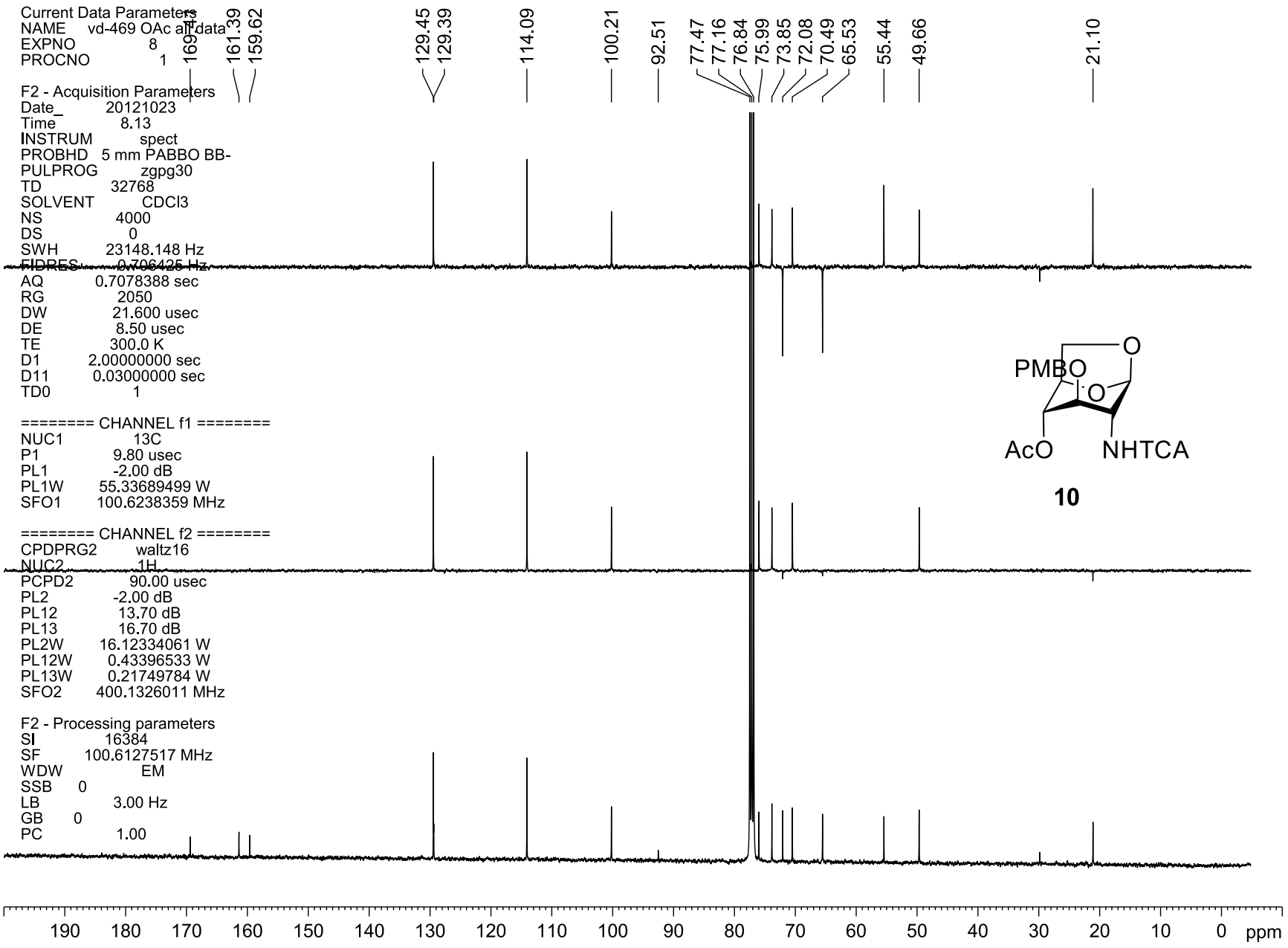
02-Aug-2013
16:59:31
2: TOF MS ES-
3.48e+003

0802_VD-510 22 (2.193)



Minimum:	30.00				-1.5			
Maximum:	100.00		5.0	10.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
486.0270	100.00	486.0278	-0.8	-1.6	11.5	46.2	0.0	C21 H19 N O6 35Cl3
488.0238	99.68	488.0248	-1.0	-2.0	11.5	49.9	0.0	C21 H19 N O6 35Cl2 37Cl
490.0229	30.67	490.0219	1.0	2.0	11.5	45.9	0.0	C21 H19 N O6 35Cl 37Cl2


7.2654
7.2428
7.2403
7.1185
7.0951
6.8723
6.8508
5.4674
4.7889
4.7209
4.6922
4.6125
4.5987
4.5524
4.5236
4.3331
4.3144
4.1488
4.1250
3.8141
3.7989
3.7962
3.7845
3.4082
2.0847
Current Data Parameters
NAME vd-469 OAc all data
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121022
Time 7.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 322
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
PMBO
O
O
AcO
NHTCA
10
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.74
0.95
2.03
1.00
1.02
1.08
1.03
1.06
1.05
1.06
0.95
3.18
1.03
3.03


Current Data Parameters
NAME vd-469 OAc alf data
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121023
Time 8.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4000
DS 0
SWH 23148.148 Hz
FIDRES 0.706426 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238359 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127517 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
169.41
161.39
159.62
129.45
129.39
114.09
100.21
92.51
77.47
77.16
76.84
75.99
73.85
72.08
70.49
65.53
55.44
49.66
21.10
PMBO
O
O
AcO
NHTCA
10
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

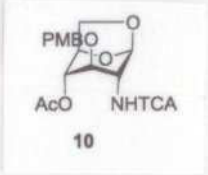
VD-469 OAc (HR-ESI neg)

Elemental Composition Report

Multiple Mass Analysis: 3 mass(es) processed
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

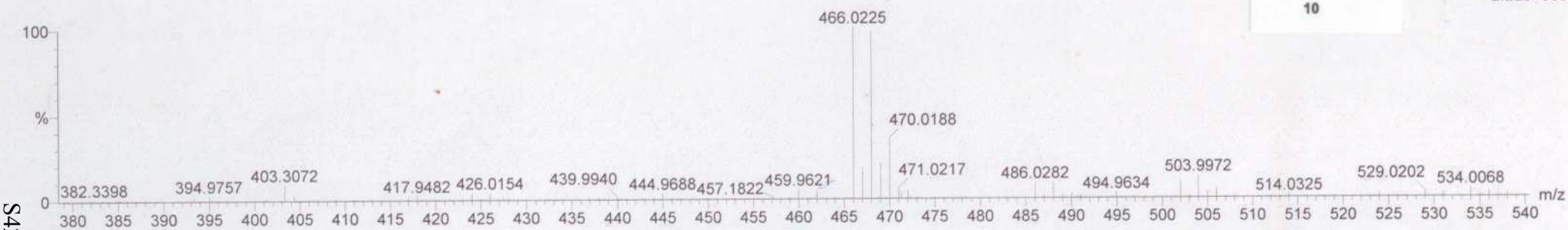
Monoisotopic Mass, Even Electron Ions
48 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 18-18 H: 0-1000 N: 1-1 O: 7-7 35Cl: 0-3 37Cl: 0-3
VD-469 OAc

KE267 $[M-H]^-$



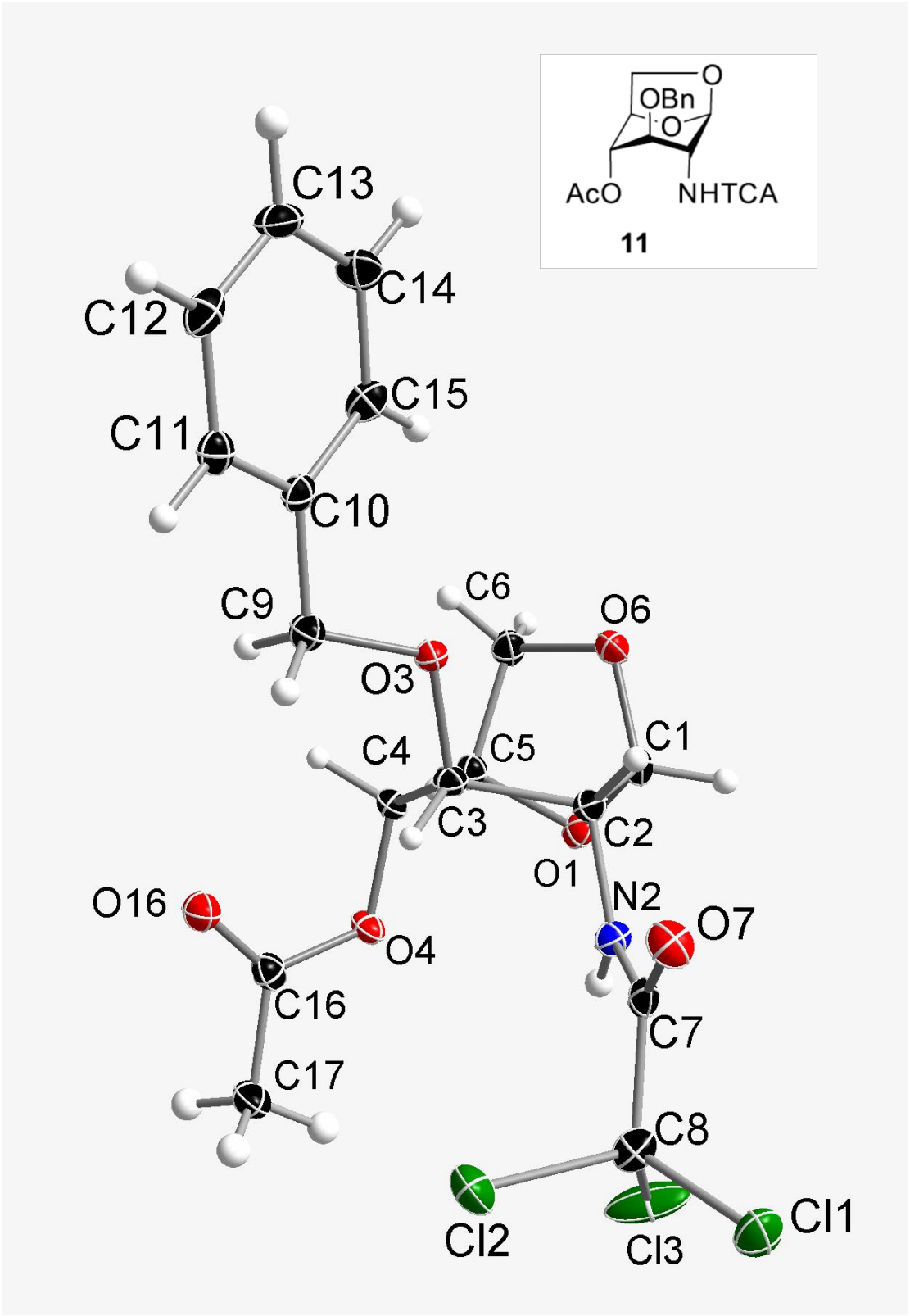
02-Aug-2013
17:10:53
2: TOF MS ES-
2.22e+003

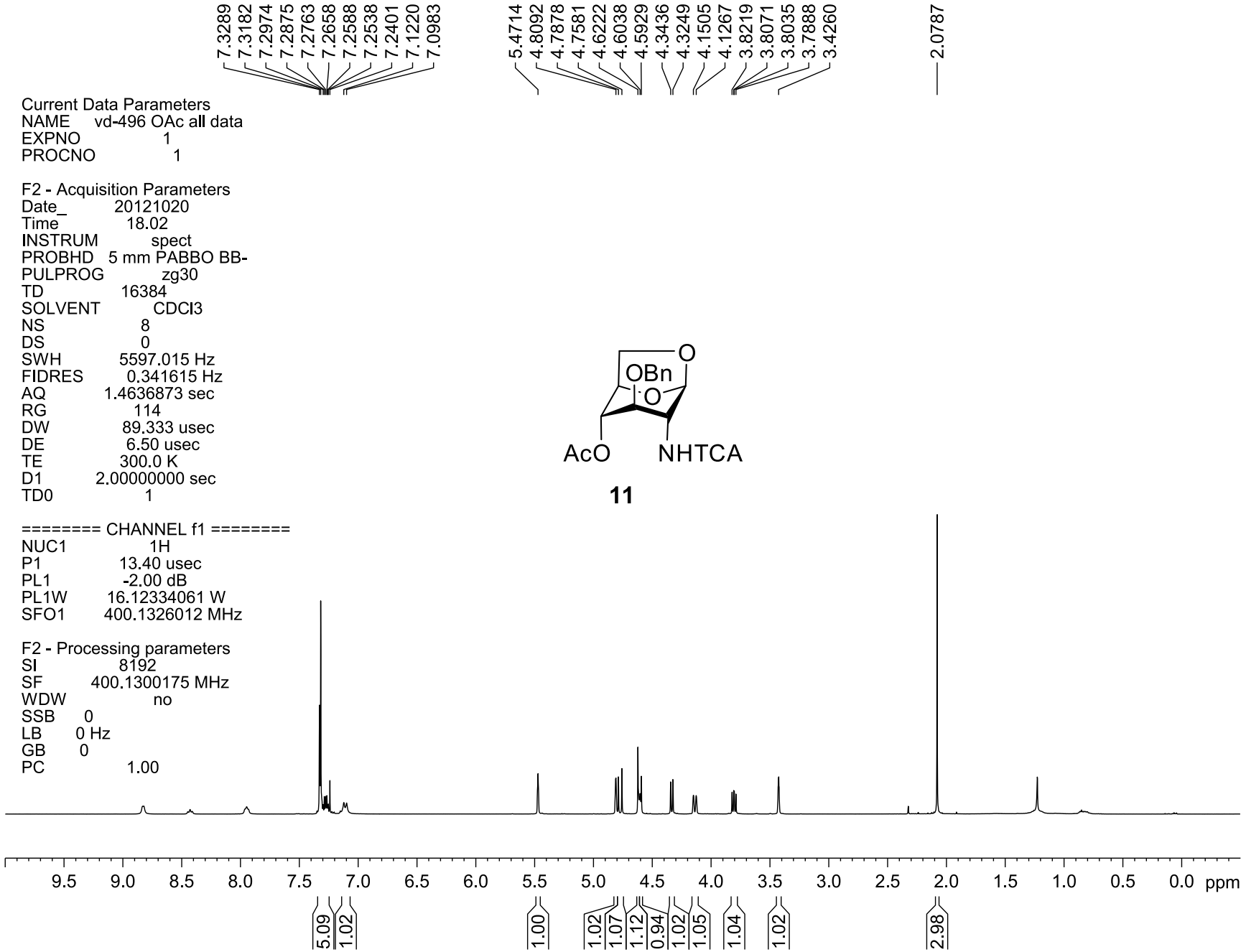
0802_VD-469 OAc 41 (4.074)

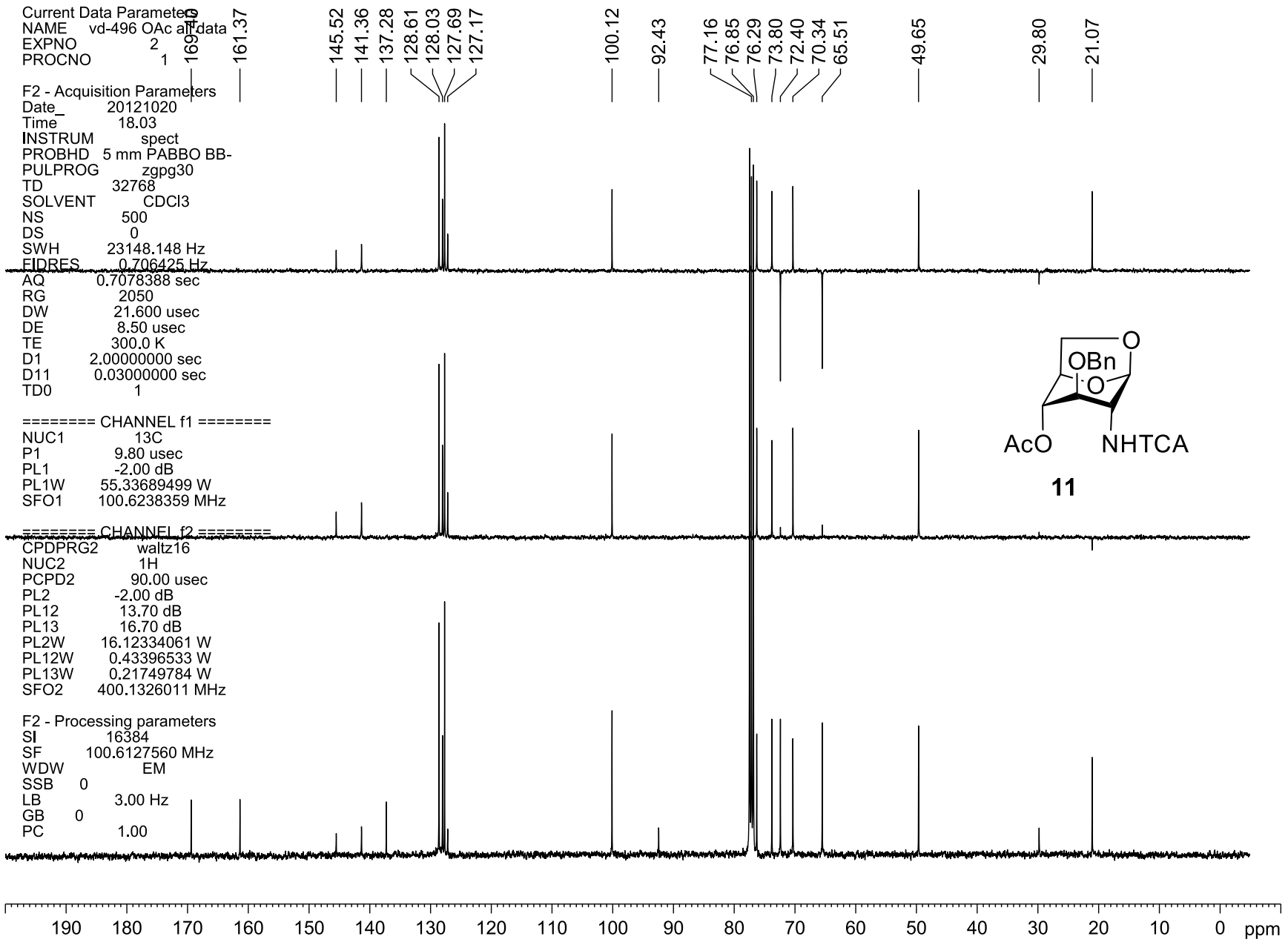


Minimum:	30.00				-1.5			
Maximum:	100.00		5.0	10.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
466.0225	100.00	466.0227	-0.2	-0.4	8.5	31.7	0.0	C18 H19 N O7 35Cl3
468.0188	97.38	468.0198	-1.0	-2.1	8.5	48.1	0.0	C18 H19 N O7 35Cl2 37Cl
470.0188	35.32	470.0168	2.0	4.3	8.5	44.9	0.0	C18 H19 N O7 35Cl 37Cl2

ORTEP diagram of **11** with the thermal ellipsoids drawn at 50% probability level.




7.3289
7.3182
7.2974
7.2875
7.2763
7.2658
7.2588
7.2538
7.2401
7.1220
7.0983
5.4714
4.8092
4.7878
4.7581
4.6222
4.6038
4.5929
4.3436
4.3249
4.1505
4.1267
3.8219
3.8071
3.8035
3.7888
3.4260
2.0787
Current Data Parameters
NAME vd-496 OAc all data
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121020
Time 18.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 8
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 114
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
O
OBn
O
AcO
NHTCA
11
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
5.09
1.02
1.00
1.02
1.07
1.12
0.94
1.02
1.05
1.04
1.02
2.98


Current Data Parameters
NAME vd-496 OAc alf data
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121020
Time 18.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 500
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238359 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127560 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
169.41
161.37
145.52
141.36
137.28
128.61
128.03
127.69
127.17
100.12
92.43
77.16
76.85
76.29
73.80
72.40
70.34
65.51
49.65
29.80
21.07
O
OBn
O
AcO
NHTCA
11
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

VD-496 OAc (HR-ESI neg)

Elemental Composition Report

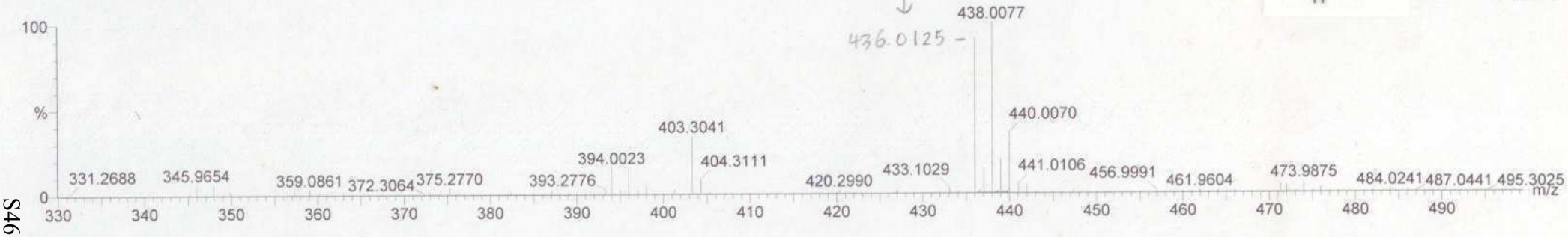
Multiple Mass Analysis: 3 mass(es) processed
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

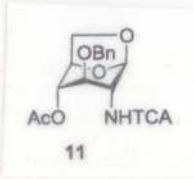
Monoisotopic Mass, Even Electron Ions
48 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 17-17 H: 0-1000 N: 1-1 O: 6-6 35Cl: 0-3 37Cl: 0-3

VD-496 OAc

KE267

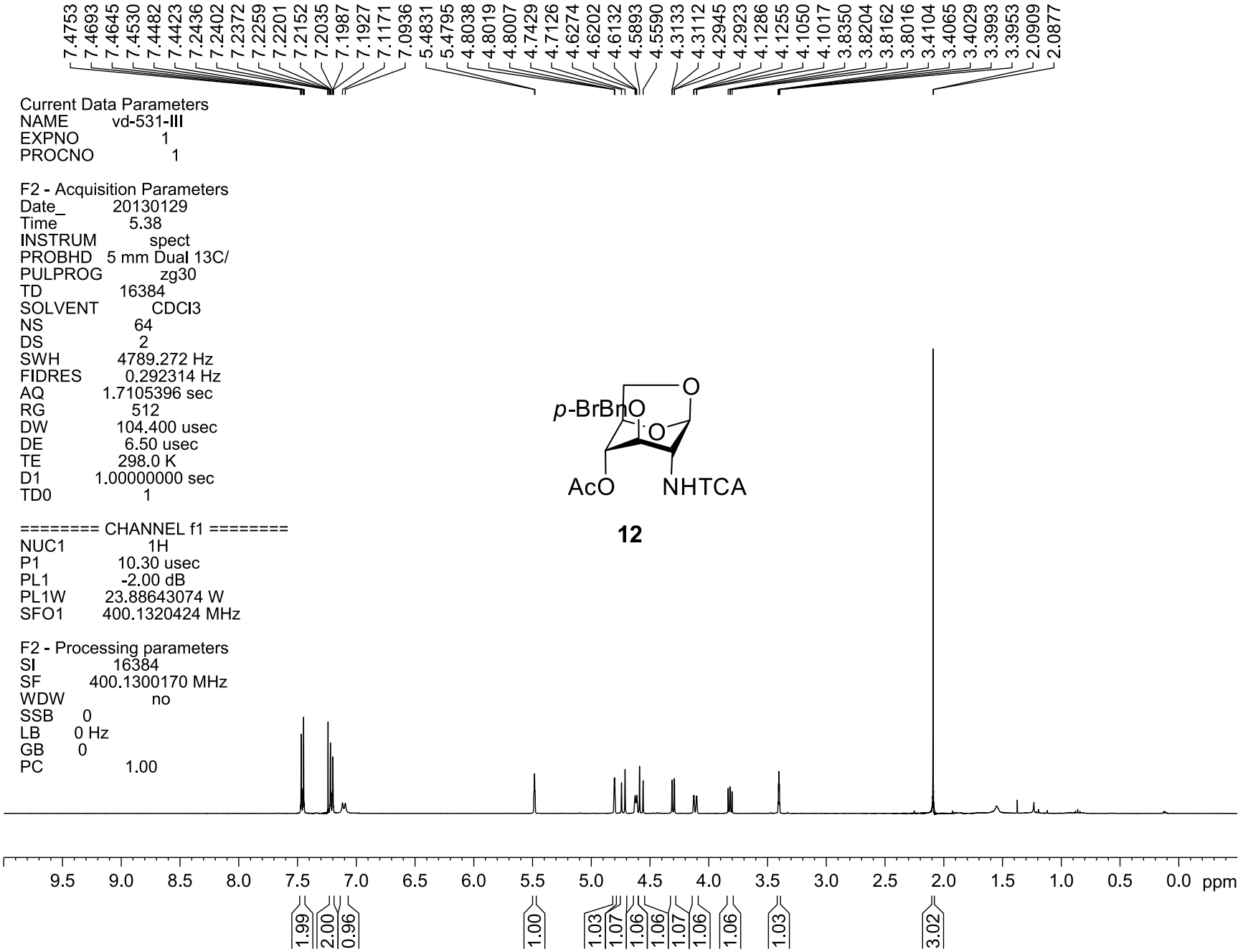
0802_VD-496 OAc 7 (0.703)

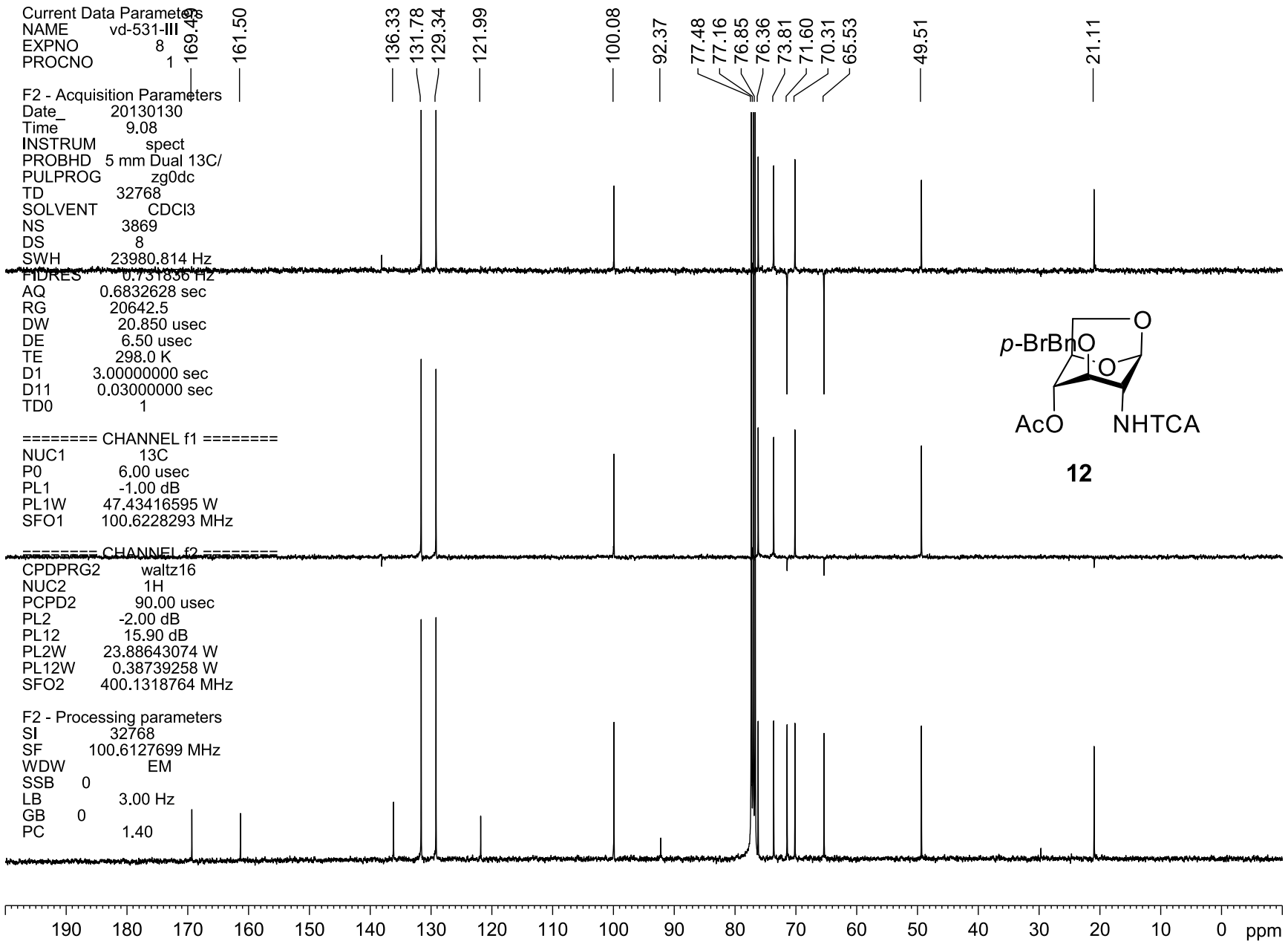




02-Aug-2013
17:22:15
2: TOF MS ES-
1.11e+004

Minimum:	35.00				-1.5			
Maximum:	100.00		5.0	10.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
436.0125	91.43	436.0121	0.4	0.9	8.5	98.7	0.0	C17 H17 N O6 35Cl3
438.0077	100.00	438.0092	-1.5	-3.4	8.5	120.8	0.0	C17 H17 N O6 35Cl2 37Cl
440.0070	35.79	440.0062	0.8	1.8	8.5	47.2	0.0	C17 H17 N O6 35Cl 37Cl2


7.4753
7.4693
7.4645
7.4530
7.4482
7.4423
7.2436
7.2402
7.2372
7.2259
7.2201
7.2152
7.2035
7.1987
7.1927
7.1171
7.0936
5.4831
5.4795
4.8038
4.8019
4.8007
4.7429
4.7126
4.6274
4.6202
4.6132
4.5893
4.5590
4.3133
4.3112
4.2945
4.2923
4.1286
4.1255
4.1050
4.1017
3.8350
3.8204
3.8162
3.8016
3.4104
3.4065
3.4029
3.3993
3.3953
2.0909
2.0877
Current Data Parameters
NAME vd-531-III
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130129
Time 5.38
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 512
DW 104.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300170 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
p-BrBnO
O
O
AcO
NHTCA
12
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.99
2.00
0.96
1.00
1.03
1.07
1.06
1.06
1.07
1.06
1.06
1.03
3.02


Current Data Parameters
NAME vd-531-III
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130130
Time 9.08
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 3869
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 20642.5
DW 20.850 usec
DE 6.50 usec
TE 298.0 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127699 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
169.49
161.50
136.33
131.78
129.34
121.99
100.08
92.37
77.48
77.16
76.85
76.36
73.81
71.60
70.31
65.53
49.51
21.11
p-BrBnO
O
O
AcO
NHTCA
12
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

VD-531 (HR-ESI neg)

Elemental Composition Report

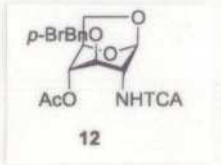
Multiple Mass Analysis: 4 mass(es) processed
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
256 formula(e) evaluated with 7 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 17-17 H: 0-1000 N: 1-1 O: 6-6 35Cl: 0-3 37Cl: 0-3 79Br: 0-1 81Br: 0-1

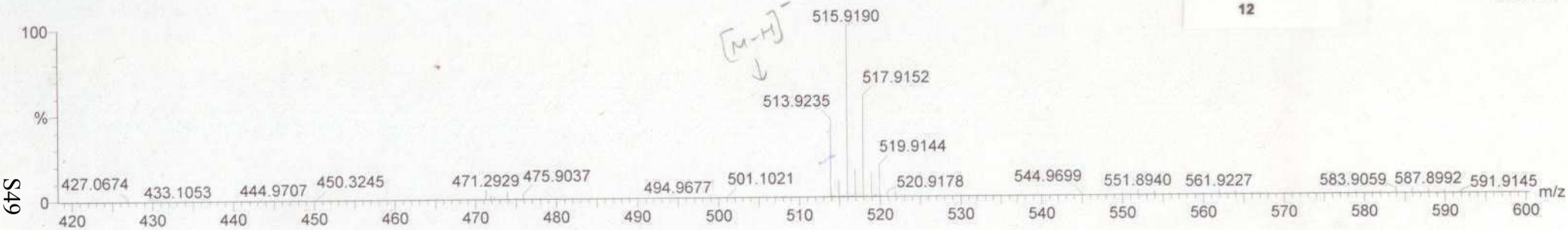
VD-531

0802_VD-531 13 (1.331)

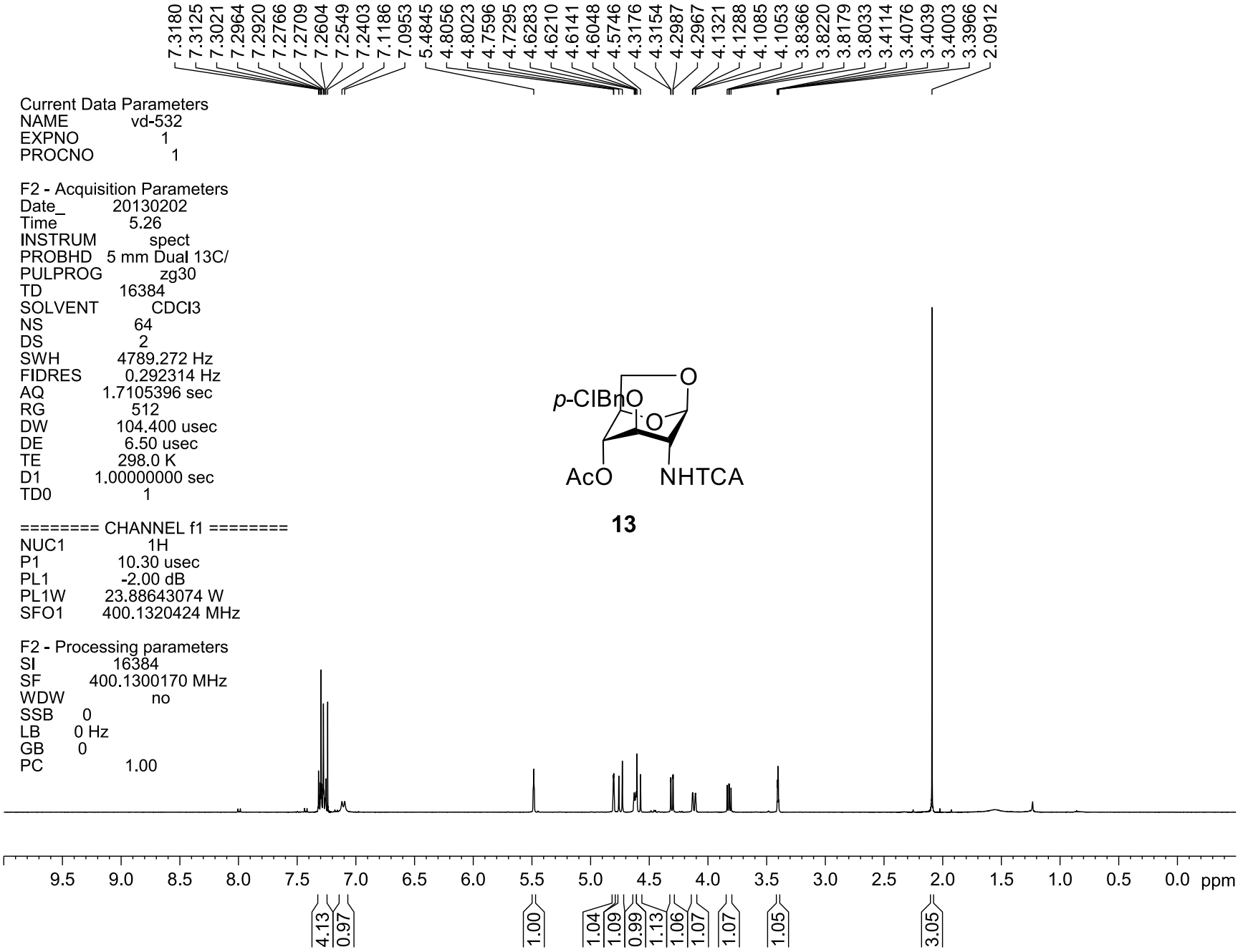
KE267

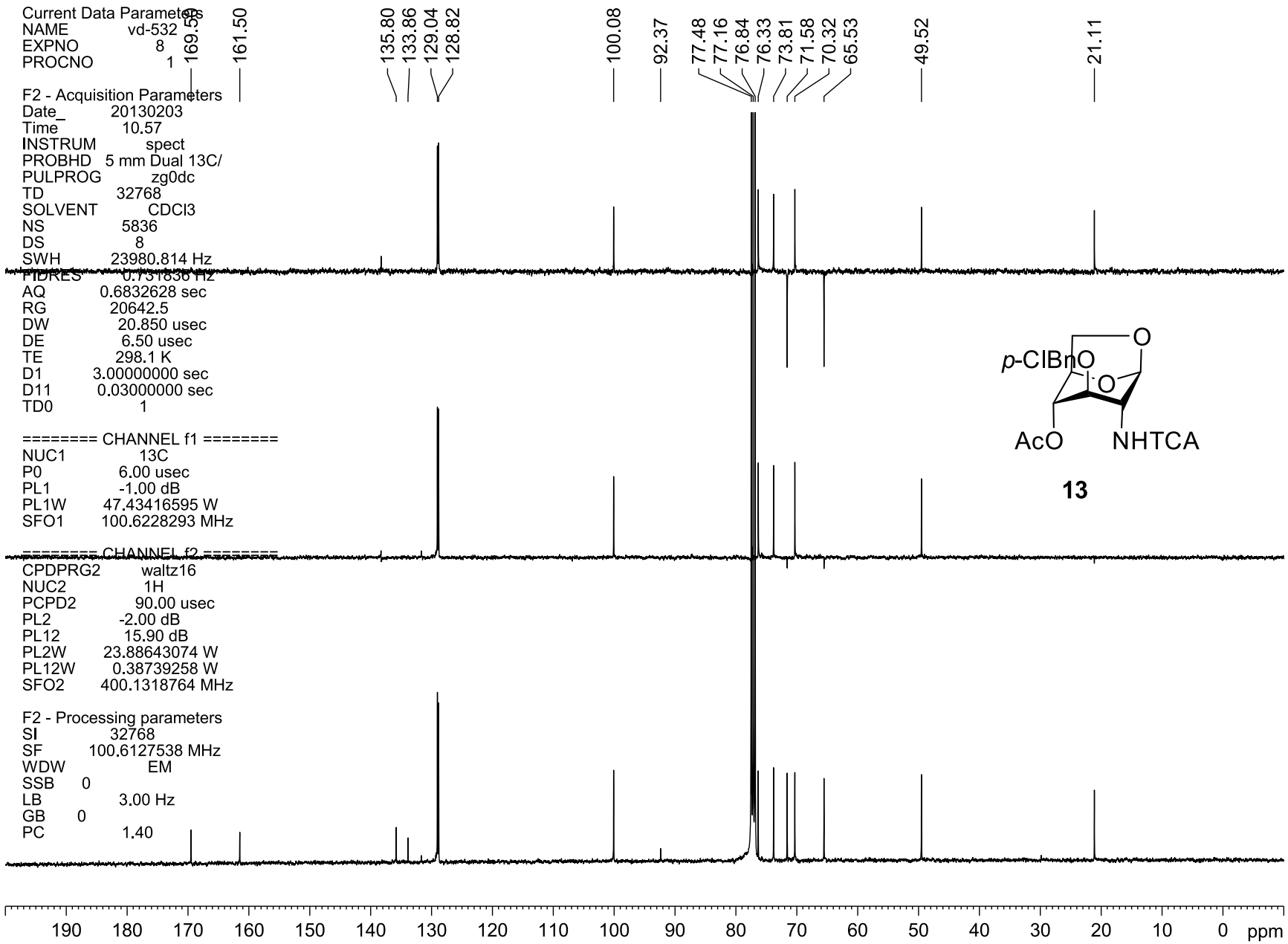


02-Aug-2013
16:02:49
2: TOF MS ES-
1.85e+004



Minimum:	18.00				-1.5			
Maximum:	100.00		5.0	10.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
513.9235	45.49	513.9227	0.8	1.6	8.5	102.0	0.0	C17 H16 N O6 35Cl3 79Br
515.9190	100.00	515.9197	-0.7	-1.4	8.5	100.9	0.6	C17 H16 N O6 35Cl2 37Cl 79Br
		515.9206	-1.6	-3.1	8.5	101.1	0.8	C17 H16 N O6 35Cl3 81Br
517.9152	58.94	517.9177	-2.5	-4.8	8.5	97.6	0.7	C17 H16 N O6 35Cl2 37Cl 81Br
		517.9168	-1.6	-3.1	8.5	97.6	0.7	C17 H16 N O6 35Cl 37Cl2 79Br
519.9144	18.53	519.9147	-0.3	-0.6	8.5	52.6	0.7	C17 H16 N O6 35Cl 37Cl2 81Br
		519.9138	0.6	1.2	8.5	52.6	0.7	C17 H16 N O6 37Cl3 79Br


7.3180
7.3125
7.3021
7.2964
7.2920
7.2766
7.2709
7.2604
7.2549
7.2403
7.1186
7.0953
5.4845
4.8056
4.8023
4.7596
4.7295
4.6283
4.6210
4.6141
4.6048
4.5746
4.3176
4.3154
4.2987
4.2967
4.1321
4.1288
4.1085
4.1053
3.8366
3.8220
3.8179
3.8033
3.4114
3.4076
3.4039
3.4003
3.3966
2.0912
Current Data Parameters
NAME vd-532
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130202
Time 5.26
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 512
DW 104.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300170 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
p-ClBnO
O
O
AcO
NHTCA
13
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.13
0.97
1.00
1.04
1.09
0.99
1.13
1.06
1.07
1.07
1.05
3.05


Current Data Parameters
NAME vd-532
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130203
Time 10.57
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 5836
DS 8
SWH 23980.814 Hz
FIDRES 0.731838 Hz
AQ 0.6832628 sec
RG 20642.5
DW 20.850 usec
DE 6.50 usec
TE 298.1 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127538 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
169.58
161.50
135.80
133.86
129.04
128.82
100.08
92.37
77.48
77.16
76.84
76.33
73.81
71.58
70.32
65.53
49.52
21.11
p-ClBnO
O
O
O
AcO
NHTCA
13
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

VD-532 (HR-ESI neg)

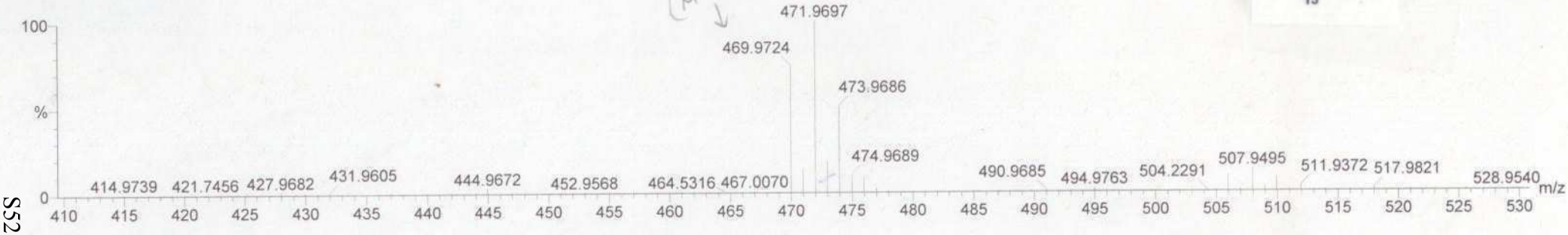
Elemental Composition Report

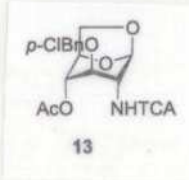
Multiple Mass Analysis: 6 mass(es) processed
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
150 formula(e) evaluated with 4 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 17-17 H: 0-1000 N: 1-1 O: 6-6 35Cl: 0-4 37Cl: 0-4
VD-532

KE267

0802_VD-532 50 (4.937)

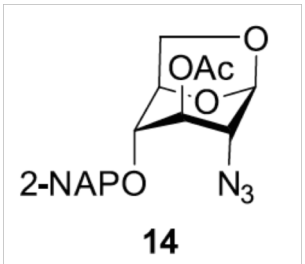




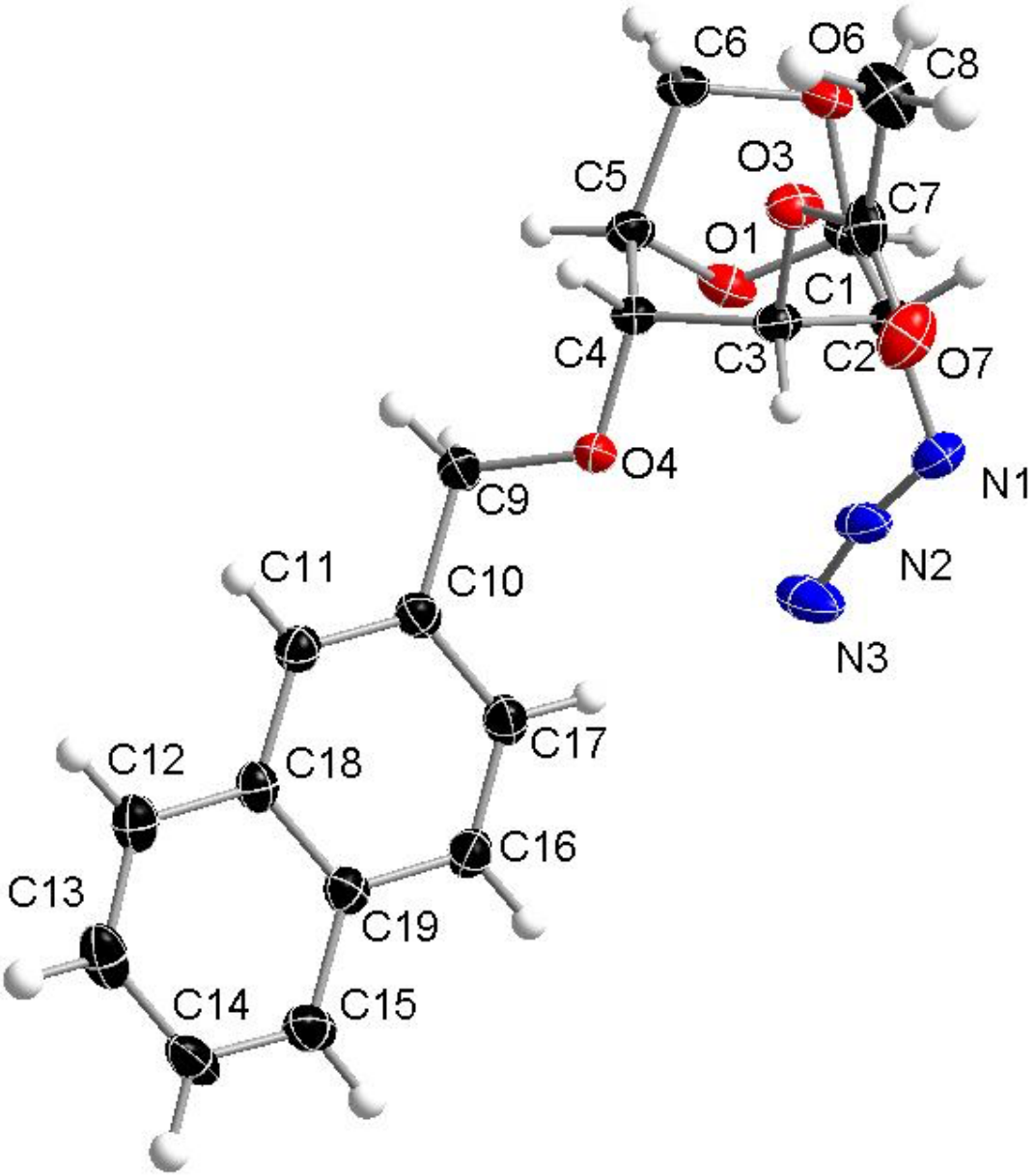
02-Aug-2013
15:51:34
2: TOF MS ES-
2.68e+003

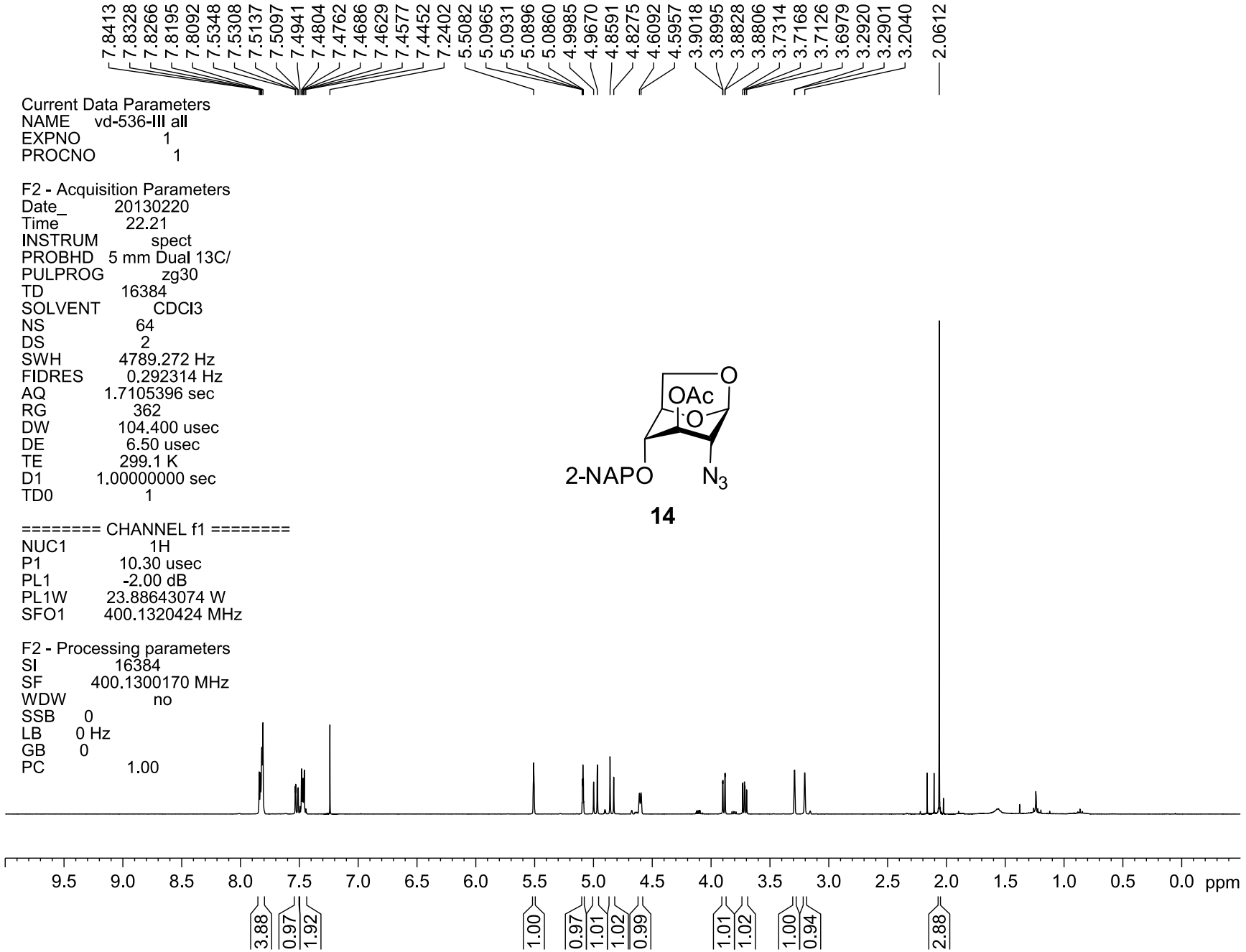
Minimum:	12.00				-1.5			
Maximum:	100.00		5.0	10.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
469.9724	73.81	469.9732	-0.8	-1.7	8.5	36.1	0.0	C17 H16 N O6 35Cl4
470.9790	13.66	---						
471.9697	100.00	471.9702	-0.5	-1.1	8.5	38.7	0.0	C17 H16 N O6 35Cl3 37Cl
472.9752	18.64	---						
473.9686	50.73	473.9673	1.3	2.7	8.5	38.1	0.0	C17 H16 N O6 35Cl2 37Cl2
507.9495	12.65	507.9469	2.6	5.1	7.5	35.1	0.0	C17 H17 N O6 35Cl4 37Cl

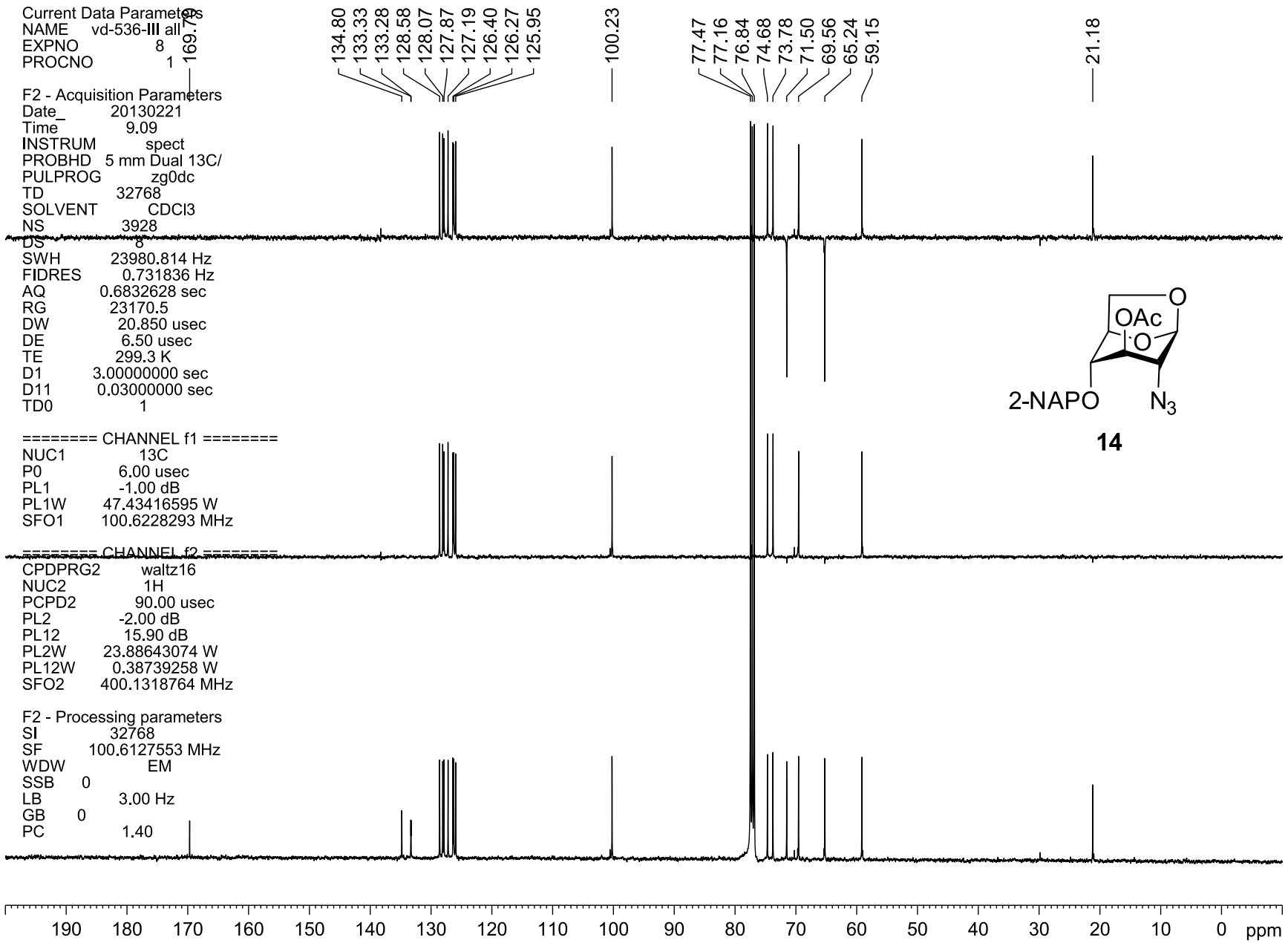
ORTEP diagram of **14** with the thermal ellipsoids drawn at 50% probability level.



14




7.8413
7.8328
7.8266
7.8195
7.8092
7.5348
7.5308
7.5137
7.5097
7.4941
7.4804
7.4762
7.4686
7.4629
7.4577
7.4452
7.2402
5.5082
5.0965
5.0931
5.0896
5.0860
4.9985
4.9670
4.8591
4.8275
4.6092
4.5957
3.9018
3.8995
3.8828
3.8806
3.7314
3.7168
3.7126
3.6979
3.2920
3.2901
3.2040
2.0612
Current Data Parameters
NAME vd-536-III all
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130220
Time 22.21
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 362
DW 104.400 usec
DE 6.50 usec
TE 299.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300170 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
O
OAc
O
2-NAPO
N3
14
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
3.88
0.97
1.92
1.00
0.97
1.01
1.02
0.99
1.01
1.02
1.00
0.94
2.88


Current Data Parameters
NAME vd-536-III all
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130221
Time 9.09
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 3928
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 23170.5
DW 20.850 usec
DE 6.50 usec
TE 299.3 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127553 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
169.77
134.80
133.33
133.28
128.58
128.07
127.87
127.19
126.40
126.27
125.95
100.23
77.47
77.16
76.84
74.68
73.78
71.50
69.56
65.24
59.15
21.18
O
OAc
O
2-NAPO
N3
14
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

VD-536 (HR-ESI)

Elemental Composition Report

Single Mass Analysis

Tolerance = 100.0 PPM / DBE: min = -1.5, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
4 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 3-3 O: 5-5 Na: 1-1

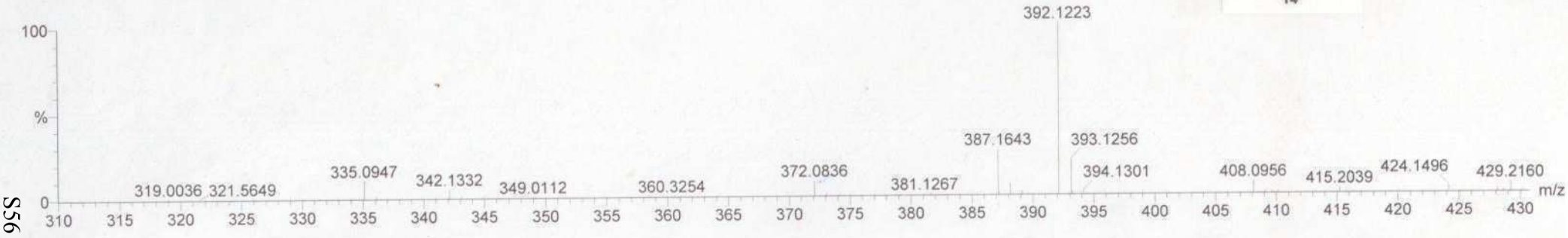
VD-536

KE267

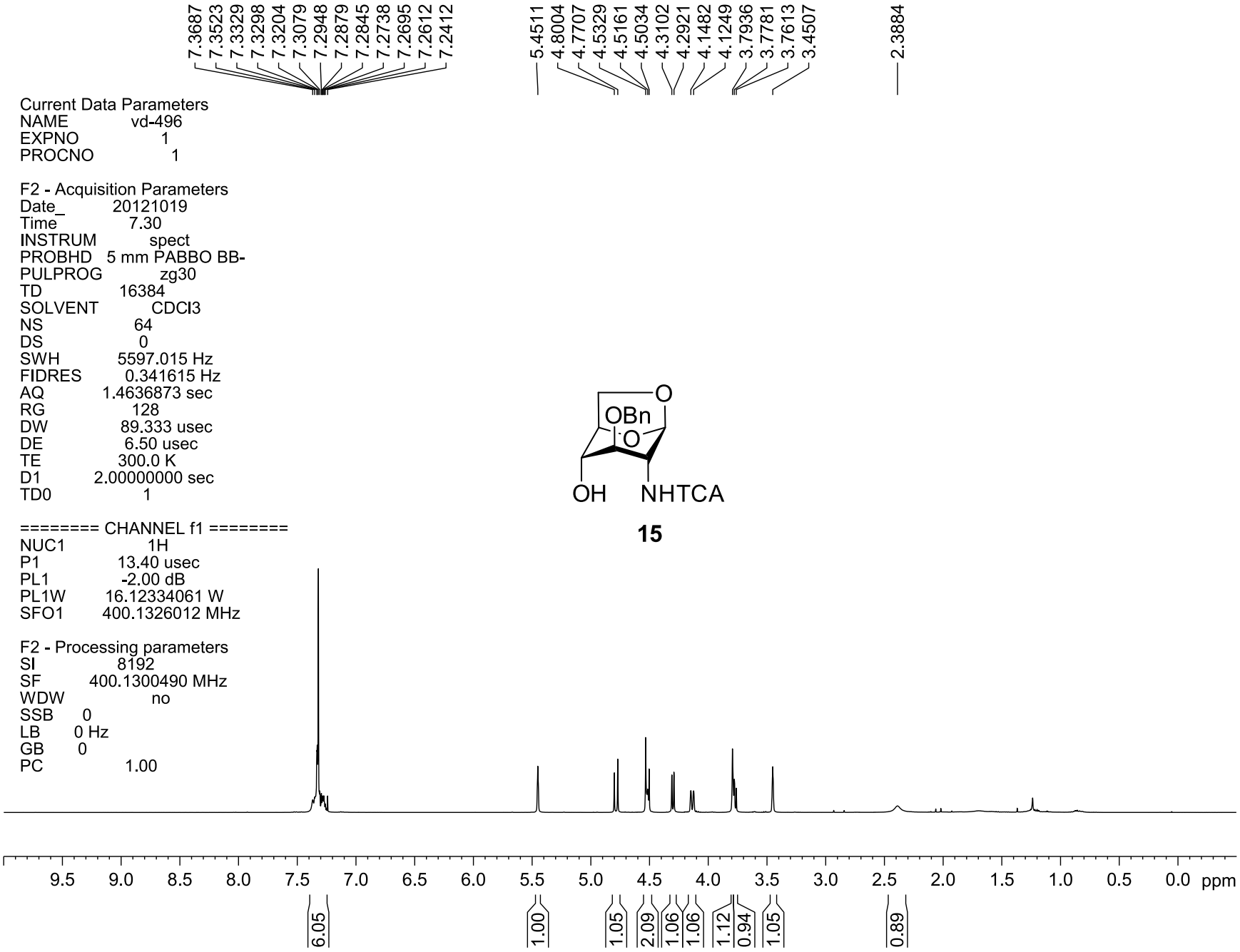
$[M+Na]^+$

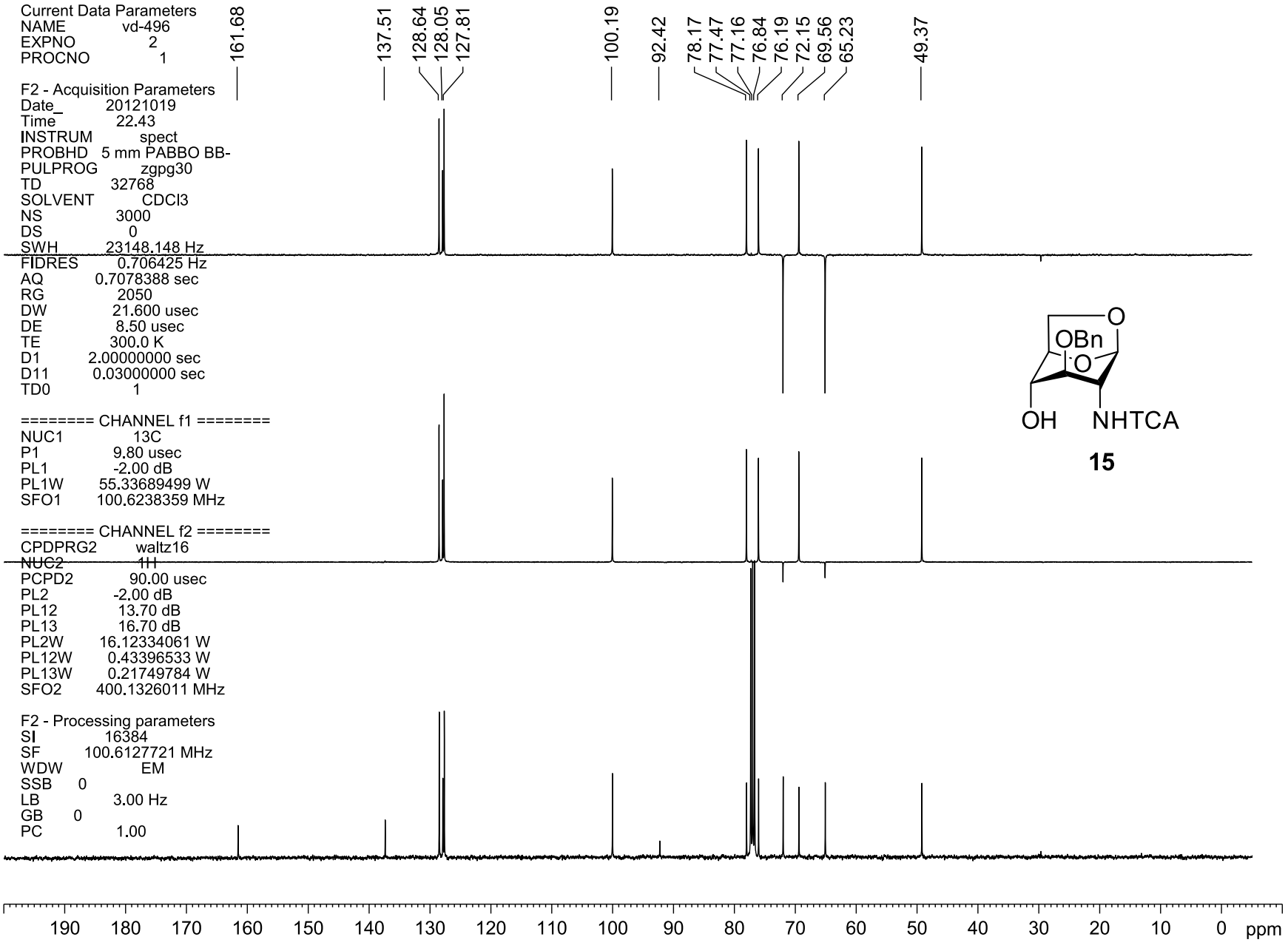
02-Aug-2013
16:25:30
1: TOF MS ES+
6.43e+003

0802_VD-536 38 (3.723) Cm (38-1)



Minimum:				-1.5			
Maximum:		5.0	100.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
392.1223	392.1222	0.1	0.3	11.5	31.0	0.0	C19 H19 N3 O5 Na

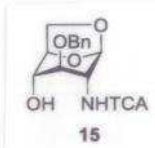

7.3687
7.3523
7.3329
7.3298
7.3204
7.3079
7.2948
7.2879
7.2845
7.2738
7.2695
7.2612
7.2412
5.4511
4.8004
4.7707
4.5329
4.5161
4.5034
4.3102
4.2921
4.1482
4.1249
3.7936
3.7781
3.7613
3.4507
2.3884
Current Data Parameters
NAME vd-496
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121019
Time 7.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 128
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
F2 - Processing parameters
SI 8192
SF 400.1300490 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
O
OBn
O
OH
NHTCA
15
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
6.05
1.00
1.05
2.09
1.06
1.06
1.12
0.94
1.05
0.89


Current Data Parameters
NAME vd-496
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121019
Time 22.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 3000
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238359 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127721 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
161.68
137.51
128.64
128.05
127.81
100.19
92.42
78.17
77.47
77.16
76.84
76.19
72.15
69.56
65.23
49.37
O
OBn
O
OH
NHTCA
15
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

VD-496 (HR-ESI)

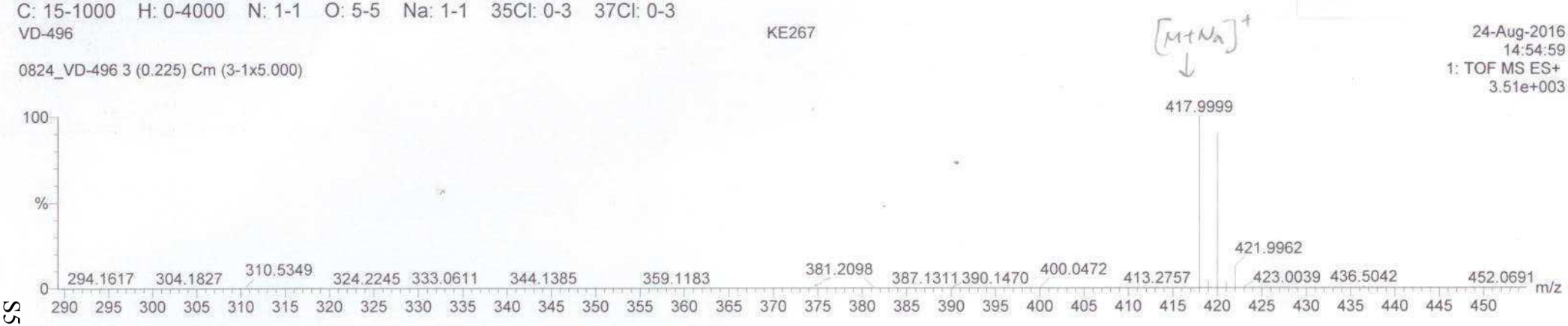
Elemental Composition Report

Multiple Mass Analysis: 3 mass(es) processed
Tolerance = 8.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

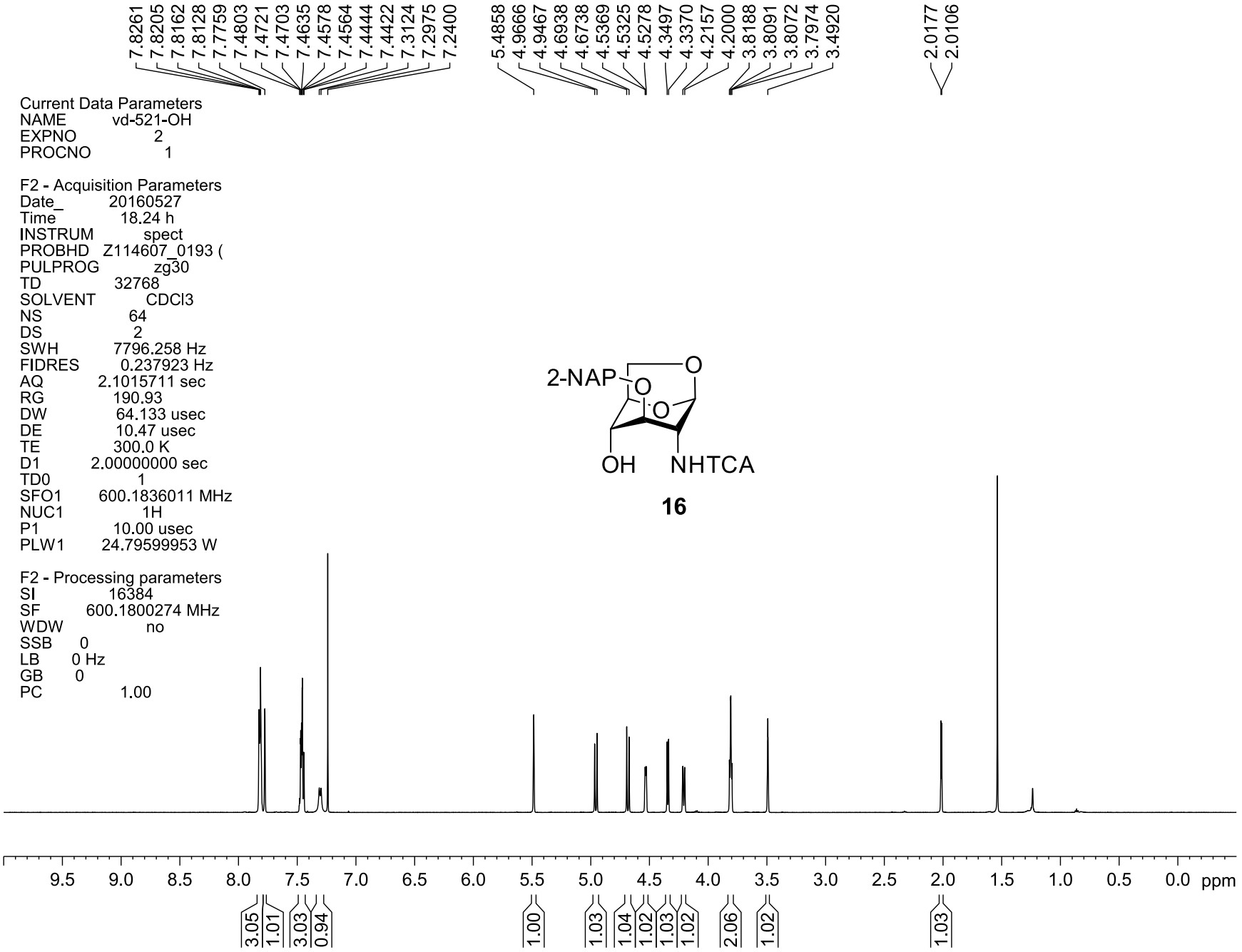


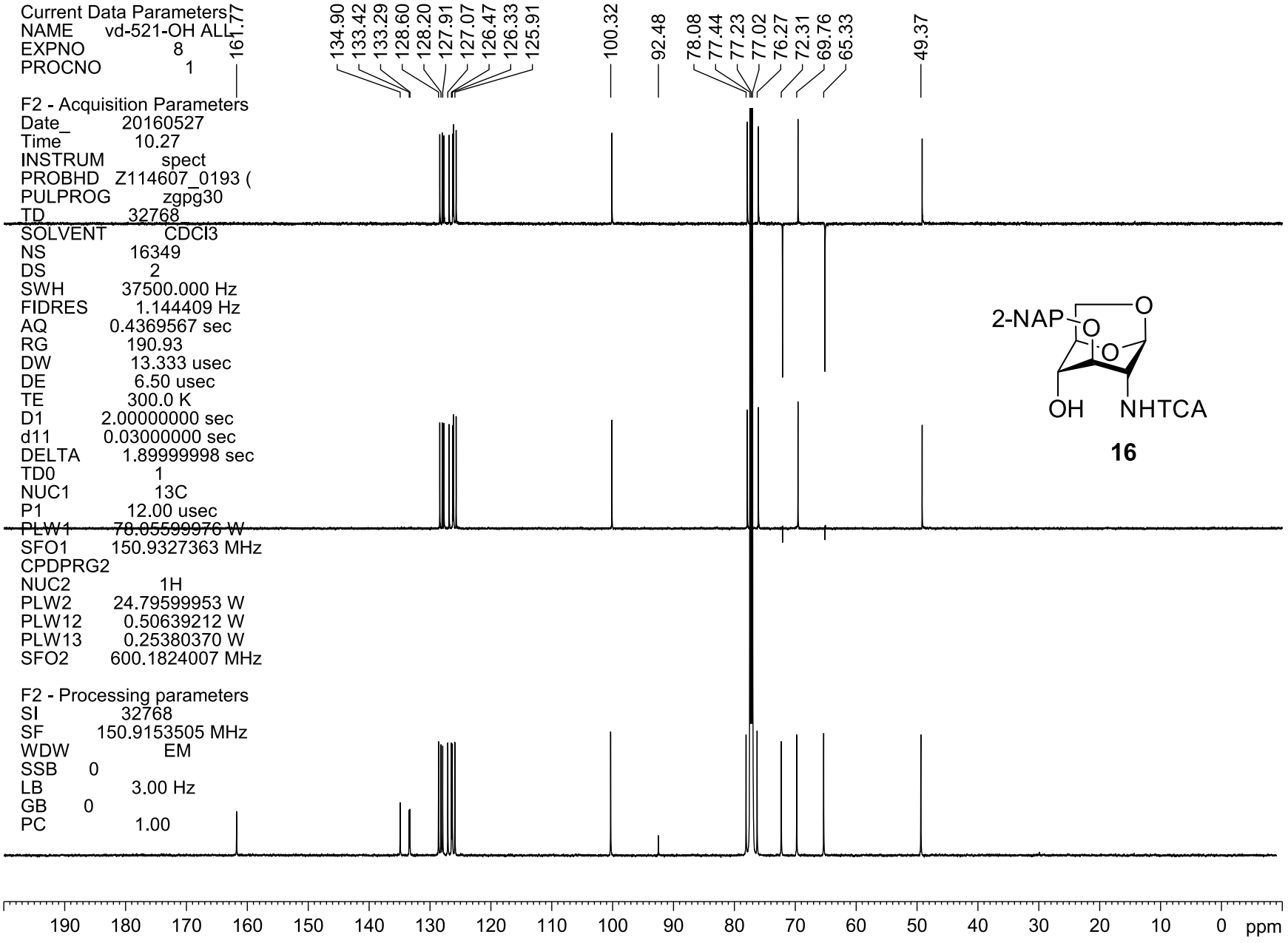
Monoisotopic Mass, Even Electron Ions
167 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 15-1000 H: 0-4000 N: 1-1 O: 5-5 Na: 1-1 35Cl: 0-3 37Cl: 0-3

VD-496 KE267 24-Aug-2016 14:54:59
0824_VD-496 3 (0.225) Cm (3-1x5.000) 1: TOF MS ES+ 3.51e+003



Minimum:	11.00				-1000.0			
Maximum:	100.00		5.0	8.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
417.9999	100.00	417.9992	0.7	1.7	6.5	25.2	0.0	C15 H16 N O5 Na 35Cl3
419.9964	90.14	419.9962	0.2	0.5	6.5	25.9	0.0	C15 H16 N O5 Na 35Cl2 37Cl
421.9962	12.41	421.9933	2.9	6.9	6.5	24.8	0.0	C15 H16 N O5 Na 35Cl 37Cl2


7.8261
7.8205
7.8162
7.8128
7.7759
7.4803
7.4721
7.4703
7.4635
7.4578
7.4564
7.4444
7.4422
7.3124
7.2975
7.2400
5.4858
4.9666
4.9467
4.6938
4.6738
4.5369
4.5325
4.5278
4.3497
4.3370
4.2157
4.2000
3.8188
3.8091
3.8072
3.7974
3.4920
2.0177
2.0106
Current Data Parameters
NAME vd-521-OH
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160527
Time 18.24 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015711 sec
RG 190.93
DW 64.133 usec
DE 10.47 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800274 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
2-NAP
O
O
O
O
OH
NHTCA
16
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
3.05
1.01
3.03
0.94
1.00
1.03
1.04
1.02
1.03
1.02
2.06
1.02
1.03


Current Data Parameters
NAME vd-521-OH ALL
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160527
Time 10.27
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16349
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369567 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO1 150.9327363 MHz
CPDPRG2
NUC2 1H
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
SFO2 600.1824007 MHz
F2 - Processing parameters
SI 32768
SF 150.9153505 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
161.77
134.90
133.42
133.29
128.60
128.20
127.91
127.07
126.47
126.33
125.91
100.32
92.48
78.08
77.44
77.23
77.02
76.27
72.31
69.76
65.33
49.37
2-NAP
O
O
O
O
OH
NHTCA
16
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

vd-521-OH (HR-ESI neg)

Elemental Composition Report

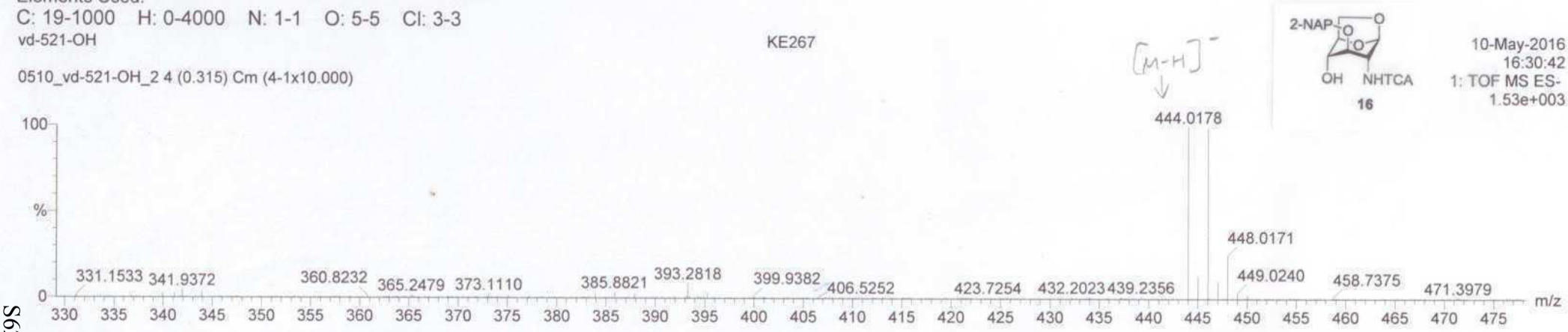
Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

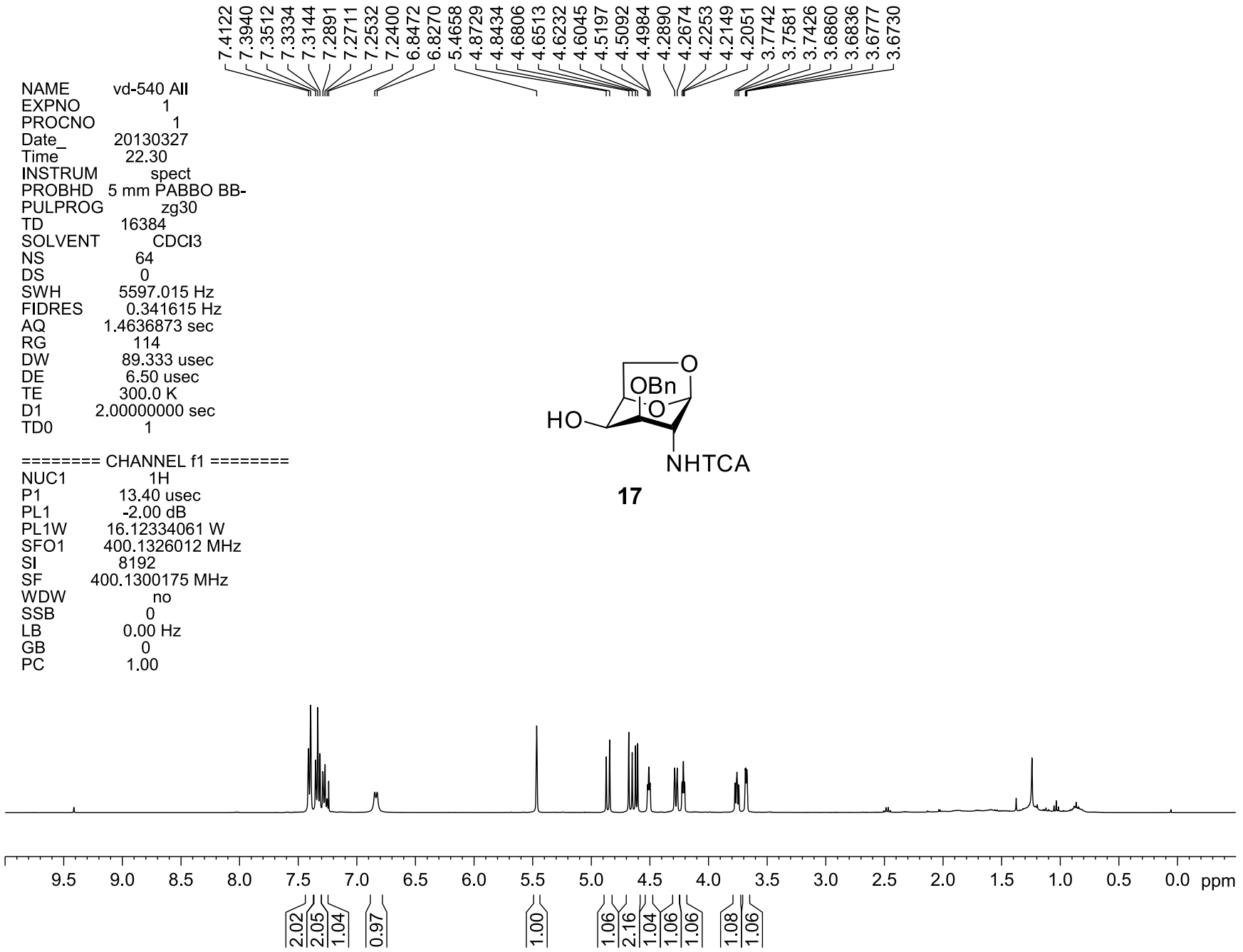
Monoisotopic Mass, Even Electron Ions
2 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 19-1000 H: 0-4000 N: 1-1 O: 5-5 Cl: 3-3

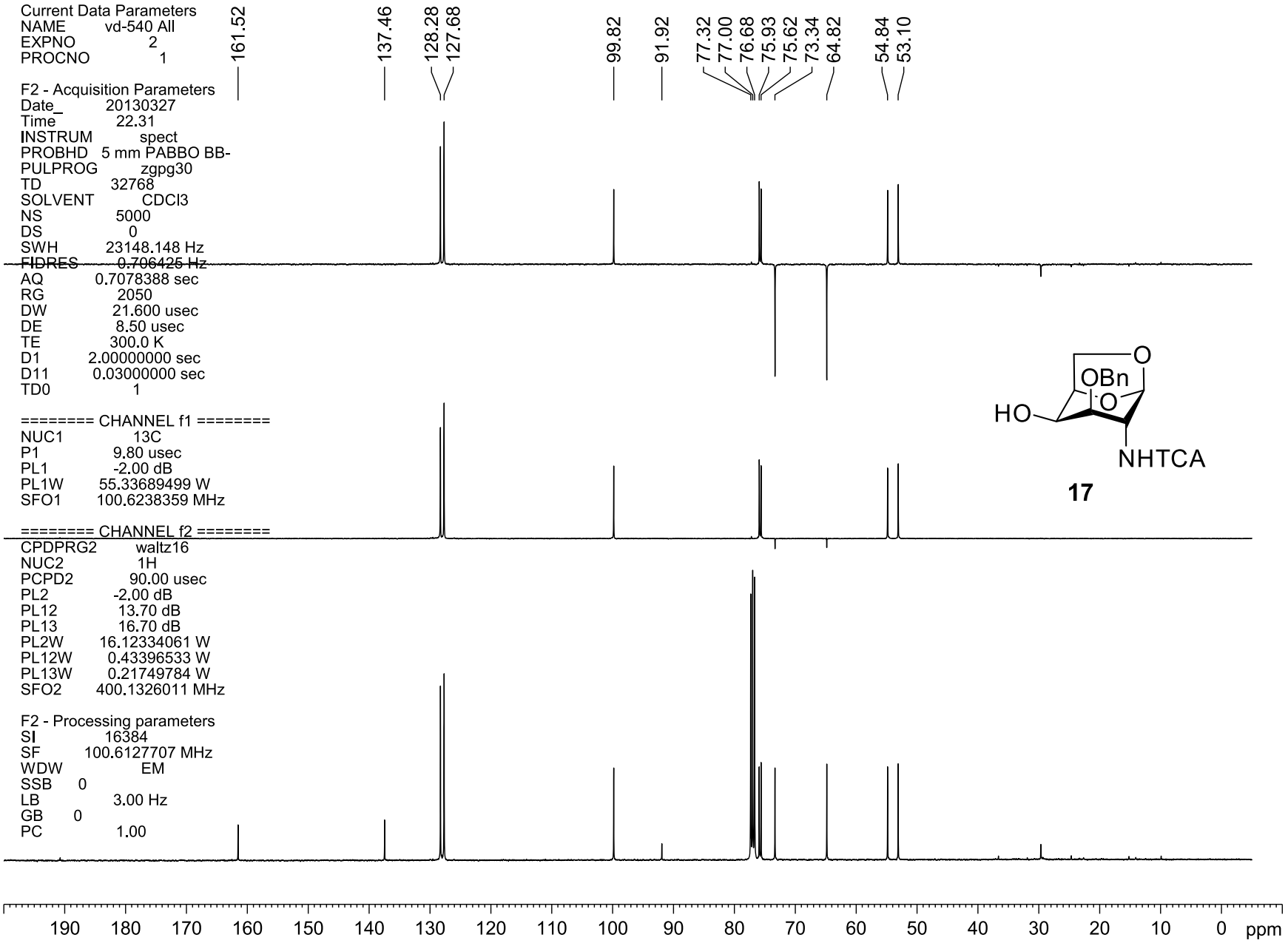
vd-521-OH KE267

0510_vd-521-OH_2 4 (0.315) Cm (4-1x10.000)



Minimum:				-1000.0			
Maximum:		5.0	10.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
444.0178	444.0172	0.6	1.4	10.5	44.8	0.0	C19 H17 N O5 Cl3


7.4122
7.3940
7.3512
7.3334
7.3144
7.2891
7.2711
7.2532
7.2400
6.8472
6.8270
5.4658
4.8729
4.8434
4.6806
4.6513
4.6232
4.6045
4.5197
4.5092
4.4984
4.2890
4.2674
4.2253
4.2149
4.2051
3.7742
3.7581
3.7426
3.6860
3.6836
3.6777
3.6730
NAME vd-540 All
EXPNO 1
PROCNO 1
Date_ 20130327
Time 22.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 114
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
O
OBn
O
HO
NHTCA
17
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.02
2.05
1.04
0.97
1.00
1.06
2.16
1.04
1.06
1.06
1.08
1.06

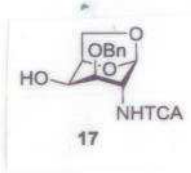

Current Data Parameters
NAME vd-540 All
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130327
Time 22.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 5000
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238359 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127707 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
161.52
137.46
128.28
127.68
99.82
91.92
77.32
77.00
76.68
75.93
75.62
73.34
64.82
54.84
53.10
O
OBn
O
HO
NHTCA
17
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

VD-540 (HR-ESI)

Elemental Composition Report

Multiple Mass Analysis: 5 mass(es) processed

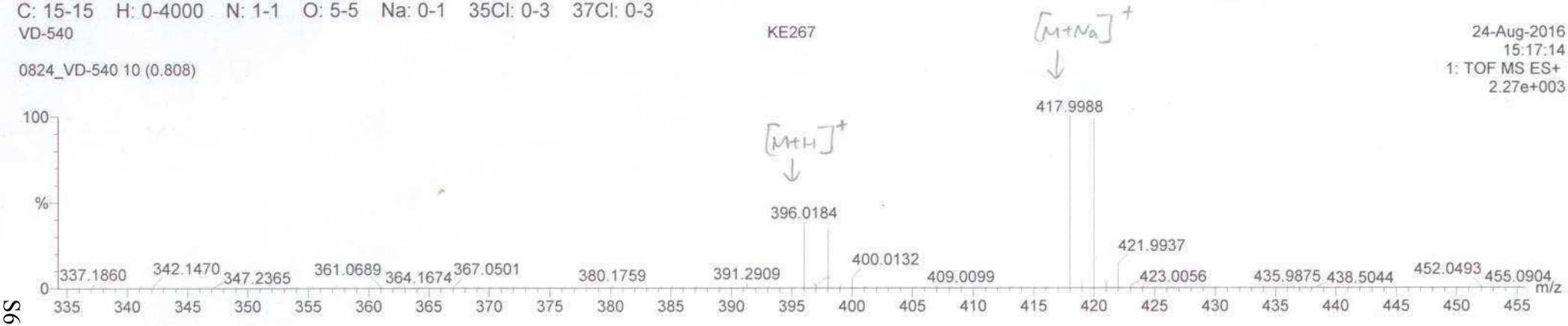
Tolerance = 11.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2



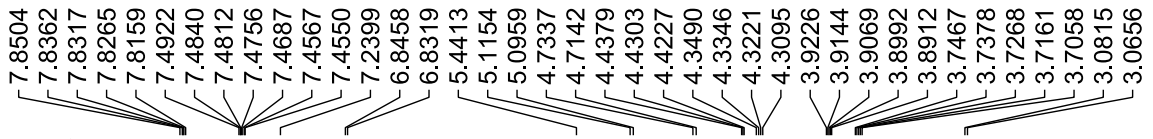
17

Monoisotopic Mass, Even Electron Ions
156 formula(e) evaluated with 5 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 15-15 H: 0-4000 N: 1-1 O: 5-5 Na: 0-1 35Cl: 0-3 37Cl: 0-3

VD-540 KE267 24-Aug-2016 15:17:14
0824_VD-540 10 (0.808) 1: TOF MS ES+ 2.27e+003



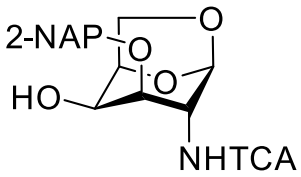
Minimum:	10.00				-1000.0			
Maximum:	100.00		5.0	11.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
396.0184	38.26	396.0172	1.2	3.0	6.5	26.3	0.0	C15 H17 N O5 35Cl3
398.0142	34.24	398.0143	-0.1	-0.3	6.5	28.6	0.0	C15 H17 N O5 35Cl2 37Cl
417.9988	100.00	417.9992	-0.4	-1.0	6.5	25.8	0.0	C15 H16 N O5 Na 35Cl3
419.9954	98.37	419.9962	-0.8	-1.9	6.5	26.8	0.0	C15 H16 N O5 Na 35Cl2 37Cl
421.9937	13.91	421.9933	0.4	0.9	6.5	21.8	0.0	C15 H16 N O5 Na 35Cl 37Cl2



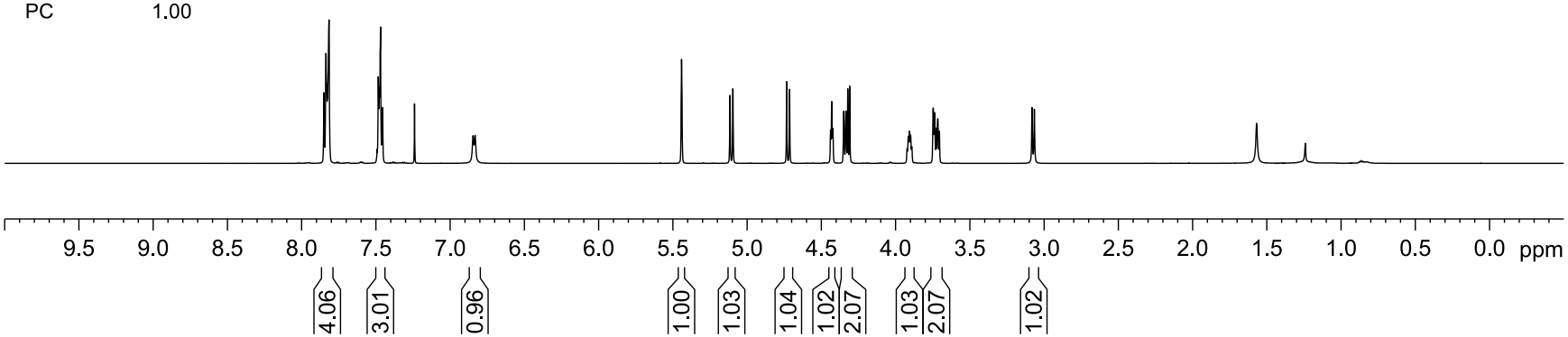
Current Data Parameters
NAME vd-550 ALL
EXPNO 1
PROCNO 1

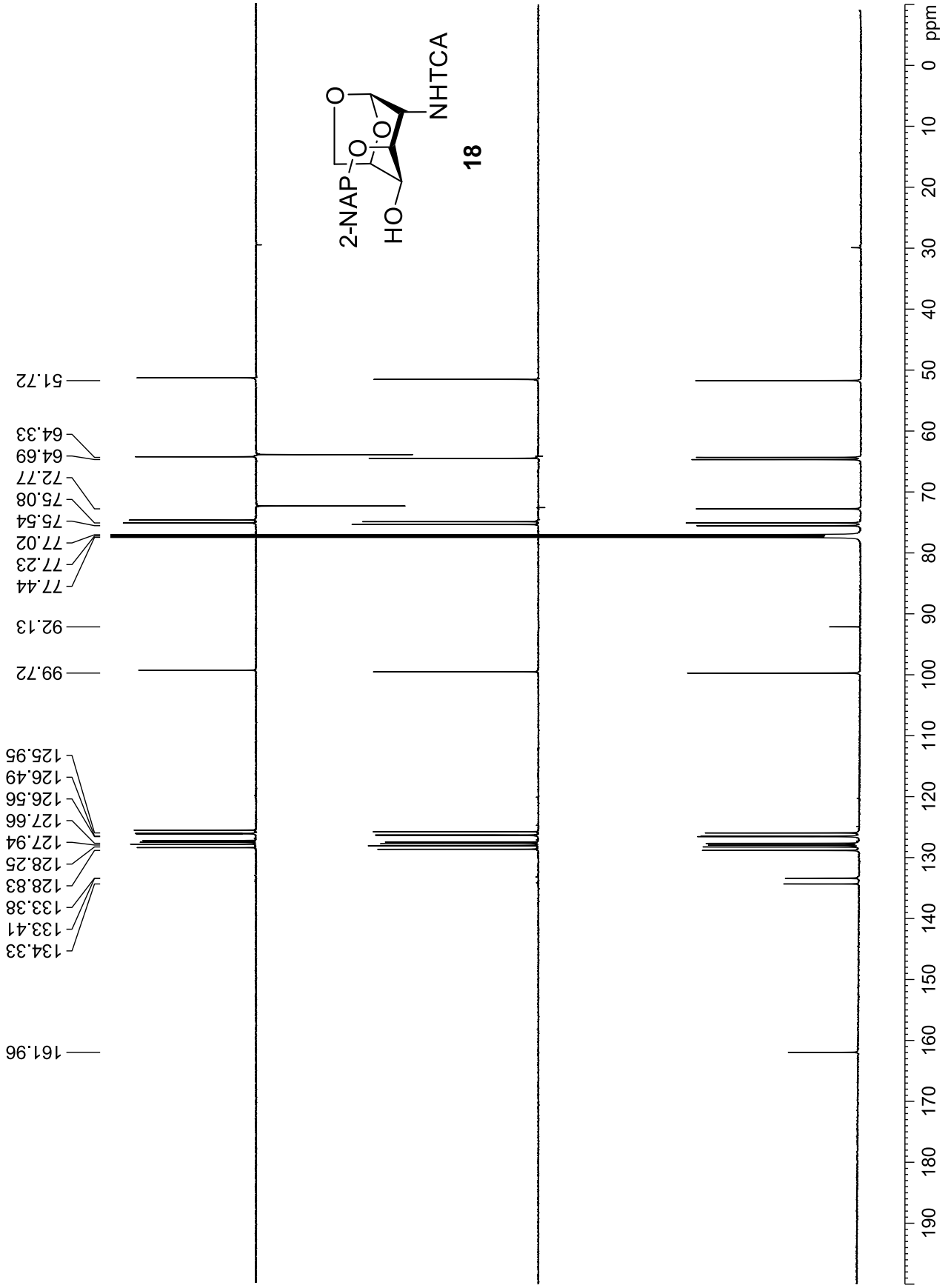
F2 - Acquisition Parameters
Date_ 20160531
Time 18.46 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015711 sec
RG 118.65
DW 64.133 usec
DE 10.47 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W

F2 - Processing parameters
SI 16384
SF 600.1800274 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



18




2-NAP
HO
NHTCA
18
51.72
64.33
64.69
72.77
75.08
75.54
77.02
77.23
77.44
92.13
99.72
125.95
126.49
126.56
127.66
127.94
128.25
128.83
133.38
133.41
134.33
161.96
ppm

vd-550 (HR-ESI neg)

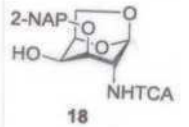
Elemental Composition Report

Multiple Mass Analysis: 3 mass(es) processed
Tolerance = 4.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
960 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 1-1000 H: 0-4000 N: 1-1 O: 5-5 35Cl: 0-3 37Cl: 0-3

vd-550

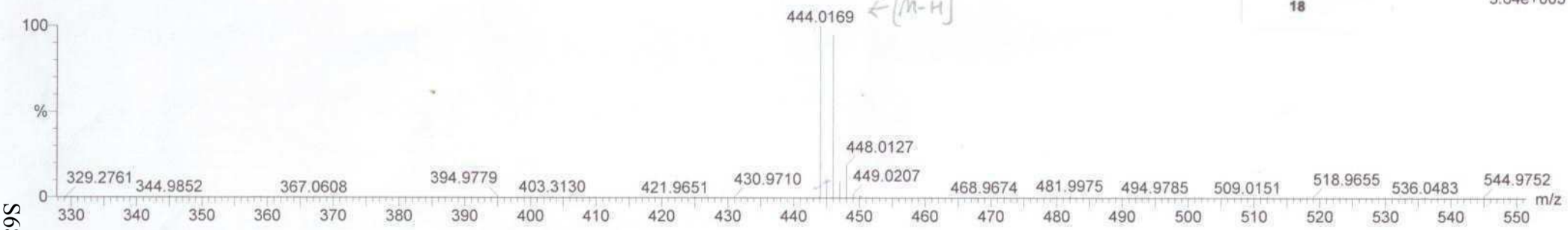
KE267



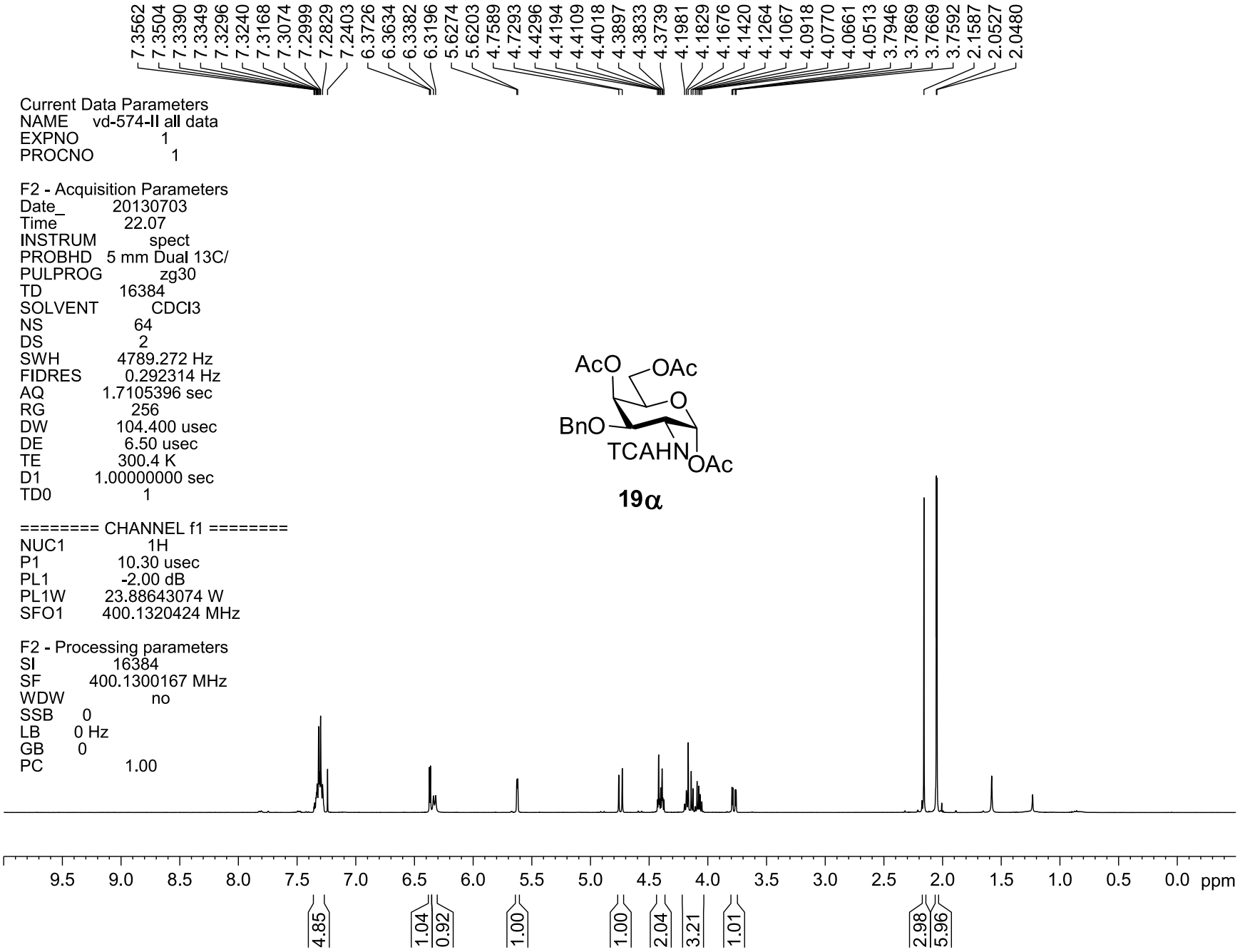
18

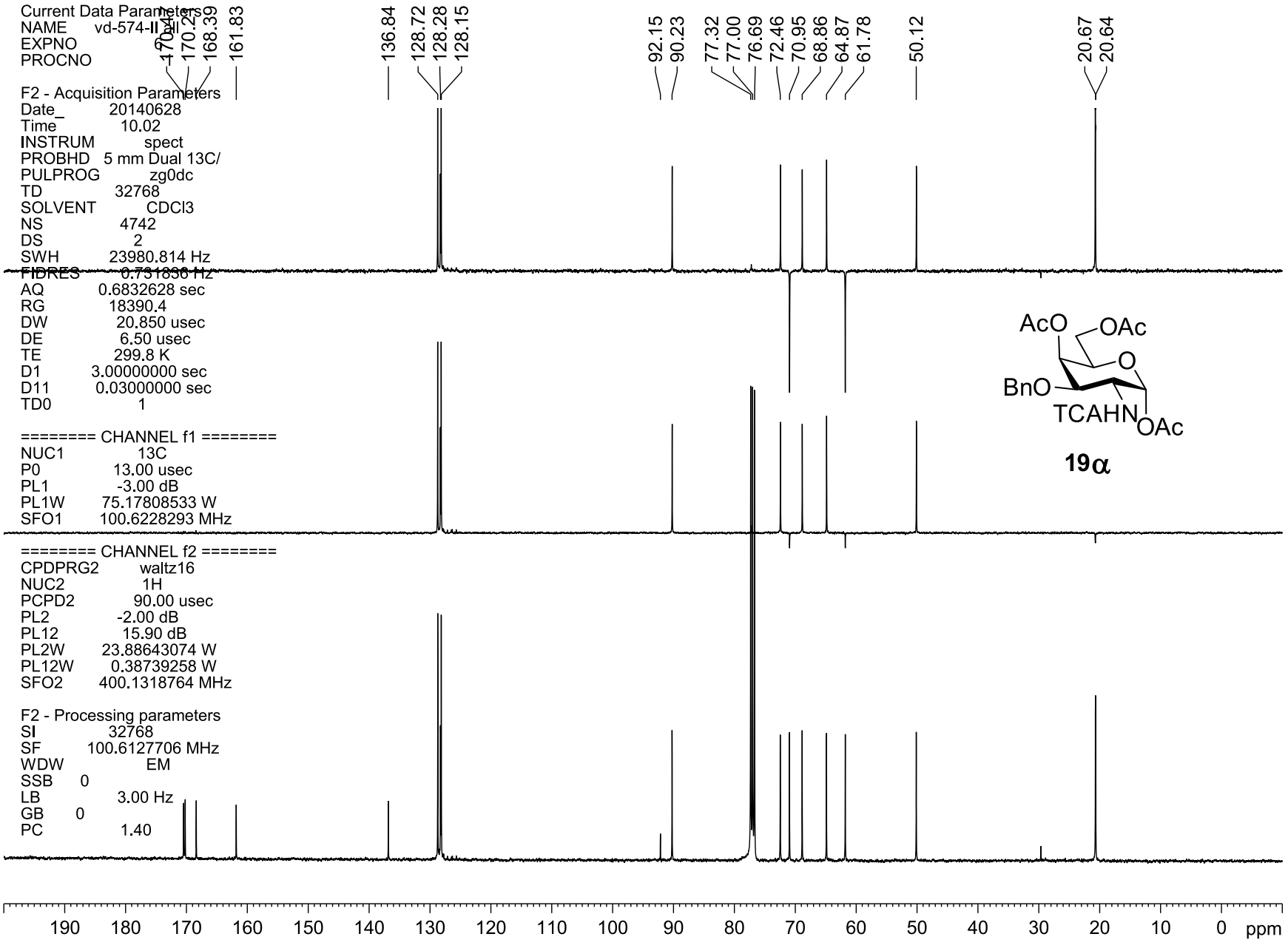
01-Jun-2016
13:25:42
2: TOF MS ES-
5.64e+003

0601_vd-550 49 (2.772)



Minimum:	12.00				-1000.0			
Maximum:	100.00		5.0	4.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
444.0169	100.00	444.0172	-0.3	-0.7	10.5	41.4	0.0	C19 H17 N O5 35Cl3
446.0146	95.17	446.0143	0.3	0.7	10.5	33.8	0.0	C19 H17 N O5 35Cl2 37Cl
448.0127	18.98	448.0113	1.4	3.1	10.5	37.6	0.0	C19 H17 N O5 35Cl 37Cl2


7.3562
7.3504
7.3390
7.3349
7.3296
7.3240
7.3168
7.3074
7.2999
7.2829
7.2403
6.3726
6.3634
6.3382
6.3196
5.6274
5.6203
4.7589
4.7293
4.4296
4.4194
4.4109
4.4018
4.3897
4.3833
4.3739
4.1981
4.1829
4.1676
4.1420
4.1264
4.1067
4.0918
4.0770
4.0661
4.0513
3.7946
3.7869
3.7669
3.7592
2.1587
2.0527
2.0480
Current Data Parameters
NAME vd-574-II all data
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130703
Time 22.07
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 256
DW 104.400 usec
DE 6.50 usec
TE 300.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300167 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
AcO
OAc
O
BnO
TCAHN
OAc
19α
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.85
1.04
0.92
1.00
1.00
2.04
3.21
1.01
2.98
5.96


Current Data Parameters
NAME vd-574-II all
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140628
Time 10.02
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 4742
DS 2
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 18390.4
DW 20.850 usec
DE 6.50 usec
TE 299.8 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 13.00 usec
PL1 -3.00 dB
PL1W 75.17808533 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127706 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
170.41
170.22
168.39
161.83
136.84
128.72
128.28
128.15
92.15
90.23
77.32
77.00
76.69
72.46
70.95
68.86
64.87
61.78
50.12
20.67
20.64
AcO
OAc
O
BnO
TCAHN
OAc
19α
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

VD-524-II

Elemental Composition Report

Single Mass Analysis

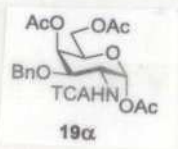
Tolerance = 100.0 PPM / DBE: min = -1.5, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
4 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1 O: 9-9 Na: 1-1 Cl: 3-3

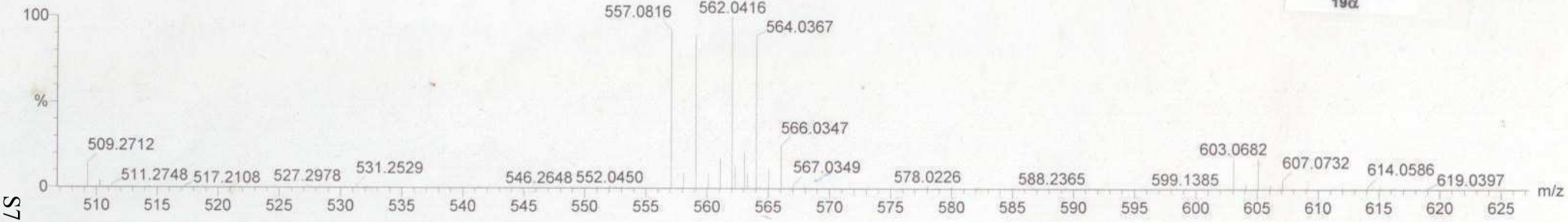
vd-574-II

KE267

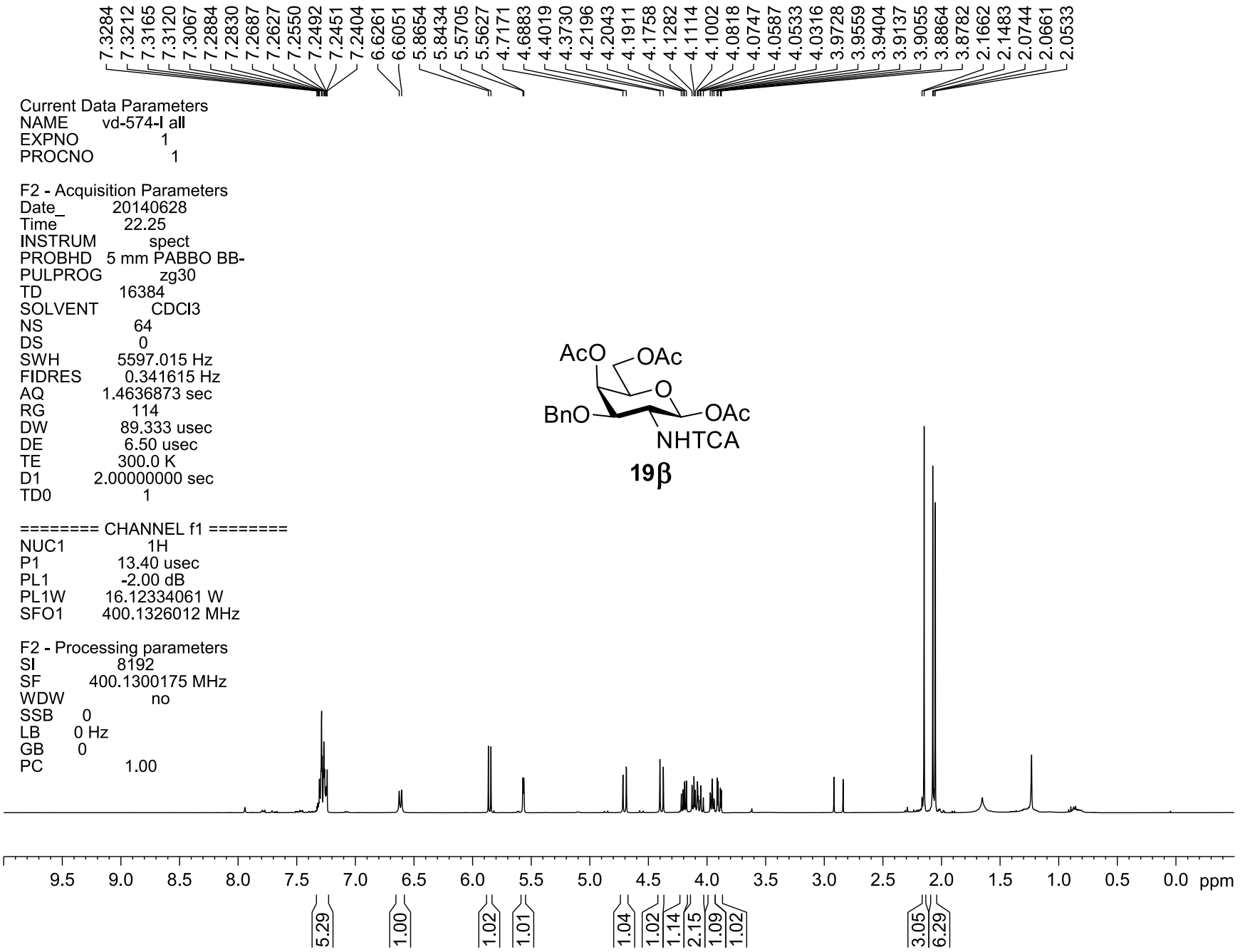
0705_vd-574-II 20 (1.913)

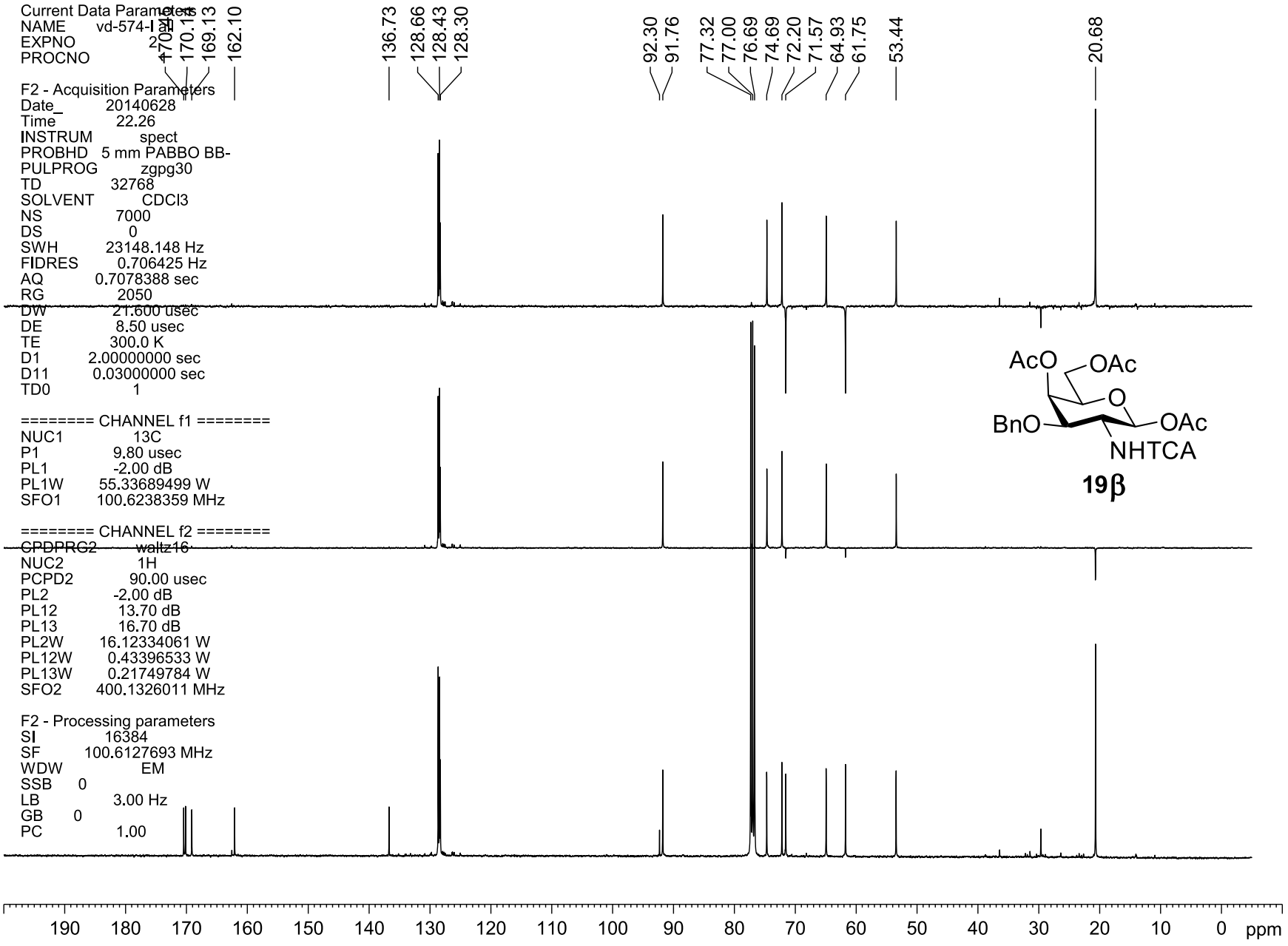


05-Jul-2013
12:58:26
1: TOF MS ES+
1.07e+004



Minimum:				-1.5			
Maximum:		5.0	100.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
562.0416	562.0414	0.2	0.4	8.5	57.7	0.0	C21 H24 N O9 Na Cl3


7.3284
7.3212
7.3165
7.3120
7.3067
7.2884
7.2830
7.2687
7.2627
7.2550
7.2492
7.2451
7.2404
6.6261
6.6051
5.8654
5.8434
5.5705
5.5627
4.7171
4.6883
4.4019
4.3730
4.2196
4.2043
4.1911
4.1758
4.1282
4.1114
4.1002
4.0818
4.0747
4.0587
4.0533
4.0316
3.9728
3.9559
3.9404
3.9137
3.9055
3.8864
3.8782
2.1662
2.1483
2.0744
2.0661
2.0533
Current Data Parameters
NAME vd-574-I all
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140628
Time 22.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 114
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
AcO
OAc
O
BnO
OAc
NHTCA
19β
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
5.29
1.00
1.02
1.01
1.04
1.02
1.14
2.15
1.09
1.02
3.05
6.29


Current Data Parameters
NAME vd-574-I-3d
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140628
Time 22.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 7000
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238359 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127693 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
170.31
170.17
169.13
162.10
136.73
128.66
128.43
128.30
92.30
91.76
77.32
77.00
76.69
74.69
72.20
71.57
64.93
61.75
53.44
20.68
AcO
OAc
O
BnO
OAc
NHTCA
19β
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

vd-574-1 (HR-ESI)

Elemental Composition Report

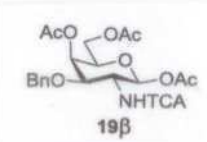
Multiple Mass Analysis: 3 mass(es) processed
Tolerance = 5.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
290 formula(e) evaluated with 3 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 18-1000 H: 0-1000 N: 1-1 O: 9-9 Na: 1-1 35Cl: 0-3 37Cl: 0-3

vd-574-1

KE267

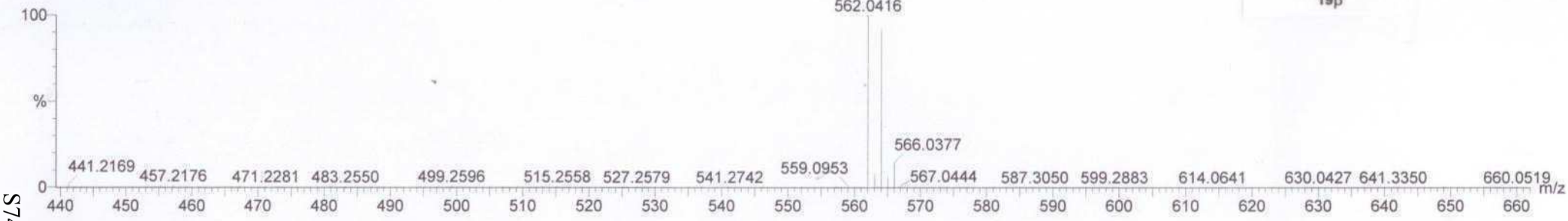
$[M+Na]^+$



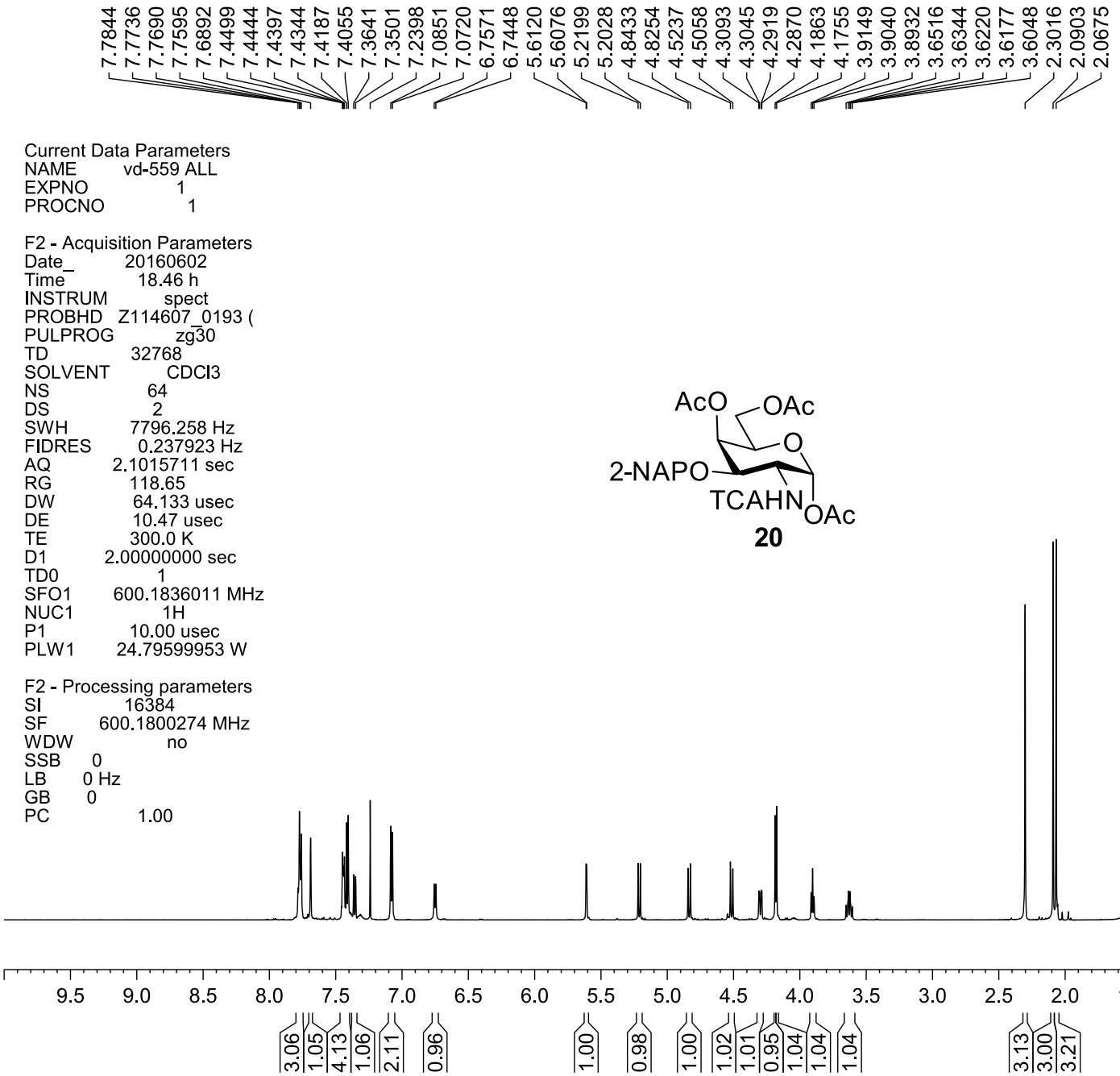
19β

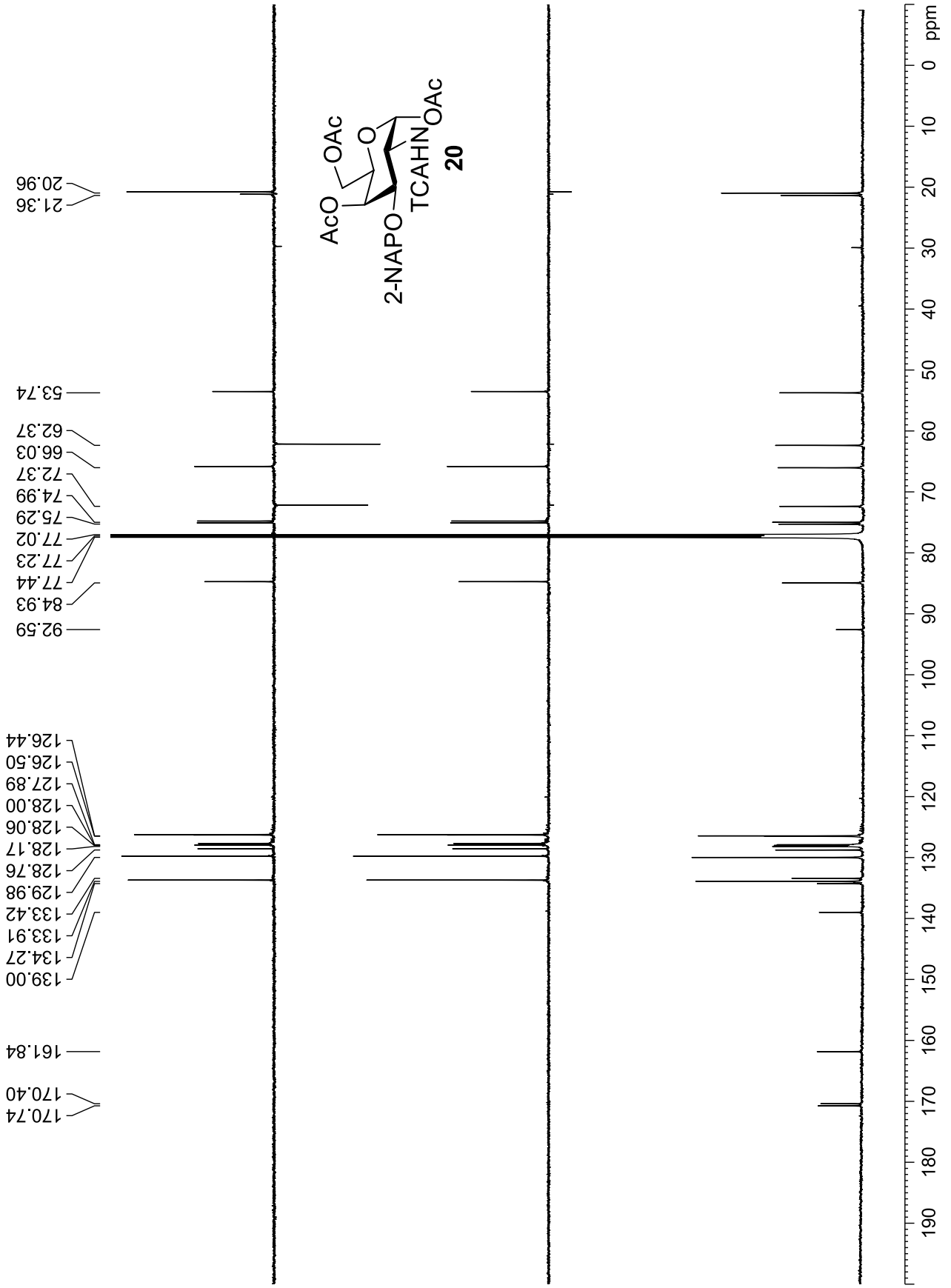
05-Jun-2015
14:34:21
1: TOF MS ES+
9.52e+003

0605_vd-574-1 88 (8.580)



Minimum:	10.00				-1000.0			
Maximum:	100.00		5.0	5.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
562.0416	100.00	562.0414	0.2	0.4	8.5	27.2	0.0	C21 H24 N O9 Na 35Cl3
564.0372	91.74	564.0385	-1.3	-2.3	8.5	28.0	0.0	C21 H24 N O9 Na 35Cl2 37Cl
566.0377	14.30	566.0355	2.2	3.9	8.5	29.8	0.0	C21 H24 N O9 Na 35Cl 37Cl2


7.7844
7.7736
7.7690
7.7595
7.6892
7.4499
7.4444
7.4397
7.4344
7.4187
7.4055
7.3641
7.3501
7.2398
7.0851
7.0720
6.7571
6.7448
5.6120
5.6076
5.2199
5.2028
4.8433
4.8254
4.5237
4.5058
4.3093
4.3045
4.2919
4.2870
4.1863
4.1755
3.9149
3.9040
3.8932
3.6516
3.6344
3.6220
3.6177
3.6048
2.3016
2.0903
2.0675
Current Data Parameters
NAME vd-559 ALL
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160602
Time 18.46 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015711 sec
RG 118.65
DW 64.133 usec
DE 10.47 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W
F2 - Processing parameters
SI 16384
SF 600.1800274 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
AcO
OAc
O
2-NAPO
TCAHN
OAc
20
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
3.06
1.05
4.13
1.06
2.11
0.96
1.00
0.98
1.00
1.02
1.01
0.95
1.04
1.04
1.04
3.13
3.00
3.21


20.96
21.36
53.74
62.37
66.03
72.37
74.99
75.29
77.02
77.23
77.44
84.93
92.59
126.44
126.50
127.89
128.00
128.06
128.17
128.76
129.98
133.42
133.91
134.27
139.00
161.84
170.40
170.74
AcO
OAc
O
TCAHN
OAc
2-NAPO
20
ppm

VD-558 (HR-ESI)

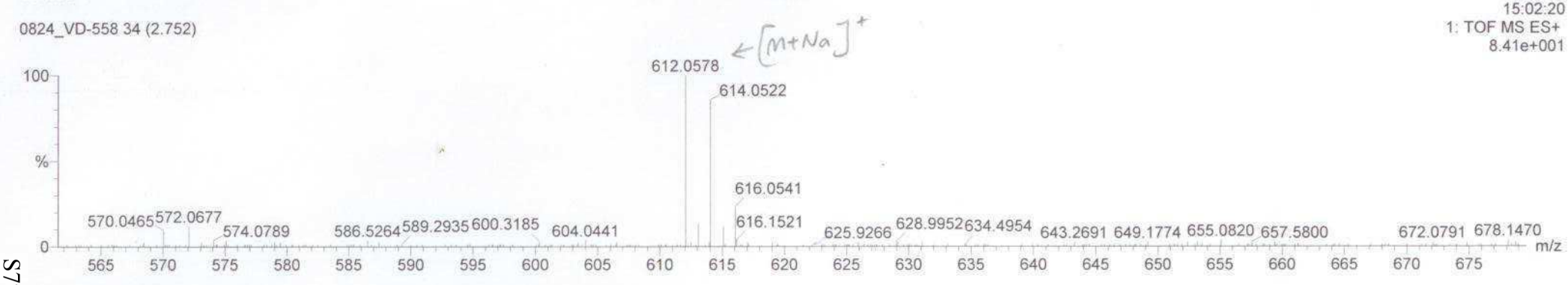
Elemental Composition Report

Multiple Mass Analysis: 3 mass(es) processed
Tolerance = 10.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

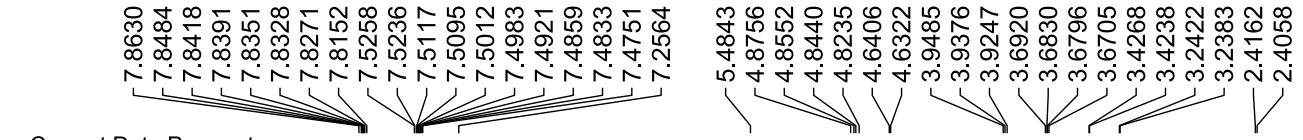
Monoisotopic Mass, Even Electron Ions
48 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 25-25 H: 0-4000 N: 1-1 O: 9-9 Na: 1-1 35Cl: 0-3 37Cl: 0-3

VD-558 KE267 24-Aug-2016 15:02:20

0824_VD-558 34 (2.752) 1: TOF MS ES+ 8.41e+001



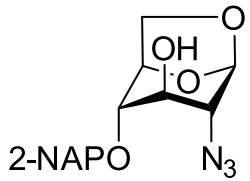
Minimum:	20.00				-1000.0			
Maximum:	100.00		5.0	10.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
612.0578	100.00	612.0571	0.7	1.1	11.5	15.4	0.0	C25 H26 N O9 Na 35Cl3
614.0522	85.54	614.0541	-1.9	-3.1	11.5	12.3	0.0	C25 H26 N O9 Na 35Cl2 37Cl
616.0541	23.20	616.0512	2.9	4.7	11.5	16.8	0.0	C25 H26 N O9 Na 35Cl 37Cl2



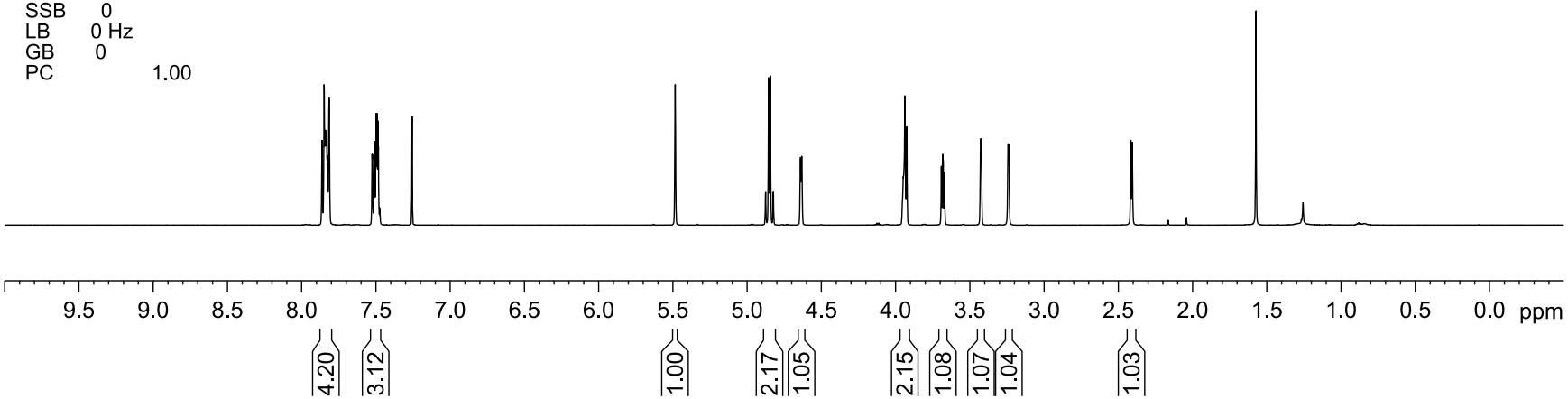
Current Data Parameters
NAME vd-536 OH
EXPNO 1
PROCNO 1

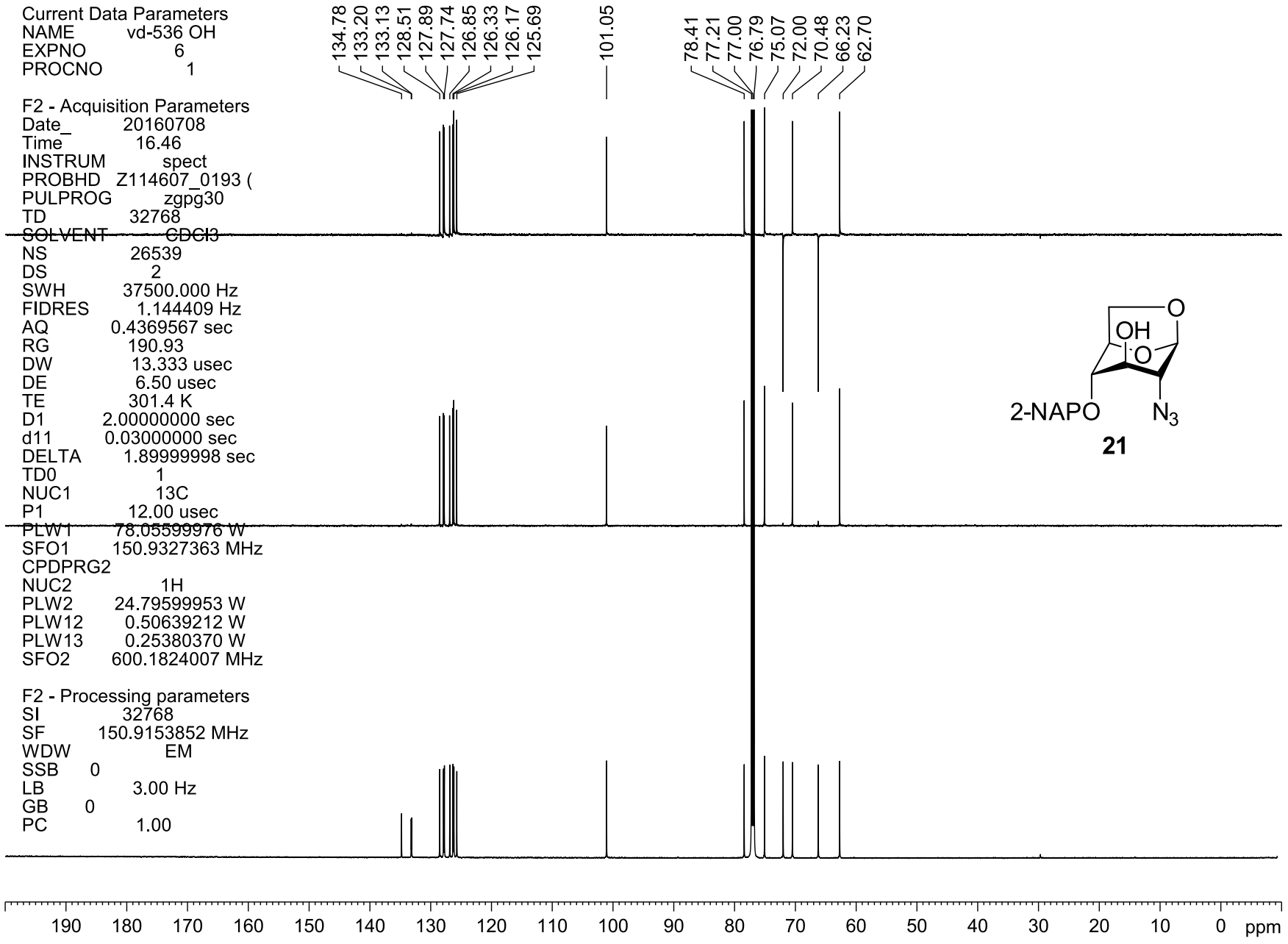
F2 - Acquisition Parameters
Date_ 20160707
Time 17.25 h
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 7796.258 Hz
FIDRES 0.237923 Hz
AQ 2.1015711 sec
RG 118.65
DW 64.133 usec
DE 10.47 usec
TE 300.7 K
D1 2.00000000 sec
TD0 1
SFO1 600.1836011 MHz
NUC1 1H
P1 10.00 usec
PLW1 24.79599953 W

F2 - Processing parameters
SI 16384
SF 600.1800175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



21




Current Data Parameters
NAME vd-536 OH
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160708
Time 16.46
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 26539
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369567 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 301.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
NUC1 13C
P1 12.00 usec
PLW1 78.05599976 W
SFO1 150.9327363 MHz
CPDPRG2
NUC2 1H
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
SFO2 600.1824007 MHz
F2 - Processing parameters
SI 32768
SF 150.9153852 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
134.78
133.20
133.13
128.51
127.89
127.74
126.85
126.33
126.17
125.69
101.05
78.41
77.21
77.00
76.79
75.07
72.00
70.48
66.23
62.70
O
OH
O
2-NAPO
N3
21
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

vd-536 OH (HR-ESI neg)

Elemental Composition Report

Single Mass Analysis

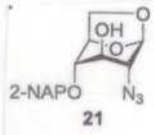
Tolerance = 40.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
19 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-4000 N: 3-3 O: 4-4

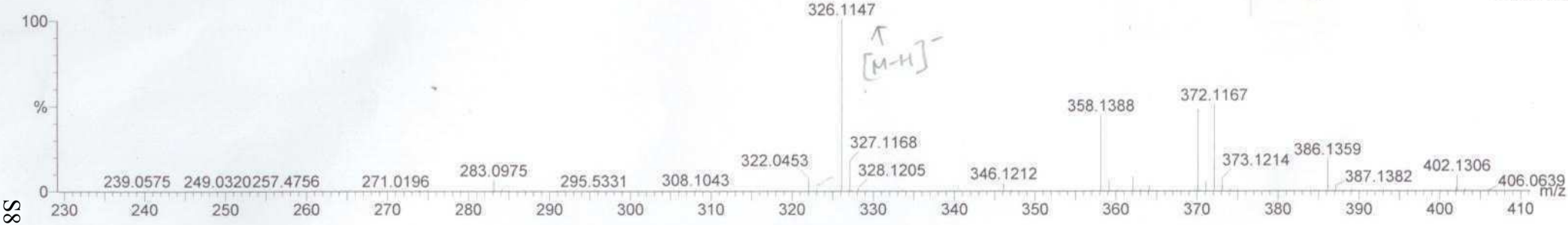
vd-536 OH

KE267

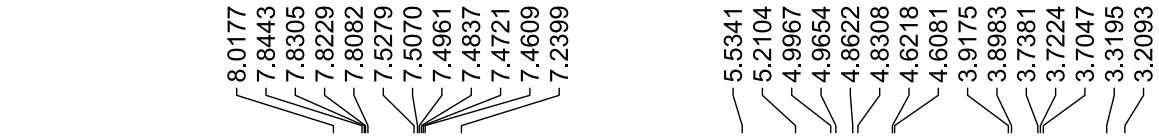
0713_vd-536 OH 13 (1.082) Cm (13-1)



13-Jul-2016
16:17:50
2: TOF MS ES-
6.46e+003



Minimum:				-1000.0			
Maximum:		5.0	40.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
326.1147	326.1141	0.6	1.8	11.5	32.7	0.0	C17 H16 N3 O4

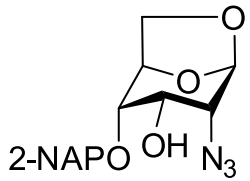


Current Data Parameters
NAME vd-847
EXPNO 1
PROCNO 1

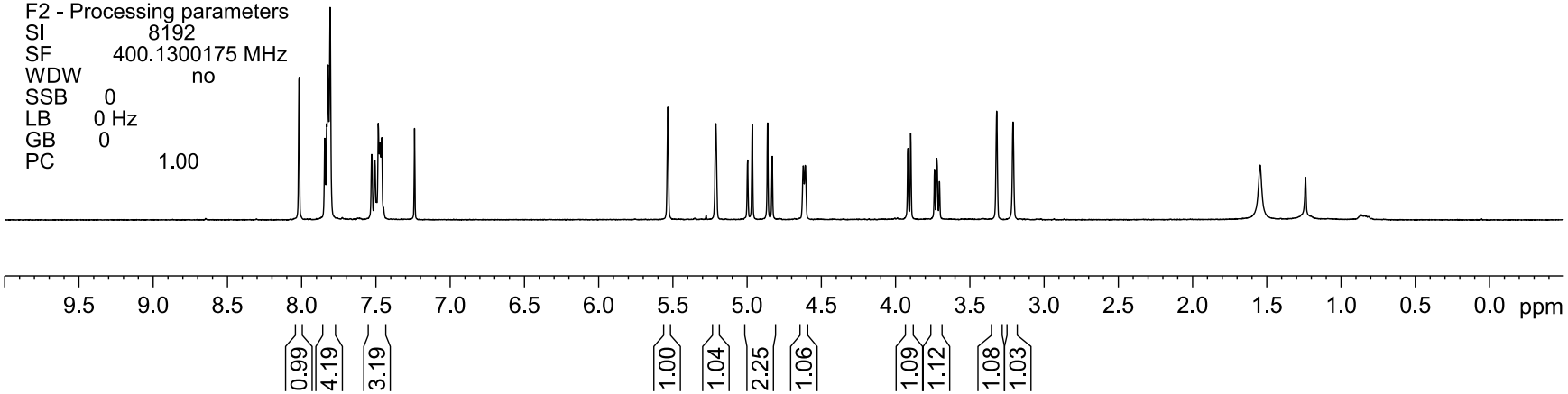
F2 - Acquisition Parameters
Date_ 20160707
Time 18.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 8
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 287
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

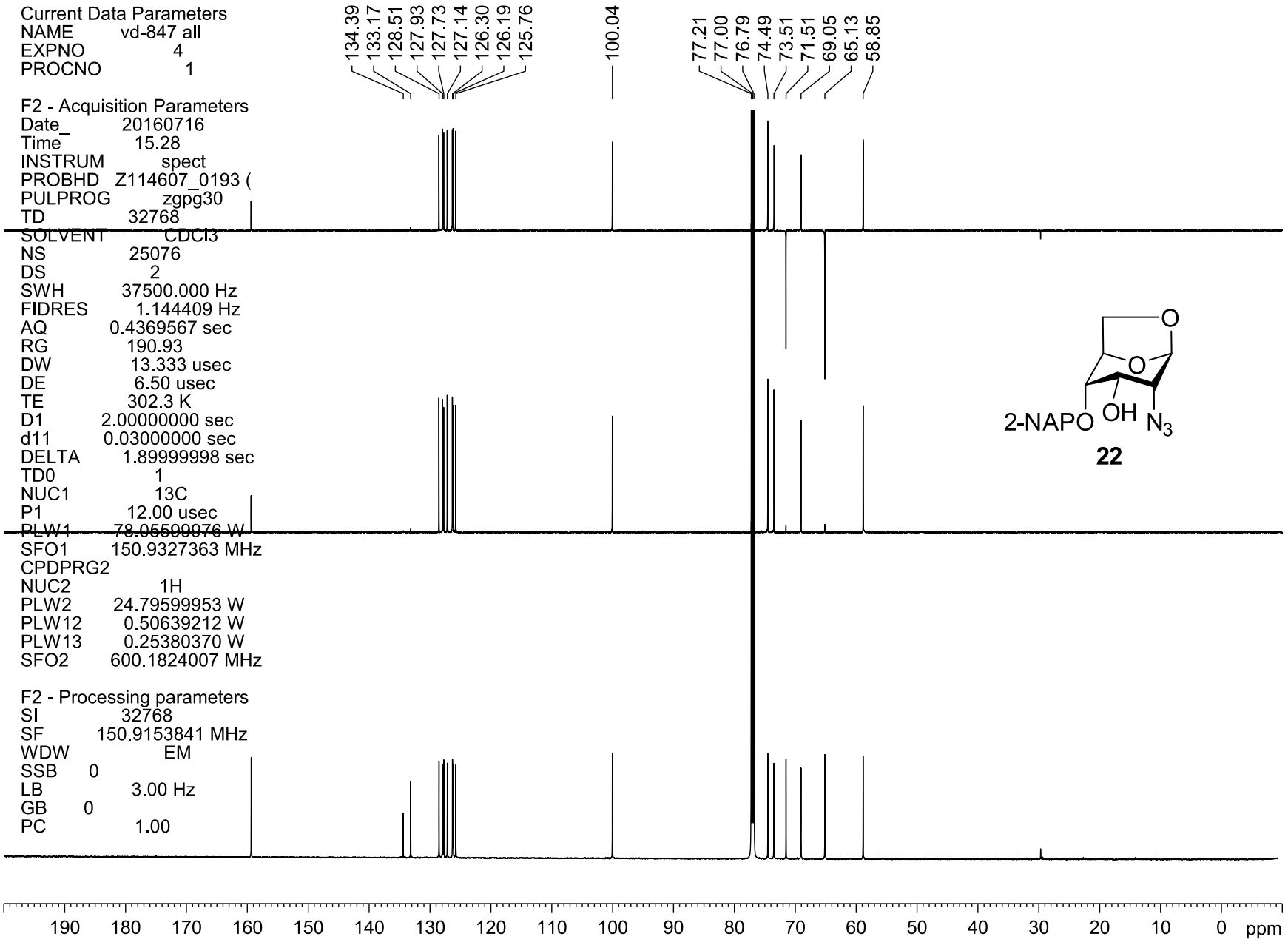
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz

F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



22




Current Data Parameters
NAME vd-847 all
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160716
Time 15.28
INSTRUM spect
PROBHD Z114607_0193 (
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 25076
DS 2
SWH 37500.000 Hz
FIDRES 1.144409 Hz
AQ 0.4369567 sec
RG 190.93
DW 13.333 usec
DE 6.50 usec
TE 302.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
NUC1 13C
P1 12.00 usec
PLW1 78.06599976 W
SFO1 150.9327363 MHz
CPDPRG2
NUC2 1H
PLW2 24.79599953 W
PLW12 0.50639212 W
PLW13 0.25380370 W
SFO2 600.1824007 MHz
F2 - Processing parameters
SI 32768
SF 150.9153841 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
134.39
133.17
128.51
127.93
127.73
127.14
126.30
126.19
125.76
100.04
77.21
77.00
76.79
74.49
73.51
71.51
69.05
65.13
58.85
O
O
2-NAPO
OH
N3
22
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

vd-847 (HR-ESI)

Elemental Composition Report

Single Mass Analysis

Tolerance = 40.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

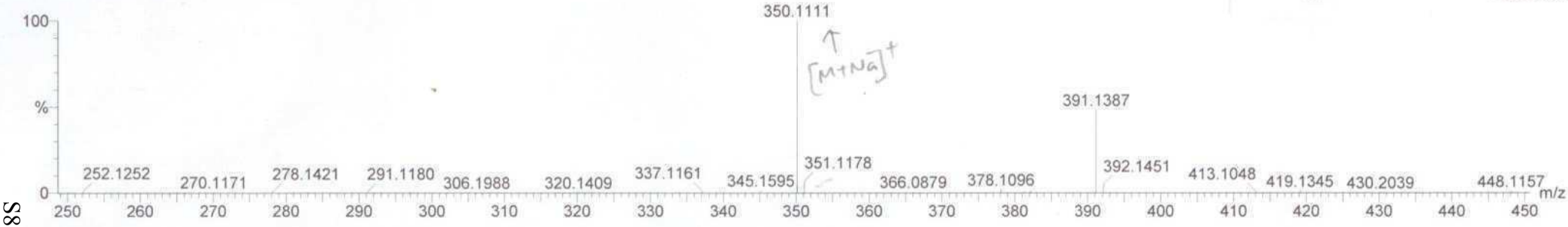
Monoisotopic Mass, Even Electron Ions
19 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-4000 N: 3-3 O: 4-4 Na: 1-1

vd-847

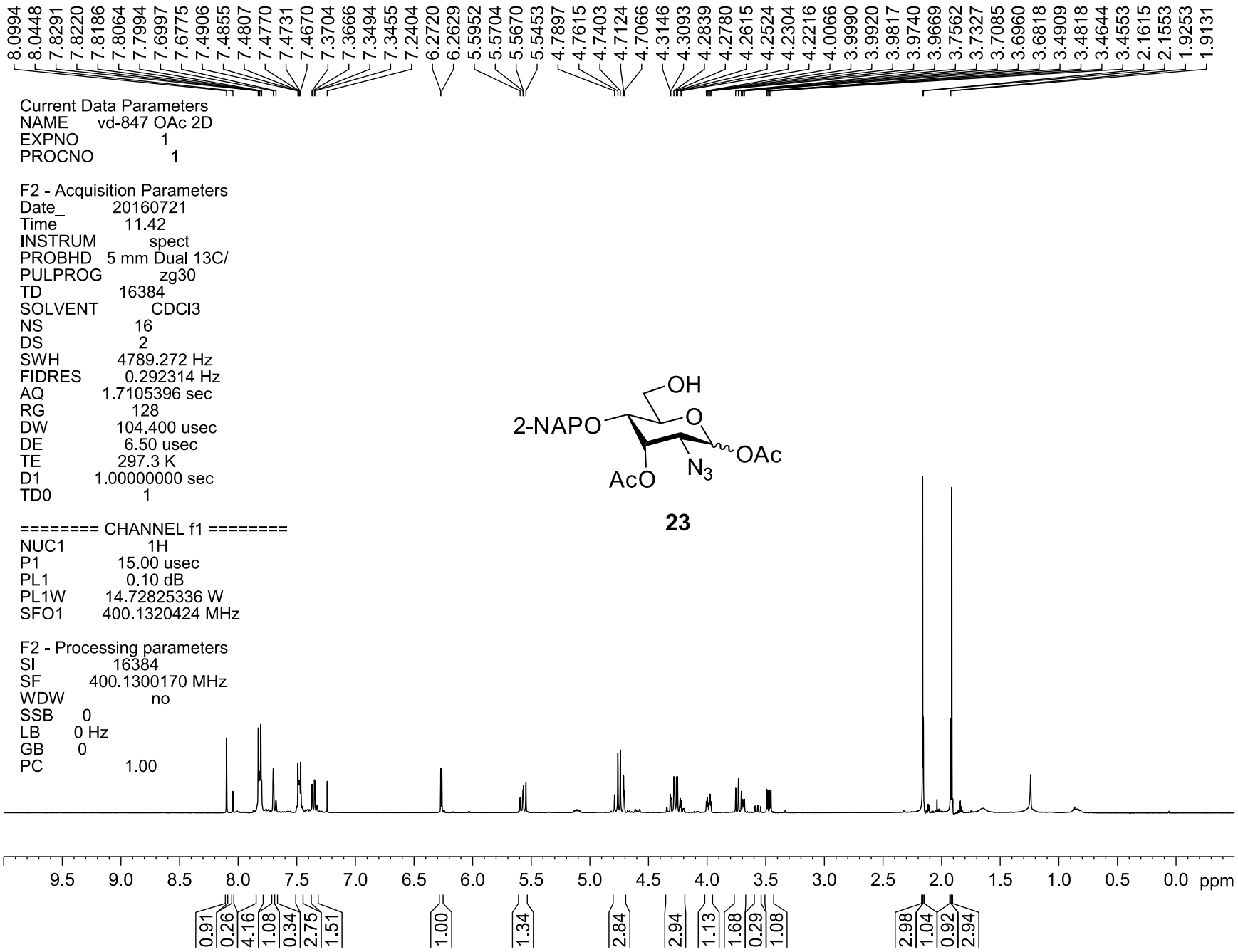
KE267

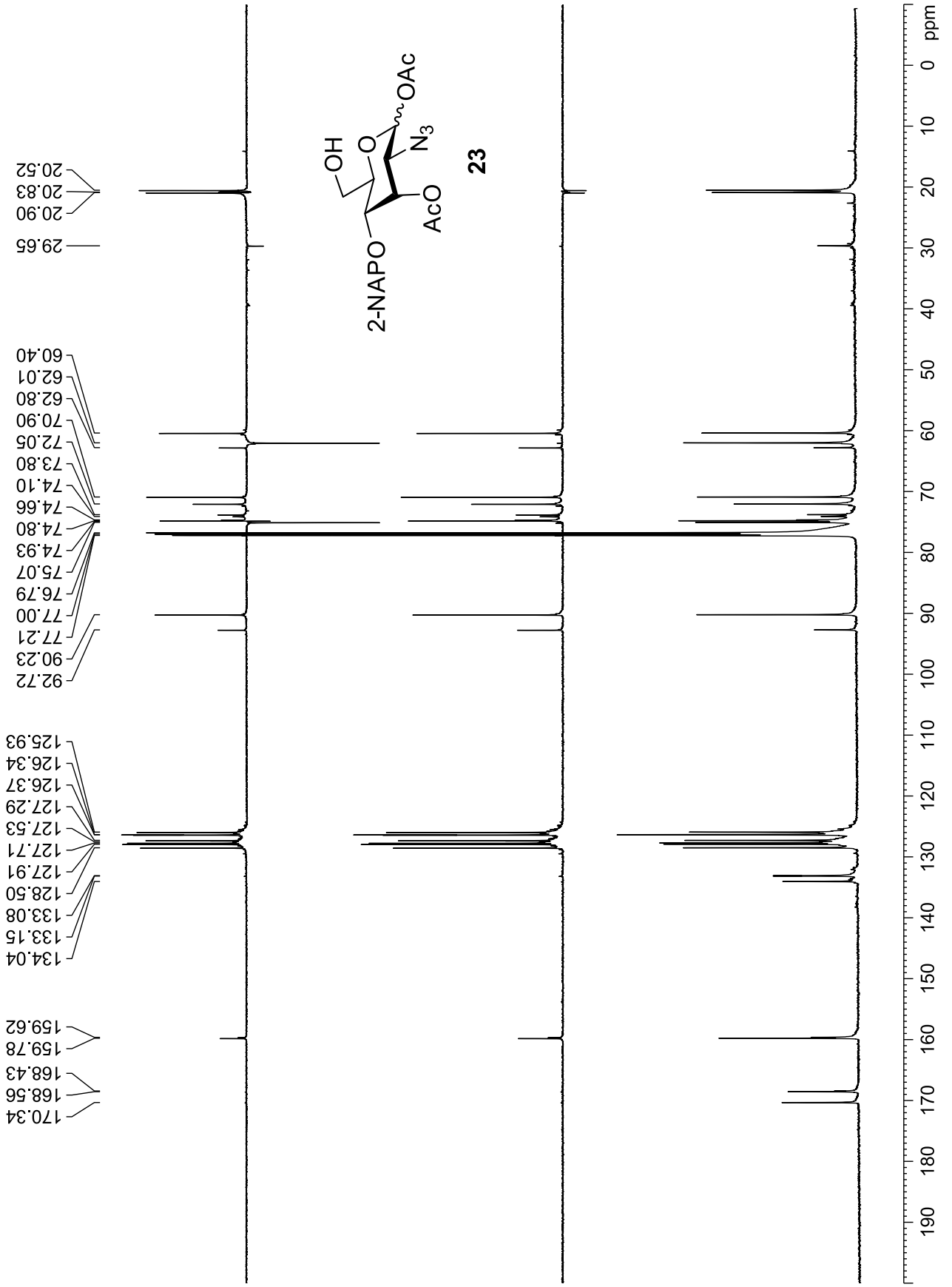
0713_vd-847 13 (1.050) Cm (13-1)

14-Jul-2016
08:34:08
1: TOF MS ES+
2.45e+003



Minimum:				-1000.0			
Maximum:		5.0	40.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
350.1111	350.1117	-0.6	-1.7	10.5	28.4	0.0	C17 H17 N3 O4 Na


8.0994
8.0448
7.8291
7.8220
7.8186
7.8064
7.7994
7.6997
7.6775
7.4906
7.4855
7.4807
7.4770
7.4731
7.4670
7.3704
7.3666
7.3494
7.3455
7.2404
6.2720
6.2629
5.5952
5.5704
5.5670
5.5453
4.7897
4.7615
4.7403
4.7124
4.7066
4.3146
4.3093
4.2839
4.2780
4.2615
4.2524
4.2304
4.2216
4.0066
3.9990
3.9920
3.9817
3.9740
3.9669
3.7562
3.7327
3.7085
3.6960
3.6818
3.4909
3.4818
3.4644
3.4553
2.1615
2.1553
1.9253
1.9131
Current Data Parameters
NAME vd-847 OAc 2D
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160721
Time 11.42
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 128
DW 104.400 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.10 dB
PL1W 14.72825336 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300170 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
OH
O
2-NAPO
OAc
AcO
N3
23
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.91
0.26
4.16
1.08
0.34
2.75
1.51
1.00
1.34
2.84
2.94
1.13
1.68
0.29
1.08
2.98
1.04
0.92
2.94


OAc
OH
O
N3
AcO
2-NAPO
23
20.52
20.83
20.90
29.65
60.40
62.01
62.80
70.90
72.05
73.80
74.10
74.66
74.80
74.93
75.07
76.79
77.00
77.21
90.23
92.72
125.93
126.34
126.37
127.29
127.53
127.71
127.91
128.50
133.08
133.15
134.04
159.62
159.78
168.43
168.56
170.34
ppm

vd-847-OAc (HR-ESI)

Elemental Composition Report

Single Mass Analysis

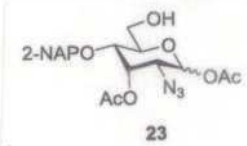
Tolerance = 10.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
19 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 5-1000 H: 0-4000 N: 3-3 O: 7-7 Na: 1-1

vd-847-OAc KE267

0726_vd-847-OAc 10 (0.790) Cm (10-1)

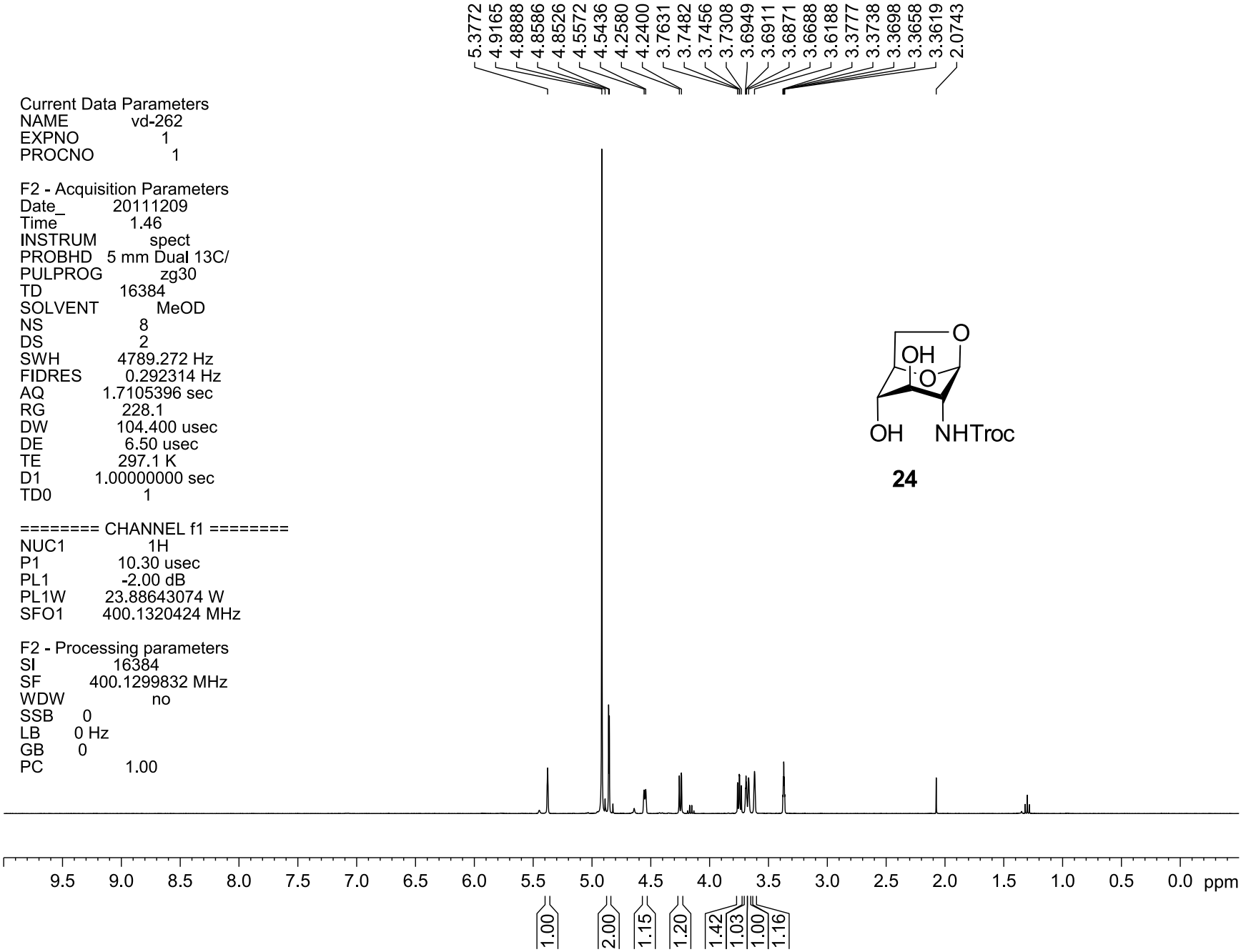
$[M+Na]^+$

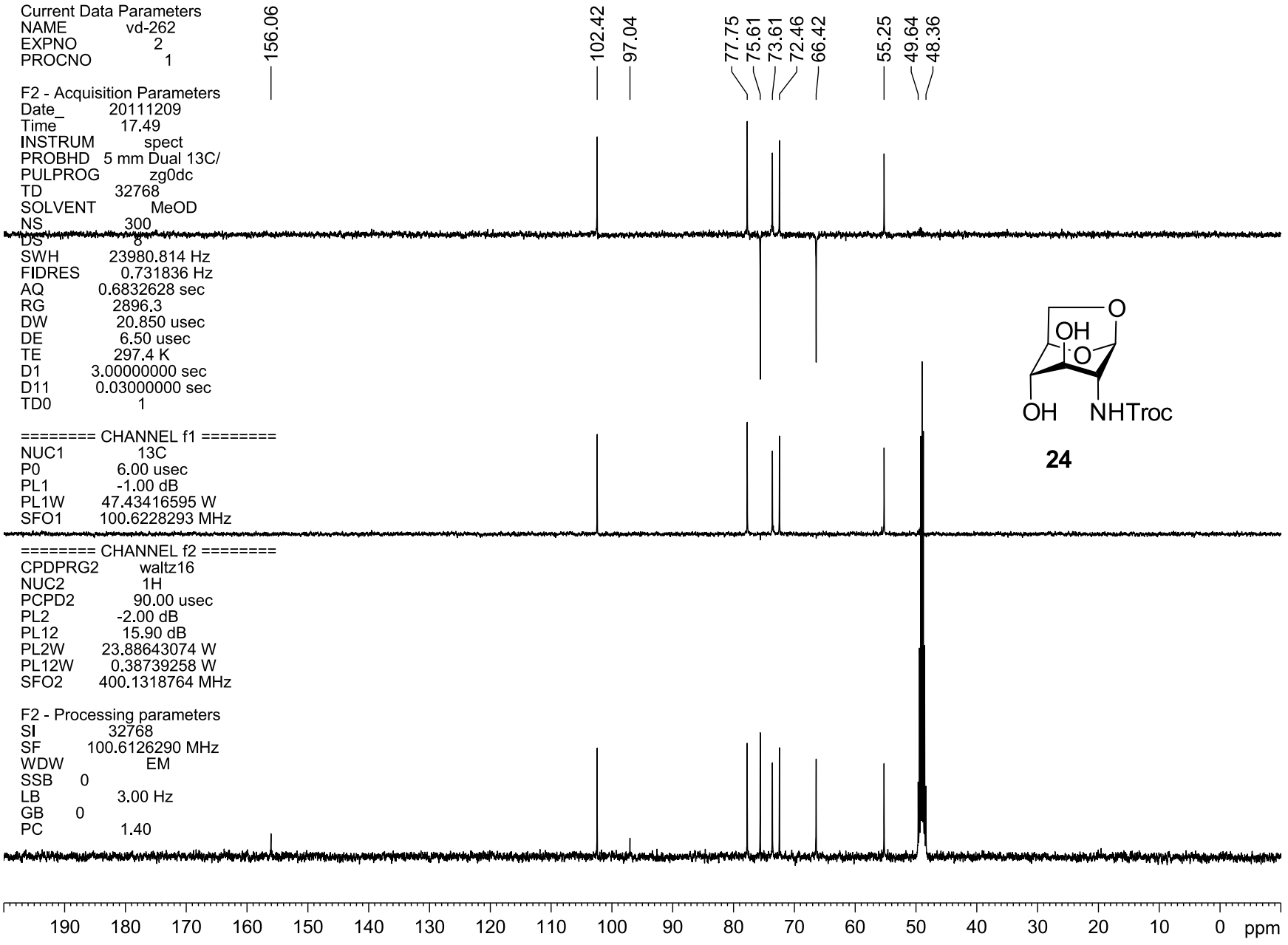


23

26-Jul-2016
17:21:28
1: TOF MS ES+
7.18e+002

Minimum:				-1000.0			
Maximum:		5.0	10.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
452.1428	452.1434	-0.6	-1.3	11.5	19.6	0.0	C21 H23 N3 O7 Na


5.3772
4.9165
4.8888
4.8586
4.8526
4.5572
4.5436
4.2580
4.2400
3.7631
3.7482
3.7456
3.7308
3.6949
3.6911
3.6871
3.6688
3.6188
3.3777
3.3738
3.3698
3.3658
3.3619
2.0743
Current Data Parameters
NAME vd-262
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111209
Time 1.46
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT MeOD
NS 8
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 228.1
DW 104.400 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1299832 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
O
OH
O
OH
NHTroc
24
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
2.00
1.15
1.20
1.42
1.03
1.00
1.16


Current Data Parameters
NAME vd-262
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111209
Time 17.49
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT MeOD
NS 300
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 2896.3
DW 20.850 usec
DE 6.50 usec
TE 297.4 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6126290 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
156.06
102.42
97.04
77.75
75.61
73.61
72.46
66.42
55.25
49.64
48.36
O
OH
O
OH
NHTroc
24
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

vd-262 (HR-ESI neg)

Elemental Composition Report

Single Mass Analysis

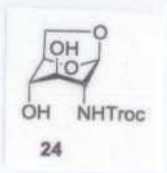
Tolerance = 100.0 PPM / DBE: min = -100.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
11 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-4000 H: 0-4000 N: 1-1 O: 6-6 Cl: 3-3

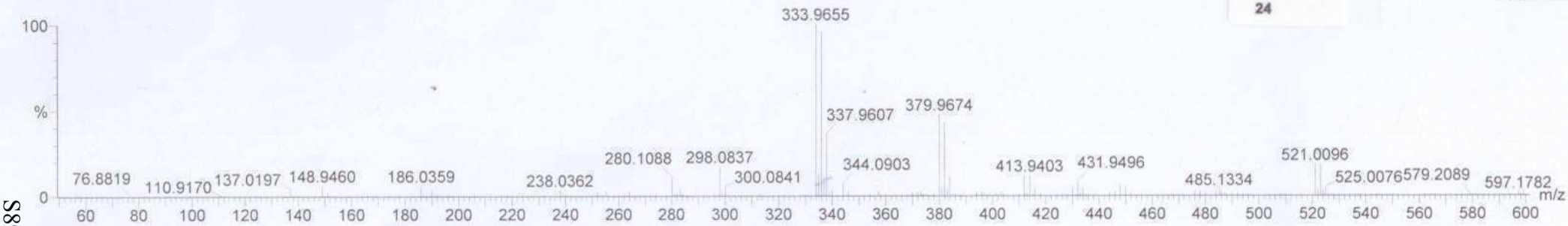
vd-262

0320_vd-262 9 (0.940) Cm (9-101)

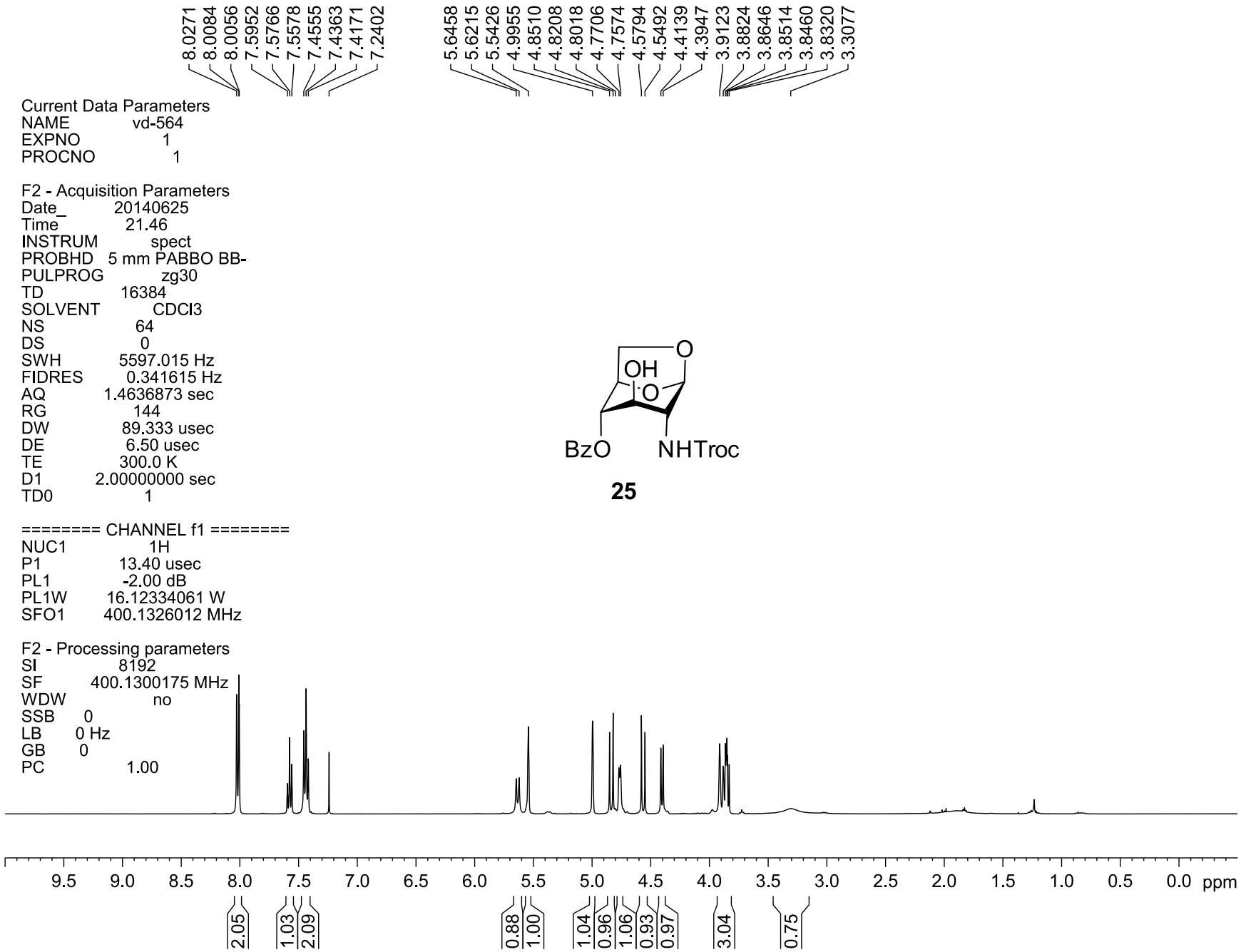
KE267 $[M-H]^-$

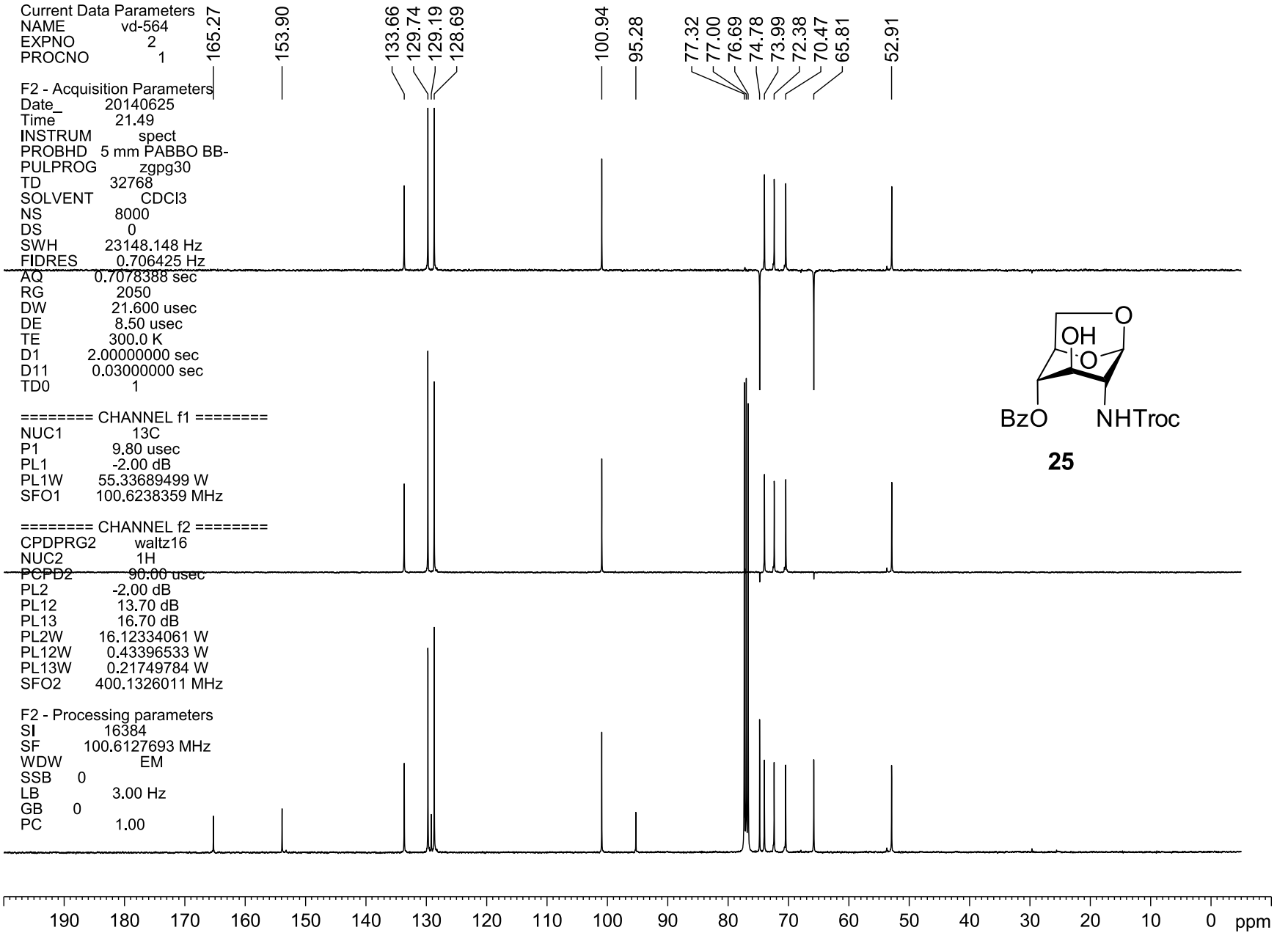


20-Mar-2012
13:09:08
2: TOF MS ES-
1.05e+004



Minimum:				-100.0			
Maximum:		5.0	100.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
333.9655	333.9652	0.3	0.9	3.5	57.2	0.0	C9 H11 N O6 Cl3


8.0271
8.0084
8.0056
7.5952
7.5766
7.5578
7.4555
7.4363
7.4171
7.2402
5.6458
5.6215
5.5426
4.9955
4.8510
4.8208
4.8018
4.7706
4.7574
4.5794
4.5492
4.4139
4.3947
3.9123
3.8824
3.8646
3.8514
3.8460
3.8320
3.3077
Current Data Parameters
NAME vd-564
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140625
Time 21.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 144
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
OH
O
O
BzO
NHTroc
25
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.05
1.03
2.09
0.88
1.00
1.04
0.96
1.06
0.93
0.97
3.04
0.75


Current Data Parameters
NAME vd-564
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140625
Time 21.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 8000
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238359 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127693 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
165.27
153.90
133.66
129.74
129.19
128.69
100.94
95.28
77.32
77.00
76.69
74.78
73.99
72.38
70.47
65.81
52.91
OH
O
O
BzO
NHTroc
25
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

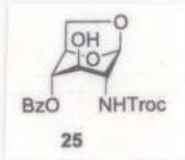
vd-564 (HR-ESI neg)

Elemental Composition Report

Multiple Mass Analysis: 3 mass(es) processed
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

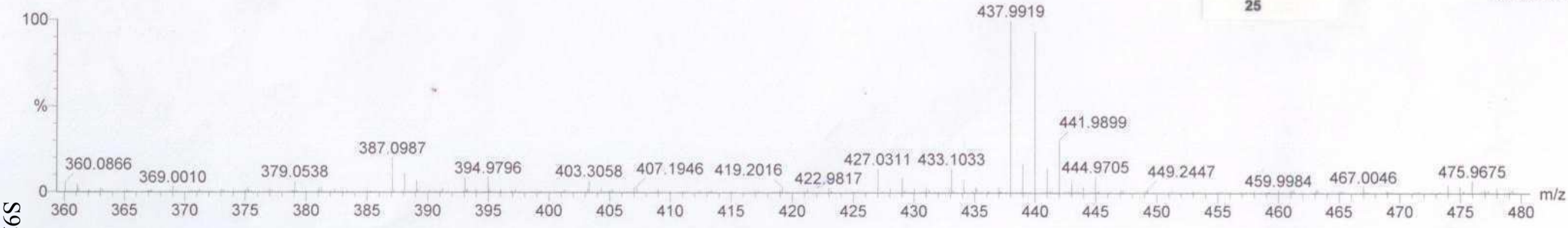
Monoisotopic Mass, Even Electron Ions
95 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-16 H: 0-1000 N: 1-1 O: 7-7 35Cl: 0-3 37Cl: 0-3

vd-564 KE267 [M-H]⁻

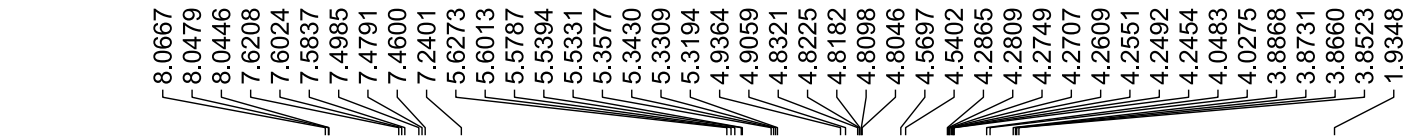


30-Sep-2013
13:28:51
2: TOF MS ES-
2.20e+003

0930_vd-564 16 (1.561)

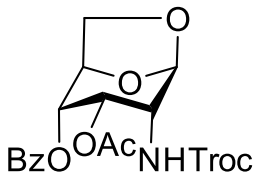


Minimum:	30.00				-1.5			
Maximum:	100.00		5.0	10.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
437.9919	100.00	437.9914	0.5	1.1	8.5	49.8	0.0	C16 H15 N O7 35Cl3
439.9887	93.81	439.9885	0.2	0.5	8.5	44.9	0.0	C16 H15 N O7 35Cl2 37Cl
441.9899	30.56	441.9855	4.4	10.0	8.5	42.6	0.0	C16 H15 N O7 35Cl 37Cl2

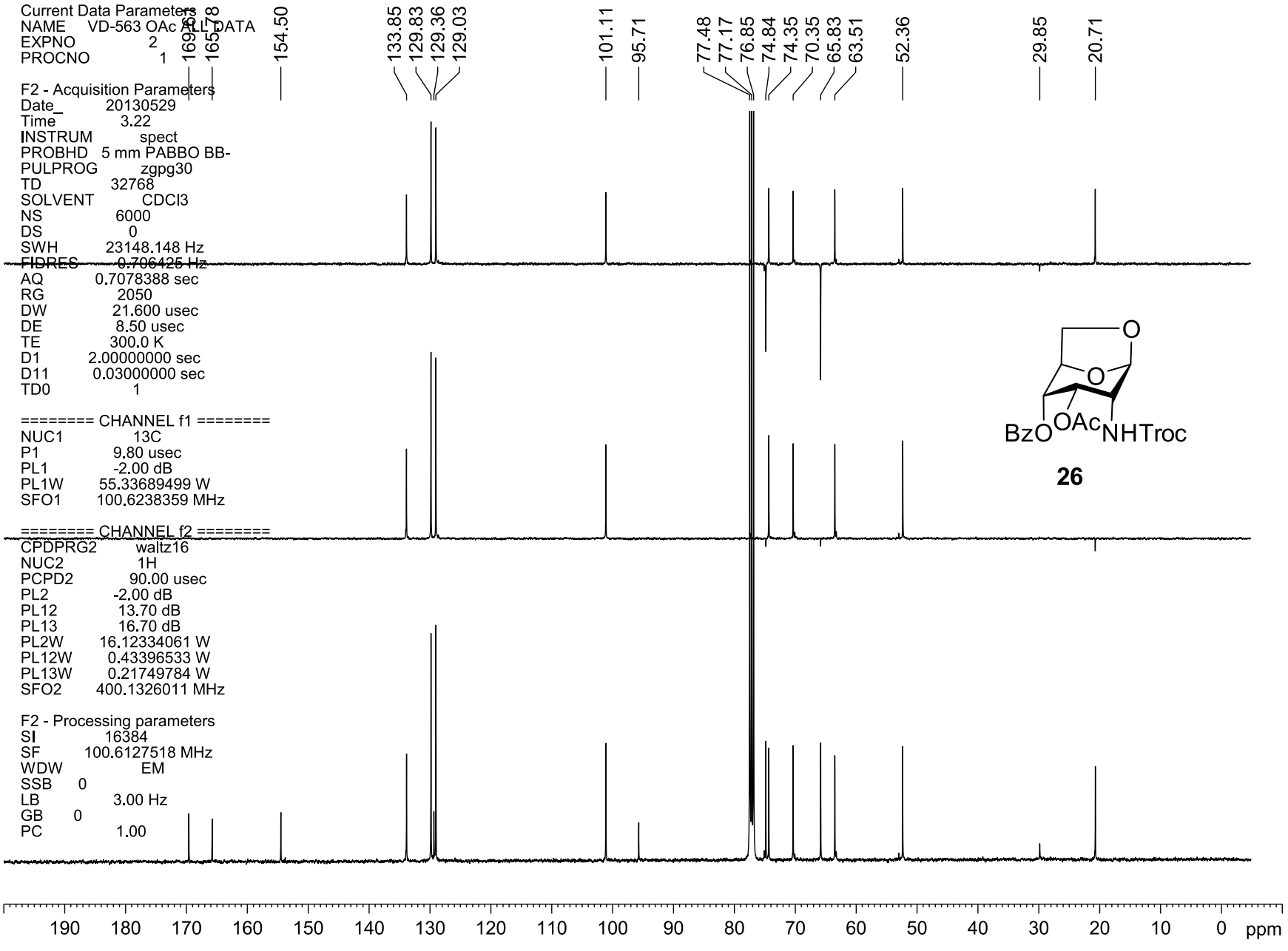


NAME VD-563 OAc ALL DATA
EXPNO 1
PROCNO 1
Date_ 20130528
Time 22.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 24
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 181
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



26


Current Data Parameters
NAME VD-563 OAc ALL DATA
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130529
Time 3.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6000
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238359 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127518 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
169.83
165.78
154.50
133.85
129.83
129.36
129.03
101.11
95.71
77.48
77.17
76.85
74.84
74.35
70.35
65.83
63.51
52.36
29.85
20.71
BzO
OAc
NHTroc
26
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

vd-563 OAc (HR-ESI)

Elemental Composition Report

Multiple Mass Analysis: 2 mass(es) processed
Tolerance = 5.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

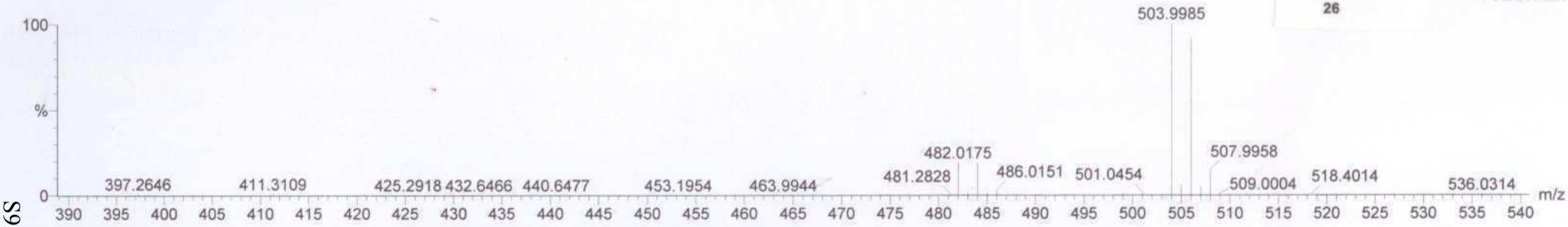
Monoisotopic Mass, Odd and Even Electron Ions
112 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 18-1000 H: 0-1000 N: 1-1 O: 8-8 Na: 1-1 35Cl: 0-3 37Cl: 0-3

vd-563 OAc KE267

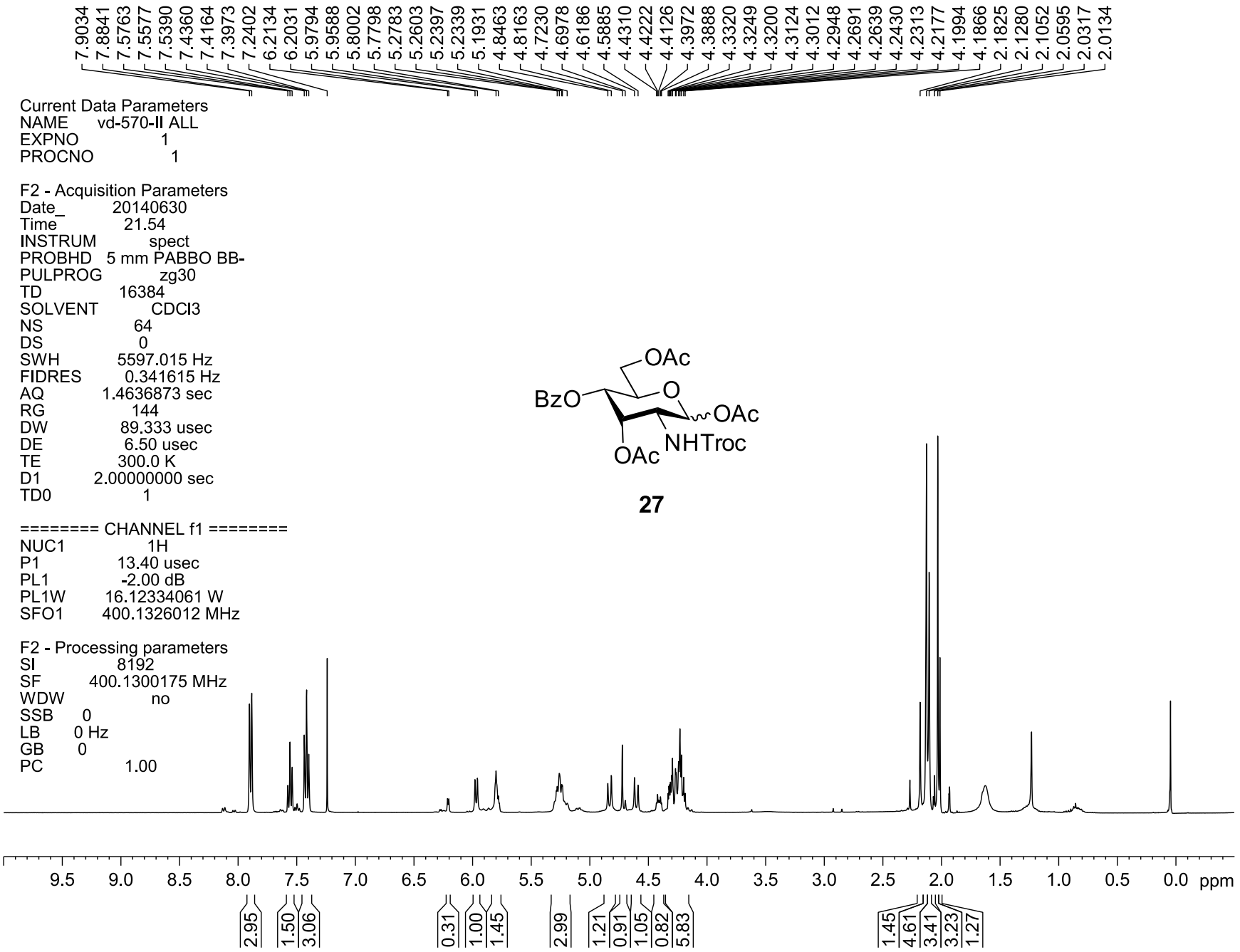
0605_vd-563 OAc 2 (0.195) Cm (2-1x10.000)

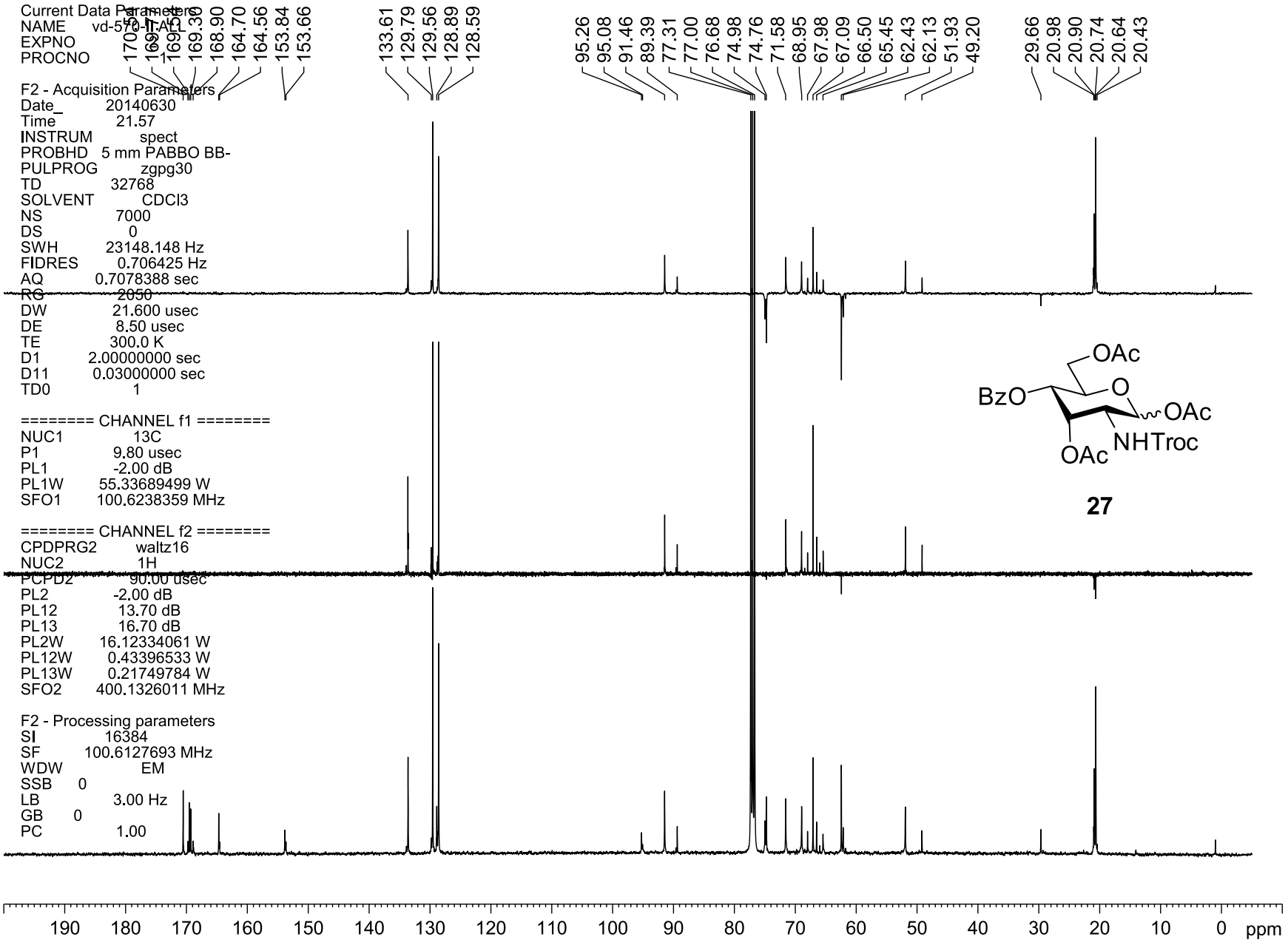
$[M+Na]^+$

05-Jun-2015
14:23:08
1: TOF MS ES+
9.90e+003



Minimum:	20.00				-1000.0			
Maximum:	100.00		5.0	5.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
503.9985	100.00	503.9996	-1.1	-2.2	8.5	33.4	0.0	C18 H18 N O8 Na 35Cl3
505.9955	91.25	505.9966	-1.1	-2.2	8.5	30.9	0.0	C18 H18 N O8 Na 35Cl2 37Cl


7.9034
7.8841
7.5763
7.5577
7.5390
7.4360
7.4164
7.3973
7.2402
6.2134
6.2031
5.9794
5.9588
5.8002
5.7798
5.2783
5.2603
5.2397
5.2339
5.1931
4.8463
4.8163
4.7230
4.6978
4.6186
4.5885
4.4310
4.4222
4.4126
4.3972
4.3888
4.3320
4.3249
4.3200
4.3124
4.3012
4.2948
4.2691
4.2639
4.2430
4.2313
4.2177
4.1994
4.1866
2.1825
2.1280
2.1052
2.0595
2.0317
2.0134
Current Data Parameters
NAME vd-570-II ALL
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140630
Time 21.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 144
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
OAc
O
BzO
OAc
OAc
NHTroc
27
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.95
1.50
3.06
0.31
1.00
1.45
2.99
1.21
0.91
1.05
0.82
5.83
1.45
4.61
3.41
3.23
1.27


Current Data Parameters
NAME vd-570-ITALL
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140630
Time 21.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 7000
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238359 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127693 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
170.03
169.70
169.61
169.30
168.90
164.70
164.56
153.84
153.66
133.61
129.79
129.56
128.89
128.59
95.26
95.08
91.46
89.39
77.31
77.00
76.68
74.98
74.76
71.58
68.95
67.98
67.09
66.50
65.45
62.43
62.13
51.93
49.20
29.66
20.98
20.90
20.74
20.64
20.43
OAc
O
BzO
OAc
OAc
NHTroc
27
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

vd-570-II (HR-ESI)

Elemental Composition Report

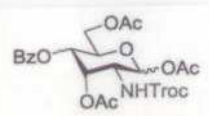
Multiple Mass Analysis: 3 mass(es) processed
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
48 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 22-22 H: 0-1000 N: 1-1 O: 11-11 Na: 1-1 35Cl: 0-3 37Cl: 0-3

vd-570-II KE267

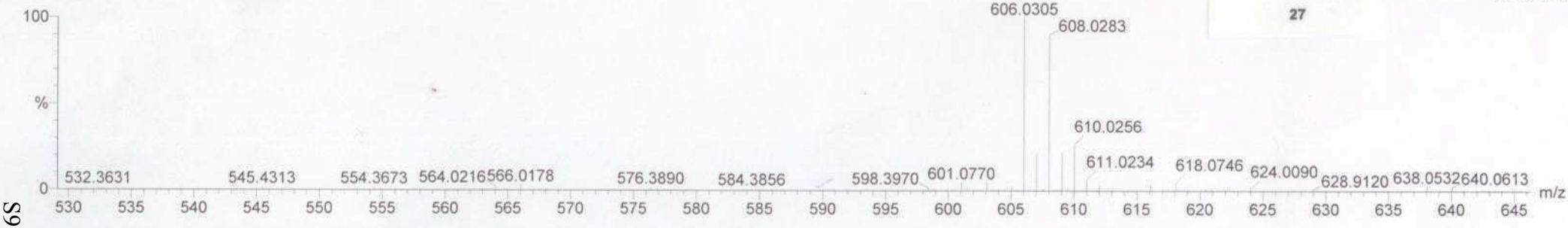
0930_vd-570-II 2 (0.190)

$[M+Na]^+$



27

30-Sep-2013
15:10:01
1: TOF MS ES+
9.50e+003



Minimum:	24.00				-1.5			
Maximum:	100.00		5.0	10.0	1000.0			
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
606.0305	100.00	606.0313	-0.8	-1.3	9.5	55.4	0.0	C22 H24 N O11 Na 35Cl3
608.0283	90.29	608.0283	0.0	0.0	9.5	41.6	0.0	C22 H24 N O11 Na 35Cl2 37Cl
610.0256	26.73	610.0254	0.2	0.3	9.5	24.0	0.0	C22 H24 N O11 Na 35Cl 37Cl2