Evolution of a Strategy for the Enantioselective Total Synthesis of (+)-Psiguadial B

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Supporting Information

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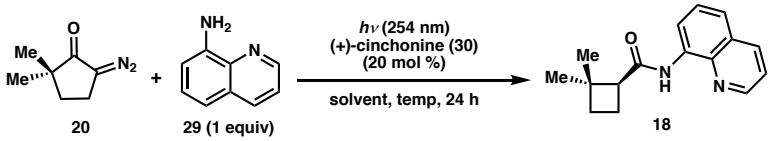
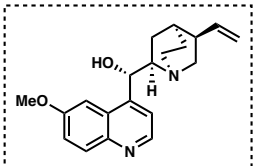
Table S1: Optimization of the Wolff rearrangement for amide 18



20 29 (1 equiv) 18

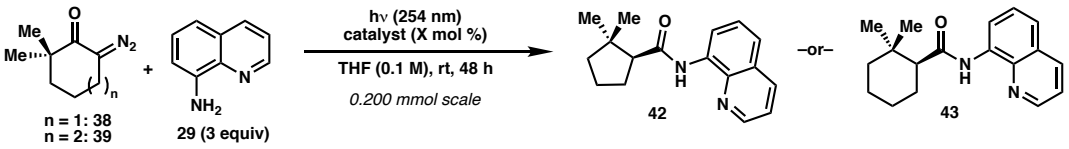
entry	solvent	temp °C	vessel[a]	atm	% yield[b]	% ee[c]
1	THF	25	quartz	N_2	63	79
2	DMF	25	quartz	N_2	76	50
3	CH_2Cl_2	25	quartz	N_2	62	47
4	toluene	25	quartz	N_2	65	59
5	dioxane	25	quartz	N_2	69	79
6	MeCN	25	quartz	N_2	43	74
7	THF	0	quartz	N_2	28	79
8	THF	25	quartz	air	18	75
9	THF	25	quartz	CO	33	69
10	THF	25	vycor	N_2	29	79
11	THF	25	pyrex	N_2	20	75



(+)-cinchonine (30)

[a] See reference 12 in text of article for discussion of photodecarbonylation.
[b] Determined by ^{1}H NMR via integration relative to an added internal standard.
[c] Determined by SFC using a chiral stationary phase.

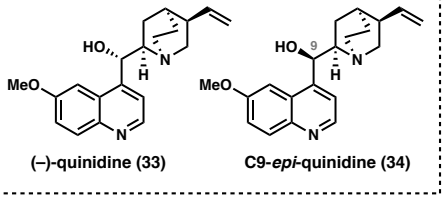
Table S2: Catalyst loading screen for amides 38 and 39



n = 1: 38
n = 2: 39

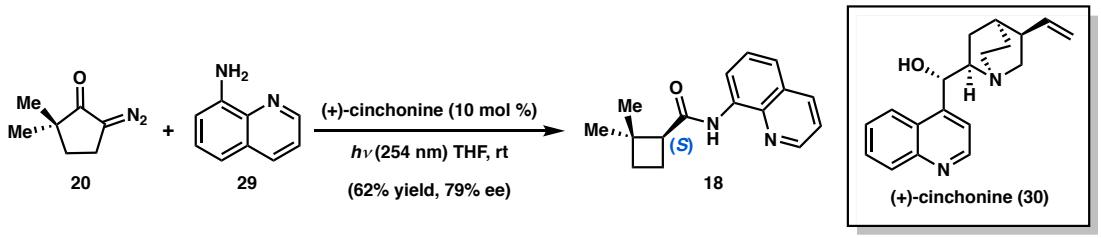
29 (3 equiv) 42 43

entry	diazo.	catalyst (mol %)	product	% isolated yield	% ee[a]
1	38	34 (50)	42	77	71
2	38	34 (30)	42	81	69
3	38	34 (20)	42	76	67
4	38	34 (10)	42	81	63
5	39	33 (50)	43	59	71
6	39	33 (30)	43	71	66
7	39	33 (20)	43	61	62
8	39	33 (10)	43	61	55

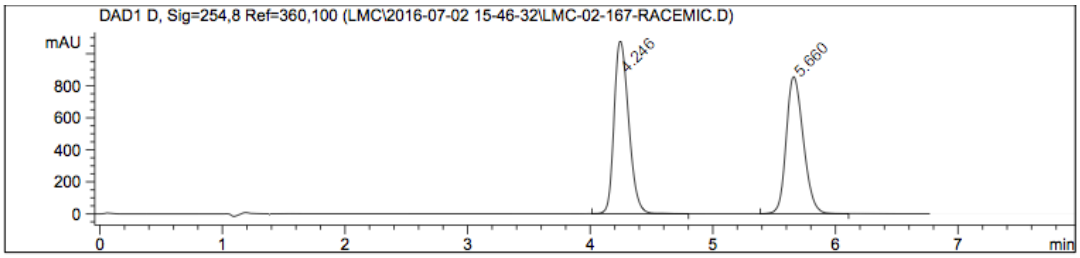


(–)-quinidine (33) C9-*epi*-quinidine (34)

[a] Determined by SFC using a chiral stationary phase.

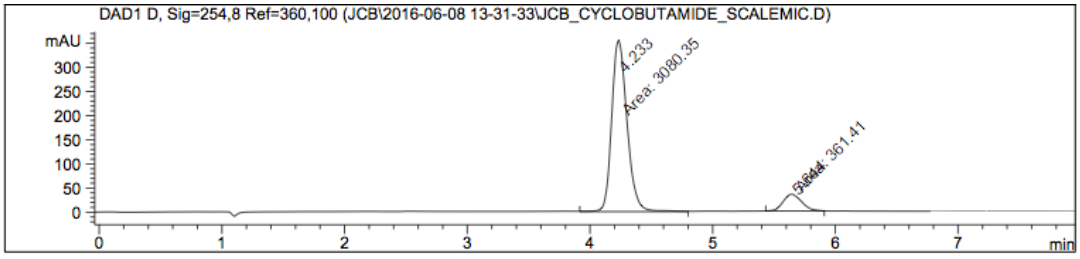


SFC data for racemic 18:



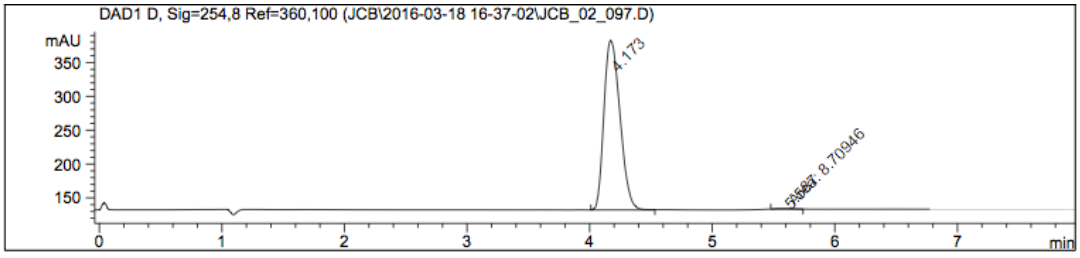
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.246	BB	0.1286	8934.03320	1078.74268	51.8271
2	5.660	BB	0.1532	8304.11426	855.84570	48.1729

Enantioenriched 18 isolated directly from reaction:

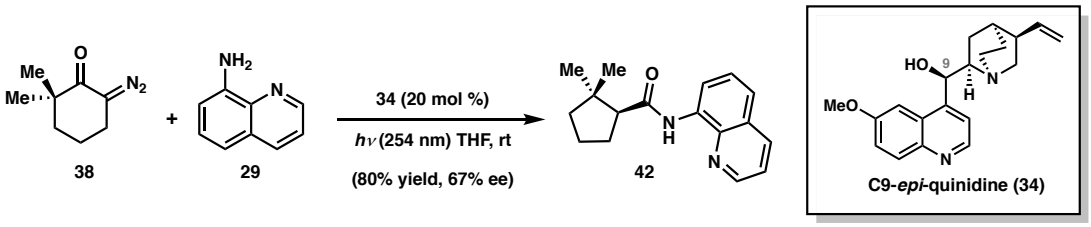


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.233	MM	0.1444	3080.35034	355.58780	89.4993
2	5.644	MM	0.1739	361.41000	34.64573	10.5007

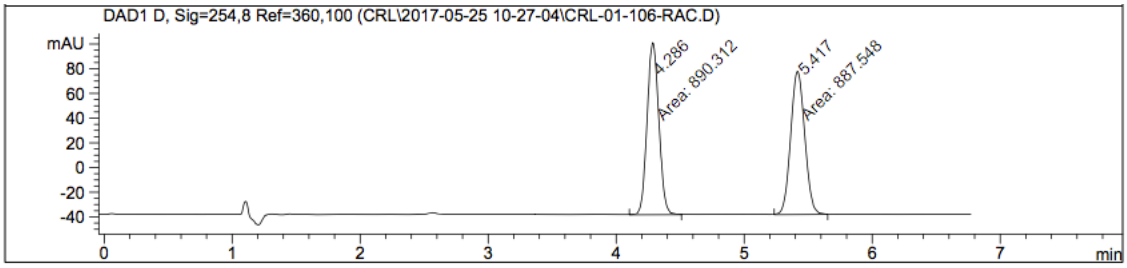
Enantiopure 18 after a single recrystallization:



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.173	BB	0.1464	2285.16455	250.58836	99.6203
2	5.587	MM	0.1518	8.70946	9.55969e-1	0.3797

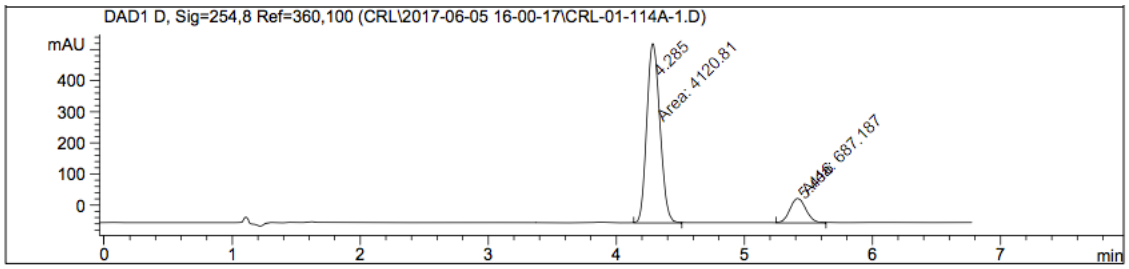


SFC data for racemic 42:



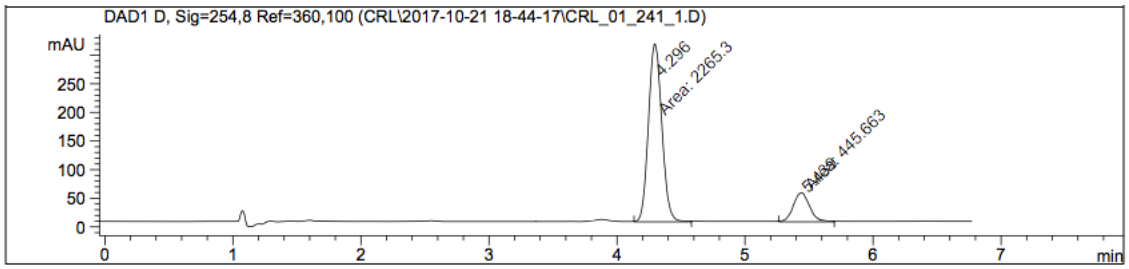
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %
1	4.286	MM	0.1063	890.31165	50.0777
2	5.417	MM	0.1274	887.54810	49.9223

Enantioenriched 42:

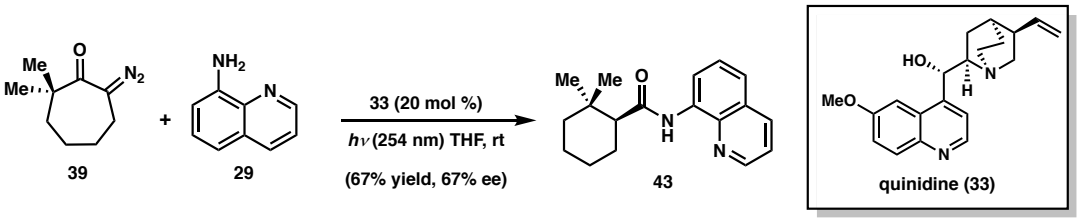


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %
1	4.285	MM	0.1189	4120.80518	85.7074
2	5.416	MM	0.1449	687.18732	14.2926

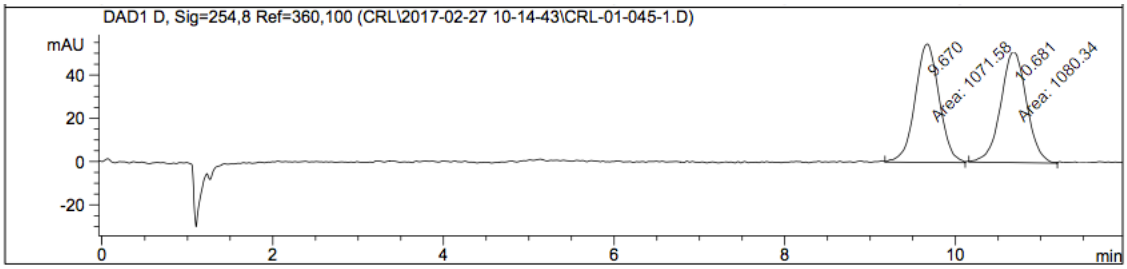
Enantioenriched 42 using 20 mol % catalyst loading:



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %
1	4.296	MM	0.1212	2265.30225	83.5607
2	5.439	MM	0.1461	445.66309	16.4393

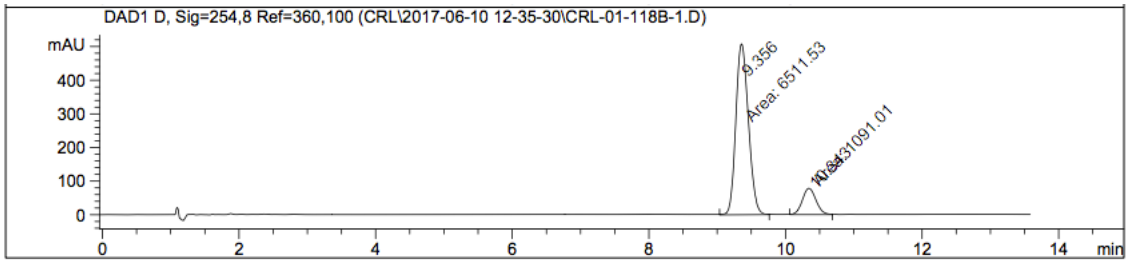


SFC data for racemic 43 (note – 12 minute run):



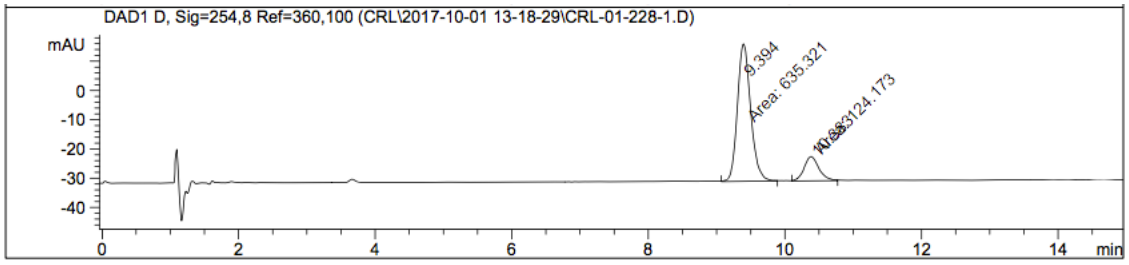
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %
1	9.670	MM	0.3268	1071.58496	49.7965
2	10.681	MM	0.3511	1080.34424	50.2035

Enantioenriched 43:

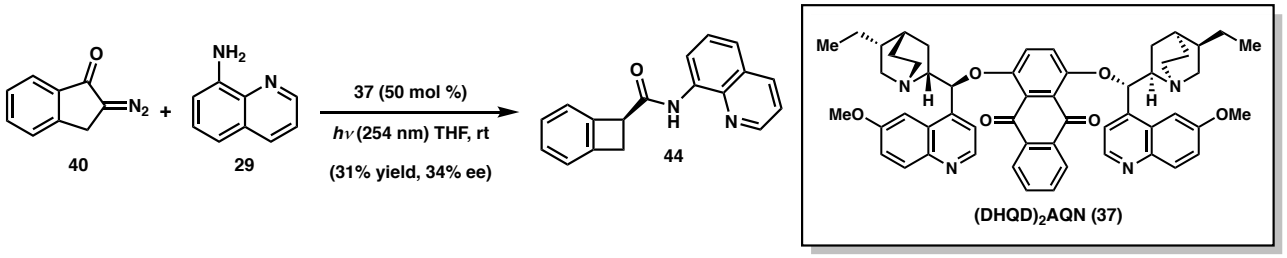


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %
1	9.356	MM	0.2135	6511.52734	85.6494
2	10.343	MM	0.2316	1091.00696	14.3506

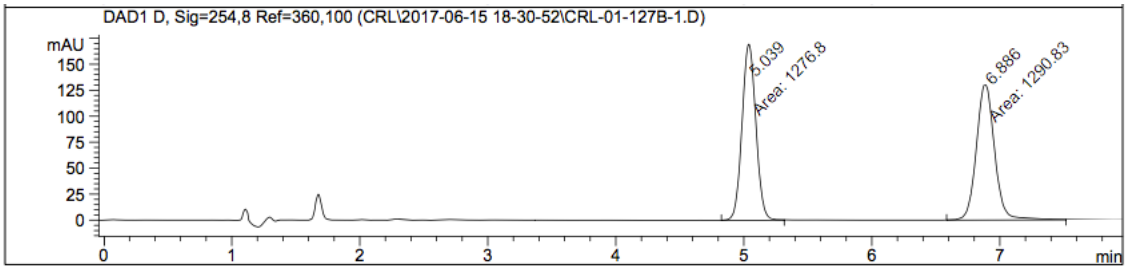
Enatioenriched 43 using 20 mol % catalyst loading:



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %
1	9.394	MM	0.2252	635.32117	83.6505
2	10.383	MM	0.2495	124.17338	16.3495

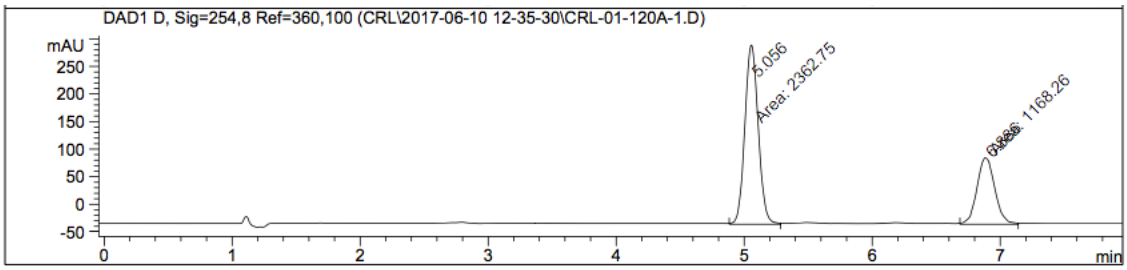


SFC data for racemic 44:

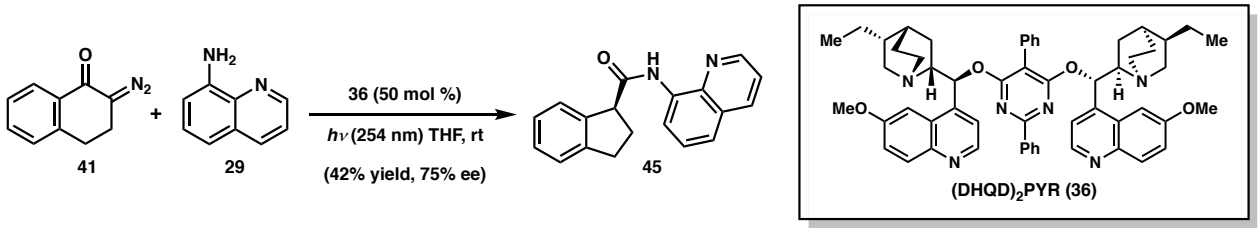


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %
1	5.039	MM	0.1254	1276.80469	49.7268
2	6.886	MM	0.1641	1290.83447	50.2732

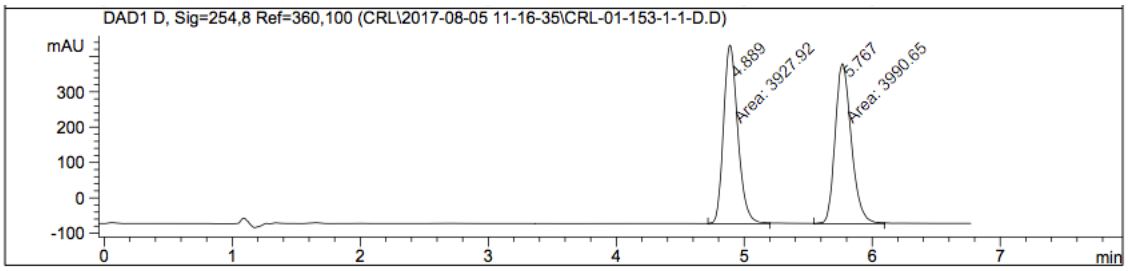
Enantioenriched 44:



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %
1	5.056	MM	0.1210	2362.74805	66.9143
2	6.886	MM	0.1605	1168.25940	33.0857

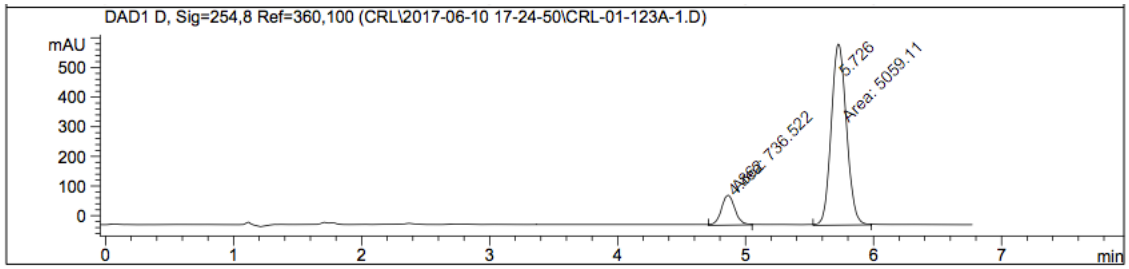


SFC data for racemic 45:



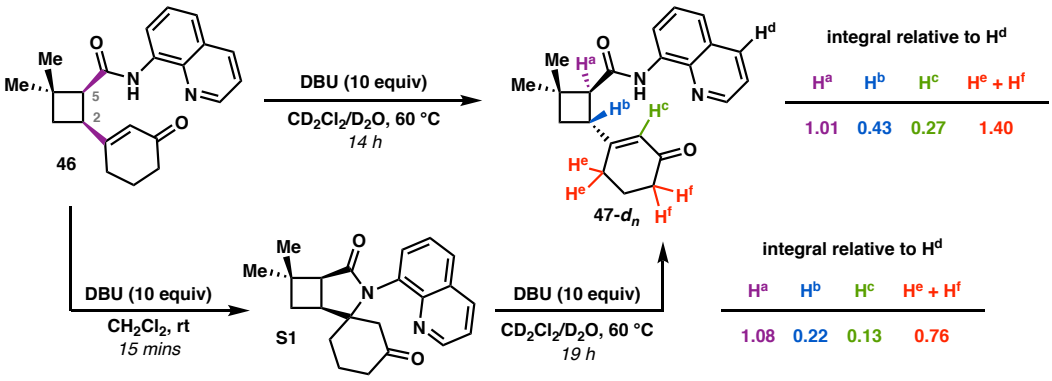
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %
1	4.889	MM	0.1294	3927.92065	49.6039
2	5.767	MM	0.1468	3990.65186	50.3961

Enatioenriched 45:

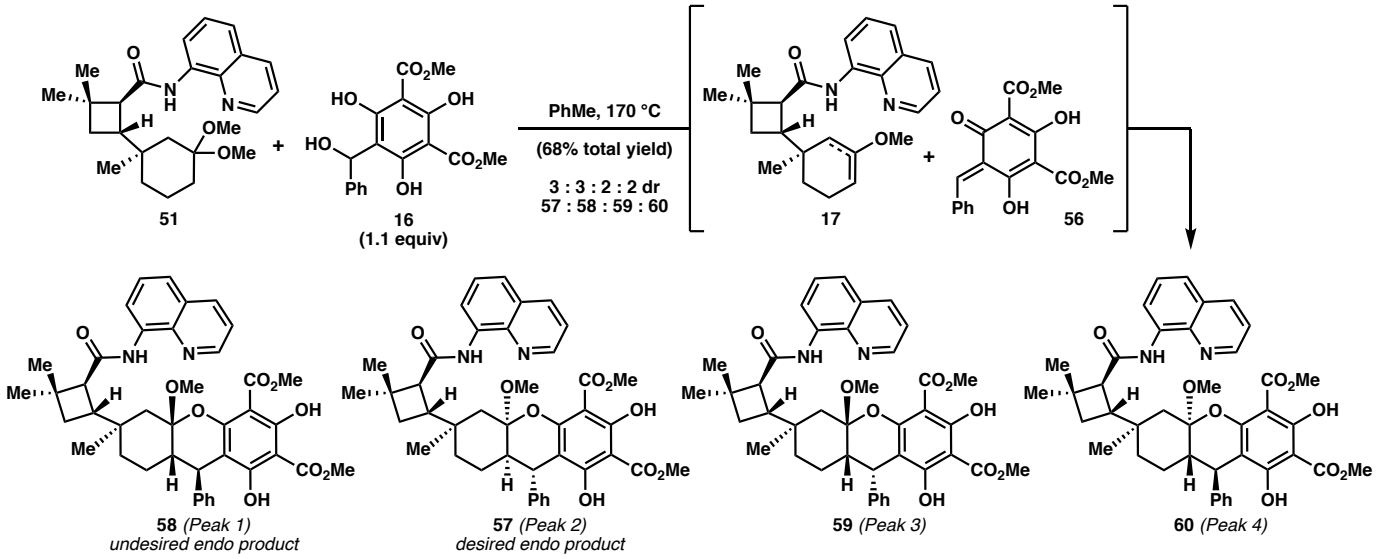


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %
1	4.862	MM	0.1218	736.52246	12.7082
2	5.726	MM	0.1379	5059.11182	87.2918

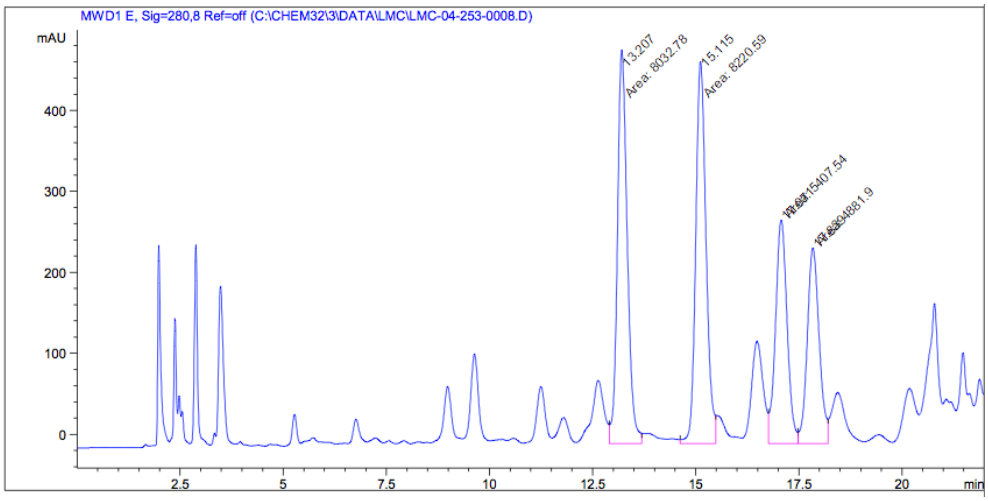
Deuterium-labeling studies to determine site of epimerization in *trans*-cyclobutane 47:



Preparation of tricyclic ketals 57–60 by thermal cycloaddition:



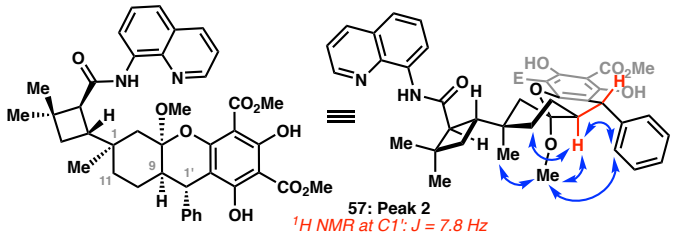
Chromatogram from HPLC separation of 57–60:



Chromatogram integration from HPLC separation of 57–60:

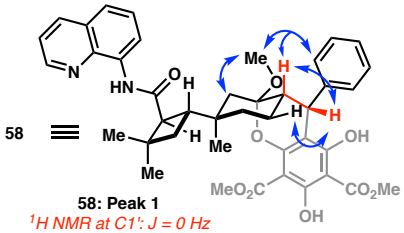
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.207	MF	0.2752	8032.78027	486.56299	30.2635
2	15.115	MF	0.2903	8220.58984	472.01566	30.9711
3	17.071	MF	0.3263	5407.54297	276.19736	20.3729
4	17.839	MF	0.3366	4881.89746	241.74495	18.3925

Summary of Key nOe correlations for stereochemical assignment of 57–60:



57: Peak 2

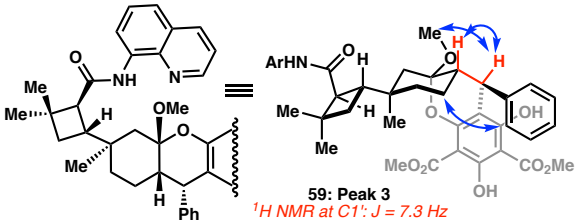
^{1}H NMR at C1': J = 7.8 Hz



58: Peak 1

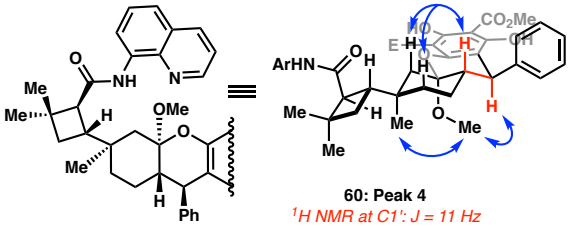
^{1}H NMR at C1': J = 0 Hz

(90 ° dihedral between C9 and C1')



59: Peak 3

^{1}H NMR at C1': J = 7.3 Hz



60: Peak 4

^{1}H NMR at C1': J = 11 Hz

(180 ° dihedral between C9 and C1')

Regio- and stereochemical assignment of Norrish–Yang product 80:

Key HMBC and nOe correlations:

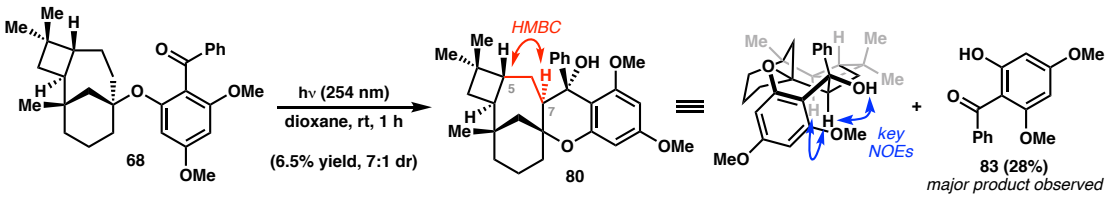
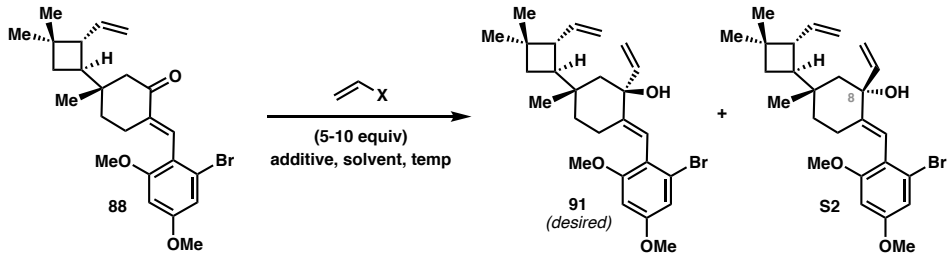


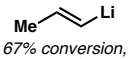
Table S3: Additional experiments toward optimization of 1,2-addition:



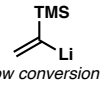
entry[a]	X	solvent	temp °C	additive	% conv.[b]	dr (91:S2)	% total yield (% yield 91)
1	MgBr	THF	–35	–	75	2.5:1	57 (41)
2	MgBr	THF	–78	–	56	4.5:1	41 (34)
3	MgBr	Et_2O	–35	–	ND	2.5:1	57 (41)
4	MgBr	CH_2Cl_2	0	–	85	1.9:1	69 (45)
5	MgBr	THF	0	–	60	2.0:1	50 (33)
6	MgBr	THF	–78	$CeCl_3$	81	1.6:1	66 (41)
7	MgBr	THF	–78	$LaCl_3$·2LiCl	100	1.7:1	59 (37)
8	Li	THF	–78	–	86	2.1:1	77 (52)
9	Li	THF	–78	$La(OTf)_3$	94	1.8:1	67 (43)
10	Li	THF	–100	–	87	1.4:1	61 (36)

[a] Test reactions conducted on 0.020 mmol scale. [b] Conversion determined by relative integration of UV LC/MS peaks, detected at 254 nm.

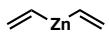
Alternative nucleophiles tested:



67% conversion, 7:3 dr

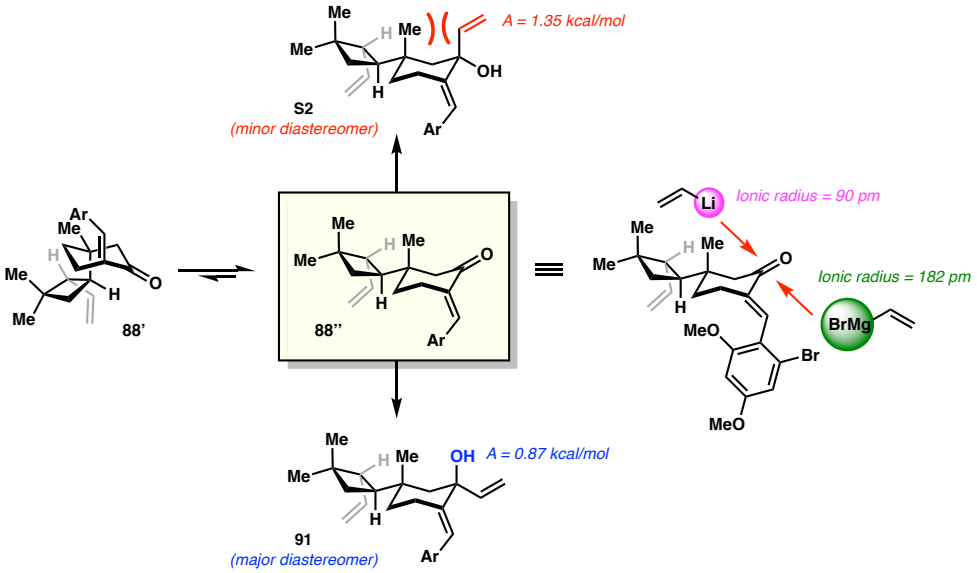


low conversion



no reaction

Conformational analysis and diasteroselectivity model for 1,2-addition:



Overview of the successful route to (+)-psiguadial B:

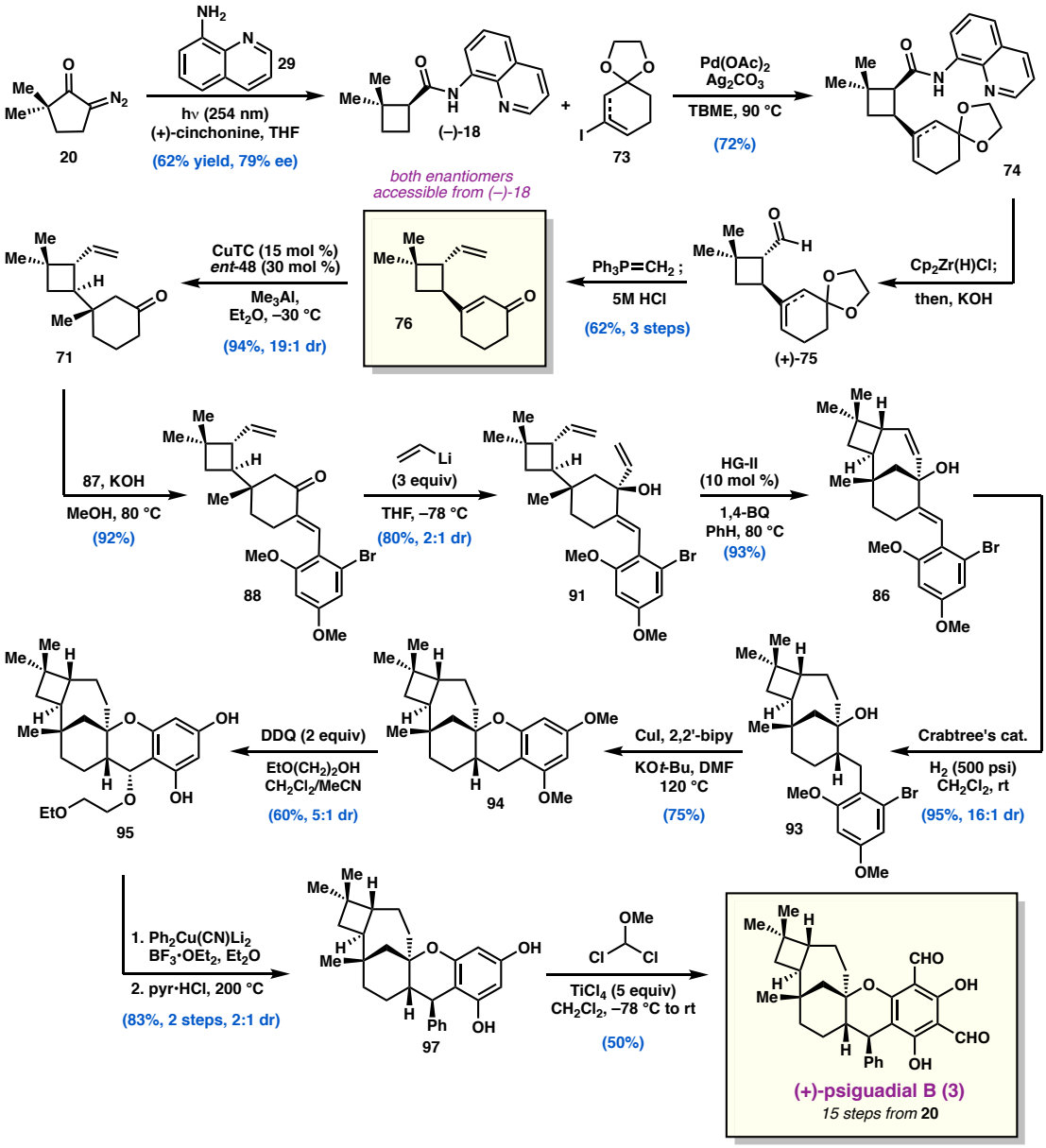
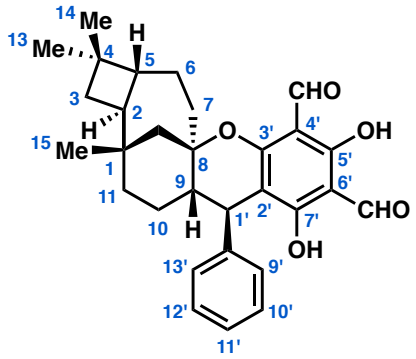


Table S4. Comparison of ^{1}H NMR spectroscopic data for natural and synthetic (+)-psiguadial B (3)

carbon number	Natural (+)-psiguadial B[1] ^{1}H NMR, 500 MHz, $CDCl_3$	Synthetic (+)-psiguadial B ^{1}H NMR, 400 MHz, $CDCl_3$
5'-OH	δ 13.51 (s, 1H)	δ 13.51 (s, 1H)
7'-OH	13.04 (s, 1H)	13.04 (s, 1H)
14', 15'	10.08 (s, 2H)	10.07 (s, 2H)
9', 13'[2]	7.23 (2H)	7.26 (dd, *J* = 14.6, 1.5 Hz, 2H)
11'	7.18 (3H)	7.23 – 7.17 (m, 1H)
10', 12'	–	7.10 (br m, 2H)
1'	3.49 (d, *J* = 11.5 Hz, 1H)	3.49 (d, *J* = 11.5 Hz, 1H)
2	2.16 (1H)	2.20 – 2.12 (m, 1H)
12	2.08 (1H)	2.09 (dd, *J* = 12.7, 2.4 Hz, 1H)
7	1.93 (1H)	1.92 (ddd, *J* = 14.9, 12.8, 4.2 Hz, 1H)
5	1.82 (m, 1H)	1.82 (ddd, *J* = 12.3, 8.8, 5.6 Hz, 1H)
9	1.68 (1H)	1.73 – 1.59 (m, 3H)
6	1.65 (1H)	–
7	1.58 (m, 1H)	–
3	1.52 (1H)	1.53 – 1.44 (m, 1H)
10	1.49 (m, 2H)	1.49 (ddd, *J* = 11.6, 8.1, 2.9 Hz, 2H)
6, 11	1.41 (2H)	–
3	1.37 (1H)	1.44 – 1.29 (m, 4H)
12	1.29 (1H)	–
11	1.10 (1H)	1.05 (dd, *J* = 7.6, 5.8 Hz, 1H)
13	1.02 (s, 3H)	1.02 (s, 3H)
14	1.01 (s, 3H)	1.00 (s, 3H)
15	0.86 (s, 3H)	0.85 (s, 3H)

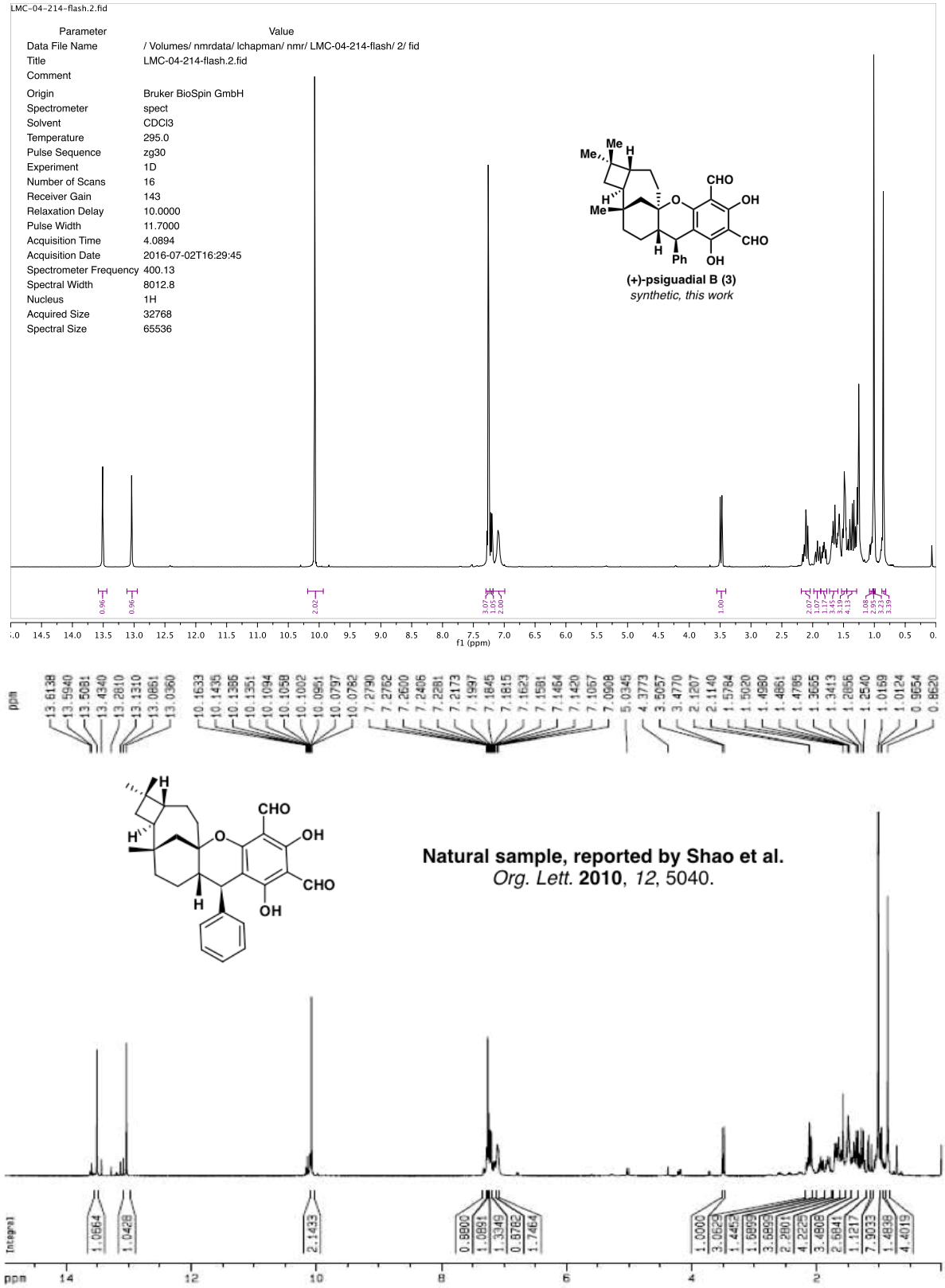


(+)-Psiguadial B (**3**) carbon numbering as reported by Shao et al.[1]

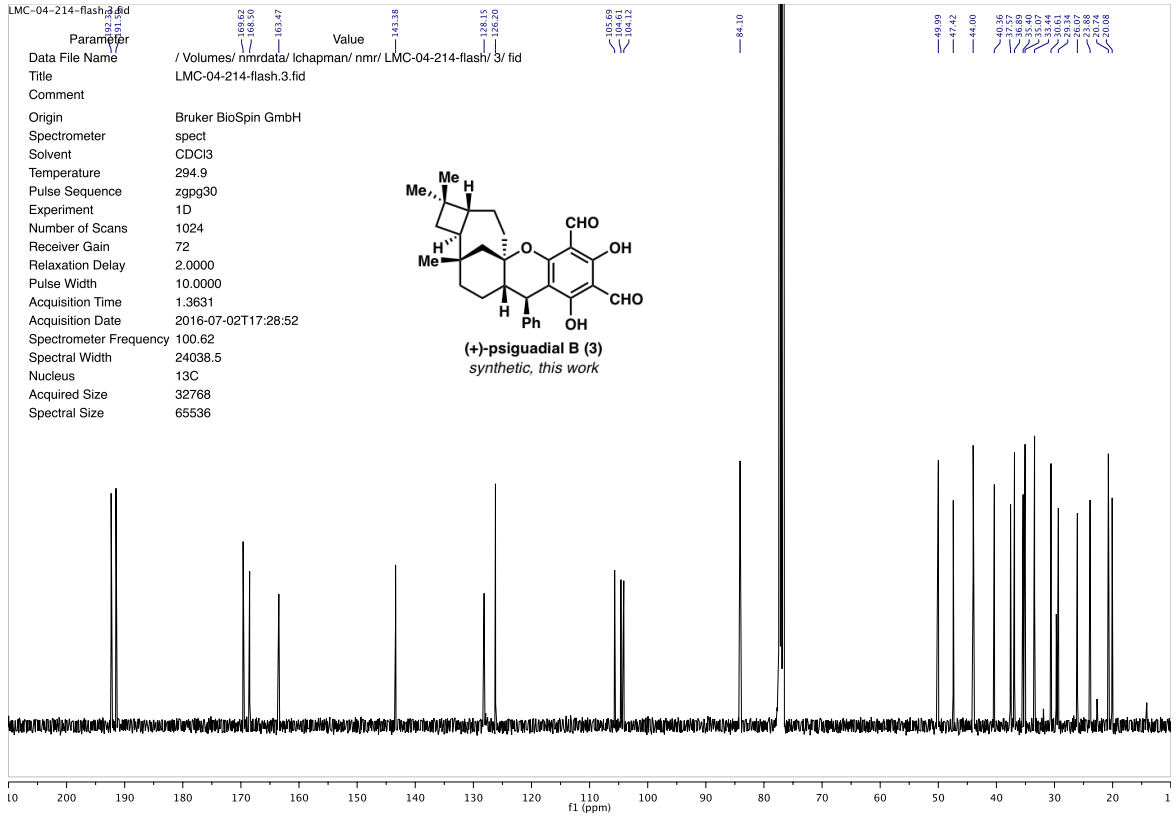
Table S5. Comparison of ^{13}C NMR spectroscopic data for natural and synthetic (+)-psiguadial B (3)

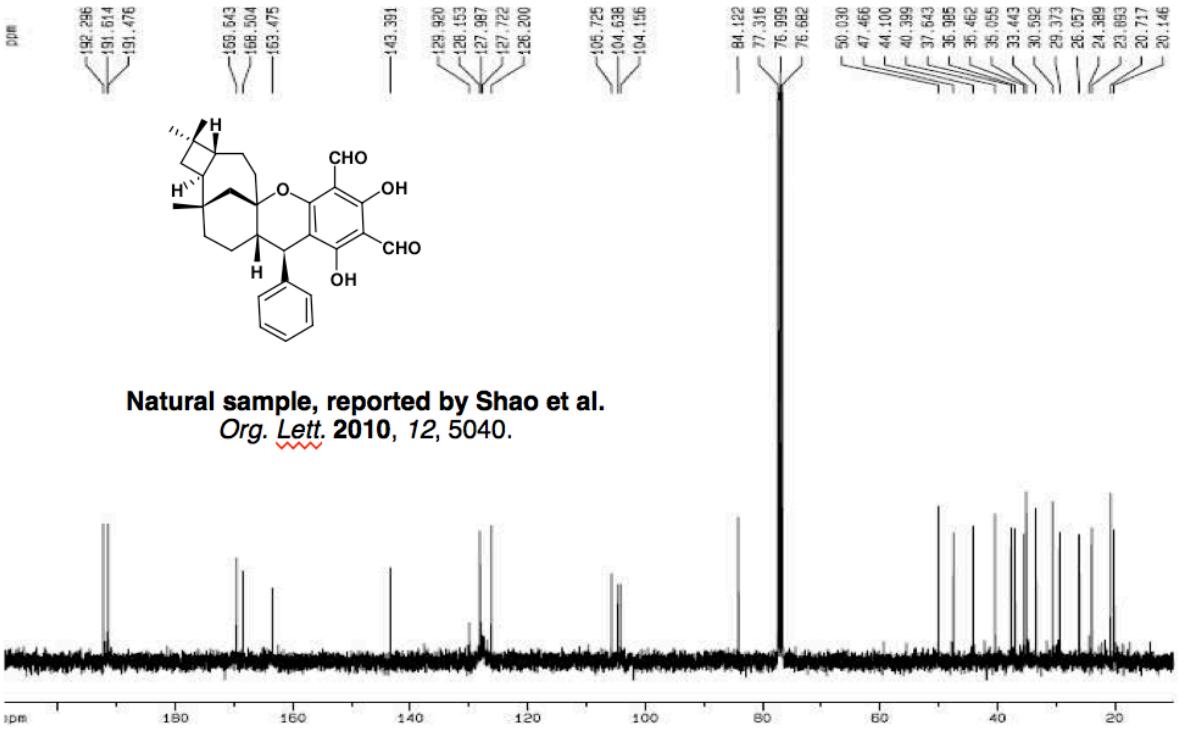
carbon number	**Natural (+)-psiguadial B**[1] ^{13}C NMR, 125 MHz, $CDCl_3$	**Synthetic (+)-psiguadial B**[3] ^{13}C NMR, 101 MHz, $CDCl_3$	Δ (ppm)
15'	192.3	192.3	0.0
14'	191.4	191.5	0.1
7'	169.6	169.6	0.0
5'	168.5	168.5	0.0
3'	163.5	163.5	0.0
8'	143.4	143.4	0.0
9', 11', 13'	128.2	128.2	0.0
10', 12'	126.2	126.2	0.0
2'	105.7	105.7	0.0
4'	104.6	104.6	0.0
6'	104.2	104.1	-0.1
8	84.1	84.1	0.0
9	50.0	50.0	0.0
12	47.5	47.4	-0.1
5	44.1	44.0	-0.1
1'	40.4	40.4	0.0
11	37.6	37.6	0.0
2	37.0	36.9	-0.1
3	35.5	35.4	-0.1
4	35.1	35.1	0.0
1	33.4	33.4	0.0
13	30.6	30.6	0.0
7	29.4	29.3	-0.1
15	26.1	26.1	0.0
10	23.9	23.9	0.0
14	20.7	20.7	0.0
6	20.1	20.1	0.0

^{1}H NMR spectral comparison of natural and synthetic (+)-psiguadial B (3):



^{13}C NMR spectral comparison of natural and synthetic (+)-psiguadial B (3):





X-Ray crystallographic data for minor ketone 50:

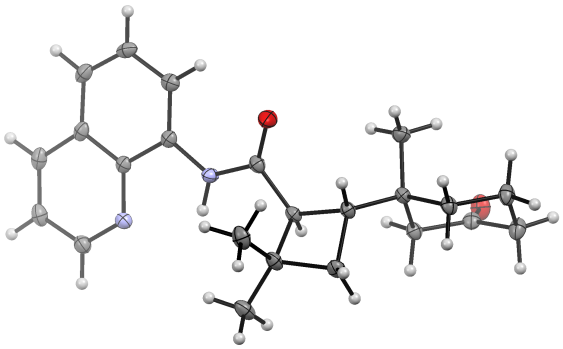


Table S6. Crystal data and structure refinement for LMC-04-038 or minor ketone **50**. Graphical representation of the structure with 50% probability thermal ellipsoids was generated using Mercury visualization software.

Identification code	LMC-04-038 or minor ketone **50**.	
Empirical formula	$C_{23}H_{28}N_2O_2$	
Formula weight	364.47	
Temperature	100 K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 6.5496(4) Å	α = 90°.
	b = 10.8428(6) Å	β = 90°.
	c = 27.6666(15) Å	γ = 90°.
Volume	1964.77(19) $Å^3$	
Z	4	
Density (calculated)	1.232 Mg/m^3	
Absorption coefficient	0.619 mm^{-1}	
F(000)	784	
Crystal size	0.25 x 0.15 x 0.1 mm^3	
Theta range for data collection	3.195 to 79.392°.	
Index ranges	-8<=h<=8, -13<=k<=13, -35<=l<=34	
Reflections collected	24424	
Independent reflections	4179 [R(int) = 0.0414]	
Completeness to theta = 67.679°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7542 and 0.7017	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4179 / 0 / 251	

Goodness-of-fit on F^2	1.021
Final R indices [I>2sigma(I)]	R1 = 0.0272, wR2 = 0.0655
R indices (all data)	R1 = 0.0300, wR2 = 0.0672
Absolute structure parameter	0.08(9)
Extinction coefficient	0.0031(3)
Largest diff. peak and hole	0.187 and -0.154 e.$Å^{-3}$

Crystallographic analysis and refinement details:

Low-temperature diffraction data (φ- and ω-scans) were collected on a Bruker AXS D8 VENTURE KAPPA diffractometer coupled to a PHOTON 100 CMOS detector with Cu-Kα radiation (λ = 1.54178 Å) from a IμS HB micro-focus sealed X-ray tube. All diffractometer manipulations, including data collection, integration, and scaling were carried out using the Bruker APEXII software.[4] Absorption corrections were applied using SADABS.[5] The structure was solved by intrinsic phasing using SHELXT14 and refined against F^2 on all data by full-matrix least squares with SHELXL-2014[6] using established refinement techniques.[7] All non-hydrogen atoms were refined anisotropically. The coordinates for the hydrogen atom bound to N1 were located in the difference Fourier synthesis and refined using a riding model. All other hydrogen atoms were included into the model at geometrically calculated positions and refined using a riding model. The isotropic displacement parameters of all hydrogen atoms were fixed to 1.2 times the *U* value of the atoms they are linked to (1.5 times for methyl groups and hydroxyl groups). Compound **50** crystallizes in the monoclinic space group $P2_12_12_1$ with one molecule in the asymmetric unit. Absolute configuration was determined by anomalous dispersion (Flack = 0.08(9)).[8] Graphical representation of the structure with 50% probability thermal ellipsoids was generated using Mercury visualization software.[9]

Table S7. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for minor ketone **50**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(2)	1276(2)	7506(1)	2689(1)	28(1)
O(1)	8526(2)	4834(1)	3832(1)	28(1)
N(1)	6907(2)	5042(1)	4555(1)	17(1)
N(2)	6036(2)	5230(1)	5486(1)	17(1)
C(1)	6977(2)	4762(1)	4073(1)	17(1)
C(19)	9262(2)	6260(1)	5671(1)	18(1)
C(8)	4149(2)	4860(1)	2948(1)	15(1)
C(23)	7898(2)	5670(1)	5348(1)	15(1)
C(15)	8440(2)	5545(1)	4849(1)	16(1)
C(7)	4752(2)	3938(1)	3343(1)	16(1)
C(2)	4949(2)	4328(1)	3880(1)	16(1)
C(17)	11684(2)	6499(1)	5023(1)	21(1)
C(18)	11180(2)	6672(1)	5498(1)	21(1)
C(16)	10309(2)	5955(1)	4692(1)	19(1)
C(3)	4183(2)	3002(1)	4028(1)	19(1)
C(22)	5507(2)	5373(2)	5942(1)	21(1)
C(10)	1519(2)	6401(2)	2662(1)	19(1)
C(14)	5948(2)	5744(2)	2867(1)	22(1)
C(21)	6728(3)	5976(2)	6290(1)	24(1)
C(9)	2231(2)	5619(1)	3083(1)	18(1)
C(6)	3268(3)	2907(1)	3511(1)	20(1)
C(13)	3699(2)	4120(2)	2484(1)	19(1)
C(20)	8601(3)	6410(2)	6153(1)	22(1)
C(12)	2998(2)	4916(2)	2060(1)	22(1)
C(4)	2696(3)	2947(2)	4449(1)	25(1)
C(5)	5955(3)	2106(2)	4103(1)	26(1)
C(11)	1133(3)	5697(2)	2200(1)	24(1)

Table S8. Bond lengths [Å] and angles [°] for minor ketone **50**.

O(2)-C(10)	1.211(2)
O(1)-C(1)	1.2176(18)
N(1)-C(1)	1.3676(19)
N(1)-C(15)	1.402(2)
N(1)-H(1)	0.90(2)
N(2)-C(23)	1.364(2)
N(2)-C(22)	1.319(2)
C(1)-C(2)	1.507(2)
C(19)-C(23)	1.417(2)
C(19)-C(18)	1.416(2)
C(19)-C(20)	1.412(2)
C(8)-C(7)	1.534(2)
C(8)-C(14)	1.535(2)
C(8)-C(9)	1.548(2)
C(8)-C(13)	1.540(2)
C(23)-C(15)	1.431(2)
C(15)-C(16)	1.373(2)
C(7)-H(7)	1.0000
C(7)-C(2)	1.5486(19)
C(7)-C(6)	1.552(2)
C(2)-H(2)	1.0000
C(2)-C(3)	1.577(2)
C(17)-H(17)	0.9500
C(17)-C(18)	1.369(2)
C(17)-C(16)	1.414(2)
C(18)-H(18)	0.9500
C(16)-H(16)	0.9500
C(3)-C(6)	1.555(2)
C(3)-C(4)	1.520(2)
C(3)-C(5)	1.527(2)
C(22)-H(22)	0.9500
C(22)-C(21)	1.410(2)
C(10)-C(9)	1.514(2)
C(10)-C(11)	1.511(2)

C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
C(21)-H(21)	0.9500
C(21)-C(20)	1.367(2)
C(9)-H(9A)	0.9900
C(9)-H(9B)	0.9900
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(13)-H(13A)	0.9900
C(13)-H(13B)	0.9900
C(13)-C(12)	1.527(2)
C(20)-H(20)	0.9500
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(12)-C(11)	1.535(2)
C(4)-H(4A)	0.9800
C(4)-H(4B)	0.9800
C(4)-H(4C)	0.9800
C(5)-H(5A)	0.9800
C(5)-H(5B)	0.9800
C(5)-H(5C)	0.9800
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900

C(1)-N(1)-C(15)	128.85(13)
C(1)-N(1)-H(1)	119.7(13)
C(15)-N(1)-H(1)	111.4(13)
C(22)-N(2)-C(23)	117.51(13)
O(1)-C(1)-N(1)	123.29(14)
O(1)-C(1)-C(2)	124.01(13)
N(1)-C(1)-C(2)	112.68(12)
C(18)-C(19)-C(23)	119.31(14)
C(20)-C(19)-C(23)	117.04(14)
C(20)-C(19)-C(18)	123.65(15)
C(7)-C(8)-C(14)	108.23(12)

C(7)-C(8)-C(9)	112.51(12)
C(7)-C(8)-C(13)	107.68(12)
C(14)-C(8)-C(9)	109.05(13)
C(14)-C(8)-C(13)	110.57(12)
C(13)-C(8)-C(9)	108.79(12)
N(2)-C(23)-C(19)	123.01(13)
N(2)-C(23)-C(15)	117.29(13)
C(19)-C(23)-C(15)	119.68(14)
N(1)-C(15)-C(23)	114.75(13)
C(16)-C(15)-N(1)	125.50(14)
C(16)-C(15)-C(23)	119.69(14)
C(8)-C(7)-H(7)	107.9
C(8)-C(7)-C(2)	121.88(12)
C(8)-C(7)-C(6)	121.41(13)
C(2)-C(7)-H(7)	107.9
C(2)-C(7)-C(6)	87.86(11)
C(6)-C(7)-H(7)	107.9
C(1)-C(2)-C(7)	119.93(12)
C(1)-C(2)-H(2)	109.5
C(1)-C(2)-C(3)	118.23(13)
C(7)-C(2)-H(2)	109.5
C(7)-C(2)-C(3)	88.51(11)
C(3)-C(2)-H(2)	109.5
C(18)-C(17)-H(17)	119.1
C(18)-C(17)-C(16)	121.76(15)
C(16)-C(17)-H(17)	119.1
C(19)-C(18)-H(18)	120.2
C(17)-C(18)-C(19)	119.62(15)
C(17)-C(18)-H(18)	120.2
C(15)-C(16)-C(17)	119.85(14)
C(15)-C(16)-H(16)	120.1
C(17)-C(16)-H(16)	120.1
C(6)-C(3)-C(2)	86.77(11)
C(4)-C(3)-C(2)	116.02(12)
C(4)-C(3)-C(6)	117.16(14)
C(4)-C(3)-C(5)	110.98(13)

C(5)-C(3)-C(2)	111.89(13)
C(5)-C(3)-C(6)	112.06(13)
N(2)-C(22)-H(22)	118.0
N(2)-C(22)-C(21)	123.90(15)
C(21)-C(22)-H(22)	118.0
O(2)-C(10)-C(9)	123.15(15)
O(2)-C(10)-C(11)	122.04(15)
C(11)-C(10)-C(9)	114.80(14)
C(8)-C(14)-H(14A)	109.5
C(8)-C(14)-H(14B)	109.5
C(8)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(22)-C(21)-H(21)	120.7
C(20)-C(21)-C(22)	118.69(15)
C(20)-C(21)-H(21)	120.7
C(8)-C(9)-H(9A)	109.4
C(8)-C(9)-H(9B)	109.4
C(10)-C(9)-C(8)	111.24(12)
C(10)-C(9)-H(9A)	109.4
C(10)-C(9)-H(9B)	109.4
H(9A)-C(9)-H(9B)	108.0
C(7)-C(6)-C(3)	89.19(11)
C(7)-C(6)-H(6A)	113.8
C(7)-C(6)-H(6B)	113.8
C(3)-C(6)-H(6A)	113.8
C(3)-C(6)-H(6B)	113.8
H(6A)-C(6)-H(6B)	111.0
C(8)-C(13)-H(13A)	108.8
C(8)-C(13)-H(13B)	108.8
H(13A)-C(13)-H(13B)	107.7
C(12)-C(13)-C(8)	113.71(13)
C(12)-C(13)-H(13A)	108.8
C(12)-C(13)-H(13B)	108.8
C(19)-C(20)-H(20)	120.1

C(21)-C(20)-C(19)	119.80(15)
C(21)-C(20)-H(20)	120.1
C(13)-C(12)-H(12A)	109.4
C(13)-C(12)-H(12B)	109.4
C(13)-C(12)-C(11)	111.00(13)
H(12A)-C(12)-H(12B)	108.0
C(11)-C(12)-H(12A)	109.4
C(11)-C(12)-H(12B)	109.4
C(3)-C(4)-H(4A)	109.5
C(3)-C(4)-H(4B)	109.5
C(3)-C(4)-H(4C)	109.5
H(4A)-C(4)-H(4B)	109.5
H(4A)-C(4)-H(4C)	109.5
H(4B)-C(4)-H(4C)	109.5
C(3)-C(5)-H(5A)	109.5
C(3)-C(5)-H(5B)	109.5
C(3)-C(5)-H(5C)	109.5
H(5A)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
C(10)-C(11)-C(12)	110.96(13)
C(10)-C(11)-H(11A)	109.4
C(10)-C(11)-H(11B)	109.4
C(12)-C(11)-H(11A)	109.4
C(12)-C(11)-H(11B)	109.4
H(11A)-C(11)-H(11B)	108.0

Table S9. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for minor ketone **50**. 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(2)	36(1)	22(1)	27(1)	0(1)	-3(1)	8(1)
O(1)	19(1)	47(1)	18(1)	-3(1)	4(1)	-2(1)
N(1)	15(1)	21(1)	16(1)	-2(1)	3(1)	-3(1)
N(2)	18(1)	17(1)	17(1)	1(1)	1(1)	1(1)
C(1)	20(1)	16(1)	16(1)	1(1)	2(1)	2(1)
C(19)	20(1)	12(1)	21(1)	2(1)	-6(1)	2(1)
C(8)	17(1)	16(1)	14(1)	1(1)	2(1)	0(1)
C(23)	16(1)	12(1)	17(1)	1(1)	-1(1)	2(1)
C(15)	17(1)	13(1)	18(1)	0(1)	-1(1)	2(1)
C(7)	19(1)	15(1)	14(1)	0(1)	1(1)	1(1)
C(2)	18(1)	15(1)	14(1)	0(1)	3(1)	0(1)
C(17)	14(1)	16(1)	33(1)	5(1)	-1(1)	-1(1)
C(18)	20(1)	16(1)	28(1)	2(1)	-9(1)	-2(1)
C(16)	18(1)	16(1)	22(1)	2(1)	2(1)	2(1)
C(3)	26(1)	14(1)	16(1)	1(1)	3(1)	-2(1)
C(22)	23(1)	23(1)	18(1)	2(1)	1(1)	0(1)
C(10)	16(1)	22(1)	19(1)	-1(1)	2(1)	4(1)
C(14)	19(1)	23(1)	25(1)	6(1)	-1(1)	-3(1)
C(21)	32(1)	24(1)	15(1)	0(1)	-1(1)	2(1)
C(9)	20(1)	18(1)	15(1)	-1(1)	2(1)	1(1)
C(6)	25(1)	14(1)	19(1)	-1(1)	1(1)	-3(1)
C(13)	21(1)	20(1)	15(1)	-2(1)	2(1)	3(1)
C(20)	29(1)	17(1)	19(1)	-1(1)	-8(1)	2(1)
C(12)	24(1)	29(1)	15(1)	-3(1)	1(1)	7(1)
C(4)	34(1)	21(1)	21(1)	1(1)	8(1)	-6(1)
C(5)	36(1)	18(1)	22(1)	3(1)	0(1)	4(1)
C(11)	24(1)	30(1)	18(1)	-3(1)	-3(1)	7(1)

Table S10. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for minor ketone **50**.

	x	y	z	U(eq)
H(7)	6064	3539	3245	19
H(2)	3872	4950	3959	19
H(17)	12993	6751	4913	25
H(18)	12112	7066	5712	25
H(16)	10677	5874	4361	22
H(22)	4228	5051	6044	25
H(14A)	6225	6198	3166	34
H(14B)	5607	6327	2609	34
H(14C)	7162	5270	2775	34
H(21)	6258	6078	6612	28
H(9A)	1121	5052	3180	21
H(9B)	2548	6159	3361	21
H(6A)	3511	2101	3353	23
H(6B)	1810	3142	3492	23
H(13A)	4948	3668	2389	22
H(13B)	2627	3502	2556	22
H(20)	9454	6811	6382	26
H(12A)	2645	4379	1783	27
H(12B)	4127	5466	1959	27
H(4A)	3400	3189	4747	38
H(4B)	2174	2105	4483	38
H(4C)	1557	3513	4389	38
H(5A)	6918	2181	3833	38
H(5B)	5430	1261	4117	38
H(5C)	6654	2302	4406	38
H(11A)	825	6286	1936	29
H(11B)	-67	5153	2242	29
H(1)	5760(30)	4900(20)	4724(7)	29

Table S11. Torsion angles [°] for minor ketone **50**.

O(2)-C(10)-C(9)-C(8)	-125.20(17)
O(2)-C(10)-C(11)-C(12)	127.00(17)
O(1)-C(1)-C(2)-C(7)	0.1(2)
O(1)-C(1)-C(2)-C(3)	106.07(18)
N(1)-C(1)-C(2)-C(7)	-178.49(13)
N(1)-C(1)-C(2)-C(3)	-72.53(17)
N(1)-C(15)-C(16)-C(17)	176.96(14)
N(2)-C(23)-C(15)-N(1)	3.66(19)
N(2)-C(23)-C(15)-C(16)	-179.03(13)
N(2)-C(22)-C(21)-C(20)	-2.0(3)
C(1)-N(1)-C(15)-C(23)	-177.11(14)
C(1)-N(1)-C(15)-C(16)	5.8(3)
C(1)-C(2)-C(3)-C(6)	-144.20(13)
C(1)-C(2)-C(3)-C(4)	97.03(17)
C(1)-C(2)-C(3)-C(5)	-31.70(18)
C(19)-C(23)-C(15)-N(1)	-174.83(13)
C(19)-C(23)-C(15)-C(16)	2.5(2)
C(8)-C(7)-C(2)-C(1)	-91.02(17)
C(8)-C(7)-C(2)-C(3)	146.90(14)
C(8)-C(7)-C(6)-C(3)	-147.60(13)
C(8)-C(13)-C(12)-C(11)	-55.22(18)
C(23)-N(2)-C(22)-C(21)	1.6(2)
C(23)-C(19)-C(18)-C(17)	0.3(2)
C(23)-C(19)-C(20)-C(21)	0.9(2)
C(23)-C(15)-C(16)-C(17)	0.0(2)
C(15)-N(1)-C(1)-O(1)	6.0(3)
C(15)-N(1)-C(1)-C(2)	-175.39(14)
C(7)-C(8)-C(9)-C(10)	-172.85(13)
C(7)-C(8)-C(13)-C(12)	177.89(13)
C(7)-C(2)-C(3)-C(6)	-20.67(11)
C(7)-C(2)-C(3)-C(4)	-139.44(14)
C(7)-C(2)-C(3)-C(5)	91.84(14)
C(2)-C(7)-C(6)-C(3)	-20.99(11)
C(2)-C(3)-C(6)-C(7)	20.61(11)

C(18)-C(19)-C(23)-N(2)	179.01(14)
C(18)-C(19)-C(23)-C(15)	-2.6(2)
C(18)-C(19)-C(20)-C(21)	-179.54(15)
C(18)-C(17)-C(16)-C(15)	-2.4(2)
C(16)-C(17)-C(18)-C(19)	2.3(2)
C(22)-N(2)-C(23)-C(19)	0.2(2)
C(22)-N(2)-C(23)-C(15)	-178.22(14)
C(22)-C(21)-C(20)-C(19)	0.7(2)
C(14)-C(8)-C(7)-C(2)	71.18(17)
C(14)-C(8)-C(7)-C(6)	-179.66(13)
C(14)-C(8)-C(9)-C(10)	67.06(16)
C(14)-C(8)-C(13)-C(12)	-64.05(17)
C(9)-C(8)-C(7)-C(2)	-49.38(18)
C(9)-C(8)-C(7)-C(6)	59.77(18)
C(9)-C(8)-C(13)-C(12)	55.69(16)
C(9)-C(10)-C(11)-C(12)	-52.89(19)
C(6)-C(7)-C(2)-C(1)	142.76(14)
C(6)-C(7)-C(2)-C(3)	20.68(11)
C(13)-C(8)-C(7)-C(2)	-169.26(13)
C(13)-C(8)-C(7)-C(6)	-60.10(17)
C(13)-C(8)-C(9)-C(10)	-53.62(16)
C(13)-C(12)-C(11)-C(10)	51.44(19)
C(20)-C(19)-C(23)-N(2)	-1.4(2)
C(20)-C(19)-C(23)-C(15)	176.96(13)
C(20)-C(19)-C(18)-C(17)	-179.26(15)
C(4)-C(3)-C(6)-C(7)	138.32(14)
C(5)-C(3)-C(6)-C(7)	-91.73(14)
C(11)-C(10)-C(9)-C(8)	54.70(18)

References

(1) Isolation of psiguadials A and B: Shao, M.; Wang, Y.; Liu, Z.; Zhang, D.-M.; Cao, H.-H.; Jiang, R.-W.; Fan, C.-L.; Zhang, X.-Q.; Chen, H.-R.; Yao, X.-S.; Ye, W.-C. *Org. Lett.* **2010**, *12*, 5040. Except where designated, multiplicities are not specified.

(2) Shao et al. report the two protons at 7.23 ppm as corresponding to the carbon signal at 126.2 ppm (carbons 10' and 12'). This is assumed to be a typographical error based on the reported plot of their HSQC spectrum and our own data showing a correlation between the proton signal at 7.26 ppm and the more downfield carbon shift at 128.2 ppm, which corresponds to carbons 9' and 13'. The remaining three aromatic proton signals are adjusted accordingly in the comparison list.

(3) The center peak of $CDCl_3$ is referenced to 76.999 ppm, as shown in the Shao et al. report, see ^{13}C spectral comparison on page S18.

(4) APEX2, Version 2 User Manual, M86-E01078, Bruker Analytical X-ray Systems, Madison, WI, June 2006.

(5) Sheldrick, G.M. *"SADABS (version 2008/1): Program for Absorption Correction for Data from Area Detector Frames"*, University of Göttingen, 2008.

(6) Sheldrick, G. M. *Acta Crystallogr A* **2008**, *64*, 112.

(7) Müller, P. *Crystallogr. Rev.* **2009**, *15*, 57.

(8) Parsons, S.; Flack, H. D.; Wagner, T. *Acta Cryst*. **2013**, *69*, 249.

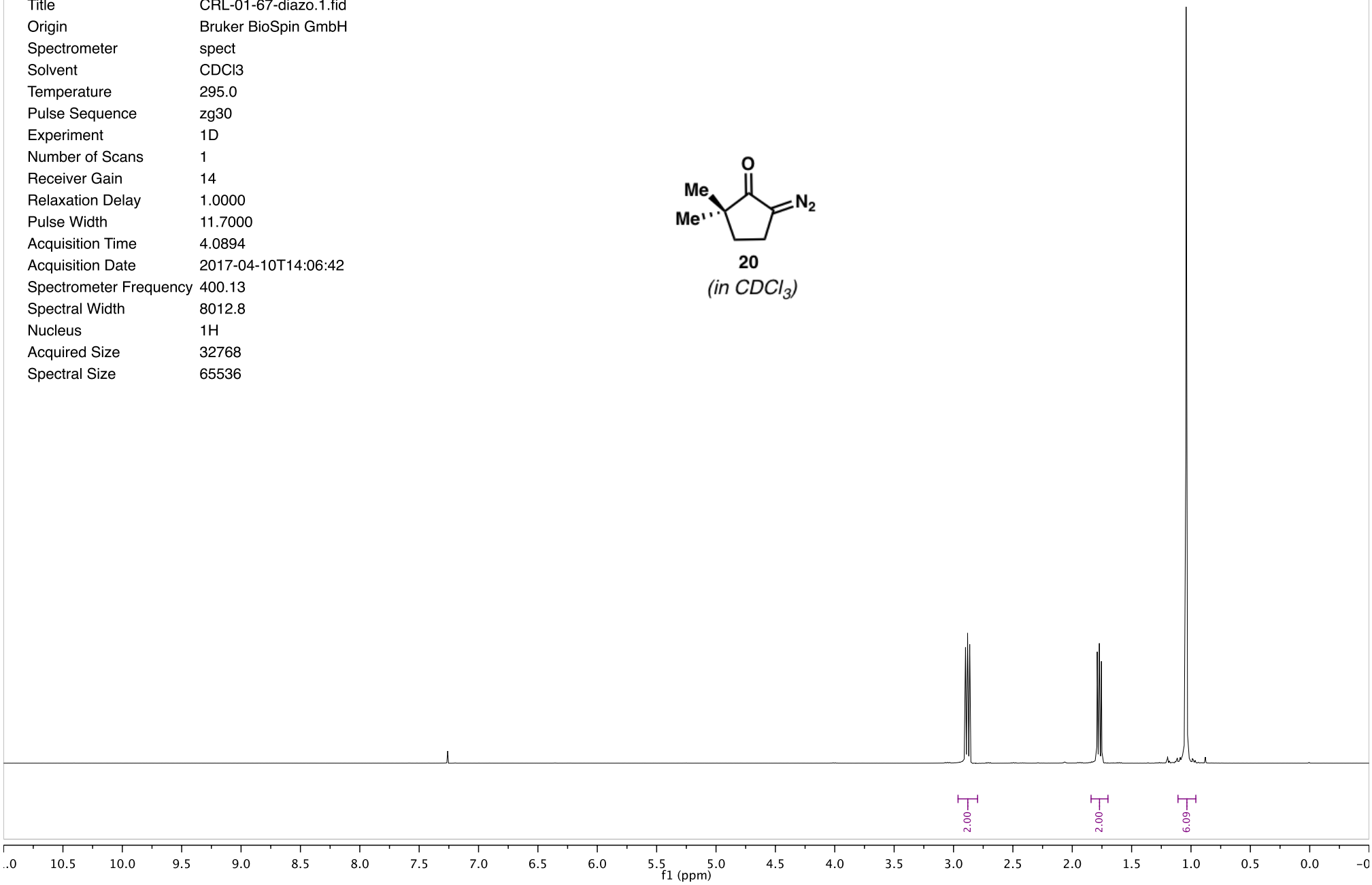
(9) Macrae, C. F.; Edgington, P. R.; McCabe, P.; Pidcock, E.; Shields, G. P.; Taylor, R.; Towler M.; van de Streek, J. *J. Appl. Cryst.* **2006**, *39*, 453.

CRL-01-67-diazo.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ CRL-01-67-diazo/ 1/ fid
Title	CRL-01-67-diazo.1.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	1
Receiver Gain	14
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-04-10T14:06:42
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

20

(in $CDCl_3$)

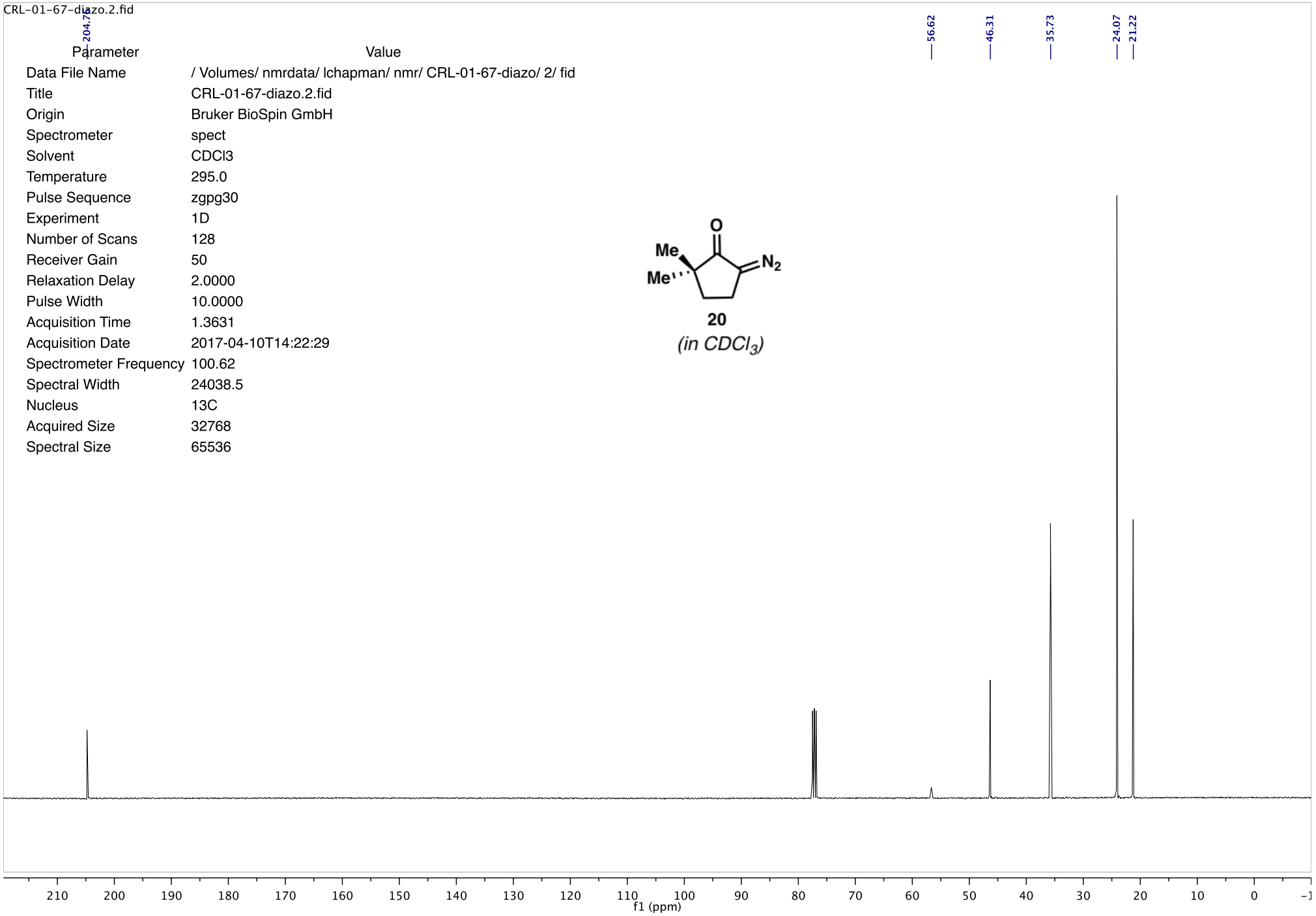


CRL-01-67-diazo.2.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ CRL-01-67-diazo/ 2/ fid
Title	CRL-01-67-diazo.2.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	50
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-04-10T14:22:29
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

20

(in $CDCl_3$)



LMC-04-273-diazo-SM.1.fid

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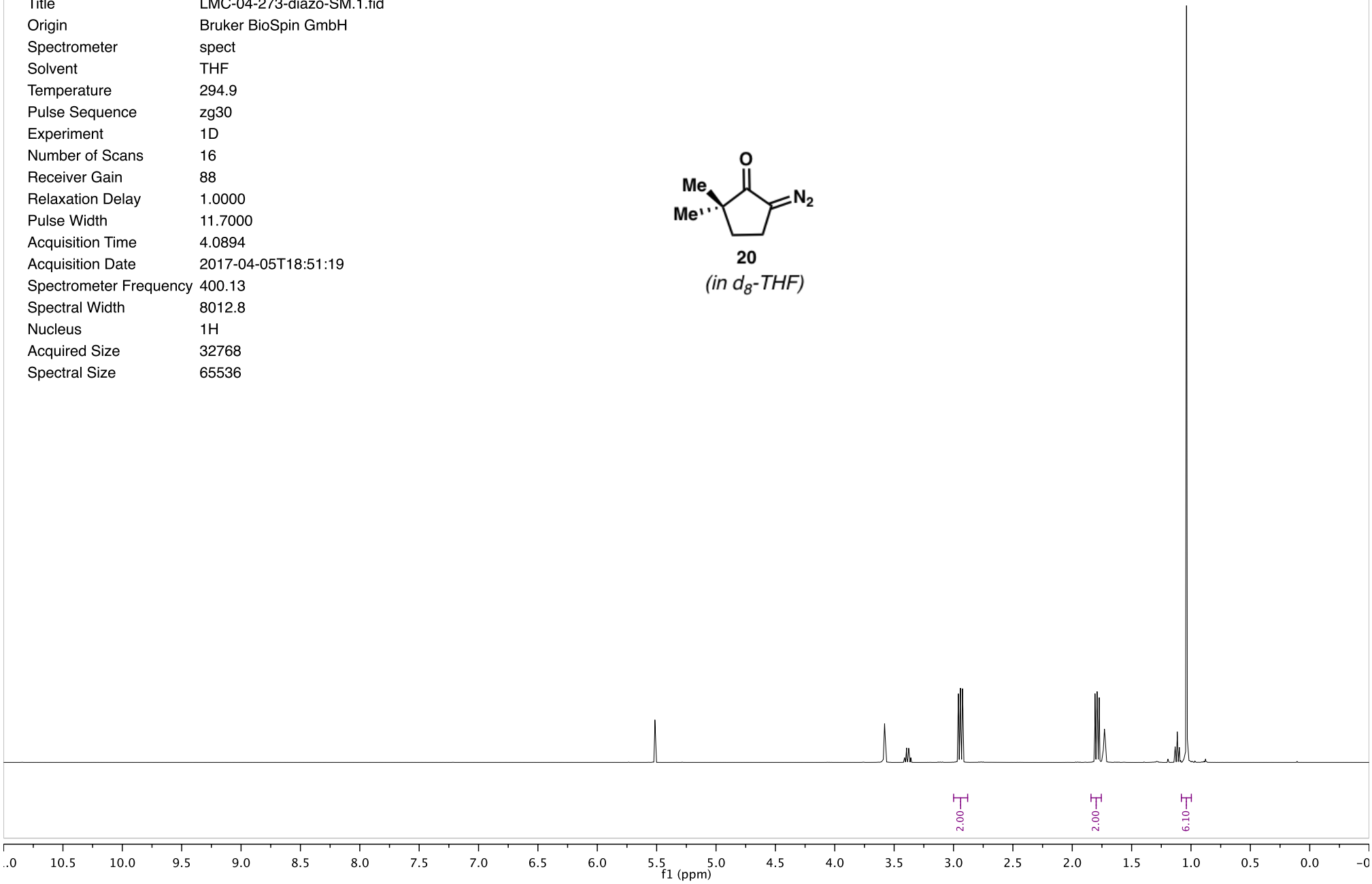
Spectrometer	spect
Solvent	THF
Temperature	294.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	88
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-04-05T18:51:19
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

20

(in d_8-THF)



LMC-04-273-diazo-SM.2.fid

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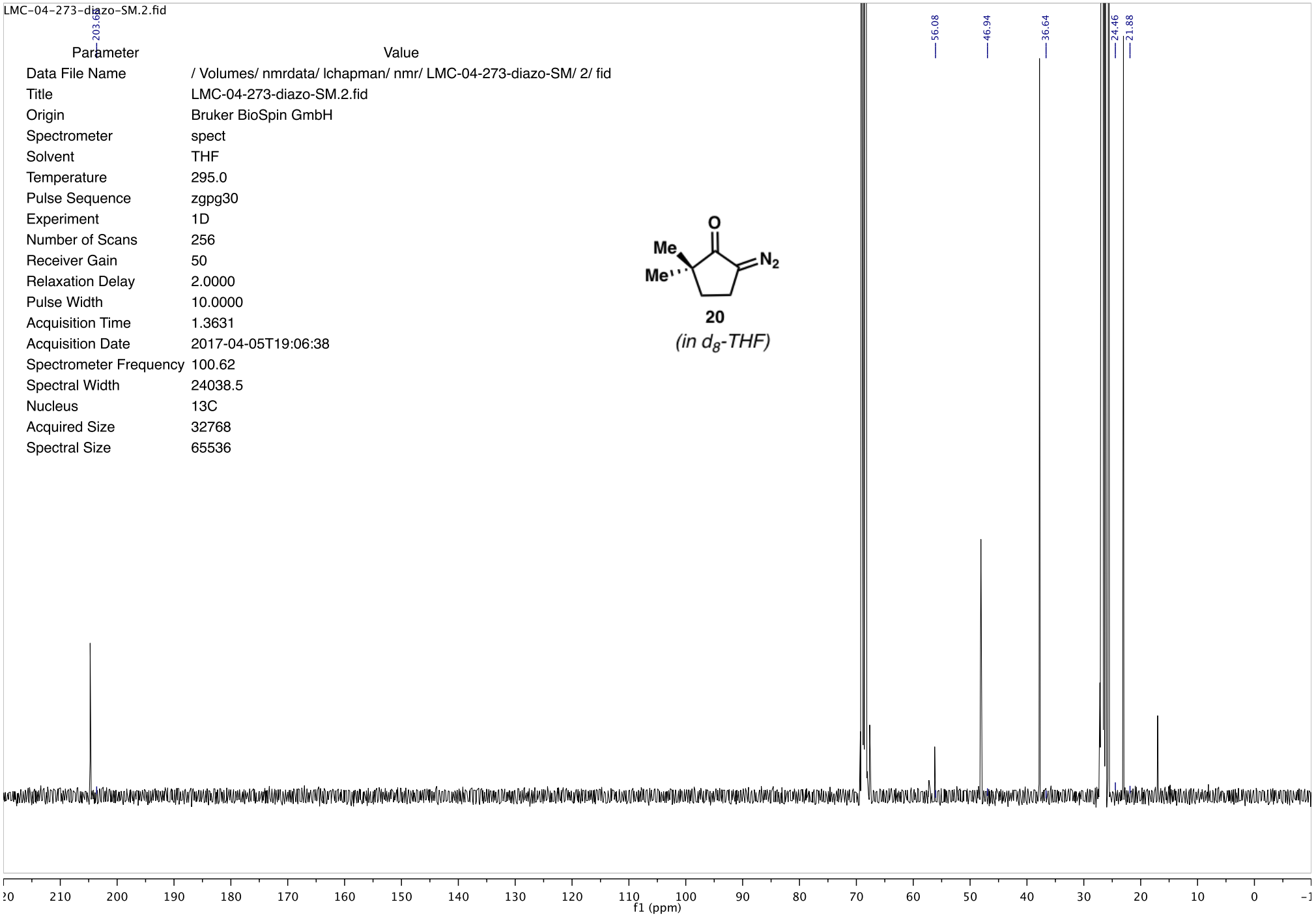
Title	LMC-04-273-diazo-SM.2.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	THF
Temperature	295.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	256
Receiver Gain	50
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-04-05T19:06:38
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

20

(in d_8-THF)



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Title	CRL_01_113_1_Nov.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	79
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-11-20T17:48:00
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1522.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

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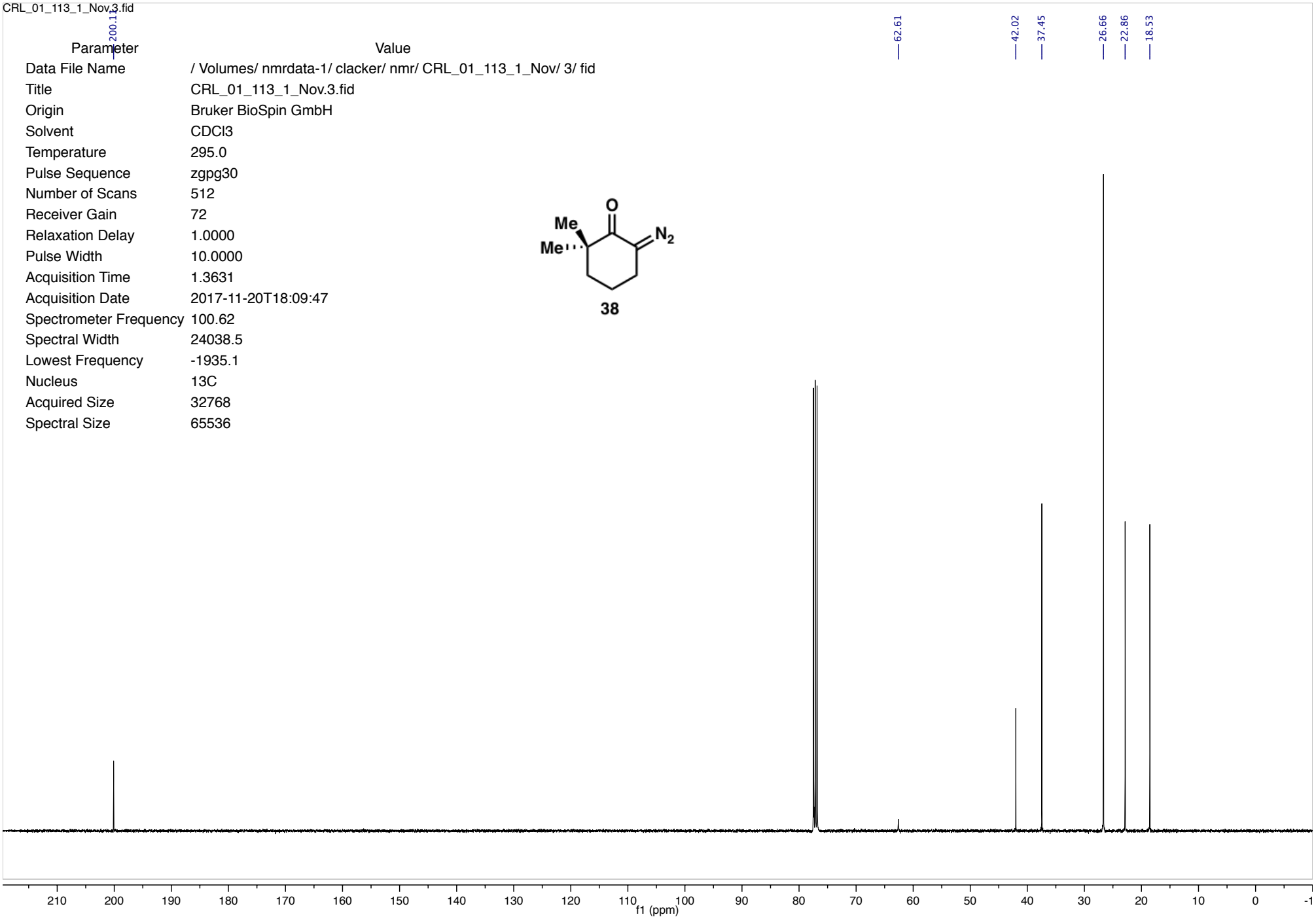
CRL_01_113_1_Nov.3.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata-1/ clacker/ nmr/ CRL_01_113_1_Nov/ 3/ fid
Title	CRL_01_113_1_Nov.3.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	512
Receiver Gain	72
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-11-20T18:09:47
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1935.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

38



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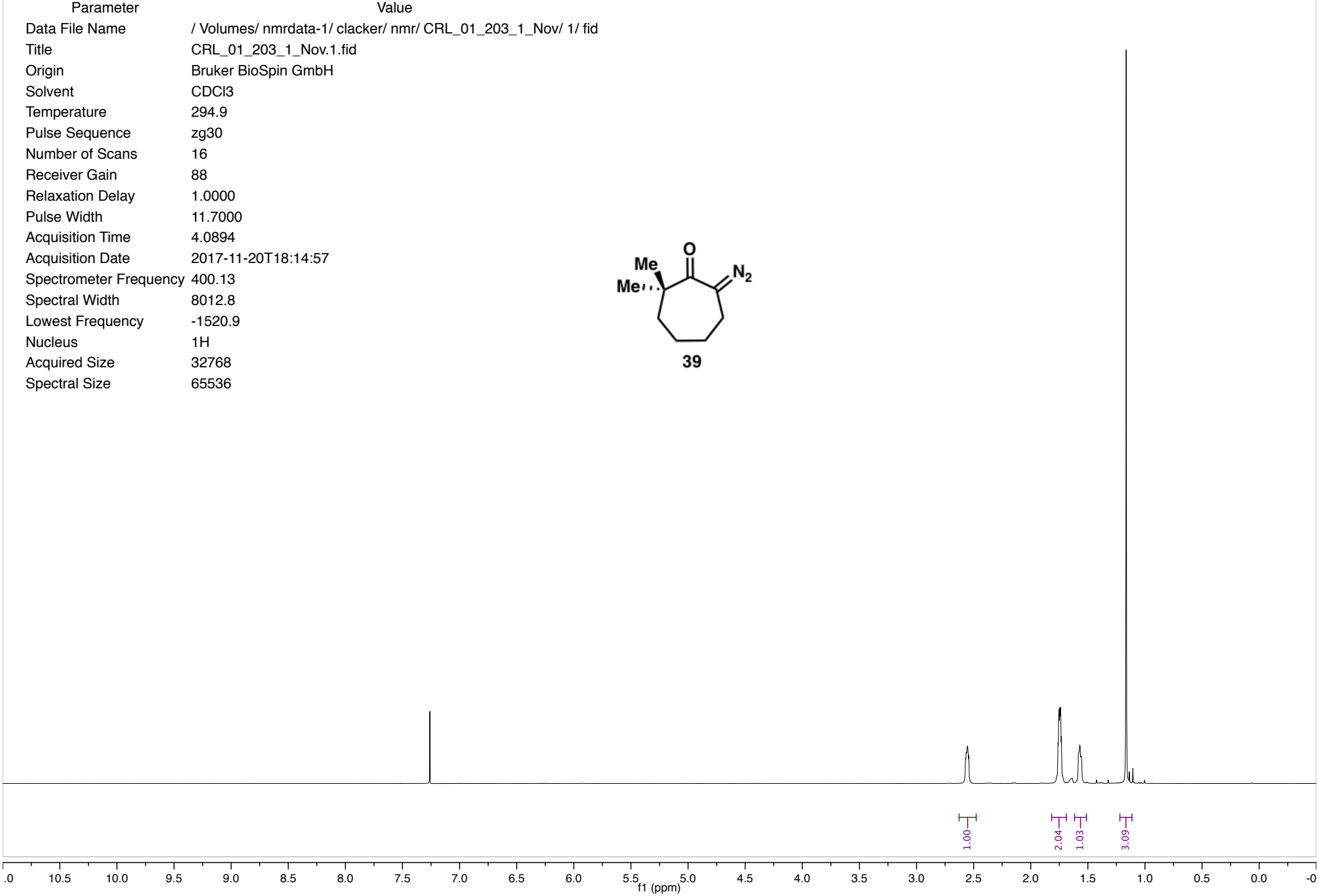
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	88
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-11-20T18:14:57
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1520.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

39



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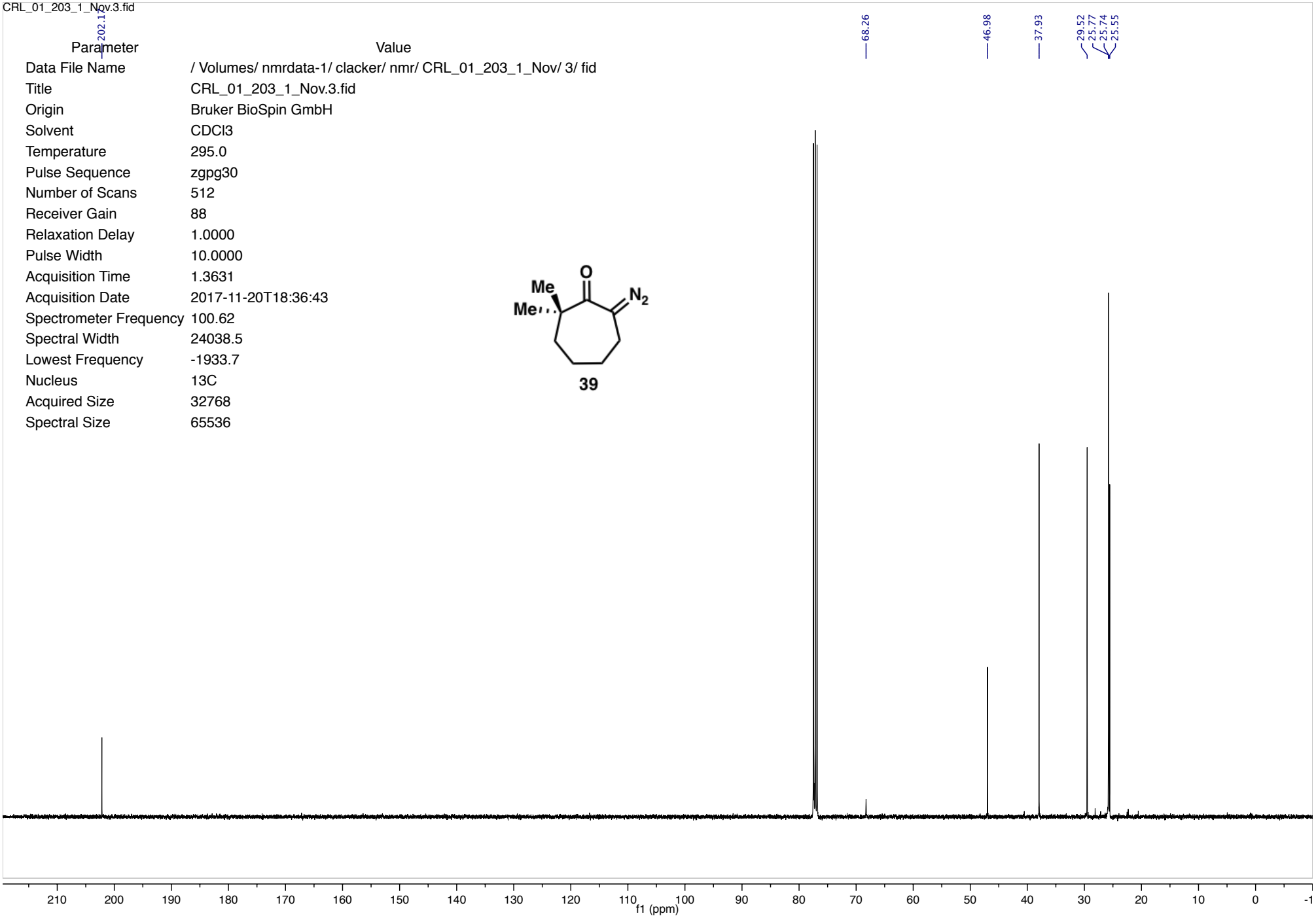
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	512
Receiver Gain	88
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-11-20T18:36:43
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1933.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

39



JCB_cyclobutamide_060916.1.fid

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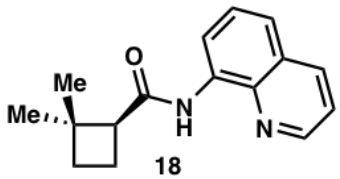
Title	JCB_cyclobutamide_060916.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	1
Receiver Gain	197
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-06-09T16:38:44
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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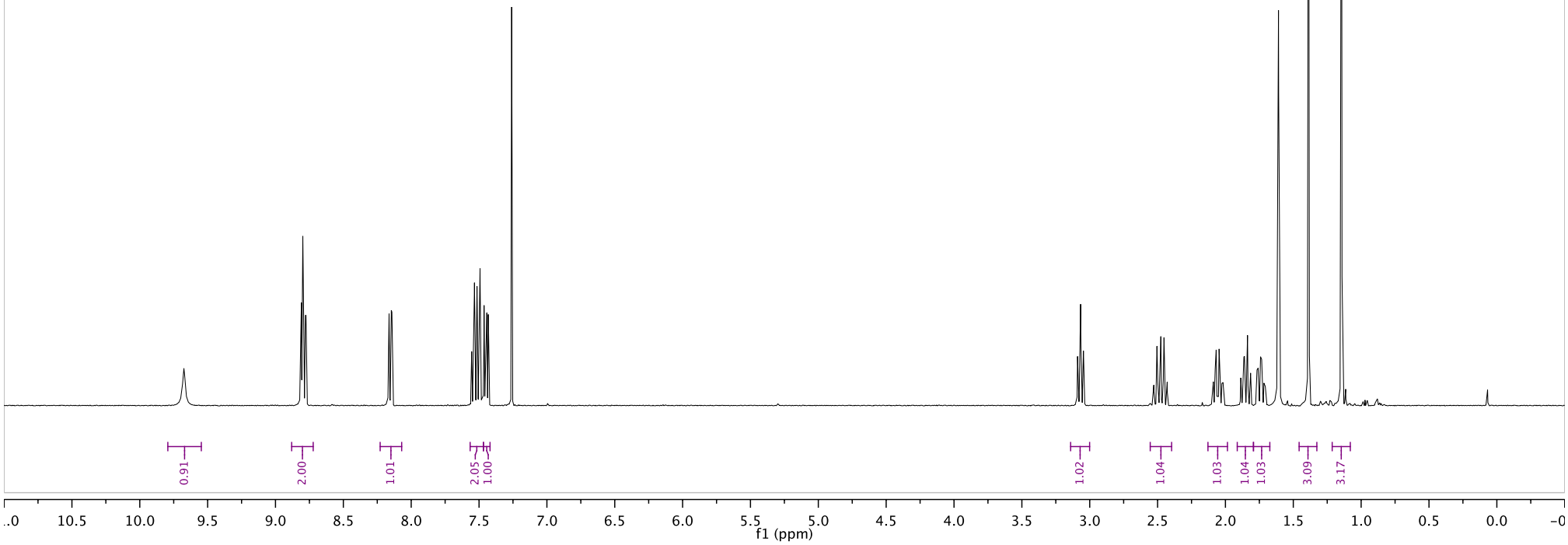

JCB_cyclobutamide_060916.3.fid

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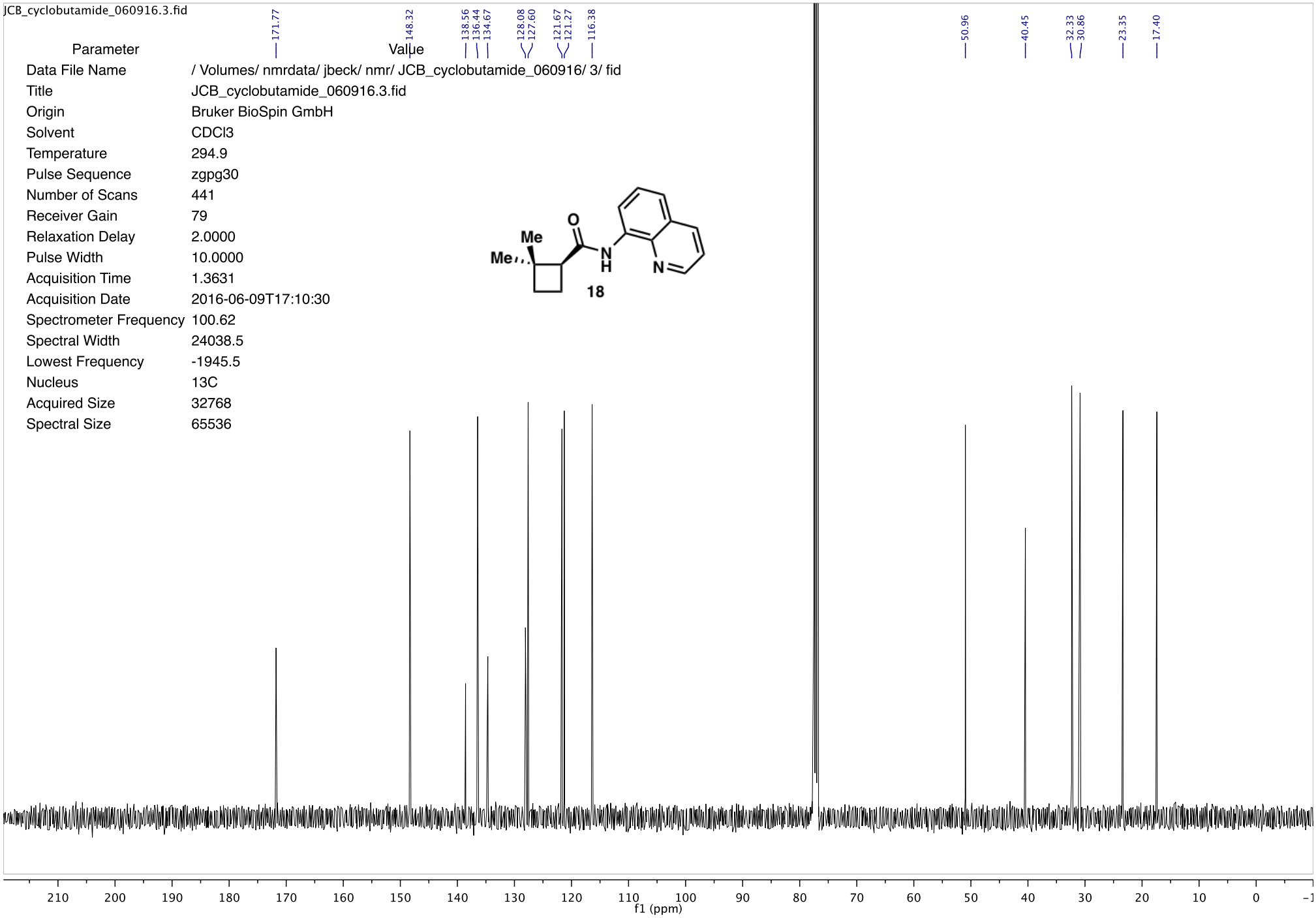
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	441
Receiver Gain	79
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-06-09T17:10:30
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1945.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

18



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Temperature	294.9
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Receiver Gain	197
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-04-19T21:04:33
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

42

CRL_01_074_2_1scan.2.fid

Parameter	Value
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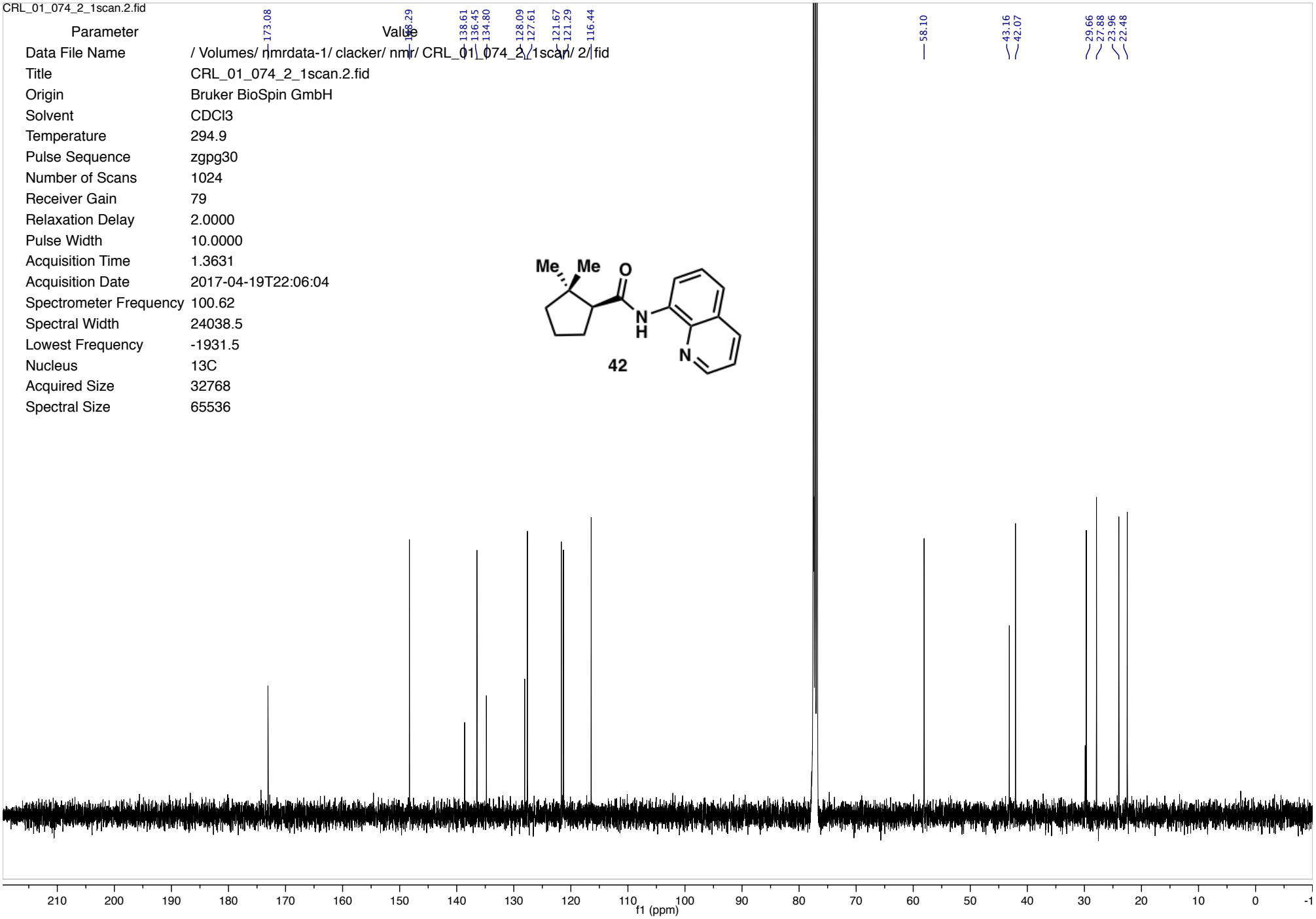
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	1024
Receiver Gain	79
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-04-19T22:06:04
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1931.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

42



CRL_01_074_4.1.fid

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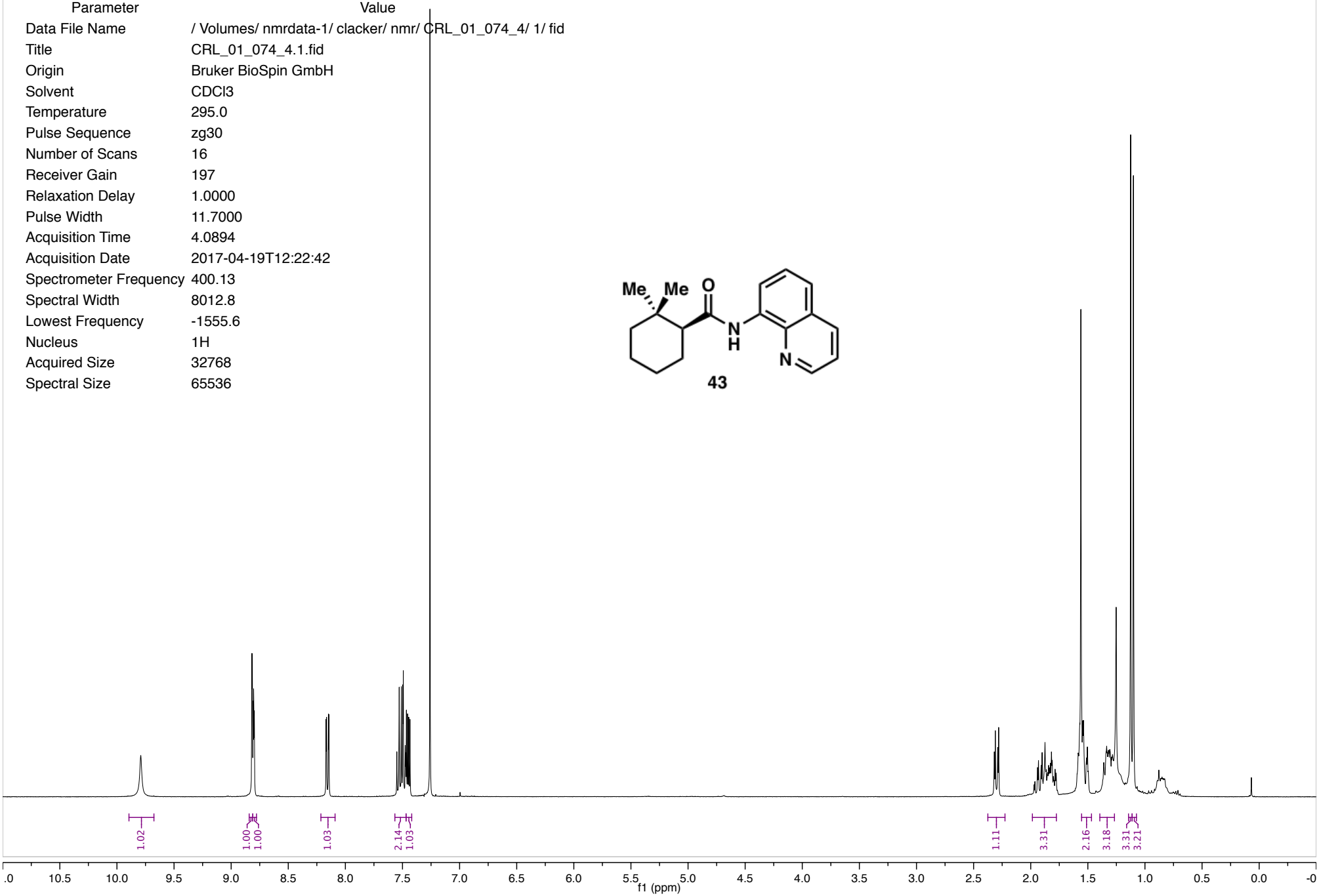
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	197
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-04-19T12:22:42
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

43



CRL_01_074_4_C.1.fid

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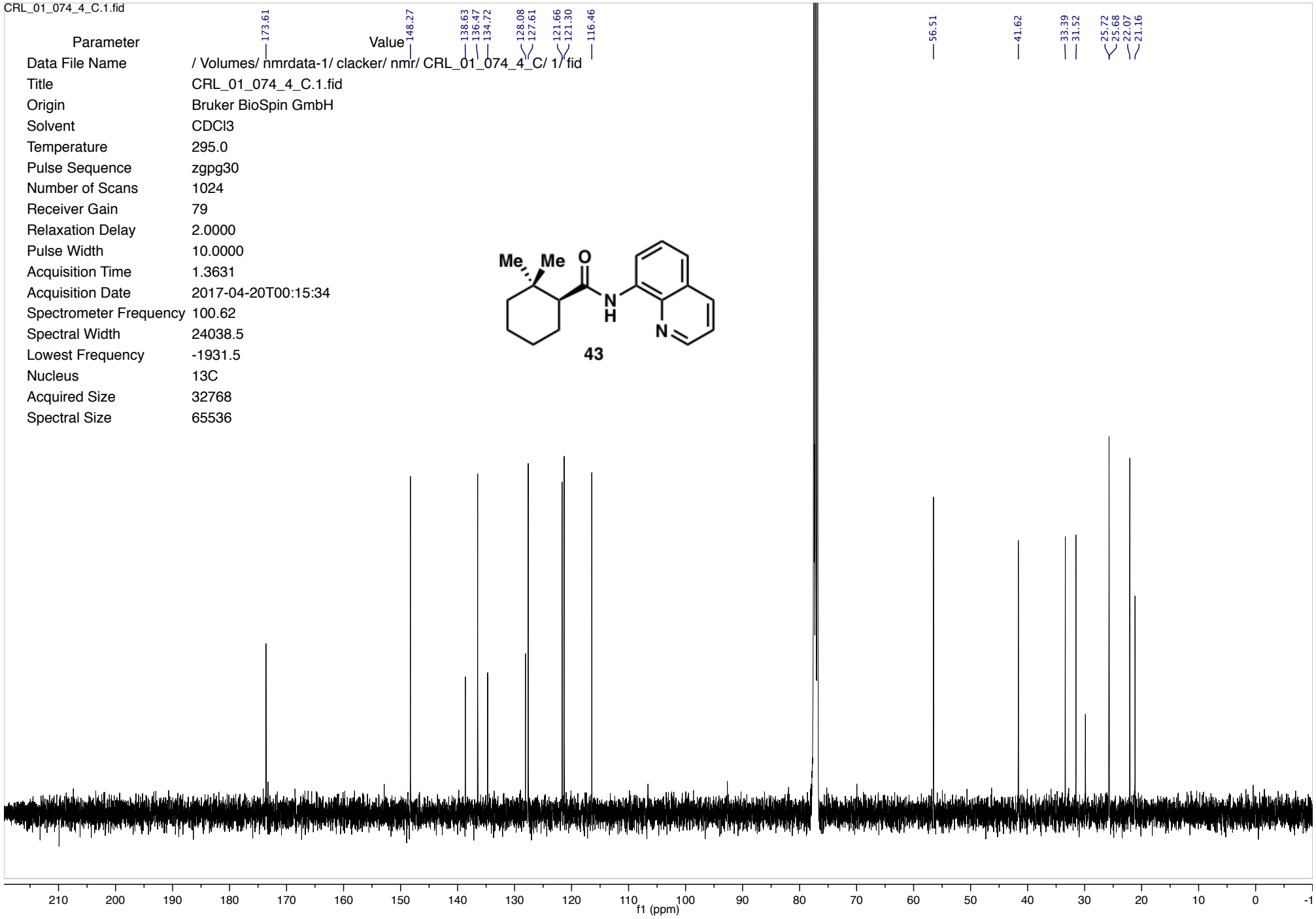
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	1024
Receiver Gain	79
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-04-20T00:15:34
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1931.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

43



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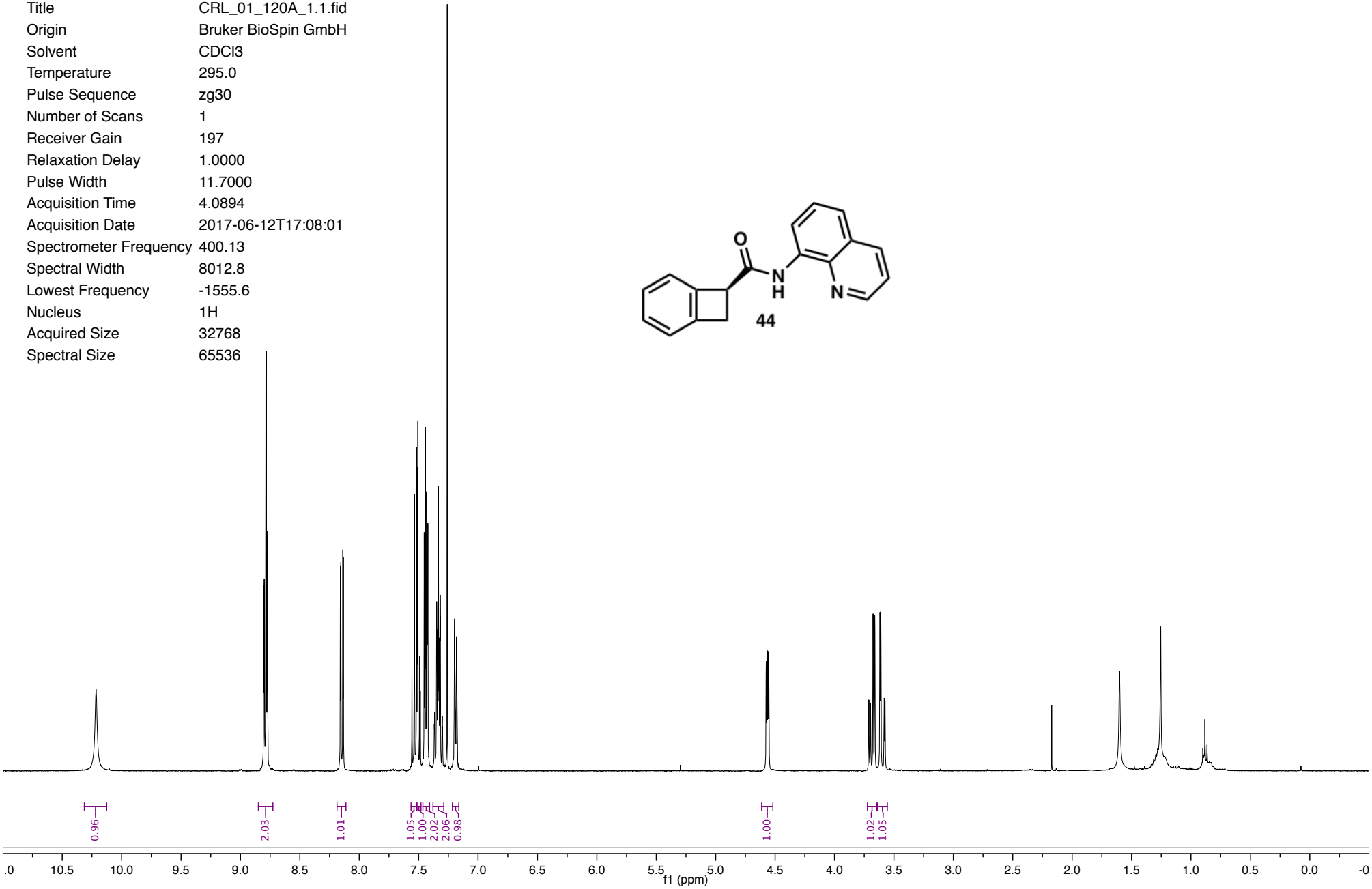
Title	CRL_01_120A_1.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	1
Receiver Gain	197
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-06-12T17:08:01
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

44



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Parameter	Value
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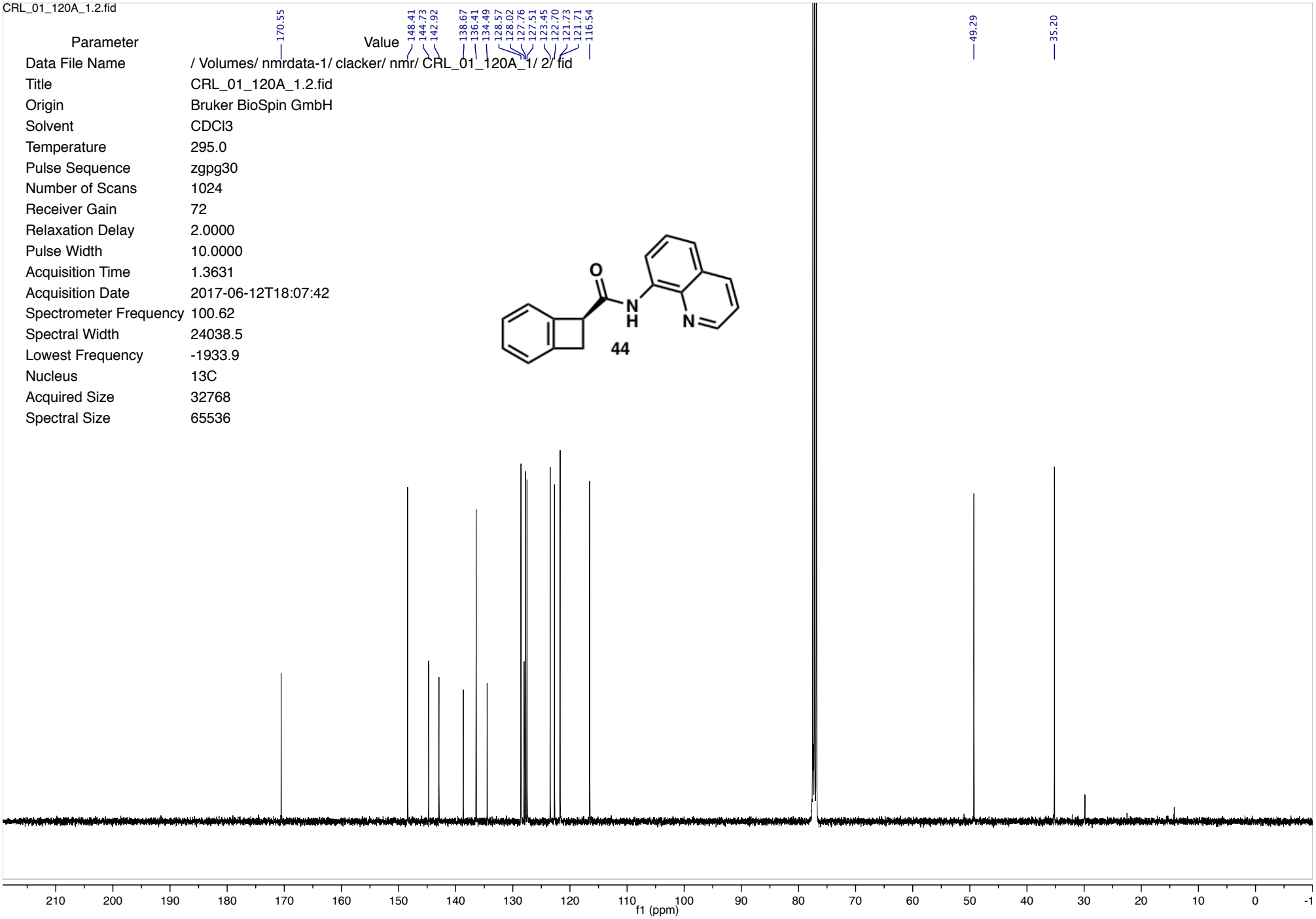
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	1024
Receiver Gain	72
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-06-12T18:07:42
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1933.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

44



CRL_01_074_3_H.1.fid

Parameter	Value
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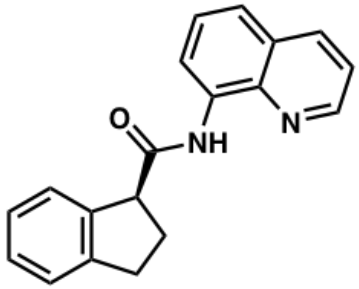
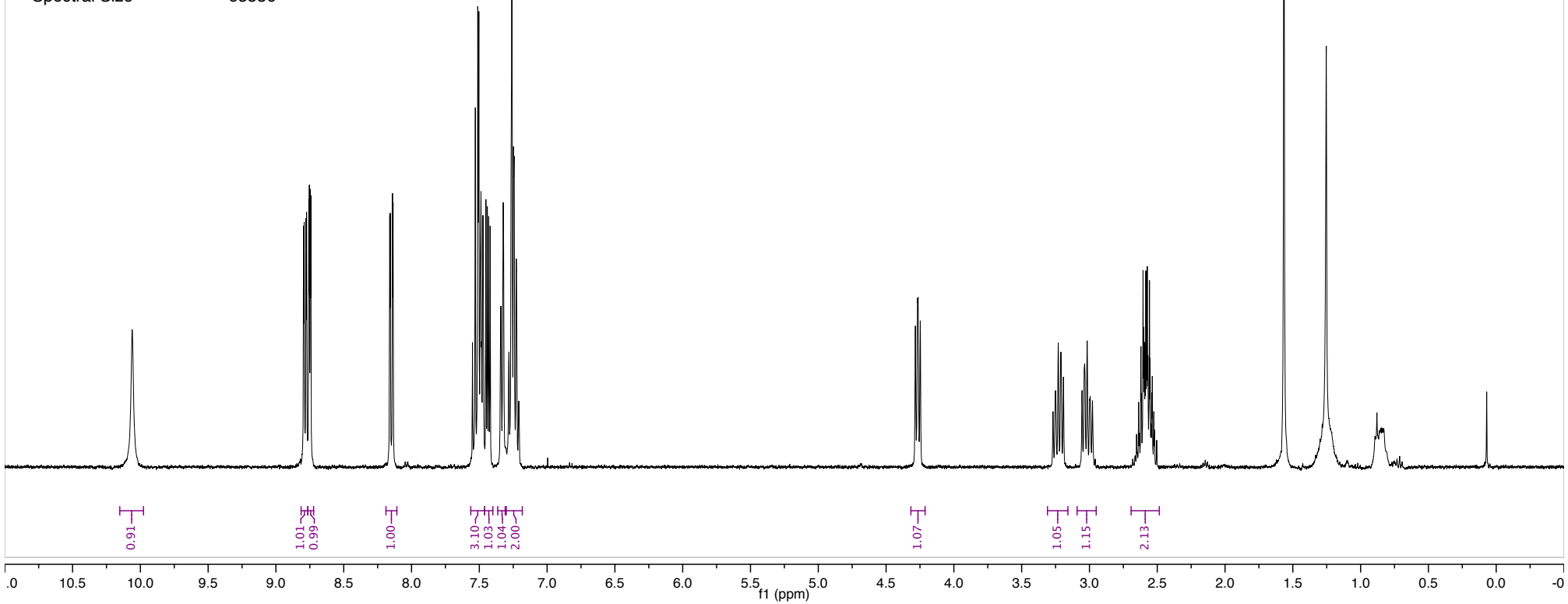
Title	CRL_01_074_3_H.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	1
Receiver Gain	197
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-04-19T22:10:20
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



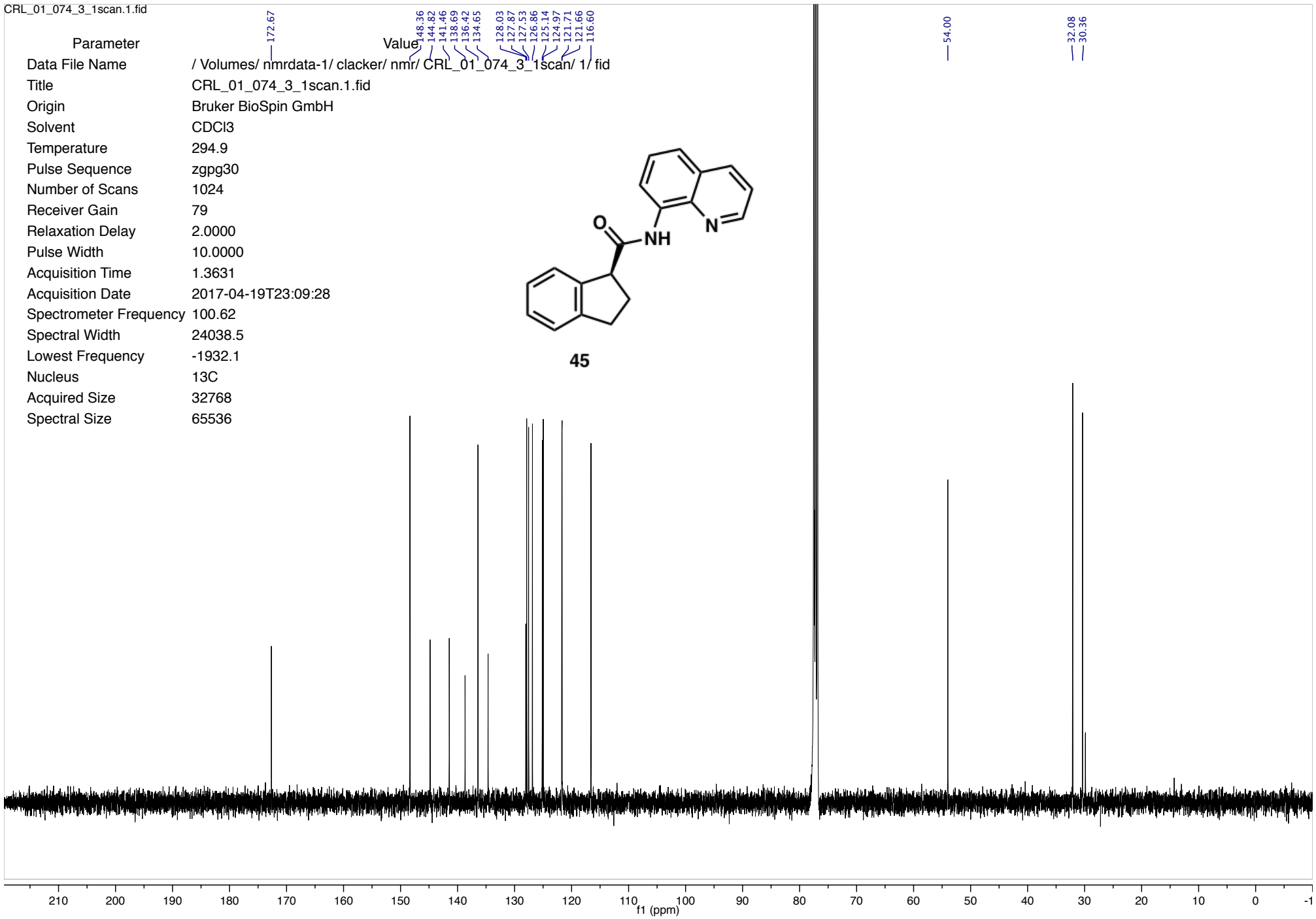
45



CRL_01_074_3_1scan.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata-1/ clacker/ nmr/ CRL_01_074_3_1scan/ 1/ fid
Title	CRL_01_074_3_1scan.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	1024
Receiver Gain	79
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-04-19T23:09:28
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1932.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

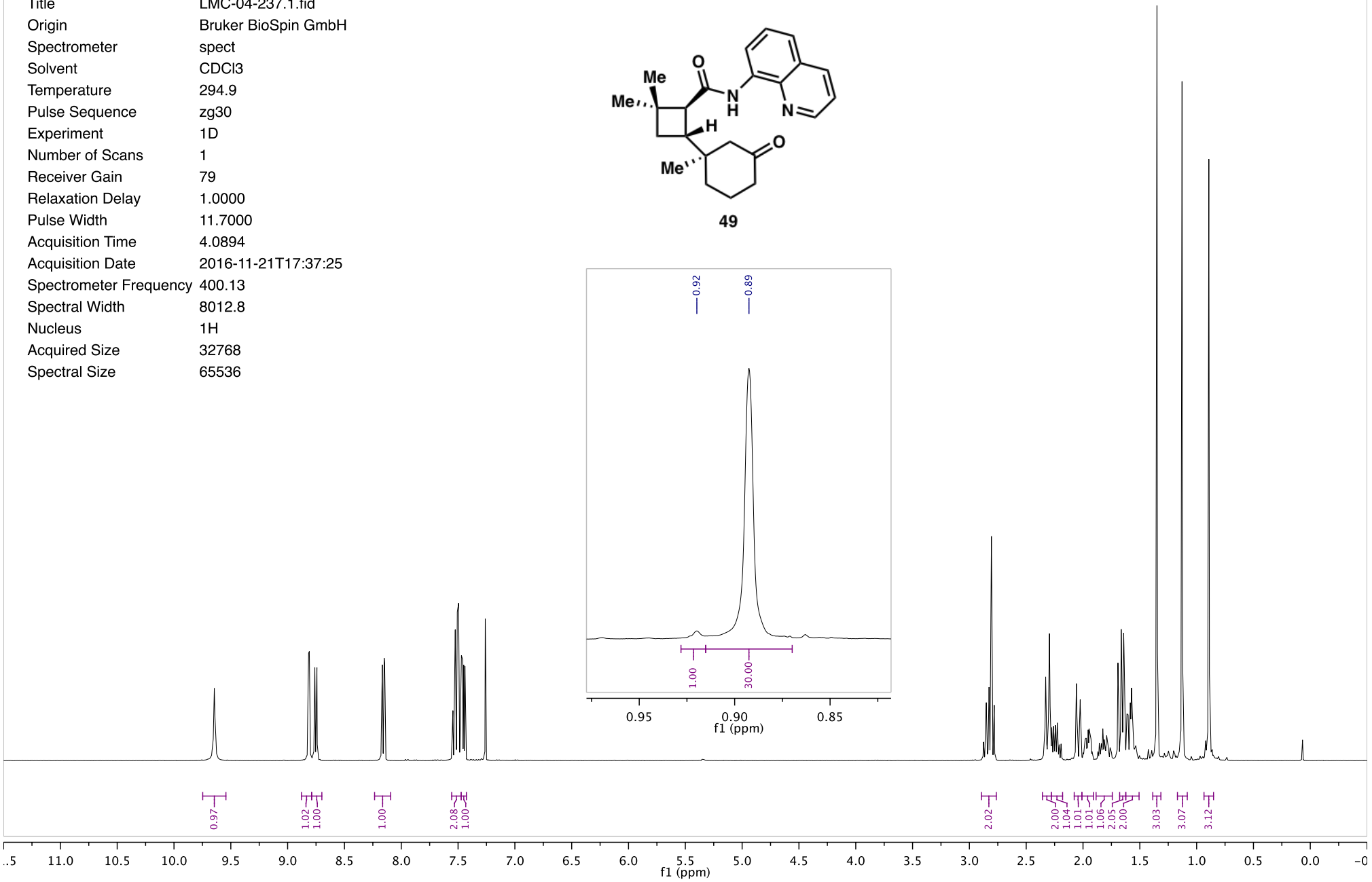
45



LMC-04-237.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-237/ 1/ fid
Title	LMC-04-237.1.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	1
Receiver Gain	79
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-11-21T17:37:25
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

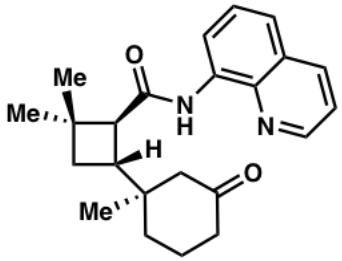
49



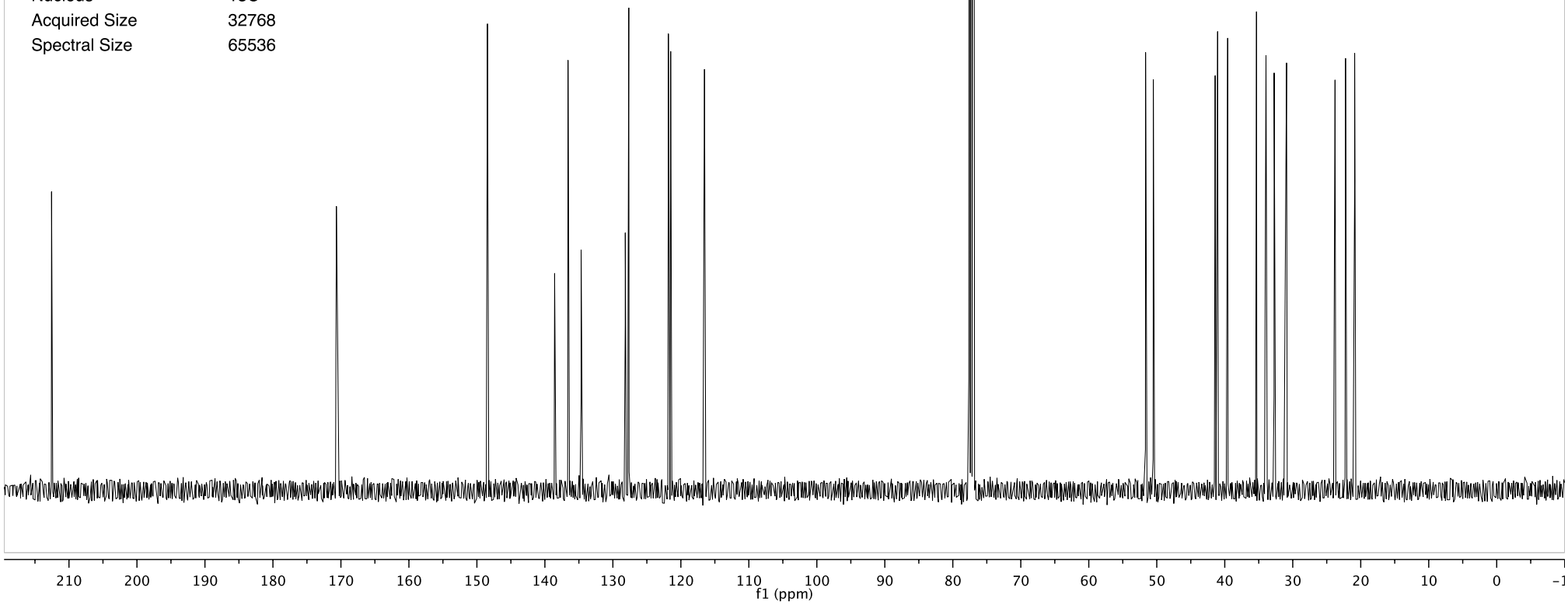
LMC-04-237.3.fid

212.47 170.55 148.33 138.46 136.48 134.56 128.07 127.55 121.72 121.39 116.42 51.53 50.40 41.30 40.94 39.46 35.22 33.82 32.63 30.80 23.67 22.13 20.78

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-237/ 3/ fid
Title	LMC-04-237.3.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	72
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-11-21T17:50:45
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



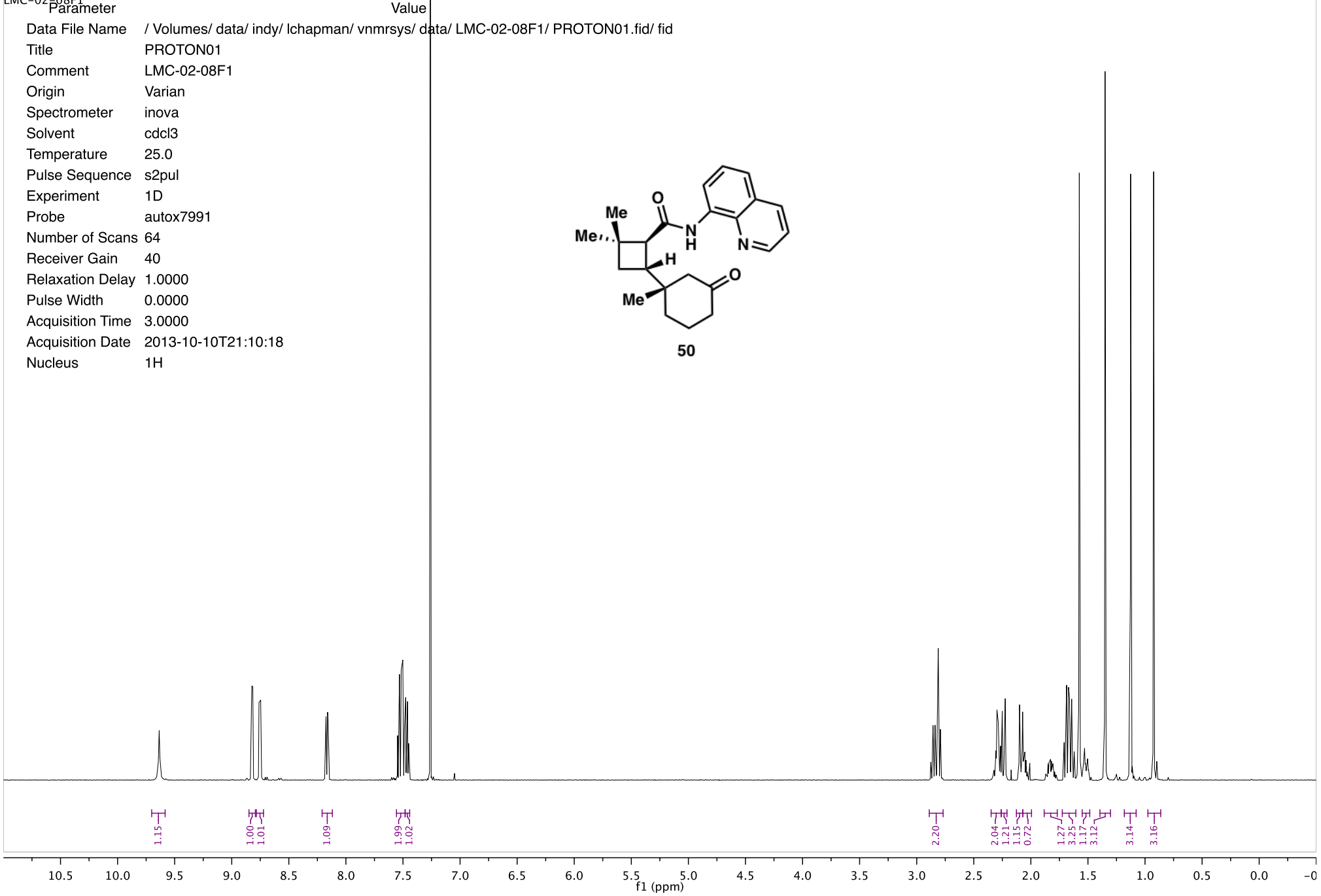
49



PROTON01
LMC-02-08F1

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ lchapman/ vnmrsys/ data/ LMC-02-08F1/ PROTON01.fid/ fid
Title	PROTON01
Comment	LMC-02-08F1
Origin	Varian
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Experiment	1D
Probe	autox7991
Number of Scans	64
Receiver Gain	40
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	3.0000
Acquisition Date	2013-10-10T21:10:18
Nucleus	1H

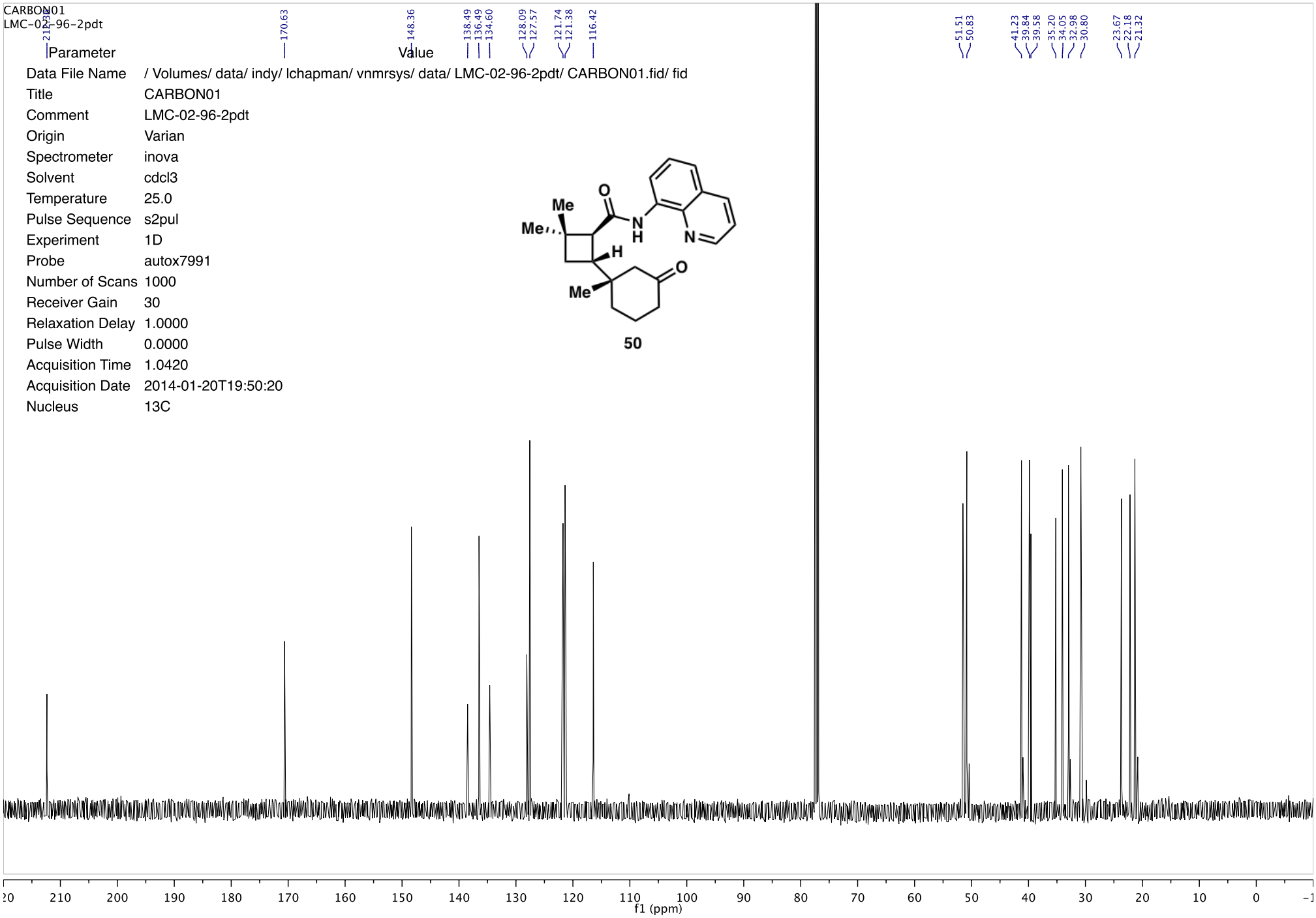
50



CARBON01
LMC-02-96-2pdt

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ lchapman/ vnmrsys/ data/ LMC-02-96-2pdt/ CARBON01.fid/ fid
Title	CARBON01
Comment	LMC-02-96-2pdt
Origin	Varian
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Experiment	1D
Probe	autox7991
Number of Scans	1000
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	1.0420
Acquisition Date	2014-01-20T19:50:20
Nucleus	13C

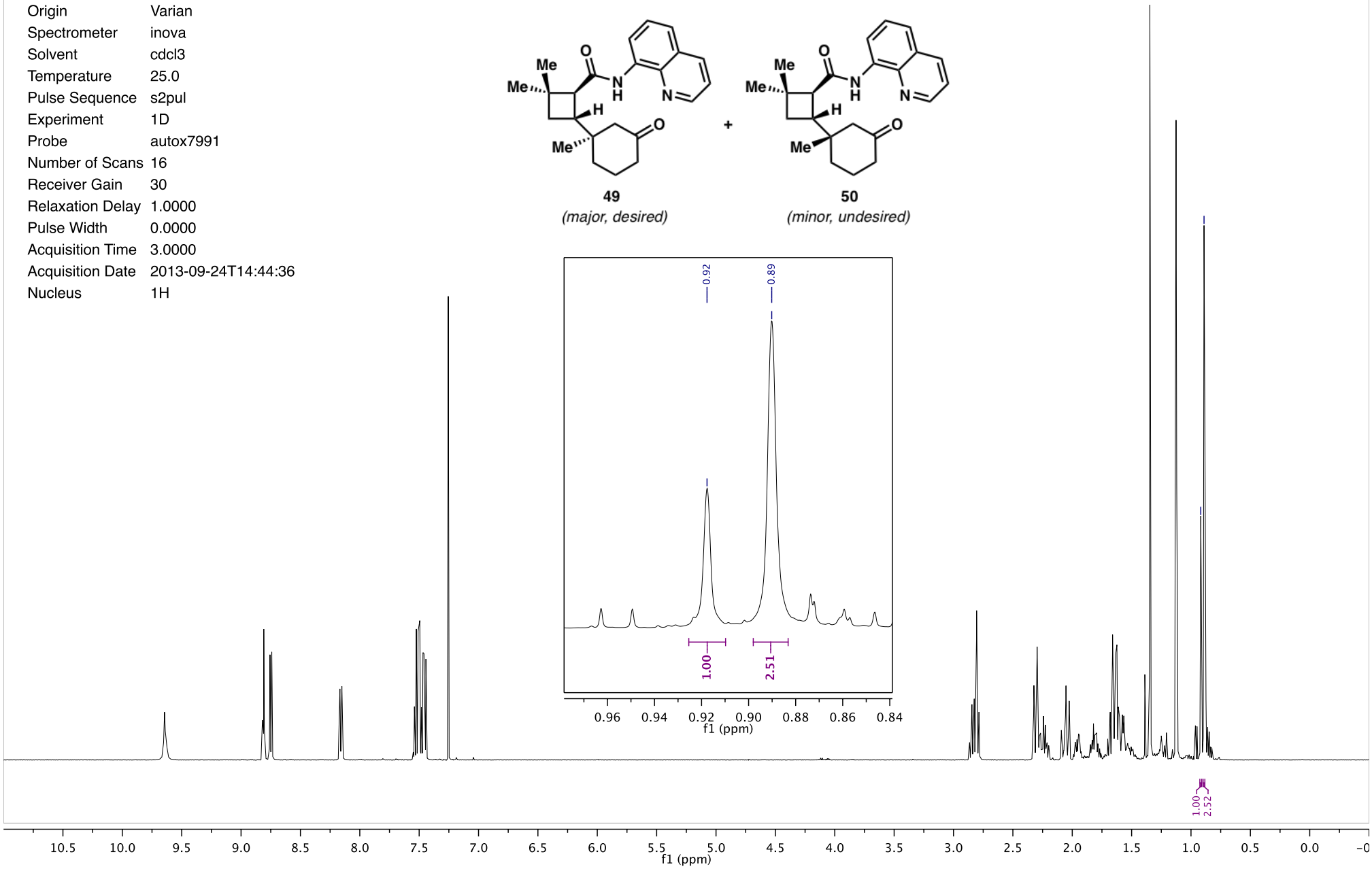
50



PROTON01

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ lchapman/ vnmrsys/ data/ LMC-02-08/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Spectrometer	inova
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Experiment	1D
Probe	autox7991
Number of Scans	16
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	3.0000
Acquisition Date	2013-09-24T14:44:36
Nucleus	1H

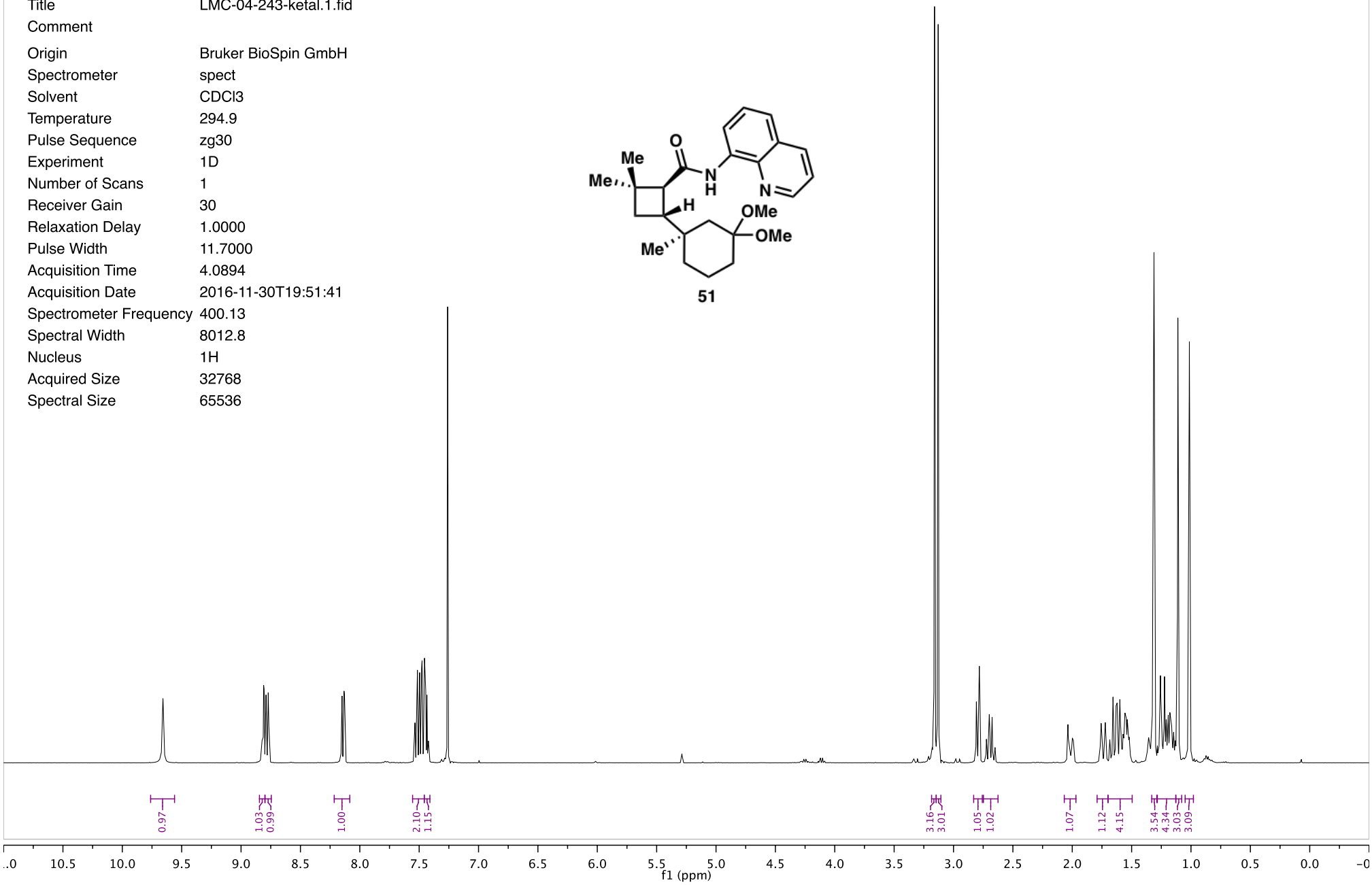
49 *(major, desired)* + **50** *(minor, undesired)*



LMC-04-243-ketal.1.fid

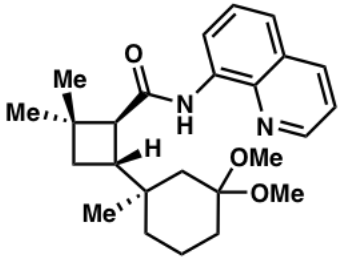
Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-243-ketal/ 1/ fid
Title	LMC-04-243-ketal.1.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	1
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-11-30T19:51:41
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

51



LMC-04-243-ketal.2.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-243-ketal/ 2/ fid
Title	LMC-04-243-ketal.2.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	88
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-11-30T19:59:38
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



51

171.04 148.28 138.51 136.40 134.74 128.04 127.56 121.65 121.18 116.36 100.81 51.27 47.86 47.29 42.29 38.58 34.82 34.66 34.02 33.26 32.47 30.75 23.85 21.38 18.79

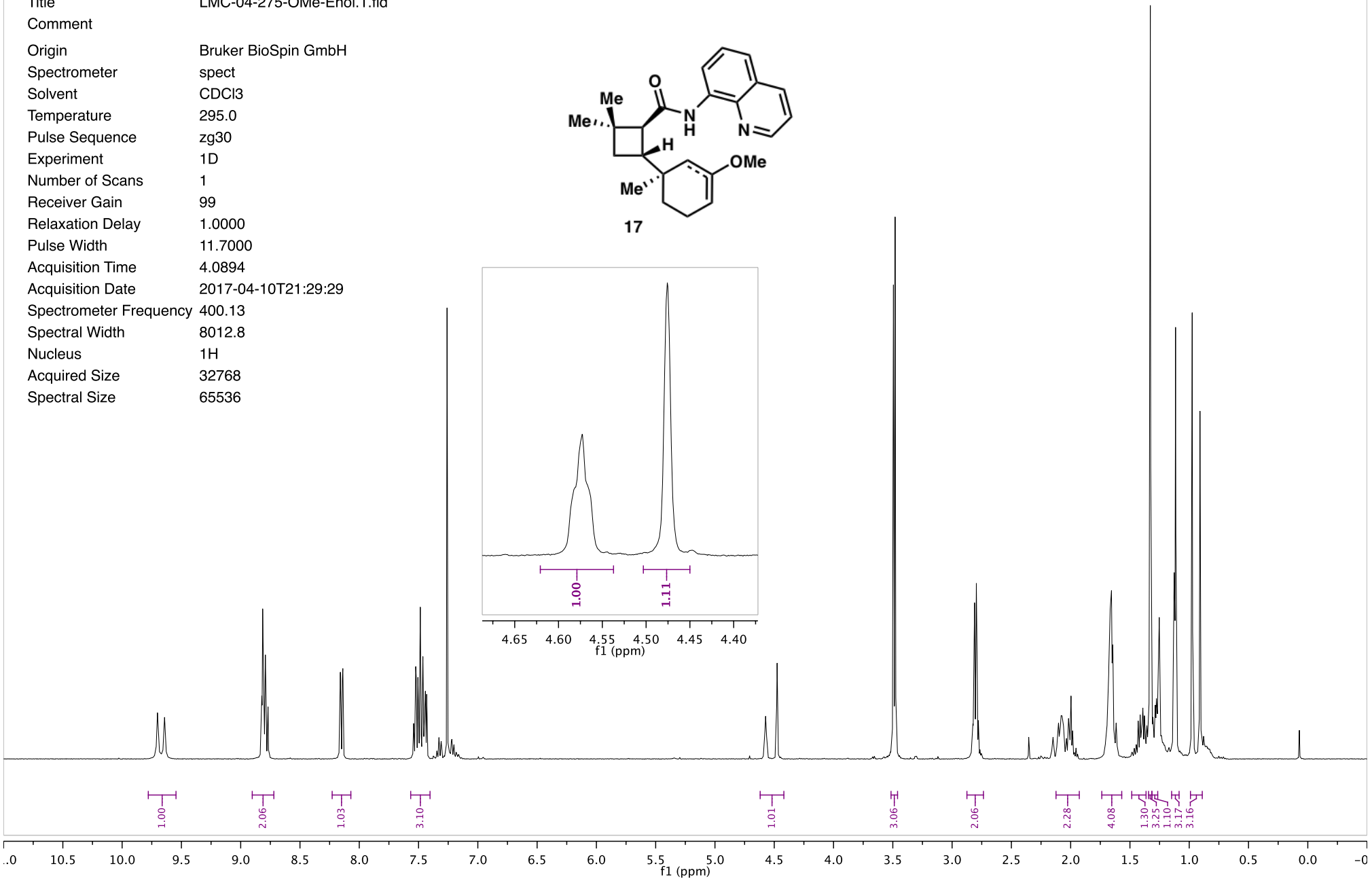
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

f1 (ppm)

LMC-04-275-OMe-Enol.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-275-OMe-Enol/ 1/ fid
Title	LMC-04-275-OMe-Enol.1.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	1
Receiver Gain	99
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-04-10T21:29:29
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

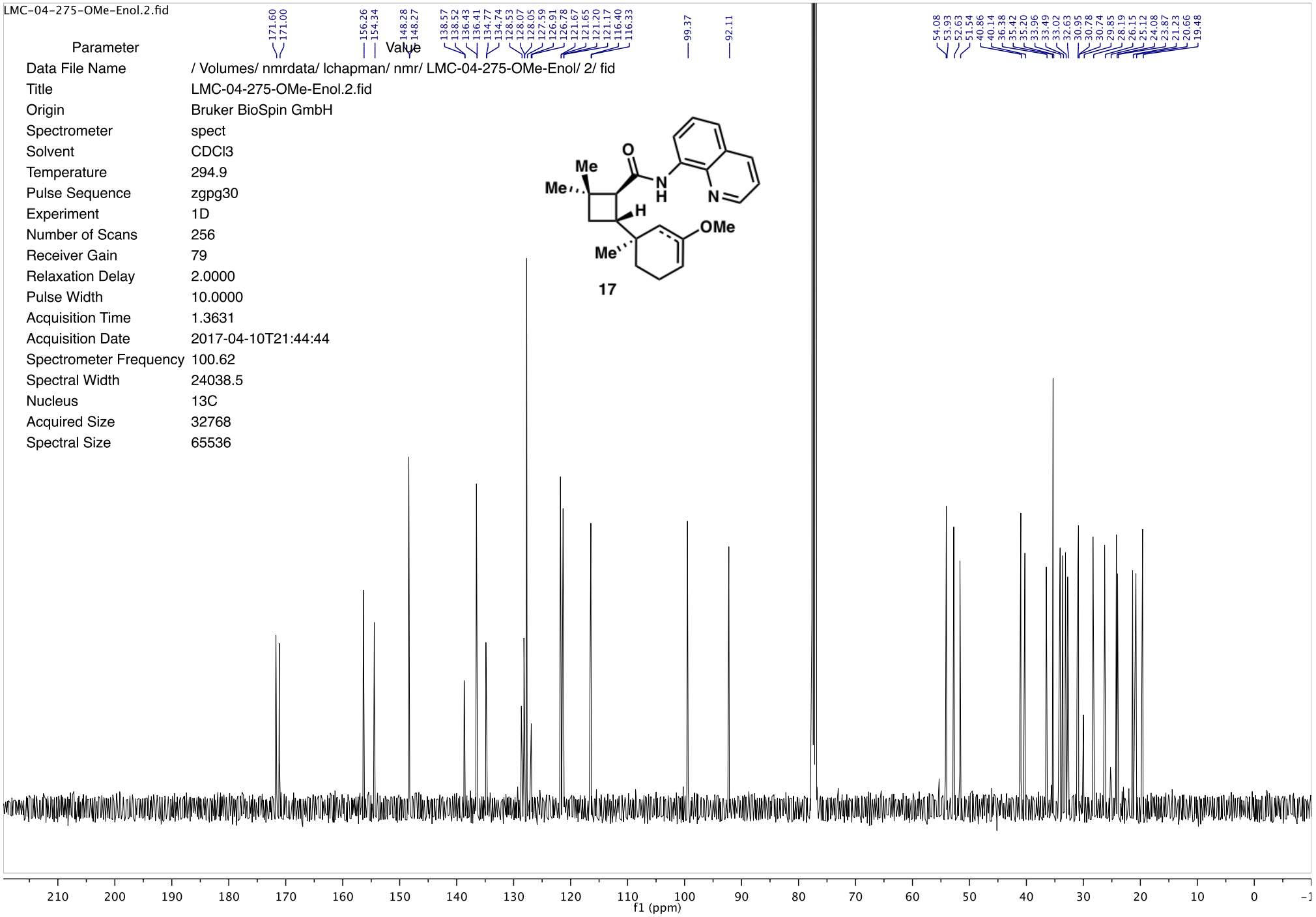
17



LMC-04-275-OMe-Enol.2.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-275-OMe-Enol/ 2/ fid
Title	LMC-04-275-OMe-Enol.2.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	256
Receiver Gain	79
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-04-10T21:44:44
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

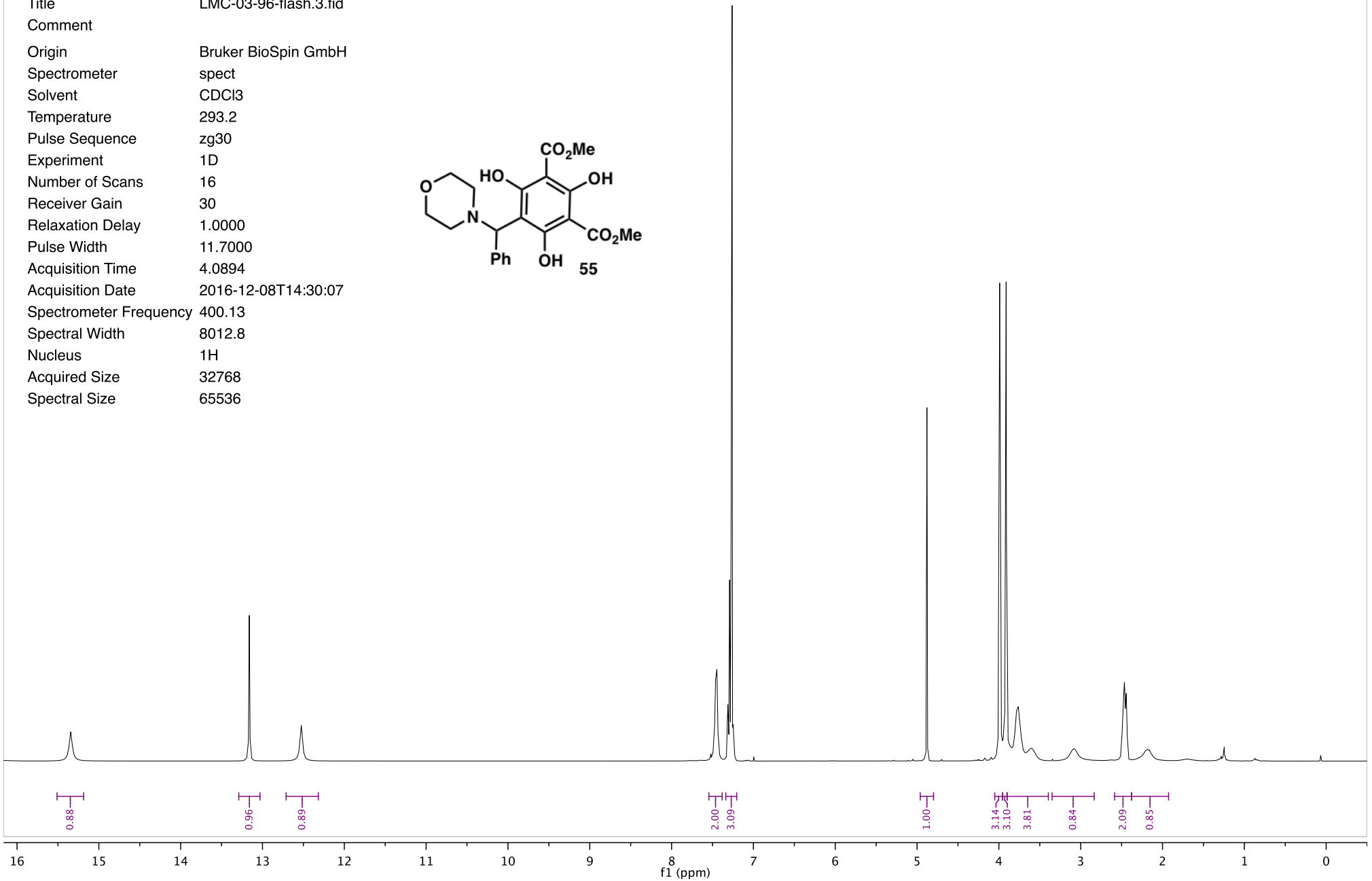
17



LMC-03-96-flash.3.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-03-96-flash/ 3/ fid
Title	LMC-03-96-flash.3.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	293.2
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-12-08T14:30:07
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

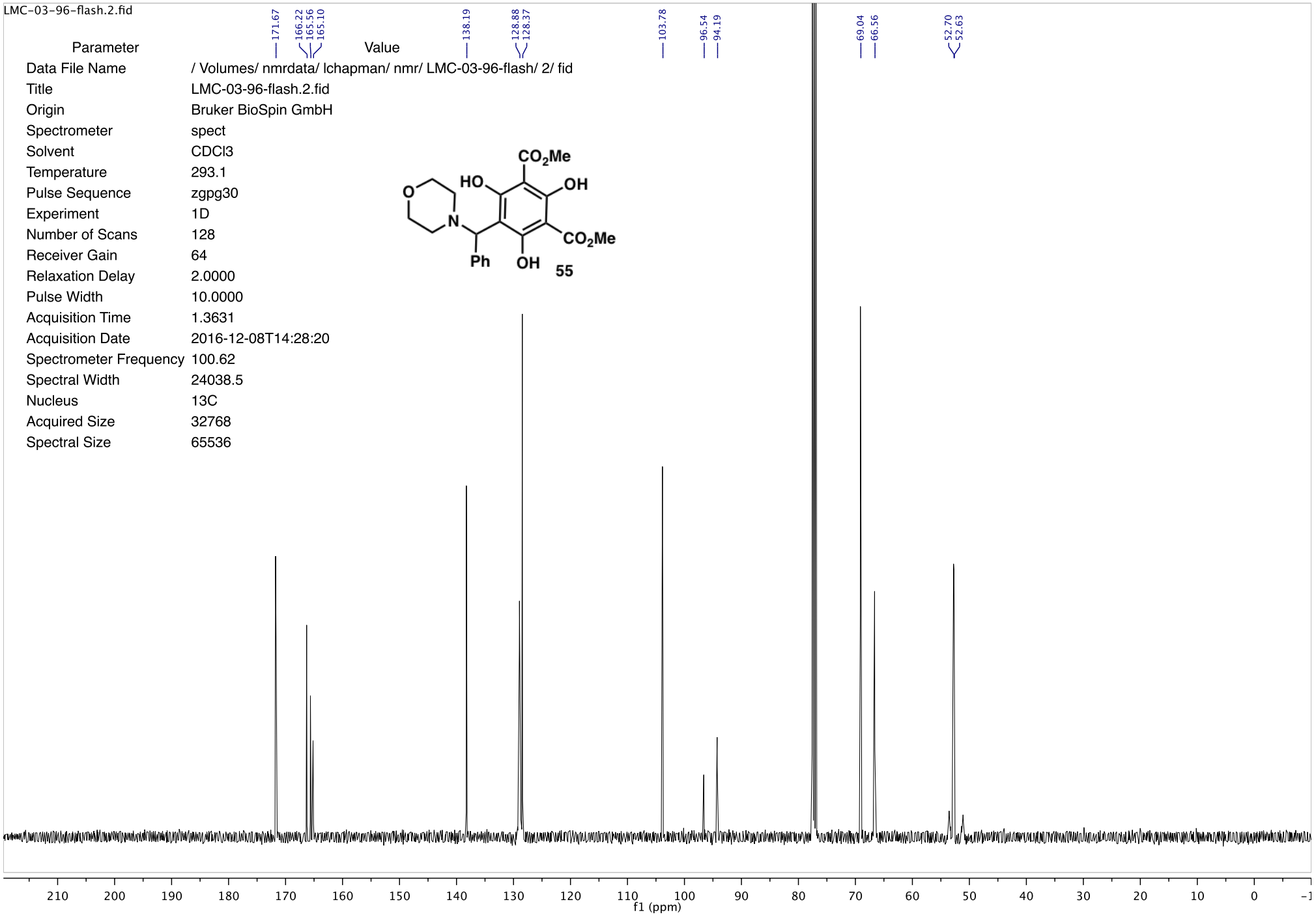
55



LMC-03-96-flash.2.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-03-96-flash/ 2/ fid
Title	LMC-03-96-flash.2.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	293.1
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	64
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-08T14:28:20
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

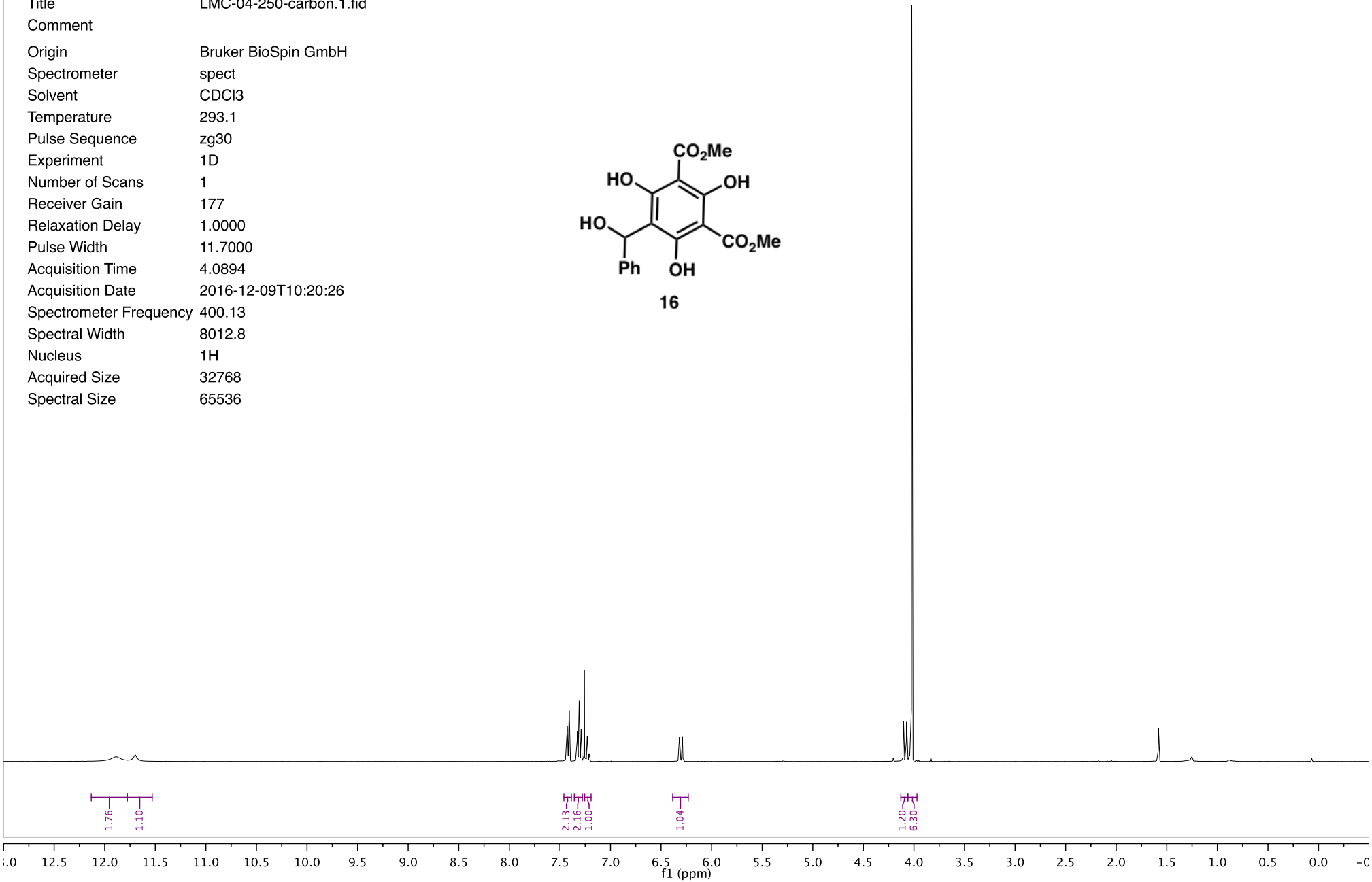
55



LMC-04-250-carbon.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-250-carbon/ 1/ fid
Title	LMC-04-250-carbon.1.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	293.1
Pulse Sequence	zg30
Experiment	1D
Number of Scans	1
Receiver Gain	177
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-12-09T10:20:26
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

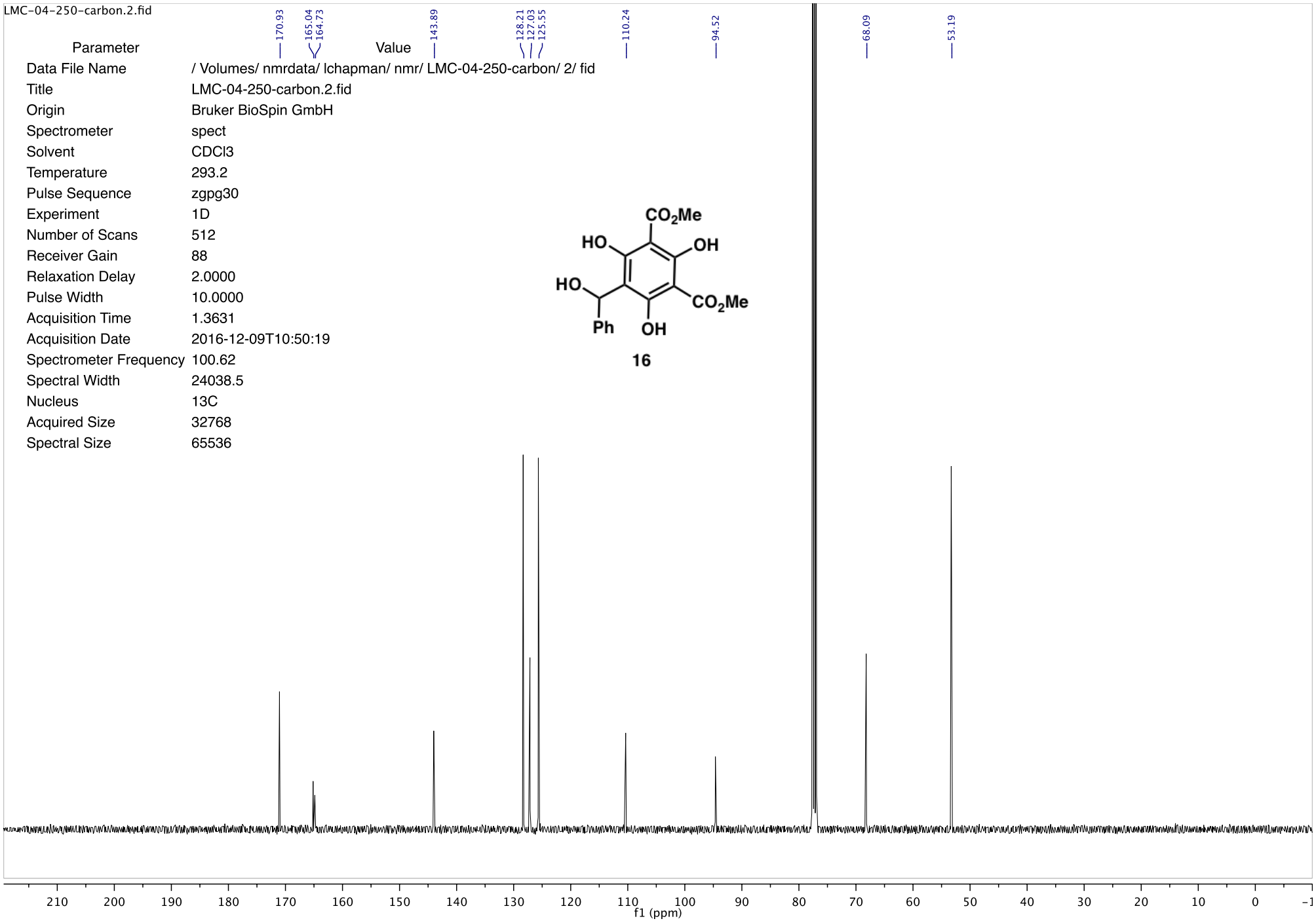
16



LMC-04-250-carbon.2.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-250-carbon/ 2/ fid
Title	LMC-04-250-carbon.2.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	293.2
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	512
Receiver Gain	88
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-09T10:50:19
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

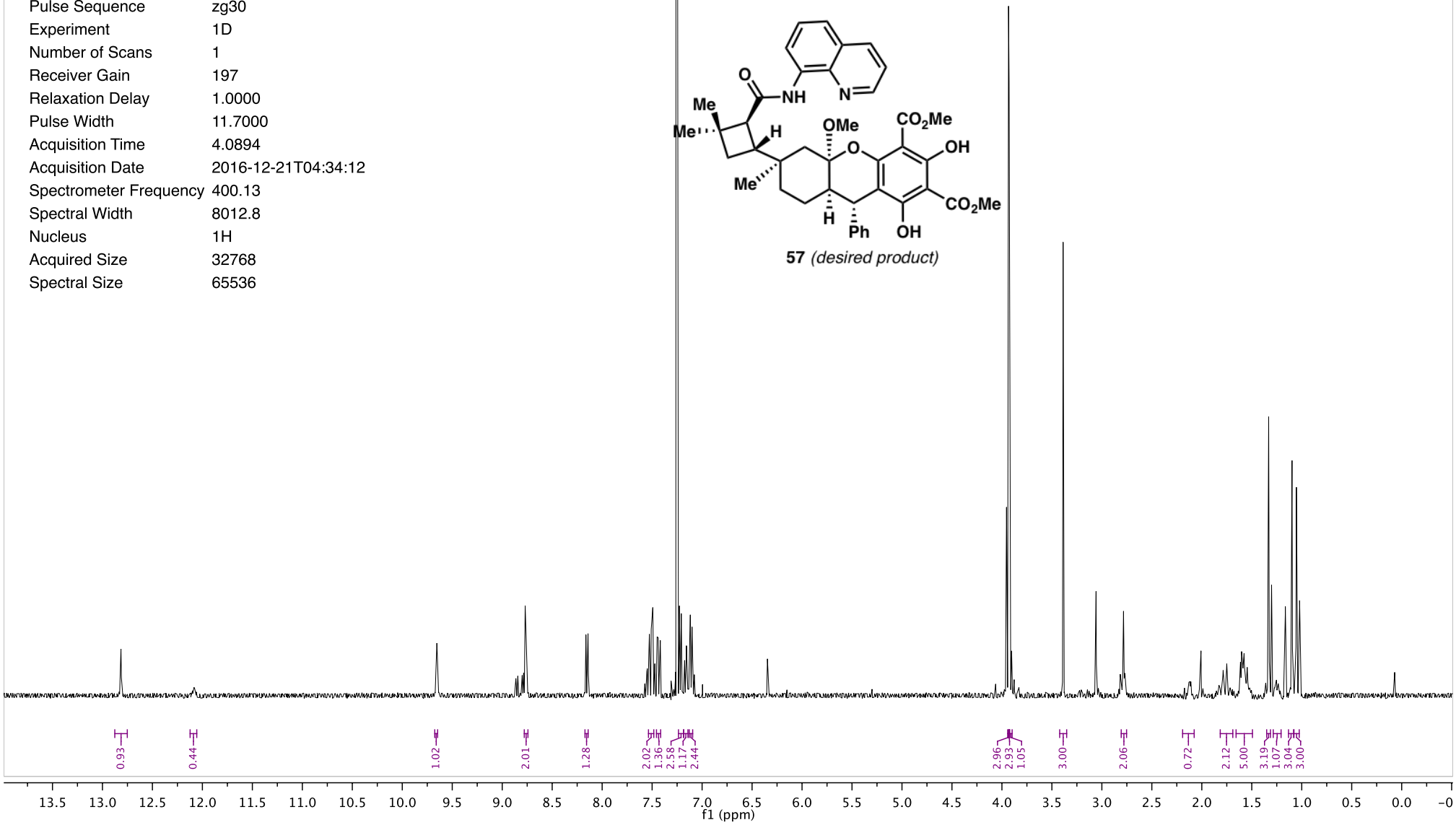
16



LMC-04-253-HPLC-P2.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-253-HPLC-P2/ 1/ fid
Title	LMC-04-253-HPLC-P2.1.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	1
Receiver Gain	197
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-12-21T04:34:12
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

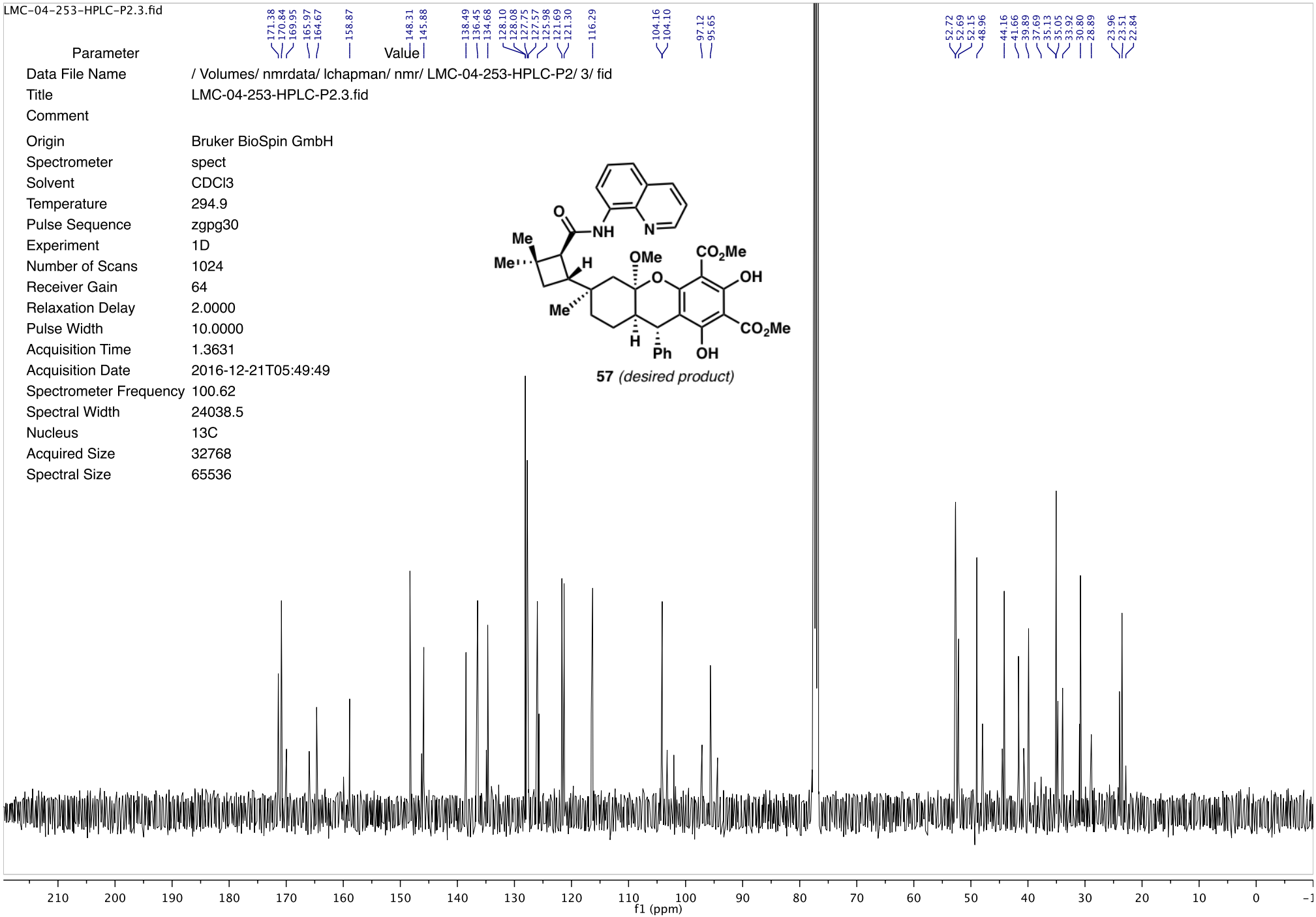
57 *(desired product)*

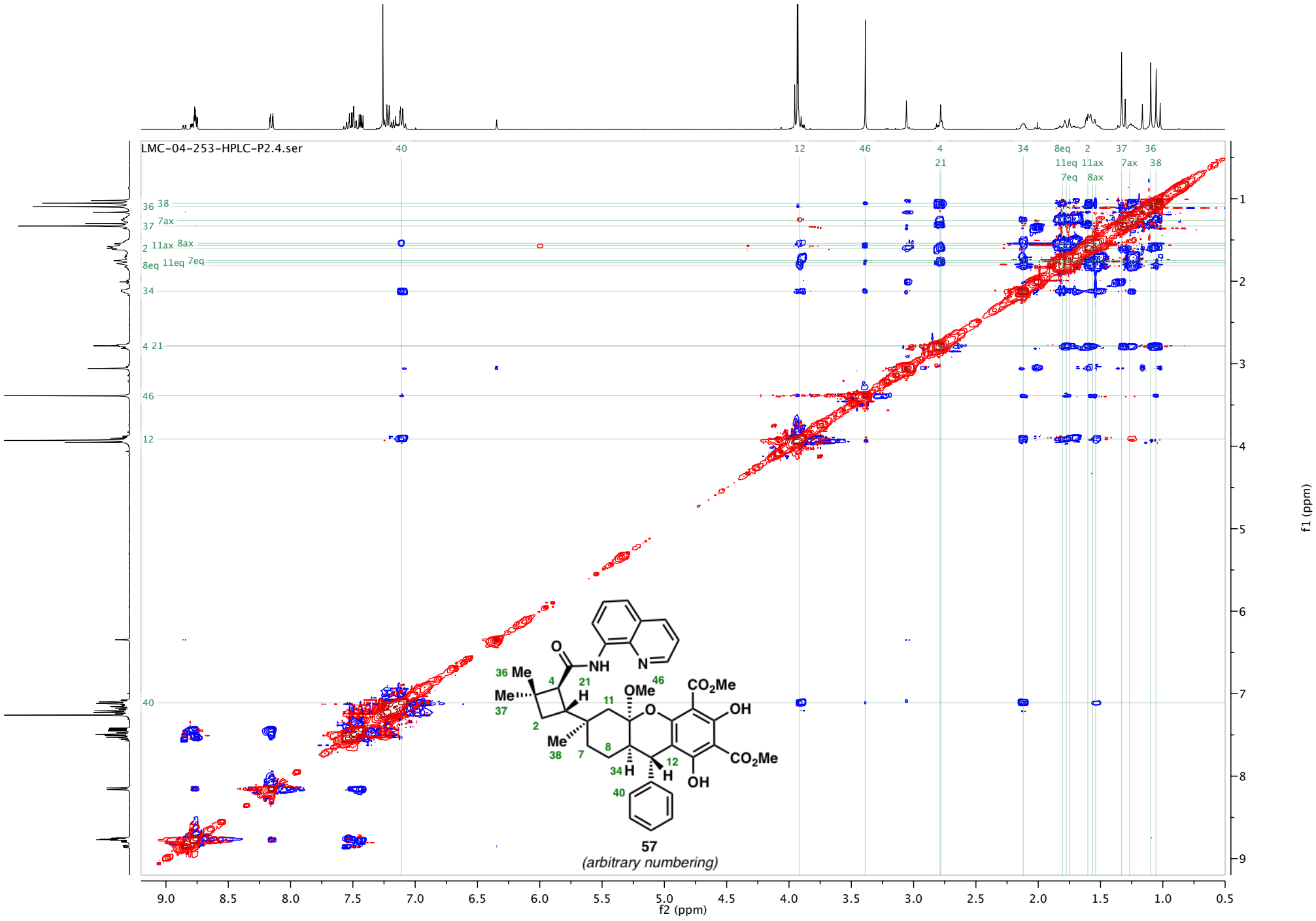


LMC-04-253-HPLC-P2.3.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-253-HPLC-P2/ 3/ fid
Title	LMC-04-253-HPLC-P2.3.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	1024
Receiver Gain	64
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-21T05:49:49
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

57 *(desired product)*

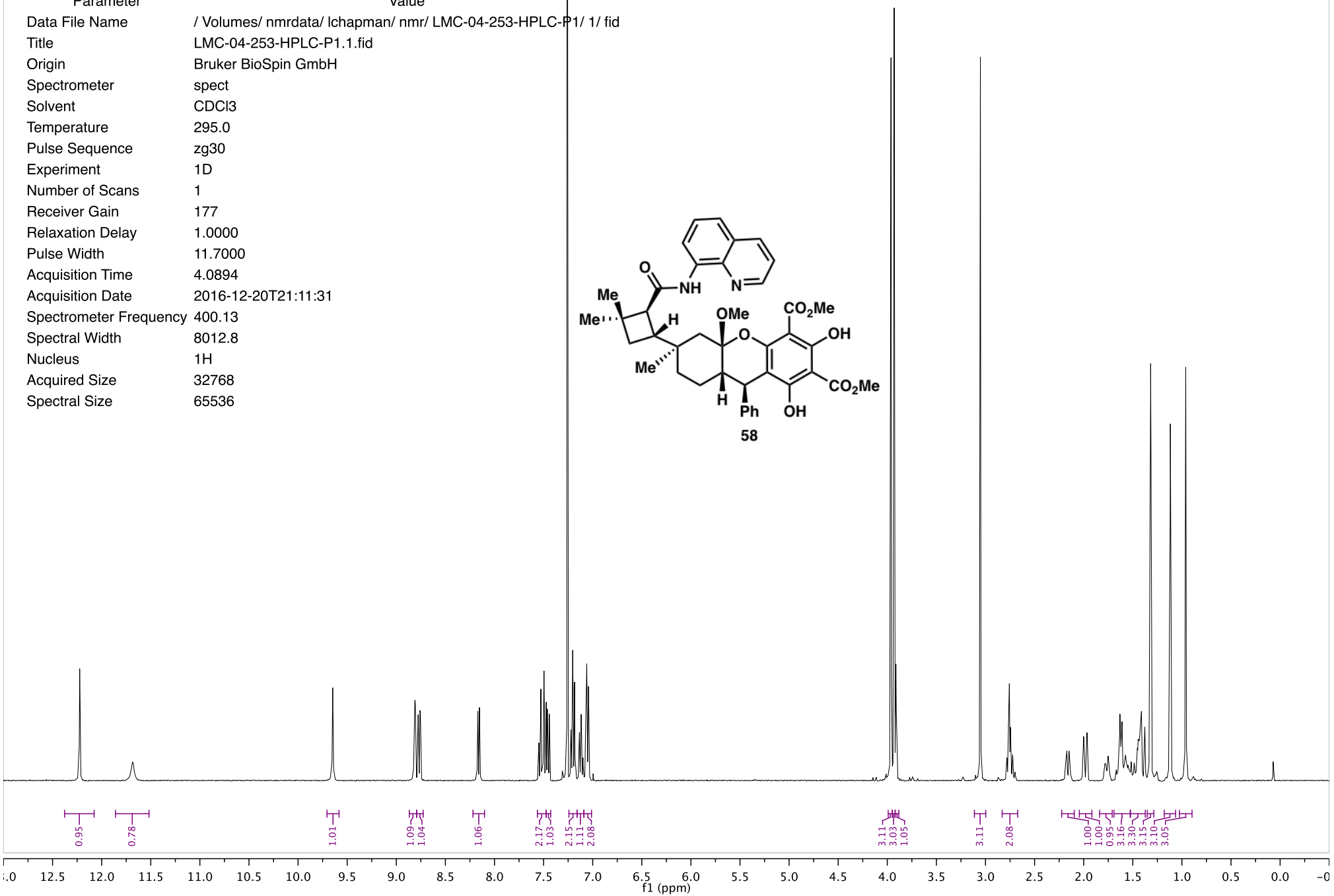



LMC-04-253-HPLC-P2.4.ser
f2 (ppm)
f1 (ppm)
57
(arbitrary numbering)

LMC-04-253-HPLC-P1.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-253-HPLC-P1/ 1/ fid
Title	LMC-04-253-HPLC-P1.1.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	1
Receiver Gain	177
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-12-20T21:11:31
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

58

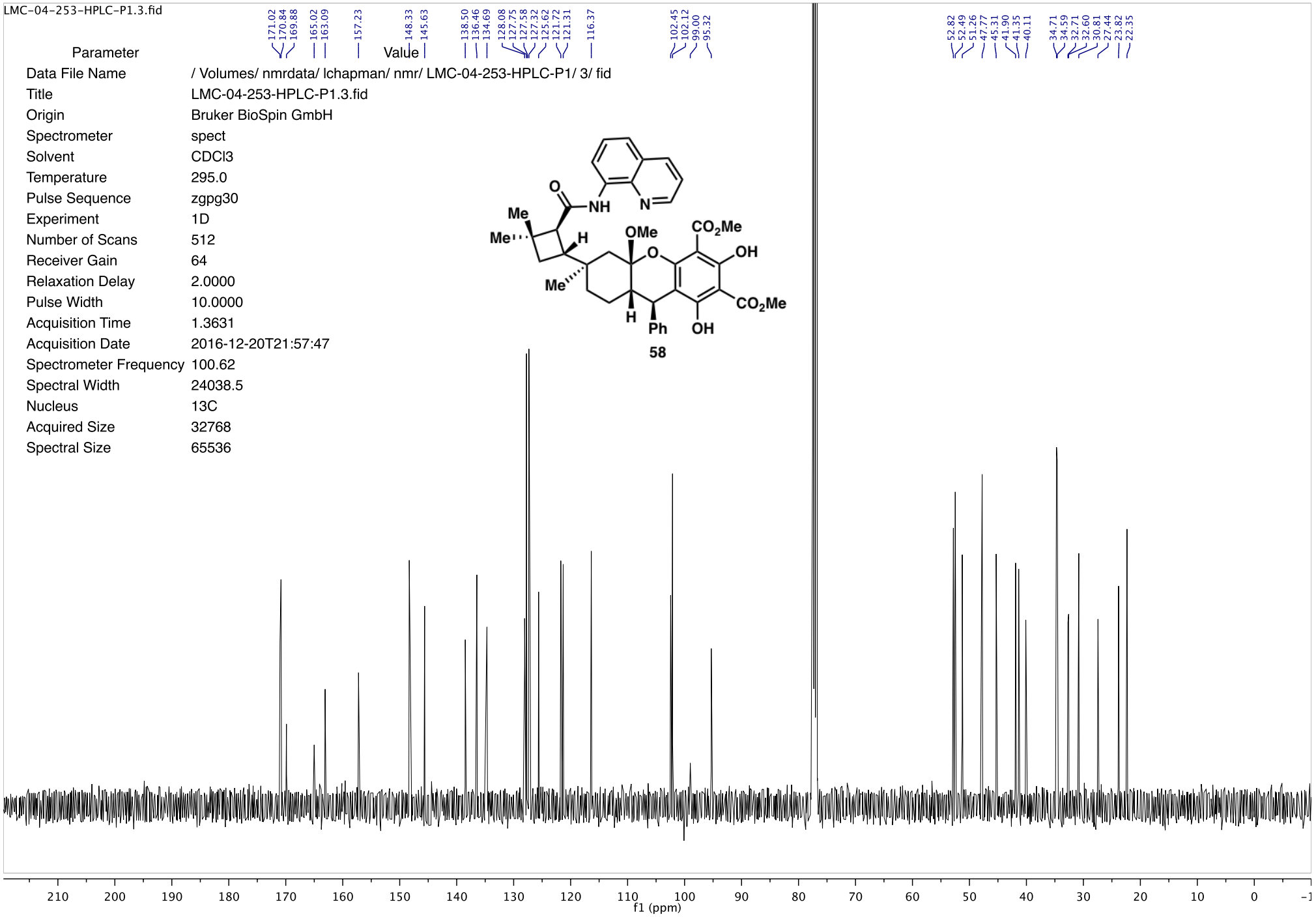


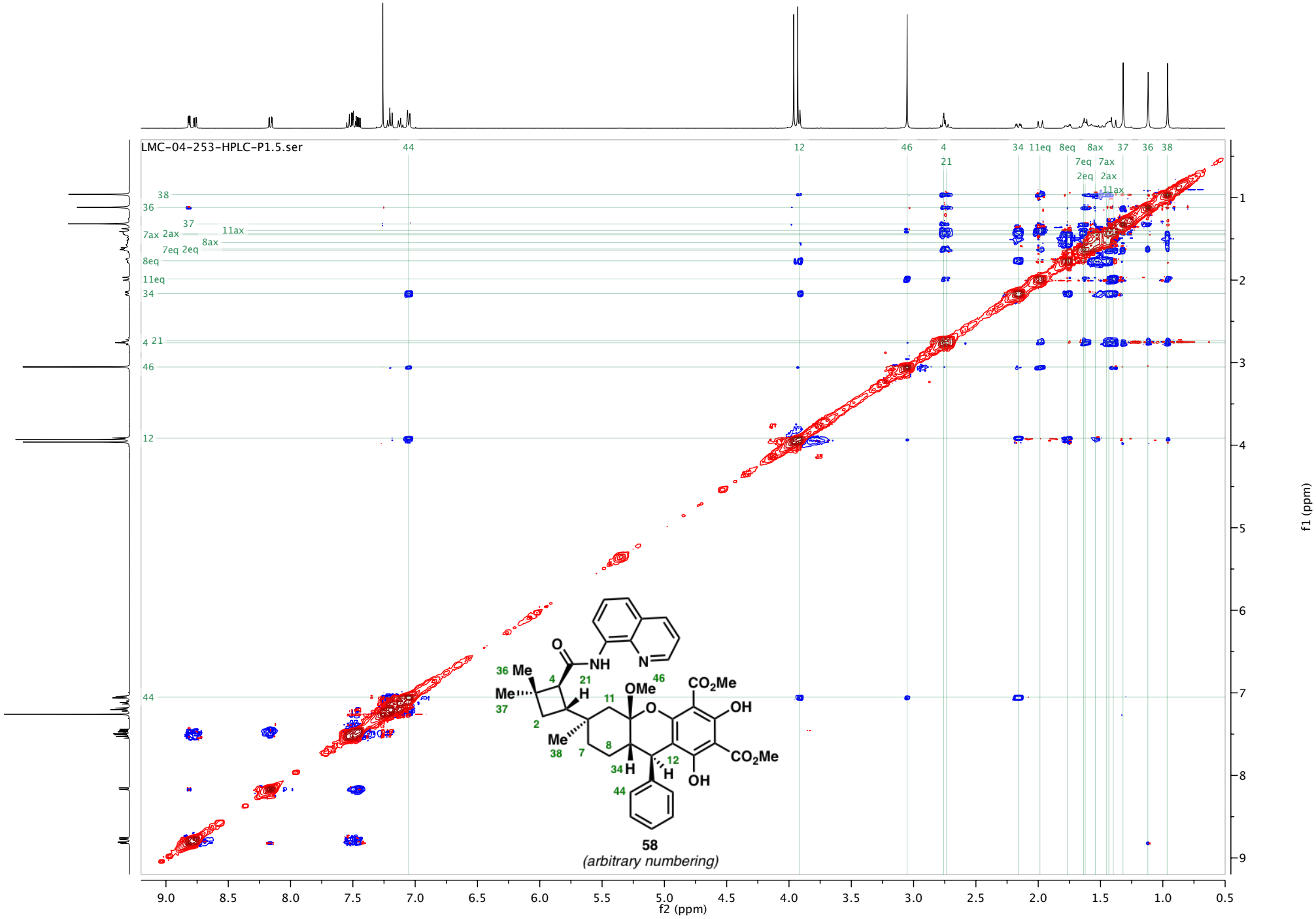
LMC-04-253-HPLC-P1.3.fid

171.02 170.84 169.88 165.02 163.09 157.23 148.33 145.63 138.50 136.46 134.69 128.08 127.75 127.58 127.32 125.62 121.72 121.31 116.37 102.45 102.12 99.00 95.32 52.82 52.49 51.26 47.77 45.31 41.90 41.35 40.11 34.71 34.59 32.71 32.60 30.81 27.44 23.82 22.35

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-253-HPLC-P1/ 3/ fid
Title	LMC-04-253-HPLC-P1.3.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	512
Receiver Gain	64
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-20T21:57:47
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

58

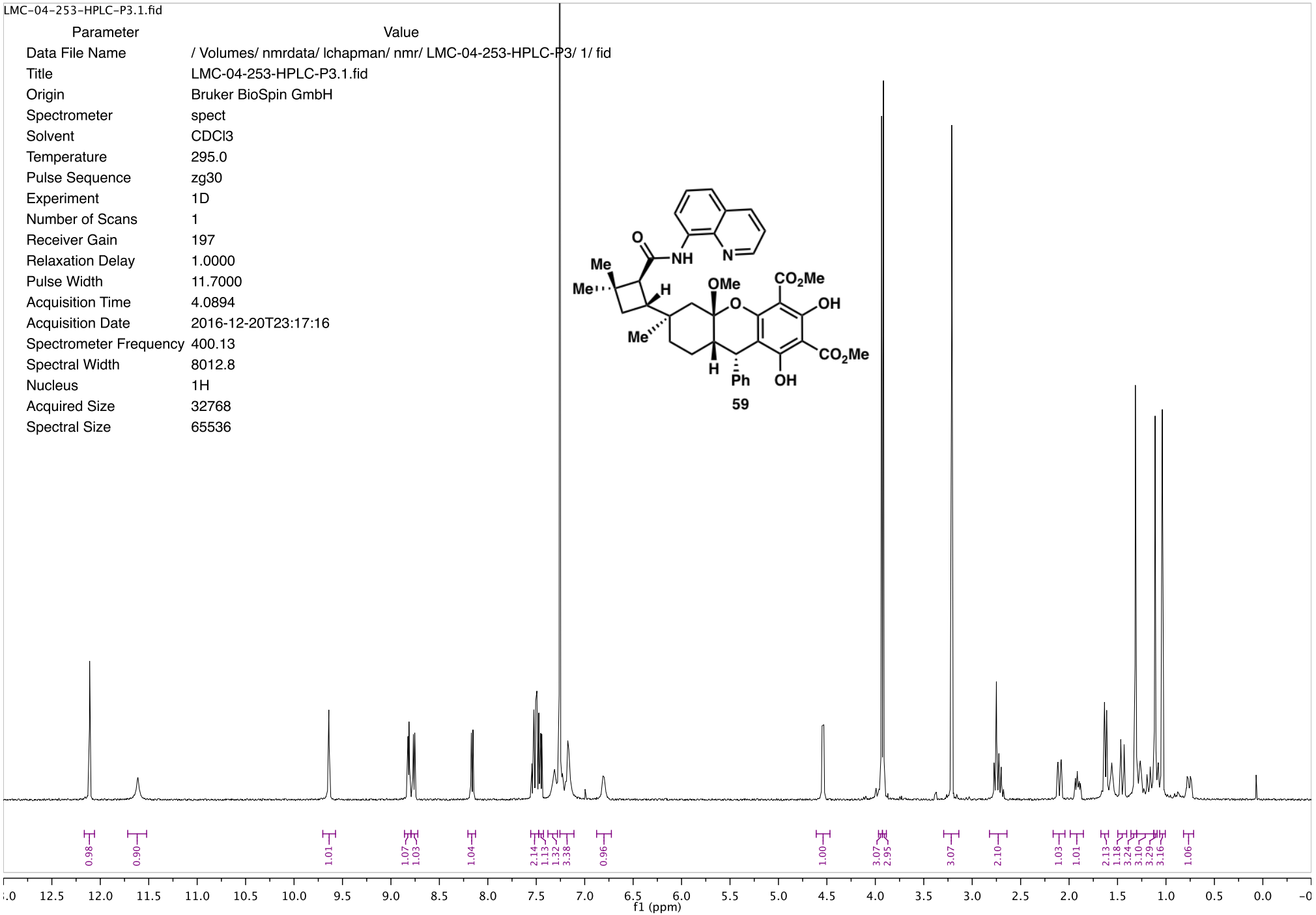



LMC-04-253-HPLC-P1.5.ser
f2 (ppm)
f1 (ppm)
58
(arbitrary numbering)

LMC-04-253-HPLC-P3.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-253-HPLC-P3/ 1/ fid
Title	LMC-04-253-HPLC-P3.1.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	1
Receiver Gain	197
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-12-20T23:17:16
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

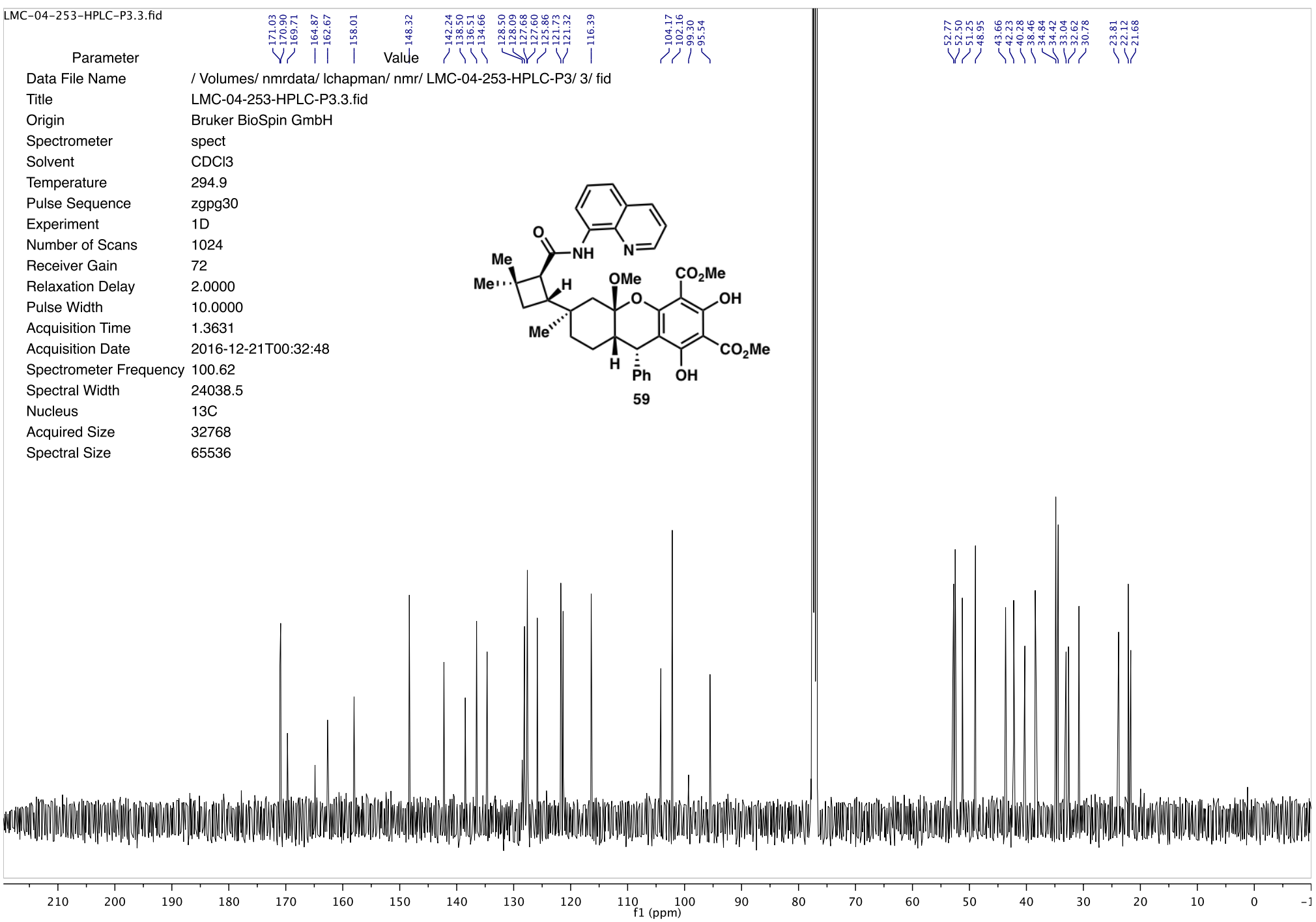
59

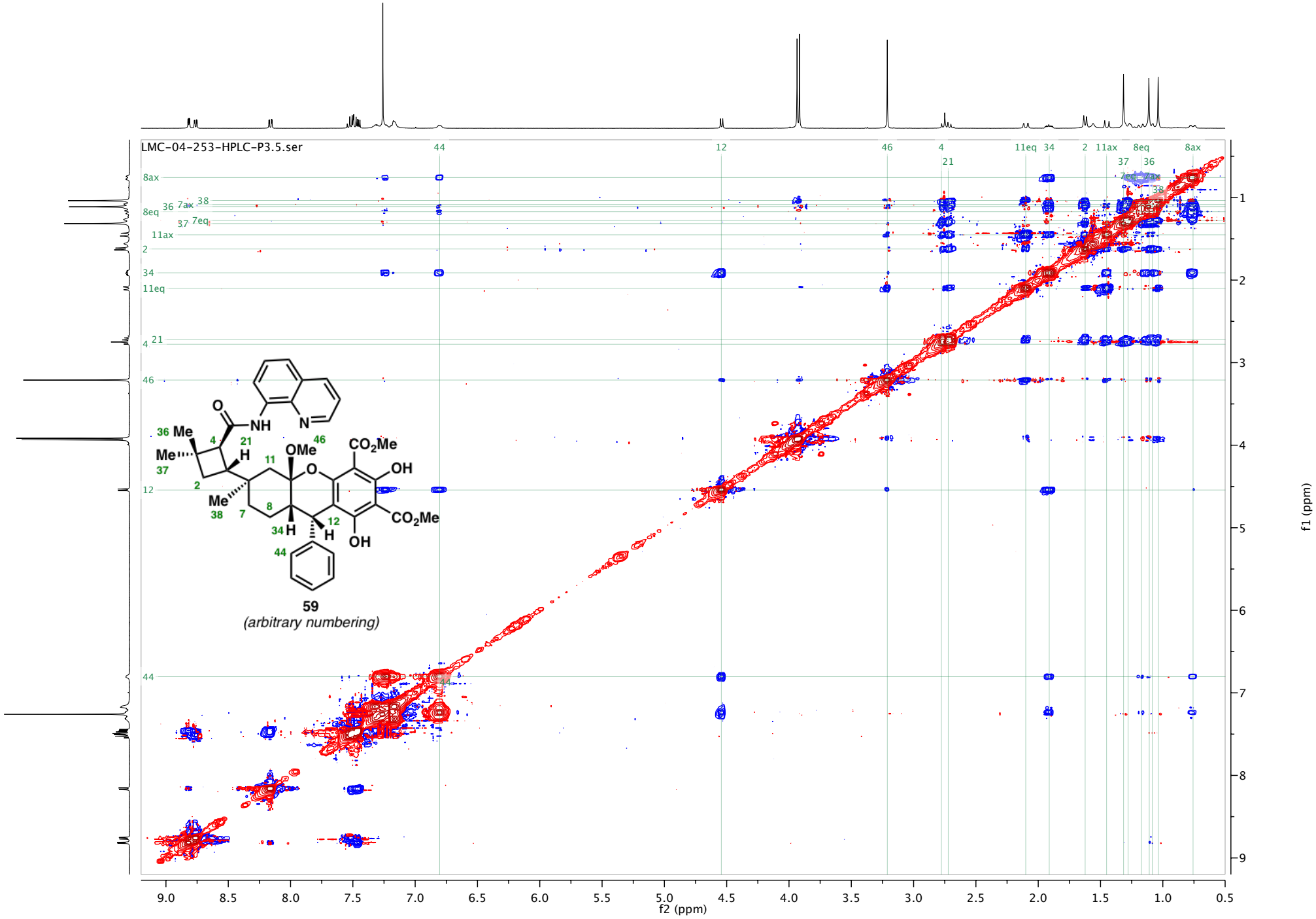


LMC-04-253-HPLC-P3.3.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-253-HPLC-P3/ 3/ fid
Title	LMC-04-253-HPLC-P3.3.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	1024
Receiver Gain	72
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-21T00:32:48
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

59

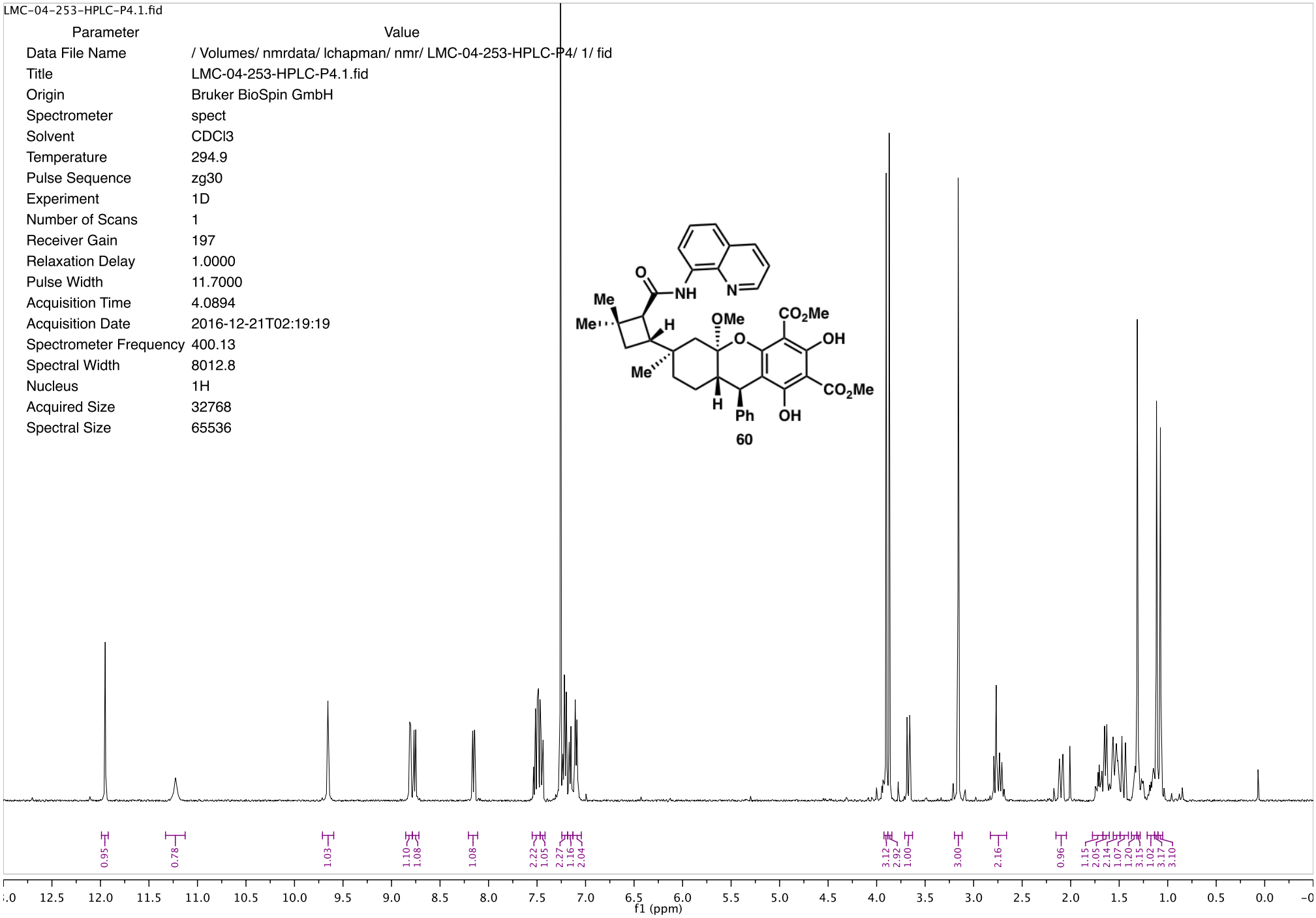



LMC-04-253-HPLC-P3.5.ser
f2 (ppm)
f1 (ppm)
59
(arbitrary numbering)

LMC-04-253-HPLC-P4.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-253-HPLC-P4/ 1/ fid
Title	LMC-04-253-HPLC-P4.1.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	1
Receiver Gain	197
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-12-21T02:19:19
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

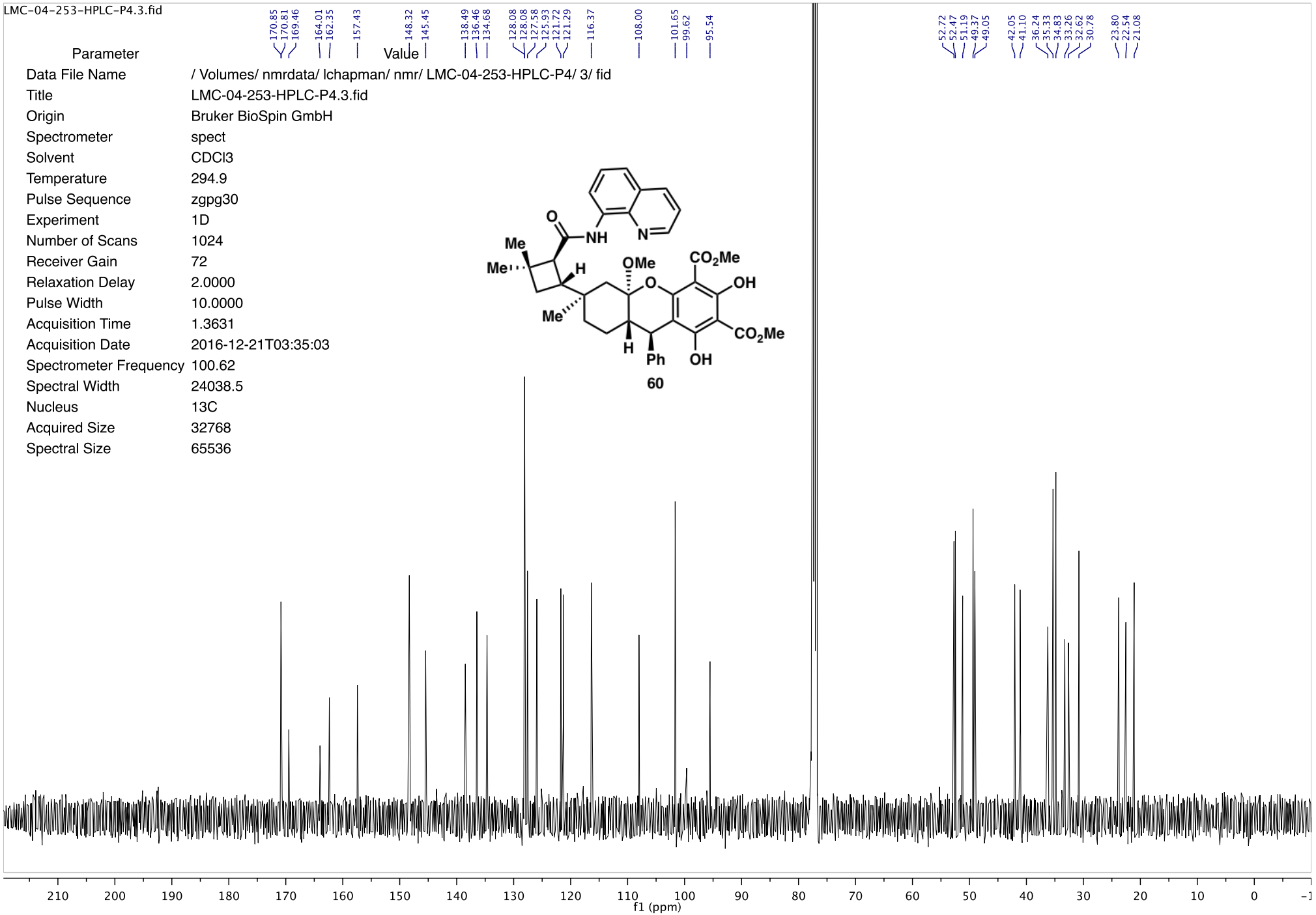
60

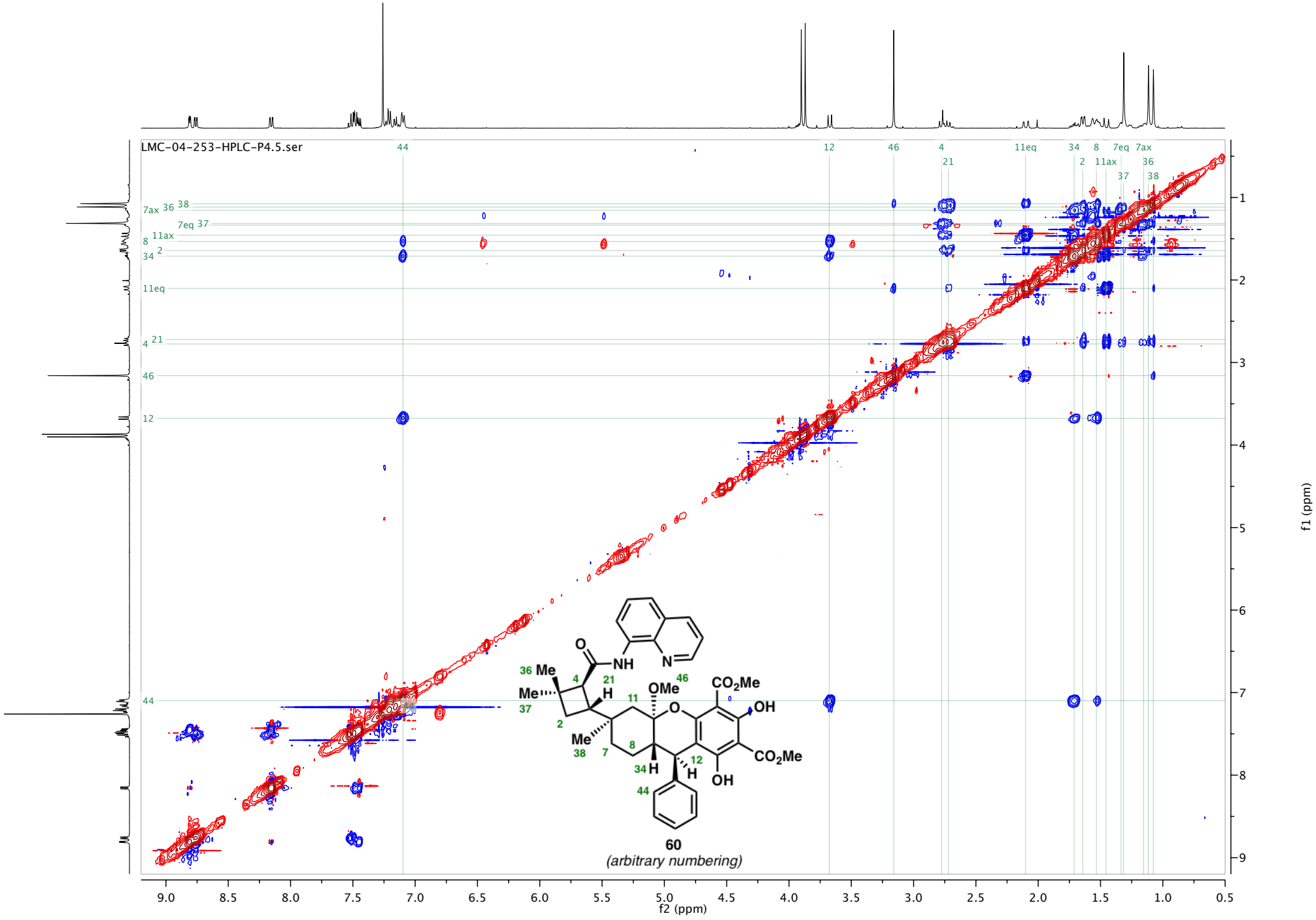


LMC-04-253-HPLC-P4.3.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-253-HPLC-P4/ 3/ fid
Title	LMC-04-253-HPLC-P4.3.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	1024
Receiver Gain	72
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-21T03:35:03
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

60

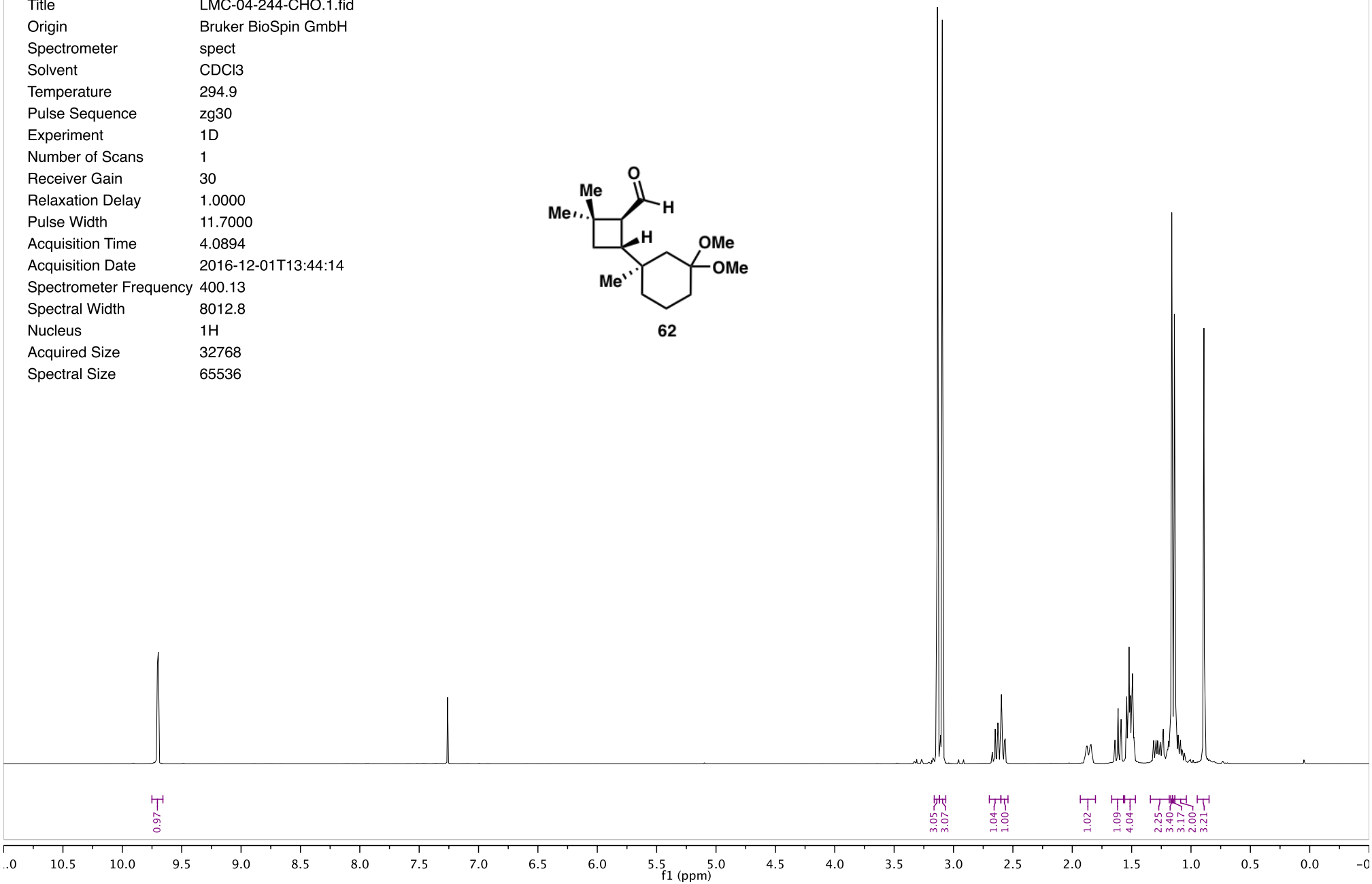



LMC-04-253-HPLC-P4.5.ser
f2 (ppm)
f1 (ppm)
60
(arbitrary numbering)

LMC-04-244-CHO.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-244-CHO/ 1/ fid
Title	LMC-04-244-CHO.1.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	1
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-12-01T13:44:14
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

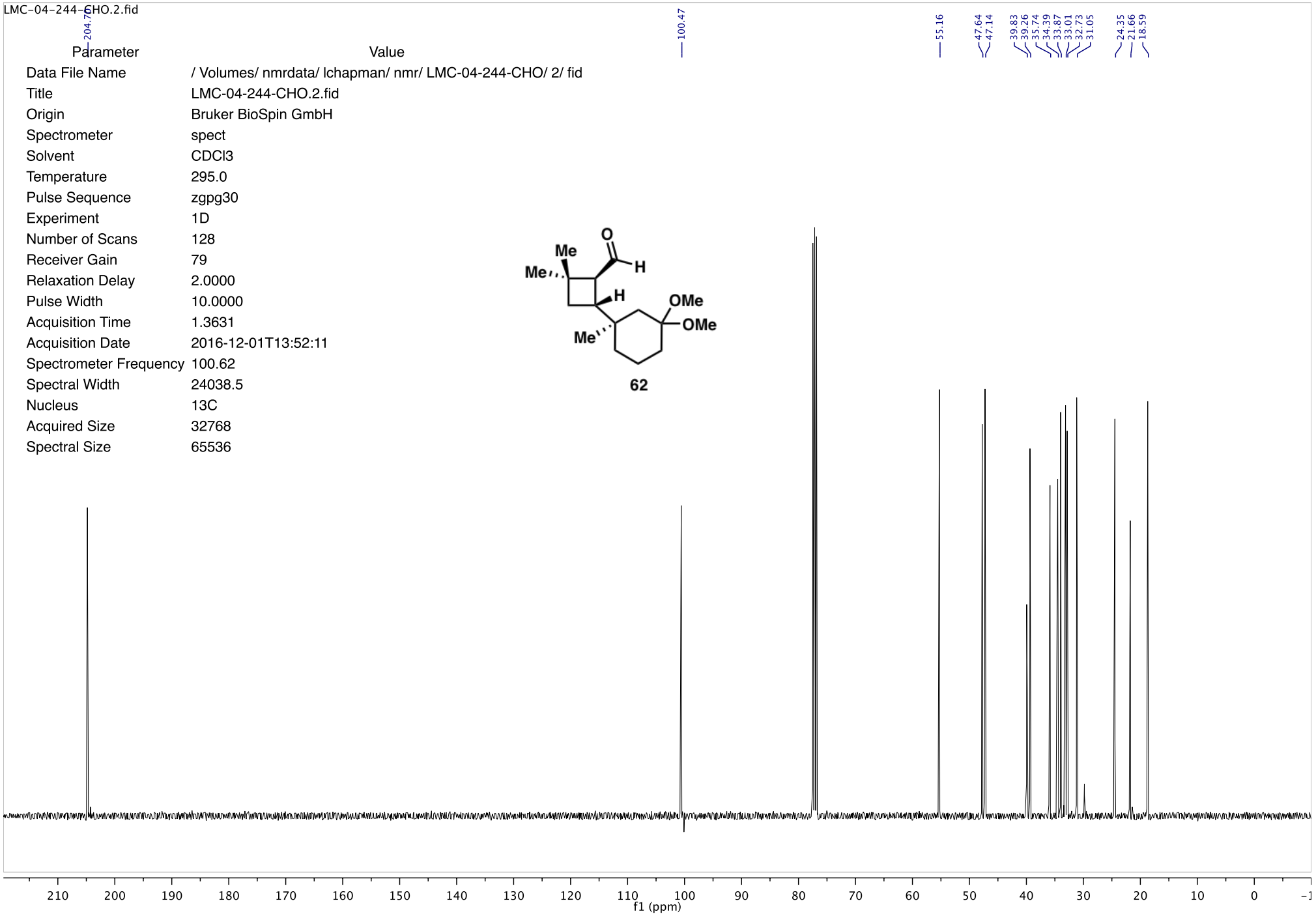
62



LMC-04-244-CHO.2.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-244-CHO/ 2/ fid
Title	LMC-04-244-CHO.2.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	79
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-01T13:52:11
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

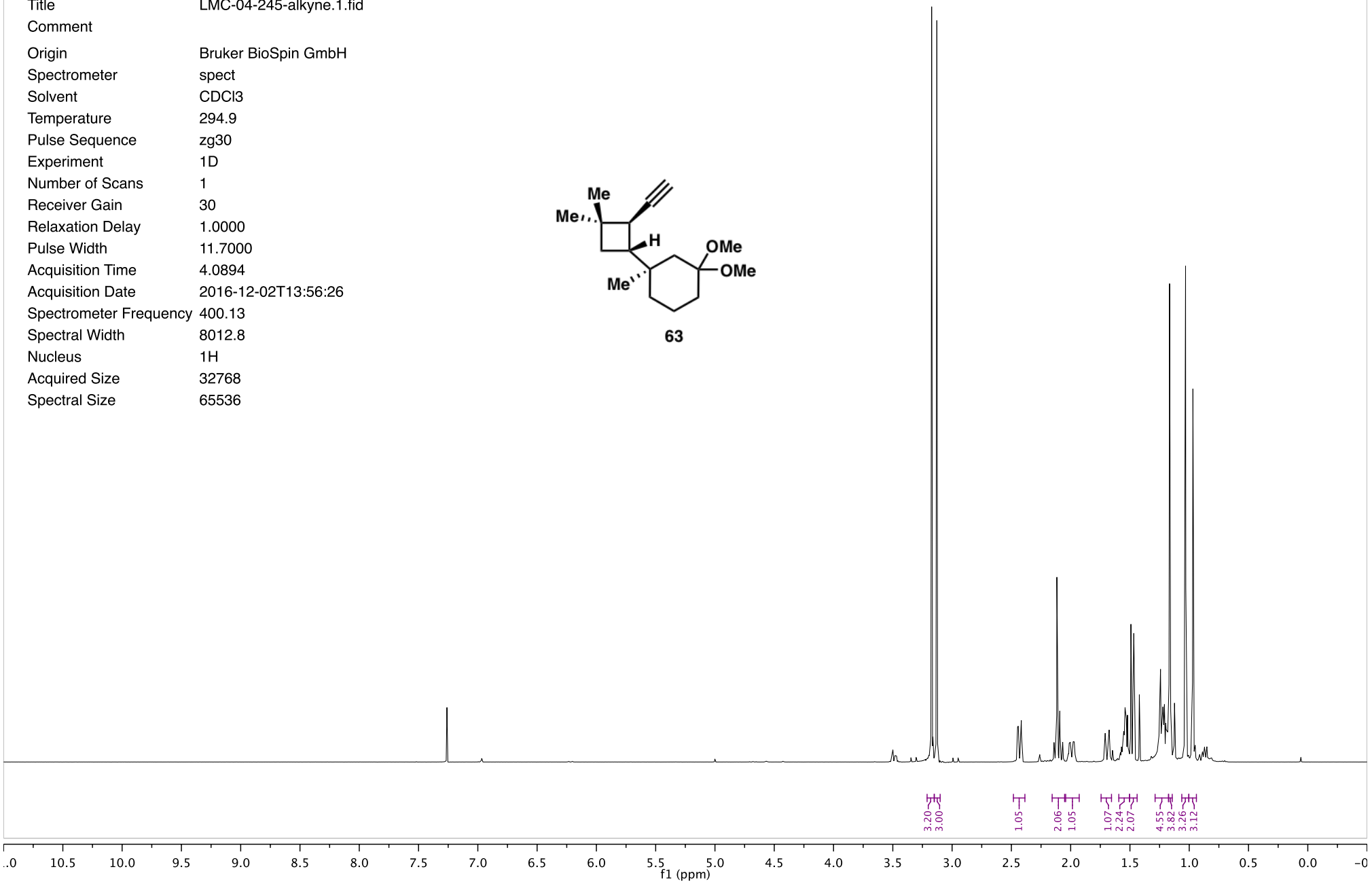
62



LMC-04-245-alkyne.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-245-alkyne/ 1/ fid
Title	LMC-04-245-alkyne.1.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	1
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-12-02T13:56:26
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

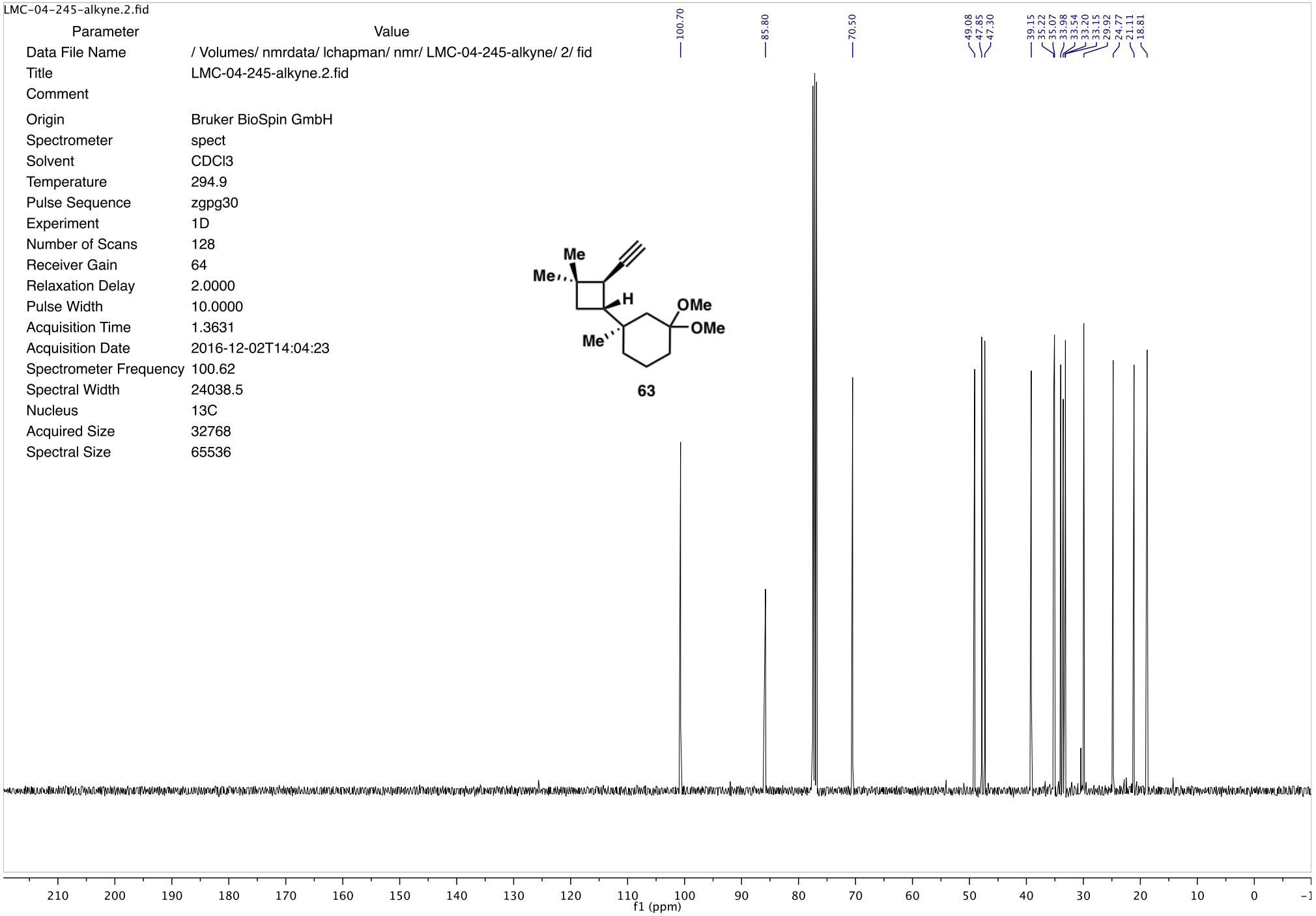
63



LMC-04-245-alkyne.2.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-245-alkyne/ 2/ fid
Title	LMC-04-245-alkyne.2.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	64
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-02T14:04:23
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

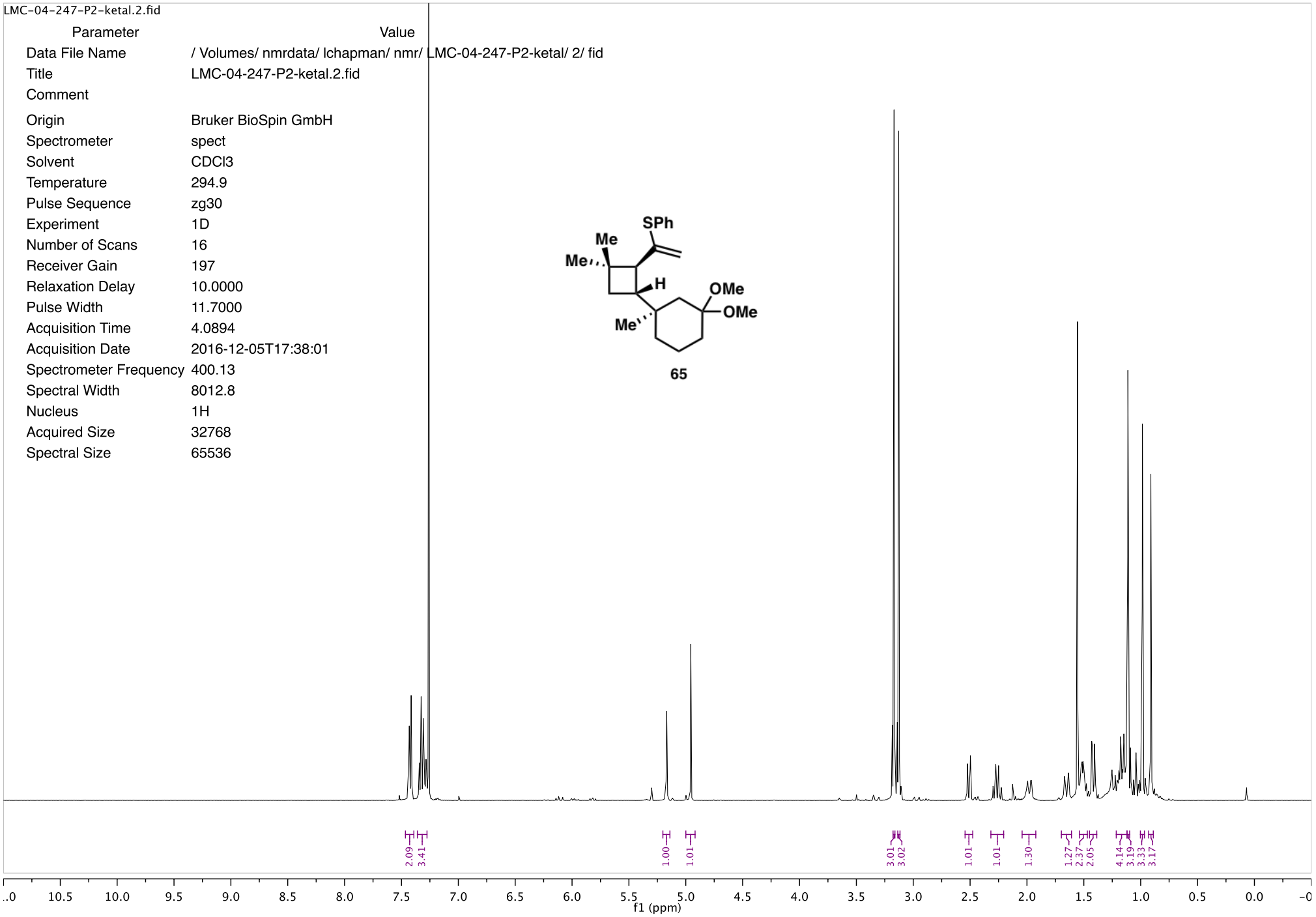
63



LMC-04-247-P2-ketal.2.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-247-P2-ketal/ 2/ fid
Title	LMC-04-247-P2-ketal.2.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	197
Relaxation Delay	10.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-12-05T17:38:01
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

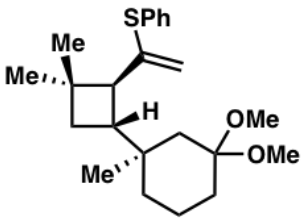
65



LMC-04-247-P2-ketal.4.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-247-P2-ketal/ 4/ fid
Title	LMC-04-247-P2-ketal.4.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	3072
Receiver Gain	72
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-06T00:56:39
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

145.88, 133.80, 133.38, 129.18, 127.93, 111.61, 100.79, 49.41, 47.88, 47.27, 45.11, 39.73, 35.61, 34.83, 34.57, 33.25, 32.43, 30.58, 23.25, 21.66, 18.88



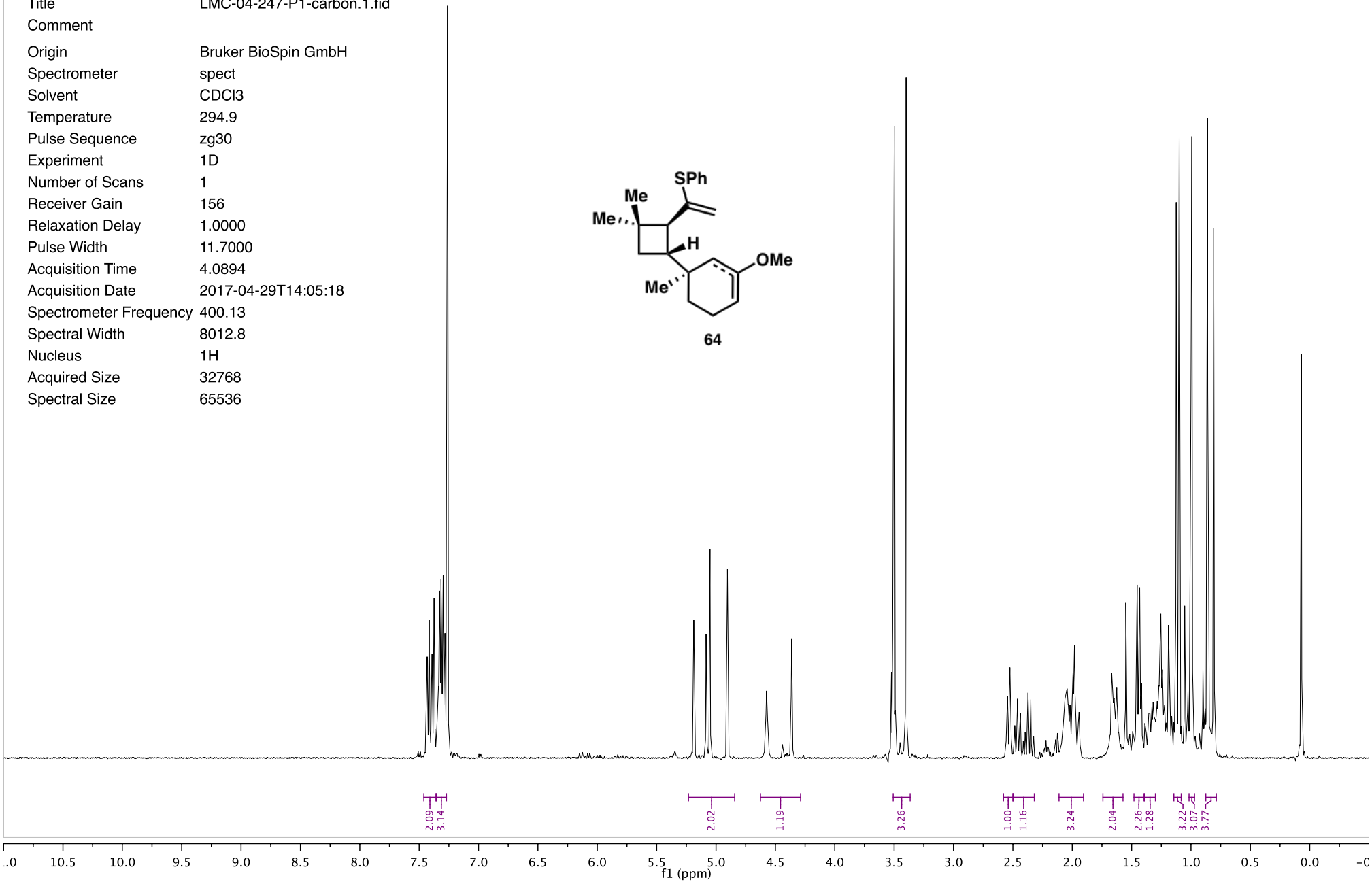
65

f1 (ppm)

LMC-04-247-P1-carbon.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-247-P1-carbon/ 1/ fid
Title	LMC-04-247-P1-carbon.1.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	1
Receiver Gain	156
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-04-29T14:05:18
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

64



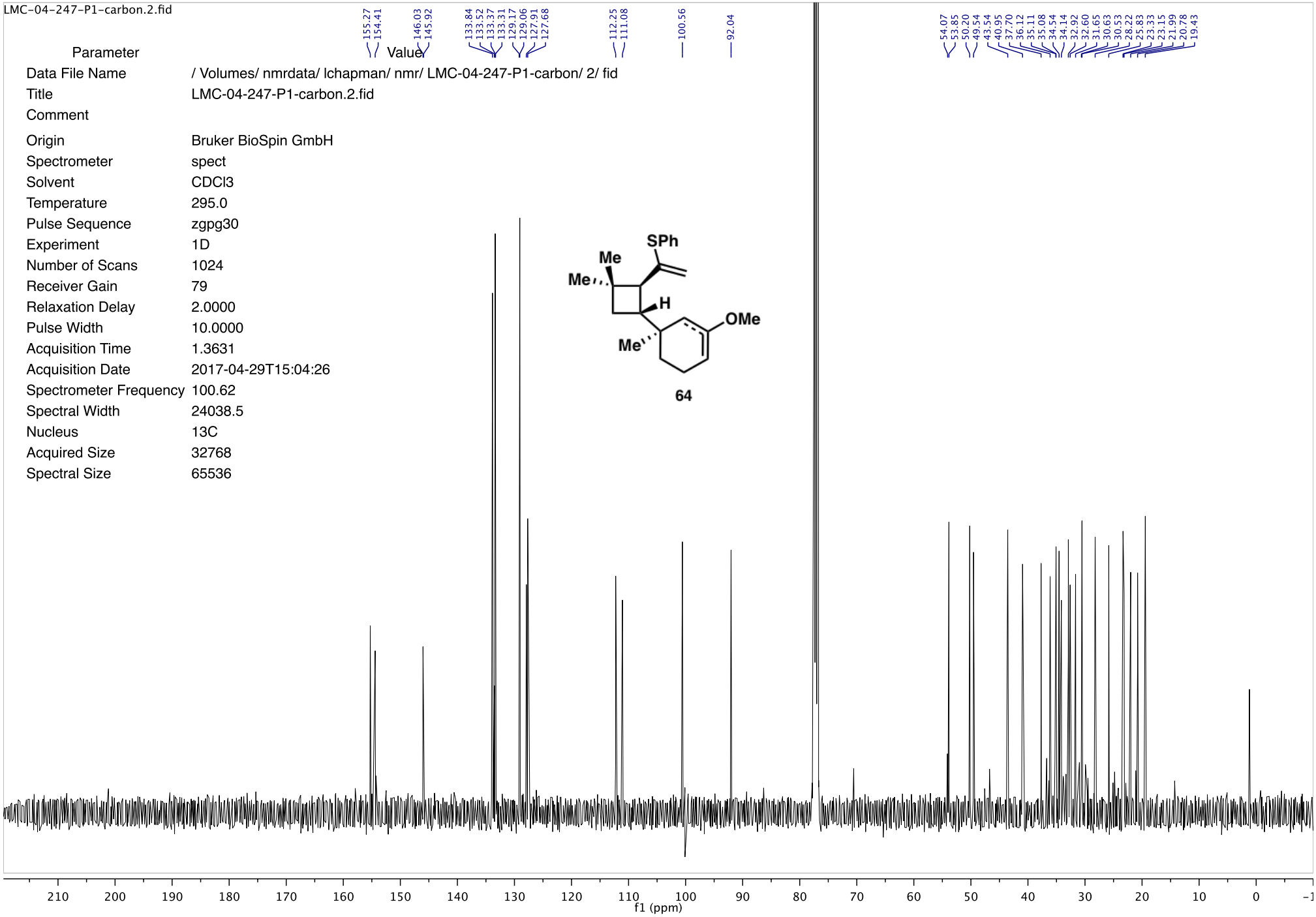
LMC-04-247-P1-carbon.2.fid

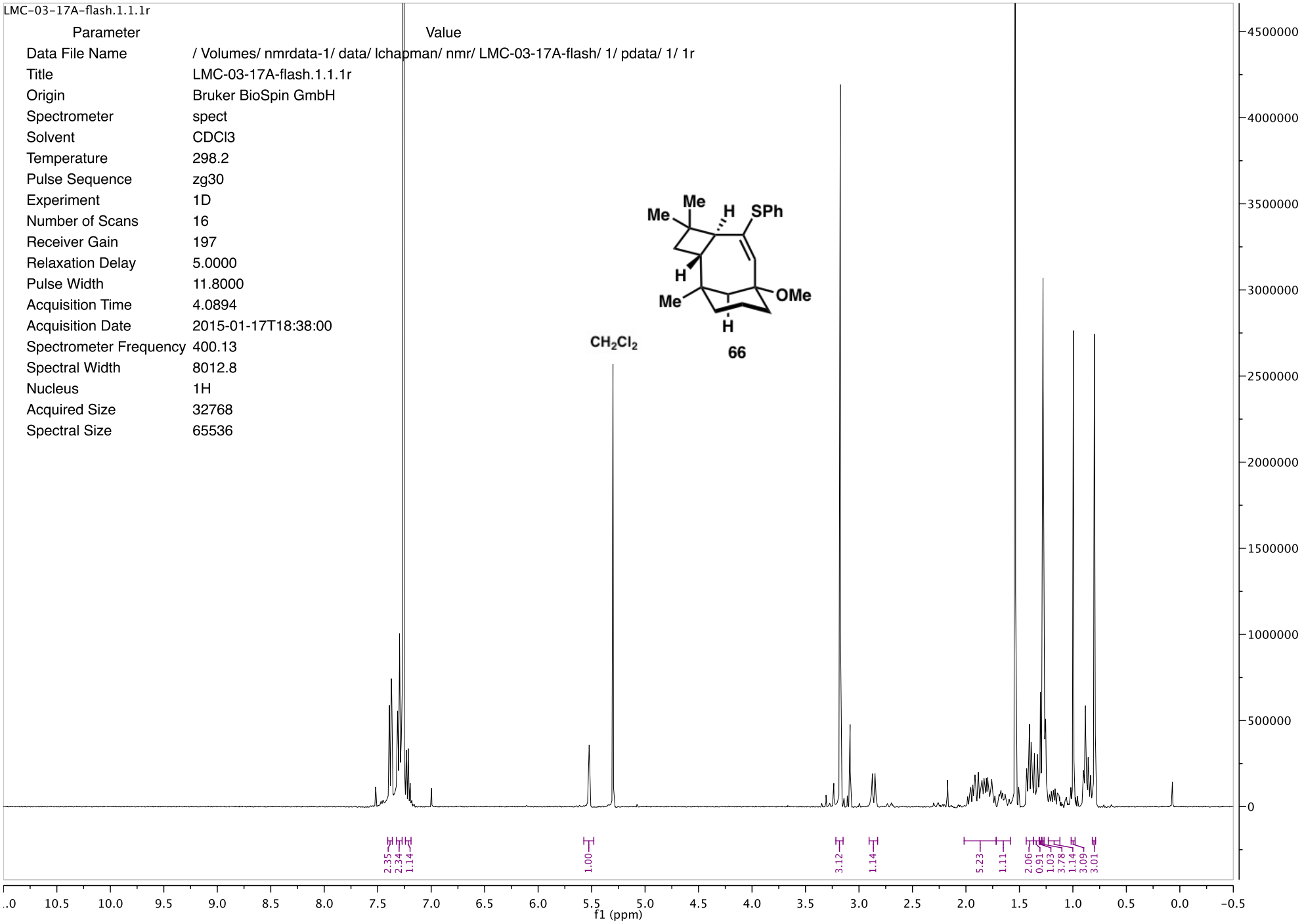
Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-247-P1-carbon/ 2/ fid
Title	LMC-04-247-P1-carbon.2.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	1024
Receiver Gain	79
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-04-29T15:04:26
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

155.27 154.41 146.03 145.92 133.84 133.52 133.37 133.31 129.17 129.06 127.91 127.68 112.25 111.08 100.56 92.04

54.07 53.85 50.20 49.54 43.54 40.95 37.70 36.12 35.11 35.08 34.54 34.14 32.92 32.60 31.65 30.63 30.53 28.22 25.83 23.33 23.15 21.99 20.78 19.43

64

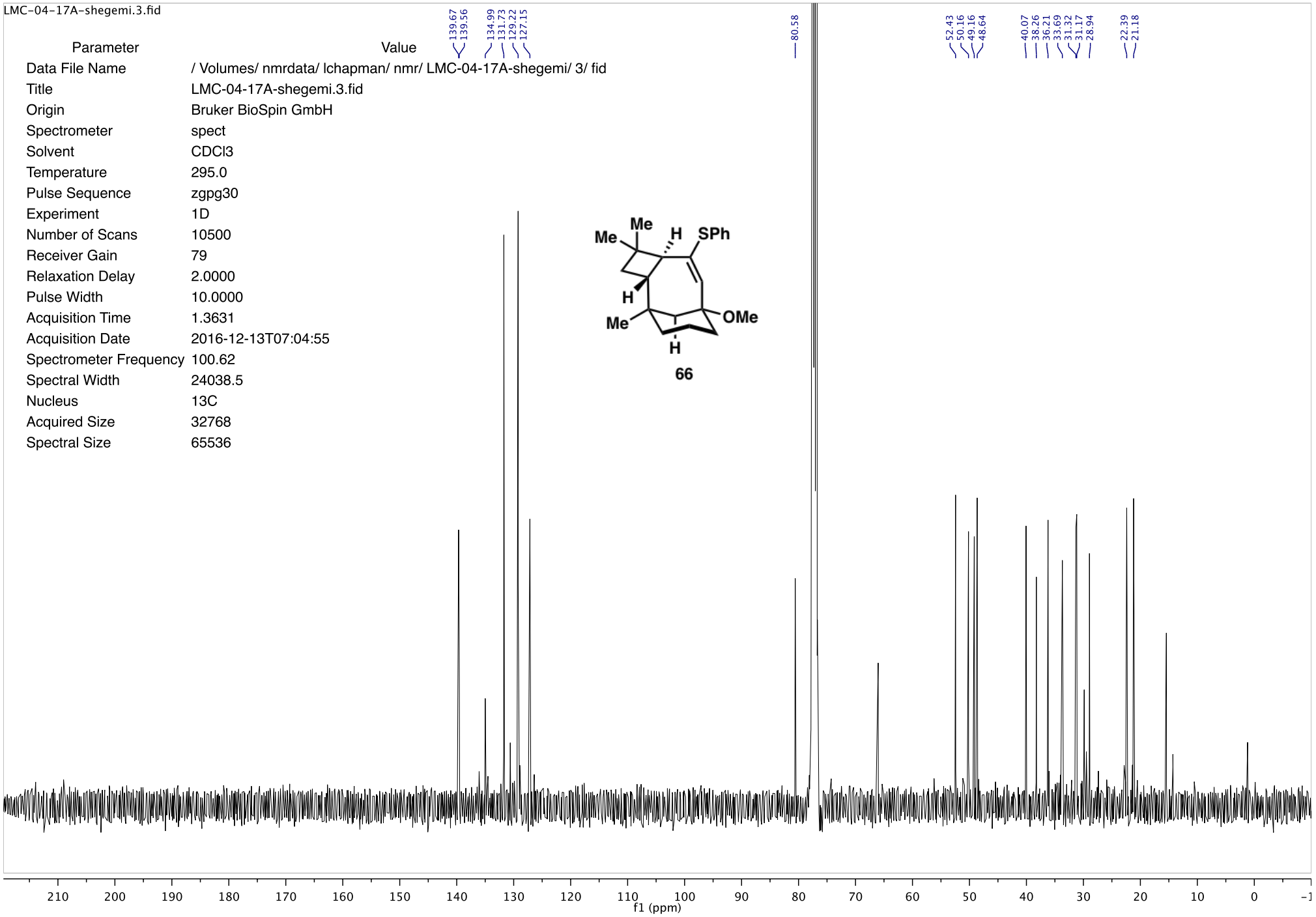


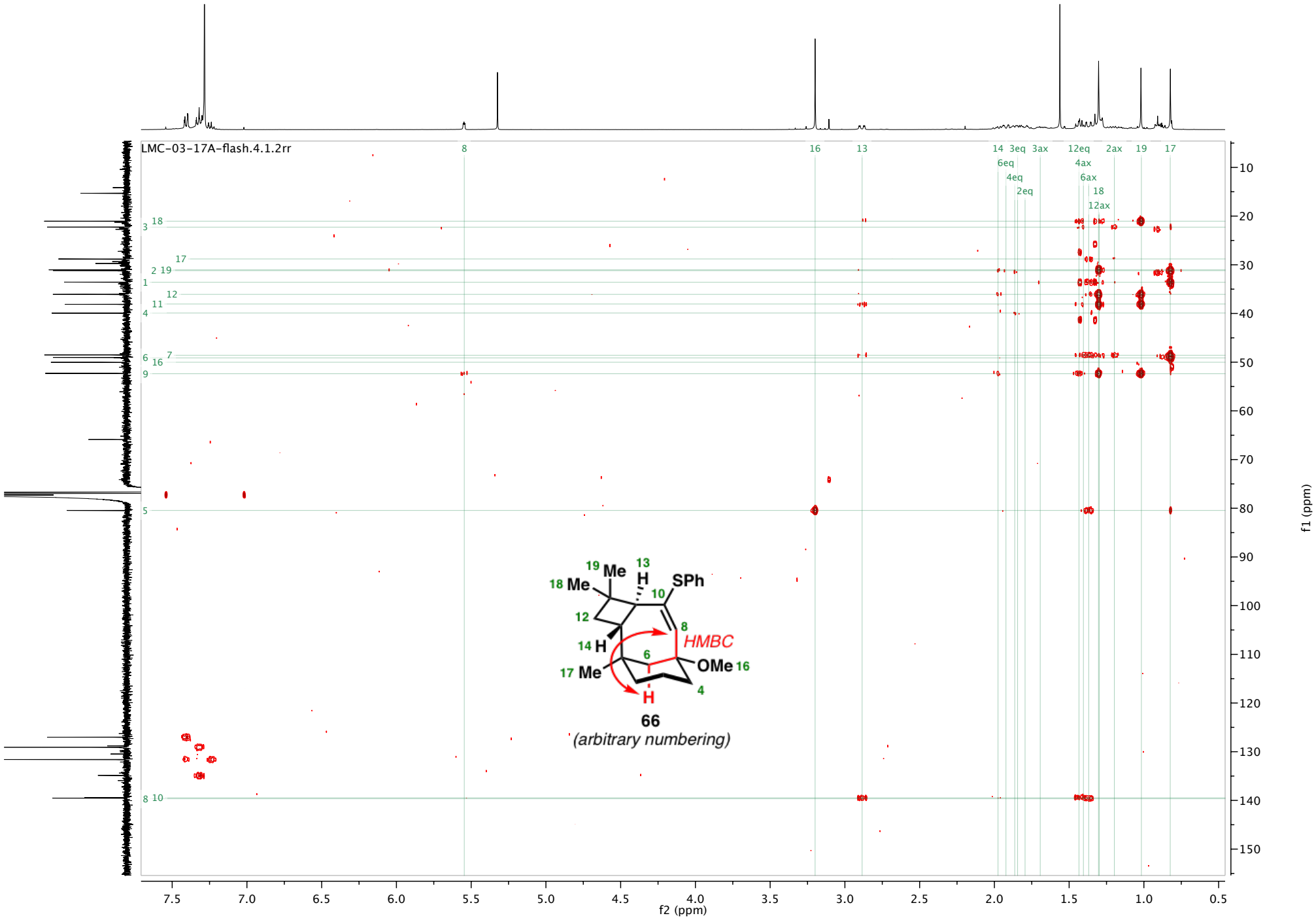

LMC-03-17A-flash.1.1.1r
Parameter Value
Data File Name / Volumes/ nmrdata-1/ data/ lchapman/ nmr/ LMC-03-17A-flash/ 1/ pdata/ 1/ 1r
Title LMC-03-17A-flash.1.1.1r
Origin Bruker BioSpin GmbH
Spectrometer spect
Solvent CDCl3
Temperature 298.2
Pulse Sequence zg30
Experiment 1D
Number of Scans 16
Receiver Gain 197
Relaxation Delay 5.0000
Pulse Width 11.8000
Acquisition Time 4.0894
Acquisition Date 2015-01-17T18:38:00
Spectrometer Frequency 400.13
Spectral Width 8012.8
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
Me
Me
H
SPh
H
Me
OMe
H
66
CH2Cl2
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
2.35
2.34
1.14
1.00
3.12
1.14
5.23
1.11
2.06
0.91
1.03
3.78
1.14
3.09
3.01
10.5
10.0
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
-0.5
f1 (ppm)

LMC-04-17A-shegemi.3.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-17A-shegemi/ 3/ fid
Title	LMC-04-17A-shegemi.3.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	10500
Receiver Gain	79
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-13T07:04:55
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

66

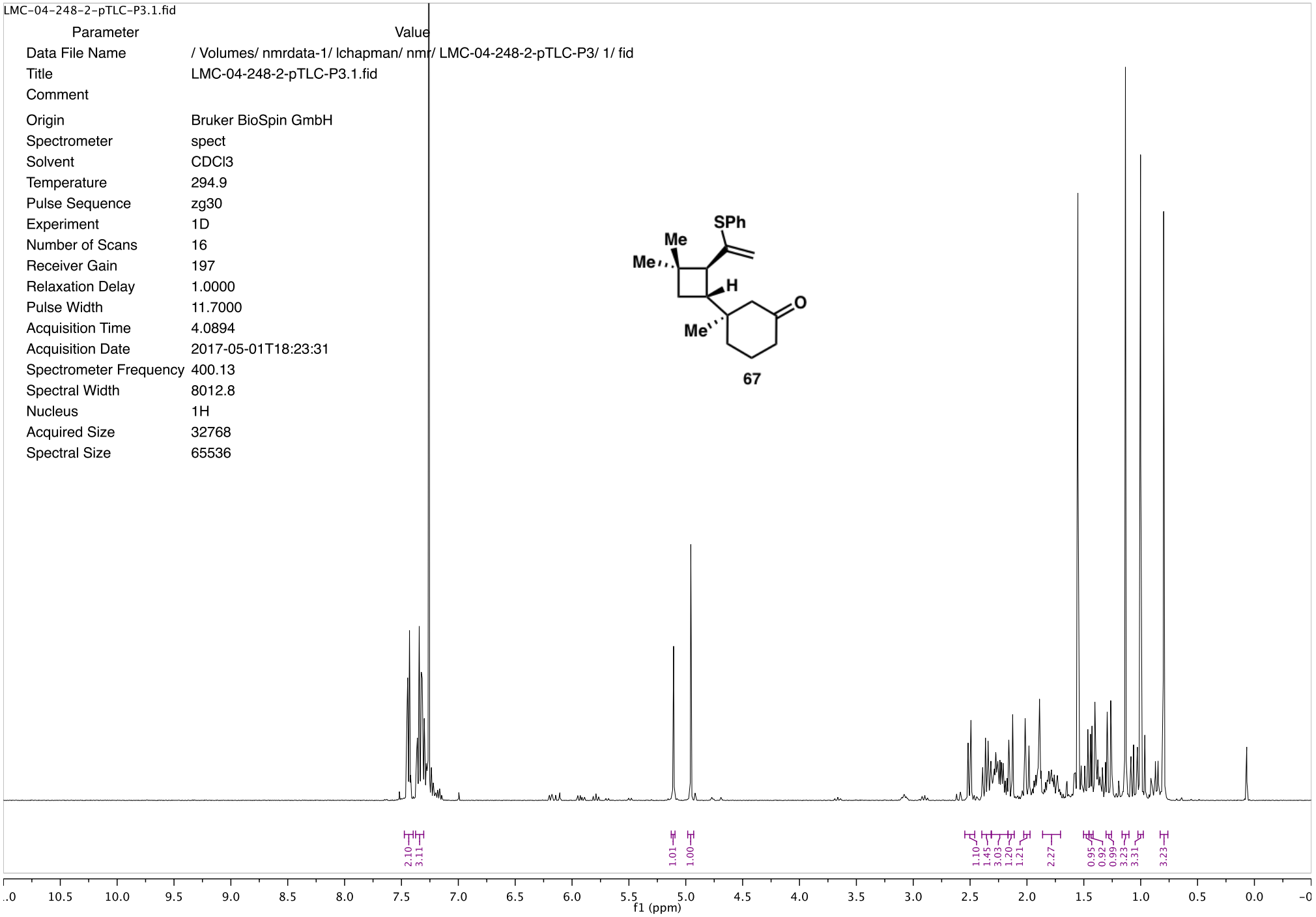



LMC-03-17A-flash.4.1.2rr
f2 (ppm)
f1 (ppm)
HMBC
66
(arbitrary numbering)

LMC-04-248-2-pTLC-P3.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata-1/ lchapman/ nmr/ LMC-04-248-2-pTLC-P3/ 1/ fid
Title	LMC-04-248-2-pTLC-P3.1.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	197
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-05-01T18:23:31
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

67

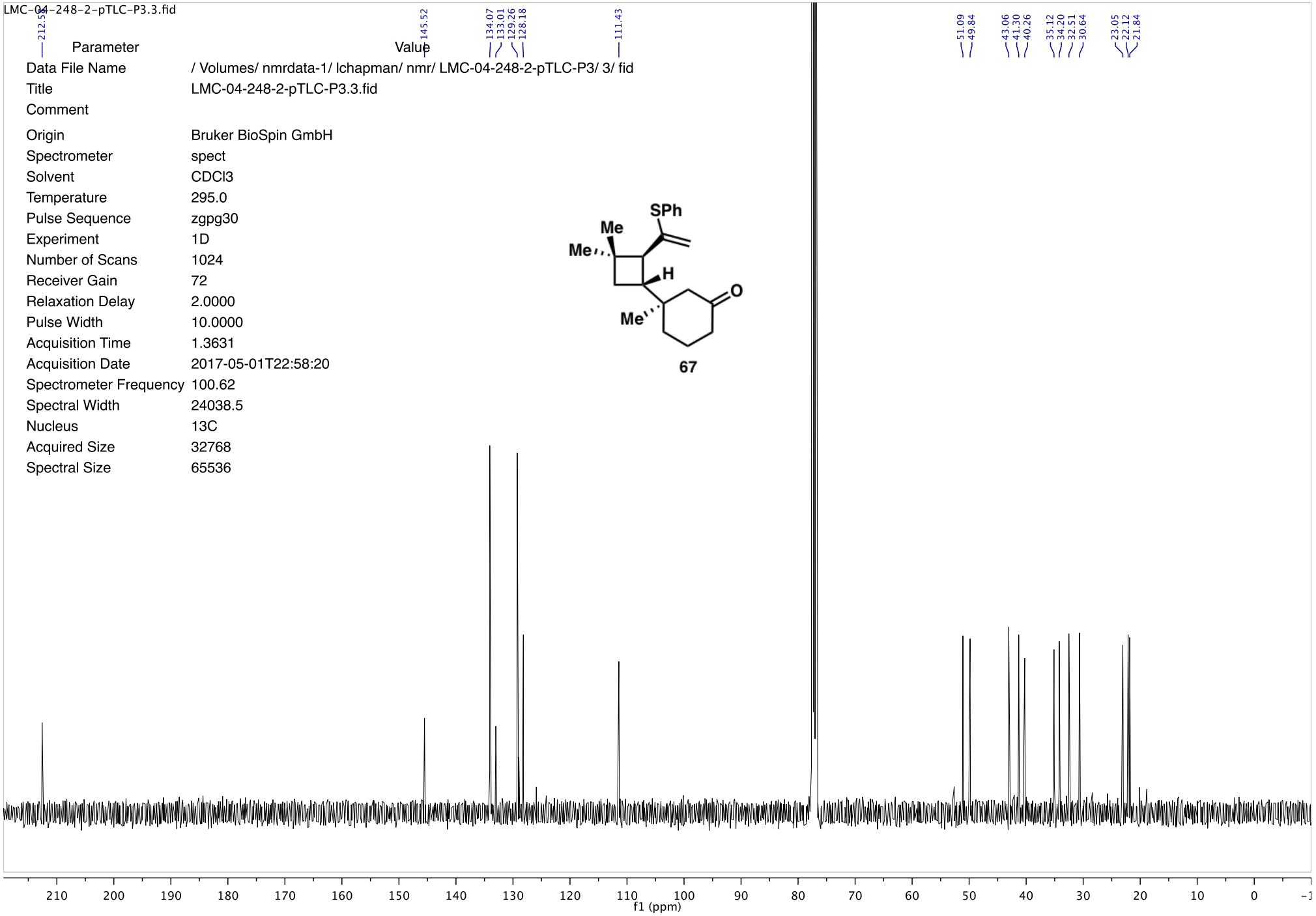


LMC-04-248-2-pTLC-P3.3.fid

212.54 145.52 134.07 133.01 129.26 128.18 111.43 51.09 49.84 43.06 41.30 40.26 35.12 34.20 32.51 30.64 23.05 22.12 21.84

Parameter	Value
Data File Name	/ Volumes/ nmrdata-1/ lchapman/ nmr/ LMC-04-248-2-pTLC-P3/ 3/ fid
Title	LMC-04-248-2-pTLC-P3.3.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	1024
Receiver Gain	72
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-05-01T22:58:20
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

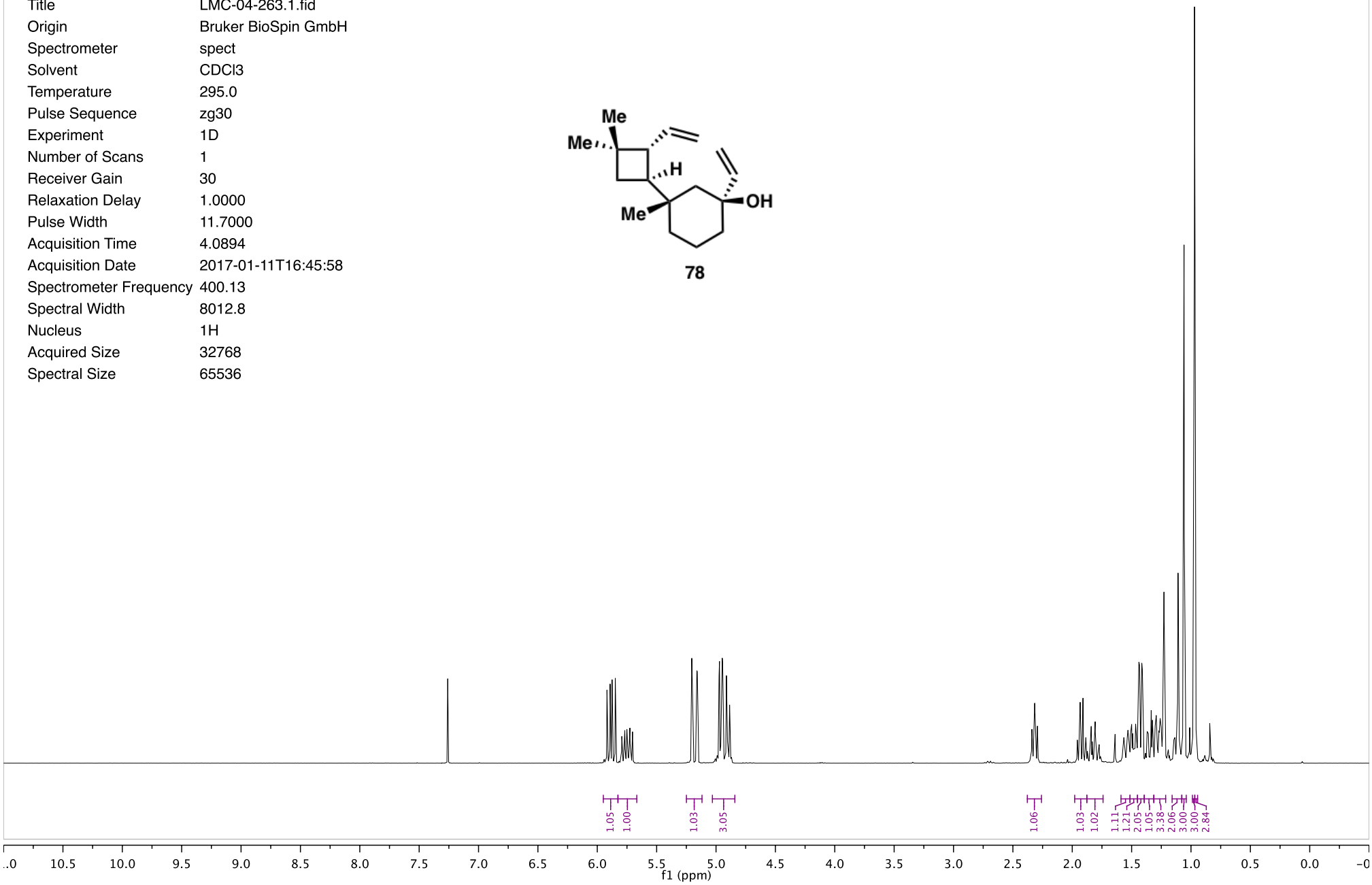
67



LMC-04-263.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-263/ 1/ fid
Title	LMC-04-263.1.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	1
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-01-11T16:45:58
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

78

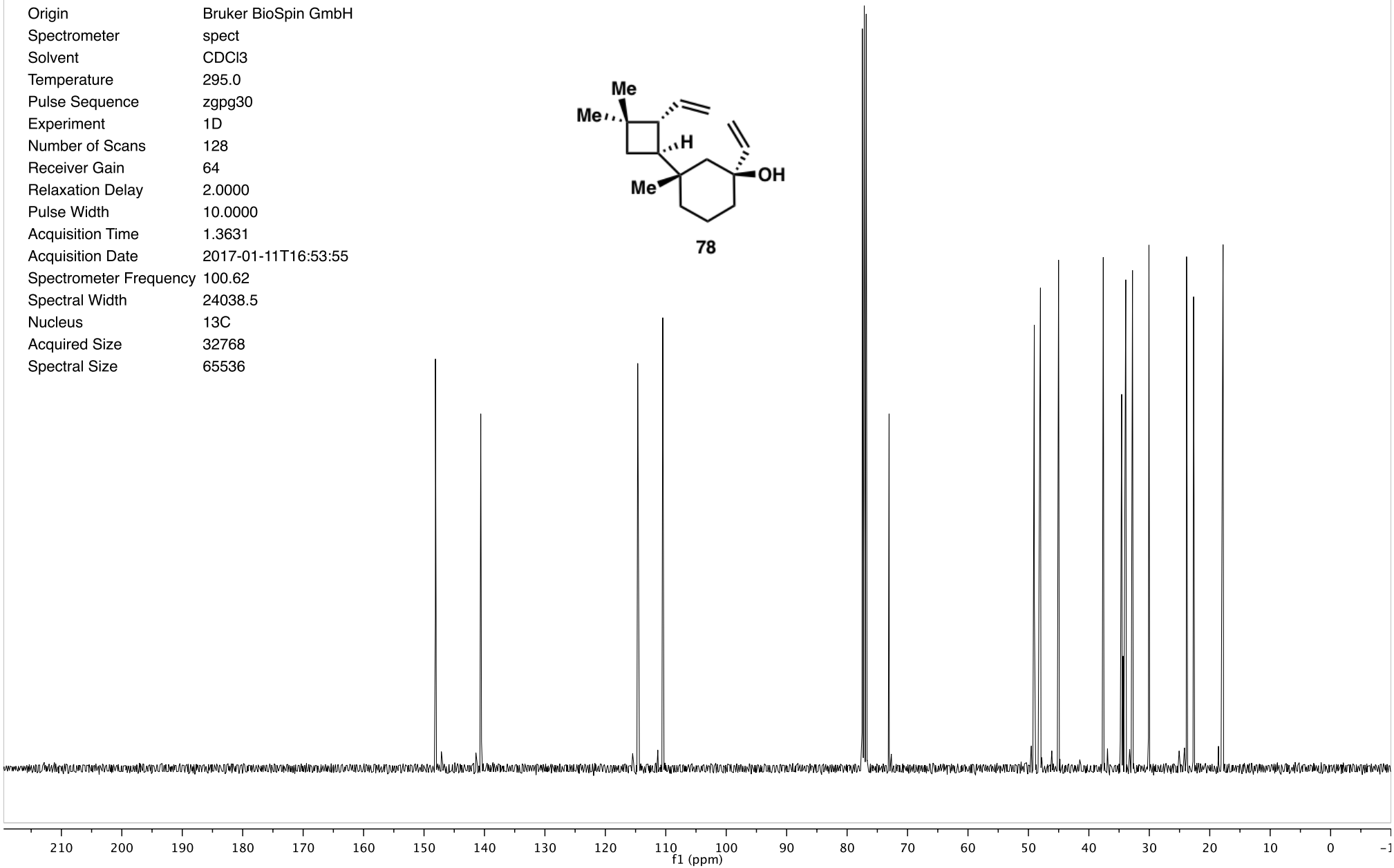


LMC-04-263.2.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-263/ 2/ fid
Title	LMC-04-263.2.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	64
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-01-11T16:53:55
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

148.12 140.61 114.64 110.50 73.06 49.03 48.01 45.01 37.62 34.57 34.34 33.89 32.78 30.06 23.83 22.67 17.80

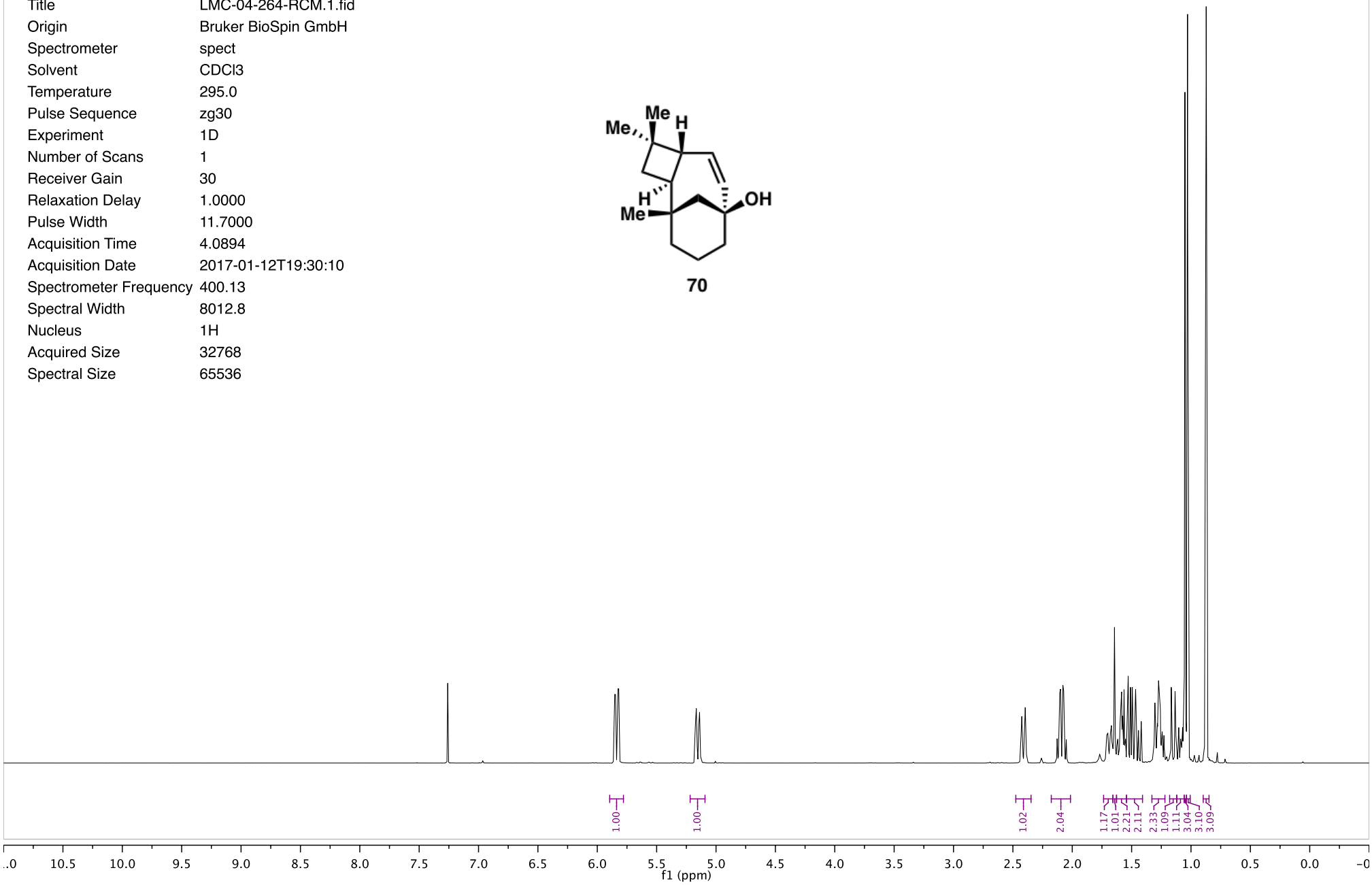
78



LMC-04-264-RCM.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-264-RCM/ 1/ fid
Title	LMC-04-264-RCM.1.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	1
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-01-12T19:30:10
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

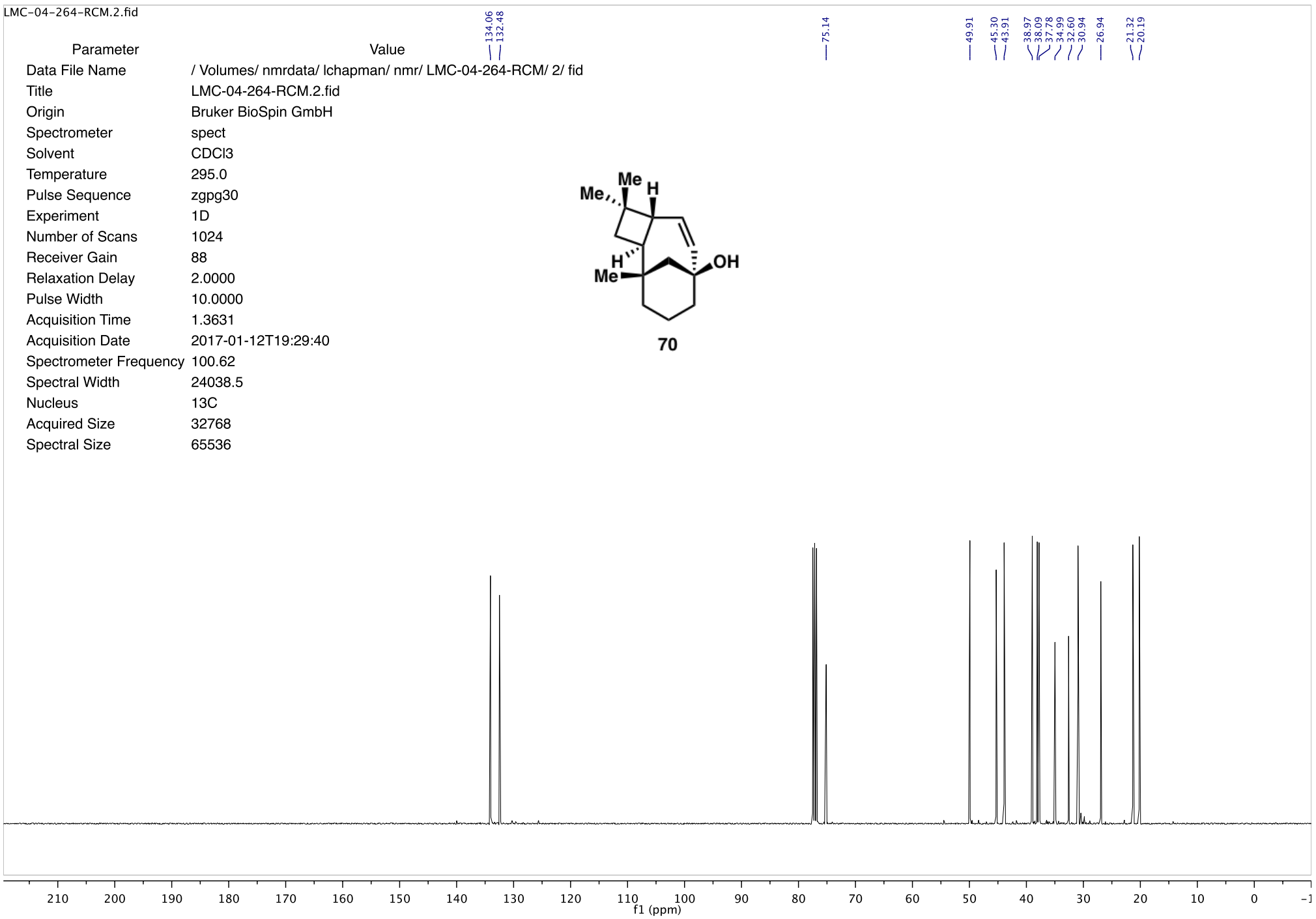
70



LMC-04-264-RCM.2.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-264-RCM/ 2/ fid
Title	LMC-04-264-RCM.2.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	1024
Receiver Gain	88
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-01-12T19:29:40
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

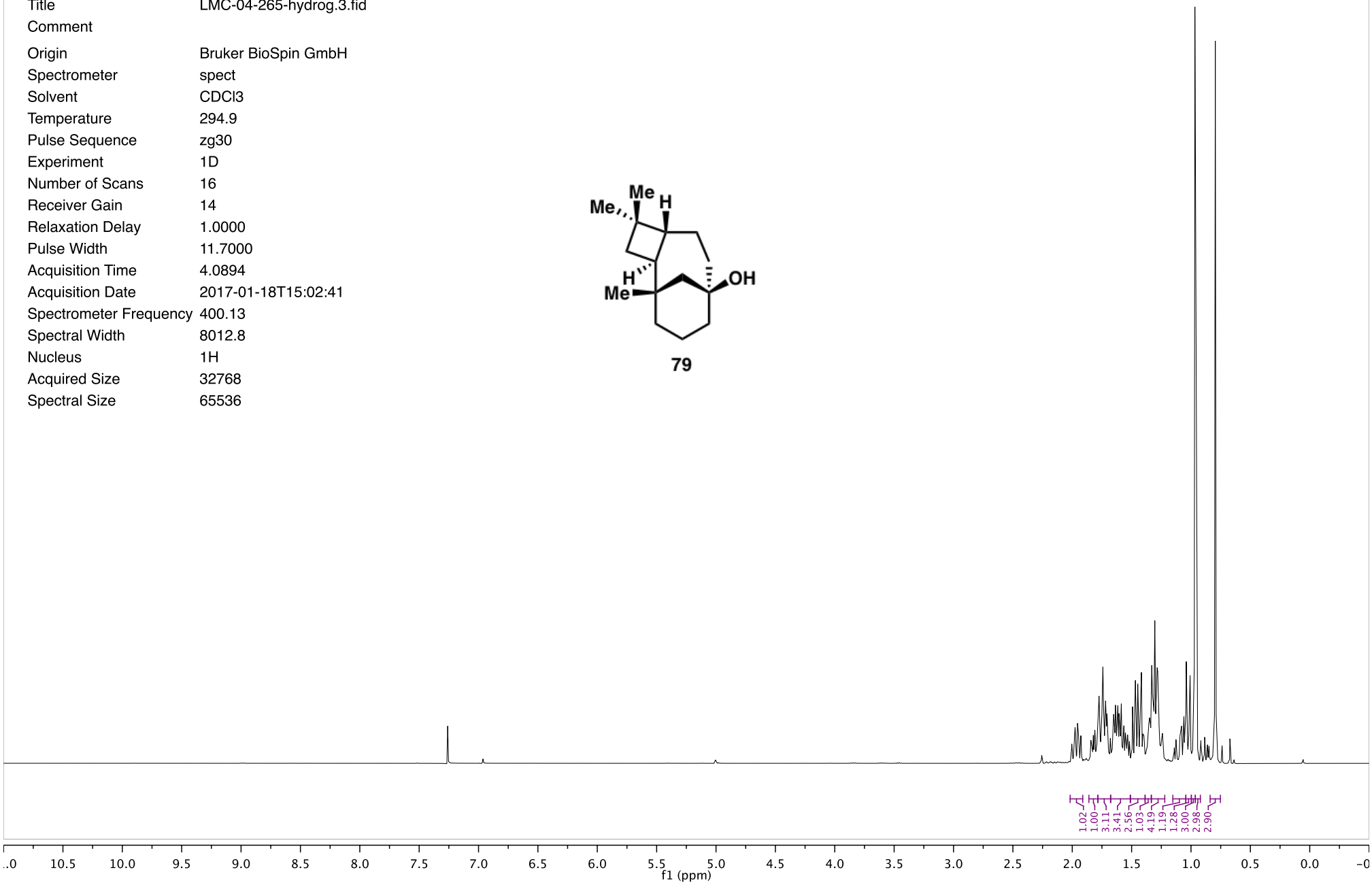
70



LMC-04-265-hydrog.3.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-265-hydrog/ 3/ fid
Title	LMC-04-265-hydrog.3.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	14
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-01-18T15:02:41
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

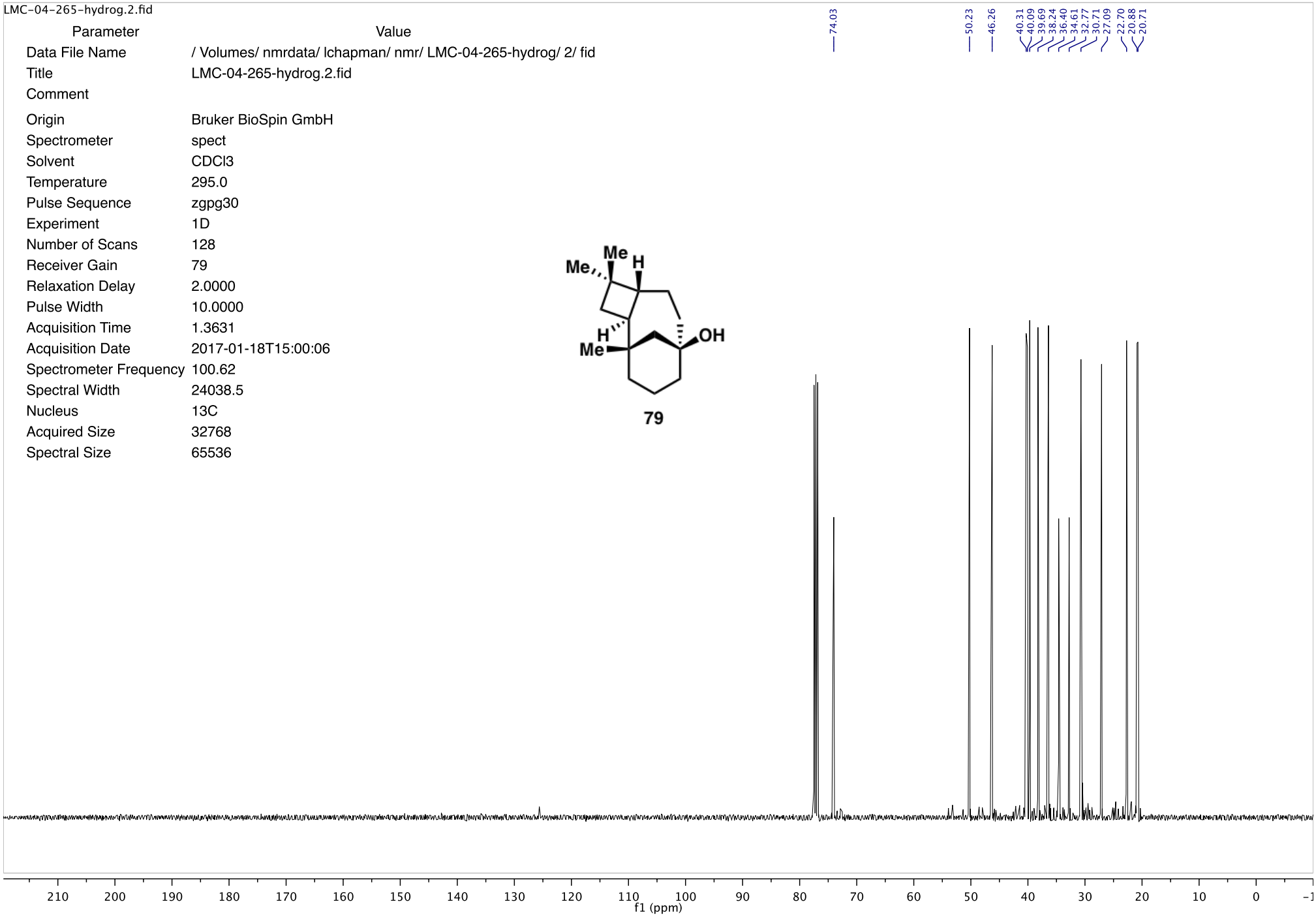
79



LMC-04-265-hydrog.2.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-265-hydrog/ 2/ fid
Title	LMC-04-265-hydrog.2.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	79
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-01-18T15:00:06
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

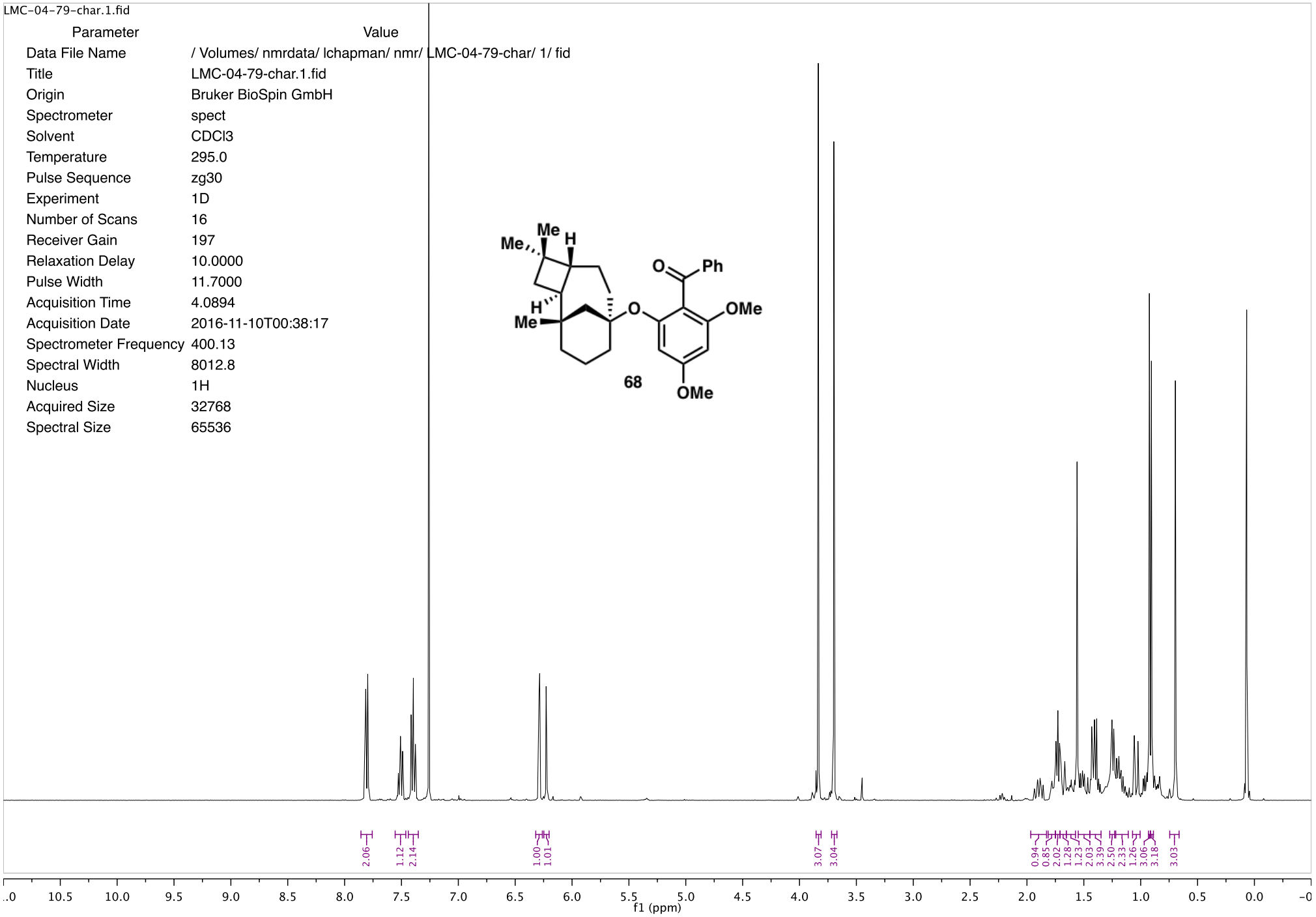
79

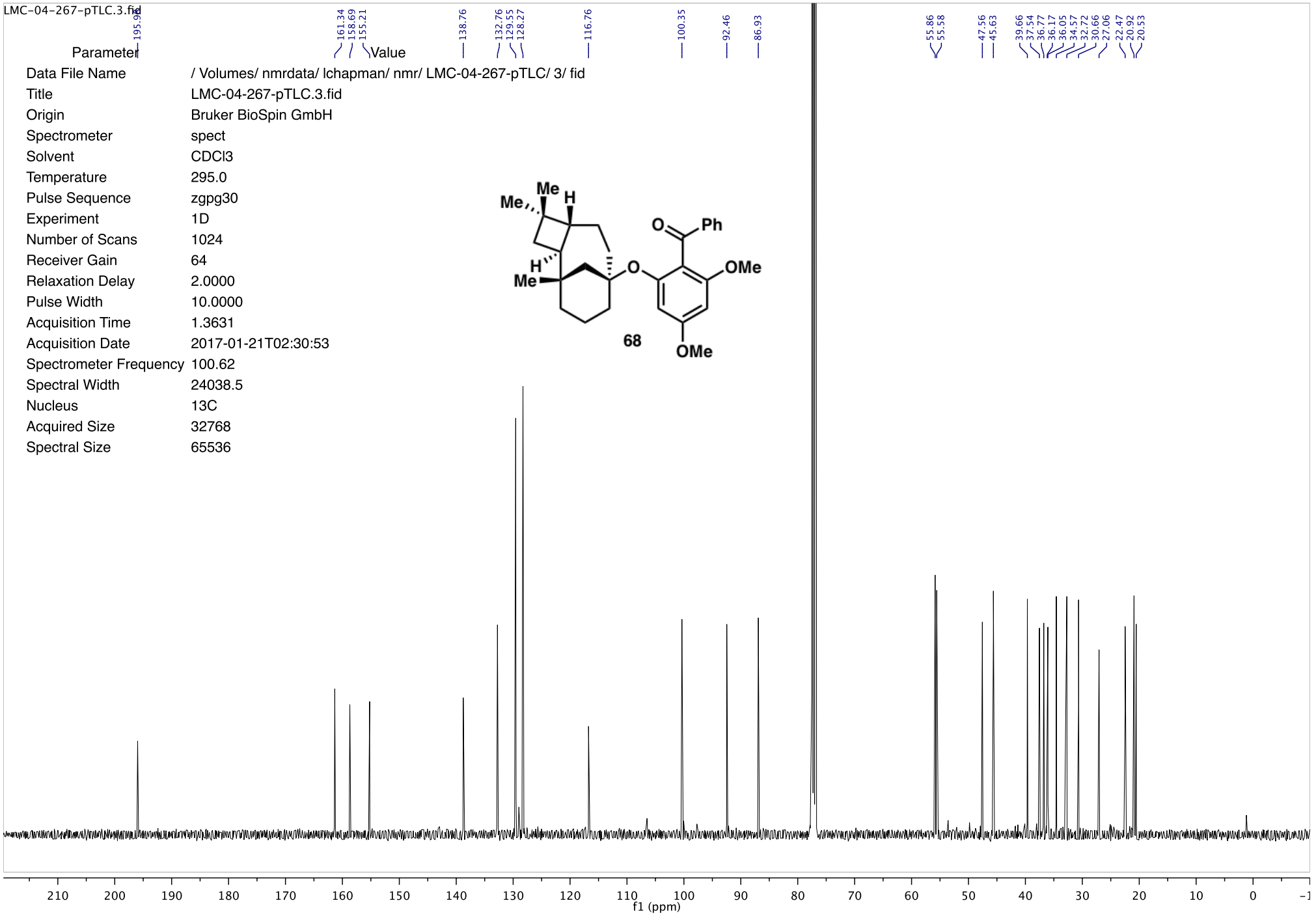


LMC-04-79-char.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-79-char/ 1/ fid
Title	LMC-04-79-char.1.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	197
Relaxation Delay	10.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-11-10T00:38:17
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

68

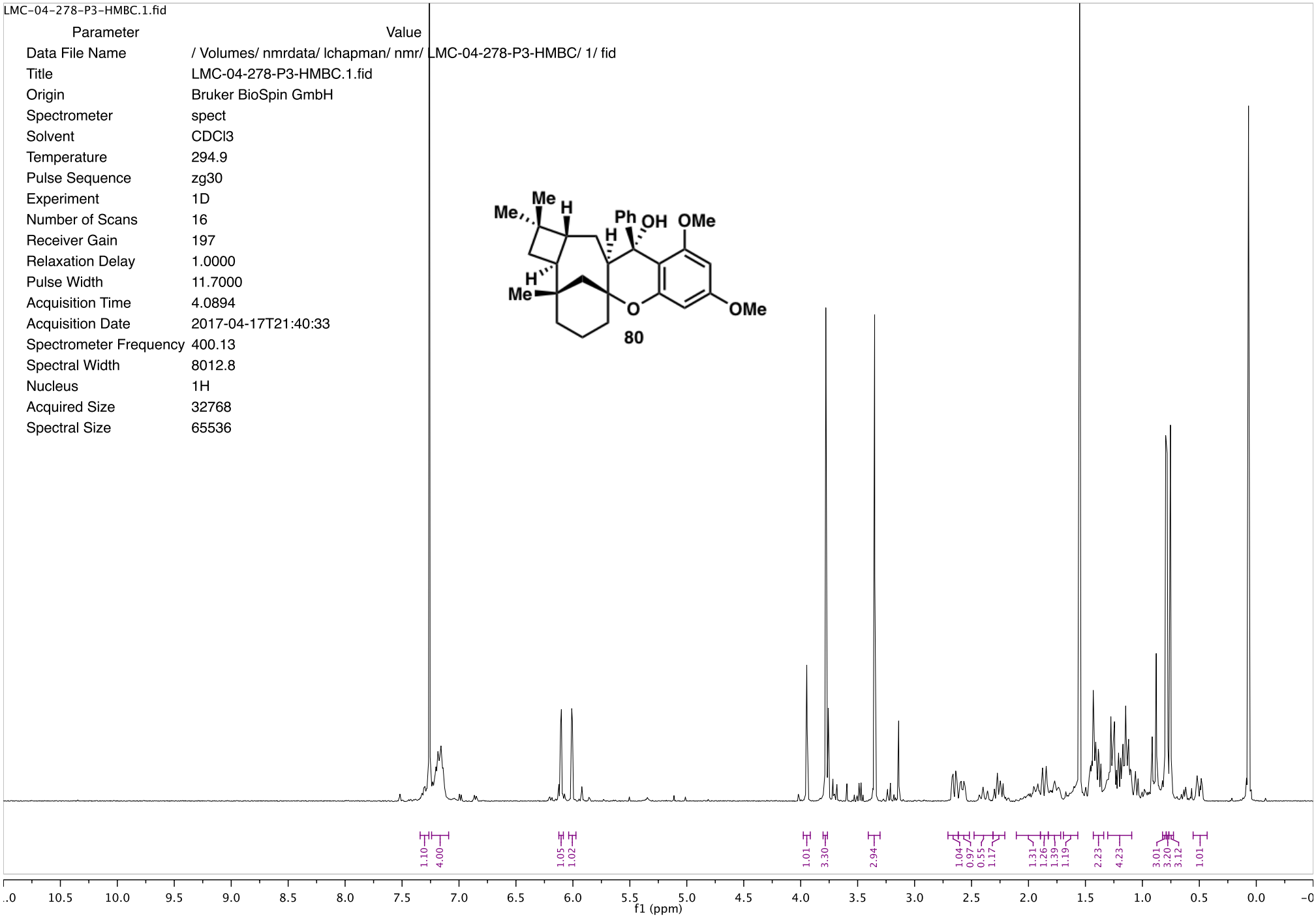



LMC-04-267-pTLC.3.fid
195.90
161.34
158.69
155.21
138.76
132.76
129.55
128.27
116.76
100.35
92.46
86.93
55.86
55.58
47.56
45.63
39.66
37.54
36.77
36.17
36.05
34.57
32.72
30.66
27.06
22.47
20.92
20.53
Parameter
Value
Data File Name / Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-267-pTLC/ 3/ fid
Title LMC-04-267-pTLC.3.fid
Origin Bruker BioSpin GmbH
Spectrometer spect
Solvent CDCl3
Temperature 295.0
Pulse Sequence zgpg30
Experiment 1D
Number of Scans 1024
Receiver Gain 64
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2017-01-21T02:30:53
Spectrometer Frequency 100.62
Spectral Width 24038.5
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
Me
H
Me
H
Me
O
Ph
O
OMe
OMe
68
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

LMC-04-278-P3-HMBC.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-278-P3-HMBC/ 1/ fid
Title	LMC-04-278-P3-HMBC.1.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	197
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-04-17T21:40:33
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

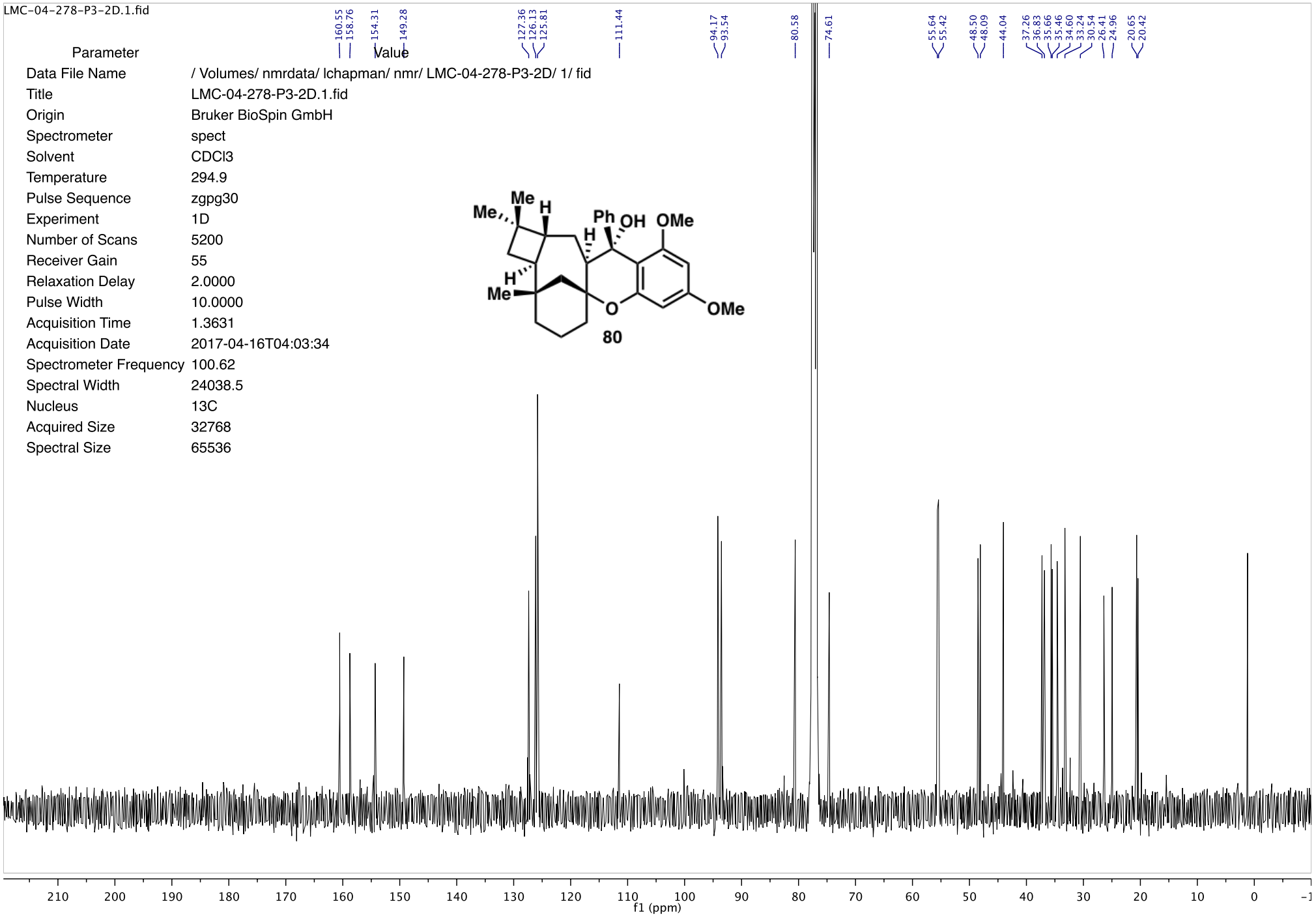
80

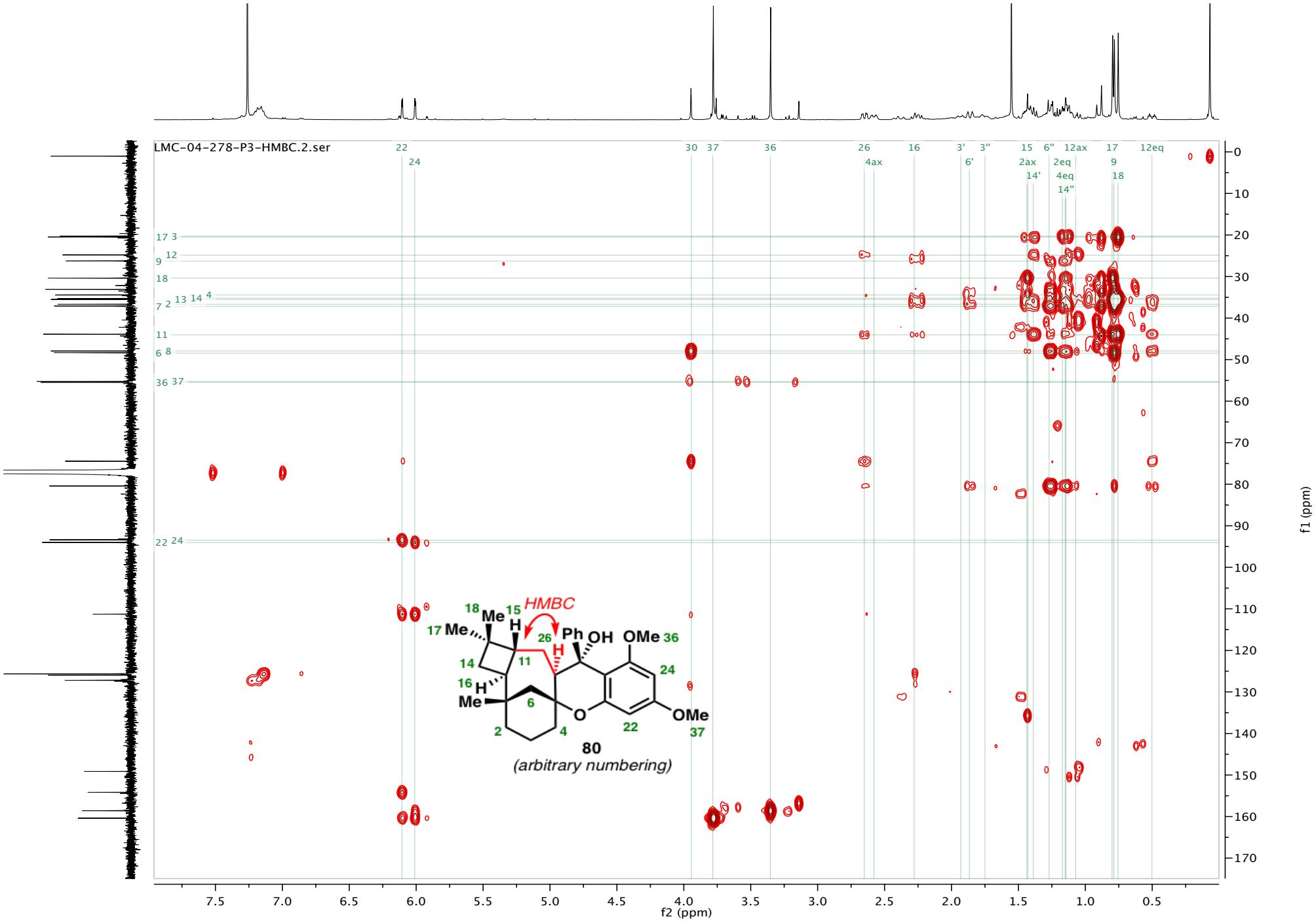


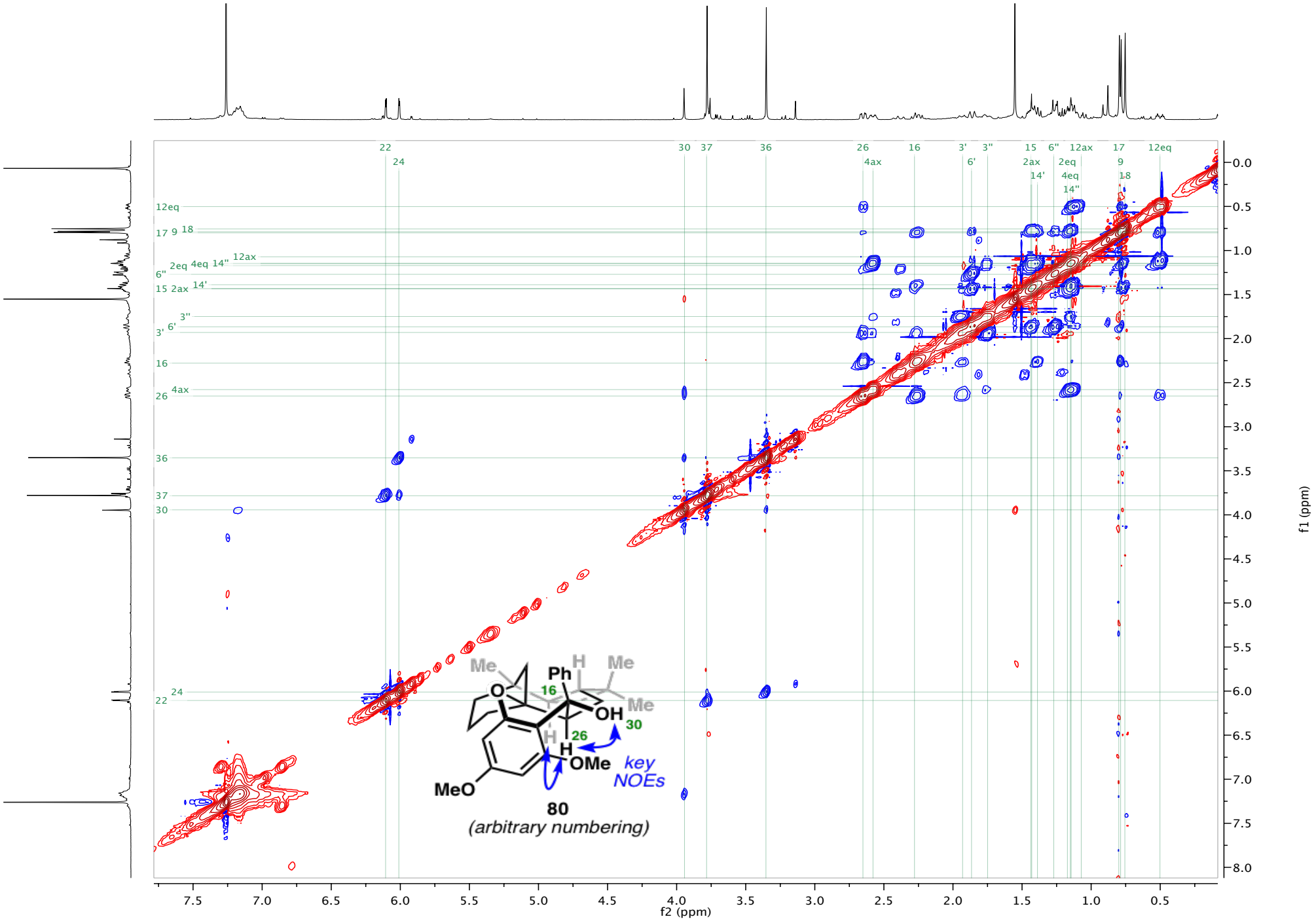
LMC-04-278-P3-2D.1.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-278-P3-2D/ 1/ fid
Title	LMC-04-278-P3-2D.1.fid
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	5200
Receiver Gain	55
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-04-16T04:03:34
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

80



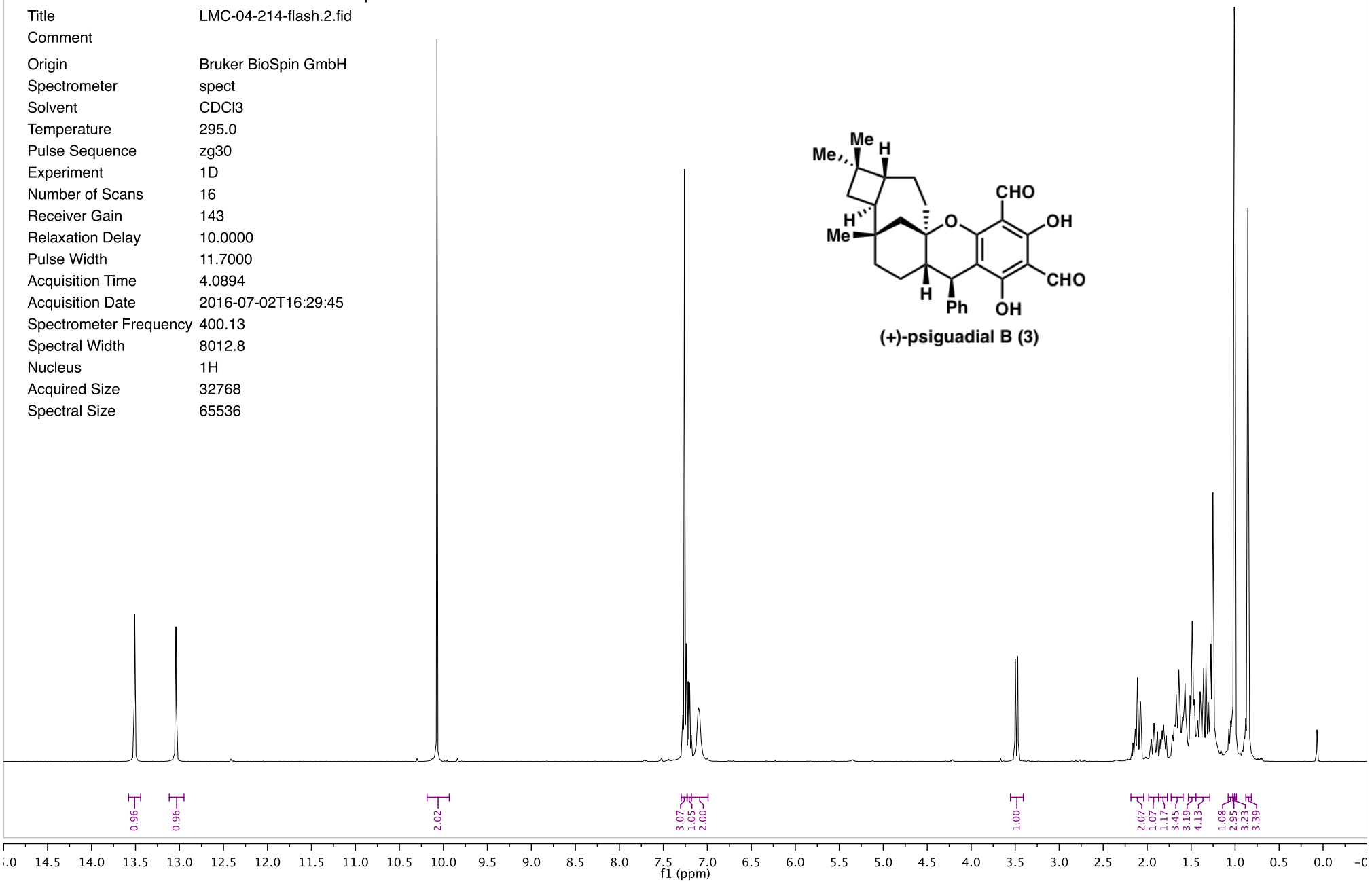

LMC-04-278-P3-HMBC.2.ser
f2 (ppm)
f1 (ppm)
HMBC
80
(arbitrary numbering)


22
24
30
37
36
26
4ax
16
3'
6'
3"
15
2ax
14'
6"
2eq
4eq
14"
12ax
17
9
18
12eq
f2 (ppm)
f1 (ppm)
Me
Ph
H
Me
O
16
OH
Me
30
26
H
OMe
MeO
key NOEs
80
(arbitrary numbering)

LMC-04-214-flash.2.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-214-flash/ 2/ fid
Title	LMC-04-214-flash.2.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	143
Relaxation Delay	10.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-07-02T16:29:45
Spectrometer Frequency	400.13
Spectral Width	8012.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

(+)-psiguadial B (3)



LMC-04-214-flash.3.fid

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ lchapman/ nmr/ LMC-04-214-flash/ 3/ fid
Title	LMC-04-214-flash.3.fid
Comment	
Origin	Bruker BioSpin GmbH
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	1024
Receiver Gain	72
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-07-02T17:28:52
Spectrometer Frequency	100.62
Spectral Width	24038.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

(+)-psiguadial B (3)