Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Synthesis of Trifluoromethylthiolated Azacalix[1]arene[3]pyridines from Cu(II)-Mediated Direct Trifluoromethylthiolation Reaction of Arenes via Reactive Arylcopper(III) Intermediate

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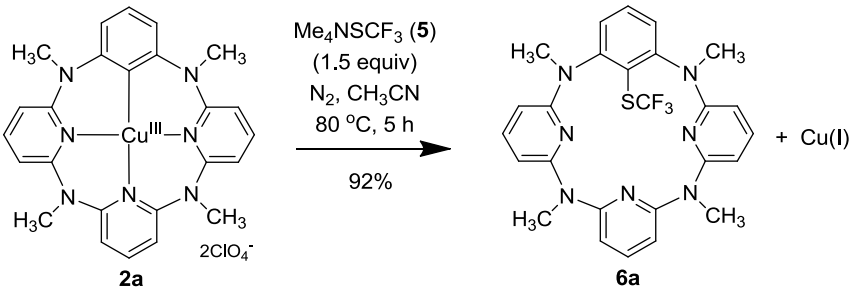
1. General Information

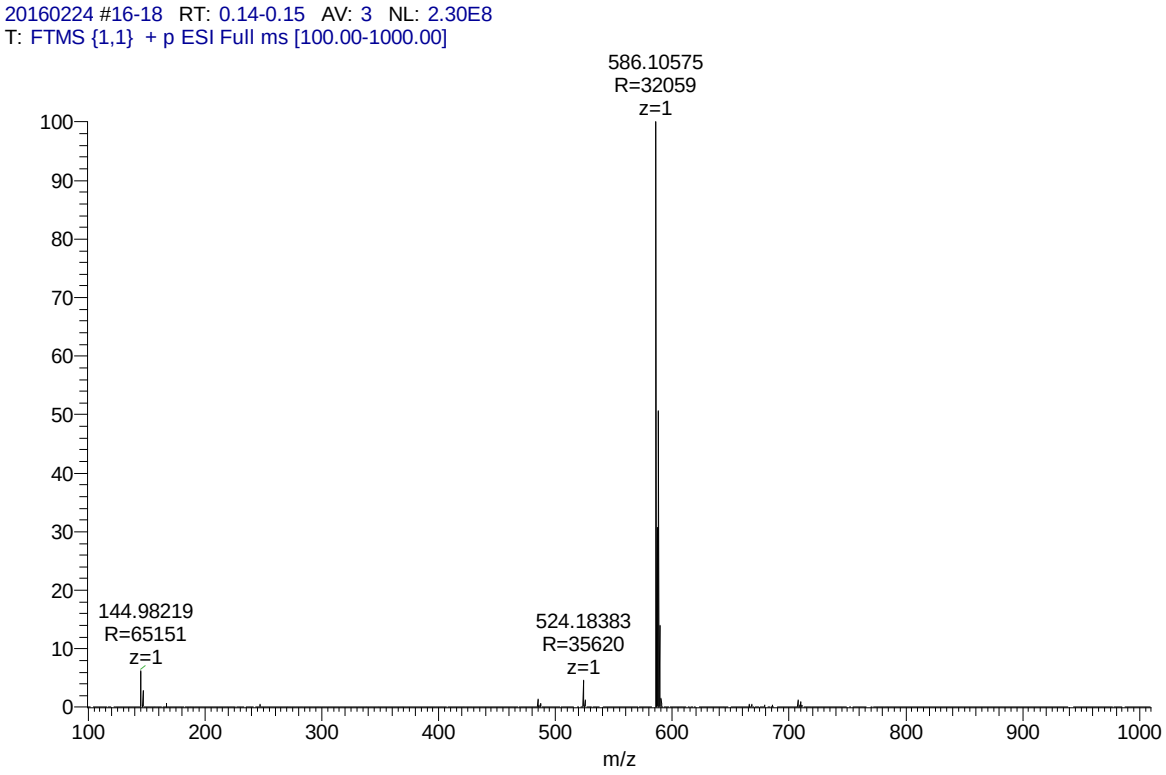
The ^{1}H and ^{13}C spectra were recorded on a 400 MHz spectrometer. Chemical shifts are reported in ppm with either tetramethylsilane or the residual solvent resonance used as an internal standard. Infrared spectra were recorded using a FT-IR spectrometer with KBr discs in the 4000-400 cm^{-1} region. Melting points are uncorrected. Flash column chromatography was performed on silica gel (100-200 mesh or 200-300 mesh).

NMe_4SCF_3[1], N-trifluoromethylthiosaccharin **3**[2] and $CuSCF_3$[3] were prepared according the literature procedures.

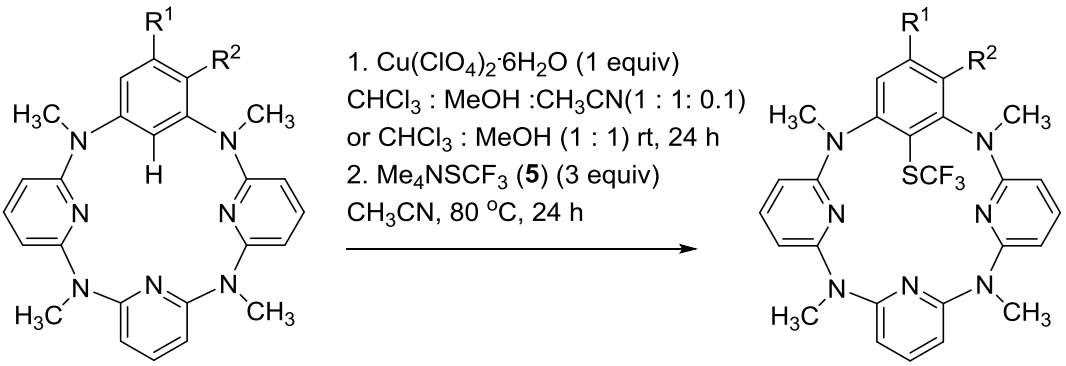
2. High resolution mass spectrum of the sample after completion of the reaction of 2a with Me_4NSCF_3 5.



2a **6a**



3. The reaction of 8a with 5 using equimolar copper(II) salt

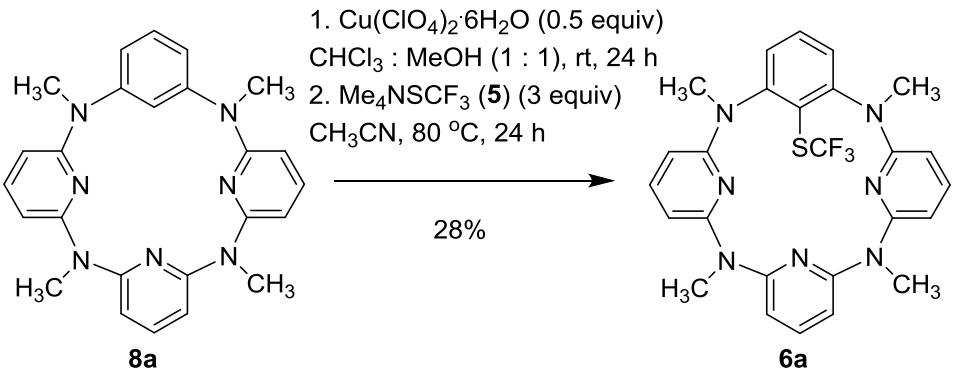


8a-f **6a-f**

entry	1	2	3	4	5	6
8	**8a**	**8b**	**8c**	**8d**	**8e**	**8f**
R^1	H	CN	Cl	H	Me	MeO
R^2	H	H	H	Cl	H	H
6	**6a**	**6b**	**6c**	**6d**	**6e**	**6f**
%[a]	89	45	51	53	57	54

[a] Isolated yield.

4. The reaction of 8a with 5 using a substoichiometric amount of copper(II) salt (50 mol%)



8a **6a**

5. References

(1). W. Tyrra and S. Kremer, Synthesis of Zero-Valent Trifluoromethyl Chalcogenato Derivatives, $[NMe_4]ECF_3$ (E = S, Se, Te), and Related Compounds, in Efficient Preparations of Fluorine Compounds (ed H. W. Roesky), 2012, John Wiley & Sons, Inc., Hoboken, NJ, USA.

(2). C.-F. Xu, B.-Q. Ma and Q. Shen, *Angew. Chem. Int. Ed.*, 2014, **53**, 9316.

(3). J. H. Clark, C. W. Jones, A. P. Kybett, M. A. McClinton, J. M. Miller, D. Bishop and R. J. Blade, *J. Fluorine Chem.*, 1990, **48**, 249.

6. Crystal data and structure refinement for each X-ray structure

structure **6a**

Empirical formula	C27 H26 Cl2 F3 N7 S
Formula weight	608.51
Temperature	173.1500 K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	P b c a
Unit cell dimensions	a = 16.558(3) Å a= 90 °.
b = 17.307(4) Å	b= 90 °.
c = 19.571(4) Å	g = 90 °.
Volume	5608.3(19) $Å^3$
Z	8
Density (calculated)	1.441 Mg/m^3
Absorption coefficient	0.358 mm^{-1}
F(000)	2512
Crystal size	0.48 x 0.44 x 0.26 mm^3
Theta range for data collection	1.995 to 27.459 °.
Index ranges	-21<=h<=21, -22<=k<=22, -25<=l<=25
Reflections collected	37998
Independent reflections	6385 [R(int) = 0.0483]
Completeness to theta = 26.000 °	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.7445
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6385 / 86 / 405
Goodness-of-fit on F^2	1.243
Final R indices [I>2sigma(I)]	R1 = 0.0895, wR2 = 0.2241
R indices (all data)	R1 = 0.0947, wR2 = 0.2349
Extinction coefficient	n/a
Largest diff. peak and hole	0.820 and -0.874 $e.Å^{-3}$

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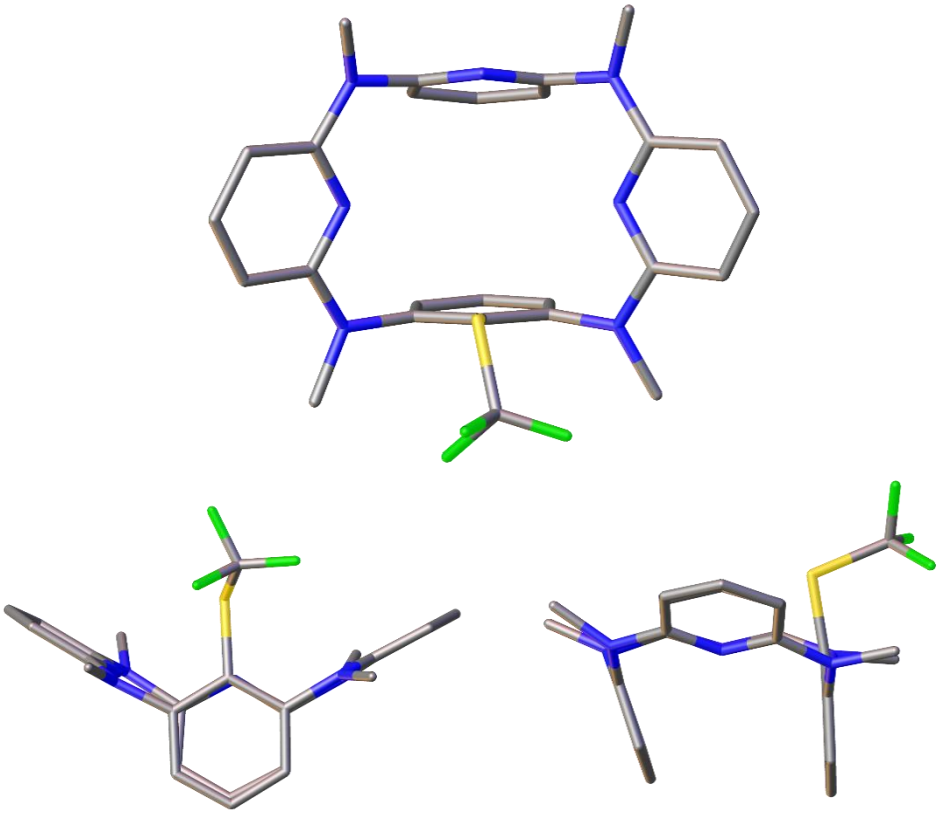

Figure 3. X-ray molecular structure of **6a** with top (top) and side (bottom) views

structure **7b**

Empirical formula	C27 H23 F6 N7 S2	
Formula weight	623.64	
Temperature	173.1500 K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 9.817(2) Å	a= 102.73(3) °.
b = 11.968(2) Å	b= 99.17(3) °.	
c = 13.149(3) Å	g = 104.43(3) °.	
Volume	1421.2(6) $Å^3$	
Z	2	
Density (calculated)	1.457 Mg/m^3	
Absorption coefficient	0.258 mm^{-1}	
F(000)	640	
Crystal size	0.39 x 0.38 x 0.11 mm^3	

Theta range for data collection	2.910 to 27.478 °.
Index ranges	-12<=h<=12, -15<=k<=15, -17<=l<=16
Reflections collected	17722
Independent reflections	6458 [R(int) = 0.0293]
Completeness to theta = 26.000 °	99.4 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.8543
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6458 / 0 / 383
Goodness-of-fit on F^2	1.290
Final R indices [I>2sigma(I)]	R1 = 0.0491, wR2 = 0.1644
R indices (all data)	R1 = 0.0544, wR2 = 0.1691
Extinction coefficient	n/a
Largest diff. peak and hole	0.384 and -0.317 e.$Å^{-3}$

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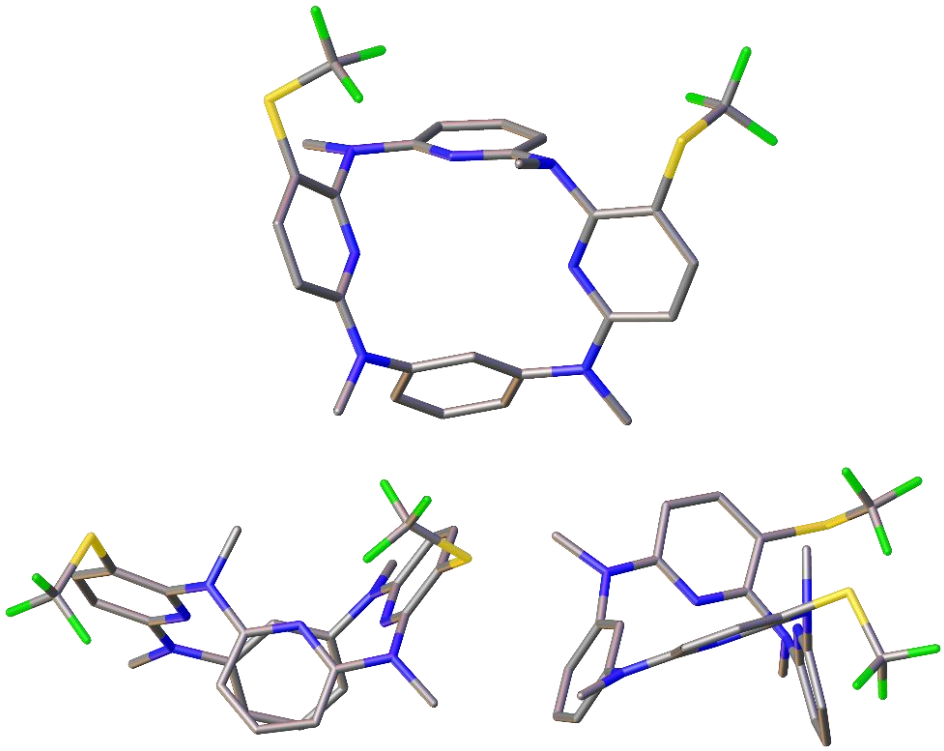

Figure S2. X-ray molecular structure of **7b** with top (top) and side (bottom) views

structure **7c**

Empirical formula	C27 H23 F6 N7 S2
Formula weight	623.64

Temperature	173.1500 K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P 1 21/c 1
Unit cell dimensions	a = 14.717(3) Å a= 90 °.
b = 15.564(3) Å	b= 103.48(3) °.
c = 12.282(3) Å	g = 90 °.
Volume	2735.8(10) $Å^3$
Z	4
Density (calculated)	1.514 Mg/m^3
Absorption coefficient	0.268 mm^{-1}
F(000)	1280
Crystal size	0.46 x 0.28 x 0.1 mm^3
Theta range for data collection	1.933 to 27.474 °.
Index ranges	-19<=h<=19, -20<=k<=20, -12<=l<=15
Reflections collected	20197
Independent reflections	6235 [R(int) = 0.0448]
Completeness to theta = 26.000 °	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.7230
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6235 / 5 / 383
Goodness-of-fit on F^2	1.181
Final R indices [I>2sigma(I)]	R1 = 0.0659, wR2 = 0.1403
R indices (all data)	R1 = 0.0733, wR2 = 0.1443
Extinction coefficient	n/a
Largest diff. peak and hole	0.405 and -0.378 $e.Å^{-3}$

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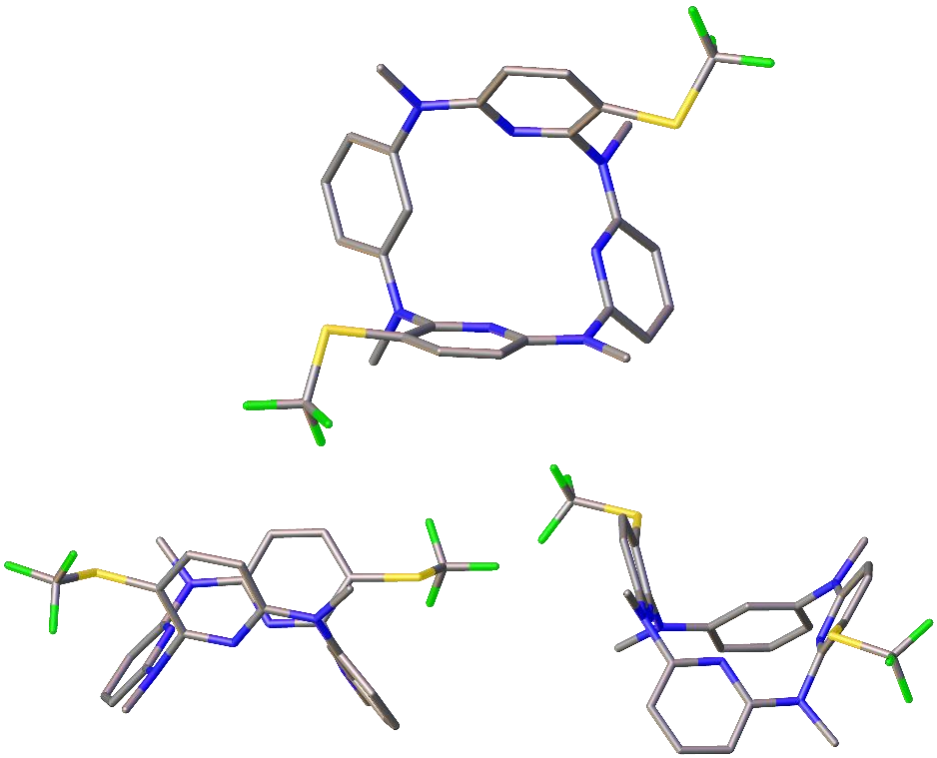


Figure S3. X-ray molecular structure of **7c** with top (top) and side (bottom) views

7. Atomic coordinates for each X-ray structure

Atomic coordinates (x 104) and equivalent isotropic displacement parameters (Å2x 103)

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

Structure of **6a**

S1	9684(1)	2084(1)	3894(1)	36(1)
N1	9053(2)	3313(2)	4904(1)	31(1)
N2	9595(2)	4120(2)	4094(1)	30(1)
N3	10258(2)	4918(2)	3341(2)	42(1)
N4	11289(2)	4015(2)	3109(1)	35(1)
N5	12218(2)	3023(2)	2868(2)	45(1)
N6	11707(2)	2161(2)	3656(1)	30(1)
N7	11208(2)	1392(2)	4511(2)	33(1)
C1	8887(2)	1487(2)	4205(2)	41(1)
C2	10157(2)	2401(2)	4656(2)	26(1)
C3	9849(2)	3026(2)	5032(2)	26(1)
C4	10293(2)	3320(2)	5571(2)	33(1)
C5	11030(2)	2999(2)	5750(2)	35(1)
C6	11325(2)	2373(2)	5388(2)	33(1)
C7	10901(2)	2065(2)	4841(2)	28(1)

C8	8444(2)	3148(2)	5427(2)	39(1)
C9	8935(2)	3891(2)	4432(2)	28(1)
C10	8170(2)	4223(2)	4317(2)	36(1)
C11	8135(2)	4804(2)	3829(2)	42(1)
C12	8800(2)	5050(2)	3479(2)	40(1)
C13	9540(2)	4692(2)	3633(2)	33(1)
C14	10288(3)	5377(3)	2718(2)	55(1)
C15	10979(2)	4522(2)	3550(2)	32(1)
C16	11930(2)	3615(2)	3316(2)	34(1)
C17	12297(2)	3719(2)	3947(2)	35(1)
C18	11966(2)	4244(2)	4390(2)	35(1)
C19	11287(2)	4658(2)	4193(2)	34(1)
C20	12308(4)	3216(3)	2145(2)	67(2)
C21	12104(2)	2261(2)	3066(2)	36(1)
C22	12389(2)	1642(2)	2672(2)	42(1)
C23	12253(2)	910(2)	2918(2)	39(1)
C24	11845(2)	786(2)	3522(2)	35(1)
C25	11587(2)	1441(2)	3882(2)	30(1)
C26	11003(2)	644(2)	4802(2)	44(1)
F1	8178(4)	1860(7)	4150(8)	80(3)
F2	8950(4)	1264(6)	4850(3)	67(2)
F3	9050(30)	756(8)	4030(20)	75(9)
Cl1	9681(1)	1148(1)	6389(1)	99(1)
C27	9664(5)	1589(3)	7182(3)	89(2)
Cl2	9330(4)	2534(2)	7132(1)	82(1)
F2A	9207(14)	928(13)	4596(18)	76(7)
Cl2A	9850(20)	2558(6)	7176(7)	93(6)
F1A	8220(9)	1825(8)	4430(17)	87(6)
F3A	8706(4)	952(5)	3754(4)	81(2)

Structure of **7b**

x	y	z	U(eq)	
S1	8192(1)	398(1)	4022(1)	34(1)
S2	7209(1)	2431(1)	10724(1)	46(1)
F1	9288(2)	1633(2)	2776(1)	69(1)
F2	10762(1)	1038(1)	3760(1)	54(1)
F3	10033(2)	2532(1)	4423(1)	59(1)

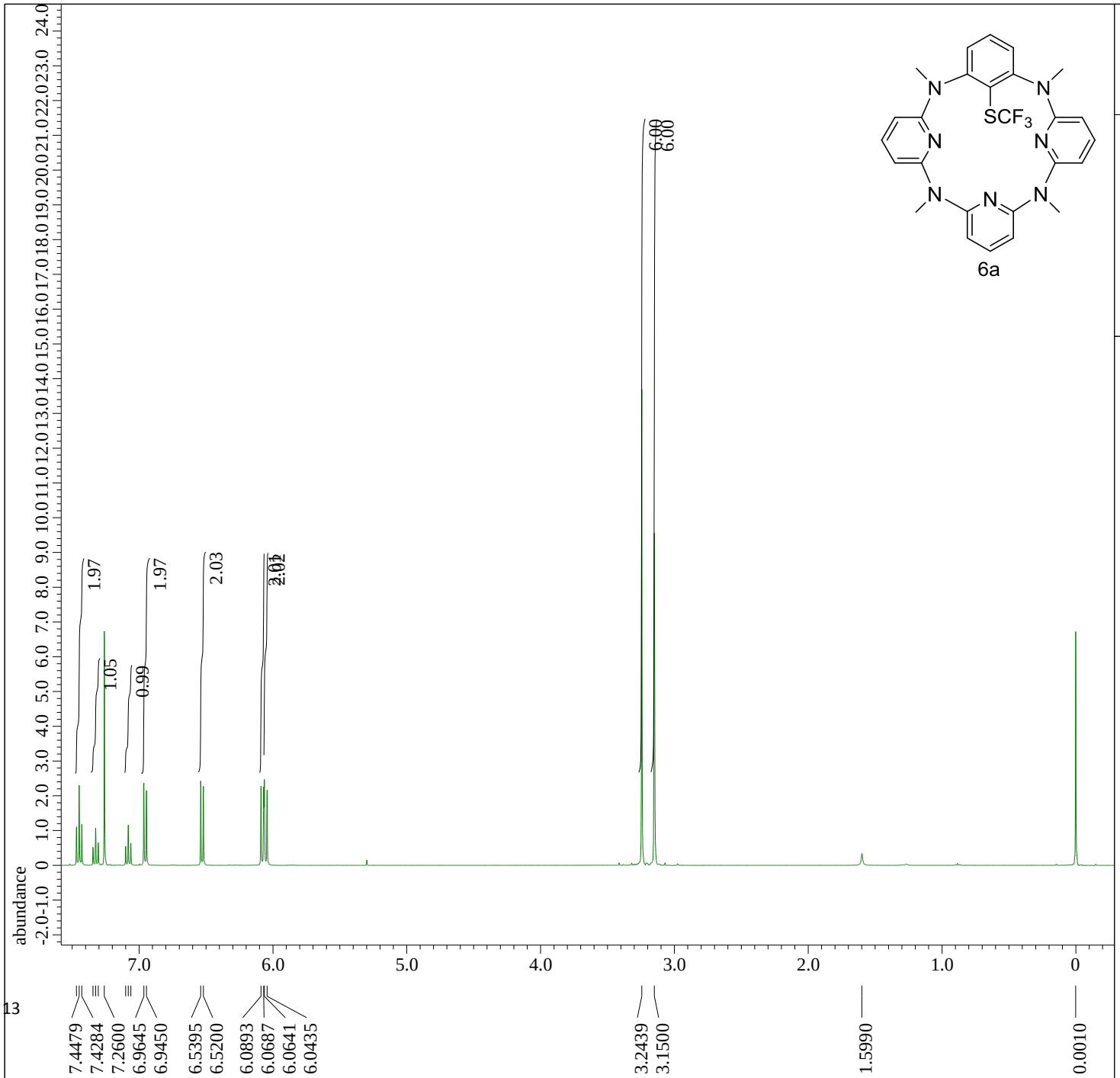
F4	7054(2)	540(2)	9187(2)	82(1)
F5	8820(2)	2032(2)	9342(1)	66(1)
F6	8840(2)	1074(2)	10525(1)	96(1)
N1	5675(2)	2425(1)	4947(1)	24(1)
N2	7481(2)	1898(1)	5953(1)	24(1)
N3	7479(2)	3099(1)	7618(1)	26(1)
N4	7372(2)	4149(1)	9276(1)	32(1)
N5	4927(2)	3382(1)	8399(1)	28(1)
N6	2546(2)	2733(1)	7479(1)	30(1)
N7	3828(2)	2998(1)	4075(1)	29(1)
C1	4833(2)	2380(2)	4016(1)	25(1)
C2	4980(2)	1734(2)	3025(1)	30(1)
C3	6006(2)	1129(2)	3029(2)	31(1)
C4	6890(2)	1180(2)	3993(1)	27(1)
C5	6667(2)	1845(1)	4936(1)	23(1)
C6	9617(2)	1450(2)	3735(2)	36(1)
C7	6857(2)	874(2)	6340(2)	34(1)
C8	7903(2)	3040(1)	6708(1)	23(1)
C9	8808(2)	4019(2)	6485(1)	27(1)
C10	9297(2)	5085(2)	7278(2)	31(1)
C11	8862(2)	5182(2)	8239(2)	31(1)
C12	7918(2)	4164(2)	8366(1)	26(1)
C13	7676(3)	5273(2)	10100(2)	47(1)
C14	5990(2)	3314(2)	9126(1)	30(1)
C15	5791(2)	2516(2)	9757(2)	36(1)
C16	4392(2)	1775(2)	9616(2)	42(1)
C17	3274(2)	1826(2)	8871(2)	37(1)
C18	3585(2)	2642(2)	8248(1)	28(1)
C19	8006(3)	1484(2)	9904(2)	48(1)
C20	1009(2)	2177(2)	7395(2)	33(1)
C21	2886(2)	3427(2)	6741(1)	26(1)
C22	2638(2)	4539(2)	6876(2)	32(1)
C23	2825(2)	5138(2)	6100(2)	34(1)
C24	3277(2)	4660(2)	5203(2)	30(1)
C25	3522(2)	3539(2)	5058(1)	26(1)
C26	3332(2)	2927(2)	5840(1)	26(1)
C27	3081(2)	3161(2)	3089(2)	42(1)

Structure of **7c**

x	y	z	U(eq)	
S1	3027(1)	6050(1)	8270(1)	36(1)
S2	1186(1)	504(1)	6513(1)	40(1)
F1	3075(1)	7666(1)	8568(2)	60(1)
F2	2395(2)	7309(1)	6892(2)	71(1)
F3	1684(1)	7174(1)	8190(2)	76(1)
F4	1796(2)	-885(1)	7661(2)	66(1)
F5	2106(1)	-857(1)	6062(2)	61(1)
F6	691(2)	-1054(1)	6186(2)	75(1)
N1	3330(1)	5027(1)	6273(2)	34(1)
N2	1848(1)	4406(1)	5841(2)	26(1)
N3	424(1)	3718(1)	5325(2)	29(1)
N4	1494(2)	2568(1)	5353(2)	32(1)
N5	2584(2)	1452(1)	5462(2)	31(1)
N6	3445(1)	2007(1)	7140(2)	28(1)
N7	4344(2)	2644(2)	8722(2)	39(1)
C1	2427(2)	4946(2)	6499(2)	27(1)
C2	2184(2)	5410(2)	7360(2)	28(1)
C3	1277(2)	5300(2)	7514(2)	28(1)
C4	664(2)	4762(2)	6830(2)	28(1)
C5	979(2)	4297(2)	6004(2)	24(1)
C6	-553(2)	3604(2)	5355(2)	35(1)
C7	832(2)	3083(2)	4733(2)	26(1)
C8	523(2)	3010(2)	3588(2)	31(1)
C9	936(2)	2388(2)	3057(2)	35(1)
C10	1626(2)	1865(2)	3653(2)	31(1)
C11	1902(2)	1961(2)	4820(2)	26(1)
C12	2988(2)	735(2)	4988(2)	38(1)
C13	2757(2)	1491(2)	6654(2)	27(1)
C14	2246(2)	985(2)	7235(2)	31(1)
C15	2524(2)	1003(2)	8406(2)	34(1)
C16	3254(2)	1514(2)	8928(2)	34(1)
C17	3674(2)	2051(2)	8270(2)	31(1)
C18	1465(2)	-616(2)	6607(3)	44(1)
C19	4755(2)	2638(2)	9929(2)	49(1)
C20	4568(2)	3338(2)	8062(2)	34(1)
C21	5495(2)	3504(2)	8061(3)	44(1)
C22	5684(2)	4193(2)	7442(3)	50(1)

C23	4988(2)	4708(2)	6837(3)	40(1)
C24	4061(2)	4526(2)	6859(2)	29(1)
C25	3866(2)	3844(2)	7477(2)	29(1)
C26	3497(2)	5760(2)	5614(3)	45(1)
C27	2513(2)	7088(2)	7960(3)	44(1)

8. Copies of ^{1}H and ^{13}C NMR spectra



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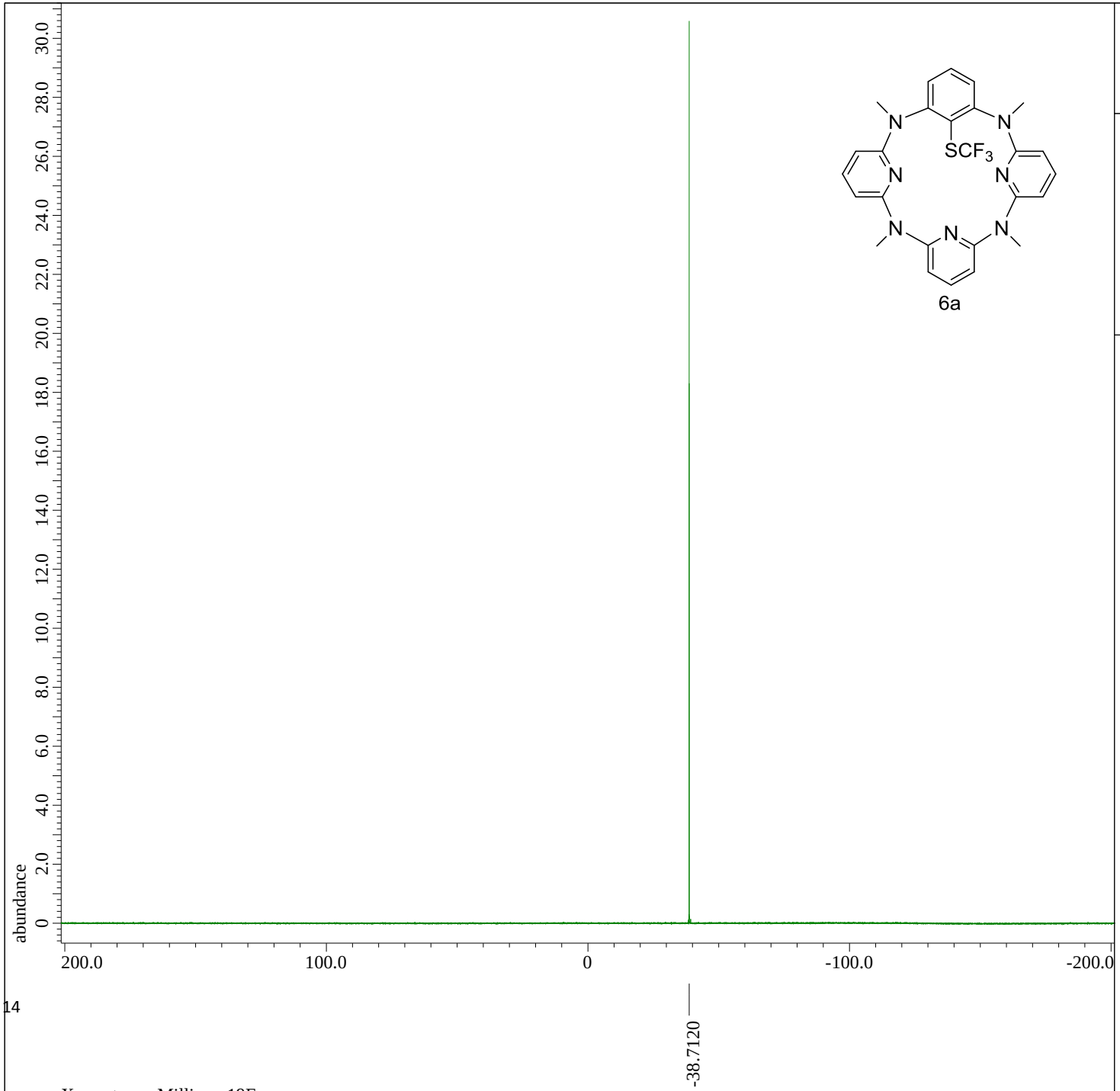

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6a

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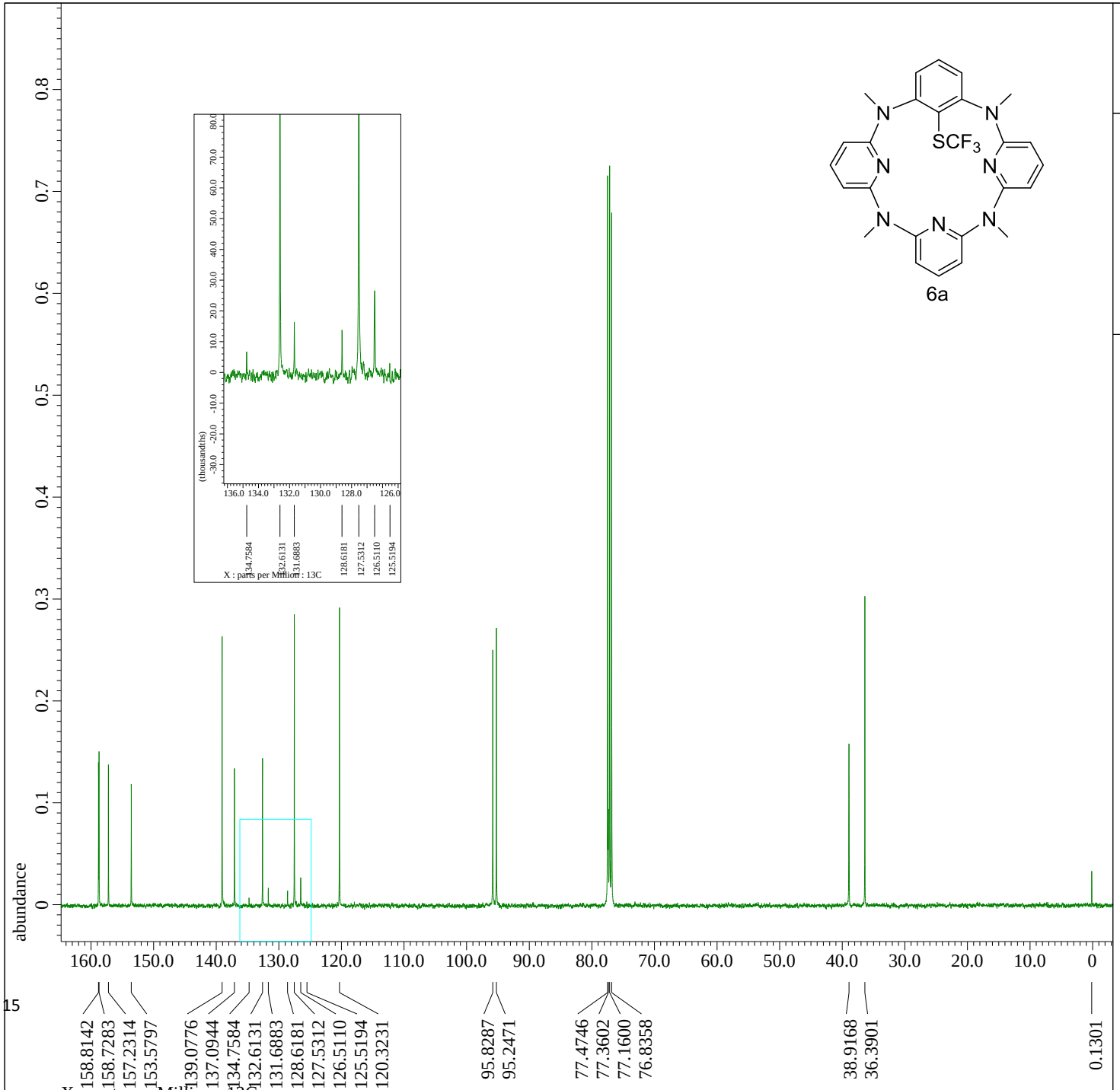
X_Sweep           = 189.39393939[kHz]
Irr_Domain        = 19F
Irr_Freq          = 376.17105393[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 19F
Tri_Freq          = 376.17105393[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 20.7[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 0.34603008[s]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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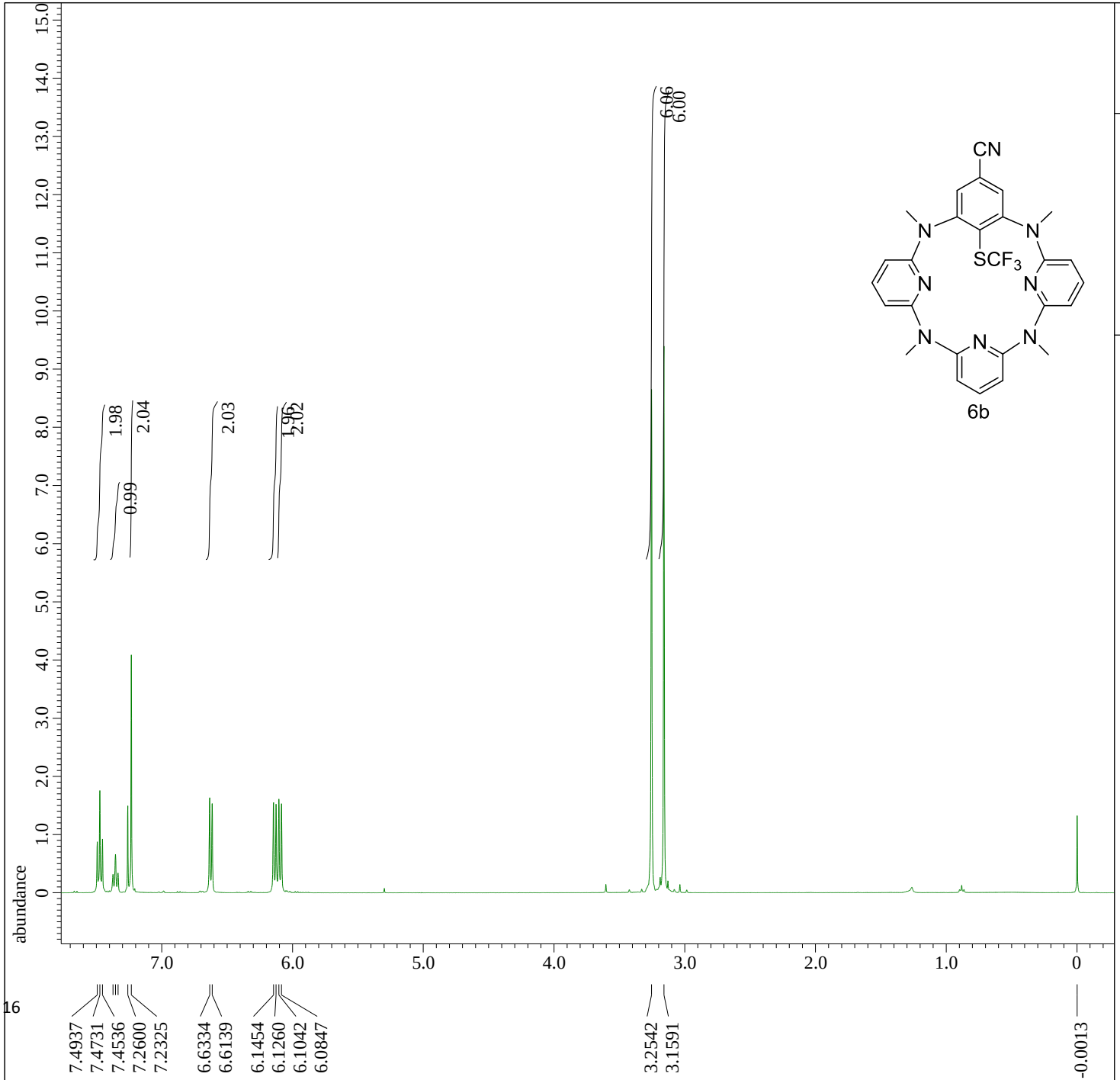
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename	= 150717-WF-467-CHECK-2-CDCL
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#330167
Solvent	= CHLOROFORM-D
Creation_Time	= 17-JUL-2015 11:03:09
Revision_Time	= 25-FEB-2016 10:38:55
Current_Time	= 25-FEB-2016 11:00:41
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 2000
Total_Scans	= 2000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 54
Temp_Get	= 21.7[dC]
X_90_Width	= 9.25[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 7.8[dB]
X_Pulse	= 3.08333333[us]
Irr_Atn_Dec	= 23.786[dB]
Irr_Atn_Noe	= 23.786[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE



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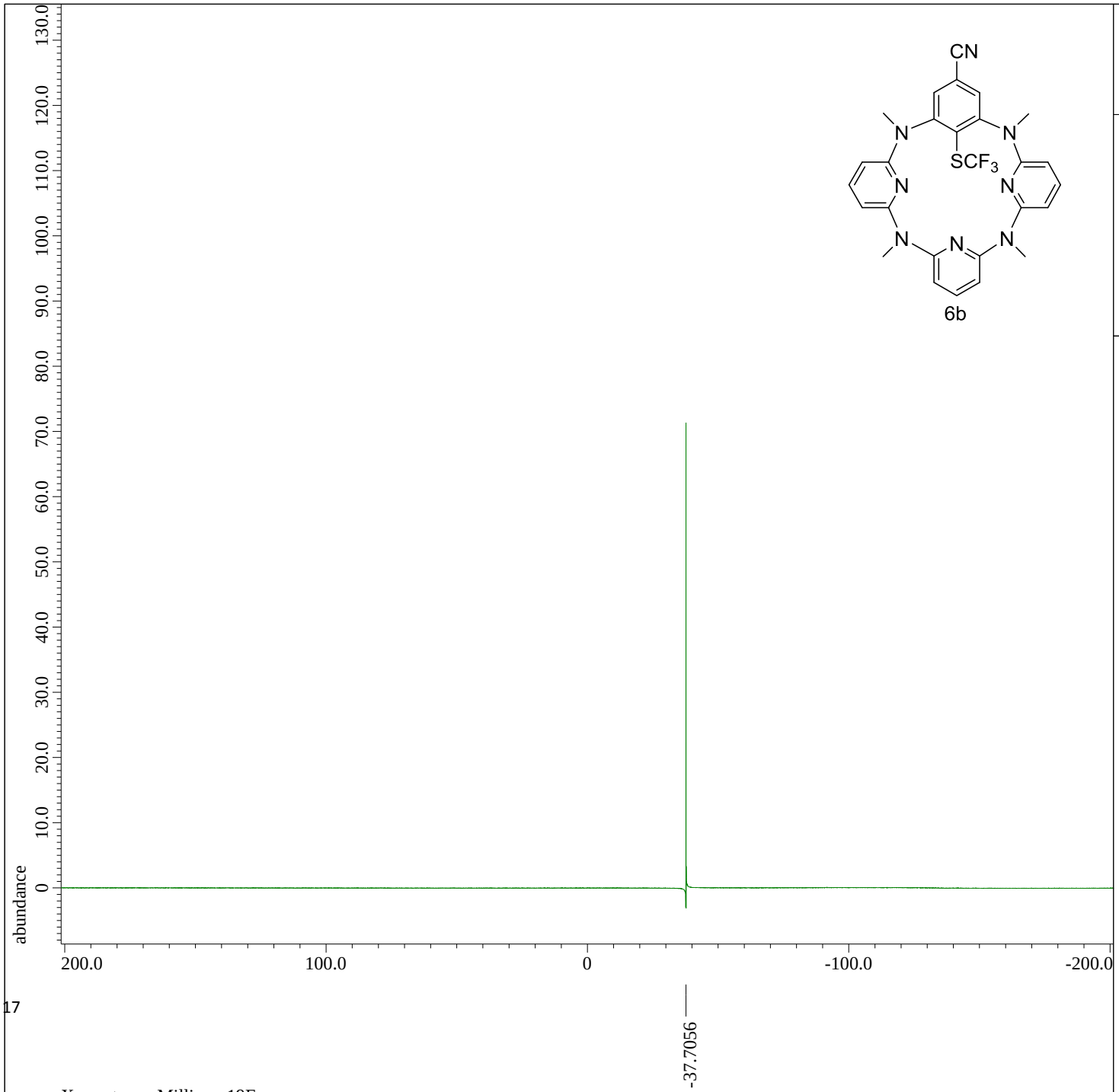
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 151210-wf-5-cn-calix-scf3-
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#808377
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2015 22:39:22
Revision_Time     = 25-FEB-2016 20:38:08
Current_Time      = 25-FEB-2016 21:37:24

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 42
Temp_Get          = 19.5[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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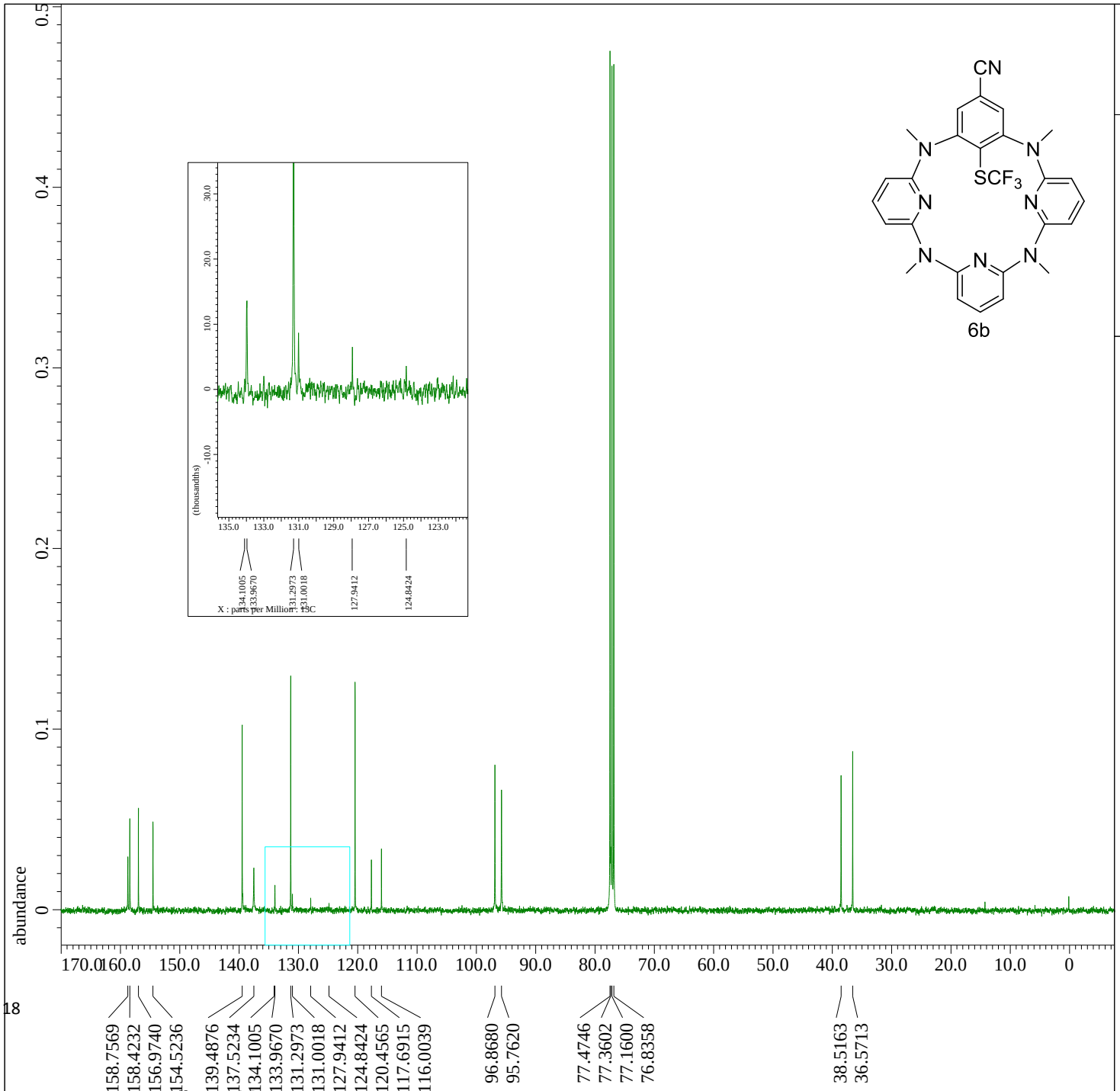
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
fft : 1 : TRUE : TRUE
fft : 1 : TRUE : TRUE
```

```
Filename          = 151210-wf-5-cn-calix-scf3-
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#810065
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2015 22:42:10
Revision_Time     = 23-DEC-2015 15:20:10
Current_Time      = 25-FEB-2016 13:37:18

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 19F
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 86.50752[ms]
X_Domain          = 19F
X_Freq            = 376.17105393[MHz]
X_Offset          = 0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 11.55968868[Hz]
X_Sweep           = 189.39393939[kHz]
Irr_Domain        = 19F
Irr_Freq          = 376.17105393[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 19F
Tri_Freq          = 376.17105393[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 19.4[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 86.50752[ms]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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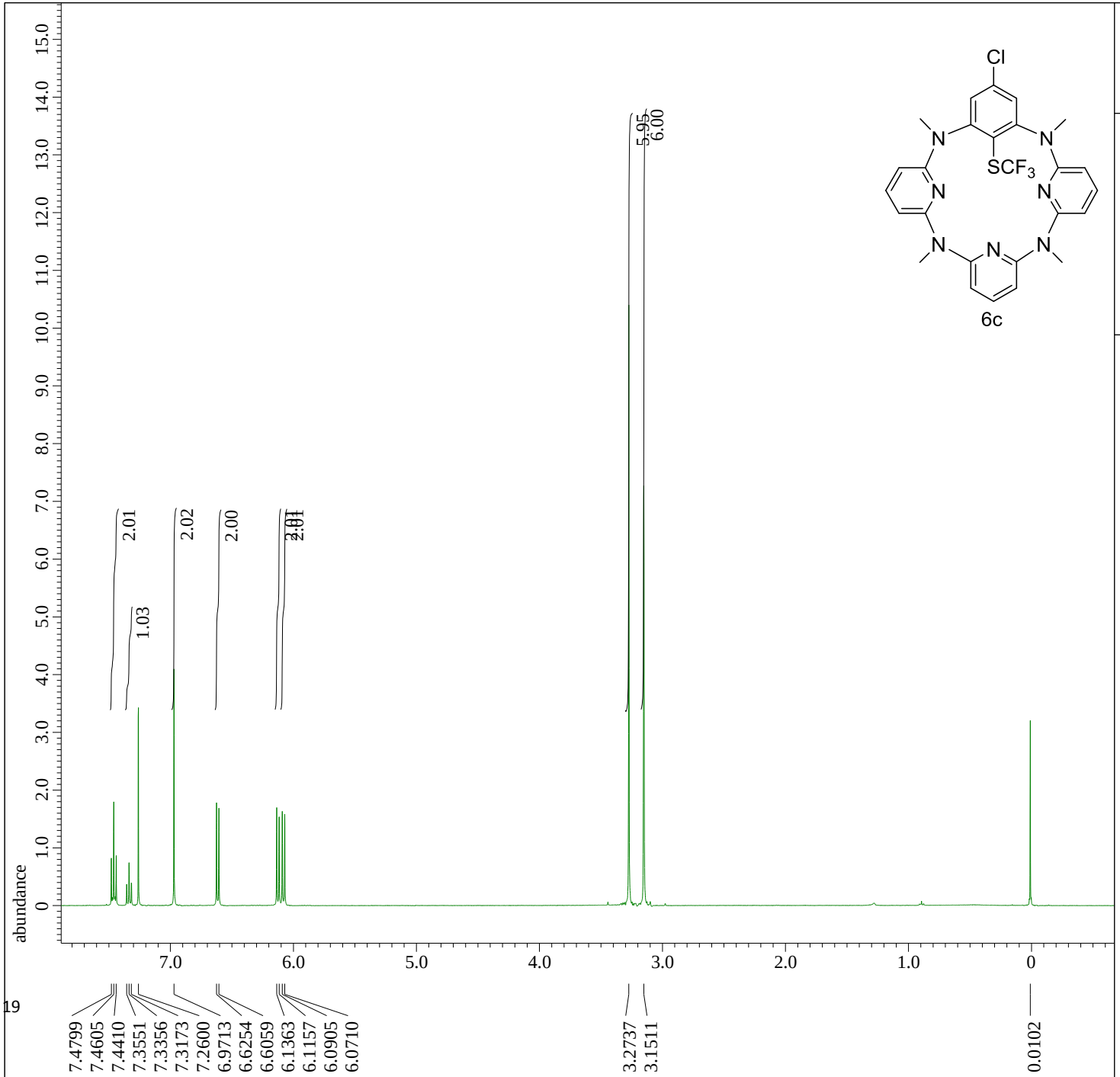
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

Filename	= 151210-wf-5-cn-calix-scf3-
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#811407
Solvent	= CHLOROFORM-D
Creation_Time	= 11-DEC-2015 00:26:14
Revision_Time	= 25-FEB-2016 13:39:13
Current_Time	= 25-FEB-2016 13:39:18
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 2000
Total_Scans	= 2000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 20.2[dC]
X_90_Width	= 11[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 7.8[dB]
X_Pulse	= 3.66666667[us]
Irr_Atn_Dec	= 23.03[dB]
Irr_Atn_Noe	= 23.03[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE



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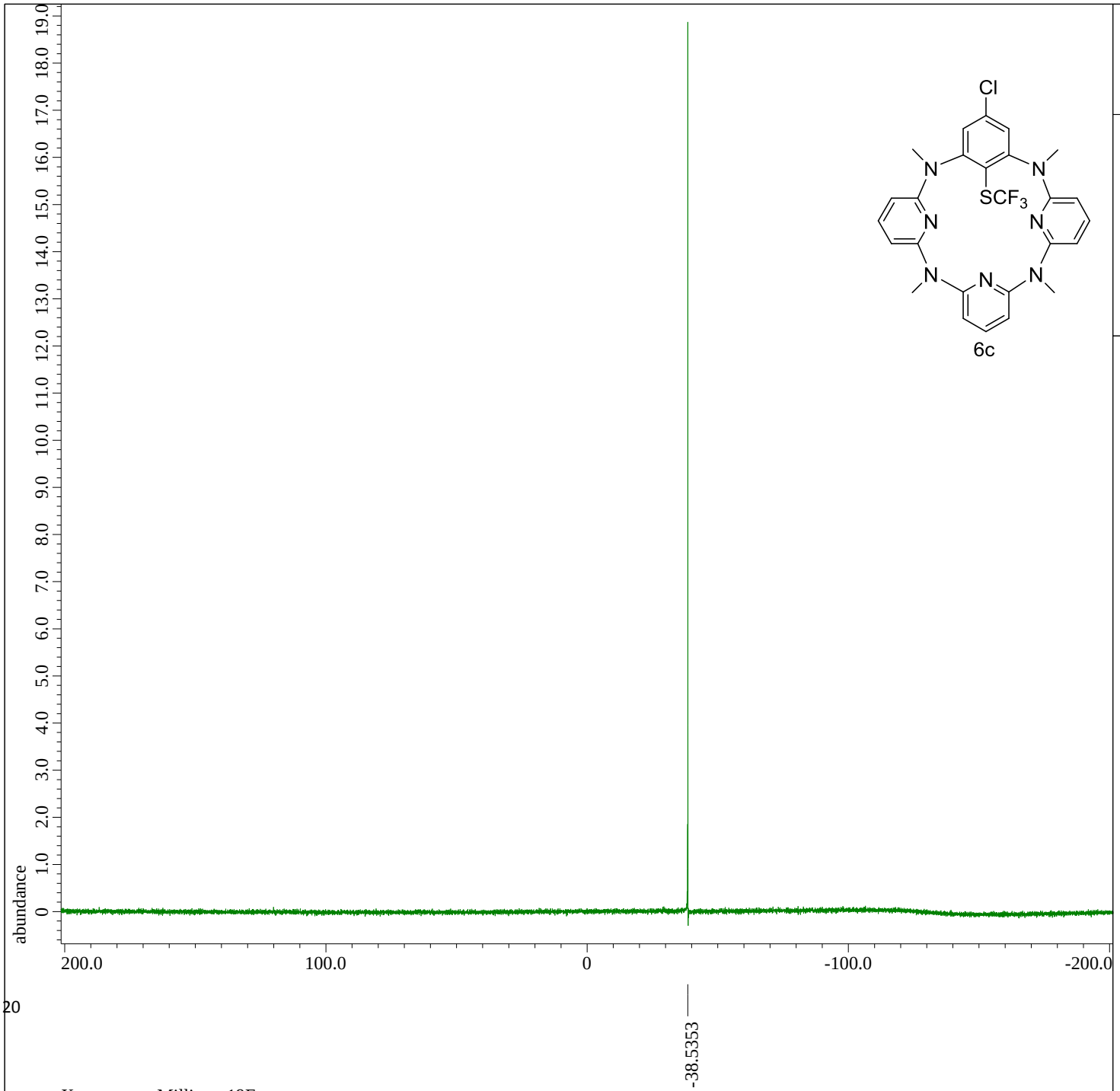
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 151212-wf-5-cl-scf3-cdcl3-
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#823337
Solvent           = CHLOROFORM-D
Creation_Time     = 12-DEC-2015 23:03:34
Revision_Time     = 25-FEB-2016 20:27:35
Current_Time      = 25-FEB-2016 21:39:21

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 45[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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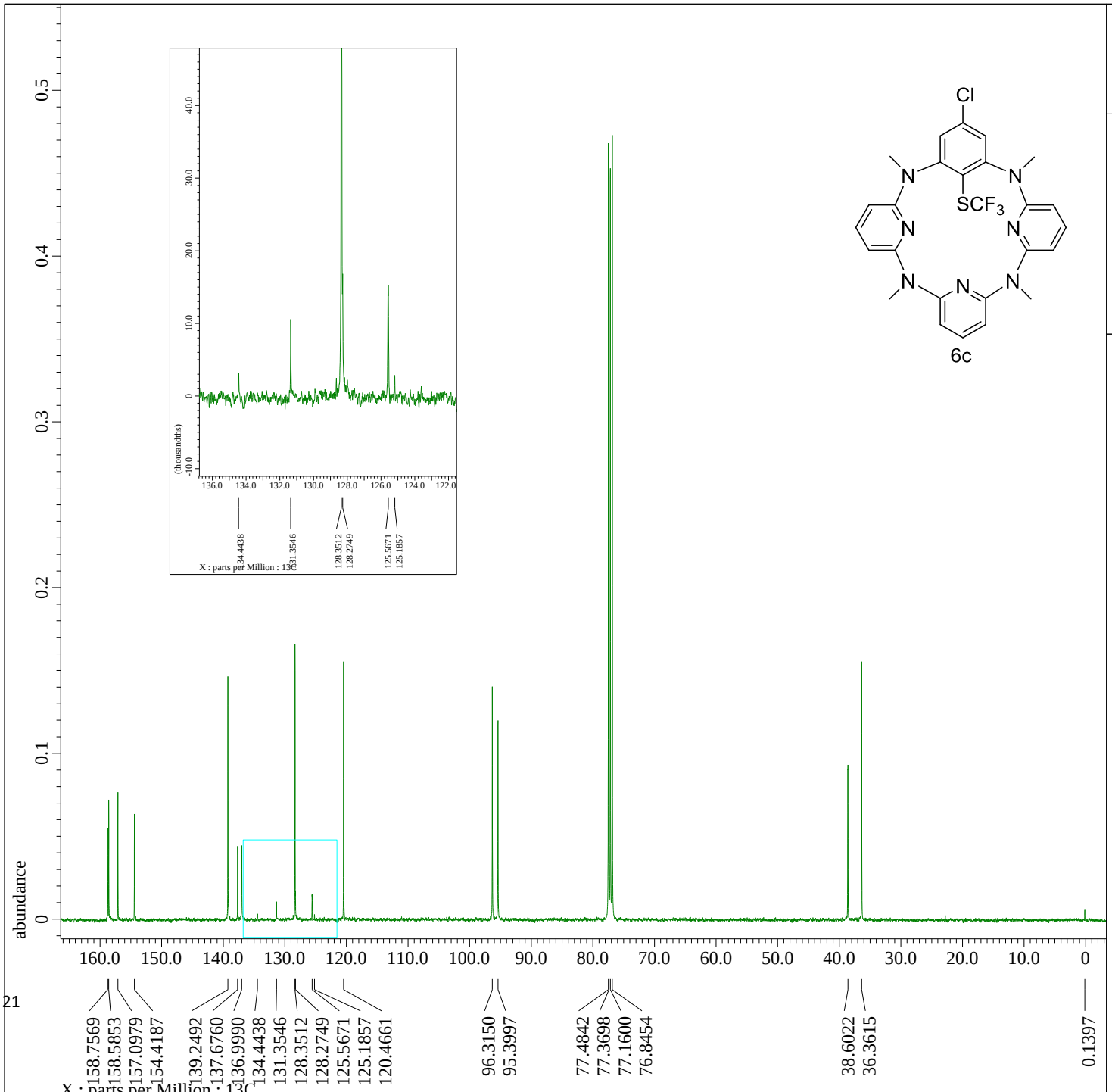
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 151212-wf-5-cl-scf3-cdcl3-
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#824476
Solvent           = CHLOROFORM-D
Creation_Time     = 12-DEC-2015 23:06:09
Revision_Time     = 23-DEC-2015 15:11:16
Current_Time      = 25-FEB-2016 13:59:33

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 19F
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 86.50752[ms]
X_Domain          = 19F
X_Freq            = 376.17105393[MHz]
X_Offset          = 0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 11.55968868[Hz]
X_Sweep           = 189.39393939[kHz]
Irr_Domain        = 19F
Irr_Freq          = 376.17105393[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 19F
Tri_Freq          = 376.17105393[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 45[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 86.50752[ms]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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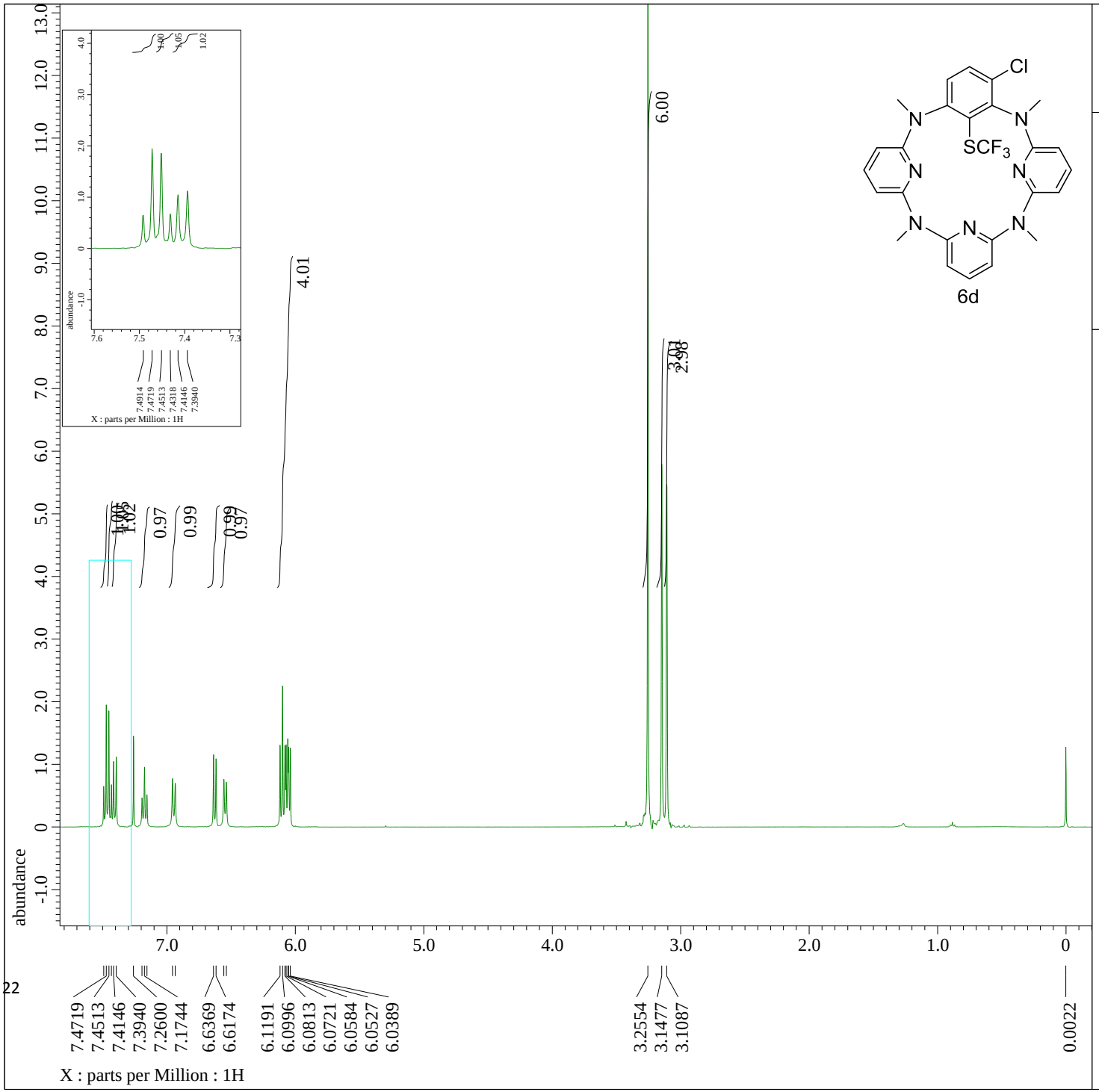
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

Filename	= 151212-wf-5-cl-scf3-cdcl3-
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#362926
Solvent	= CHLOROFORM-D
Creation_Time	= 13-DEC-2015 13:08:18
Revision_Time	= 25-FEB-2016 14:05:40
Current_Time	= 25-FEB-2016 14:06:07
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= TRUE
Scans	= 3375
Total_Scans	= 3375
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 20.3[dC]
X_90_Width	= 11[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 7.8[dB]
X_Pulse	= 3.66666667[us]
Irr_Atn_Dec	= 23.03[dB]
Irr_Atn_Noe	= 23.03[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE



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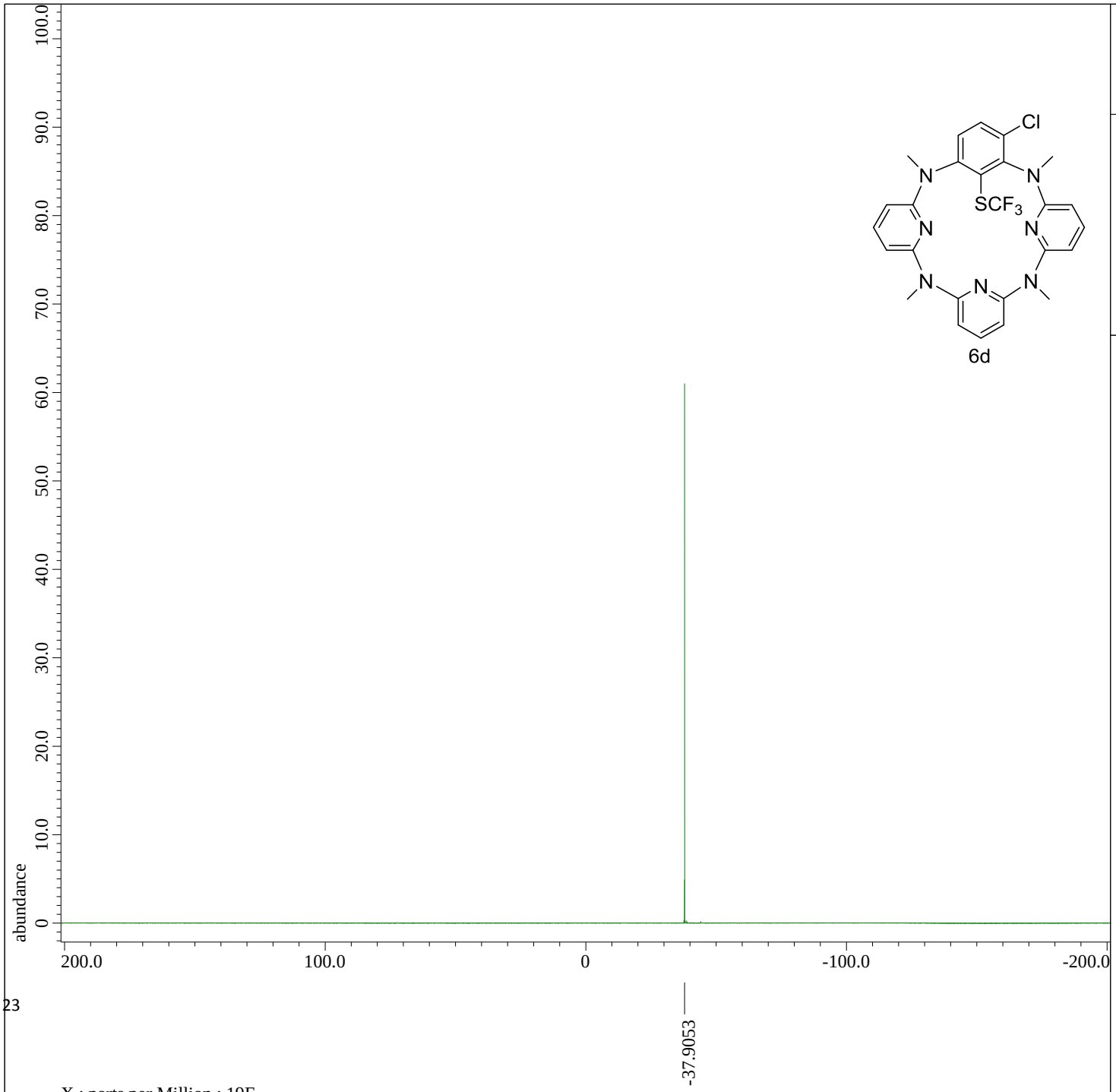
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

Filename	= 151210-wf-4-cl-calix-scf3-
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= S#821134
Solvent	= CHLOROFORM-D
Creation_Time	= 14-DEC-2015 23:00:23
Revision_Time	= 29-FEB-2016 13:11:21
Current_Time	= 29-FEB-2016 13:11:25
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 40
Temp_Get	= 19.6[dC]
X_90_Width	= 12[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 3.4[dB]
X_Pulse	= 6[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE



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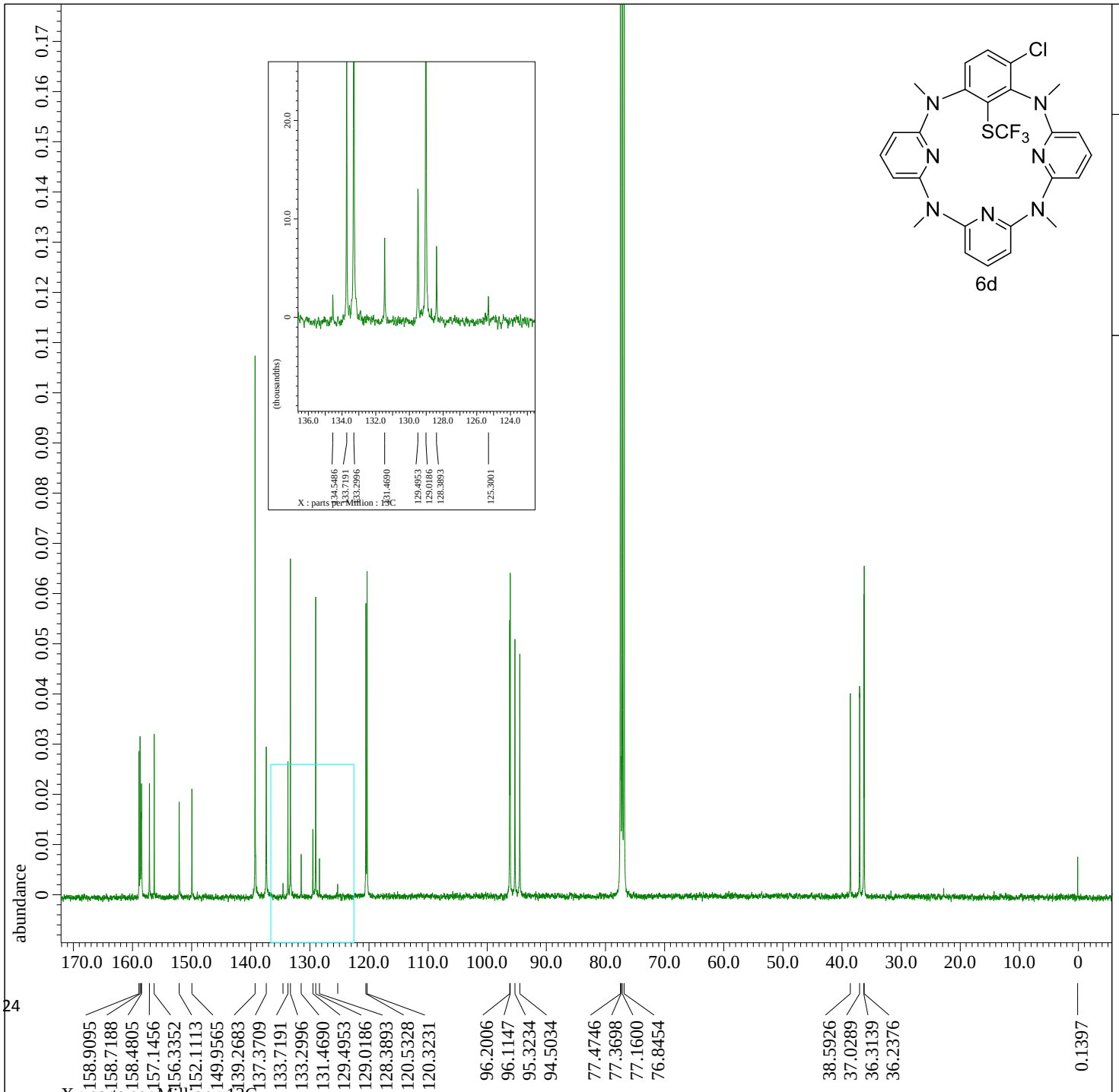
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename	= 151210-wf-4-cl-calix-scf3-
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= S#822646
Solvent	= CHLOROFORM-D
Creation_Time	= 14-DEC-2015 23:03:11
Revision_Time	= 23-DEC-2015 15:15:45
Current_Time	= 25-FEB-2016 14:43:11
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 52428
Dim_Title	= 19F
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 0.34603008[s]
X_Domain	= 19F
X_Freq	= 376.17105393[MHz]
X_Offset	= 0[ppm]
X_Points	= 65536
X_Prescans	= 1
X_Resolution	= 2.88992217[Hz]
X_Sweep	= 189.39393939[kHz]
Irr_Domain	= 19F
Irr_Freq	= 376.17105393[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 19F
Tri_Freq	= 376.17105393[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 50
Temp_Get	= 19.5[dC]
X_90_Width	= 12[us]
X_Acq_Time	= 0.34603008[s]
X_Angle	= 45[deg]
X_Atn	= 3.6[dB]
X_Pulse	= 6[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE



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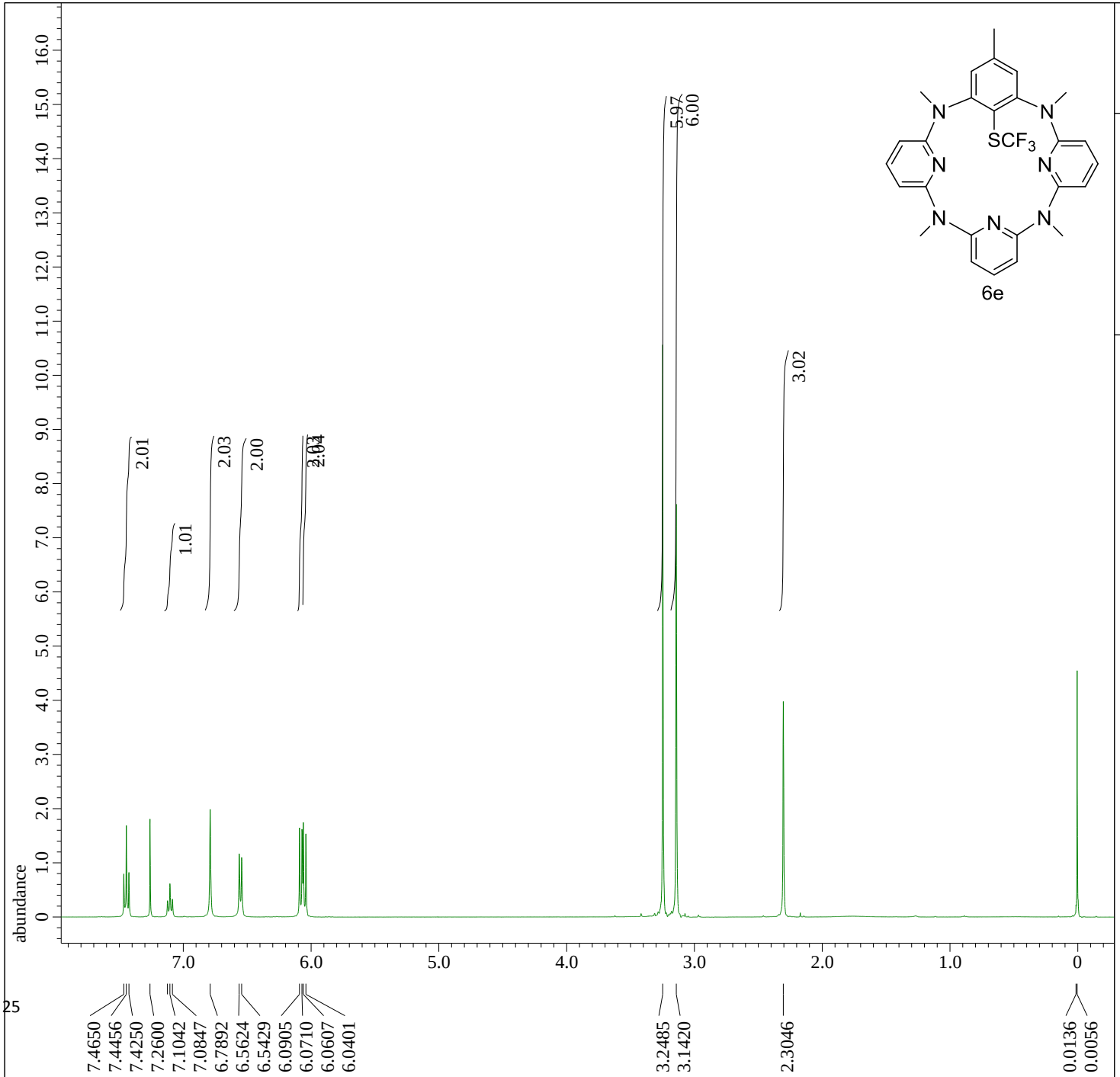
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
fft : 1 : TRUE : TRUE
```

Filename	= 151210-wf-4-cl-calix-scf3-
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#824311
Solvent	= CHLOROFORM-D
Creation_Time	= 15-DEC-2015 07:33:26
Revision_Time	= 25-FEB-2016 14:38:05
Current_Time	= 25-FEB-2016 14:38:17
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 10000
Total_Scans	= 10000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 20.1[dC]
X_90_Width	= 11[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 7.8[dB]
X_Pulse	= 3.66666667[us]
Irr_Atn_Dec	= 23.03[dB]
Irr_Atn_Noe	= 23.03[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE



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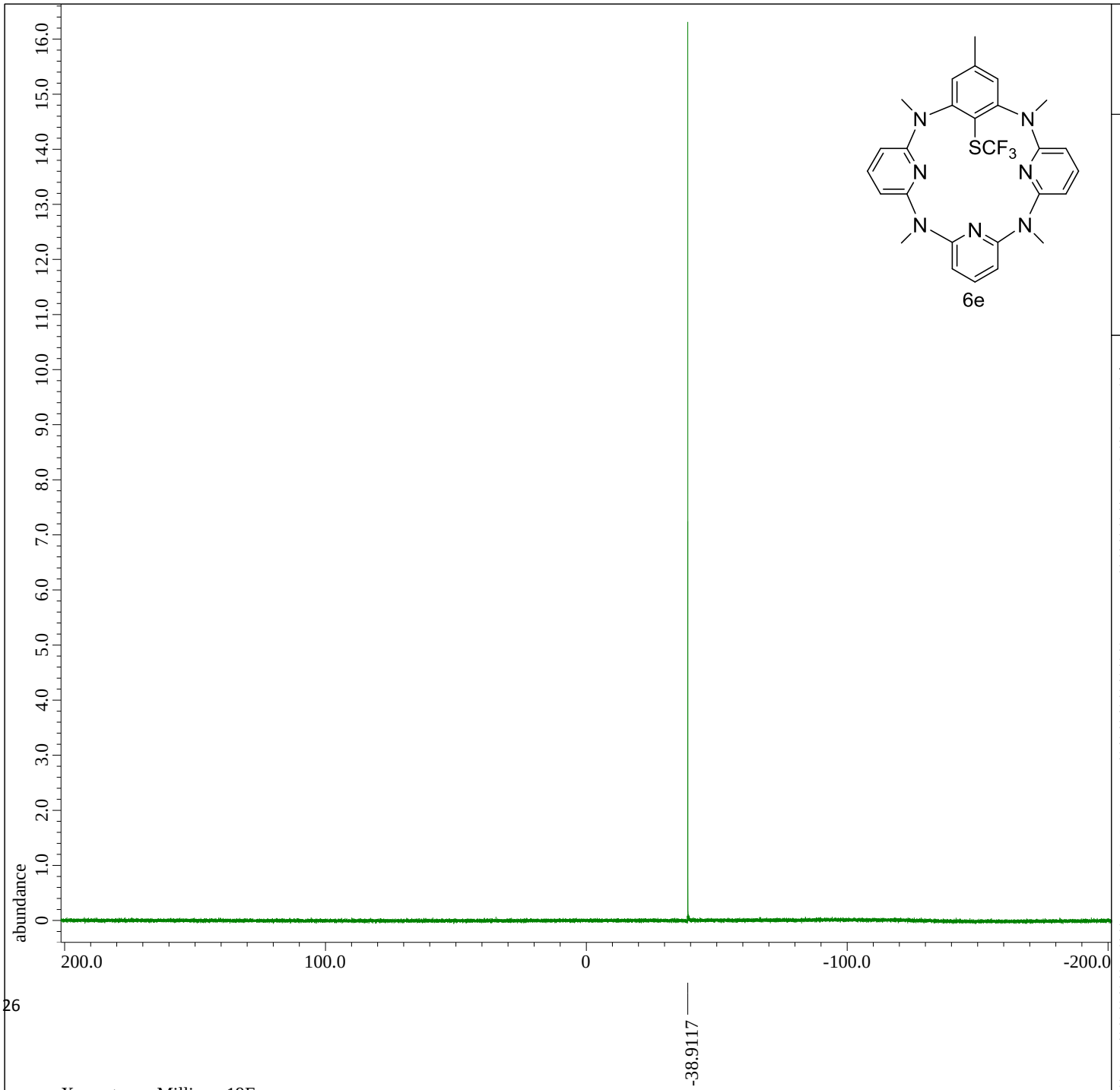
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 151208-wf-5-me-calix-scf3-
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#344553
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2015 09:47:26
Revision_Time     = 25-FEB-2016 20:25:52
Current_Time      = 25-FEB-2016 21:40:44

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 25.3[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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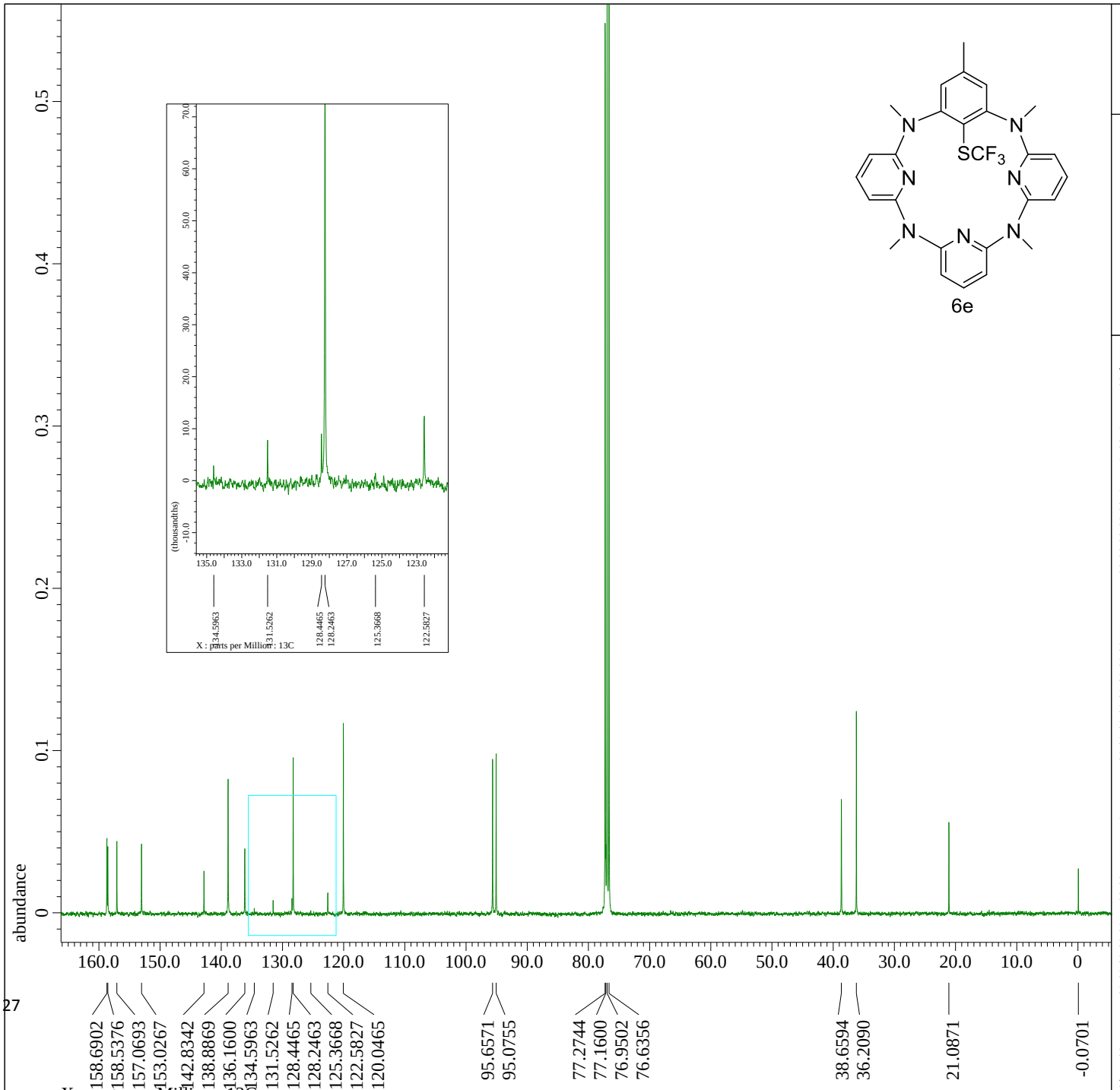
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 151208-wf-5-me-calix-scf3-
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#811654
Solvent           = CHLOROFORM-D
Creation_Time     =  8-DEC-2015 22:45:04
Revision_Time     = 23-DEC-2015 15:02:28
Current_Time      = 25-FEB-2016 12:41:32

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 52428
Dim_Title         = 19F
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 0.34603008[s]
X_Domain          = 19F
X_Freq            = 376.17105393[MHz]
X_Offset          = 0[ppm]
X_Points          = 65536
X_Prescans        = 1
X_Resolution      = 2.88992217[Hz]
X_Sweep           = 189.39393939[kHz]
Irr_Domain        = 19F
Irr_Freq          = 376.17105393[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 19F
Tri_Freq          = 376.17105393[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 52
Temp_Get          = 18.8[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 0.34603008[s]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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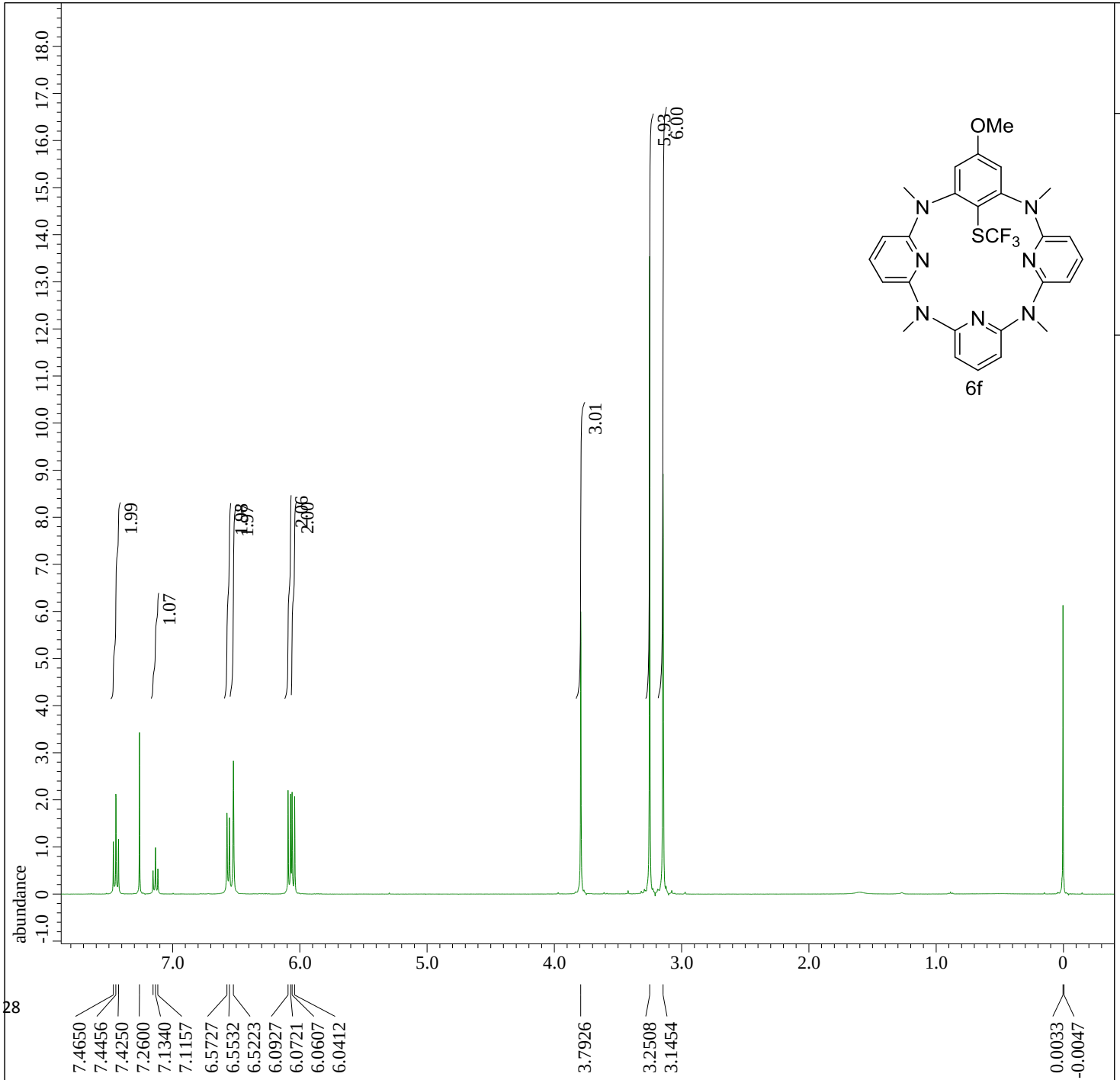
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 151208-wf-5-me-calix-scf3-
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#346651
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2015 12:47:44
Revision_Time     = 25-FEB-2016 12:44:36
Current_Time      = 25-FEB-2016 12:44:42

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 3503
Total_Scans       = 3503

Relaxation_Delay = 2[s]
Recvr_Gain        = 52
Temp_Get          = 26[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 3.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
```



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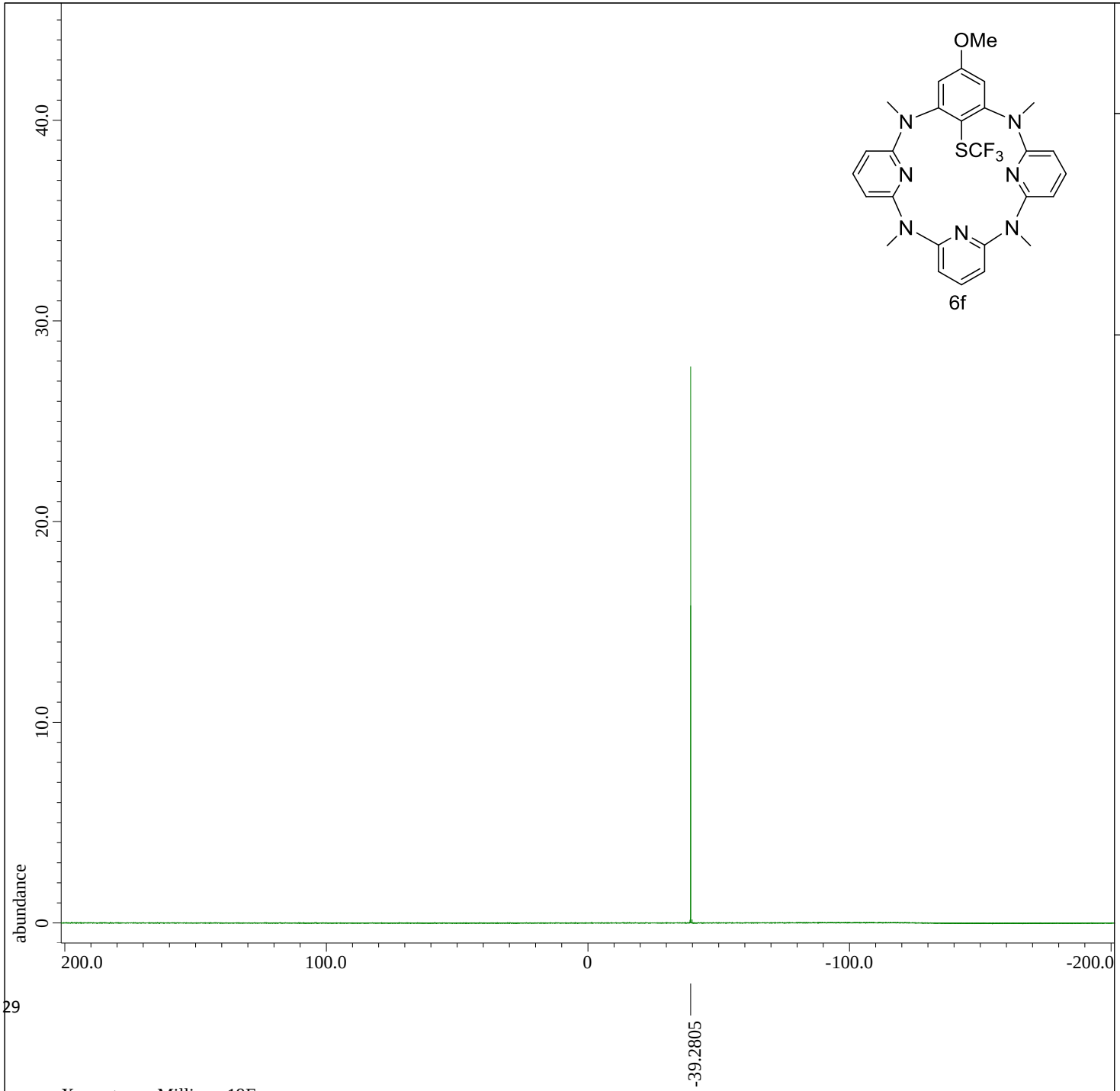
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 151207-wf-5-ome-calix-scf3
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#792125
Solvent           = CHLOROFORM-D
Creation_Time     =  7-DEC-2015 22:12:01
Revision_Time     = 25-FEB-2016 20:24:23
Current_Time      = 25-FEB-2016 21:41:30

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 24.9[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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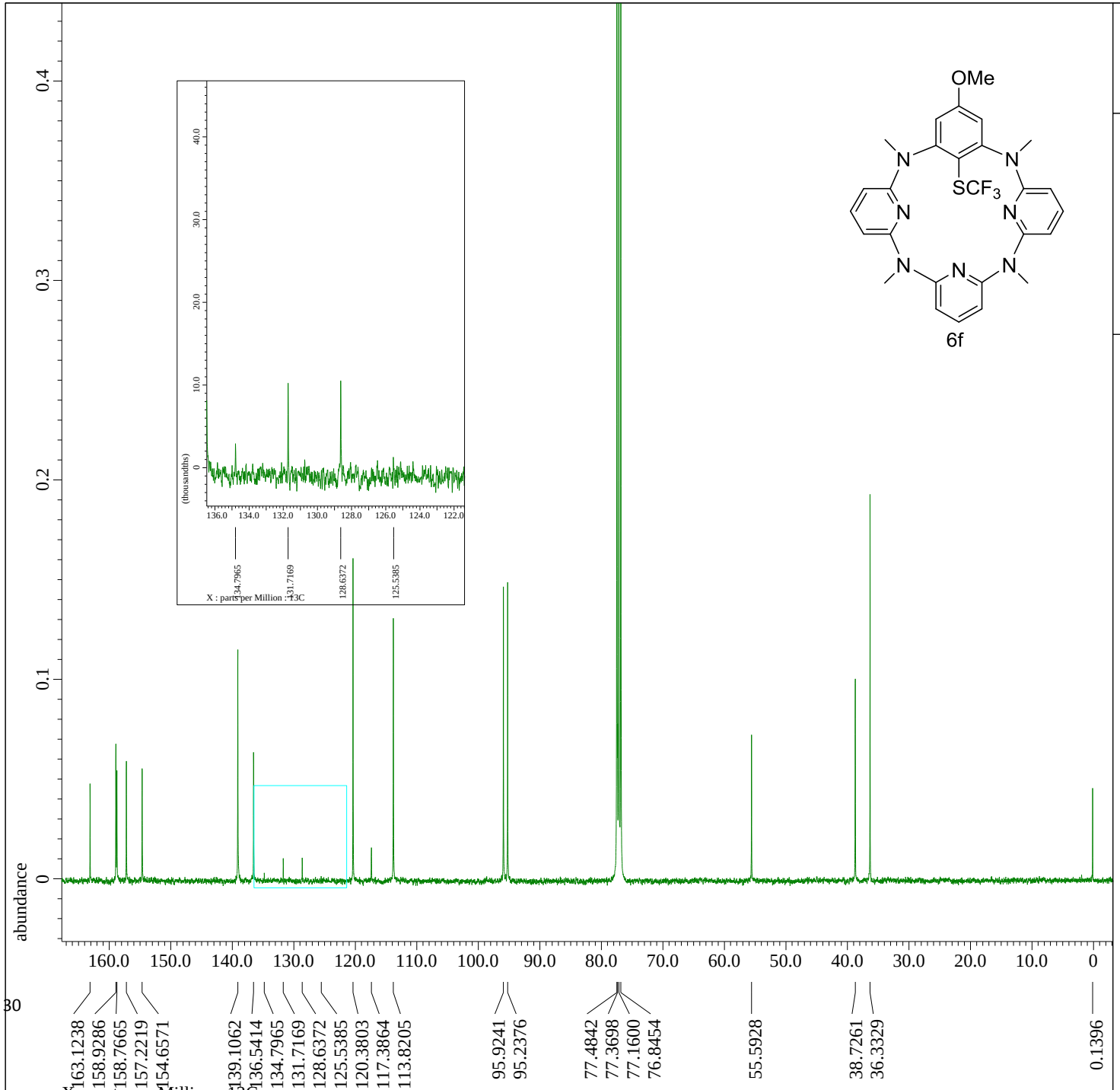
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 151207-wf-5-ome-calix-scf3
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#793569
Solvent           = CHLOROFORM-D
Creation_Time     =  7-DEC-2015 22:14:45
Revision_Time     = 23-DEC-2015 14:52:38
Current_Time      = 25-FEB-2016 14:57:16

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 52428
Dim_Title         = 19F
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 0.34603008[s]
X_Domain          = 19F
X_Freq            = 376.17105393[MHz]
X_Offset          = 0[ppm]
X_Points          = 65536
X_Prescans        = 1
X_Resolution      = 2.88992217[Hz]
X_Sweep           = 189.39393939[kHz]
Irr_Domain        = 19F
Irr_Freq          = 376.17105393[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 19F
Tri_Freq          = 376.17105393[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 50
Temp_Get          = 24.9[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 0.34603008[s]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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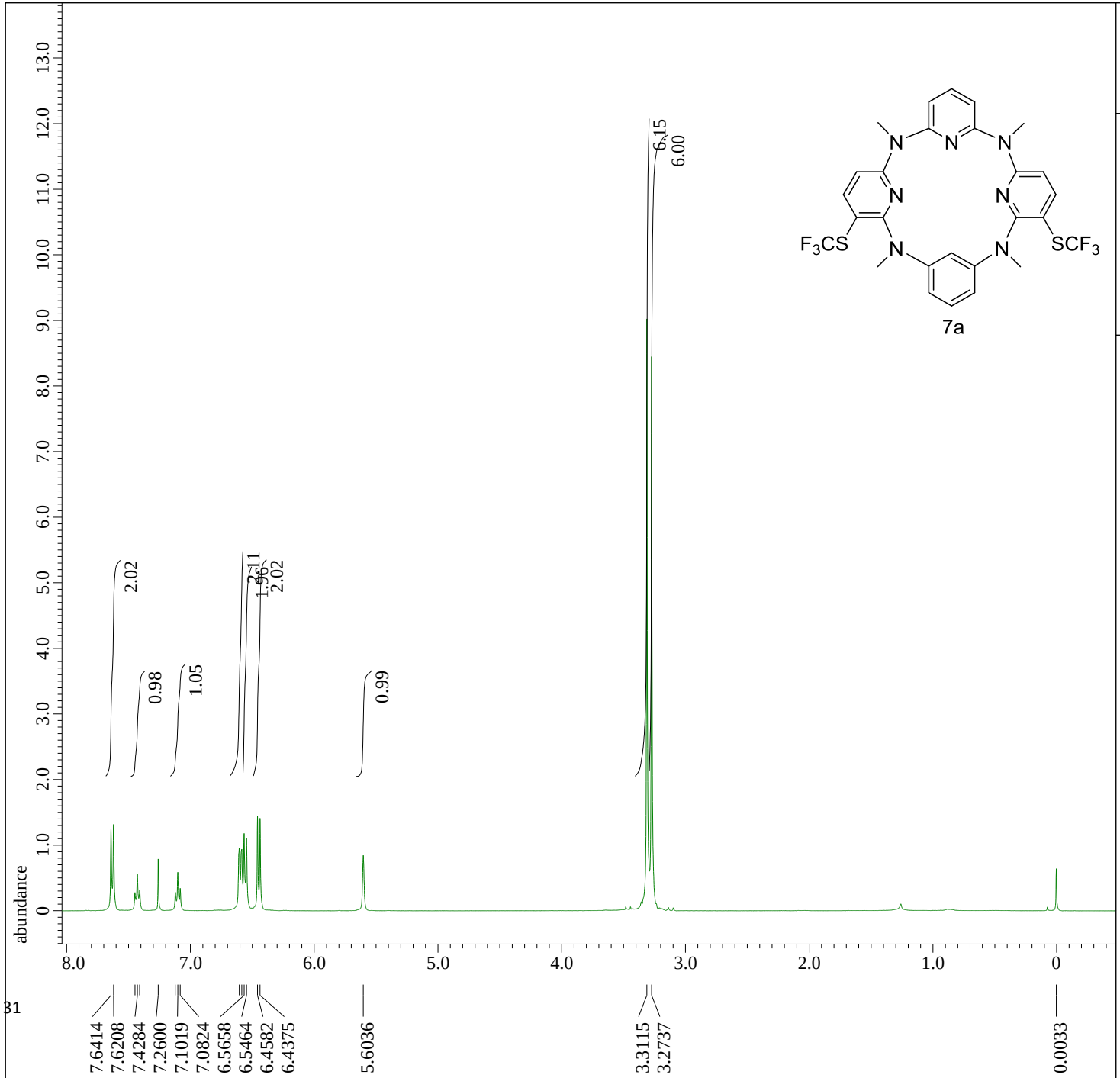
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 151207-wf-5-ome-calix-scf3
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#795942
Solvent           = CHLOROFORM-D
Creation_Time     =  8-DEC-2015 06:46:27
Revision_Time     = 25-FEB-2016 14:59:28
Current_Time      = 25-FEB-2016 14:59:33

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

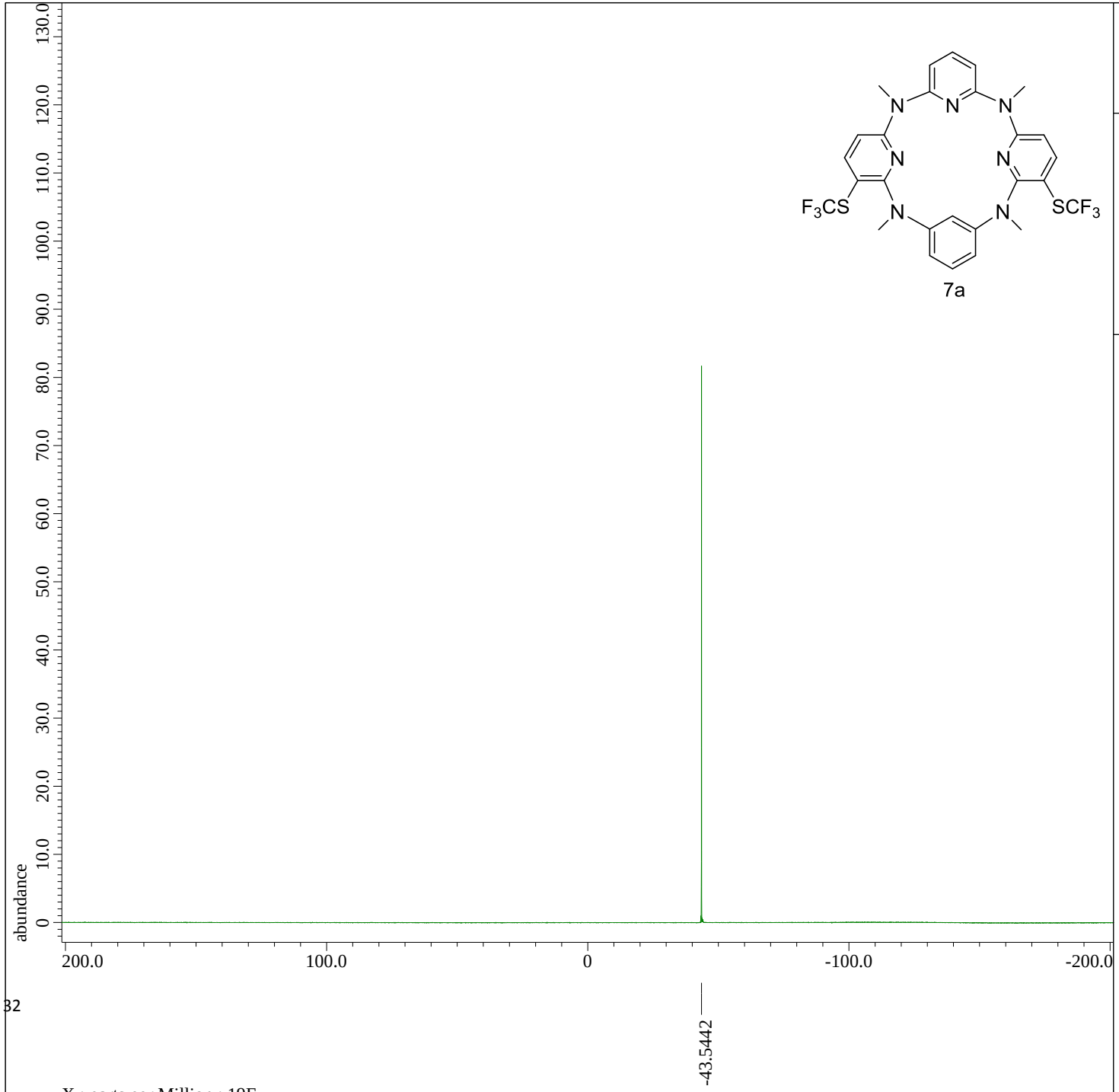
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 10000
Total_Scans       = 10000

Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
Temp_Get          = 26.7[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 3.66666667[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
```



---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename	= 160223-wf-719-2-cdcl3-h-CH
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= S#10893
Solvent	= CHLOROFORM-D
Creation_Time	= 24-FEB-2016 00:20:16
Revision_Time	= 25-FEB-2016 20:34:57
Current_Time	= 25-FEB-2016 21:42:24
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 38
Temp_Get	= 18.8[dC]
X_90_Width	= 12[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 3.4[dB]
X_Pulse	= 6[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE



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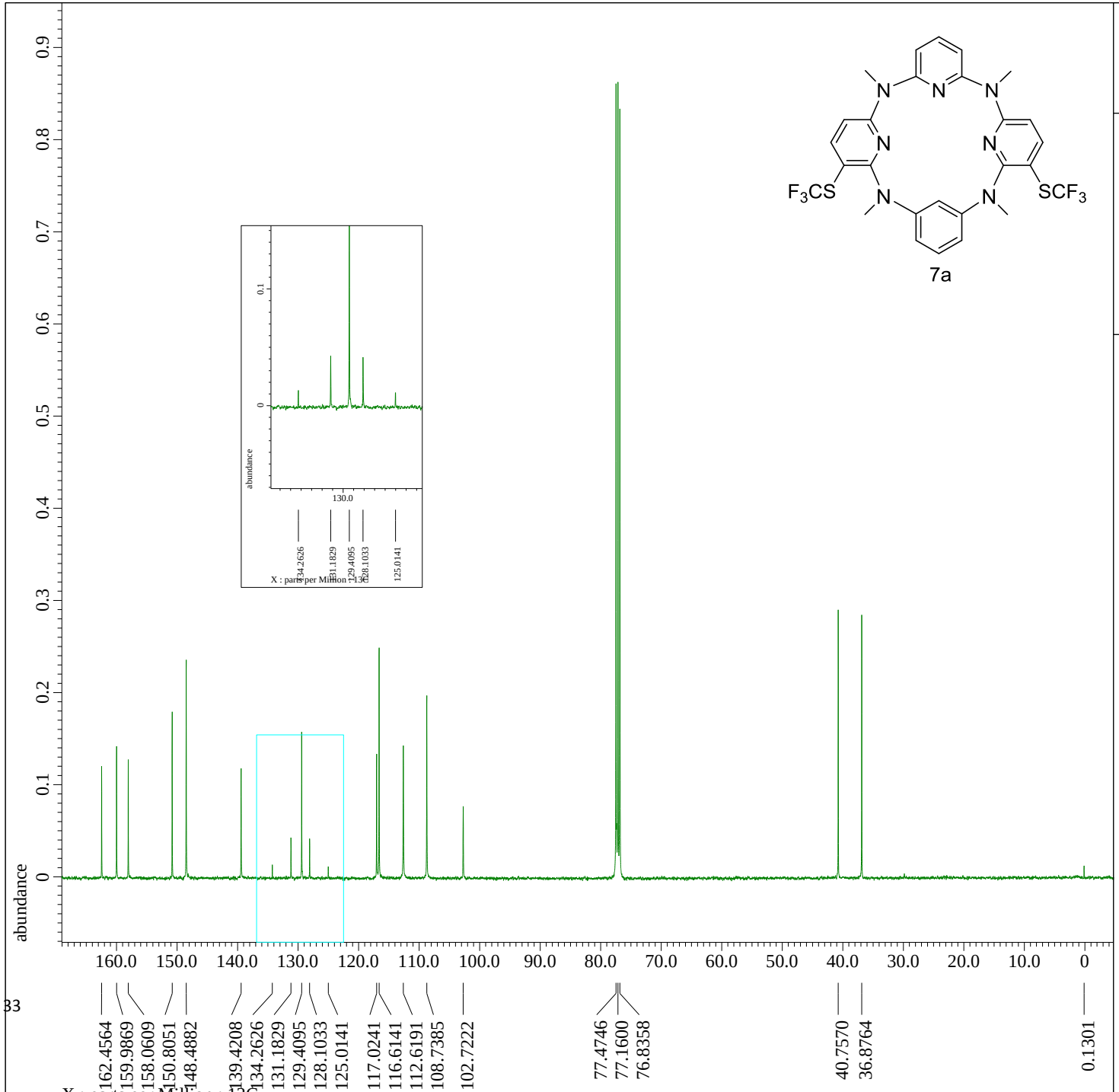
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 160222-wf-719-2-cdcl3-f-5.
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#830898
Solvent           = CHLOROFORM-D
Creation_Time     = 22-FEB-2016 23:07:12
Revision_Time     = 25-FEB-2016 15:54:04
Current_Time      = 25-FEB-2016 16:48:07

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 19F
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

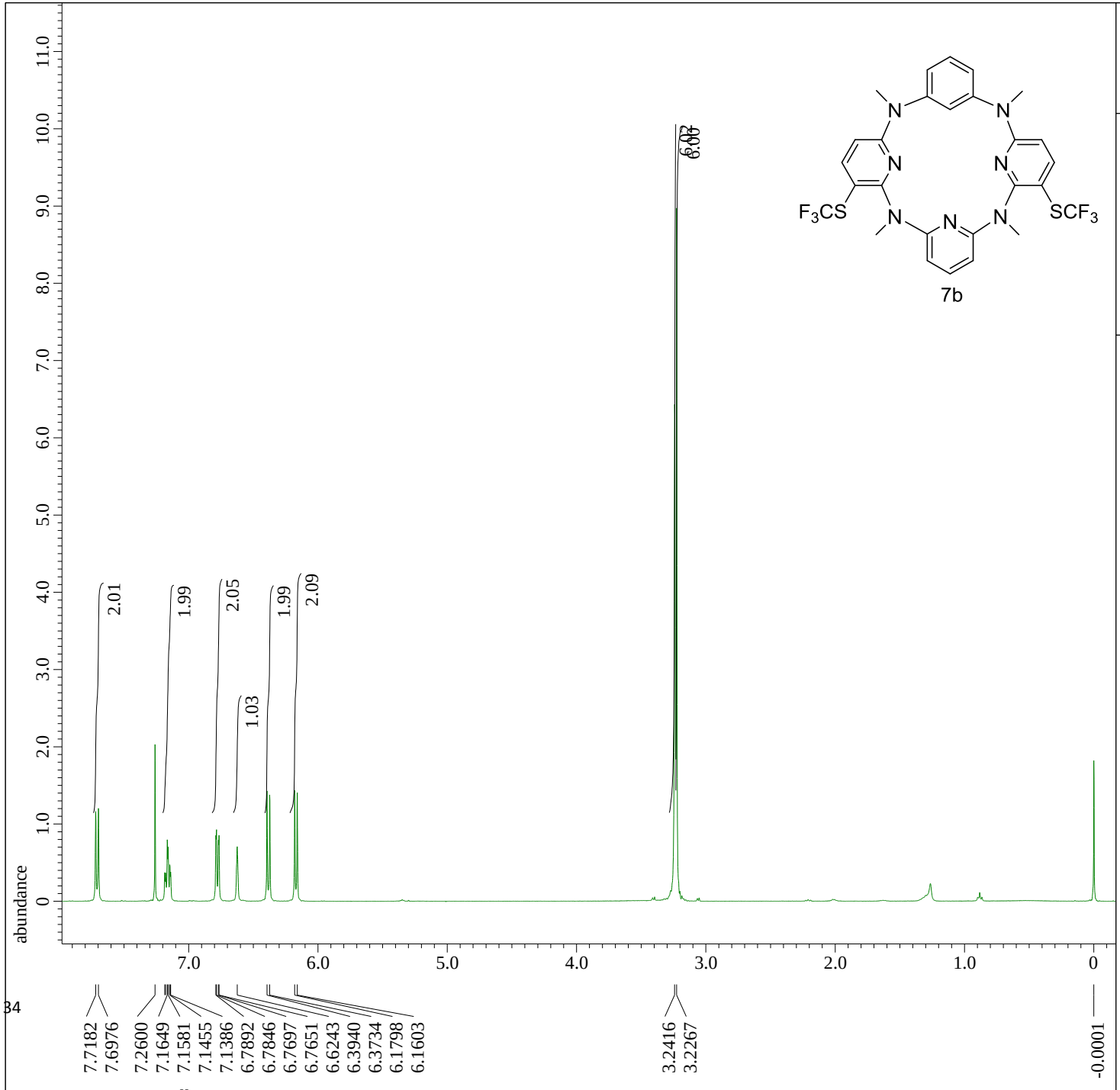
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 86.50752[ms]
X_Domain          = 19F
X_Freq            = 376.17105393[MHz]
X_Offset          = 0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 11.55968868[Hz]
X_Sweep           = 189.39393939[kHz]
Irr_Domain        = 19F
Irr_Freq          = 376.17105393[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 19F
Tri_Freq          = 376.17105393[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 19.2[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 86.50752[ms]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

Filename	= 160223-wf-719-2-cdcl-C-CHE
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= -
Solvent	= CHLOROFORM-D
Creation_Time	= 24-FEB-2016 04:37:22
Revision_Time	= 25-FEB-2016 16:49:04
Current_Time	= 25-FEB-2016 16:49:09
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= TRUE
Scans	= 5000
Total_Scans	= 5000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 56
Temp_Get	= 19.1[dC]
X_90_Width	= 11[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 7.8[dB]
X_Pulse	= 3.66666667[us]
Irr_Atn_Dec	= 23.03[dB]
Irr_Atn_Noe	= 23.03[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE



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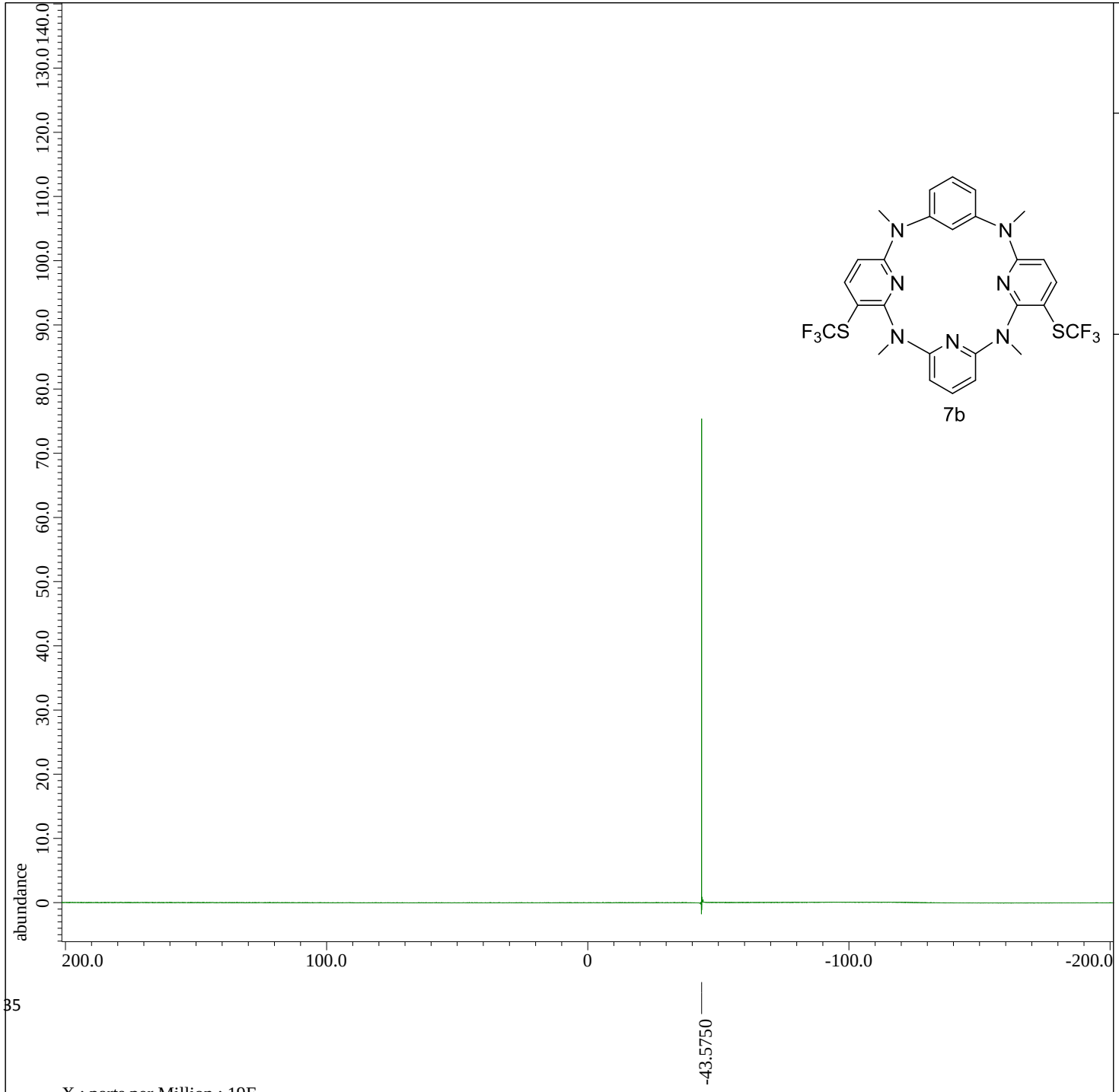
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 160224-wf-719-1-cdcl3-h-5.
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#595194
Solvent           = CHLOROFORM-D
Creation_Time     = 24-FEB-2016 16:33:52
Revision_Time     = 25-FEB-2016 20:30:50
Current_Time      = 25-FEB-2016 21:43:08

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 19.5[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



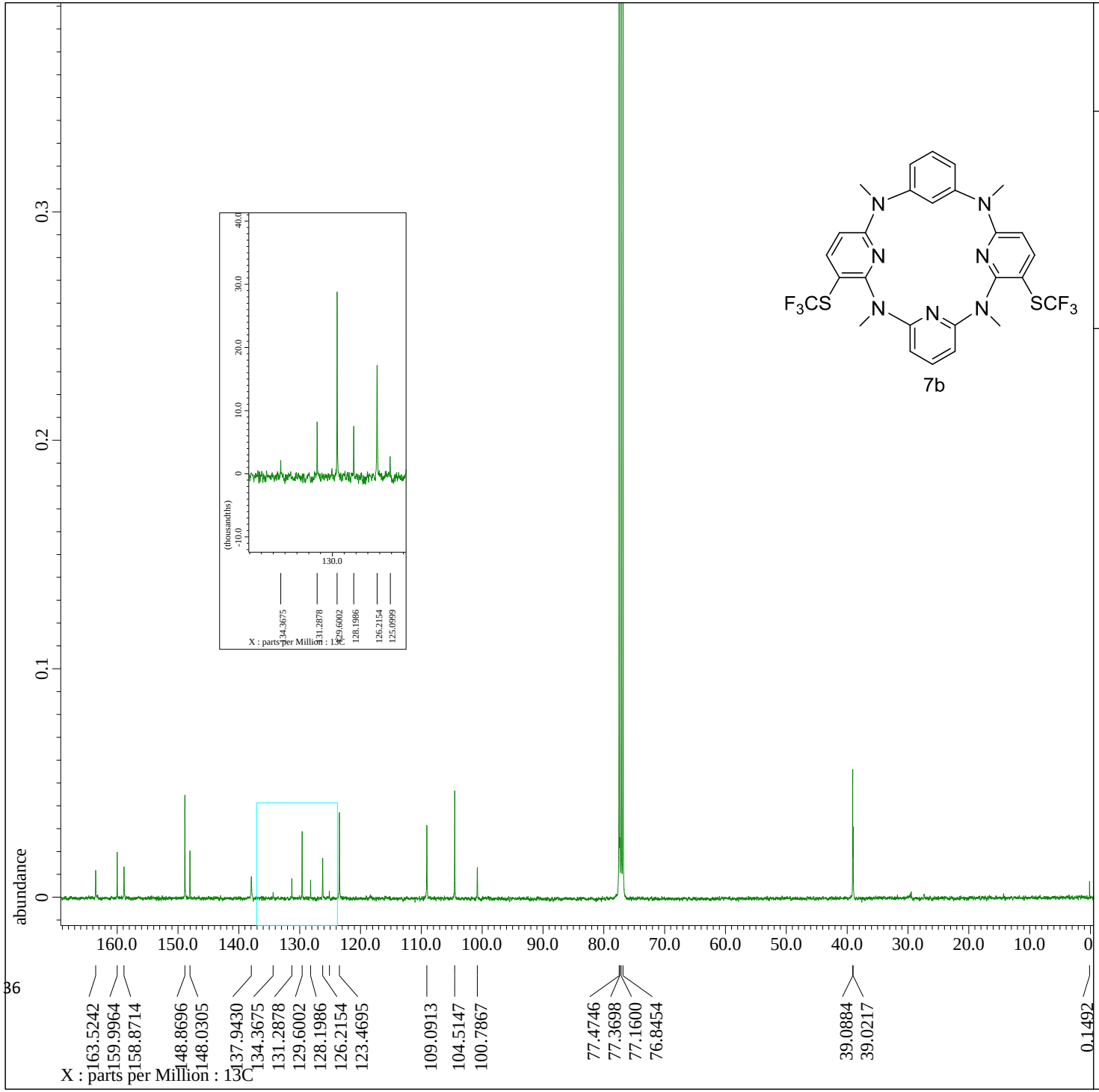

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 160224-wf-719-1-cdcl3-f-5.
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#596491
Solvent           = CHLOROFORM-D
Creation_Time     = 24-FEB-2016 16:36:39
Revision_Time     = 25-FEB-2016 08:15:48
Current_Time      = 25-FEB-2016 16:36:59

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 19F
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 86.50752[ms]
X_Domain          = 19F
X_Freq            = 376.17105393[MHz]
X_Offset          = 0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 11.55968868[Hz]
X_Sweep           = 189.39393939[kHz]
Irr_Domain        = 19F
Irr_Freq          = 376.17105393[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 19F
Tri_Freq          = 376.17105393[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 48
Temp_Get          = 19.6[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 86.50752[ms]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



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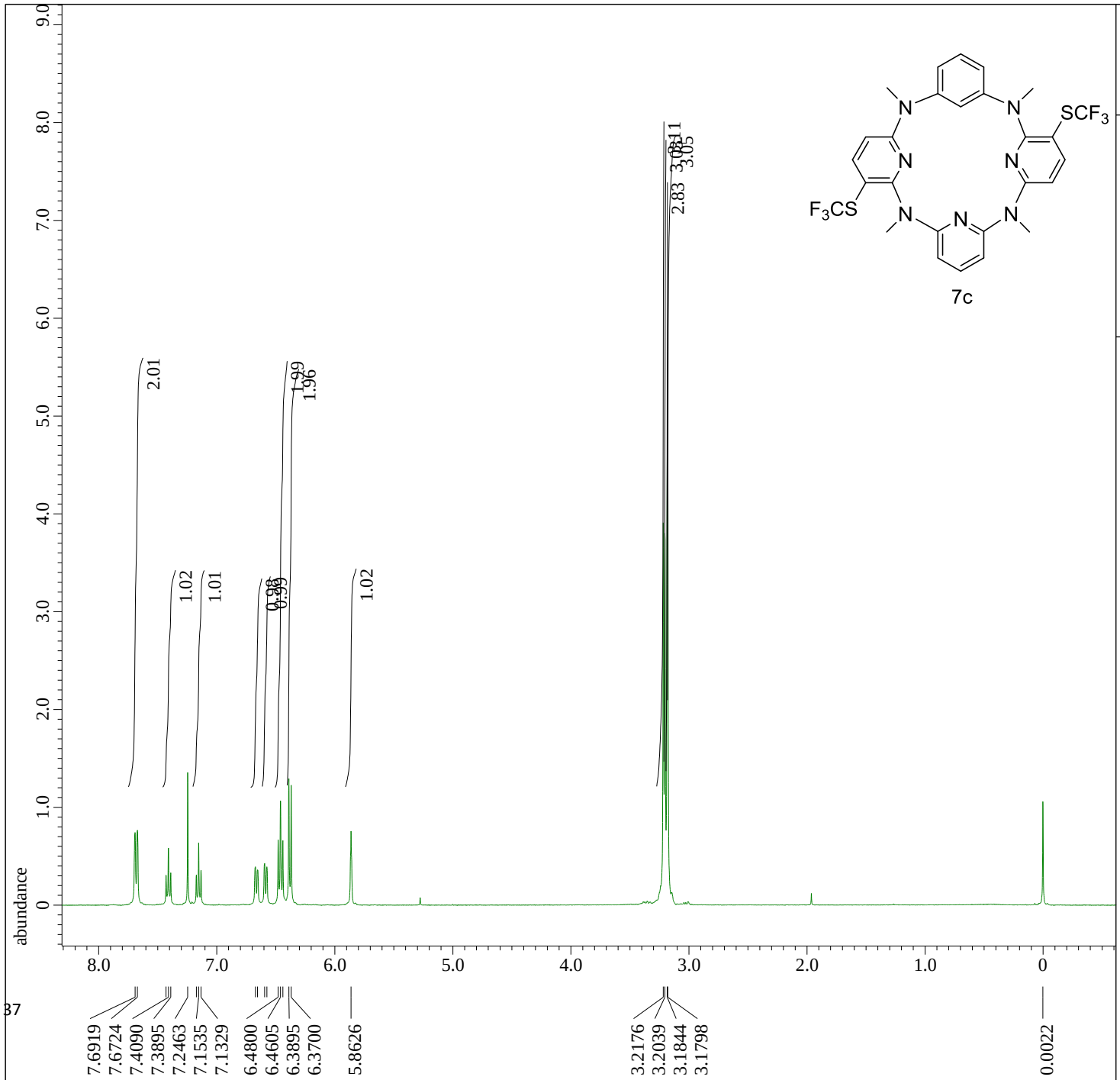
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

Filename	= 160224-wf-719-1-cdcl3-c-4.
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#840978
Solvent	= CHLOROFORM-D
Creation_Time	= 25-FEB-2016 03:37:46
Revision_Time	= 25-FEB-2016 16:38:44
Current_Time	= 25-FEB-2016 16:38:50
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 5000
Total_Scans	= 5000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 19.4[dC]
X_90_Width	= 11[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 7.8[dB]
X_Pulse	= 3.66666667[us]
Irr_Atn_Dec	= 23.03[dB]
Irr_Atn_Noe	= 23.03[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE



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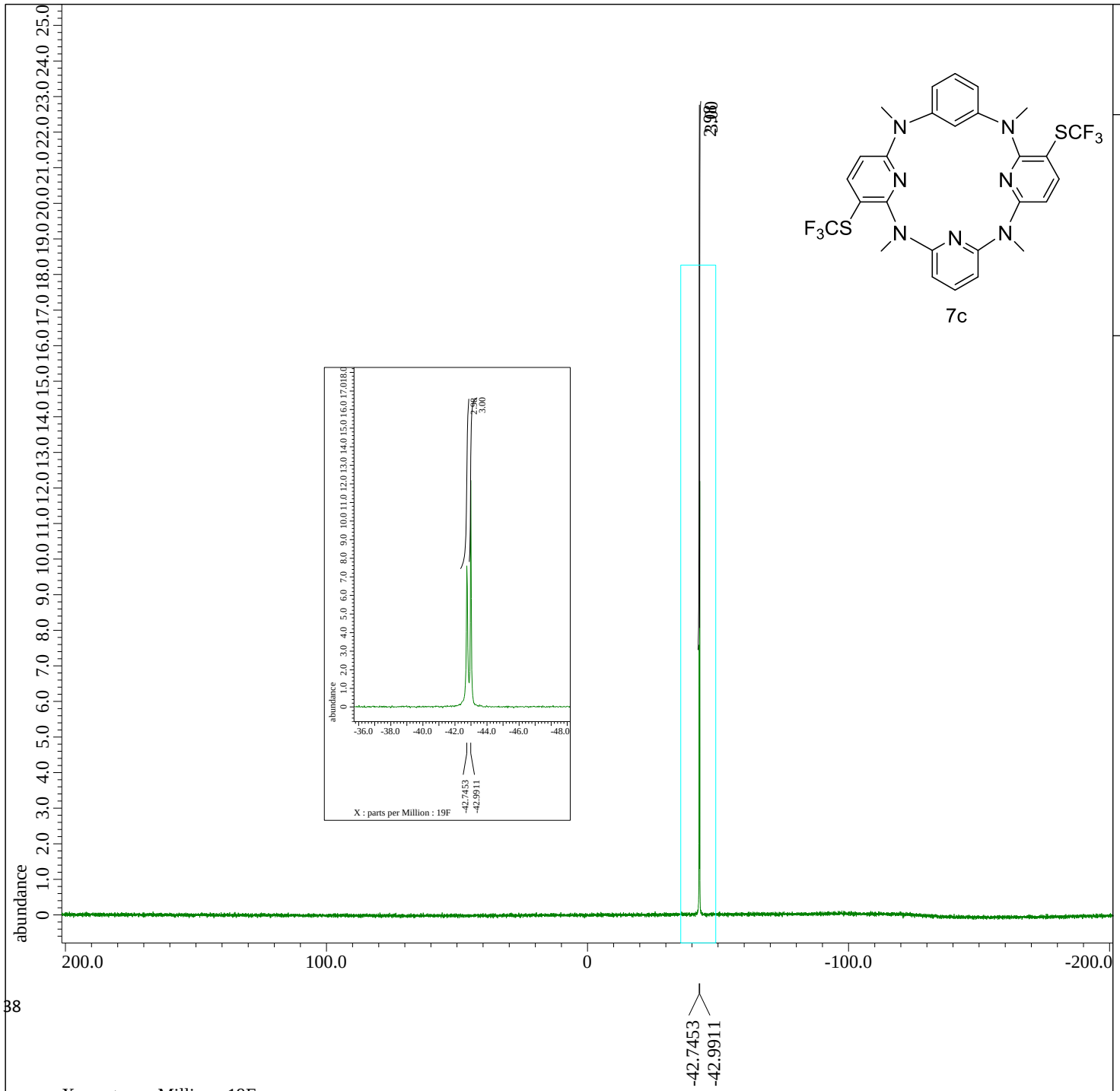
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 160225-wf-719-3-check-55-h
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#822411
Solvent           = CHLOROFORM-D
Creation_Time     = 25-FEB-2016 22:52:19
Revision_Time     = 26-FEB-2016 15:36:12
Current_Time      = 26-FEB-2016 16:33:49

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 55[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



38

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

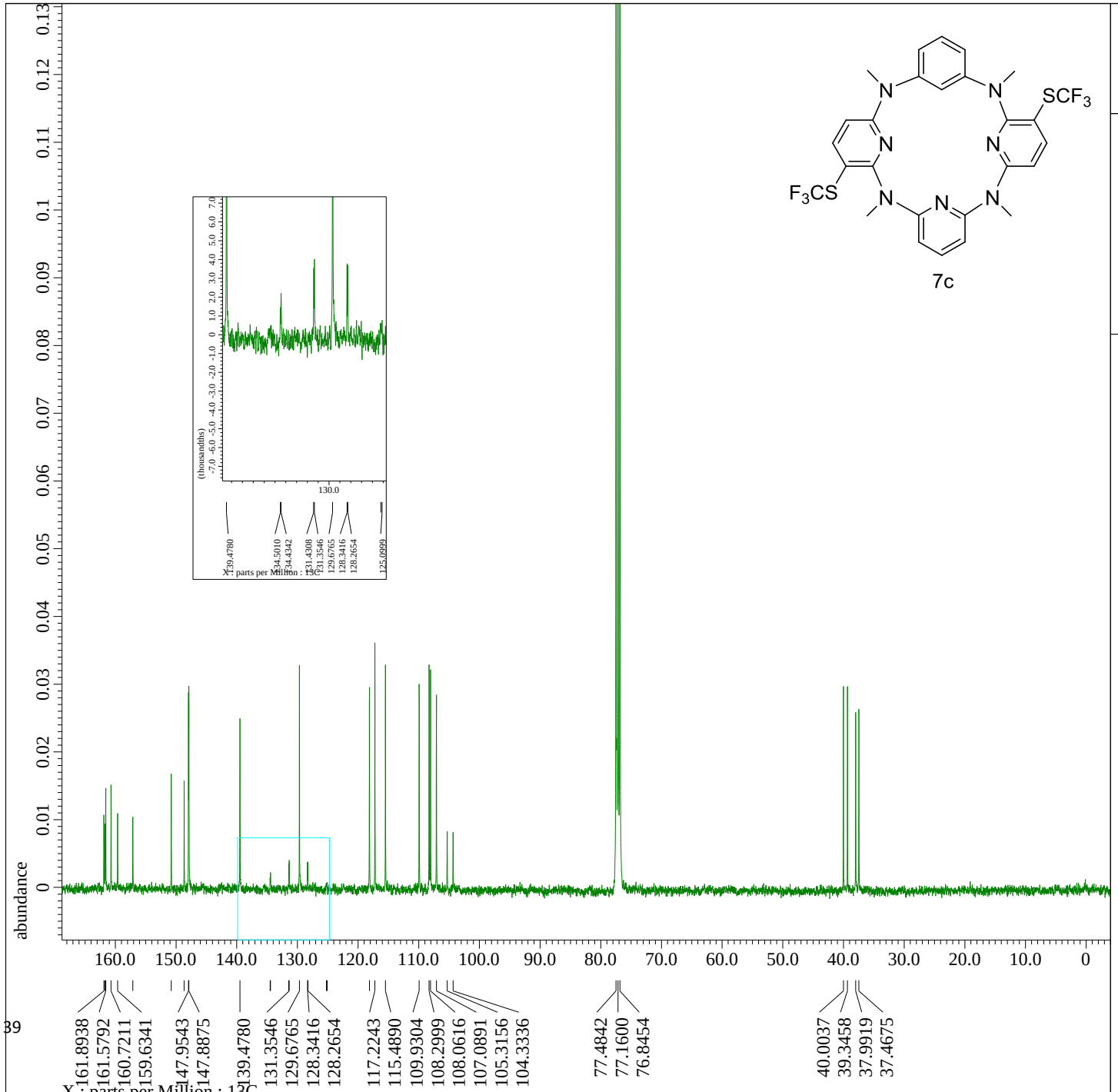
```
Filename          = 160225-wf-719-3-f-6.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#327735
Solvent           = CHLOROFORM-D
Creation_Time     = 25-FEB-2016 09:08:35
Revision_Time     = 26-FEB-2016 16:18:45
Current_Time      = 26-FEB-2016 16:18:49

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 19F
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 86.50752[ms]
X_Domain          = 19F
X_Freq            = 376.17105393[MHz]
X_Offset          = 0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 11.55968868[Hz]
X_Sweep           = 189.39393939[kHz]
Irr_Domain        = 19F
Irr_Freq          = 376.17105393[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 19F
Tri_Freq          = 376.17105393[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 18.7[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 86.50752[ms]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```

X : parts per Million : 19F



7c


```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

Filename	= 160225-wf-719-3-check-55-c
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#319245
Solvent	= CHLOROFORM-D
Creation_Time	= 26-FEB-2016 15:24:56
Revision_Time	= 26-FEB-2016 16:16:50
Current_Time	= 26-FEB-2016 16:16:58
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= TRUE
Scans	= 7696
Total_Scans	= 7696
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 55[dC]
X_90_Width	= 11[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 7.8[dB]
X_Pulse	= 3.66666667[us]
Irr_Atn_Dec	= 23.03[dB]
Irr_Atn_Noe	= 23.03[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE