

Supporting Information

Flow Update for the Carbonylation of 1-Silyl-Substituted Organolithiums under CO Pressure

Takahide Fukuyama*, Takenori Totoki, and Ilhyong Ryu*

*Department of Chemistry, Graduate School of Science, Osaka Prefecture
University, Sakai, Osaka 599-8531, Japan*

ryu@c.s.osakafu-u.ac.jp

Table of Contents

General Information	S2
Typical Procedure for Table 1	S3-4
Typical Procedure for Table 2	S4-5
Spectrum Data	S6-8
References	S8
Spectrum Chart	S9-22

General Information

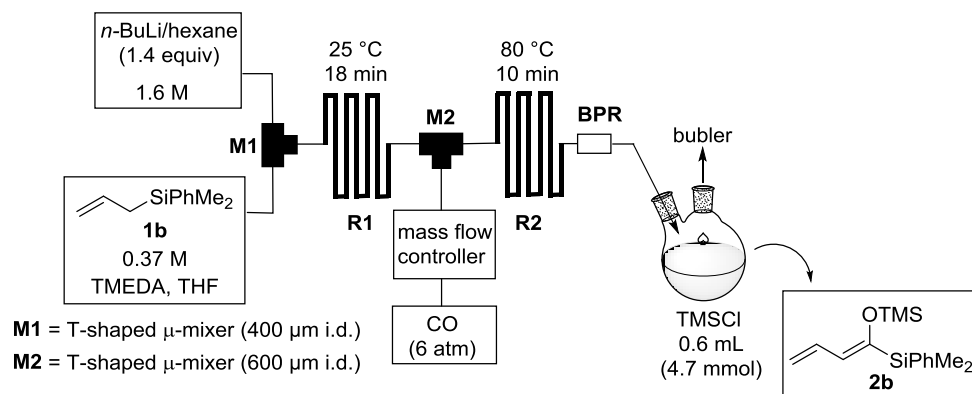
¹H NMR spectra were recorded using JEOL ECP-500 (500 MHz) or JEOL ECS-400 (400 MHz) spectrometers in CDCl₃ and are referenced at 7.26 ppm for CHCl₃. ¹³C NMR spectra were recorded using JEOL ECP-500 (125 MHz) or JEOL ECS-400 (100 MHz) spectrometers in CDCl₃ and are referenced at 77.16 ppm for CHCl₃. Chemical shifts are reported in parts per million (δ). Splitting patterns are indicated as follows: br, broad; s, singlet; d, doublet; t, triplet; m, multiplet. Infrared spectra were obtained on a JASCO FT/IR-4100 spectrometer; absorptions were reported in reciprocal centimeters. Both conventional and high-resolution mass spectra were recorded with a JEOL MS-700 spectrometer. The melting point was measured according to the BÜCHI Melting Point B-540. The products were purified by flash column chromatography on silica gel (Kanto Chem. Co. Silica Gel 60N (spherical, neutral, 40-50 μm)) and, if necessary, were further purified by preparative HPLC (Japan Analytical Industry Co., Ltd., LC-908) with GPC columns using CHCl₃ as an eluent.

THF was distilled from sodium and benzophenone prior to use. *N,N,N',N'*-tetramethylethylenediamine (TMEDA) and trimethylchlorosilane (TMSCl) were distilled from CaH₂. The starting materials **1b**¹ and **1c**² were prepared according to the literature procedures. Other reagents were commercially available and used without further purification.

Stainless steel T-shaped micromixers with inner diameter of 400 and 600 μm were purchased from MiChS Co., Ltd.³ Stainless steel and PTFE microtube reactors with inner diameters of 1000 μm were purchased from GL Sciences Inc. The microreactor and microtube reactors were connected with PEEK fittings (GL Sciences Inc., 1/16"). Back-pressure regulators (40 and 75 psi) were purchased from M & S Instruments Inc. Solutions were introduced to the flow microreactor system using syringe pumps, YSP-101 and YSP-301 (YMC Co., Ltd.), equipped with gastight syringes. These syringes were purchased from SGE Analytical Science Pty. Ltd.

Carbon monoxide was delivered to the micromixer at a constant rate through a mass flow controller, MiChS GFC-1, from a CO gas cylinder. The pressure of the system was controlled by a back-pressure regulator and was monitored by a pressure monitor on the mass flow controller. Residence time at **R2** was estimated according to the equation: $t \text{ (min)} = \text{inner volume of } \mathbf{R2} \text{ (mL)} / [\text{liquid flow rate (mL min}^{-1}) + \text{CO flow rate at 25 } ^\circ\text{C, 1 atm (mL min}^{-1}) / \text{pressure of CO (atm)}]$.

Typical Procedure for Two-Consecutive-Flow Reaction (Table 1, entry 3)



Allyl(phenyl)dimethylsilane (**1b**) (0.704 g, 3.99 mmol) was dissolved in THF (9 mL) and TMEDA (1.8 mL, 12 mmol) and then placed in a syringe, which was then attached to a syringe pump. A THF/TMEDA solution of **1b** (flow rate: 0.2 mL min⁻¹) and a hexane solution of *n*-BuLi (flow rate: 0.065 mL min⁻¹) were mixed in **M1** (400 μ m i.d.) at 25 °C using syringe pumps. The resultant reaction mixture was fed into **R1** (channel diameter = 1000 μ m, length = 6 m) and was then mixed with pressurized carbon monoxide (6 atm, 7.46 mL min⁻¹ (in terms of 1 atm), 4.5 equiv) in **M2** (600 μ m i.d.), which was supplied through a mass flow controller. The reaction mixture was passed through **R2** (channel diameter = 1000 μ m, length = 20 m), which was connected to a back-pressure regulator (75 psi), and was collected from the outlet. The reaction mixture eluted during the first 5 min was discarded and the following portion was collected for a 5 min period in a glass flask that contained TMSCl (0.6 mL, 4.7 mmol). The collected reaction mixture was stirred at 25 °C for 1 h and then aqueous workup with ether and a NaHCO₃ aqueous solution was conducted. The ethereal solution was dried over MgSO₄. The filtration and evaporation of the solvents gave a crude reaction mixture, which was purified by flash column chromatography on SiO₂ (hexane) to give **2b** (93.3 mg, 91%).

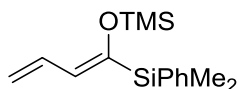
Table S1. Details of Continuous Microflow Reaction for Table 1 in the Manuscript

entry	length (m)		CO (atm)	flow rate (mL min ⁻¹)		
	R1	R2		1b	<i>n</i> -BuLi	CO ^a
1	6	10	4	0.15	0.048	5.55 (1.39)
2	10.5	20	4	0.2	0.065	7.46 (1.87)
3	6	20	6	0.2	0.065	7.46 (1.24)

Inner diameters of **R1** and **R2** are 1000 μ m.

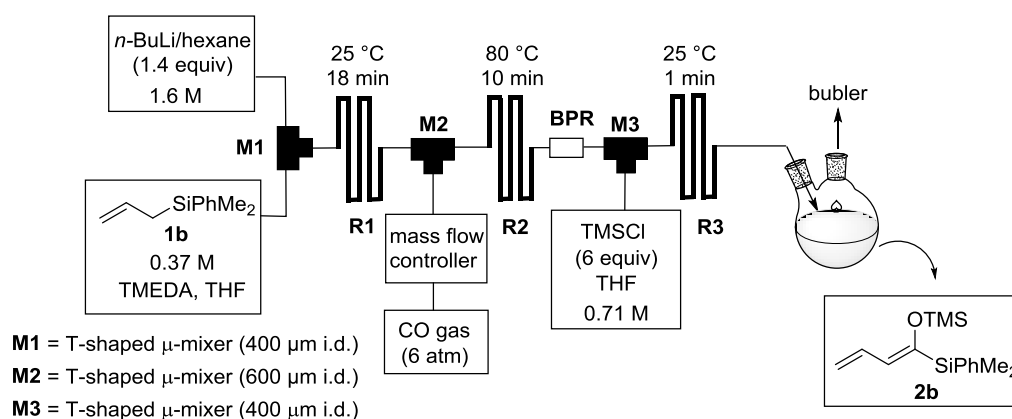
^aFlow rate at standard condition of 25 °C and 1 atm. Calculated flow rate at actual pressure at 25 °C is shown in parentheses.

(E)-((1-(Dimethylphenylsilyl)-1,3-butadienyl)oxy)trimethylsilane (2b)



colorless oil; $R_f = 0.1$ (hexane); ^1H NMR (400 MHz, CDCl_3) δ 0.05 (s, 9H), 0.41 (s, 6H), 4.98 (dd, $J = 10.4, 1.2$ Hz, 1H), 5.13 (dd, $J = 17.6, 2.4$ Hz, 1H), 5.79 (d, $J = 10.4$ Hz, 1H), 6.66 (ddd, $J = 17.2, 10.6, 10.4$ Hz, 1H), 7.30-7.40 (m, 3H), 7.50-7.58 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ -3.13, 0.97, 115.2, 126.4, 127.9, 129.4, 130.6, 134.4, 137.0, 158.6. These spectral data are consistent with those previously reported in the literature.⁴

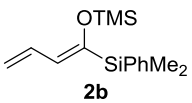
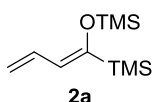
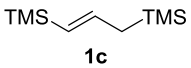
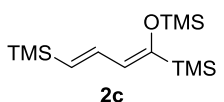
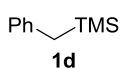
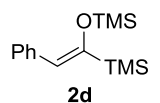
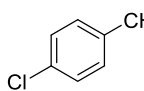
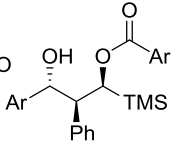
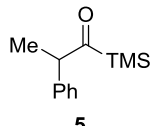
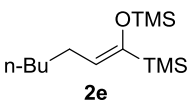
Typical Procedure for Three-Consecutive-Flow Reactions (Table 2, entry 1)



Allyl(phenyl)dimethylsilane (**1b**) (0.697 g, 3.95 mmol) was dissolved in THF (9 mL) and TMEDA (1.8 mL, 12 mmol) and then placed in a syringe, which was then attached to a syringe pump. A THF/TMEDA solution of **1b** (flow rate: 0.2 mL min^{-1}) and a hexane solution of *n*-BuLi (flow rate: 0.065 mL min^{-1}) were mixed in a **M1** (400 μm i.d.) at 25 °C using syringe pumps. The resultant reaction mixture was fed into **R1** (channel diameter = 1000 μm , length = 6 m) and was then mixed with pressurized carbon monoxide (6 atm, 7.46 mL min^{-1} (in terms of 1 atm), 4.5 equiv) in **M2** (600 μm i.d.), which was supplied through a mass flow controller. The reaction mixture was passed through **R2** (channel diameter = 1000 μm , length = 20 m), which was connected to a back-pressure regulator (75 psi), and was quenched by mixing with a THF solution of TMSCl (flow rate: 0.625 mL min^{-1}) in **M3** (400 μm i.d.), and then fed into **R3** (channel diameter = 1000 μm , length = 11 m). A mixture of the product was collected from the outlet. The reaction mixture eluted during the first 5 min was discarded and the following portion was collected for a 5 min period. After collection, an aqueous

workup with ether and a NaHCO₃ aqueous solution was conducted. The ethereal solution was dried over MgSO₄. The filtration and evaporation of the solvents, gave a crude reaction mixture, which was purified by flash column chromatography on SiO₂ (hexane) to give **2b** (94.2 mg, 93%).

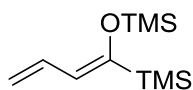
Table S2. Details of Continuous Microflow Reaction for Table 2 and Scheme 4 in the Manuscript

entry	1	length (m)			electrophiles	products
		R1	R2	R3		
1	1b	6	20	11	TMSCl	 2b
2	1a	6	10	11	TMSCl	 2a
3	 1c	6	20	11	TMSCl	 2c
4	 1d	10.5	10	11	TMSCl	 2d
5	1d	10.5	10	11		 4 Ar = <i>p</i> -Cl-C ₆ H ₄
6	1d	10.5	10	11	Mel	 5
Scheme 4	1e	10.5	10	11	TMSCl	 2e

Flow rate: A THF/TMEDA solution of **1** (0.2 mL min⁻¹), A hexane solution of *n*-BuLi (0.065 mL min⁻¹), CO (7.46 sccm, where sccm denotes mL min⁻¹ at the standard condition of 25 °C and 1 atm.), electrophiles (0.625 mL min⁻¹).
Inner diameters of **R1**, **R2**, and **R3** are 1000 μm.

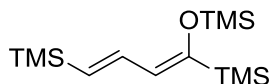
Spectrum Data

(*E*)-Trimethyl((1-(trimethylsilyl)-1,3-butadien-1-yl)oxy)silane (2a)



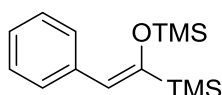
colorless oil; $R_f = 0.25$ (hexane); ^1H NMR (400 MHz, CDCl_3) δ 0.13 (s, 9H), 0.21 (s, 9H), 4.96 (dd, $J = 10.8, 1.6$ Hz, 1H), 5.13 (dd, $J = 16.8, 1.6$ Hz, 1H), 5.75 (d, $J = 10.4$ Hz, 1H), 6.66 (ddd, $J = 16.8, 10.6, 10.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ -1.73, 0.99, 114.8, 125.0, 130.6, 160.4. These spectral data are consistent with those previously reported in the literature.⁴

((1*E*,3*E*)-1-((Trimethylsilyl)oxy)-1,3-butadien-1,4-diyl)bis(trimethylsilane) (2c)



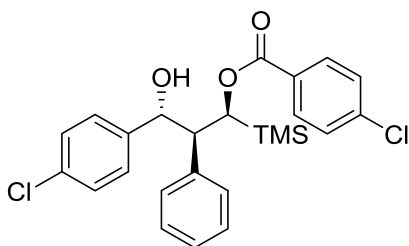
colorless oil; $R_f = 0.25$ (hexane); ^1H NMR (400 MHz, CDCl_3) δ 0.07 (s, 9H), 0.12 (s, 9H), 0.21 (s, 9H), 5.76 (d, $J = 10.0$ Hz, 1H), 5.77 (d, $J = 18.4$ Hz, 1H), 6.88 (dd, $J = 18.4, 10.0$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ -1.75, -0.99, 1.16, 127.1, 131.2, 137.6, 160.5; IR (neat): 1604, 1554 cm^{-1} ; EIMS m/z (relative intensity) 286 (M^+ , 29), 183 (14), 148 (11), 147 (74), 73 (100). HRMS (EI) m/z calcd for $\text{C}_{13}\text{H}_{30}\text{OSi}_3$ (M^+): 286.1604, found: 286.1605.

(*E*)-Trimethyl((2-phenyl-1-(trimethylsilyl)ethenyl)oxy)silane (2d)



colorless oil; $R_f = 0.125$ (hexane); ^1H NMR (400 MHz, CDCl_3) δ 0.06 (s, 9H), 0.20 (s, 9H), 5.93 (s, 1H), 7.16 (t, $J = \text{Hz}$, 1H), 7.28 (t, $J = 7.2\text{Hz}$, 2H), 7.50 (d, $J = 6.8$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ -1.28, 1.16, 123.4, 126.4, 128.1, 129.1, 136.5, 159.9. These spectral data are consistent with those previously reported in the literature.⁵

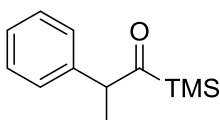
(1*S*,2*S*,3*R*)-rel-1-(4-Chlorobenzoate)-3-(4-Chlorophenyl)-2-phenyl-1-(trimethylsilyl)-1,3-propanediol (4)



White solid; m.p. 105-108 °C; R_f = 0.1 (hexane : EtOAc = 50 : 1); ^1H NMR (400 MHz, CDCl_3) δ -0.17 (s, 9H), 2.95 (dd, J = 10.0, 2.4 Hz, 1H), 4.56 (dd, J = 10.0, 3.2 Hz, 1H), 4.72 (d, J = 3.2 Hz, 1H), 5.74 (d, J = 2.4 Hz, 1H), 6.92-6.94 (m, 4H), 7.04 (d, J = 8.8 Hz, 2H), 7.13-7.14 (m, 3H), 7.54 (dd, J = 6.8, 2.4 Hz, 2H), 8.11 (dd, J = 6.8, 2.4 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ -3.11, 57.05, 69.81, 73.65, 127.2, 128.0, 128.1, 128.2, 128.4, 129.4, 129.9, 131.4, 132.7, 138.6, 140.4, 140.5, 168.3; IR (neat): 3477, 1695 cm^{-1} ; EIMS m/z (relative intensity) 457 ($\text{M}^+ - \text{CH}_3$, 1), 177 (21), 176 (96), 162 (14), 161 (83), 145 (15), 139 (100), 113 (11), 111 (21), 77 (24), 73 (35). HRMS (EI) m/z calcd for $\text{C}_{24}\text{H}_{23}\text{Cl}_2\text{O}_3\text{Si}$ ($\text{M}^+ - \text{CH}_3$): 457.0794, found: 457.0804.

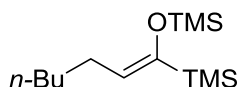
The stereoconfiguration of this compound was determined by referring to the ^1H NMR spectrum of the related compound in literature.⁶

2-Phenyl-1-(trimethylsilyl)-1-propanone (5)



yellow oil; R_f = 0.125 (hexane : EtOAc = 50 : 1); ^1H NMR (400 MHz, CDCl_3) δ -0.02 (s, 9H), 1.28 (d, J = 7.2 Hz, 3H), 4.01 (q, J = 7.2 Hz, 1H), 7.12-7.13 (m, 2H), 7.24-7.26 (m, 1H), 7.30-7.34 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ -2.51, 16.3, 57.5, 127.0, 128.7, 139.1, 244.5. These spectral data are consistent with those previously reported in the literature.⁷

Trimethyl(((1*E*)-1-(trimethylsilyl)-1-hepten-1-yl)oxy)silane (2e)



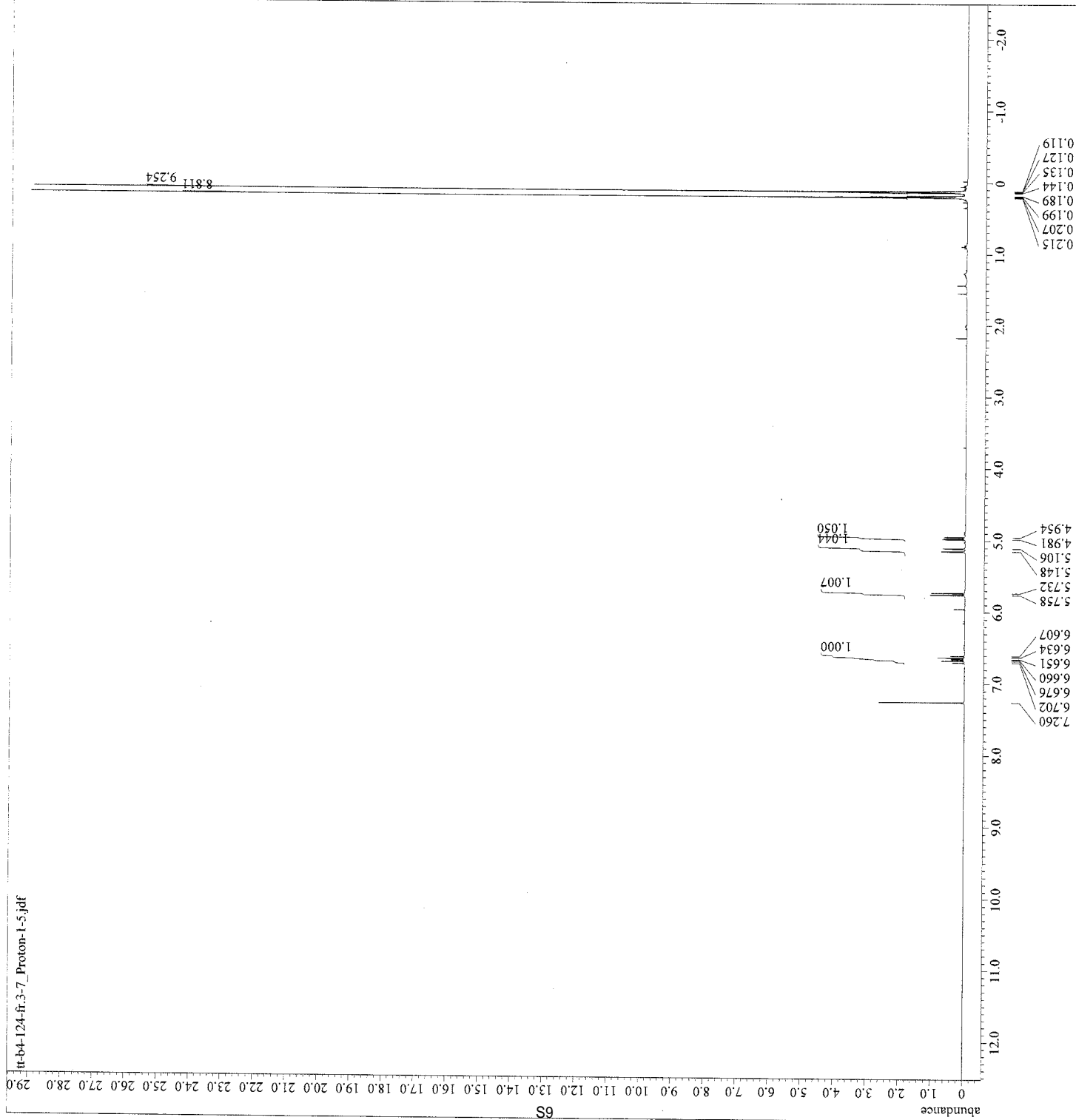
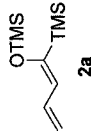
colorless oil; ^1H NMR (500 MHz, CDCl_3) δ 0.09 (s, 9H), 0.18 (s, 9H), 0.89 (t, $J = 6.4$ Hz, 3H), 1.30-1.33 (m, 6H), 2.05-2.06 (m, 2H), 5.01 (t, $J = 6.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ -1.51, 1.07, 14.2, 22.7, 25.9, 29.4, 32.0, 125.9, 156.5; IR (neat): 1616 cm^{-1} ; EIMS m/z (relative intensity) 258 (M^+ , 11), 201 (16), 185 (61), 184 (11), 148 (11), 147 (71), 133 (13), 73(100). HRMS (EI) m/z calcd for $\text{C}_{13}\text{H}_{30}\text{OSi}_2$ (M^+): 258.1835, found: 258.1826.

References

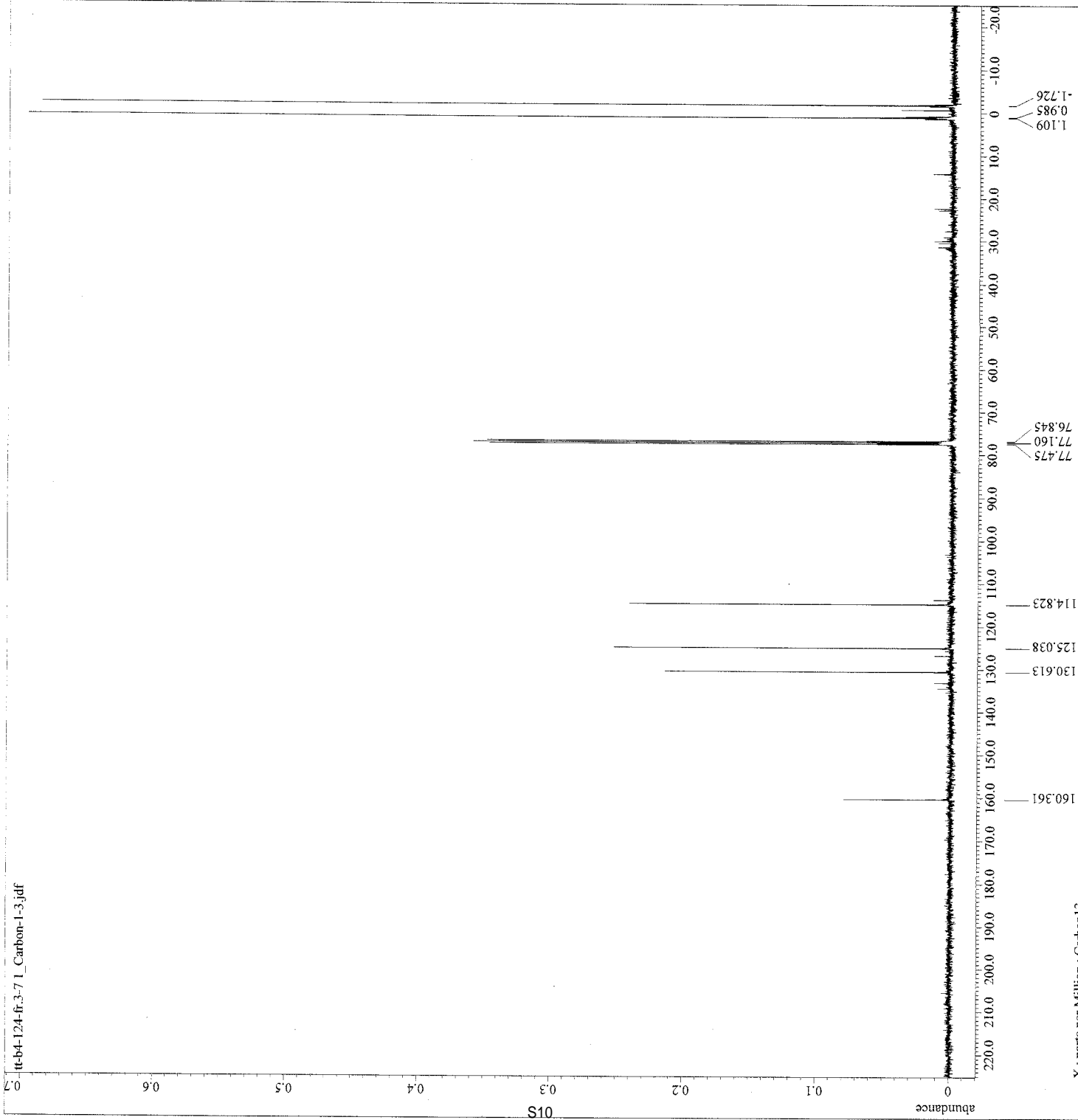
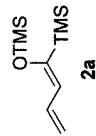
- (1) Soderquist, J. A.; Hassner, A. *J. Org. Chem.* **1983**, *48*, 1801.
- (2) Ihara, E.; Koyama, K.; Yasuda, H.; Kanehisa, N.; Kai, Y. *J. Organomet. Chem.* **1999**, *574*, 40.
- (3) http://www.michs.jp/index_en.html
- (4) Ryu, I.; Yamamoto, H.; Sonoda, N.; Murai, S. *Organometallics* **1996**, *15*, 5459.
- (5) Murai, S.; Ryu, I.; Iriguchi, J.; Sonoda, N. *J. Am. Chem. Soc.* **1984**, *106*, 2440.
- (6) Honda, M.; Iwamoto, R.; Nogami, Y.; Segi, M. *Chem. Lett.* **2005**, *34*, 466.
- (7) Nakada, M.; Urano, Y.; Kobayashi, S.; Ohno, M. *J. Am. Chem. Soc.* **1988**, *110*, 4826.



Filename = C:\Users\delia\Documents\J
 Author = delia
 Experiment = proton_jyp
 Sample Id = tt-b4-124-ft.3-7
 Solvent = CHLOROFORM-D
 Creation Time = 24-JAN-2013 20:20:11
 Revision Time = 12-MAR-2013 16:46:18
 Current Time = 12-MAR-2013 16:52:14
 Comment = single pulse
 Data Format = ID COMEX
 Data Size = 13107
 Data Title = Proton
 Data Units = [ppm]
 Dimensions =
 Status = NMR-EGS400
 Spectrometer = DELTA2_NMR
 Field Strength = 9.4249681[T] (400[MHz])
 X_Acq_Duration = 2.1757952[s]
 X_Domain = 1H
 X_Freq = 401.28219856[MHz]
 X_Offset = 16384
 X_Points = 1
 X_Prescans = 1
 X_Resolution = 0.45960208[Hz]
 X_Sweep = 7.53012048[kHz]
 X_Sweep_Clip = 6.02409639[kHz]
 Irr_Domain = Proton
 Irr_Freq = 401.28219856[MHz]
 Irr_Offset = 16384
 Irr_Points = 1
 Tri_Domain = Proton
 Tri_Freq = 401.28219856[MHz]
 Tri_Offset = 5[ppm]
 Tri_Clipped = FALSE
 Scans = 8
 Total_Scans = 8
 Relaxation_Delay = 5[s]
 Recv_Gain = 44
 Temp_Get = 20.1[degC]
 X_90_Width = 9.25[us]
 X_Acq_Time = 2.1757952[s]
 X_Angle = 45[deg]
 X_Pulse = 4.8[us]
 X_Pulse2 = 4.825[us]
 Irr_Mode = Off
 Tri_Mode = Off
 Dante_Preset = FALSE
 Initial_Wait = 1[s]
 Repetition_Time = 7.1757952[s]

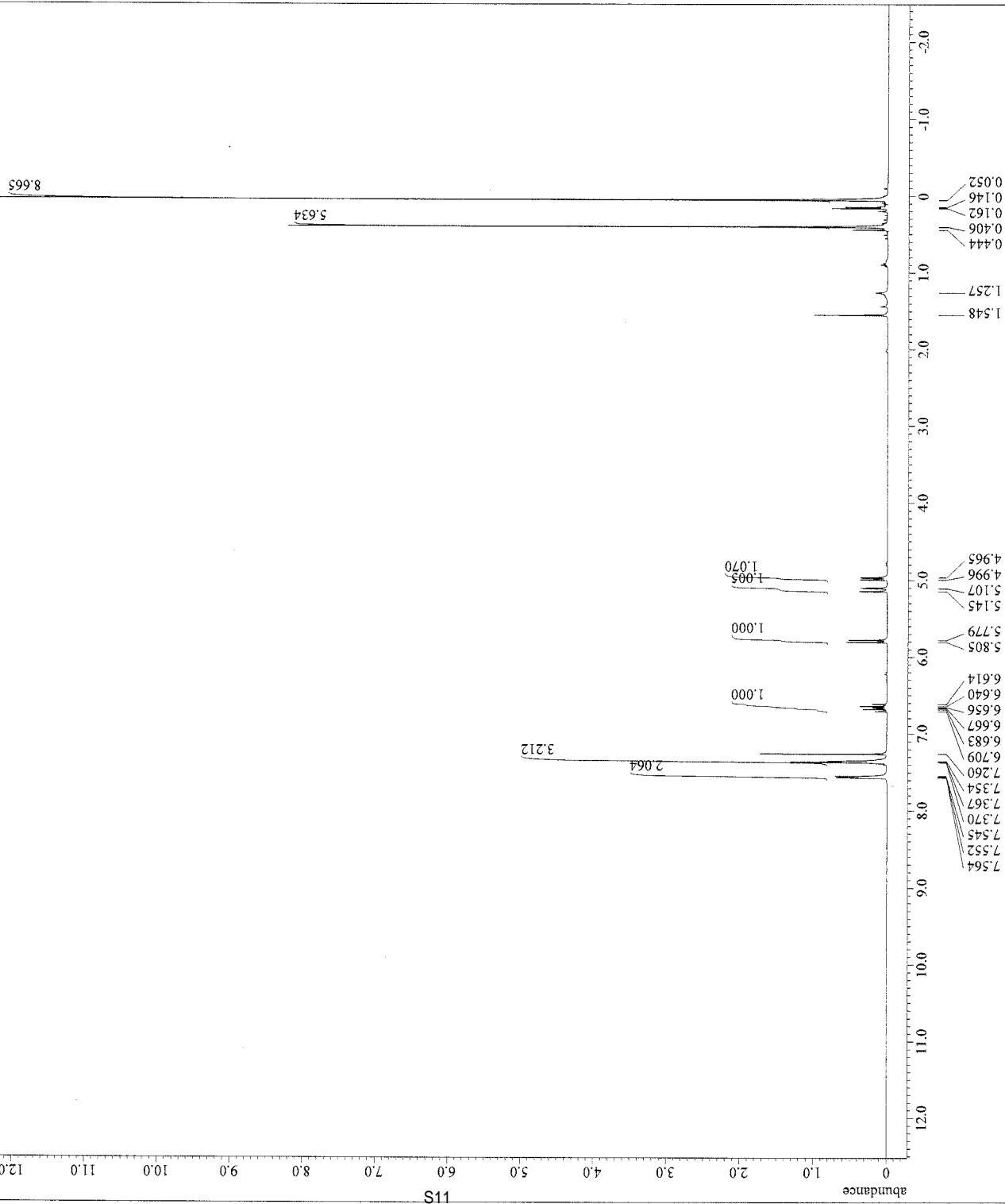
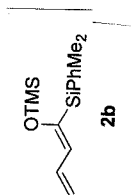


Filename = C:\Users\delia\Documents\J
 Author = delta
 Experiment = carbon_jsp
 Sample Id = tt-b4-124-fr.3-71
 Solvent = CHLOROFORM-D
 Creation Time = 24-JAN-2013 20:54:49
 Revision Time = 12-MAR-2013 16:51:26
 Current Time = 12-MAR-2013 16:51:59
 Comment = single pulse decoupled gat
 Data Format = ID COMPLEX
 Data Size = 26214
 Data Title = Carbon13
 Data Units = ppm
 Dimensions = 2
 Size = 256-KS400
 Spectrometer = DELTA2_NMR
 Field Strength = 9.42499681[T] (400[MHz])
 X_Acq_Duration = 1.03809024[s]
 X_Domain = 13C
 X_Freq = 100.90247863[MHz]
 X_Offset = 32766
 X_Points = 4
 X_Prescans = 4
 X_Resolution = 0.96330739[Hz]
 X_Sweep = 31.56565657[kHz]
 X_Sweep_Clip = 25.25252525[kHz]
 Irr_Domain = proton
 Irr_Freq = 401.28219856[MHz]
 Irr_Offset = 51200
 Clipped = FALSE
 Scans = 256
 Total Scans = 256
 Relaxation Delay = 2[s]
 Recycle Gain = 20
 T1_Rho = 2.41[sec]
 T1_Rho_2 = 8.75[us]
 X_90_Width = 1.03809024[s]
 X_Acq_Time = 30[deg]
 X_Angle = 5.2[deg]
 X_Pulse = 2.91666667[us]
 Irr_Atn_Dec = 22.6911[deg]
 Irr_Atn_Dec_2 = 22.6911[deg]
 Irr_Noise = 0.115[ms]
 Irr_Width = 0.115[ms]
 Decoupling = TRUE
 Initial Wait = 1[s]
 Noe = TRUE
 Noe_Time = 2[s]
 Acquisition Time = 3.03809024[s]





FileNames	= C:\Users\deltaelta\Documents\deltaelta
Author	= proton_jxp
Experiment	= tt-b4-106-fr.3-9
Sample_Id	= CHLOROFORM-D
Solvent	= CHLOROFORM-D
Creation_Time	= 10-DEC-2012 21:30:03
Acquisition_Time	= 12-MAR-2013 16:52:26
Current_Time	= 12-MAR-2013 16:52:26
Comment	= single pulse
Data_Format	= 1D COMEXX
Dim_Size	= 13107
Dim_Title	= Proton
Dimensions	= X (ppm)
SI_Site	= X
Spectrometer	= JNM-ECX400
Field Strength	= DELTA2_NMR
Acq_Duration	= 9.42495681 [T] (400 [MHz])
Chemical_Shift	= 1.757952 [s]
Freq	= 401.28219856 [MHz]
X_Offset	= 5 [ppm]
X_Points	= 10384
X_Prescans	= 1
X_Resolution	= 0.45602008 [Hz]
Acq_Sweep	= 6.02404008 [Hz]
Clipped	= 0.02404008 [Hz]
Irq_Freq	= Proton
Irq_Domain	= 401.28219856 [MHz]
Irq_Offset	= 5 [ppm]
Trf_Domain	= Proton
Trf_Freq	= 401.28219856 [MHz]
Trf_Offset	= 5 [ppm]
Clipped	= PULSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5 [s]
Recov_Gain	= 46
X_90_Time	= 5 [sG]
X_Acq_Time	= 9.25 [us]
X_Angle	= 2.11757952 [s]
X_Pulse	= 45 [deg]
X_Atn	= 0.8 [dB]
X_Pulse	= 4.625 [us]
Irq_Mode	= OFF
Dance_Preset	= PULSE
Initial Wait	= 1 [s]
Repetition Time	= 7.1757952 [s]



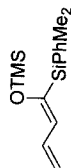
X : parts per Million : Proton



--- PROCESSING PARAMETERS ---
acquire = 1
fft = 1
ppm
machinephase

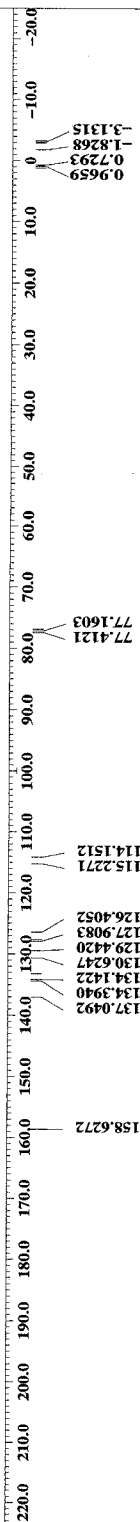
7.7-134-20-1

--- ACQUISITION PARAMETERS ---
File Name = 1d_13c_spectrum_copy.30
Author =
Sample ID = tt-b4-20-1
Content = Single Pulse with Broad
Creation Date = 20-JUN-2012 18:51:54
Revision Date = 23-JUN-2012 18:56:05
Spec Site = ECP500
Spec Type = DELTA_NMR
Data Format = ID COMPLEX
Pulse Program = zgpg30
Dim Title = 13C
Dim Size = 32768
Dim Units = [ppm]
Scans = 201.0
Acq Return = 13C
X Domain = 13C
X Offset = 100 [ppm]
X Freq = 125.77787547 [MHz]
X Sweep = 31.44654088 [kHz]
Solvent = CHLOROFORM-D
Temp Set = 127 [K]
Temp Get = 23.5 [C]
Recvr Gain = 30
Field Strength = 11.7473579 [T]
Filter Mode = BUTTERWORTH
Filter Width = 15.7206221 [kHz]



(Millions)

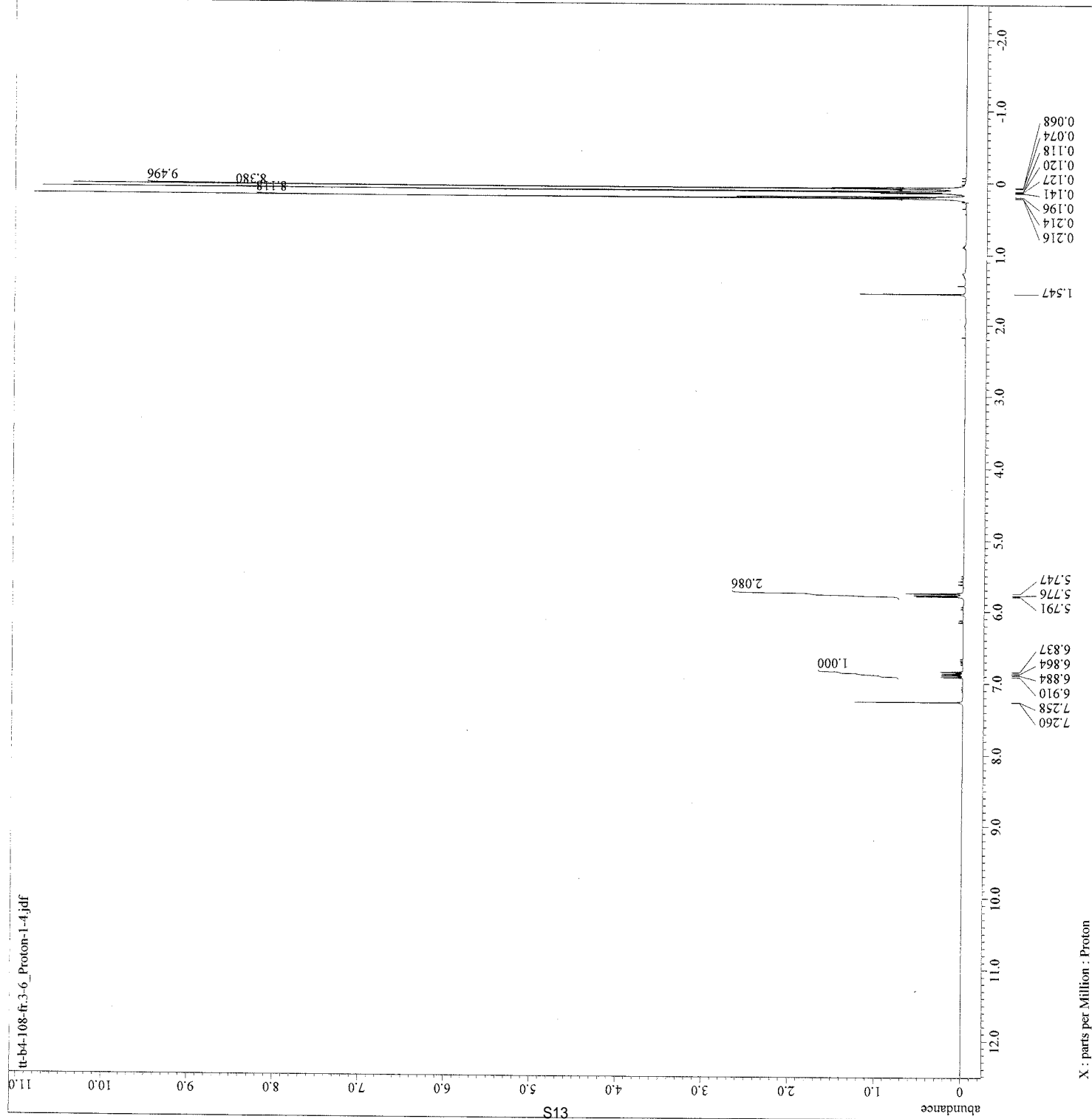
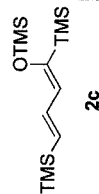
10.0 30.0 50.0 70.0 90.0 110.0 130.0 150.0 170.0 190.0 210.0 230.0 250.0 270.0 290.0 310.0 330.0 350.0



X : parts per Million : 13C



Filename = C:\Users\delta\Documents\J
 Author = delta
 Experiment = 108-fr.3-6
 Sample Id = tt-b4-108-fr.3-6
 Solvent = CHLOROFORM-D
 Creation Time = 11-DEC-2012 19:25:45
 Revision Time = 12-MAR-2013 16:43:34
 Current Time = 12-MAR-2013 16:52:20
 Comment = sample pulse
 Data Format = ID COMPLEX
 Data Size = 13107
 Data Title = Proton
 Data Units = [ppm]
 Dimensions = X
 Site = JNM-ECS400
 Spectrometer = DELTA2_NMR
 Field Strength = 9.42495681 [T] (400 [MHz])
 X Acq Duration = 2.1757952 [s]
 X Domain = 1H
 X Freq = 401.28219856 [MHz]
 X Offset = 5 [ppm]
 X Points = 16384
 X Resolution = 0.45960208 [Hz]
 X Sweep = 7.53012048 [kHz]
 X Sweep Clipped = 6.02409639 [kHz]
 Irr Domain = Proton
 Irr Freq = 401.28219856 [MHz]
 Irr Offset = 5 [ppm]
 Irr Points = 16384
 Irr Domain = Proton
 Tri Freq = 401.28219856 [MHz]
 Tri Offset = 5 [ppm]
 Clipped = FALSE
 Scans = 8
 Total Scans = 8
 Relaxation Delay = 5 [s]
 Recor Gain = 41
 Temp Set = 20.8 [dC]
 X 90 Width = 9.25 [us]
 X Acq Time = 2.1757952 [s]
 X Angle = 45 [deg]
 X Attn = 0.8 [dB]
 X Pulse = 4.625 [us]
 X Mode = Off
 Tri Mode = Off
 Dantec Presat = FALSE
 Initial Wait = 1 [s]
 Repetition Time = 7.1757952 [s]





----- PROCESSING PARAMETERS -----

```

      dc_balance
      ssexp : 5[Hz]
      fft : 1
      ppm
      machinephase

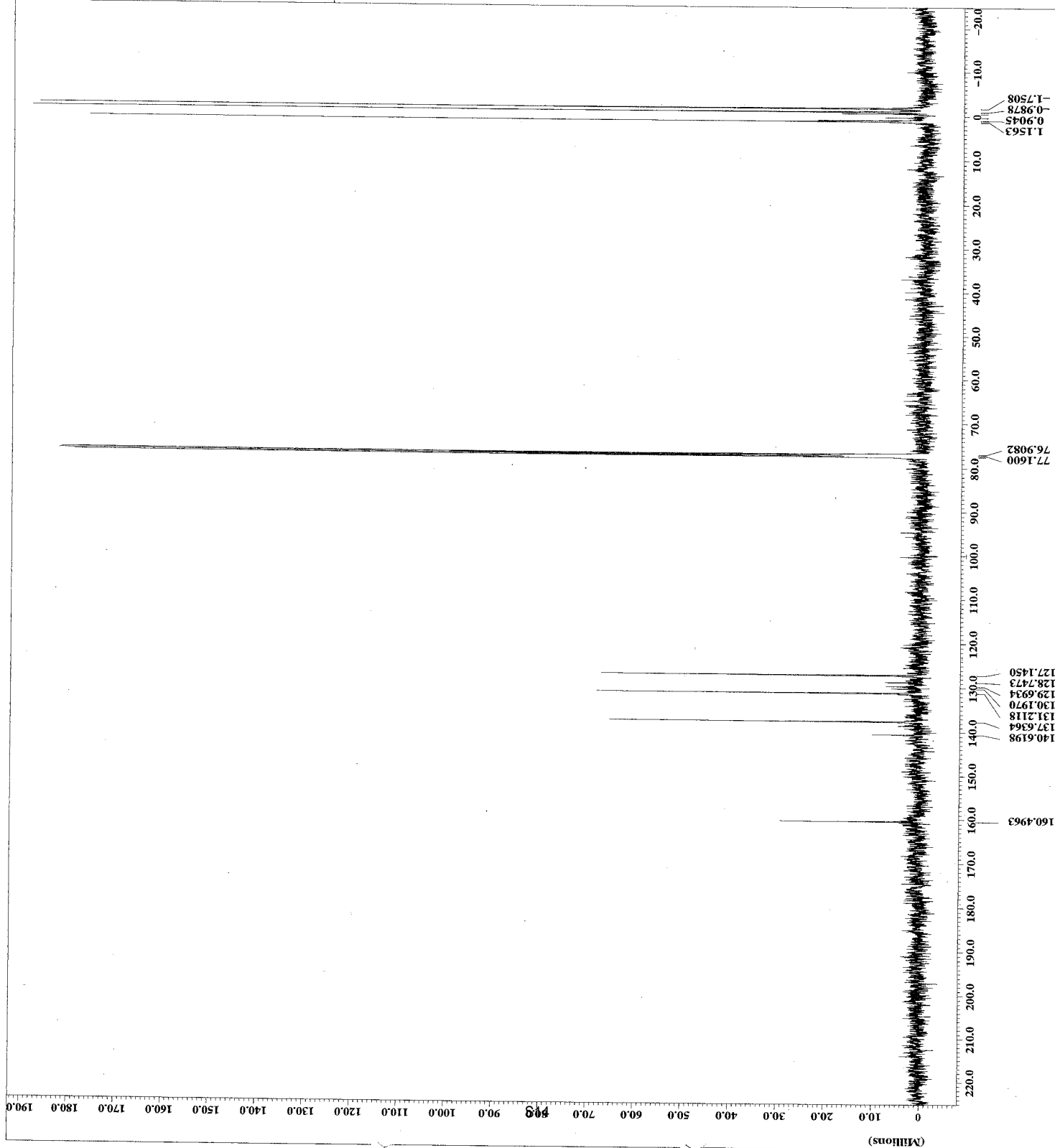
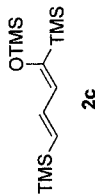
```

----- ACQUISITION PARAMETERS

```

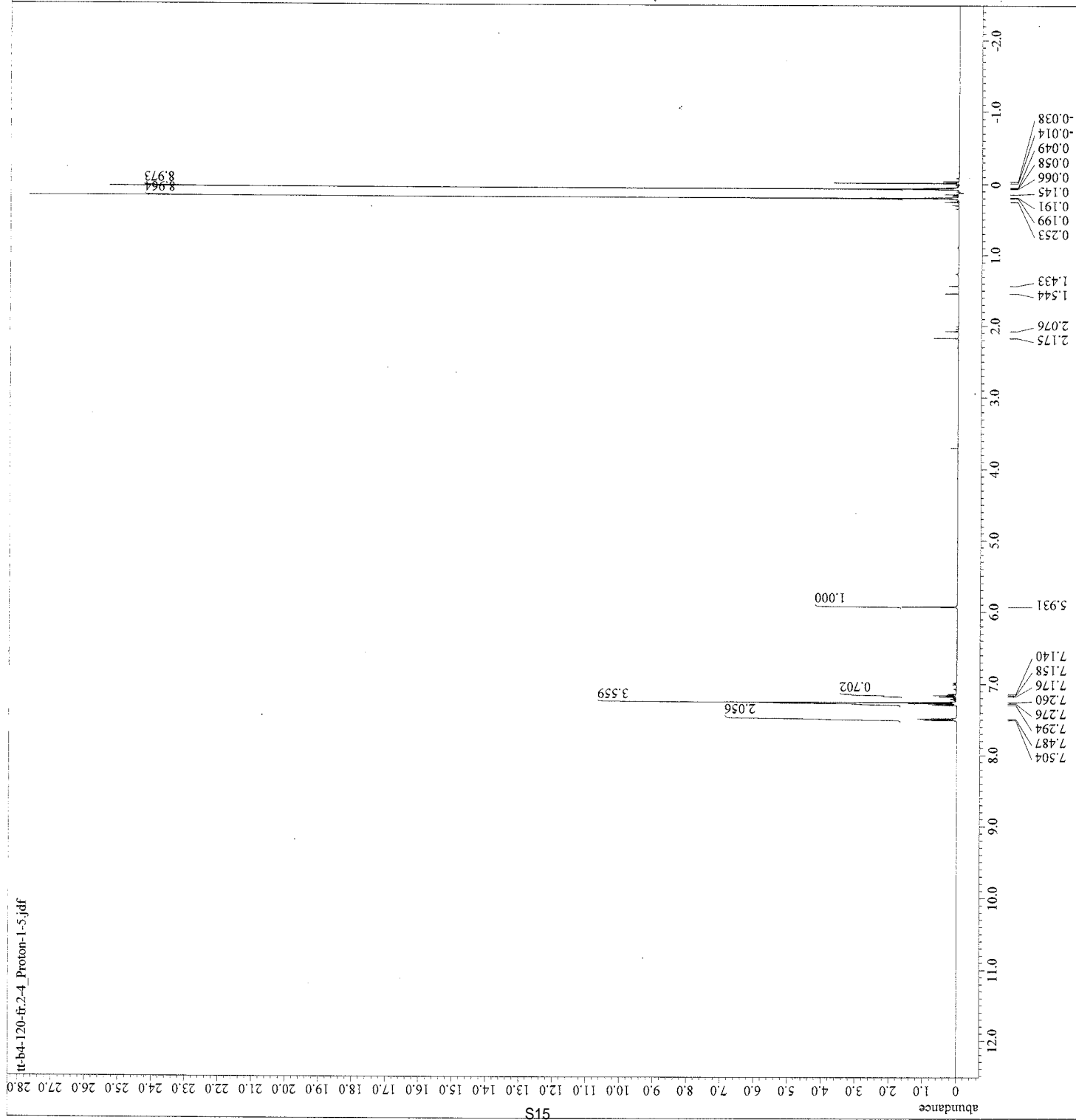
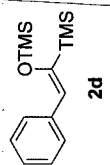
# =====
# File Name      = 14_13c_spectrum.106
# Author         =
# Sample ID     = VV-84-74-FR-3-4
# Contactor     = Steve Pulse with Broad
# Revision Date = 7-MAR-2012 13:28:35
# Revision Date = 10-NOV-2012 15:05:29
# Spec Site     = ECF500
# Spec Type     = DELTA_NMR
# Spec Type     = 1D COMPLEX
# Dimensions    = X
# Dimensions    = Y
# Spin Units    = 32768
# Spin Units    = [cps]
# Scans         = 304
# Scans         = 13C
# C1Acq         = 125.77878547[MHz]
# C1Freq        = 125.77878547[MHz]
# C1Sweep       = 31.44634089[MHz]
# C1Prgm        = 16[Hs]
# C1Prgm        = 24.2[dC]
# Temp_spt     = 30
# Temp_spt     = 7473579.7p
# AcqGain       = 1.0
# AcqGain       = 1.0
# Filter width  = 45.72066221[MHz]
#

```



X : parts per Million : 13C

FileNames	= C:\Users\deltaelta\Documents\ex
Author	= delta
Experiment	= proton_jxp
Sample_Id	= tr-b4-120-fr_2-4
Solvent	= CHOROFORM-D
Acquisition_Time	= 12-MAR-2013 19:11:48
Resting_Time	= 12-MAR-2013 19:33:46
Current_Time	= 12-MAR-2013 18:33:25
Comment	= single pulse
Data_Format	= ID_COMPLEX
Dat_Size	= 13107
Pulse_Program	= Proton
Rm_Tools	= X
Dimensions	= X
Site	= JMR-ECS400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.42495681 [T] (400 [MHz])
Acq_Duration	= 1.1757952 [s]
Q_Offset	= 18
Freq	= 401.28219856 [MHz]
X_Offset	= 5 [ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45460208 [Hz]
X_Sweep	= 0.00000000 [Hz]
X_Sweep_Clippped	= 6.024045639 [kHz]
Irr_Domain	= Proton
Irr_Freq	= 401.28219856 [MHz]
Irr_Offset	= 5 [ppm]
Tri_Domain	= Proton
Tri_Freq	= 401.28219856 [MHz]
Tri_Offset	= 5 [ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5 [s]
Recvr_Gain	= 48
Recv_Units	= 7.25 [dB]
X_90_Width	= 9.25 [us]
X_AcqTime	= 2.1757952 [s]
X_Angle	= 45 [deg]
X_Pulse	= 0.8 [db]
X_Align	= 4.625 [us]
Irr_Mode	= Off
Probe Preset	= WALTSE
Initial Wait	= 1 [s]
Repetition Time	= 7.1757952 [s]



X: parts per Million : Proton

```

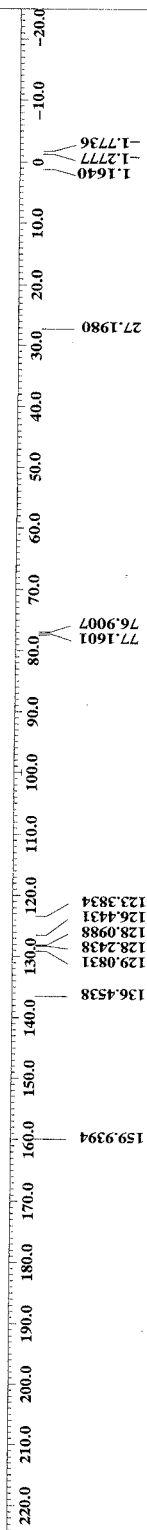
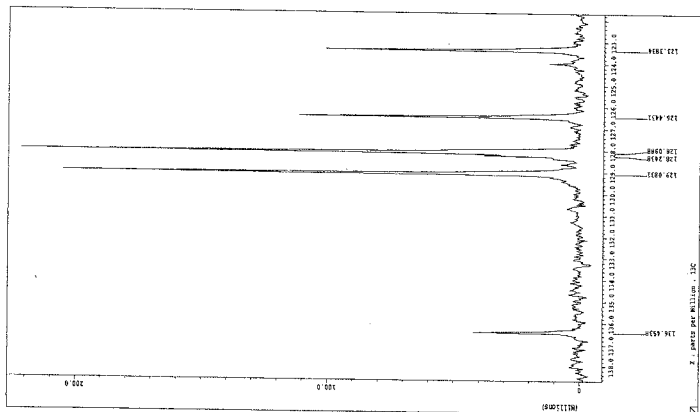
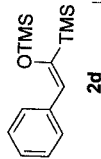
----- PROCESSING PARAMETERS -----
dc_balance
sexp : 5[Hz]
fft : 1
ppm
machinephase

```

```

===== ACQUISITION PARAMETERS =====
File Name      = 1d_13c_spectrum.11
Author         =
Sample ID      = tr-54-120-efr-2-4
Experiment     = Single Pulse with Broad
                16-VAN-2013 13:49:52
Creation Date  =
Revision Date  =
Spec Site     = 21-VAN-2013 22:46:08
ECP50         = ECP500
Spec Type     = DEPTA-WO
Data Format    = 1D COMPLEX
Dimensions    = 13c
Dim1 title    = 13c
Dim2 title    = 32F88
Dim3 title    = 32F88
Dim4 title    = 400m
Dim5 title    = 400m
Dim6 title    = 400m
Mod-return    = 1
X.domain      = 13c
X.offset      = 100 [ppm]
X.sweep       = 25.473473 [MHz]
Y.sweep       = 34.4455088 [kHz]
Solvent       = CHOROFORM-D
Spin_get      = 17 [Hz]
Temp_get      = 30.0 [C]
Pulse_prog    = zgpg30
Pulse_gain    = 30
Pulse_length  = 30.7473579 [m]
BUTYRNOGTH    = 15.72066221 [kHz]
Filter_mode   =
Filter_width  =

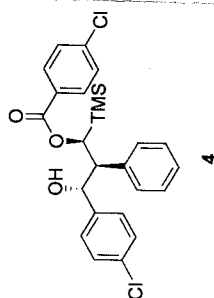
```



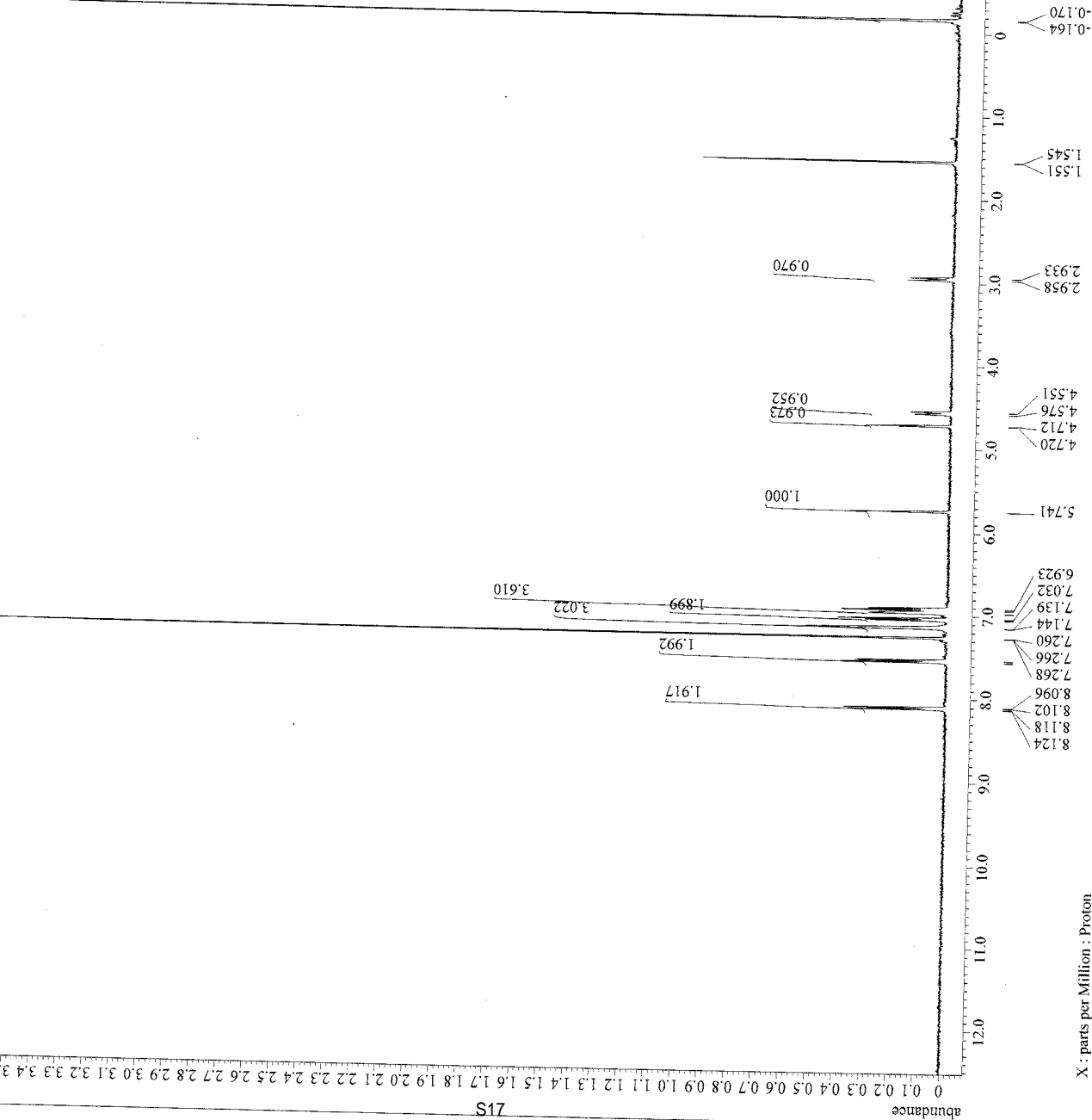
X : parts per Million : 13C



File Name = C:\Users\delta\Documents\3
 Author = delta
 Experiment = proton_1-4
 Sample Id = ester
 Solvent = CHLOROFORM-D
 Creation Time = 18-MAR-2013 13:57:42
 Revision Time = 18-MAR-2013 13:57:10
 Current Time = 18-MAR-2013 13:57:21
 Comment = single pulse
 Data Format = ID COMEXX
 Dim Size = 13107
 Dim Title = Proton
 Dim Units = [ppm]
 Dimensions = X
 Site = 004-PCS400
 Spectrometer = DPX400_NMR
 Field Strength = 9.42499681[T] (400[Mhz])
 X_Acq_Duration = 2.1757952[s]
 X_Domain = 1H
 X_Freq = 401.28219856[Mhz]
 X_Offset = 1.0584
 X_Points = 1
 X_Prescans = 0.45960208[Hz]
 X_Resolution = 7.53012048[kHz]
 X_Sweep_Clip = 6.02409639[kHz]
 X_Sweep_Domain = Proton
 X_Sweep_Freq = 401.28219856[Mhz]
 X_Sweep_Offset = 5[ppm]
 X_Sweep_Points = 1
 X_Sweep_Tri_Domain = 401.28219856[Mhz]
 X_Sweep_Tri_Freq = 401.28219856[Mhz]
 X_Sweep_Tri_Offset = 5[ppm]
 X_Sweep_Tri_Points = FALSE
 X_Sweep_Tri_Scans = 8
 X_Sweep_Tri_Total_Scans = 8
 Relaxation_Delay = 5[s]
 Recv_Gain = 56
 Temp_Cet = 20.6[dc]
 X_90_Width = 9.25[us]
 X_Acq_Time = 2.1757952[s]
 X_Angle = 45[deg]
 X_Acquisition = 0.8[db]
 X_Pulse = 4.025[us]
 X_Pulse_Mode = Off
 X_Pulse_Tri_Mode = Off
 X_Pulse_Tri_Presat = FALSE
 Initial Wait = 1[s]
 Repetition Time = 7.1757952[s]



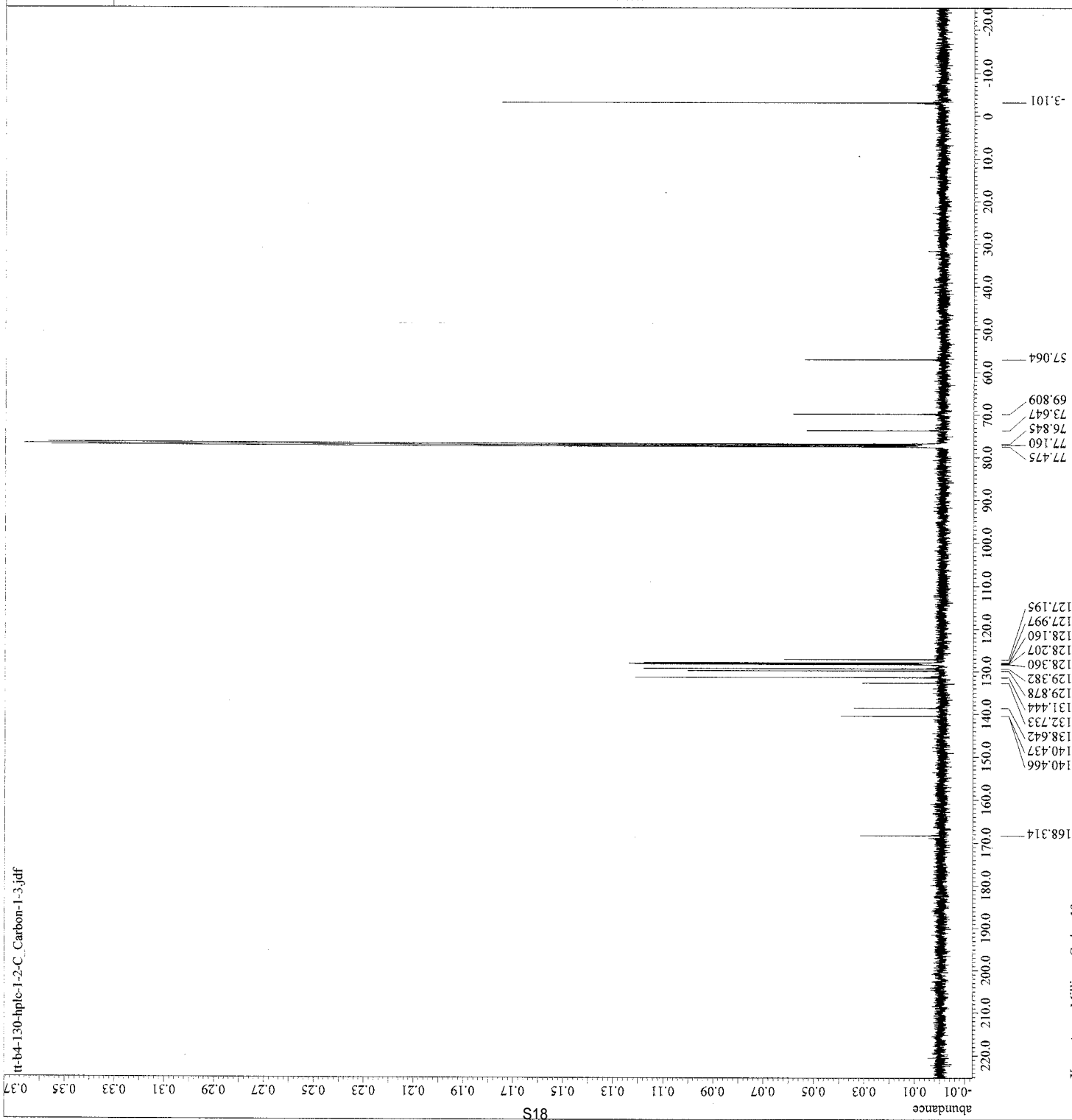
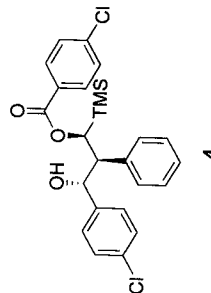
8.466



217



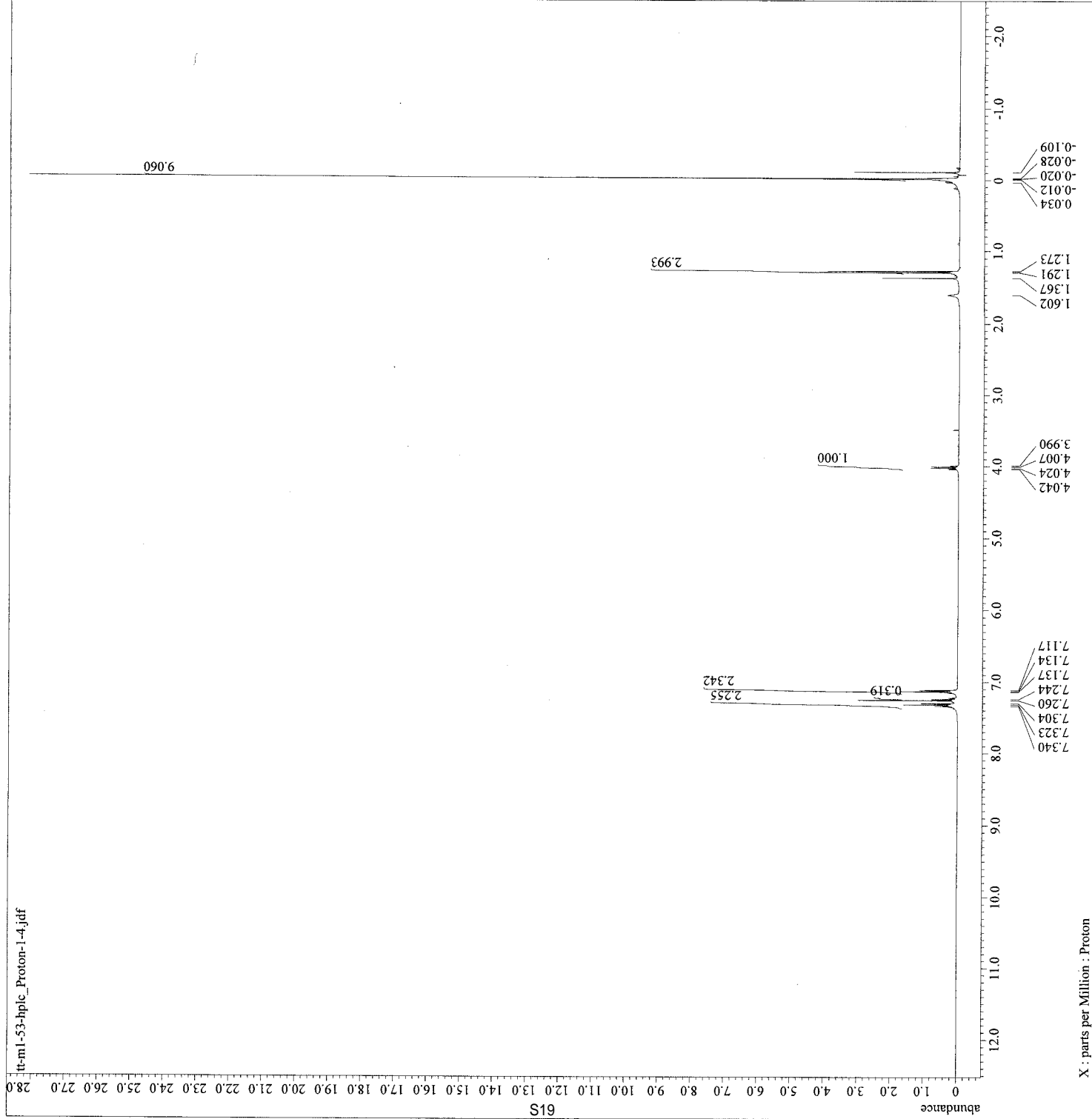
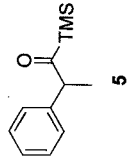
FileNames	= C:\Users\dalra\Documents\A
Author	= delta
Experiment	= carbon_jsp
Sample_Id	= tt-bd-130-hplc-1-2-C
Solvent	= CHLOROFORM-D
Creation_Time	= 11-MAR-2013 11:05:18
Revision_Time	= 12-MAR-2013 18:27:21
Current_Time	= 12-MAR-2013 18:27:21
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim Size	= 76214
Dim Title	= Carbon13
Dim Units	= [ppm]
Dimensions	X = 100[ppm] Y = 100[ppm] Z = 100[ppm]
SpecName	= X-MG-PCS400
Spectrometer	= DELTA2_NMR
Field Strength	= 9.42499681[T] (400[MHz])
Acq Duration	= 1.03809024[s]
X Domain	= 13C = 100.90247863[MHz] = 100[ppm] = 100[ppm] = 4 = 4
X Freq	= 0.96330739[Bz]
X Offset	= 31.5655657[Bz]
X Prescan	= 25.25252525[kHz]
X Resolution	= Proton
X Sweep	= 401.28219856[MHz]
X Sweep_Clippped	= 51[ppm] = 25.25252525[kHz] = 256
Irr Freq	= 401.28219856[MHz]
Irr Domain	= Proton
Irr Onset	= 51[ppm]
Acq'd Time	= 256
Total_Scans	= 256
Relaxation_Delay	= 2[s]
Recovr Gain	= 50
Temp	= 20.5[degC]
X_90_Width	= 8.75[us]
Acq Time	= 30.6809024[s]
Noise	= 5.2[dB]
X Att	= 2.91666667[us]
X Pulse	= 22.691[dB]
Irr Att Dec	= 22.691[dB]
Irr Att	= 22.691[dB]
Irr Noise	= WALTZ
Irr Width	= 0.115[msec]
Decoupling	= WALTZ
Dithing	= 11.1[us]
Initial_Wait	= 7008
Noe Time	= 7008
Repetition Time	= 3.03809024[s]



X : parts per Million : Carbon13

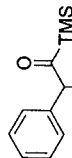


Filename = tt-ml-53-hplc_Proton-1-4.j
 Author = delta
 Experiment = proton_jxp
 Sample Id = tt-ml-53-hplc
 Solvent = CHLOROFORM-D
 Creation_Time = 26-AUG-2013 11:55:17
 Revision_Time = 26-AUG-2014 18:58:37
 Current_Time = 26-AUG-2014 19:02:26
 Comment = single pulse
 Data Format = 1D COMPLEX
 Dim Size = 13107
 Dim Title = Proton
 Dim Units = [ppm]
 Dimensions = 1
 Name = RM-RS2400
 Spectrometer = DELTA2_NMR
 Field Strength = 9.42499681[T] (400[Mhz])
 X_Acq_Duration = 2.1757952[s]
 X_Domain = 1H
 X_Freq = 401.28219856[Mhz]
 X_Offset = 1.6384
 X_Prescans = 1
 X_Resolution = 0.45960208[Hz]
 X_Sweep = 7.53012048[Hz]
 X_Sweep_Clip = 6.02409639[Mhz]
 Irq_Domain = Proton
 Irq_Freq = 401.28219856[Mhz]
 Irq_Offset = 5.1111
 Irq_Prescans = 5
 Tri_Domain = Proton
 Tri_Freq = 401.28219856[Mhz]
 Tri_Offset = 5[ppm]
 Clipped = FALSE
 Scans = 8
 Total_Scans = 8
 Relaxation_Delay = 5[s]
 Recvr Gain = 42
 Temp_Get = 21.6[degC]
 X_90_Width = 9.25[us]
 X_Acq_Time = 2.1757952[s]
 X_Angle = 45[deg]
 X_Cycle = 1
 X_Pulse = 4.625[us]
 Irq_Mode = Off
 Tri_Mode = Off
 Dante_Preset = FALSE
 Initial_Wait = 1[s]
 Repetition_Time = 7.1757952[s]

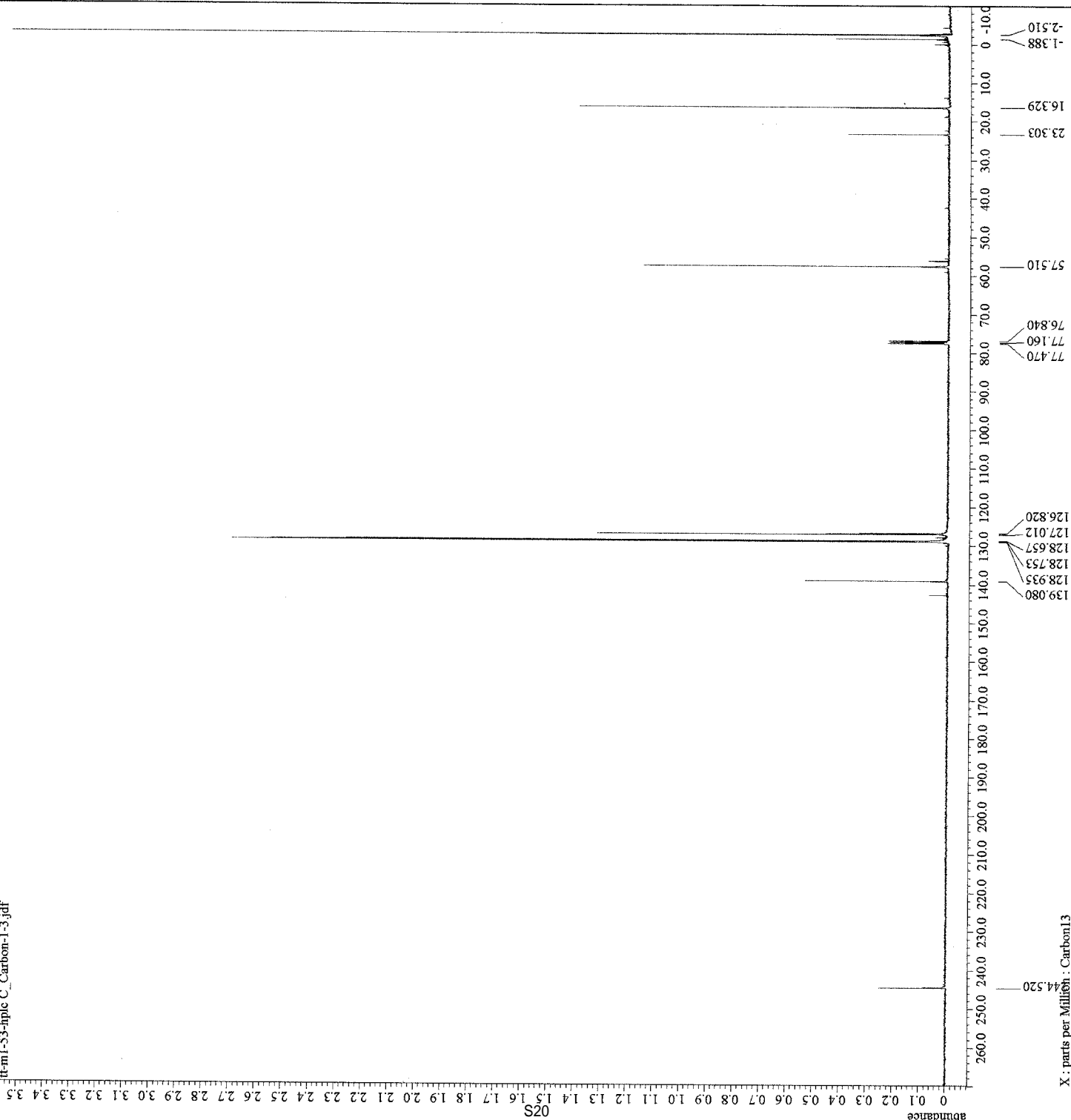




Filename = \\Users\delta\Documents\JEO
 Author = delta
 Experiment = carbon_jpp
 Sample_Id = tt-m1-53-hplc_C
 Solvent = CHLOROFORM-D
 Creation_Time = 26-AUG-2013 12:02:15
 Revision_Time = 26-AUG-2013 13:04:06
 Current_Time = 26-AUG-2013 13:04:52
 Comment = single pulse decoupled gat
 Data_Format = 1D COMEXX
 Dia_Size = 26214
 Dia_Title = Carbon13
 Dia_Units = [ppm]
 Dimensions = X
 Site = JNM-EC5400
 Spectrometer = DELTA2_MK2
 Field_Strength = 9.42499681[T] (400 [MHz])
 X_Acq_Duration = 0.92798976[s]
 X_Domain = 13C
 X_Freq = 100.62647663 [MHz]
 X_Offset = 130.1 [ppm]
 X_Points = 32768
 X_Frescan = 4
 X_Resolution = 1.0775981 [Hz]
 X_Sweep = 35.31073446 [kHz]
 X_Sweep_Clipped = 28.24858757 [kHz]
 X_Domain = Proton
 Xir_Freq = 401.28219856 [MHz]
 Xir_Offset = 5 [ppm]
 Xir_Clipped = FALSE
 Scans = 256
 Total_Scans = 256
 Relaxation_Delay = 2[s]
 Recvr_Gain = 50
 Temp_Get = 21.9 [deg]
 X_P0_Width = 8.75 [us]
 X_Acq_Time = 0.92798976[s]
 X_Pulse = 20 [deg]
 X_Atr = 2.91666667 [us]
 Xir_Atr_Dec = 22.691 [ns]
 Xir_Atr_Noe = 22.691 [ns]
 Xir_Noise = WALTZ
 Xir_Fwidth = 0.115 [ms]
 Decoupling = TRUE
 Initial_Wait = 1[s]
 Noe = TRUE
 Noe_Time = 2[s]
 Repetition_Time = 2.92798976[s]



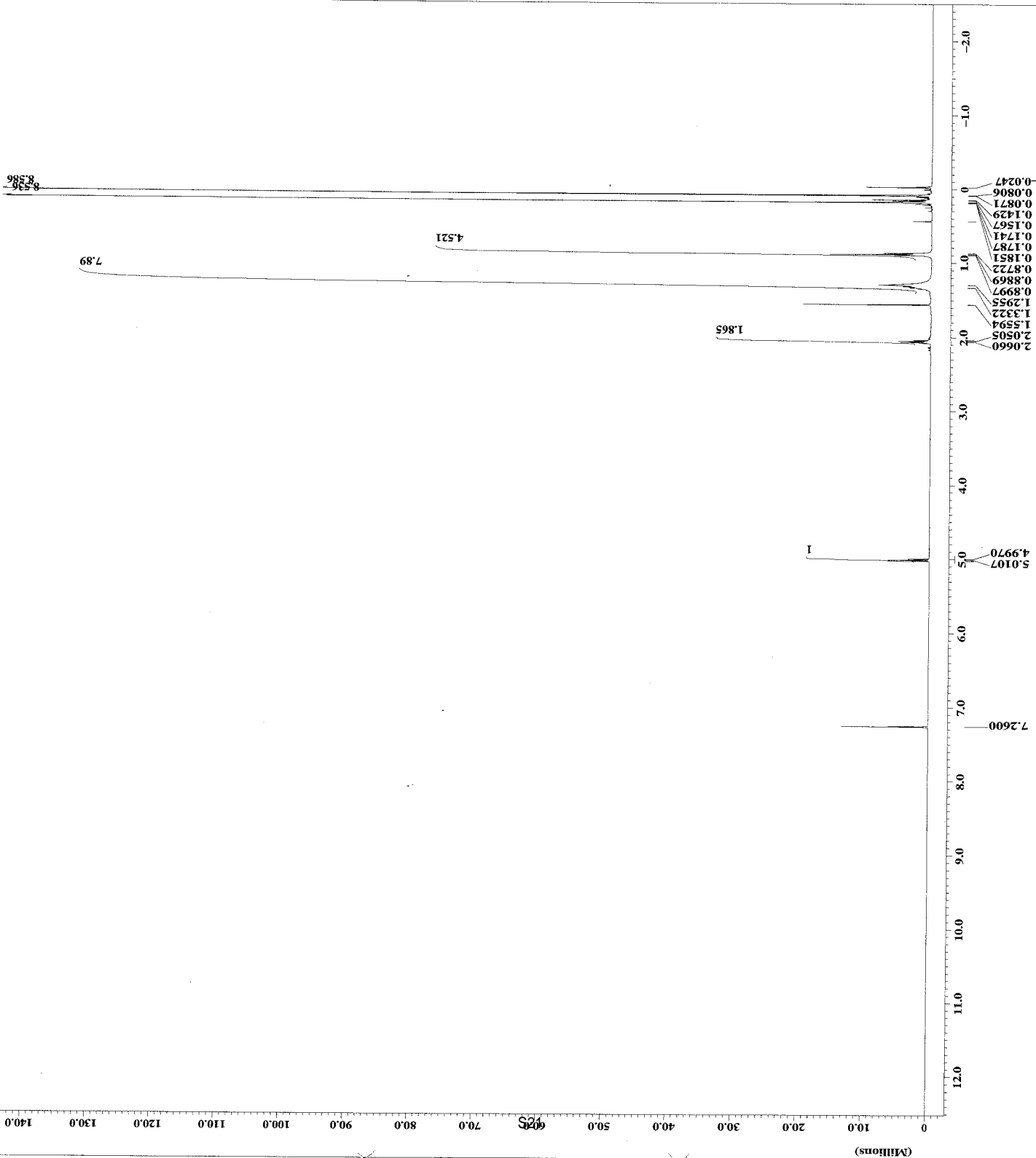
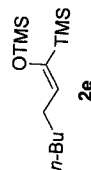
5



```

===== ACQUISITION PARAMETERS =====
File Name      = 1d_spectrum.235
Author
Sample ID      = tc-ml-58-distillate
Content        = Single Pulse Experiment
Creation Date   = 29-AUG-2013 14:14:45
Revision Date  = 1-SEP-2013 22:19:27
Spec Site      = ECF5050
Soc Type       = DELTA NMR
Data Format     = 1D COMPLEX
Dimensions     = 1
Dim 1 title    = X
Dim 1 size     = 18384
Dim 2 title    = 13C
Dim 2 size     = 8
Dim 3 title    = Scans
Dim 3 size     = 1
Mod return     = 1
X domain        = 1H
X offset        = 5 [ppm]
X sweep         = 7.50750750 [MHz]
X resolution    = 7.50750751 [kHz]
X solvent       = CHLOROFORM-D
X spin.get      = 15 [Hz]
X proc.get      = 465 [G]
Y domain        = 1H
Y offset        = 5 [ppm]
Y sweep         = 7.50750750 [MHz]
Y resolution    = 7.50750751 [kHz]
Y solvent       = CHLOROFORM-D
Y spin.get      = 15 [Hz]
Y proc.get      = 465 [G]
Field strength  = 11.7473579 [T]
Filter mode     = BUTTERWORTH
Filter width    = 3.75119936 [MHz]
=====

```



X : parts per Million : 1H

Filename = Users\delta\Documents\JEO
 File = delta
 Experiment = tt-b4-151-fr.2-3
 Sample Id = CHELOFORM-D
 Solvent = 14-MAR-2013 23:17:32
 Revision Time = 15-MAR-2013 00:01:11
 Current Time = 15-MAR-2013 00:01:20
 Comment = single pulse decoupled gat
 Data Format = ID_CPMZEX
 Dim Size = 26214
 Dim Title = Carbon13
 Dim Units = [ppm]
 Dimensions = X
 Site = JNM-SCS400
 Spectrometer = DELTA2_NMR
 Field Strength = 9.42499681[T] (400[MHz])
 X Acq Duration = 1.03809024[s]
 X Domain = 13C
 X Freq = 100.90247863[MHz]
 X Offset = 100[ppm]
 X Points = 32768
 X Resolution = 0.96330739[Hz]
 X Sweep = 31.56565657[KHz]
 X Sweep Clipped = 25.25252525[KHz]
 Irr Domain = Proton
 Irr Freq = 401.28219856[MHz]
 Irr Offset = 5[ppm]
 Clipped = 0.452
 Scans = 947
 Total Scans = 947
 Relaxation Delay = 2[s]
 Recvr Gain = 50
 Temp Get = 20[dc]
 X Acq Width = 0.75[us]
 X Acq Time = 1.03809024[s]
 X Angle = 30[deg]
 X Atn = 5.2[db]
 X Pulse = 2.91666667[us]
 Irr Atn Dec = 22.691[db]
 Irr Atn Noe = 22.691[db]
 Irr Noise = WALTZ
 Irr Power = 1.5[db]
 Decoupling = TRUE
 Initial Wait = 1[s]
 Noe = TRUE
 Noe Time = 2[s]
 Repetition Time = 3.03809024[s]

