Supporting Information

Radical Addition of Alkyl halides to formaldehyde in the Presence of Cyanoborohydride as a Radical Mediator. A New Protocol for Hydroxymethylation Reaction

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Table of Contents

General Techniques. Thin layer chromatography (TLC) was performed on Merck precoated plates (silica gel 60 F254, Art 5715, 0.25 mm) and were visualized by fluorescence quenching under UV light or by staining with *p*-anisaldeyde/AcOH/H_2SO_4/EtOH, or $12MoO_3 \cdot H_3PO_4$/EtOH. The products were purified by flash chromatography on silica gel (Kanto Chem. Co. Silica Gel 60N (spherical, neutral, 40-50 mm)) and, if necessary, were further purified by recycling preparative HPLC (Japan Analytical Industry Co. Ltd., LC-918) equipped with GPC columns (JAIGEL-1H + JAIGEL-2H columns) using $CHCl_3$ as eluent. ^{1}H NMR spectra were recorded with a JEOL JMN-ECS400 (400 MHz) spectrometer or a Varian MR400 (400 MHz) spectrometer and referenced to the solvent peak at 7.26 ppm. ^{13}C NMR spectra were recorded with a JEOL JMN-ECS500 (100 MHz) spectrometer or a Varian MR400 (100 MHz) spectrometer and referenced to the solvent peak at 77.00 ppm. Splitting patterns are indicated as follows: br, broad; s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Infrared spectra were recorded on a JASCO FT/IR-4100 spectrometer and are reported as wavenumber (cm^{-1}). High resolution mass spectra were recorded with a JEOL MS700 spectrometer. Mp was measured in capillary using BUCHI Melting Point B-540.

Acetonitrile (MeCN) was distilled over calcium hydride. Borohydrides were prepared in a similar manner as described for corresponding borohydride[1]. Alkyl iodides **1a, 1b**, **1c, 1d, 1e,** and **1f** were prepared from the corresponding alcohol according to the literature methods.[2] Alkyl iodides **1j** and **1k** were prepared from cyclohexene and corresponding alcohol to the literature method.[3] Other reagents were commercially available and used without further purification.

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[1] Hutchins, R. O.; Kandasamy, D. *J. Am. Chem. Soc.* **1973**, *95*, 6131.

[2] (a) Olah, G. A.; Narang, S. C.; Gupta, B. G. B.; Malhotra, R. *J. Org. Chem.* **1979**, *44*, 1247. (b) Lukach, A. E.; Rossi, R. A. *J. Org. Chem.* **1999**, *64,* 5826.

[3] Gerald, H.; Mary-Jeanne, L.; Anne, R.; Rex T, W. *Tetrahedron* **1993**, *20,* 4229.

General Procedure.

Method A

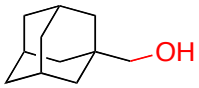
A magnetic stirring bar, 1-iodoadamantane (**1a**) (66 mg, 0.25 mmol), *n*-Bu_4NBH_3CN (282 mg, 1.0 mmol), and paraformaldehyde (113 mg, 3.8 mmol) were placed in a Pyrex test tube. Purged three times with nitrogen, then CH_3CN (1 mL) was added to the mixture. The mixture was irradiated by black light (15 W) with stirring for 3 h under nitrogen. Methanol (1 mL) was added to the reaction mixture and stirring for 10 min. The reaction mixture was filterated through silica pad, then concentrated. The residue was purified by flash chromatography on SiO_2 (hexane/AcOEt = 10) to give 1-adamantanemethanol **1b** (35 mg, 0.21 mmol, 85%).

Method B

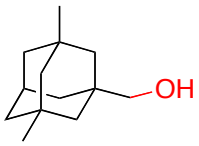
A magnetic stirring bar, 1-bromodoadamantane (**1a**) (109 mg, 0.51 mmol), *n*-Bu_4NBH_3CN (562 mg, 2.0 mmol), and paraformaldehyde (225 mg, 7.6 mmol) were placed in a quartz test tube. Purged three times with nitrogen, then CH_3CN (2 mL) was added to the mixture. The mixture was irradiated by low pressure Hg lamp (6 W) with stirring for 12 h under nitrogen. Methanol (1 mL) was added to the reaction mixture and stirring for 10 min. The reaction mixture was filterated through silica pad, then concentrated. The residue was purified by flash chromatography on SiO_2 (hexane/AcOEt = 10) to give 1-adamantanemethanol **1b** (71 mg, 0.43 mmol, 85%).

Method C

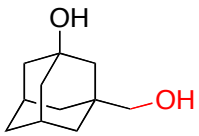
A magnetic stirring bar, 1-iodoadamantane (**1a**) (65 mg, 0.25 mmol), *n*-Bu_4NBH_3CN (140 mg, 0.5 mmol), paraformaldehyde (56 mg, 1.9 mmol) and AIBN (8 mg, were placed in a Pyrex test tube. Purged three times with nitrogen, then CH_3CN (1 mL) was added to the mixture. The mixture was heated at 80 ºC for 3 h under nitrogen. Methanol (1 mL) was added to the reaction mixture and stirring for 10 min The reaction mixture was filterated through silica pad, then concentrated. The residue was purified by flash chromatography on SiO_2 (hexane/AcOEt = 10) to give 1-adamantanemethanol **1b** (36 mg, 0.22 mmol, 87%).



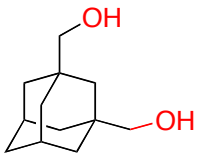
1-Adamantanmethanol (2a). colorless solid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.45-1.58 (m, 6H), 1.60-1.70 (m, 3H), 1.70-1.80 (m, 3H), 1.96-2.04 (m, 3H), 3.20 (s, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 28.17, 34.47, 37.16, 39.02, 73.86. This product is commercially available and the 1H- and ^{13}C NMR spectra are consistent with those of the authentic sample.



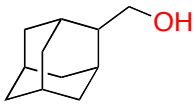
3,5-Dimethyladamantane-1-methanol (2b). colorless solid; mp. 58-59 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.82 (s, 6H), 1.07-1.13 (m, 3H), 1.14-1.20 (m, 3H), 1.30-1.36 (m, 7H), 2.06-2.11 (m, 1H), 3.24 (s, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 29.30, 30.58, 30.91, 36.33, 37.65, 43.38, 45.32, 51.40, 73.30.



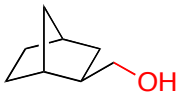
3-Hydroxy-1-adamantanemethanol (2c). colorless solid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.44-1.57 (m, 10H), 1.64-1.73 (m, 4H), 2.24-2.25 (m, 2H), 3.29 (s, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 30.35, 35.59, 37.71, 38.44, 44.85, 46.66, 68.84, 68.84, 72.66; FT-IR (KBr): 3374, 3307 cm^{-1}; MS (EI) *m/z* (rel intensity) 182 (M^+, 32), 151 (100), 107 (12), 95 (33), 91 (11), 77 (11) ; HRMS calcd for $C_{11}H_{18}O_2$ (M^+) 182.1307, found 182.1307.



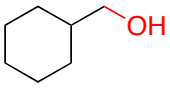
1,3-Adamantanedimethanol (2d). colorless solid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.28 (bs, 2H), 1.43-1.64 (m, 12H), 2.11 (brs, 2H), 3.26 (s, 4H); ^{113}C NMR ($CDCl_3$, 100 MHz): δ 28.12, 34.97, 36.66, 38.62, 40.44, 73.48. FT-IR (KBr): 3314 cm^{-1}; MS (EI) *m/z* (rel intensity) 196 (M^+, 17), 165 (100), 147 (28), 104 (31), 90 (20) 81 (11), 79 (18); HRMS calcd for $C_{12}H_{20}O_2$ (M^+) 196.1463, found 196.1470.



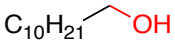
2-Adamantanemethanol (2e). colorless solid; mp. 82-84 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.25 (bs, 1H), 1.52-1.59 (m, 2H), 1.71-1.95 (m, 13H), 3.73 (d, *J* = 8Hz, 2H); ^{13}C NMR (CDCl3, 100 MHz): δ 27.93, 28.37, 29.19, 31.91, 38.16, 38.86, 47.10, 65.08.



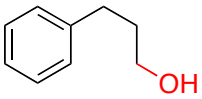
2-Norbornanemethanol (exo / endo = 99 / 1) (2f). colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.94-1.20 (m, 1H), 1.06-1.12 (m, 1H), 1.12-1.23 (m, 2H), 1.23-1.28 (m, 1H), 1.32-1.42 (m, 2H), 1.45-1.58 (m, 2H), 1.61-1.70 (m, 1H), 2.14 (m, 1H), 2.18-2.24 (m, 1H), 3.28-3.42 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): d 28.91, 29.82, 33.99, 35.13, 36.09, 38.09, 44.89, 66.92.



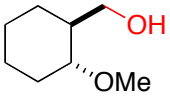
Cyclohexanemethanol (2g). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.87-0.96 (m, 2H), 1.13-1.28 (m, 2H), 1.42-1.73 (m, 1H), 1.72-1.75 (m, 6H), 2.65-2.90 (m, 1H), 3.42 (d, *J* = 6.0 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 25.81. 26.57, 29.52, 40.45, 68.78. This product is commercially available and the ^{1}H- and ^{13}C-NMR spectra are consistent with those of the authentic sample.



1-Undecanol (2h). colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.88 (t, *J* = 6 Hz, 3H), 1.18-1.50 (m, 16H), 1.53-1.58 (m, 2H), 3.64 (t, *J* = 6.4 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 14.10, 22.67, 25.71, 29.32, 29.42, 29.60, 31.90, 32.77, 63.07. This product is commercially available and the ^{1}H- and ^{13}C-NMR spectra are consistent with those of the authentic sample.

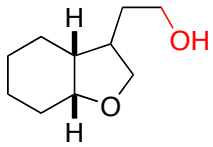


3-Phenyl-propane-1-ol (2i). colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.90 (q, *J* = 6.8 Hz, 2H), 2.72 (t, *J* = 7.6 Hz, 2H), 3.69 (t, *J* = 6.4 Hz, 2H), 7.17-7.31 (m, 5H).; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 32.06, 34.22, 62.31, 125.86, 128.40, 128.42, 141.79. This product is commercially available and the ^{1}H- and ^{13}C-NMR spectra are consistent with those of the authentic sample.



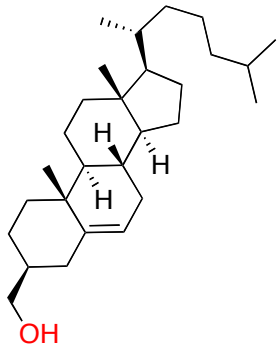
2-methoxycyclohexyl methanol (2j)[4] colorless oil;; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.92 (dq, *J* = 3.2, 12.6 Hz, 1H), 1.05-1.28 (m, 3H), 1.54-1.85 (m, 4H), 2.14-2.20 (m, 1H), 3.06 (dt, *J* = 3.6, 9.8 Hz, 1H), 3.37 (s, 3H), 3.47-3.56 (m, 1H), 3.61 (t, *J* = 8 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 24.27, 25.13, 27.70, 30.01, 45.07, 55.60, 68.71, 86.06.

[4] Christoph, R. *J. Orgmetal. Chem.* **1986**, *310*, 135.

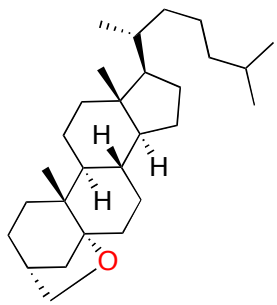


2-octahydrobenzofuran-3-yl)ethanol (2k) (dr = 75/25)

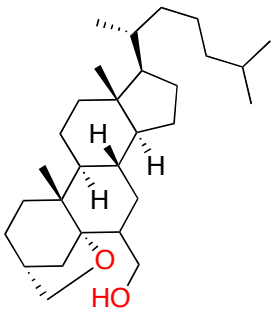
colorless oil; (major isomer)^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.04-1.19 (m, 2H), 1.22-1.79 (m, 7H), 1.83-1.90 (m, 1H), 1.94-2.00 (m, 1H), 2.39-2.50 (m, 1H), 3.55 (dd, *J* = 2.4, 7.2 Hz, 1H), 3.59-3.69 (m, 2H), 3.97 (t, *J* = 8.4 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 20.43, 22.03, 24.46, 28.48, 30.57, 39.97, 40.61, 62.10, 70.92, 78.16; FT-IR (neat) : 3398, 2930, 2860, 1446, 1433, 1157 cm^{-1}; MS (EI) *m/z* (rel intensity) 170 (M^+, 23), 125 (100), 98 (15), 83 (20), 81 (24) ; HRMS calcd for $C_{10}H_{18}O_2$ (M^+) 170.1307, found 170.1299.



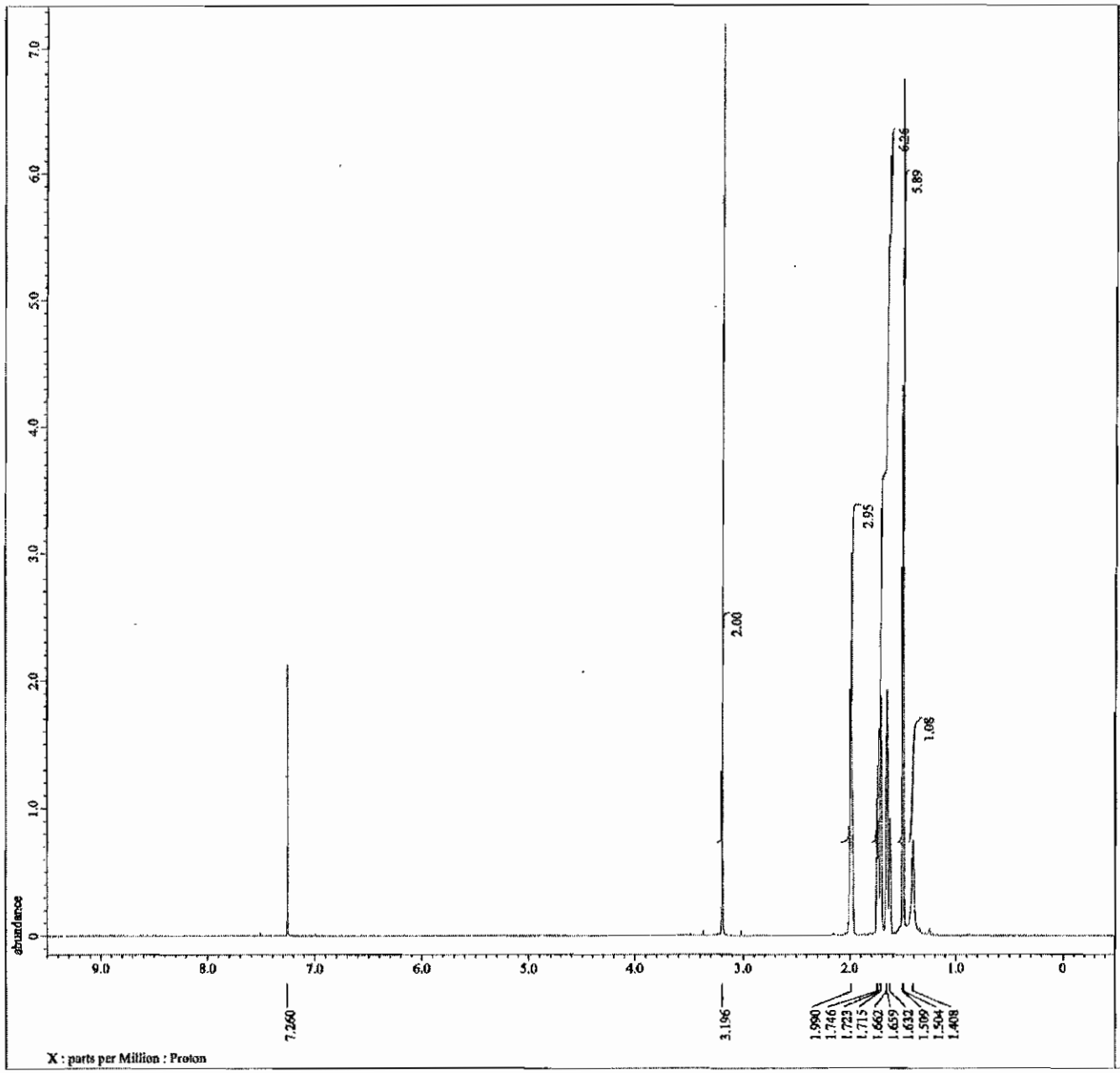
3-hydroxymethylcholest-5-ene (dr = 93/7) (2l). colorless solid; mp. 108-109 ºC; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.67 (s, 3H), 0.85-2.10 (m, 41H), 3.48 (t, *J* = 5.6 Hz, 2H), 5.31-5.33 (m, 1H); ^{13}C NMR($CDCl_3$, 100 MHz): δ 11.85, 18.70, 19.45, 22.55, 22.82, 23.82, 24.27, 25.34, 28.00, 28.23, 30.94, 31.87, 35.79, 36.18, 37.34, 39.00, 39.50, 39.80, 42.11, 42.29, 50.38, 56.14, 56.80, 68.50, 119.95, 142.30 ; FT-IR (KBr): 3338, 2931, 1458, 1376, 1332, 1141, 1066, 999, 960, 834 cm^{-1}; MS (EI) *m/z* (rel intensity) 400 (M^+, 100), 385 (88), 301 (45), 245 (45), 95 (45), 81 (36); HRMS calcd for $C_{28}H_{48}O$ (M+) 400.3705, found 400.3714.



(3) colorless solid; mp. 90-91 ºC; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.63 (s, 3H), 0.85 (d, *J* = 2 Hz, 3H), 0.87 (d, *J* = 2 Hz, 3H), 0.89 (d, *J* = 6 Hz, 3H), 0.90 (s, 3H), 0.95-1.62 (m, 27H), 1.75-1.85 (m, 1H), 1.89 (d, *J* = 11.6 Hz, 1H), 1.96 (dt, *J* = 2.8, 12.0 Hz, 1H), 2.31 (brs, 1H), 3.73-3.81 (m, 2H); ^{13}C NMR($CDCl_3$, 100 MHz): δ 11.89, 16.53, 18.65, 20.60, 22.55, 22.83, 23.92, 24.21, 27.44, 28.00, 28.25, 28.44, 32.51, 32.81, 34.85, 35.84, 36.16, 36.22, 39.49, 39.90, 41.09, 42.46, 47.86, 55.95, 56.23, 71.67, 85.4; FT-IR (KBr): 2920, 2857, 1459, 1382, 1116, 1037, 970, 945, 860, 791, 760 cm^{-1}; MS (EI) *m/z* (rel intensity) 400 (M^+, 100), 385 (22), 260 (12), 244 (15), 245 (20), 95 (11); HRMS calcd for $C_{28}H_{48}O$ (M^+) 400.3705, found 400.3711.



(**4**) colorless solid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.64 (s, 3H), 0.84-0.91 (m ,12H), 0.96-1.37 (m, 17H), 1.48-1.67 (m, 7H), 1.70-1.88 (m, 3H), 1.95-2.02 (m, 2H), 2.39 (brs, 1H), 3.47 (dt, *J* = 2.4, 9.6 Hz, 1H), 3.69-3.82 (m, 2H), 4.08 (dd, *J* = 2.4, 11 Hz, 1H); ^{13}C NMR($CDCl_3$, 100 MHz): δ 11.88, 16.38, 18.64, 20.59, 22.53, 22.81, 23.90, 24.14, 27.06, 27.98, 28.25, 31.91, 32.72, 34.07, 34.25, 35.76, 35.82, 36.14, 37.29, 39.46, 39.86, 41,77, 42.34, 47.84, 55.99, 56.19, 64.58, 71.40, 89.96; FT-IR ($CHCl_3$): 3019, 2399, 1521, 1424, 1212, 1046, 929 cm^{-1}; MS (EI) *m/z* (rel intensity) 430 (M^+, 100), 412 (18), 400 (21), 399 (25), 275 (13), 95 (17), 81 (15); HRMS (FAB) calcd for C28H48O (M-H^+) 431.3811, found 431.3871.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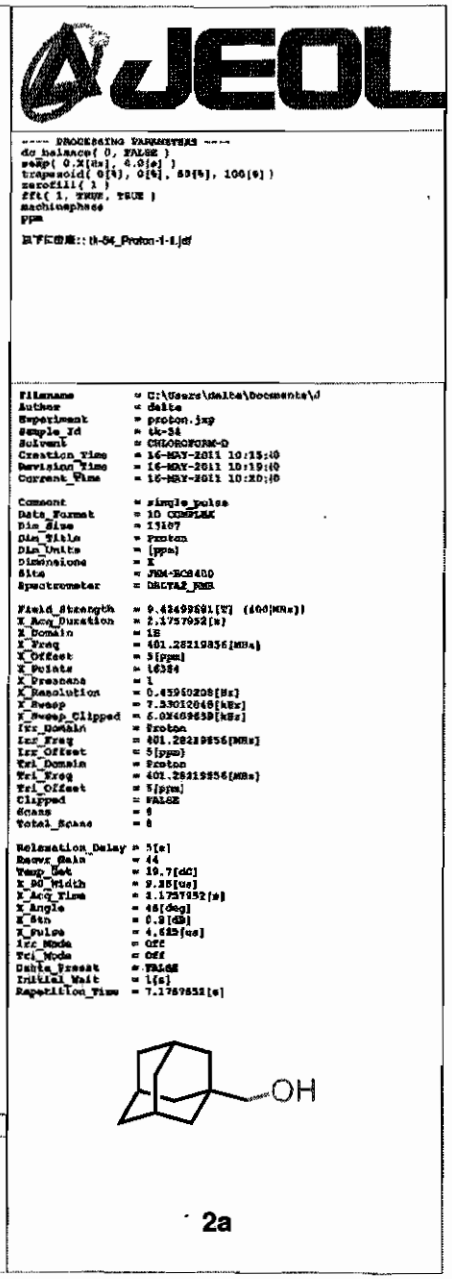
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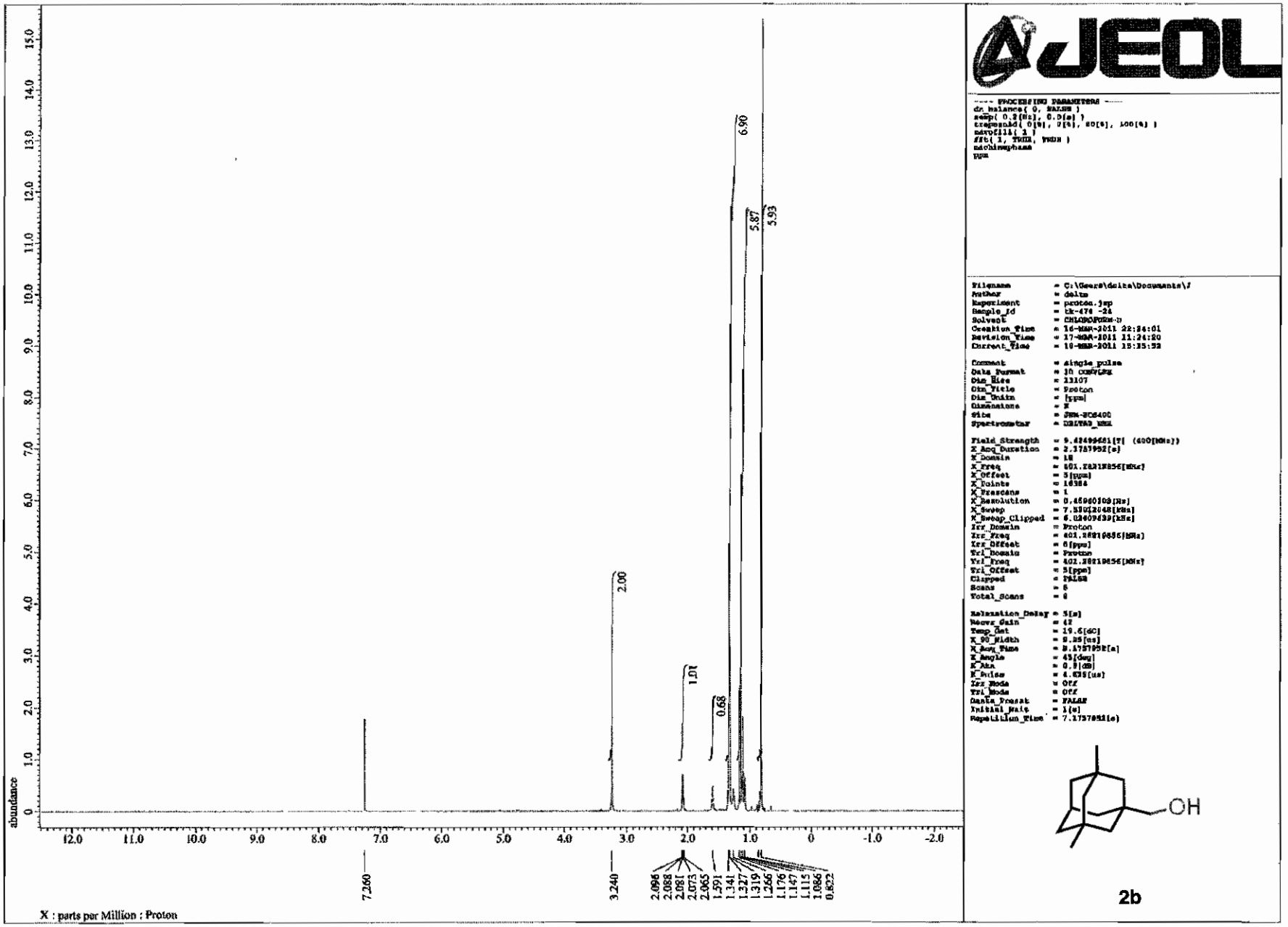
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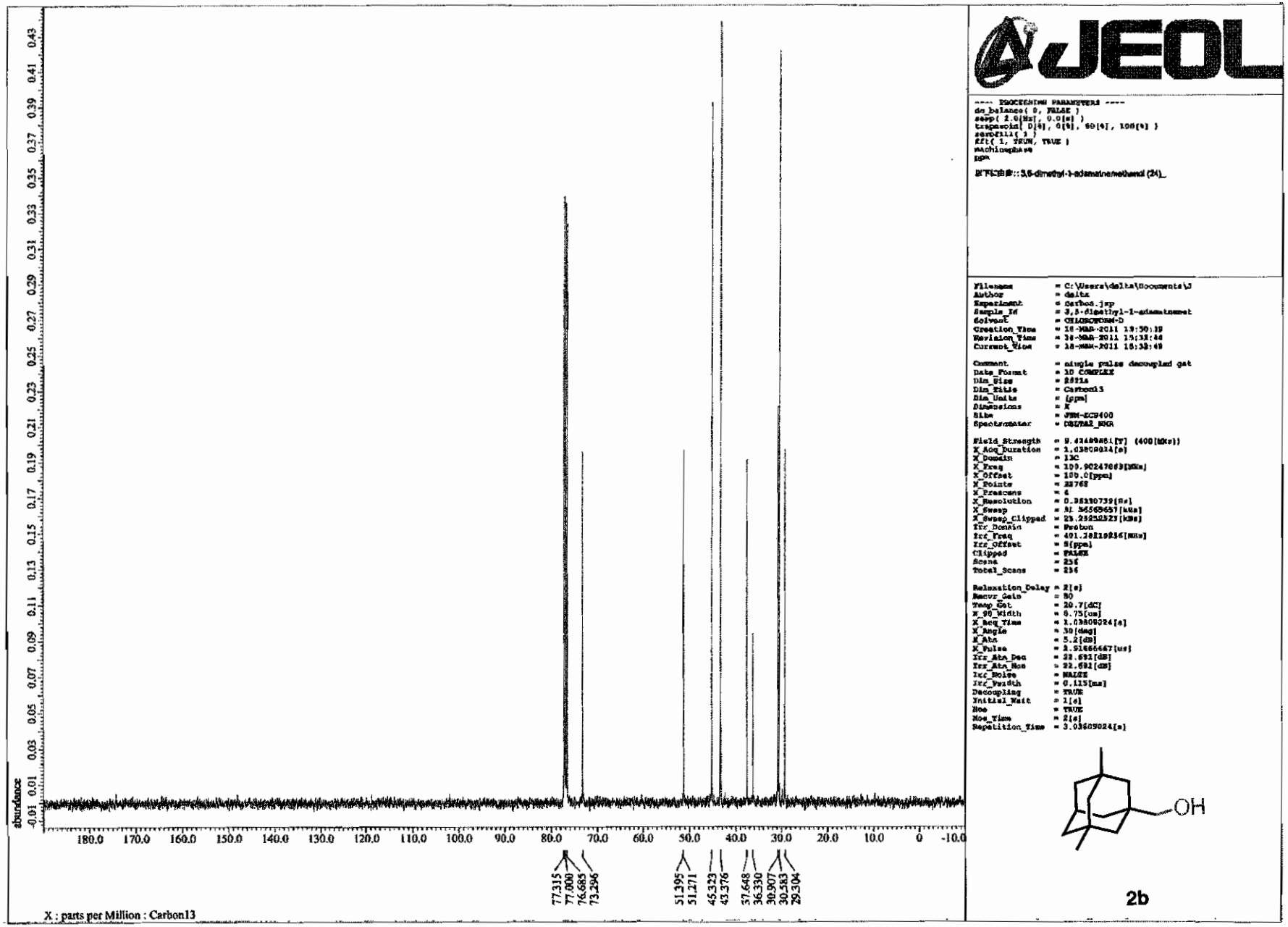
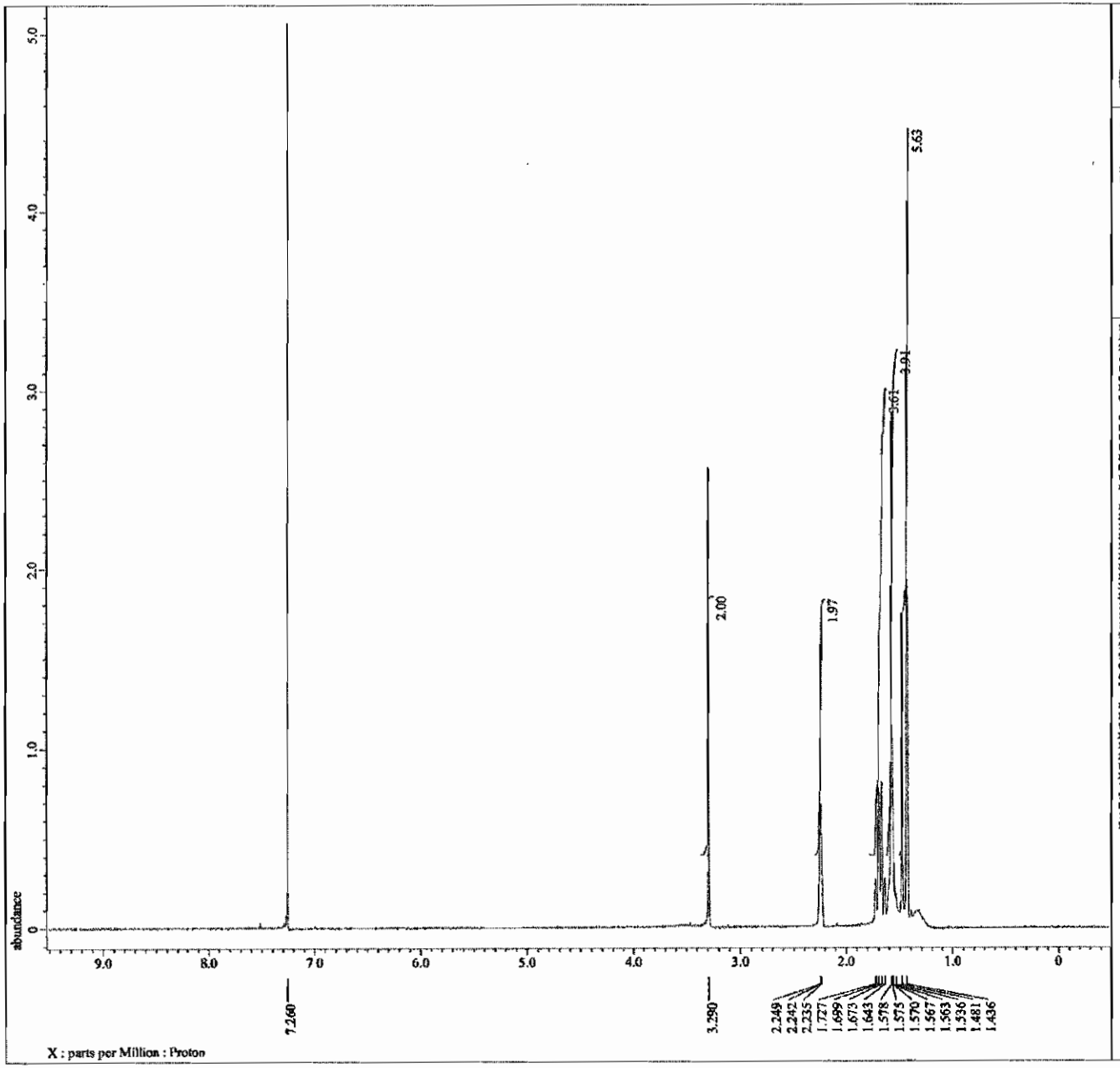
Irr_Offset = 6[ppm]
Tri_Domain = Proton
Tri_Freq = 401.2821856[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 42
Temp_Get = 19.6[dC]
X_90_Width = 9.25[us]
X_Acq_Time = 2.1757952[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 4.625[us]
Irr_Mode = Off
Tri_Mode = Off
Delta_Preset = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.1757952[s]
OH
2b

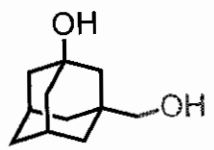

JEOL
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
expp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 50[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = C:\Users\delta\Documents\J
Author = delta
Experiment = carbon.jxp
Sample_Id = 3,5-dimethyl-1-adamantanmet
Solvent = CHLOROFORM-D
Creation_Time = 18-MAR-2011 13:50:19
Revision_Time = 18-MAR-2011 15:32:44
Current_Time = 18-MAR-2011 15:32:49
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 25714
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JEM-ECP400
Spectrometer = DELTA2_NMR
Field_Strength = 9.424899861[T] (400[MHz])
X_Acq_Duration = 1.038809024[s]
X_Domain = 13C
X_Freq = 100.9024789[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.96569657[kHz]
X_Sweep_Clipped = 25.58252529[kHz]
Irr_Domain = Proton
Irr_Freq = 401.2819236[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 20.7[dC]
X_90_Width = 6.75[us]
X_Acq_Time = 1.038809024[s]
X_Angle = 30[deg]
X_Atn = 5.2[dB]
X_Pulse = 2.51666667[us]
Irr_Atn_Dec = 22.69[dB]
Irr_Atn_Noe = 22.69[dB]
Irr_Noise = NAIZE
Irr_Width = 0.125[ms]
Decoupling = TRUE
Initial_Wait = 2[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.038809024[s]
OH
2b
abundance
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
77.315
77.000
76.685
73.296
51.395
51.271
45.323
43.376
37.648
36.330
30.907
30.583
29.304
X : parts per Million : Carbon13



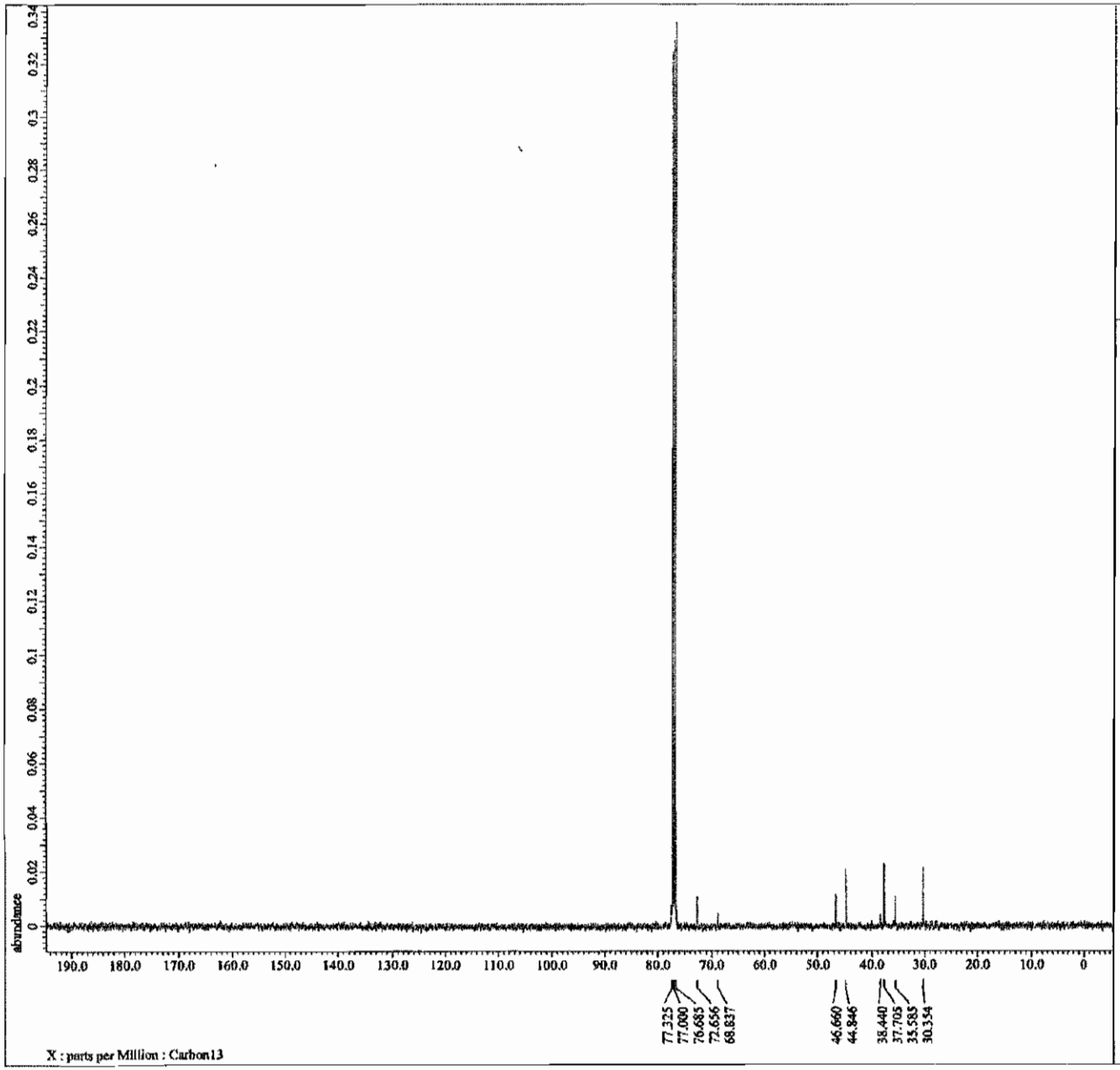
JEOL

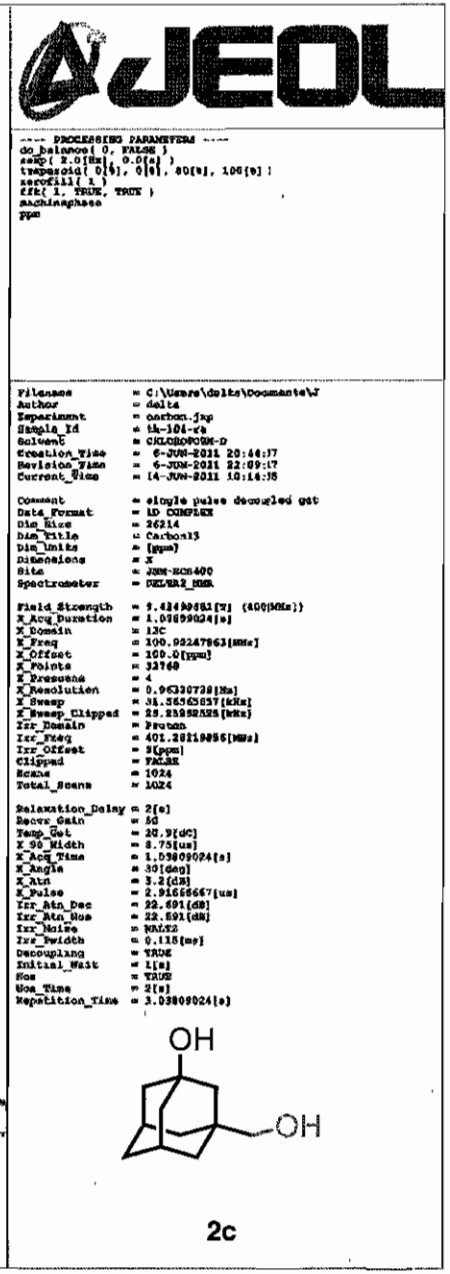
```
----- PROCESSING PARAMETERS -----
dc_balance( 0, FALSE )
expo( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

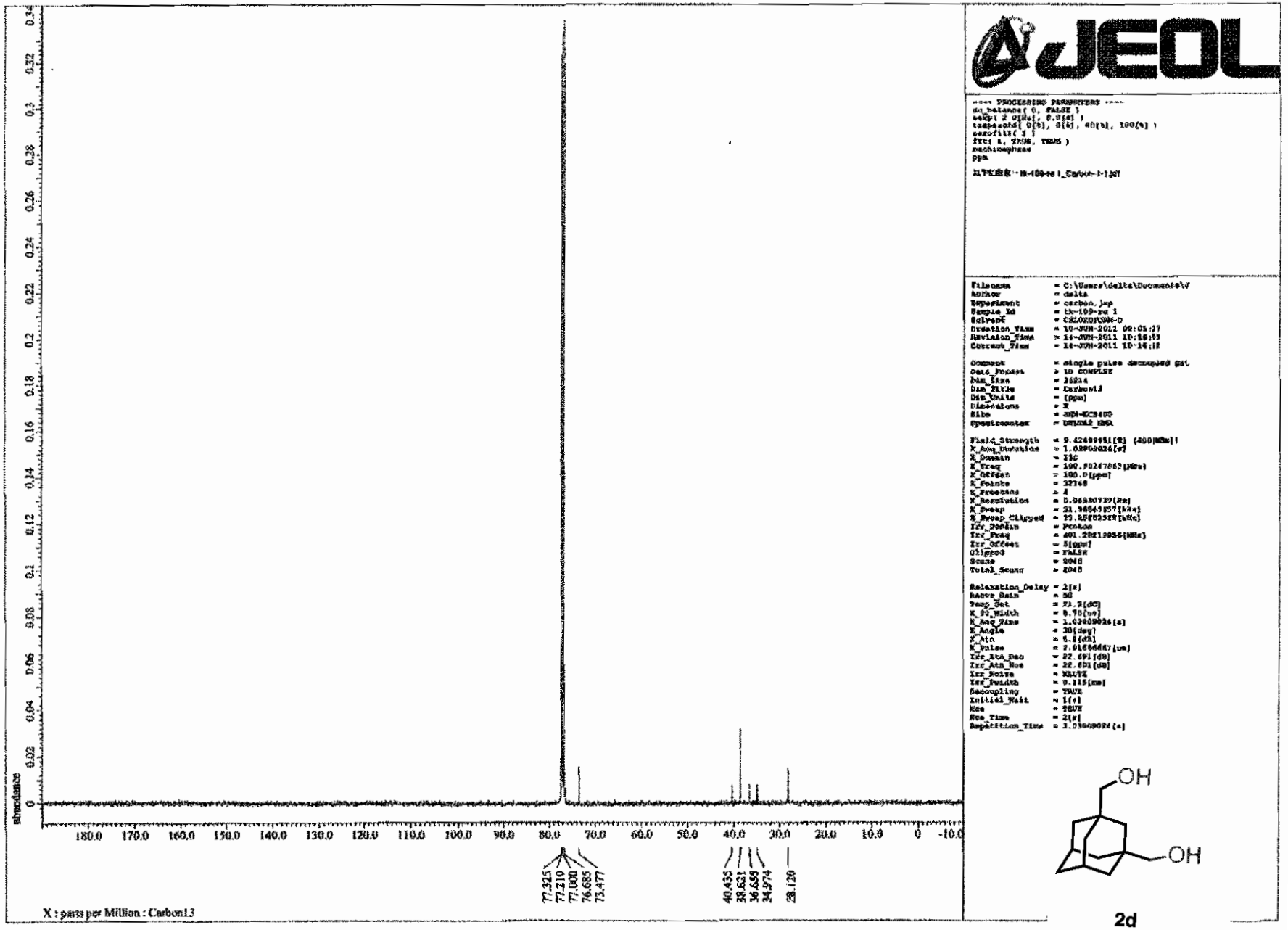
Filename	= C:\Users\delta\Documents\d
Author	= delta
Experiment	= proton.jxp
Sample_Id	= tk-104-ac
Solvent	= CHLOROFORM-D
Creation_Time	= 6-JUN-2011 20:42:5
Revision_Time	= 6-JUN-2011 20:47:1
Current_Time	= 14-JUN-2011 10:15:1
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.42499631[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_Prescans	= 1
X_Resolution	= 0.45960208[Hz]
X_Sweep	= 7.53012348[kHz]
X_Sweep_Clipped	= 6.02409639[kHz]
1fr_Domain	= Proton
1fr_Freq	= 401.28219856[MHz]
1fr_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]
Recve_Gain	= 56
Temp_Set	= 20.0[deg]
X_90_Width	= 9.25[us]
X_Acq_Time	= 2.1757952[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 4.625[us]
1fr_Mode	= Off
Tri_Mode	= Off
Data_Format	= DA1-8E
Initial_Wait	= 1[s]
Repetition_Time	= 7.1757952[s]

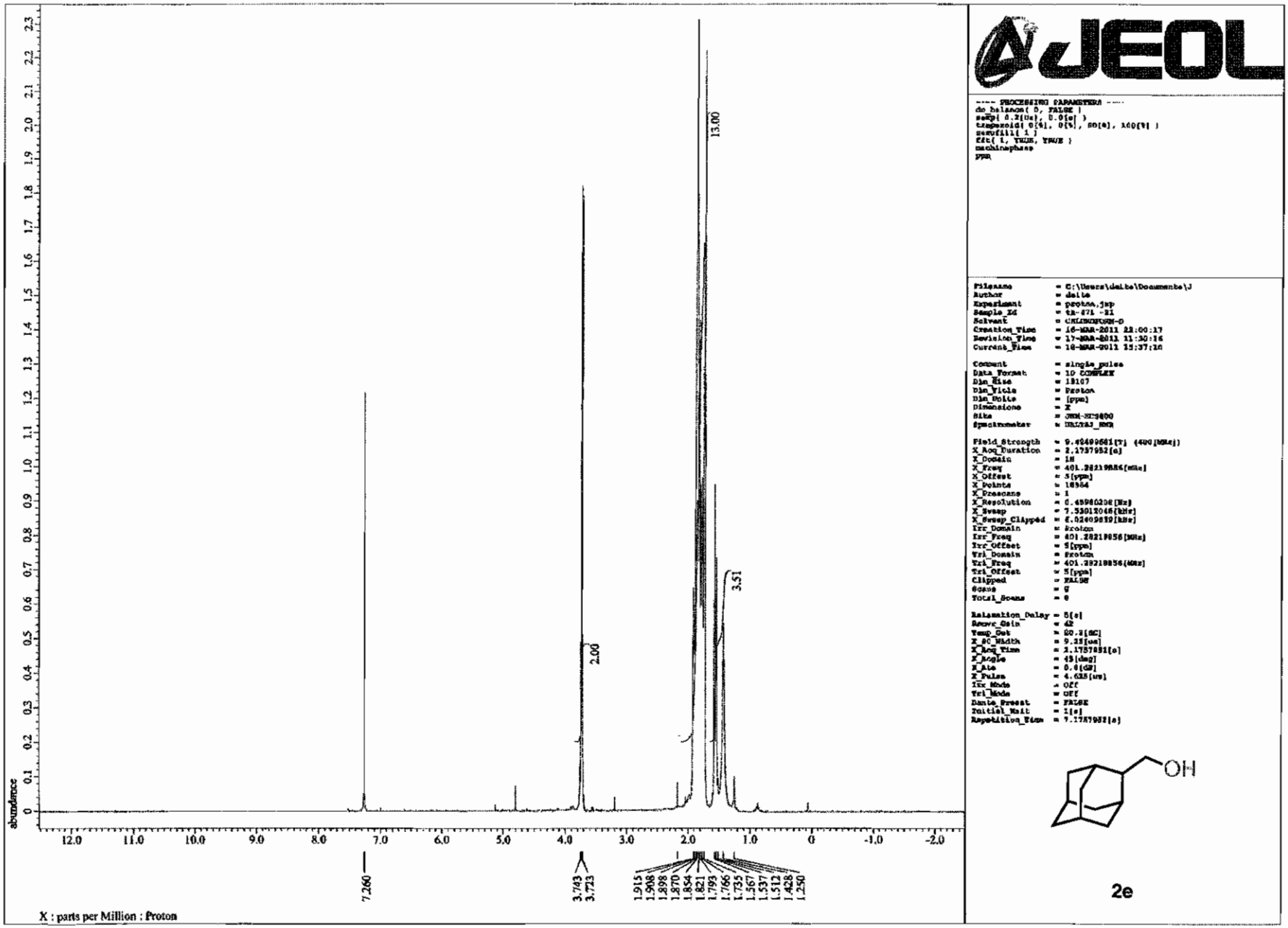


2c


abundance
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
77.325
77.000
76.685
72.656
68.837
46.660
44.846
38.440
37.705
35.585
30.354
X : parts per Million : Carbon13


JEOL
--- PROCESSING PARAMETERS ---
do_baseno(0, FALSE)
setp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
sachimaphase
ppm
Filename = C:\Users\delta\Documents\J
Author = delta
Experiment = carbon.jxp
Sample_Id = th-101-ra
Solvent = CHLOROFORM-D
Creation_Time = 6-JUN-2011 20:44:37
Revision_Time = 6-JUN-2011 22:09:17
Current_Time = 14-JUN-2011 10:14:35
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.4149961[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.9324786[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330738[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Freq = 401.28219856[MHz]
Irr_Offset = 3[ppm]
Clipped = FALSE
Scans = 1024
Total_Scans = 1024
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 20.9[dC]
X_90_Width = 8.75[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.2[dB]
X_Pulse = 2.91666667[us]
Irr_Atn_Dec = 22.69[dB]
Irr_Atn_Noe = 22.69[dB]
Irr_Noise = WALTZ
Irr_Width = 0.118[ms]
Decouplang = TRUE
Initial_Wait = 1[s]
Noe = TAUE
Noe_Time = 2[s]
Repetition_Time = 3.03809024[s]
OH
OH
2c


JEOL
abundance
X : parts per Million : Carbon13
77.325
77.210
77.000
76.685
73.477
40.435
38.631
36.655
34.974
28.120
OH
OH
2d

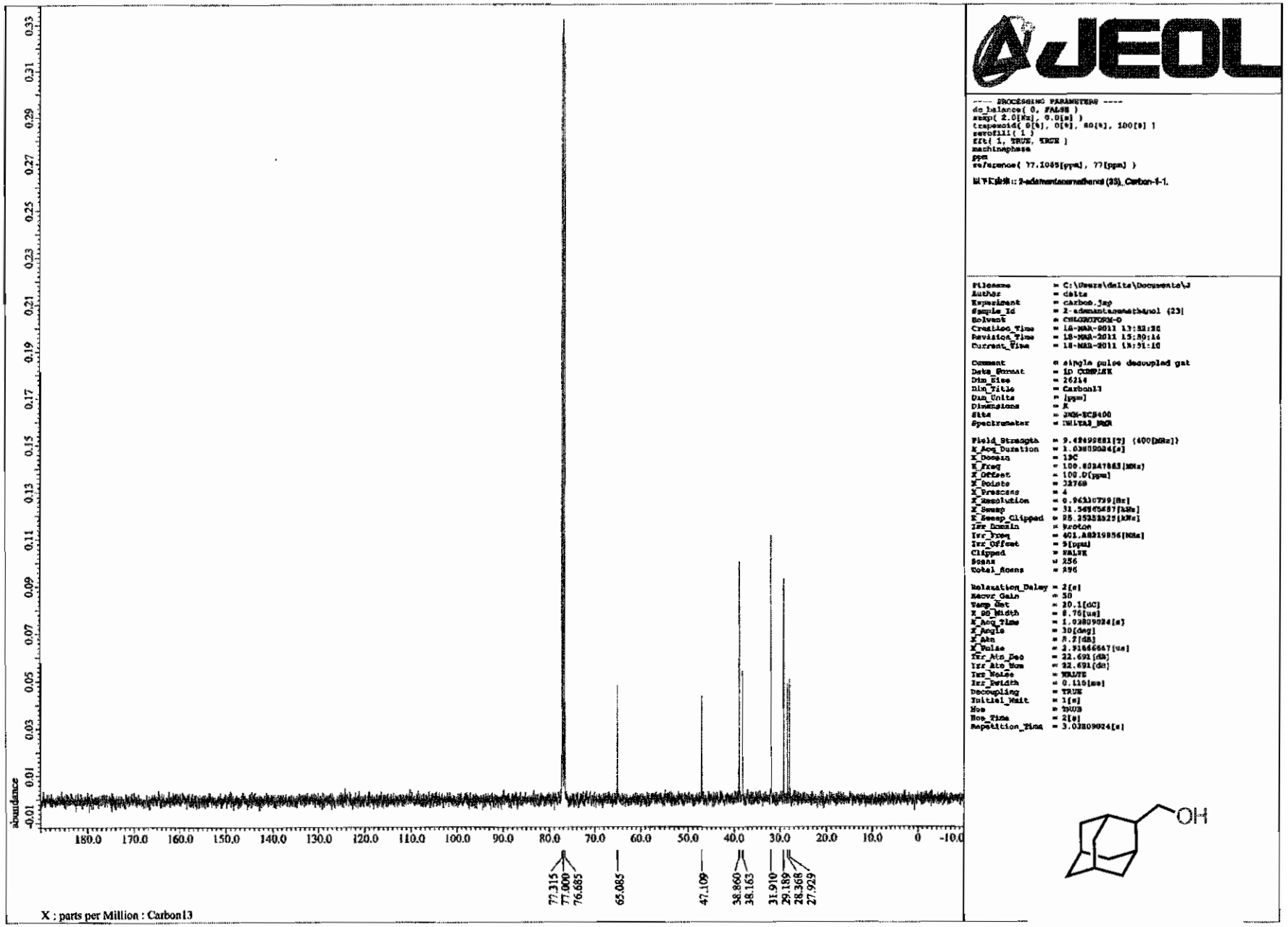


JEOL

```
---- PROCESSING PARAMETERS ----
do_balance( 0, FALSE )
setP( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[s], 160[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
PPR
```

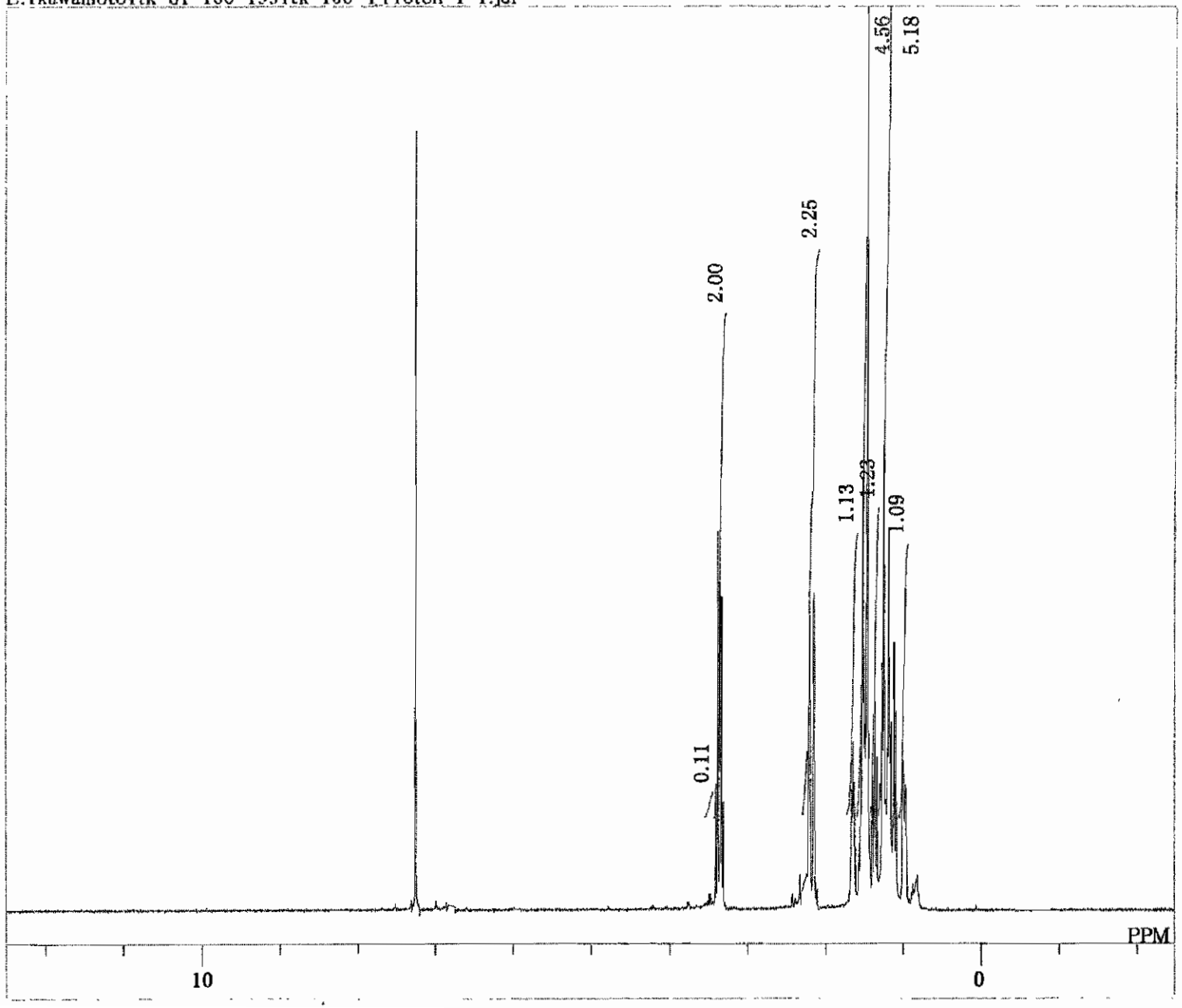
```
Filename          = C:\Users\delbe\Documents\J
Author            = delbe
Experiment        = proton.jxp
Sample_Id         = th-471 -21
Solvent           = CHLOROFORM-D
Creation_Time     = 16-MAR-2011 21:00:17
Revision_Time     = 17-MAR-2011 11:30:16
Current_Time      = 18-MAR-2011 15:37:16

Content           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 18107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = MAGRAG_NMR

Field_Strength    = 9.40490681[T] (400[MHz])
X_Acq_Duration    = 2.1737952[s]
X_Domain          = 1H
X_Freq            = 401.28219856[MHz]
X_Offset          = 5[ppm]
X_Points          = 18584
X_Prescans        = 1
X_Resolution      = 0.45986208[Hz]
X_Sweep           = 7.53012048[kHz]
X_Sweep_Clipped   = 6.02409639[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
dscans            = 0
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 42
Temp_Set          = 60.0[degC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 2.1737952[s]
X_Angle           = 45[deg]
X_Atn             = 0.0[dB]
X_Pulse           = 4.625[us]
Irr_Mode          = Off
Tri_Mode          = Off
Decpl_Preset      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.1737952[s]
```


2e

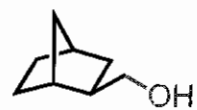

JEOL
---- PROCESSING PARAMETERS ----
do_balance(0, FALSE)
expo(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
reference(77.1045[ppm], 77[ppm])
Filename = C:\Users\delta\Documents\J
Author = delta
Experiment = carbon.jxp
Sample_Id = 2-adamantanemethanol (23)
Solvent = CHLOROFORM-D
Creation_Time = 16-MAR-2011 13:32:36
Revision_Time = 16-MAR-2011 15:50:14
Current_Time = 16-MAR-2011 15:51:10
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.4899881[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.6024785[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96310799[Hz]
X_Sweep = 31.5495487[kHz]
X_Sweep_Clipped = 25.2525825[kHz]
Irr_Domain = Proton
Irr_Freq = 401.8819856[MHz]
Irr_Offset = 5[ppm]
Clipped = VALUE
Scans = 256
Total_Scans = 896
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Set = 20.1[dC]
X_90_Width = 8.75[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 8.7[dB]
X_Pulse = 2.91666667[us]
Irr_Atn_Dec = 22.69[dB]
Irr_Atn_Hom = 22.69[dB]
Irr_Mode = WALTZ
Irr_Width = 0.116[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Mom = NONE
Mom_Time = 2[s]
Repetition_Time = 3.03809024[s]
abundance
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
77.315
77.000
76.685
65.085
47.109
38.860
38.163
31.910
29.189
28.368
27.929
X : parts per Million : Carbon13
OH
2e

single_pulse

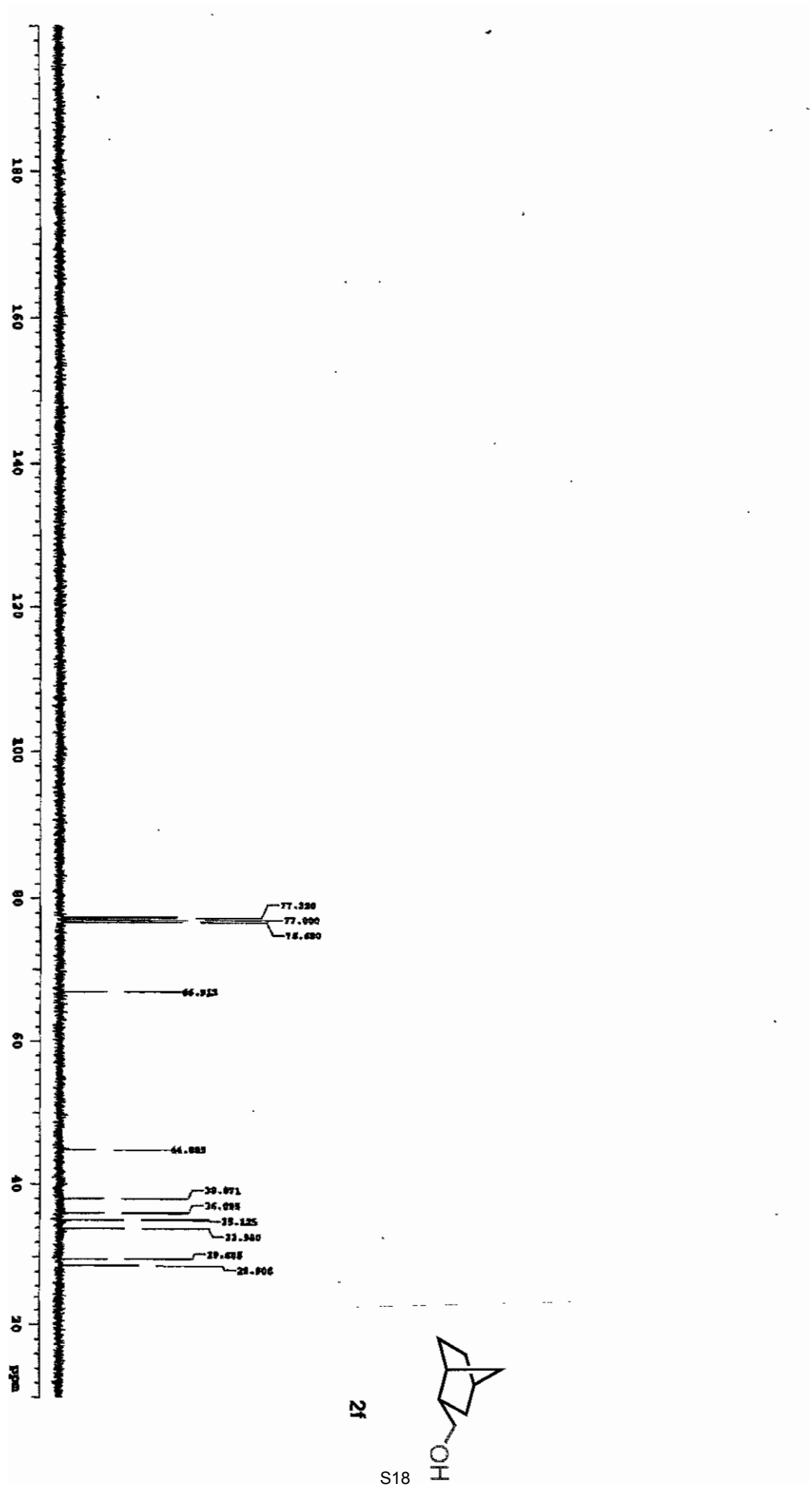
E:¥kawamoto¥tk-d1-100-199¥tk-100-4_Proton-1-1.jdf



DFILE	tk-100-4_Proton-1-1.jdf
COMNT	single_pulse
DATIM	2011-05-31 15:28:22
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	401.28 MHz
OBSET	4.20 KHz
OBFIN	4.97 Hz
POINT	16384
FREQU	7530.12 Hz
SCANS	8
ACQTM	2.1758 sec
PD	5.0000 sec
PW1	4.62 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	46

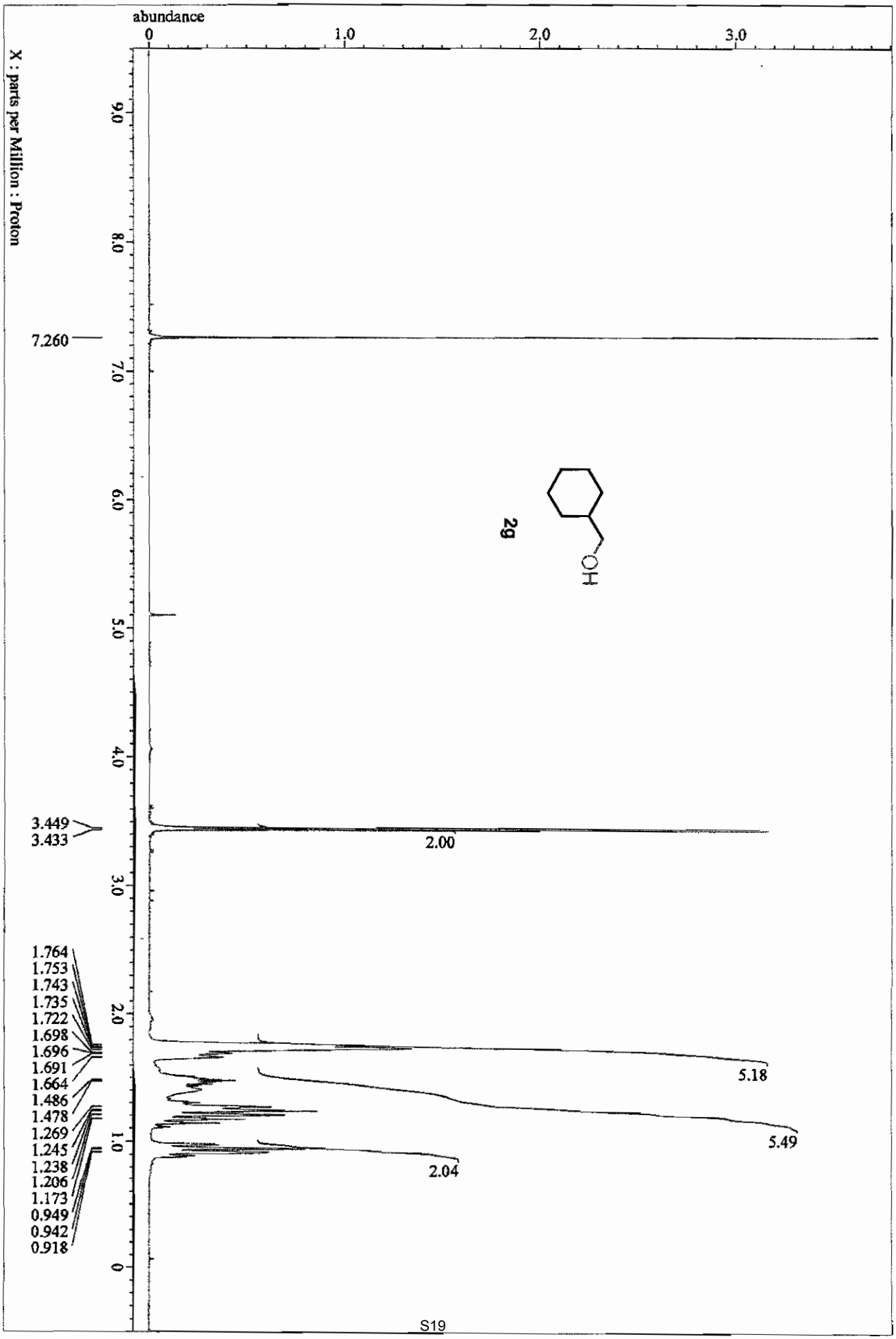


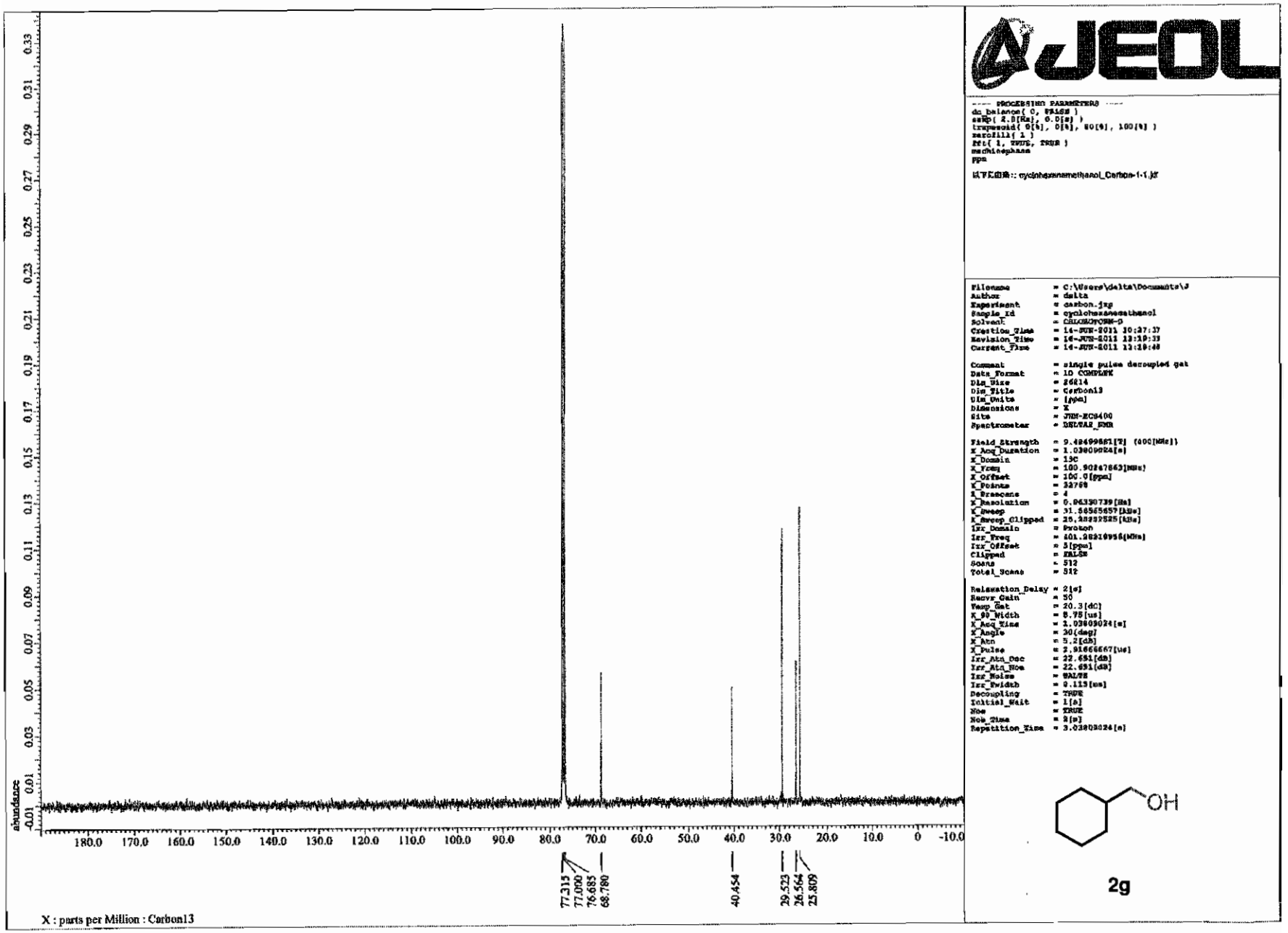
2f

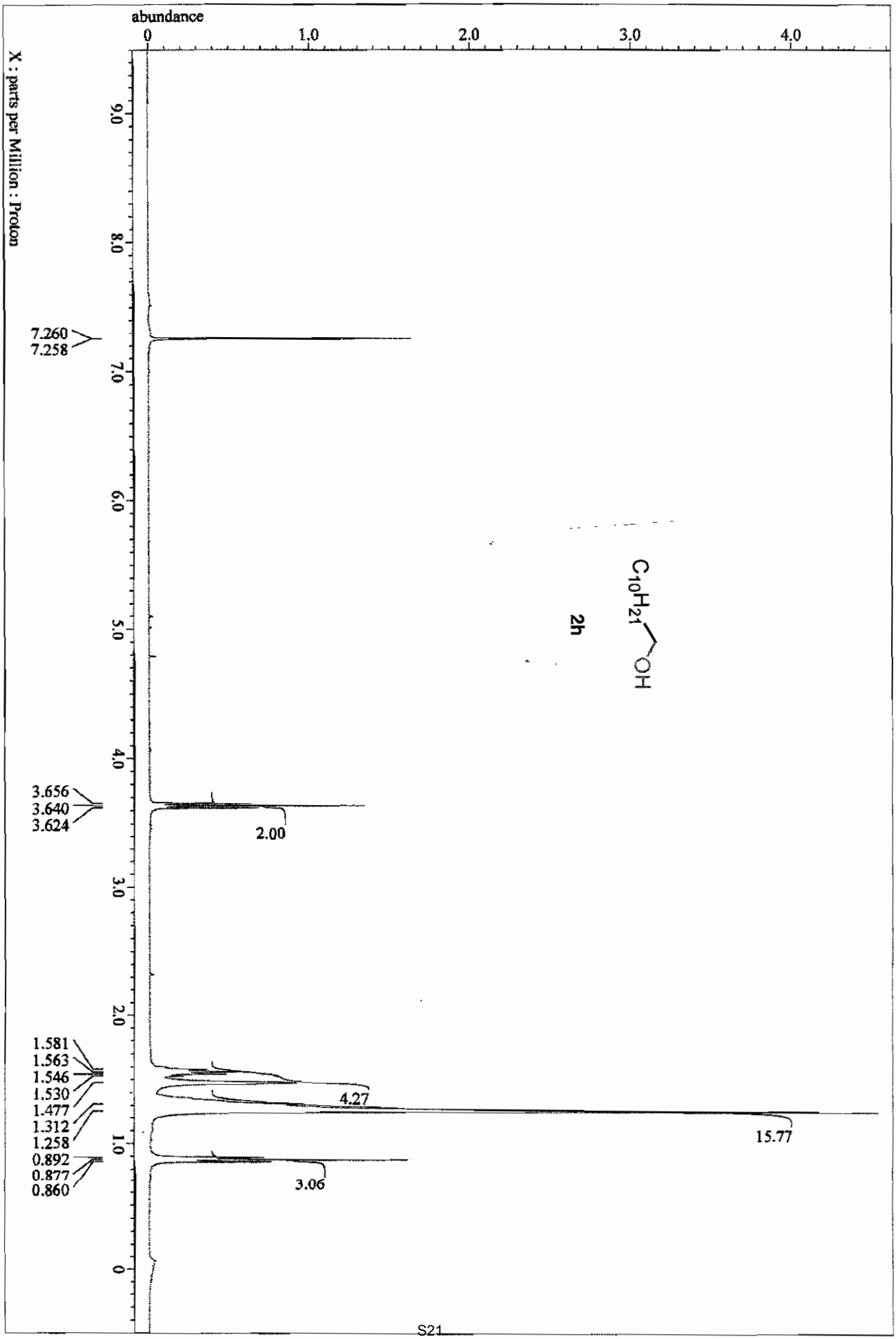

180
160
140
120
100
80
60
40
20
ppm
77.320
77.000
76.680
66.953
44.883
39.071
36.095
35.125
33.980
29.695
28.906

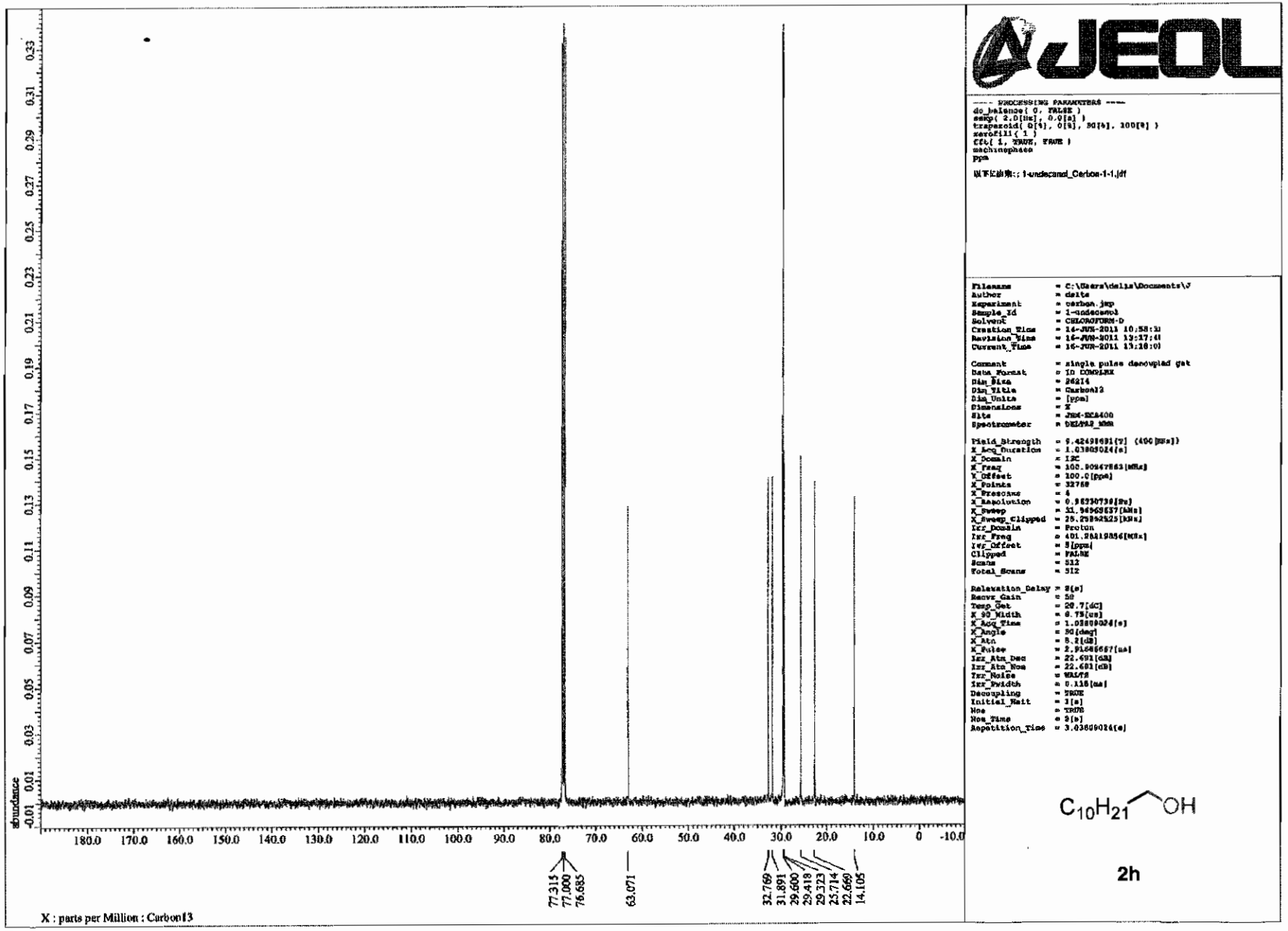
2f

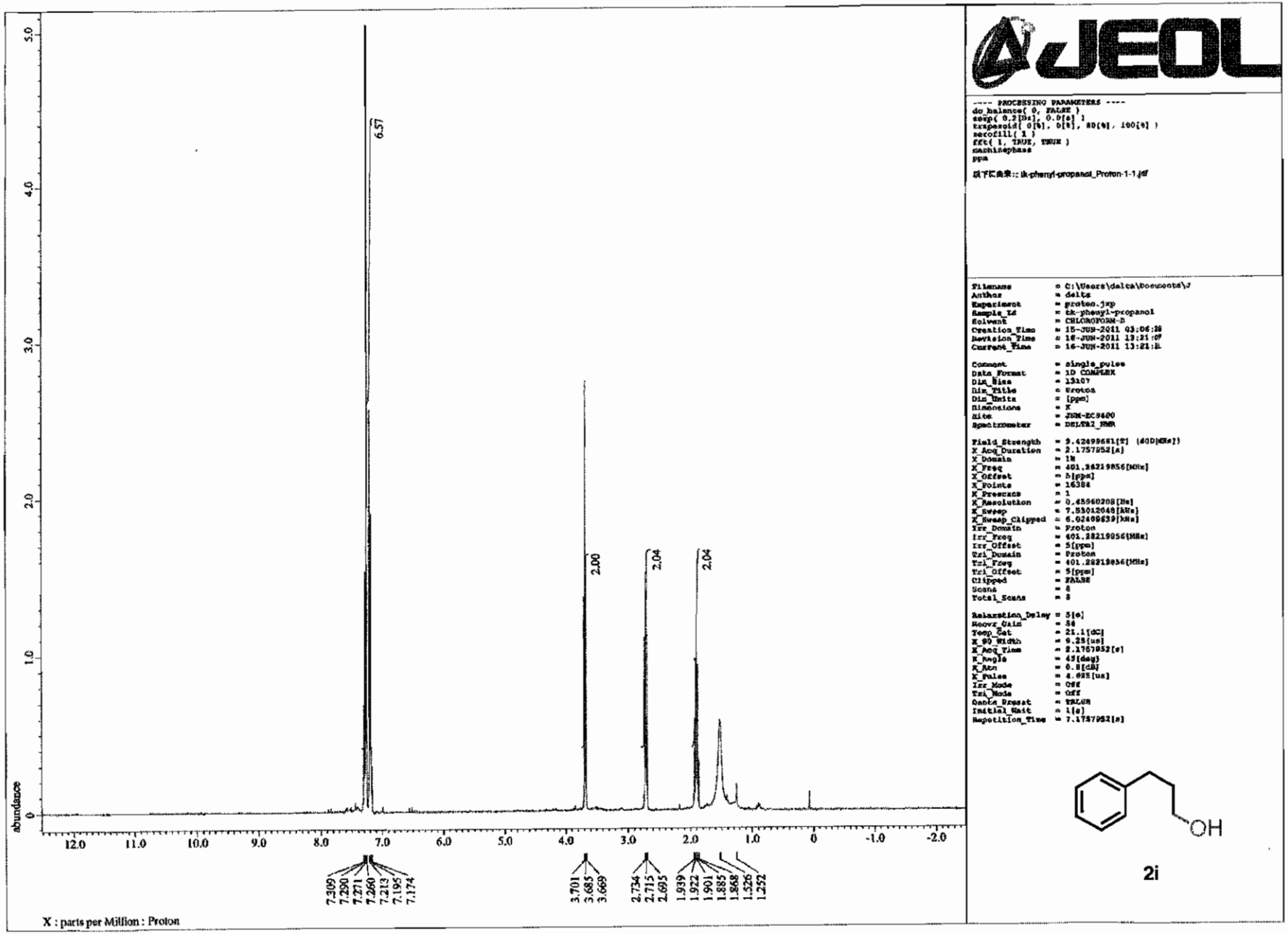
OH

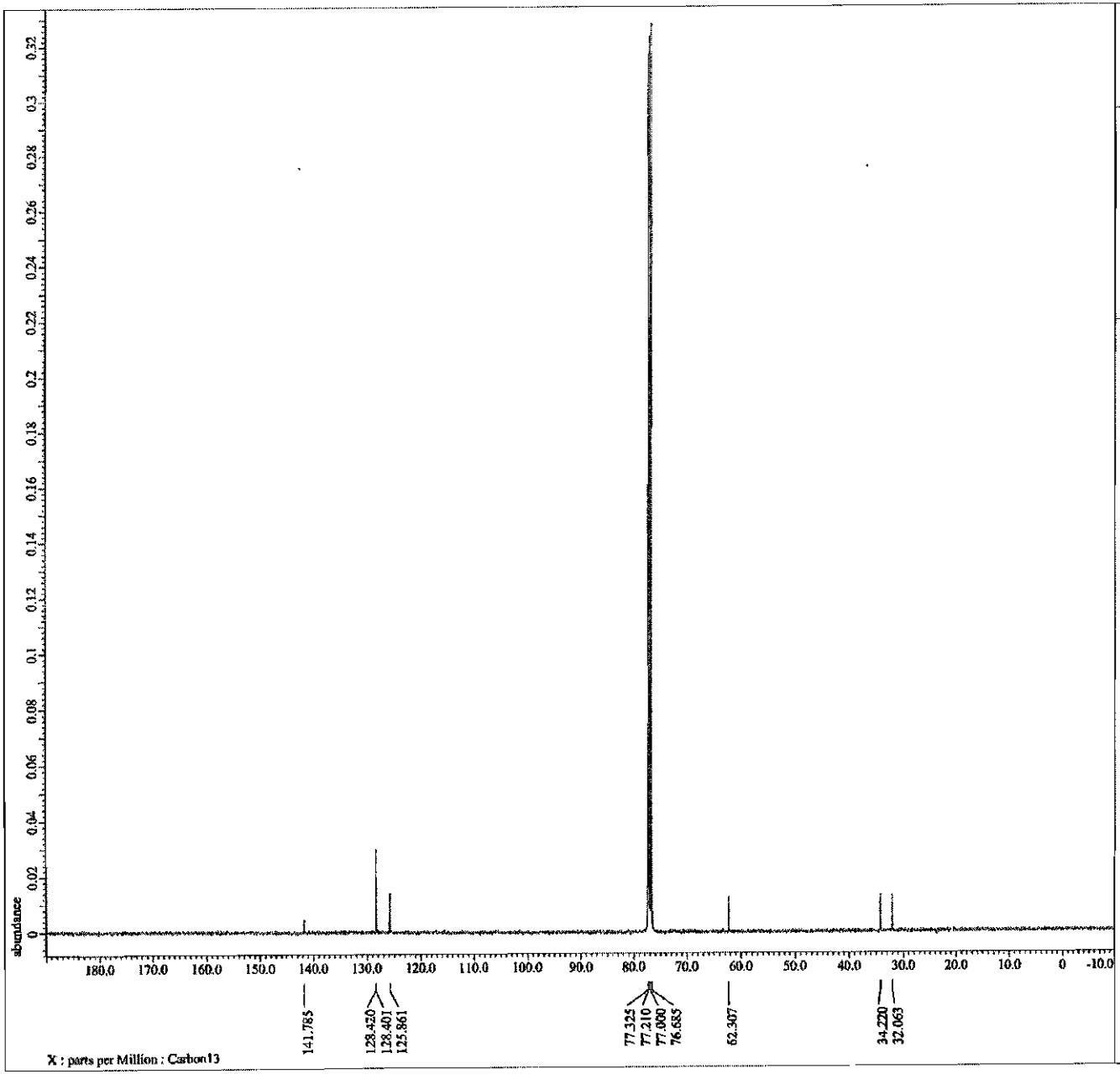

abundance
0
1.0
2.0
3.0
X : parts per Million : Proton
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.260
3.449
3.433
1.764
1.753
1.743
1.735
1.722
1.698
1.696
1.691
1.664
1.486
1.478
1.269
1.245
1.238
1.206
1.173
0.949
0.942
0.918
2g
OH
2.00
5.18
5.49
2.04

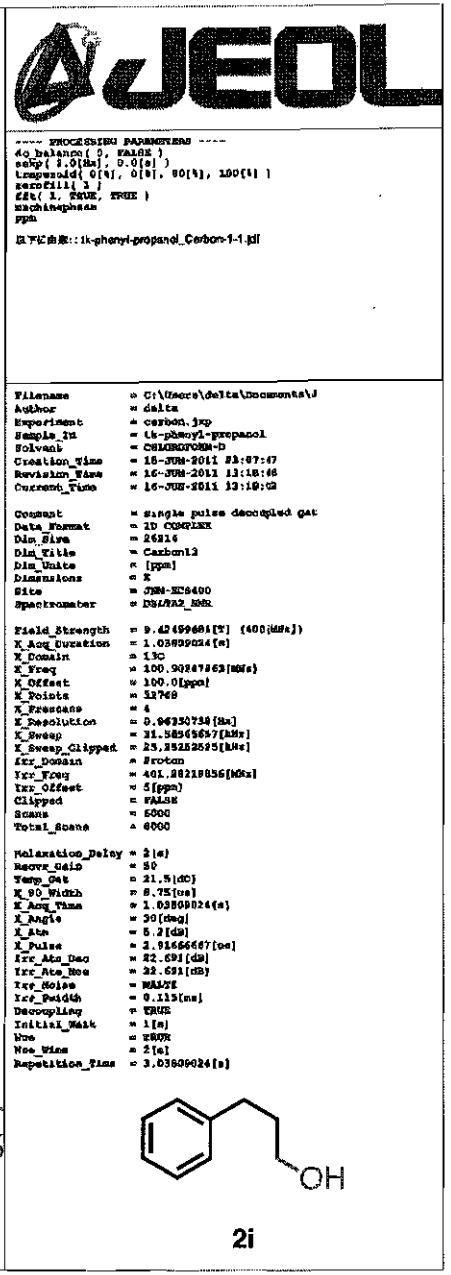

JEOL
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
asRp(2.8[Hz], 6.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zeroFill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
cyclohexanemethanol_Carbon-1-1.jd
Filename = C:\Users\delta\Documents\J
Author = delta
Experiment = carbon.jxp
Sample_Id = cyclohexanemethanol
Solvent = CHLOROFORM-D
Creation_Time = 14-JUN-2011 10:37:37
Revision_Time = 14-JUN-2011 13:19:33
Current_Time = 14-JUN-2011 13:19:48
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 65614
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-EC5400
Spectrometer = DELTA2_NMR
Field_Strength = 9.48499881[T] (400[MHz])
X_Acq_Duration = 1.03809824[s]
X_Domain = 13C
X_Freq = 100.90247843[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.23232525[kHz]
Irr_Domain = Proton
Irr_Freq = 401.28210956[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 512
Total_Scans = 512
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Set = 20.3[dC]
X_90_Width = 8.75[us]
X_Acq_Time = 1.03809824[s]
X_Angle = 30[deg]
X_Atn = 5.2[dB]
X_Pulse = 2.91666667[us]
Irr_Atn_Dec = 22.65[dB]
Irr_Atn_Noe = 22.65[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.03809824[s]
OH
2g
abundance
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
77.315
77.000
76.685
68.780
40.454
29.523
26.564
25.809
X : parts per Million : Carbon13

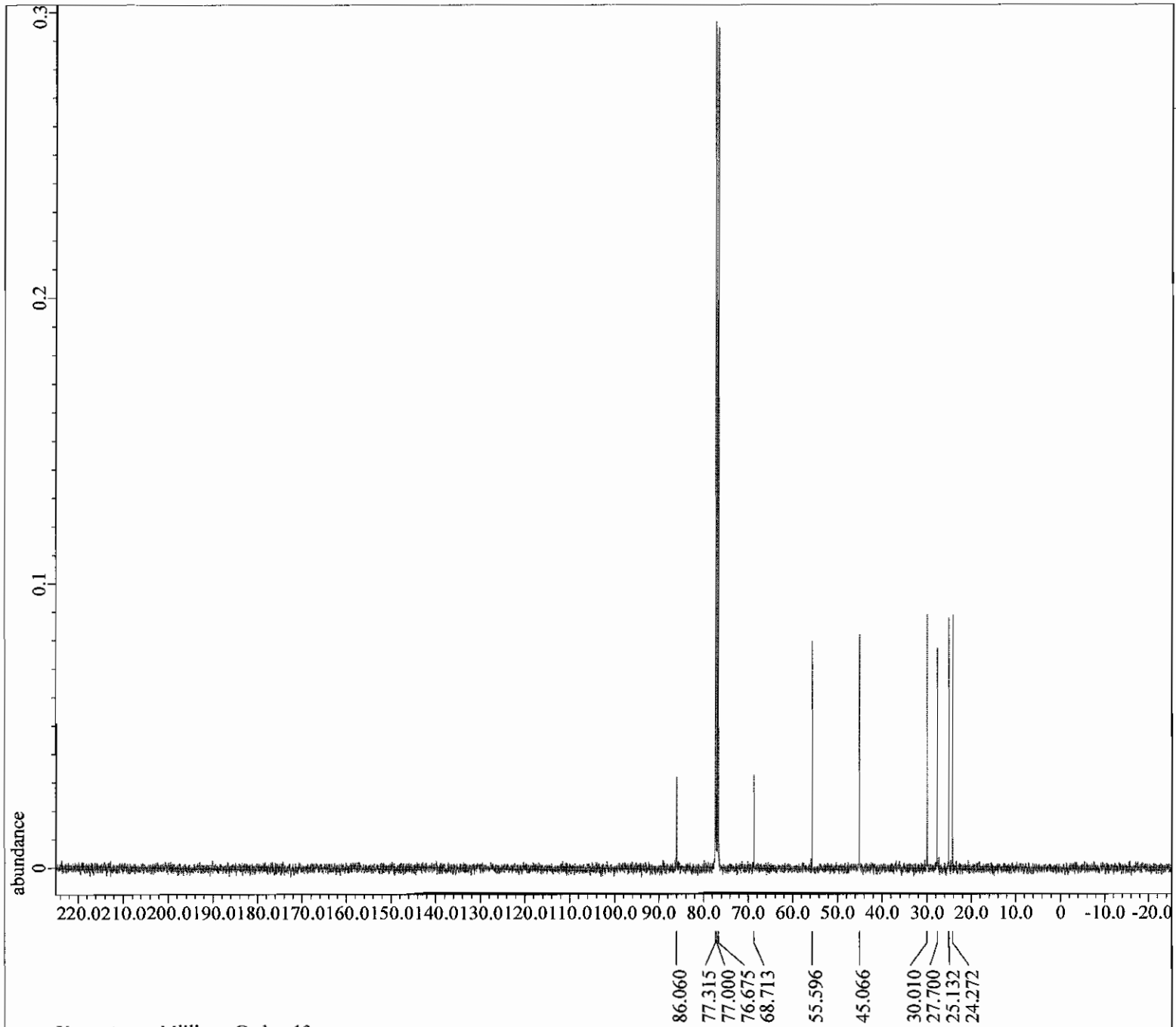

abundance
0
1.0
2.0
3.0
4.0
X : parts per Million : Proton
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.260
7.258
3.656
3.640
3.624
1.581
1.563
1.546
1.530
1.477
1.312
1.258
0.892
0.877
0.860
2.00
4.27
15.77
3.06
C10H21
OH
2h


JEOL
---- PROCESSING PARAMETERS ----
do_baseline(0, FALSE)
emhp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 90[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に結果:; 1-undecanol_Carbon-1-1.jdf
Filename = C:\Users\delia\Documents\J
Author = delia
Experiment = carbon.jxp
Sample_Id = 1-undecanol
Solvent = CHLOROFORM-D
Creation_Time = 14-JUN-2011 10:58:31
Revision_Time = 16-JUN-2011 13:17:41
Current_Time = 16-JUN-2011 13:18:03
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA400
Spectrometer = DELTA2_NMR
Field_Strength = 9.42491691[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.90947863[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330736[Hz]
X_Sweep = 31.94969637[kHz]
X_Sweep_Clipped = 25.75892525[kHz]
Irr_Domain = Proton
Irr_Freq = 401.25819856[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 512
Total_Scans = 512
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 20.7[dC]
X_90_Width = 8.75[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 90[deg]
X_Atn = 5.2[dB]
X_Pulse = 2.91666667[us]
Irr_Atn_Dec = 22.60[dB]
Irr_Atn_Noe = 22.60[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.116[ms]
Decoupling = TRUE
Initial_Wait = 2[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.03809024[s]
C10H21OH
2h
abundance
X : parts per Million : Carbon13
77.315
77.000
76.685
63.071
32.769
31.891
29.600
29.418
29.323
25.714
22.669
14.105


6.57
2.00
2.04
2.04
abundance
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
7.309
7.290
7.271
7.260
7.213
7.195
7.174
3.701
3.685
3.669
2.734
2.715
2.695
1.939
1.922
1.901
1.885
1.868
1.526
1.252
X : parts per Million : Proton
JEOL
---- PROCESSING PARAMETERS ----
do_baiance(0, FALSE)
sesp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
manualphase
ppa
Filename = C:\Users\delta\Documents\J
Author = delta
Experiment = proton.jxp
Sample_Id = ch-phenyl-propanol
Solvent = CHLOROFORM-D
Creation_Time = 15-JUN-2011 03:06:28
Revision_Time = 16-JUN-2011 13:21:07
Current_Time = 16-JUN-2011 13:21:2
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-EC9400
Spectrometer = DELTA2_NMR
Field_Strength = 9.424988681[T] (400[MHz])
X_Acq_Duration = 2.1757952[s]
X_Domain = 1H
X_Freq = 401.38219856[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45960208[Hz]
X_Sweep = 7.53012048[kHz]
X_Sweep_Clipped = 6.02409639[kHz]
Irr_Domain = Proton
Irr_Freq = 401.38219856[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 401.38219856[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 54
Temp_Set = 21.1[dC]
X_90_Width = 9.25[us]
X_Acq_Time = 2.1757952[s]
X_Angle = 45[deg]
X_Atn = 0.5[dB]
X_Pulse = 4.62[us]
Irr_Mode = Off
Tri_Mode = Off
Decpl_Preset = VALUE
Initial_Wait = 1[s]
Repetition_Time = 7.1757952[s]
OH
2i

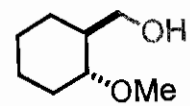

abundance
0.32
0.3
0.28
0.26
0.24
0.22
0.2
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
141.785
128.420
128.401
125.861
77.325
77.210
77.000
76.685
62.307
34.220
32.063
X : parts per Million : Carbon13


JEOL
---- PROCESSING PARAMETERS ----
do_baseline(0, FALSE)
setup(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(2)
fft(1, TRUE, TRUE)
Machinephase
ppm
Filename = C:\Users\delta\Documents\J
Author = delta
Experiment = carbon.jxp
Sample_Id = 1k-phenyl-propanol
Solvent = CHLOROFORM-D
Creation_Time = 16-JUN-2011 23:07:47
Revision_Time = 16-JUN-2011 13:18:46
Current_Time = 16-JUN-2011 13:19:02
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26316
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.42499681[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.90247363[MHz]
X_Offset = 100.0[ppm]
X_Points = 52768
X_Prescans = 4
X_Resolution = 0.96330738[Hz]
X_Sweep = 31.58965637[kHz]
X_Sweep_Clipped = 25.35252595[kHz]
Irr_Domain = Proton
Irr_Freq = 400.28219836[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 5000
Total_Scans = 6000
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21.5[dC]
X_90_Width = 8.75[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 5.2[dB]
X_Pulse = 2.91666667[us]
Irr_Atn_Dec = 22.69[dB]
Irr_Atn_Noe = 22.69[dB]
Irr_Noise = WALTZ
Irr_Width = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.03809024[s]
OH
2i

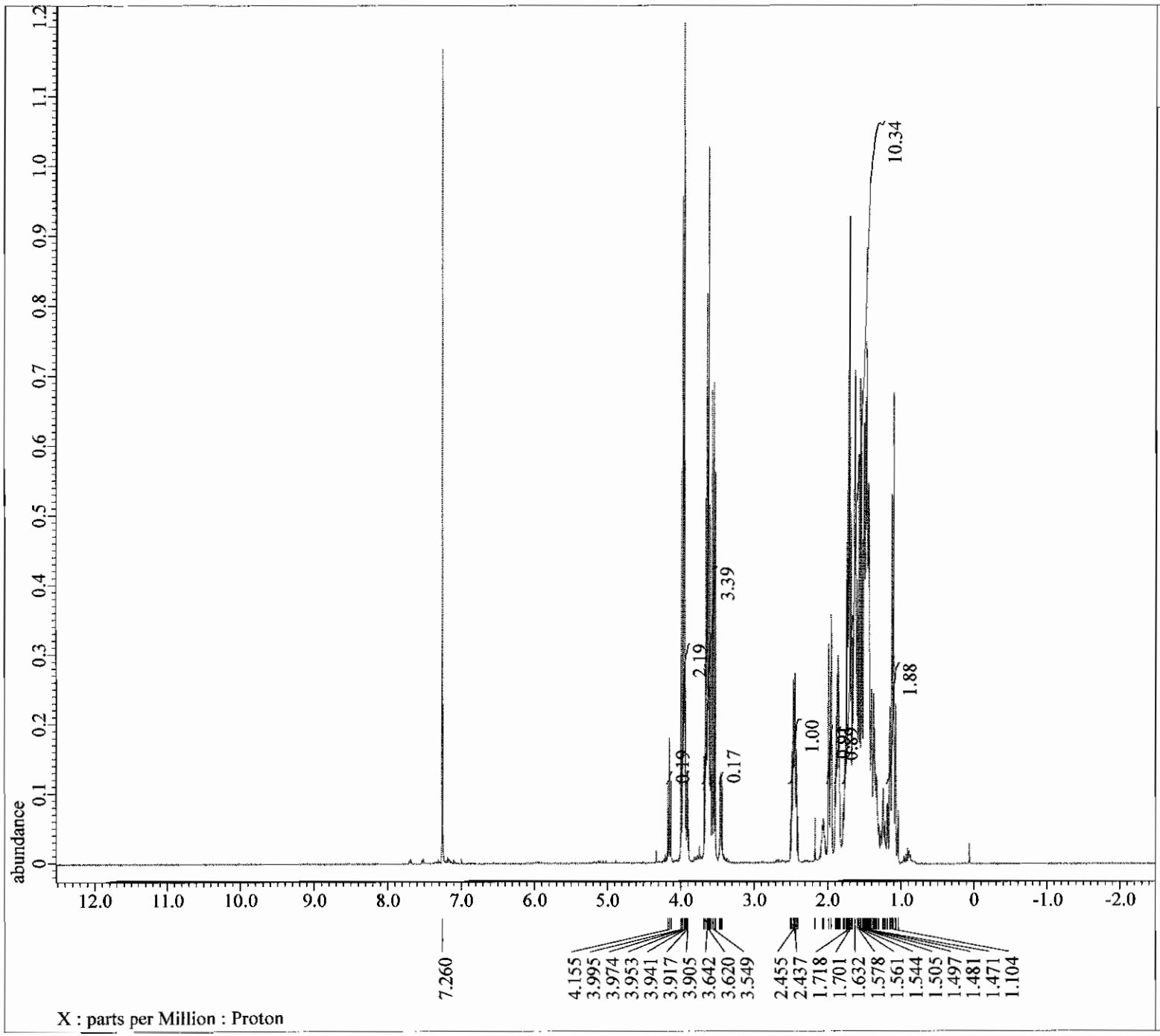


JEOL

Filename	= /Volumes/NO NAME/kawamoto/
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= tk-213-fr3
Solvent	= CHLOROFORM-D
Creation_Time	= 26-AUG-2011 13:00:20
Revision_Time	= 26-AUG-2011 16:40:31
Current_Time	= 31-OCT-2011 23:04:11
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
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	= 100.0[ppm]
X_Points	= 32768
X_Prescans	= 4
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	= FALSE
Scans	= 512
Total_Scans	= 512
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 21.9[dC]
X_90_Width	= 8.75[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_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.03809024[s]

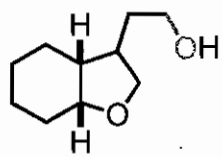


2j

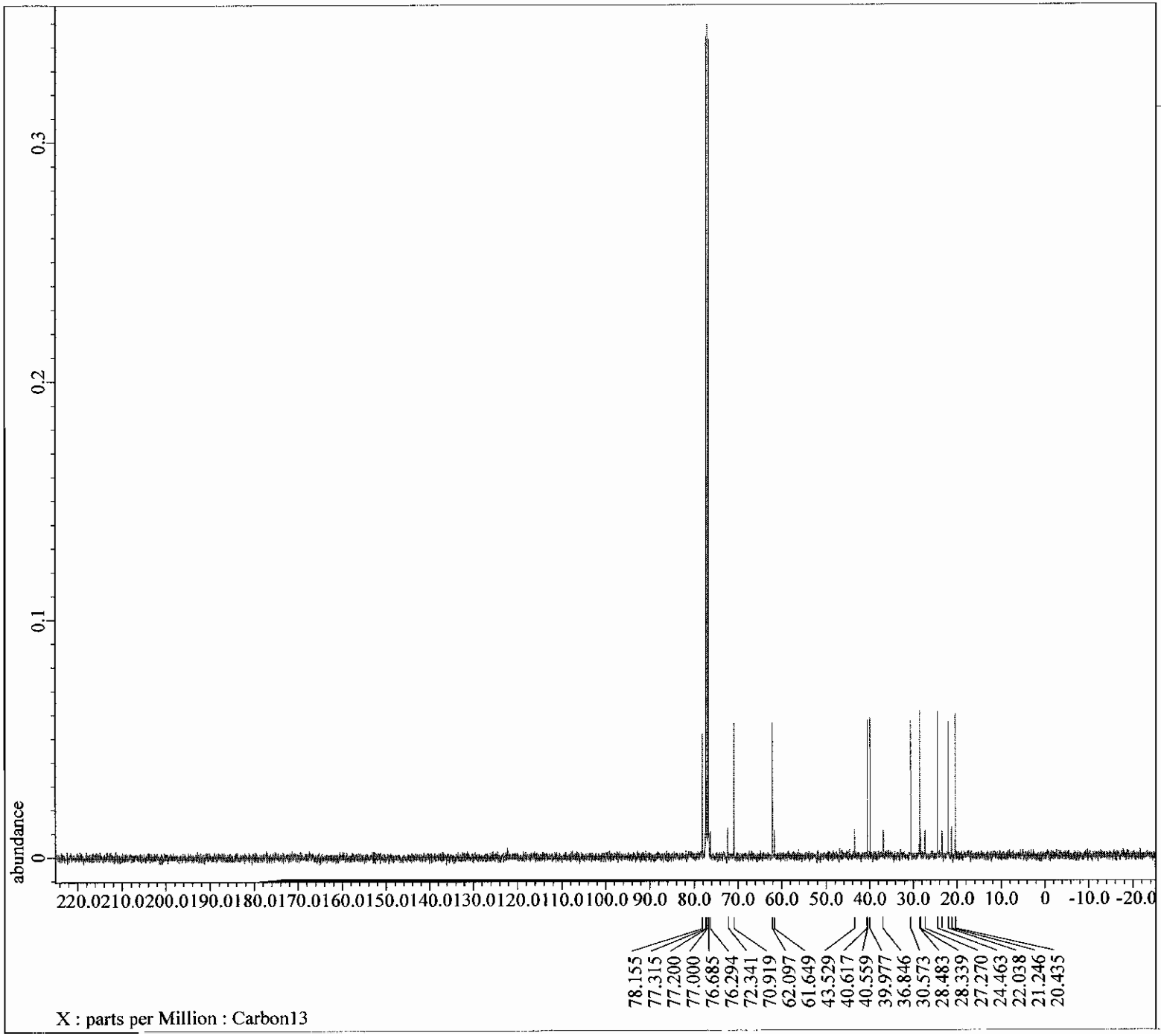


JEOL

Filename	= /Volumes/NO NAME/kawamoto/
Author	= delta
Experiment	= proton.jxp
Sample_Id	= tk-201-HPLC1
Solvent	= CHLOROFORM-D
Creation_Time	= 29-JUL-2011 22:37:51
Revision_Time	= 29-JUL-2011 22:45:31
Current_Time	= 31-OCT-2011 23:03:34
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Unita	= [ppm]
Dimensiona	= X
Site	= JNM-ECS400
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	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
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]
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	= 40
Temp_Get	= 21.4[dC]
X_90_Width	= 9.25[us]
X_Acq_Time	= 2.1757952[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 4.625[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.1757952[s]

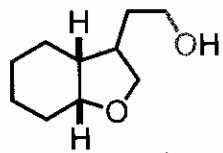


2k

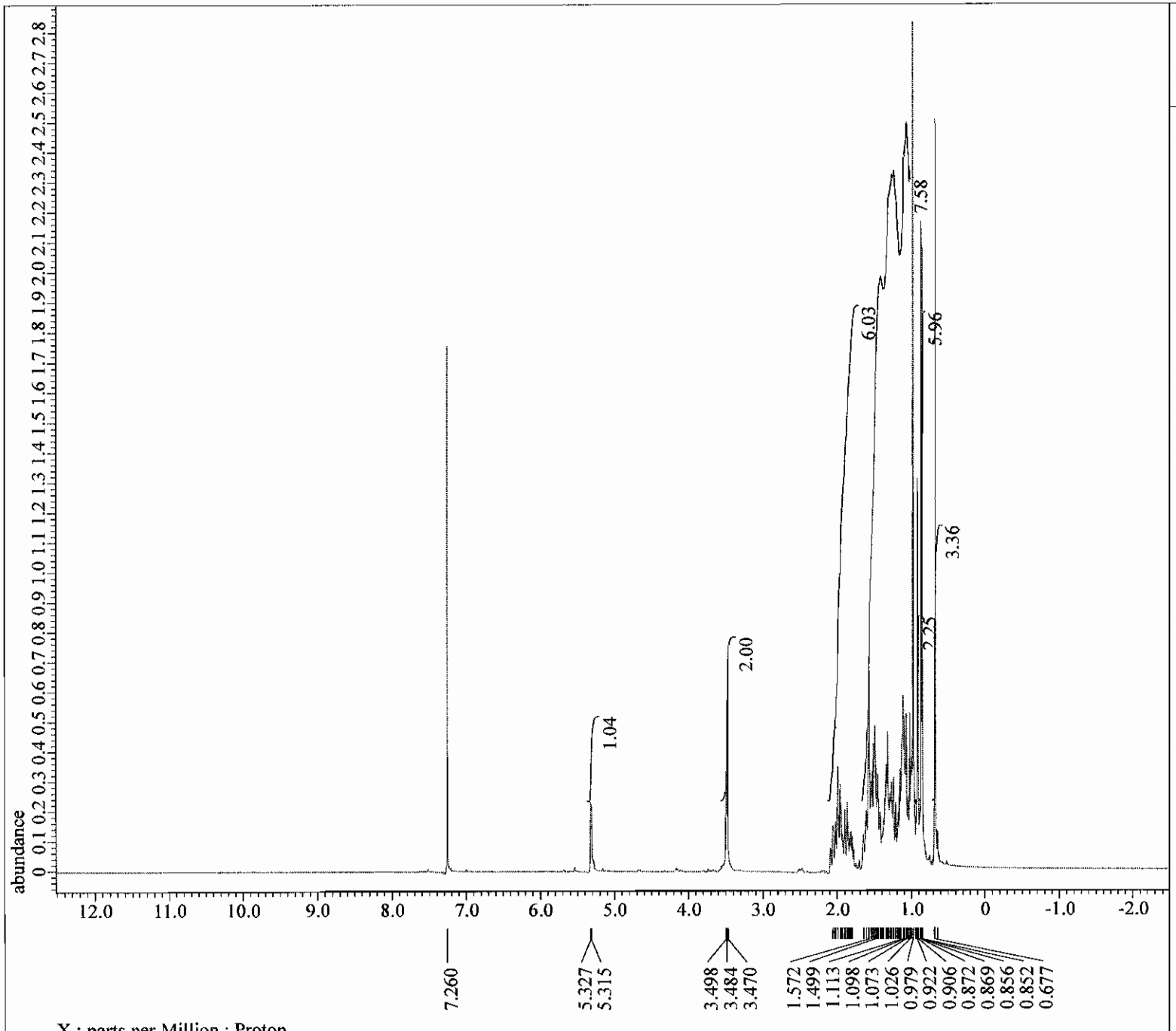


JEOL

Filename	= /Volumes/NO NAME/kawamoto/
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= tk-201-HPLC1
Solvent	= CHLOROFORM-D
Creation_Time	= 1-AUG-2011 16:04:00
Revision_Time	= 31-OCT-2011 23:03:08
Current_Time	= 31-OCT-2011 23:03:20
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
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	= 100.0[ppm]
X_Points	= 32768
X_Prescans	= 4
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	= FALSE
Scans	= 512
Total_Scans	= 512
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 22.5[dC]
X_90_Width	= 8.75[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_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.03809024[s]

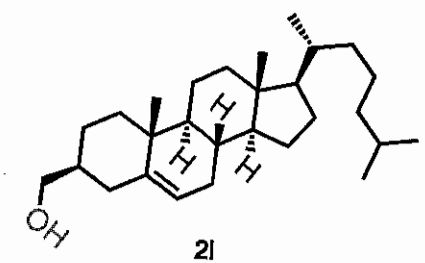


2k

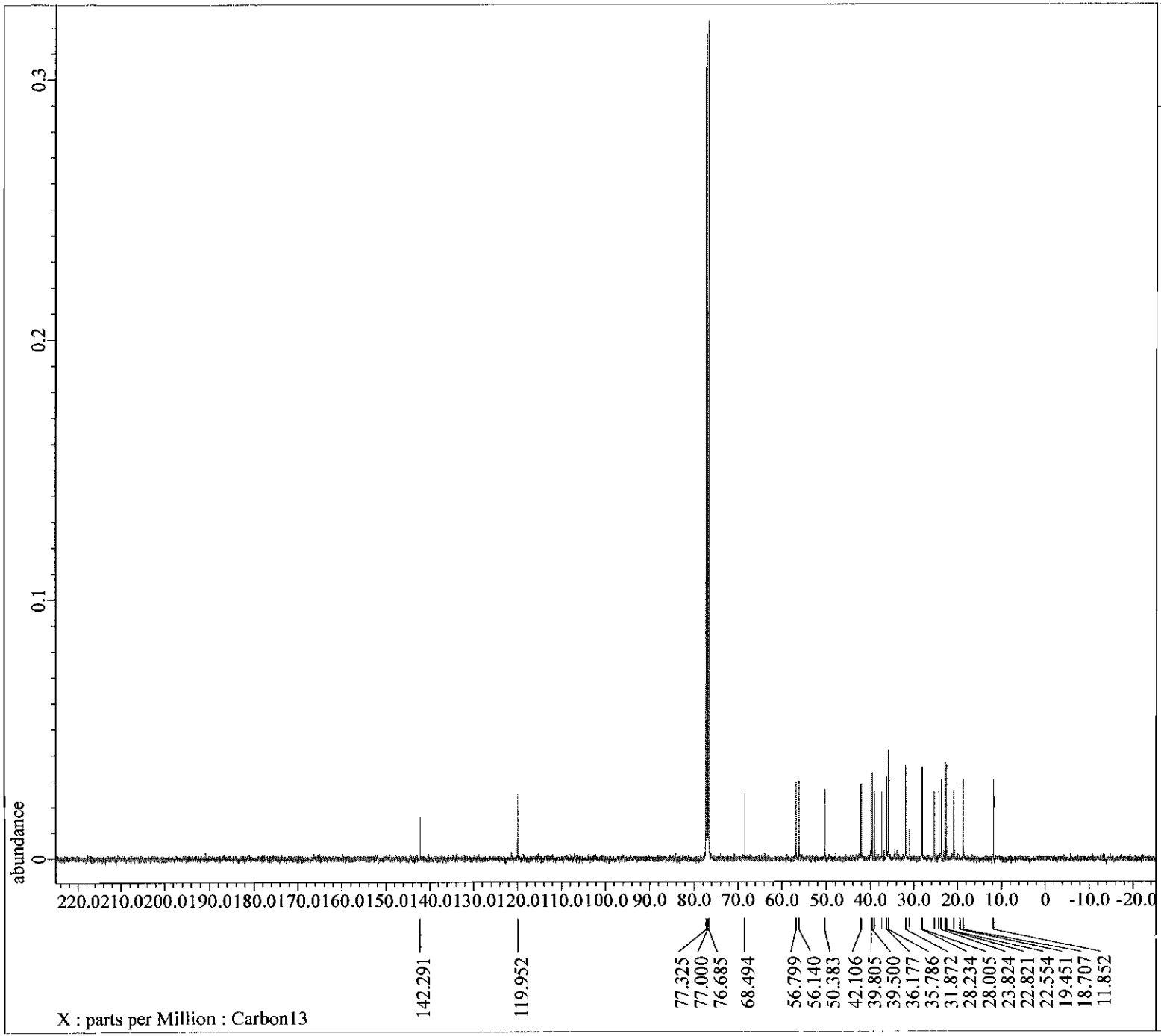


JEOL

Filename	= /Volumee/NO NAME/kawamoto/
Author	= delta
Experiment	= proton.jxp
Sample_Id	= tk-236-solid
Solvent	= CHLOROFORM-D
Creation_Time	= 14-SEP-2011 20:23:39
Reviaion_Time	= 14-SEP-2011 21:22:22
Current_Time	= 31-OCT-2011 23:01:00
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
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	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
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]
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	= 40
Temp_Get	= 22.1[dC]
X_90_Width	= 9.25[us]
X_Acq_Time	= 2.1757952[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 4.625[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.1757952[s]

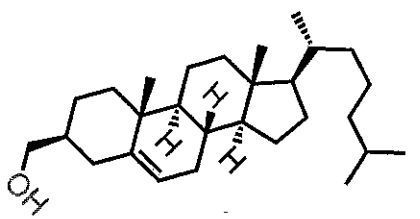


2l

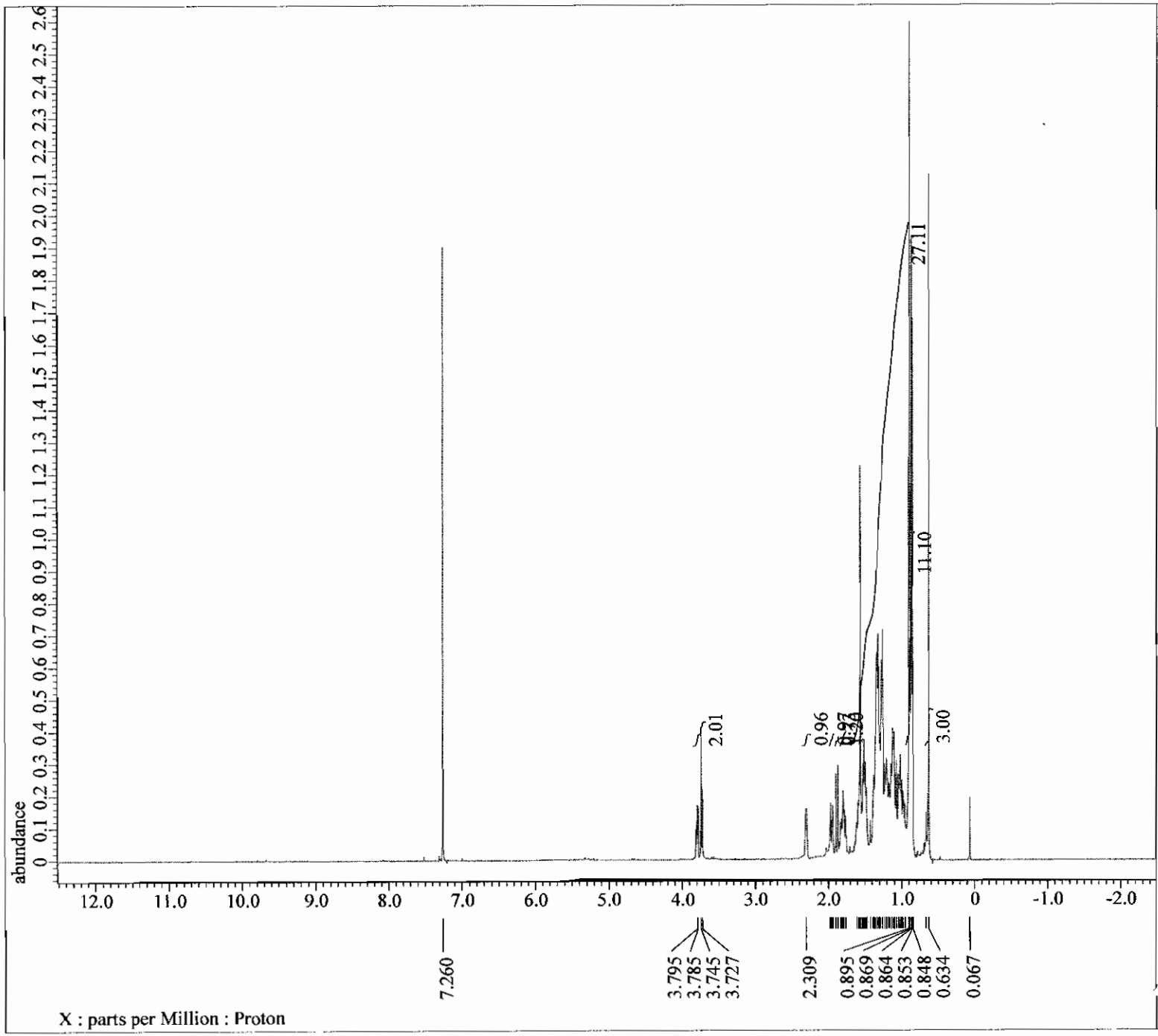


JEOL

Filename	= /Volumes/NO NAME/kawamoto/
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= tk-236-solid
Solvent	= CHLOROFORM-D
Creation_Time	= 15-SEP-2011 09:58:41
Revision_Time	= 22-OCT-2011 21:25:09
Current_Time	= 31-OCT-2011 23:00:44
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
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	= 100.0[ppm]
X_Points	= 32768
X_Prescans	= 4
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	= FALSE
Scans	= 1024
Total_Scans	= 1024
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 22.2[dC]
X_90_Width	= 8.75[us]
X_Acq_Time	= 1.03809024[s]
X_Angle	= 30[deg]
X_Atn	= 5.2[dB]
X_Pulse	= 2.91666667[us]
Irr_Atn_Dec	= 22.69[dB]
Irr_Atn_Noe	= 22.69[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.03809024[s]

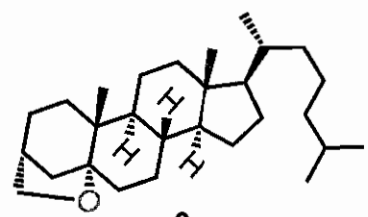


2l

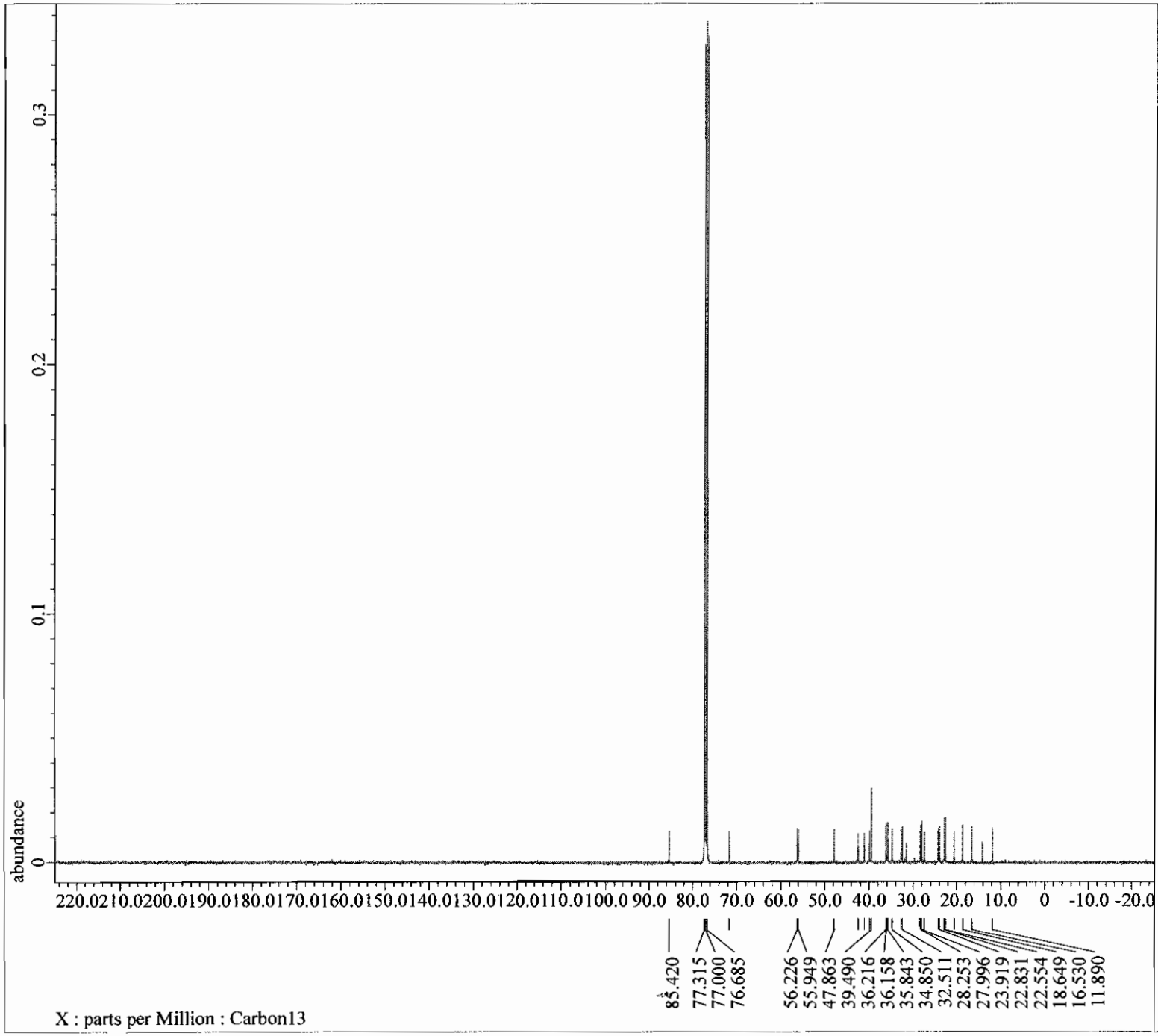


JEOL

Filename	= /Volumes/NO NAME/kawamoto/
Author	= delta
Experiment	= proton.jxp
Sample_Id	= tk-269-fr10-11
Solvent	= CHLOROFORM-D
Creation_Time	= 26-OCT-2011 19:05:59
Revision_Time	= 31-OCT-2011 22:55:17
Current_Time	= 31-OCT-2011 22:55:45
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
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	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
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]
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	= 21.9[dC]
X_90_Width	= 9.25[us]
X_Acq_Time	= 2.1757952[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 4.625[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.1757952[s]

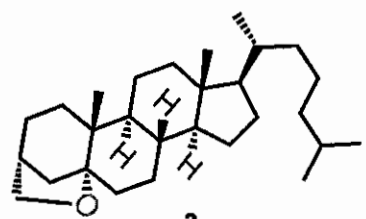


3

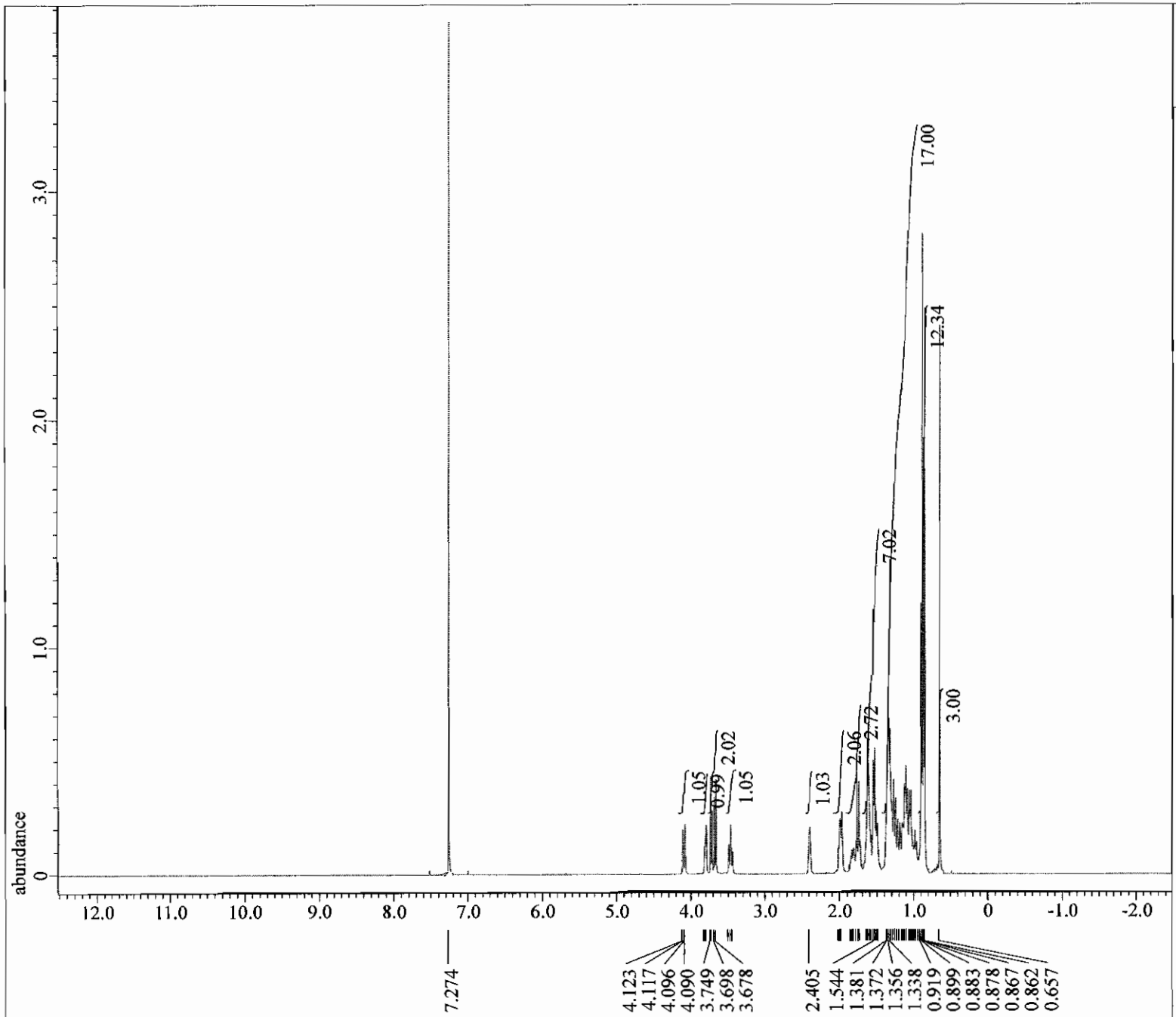


JEOL

Filename	= /Volumes/NO NAME/kawamoto/
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= tk-269-fr10-11
Solvent	= CHLOROFORM-D
Creation_Time	= 27-OCT-2011 02:05:33
Revision_Time	= 31-OCT-2011 22:56:43
Current_Time	= 31-OCT-2011 22:56:58
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
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	= 100.0[ppm]
X_Points	= 32768
X_Prescans	= 4
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	= FALSE
Scans	= 4000
Total_Scans	= 4000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 22[dC]
X_90_Width	= 8.75[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_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.03809024[s]

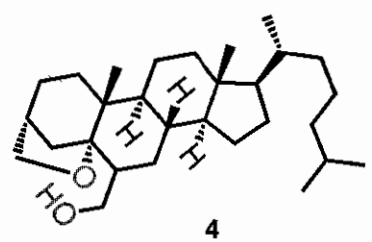


3



JEOL

Filename	= /Volumes/NO NAME/kawamoto/
Author	= delta
Experiment	= proton.jxp
Sample_Id	= tk-236-liquid
Solvent	= CHLOROFORM-D
Creation_Time	= 14-SEP-2011 20:16:08
Revision_Time	= 14-SEP-2011 21:20:10
Current_Time	= 31-OCT-2011 23:00:25
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
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	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
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]
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	= 38
Temp_Get	= 21.9[dC]
X_90_Width	= 9.25[us]
X_Acq_Time	= 2.1757952[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 4.625[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.1757952[s]



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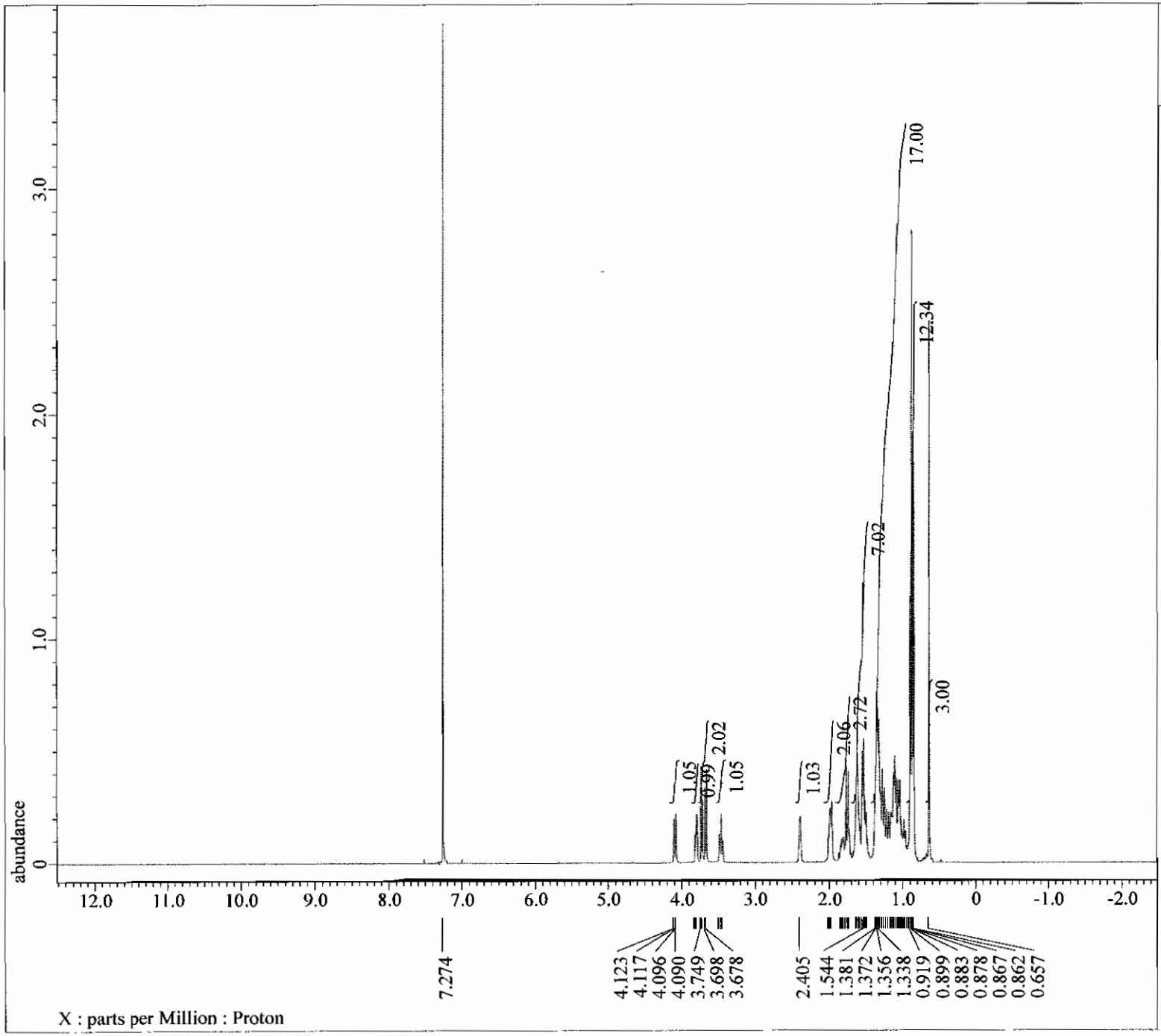
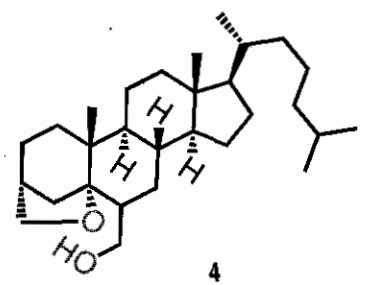


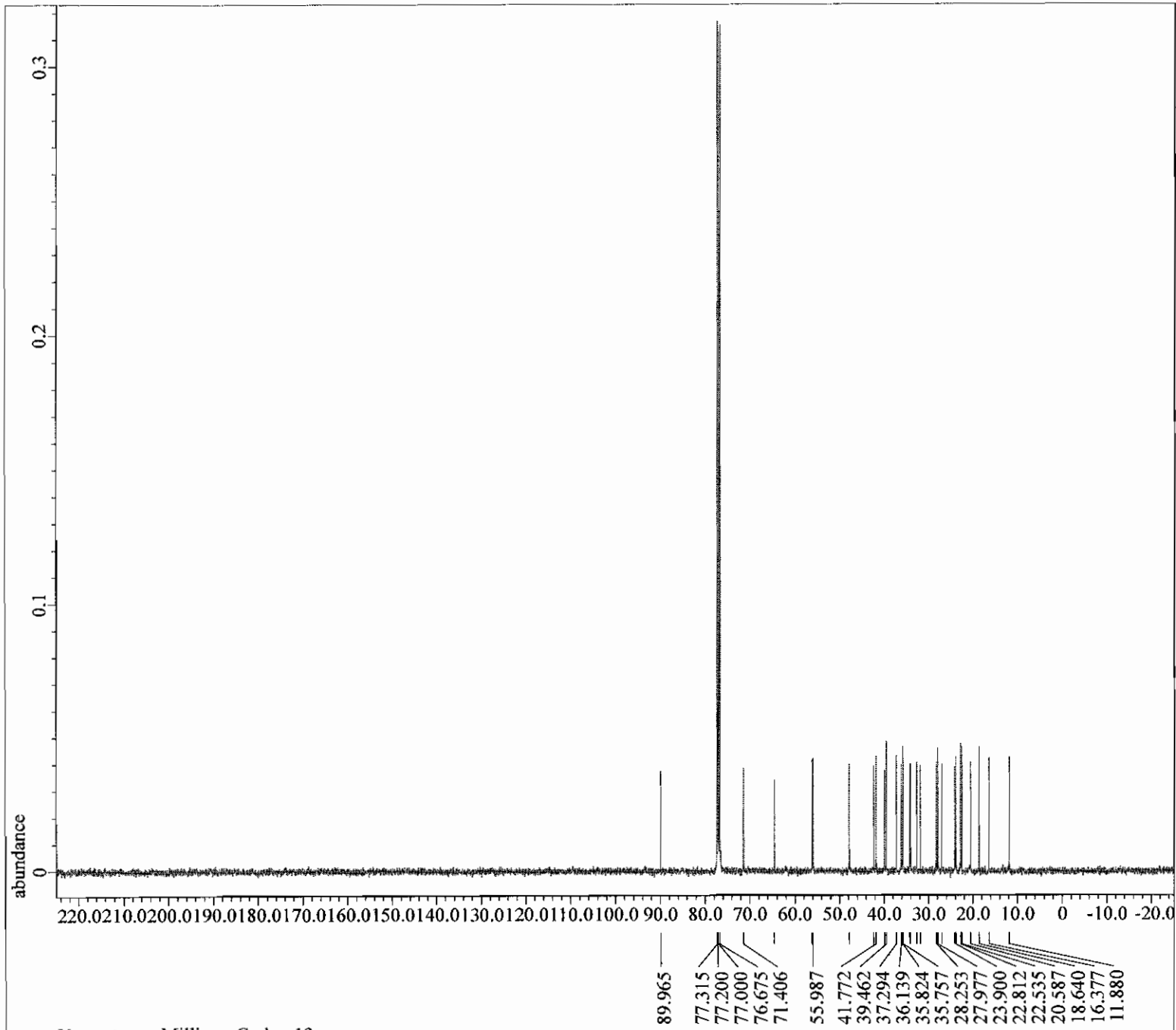
JEOL

Filename	= /Volumes/NO NAME/kawamoto/
Author	= delta
Experiment	= proton.jxp
Sample_Id	= tk-236-liquid
Solvent	= CHLOROFORM-D
Creation_Time	= 14-SEP-2011 20:16:08
Revision_Time	= 14-SEP-2011 21:20:10
Current_Time	= 31-OCT-2011 23:01:37
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
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	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
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]
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	= 38
Temp_Get	= 21.9[dC]
X_90_Width	= 9.25[us]
X_Acq_Time	= 2.1757952[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 4.625[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.1757952[s]

S33

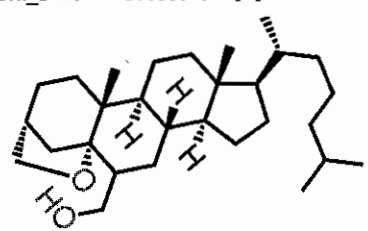


4



JEOL

Filename	= /Volumes/NO NAME/kawamoto/
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= tk-236-liquid
Solvent	= CHLOROFORM-D
Creation_Time	= 14-SEP-2011 21:19:54
Revision_Time	= 14-SEP-2011 22:15:46
Current_Time	= 31-OCT-2011 22:58:35
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-EC8400
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	= 100.0[ppm]
X_Points	= 32768
X_Prescans	= 4
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	= FALSE
Scans	= 1024
Total_Scans	= 1024
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 22[dC]
X_90_Width	= 8.75[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_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.03809024[s]



4