Supplementary Information

Effects of 2-substitution on 14-*epi*-19-nortachysterol-mediated biological events: Based on synthesis and X-ray co-crystallographic analysis with human vitamin D receptor

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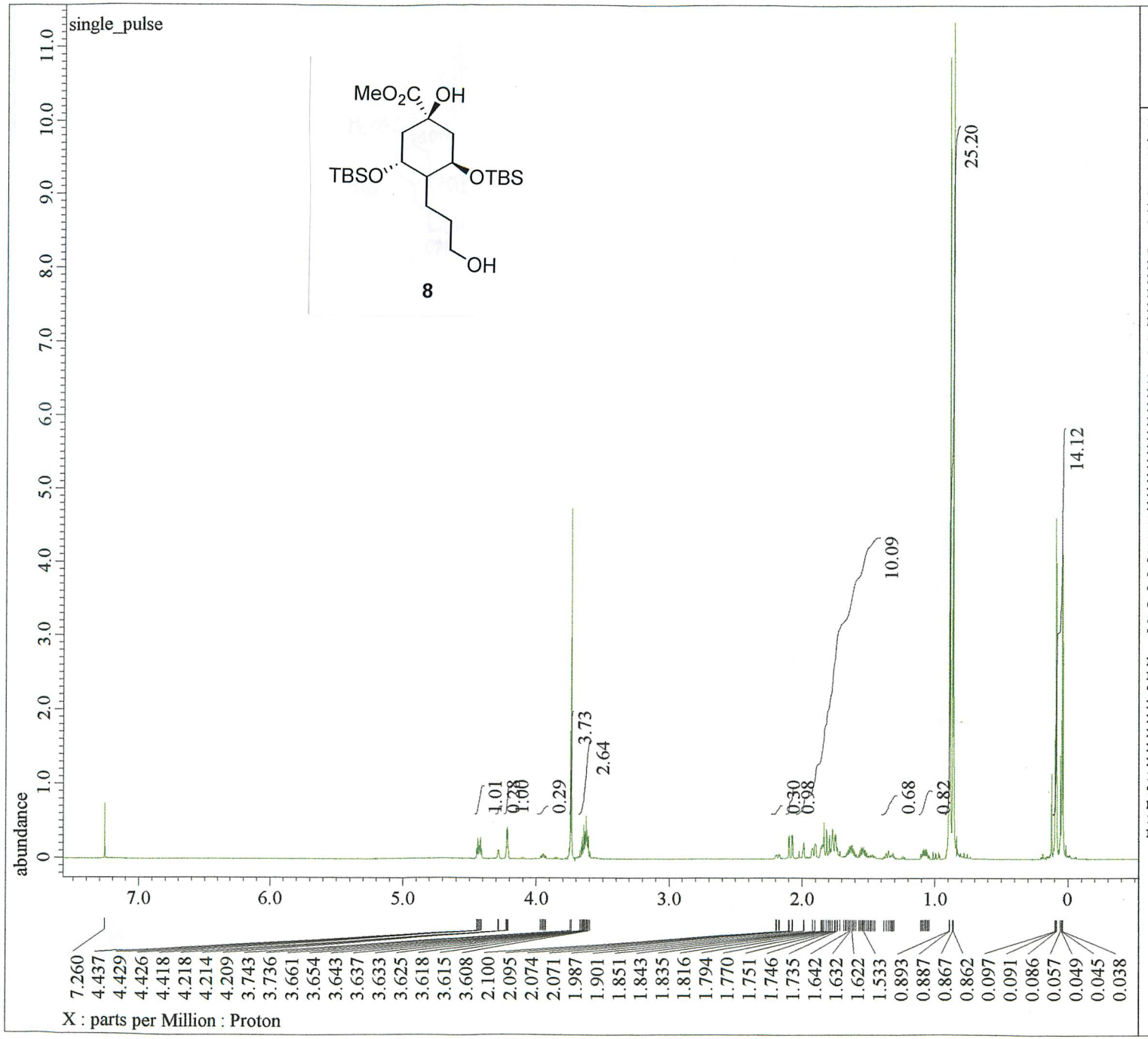
General Remarks: ^{1}H and ^{13}C NMR spectra were recorded on JEOL AL-400 NMR (400 MHz) and ECP-600 NMR (600 MHz) spectrometers. ^{1}H NMR spectra were referenced with $(CH_3)_4Si$ (δ 0.00 ppm) as an internal standard. ^{13}C NMR spectra were referenced with deuterated solvent (δ 77.0 ppm for $CDCl_3$ and 49.3 ppm for CD_3OD). IR spectra were recorded on JASCO FT-IR-800 Fourier Transform Infrared Spectrophotometer. High resolution mass spectra were obtained on SHIMADZU LCMS-IT-TOF mass spectrometer in positive electrospray ionization (ESI) method. Optical rotations were measured on a JASCO DIP-370 digital polarimeter. Column chromatography was performed on silica gel 60N (Kanto Chemical Co., Inc., 100-210 μm) or silica gel 60 (Merck, 0.040-0.063 mm). Preparative thin layer chromatography was performed on silica gel 60 F_{254} (Merck, 0.5 mm). High performance liquid chromatography (HPLC) was carried out on a SHIMADZU HPLC system consisting of the following equipments: pump, LC-6AD; detector, SPD-10A; column, YMC-Pack ODS-A. All experiments were performed under anhydrous conditions in an atmosphere of argon, unless otherwise mentioned.



JEOL RESONANCE

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Author	= y-yakka
Experiment	= proton.jxp
Sample_Id	= 41KWG180223-Aring-OH-data
Solvent	= CHLOROFORM-D
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Spectrometer	= DELTA2_NMR
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X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.68733284[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 22
Temp_Get	= 22.3[dC]
X_90_Width	= 8.1[us]
X_Acq_Time	= 1.4548992[s]
X_Angle	= 45[deg]
X_Atn	= 2.5[dB]
X_Pulse	= 4.05[us]
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Tri_Mode	= Off
Dante_Presat	= FALSE
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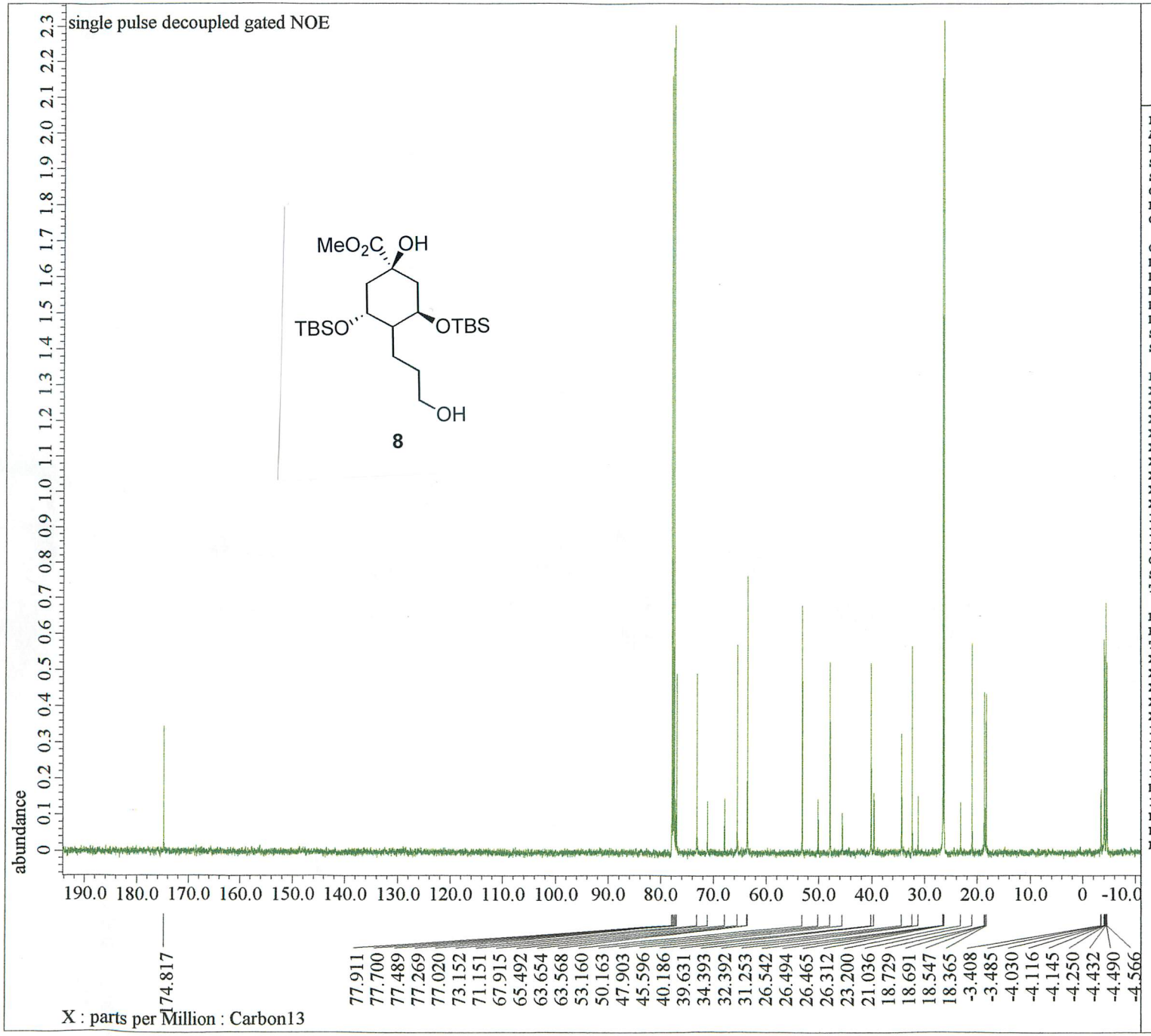

JEOL RESONANCE

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Author            = y-yakka
Experiment        = carbon.jxp
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Solvent           = CHLOROFORM-D
Creation_Time     = 23-FEB-2018 12:42:32
Revision_Time     = 23-FEB-2018 13:07:07
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Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.3[dC]
X_90_Width        = 11.1[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 12[dB]
X_Pulse           = 3.7[us]
Irr_Atn_Dec       = 21.947[dB]
Irr_Atn_Noe       = 21.947[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69206016[s]
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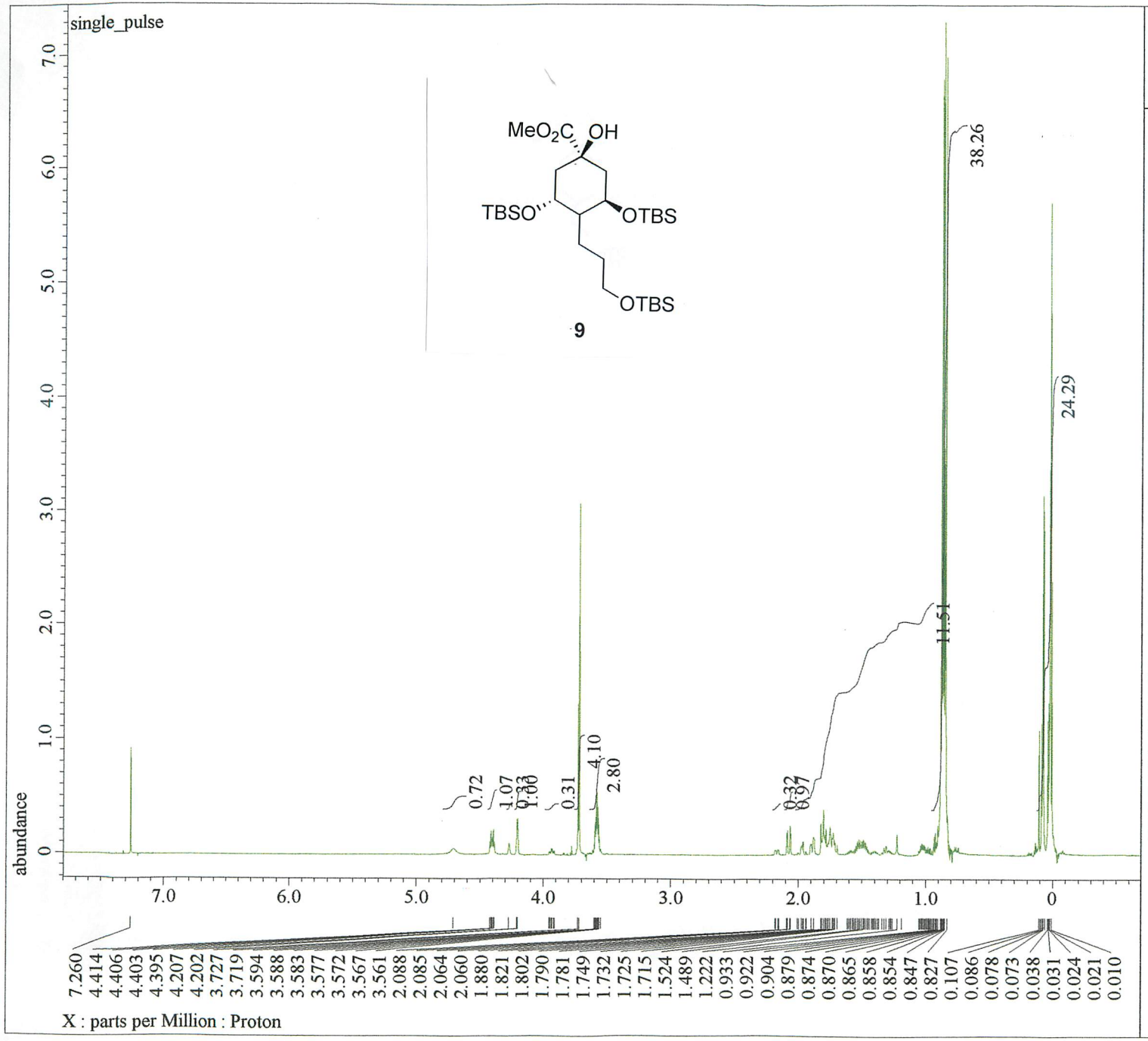

JEOL RESONANCE

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Author	= y-yakka
Experiment	= proton.jxp
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Solvent	= CHLOROFORM-D
Creation_Time	= 23-FEB-2018 18:02:18
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Spectrometer	= DELTA2_NMR
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X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.68733284[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 14
Temp_Get	= 22.1[dC]
X_90_Width	= 8.1[us]
X_Acq_Time	= 1.4548992[s]
X_Angle	= 45[deg]
X_Atn	= 2.5[dB]
X_Pulse	= 4.05[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 6.4548992[s]

single pulse decoupled gated NOE

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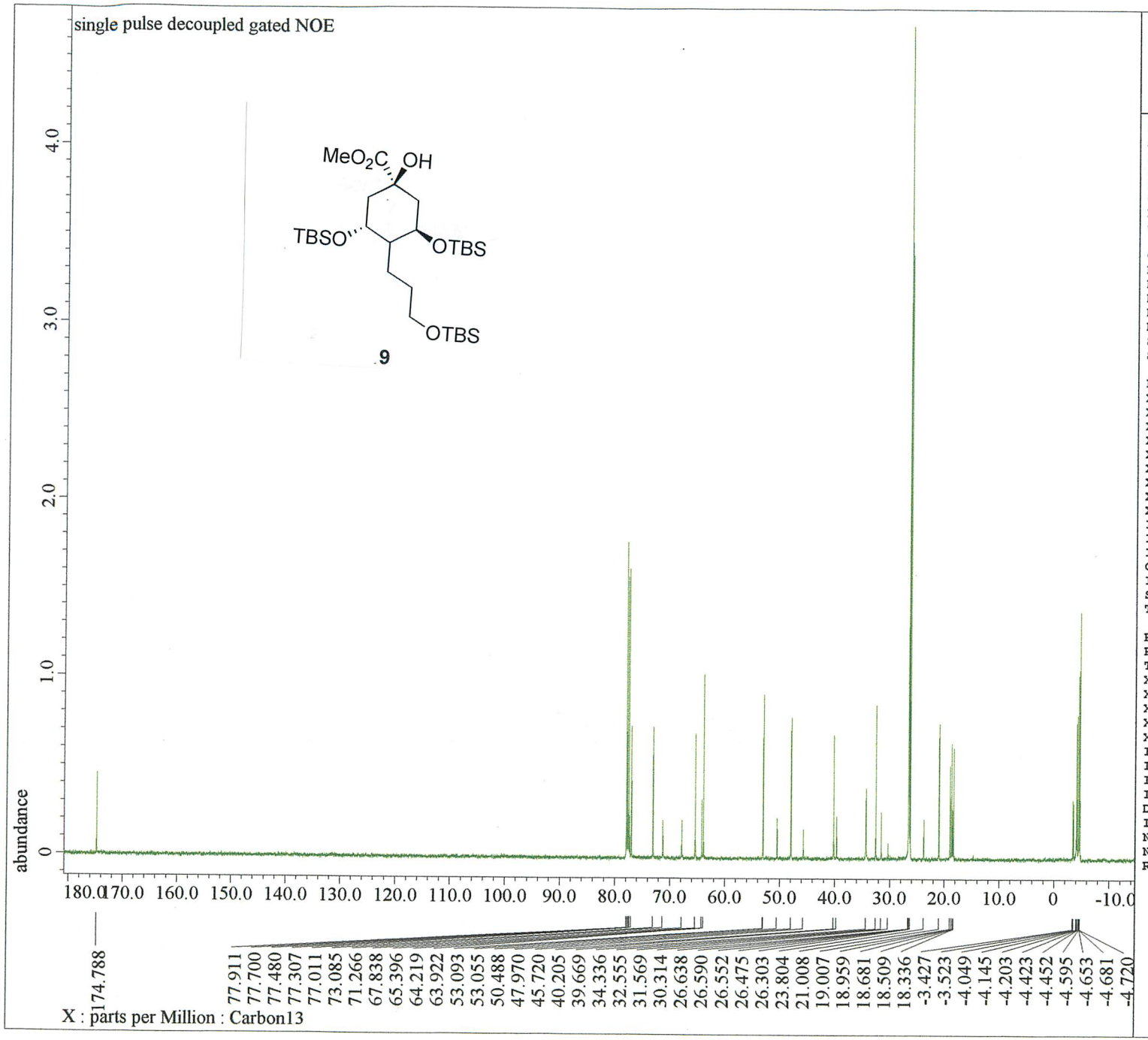

JEOL RESONANCE

Filename	= 41KWG180223-aring-OTBS-dat
Author	= y-yakka
Experiment	= carbon.jxp
Sample_Id	= 41KWG180223-aring-OTBS-dat
Solvent	= CHLOROFORM-D
Creation_Time	= 23-FEB-2018 18:05:48
Revision_Time	= 23-FEB-2018 18:22:01
Current_Time	= 23-FEB-2018 18:23:46
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Data_Format	= 1D COMPLEX
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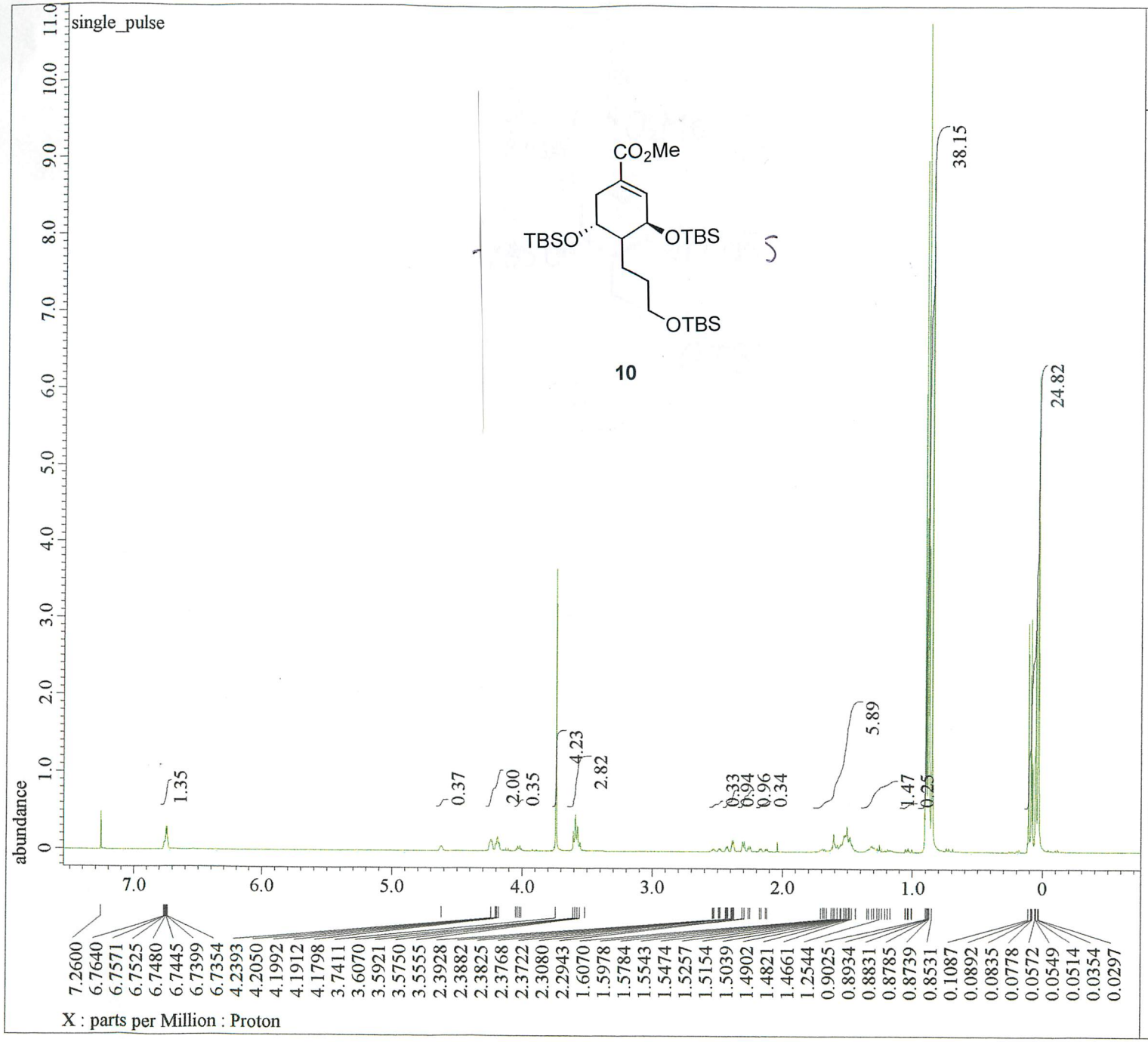
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 0[s]
X_Domain	= 13C
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Incomplete_Copy	= TRUE
Scans	= 285
Total_Scans	= 285
Relaxation_Delay	= 2[s]
Recvr_Gain	= 58
Temp_Get	= 22.2[dC]
X_90_Width	= 11.1[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 12[dB]
X_Pulse	= 3.7[us]
Irr_Atn_Dec	= 21.947[dB]
Irr_Atn_Noe	= 21.947[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 76[us]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.69206016[s]



JEOL RESONANCE

Filename	= 41KWG180227-aring-3_Proton
Author	= y-yakka
Experiment	= proton.jxp
Sample_Id	= 41KWG180227-aring-3
Solvent	= CHLOROFORM-D
Creation_Time	= 27-FEB-2018 17:30:36
Revision_Time	= 27-FEB-2018 17:44:00
Current_Time	= 27-FEB-2018 17:46:58
Comment	= single_pulse
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Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
Spectrometer	= DELTA2_NMR
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]
X_Sweep_Clipped	= 6.00240096[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 22
Temp_Get	= 24[dC]
X_90_Width	= 6.3[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 1.1[dB]
X_Pulse	= 3.15[us]
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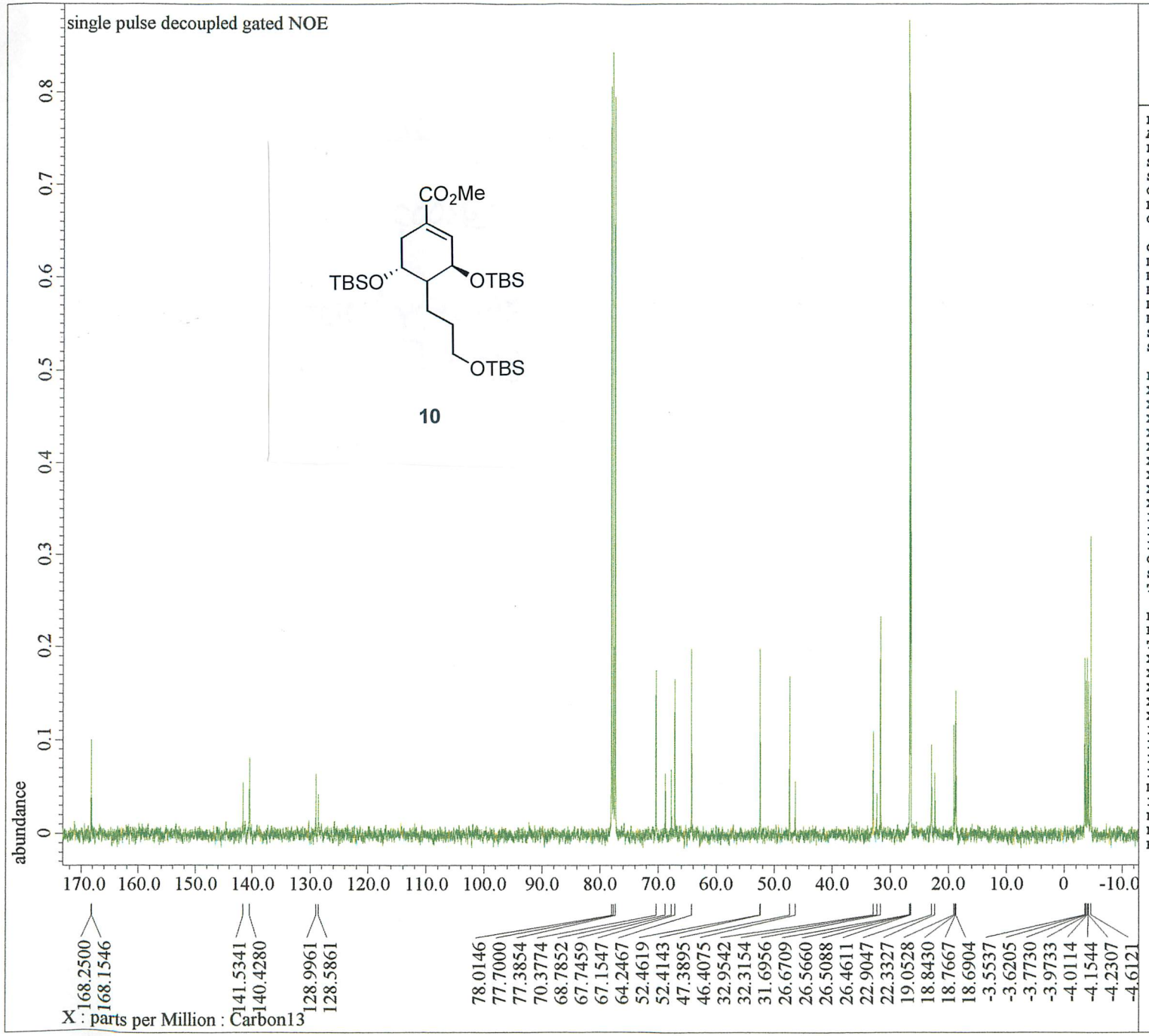
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]



JEOL RESONANCE

Filename	= 41KWG180227-aring-3_Carbon
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Experiment	= carbon.jxp
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Solvent	= CHLOROFORM-D
Creation_Time	= 27-FEB-2018 17:34:40
Revision_Time	= 27-FEB-2018 17:48:58
Current_Time	= 27-FEB-2018 17:49:10
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
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Spectrometer	= DELTA2_NMR
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]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 256
Total_Scans	= 256
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 23.5[dC]
X_90_Width	= 10.9[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 3.8[dB]
X_Pulse	= 3.63333333[us]
Irr_Atn_Dec	= 26.327[dB]
Irr_Atn_Noe	= 26.327[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
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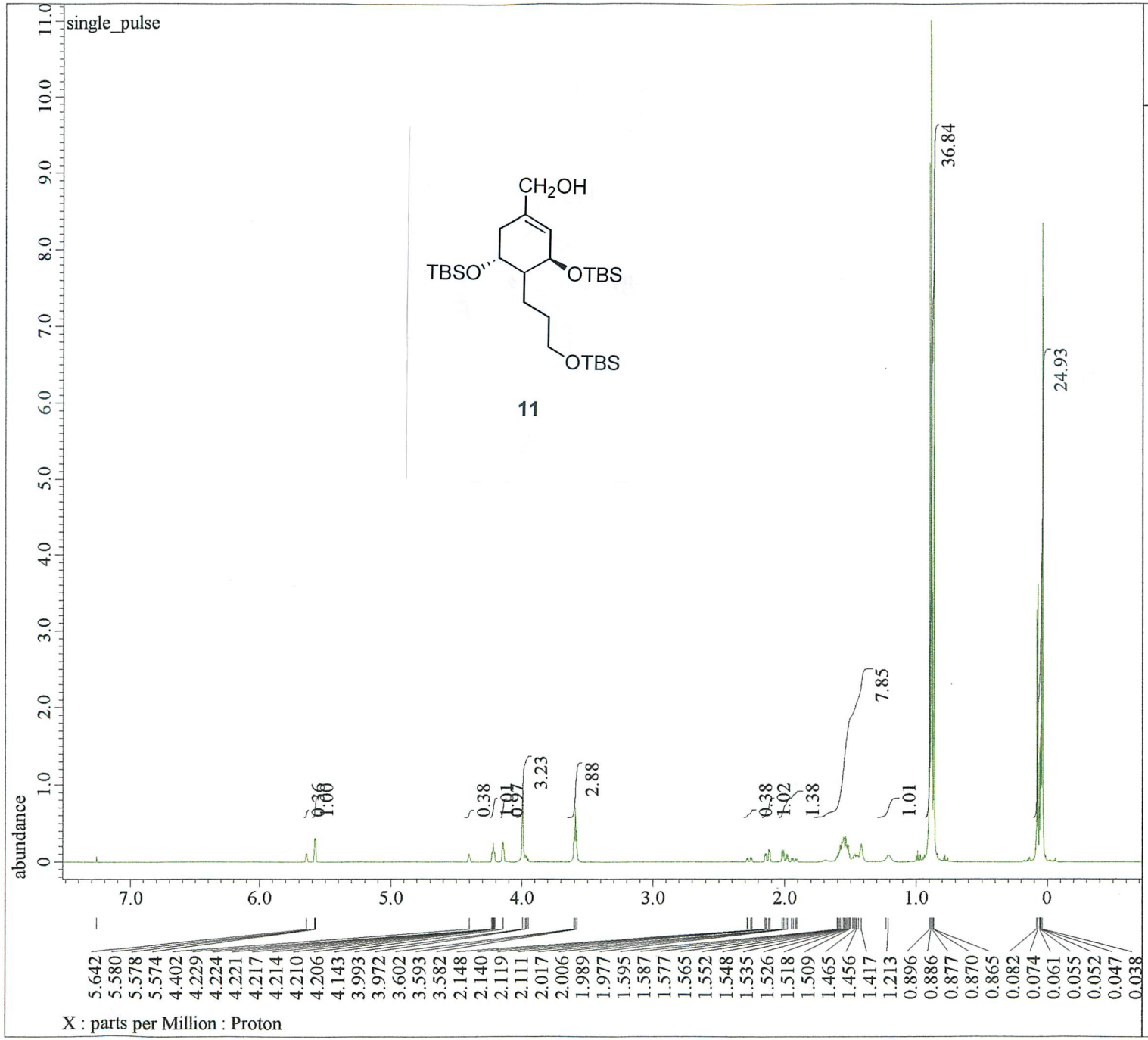
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.04333312[s]



JEOL RESONANCE

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Author	= y-yakka
Experiment	= proton.jxp
Sample_Id	= 41KWG180228-aring4data
Solvent	= CHLOROFORM-D
Creation_Time	= 28-FEB-2018 16:22:25
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Current_Time	= 28-FEB-2018 16:37:33
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Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 1.4548992[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.68733284[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 20
Temp_Get	= 22.4[dC]
X_90_Width	= 8.1[us]
X_Acq_Time	= 1.4548992[s]
X_Angle	= 45[deg]
X_Atn	= 2.5[dB]
X_Pulse	= 4.05[us]
Irr_Mode	= Off
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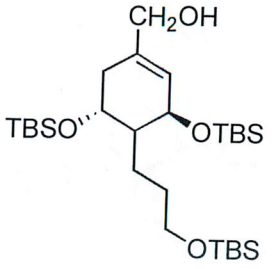
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 6.4548992[s]

single pulse decoupled gated NOE



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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

137.935
125.335
124.406
77.911
77.700
77.489
70.624
69.006
67.493
67.359
67.168
67.082
64.420
64.372
48.258
47.453
35.389
33.781
32.000
31.578
26.676
26.657
26.599
26.504
22.817
22.367
19.055
18.863
18.786
18.729
-3.063
-3.523
-3.791
-3.906
-4.049
-4.097
-4.136
-4.576

X : parts per Million : Carbon13

JEOL RESONANCE

Filename	= 41KWG180228-aring4data_Car
Author	= y-yakka
Experiment	= carbon.jxp
Sample_Id	= 41KWG180228-aring4data
Solvent	= CHLOROFORM-D
Creation_Time	= 28-FEB-2018 16:25:44
Revision_Time	= 28-FEB-2018 16:54:16
Current_Time	= 28-FEB-2018 16:54:39
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Dimensions	= X
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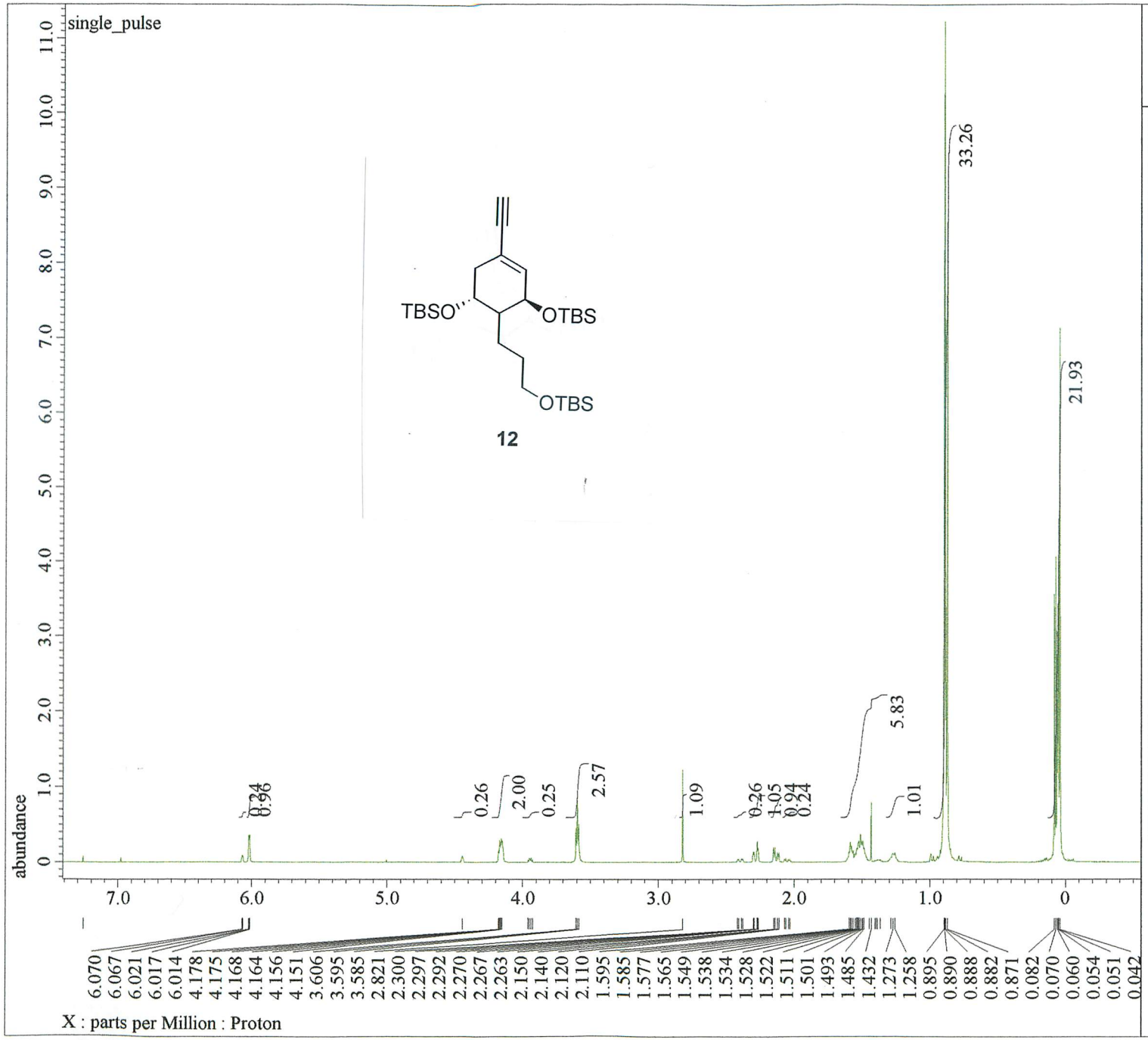
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 0.69206016[s]
X_Domain	= 13C
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 512
Total_Scans	= 512
Relaxation_Delay	= 2[s]
Recvr_Gain	= 58
Temp_Get	= 23.8[dC]
X_90_Width	= 11.1[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 12[dB]
X_Pulse	= 3.7[us]
Irr_Atn_Dec	= 21.947[dB]
Irr_Atn_Noe	= 21.947[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 76[us]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.69206016[s]



JEOL RESONANCE

Filename	= 41KWG180301-sawadaaring5_P
Author	= y-yakka
Experiment	= proton.jxp
Sample_Id	= 41KWG180301-sawadaaring5
Solvent	= CHLOROFORM-D
Creation_Time	= 1-MAR-2018 14:08:52
Revision_Time	= 1-MAR-2018 14:14:04
Current_Time	= 1-MAR-2018 14:14:12
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Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 1.4548992[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.68733284[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 20
Temp_Get	= 22.6[dC]
X_90_Width	= 8.1[us]
X_Acq_Time	= 1.4548992[s]
X_Angle	= 45[deg]
X_Atn	= 2.5[dB]
X_Pulse	= 4.05[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
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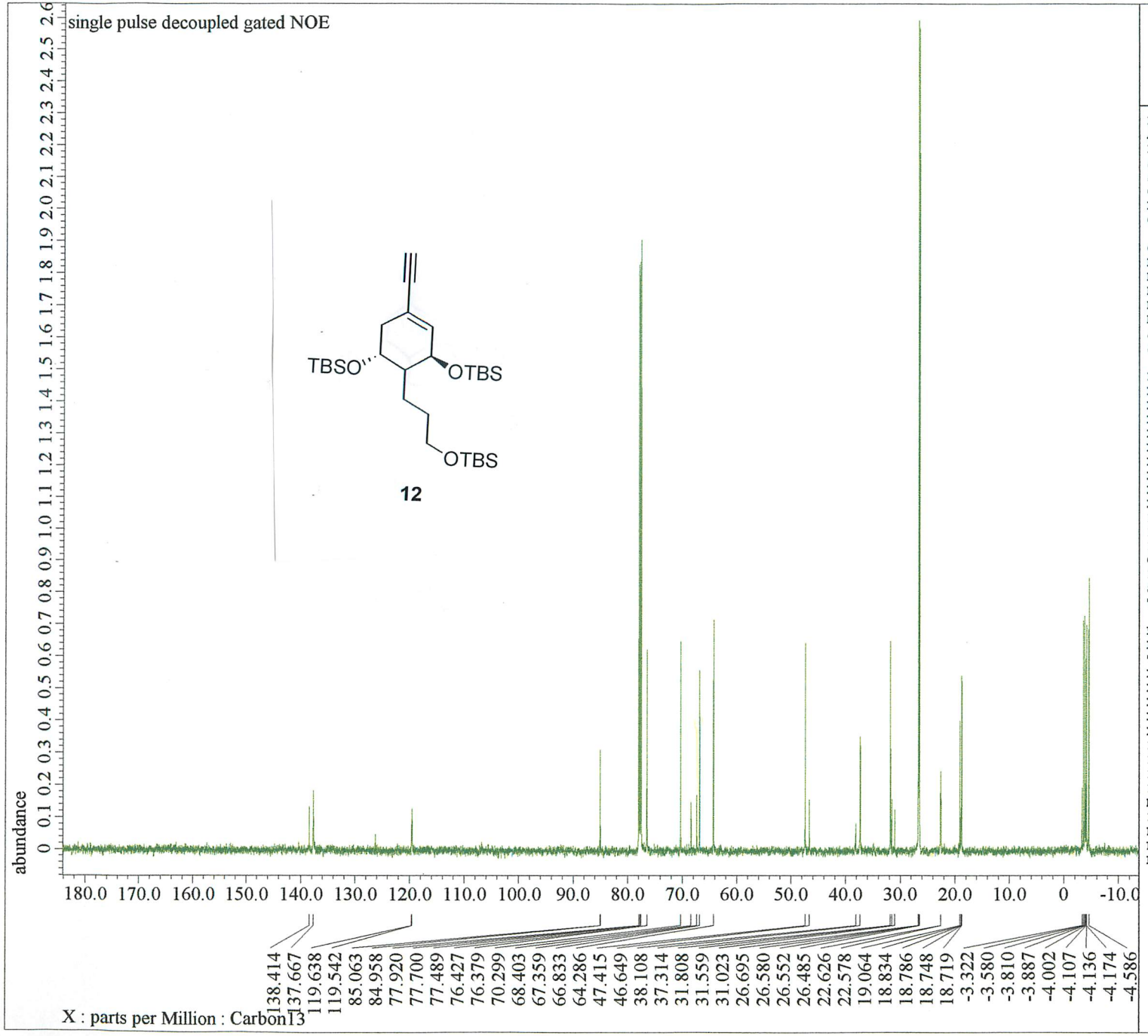

JEOL RESONANCE

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Experiment	= carbon.jxp
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Solvent	= CHLOROFORM-D
Creation_Time	= 1-MAR-2018 14:12:23
Revision_Time	= 1-MAR-2018 14:32:58
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Comment	= single pulse decoupled gat
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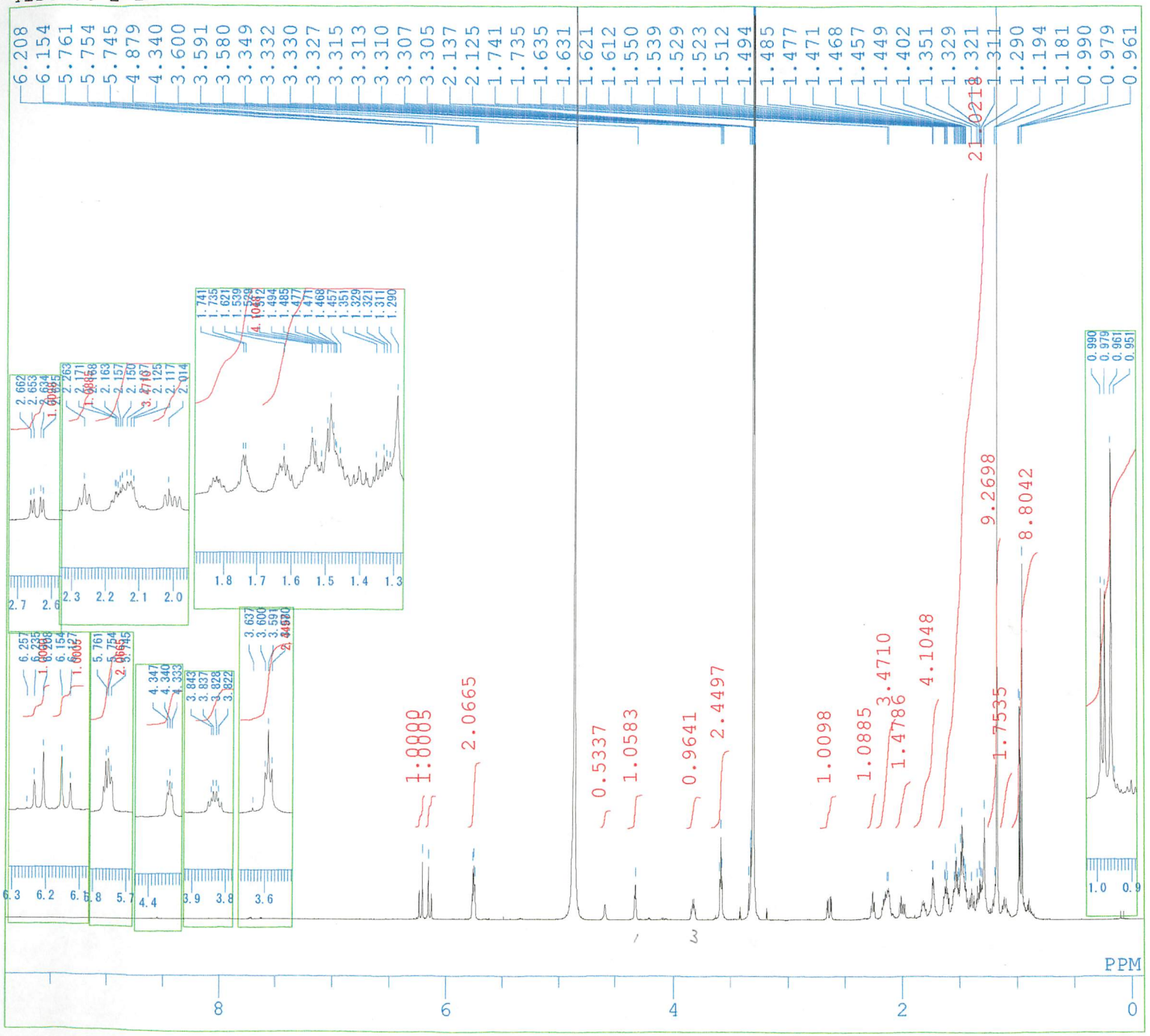
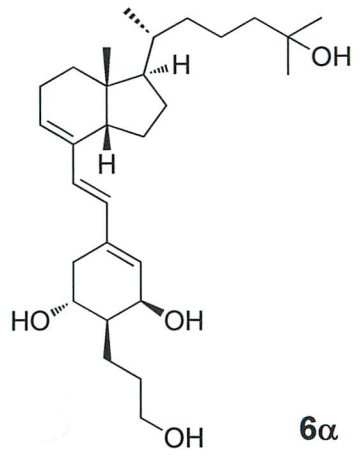
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 0[s]
X_Domain	= 13C
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Incomplete_Copy	= TRUE
Scans	= 256
Total_Scans	= 256
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 22.7[dC]
X_90_Width	= 11.1[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 12[dB]
X_Pulse	= 3.7[us]
Irr_Atn_Dec	= 21.947[dB]
Irr_Atn_Noe	= 21.947[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 76[us]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.69206016[s]

\\10.20.10.246\moc600\41D3136-2-2.als
41D3136-2-2

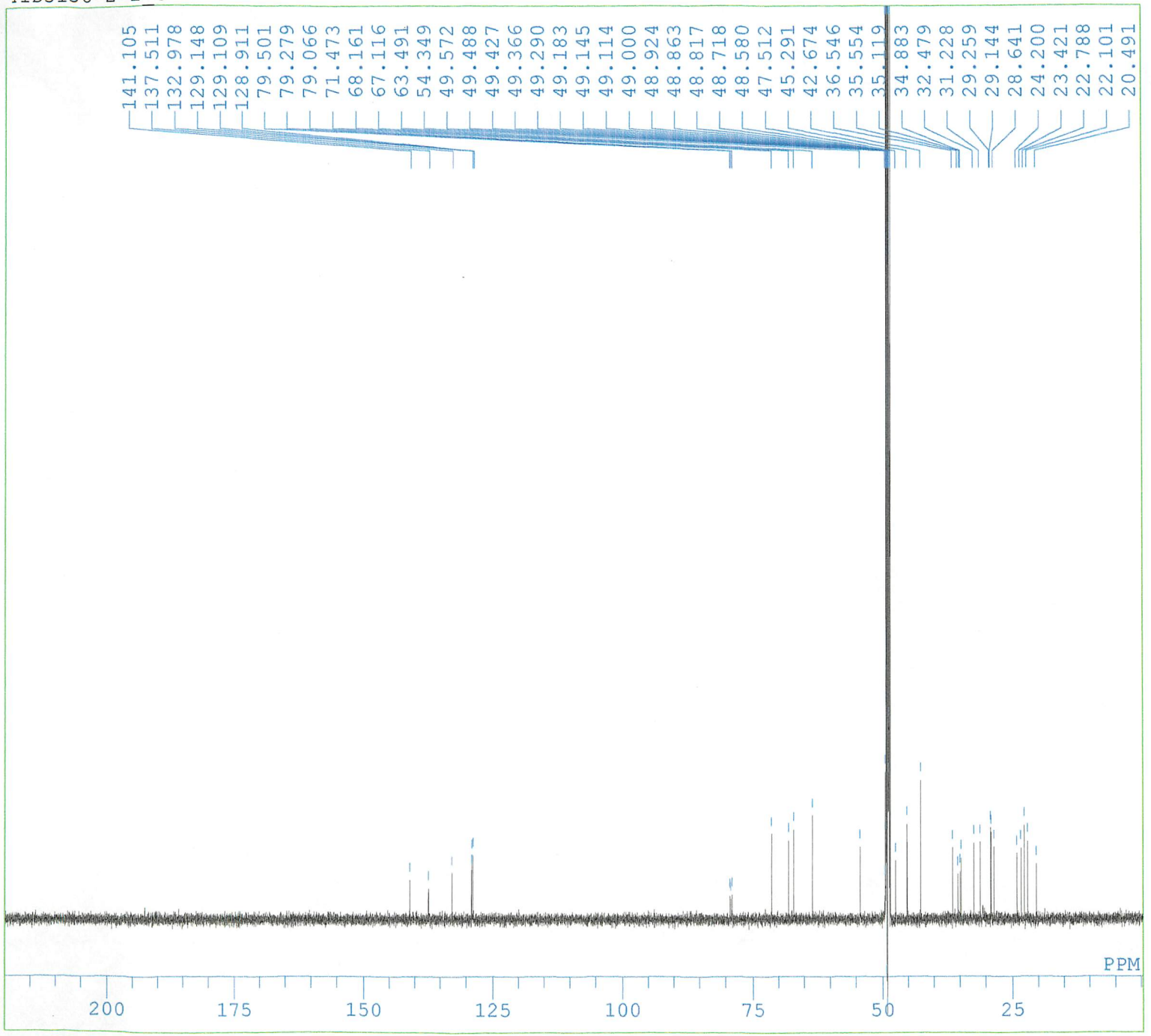


DFILE \\10.20.10.246\moc600\41D313
COMNT 41D3136-2-2
DATIM 3-JUN-2010 13:29:12
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 600.00 MHz
OBSET 175.00 KHz
OBFIN 305.5 Hz
POINT 32768
FREQU 9009.0 Hz
SCANS 512
ACQTM 3.637 sec
PD 4.000 sec
PW1 6.2 us
IRNUC 1H
CTEMP 22.1 c
SLVNT CD3OD
EXREF 3.31 ppm
BF 0.12 Hz
RGAIN 29

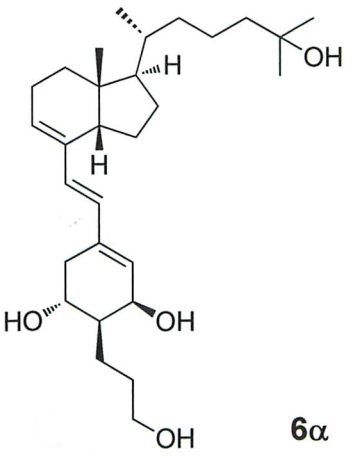


6α

\\10.20.10.246\moc600\41D3136-2-2_C.als
41D3136-2-2_C

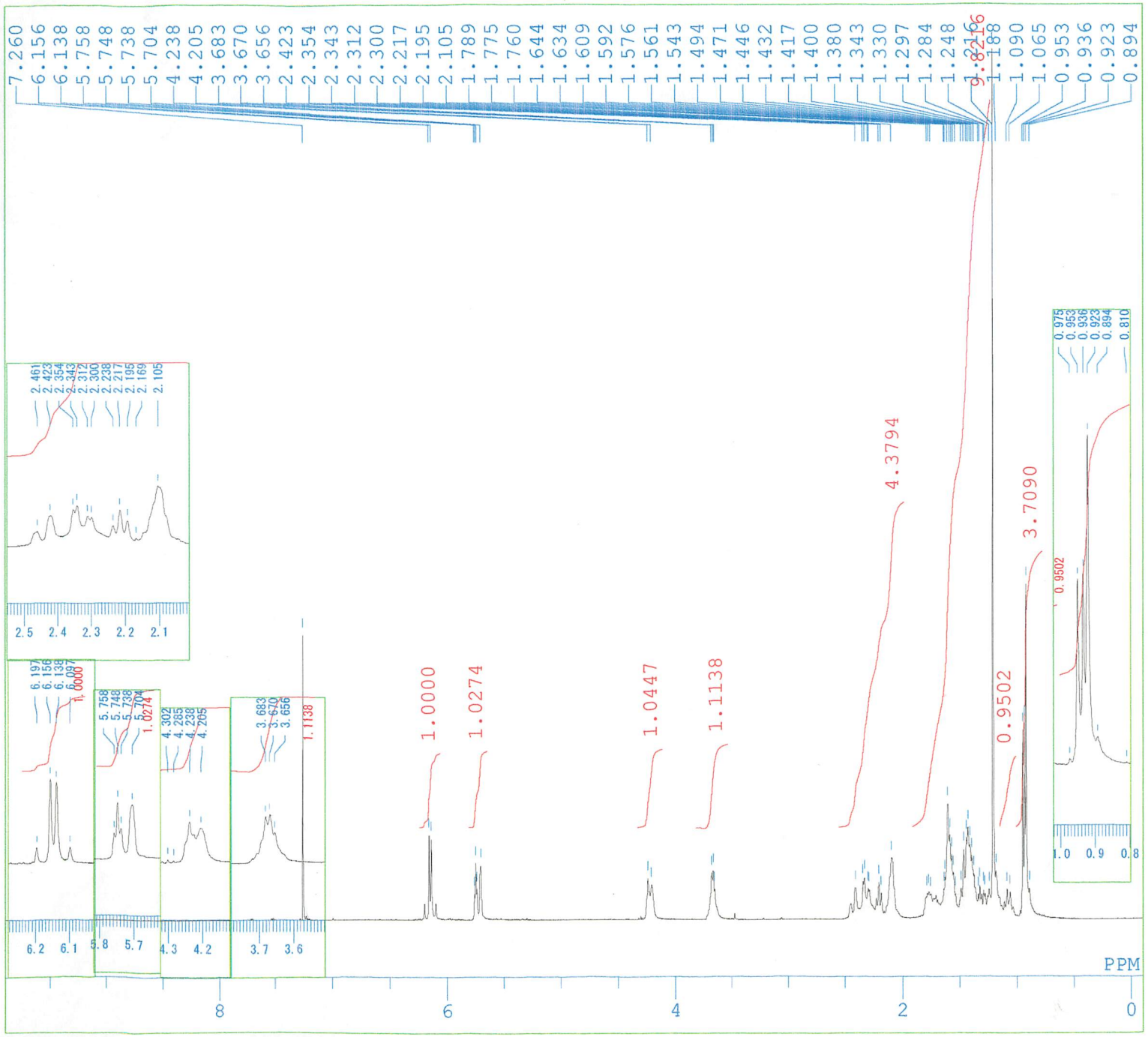


DFILE	\\10.20.10.246\moc600\41D313
COMNT	41D3136-2-2_C
DATIM	7-JUN-2010 10:50:42
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.00 MHz
OBSET	928.00 KHz
OBFIN	521.7 Hz
POINT	32768
FREQU	37735.8 Hz
SCANS	59000
ACQTM	0.868 sec
PD	3.000 sec
PW1	3.7 us
IRNUC	1H
CTEMP	24.5 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	0.12 Hz
RGAIN	30

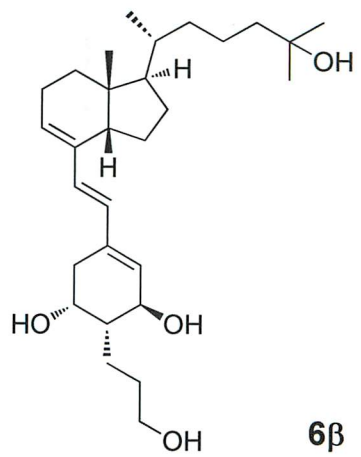


6α

\\10.20.10.243\40041\sawada\3\41d3136-2-1.als

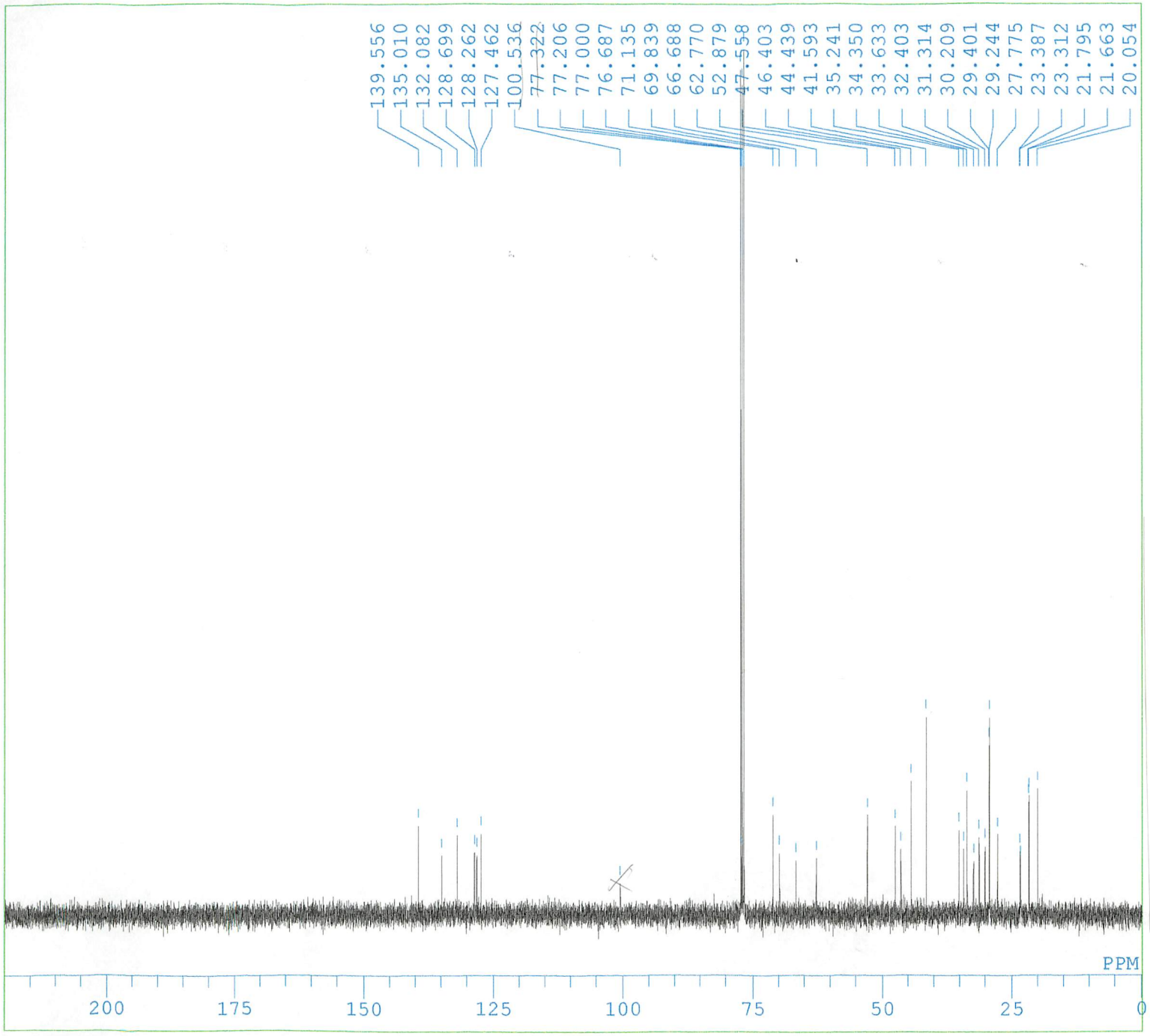


DFILE \\10.20.10.243\40041\sawada\
COMNT
DATIM Mon May 17 19:31:17 2010
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.0 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 16
ACQTM 4.096 sec
PD 2.904 sec
PW1 6.2 us
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 16

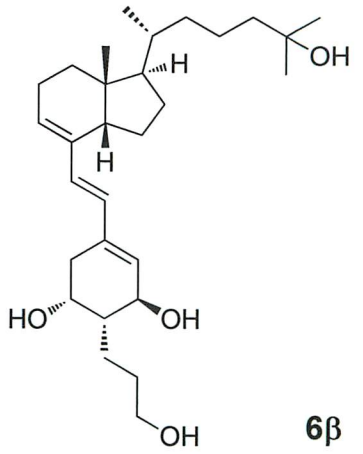


6β

\\10.20.10.243\40041\sawada\3\41d3136-2-1bcm.als



DFILE	\\10.20.10.243\40041\sawada\
COMNT	
DATIM	Mon May 17 20:50:44 2010
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.0 Hz
POINT	32768
FREQU	27173.9 Hz
SCANS	1200
ACQTM	1.206 sec
PD	1.794 sec
PW1	4.6 us
IRNUC	1H
CTEMP	23.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	25



6β