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Supporting Information

Palladium-catalyzed dehydrative *N*-benzylation/C-H benzylation cascade of 2-morpholinoanilines on water

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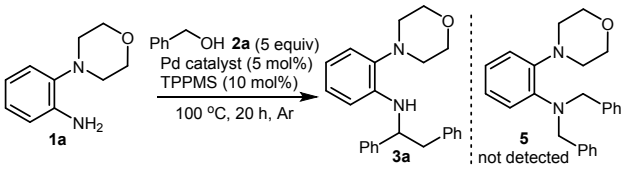
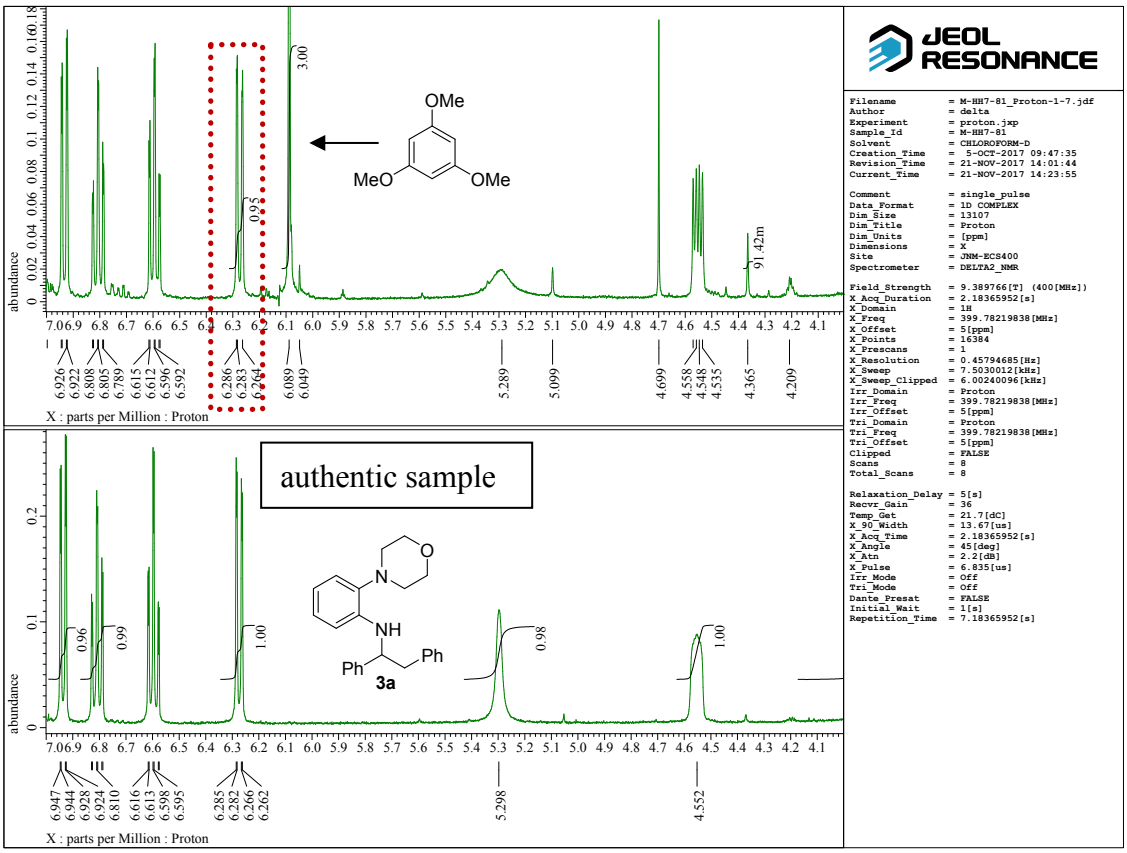
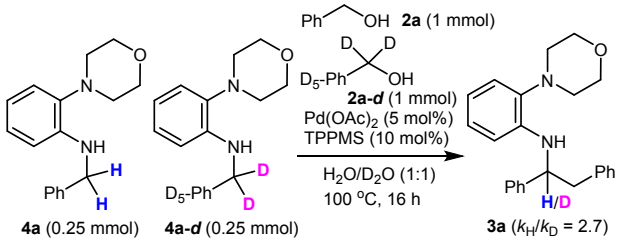
Effects of catalysts and solvents (see Table 1, entry 8).



A mixture of 2-morpholinoaniline **1a** (178 mg, 1 mmol), palladium(II) acetate (11 mg, 0.05 mmol), sodium diphenylphosphinobenzene-3-sulfonate (TPPMS, 36 mg, 0.1 mmol) and benzyl alcohol **2a** (515 μL, 5 mmol) in H_2O (4 mL) was heated at 100 °C for 20 h in a sealed tube under Ar. After the reaction mixture was cooled, 1,3,5-trimethoxybenzene (168.2 mg, 1 mmol, internal standard) was added to the reaction mixture, which was extracted with $CDCl_3$ (8 mL), then the organic layer was analyzed by ^{1}H-NMR spectroscopy.

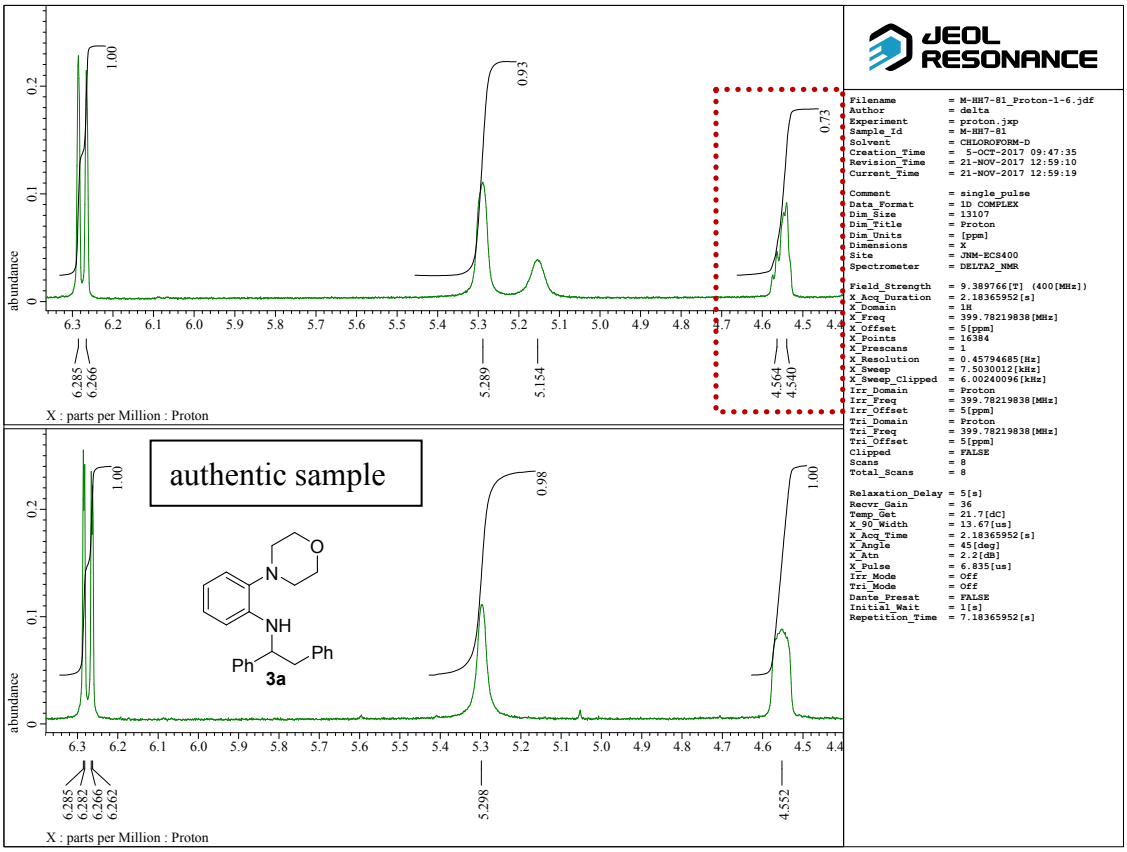


Competitive deuterium labeling experiment (see Scheme 4A).

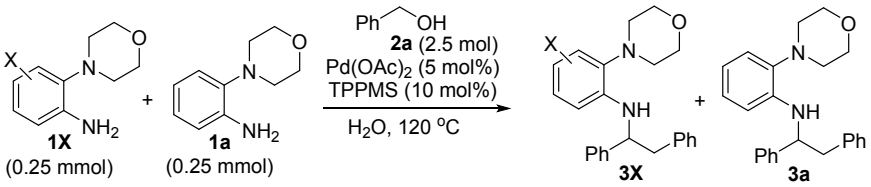


A mixture of **4a** (67.1 mg, 0.25 mmol), **4a-*d*** (68.9 mg, 0.25 mmol), $Pd(OAc)_2$ (6 mg, 0.025 mmol), sodium diphenylphosphinobenzene-3-sulfonate (TPPMS, 18 mg, 0.05 mmol), benzyl alcohol **2a** (108 mg, 1.0 mmol), and **2a-*d*** (115 mg, 1.0 mmol) in H_2O (1 mL) and D_2O (1 mL) was heated at 100 °C for 16 h in a sealed tube under air. After cooling, the reaction mixture was poured into water and extracted with EtOAc. The organic layer was washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The residue was washed with hexanes, then purified by flash column chromatography (silica gel, hexanes/EtOAc) to give desired product **3a**.

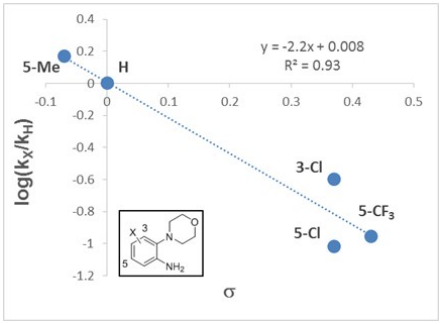
Signal δ	6.27 (Ar-**H**)	4.55 (methine-**H**)
Integral value	1.00 (1H)	0.73 (1H): k_H/k_D = 0.73/0.27 = 2.7



Hammett study (see Figure 4).

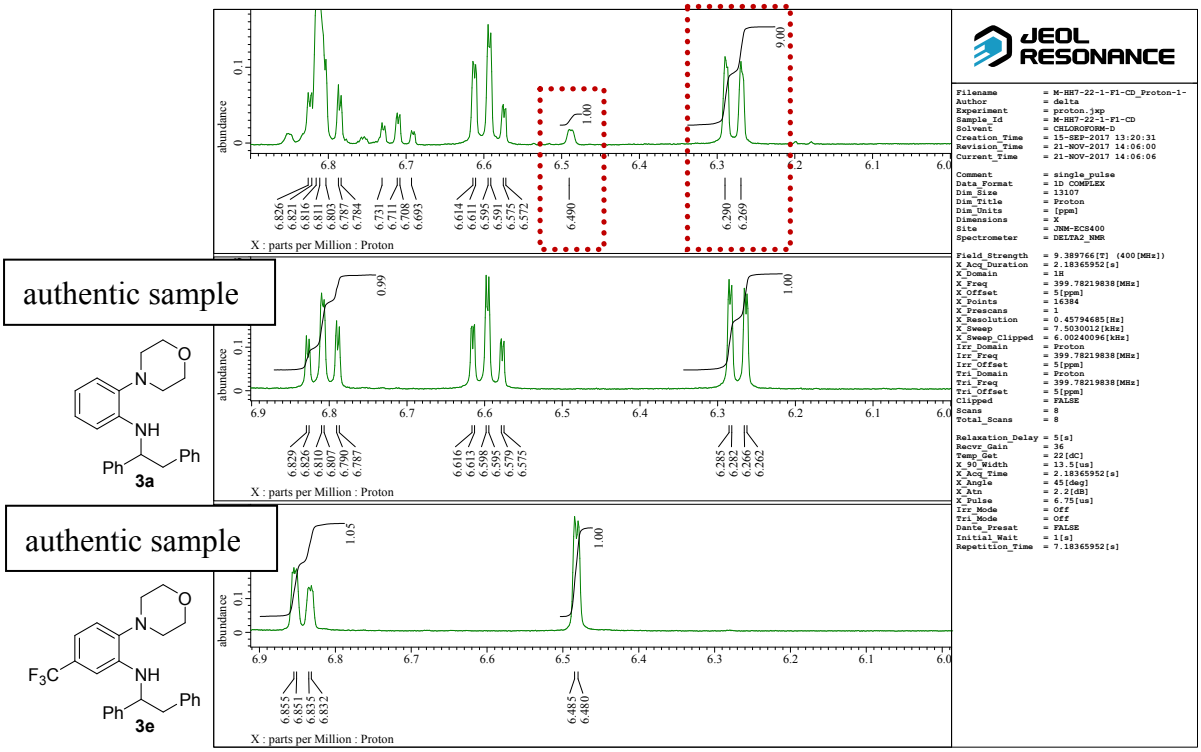


A mixture of 3- or 5-substituted 2-morpholinoanilines **1** (X = Me, Cl and CF_3 groups) (0.25 mmol), $Pd(OAc)_2$ (6 mg, 0.025 mmol), sodium diphenylphosphinobenzene-3-sulfonate (TPPMS, 18 mg, 0.05 mmol) and benzylic alcohol **2a** (258 μL, 2.5 mmol) in H_2O (2 mL) was heated at 120 °C in a sealed tube under air. After cooling, the reaction mixture was poured into water and extracted with EtOAc. The organic layer was washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The residue was analyzed by ^{1}H-NMR spectroscopy.

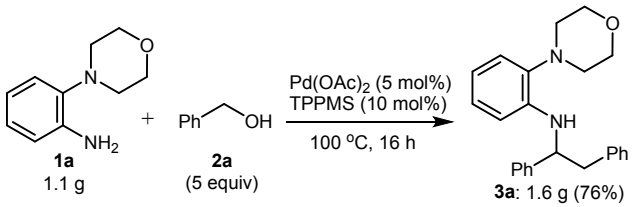


	σ	$\log(k_X/k_H)$
5-Me (m)	-0.07	0.17
H	0	0
3-Cl (m)	0.37	-0.6
5-Cl (m)	0.37	-1.02
5-CF_3 (m)	0.43	-0.954

X = CF_3: $\log(k_X/k_H) = \log(1/9) = -0.95$



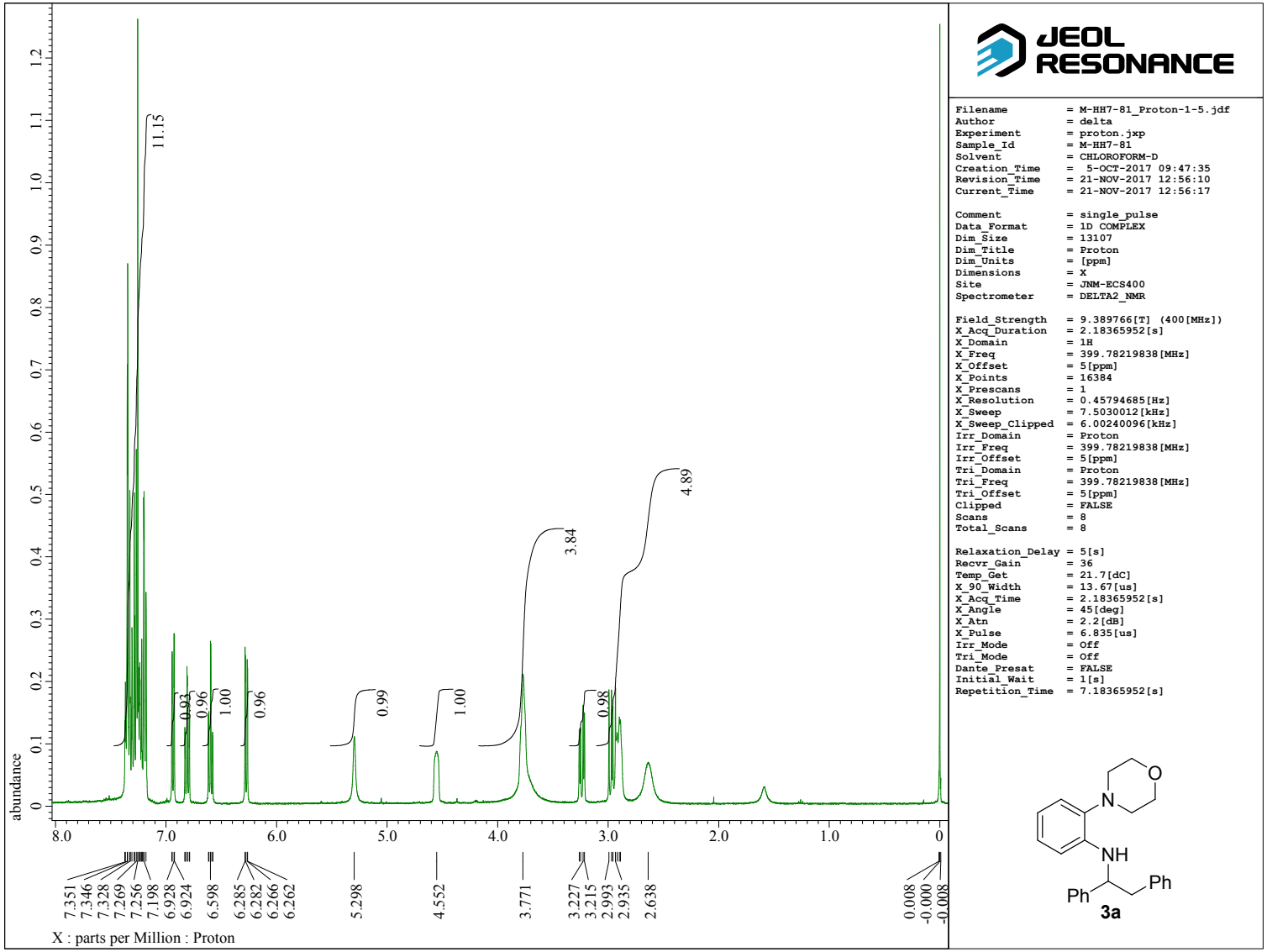
Scale-up experiment (see Scheme 9).



A mixture of 2-morpholinoaniline (1.07 g, 6 mmol), palladium(II) acetate (67.4 mg, 0.3 mmol), TPPMS (218.6 mg, 0.6 mmol), and benzyl alcohol (**2a**, 3.09 mL, 30 mmol) in H_2O (24 mL) was heated at 100 °C for 16 h under Ar. After cooling, the reaction mixture was poured into water and extracted with EtOAc. The organic layer was washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The residue was recrystallized from hexane/AcOEt to give desired product **3a** (1.63 g, 4.55 mmol, 76%) as a pale yellow solid.

After 10 min

After 16 h



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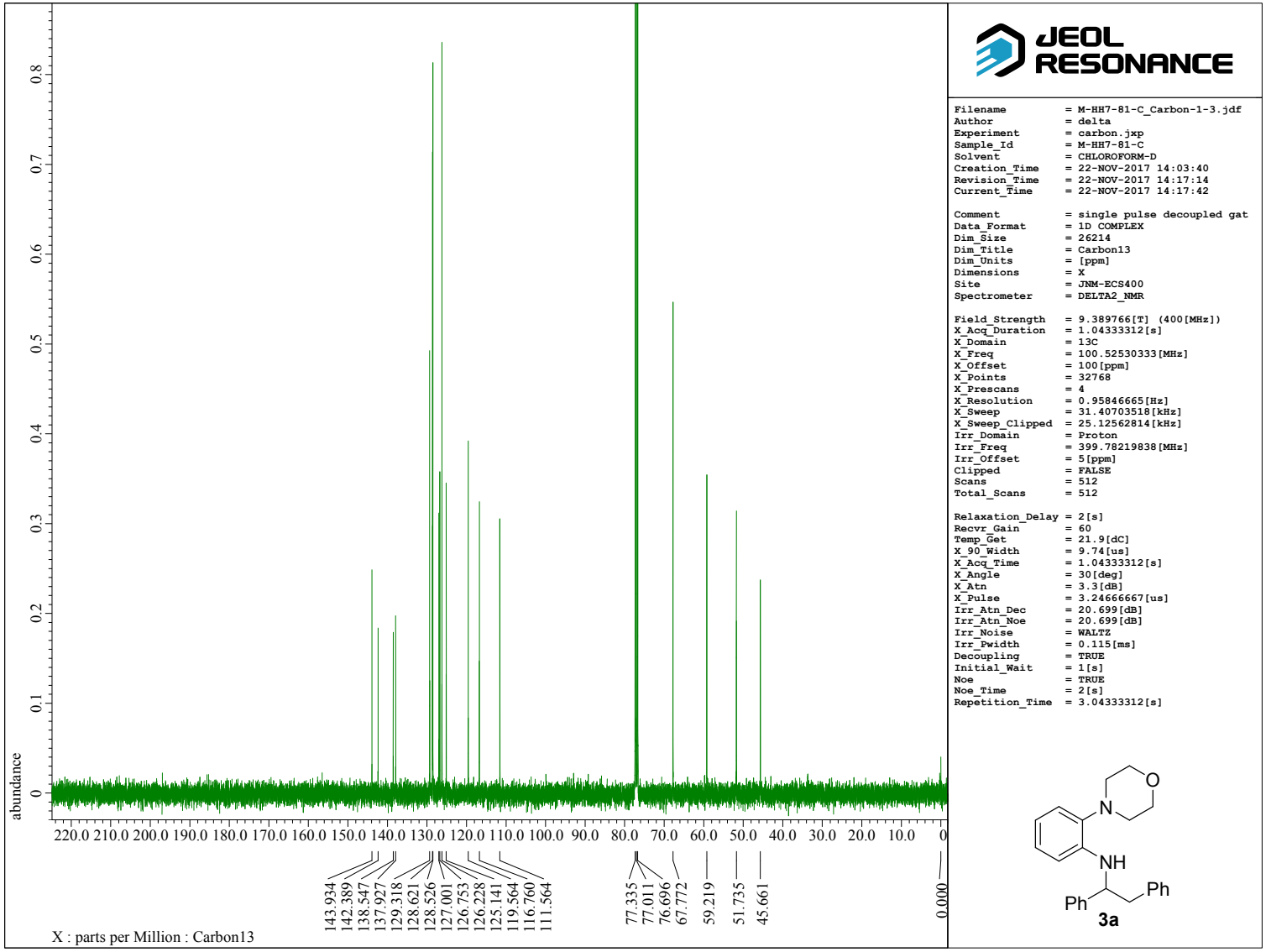
3a



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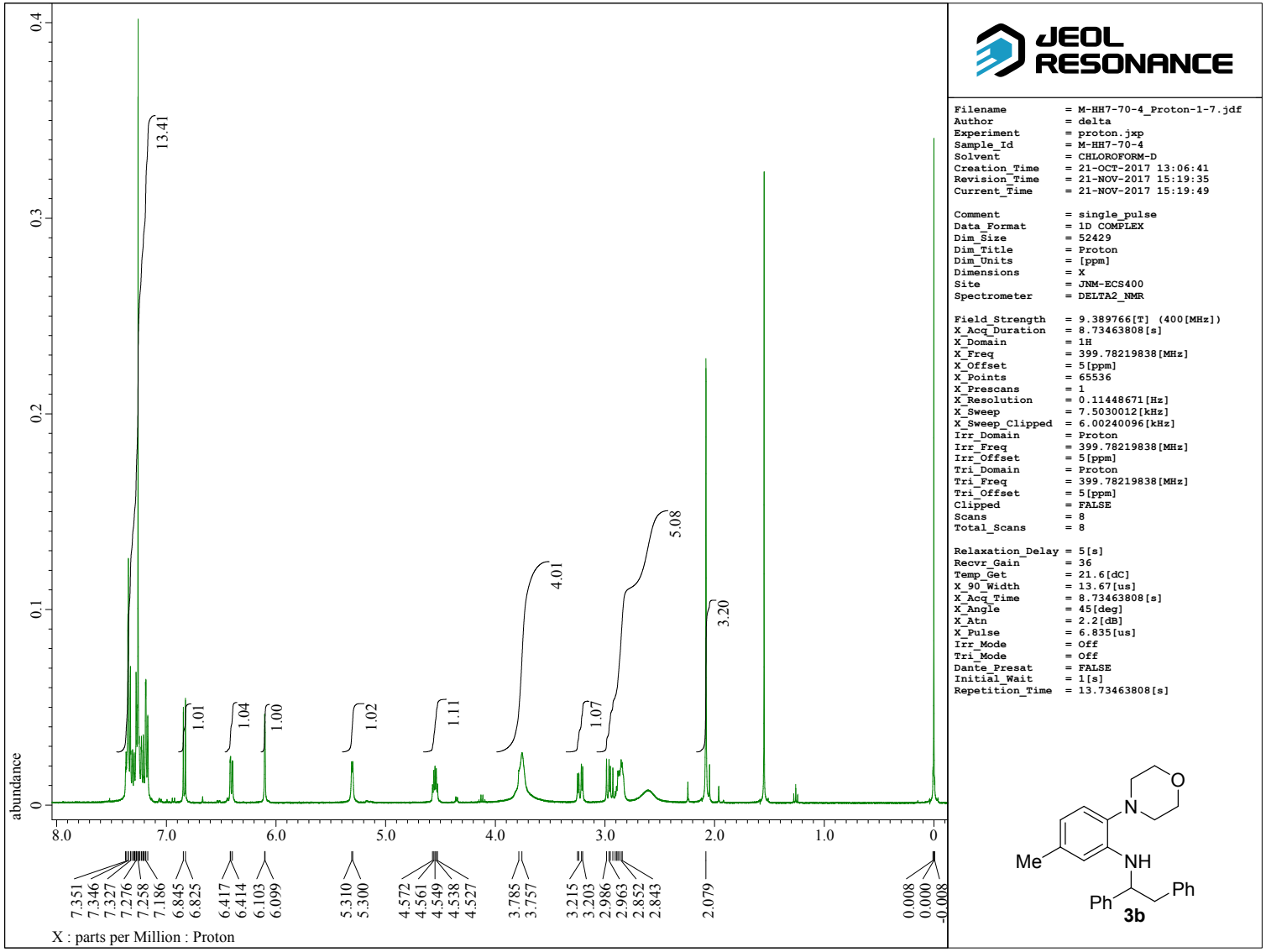
3a



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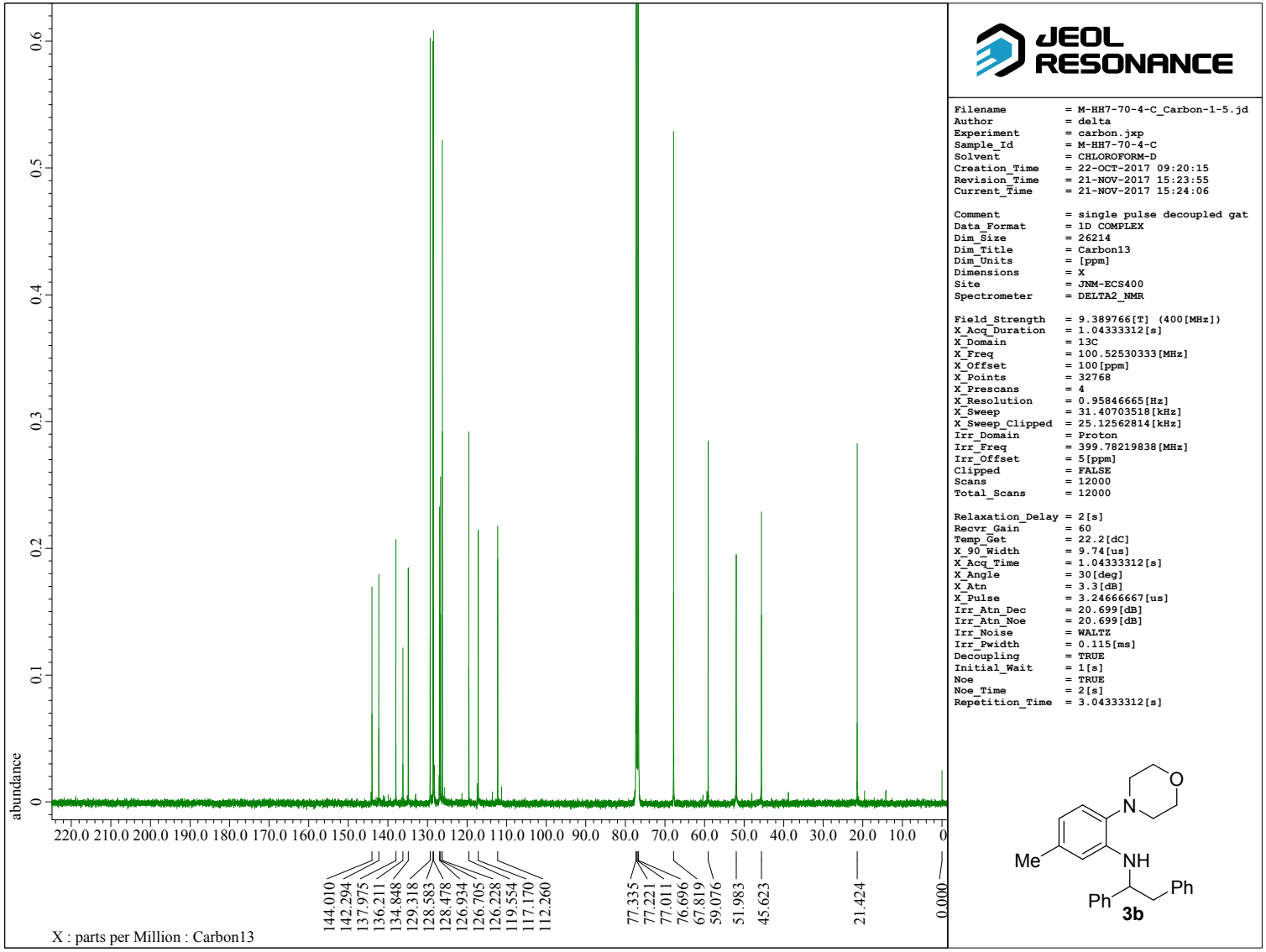
3b



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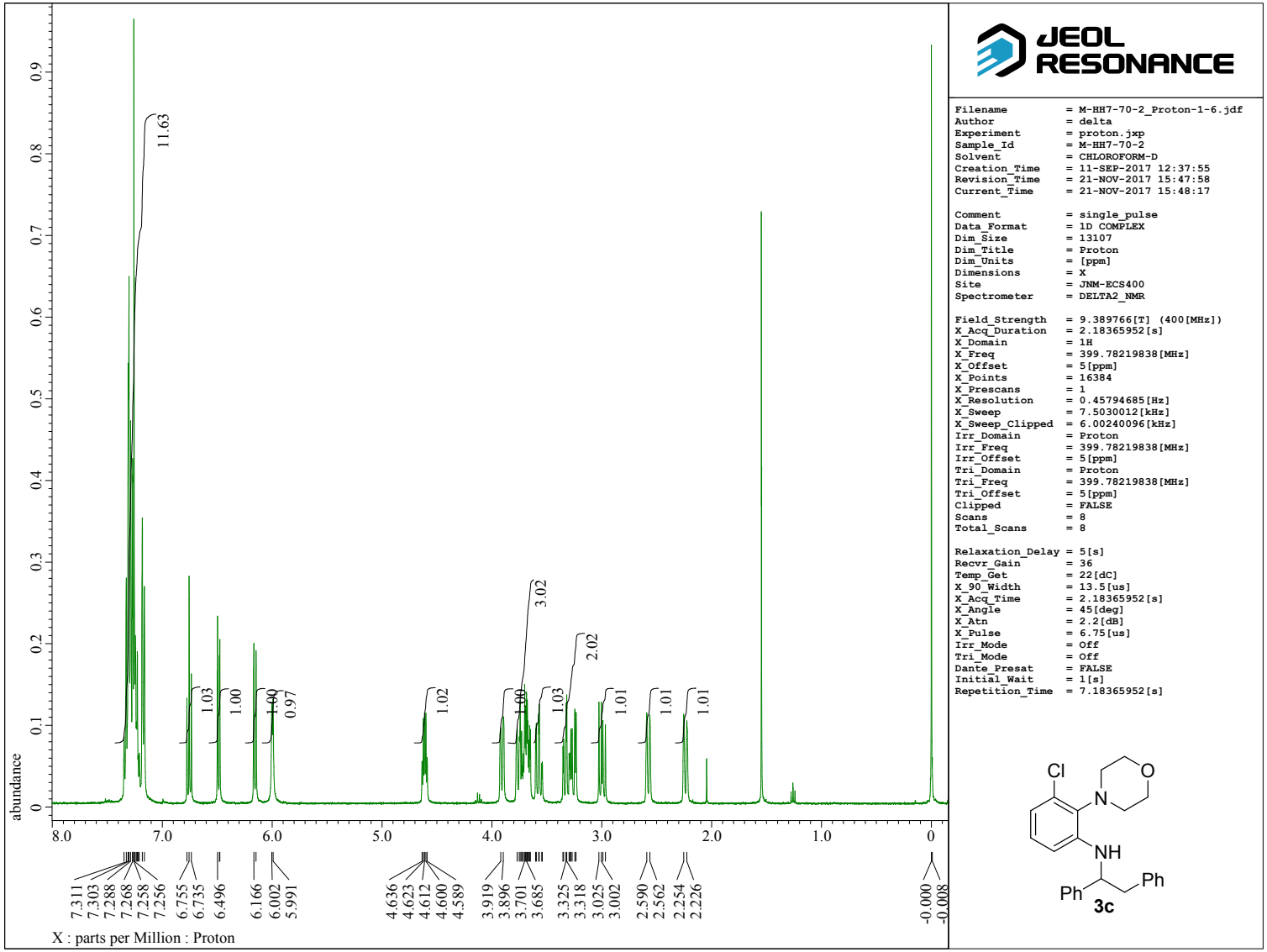
3b



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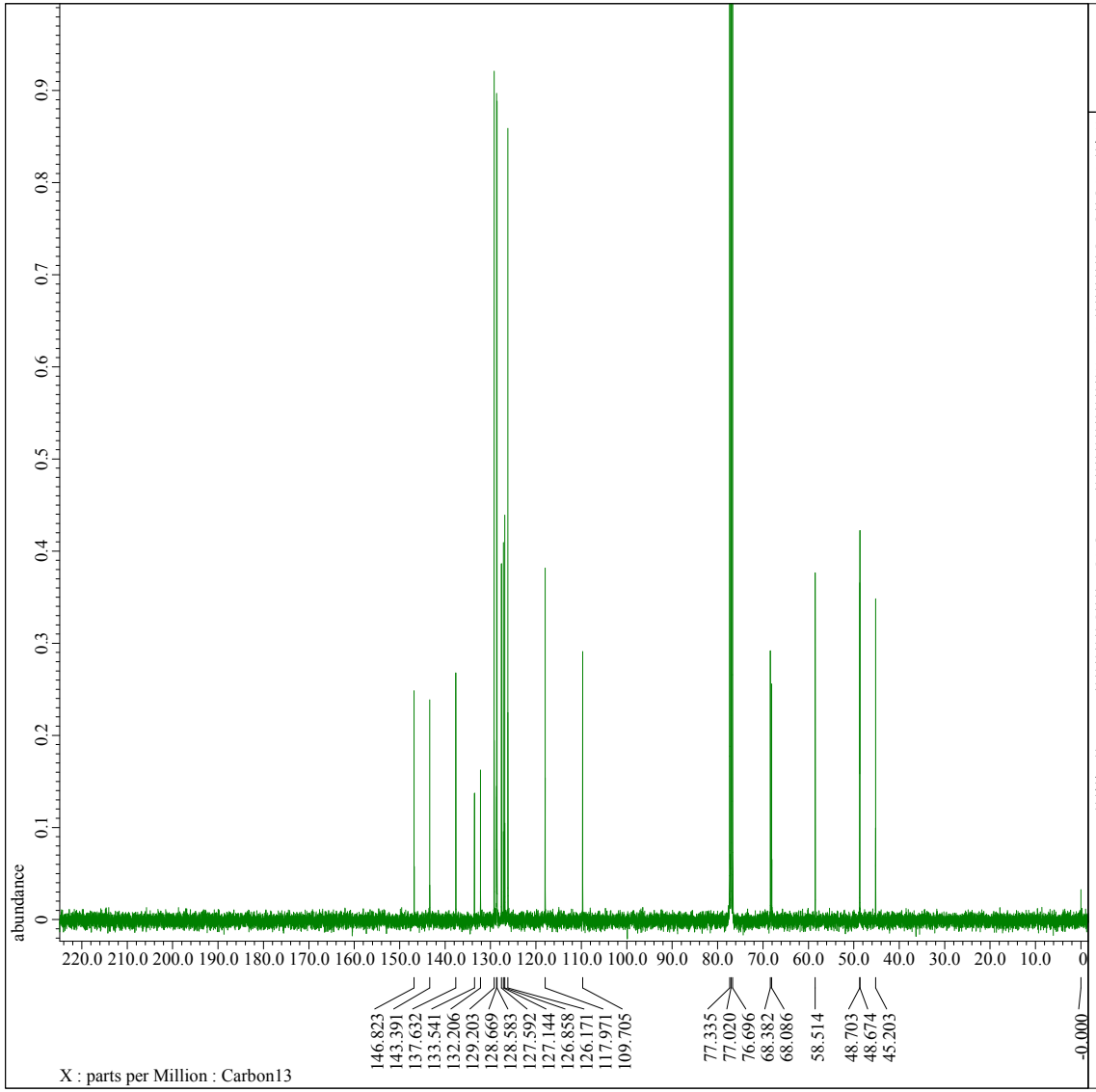

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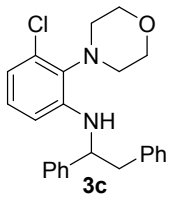

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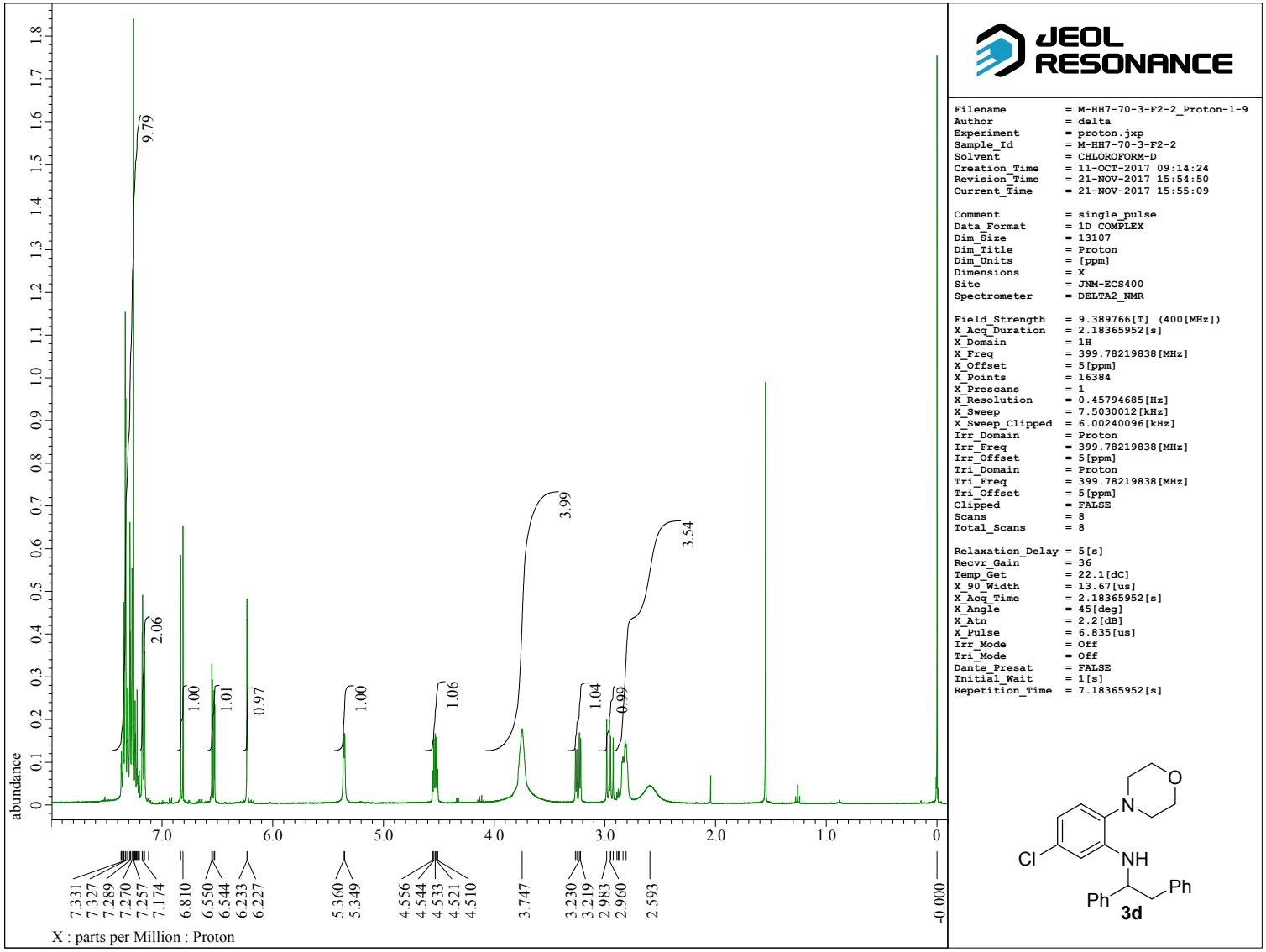

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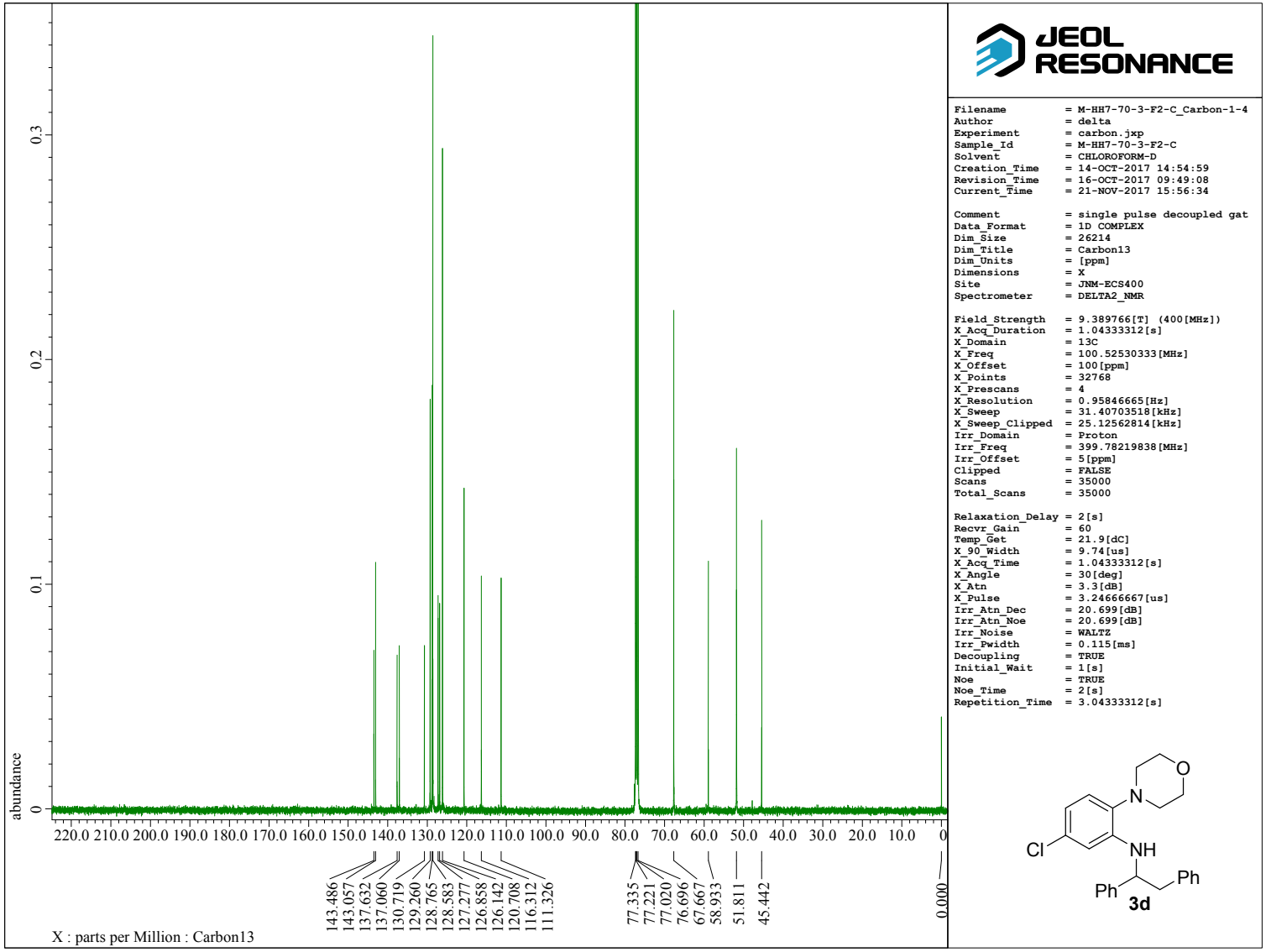

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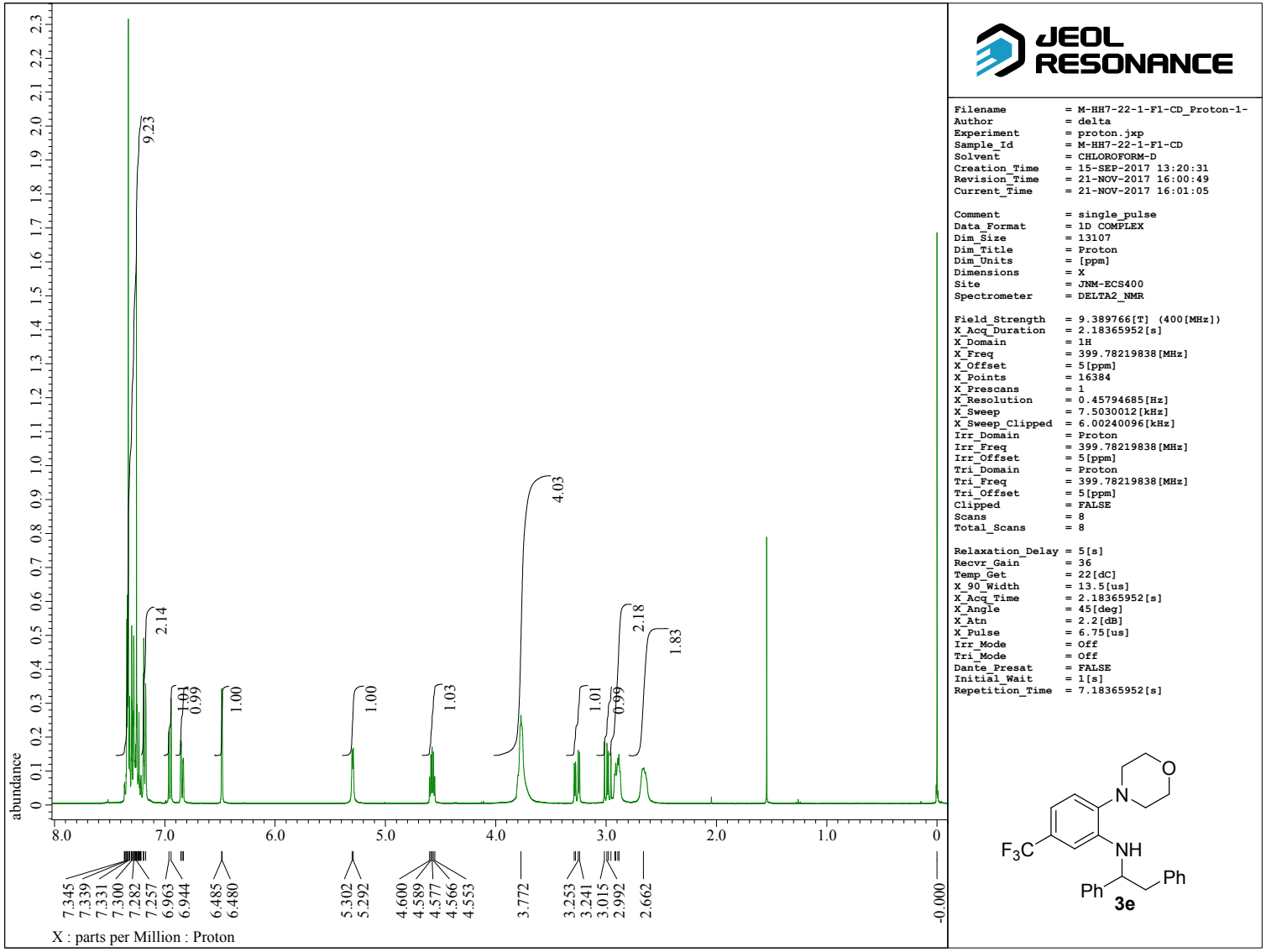
Sample_Id         = M-HH7-70-3-F2-C
Solvent           = CHLOROFORM-D
Creation_Time     = 14-OCT-2017 14:54:59
Revision_Time     = 16-OCT-2017 09:49:08
Current_Time      = 21-NOV-2017 15:56:34

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    = 9.389766[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             = 35000
Total_Scans       = 35000

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 21.9[dC]
X_90_Width        = 9.74[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 3.3[dB]
X_Pulse           = 3.24666667[us]
Irr_Atn_Dec       = 20.699[dB]
Irr_Atn_Noe       = 20.699[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```

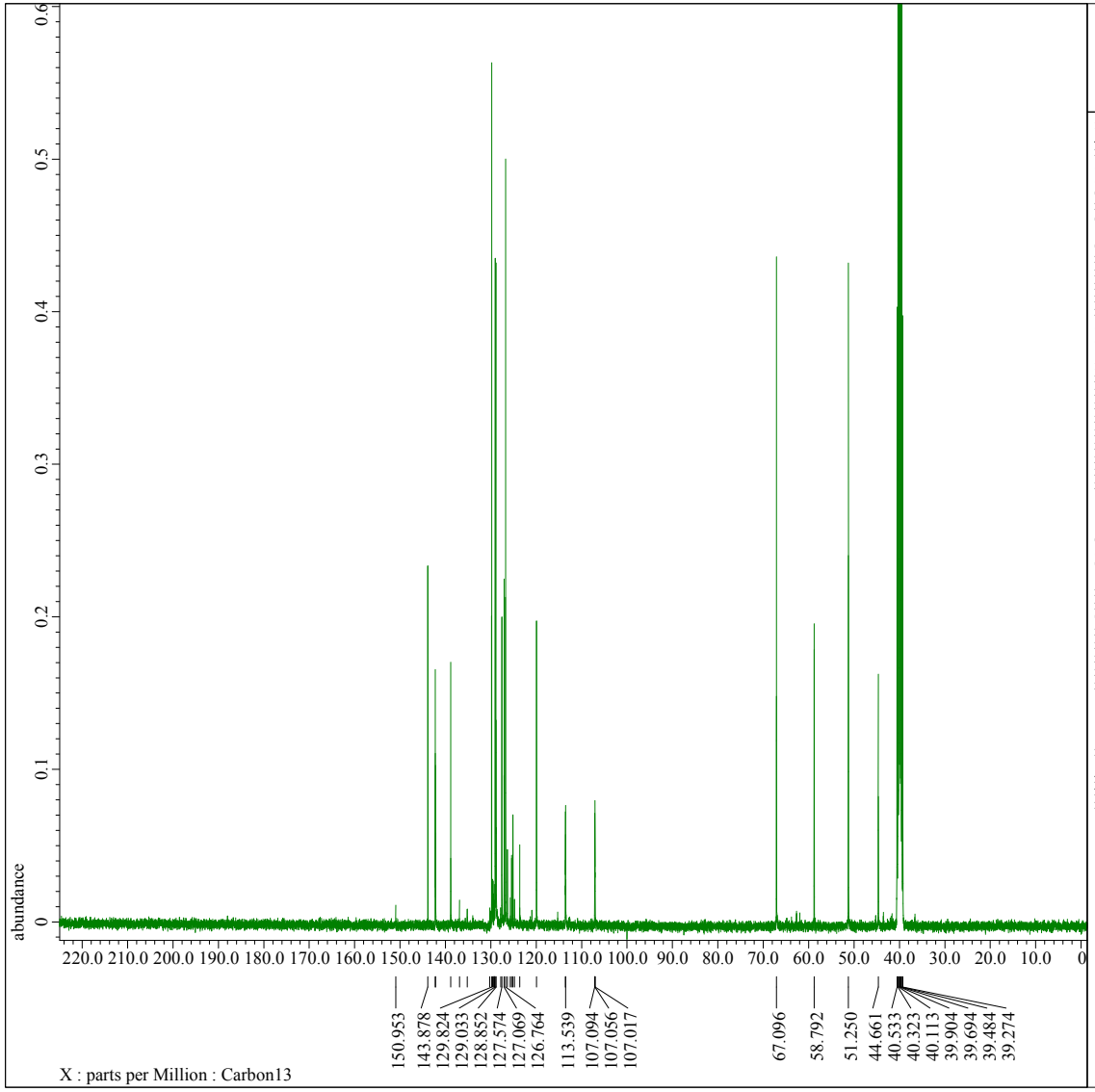

3d



JEOL RESONANCE

Filename	= M-HH7-22-1-F1-CD_Proton-1-
Author	= delta
Experiment	= proton.jxp
Sample_Id	= M-HH7-22-1-F1-CD
Solvent	= CHLOROFORM-D
Creation_Time	= 15-SEP-2017 13:20:31
Revision_Time	= 21-NOV-2017 16:00:49
Current_Time	= 21-NOV-2017 16:01:05
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	= 9.389766[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	= 36
Temp_Get	= 22[dC]
X_90_Width	= 13.5[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2.2[dB]
X_Pulse	= 6.75[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]

3e



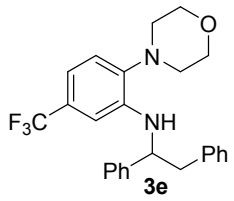
JEOL RESONANCE

```
Filename          = M-HH7-22-1-F1_Carbon-1-3.j
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = M-HH7-22-1-F1
Solvent           = DMSO-D6
Creation_Time     = 26-SEP-2017 23:33:13
Revision_Time     = 21-NOV-2017 16:03:01
Current_Time      = 21-NOV-2017 16:03:26

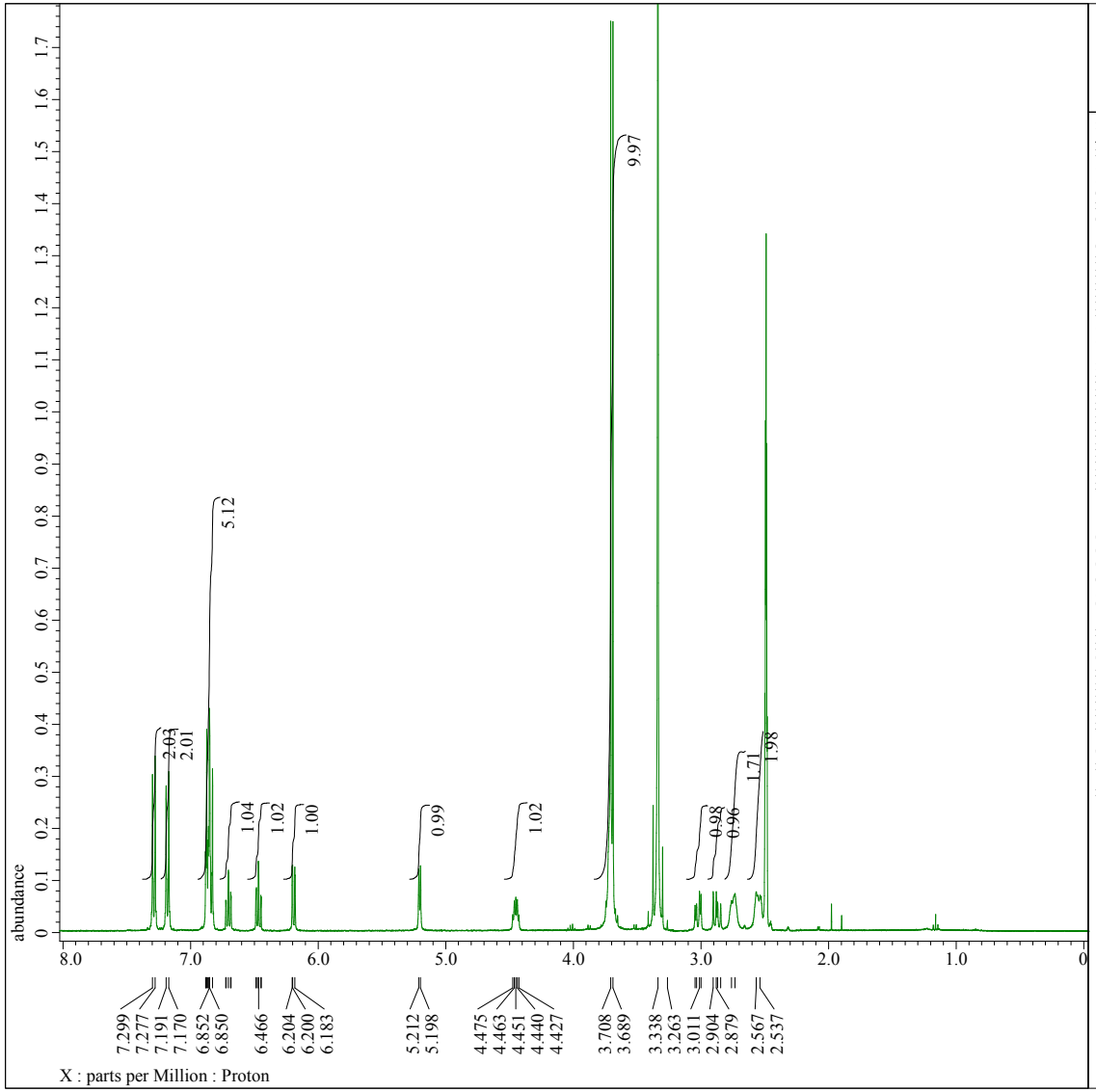
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[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             = 8000
Total_Scans       = 8000

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.4[dC]
X_90_Width        = 9.74[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 3.3[dB]
X_Pulse           = 3.24666667[us]
Irr_Atn_Dec       = 20.699[dB]
Irr_Atn_Noe       = 20.699[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```

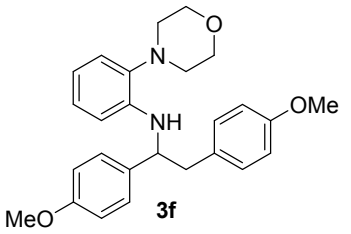


3e

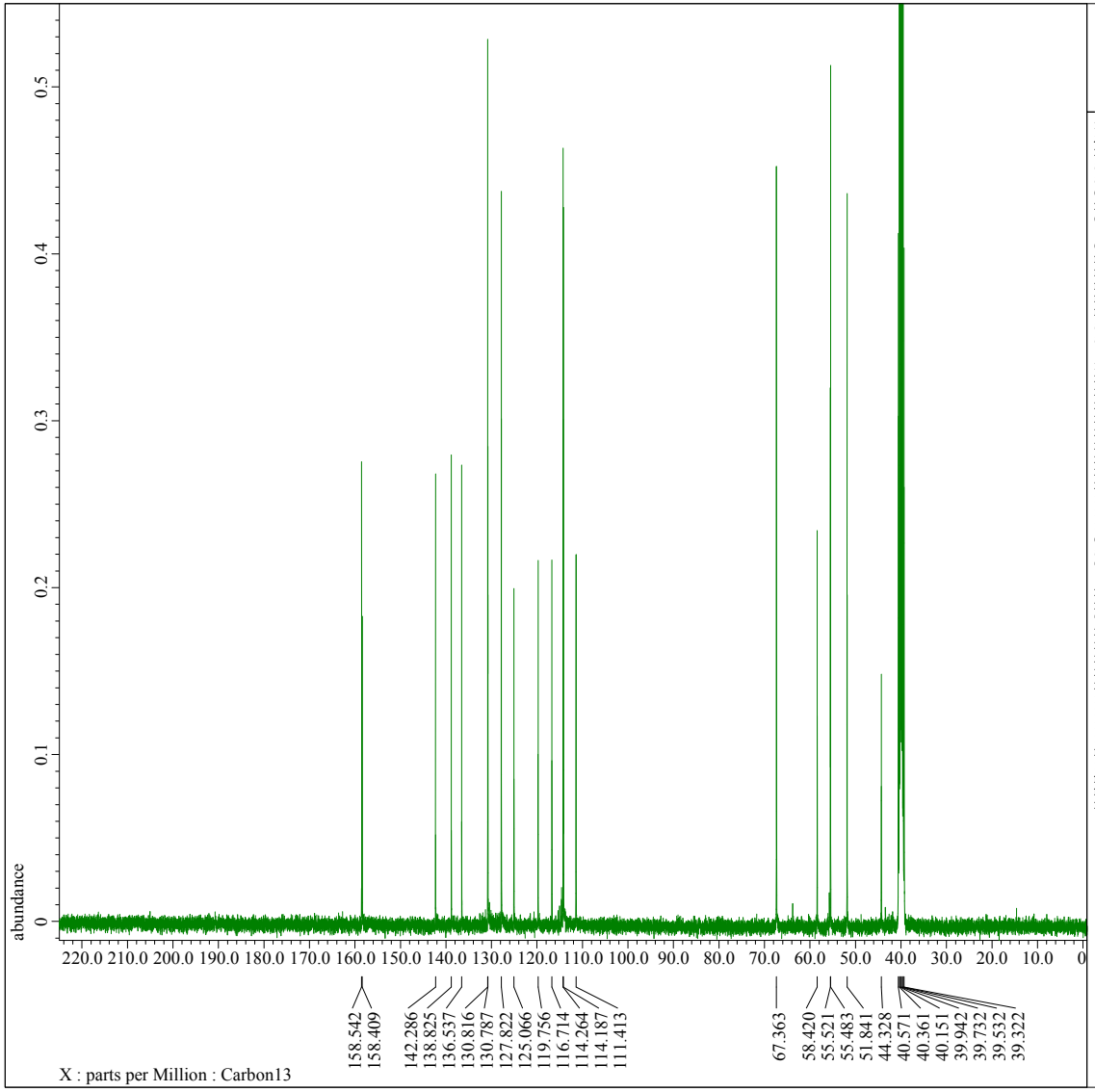


JEOL RESONANCE

Filename	= M-HH7-21-1-F2_Proton-1-4.j
Author	= delta
Experiment	= proton.jxp
Sample_Id	= M-HH6-21-1-F2
Solvent	= DMSO-D6
Creation_Time	= 11-NOV-2016 13:59:50
Revision_Time	= 21-NOV-2017 16:08:01
Current_Time	= 21-NOV-2017 16:08:20
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	= 9.389766[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	= 34
Temp_Get	= 20.2[dC]
X_90_Width	= 13.1[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2.2[dB]
X_Pulse	= 6.55[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]



3f



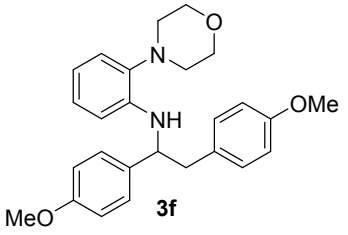
JEOL RESONANCE

```
Filename          = M-HH7-21-1-F2-C_Carbon-1-4
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = M-HH7-21-1-F2-C
Solvent           = DMSO-D6
Creation_Time     =  2-OCT-2017 02:44:43
Revision_Time     =  2-OCT-2017 12:59:03
Current_Time      = 21-NOV-2017 16:09:19

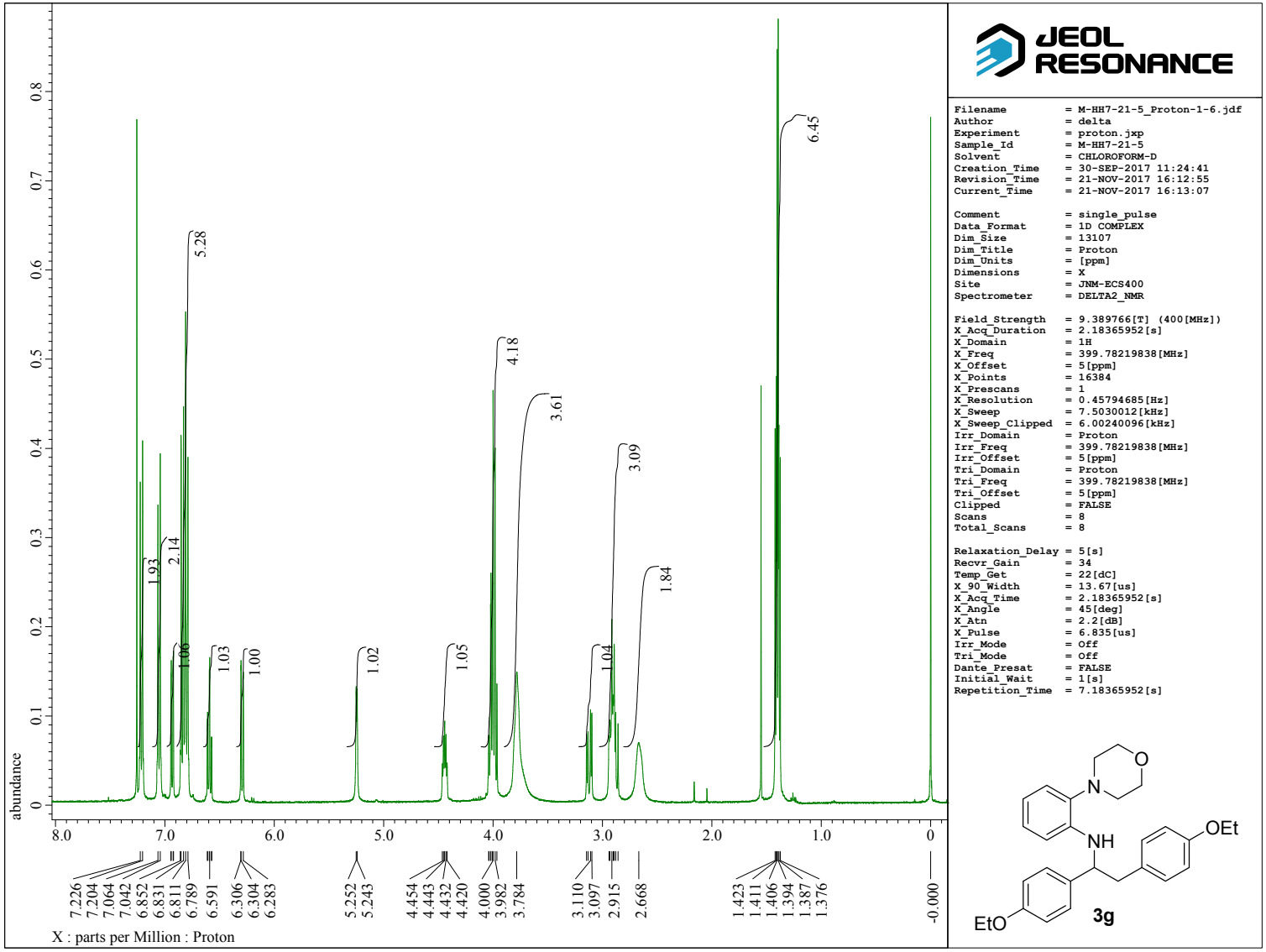
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[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             = 4000
Total_Scans       = 4000

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.2[dC]
X_90_Width        = 9.74[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 3.3[dB]
X_Pulse           = 3.24666667[us]
Irr_Atn_Dec       = 20.699[dB]
Irr_Atn_Noe       = 20.699[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



3f



JEOL RESONANCE

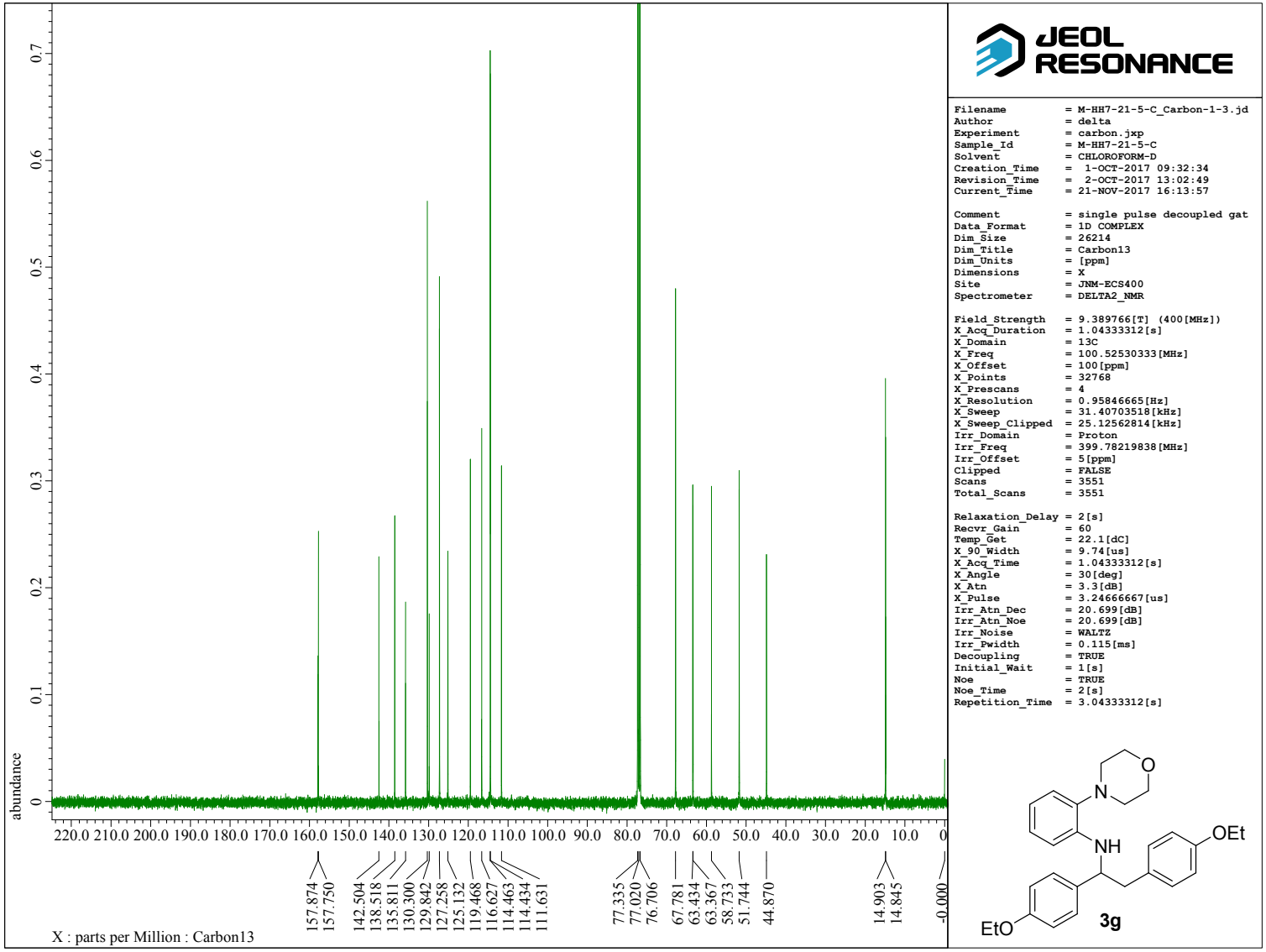
```
Filename          = M-HH7-21-5_Proton-1-6.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = M-HH7-21-5
Solvent           = CHLOROFORM-D
Creation_Time     = 30-SEP-2017 11:24:41
Revision_Time     = 21-NOV-2017 16:12:55
Current_Time      = 21-NOV-2017 16:13:07

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    = 9.389766[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        = 34
Temp_Get          = 22[dC]
X_90_Width        = 13.67[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2.2[dB]
X_Pulse           = 6.835[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```


3g



JEOL RESONANCE

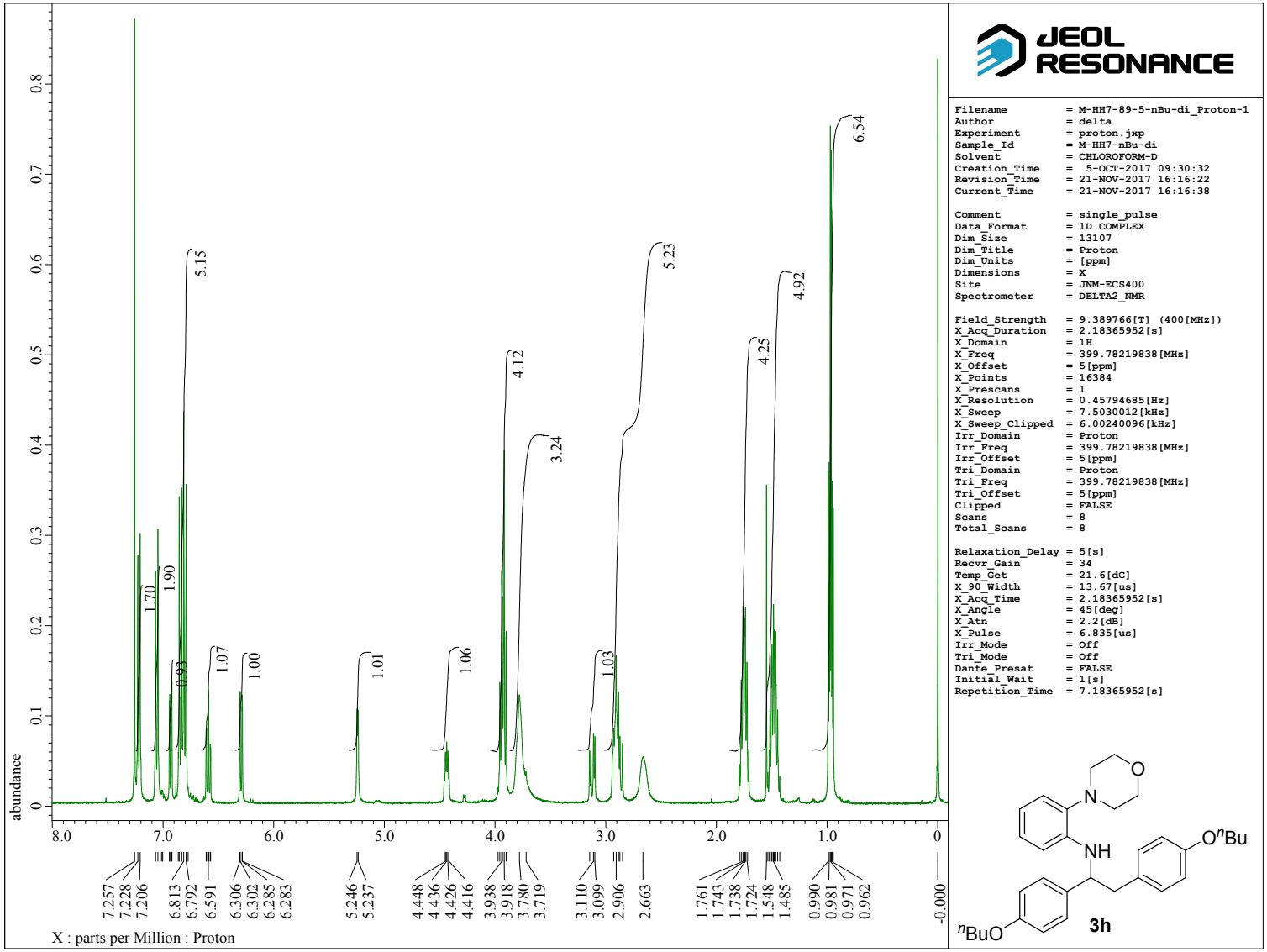
```
Filename          = M-HH7-21-5-C_Carbon-1-3.jd
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = M-HH7-21-5-C
Solvent           = CHLOROFORM-D
Creation_Time     =  1-OCT-2017 09:32:34
Revision_Time     =  2-OCT-2017 13:02:49
Current_Time      = 21-NOV-2017 16:13:57

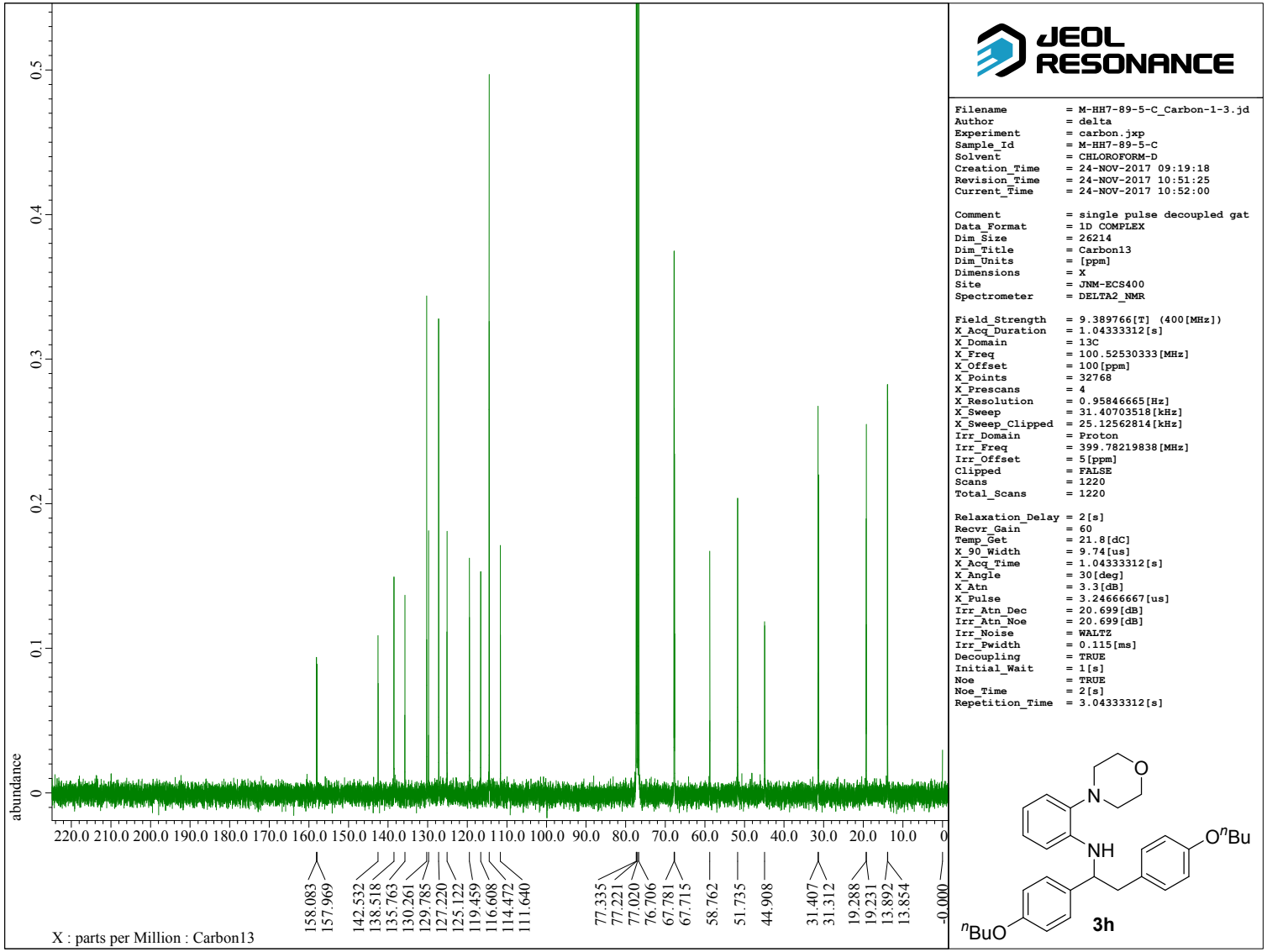
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[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             = 3551
Total_Scans       = 3551

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.1[dC]
X_90_Width        = 9.74[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 3.3[dB]
X_Pulse           = 3.24666667[us]
Irr_Atn_Dec       = 20.699[dB]
Irr_Atn_Noe       = 20.699[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```


3g


abundance
X : parts per Million : Proton
JEOL RESONANCE
Filename = M-HH7-89-5-nBu-di_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = M-HH7-nBu-di
Solvent = CHLOROFORM-D
Creation_Time = 5-OCT-2017 09:30:32
Revision_Time = 21-NOV-2017 16:16:22
Current_Time = 21-NOV-2017 16:16:38
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 = 9.389766[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 = 34
Temp_Get = 21.6[dC]
X_90_Width = 13.67[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2.2[dB]
X_Pulse = 6.835[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
3h



JEOL RESONANCE

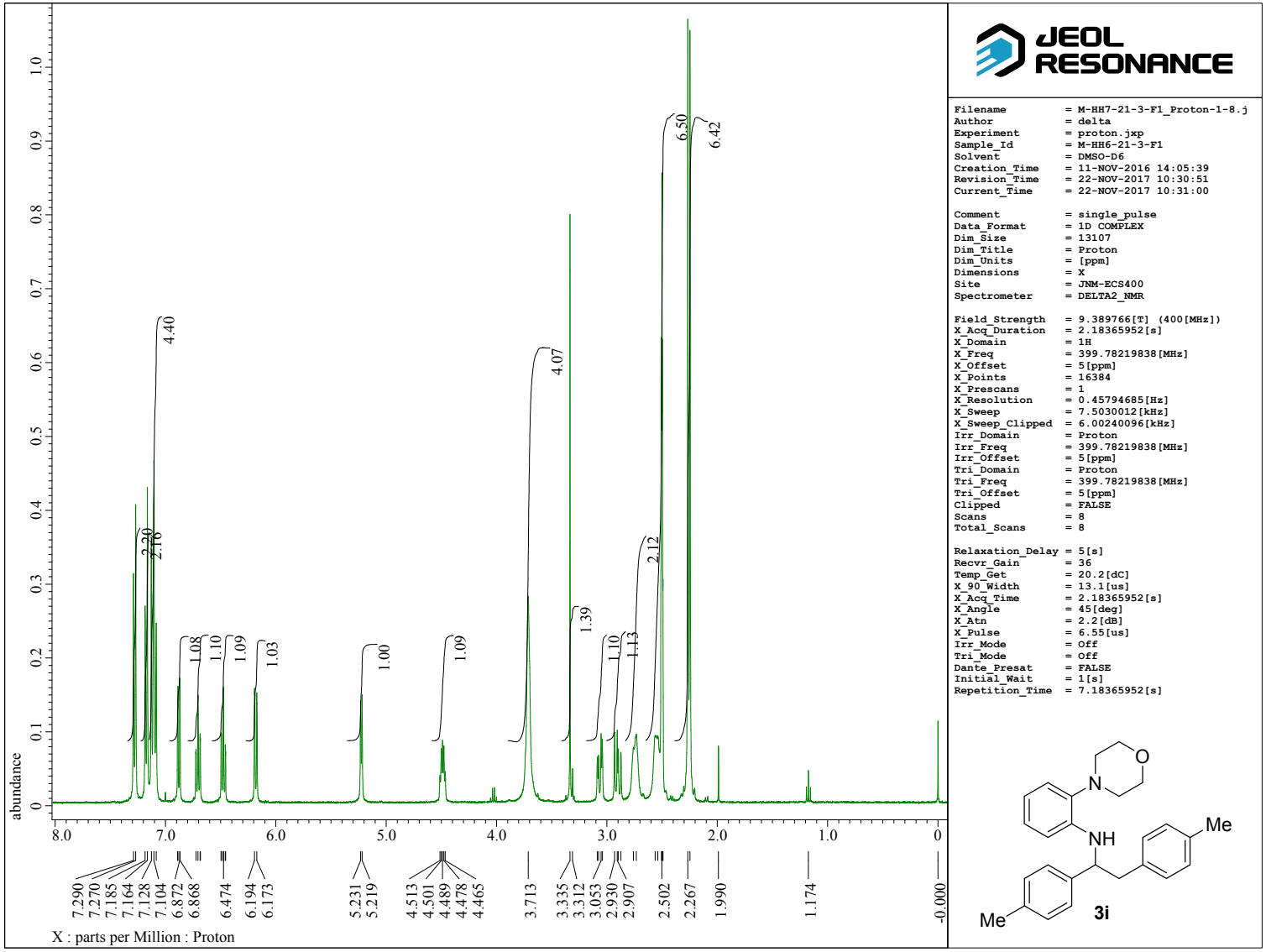
```
Filename          = M-HH7-89-5-C_Carbon-1-3.jd
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = M-HH7-89-5-C
Solvent           = CHLOROFORM-D
Creation_Time     = 24-NOV-2017 09:19:18
Revision_Time     = 24-NOV-2017 10:51:25
Current_Time      = 24-NOV-2017 10:52:00

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    = 9.389766[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             = 1220
Total_Scans       = 1220

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 21.8[dC]
X_90_Width        = 9.74[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 3.3[dB]
X_Pulse           = 3.24666667[us]
Irr_Atn_Dec       = 20.699[dB]
Irr_Atn_Noe       = 20.699[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```

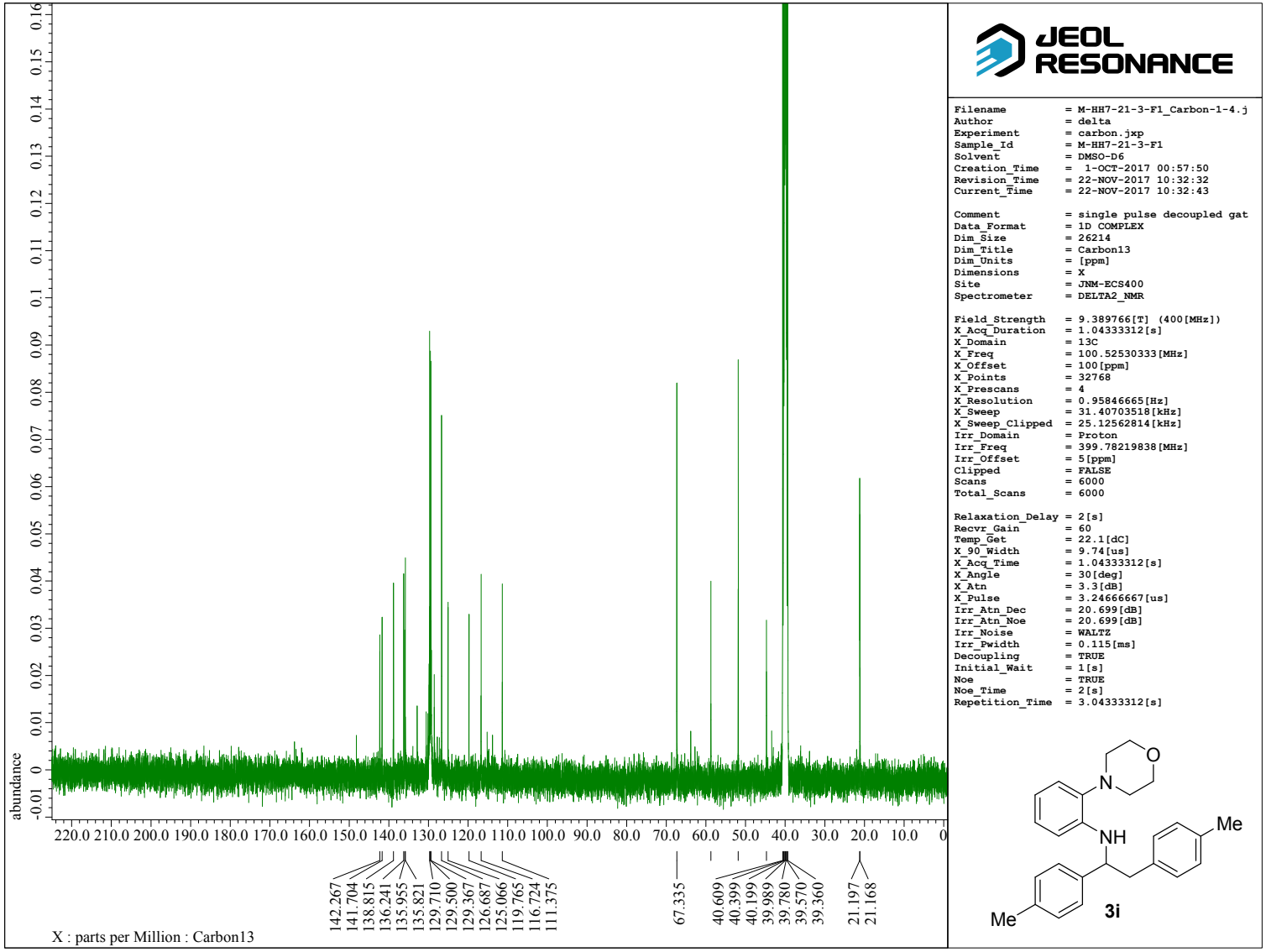

3h



JEOL RESONANCE

Filename	= M-HH7-21-3-F1_Proton-1-8.j
Author	= delta
Experiment	= proton.jxp
Sample_Id	= M-HH6-21-3-F1
Solvent	= DMSO-D6
Creation_Time	= 11-NOV-2016 14:05:39
Revision_Time	= 22-NOV-2017 10:30:51
Current_Time	= 22-NOV-2017 10:31:00
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	= 9.389766[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	= 36
Temp_Get	= 20.2[dC]
X_90_Width	= 13.1[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2.2[dB]
X_Pulse	= 6.55[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]

3i



JEOL RESONANCE

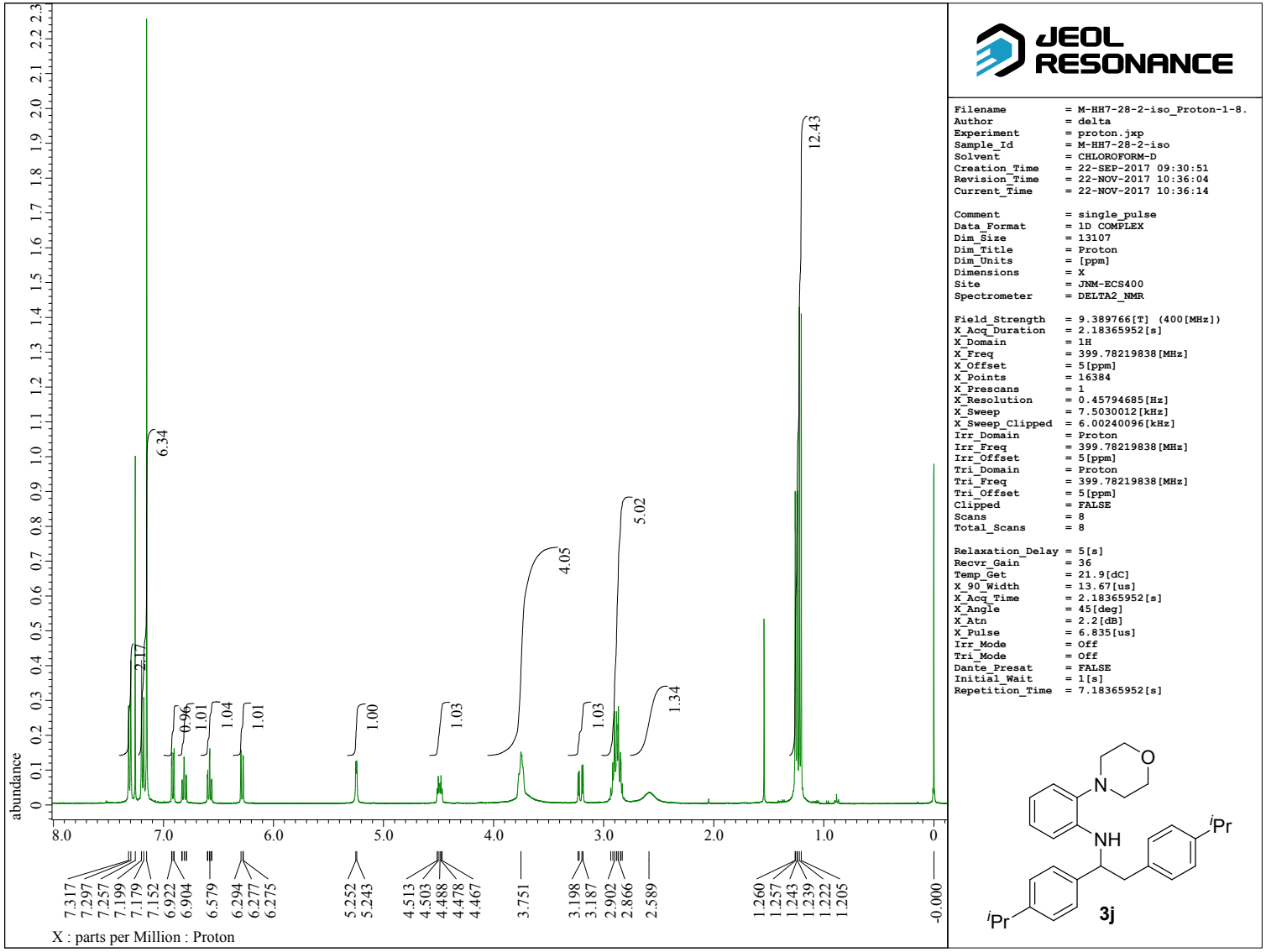
```
Filename          = M-HH7-21-3-F1_Carbon-1-4.j
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = M-HH7-21-3-F1
Solvent           = DMSO-D6
Creation_Time     =  1-OCT-2017 00:57:50
Revision_Time     = 22-NOV-2017 10:32:32
Current_Time      = 22-NOV-2017 10:32:43

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    = 9.389766[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             = 6000
Total_Scans       = 6000

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.1[dC]
X_90_Width        = 9.74[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 3.3[dB]
X_Pulse           = 3.24666667[us]
Irr_Atn_Dec       = 20.699[dB]
Irr_Atn_Noe       = 20.699[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```

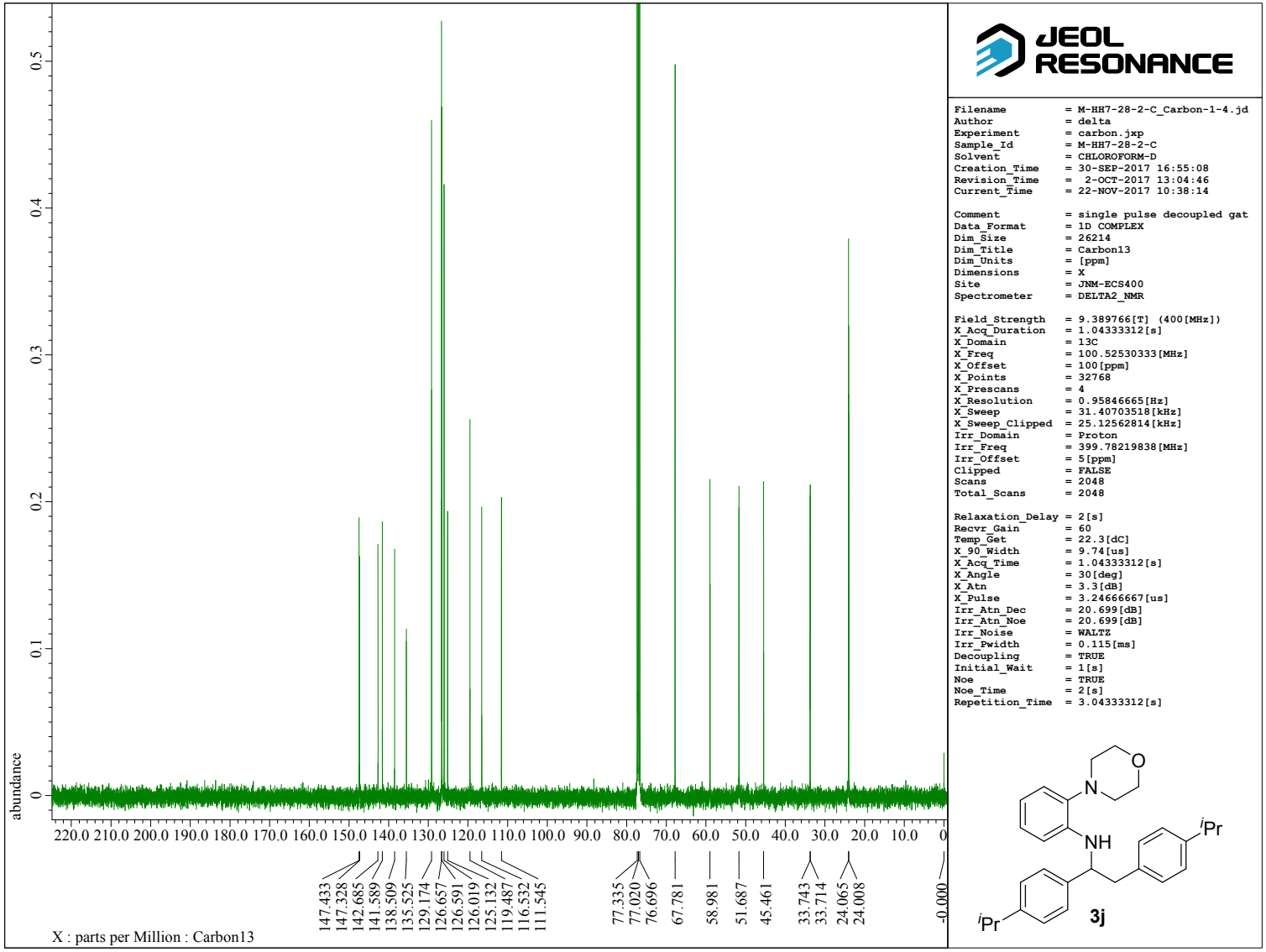

3i



JEOL RESONANCE

Filename	= M-HH7-28-2-iso_Proton-1-8.
Author	= delta
Experiment	= proton.jxp
Sample_Id	= M-HH7-28-2-iso
Solvent	= CHLOROFORM-D
Creation_Time	= 22-SEP-2017 09:30:51
Revision_Time	= 22-NOV-2017 10:36:04
Current_Time	= 22-NOV-2017 10:36:14
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	= 9.389766[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	= 36
Temp_Get	= 21.9[dC]
X_90_Width	= 13.67[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2.2[dB]
X_Pulse	= 6.835[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]

3j



JEOL RESONANCE

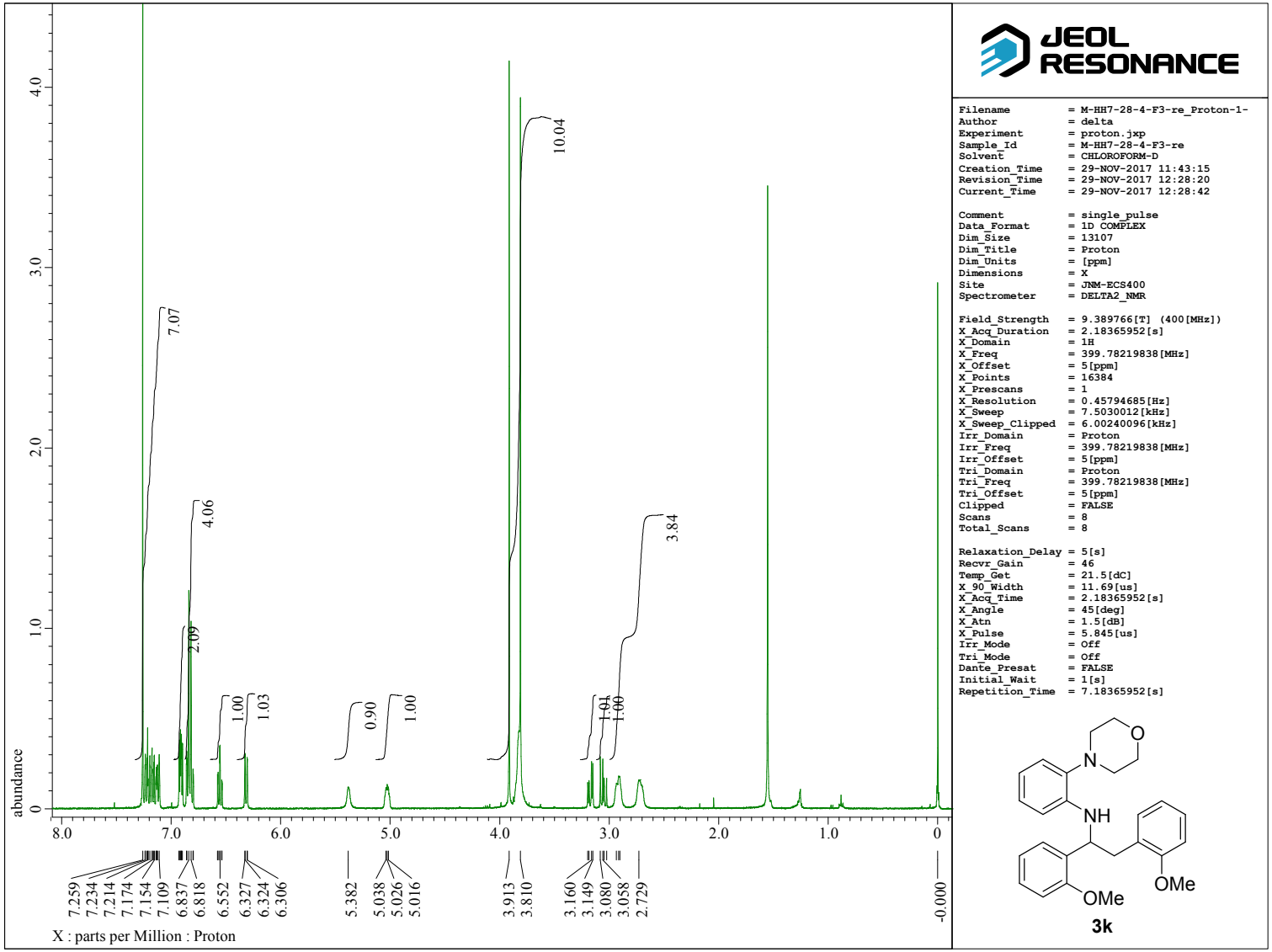
```
Filename          = M-HH7-28-2-C_Carbon-1-4.jd
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = M-HH7-28-2-C
Solvent           = CHLOROFORM-D
Creation_Time     = 30-SEP-2017 16:55:08
Revision_Time     =  2-OCT-2017 13:04:46
Current_Time      = 22-NOV-2017 10:38:14

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    = 9.389766[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             = 2048
Total_Scans       = 2048

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.3[dC]
X_90_Width        = 9.74[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 3.3[dB]
X_Pulse           = 3.24666667[us]
Irr_Atn_Dec       = 20.699[dB]
Irr_Atn_Noe       = 20.699[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```


3j



JEOL RESONANCE

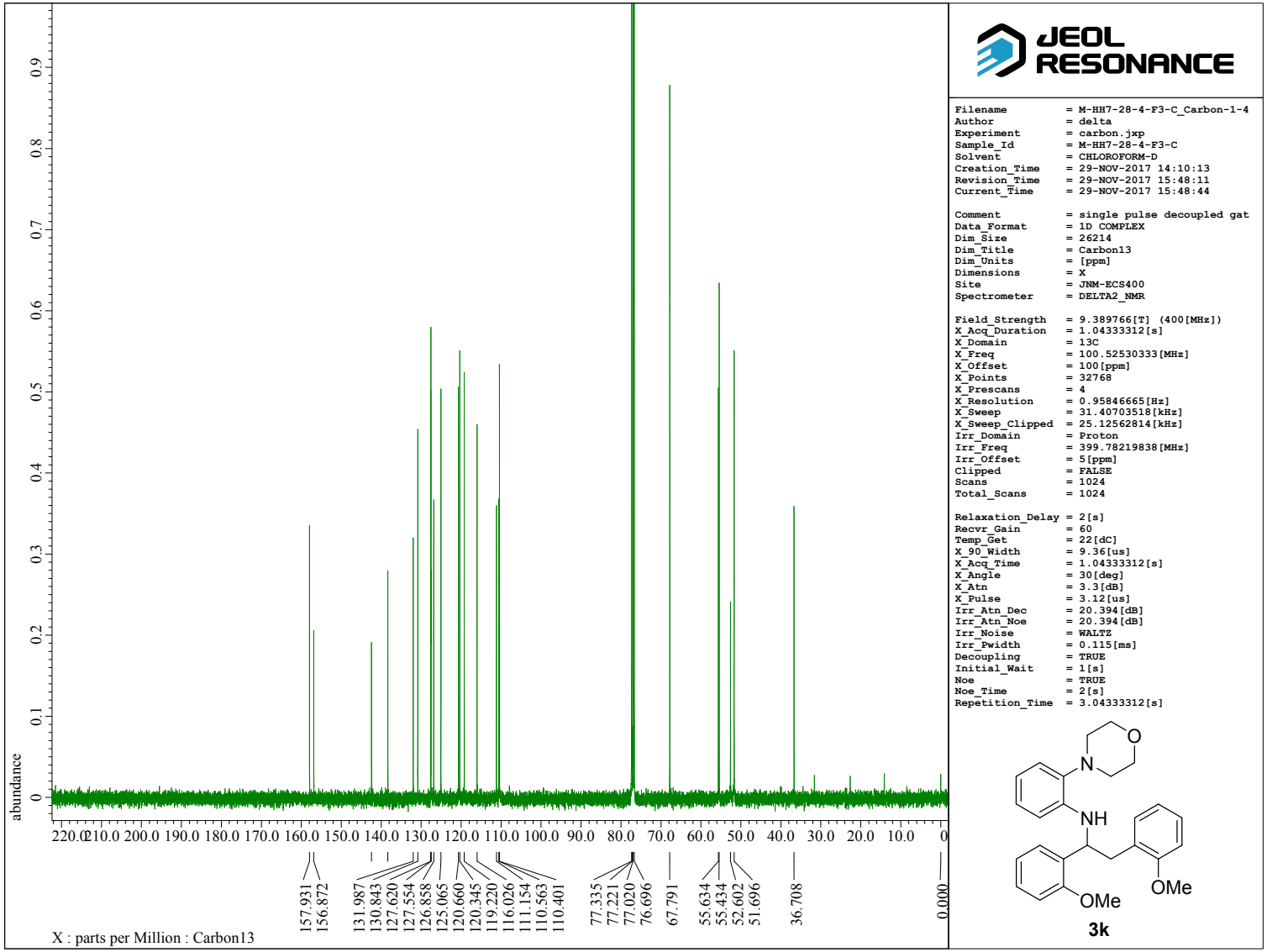
```
Filename          = M-HH7-28-4-F3-re_Proton-1-
Author            = delta
Experiment        = proton.jxp
Sample_Id         = M-HH7-28-4-F3-re
Solvent           = CHLOROFORM-D
Creation_Time     = 29-NOV-2017 11:43:15
Revision_Time     = 29-NOV-2017 12:28:20
Current_Time      = 29-NOV-2017 12:28:42

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    = 9.389766[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        = 46
Temp_Get          = 21.5[dC]
X_90_Width        = 11.69[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1.5[dB]
X_Pulse           = 5.845[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```


3k



JEOL RESONANCE

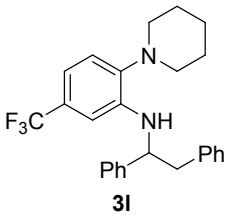
```
Filename          = M-HH7-28-4-F3-C_Carbon-1-4
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = M-HH7-28-4-F3-C
Solvent           = CHLOROFORM-D
Creation_Time     = 29-NOV-2017 14:10:13
Revision_Time     = 29-NOV-2017 15:48:11
Current_Time      = 29-NOV-2017 15:48:44

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

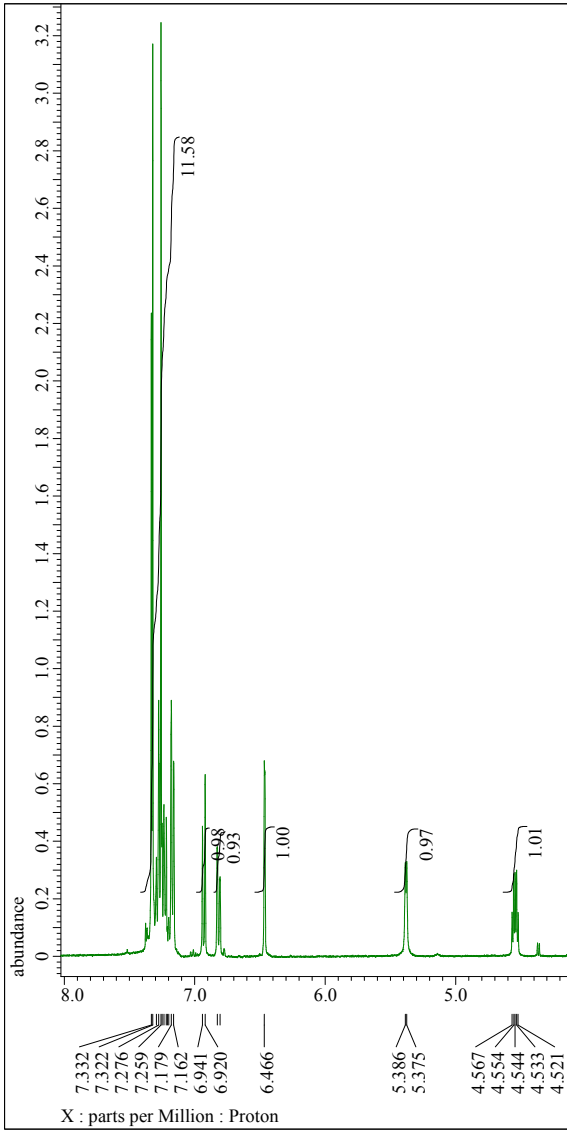
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

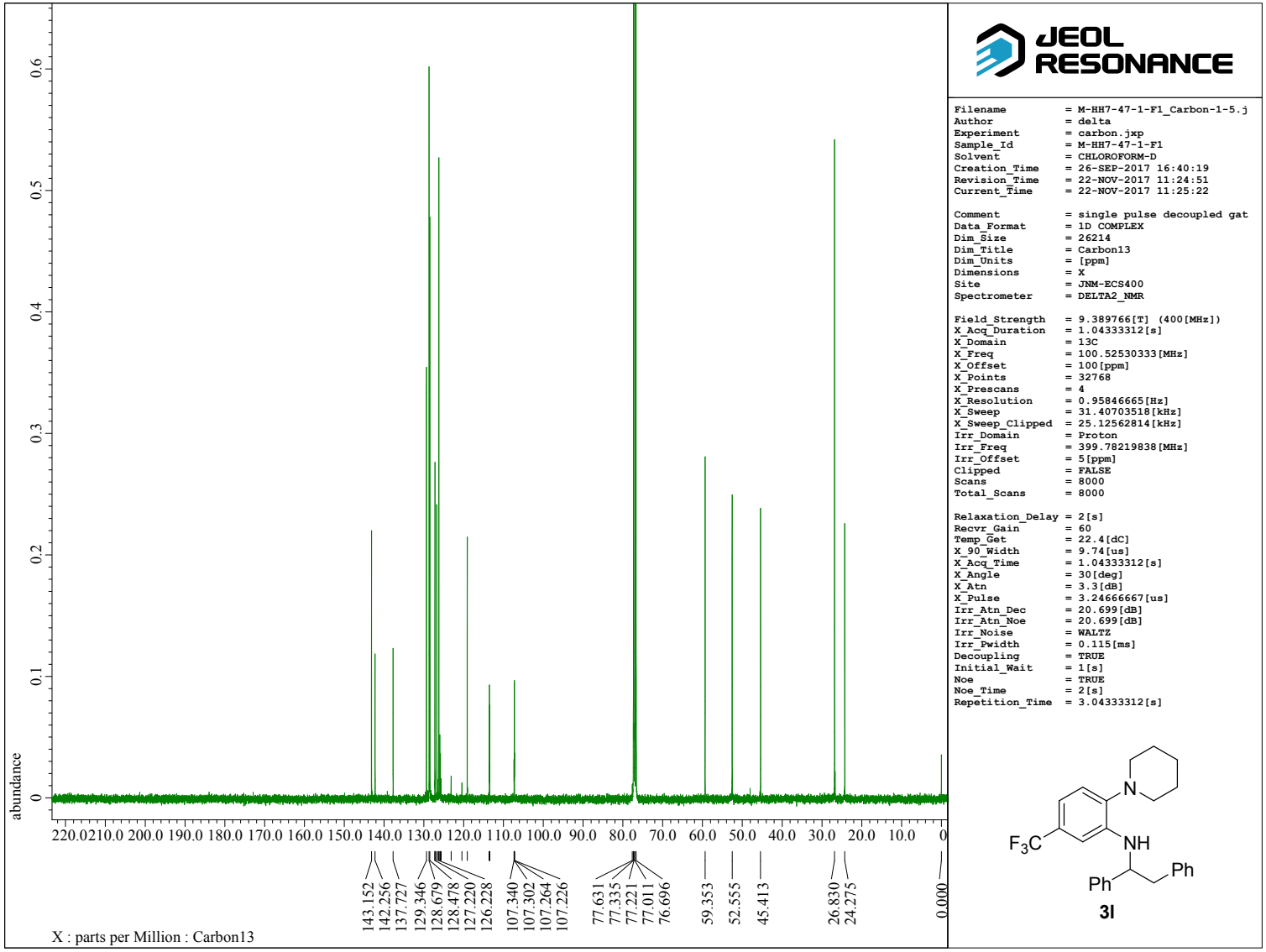
Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22[dC]
X_90_Width        = 9.36[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 3.3[dB]
X_Pulse           = 3.12[us]
Irr_Atn_Dec       = 20.394[dB]
Irr_Atn_Noe       = 20.394[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```


3k


N
F3C
NH
Ph
Ph

3l


abundance
11.58
0.98
0.93
1.00
0.97
1.01
8.0
7.0
6.0
5.0
7.332
7.322
7.276
7.259
7.179
7.162
6.941
6.920
6.466
5.386
5.375
4.567
4.554
4.544
4.533
4.521
X : parts per Million : Proton



JEOL RESONANCE

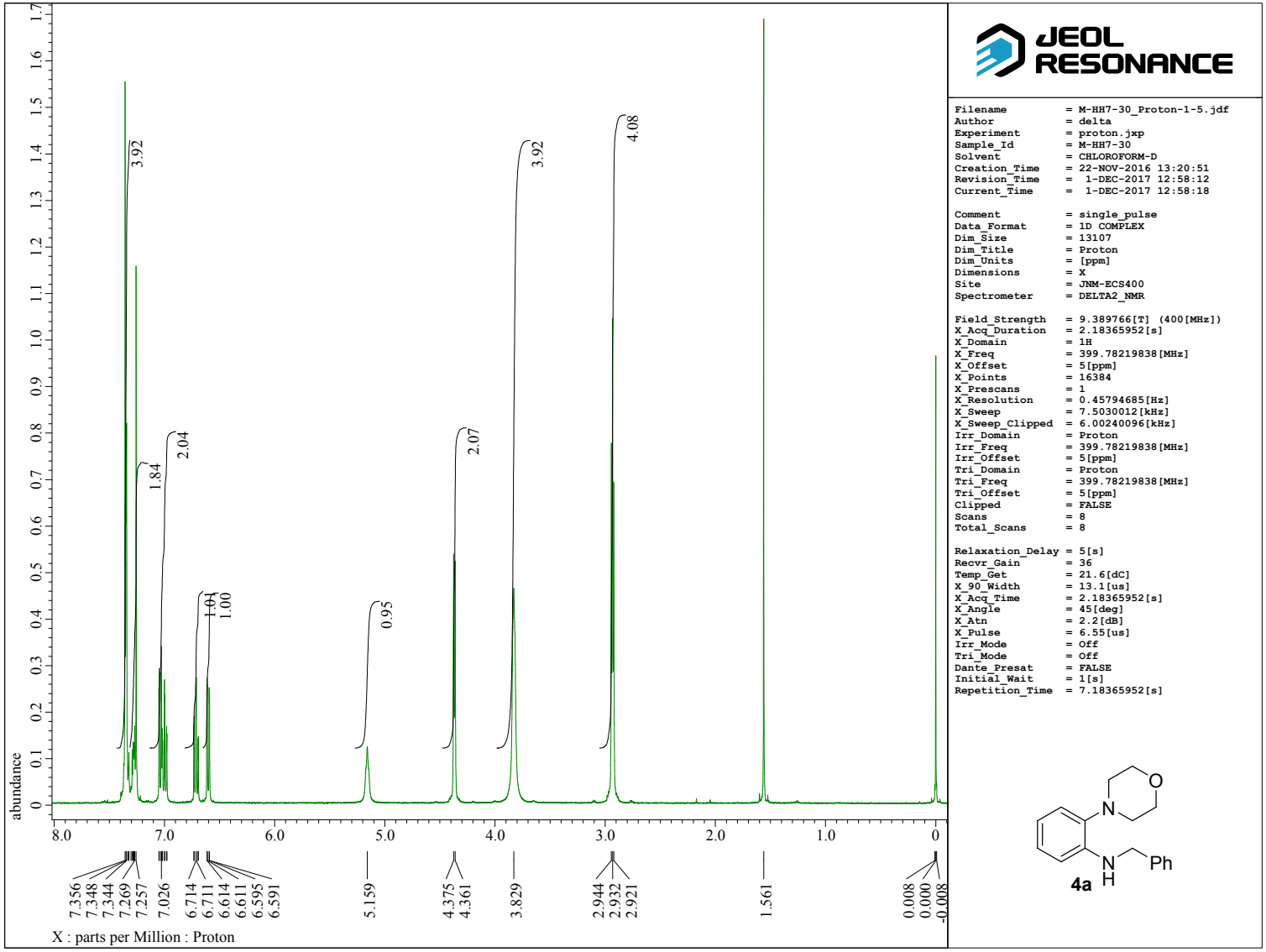
```
Filename          = M-HH7-47-1-F1_Carbon-1-5.j
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = M-HH7-47-1-F1
Solvent           = CHLOROFORM-D
Creation_Time     = 26-SEP-2017 16:40:19
Revision_Time     = 22-NOV-2017 11:24:51
Current_Time      = 22-NOV-2017 11:25:22

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    = 9.389766[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             = 8000
Total_Scans       = 8000

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.4[dC]
X_90_Width        = 9.74[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 3.3[dB]
X_Pulse           = 3.24666667[us]
Irr_Atn_Dec       = 20.699[dB]
Irr_Atn_Noe       = 20.699[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```

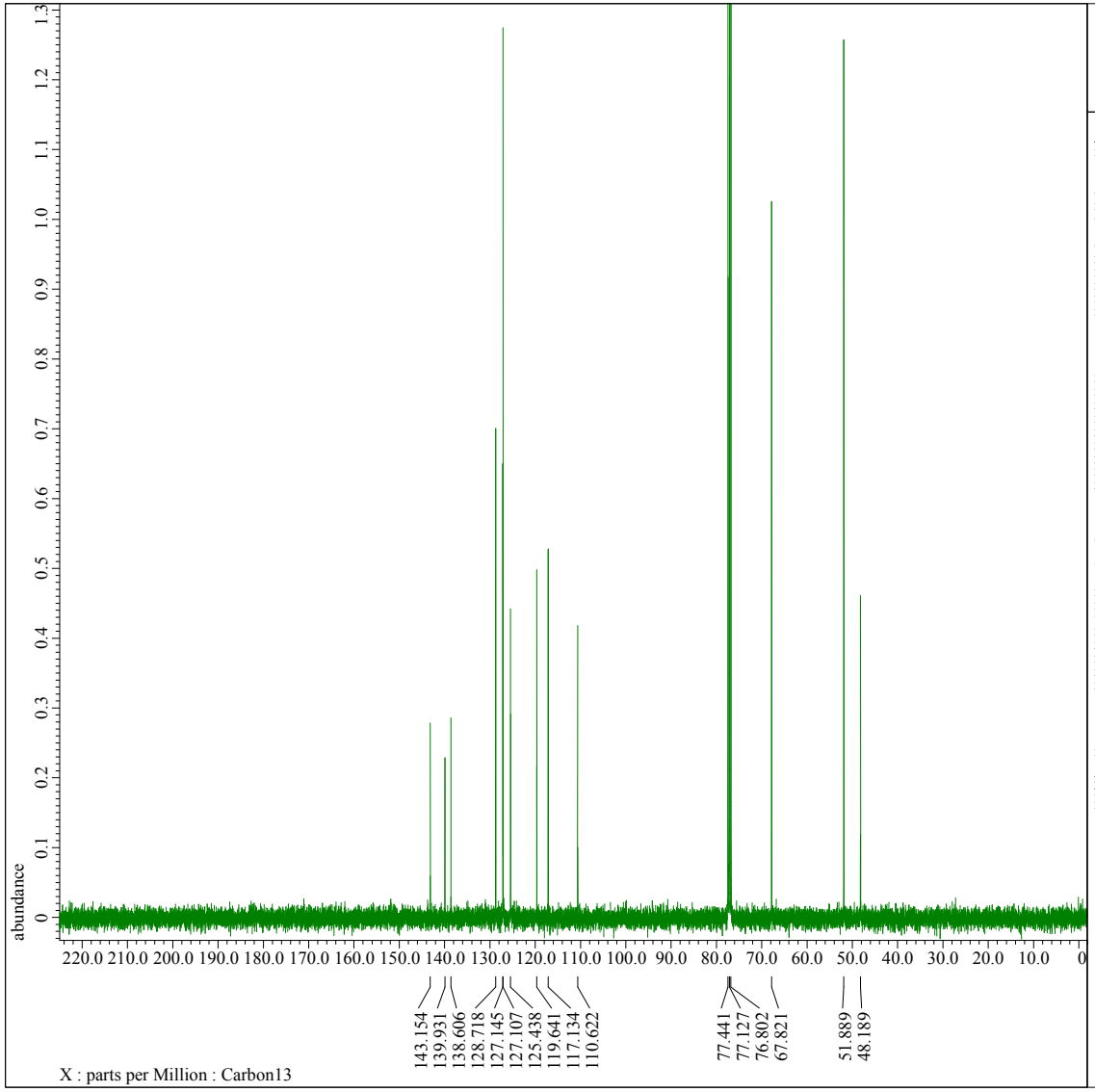

3l



JEOL RESONANCE

Filename	= M-HH7-30_Proton-1-5.jdf
Author	= delta
Experiment	= proton.jxp
Sample_Id	= M-HH7-30
Solvent	= CHLOROFORM-D
Creation_Time	= 22-NOV-2016 13:20:51
Revision_Time	= 1-DEC-2017 12:58:12
Current_Time	= 1-DEC-2017 12:58:18
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	= 9.389766[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	= 36
Temp_Get	= 21.6[dC]
X_90_Width	= 13.1[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2.2[dB]
X_Pulse	= 6.55[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]

4a



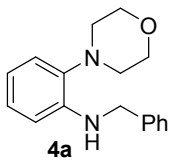
JEOL RESONANCE

```
Filename          = M-HH7-30-C_Carbon-1-2.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = M-HH7-30-C
Solvent           = CHLOROFORM-D
Creation_Time     = 24-NOV-2017 10:28:32
Revision_Time     = 24-NOV-2017 10:49:51
Current_Time      = 24-NOV-2017 10:50:13

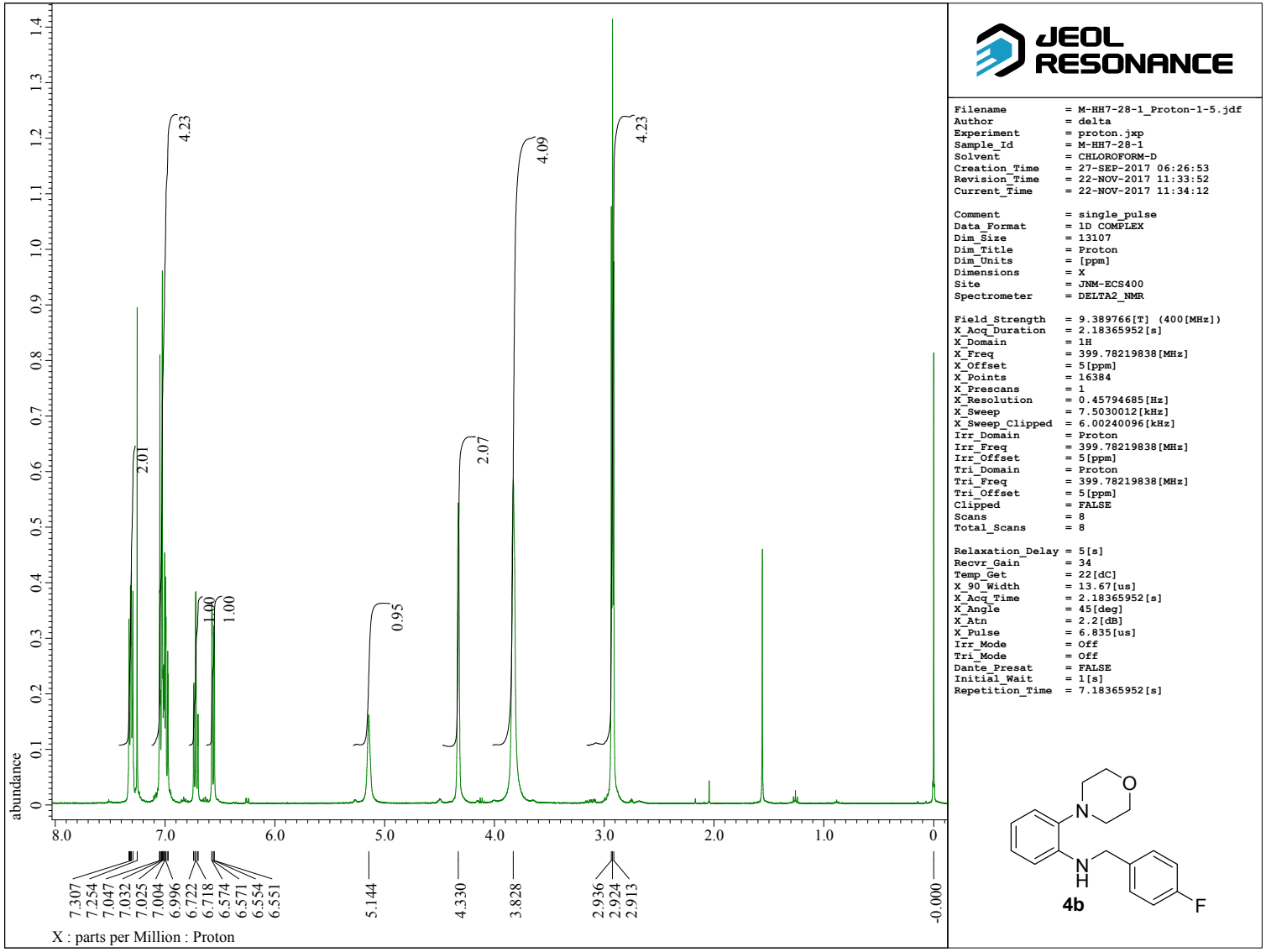
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[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             = 352
Total_Scans       = 352

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 21.9[dC]
X_90_Width        = 9.74[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 3.3[dB]
X_Pulse           = 3.24666667[us]
Irr_Atn_Dec       = 20.699[dB]
Irr_Atn_Noe       = 20.699[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



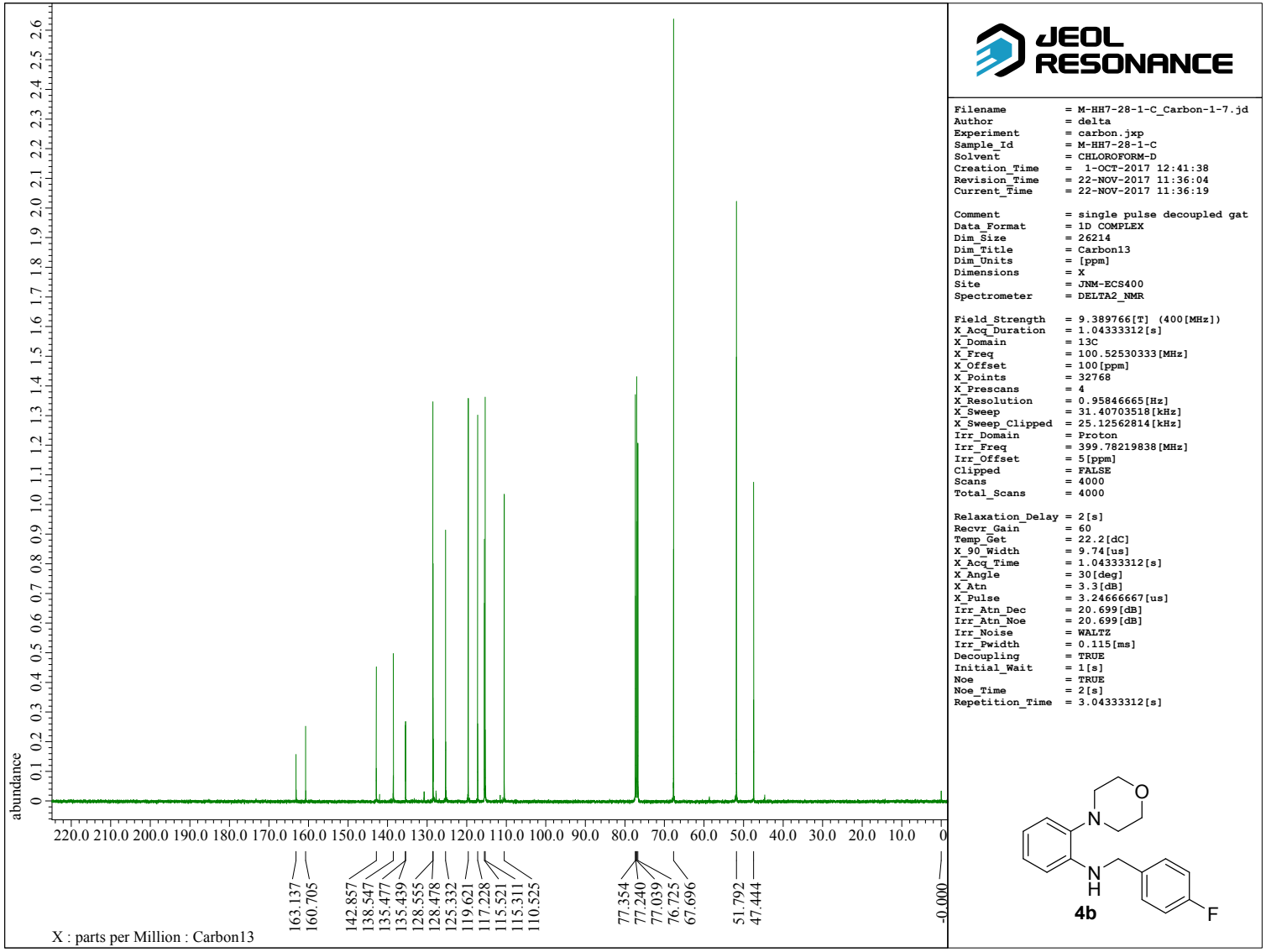
4a



JEOL RESONANCE

Filename	= M-HH7-28-1_Proton-1-5.jdf
Author	= delta
Experiment	= proton.jxp
Sample_Id	= M-HH7-28-1
Solvent	= CHLOROFORM-D
Creation_Time	= 27-SEP-2017 06:26:53
Revision_Time	= 22-NOV-2017 11:33:52
Current_Time	= 22-NOV-2017 11:34:12
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	= 9.389766[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	= 34
Temp_Get	= 22[dC]
X_90_Width	= 13.67[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2.2[dB]
X_Pulse	= 6.835[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]

4b



JEOL RESONANCE

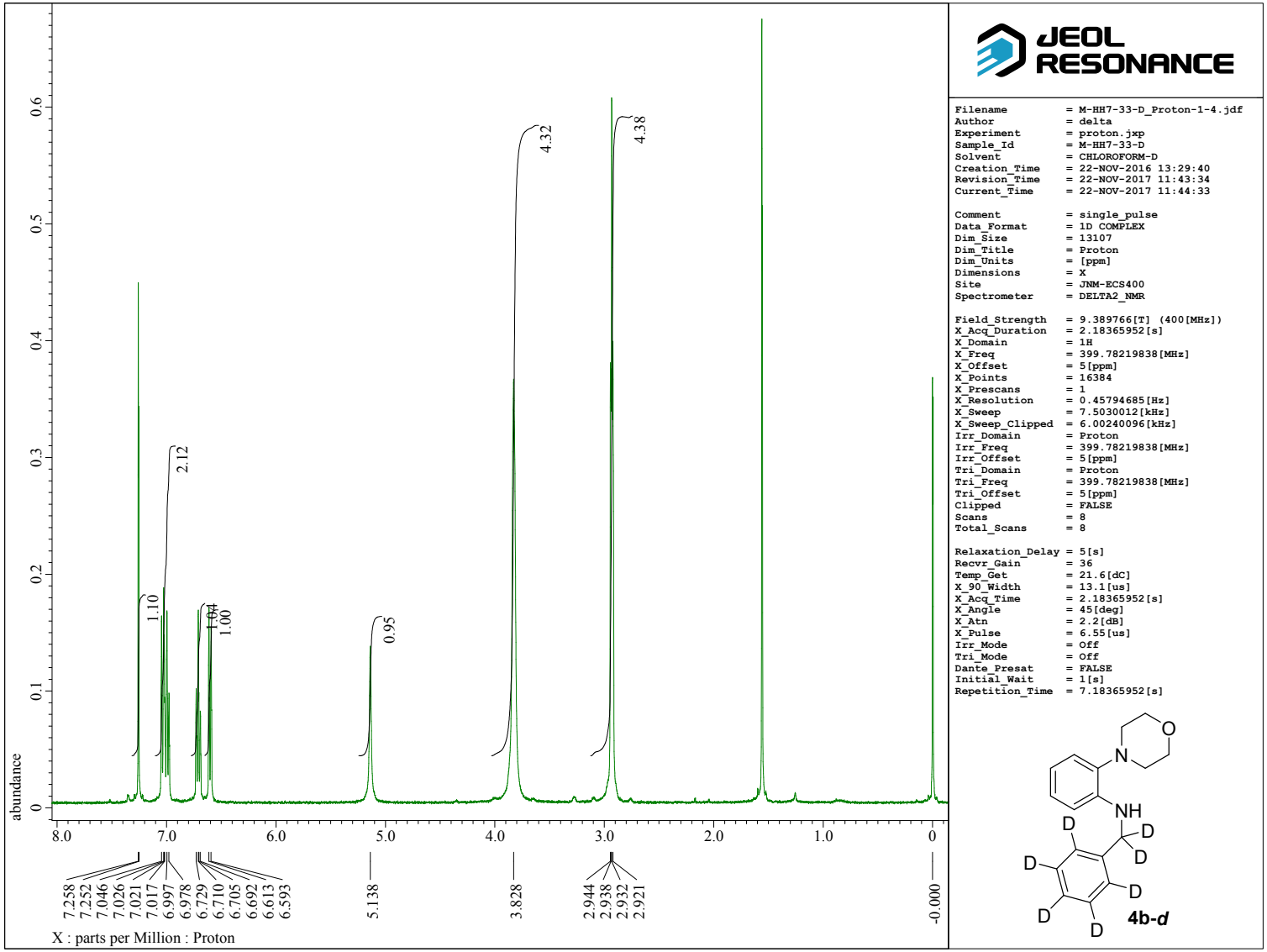
```
Filename          = M-HH7-28-1-C_Carbon-1-7.jd
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = M-HH7-28-1-C
Solvent           = CHLOROFORM-D
Creation_Time     =  1-OCT-2017 12:41:38
Revision_Time     = 22-NOV-2017 11:36:04
Current_Time      = 22-NOV-2017 11:36:19

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    = 9.389766[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             = 4000
Total_Scans       = 4000

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.2[dC]
X_90_Width        = 9.74[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 3.3[dB]
X_Pulse           = 3.24666667[us]
Irr_Atn_Dec       = 20.699[dB]
Irr_Atn_Noe       = 20.699[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```

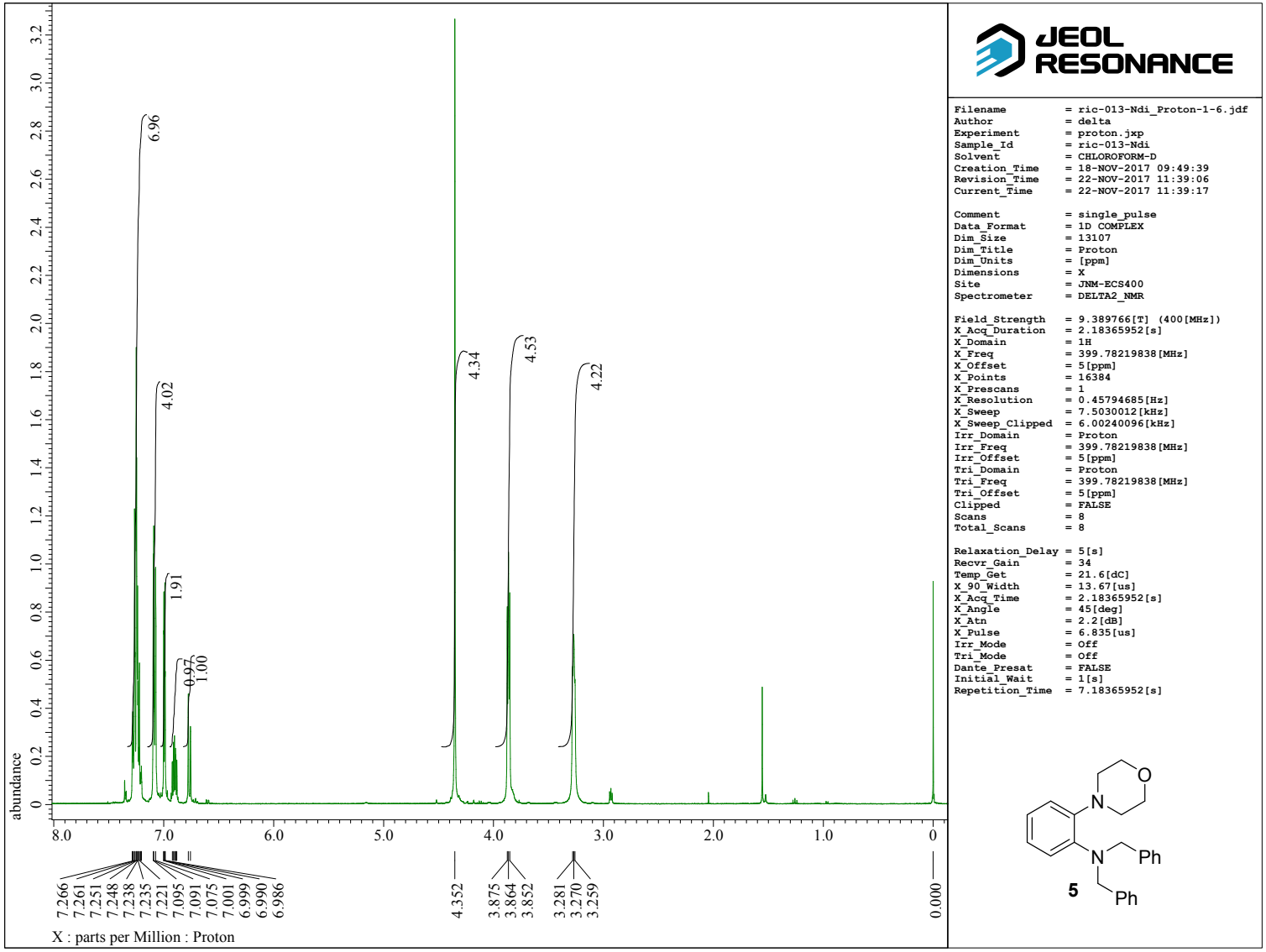

4b



JEOL RESONANCE

Filename	= M-HH7-33-D_Proton-1-4.jdf
Author	= delta
Experiment	= proton.jxp
Sample_Id	= M-HH7-33-D
Solvent	= CHLOROFORM-D
Creation_Time	= 22-NOV-2016 13:29:40
Revision_Time	= 22-NOV-2017 11:43:34
Current_Time	= 22-NOV-2017 11:44:33
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	= 9.389766[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	= 36
Temp_Get	= 21.6[dC]
X_90_Width	= 13.1[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2.2[dB]
X_Pulse	= 6.55[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]

4b-*d*



JEOL RESONANCE

Filename	= ric-013-Ndi_Proton-1-6.jdf
Author	= delta
Experiment	= proton.jxp
Sample_Id	= ric-013-Ndi
Solvent	= CHLOROFORM-D
Creation_Time	= 18-NOV-2017 09:49:39
Revision_Time	= 22-NOV-2017 11:39:06
Current_Time	= 22-NOV-2017 11:39:17
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	= 9.389766[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	= 34
Temp_Get	= 21.6[dC]
X_90_Width	= 13.67[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2.2[dB]
X_Pulse	= 6.835[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]

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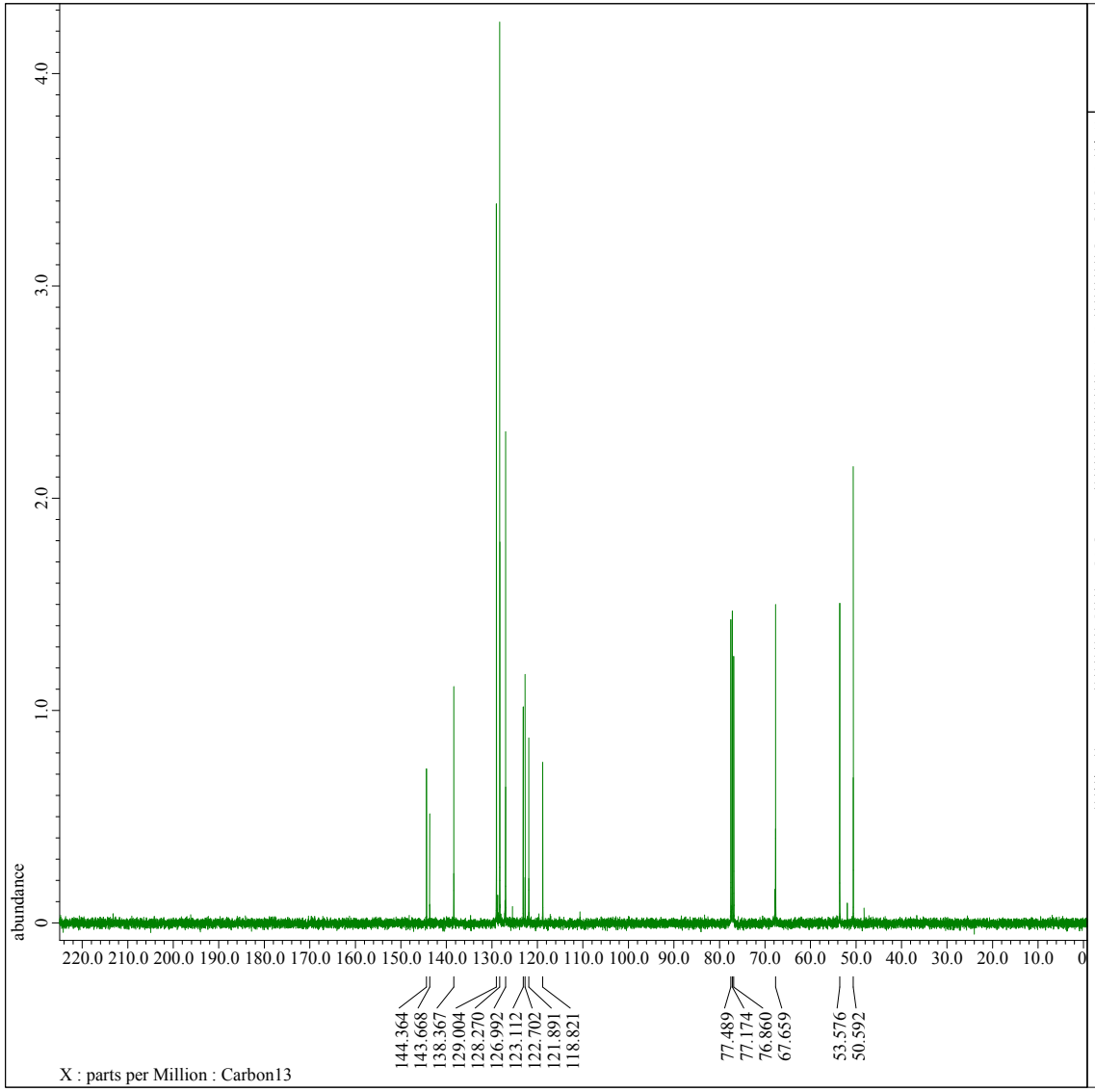


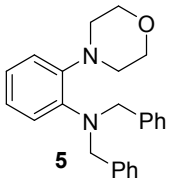
JEOL RESONANCE

```
Filename          = M-ric-013-C_Carbon-1-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = M-ric-013-C
Solvent           = CHLOROFORM-D
Creation_Time     = 22-NOV-2017 10:23:28
Revision_Time     = 22-NOV-2017 12:24:00
Current_Time      = 22-NOV-2017 12:24:17

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    = 9.389766[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             = 160
Total_Scans       = 160

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 21.8[dC]
X_90_Width        = 9.74[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 3.3[dB]
X_Pulse           = 3.24666667[us]
Irr_Atn_Dec       = 20.699[dB]
Irr_Atn_Noe       = 20.699[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



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