Preparation of C_2-Symmetric Allenes by Palladium-Catalyzed Double Nucleophilic Substitution on 3-Bromopenta-2,4-dienyl Acetate

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Supporting Information

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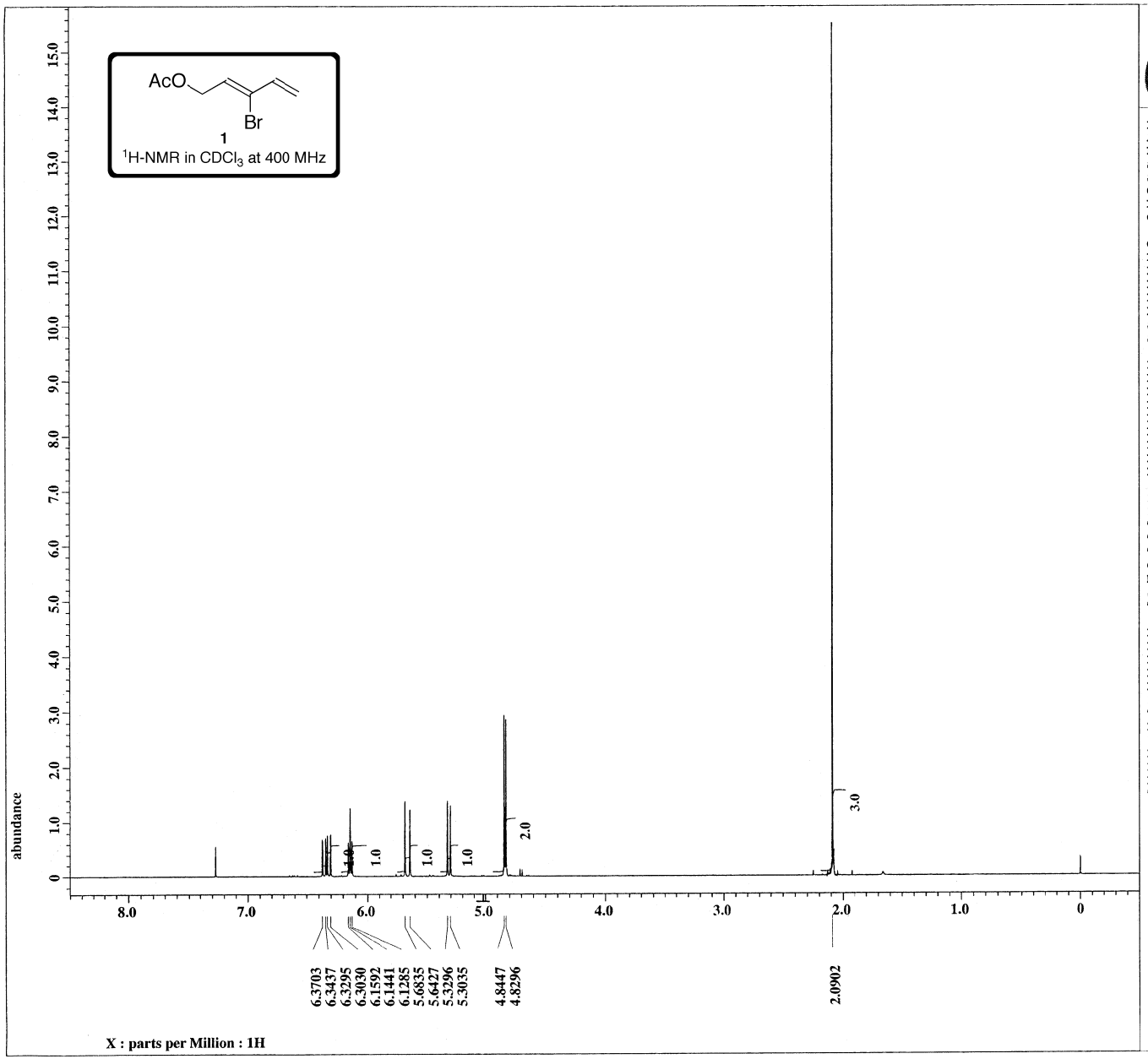
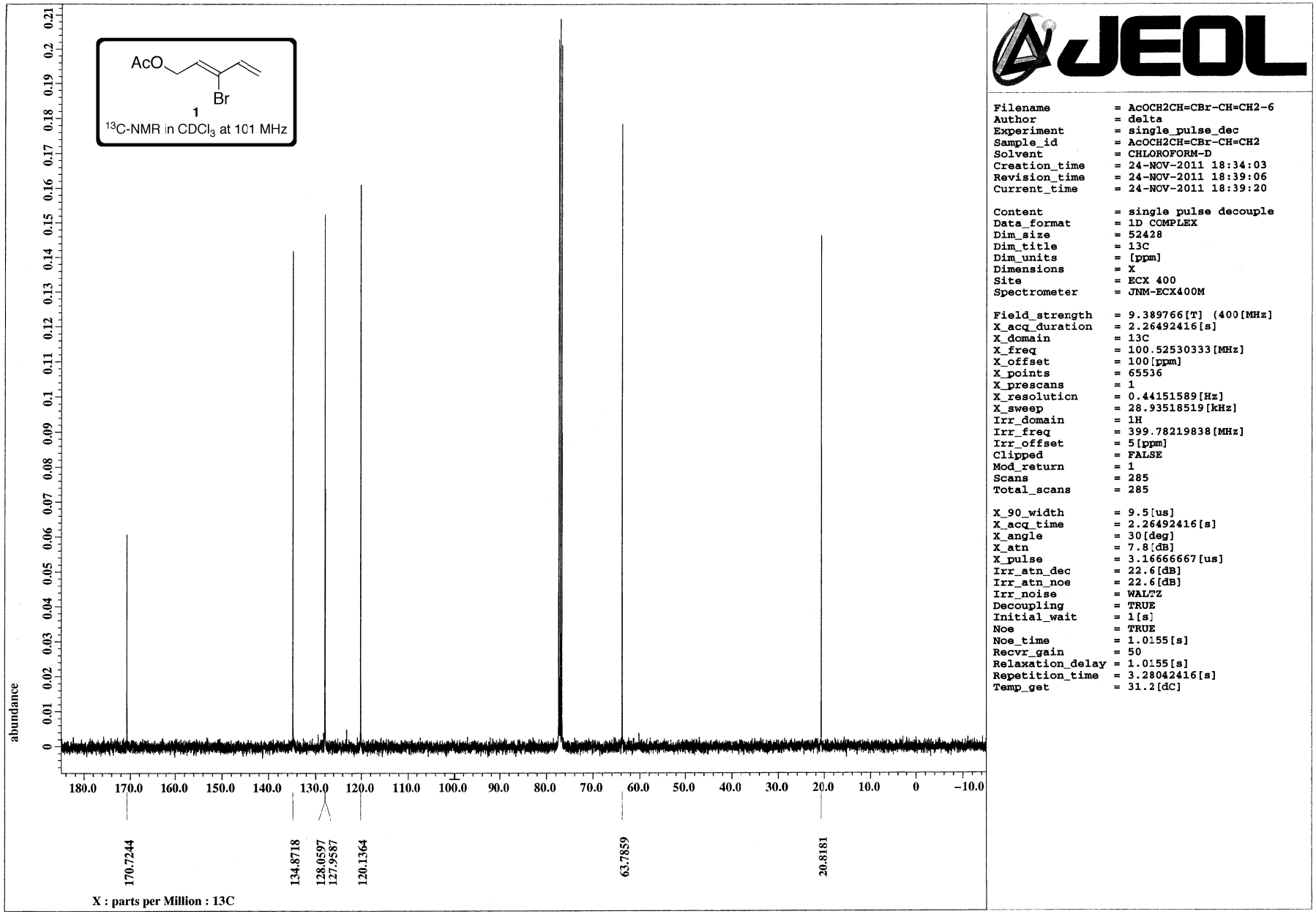
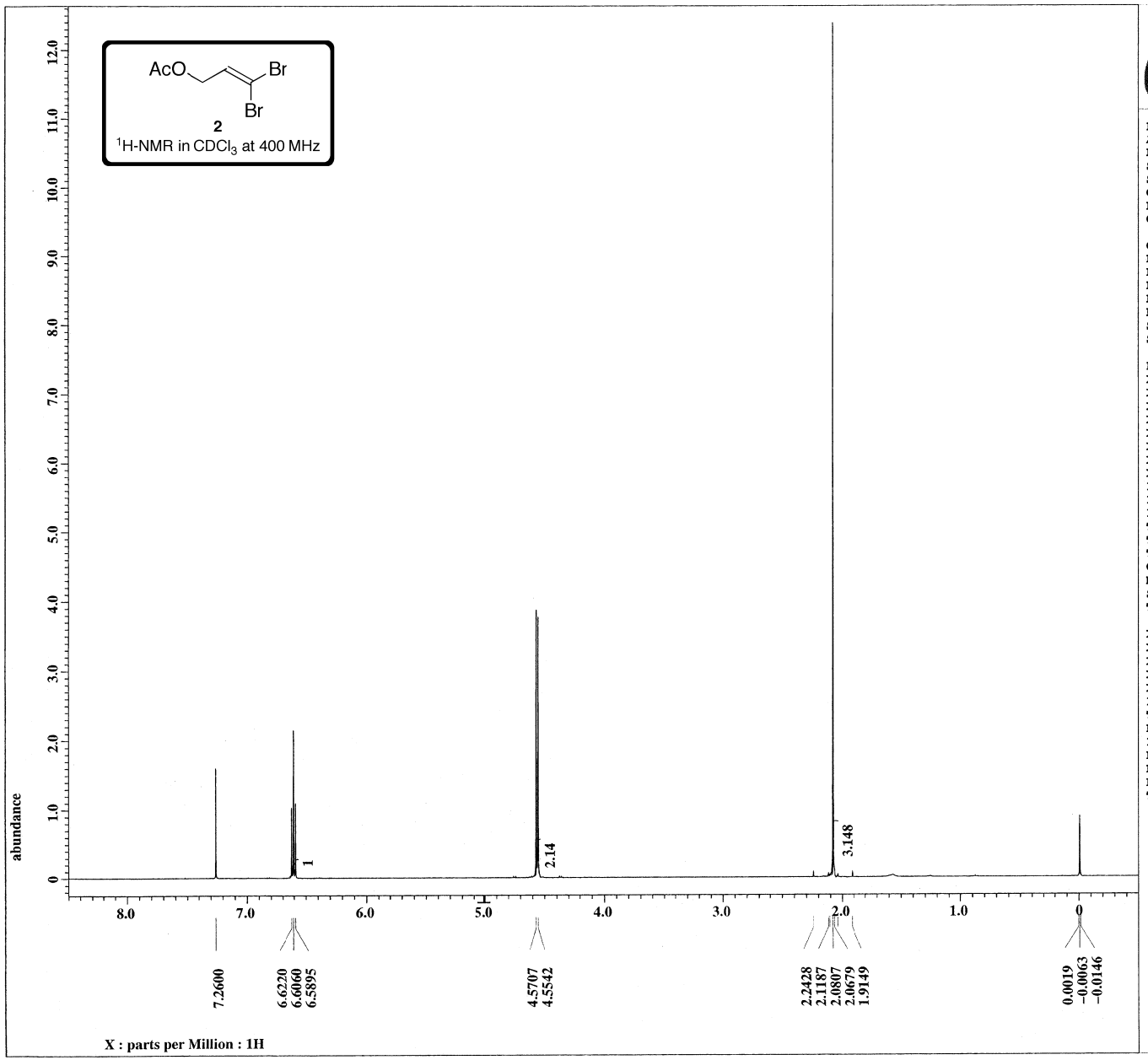


JEOL

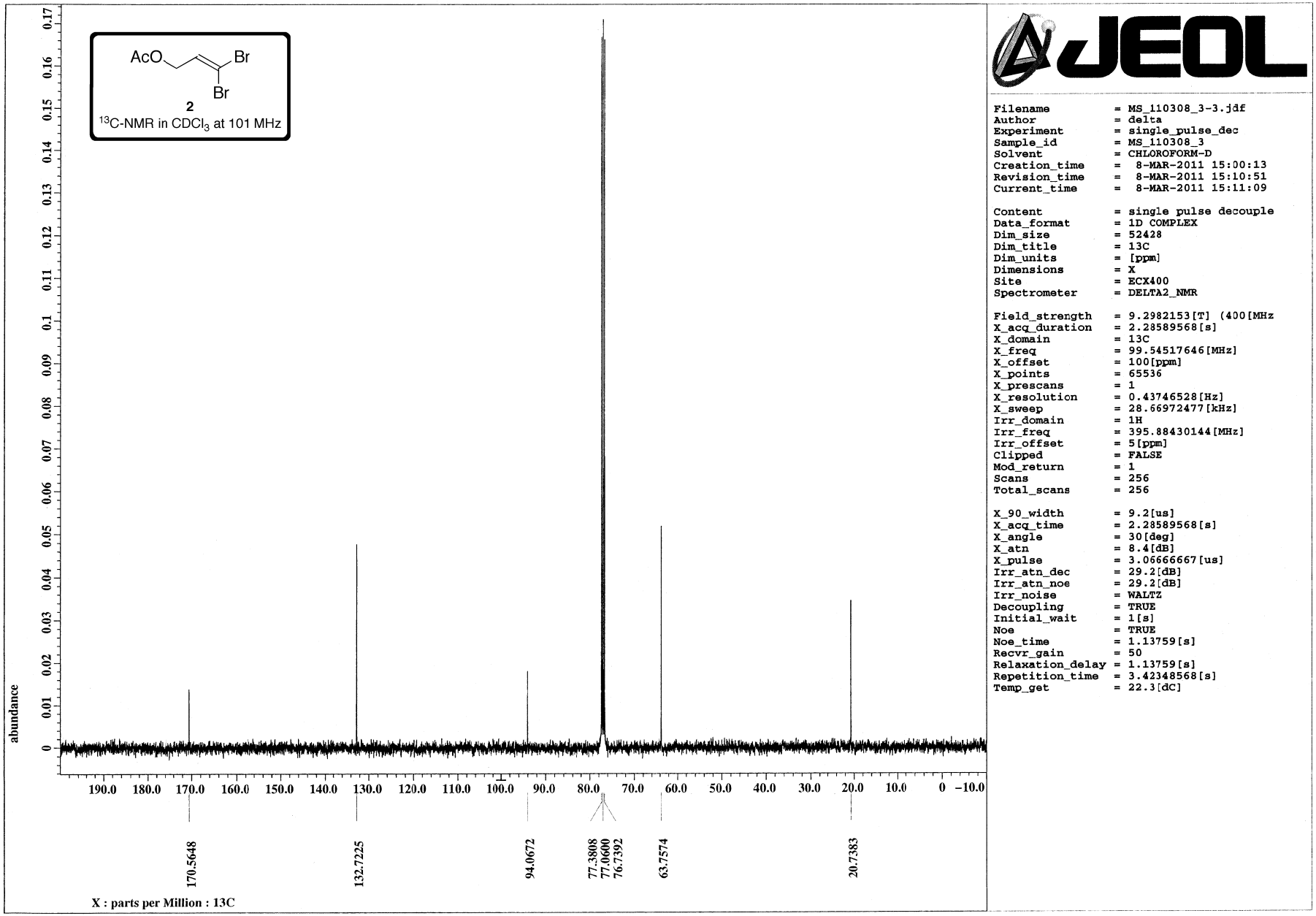
```
Filename          = AcOCH2CH=CBr-CH=CH2-6
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = AcOCH2CH=CBr-CH=CH2
Solvent           = CHLOROFORM-D
Creation_time     = 24-NOV-2011 18:18:10
Revision_time     = 24-NOV-2011 18:35:24
Current_time      = 24-NOV-2011 18:35:47

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400M

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 5.46308096[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.18304689[Hz]
X_sweep           = 5.99808061[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 11.5[us]
X_acq_time        = 5.46308096[s]
X_angle           = 45[deg]
X_atn             = 2.6[dB]
X_pulse           = 5.75[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 1.66961[s]
Repetition_time   = 7.13269096[s]
Temp_get          = 30.9[dC]
```

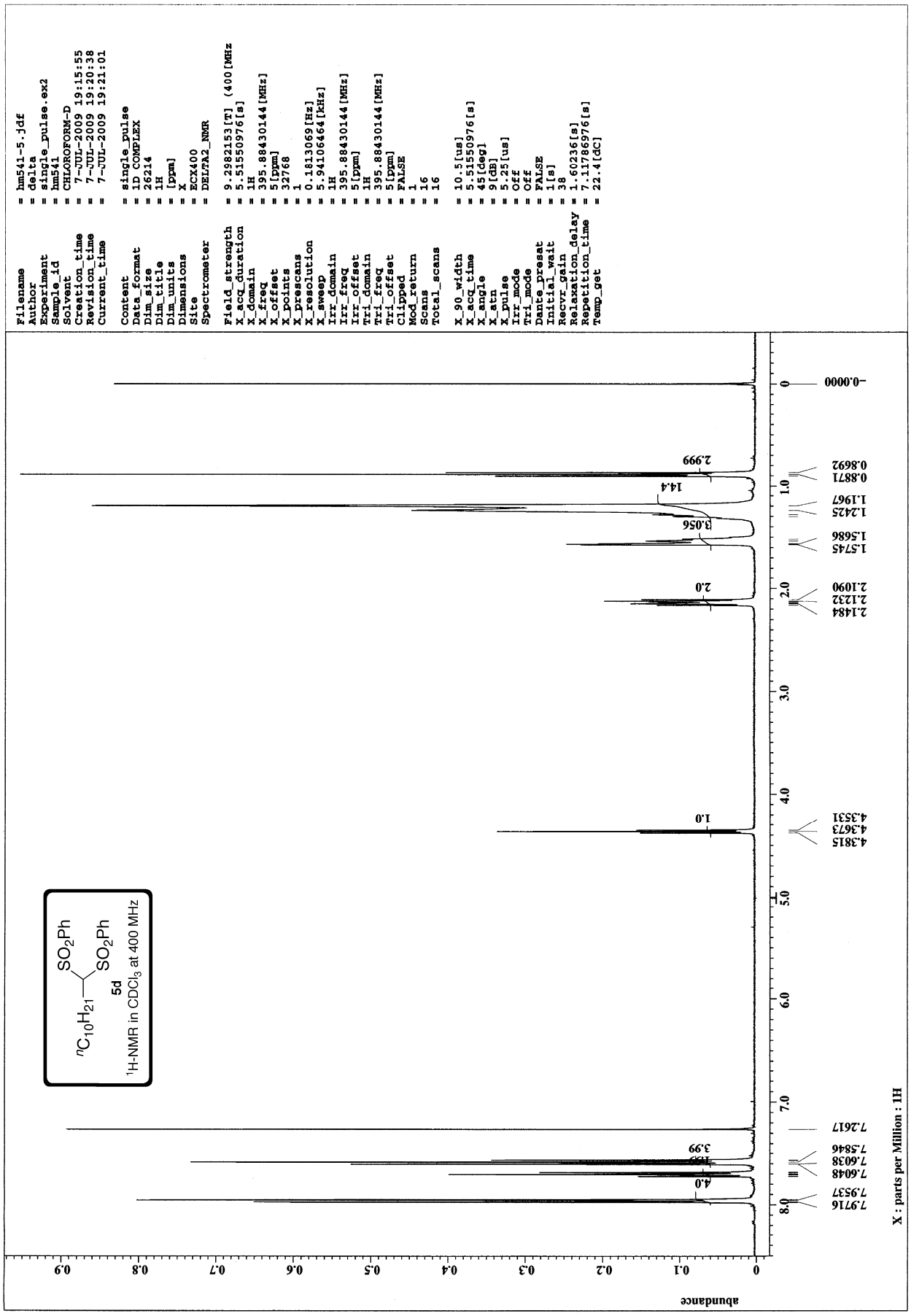

AcO
Br
1
13C-NMR in CDCl3 at 101 MHz
abundance
0 0.01 0.02 0.03 0.04 0.05 0.06 0.07 0.08 0.09 0.1 0.11 0.12 0.13 0.14 0.15 0.16 0.17 0.18 0.19 0.2 0.21
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 −10.0
170.7244
134.8718
128.0597
127.9587
120.1364
63.7859
20.8181
X : parts per Million : 13C
JEOL
Filename = AcOCH2CH=CBr-CH=CH2-6
Author = delta
Experiment = single_pulse_dec
Sample_id = AcOCH2CH=CBr-CH=CH2
Solvent = CHLOROFORM-D
Creation_time = 24-NOV-2011 18:34:03
Revision_time = 24-NOV-2011 18:39:06
Current_time = 24-NOV-2011 18:39:20
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = JNM-ECX400M
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 2.26492416[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 1
X_resolution = 0.44151589[Hz]
X_sweep = 28.93518519[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 285
Total_scans = 285
X_90_width = 9.5[us]
X_acq_time = 2.26492416[s]
X_angle = 30[deg]
X_atn = 7.8[dB]
X_pulse = 3.16666667[us]
Irr_atn_dec = 22.6[dB]
Irr_atn_noe = 22.6[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 1.0155[s]
Recvr_gain = 50
Relaxation_delay = 1.0155[s]
Repetition_time = 3.28042416[s]
Temp_get = 31.2[dC]

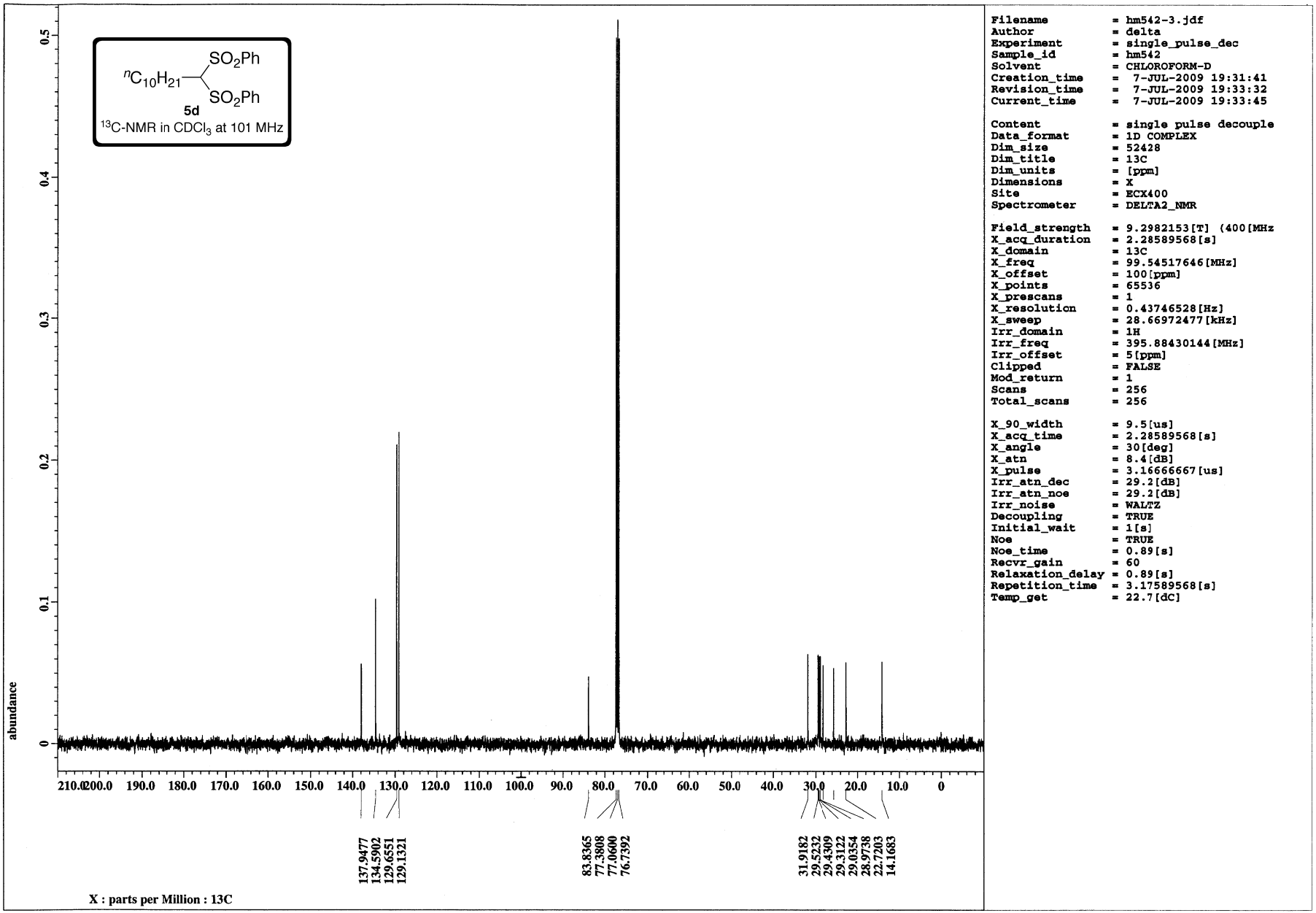


JEOL

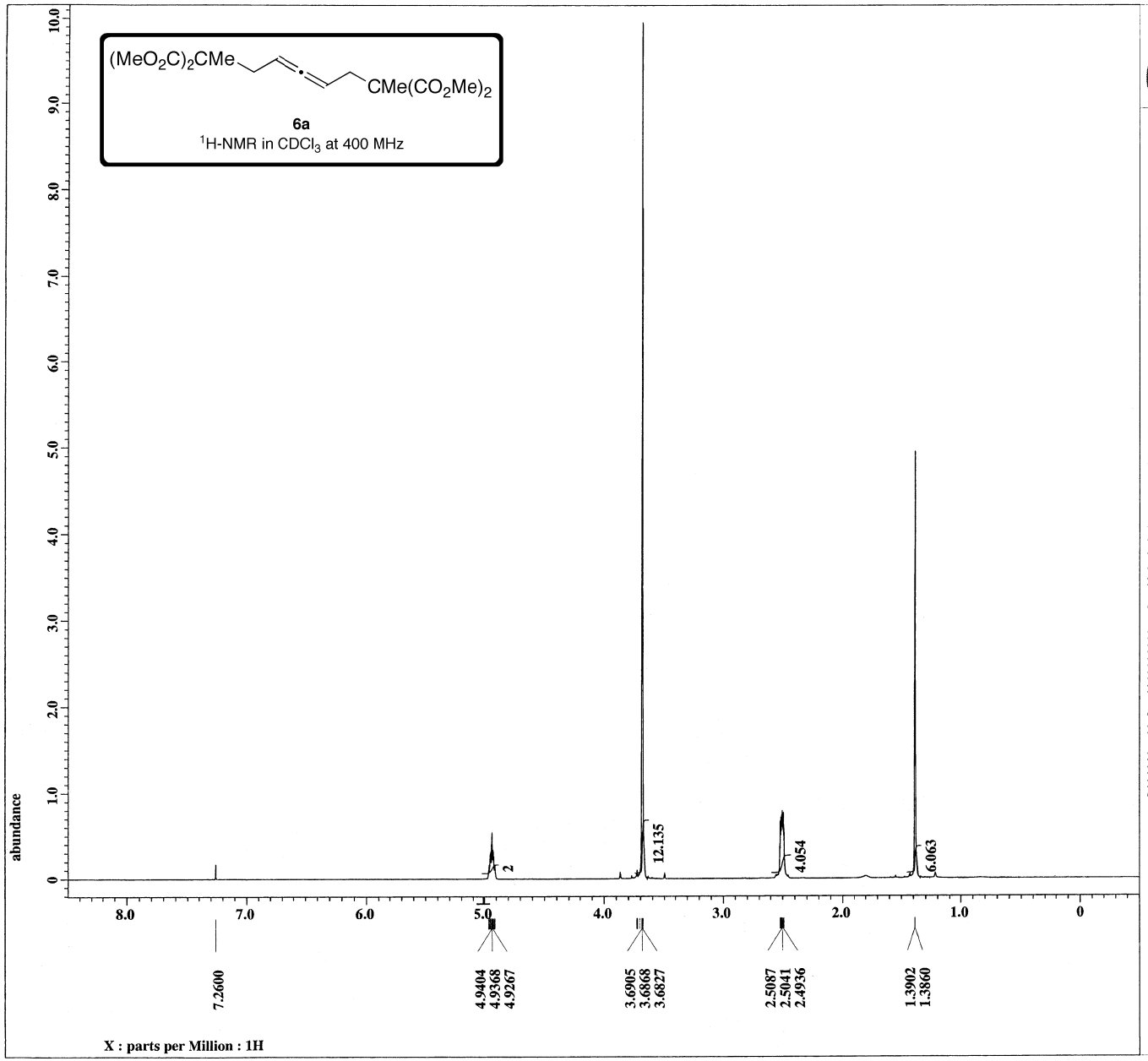
```
Filename          = MS_110308_2-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = MS_110308_2
Solvent           = CHLOROFORM-D
Creation_time     =  8-MAR-2011 14:43:43
Revision_time     =  8-MAR-2011 14:55:46
Current_time      =  8-MAR-2011 14:55:57

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400
Spectrometer      = DELTA2_NMR

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 5.51550976[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.1813069[Hz]
X_sweep           = 5.94106464[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 11[us]
X_acq_time        = 5.51550976[s]
X_angle           = 45[deg]
X_atn             = 9[dB]
X_pulse           = 5.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 44
Relaxation_delay  = 1.60236[s]
Repetition_time   = 7.11786976[s]
Temp_get          = 21.8[dC]
```


AcO
Br
Br
2
^{13}C-NMR in $CDCl_3$ at 101 MHz
abundance
0 0.01 0.02 0.03 0.04 0.05 0.06 0.07 0.08 0.09 0.1 0.11 0.12 0.13 0.14 0.15 0.16 0.17
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 −10.0
170.5648
132.7225
94.0672
77.3808
77.0600
76.7392
63.7574
20.7383
X : parts per Million : 13C
JEOL
Filename = MS_110308_3-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = MS_110308_3
Solvent = CHLOROFORM-D
Creation_time = 8-MAR-2011 15:00:13
Revision_time = 8-MAR-2011 15:10:51
Current_time = 8-MAR-2011 15:11:09
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX400
Spectrometer = DELTA2_NMR
Field_strength = 9.2982153[T] (400[MHz
X_acq_duration = 2.28589568[s]
X_domain = 13C
X_freq = 99.54517646[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 1
X_resolution = 0.43746528[Hz]
X_sweep = 28.66972477[kHz]
Irr_domain = 1H
Irr_freq = 395.88430144[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 256
Total_scans = 256
X_90_width = 9.2[us]
X_acq_time = 2.28589568[s]
X_angle = 30[deg]
X_atn = 8.4[dB]
X_pulse = 3.06666667[us]
Irr_atn_dec = 29.2[dB]
Irr_atn_noe = 29.2[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 1.13759[s]
Recvr_gain = 50
Relaxation_delay = 1.13759[s]
Repetition_time = 3.42348568[s]
Temp_get = 22.3[dC]

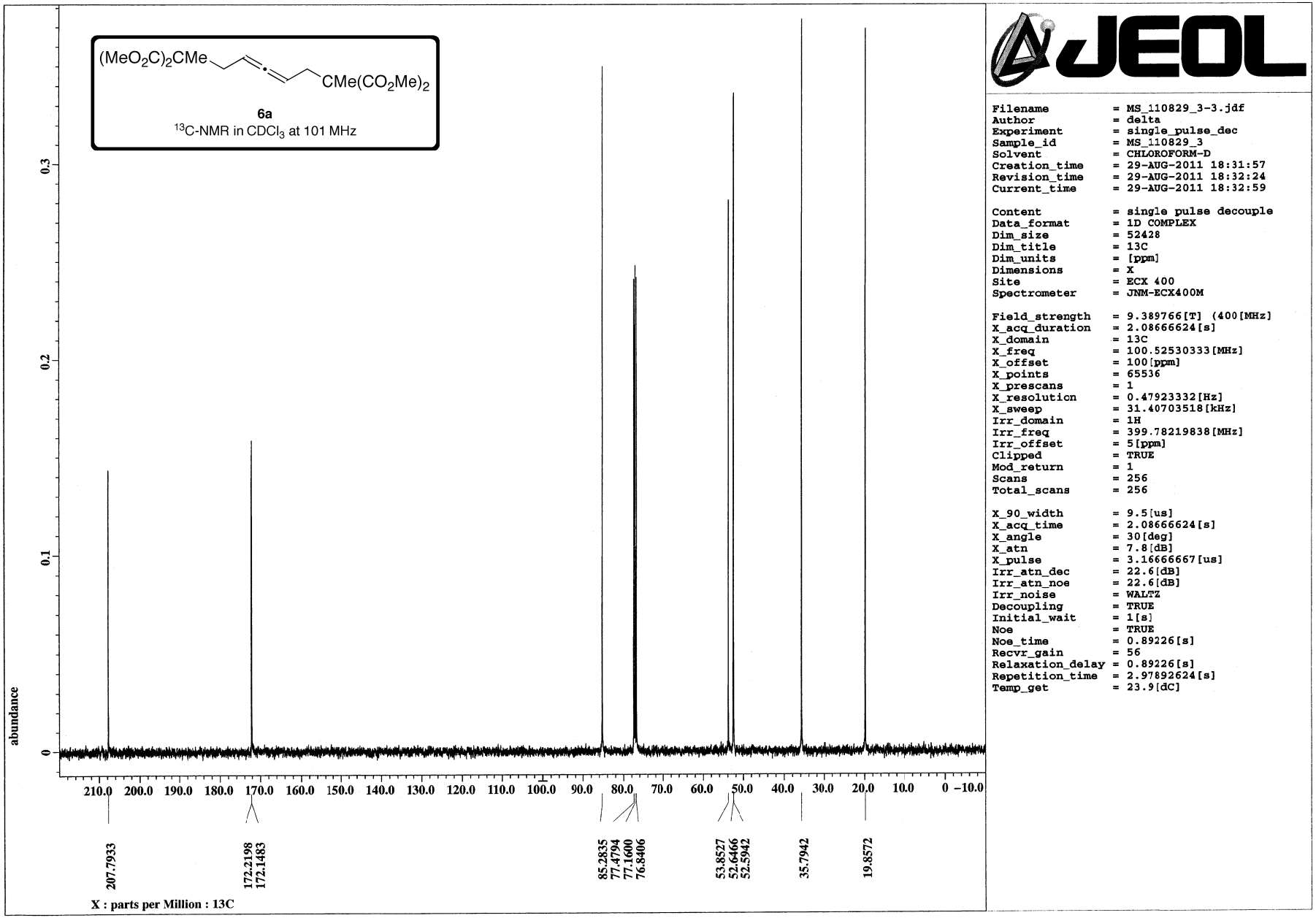

Filename = hm541-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = hm541
Solvent = CHLOROFORM-D
Creation_time = 7-JUL-2009 19:15:55
Revision_time = 7-JUL-2009 19:20:38
Current_time = 7-JUL-2009 19:21:01
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX400
Spectrometer = DELTA2_NMR
Field_strength = 9.2982153[T] (400[MHz
X_acq_duration = 5.51550976[s]
X_domain = 1H
X_freq = 395.88430144[MHz]
X_offset = 5[ppm]
X_points = 32768
X_prescans = 1
X_resolution = 0.18130069[Hz]
X_sweep = 5.94106464[kHz]
Irr_domain = 1H
Irr_freq = 395.88430144[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 395.88430144[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 10.5[us]
X_acq_time = 5.51550976[s]
X_angle = 45[deg]
X_atn = 9[dB]
X_pulse = 5.25[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 38
Relaxation_delay = 1.60236[s]
Repetition_time = 7.11786976[s]
Temp_get = 22.4[dC]
$^{n}C_{10}H_{21}$
SO2Ph
SO2Ph
5d
1H-NMR in CDCl3 at 400 MHz
abundance
X : parts per Million : 1H
7.9716
7.9537
7.6048
7.6038
7.5846
7.2617
4.3815
4.3673
4.3531
2.1484
2.1232
2.1090
1.5745
1.5686
1.2425
1.1967
0.8871
0.8692
-0.0000
4.0
3.99
1.0
2.0
3.056
14.4
2.999




```
Filename          = hm542-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = hm542
Solvent           = CHLOROFORM-D
Creation_time     =  7-JUL-2009 19:31:41
Revision_time     =  7-JUL-2009 19:33:32
Current_time      =  7-JUL-2009 19:33:45

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 52428
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400
Spectrometer      = DELTA2_NMR

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 2.28589568[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 65536
X_prescans        = 1
X_resolution      = 0.43746528[Hz]
X_sweep           = 28.66972477[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 256
Total_scans       = 256

X_90_width        = 9.5[us]
X_acq_time        = 2.28589568[s]
X_angle           = 30[deg]
X_atn             = 8.4[dB]
X_pulse           = 3.16666667[us]
Irr_atn_dec       = 29.2[dB]
Irr_atn_noe       = 29.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 0.89[s]
Recvr_gain        = 60
Relaxation_delay  = 0.89[s]
Repetition_time   = 3.17589568[s]
Temp_get          = 22.7[dC]
```

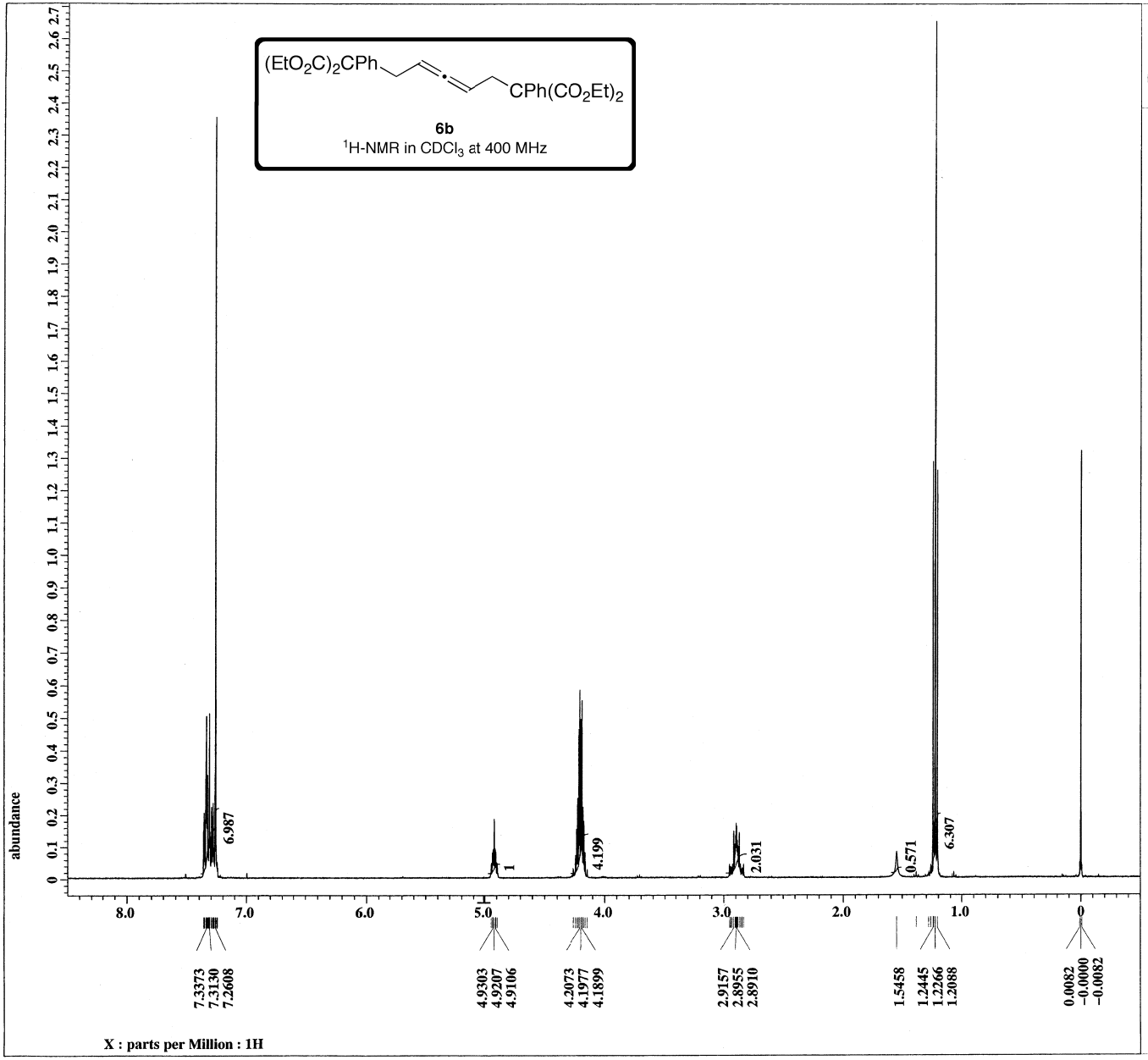



```
Filename          = MS_110829_2-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = MS_110829_2
Solvent           = CHLOROFORM-D
Creation_time     = 29-AUG-2011 18:16:07
Revision_time     = 29-AUG-2011 18:17:54
Current_time      = 29-AUG-2011 18:18:11

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400M

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 5.46308096[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.18304689[Hz]
X_sweep           = 5.99808061[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 11.5[us]
X_acq_time        = 5.46308096[s]
X_angle           = 45[deg]
X_atn             = 2.6[dB]
X_pulse           = 5.75[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 32
Relaxation_delay  = 1.66961[s]
Repetition_time   = 7.13269096[s]
Temp_get          = 23.7[dC]
```



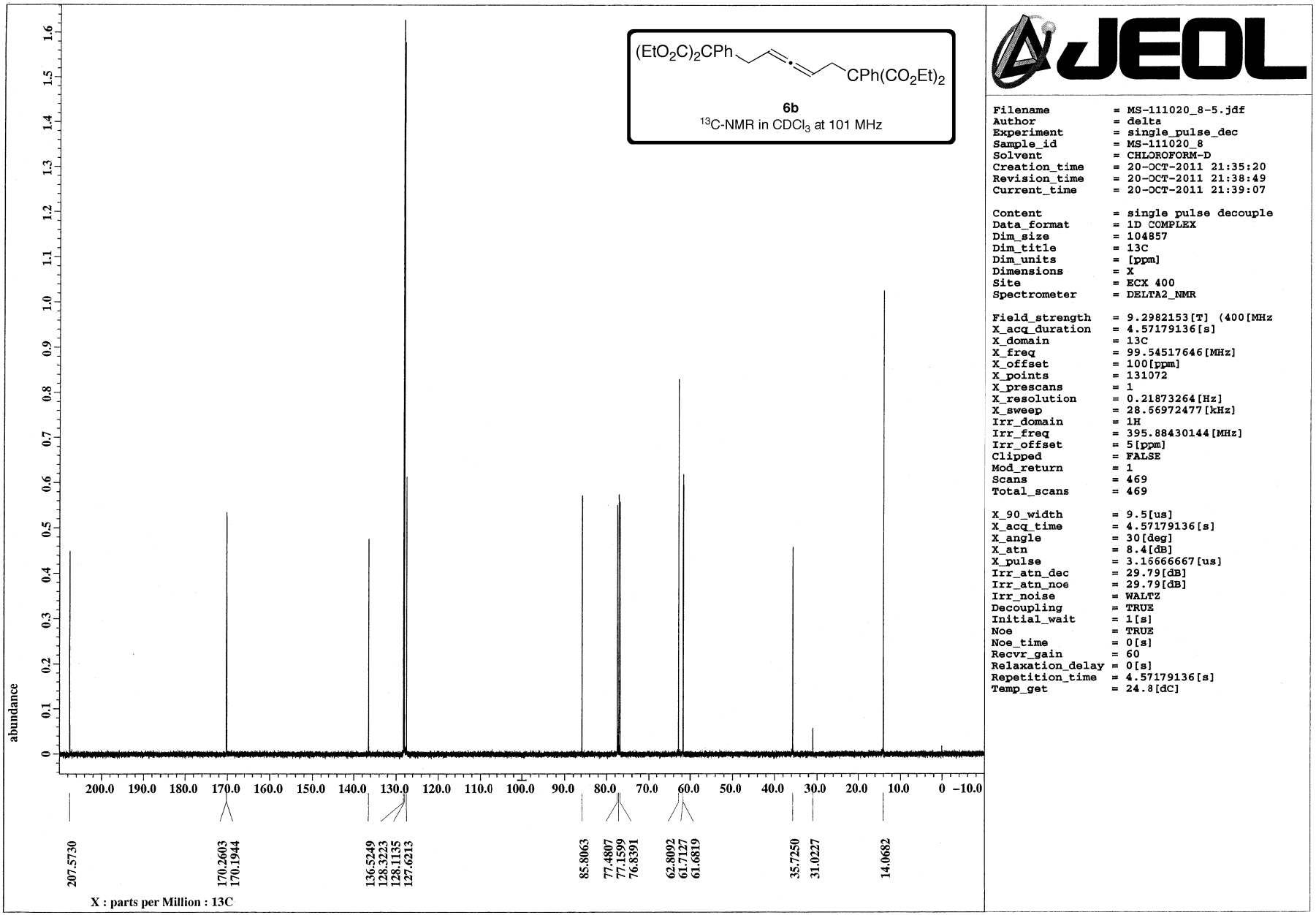
JEOL

```
Filename          = MS_110829_3-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = MS_110829_3
Solvent           = CHLOROFORM-D
Creation_time     = 29-AUG-2011 18:31:57
Revision_time     = 29-AUG-2011 18:32:24
Current_time      = 29-AUG-2011 18:32:59

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 52428
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400M

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.08666624[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 65536
X_prescans        = 1
X_resolution      = 0.47923332[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 256
Total_scans       = 256

X_90_width        = 9.5[us]
X_acq_time        = 2.08666624[s]
X_angle           = 30[deg]
X_atn             = 7.8[dB]
X_pulse           = 3.16666667[us]
Irr_atn_dec       = 22.6[dB]
Irr_atn_noe       = 22.6[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 0.89226[s]
Recvr_gain        = 56
Relaxation_delay  = 0.89226[s]
Repetition_time   = 2.97892624[s]
Temp_get          = 23.9[dC]
```



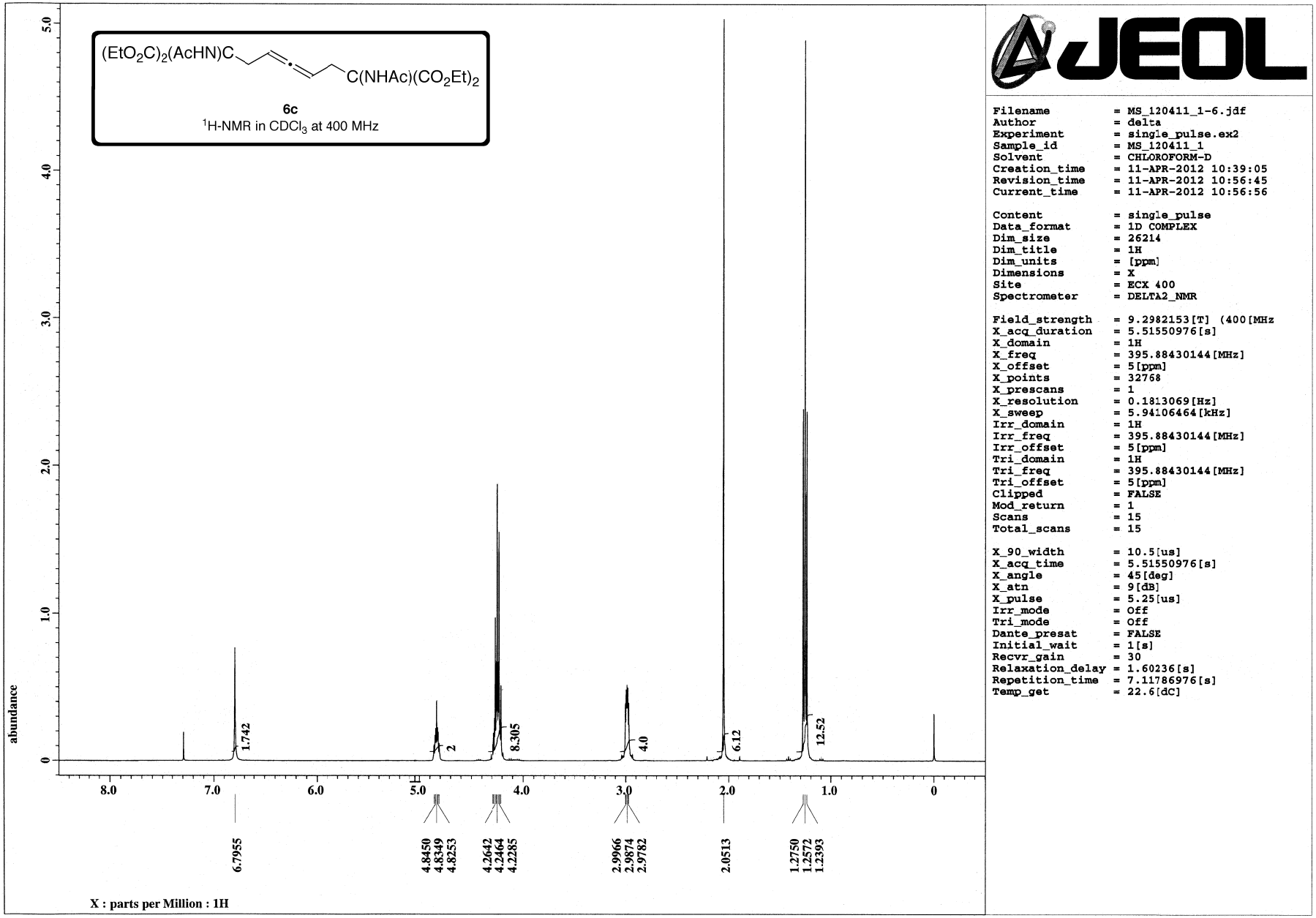
JEOL

```
Filename          = MS_1119_2-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = MS_1119_2
Solvent           = CHLOROFORM-D
Creation_time     = 19-OCT-2011 22:26:53
Revision_time     = 19-OCT-2011 22:30:45
Current_time      = 19-OCT-2011 22:30:58

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400M

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 5.46308096[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.18304689[Hz]
X_sweep           = 5.99808061[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 11.5[us]
X_acq_time        = 5.46308096[s]
X_angle           = 45[deg]
X_atn             = 2.6[dB]
X_pulse           = 5.75[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 1.66961[s]
Repetition_time   = 7.13269096[s]
Temp_get          = 21.6[dC]
```



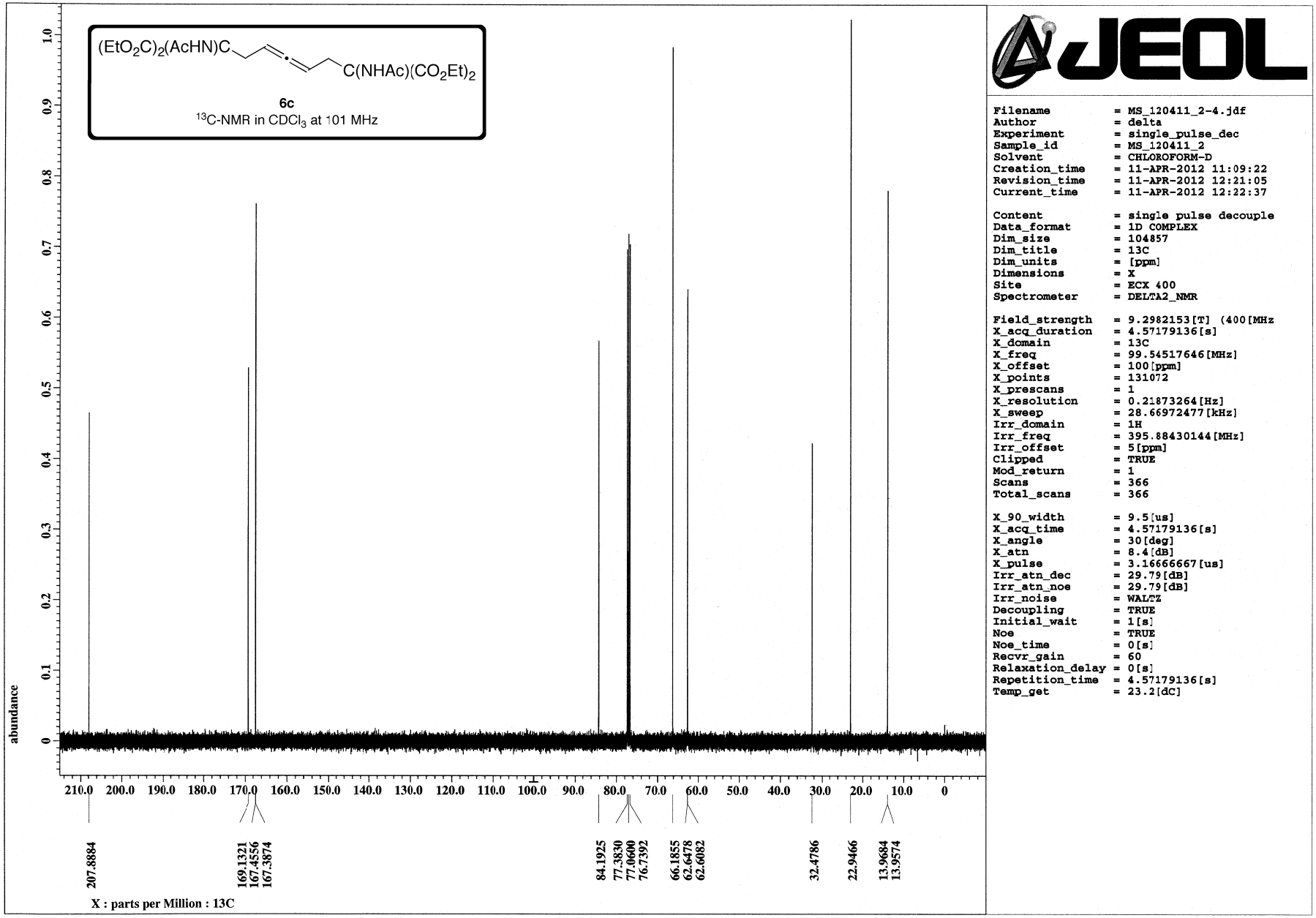
JEOL

Filename	= MS-111020_8-5.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= MS-111020_8
Solvent	= CHLOROFORM-D
Creation_time	= 20-OCT-2011 21:35:20
Revision_time	= 20-OCT-2011 21:38:49
Current_time	= 20-OCT-2011 21:39:07
Content	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 104857
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= DELTA2_NMR
Field_strength	= 9.2982153[T] (400[MHz
X_acq_duration	= 4.57179136[s]
X_domain	= 13C
X_freq	= 99.54517646[MHz]
X_offset	= 100[ppm]
X_points	= 131072
X_prescans	= 1
X_resolution	= 0.21873264[Hz]
X_sweep	= 28.56972477[kHz]
Irr_domain	= 1H
Irr_freq	= 395.88430144[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 469
Total_scans	= 469
X_90_width	= 9.5[us]
X_acq_time	= 4.57179136[s]
X_angle	= 30[deg]
X_atn	= 8.4[dB]
X_pulse	= 3.16666667[us]
Irr_atn_dec	= 29.79[dB]
Irr_atn_noe	= 29.79[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 0[s]
Recvr_gain	= 60
Relaxation_delay	= 0[s]
Repetition_time	= 4.57179136[s]
Temp_get	= 24.8[dC]



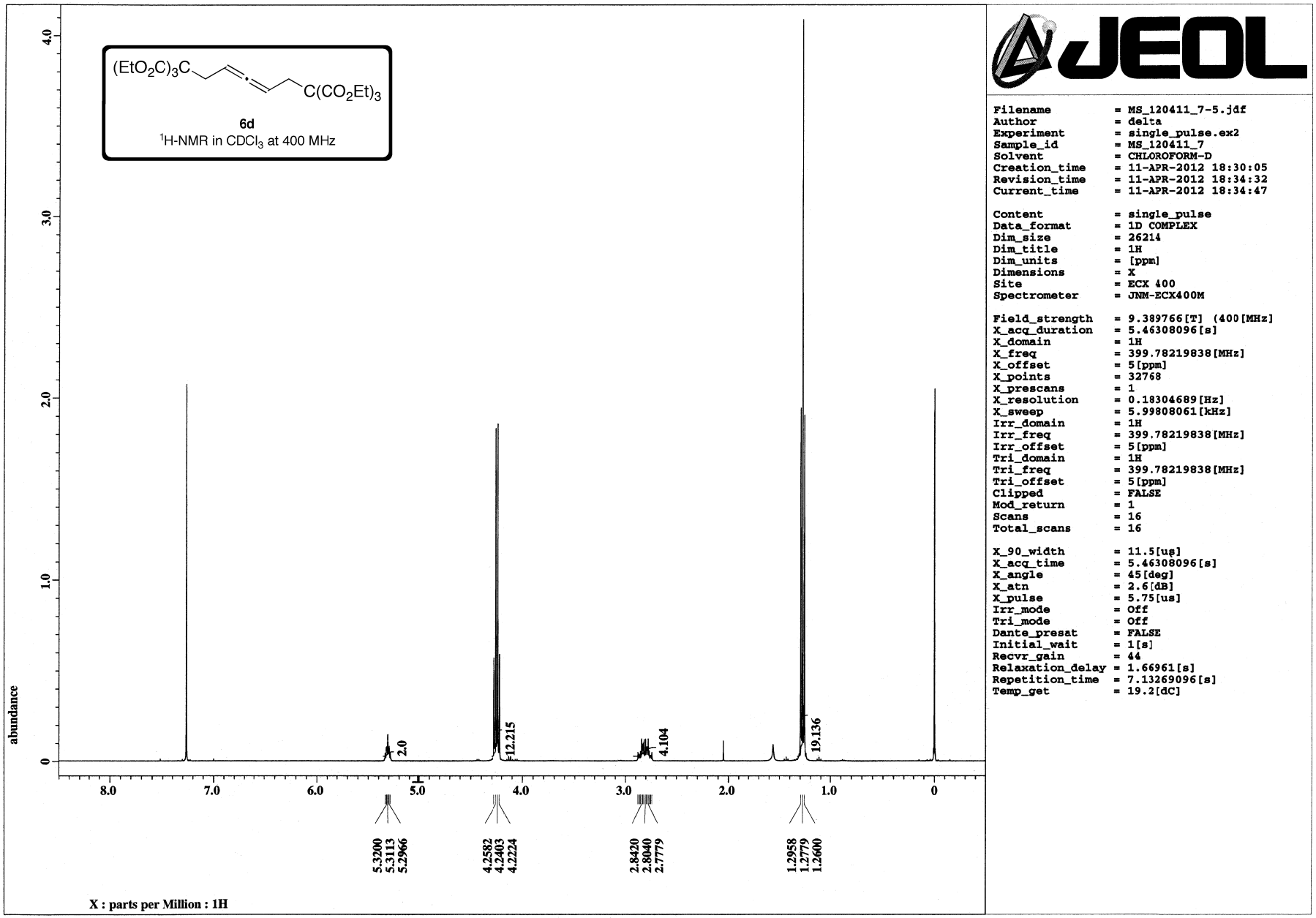
JEOL

```
Filename          = MS_120411_1-6.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = MS_120411_1
Solvent           = CHLOROFORM-D
Creation_time     = 11-APR-2012 10:39:05
Revision_time     = 11-APR-2012 10:56:45
Current_time      = 11-APR-2012 10:56:56

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = DELTA2_NMR

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 5.51550976[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.1813069[Hz]
X_sweep           = 5.94106464[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 15
Total_scans       = 15

X_90_width        = 10.5[us]
X_acq_time        = 5.51550976[s]
X_angle           = 45[deg]
X_atn             = 9[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 30
Relaxation_delay  = 1.60236[s]
Repetition_time   = 7.11786976[s]
Temp_get          = 22.6[dC]
```



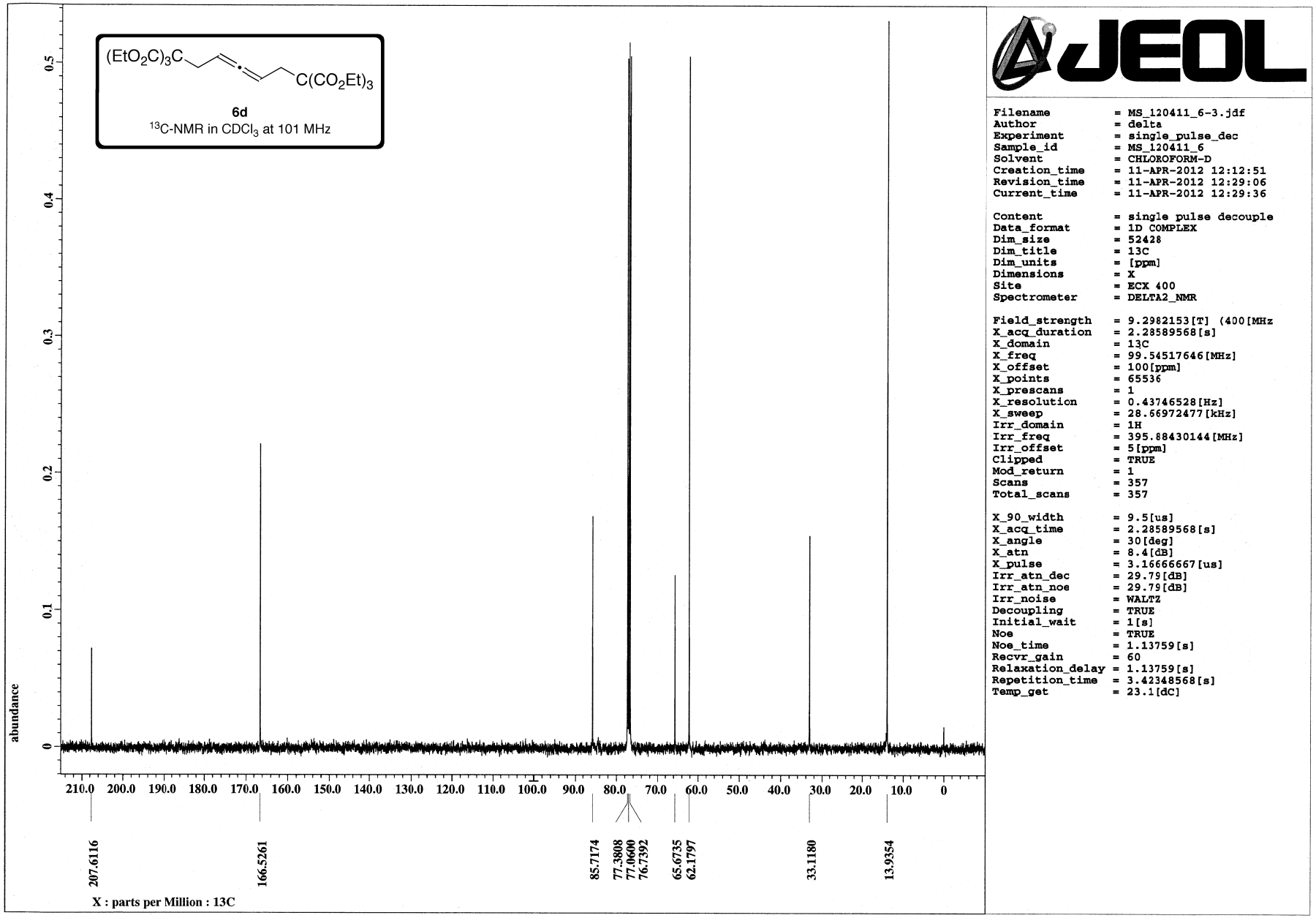
JEOL

```
Filename          = MS_120411_2-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = MS_120411_2
Solvent           = CHLOROFORM-D
Creation_time     = 11-APR-2012 11:09:22
Revision_time     = 11-APR-2012 12:21:05
Current_time      = 11-APR-2012 12:22:37

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 104857
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = DELTA2_NMR

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 4.57179136[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 131072
X_prescans        = 1
X_resolution      = 0.21873264[Hz]
X_sweep           = 28.66972477[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 366
Total_scans       = 366

X_90_width        = 9.5[us]
X_acq_time        = 4.57179136[s]
X_angle           = 30[deg]
X_atn             = 8.4[dB]
X_pulse           = 3.16666667[us]
Irr_atn_dec       = 29.79[dB]
Irr_atn_noe       = 29.79[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 0[s]
Recvr_gain        = 60
Relaxation_delay  = 0[s]
Repetition_time   = 4.57179136[s]
Temp_get          = 23.2[dC]
```



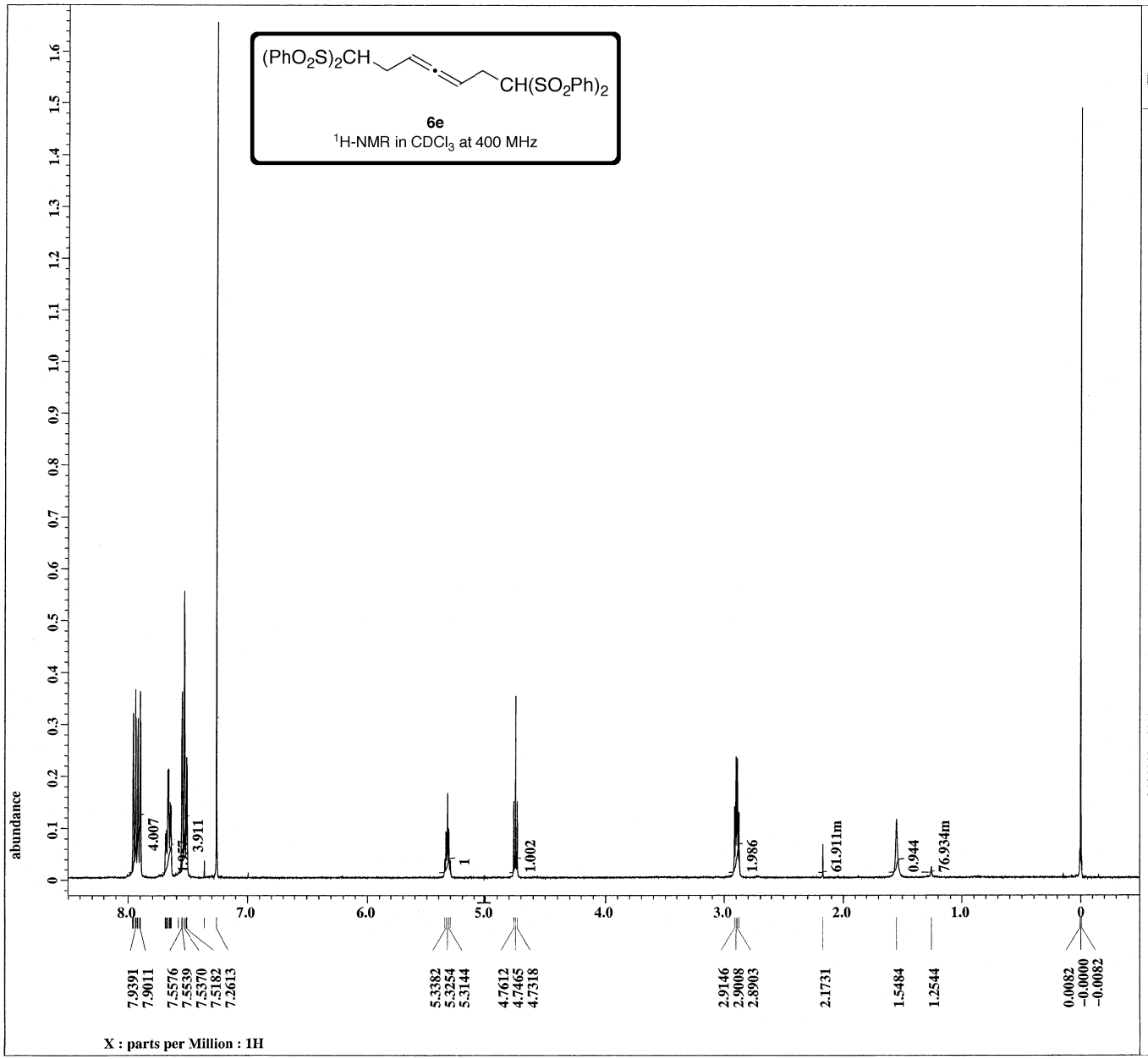
JEOL

```
Filename          = MS_120411_7-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = MS_120411_7
Solvent           = CHLOROFORM-D
Creation_time     = 11-APR-2012 18:30:05
Revision_time     = 11-APR-2012 18:34:32
Current_time      = 11-APR-2012 18:34:47

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400M

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 5.46308096[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.18304689[Hz]
X_sweep           = 5.99808061[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 11.5[us]
X_acq_time        = 5.46308096[s]
X_angle           = 45[deg]
X_atn             = 2.6[dB]
X_pulse           = 5.75[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 44
Relaxation_delay  = 1.66961[s]
Repetition_time   = 7.13269096[s]
Temp_get          = 19.2[dC]
```



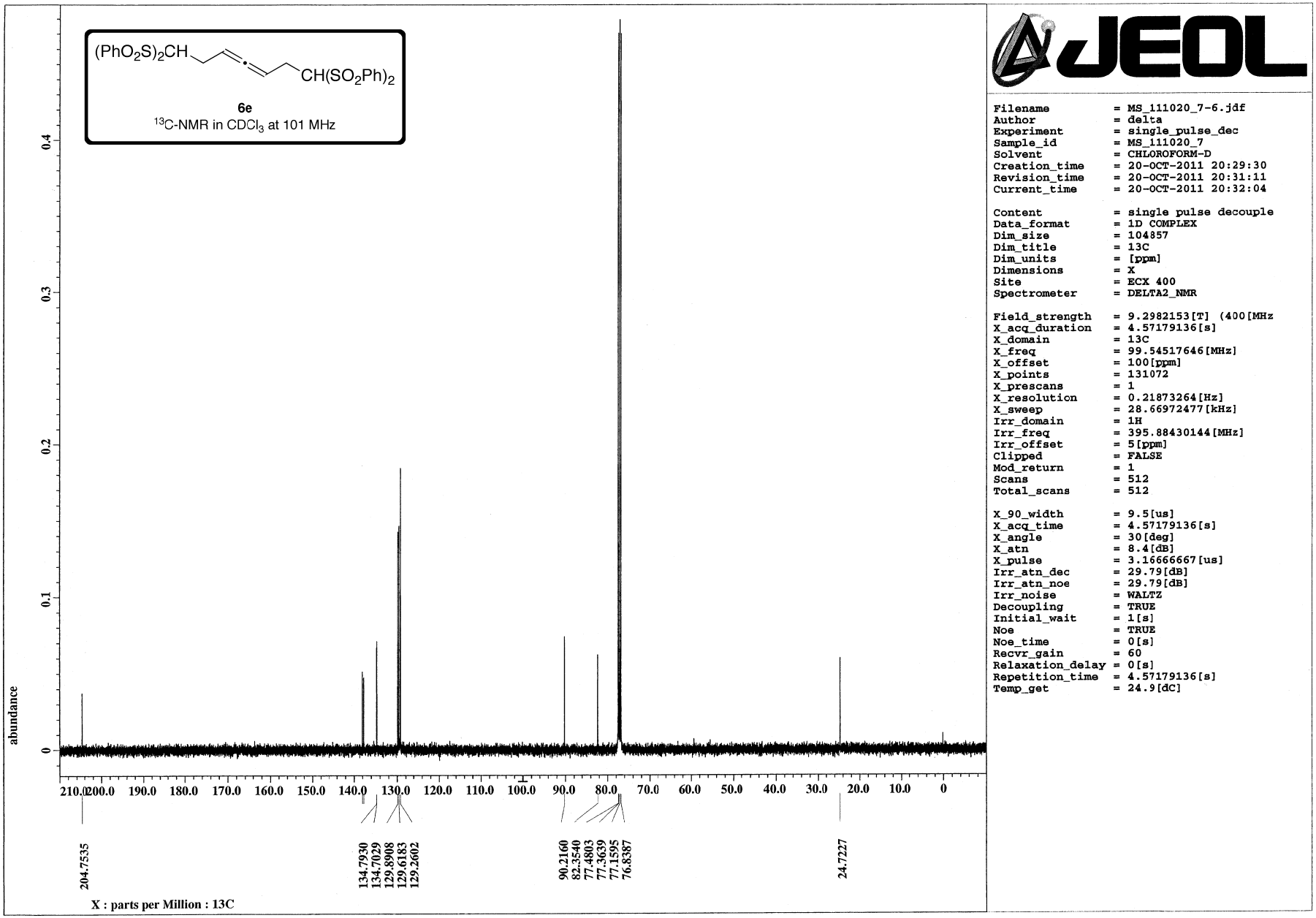
JEOL

```
Filename          = MS_120411_6-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = MS_120411_6
Solvent           = CHLOROFORM-D
Creation_time     = 11-APR-2012 12:12:51
Revision_time     = 11-APR-2012 12:29:06
Current_time      = 11-APR-2012 12:29:36

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 52428
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = DELTA2_NMR

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 2.28589568[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 65536
X_prescans        = 1
X_resolution      = 0.43746528[Hz]
X_sweep           = 28.66972477[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 357
Total_scans       = 357

X_90_width        = 9.5[us]
X_acq_time        = 2.28589568[s]
X_angle           = 30[deg]
X_atn             = 8.4[dB]
X_pulse           = 3.16666667[us]
Irr_atn_dec       = 29.79[dB]
Irr_atn_noe       = 29.79[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1.13759[s]
Recvr_gain        = 60
Relaxation_delay  = 1.13759[s]
Repetition_time   = 3.42348568[s]
Temp_get          = 23.1[dC]
```



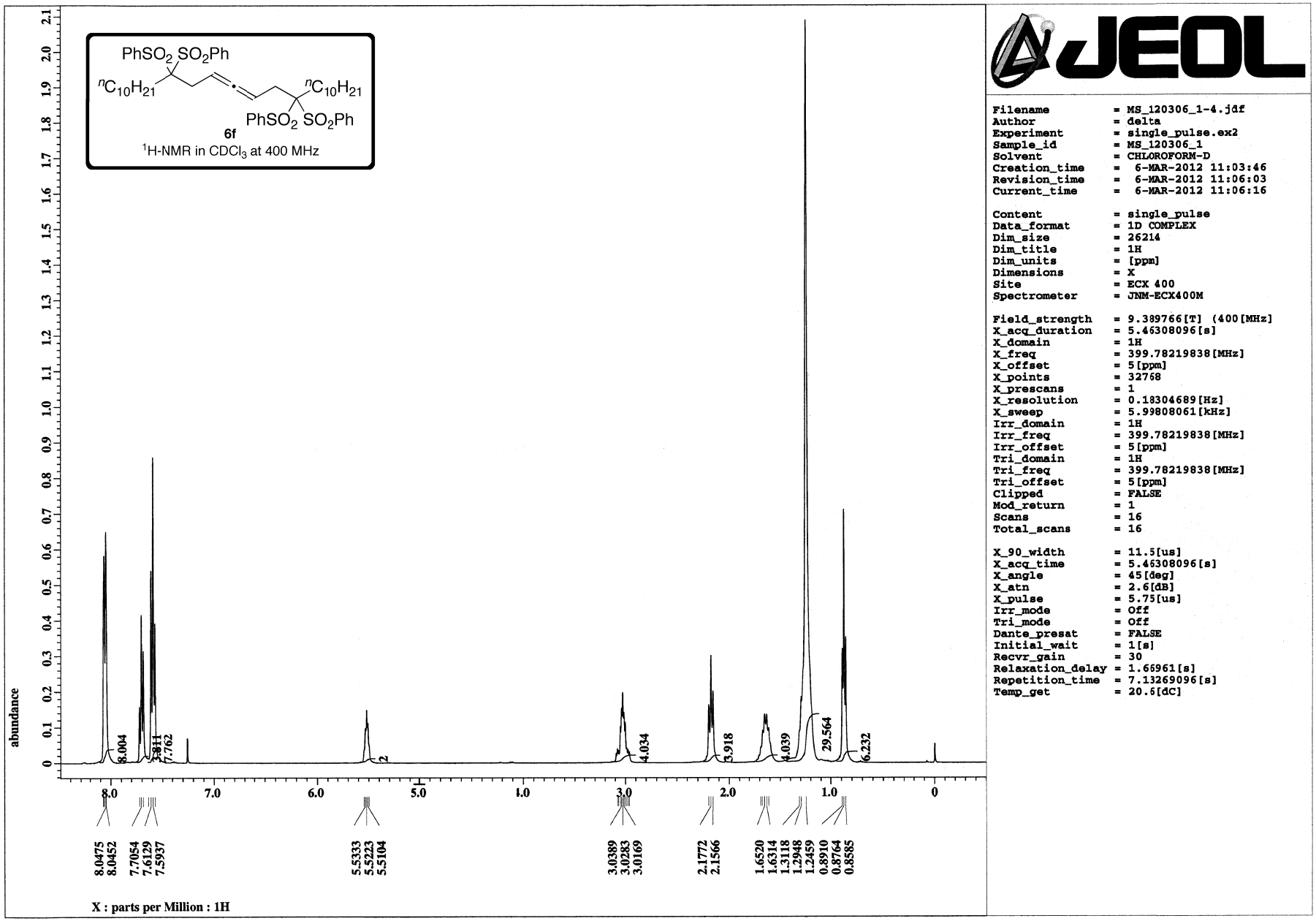
JEOL

```
Filename          = MS_111020_4-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = MS_111020_4
Solvent           = CHLOROFORM-D
Creation_time     = 20-OCT-2011 18:05:02
Revision_time     = 20-OCT-2011 18:07:33
Current_time      = 20-OCT-2011 18:07:45

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = DELTA2_NMR

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 5.51550976[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.1813069[Hz]
X_sweep           = 5.94106464[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10.5[us]
X_acq_time        = 5.51550976[s]
X_angle           = 45[deg]
X_atn             = 9[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 44
Relaxation_delay  = 1.60236[s]
Repetition_time   = 7.11786976[s]
Temp_get          = 24.4[dC]
```

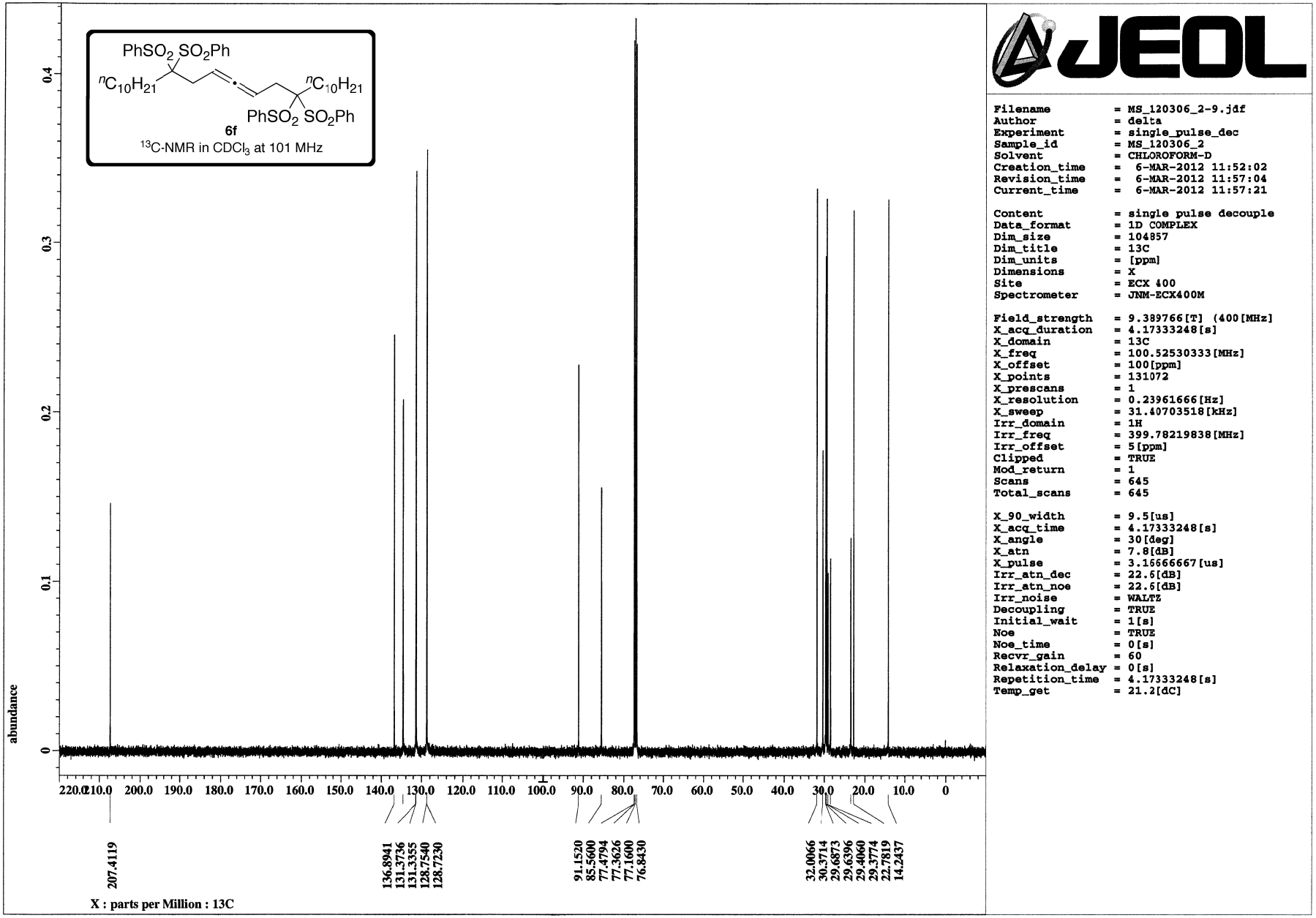


JEOL

```
Filename          = MS_111020_7-6.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = MS_111020_7
Solvent           = CHLOROFORM-D
Creation_time     = 20-OCT-2011 20:29:30
Revision_time     = 20-OCT-2011 20:31:11
Current_time      = 20-OCT-2011 20:32:04

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 104857
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = DELTA2_NMR

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 4.57179136[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 131072
X_prescans        = 1
X_resolution      = 0.21873264[Hz]
X_sweep           = 28.66972477[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 9.5[us]
X_acq_time        = 4.57179136[s]
X_angle           = 30[deg]
X_atn             = 8.4[dB]
X_pulse           = 3.16666667[us]
Irr_atn_dec       = 29.79[dB]
Irr_atn_noe       = 29.79[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 0[s]
Recvr_gain        = 60
Relaxation_delay  = 0[s]
Repetition_time   = 4.57179136[s]
Temp_get          = 24.9[dC]
```

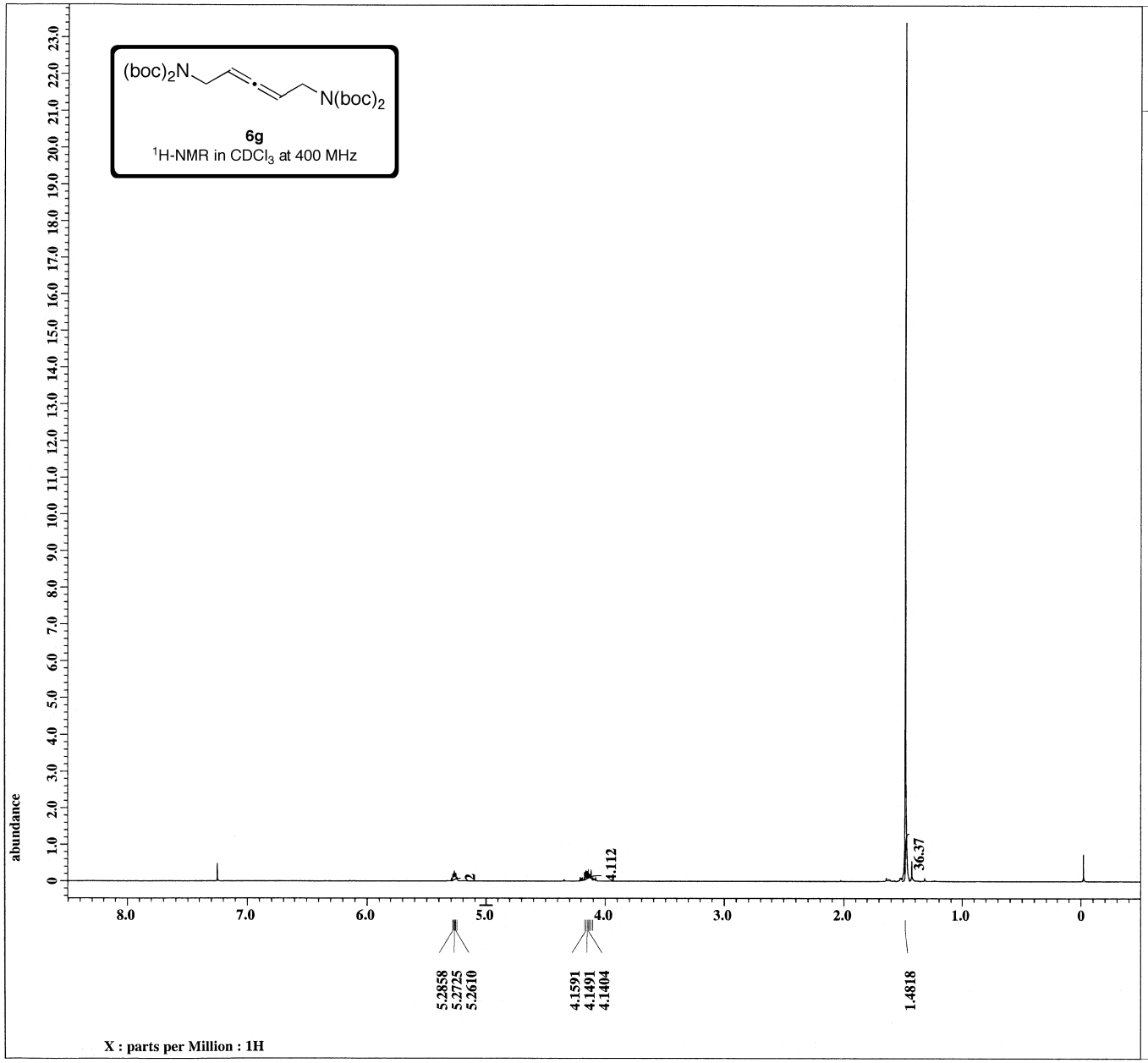



```
Filename          = MS_120306_1-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = MS_120306_1
Solvent           = CHLOROFORM-D
Creation_time     =  6-MAR-2012 11:03:46
Revision_time     =  6-MAR-2012 11:06:03
Current_time      =  6-MAR-2012 11:06:16

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400M

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 5.46308096[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.18304689[Hz]
X_sweep           = 5.99808061[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 11.5[us]
X_acq_time        = 5.46308096[s]
X_angle           = 45[deg]
X_atn             = 2.6[dB]
X_pulse           = 5.75[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 30
Relaxation_delay  = 1.66961[s]
Repetition_time   = 7.13269096[s]
Temp_get          = 20.6[dC]
```



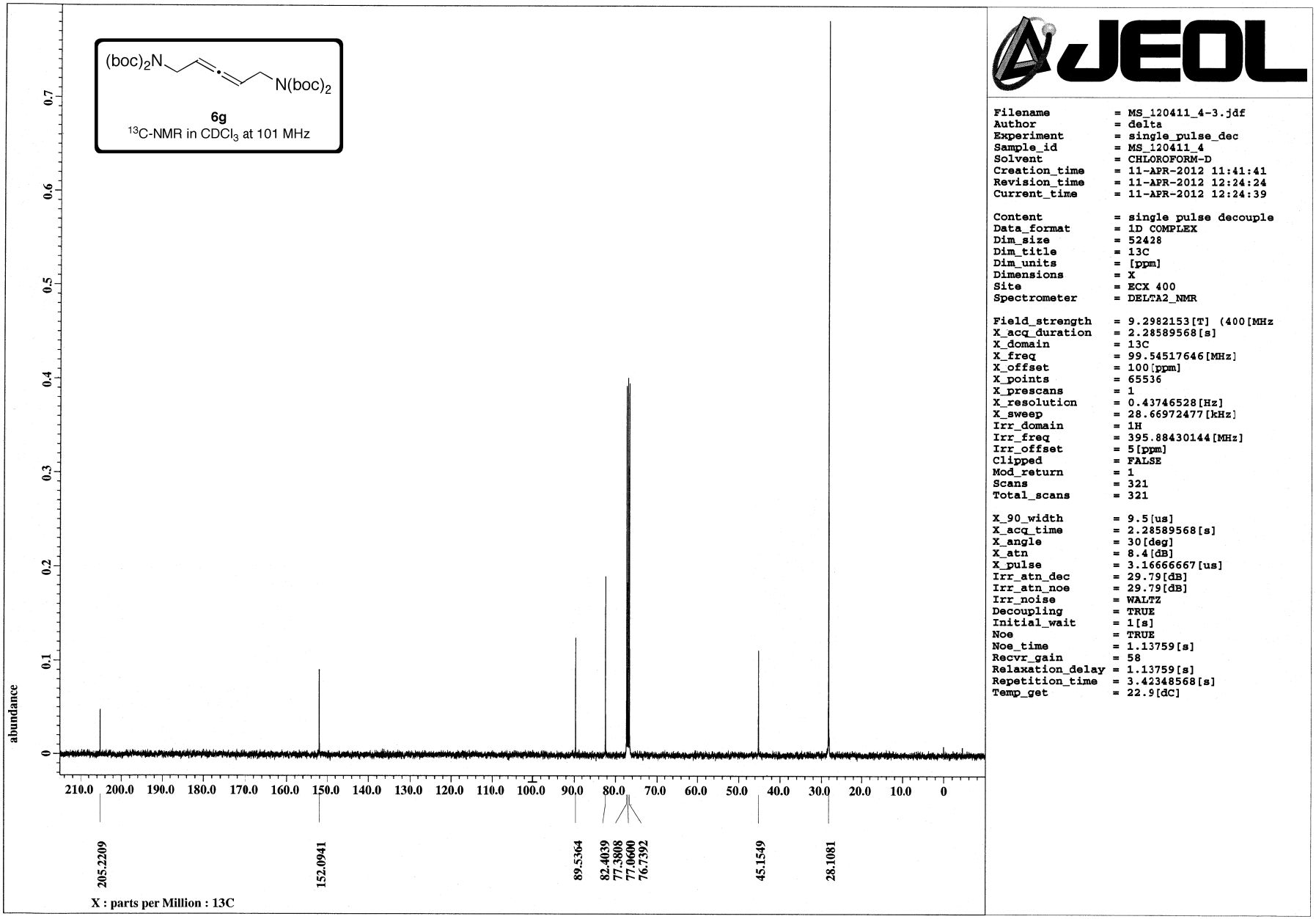
JEOL

```
Filename          = MS_120306_2-9.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = MS_120306_2
Solvent           = CHLOROFORM-D
Creation_time     =  6-MAR-2012 11:52:02
Revision_time     =  6-MAR-2012 11:57:04
Current_time      =  6-MAR-2012 11:57:21

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 104857
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400M

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 4.17333248[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 131072
X_prescans        = 1
X_resolution      = 0.23961666[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 645
Total_scans       = 645

X_90_width        = 9.5[us]
X_acq_time        = 4.17333248[s]
X_angle           = 30[deg]
X_atn             = 7.8[dB]
X_pulse           = 3.16666667[us]
Irr_atn_dec       = 22.5[dB]
Irr_atn_noe       = 22.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 0[s]
Recvr_gain        = 60
Relaxation_delay  = 0[s]
Repetition_time   = 4.17333248[s]
Temp_get          = 21.2[dC]
```



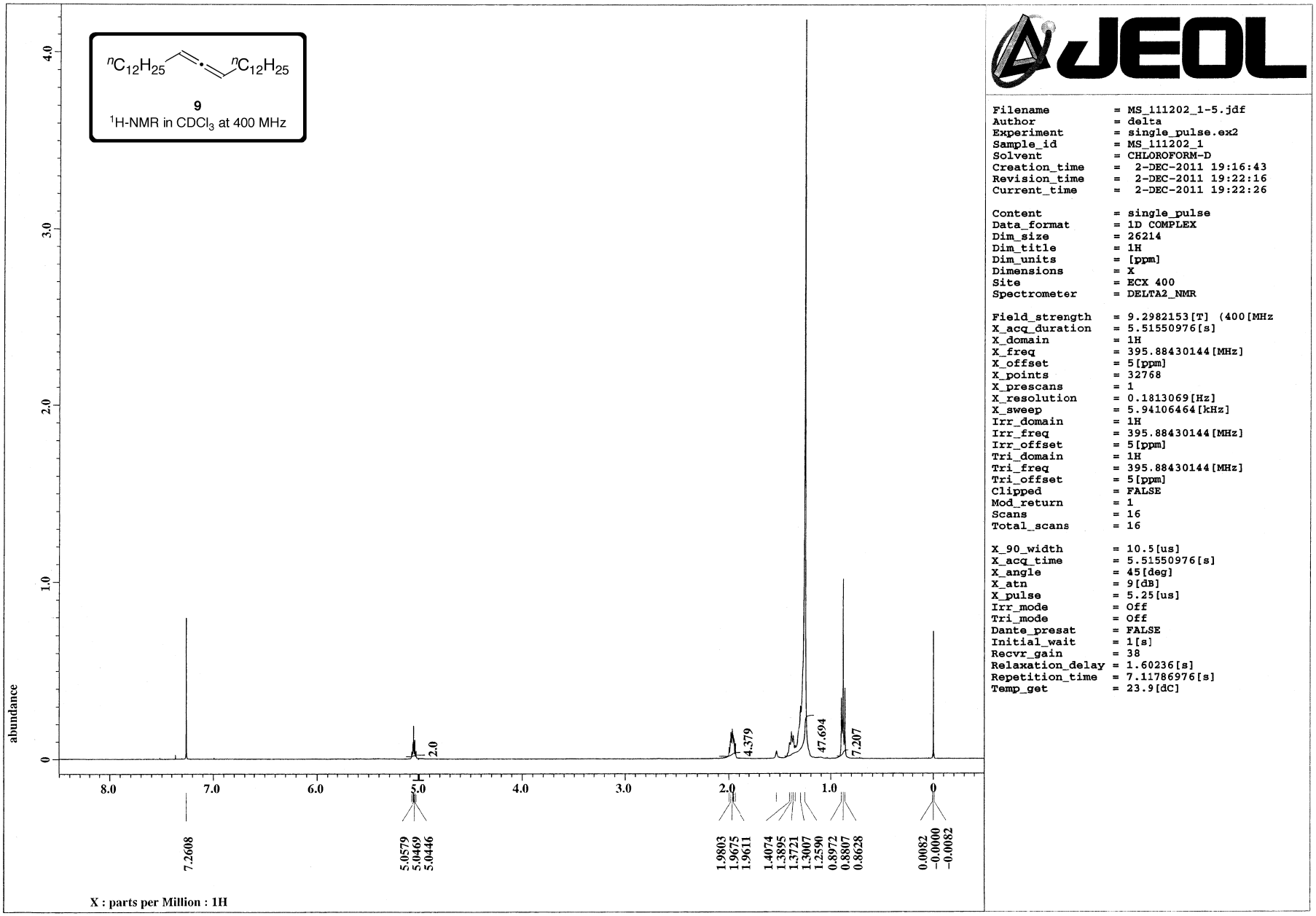
JEOL

```
Filename          = MS_120411_3-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = MS_120411_3
Solvent           = CHLOROFORM-D
Creation_time     = 11-APR-2012 11:19:47
Revision_time     = 11-APR-2012 11:42:31
Current_time      = 11-APR-2012 11:43:48

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = DELTA2_NMR

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 5.51550976[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.1813069[Hz]
X_sweep           = 5.94106464[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10.5[us]
X_acq_time        = 5.51550976[s]
X_angle           = 45[deg]
X_atn             = 9[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 1.60236[s]
Repetition_time   = 7.11786976[s]
Temp_get          = 22.7[dC]
```



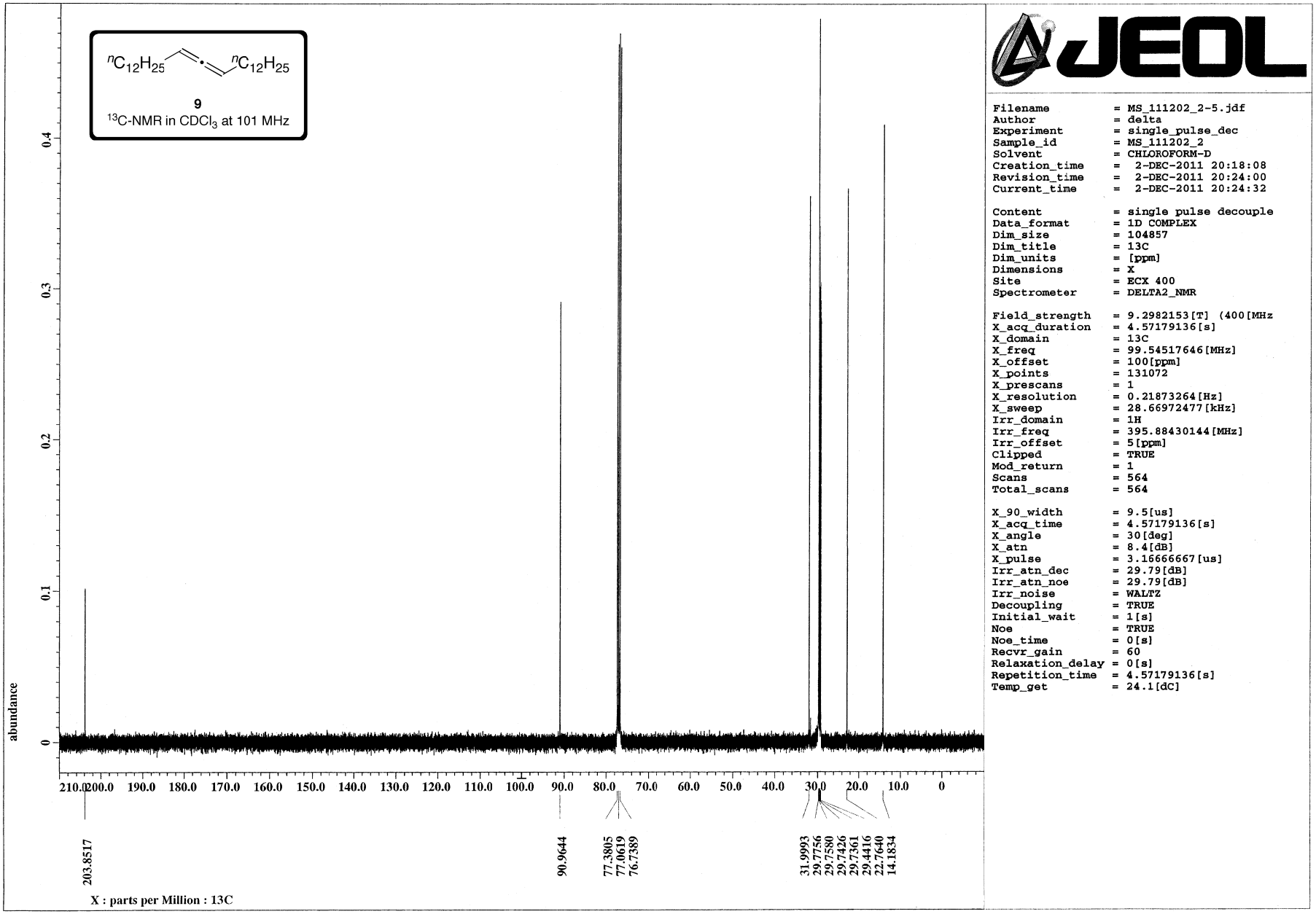
JEOL

Filename	= MS_120411_4-3.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= MS_120411_4
Solvent	= CHLOROFORM-D
Creation_time	= 11-APR-2012 11:41:41
Revision_time	= 11-APR-2012 12:24:24
Current_time	= 11-APR-2012 12:24:39
Content	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 52428
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= DELTA2_NMR
Field_strength	= 9.2982153[T] (400[MHz
X_acq_duration	= 2.28589568[s]
X_domain	= 13C
X_freq	= 99.54517646[MHz]
X_offset	= 100[ppm]
X_points	= 65536
X_prescans	= 1
X_resolution	= 0.43746528[Hz]
X_sweep	= 28.66972477[kHz]
Irr_domain	= 1H
Irr_freq	= 395.88430144[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 321
Total_scans	= 321
X_90_width	= 9.5[us]
X_acq_time	= 2.28589568[s]
X_angle	= 30[deg]
X_atn	= 8.4[dB]
X_pulse	= 3.16666667[us]
Irr_atn_dec	= 29.79[dB]
Irr_atn_noe	= 29.79[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 1.13759[s]
Recvr_gain	= 58
Relaxation_delay	= 1.13759[s]
Repetition_time	= 3.42348568[s]
Temp_get	= 22.9[dC]



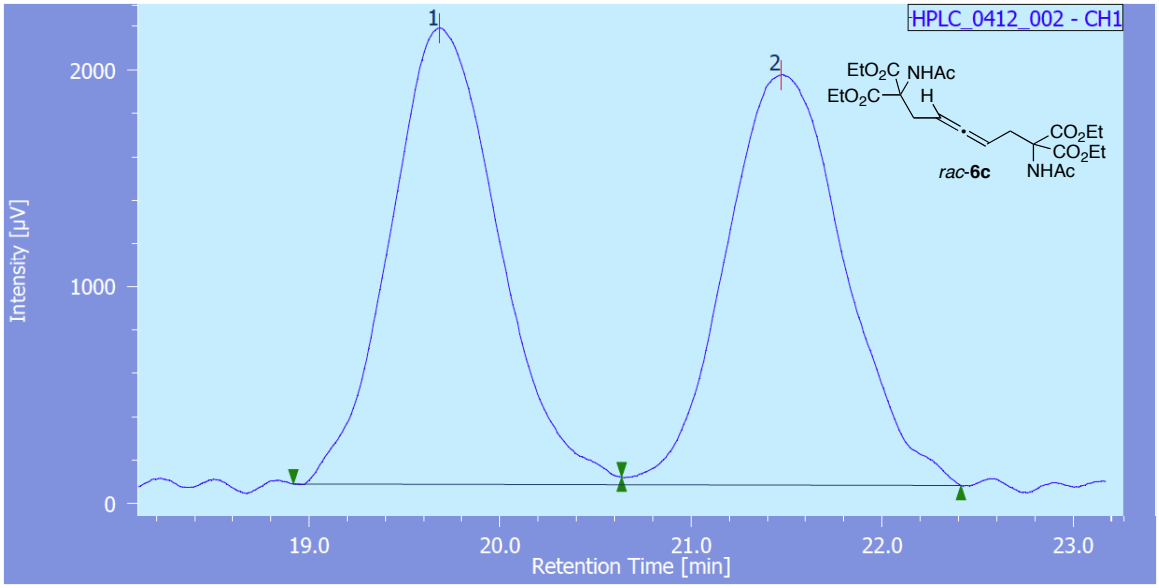
JEOL

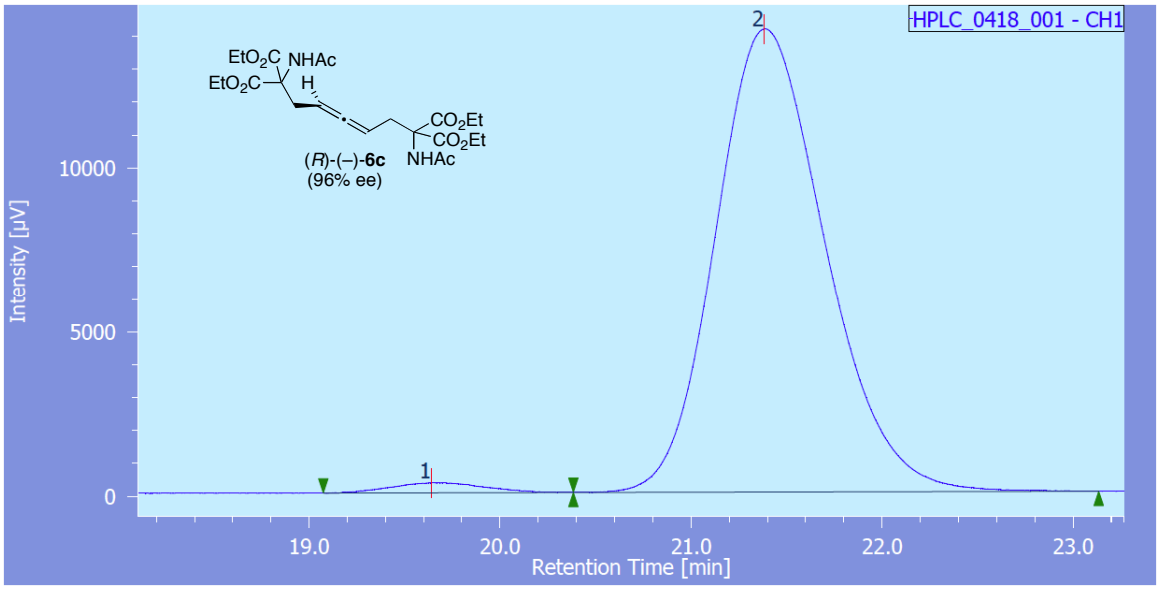
```
Filename          = MS_111202_1-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = MS_111202_1
Solvent           = CHLOROFORM-D
Creation_time     =  2-DEC-2011 19:16:43
Revision_time     =  2-DEC-2011 19:22:16
Current_time      =  2-DEC-2011 19:22:26

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = DELTA2_NMR

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 5.51550976[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.1813069[Hz]
X_sweep           = 5.94106464[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 10.5[us]
X_acq_time        = 5.51550976[s]
X_angle           = 45[deg]
X_atn             = 9[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 1.60236[s]
Repetition_time   = 7.11786976[s]
Temp_get          = 23.9[dC]
```


$^{n}C_{12}H_{25}$ $^{n}C_{12}H_{25}$
9
^{13}C-NMR in $CDCl_3$ at 101 MHz
abundance
0
0.1
0.2
0.3
0.4
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
203.8517
90.9644
77.3805
77.0619
76.7389
31.9993
29.7756
29.7580
29.7426
29.7361
29.4416
22.7640
14.1834
X : parts per Million : 13C
JEOL
Filename = MS_111202_2-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = MS_111202_2
Solvent = CHLOROFORM-D
Creation_time = 2-DEC-2011 20:18:08
Revision_time = 2-DEC-2011 20:24:00
Current_time = 2-DEC-2011 20:24:32
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 104857
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = DELTA2_NMR
Field_strength = 9.2982153[T] (400[MHz
X_acq_duration = 4.57179136[s]
X_domain = 13C
X_freq = 99.54517646[MHz]
X_offset = 100[ppm]
X_points = 131072
X_prescans = 1
X_resolution = 0.21873264[Hz]
X_sweep = 28.66972477[kHz]
Irr_domain = 1H
Irr_freq = 395.88430144[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 564
Total_scans = 564
X_90_width = 9.5[us]
X_acq_time = 4.57179136[s]
X_angle = 30[deg]
X_atn = 8.4[dB]
X_pulse = 3.16666667[us]
Irr_atn_dec = 29.79[dB]
Irr_atn_noe = 29.79[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 0[s]
Recvr_gain = 60
Relaxation_delay = 0[s]
Repetition_time = 4.57179136[s]
Temp_get = 24.1[dC]

Chiral HPLC Analysis of (*R*)-(–)-6c (Scheme 4).

chiral column: Chiralpak AD-H; eluent: hexane/^{i}PrOH = 4/1; flow rate: 0.8 mL/min.





#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*S*)-isomer	1	19.642	10431	304	1.821	2.113	7068	1.763	1.124
2	(*R*)-isomer	1	21.383	562317	14083	98.179	97.887	6679	N/A	1.191