Supporting Information

Selective Formylation of Azacalixpyridine Macrocycles and Their Transformation to Molecular Semi-cages

Wen-Sheng Ren, Liang Zhao, Mei-Xiang Wang*

Key Laboratory of Bioorganic Phosphorus Chemistry and Chemical Biology (Ministry of Education), Department of Chemistry, Tsinghua University, Beijing 100084, China

wangmx@mail.tsinghua.edu.cn

Table of Contents

1. General Information……..…….S1

2. Copies of ^{1}H and ^{13}C NMR spectra……..…….S2-S24

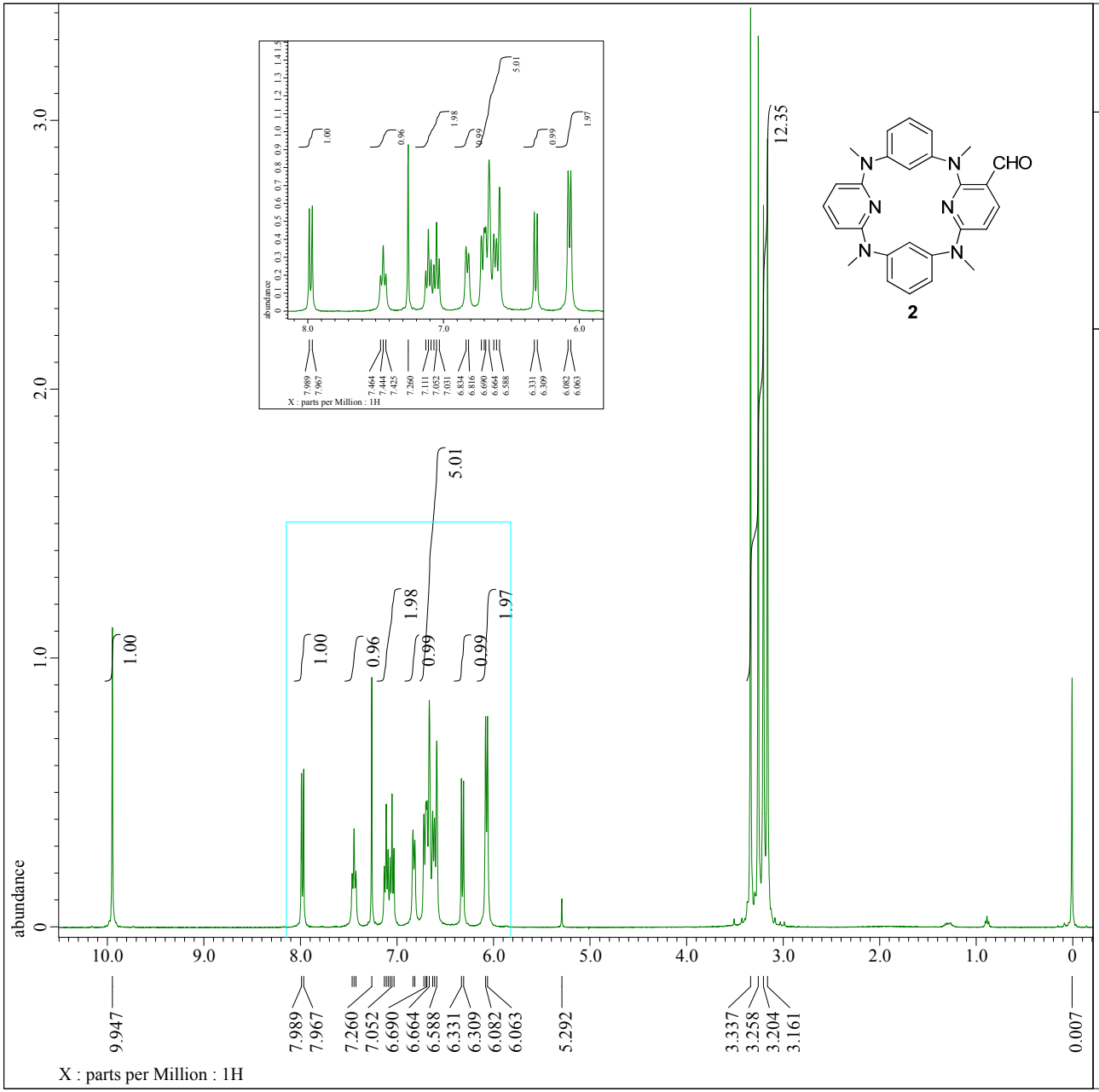
3. Crystal data and structure refinement for each X-ray structure…….. ……S25-S30

4. Atomic coordinates for each X-ray structure…….. ……S31-S36

1. General Information

All commercially available reagents were used as received. TLC analysis was performed on pre-coated, glass-backed silica gel plates and visualized with UV light. Flash column chromatography was performed on silica gel (100-200). Anhydrous DMF, THF and 1,2-dichloroethane were dried by 4 Å molecular sieve. ^{1}H NMR and ^{13}C NMR spectra were recorded using 400 MHz spectrometers. Chemical shifts are reported in ppm versus tetramethylsilane with either tetramethylsilane or the residual solvent resonance used as an internal standard. Infrared spectra were recorded using a FT-IR spectrometer with KBr discs in the 4000-400 cm^{-1} region. Melting points are uncorrected.

2. Copies of ^{1}H and ^{13}C NMR spectra

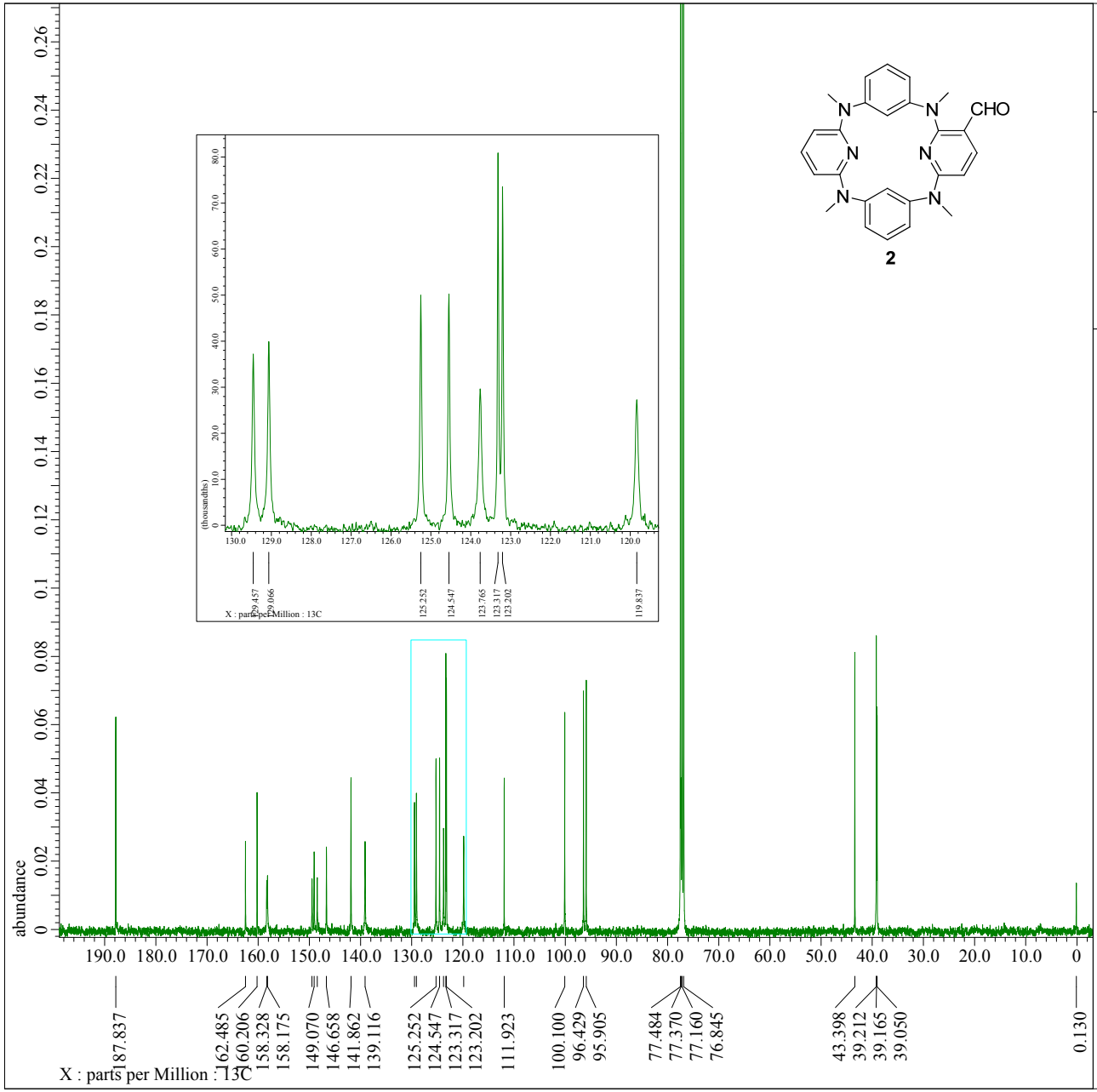


2

JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

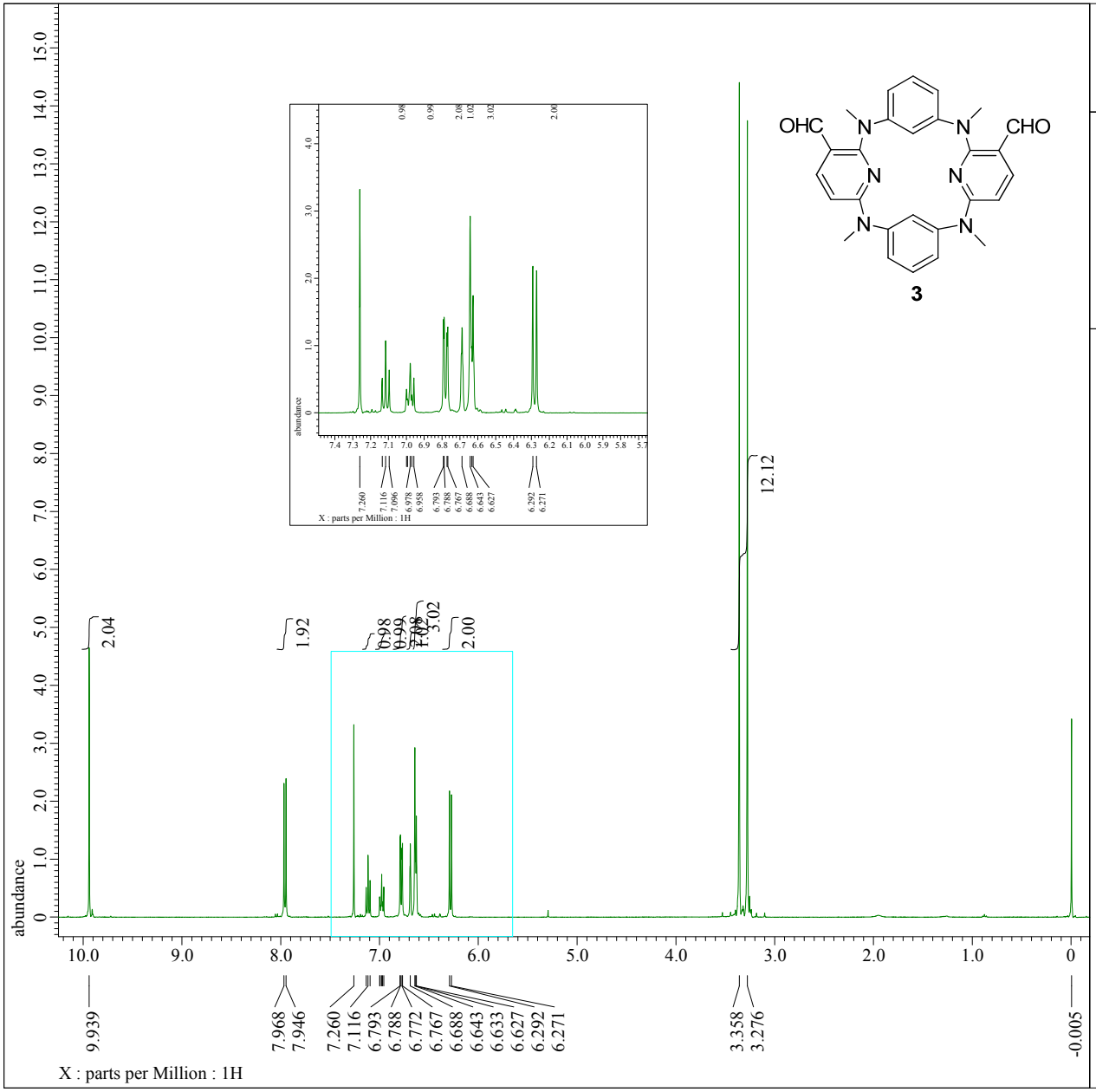
```
Filename          = C:\Users\RWS\Desktop\核磁数据\
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#284483
Solvent           = CHLOROFORM-D
Creation_Time     = 15-FEB-2014 07:11:25
Revision_Time     = 15-JUN-2015 11:21:31
Current_Time      = 15-JUN-2015 11:22:25

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
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
Incomplete_Copy   = TRUE
Scans             = 5
Total_Scans       = 5

Relaxation_Delay  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 22.2[dC]
X_90_Width        = 10.75[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.375[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```



2

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

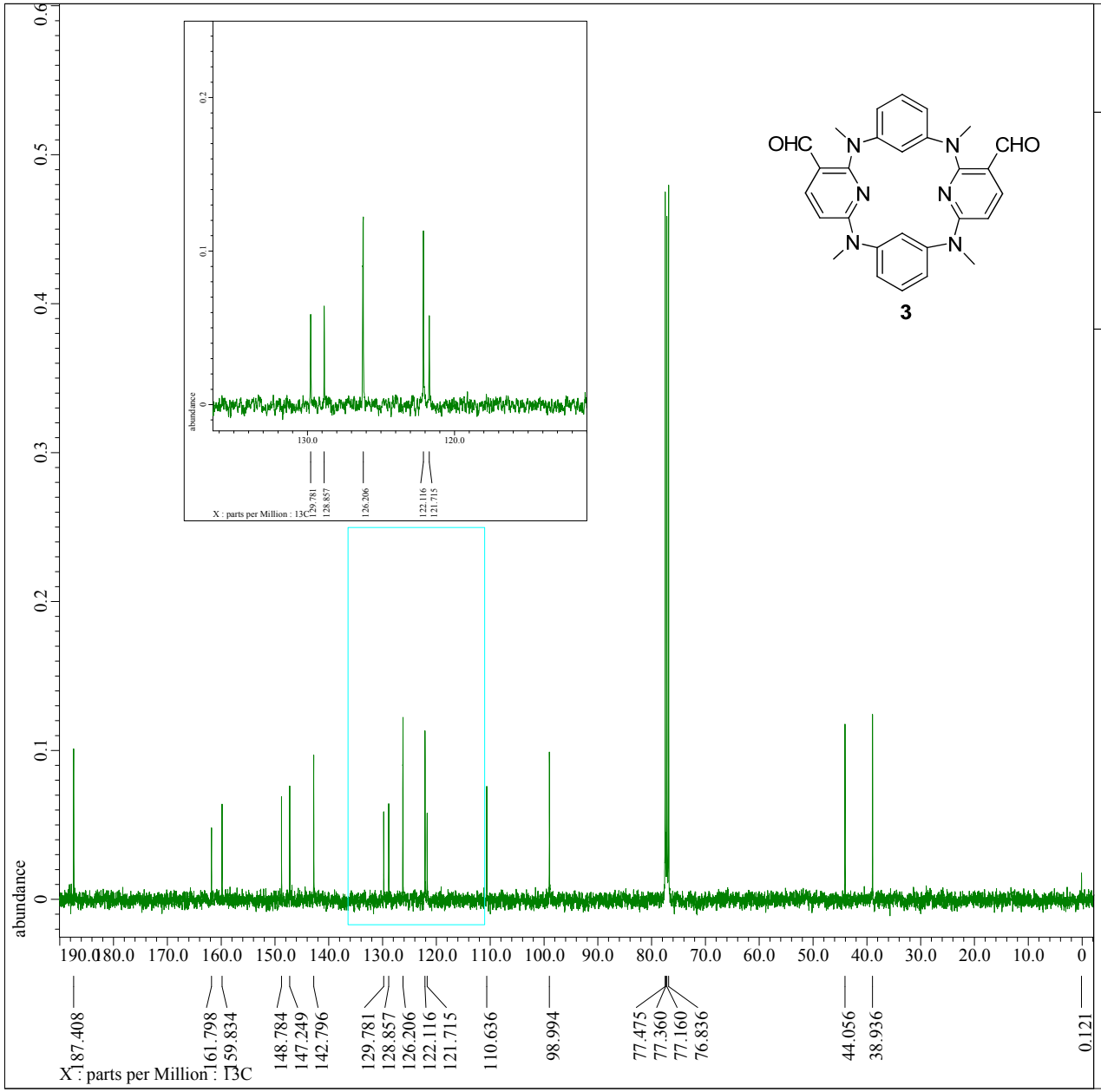
```
Filename          = C:\Users\RWS\Desktop\核磁\rw
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#287455
Solvent           = CHLOROFORM-D
Creation_Time     = 15-FEB-2014 09:07:35
Revision_Time     = 14-JUN-2015 19:05:57
Current_Time      = 14-JUN-2015 19:06:19

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 2177
Total_Scans       = 2177

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21.3[dC]
X_90_Width        = 8.75[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 2.91666667[us]
Irr_Atn_Dec       = 23.98[dB]
Irr_Atn_Noe       = 23.98[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

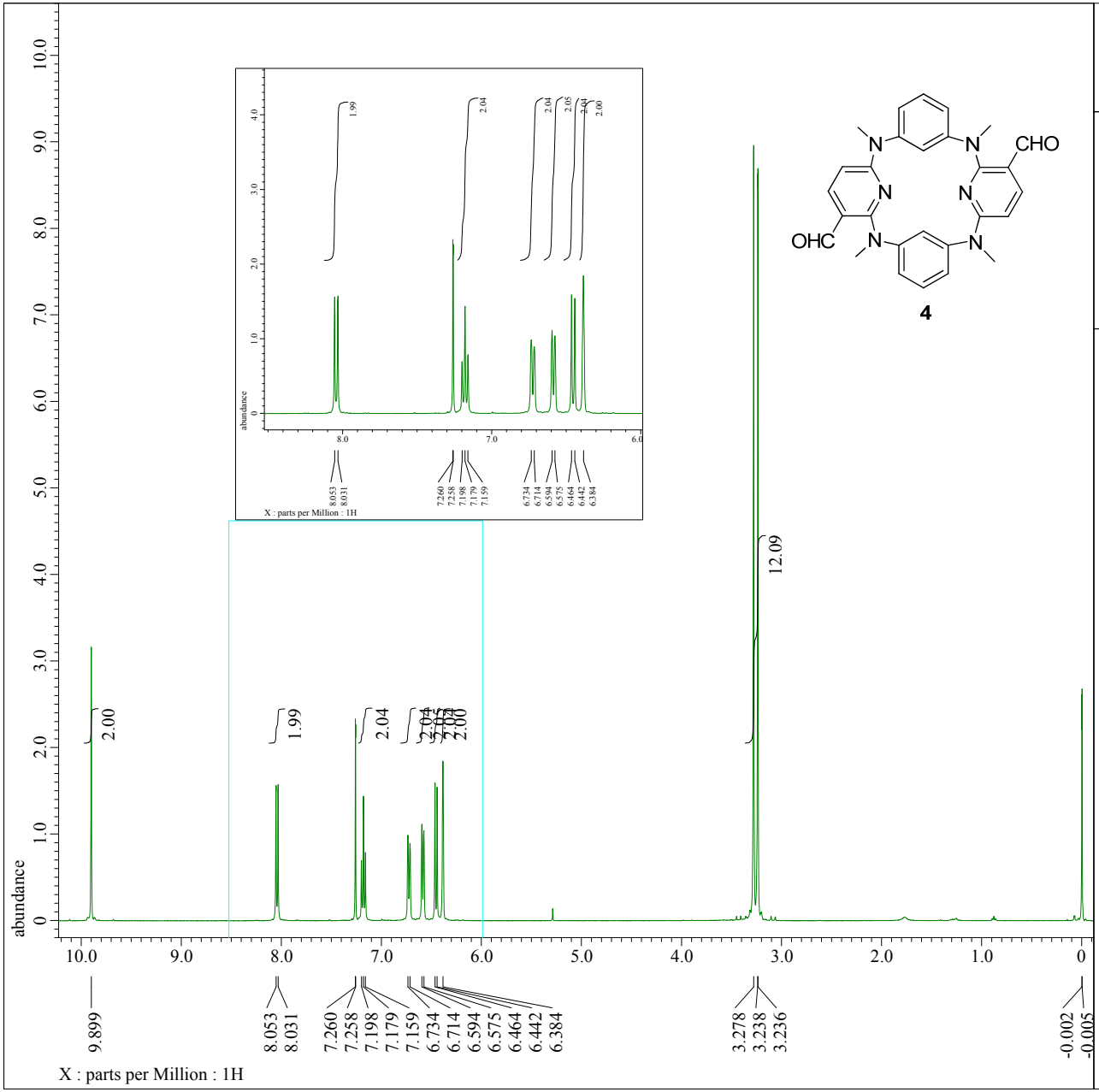
```
Filename          = C:\Users\RWS\Desktop\核磁数据\
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#381099
Solvent           = CHLOROFORM-D
Creation_Time     = 15-FEB-2014 09:52:53
Revision_Time     = 15-JUN-2015 19:34:59
Current_Time      = 15-JUN-2015 19:35:31

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
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
Scans             = 9
Total_Scans       = 9

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 21[dC]
X_90_Width        = 10.75[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.375[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



3

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = C:\Users\RWS\Desktop\核磁数据\
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#382734
Solvent           = CHLOROFORM-D
Creation_Time     = 15-FEB-2014 10:01:20
Revision_Time     = 15-JUN-2015 11:34:35
Current_Time      = 15-JUN-2015 11:34:55

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 106
Total_Scans       = 106

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21.2[dC]
X_90_Width        = 8.75[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 2.91666667[us]
Irr_Atn_Dec       = 23.98[dB]
Irr_Atn_Noe       = 23.98[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```

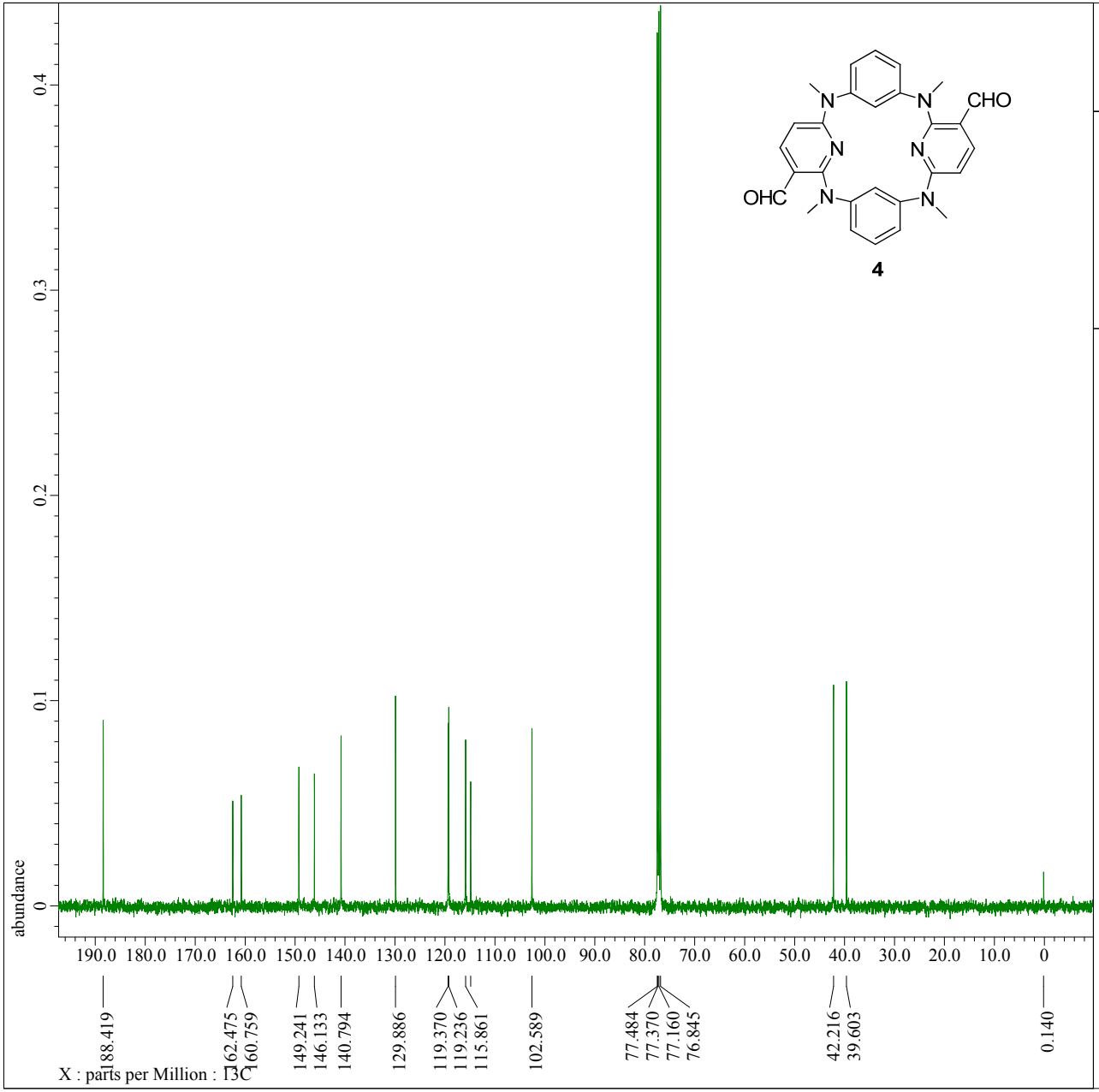


4

JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = C:\Users\RWS\Desktop\核磁数据\
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#361534
Solvent           = CHLOROFORM-D
Creation_Time     = 15-FEB-2014 09:21:06
Revision_Time     = 15-JUN-2015 11:59:37
Current_Time      = 15-JUN-2015 12:04:40

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
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
Scans             = 16
Total_Scans       = 16

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 21[dC]
X_90_Width        = 10.75[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.375[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```

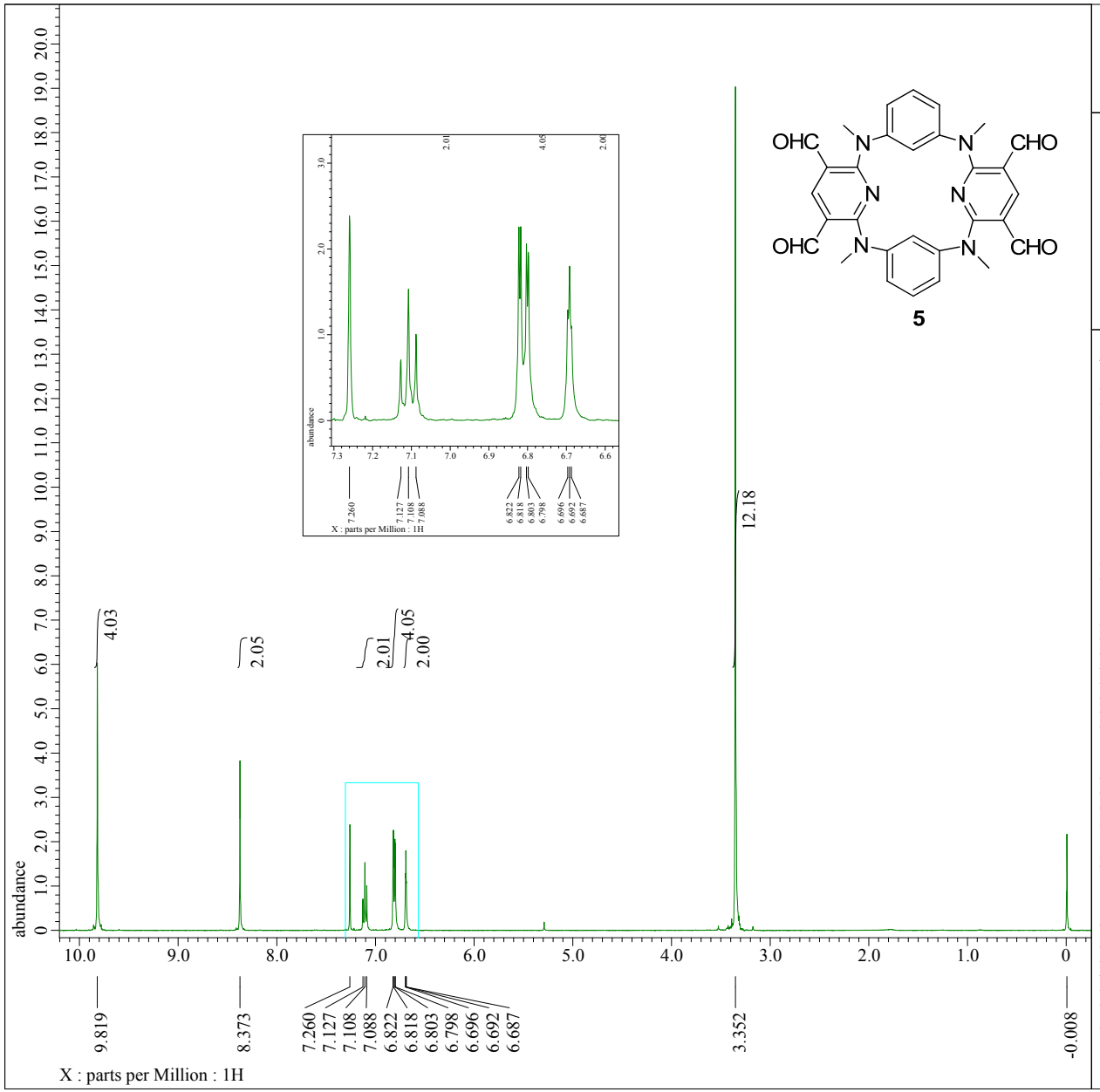


4

JEOL RESONANCE

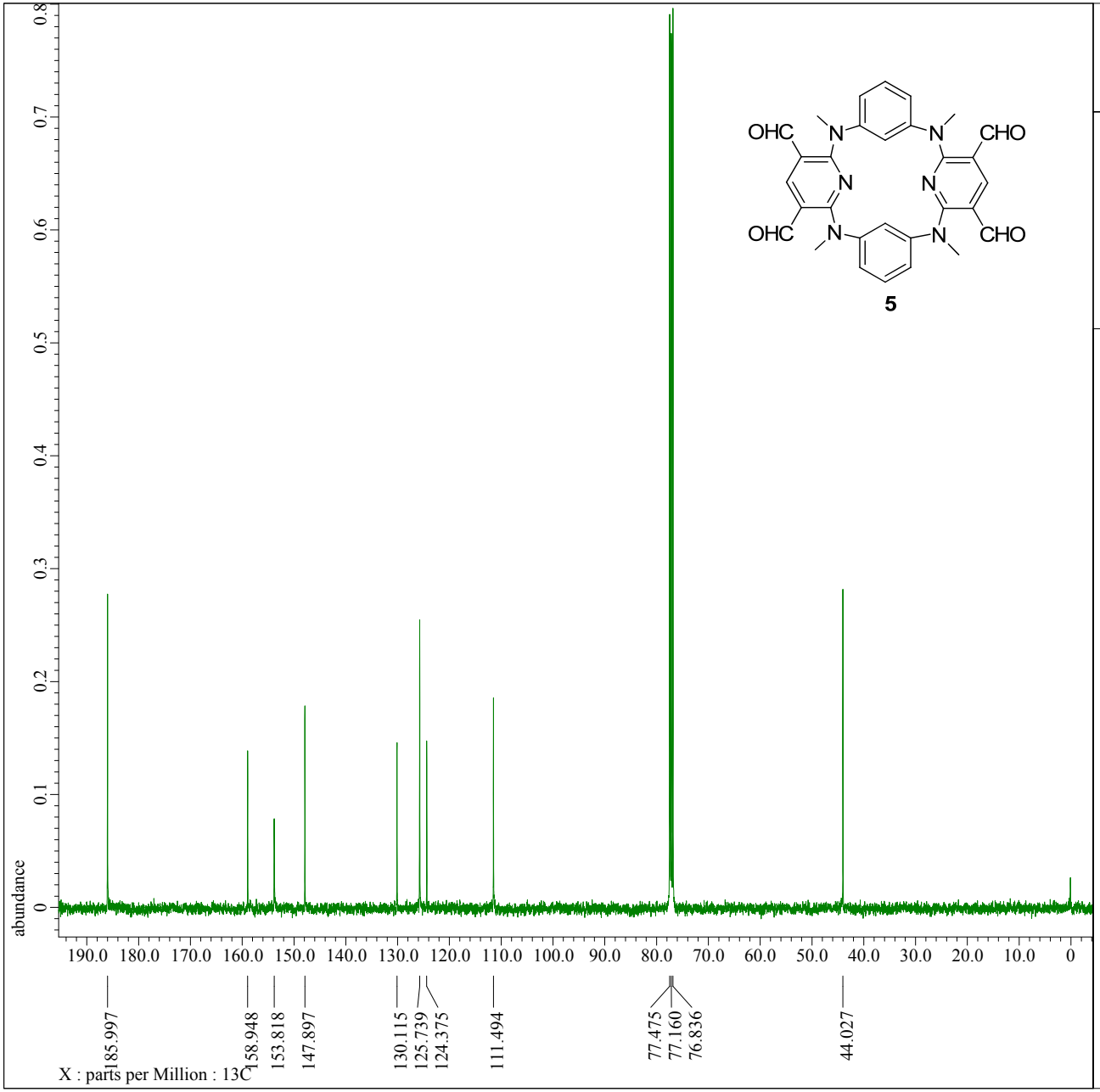
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = C:\Users\RWS\Desktop\核磁\r w
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#364113
Solvent           = CHLOROFORM-D
Creation_Time     = 15-FEB-2014 09:45:48
Revision_Time     = 14-JUN-2015 19:32:29
Current_Time      = 14-JUN-2015 19:32:48

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 412
Total_Scans       = 412

Relaxation_Delay = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21.3[dC]
X_90_Width        = 8.75[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 2.91666667[us]
Irr_Atn_Dec       = 23.98[dB]
Irr_Atn_Noe       = 23.98[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = C:\Users\RWS\Desktop\核磁数据\
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#261542
Solvent           = CHLOROFORM-D
Creation_Time     = 15-FEB-2014 06:33:03
Revision_Time     = 15-JUN-2015 18:58:36
Current_Time      = 15-JUN-2015 18:58:52

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
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
Incomplete_Copy   = TRUE
Scans             = 2
Total_Scans       = 2

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 25.7[dC]
X_90_Width        = 10.75[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.375[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```

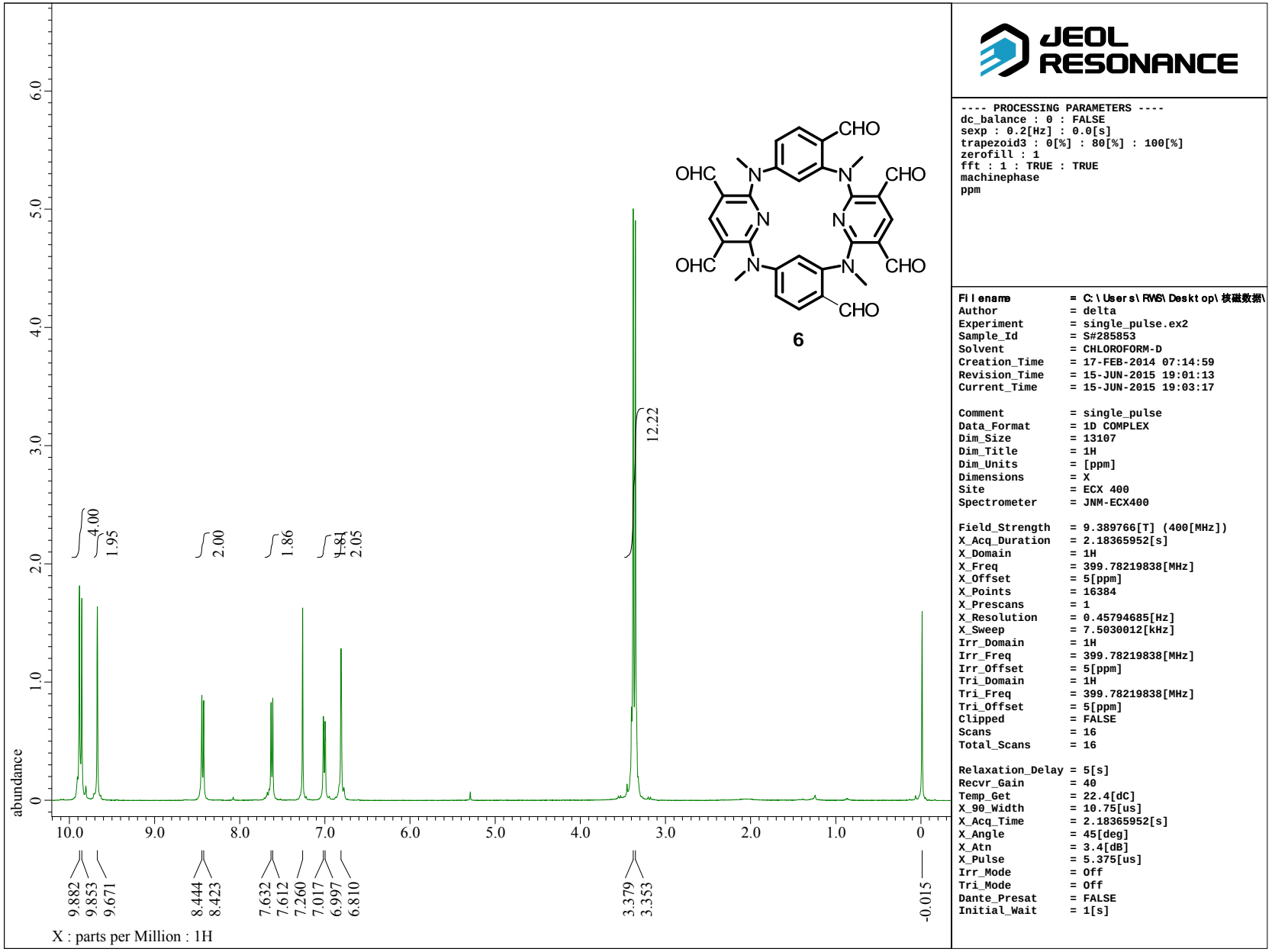


5

JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

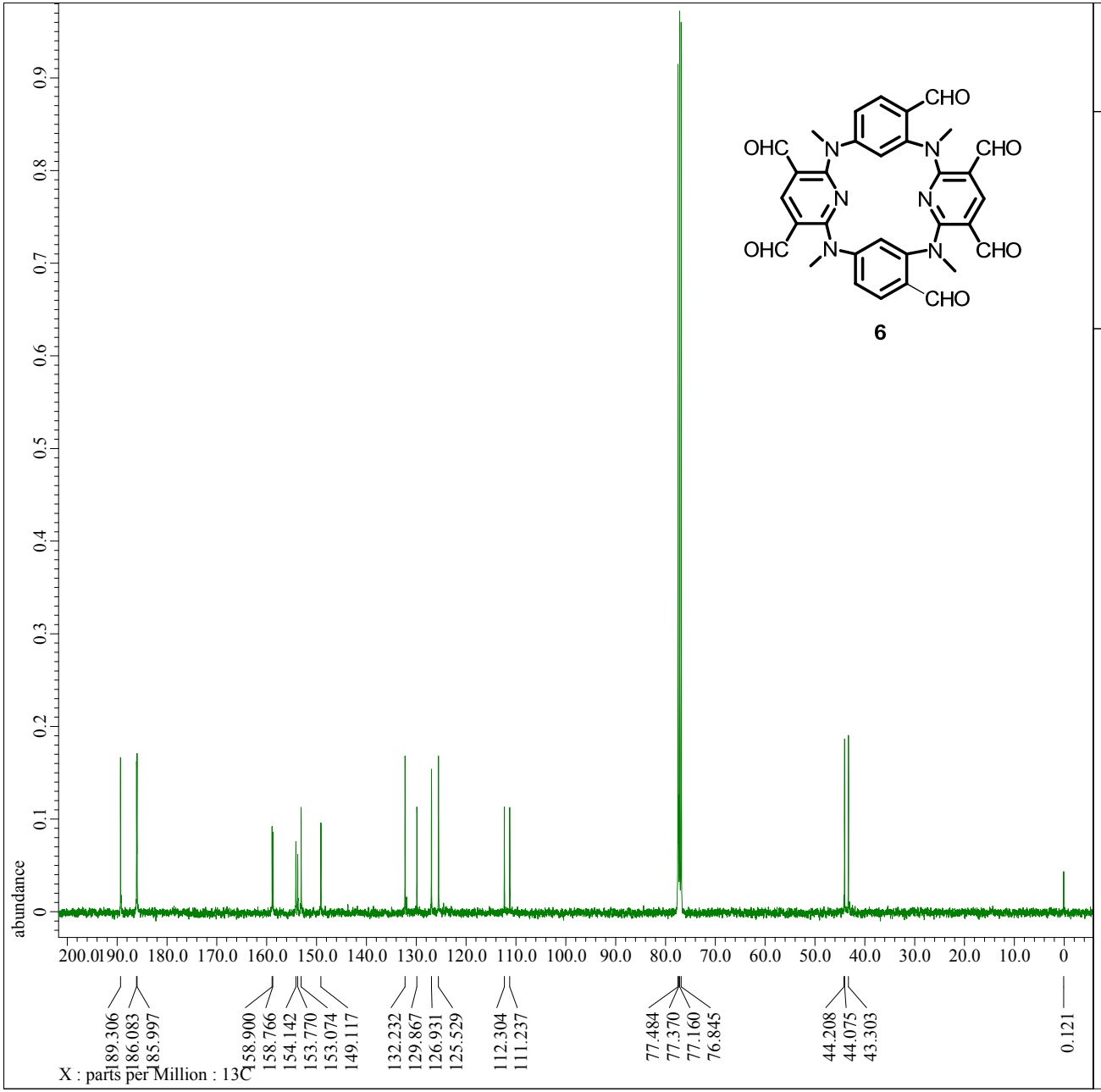
```
Filename          = C:\Users\RWS\Desktop\核磁\r w
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#264536
Solvent           = CHLOROFORM-D
Creation_Time     = 15-FEB-2014 07:06:03
Revision_Time     = 14-JUN-2015 19:41:38
Current_Time      = 14-JUN-2015 19:41:53

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 530
Total_Scans       = 530

Relaxation_Delay  = 2[s]
Recvr_Gain        = 56
Temp_Get          = 22.5[dC]
X_90_Width        = 8.75[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 2.91666667[us]
Irr_Atn_Dec       = 23.98[dB]
Irr_Atn_Noe       = 23.98[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = C:\Users\RWS\Desktop\核磁数据\
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#285853
Solvent           = CHLOROFORM-D
Creation_Time     = 17-FEB-2014 07:14:59
Revision_Time     = 15-JUN-2015 19:01:13
Current_Time      = 15-JUN-2015 19:03:17

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
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
Scans             = 16
Total_Scans       = 16

Relaxation_Delay = 5[s]
Recvr_Gain        = 40
Temp_Get          = 22.4[dC]
X_90_Width        = 10.75[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.375[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```

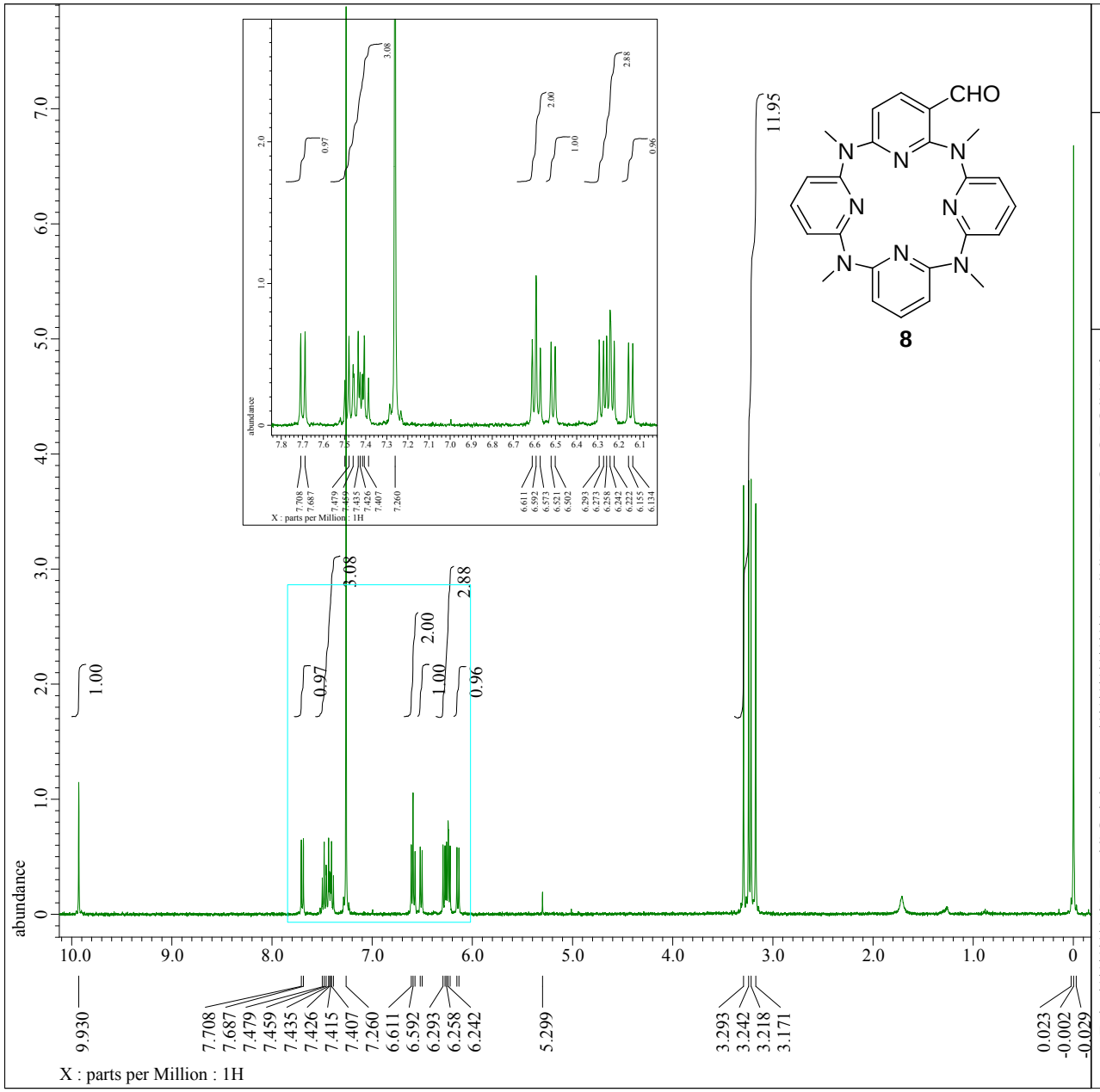


6

JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = C:\Users\RWS\Desktop\核磁\r w
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#287935
Solvent           = CHLOROFORM-D
Creation_Time     = 17-FEB-2014 08:08:47
Revision_Time     = 14-JUN-2015 20:01:34
Current_Time      = 14-JUN-2015 20:02:26

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1000
Total_Scans       = 1000

Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
Temp_Get          = 22.5[dC]
X_90_Width        = 8.75[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 2.91666667[us]
Irr_Atn_Dec       = 23.98[dB]
Irr_Atn_Noe       = 23.98[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```

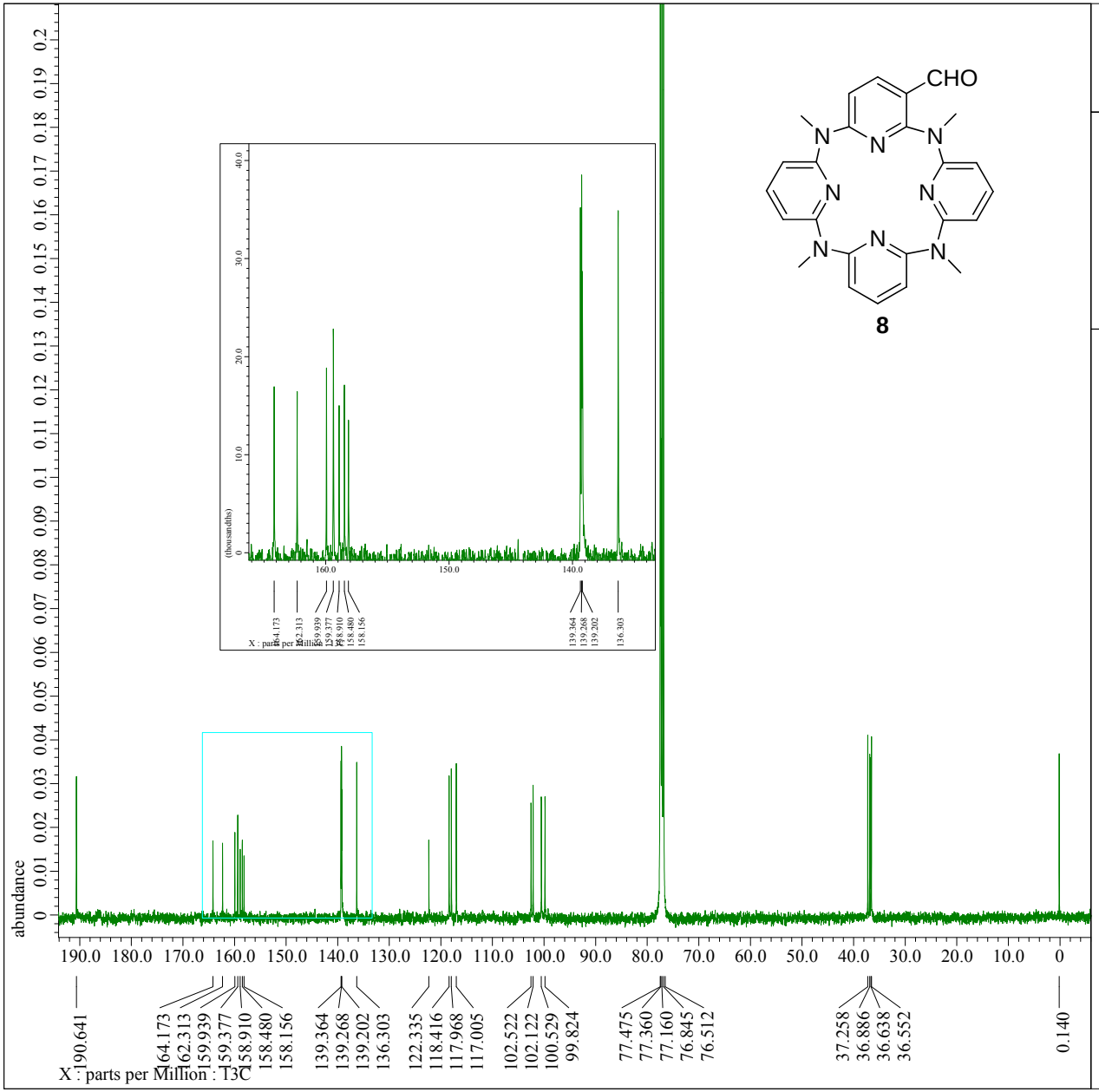


8

JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

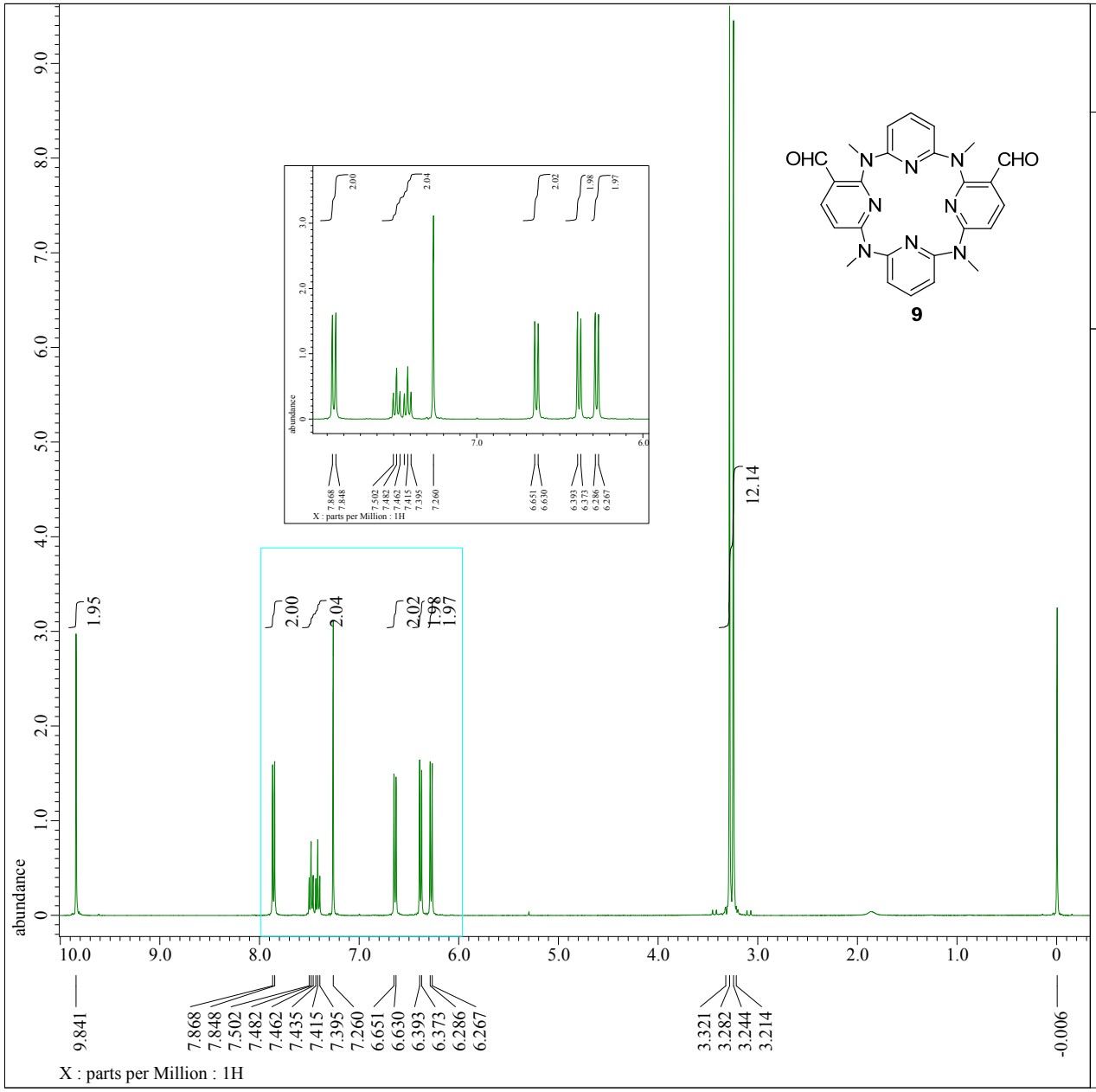
```
Filename          = C:\Users\RWS\Desktop\核磁数据\
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#726646
Solvent           = CHLOROFORM-D
Creation_Time     = 24-APR-2014 19:28:35
Revision_Time     = 15-JUN-2015 19:10:22
Current_Time      = 15-JUN-2015 19:10:26

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
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
Scans             = 32
Total_Scans       = 32

Relaxation_Delay  = 5[s]
Recvr_Gain        = 72
Temp_Get          = 18.2[dC]
X_90_Width        = 10.75[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.375[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



JEOL RESONANCE

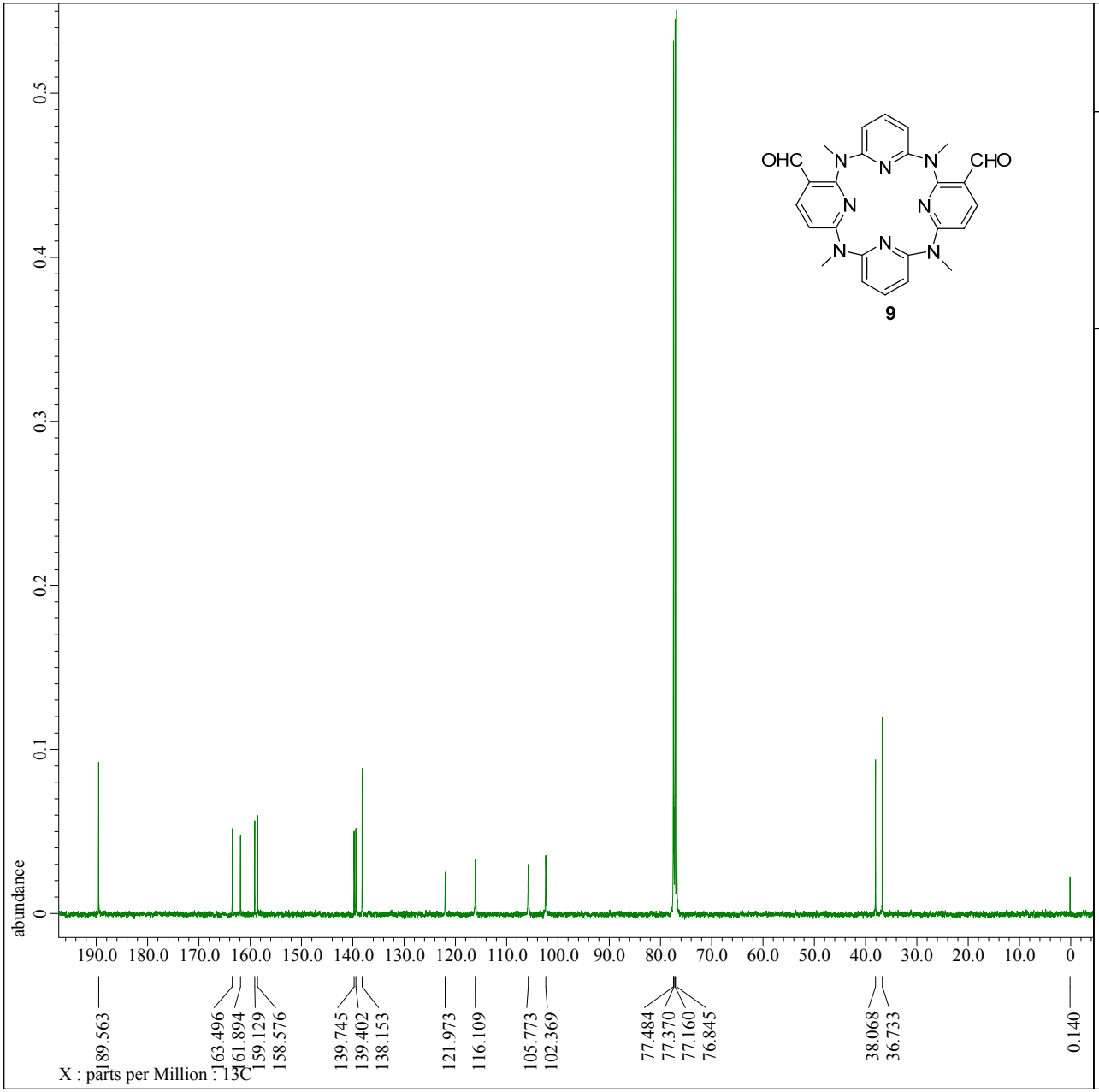
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

Filename	= C:\Users\RWS\Desktop\核磁数据\
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#16078
Solvent	= CHLOROFORM-D
Creation_Time	= 25-APR-2014 07:20:04
Revision_Time	= 15-JUN-2015 19:12:45
Current_Time	= 15-JUN-2015 19:14:25
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
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]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 9020
Total_Scans	= 9020
Relaxation_Delay	= 2[s]
Recvr_Gain	= 56
Temp_Get	= 16.1[dC]
X_90_Width	= 8.75[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 7.8[dB]
X_Pulse	= 2.91666667[us]
Irr_Atn_Dec	= 23.98[dB]
Irr_Atn_Noe	= 23.98[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

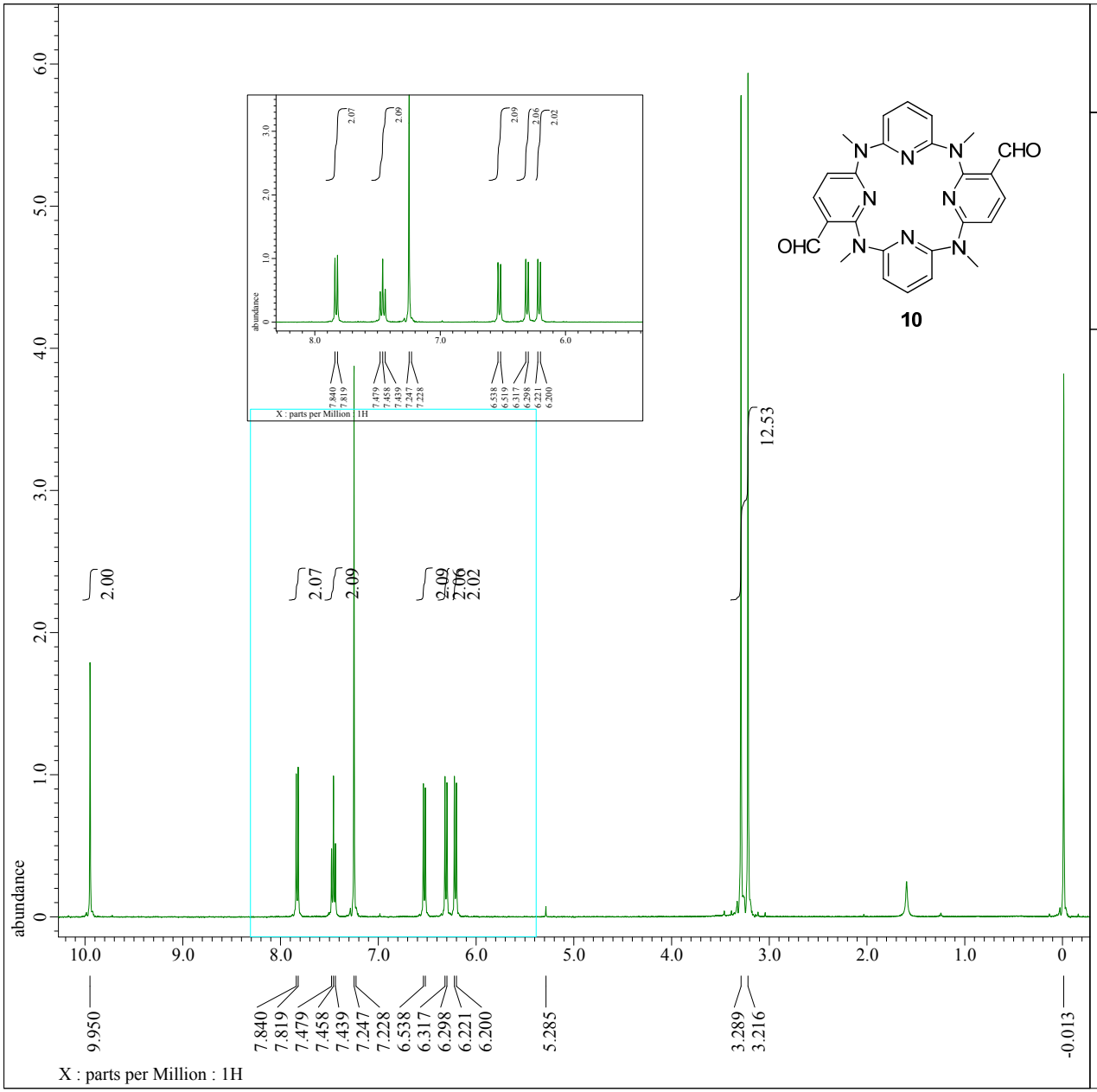
```
Filename          = C:\Users\RWS\Desktop\核磁数据\
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#323531
Solvent           = CHLOROFORM-D
Creation_Time     = 27-APR-2014 08:14:51
Revision_Time     = 15-JUN-2015 19:16:04
Current_Time      = 15-JUN-2015 19:17:03

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
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
Scans             = 14
Total_Scans       = 14

Relaxation_Delay  = 5[s]
Recvr_Gain        = 42
Temp_Get          = 17.6[dC]
X_90_Width        = 10.75[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.375[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```

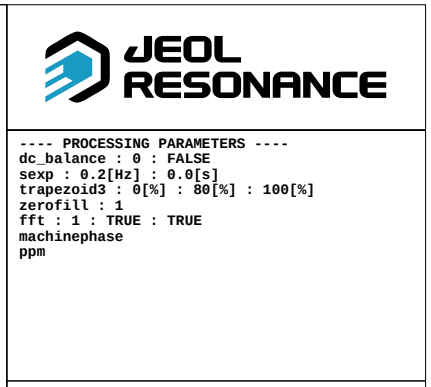


JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

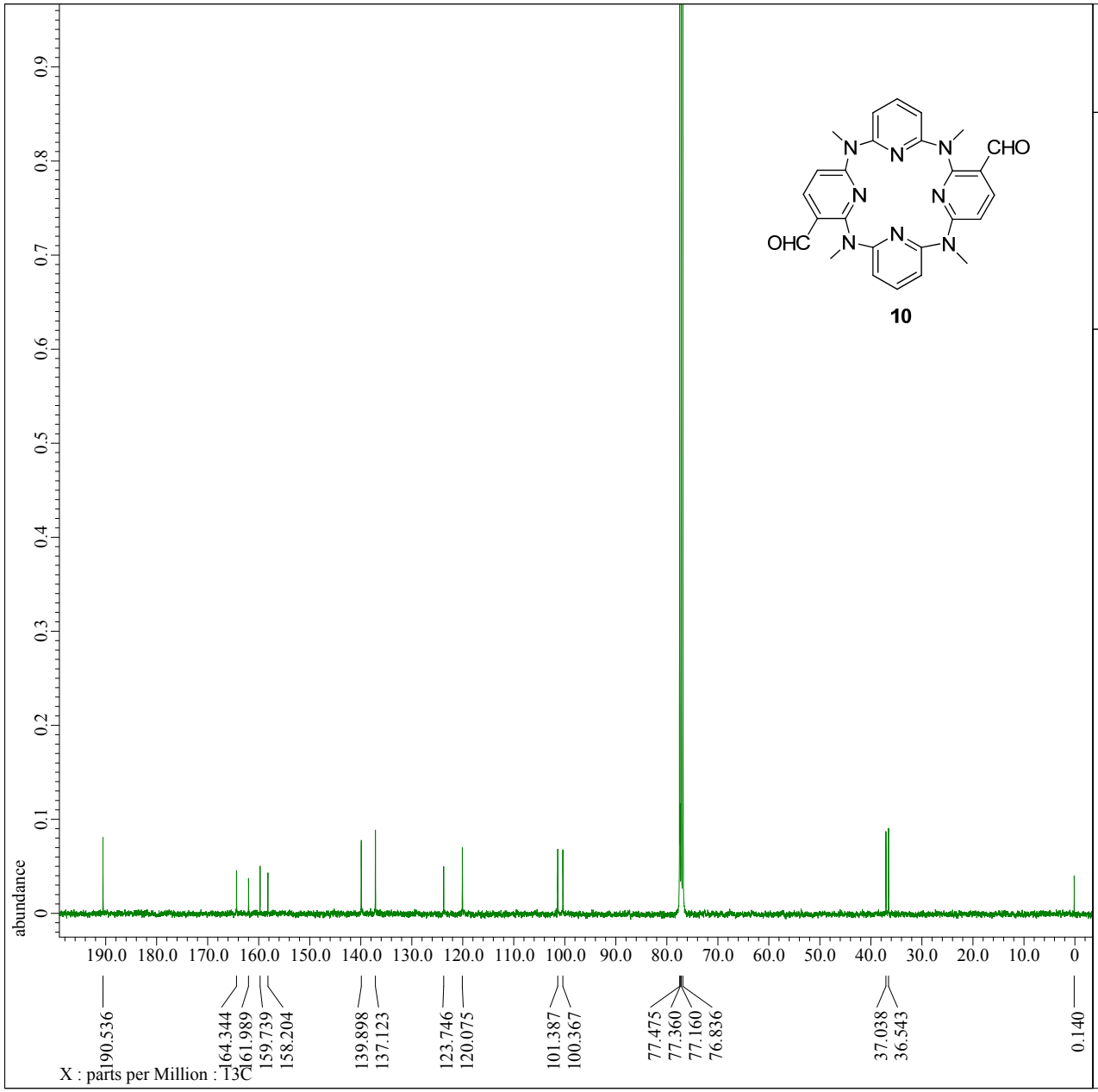
```
Filename          = C:\Users\RWS\Desktop\核磁\rw
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#636017
Solvent           = CHLOROFORM-D
Creation_Time     = 27-APR-2014 18:38:39
Revision_Time     = 14-JUN-2015 23:46:09
Current_Time      = 14-JUN-2015 23:46:36

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 2032
Total_Scans       = 2032

Relaxation_Delay  = 2[s]
Recvr_Gain        = 52
Temp_Get          = 18.1[dC]
X_90_Width        = 8.75[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 2.91666667[us]
Irr_Atn_Dec       = 23.98[dB]
Irr_Atn_Noe       = 23.98[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```





```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = C:\Users\RWS\Desktop\核磁数据\
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#722656
Solvent           = CHLOROFORM-D
Creation_Time     = 26-MAR-2014 19:21:39
Revision_Time     = 15-JUN-2015 19:19:52
Current_Time      = 15-JUN-2015 19:20:10

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
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
Incomplete_Copy   = TRUE
Scans             = 5
Total_Scans       = 5

Relaxation_Delay = 5[s]
Recvr_Gain        = 46
Temp_Get          = 24.3[dC]
X_90_Width        = 10.75[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.375[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
```

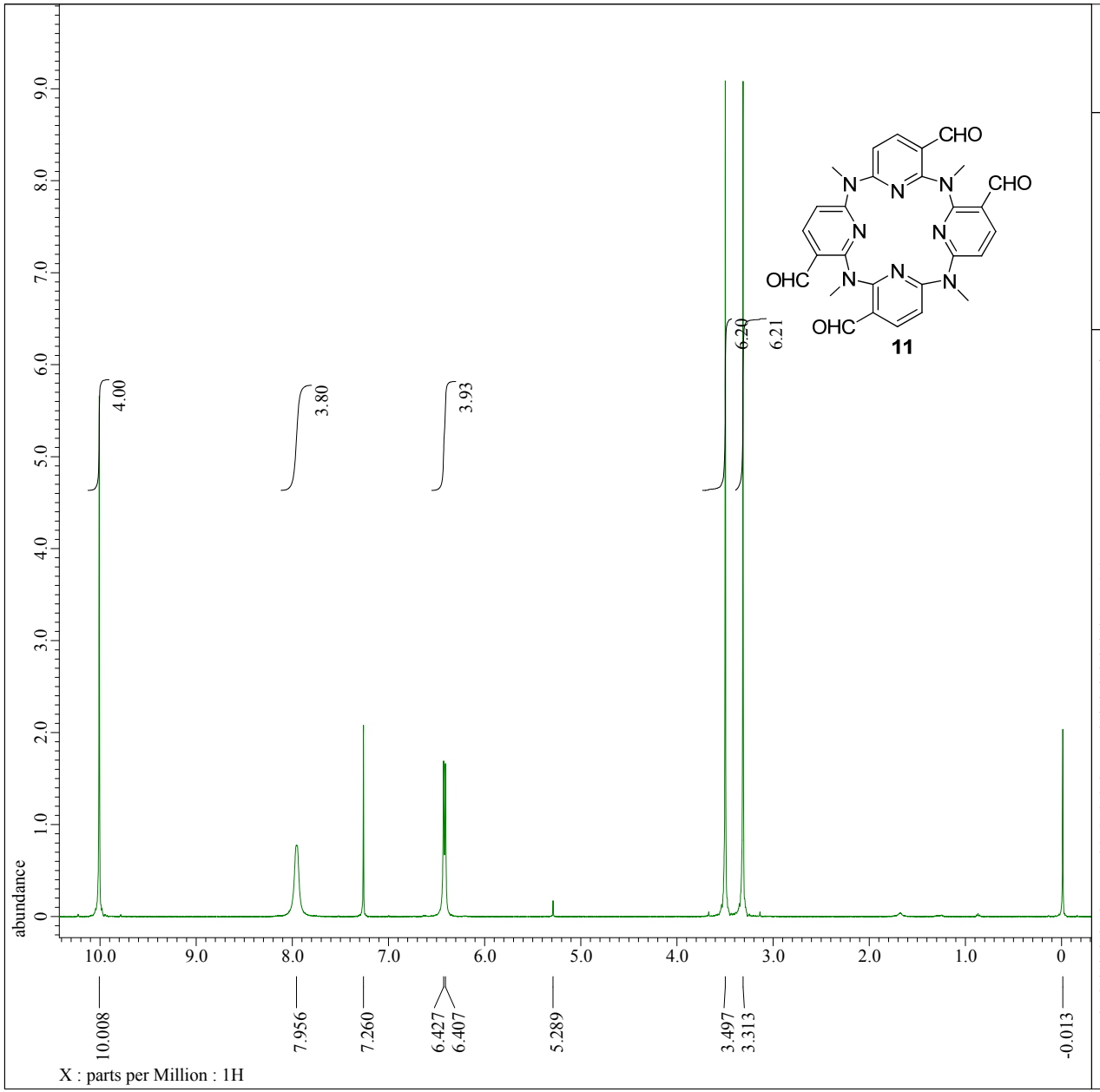


10

JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

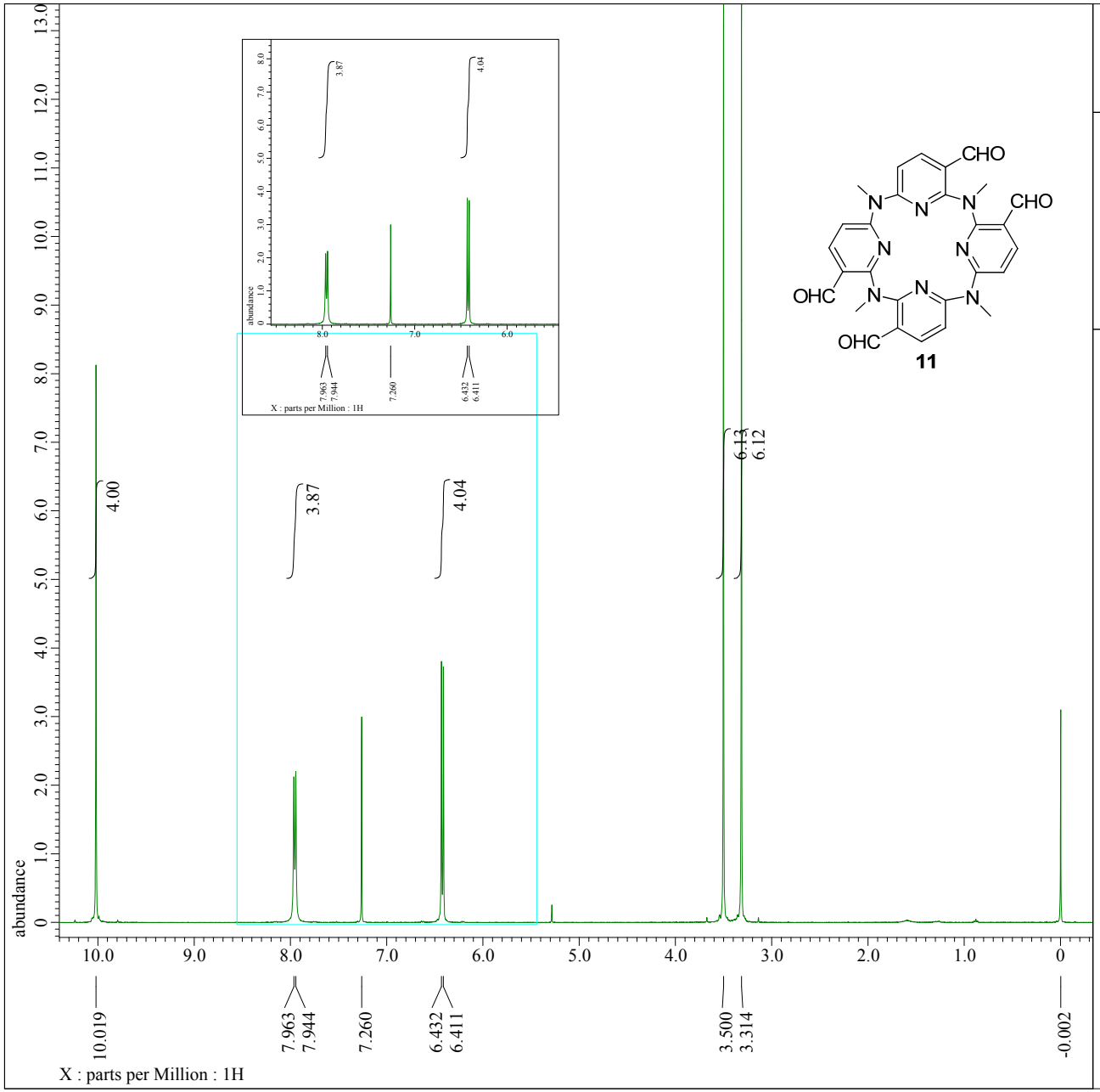
```
Filename          = C:\Users\RWS\Desktop\核磁\rw
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#246914
Solvent           = CHLOROFORM-D
Creation_Time     = 27-MAR-2014 08:02:32
Revision_Time     = 15-JUN-2015 00:05:51
Current_Time      = 15-JUN-2015 00:06:25

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 2213
Total_Scans       = 2213

Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
Temp_Get          = 20.9[dC]
X_90_Width        = 8.75[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 2.91666667[us]
Irr_Atn_Dec       = 23.98[dB]
Irr_Atn_Noe       = 23.98[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



JEOL RESONANCE

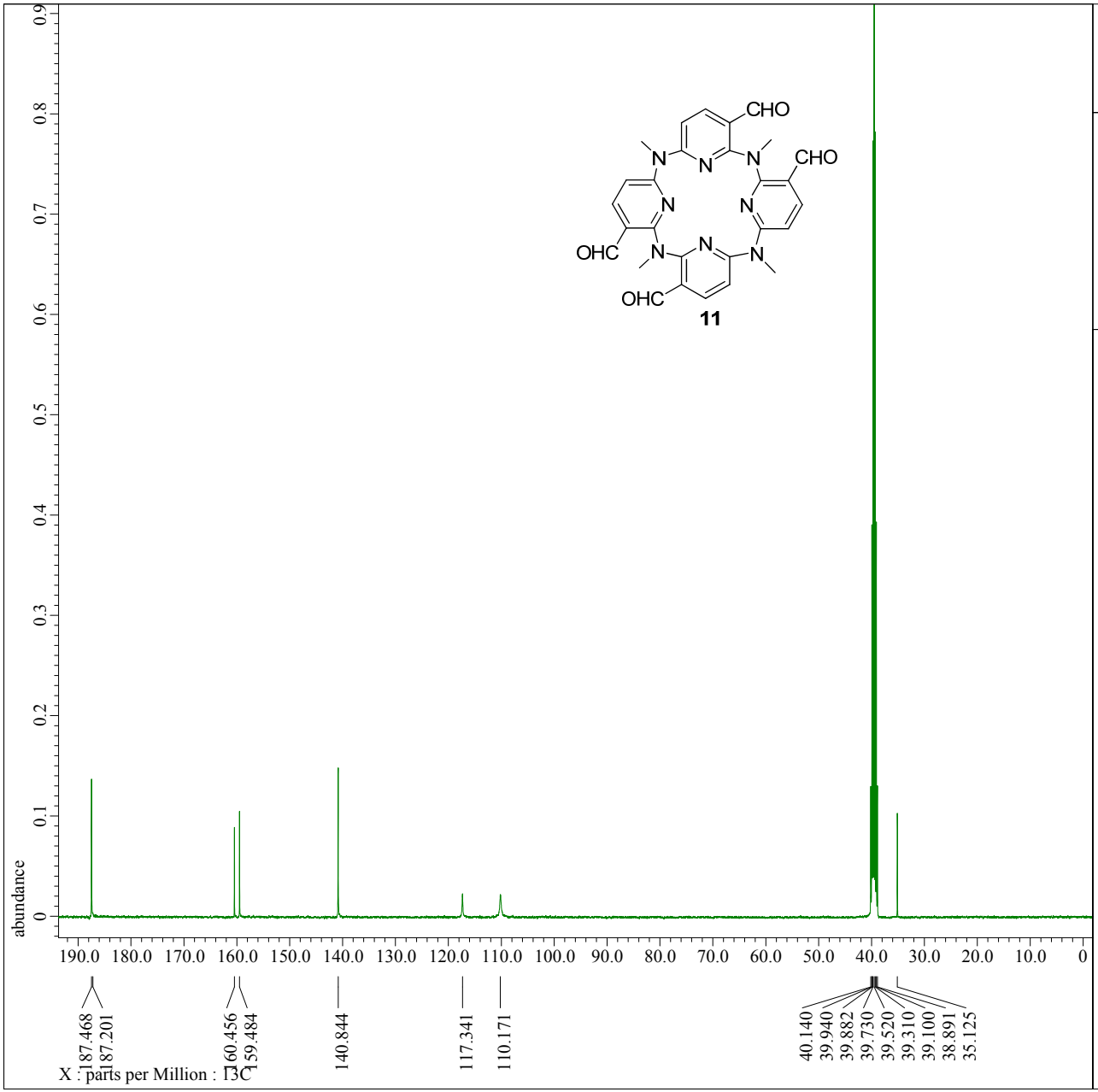
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = C:\Users\RWS\Desktop\核磁数据\
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#273574
Solvent           = CHLOROFORM-D
Creation_Time     = 20-MAR-2014 06:53:50
Revision_Time     = 15-JUN-2015 19:22:30
Current_Time      = 15-JUN-2015 19:22:54

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
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
Scans             = 11
Total_Scans       = 11

Relaxation_Delay  = 5[s]
Recvr_Gain        = 58
Temp_Get          = 24.9[dC]
X_90_Width        = 10.75[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.375[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

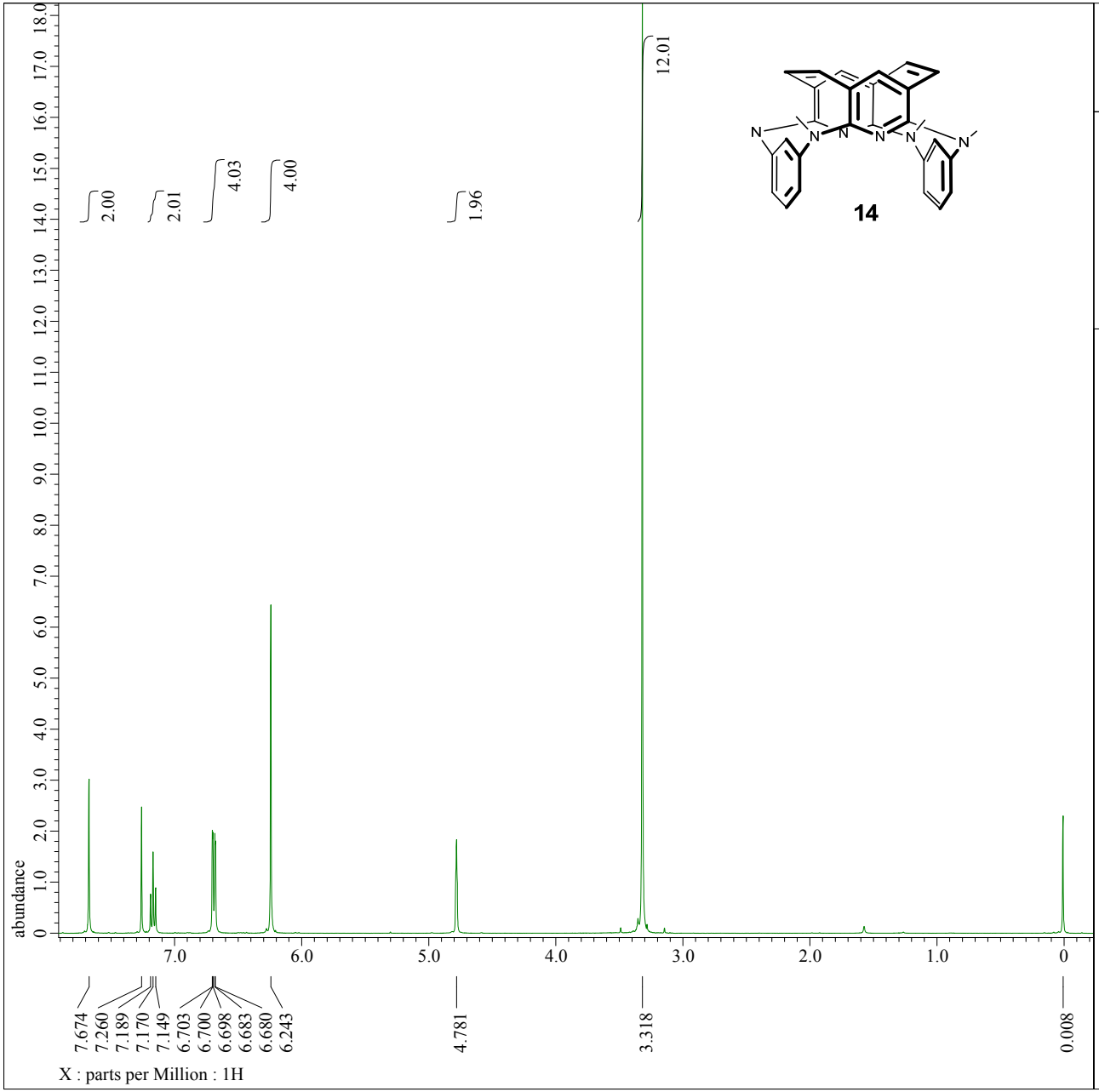
```
Filename          = C:\Users\RWS\Desktop\核磁\r w
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#277417
Solvent           = CHLOROFORM-D
Creation_Time     = 20-MAR-2014 07:00:22
Revision_Time     = 14-JUN-2015 23:57:10
Current_Time      = 14-JUN-2015 23:57:19

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
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
Scans             = 12
Total_Scans       = 12

Relaxation_Delay  = 5[s]
Recvr_Gain        = 54
Temp_Get          = 50[dC]
X_90_Width        = 10.75[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.375[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

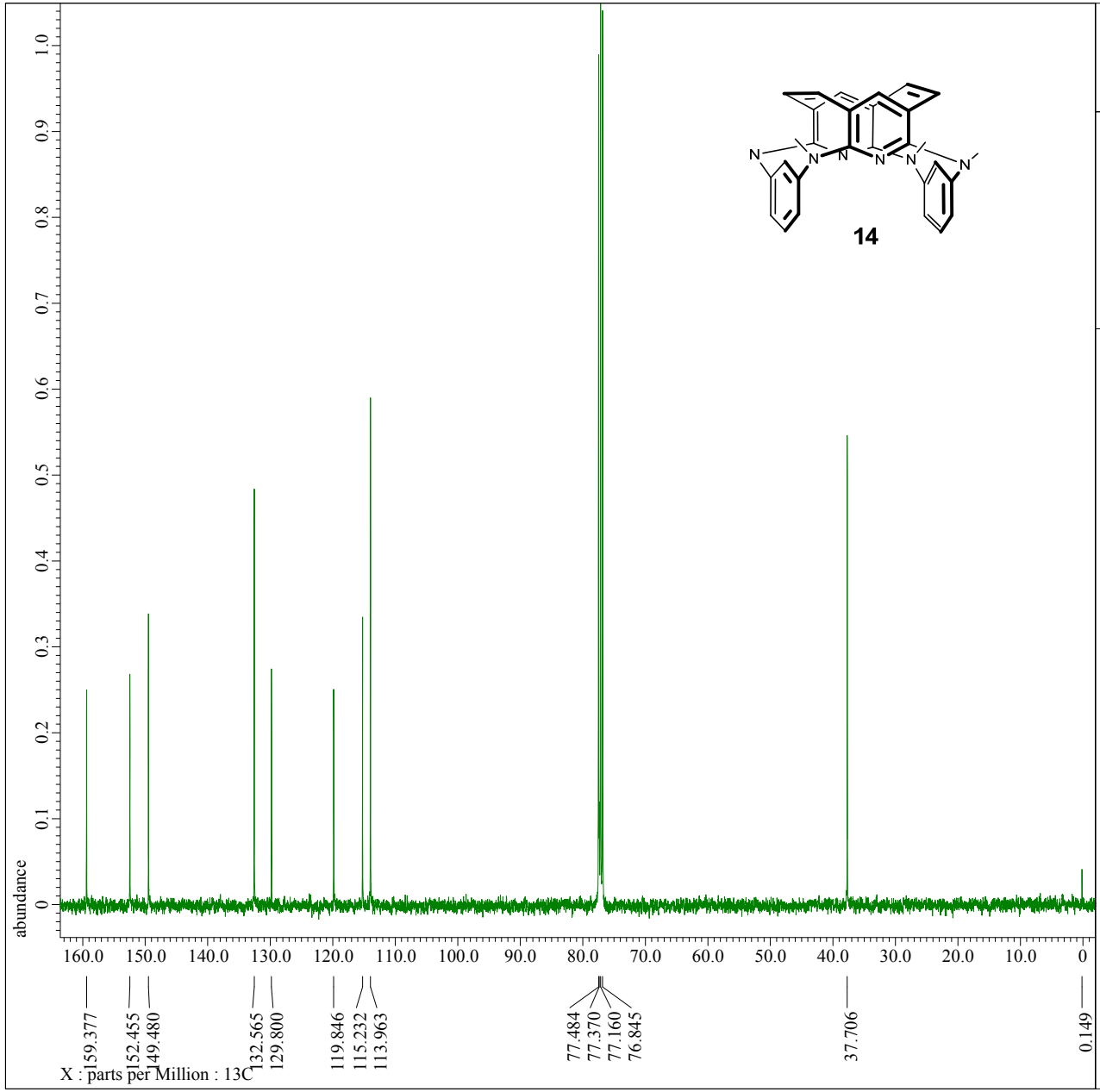
```
Filename          = C:\Users\RWS\Desktop\核磁\rw
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#818418
Solvent           = DMSO-D6
Creation_Time     = 21-MAR-2014 06:18:32
Revision_Time     = 15-JUN-2015 00:03:10
Current_Time      = 15-JUN-2015 00:03:59

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 9783
Total_Scans       = 9783

Relaxation_Delay  = 2[s]
Recvr_Gain        = 54
Temp_Get          = 120[dC]
X_90_Width        = 8.75[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 2.91666667[us]
Irr_Atn_Dec       = 23.98[dB]
Irr_Atn_Noe       = 23.98[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



JEOL RESONANCE

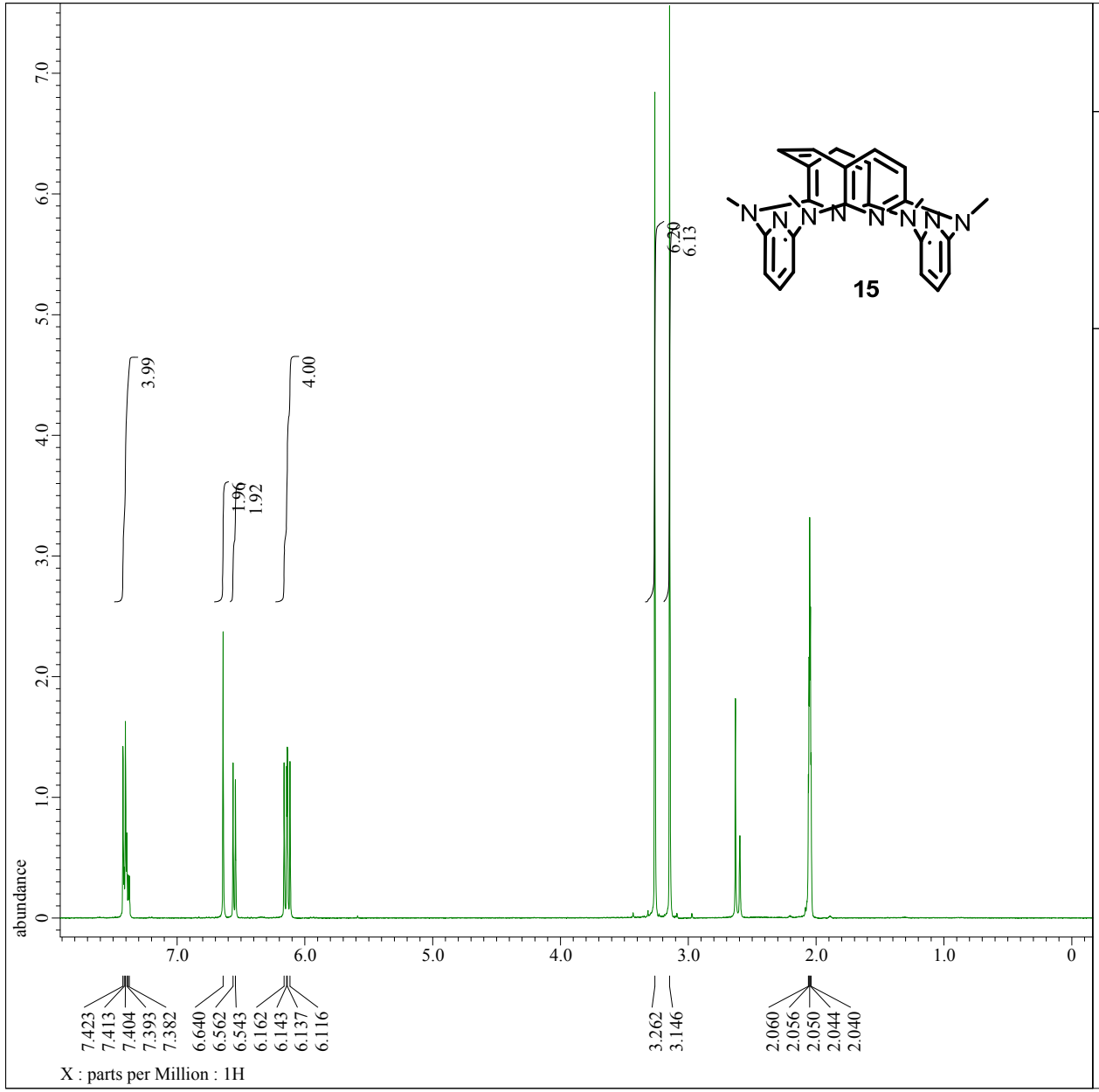
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = C:\Users\RWS\Desktop\核磁\rw
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#270039
Solvent           = CHLOROFORM-D
Creation_Time     = 17-FEB-2014 06:48:38
Revision_Time     = 17-FEB-2014 07:35:51
Current_Time      = 14-JUN-2015 20:04:57

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
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
Scans             = 16
Total_Scans       = 16

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 22.5[dC]
X_90_Width        = 10.75[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.375[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

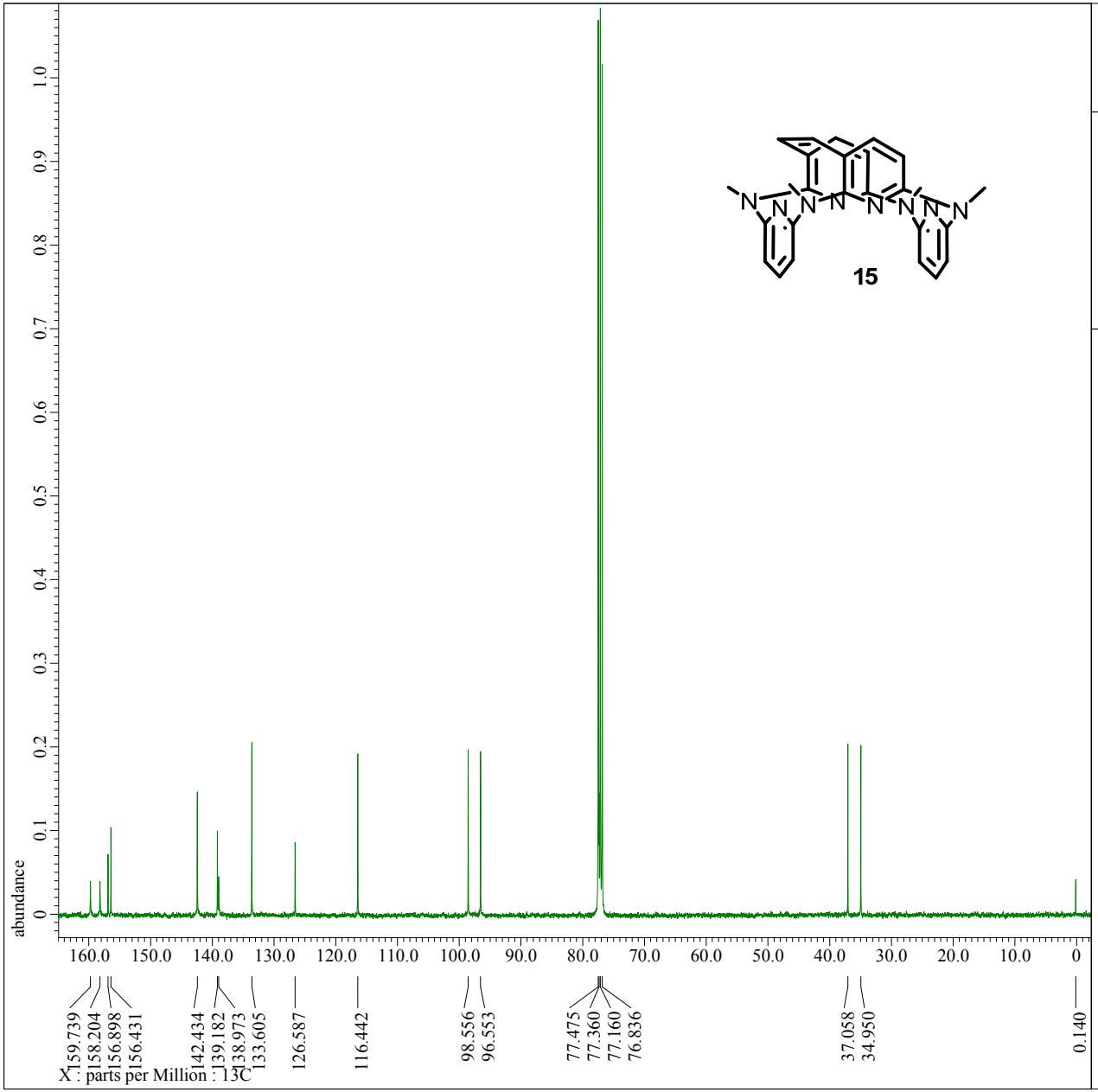
```
Filename          = C:\Users\RWS\Desktop\核磁\rw
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#272122
Solvent           = CHLOROFORM-D
Creation_Time     = 17-FEB-2014 07:07:57
Revision_Time     = 14-JUN-2015 20:05:53
Current_Time      = 14-JUN-2015 20:06:12

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 320
Total_Scans       = 320

Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
Temp_Get          = 22.6[dC]
X_90_Width        = 8.75[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 2.91666667[us]
Irr_Atn_Dec       = 23.98[dB]
Irr_Atn_Noe       = 23.98[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = C:\Users\RWS\Desktop\RWS20
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#339398
Solvent           = ACETONE-D6
Creation_Time     = 30-JUN-2015 09:36:43
Revision_Time     =  2-JUL-2015 00:05:53
Current_Time      =  2-JUL-2015 00:12:54

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
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
Scans             = 16
Total_Scans       = 16

Relaxation_Delay = 5[s]
Recvr_Gain        = 54
Temp_Get          = 50[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



15

JEOL RESONANCE

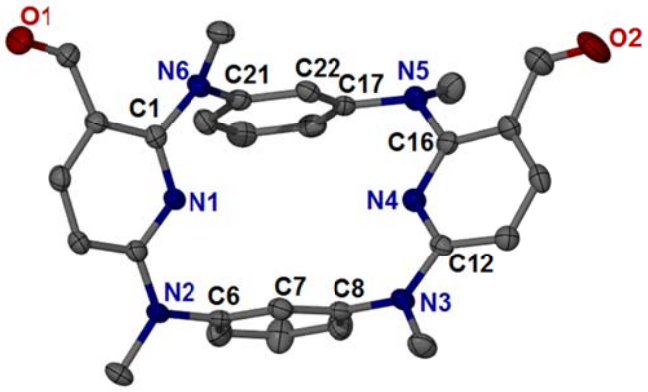
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = C:\Users\RWS\Desktop\RWS20
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#244558
Solvent           = CHLOROFORM-D
Creation_Time     =  1-JUL-2015 09:28:36
Revision_Time     =  2-JUL-2015 00:14:53
Current_Time      =  2-JUL-2015 00:15:12

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

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]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 2961
Total_Scans       = 2961

Relaxation_Delay = 2[s]
Recvr_Gain        = 58
Temp_Get          = 21.3[dC]
X_90_Width        = 9.25[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 7.8[dB]
X_Pulse           = 3.08333333[us]
Irr_Atn_Dec       = 23.786[dB]
Irr_Atn_Noe       = 23.786[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```

3. Crystal data and structure refinement for each X-ray structure

Structure of 3

Empirical formula	C28 H26 N6 O2	
Formula weight	478.56	
Temperature	180.00(10) K	
Crystal system, space group	Triclinic, P-1	
Unit cell dimensions	a = 8.9573(9) Å	a= 108.560(9)°.
	b = 10.2459(9) Å	b= 91.146(8)°.
	c = 13.7280 (14) Å	g = 96.44(8)°.
Volume	1184.8(2) $Å^3$	
Z	2	
Density (calculated)	1.3414 Mg/m^3	
Completeness to theta = 27.51°	99.25 %	
Goodness-of-fit on F^2	1.121	
Final R indices [I>2sigma(I)]	R1 = 0.0513,	
R indices (all data)	wR2 = 0.1155	

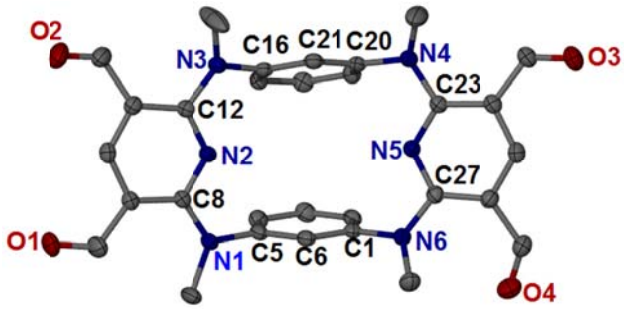


X-ray molecular structure of **3** with top view. The molecular is depicted in stick-ellipsoid style at 50% probability level for all atoms. Selected bond lengths [Å]: C(1)-N(6), 1.407; C(21)-N(6), 1.429; C(17)-N(5), 1.436; C(16)-N(5), 1.415; C(12)-N(3), 1.367; C(8)-N(3), 1.437; C(6)-N(2), 1.439; C(5)-N(2), 1.362. Selected angles (deg): C(21)-N(6)-C(1), 117.3; N(6)-C(21)-C(22), 122.9; N(5)-C(17)-C(22), 116.8; C(17)-N(5)-C(16), 114.3; N(5)-C(16)-N(4), 116.1; N(4)-C(12)-N(3), 116.4; C(12)-N(3)-C(8), 120.4; N(3)-C(8)-C(7), 120.3; N(2)-C(6)-C(7), 119.7; C(6)-N(2)-C(5), 119.8; N(2)-C(5)-N(1), 115.6; N(6)-C(1)-N(1), 114.9.

Structure of 5

Empirical formula	C30 H26 N6 O4
Formula weight	534.57
Temperature	173.1500 K

Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 10.070(3) Å	a= 70.851(7)°.
	b = 10.403(3) Å	b= 79.601(9)°.
	c = 12.774(3) Å	g = 88.998(11)°.
Volume	1242.1(5) $Å^3$	
Z	2	
Density (calculated)	1.429 Mg/m^3	
Absorption coefficient	0.098 mm^{-1}	
F(000)	560	
Crystal size	0.39 x 0.23 x 0.16 mm^3	
Theta range for data collection	2.866 to 27.521°.	
Index ranges	-13<=h<=13, -13<=k<=13, -16<=l<=16	
Reflections collected	15940	
Independent reflections	5699 [R(int) = 0.0322]	
Completeness to theta = 26.000°	99.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.0000 and 0.8782	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5699 / 0 / 365	
Goodness-of-fit on F^2	1.130	
Final R indices [I>2sigma(I)]	R1 = 0.0613, wR2 = 0.1468	
R indices (all data)	R1 = 0.0675, wR2 = 0.1509	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.471 and -0.251 $e.Å^{-3}$	

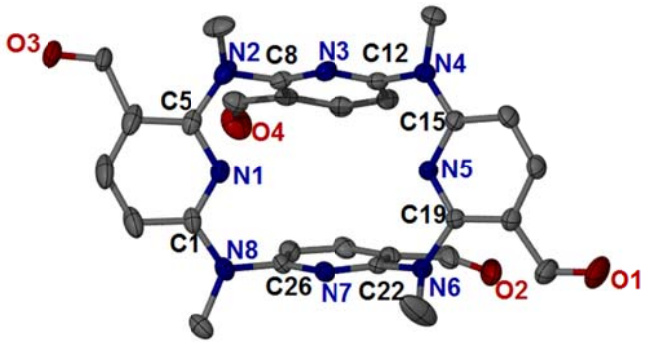


X-ray molecular structure of **5** with top view. The molecular is depicted in stick-ellipsoid style at 50% probability level for all atoms. Selected bond lengths [Å]: N(3)-C(12), 1.380; N(3)-C(16), 1.435; N(4)-C(20),

1.444; N(4)-C(23), 1.380; N(5)-C(27), 1.337; N(6)-C(1), 1.436; N(1)-C(5), 1.446; N(1)-C(8), 1.359. Selected angles (deg): C(12)-N(3)-C(16), 118.8; C(21)-C(16)-N(3), 120.3; C(21)-C(20)-N(4), 120.5; C(23)-N(4)-C(20), 116.5; N(5)-C(23)-N(4), 115.2; N(5)-C(27)-N(6), 115.2; C(27)-N(6)-C(1), 117.9; C(6)-C(1)-N(6), 121.6; C(6)-C(5)-N(1), 119.2; C(8)-N(1)-C(5), 118.4; N(2)-C(8)-N(1), 113.4; N(2)-C(12)-N(3), 115.0.

Structure of 11

Empirical formula	C29 H25 Cl3 N8 O4	
Formula weight	655.92	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	Triclinic, P-1	
Unit cell dimensions	a = 9.641(3) Å	a= 82.779(12)°.
	b = 11.263(4) Å	b= 83.587(14)°.
	c = 13.731 (5) Å	g = 81.730(11)°.
Volume	1457.0(9) $Å^3$	
Z	2	
Density (calculated)	1.495 Mg/m^3	
Absorption coefficient	0.366 mm^{-1}	
F(000)	676	
Crystal size	0.52 x 0.43 x 0.13 mm^3	
Theta range for data collection	2.82 to 27.51°.	
Index ranges	-12<=h<=12, -14<=k<=14, -17<=l<=17	
Reflections collected	19278	
Independent reflections	6656 [R(int) = 0.0434]	
Completeness to theta = 27.51°	99.2 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.0000 and 0.6232	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6656 / 6 / 448	
Goodness-of-fit on F^2	1.121	
Final R indices [I>2sigma(I)]	R1 = 0.0674, wR2 = 0.1602	
R indices (all data)	R1 = 0.0736, wR2 = 0.1653	
Largest diff. peak and hole	0.545 and -0.496 e.$Å^{-3}$	

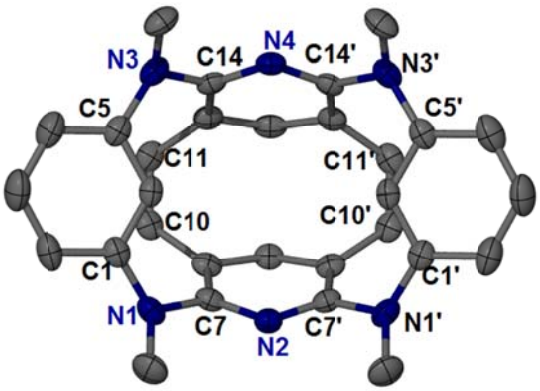


X-ray molecular structure of **11** with top view. A solvent molecule was omitted for clarity. The molecular is depicted in stick-ellipsoid style at 50% probability level for all atoms. Selected bond lengths [Å]: N(2)-C(8), 1.414; N(4)-C(12), 1.427; N(4)-C(15), 1.372; N(6)-C(19), 1.398; N(6)-C(22), 1.406; N(8)-C(26), 1.429; N(8)-C(1), 1.360; N(2)-C(5), 1.398. Selected angles (deg): C(5)-N(2)-C(8), 116.2; N(3)-C(8)-N(2), 117.6; N(3)-C(12)-N(4), 116.0; C(15)-N(4)-C(12), 117.8; N(5)-C(15)-N(4), 115.1; N(5)-C(19)-N(6), 114.7; C(19)-N(6)-C(22), 116.1; N(7)-C(22)-N(6), 117.3; N(7)-C(26)-N(8), 115.5; C(1)-N(8)-C(26), 118.4; N(1)-C(1)-N(8), 115.4; N(1)-C(5)-N(2), 115.2.

Structure of 14

Empirical formula	C61 H53 Cl3 N12	
Formula weight	1060.50	
Temperature	173.1500 K	
Wavelength	0.71073 Å	
Crystal system	Tetragonal	
Space group	P 42 n m	
Unit cell dimensions	a = 19.890(3) Å	a= 90°.
	b = 19.890(3) Å	b= 90°.
	c = 6.3216(13) Å	g = 90°.
Volume	2500.9(9) $Å^3$	
Z	2	
Density (calculated)	1.408 Mg/m^3	
Absorption coefficient	0.240 mm^{-1}	
F(000)	1108	
Crystal size	0.47 x 0.15 x 0.05 mm^3	
Theta range for data collection	2.290 to 27.493°.	
Index ranges	-25<=h<=25, -24<=k<=25, -8<=l<=8	
Reflections collected	19317	
Independent reflections	2974 [R(int) = 0.0533]	

Completeness to theta = 25.242°	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.7602
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2974 / 7 / 188
Goodness-of-fit on F^2	1.130
Final R indices [I>2sigma(I)]	R1 = 0.0573, wR2 = 0.1457
R indices (all data)	R1 = 0.0587, wR2 = 0.1468
Absolute structure parameter	1.0(3)
Extinction coefficient	n/a
Largest diff. peak and hole	0.415 and -0.570 e.$Å^{-3}$

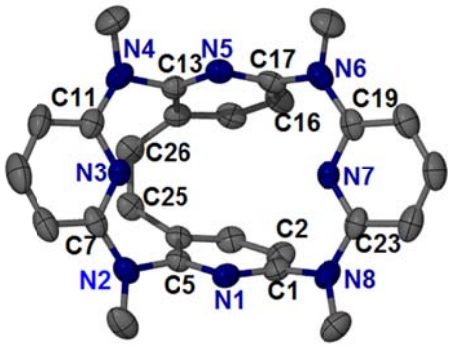


X-ray molecular structure of **14** with top views. A solvent molecule was omitted for clarity. The molecular is depicted in stick-ellipsoid style at 50% probability level for all atoms. Selected bond lengths [Å]: N(1)-C(1), 1.416; N(1)-C(7), 1.421; N(3)-C(5), 1.430; N(3)-C(14), 1.418; C(10)-C(11), 1.350. Selected angles (deg): C(1)-N(1)-C(7), 115.2; N(2)-C(7)-N(1), 116.9; N(4)-C(14)-N(3), 115.6; C(14)-N(3)-C(5), 114.4.

Structure of 15

Empirical formula	C26 H24 N8	
Formula weight	448.53	
Temperature	173.1500 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 17.774(5) Å	a= 90°.
	b = 11.314(3) Å	b= 106.667(4)°.
	c = 11.548(3) Å	g = 90°.
Volume	2224.7(10) $Å^3$	

Z	4
Density (calculated)	1.339 Mg/m^3
Absorption coefficient	0.085 mm^{-1}
F(000)	944
Crystal size	0.37 x 0.33 x 0.05 mm^3
Theta range for data collection	2.575 to 27.470°.
Index ranges	-22<=h<=23, -14<=k<=14, -14<=l<=14
Reflections collected	14195
Independent reflections	5010 [R(int) = 0.0443]
Completeness to theta = 26.000°	98.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.8738
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5010 / 0 / 311
Goodness-of-fit on F^2	1.385
Final R indices [I>2sigma(I)]	R1 = 0.0854, wR2 = 0.2158
R indices (all data)	R1 = 0.1103, wR2 = 0.2293
Extinction coefficient	n/a
Largest diff. peak and hole	0.192 and -0.204 $e.Å^{-3}$



X-ray molecular structure of **15** with top view. The molecular is depicted in stick-ellipsoid style at 50% probability level for all atoms. Selected bond lengths [Å]: N(4)-C(13), 1.440; N(5)-C(13), 1.331; N(5)-C(17), 1.337; N(6)-C(17), 1.428; N(6)-C(19), 1.389; N(7)-C(19), 1.342; N(7)-C(23), 1.331; N(8)-C(23), 1.398; N(8)-C(1), 1.418; N(1)-C(1), 1.340; N(1)-C(5), 1.336; N(2)-C(5), 1.425; N(2)-C(7), 1.389; N(3)-C(7), 1.327; N(3)-C(11), 1.328; N(4)-C(11), 1.385; C(25)-C(26), 1.333. Selected angles (deg): C(11)-N(4)-C(13), 115.9; N(5)-C(13)-N(4), 115.3; N(5)-C(17)-N(6), 117.6; C(19)-N(6)-C(17), 118.3; N(7)-C(19)-N(6), 116.5; N(7)-C(23)-N(8), 115.8; C(23)-N(8)-C(1), 116.6; N(1)-C(1)-N(8), 117.7; N(1)-C(5)-N(2), 115.8;

C(7)-N(2)-C(5), 116.9; N(3)-C(7)-N(2), 114.1; N(3)-C(11)-N(4), 113.8. Selected atomic distances [Å]: C(4)-C(14), 2.867; C(2)-C(16), 3.639.

4. Atomic coordinates for each X-ray structure

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

Structure of 3

	x	y	z	U(eq)
O1	14306(2)	7976(2)	-355(2)	38(6)
O2	8096(3)	4340(3)	5863(2)	62(9)
N1	10126(3)	9272(3)	1682(2)	24(6)
N2	9290(3)	11396(3)	2071(2)	28(6)
N3	7651(3)	9948(2)	4977(2)	25(6)
N4	7242(3)	7593(3)	4161(2)	26(6)
N5	6925(3)	5238(3)	3208(2)	28(7)
N6	10743(3)	7050(3)	1332(2)	26(6)
C1	11071(3)	8379(3)	1239(3)	23(7)
C2	12304(3)	8714(3)	708(3)	24(7)
C3	12569(3)	10112(3)	740(3)	26(7)
C4	11623(3)	11038(3)	1194(3)	27(7)
C5	10349(3)	10565(3)	1640(2)	24(7)
C6	8101(3)	10892(3)	2599(3)	26(7)
C7	8425(3)	10711(3)	3528(3)	25(7)
C8	7310(3)	10161(3)	4016(3)	25(7)
C9	5850(3)	9822(3)	3581(3)	34(9)
C10	5541(4)	10022(4)	2659(3)	40(9)
C11	6651(4)	10553(4)	2159(3)	34(8)
C12	7510(3)	8639(3)	5046(3)	22(7)
C13	7638(3)	8419(3)	6009(3)	27(8)
C14	7652(3)	7080(3)	5996(3)	29(8)
C15	7516(3)	5979(3)	5076(3)	29(8)
C16	7230(3)	6296(3)	4171(3)	25(7)
C17	7275(3)	5681(3)	2337(3)	25(7)
C18	6208(3)	5710(3)	1622(3)	30(8)
C19	6638(4)	6207(3)	825(3)	33(8)
C20	8106(4)	6653(3)	743(3)	29(8)
C21	9211(3)	6606(3)	1462(3)	26(7)

C22	8785(3)	6112(3)	2248(3)	25(7)
C23	11924(3)	6383(3)	1660(3)	31(8)
C24	9330(4)	12803(3)	2025(3)	34(9)
C25	8219(4)	11153(3)	5860(3)	33(8)
C26	5509(4)	4330(3)	3114(3)	41(10)
C27	13180(3)	7706(3)	62(3)	28(8)
C28	7824(4)	4618(4)	5076(3)	44(10)

Structure of 5

	x	y	z	U(eq)
O1	-309(2)	-1734(2)	8250(1)	44(1)
O2	2078(2)	-1970(2)	11263(2)	56(1)
O3	8450(2)	9907(2)	5522(1)	45(1)
O4	4368(2)	9345(2)	2627(1)	44(1)
N1	918(2)	2536(2)	6809(1)	27(1)
N2	2071(2)	2213(2)	8234(1)	24(1)
N3	3280(2)	2222(2)	9585(1)	25(1)
N4	5651(2)	6678(2)	7605(1)	29(1)
N5	4662(2)	6904(2)	6075(1)	24(1)
N6	3651(2)	6749(2)	4657(1)	26(1)
C1	2538(2)	6015(2)	5505(1)	23(1)
C2	1683(2)	6729(2)	6075(2)	26(1)
C3	591(2)	6058(2)	6890(2)	29(1)
C4	336(2)	4671(2)	7143(2)	30(1)
C5	1196(2)	3974(2)	6573(2)	25(1)
C6	2293(2)	4628(2)	5750(2)	24(1)
C7	37(2)	2221(2)	6128(2)	41(1)
C8	1303(2)	1635(2)	7732(2)	24(1)
C9	961(2)	200(2)	8168(2)	27(1)
C10	1366(2)	-503(2)	9180(2)	27(1)
C11	2105(2)	81(2)	9732(2)	27(1)
C12	2476(2)	1498(2)	9185(2)	24(1)
C13	194(2)	-601(2)	7705(2)	40(1)
C14	2277(2)	-741(2)	10877(2)	38(1)
C15	4355(3)	1633(2)	10161(3)	66(1)
C16	3276(2)	3682(2)	9184(1)	23(1)
C17	2092(2)	4321(2)	9424(2)	26(1)
C18	2105(2)	5730(2)	9094(2)	29(1)

C19	3283(2)	6503(2)	8514(2)	29(1)
C20	4453(2)	5863(2)	8264(1)	26(1)
C21	4459(2)	4448(2)	8613(1)	24(1)
C22	6933(2)	6262(3)	7977(2)	50(1)
C23	5635(2)	7342(2)	6479(2)	24(1)
C24	6616(2)	8385(2)	5770(2)	27(1)
C25	6491(2)	8914(2)	4645(2)	28(1)
C26	5495(2)	8481(2)	4203(2)	26(1)
C27	4594(2)	7393(2)	4979(2)	23(1)
C28	7592(2)	9074(2)	6161(2)	32(1)
C29	5324(2)	9332(2)	3075(2)	33(1)
C30	3922(2)	6489(2)	3582(2)	35(1)

Structure of 11

	x	y	z	U(eq)
O1	5785(2)	2087(2)	2809(2)	70(1)
O2	6242 (2)	5040(2)	4171(2)	49(1)
O3	-901(4)	13831(3)	1771(3)	51(1)
O3A	-1052(5)	12980(4)	2560(4)	49(2)
O4	2399(2)	11016(2)	5011(2)	53(1)
N1	2484(2)	10283(2)	2147(1)	29(1)
N2	196(2)	10307(2)	2850(1)	32(1)
N3	478(2)	8226(2)	3376(1)	28(1)
N4	791(2)	6132(2)	3790(1)	31(1)
N5	3050(2)	5896(1)	3065(1)	26(1)
N6	5292(2)	5862(2)	2286(1)	31(1)
N7	5127(2)	7961(2)	1922(1)	28(1)
N8	4777(2)	10055(2)	1498(1)	32(1)
C1	3500(2)	10750(2)	1538(2)	32(1)
C2	3242(3)	11865(2)	945(2)	46(1)
C3	1932(3)	12500(2)	1080(2)	54(1)
C4	874(3)	12074(2)	1752(2)	42(1)
C5	1201(2)	10903(2)	2240(2)	30(1)
C6	-556(10)	12803(8)	2155(7)	35(2)
C6A	-205(10)	12991(9)	1844(8)	38(2)
C7	-1155(2)	10229(3)	2473(2)	44(1)
C8	725(2)	9326(2)	3513(2)	26(1)
C9	1472(2)	9542(2)	4277(2)	26(1)

C10	2034(2)	8552(2)	4888(2)	29(1)
C11	1826(2)	7405(2)	4732(2)	30(1)
C12	1036(2)	7300(2)	3973(2)	27(1)
C13	1623(2)	10782(2)	4453(2)	32(1)
C14	-655(2)	5871(2)	3838(2)	35(1)
C15	1920(2)	5369(2)	3430(2)	26(1)
C16	1870(2)	4121(2)	3426(2)	32(1)
C17	3088(2)	3436(2)	3121(2)	32(1)
C18	4315(2)	3952(2)	2797(2)	31(1)
C19	4211(2)	5217(2)	2732(2)	27(1)
C20	5666(3)	3184(2)	2692(2)	46(1)
C21	5986(4)	5623(3)	1329(3)	77(1)
C22	5298(2)	7004(2)	2597(2)	26(1)
C23	5464(2)	7105(2)	3586(2)	28(1)
C24	5333(2)	8255(2)	3880(2)	31(1)
C25	5093(2)	9253(2)	3197(2)	31(1)
C26	5010(2)	9054(2)	2237(2)	27(1)
C27	5864(2)	6077(2)	4325(2)	38(1)
C28	6003(3)	10430(2)	879(2)	39(1)
C31	1544(6)	7275(5)	1057(5)	41(1)
C31A	908(7)	7286(6)	1144(6)	58(2)
Cl1	3000(2)	6636(2)	338(1)	71(1)
Cl1A	1935(6)	6087(4)	618(3)	152(3)
Cl2	978(1)	8677(1)	434(1)	69(1)
Cl3	261(3)	6302(2)	1239(1)	83(1)
Cl3A	-850(5)	7058(4)	1329(2)	112(2)

Structure of 14

	x	y	z	U(eq)
Cl1	4498(1)	4498(1)	1154(9)	159(2)
Cl2	5000	5000	5015(7)	120(2)
N1	487(1)	2146(2)	3342(5)	33(1)
N2	1251(1)	1251(1)	3683(7)	29(1)
N3	2190(2)	3836(1)	3497(5)	34(1)
N4	3072(1)	3072(1)	3916(7)	30(1)
C1	660(2)	2812(2)	2770(6)	32(1)
C2	183(2)	3293(2)	2193(7)	45(1)
C3	392(2)	3954(2)	1934(8)	50(1)

C4	1045(2)	4158(2)	2346(7)	45(1)
C5	1518(2)	3672(2)	2873(5)	32(1)
C6	1329(2)	3005(2)	2883(5)	29(1)
C7	978(2)	1798(2)	4556(5)	29(1)
C8	1184(2)	2053(2)	6536(5)	30(1)
C9	1674(2)	1674(2)	7560(7)	28(1)
C10	1047(2)	2744(2)	7337(5)	32(1)
C11	1525(2)	3225(2)	7399(6)	34(1)
C12	2231(2)	3102(2)	6650(5)	30(1)
C13	2598(2)	2598(2)	7649(8)	31(1)
C14	2510(2)	3337(2)	4743(5)	28(1)
C15	90(2)	1755(2)	1843(7)	48(1)
C16	2600(2)	4156(2)	1851(7)	41(1)
C17	5200(4)	4800(4)	2400(30)	57(3)

Structure of 15

	x	y	z	U(eq)
N1	3941(1)	6529(2)	7712(2)	37(1)
N2	3452(1)	4662(2)	7984(2)	39(1)
N3	2152(1)	5129(2)	7298(2)	34(1)
N4	902(1)	5639(2)	6271(2)	43(1)
N5	1254(1)	7600(2)	6046(2)	38(1)
N6	1732(2)	9557(2)	6096(2)	45(1)
N7	3060(1)	9113(2)	6822(2)	36(1)
N8	4350(1)	8493(2)	7552(2)	46(1)
C1	4078(2)	7392(2)	7003(2)	38(1)
C2	3932(2)	7266(3)	5768(2)	43(1)
C3	3528(2)	6274(2)	5232(2)	39(1)
C4	3274(2)	5433(2)	5919(2)	34(1)
C5	3552(2)	5570(2)	7186(2)	34(1)
C6	4109(2)	4451(3)	9067(3)	56(1)
C7	2693(2)	4461(2)	8042(2)	39(1)
C8	2502(2)	3637(3)	8812(3)	54(1)
C9	1716(2)	3480(3)	8689(3)	62(1)
C10	1141(2)	4093(3)	7857(3)	54(1)
C11	1393(2)	4941(2)	7155(2)	40(1)
C12	137(2)	5971(3)	6389(3)	64(1)
C13	1284(1)	6485(2)	5698(2)	35(1)

C14	1670(2)	6114(2)	4852(2)	36(1)
C15	1913(2)	7012(2)	4222(2)	40(1)
C16	1893(2)	8178(3)	4572(2)	43(1)
C17	1617(2)	8411(2)	5556(2)	38(1)
C18	1066(2)	10173(3)	6297(3)	60(1)
C19	2497(2)	9940(2)	6623(2)	41(1)
C20	2669(2)	11118(3)	6963(3)	52(1)
C21	3438(2)	11410(3)	7494(3)	56(1)
C22	4025(2)	10576(3)	7698(3)	53(1)
C23	3804(2)	9415(2)	7346(2)	41(1)
C24	5047(2)	8507(3)	8592(3)	53(1)
C25	2675(2)	4560(2)	5293(2)	40(1)
C26	1931(2)	4874(2)	4777(2)	42(1)