

## Supplementary Materials:

# Labdane-type Diterpenes, Galangalditerpenes A–C, with Melanogenesis Inhibitory Activity from the Fruit of *Alpinia galanga*

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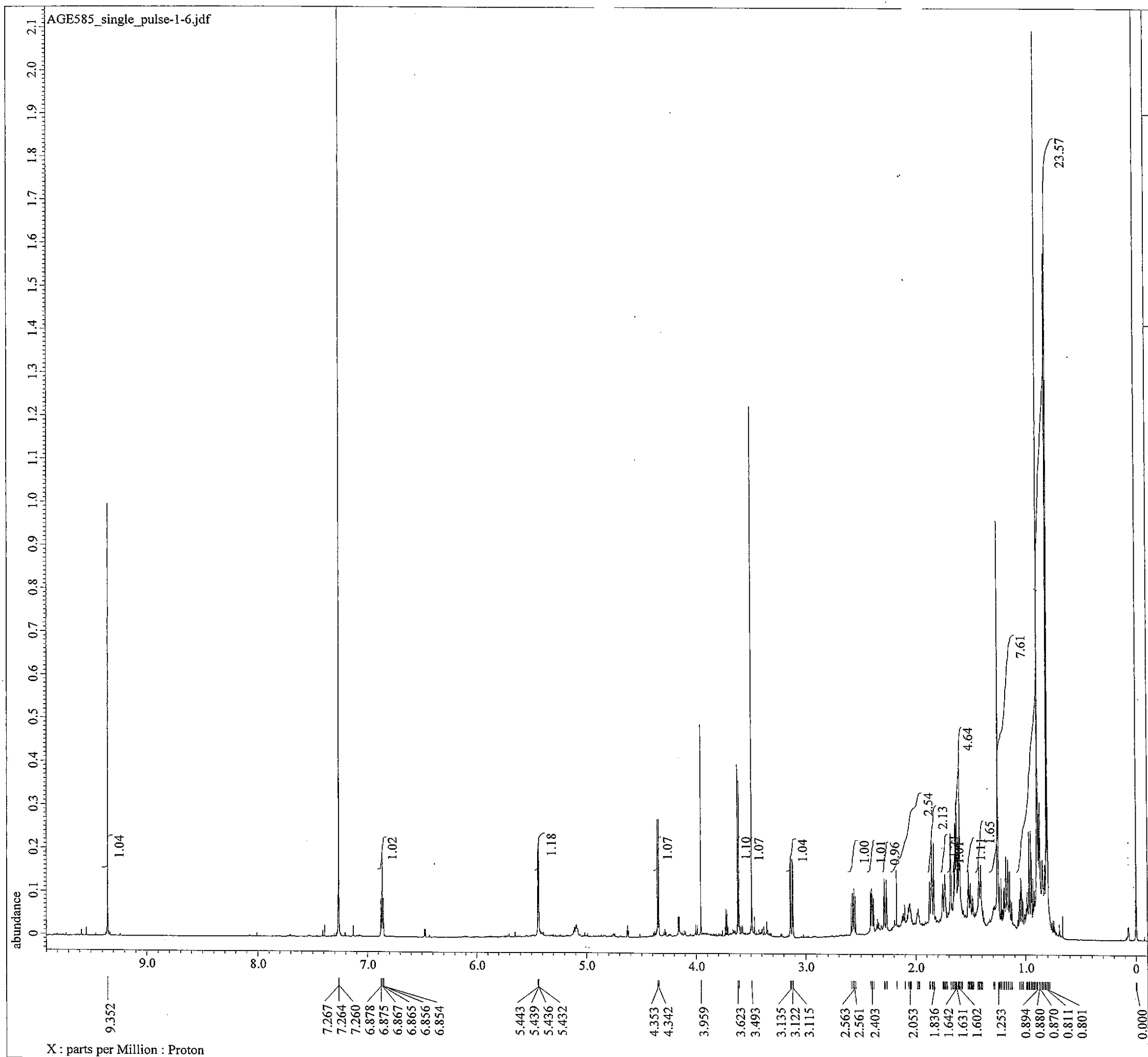
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**Figure S1.** NMR spectra of galangalditerpenes A–C (1–3)

Galangalditerpene A (1)	• • • • •	1 – 6
<sup>1</sup> H NMR spectrum	• • • • •	1
<sup>13</sup> C NMR and DEPT spectra	• • • • •	2
<sup>1</sup> H– <sup>1</sup> H COSY spectrum	• • • • •	3
HSQC spectrum	• • • • •	4
HMBC spectrum	• • • • •	5
NOESY spectrum	• • • • •	6
Galangalditerpene B (2)	• • • • •	7 – 12
<sup>1</sup> H NMR spectrum	• • • • •	7
<sup>13</sup> C NMR and DEPT spectra	• • • • •	8
<sup>1</sup> H– <sup>1</sup> H COSY spectrum	• • • • •	9
HSQC spectrum	• • • • •	10
HMBC spectrum	• • • • •	11
NOESY spectrum	• • • • •	12
Galangalditerpene C (3)	• • • • •	13 – 18
<sup>1</sup> H NMR spectrum	• • • • •	13
<sup>13</sup> C NMR and DEPT spectra	• • • • •	14
<sup>1</sup> H– <sup>1</sup> H COSY spectrum	• • • • •	15
HSQC spectrum	• • • • •	16
HMBC spectrum	• • • • •	17
NOESY spectrum	• • • • •	18



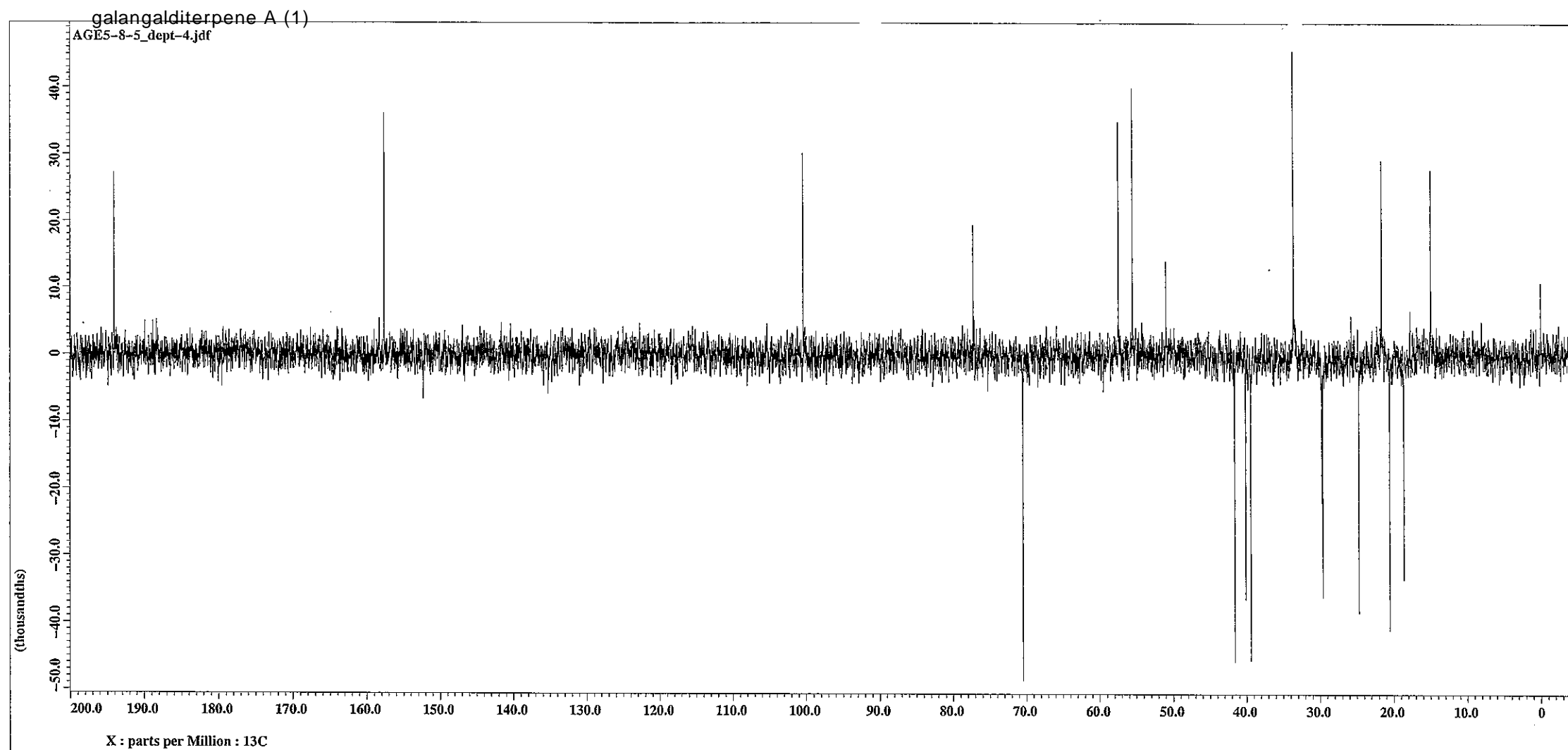
----- PROCESSING PARAMETERS -----  
dc\_balance( 0, FALSE )  
sexf( 0.2[Hz], 0.0[s] )  
trapezoid( 0[%], 0[%], 80[%], 100[%] )  
zerofill( 2 )  
fft( 1, TRUE, TRUE )  
machinephase  
ppm  
phase( -3, 0, 35.02327[%] )  
以下に由来: AGE585\_single\_pulse-1-1.jdf

Filename = C:\Users\datum\Documents\J  
Author = delta  
Experiment = single\_pulse.jxp  
Sample Id = AGE585  
Solvent = CHLOROFORM-D  
Creation Time = 15-FEB-2012 12:47:44  
Revision Time = 15-FEB-2012 13:00:54  
Current Time = 15-FEB-2012 13:01:32

Comment = single\_pulse  
Data Format = 1D COMPLEX  
Dim Size = 52429  
Dim Title = Proton  
Dim Units = [ppm]  
Dimensions = X  
Site = JNM-ECA800  
Spectrometer = DELTA2\_NMR

Field Strength = 18.79305132[T] (800[MHz])  
X\_Acq\_Duration = 2.18103808[s]  
X\_Domain = 1H  
X\_Freq = 800.14000039[MHz]  
X\_Offset = 5[ppm]  
X Points = 32768  
X\_Prescans = 1  
X Resolution = 0.45849727[Hz]  
X\_Sweep = 15.02403846[kHz]  
X\_Sweep\_Clippped = 12.01923077[kHz]  
Irr\_Domain = Proton  
Irr\_Freq = 800.14000039[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = Proton  
Tri\_Freq = 800.14000039[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total\_Scans = 8

Relaxation\_Delay = 5[s]  
Recvr\_Gain = 48  
Temp\_Get = 19.7[dc]  
X\_90\_Width = 14[us]  
X\_Acq\_Time = 2.18103808[s]  
X\_Angle = 45[deg]  
X\_Atn = 1.3[db]  
X\_Pulse = 7[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 7.18103808[s]



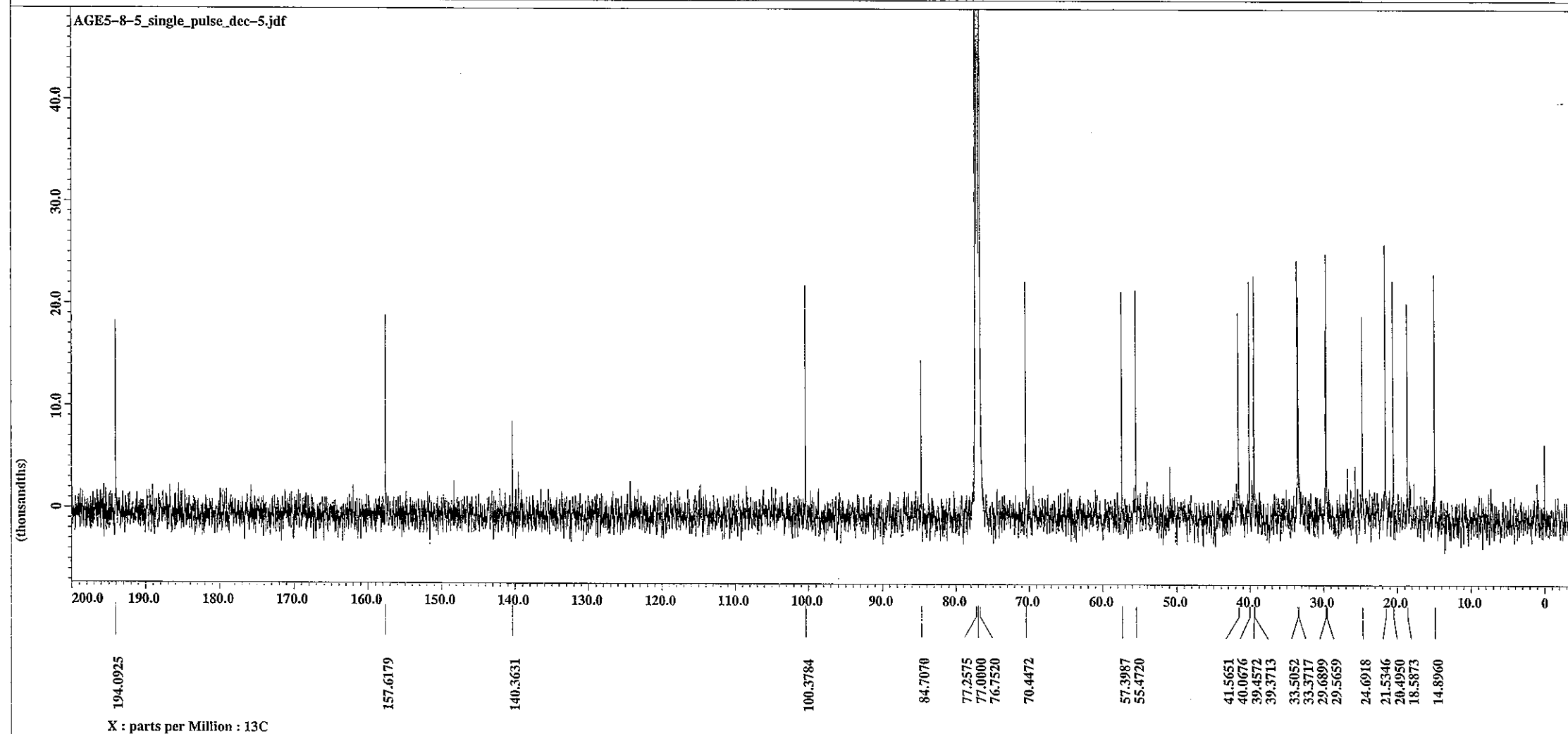
----- PROCESSING PARAMETERS -----  
dc\_balance : 0 : FALSE  
secp : 2.0[Hz] : 0.0[s]  
trapezoid3 : 0[%] : 80[%] : 100[%]  
zerofill : 1  
fft : 1 : TRUE : TRUE  
machinephase  
ppm

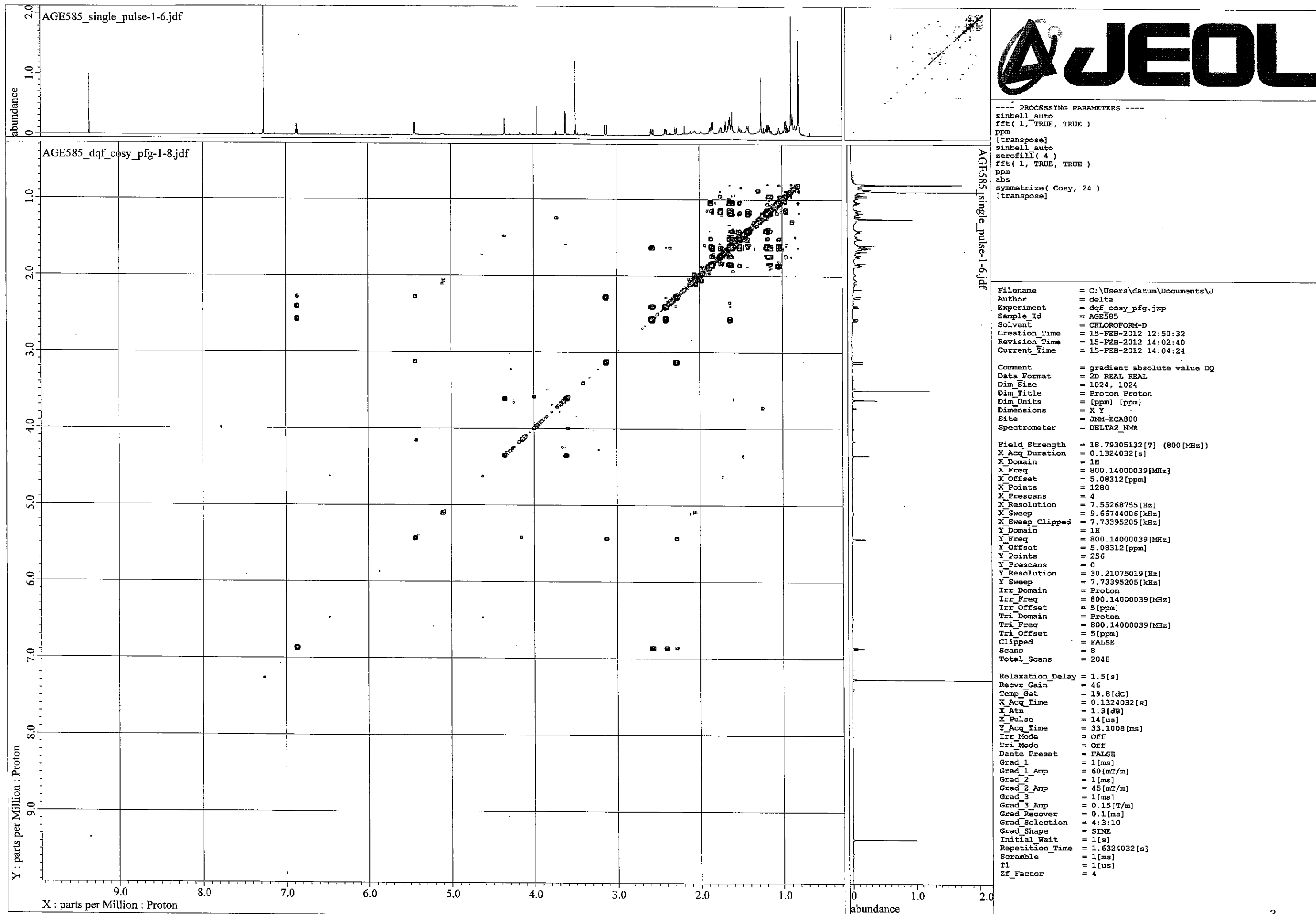
Filename = AGE5-8-5\_single\_pulse  
Author = delta  
Experiment = single\_pulse\_dec  
Sample\_id = 001  
Solvent = CHLOROFORM-D  
Creation\_time = 14-FEB-2012 18:07:12  
Revision\_time = 15-FEB-2012 08:45:47  
Current\_time = 15-FEB-2012 08:47:01

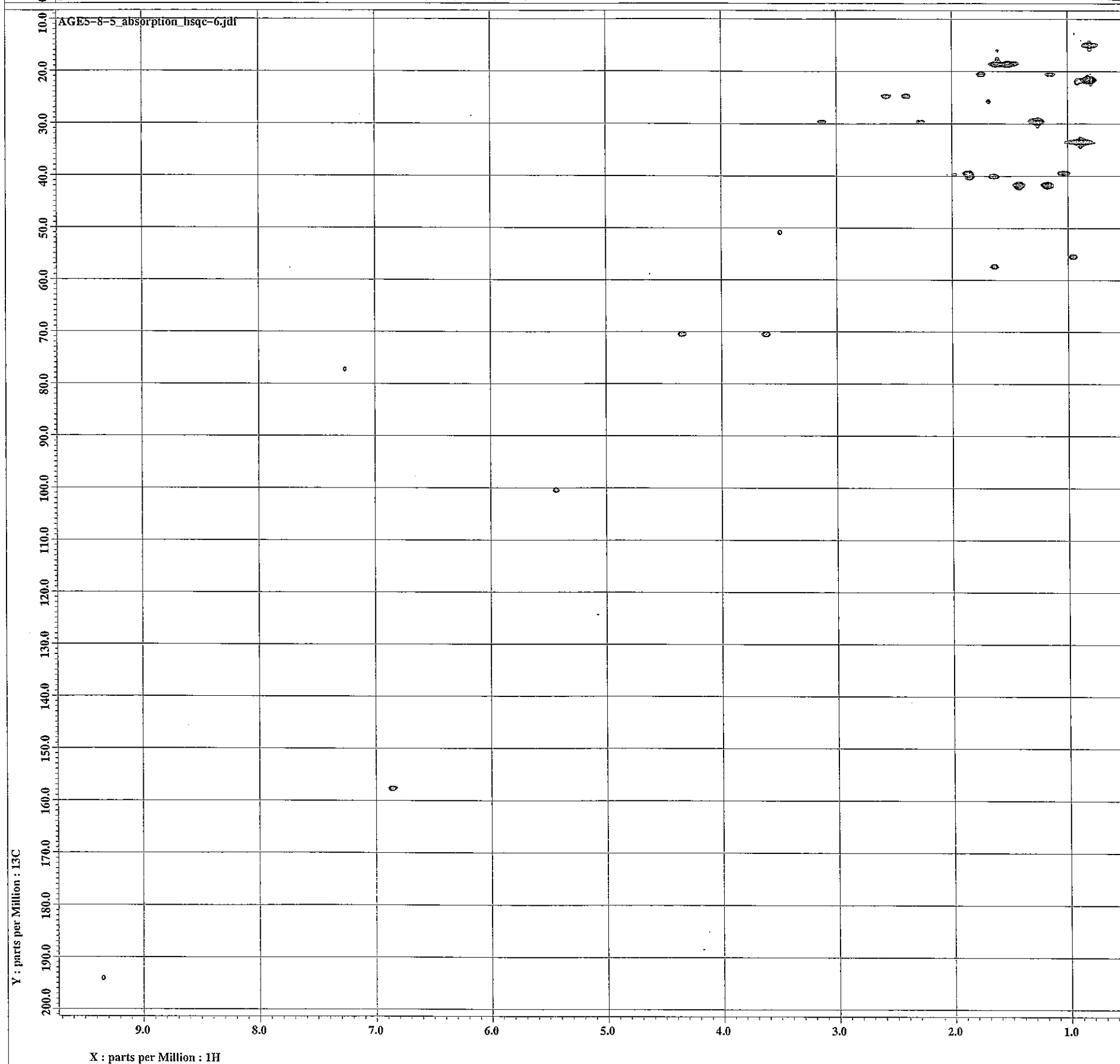
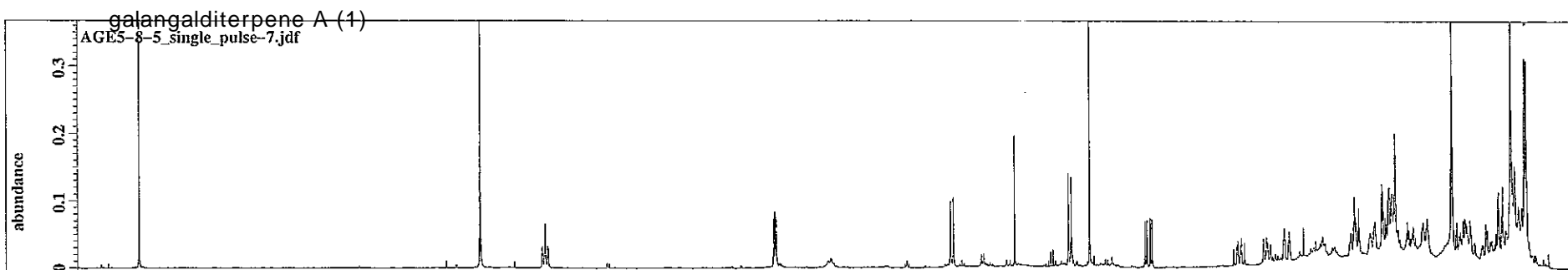
Comment = single pulse decouple  
Data\_format = 1D COMPLEX  
Dim\_size = 26214  
Dim\_title = 13C  
Dim\_units = [ppm]  
Dimensions = X  
Site = ECA500  
Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH]  
X\_acq\_duration = 0.83361792[s]  
X\_domain = 13C  
X\_freq = 125.76529768[MHz]  
X\_offset = 100[ppm]  
X\_points = 32768  
X\_prescans = 4  
X\_resolution = 1.19959034[Hz]  
X\_sweep = 39.3081761[kHz]  
Irr\_domain = 1H  
Irr\_freq = 500.15991521[MHz]  
Irr\_offset = 5.0[ppm]  
Clipped = FALSE  
Mod\_return = 1  
Scans = 5000  
Total\_scans = 5000

X\_90\_width = 12[us]  
X\_acq\_time = 0.83361792[s]  
X\_angle = 30[deg]  
X\_atn = 8.4[dB]  
X\_pulse = 4[us]  
Irr\_atn\_dec = 20.84[dB]  
Irr\_atn\_noe = 20.84[dB]  
Irr\_noise = WALTZ  
Decoupling = TRUE  
Initial\_wait = 1[s]  
Noe = TRUE  
Noe\_time = 2[s]  
Recvr\_gain = 58  
Relaxation\_delay = 2[s]  
Repetition\_time = 2.83361792[s]  
Temp\_get = 23.3[degC]







----- PROCESSING PARAMETERS -----  
sexp : 10.0[Hz] : 0.0[s]  
trapezoid3 : 0[%] : 80[%] : 100[%]  
zerofill : 1  
fft : 1 : TRUE : TRUE  
ppm  
machinephase  
phase : -50 : 0 : 95.93023[ppm]  
phase : 80 : -10 : 5.07525[ppm]  
[transpose]  
sexp : 50.0[Hz] : 0.0[s]  
trapezoid3 : 0[%] : 80[%] : 100[%]  
zerofill : 2  
fft : 1 : TRUE : TRUE  
ppm  
machinephase  
phase : 0 : 0 : 104.96322[ppm]  
[transpose]

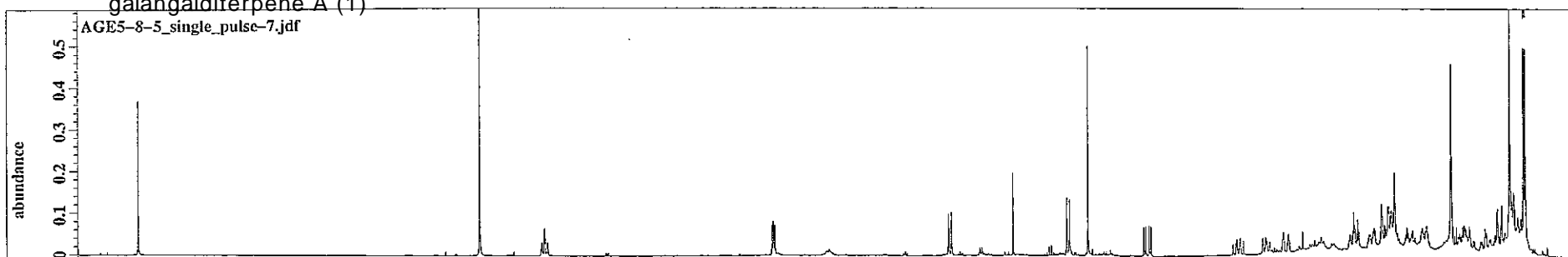
Filename = AGE5-8-5\_absorption\_h  
Author = delta  
Experiment = hsqc\_dec\_phase\_p  
Sample\_id = 001  
Solvent = CHLOROFORM-D  
Creation\_time = 14-FEB-2012 22:38:10  
Revision\_time = 15-FEB-2012 08:53:01  
Current\_time = 15-FEB-2012 08:53:49  
  
Comment = absorption\_hsqc  
Data\_format = 2D COMPLEX COMPLEX  
Dim\_size = 819, 512  
Dim\_title = 1H 13C  
Dim\_units = [ppm] [ppm]  
Dimensions = X Y  
Site = ECA500  
Spectrometer = JNM-ECA500  
  
Field\_strength = 11.7473579[T] (500[MH  
X\_acq\_duration = 0.176128[s]  
X\_domain = 1H  
X\_freq = 500.15991521[MHz]  
X\_offset = 5.0639[ppm]  
X\_points = 1024  
X\_prescans = 4  
X\_resolution = 5.67768895[Hz]  
X\_sweep = 5.81395349[kHz]  
Y\_domain = 13C  
Y\_freq = 125.76529768[MHz]  
Y\_offset = 104.77438[ppm]  
Y\_points = 256  
Y\_prescans = 0  
Y\_resolution = 94.99635214[Hz]  
Y\_sweep = 24.31906615[kHz]  
Tri\_domain = 1H  
Tri\_freq = 500.15991521[MHz]  
Tri\_offset = 5.0[ppm]  
Clipped = FALSE  
Mod\_return = 1  
Scans = 8  
Total\_scans = 2048  
  
X\_acq\_time = 0.176128[s]  
X\_atn = 2.4[dB]  
X\_pulse = 12[us]  
Y\_acq\_time = 10.52672[ms]  
Y\_atn = 8.4[dB]  
Y\_pl\_correction = 360  
Y\_pulse = 12[us]  
Irr\_atn\_dec = 28[dB]  
Irr\_noise = MPF10  
Tri\_mode = Off  
Composite\_pulse = FALSE  
Dante\_presat = FALSE  
Decoupling = TRUE  
Grad\_1\_amp = 0.105[T/m]  
Grad\_2\_amp = 30[mT/m]  
Initial\_wait = 1[s]  
J\_constant = 140[Hz]  
Purge = 0[ms]  
Recvr\_gain = 42  
Relaxation\_delay = 1.5[s]  
Repetition\_time = 1.676128[s]  
T1 = 0.88[us]  
T1\_cal1 = -9.4[us]  
T1\_cal2 = 0.88[us]  
T1\_cal3 = 11.16[us]  
T1\_cal4 = 21.44[us]  
T1\_cal5 = 31.72[us]  
T1\_cal6 = 42.0[us]  
Temp\_get = 22.4[dc]

(thousandths)

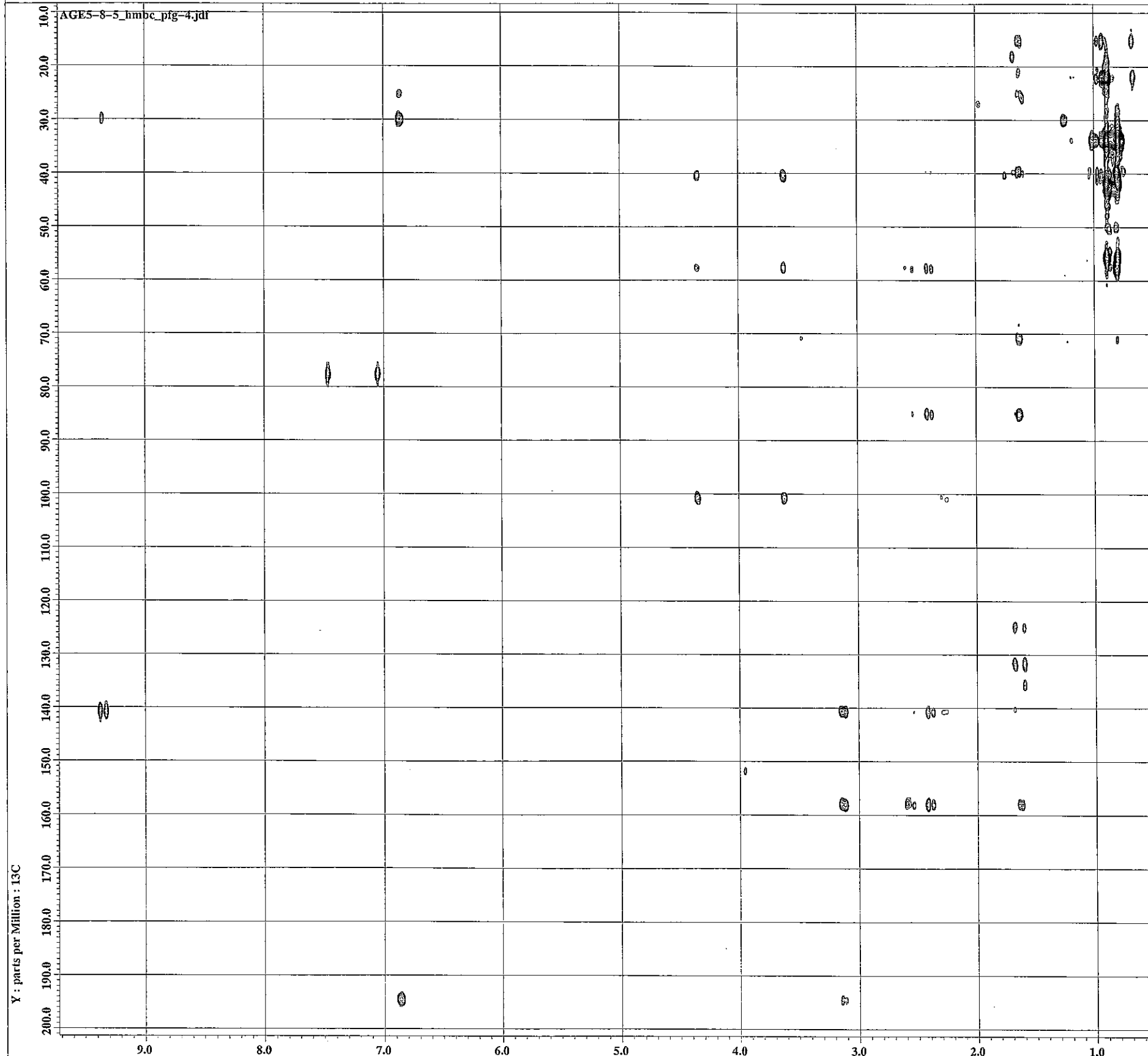
## galangalditerpene A (1)

AGE5-8-5\_single\_pulse-7.jdf

abundance



AGE5-8-5\_hmbc\_pfg-4.jdf



Y : parts per Million : 13C

X : parts per Million : 1H



----- PROCESSING PARAMETERS -----  
gauss : 5.0[Hz] : 0.0[s]  
sinbell\_auto  
zerofill : 1  
fft : 1 : TRUE : TRUE  
ppm  
[transpose]  
sinbell4 : -60 : 160  
trapezoid3 : 5[%] : 80[%] : 100[%]  
zerofill : 2  
fft : 1 : TRUE : TRUE  
ppm  
abs  
[transpose]

Filename = AGE5-8-5\_hmbc\_pfg-4.j  
Author = delta  
Experiment = hmbc\_pfg.ex2  
Sample\_id = 001  
Solvent = CHLOROFORM-D  
Creation\_time = 15-FEB-2012 03:00:58  
Revision\_time = 15-FEB-2012 08:59:18  
Current\_time = 15-FEB-2012 09:00:02

Comment = gradient enhanced HMB  
Data\_format = 2D REAL REAL  
Dim\_size = 1638, 512  
Dim\_title = 1H 13C  
Dim\_units = [ppm] [ppm]  
Dimensions = X Y  
Site = ECA500  
Spectrometer = JNM-ECA500

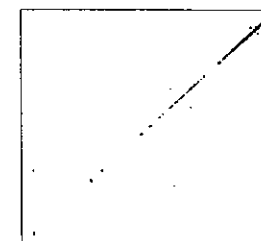
Field\_strength = 11.7473579[T] (500[MH  
X\_acq\_duration = 0.352256[s]  
X\_domain = 1H  
X\_freq = 500.15991521[MHz]  
X\_offset = 5.0639[ppm]  
X\_points = 2048  
X\_prescans = 4  
X\_resolution = 2.83884448[Hz]  
X\_sweep = 5.81395349[kHz]  
Y\_domain = 13C  
Y\_freq = 125.76529768[MHz]  
Y\_offset = 104.77438[ppm]  
Y\_points = 256  
Y\_prescans = 0  
Y\_resolution = 94.99635214[Hz]  
Y\_sweep = 24.31906615[kHz]  
Tri\_domain = 1H  
Tri\_freq = 500.15991521[MHz]  
Tri\_offset = 5.0[ppm]  
Clipped = FALSE  
Mod\_return = 1  
Scans = 32  
Total\_scans = 8192

X\_acq\_time = 0.352256[s]  
X\_atn = 2.4[dB]  
X\_pulse = 12[us]  
Y\_acq\_time = 10.52672[ms]  
Y\_atn = 8.4[dB]  
Y\_pulse = 12[us]  
Tri\_mode = Off  
Dante\_presat = FALSE  
Delta = 62.5[ms]  
Grad\_1 = 1[ms]  
Grad\_1\_amp = 0.18[T/m]  
Grad\_2 = 1[ms]  
Grad\_2\_amp = 0.18[T/m]  
Grad\_3 = 1[ms]  
Grad\_3\_amp = 90.54325956[mT/m]  
Grad\_recover = 0.1[ms]  
Grad\_selection = 13C = 1.988:1.98  
Grad\_shape = SINE  
Initial\_wait = 1[s]  
J\_constant = 140[Hz]  
Long\_range\_j = 8[Hz]  
Recvr\_gain = 42  
Refocus\_comp = 2.57142857[ms]  
Relaxation\_delay = 1.5[s]  
Repetition\_time = 1.852256[s]  
T1 = 1[us]  
Templ = 1.988  
Temp\_get = 22[dc]

(thousandths)

## galangalditerpene A (1)

AGE5-8-5\_single\_pulse-7.jdf



## ----- PROCESSING PARAMETERS -----

sexp : 5.0[Hz] : 0.0[s]  
trapezoid3 : 0[%] : 80[%] : 100[%]  
zerofill : 1  
fft : 1 : TRUE : TRUE

ppm  
machinephase  
phase : -1.25 : 0 : 85.78384[%]  
phase : 1 : 0 : 5.07856[ppm]  
[transpose]

sexp : 5.0[Hz] : 0.0[s]  
trapezoid3 : 0[%] : 80[%] : 100[%]  
zerofill : 2  
fft : 1 : TRUE : TRUE

ppm  
machinephase  
phase : 0 : 0 : 5.07562[ppm]  
[transpose]

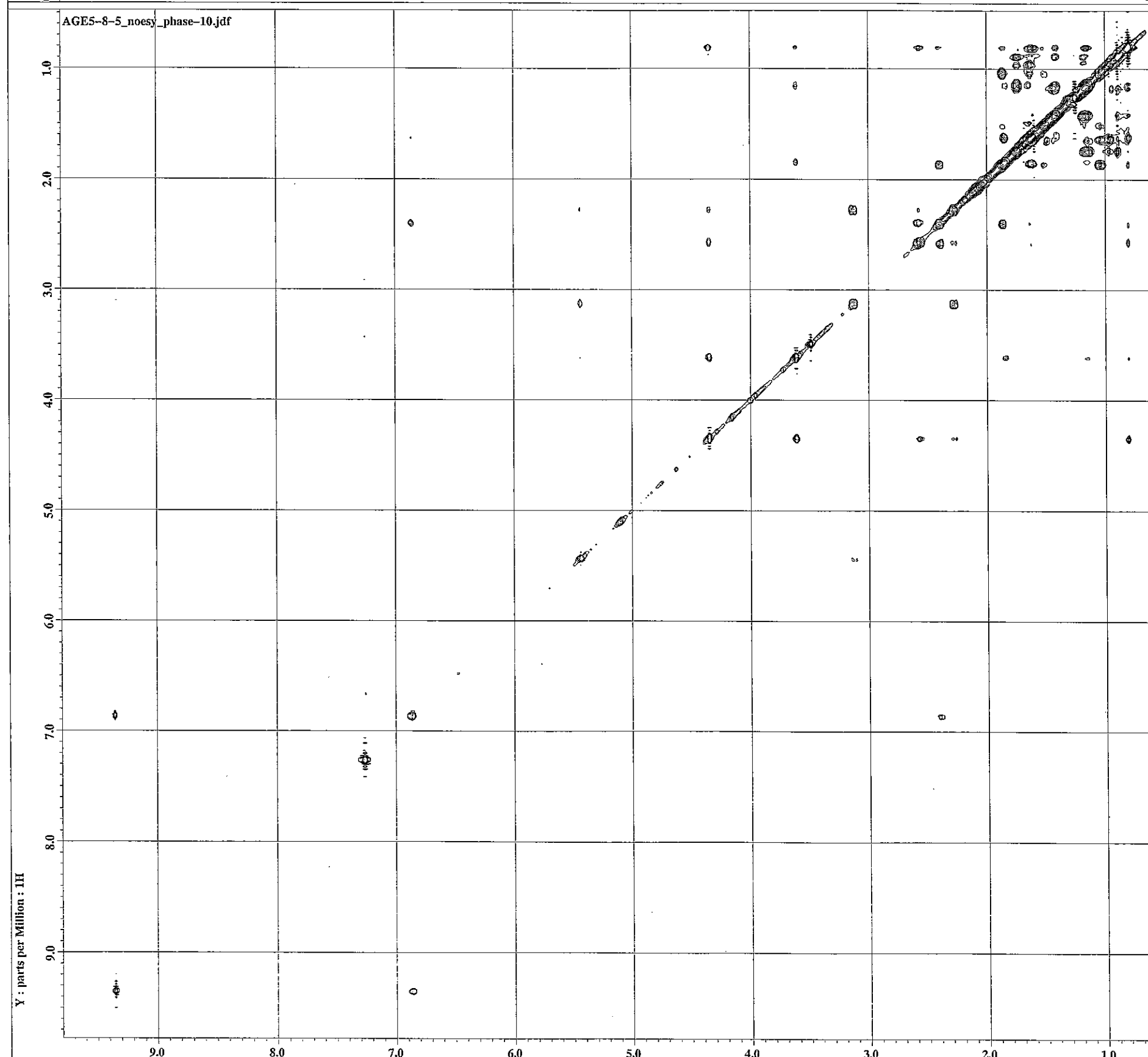
Filename = AGE5-8-5\_noesy\_phase-  
Author = delta  
Experiment = noesy\_phase.ex2  
Sample\_id = 001  
Solvent = CHLOROFORM-D  
Creation\_time = 15-FEB-2012 09:32:00  
Revision\_time = 15-FEB-2012 10:01:35  
Current\_time = 15-FEB-2012 10:03:11

Comment = phase sensitive noesy  
Data\_format = 2D COMPLEX COMPLEX  
Dim\_size = 819, 512  
Dim\_title = 1H 1H  
Dim\_units = [ppm] [ppm]  
Dimensions = X Y  
Site = ECA500  
Spectrometer = JNM-ECA500

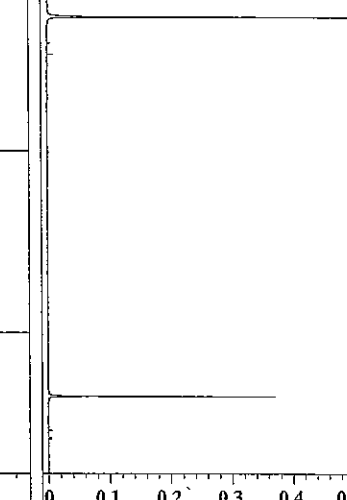
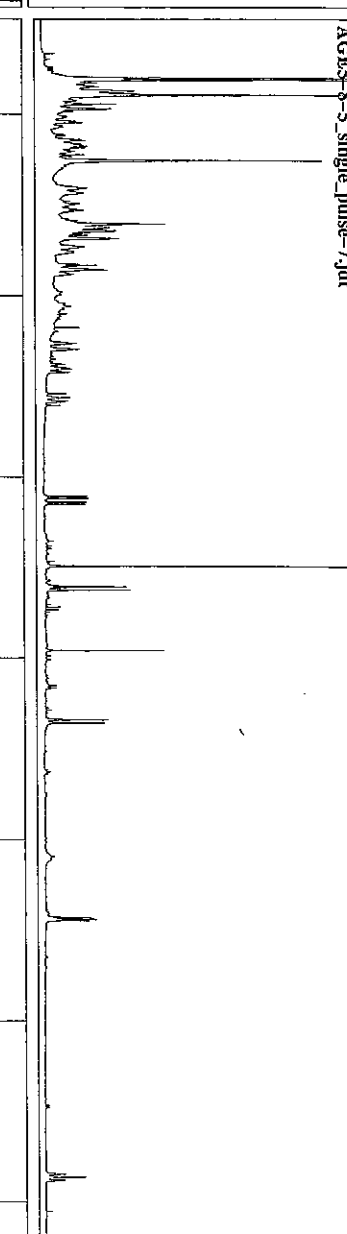
Field\_strength = 11.7473579[T] (500[MH  
X\_acq\_duration = 0.1363968[s]  
X\_domain = 1H  
X\_freq = 500.15991521[MHz]  
X\_offset = 5.0639[ppm]  
X\_points = 1024  
X\_prescans = 4  
X\_resolution = 7.3315503[Hz]  
X\_sweep = 7.50750751[kHz]  
Y\_domain = 1H  
Y\_freq = 500.15991521[MHz]  
Y\_offset = 5.0639[ppm]  
Y\_points = 256  
Y\_prescans = 0  
Y\_resolution = 23.44687875[Hz]  
Y\_sweep = 6.00240096[kHz]  
Irr\_domain = 1H  
Irr\_freq = 500.15991521[MHz]  
Irr\_offset = 5.0[ppm]  
Tri\_domain = 1H  
Tri\_freq = 500.15991521[MHz]  
Tri\_offset = 5.0[ppm]  
Clipped = FALSE  
Mod\_return = 1  
Scans = 16  
Total\_scans = 4096

X\_acq\_time = 0.1363968[s]  
X\_atn = 2.4[db]  
X\_pulse = 12[us]  
Y\_acq\_time = 42.6496[ms]  
Y\_p0\_correction = 0  
Y\_p1\_correction = 180  
Irr\_mode = Off  
Tri\_mode = Off  
Dante\_presat = FALSE  
Initial\_wait = 1[s]  
Mix\_time = 0.7[s]  
Recvr\_gain = 42  
Relaxation\_delay = 2[s]  
Repetition\_time = 2.1363968[s]  
Scramble = 1[ms]  
T1 = 67.94[us]  
T1\_cal1 = 67.94[us]  
T1\_cal2 = 0.15124[ms]  
T1\_cal3 = 0.23454[ms]  
T1\_cal4 = 0.31784[ms]  
T1\_cal5 = 0.40114[ms]  
T1\_cal6 = 0.48444[ms]  
Temp\_get = 22[dc]

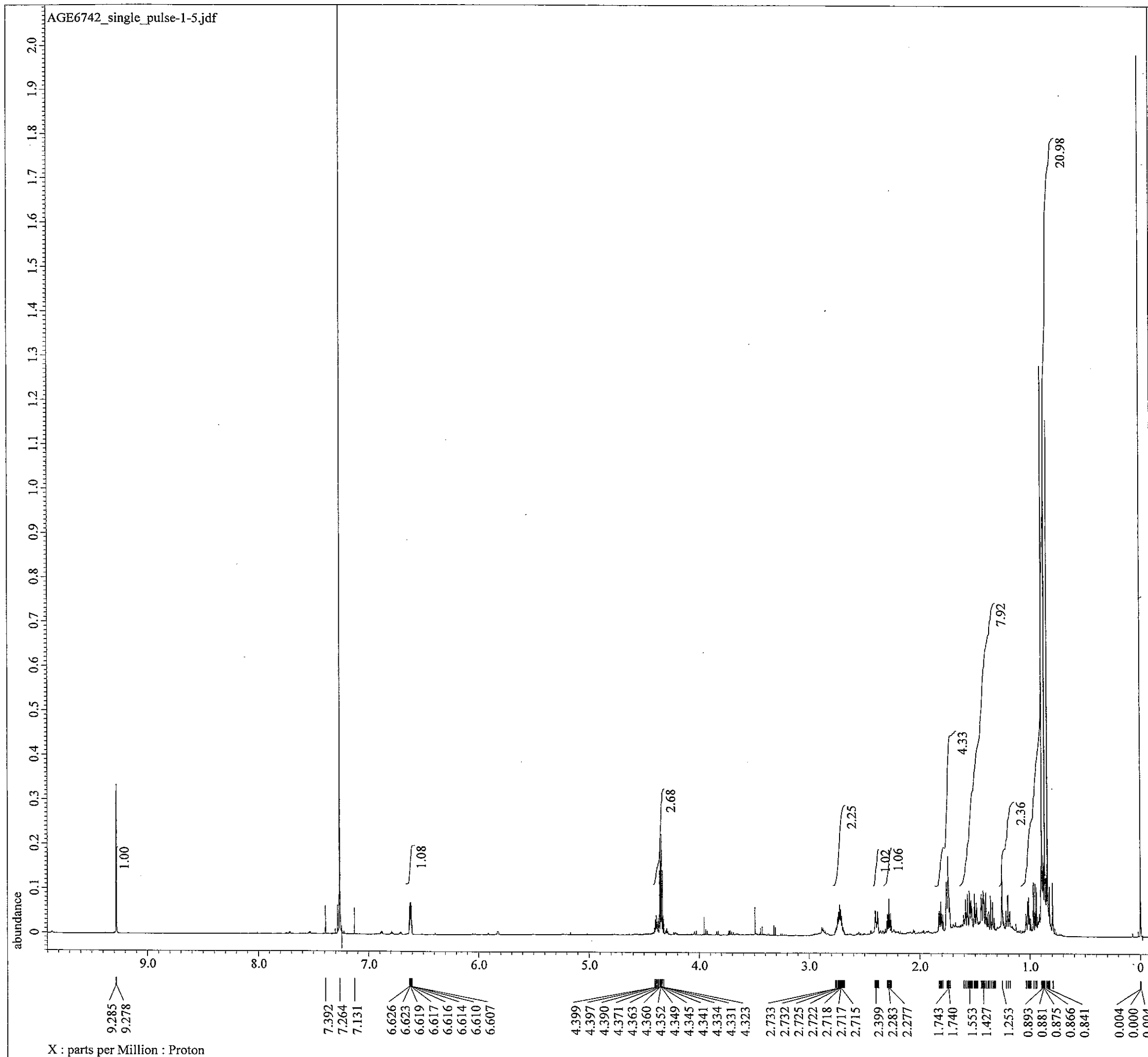
AGE5-8-5\_noesy\_phase-10.jdf



AGE5-8-5\_single\_pulse-7.jdf



abundance



----- PROCESSING PARAMETERS -----  
dc balance( 0, FALSE )  
sext( 0.2[Hz], 0.0[s] )  
trapezoid( 0[%], 0[%], 80[%], 100[%] )  
zerofill( 1 )  
fft( 1, TRUE, TRUE )  
machinephase  
ppm  
phase( -4, 1, 35.02327[%] )

以下に由来: AGE6742\_single\_pulse-1-1.jdf

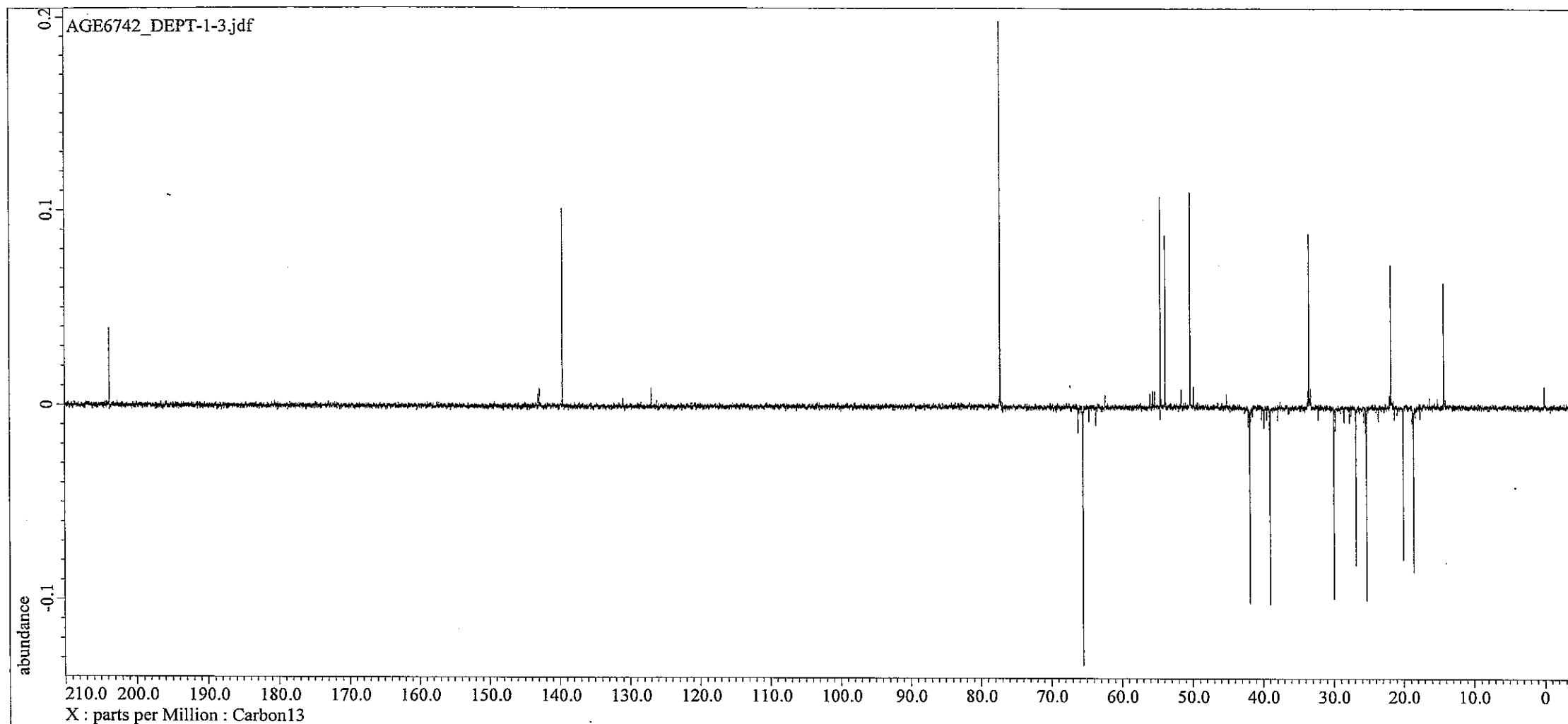
Filename = C:\Users\delta\Documents\J  
Author = delta  
Experiment = single\_pulse.jpg  
Sample\_Id = AGE6742  
Solvent = CHLOROFORM-D  
Creation\_Time = 20-SEP-2013 14:22:42  
Revision\_Time = 20-SEP-2013 15:46:00  
Current\_Time = 20-SEP-2013 15:46:11

Comment = single\_pulse  
Data\_Format = 1D COMPLEX  
Dim Size = 52429  
Dim Title = Proton  
Dim Units = [ppm]  
Dimensions = X  
Site = JNM-ECA800  
Spectrometer = DELTA2\_NMR

Field Strength = 18.79305132[T] (800[MHz])  
X\_Acq\_Duration = 4.36207616[s]  
X\_Domain = 1H  
X\_Freq = 800.14000039[MHz]  
X\_Offset = 5[ppm]  
X\_Points = 65536  
X\_Prescans = 1  
X\_Resolution = 0.22924863[Hz]  
X\_Sweep = 15.02403846[kHz]  
X\_Sweep\_Clippped = 12.01923077[kHz]  
Irr\_Domain = Proton  
Irr\_Freq = 800.14000039[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = Proton  
Tri\_Freq = 800.14000039[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 16  
Total\_Scans = 16

Relaxation\_Delay = 5[s]  
Recvr Gain = 46  
Temp\_Get = 22[dc]  
X\_90\_Width = 15.5[us]  
X\_Acq\_Time = 4.36207616[s]  
X\_Angle = 45[deg]  
X\_Atn = 3.2[dB]  
X\_Pulse = 7.75[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 9.36207616[s]





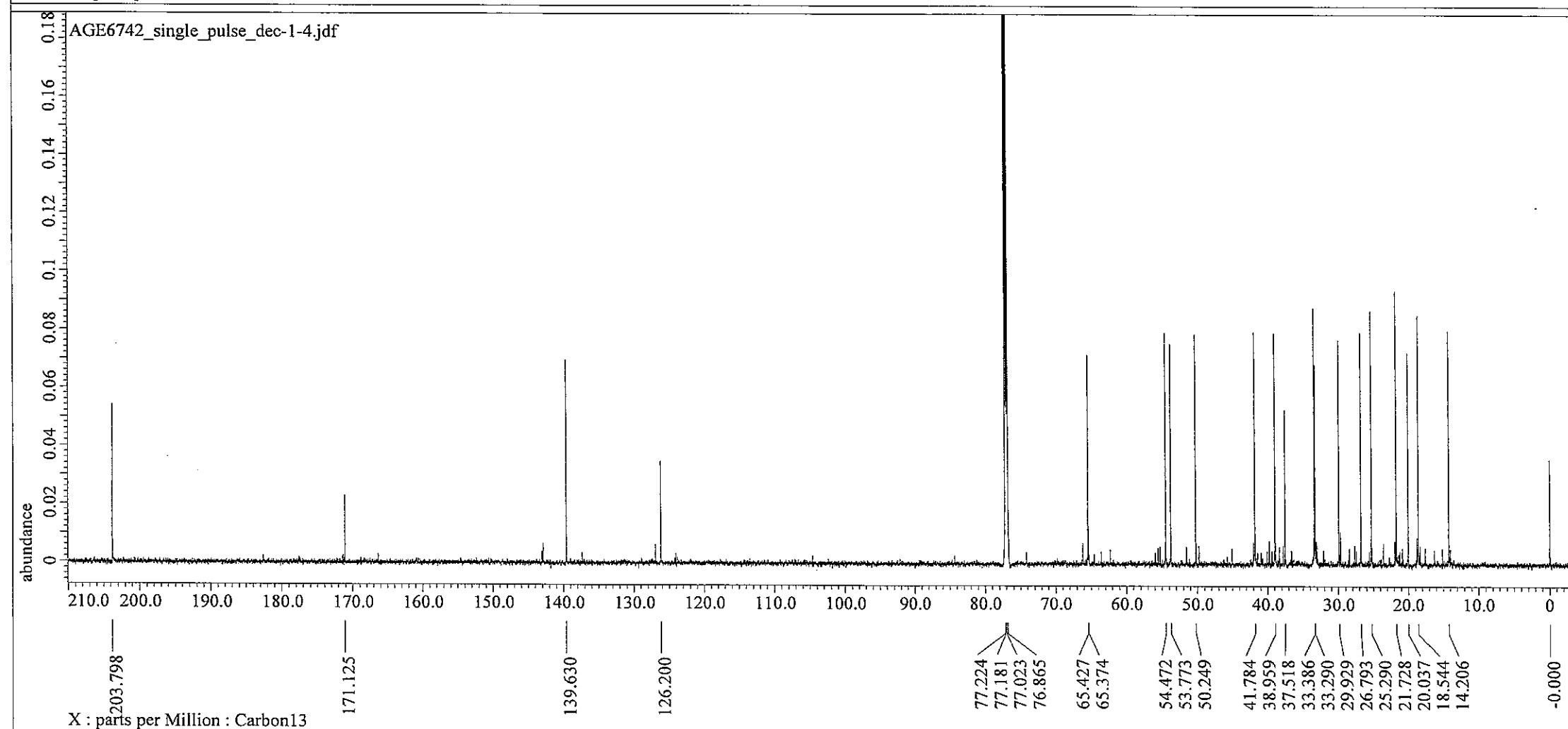
----- PROCESSING PARAMETERS -----  
dc\_balance( 0, FALSE )  
sexp( 2.0[Hz], 0.0[s] )  
trapezoid( 0[%], 0[%], 80[%], 100[%] )  
zerofill( 1 )  
fft( 1, TRUE, TRUE )  
machinephase  
ppm  
phase( 0.5, 0, 59.29274[%] )  
base\_correct( None, 10, Smooth )

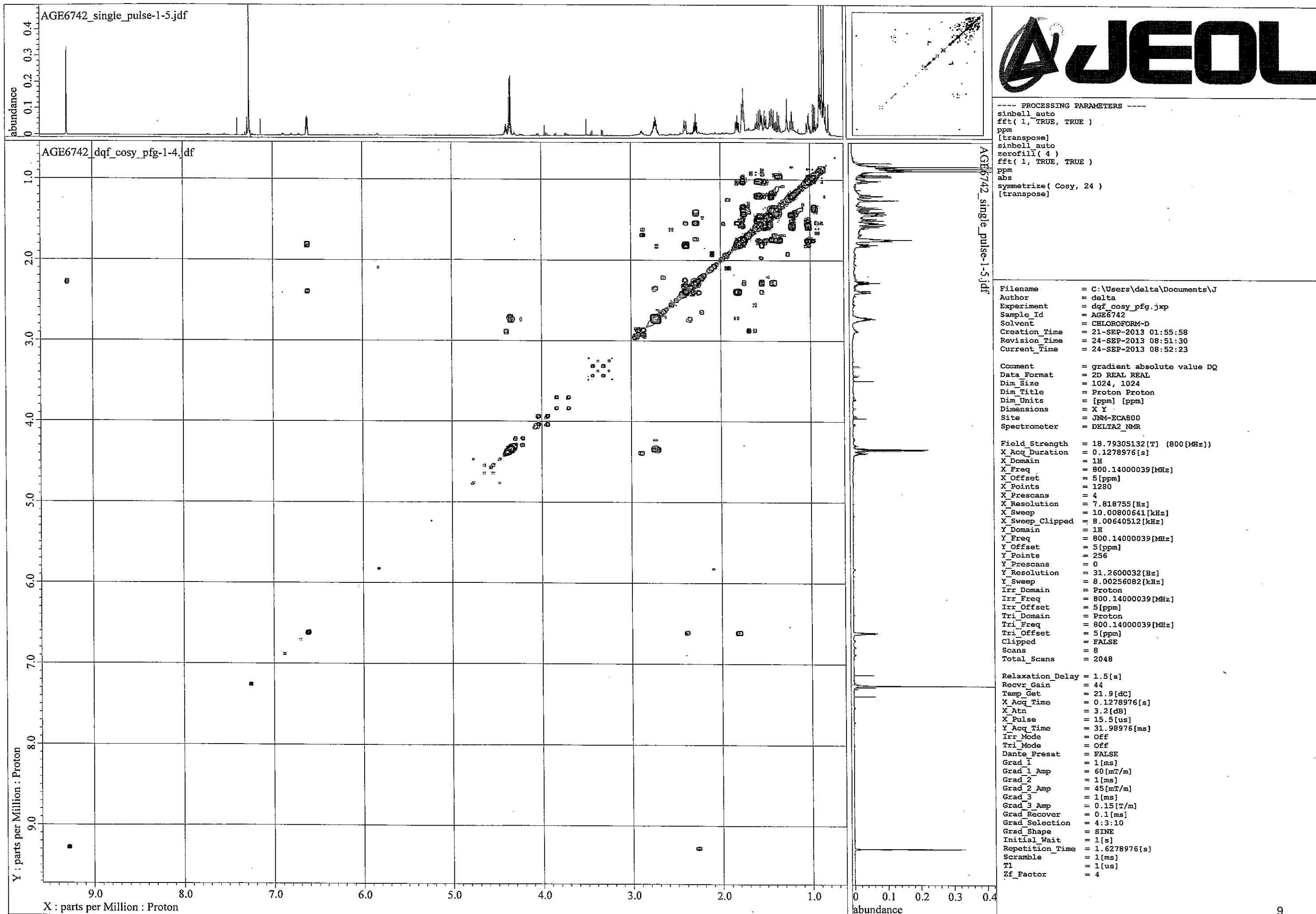
Filename = C:\Users\delta\Documents\J  
Author = delta  
Experiment = single\_pulse\_dec.jxp  
Sample Id = AGE6742  
Solvent = CHLOROFORM-D  
Creation Time = 20-SEP-2013 14:25:55  
Revision Time = 24-SEP-2013 08:48:55  
Current Time = 24-SEP-2013 08:49:36

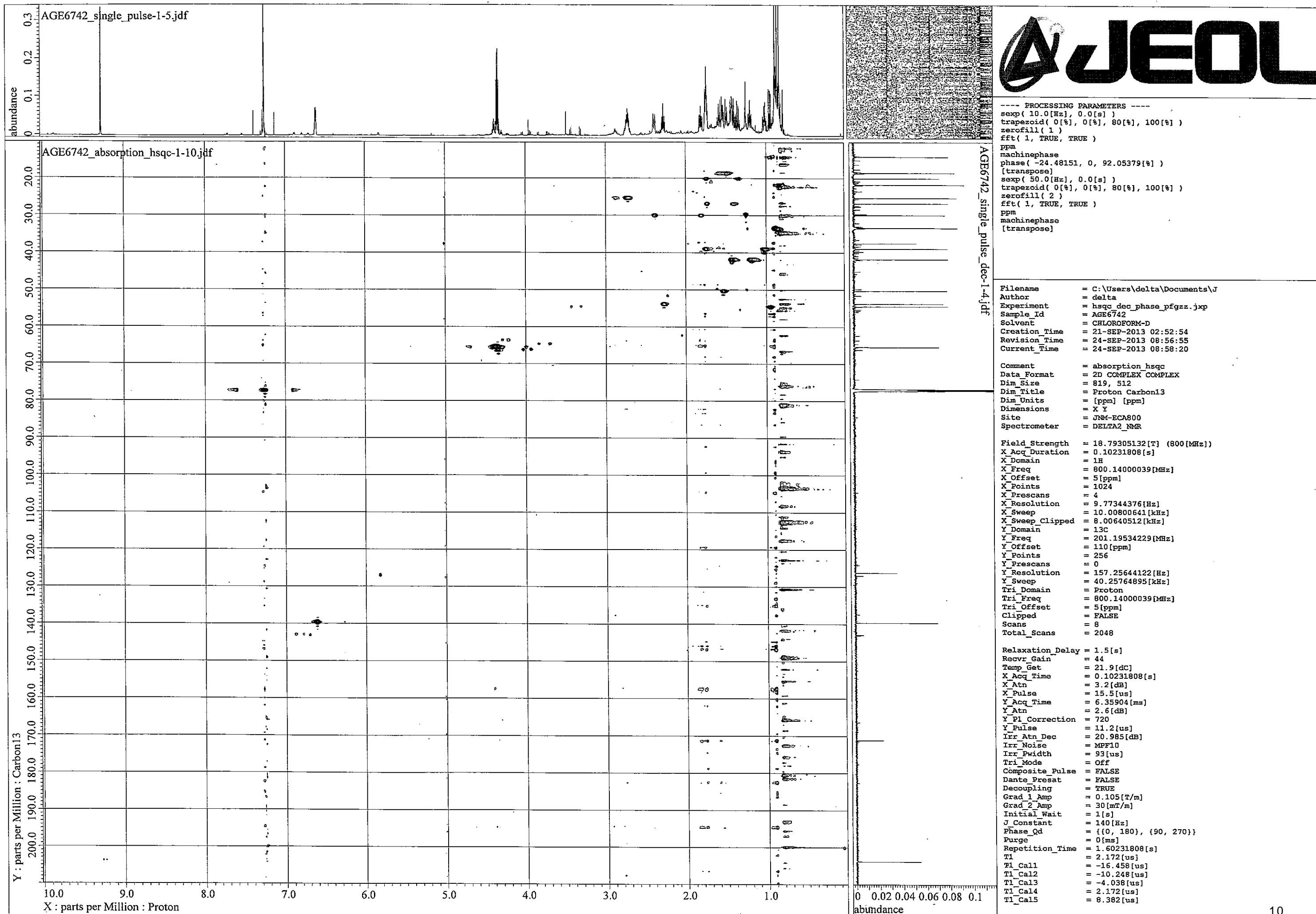
Comment = single pulse decoupled gat  
Data Format = 1D REAL  
Dim Size = 52429  
Dim Title = Carbon13  
Dim Units = [ppm]  
Dimensions = X  
Site = JNM-ECA800  
Spectrometer = DELTA2\_NMR

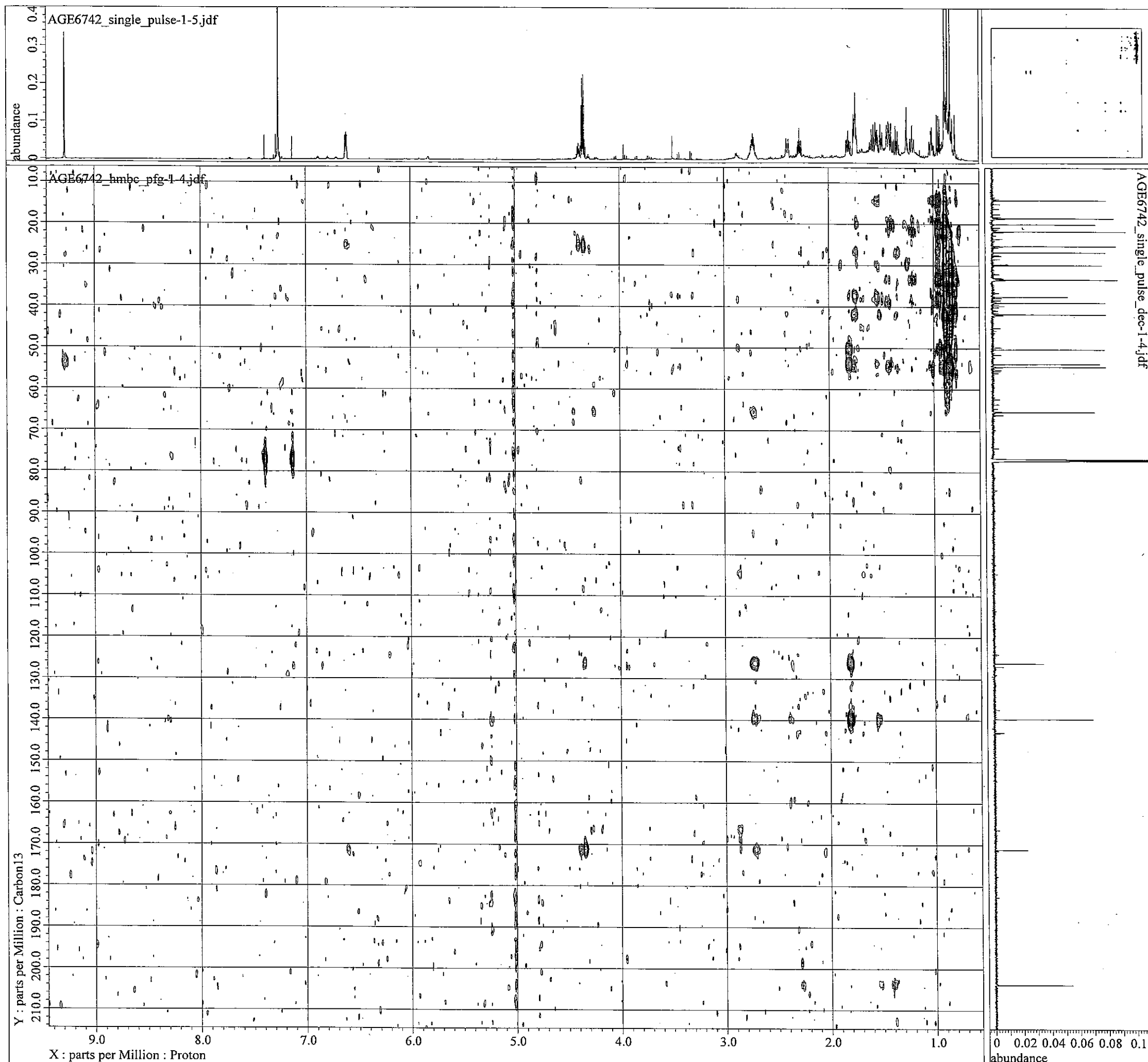
Field Strength = 18.79305132[T] (800[MHz])  
X\_Acq\_Duration = 1.03809024[s]  
X\_Domain = 13C  
X\_Freq = 201.19534229[MHz]  
X\_Offset = 100[ppm]  
X\_Points = 65536  
X\_Prescans = 4  
X\_Resolution = 0.96330739[Hz]  
X\_Sweep = 63.13131313[kHz]  
X\_Sweep\_Clipped = 50.50505051[kHz]  
Irr\_Domain = Proton  
Irr\_Freq = 800.14000039[MHz]  
Irr\_Offset = 5[ppm]  
Clipped = TRUE  
Scans = 10000  
Total\_Scans = 10000

Relaxation\_Delay = 2[s]  
Recvr\_Gain = 58  
Temp\_Get = 22.6[dC]  
X\_90\_Width = 11.2[us]  
X\_Acq\_Time = 1.03809024[s]  
X\_Angle = 30[deg]  
X\_Atn = 2.6[dB]  
X\_Pulse = 3.73333333[us]  
Irr\_Atn\_Dec = 14.51[dB]  
Irr\_Atn\_No = 14.51[dB]  
Irr\_Noise = WALTZ  
Irr\_Pwidth = 57[us]  
Decoupling = TRUE  
Initial\_Wait = 1[s]  
Noe = TRUE  
Noe Time = 2[s]  
Repetition\_Time = 3.03809024[s]









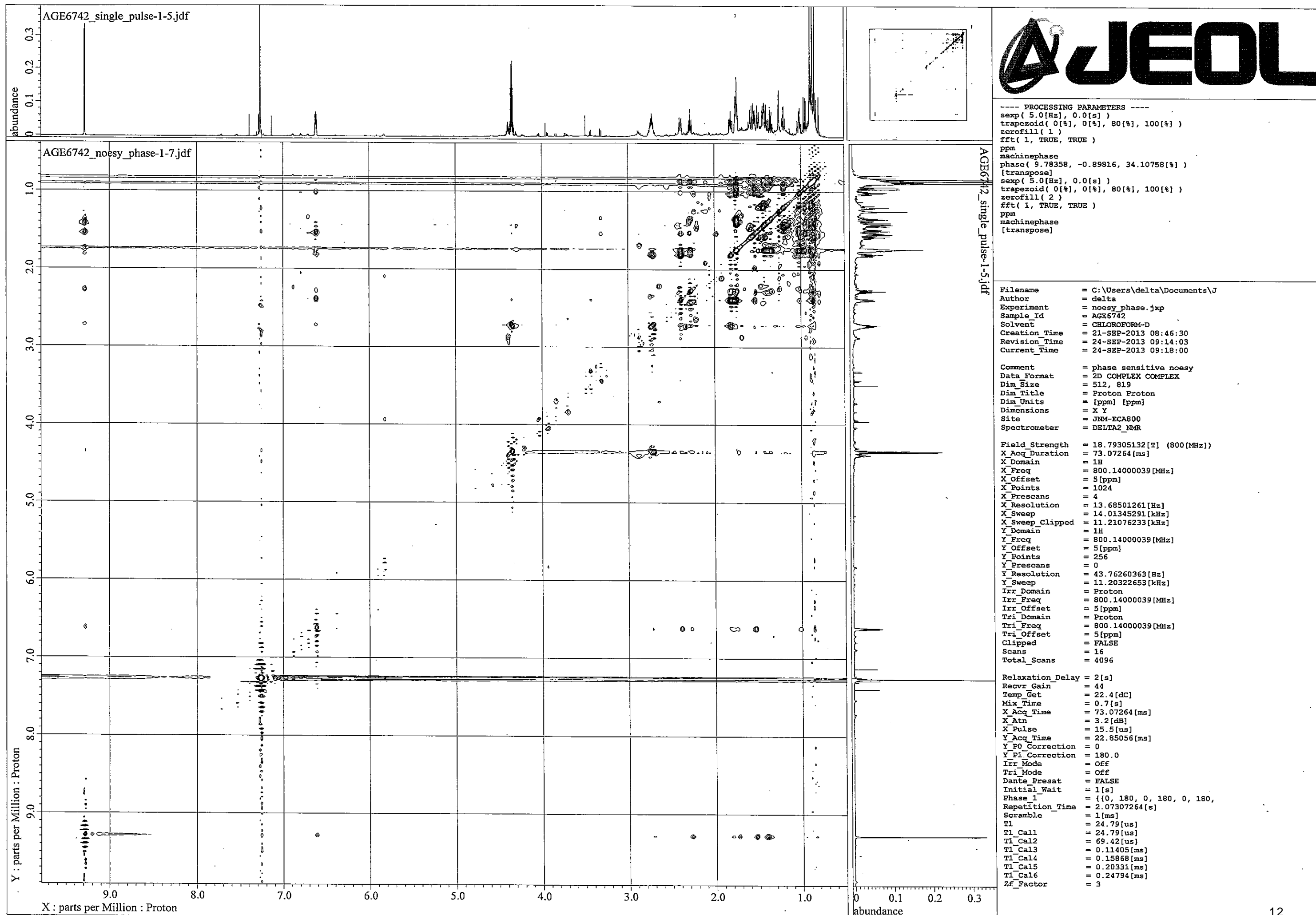
---- PROCESSING PARAMETERS ----  
gauss( 5.0[Hz], 0.0[s] )  
sinbell auto  
zerofill( 1 )  
fft( 1, TRUE, TRUE )  
ppm  
[transpose]  
sinbell4( -60, 160 )  
trapezoid( 0[%], 5[%], 80[%], 100[%] )  
zerofill( 2 )  
fft( 1, TRUE, TRUE )  
ppm  
abs  
[transpose]

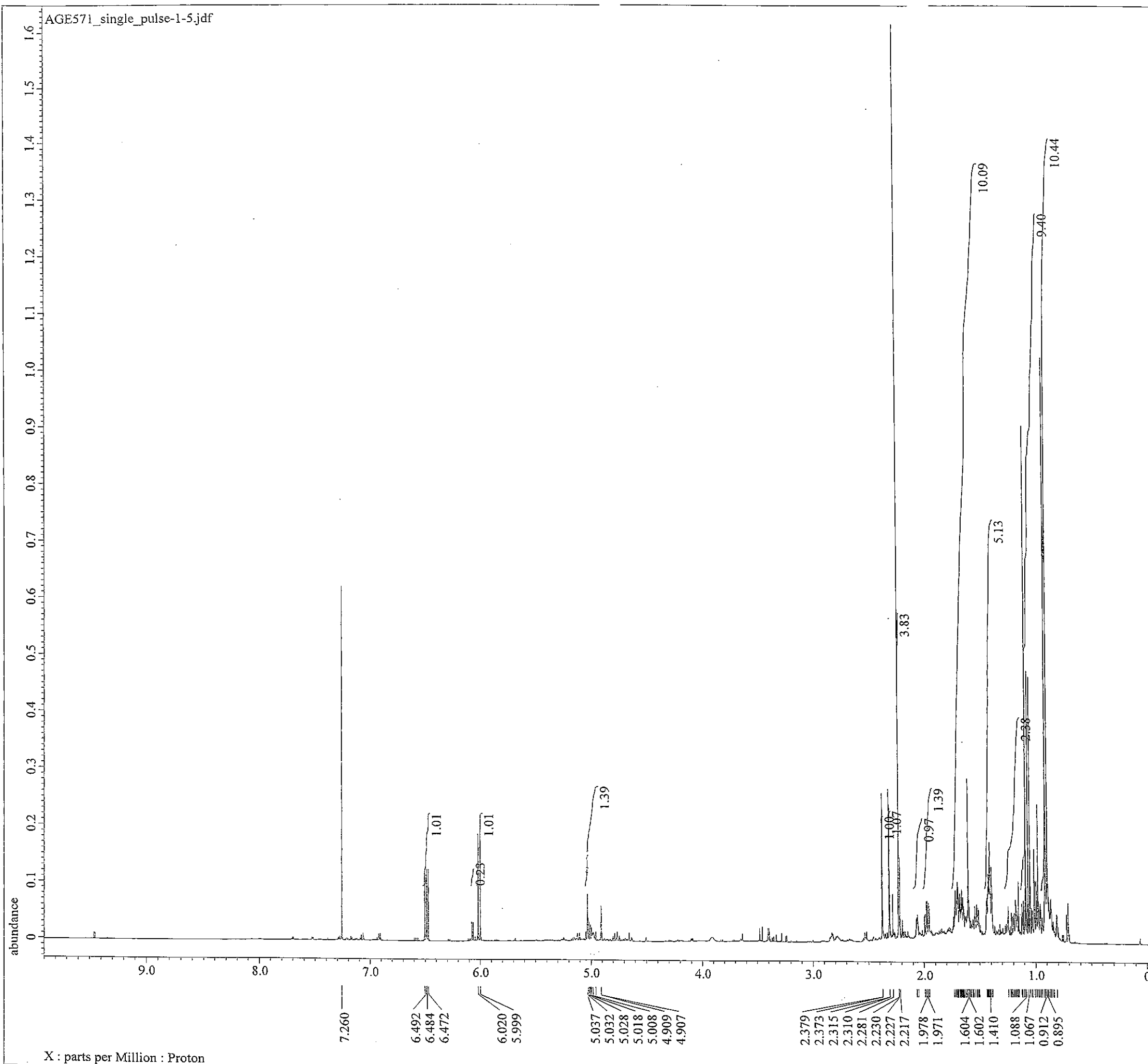
Filename = C:\Users\delta\Documents\J  
Author = delta  
Experiment = hmhc\_pfg.jxp  
Sample Id = AGE6742  
Solvent = CHLOROFORM-D  
Creation Time = 21-SEP-2013 04:43:50  
Revision Time = 24-SEP-2013 09:01:49  
Current Time = 24-SEP-2013 09:03:13

Comment = gradient enhanced HMBC  
Data Format = 2D REAL REAL  
Dim Size = 512, 1638  
Dim Title = Carbon13 Proton  
Dim Units = [ppm] [ppm]  
Dimensions = X Y  
Site = JNM-ECA800  
Spectrometer = DELTA2 NMR

Field Strength = 18.79305132[T] (800[MHz])  
X Acq Duration = 0.20463616[s]  
X Domain = 1H  
X Freq = 800.14000039[MHz]  
X Offset = 5[ppm]  
X Points = 2048  
X Prescans = 4  
X Resolution = 4.88672188[Hz]  
X Sweep = 10.00800641[kHz]  
X Sweep Clipped = 8.00640512[kHz]  
Y Domain = 13C  
Y Freq = 201.19534229[MHz]  
Y Offset = 100[ppm]  
Y Points = 256  
Y Prescans = 0  
Y Resolution = 196.49144869[Hz]  
Y Sweep = 50.30181087[kHz]  
Tri Domain = Proton  
Tri Freq = 800.14000039[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 32  
Total Scans = 8192

Relaxation Delay = 1.5[s]  
Recvr Gain = 44  
Temp Get = 21.9[dc]  
X Acq Time = 0.20463616[s]  
X Atn = 3.2[dB]  
X Pulse = 15.5[us]  
Y Acq Time = 5.08928[ms]  
Y Atn = 2.6[dB]  
Y Pulse = 11.2[us]  
Tri Mode = Off  
Dante Presat = FALSE  
Delta = 62.5[ms]  
Delta 1 = 3.83288616[ms]  
Delta 2 = 3.43642612[ms]  
Delta 3 = 3.11429461[ms]  
Grad 1 = 1[ms]  
Grad 1 Amp = 0.18[T/m]  
Grad 2 = 1[ms]  
Grad 2 Amp = 0.18[T/m]  
Grad 3 = 1[ms]  
Grad 3 Amp = 90.54325956[mT/m]  
Grad Lpf = 1[ms]  
Grad Lpf1 Amp = 45[mT/m]  
Grad Lpf2 Amp = -25.71428571[mT/m]  
Grad Lpf3 Amp = -12.85714286[mT/m]  
Grad Lpf4 Amp = -6.42857143[mT/m]  
Grad Recover = 0.1[ms]  
Grad Selection = 13C = 1.988:1.988:1  
Grad Shape = SINE  
Initial Wait = 1[s]





----- PROCESSING PARAMETERS -----  
dc\_balance( 0, FALSE )  
sexp( 0.2[Hz], 0.0[s] )  
trapezoid( 0[%], 0[%], 80[%], 100[%] )  
zerofill( 1 )  
fft( 1, TRUE, TRUE )  
machinephase  
ppm  
phase( -6, 0, 68.61601[%] )

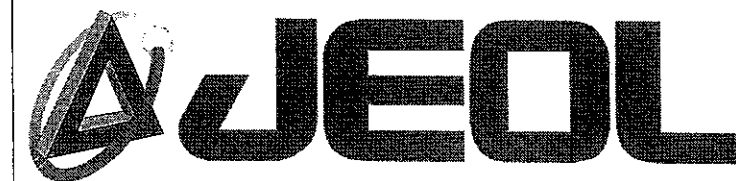
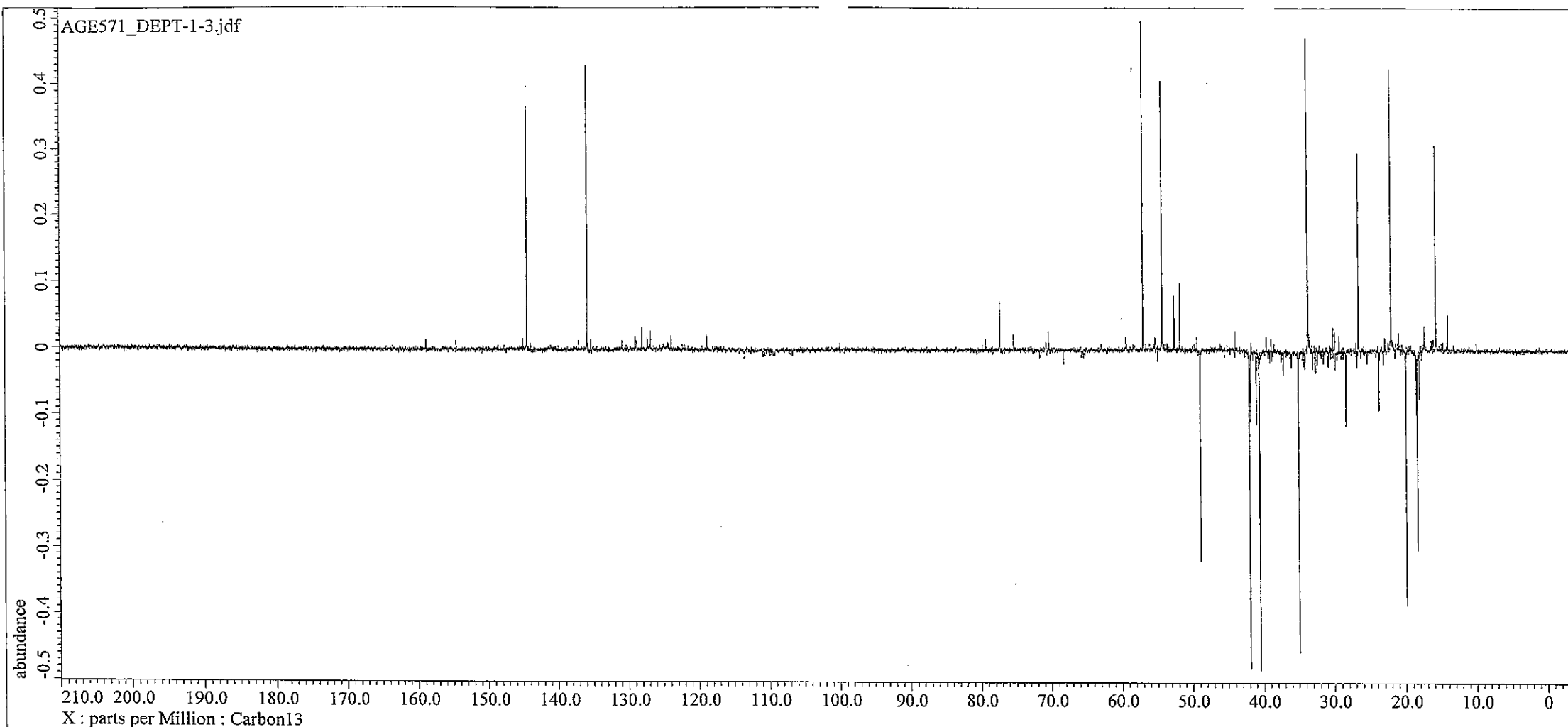
以下に由来: AGE571\_single\_pulse-1-1.jdf

Filename = C:\Users\delta\Documents\J  
Author = delta  
Experiment = single\_pulse.jxp  
Sample Id = AGE571  
Solvent = CHLOROFORM-D  
Creation Time = 15-NOV-2012 10:45:39  
Revision Time = 15-NOV-2012 11:02:40  
Current Time = 15-NOV-2012 11:03:06

Comment = single\_pulse  
Data Format = 1D COMPLEX  
Dim Size = 52429  
Dim Title = Proton  
Dim Units = [ppm]  
Dimensions = X  
Site = JNM-ECA800  
Spectrometer = DELTA2\_NMR

Field Strength = 18.79305132[T] (800[MHz])  
X\_Acq\_Duration = 4.36207616[s]  
X\_Domain = 1H  
X\_Freq = 800.14000039[MHz]  
X\_Offset = 5[ppm]  
X Points = 65536  
X Prescans = 1  
X Resolution = 0.22924863[Hz]  
X Sweep = 15.02403846[kHz]  
X Sweep Clipped = 12.01923077[kHz]  
Irr\_Domain = Proton  
Irr\_Freq = 800.14000039[MHz]  
Irr\_Offset = 5[ppm]  
Tri\_Domain = Proton  
Tri\_Freq = 800.14000039[MHz]  
Tri\_Offset = 5[ppm]  
Clipped = FALSE  
Scans = 32  
Total\_Scans = 32

Relaxation\_Delay = 5[s]  
Recvr\_Gain = 36  
Temp\_Get = 20.9[dC]  
X\_90\_Width = 16[us]  
X\_Acq\_Time = 4.36207616[s]  
X\_Angle = 45[deg]  
X\_Atn = 3[dB]  
X\_Pulse = 8[us]  
Irr\_Mode = Off  
Tri\_Mode = Off  
Dante\_Presat = FALSE  
Initial\_Wait = 1[s]  
Repetition\_Time = 9.36207616[s]

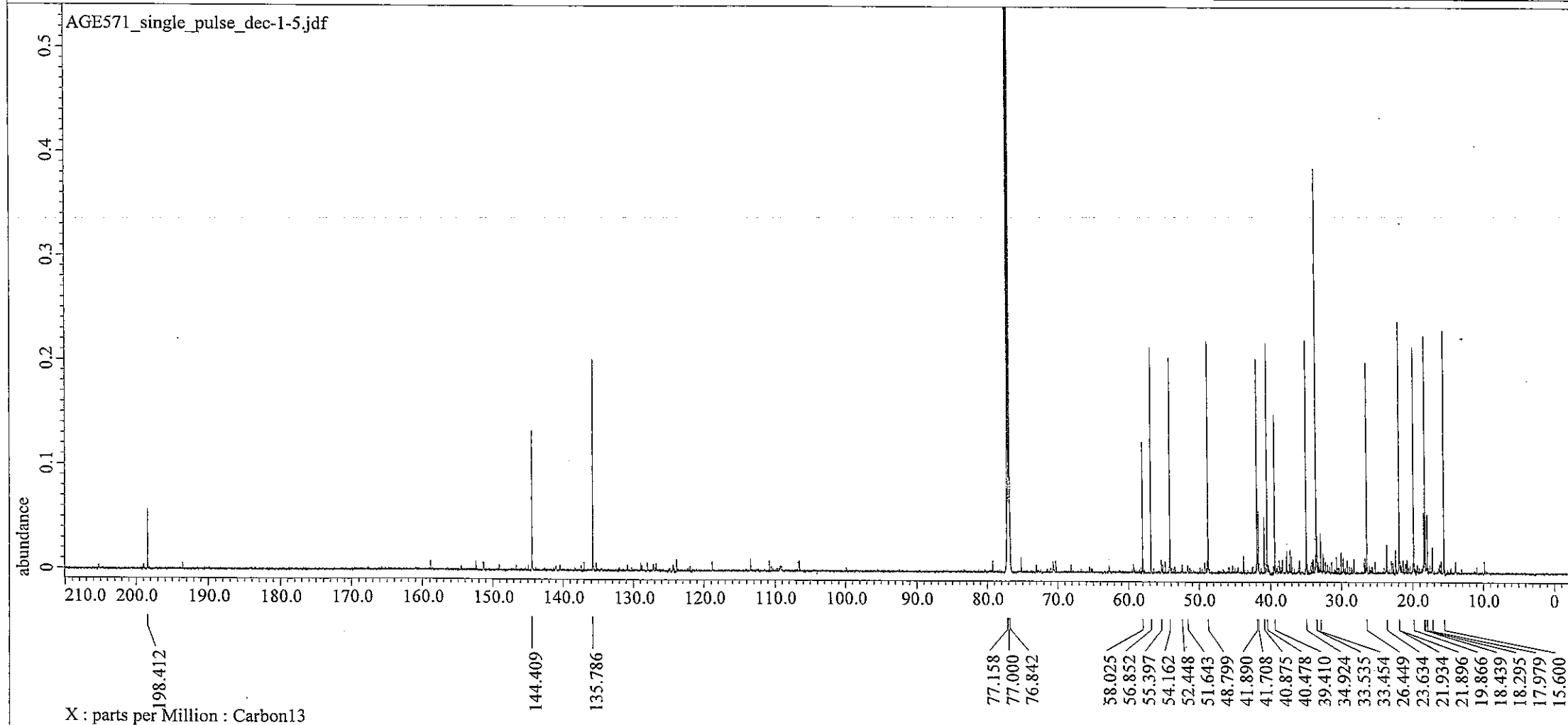


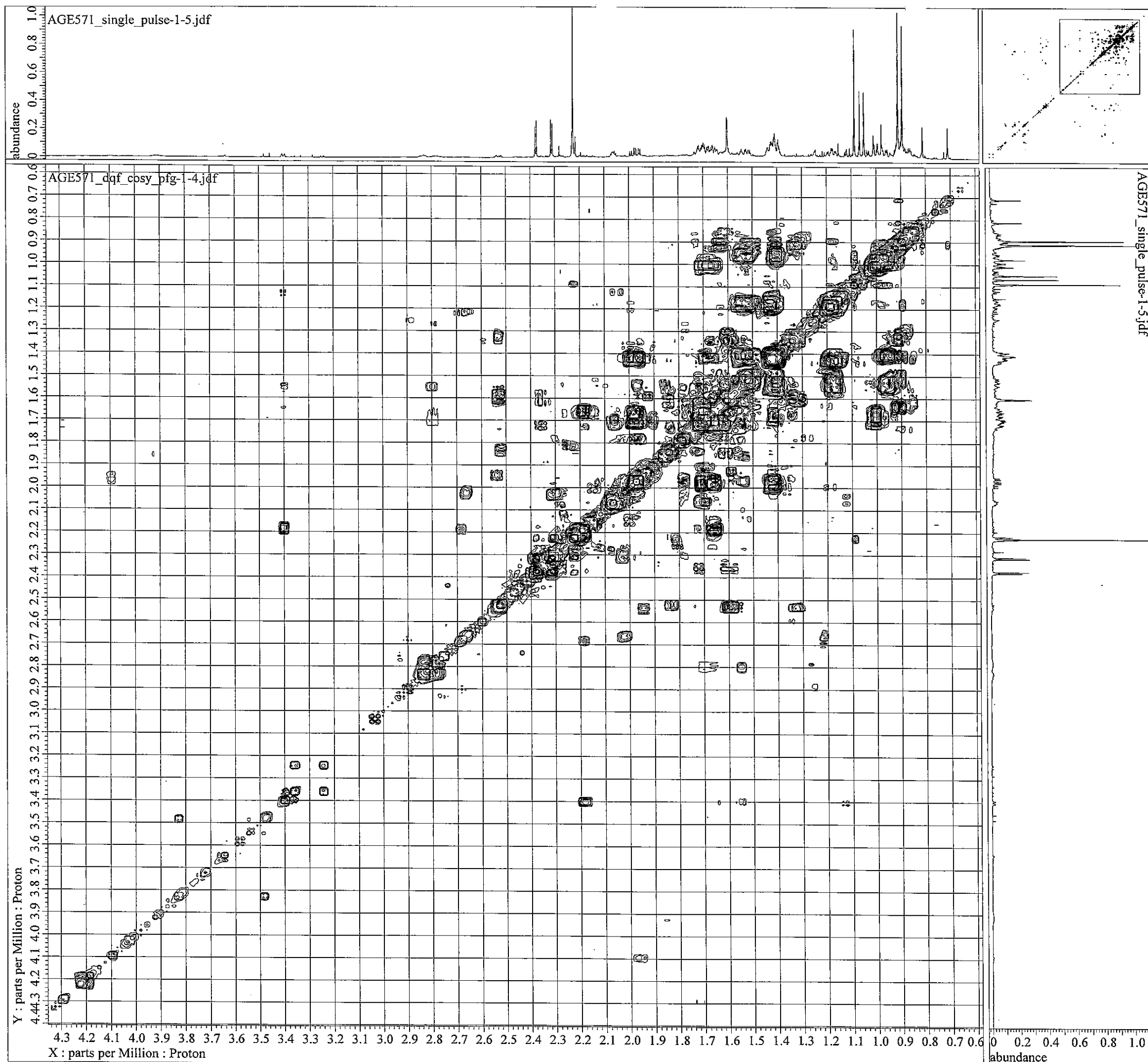
----- PROCESSING PARAMETERS -----  
dc\_balance( 0, FALSE )  
sexp( 2.0[Hz], 0.0[s] )  
trapezoid( 0[%], 0[%], 80[%], 100[%] )  
zerofill( 1 )  
fft( 1, TRUE, TRUE )  
machinephase  
ppm  
phase( 0.75, 0, 58.98566[%] )  
base\_correct( None, 10, Smooth )

Filename = C:\Users\delta\Documents\J  
Author = delta  
Experiment = single\_pulse\_dec.jxp  
Sample\_Id = AGE571  
Solvent = CHLOROFORM-D  
Creation\_Time = 15-NOV-2012 10:51:21  
Revision\_Time = 16-NOV-2012 10:39:18  
Current\_Time = 16-NOV-2012 10:41:49  
  
Comment = single pulse decoupled gat  
Data\_Format = 1D REAL  
Dim\_Size = 52429  
Dim\_Title = Carbon13  
Dim\_Units = [ppm]  
Dimensions = X  
Site = JNM-ECAB00  
Spectrometer = DELTA2\_NMR

Field\_Strength = 18.79305132[T] (800[MHz])  
X\_Acq\_Duration = 1.03809024[s]  
X\_Domain = 13C  
X\_Freq = 201.19534229[MHz]  
X\_Offset = 100[ppm]  
X\_Points = 65536  
X\_Prescans = 4  
X\_Resolution = 0.96330739[Hz]  
X\_Sweep = 63.13131313[kHz]  
X\_Sweep\_Clipped = 50.50505051[kHz]  
Irr\_Domain = Proton  
Irr\_Freq = 800.14000039[MHz]  
Irr\_Offset = 5[ppm]  
Clipped = TRUE  
Scans = 10000  
Total\_Scans = 10000

Relaxation\_Delay = 2[s]  
Recvr\_Gain = 60  
Temp\_Get = 21.1[degC]  
X\_90\_Width = 11.2[us]  
X\_Acq\_Time = 1.03809024[s]  
X\_Angle = 30[deg]  
X\_Atn = 2.6[dB]  
X\_Pulse = 3.73333333[us]  
Irr\_Atn\_Dec = 14.35[dB]  
Irr\_Atn\_Noe = 14.35[dB]  
Irr\_Noise = WALTZ  
Irr\_Pwidth = 57[us]  
Decoupling = TRUE  
Initial\_Wait = 1[s]  
Noe = TRUE  
Noe\_Time = 2[s]  
Repetition\_Time = 3.03809024[s]





----- PROCESSING PARAMETERS -----  
sinbell\_auto  
fft( 1, TRUE, TRUE )  
ppm  
[transpose]  
sinbell\_auto  
zerofill( 4 )  
fft( 1, TRUE, TRUE )  
ppm  
abs  
symmetrize( Cosy, 24 )  
[transpose]


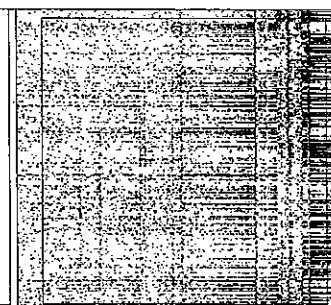
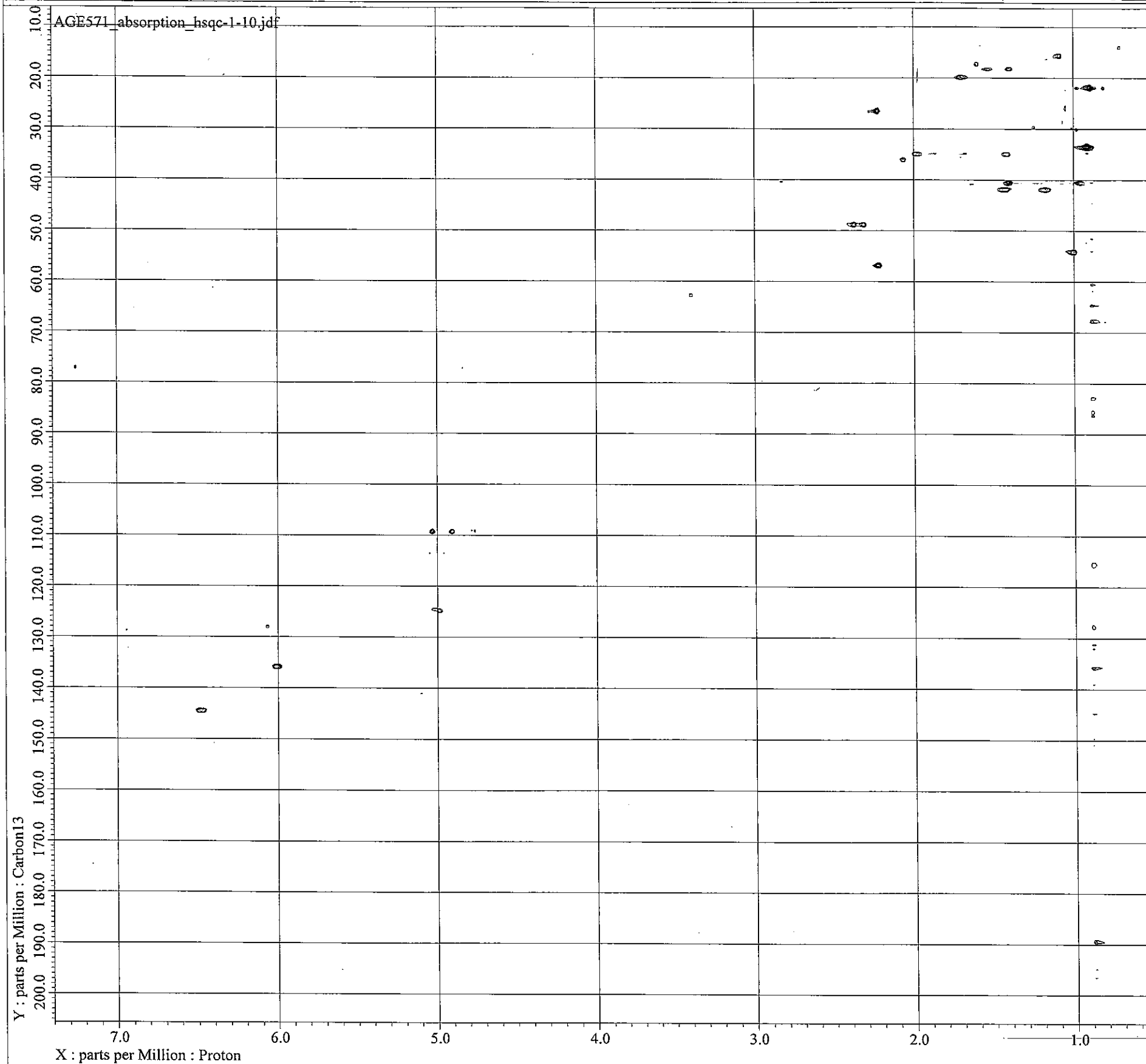
Filename = C:\Users\delta\Documents\J  
Author = delta  
Experiment = dqf\_cpsy\_pfg.jxp  
Sample Id = AGE571  
Solvent = CHLOROFORM-D  
Creation Time = 15-NOV-2012 21:50:15  
Revision Time = 16-NOV-2012 10:43:31  
Current Time = 16-NOV-2012 10:47:30

Comment = gradient absolute value DQ  
Data Format = 2D REAL REAL  
Dim Size = 1024, 1024  
Dim Title = Proton Proton  
Dim Units = [ppm] [ppm]  
Dimensions = X Y  
Site = JNM-ECA800  
Spectrometer = DELTA2\_NMR

Field Strength = 18.79305132[T] (800[MHz])  
X Acq Duration = 0.1599488[s]  
X Domain = 1H  
X Freq = 800.14000039[MHz]  
X Offset = 4[ppm]  
X Points = 1280  
X Prescans = 4  
X Resolution = 6.25200064[Hz]  
X Sweep = 8.00256082[kHz]  
X Sweep\_Clippped = 6.40204866[kHz]  
Y Domain = 1H  
Y Freq = 800.14000039[MHz]  
Y Offset = 4[ppm]  
Y Points = 256  
Y Prescans = 0  
Y Resolution = 25.00480092[Hz]  
Y Sweep = 6.40122904[kHz]  
Irr Domain = Proton  
Irr Freq = 800.14000039[MHz]  
Irr Offset = 5[ppm]  
Tri Domain = Proton  
Tri Freq = 800.14000039[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 8  
Total Scans = 2048

Relaxation Delay = 1.5[s]  
Recvr Gain = 34  
Temp Get = 20.8[dC]  
X Acq Time = 0.1599488[s]  
X Atn = 3[dB]  
X Pulse = 16[us]  
Y Acq Time = 39.99232[ms]  
Irr Mode = Off  
Tri Mode = Off  
Dante Presat = FALSE  
Grad 1 = 1[ms]  
Grad 1 Amp = 60[mT/m]  
Grad 2 = 1[ms]  
Grad 2 Amp = 45[mT/m]  
Grad 3 = 1[ms]  
Grad 3 Amp = 0.15[T/m]  
Grad Recover = 0.1[ms]  
Grad Selection = 4:3:10  
Grad Shape = SINE  
Initial Wait = 1[s]  
Repetition Time = 1.6599488[s]  
Scramble = 1[ms]  
Ti = 1[us]  
Zf Factor = 4





**JEOL**

```

---- PROCESSING PARAMETERS ----
sexp( 10.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
machinephase
phase( 3.16575, -60.417, 88.75306[%] )
[transposel
sexp( 50.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 2 )
fft( 1, TRUE, TRUE )
ppm
machinephase
[transposel

```

```

Filename           = C:\Users\delta\Documents\J
Author             = delta
Experiment         = hsqc dec_phase_pfgzz.jxp
Sample_Id          = AGES71
Solvent            = CHLOROFORM-D
Creation_Time      = 15-NOV-2012 22:48:26
Revision_Time      = 16-NOV-2012 10:54:52
Current_Time       = 16-NOV-2012 10:56:20

Comment            = absorption hsqc
Data Format         = 2D COMPLEX COMPLEX
Dim_Size           = 819, 512
Dim_Title          = Proton Carbon13
Dim_Units          = [ppm] [ppm]
Dimensions         = X Y
Site               = JNM-ECA800
Spectrometer       = DELTA2 NMR

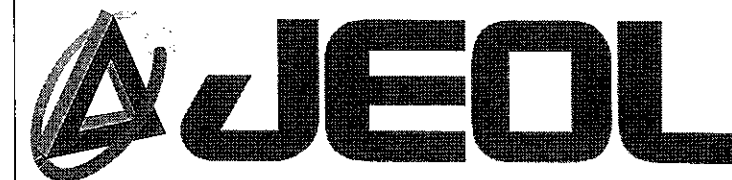
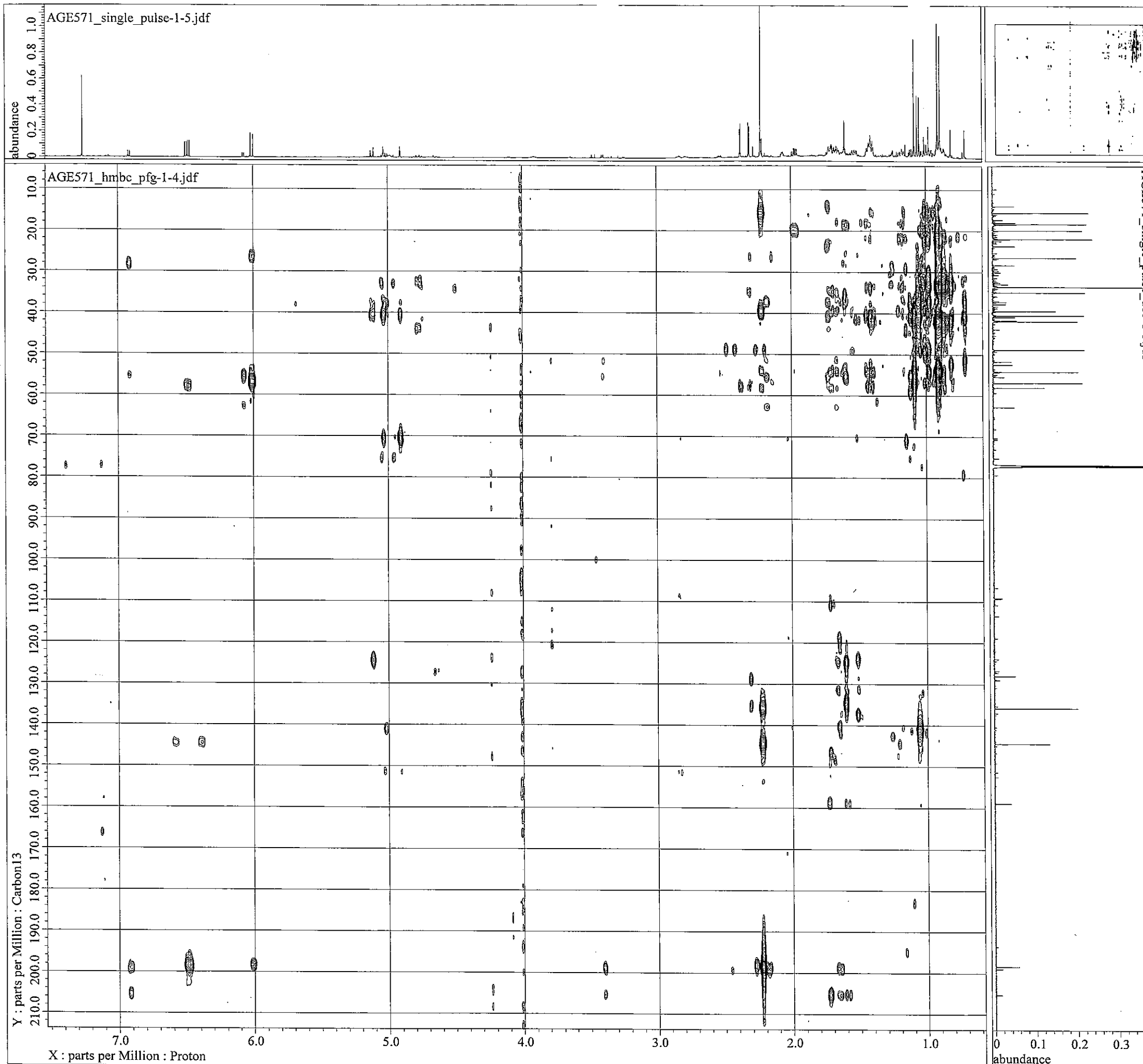
```

```
Field Strength      = 18.79305132[T] (800[MHz])
X Acq Duration      = 0.12787712[s]
X Domain            = 1H
X Freq              = 800.14000039[MHz]
X Offset            = 4[ppm]
X Points            = 1024
X Prescans          = 4
X Resolution         = 7.82000721[Hz]
X Sweep             = 8.00768738[kHz]
X Sweep_Clippped    = 6.4061499[kHz]
Y Domain            = 13C
Y Freq              = 201.19534229[MHz]
Y Offset            = 105.0 [ppm]
Y Points            = 256
Y Prescans          = 0
Y Resolution         = 165.23900169[Hz]
Y Sweep             = 42.30118443[kHz]
Tri_Domain          = Proton
Tri_Freq            = 800.14000039[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total Scans         = 2048
```

```

Relaxation_Delay = 1.5[s]
Recvr Gain       = 34
Temp_Get         = 20.8[dC]
X_Acq_Time       = 0.12787712[s]
X_Atn            = 3[dB]
X_Pulse          = 16[us]
Y_Acq_Time       = 6.05184[ms]
Y_Atn            = 2.6[dB]
Y_P1_Correction  = 720
Y_Pulse          = 11.2[us]
Irr_Atn_Dec      = 20.985[dB]
Irr_Noise        = MPF10
Irr_Pwidth       = 93[us]
Tri_Mode         = Off
Composite_Pulse   = FALSE
Dante_Preset     = FALSE
Decoupling       = TRUE
Grad_1_Amp       = 0.105[T/m]
Grad_2_Amp       = 30[mT/m]
Initial_Wait     = 1[s]
J_Constant       = 140[Hz]
Phase_Qd         = ({0, 180}, {90, 270})
Purge            = 0[ms]
Repetition_Time  = 1.62787712[s]
T1               = 0.472[us]
T1_Cal1          = -17.258[us]
T1_Cal2          = -11.348[us]
T1_Cal3          = -5.438[us]
T1_Cal4          = 0.472[us]
T1_Cal5          = 6.382[us]

```



----- PROCESSING PARAMETERS -----  
gauss( 5.0[Hz], 0.0[s] )  
sinbell auto  
zerofill( 1 )  
fft( 1, TRUE, TRUE )  
ppm  
[transpose]  
sinbell4( -60, 160 )  
trapezoid( 0[%], 5[%], 80[%], 100[%] )  
zerofill( 2 )  
fft( 1, TRUE, TRUE )  
ppm  
abs  
[transpose]

Filename = C:\Users\delta\Documents\J  
Author = delta  
Experiment = hmhc\_pfg.jxp  
Sample Id = AGE571  
Solvent = CHLOROFORM-D  
Creation Time = 16-NOV-2012 00:41:09  
Revision Time = 16-NOV-2012 10:58:53  
Current Time = 16-NOV-2012 11:00:04

Comment = gradient enhanced HMQC  
Data Format = 2D REAL REAL  
Dim Size = 512, 1638  
Dim Title = Carbon13 Proton  
Dim Units = [ppm] [ppm]  
Dimensions = X Y  
Site = JNM-ECA800  
Spectrometer = DELTA2\_NMR

Field Strength = 18.79305132[T] (800[MHz])  
X Acq Duration = 0.25575424[s]  
X Domain = 1H  
X Freq = 800.14000039[MHz]  
X Offset = 4[ppm]  
X Points = 2048  
X Prescans = 4  
X Resolution = 3.9100036[Hz]  
X Sweep = 8.00768738[kHz]  
X Sweep\_Clippped = 6.4061499[kHz]  
Y Domain = 13C  
Y Freq = 201.19534229[MHz]  
Y Offset = 100[ppm]  
Y Points = 256  
Y Prescans = 0  
Y Resolution = 196.49144869[Hz]  
Y Sweep = 50.30181087[kHz]  
Tri Domain = Proton  
Tri Freq = 800.14000039[MHz]  
Tri Offset = 5[ppm]  
Clipped = FALSE  
Scans = 32  
Total\_Scans = 8192

Relaxation\_Delay = 1.5[s]  
Recvr Gain = 34  
Temp\_Get = 20.7[dc]  
X Acq Time = 0.25575424[s]  
X Atn = 3[dB]  
X Pulse = 16[us]  
Y Acq Time = 5.08928[ms]  
Y Atn = 2.6[dB]  
Y Pulse = 11.2[us]  
Tri Mode = Off  
Dante\_Presat = FALSE  
Delta = 62.5[ms]  
Delta\_1 = 3.83288616[ms]  
Delta\_2 = 3.43642612[ms]  
Delta\_3 = 3.11429461[ms]  
Grad\_1 = 1[ms]  
Grad\_1\_Amp = 0.18[T/m]  
Grad\_2 = 1[ms]  
Grad\_2\_Amp = 0.18[T/m]  
Grad\_3 = 1[ms]  
Grad\_3\_Amp = 90.54325956[mT/m]  
Grad\_Lpf = 1[ms]  
Grad\_Lpf1\_Amp = 45[mT/m]  
Grad\_Lpf2\_Amp = -25.71428571[mT/m]  
Grad\_Lpf3\_Amp = -12.85714286[mT/m]  
Grad\_Lpf4\_Amp = -6.42857143[mT/m]  
Grad\_Recover = 0.1[ms]  
Grad\_Selection = 13C = 1.988:1.988:1  
Grad\_Shape = SINE  
Initial\_Wait = 1[s]

abundance

