

Electronic Supplementary Information for:

Control of molecular packing of chloroboron(III) and fluoroboron(III) subnaphthalocyanines by designing peripheral substituents

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----- PROCESSING PARAMETERS -----
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sweep : 0.2 [Hz] : 0.0 [s]
trapzoids : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

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Experiment = single_pulse.ex2
Sample_id = #415683
Solvent = CHLOROFORM-D
Creation_time = 5-OCT-2016 10:13:09
Revision_time = 7-JUL-2017 18:54:42
Current_time = 7-JUL-2017 18:55:04

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Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579 [G] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [kHz]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [dB]
X_pulse = 3.12 [us]
Irr_mode = Off
Irr_pulse = Off
Dante_preset = FALSE
Initial_wait = 1 [s]
Recvr_gain = 56
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 20.3 [dcl]

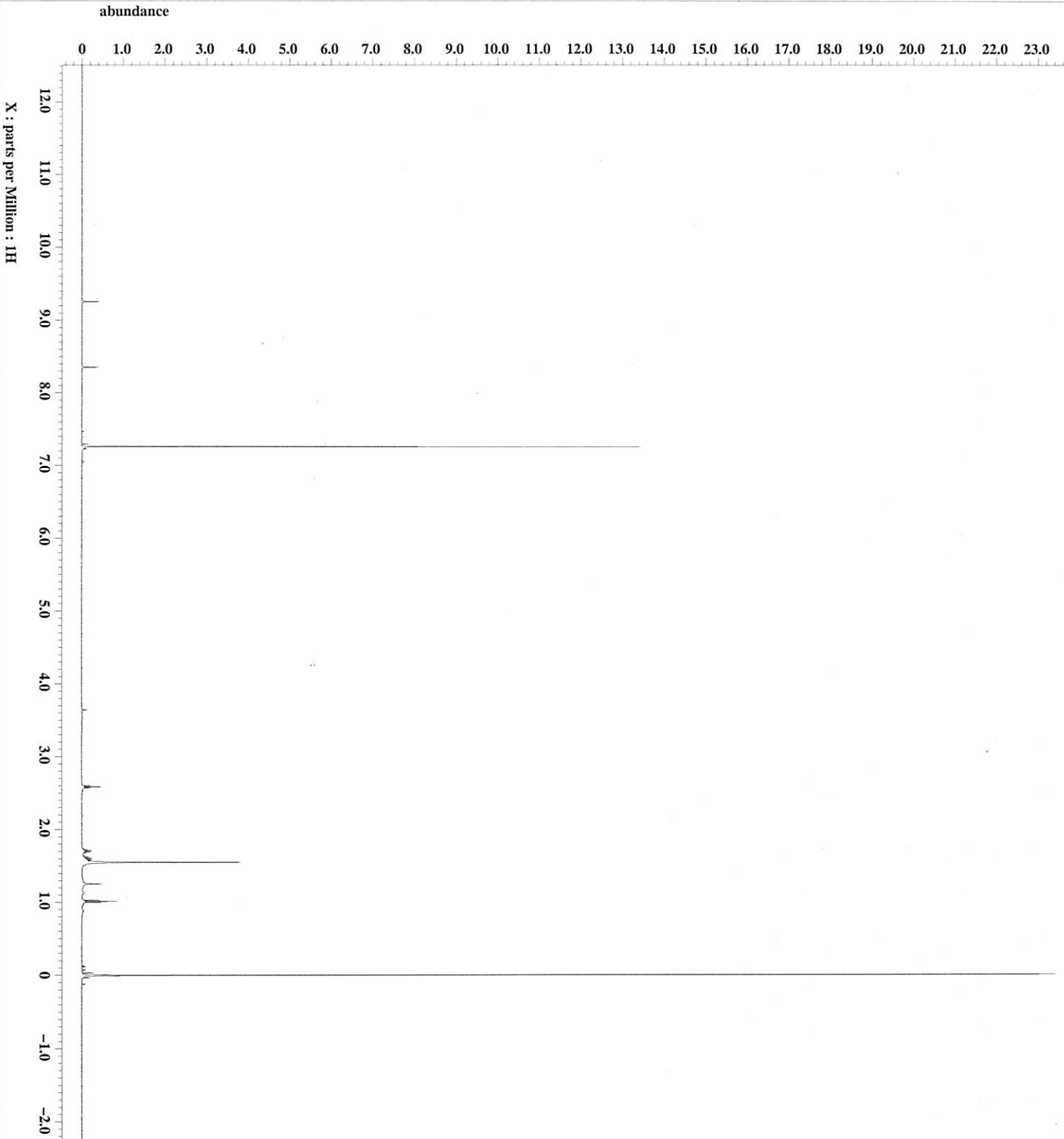


Figure S1. ¹H NMR of 1a.



----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
sweep : 0.2 [Hz] : 0.0 [s]
c1apezoid3 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
ift : 1 : TRUE : TRUE
machinephase
ppm

Filename = 20161005hekisin-2.fdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#415683
Solvent = CHLOROFORM-D
Creation_time = 5-OCT-2016 10:13:09
Revision_time = 7-JUL-2017 18:54:42
Current_time = 7-JUL-2017 18:55:19

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579 [T] (500 [MHZ])
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.01 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [kHz]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.01 [ppm]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.01 [ppm]
Mod_return = FALSE
Scans = 1
Recal_scans = 8

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [dB]
X_pulse = 3.12 [us]
Irr_mode = OET
Irr_mode = Off
Dante_presat = FALSE
Initial_wait = 1 [s]
Recvr_gain = 56
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 20.3 [dcl]

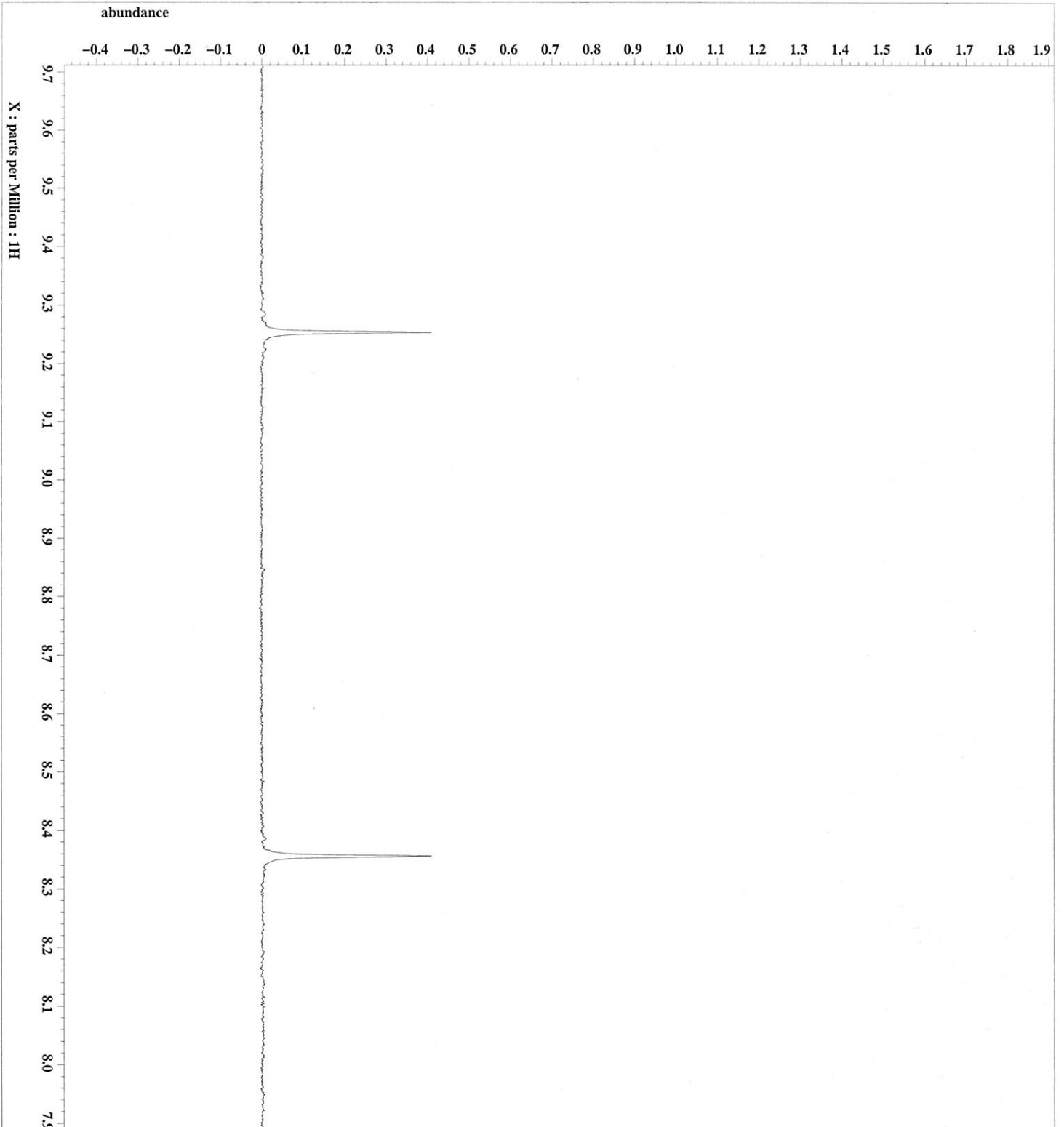


Figure S1. ¹H NMR of **1a** (Low-field region).

```

---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sweep : 0.2 [Hz] : 0.0 [s]
cpdphase3 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
  
```

```

Filename = 20161005hekisin-2.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#415683
Solvent = CHLOROFORM-D
Creation_time = 5-OCT-2016 10:13:09
Revision_time = 7-JUL-2017 18:54:42
Current_time = 7-JUL-2017 18:55:43
  
```

```

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500
  
```

```

Field_strength = 11.7473579 [T] (500 [MH])
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 1.5727737 [Hz]
X_sweep = 9.38438438 [kHz]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Irr_domain = 1H
Tri_freq = 500.15991521 [MHz]
Tri_offset = 5.0 [ppm]
Clipped = FALSE
Mod_return = 1
Scans = 1
Total_scans = 8
  
```

```

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [dB]
X_pulse = 3.12 [us]
Irr_mode = OTE
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1 [s]
Recvr_gain = 56
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 20.3 [dc]
  
```

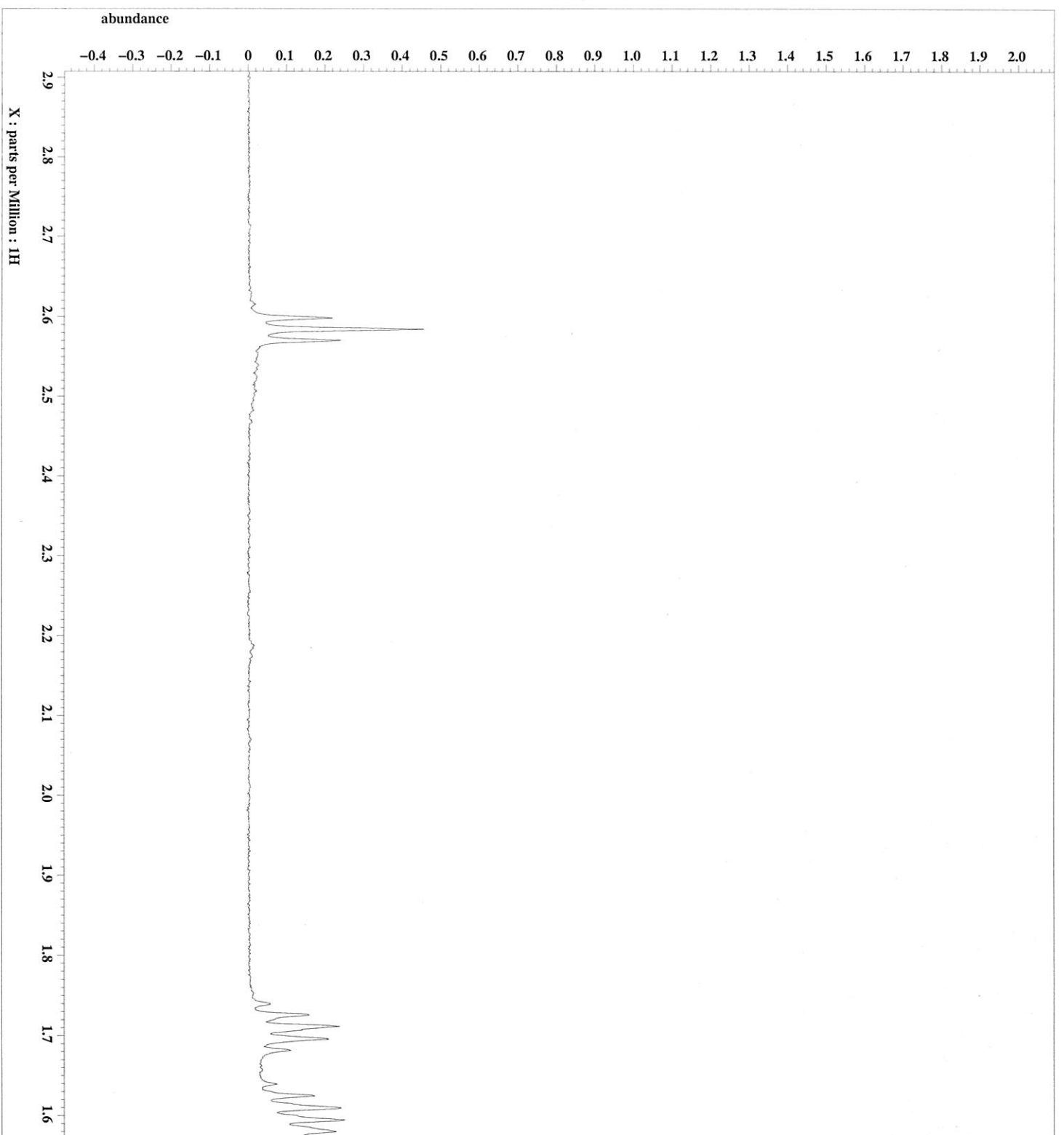


Figure S1. ¹H NMR of **1a** (Up-field region).

```

---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
semp : 0.2 [Hz] : 0.0 [s]
trepzoid3 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
  
```

```

Filename = 20161005hekisin-2.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = SH415683
Solvent = CHLOROFORM-D
Creation_time = 5-OCT-2016 10:13:09
Revision_time = 7-JUL-2017 18:54:42
Current_time = 7-JUL-2017 18:56:19
  
```

```

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500
  
```

```

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [kHz]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Irr_domain = 1H
Tri_domain = 1H
Tri_freq = 500.15991521 [MHz]
Tri_offset = 5.0 [ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
  
```

```

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [dB]
X_pulse = 3.12 [us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1 [s]
Recvr_gain = 56
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 20.3 [dC]
  
```

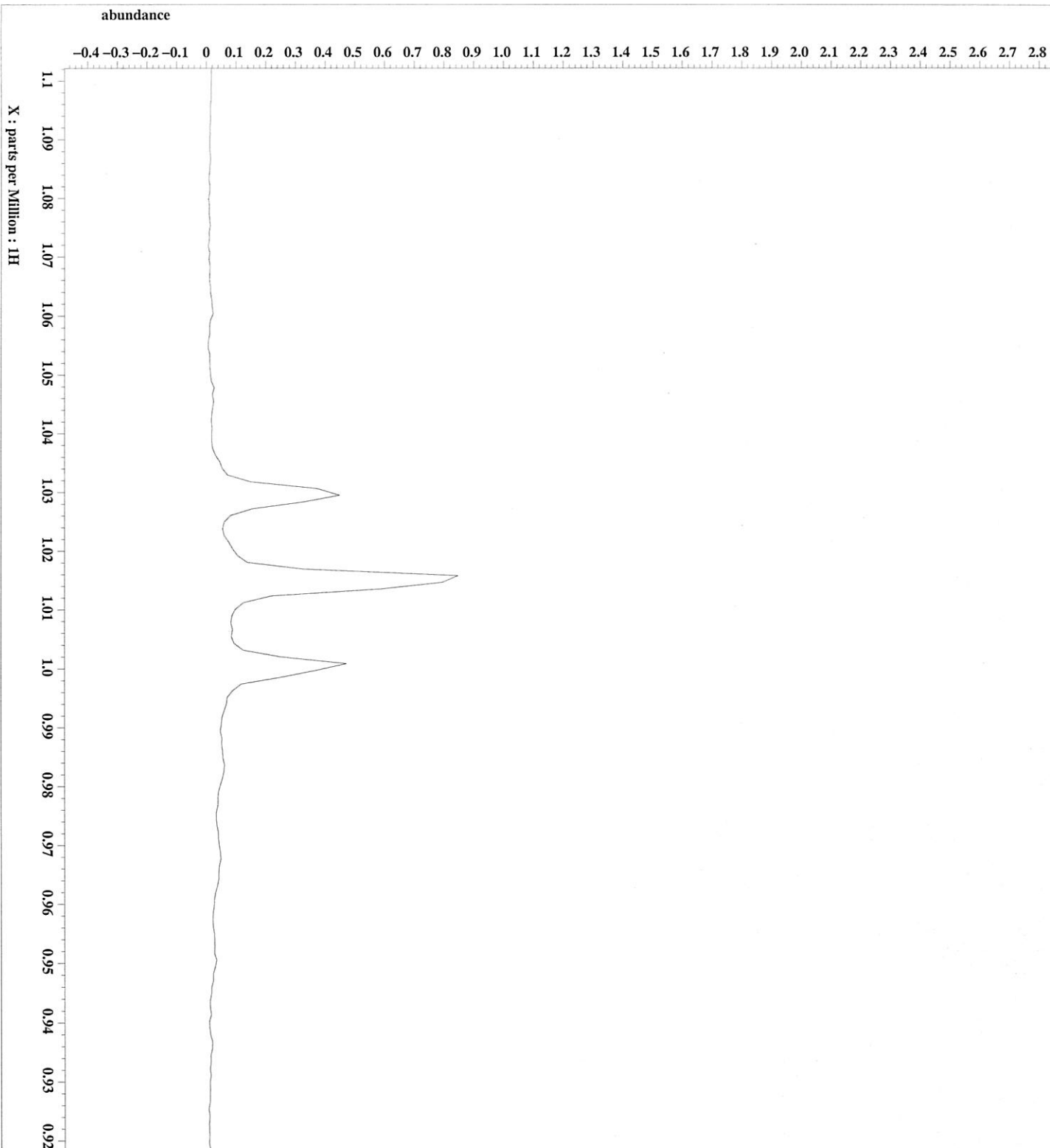
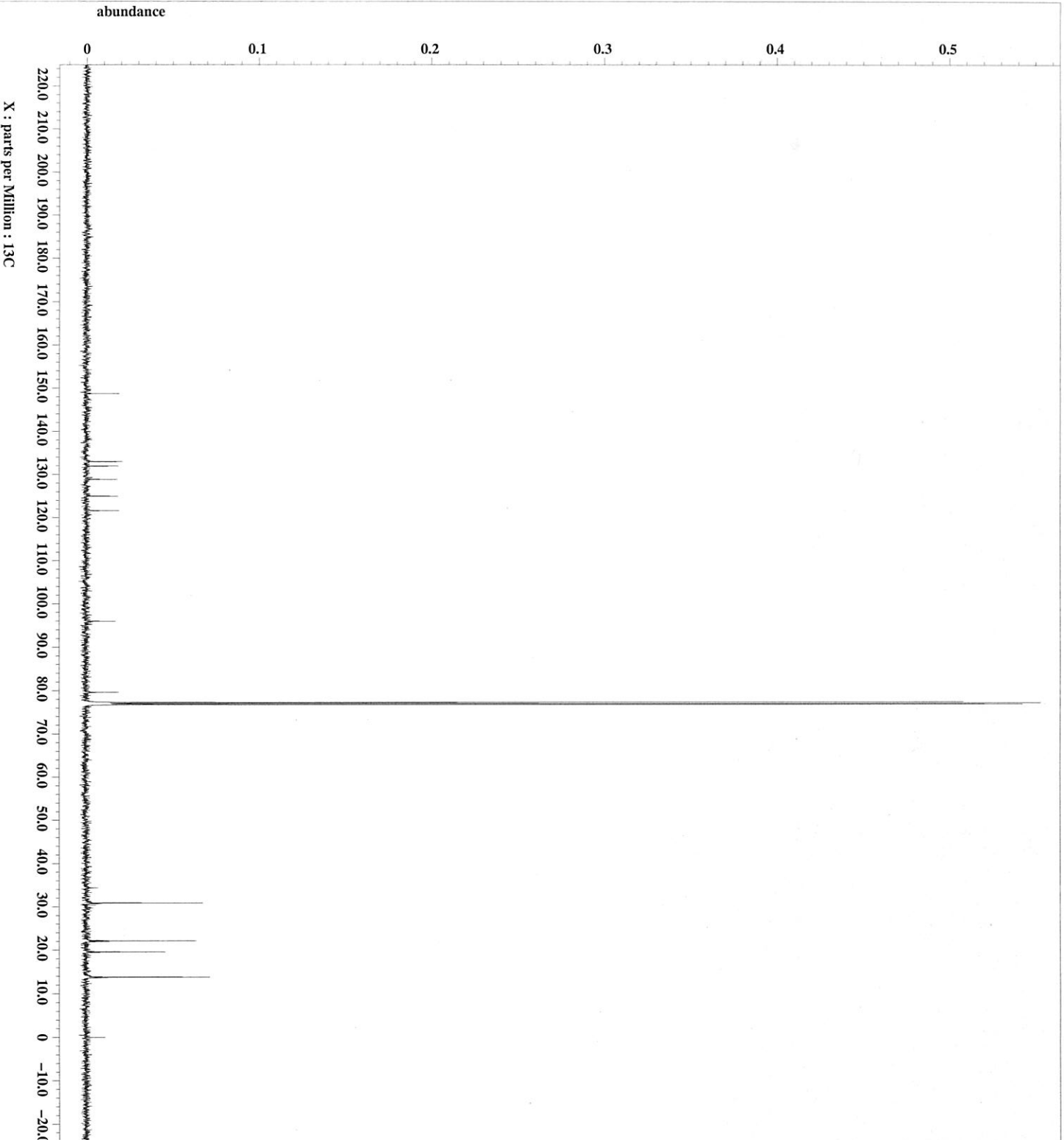


Figure S1. ¹H NMR of **1a** (Low-field region 2).



----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
semp : 2.0 [Hz] : 0.0 [s]
trapzfold3 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
ift : 1 : TRUE : TRUE
machinephase
ppm



Filename = c4h9-cl13c-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#590775
Solvent = CHLOROFORM-D
Creation_time = 24-MAY-2017 15:18:41
Revision_time = 7-JUL-2017 18:57:06
Current_time = 7-JUL-2017 18:57:15
Comment = single pulse decouple
Data_format = 1D COMPLEX
Data_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_prescan_resolution = 1.19959034 [Hz]
X_sweep_resolution = 39.3081761 [kHz]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Clipped = FALSE
Mod_return = 1
Scans = 800
Total_scans = 800
X_90_width = 10.87 [us]
X_acq_time = 0.83361792 [s]
X_angle = 30 [deg]
X_atn = 6.5 [dB]
X_pulse = 3.62333333 [us]
Irr_atn_dec = 26.772 [dB]
Irr_atn_noe = 26.772 [dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1 [s]
Noe_time = TRUE
Noe = 2 [s]
Recvr_gain = 52
Relaxation_delay = 2 [s]
Repetition_time = 2.83361792 [s]
Temp_get = 21 [dC]

Figure S2. ¹³C NMR of 1a.



----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
secp : 0.2 [Hz] : 0.0 [s]
trapzoid3 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename = 20161005tetra-2.fdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#407175
Solvent = CHLOROFORM-D
Creation_time = 5-OCT-2016 10:00:18
Revision_time = 7-JUL-2017 18:58:52
Current_time = 7-JUL-2017 19:00:07

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Spectrometer = ECA500
Site = JNM-ECA500

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [Hz]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Irr_domain = 1H
Tri_freq = 500.15991521 [MHz]
Tri_offset = 5.0 [ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [dB]
X_pulse = 3.12 [us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1 [s]
Recvr_gain = 58
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_set = 20.2 [dC]

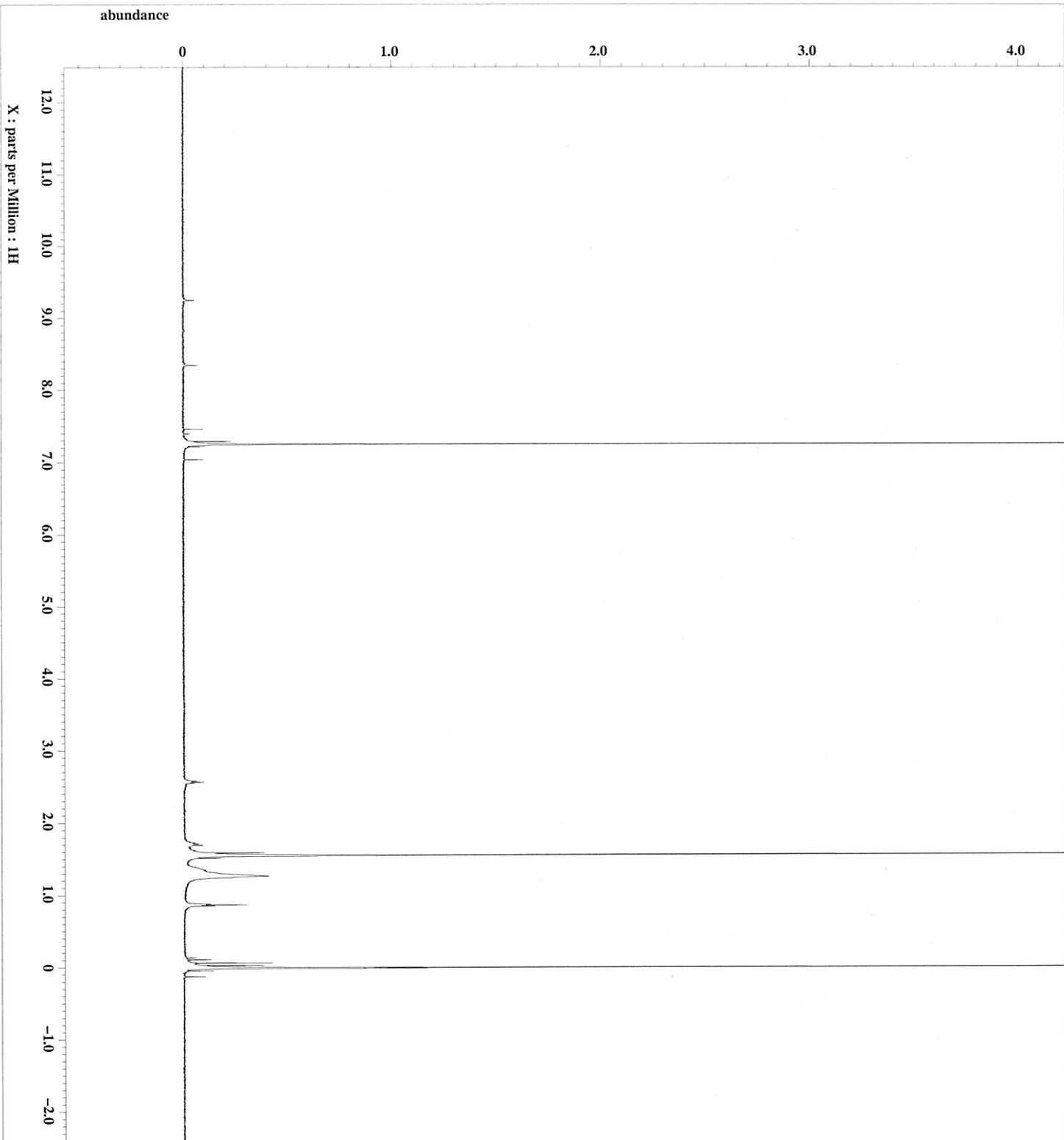


Figure S3. ¹H NMR of **1b**.


```

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

```

Filename = 20161005tetra-2.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#407175
Solvent = CHLOROFORM-D
Creation_time = 5-OCT-2016 10:00:18
Revision_time = 7-JUL-2017 18:58:52
Current_time = 7-JUL-2017 19:00:21
  
```

```

Comment = single_pulse
Data_format = ID COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500
  
```

```

Field_strength = 11.747379 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [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 = 8
Total_scans = 8
  
```

```

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [dB]
X_pulse = 3.12 [us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1 [s]
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 20.2 [dc]
  
```

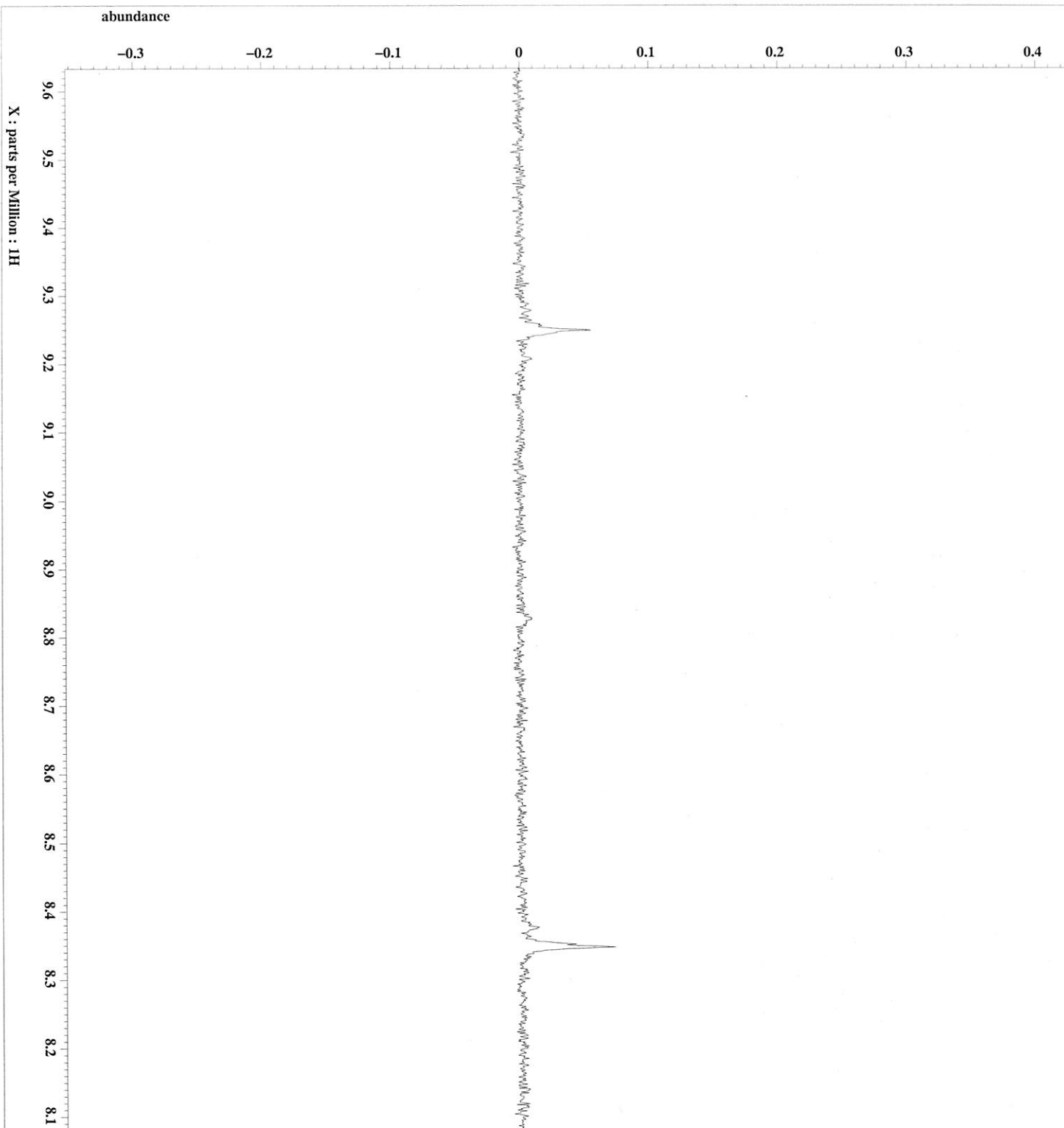


Figure S3. ¹H NMR of **1b** (Low-field region).



```

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

```

Filename = 20161005tetra-2.fdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#407175
Solvent = CHLOROFORM-D
Creation time = 5-OCT-2016 10:00:18
Revision time = 7-OUL-2017 18:58:52
Current_time = 7-OUL-2017 19:00:29
  
```

```

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Spectrometer = ECA500
Site = JNM-ECA500
  
```

```

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_preampl = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [kHz]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Mod_return = FALSE
Scans = 1
Total_scans = 8
X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [db]
X_pulse = 3.12 [us]
Irr_mode = Off
Irr_mode = FALSE
Dante_presat = 58
Initial_wait = 1 [s]
Recvr_gain = 5 [s]
Relaxation_delay = 6.74587904 [s]
Repetition_time = 20.2 [dc]
Temp_get
  
```

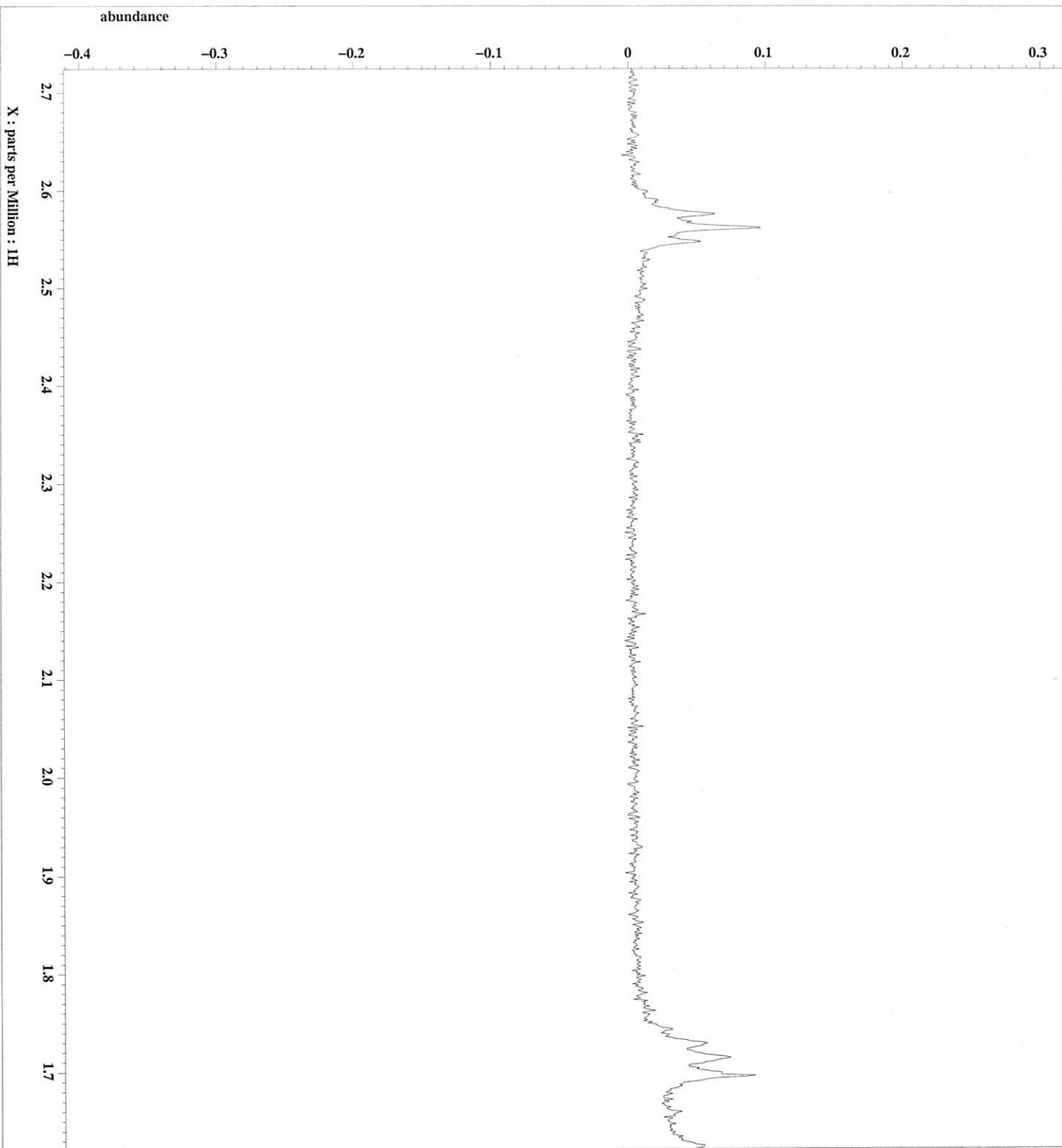


Figure S3. ¹H NMR of **1b** (Up-field region).

```

---- PROCESSING PARAMETERS ----
dc balance : 0 : FALSE
smp : 0.2[Hz] : 0.0[ls]
trapzoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
  
```

```

Filename = 20161005tetra-2.fdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#407175
Solvent = CHLOROFORM-D
Creation_time = 5-OCT-2016 10:00:18
Revision_time = 7-JUL-2017 18:58:52
Current_time = 7-JUL-2017 19:00:42
  
```

```

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500
  
```

```

Field_strength = 11.7473579 [T] (500 [MH]
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [kHz]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Irr_domain = 1H
Tri_freq = 500.15991521 [MHz]
Tri_offset = 5.0 [ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
  
```

```

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [db]
X_pulse = 3.12 [us]
Irr_mode = Off
Tri_mode = FALSE
Dante_presat = 1 [ls]
Recvr_gain = 58
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 20.2 [dC]
  
```

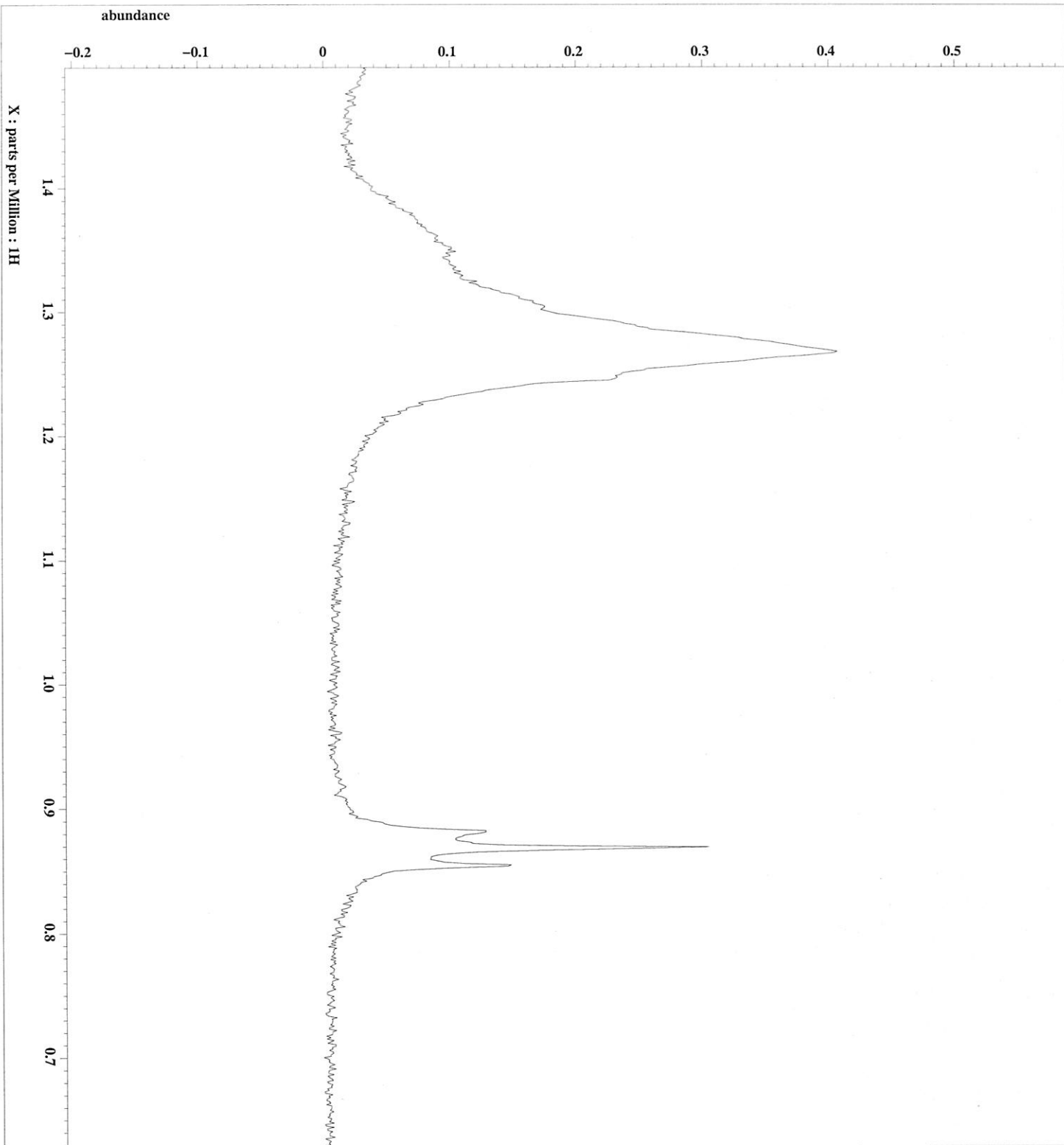


Figure S3. ¹H NMR of **1b** (Up-field region 2).



```

---- PROCESSING PARAMETERS ----
dc balance : 0 : FALSE
smp : 2.0 [Hz] : 0.0 [s]
trapzoid3 : 0 [%] : 80 [%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
  
```

```

Filename = c12h25-c113c-2.jdf
Author =
Experiment = single_pulse_dec
Sample_id = S#747930
Solvent = CHLOROFORM-D
Creation_time = 2-AUG-2016 08:08:26
Revision_time = 7-JUL-2017 19:02:07
Current_time = 7-JUL-2017 19:02:33
  
```

```

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 = 16000
Total_scans = 16000
  
```

```

X_90_width = 10.87 [us]
X_acq_time = 0.83361792 [s]
X_angle = 30 [deg]
X_atn = 6.5 [db]
X_pulse = 3.62333333 [us]
Irr_atn_dec = 26.772 [db]
Irr_atn_noe = 26.772 [db]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1 [s]
Noe_time = TRUE
Noe_gain = 2 [s]
Relaxation_delay = 50
Relaxation_delay = 2 [s]
Repetition_time = 2.83361792 [s]
Temp_set = 21.5 [dC]
  
```

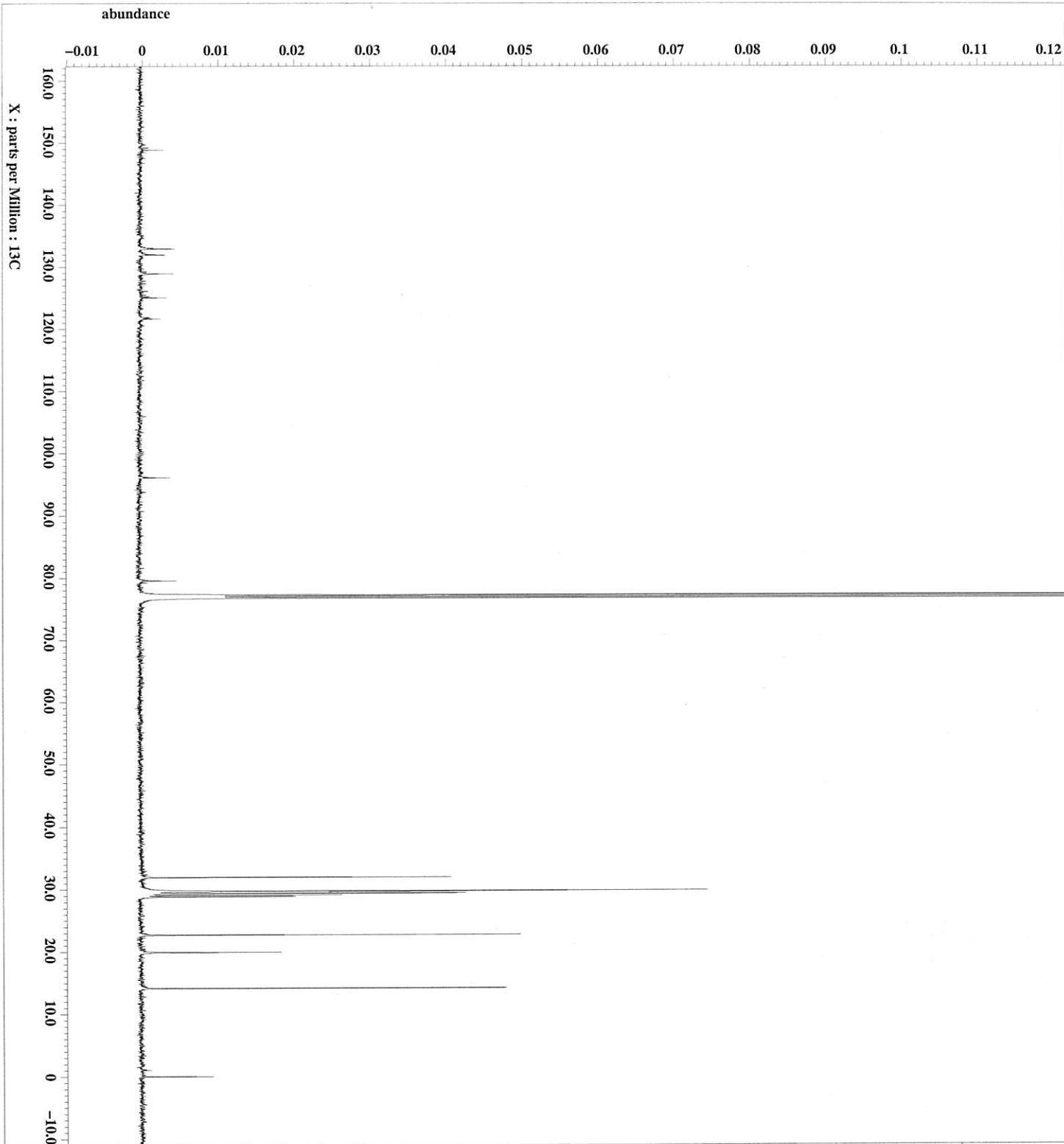


Figure S4. ¹³C NMR of 1b.

```

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

```

Filename = c12h25-c113c-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#747930
Solvent = CHLOROFORM-D
Creation_time = 2-AUG-2016 08:08:26
Revision_time = 7-JUL-2017 19:02:07
Current_time = 7-JUL-2017 19:03:11
  
```

```

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 = 16000
Total_scans = 16000
  
```

```

X_90_width = 10.87 [us]
X_acq_time = 0.83361792 [s]
X_angle = 30 [deg]
X_atn = 6.5 [db]
X_pulse = 3.62333333 [us]
Irr_atn_dec = 26.772 [db]
Irr_atn_noe = 26.772 [db]
WALTZ = TRUE
Decoupling = TRUE
Initial_wait = 1 [s]
Noe = TRUE
Noe_time = 2 [s]
Recvr_gain = 50
Relaxation_delay = 2 [s]
Repetition_time = 2.83361792 [s]
Temp_get = 21.5 [dC]
  
```

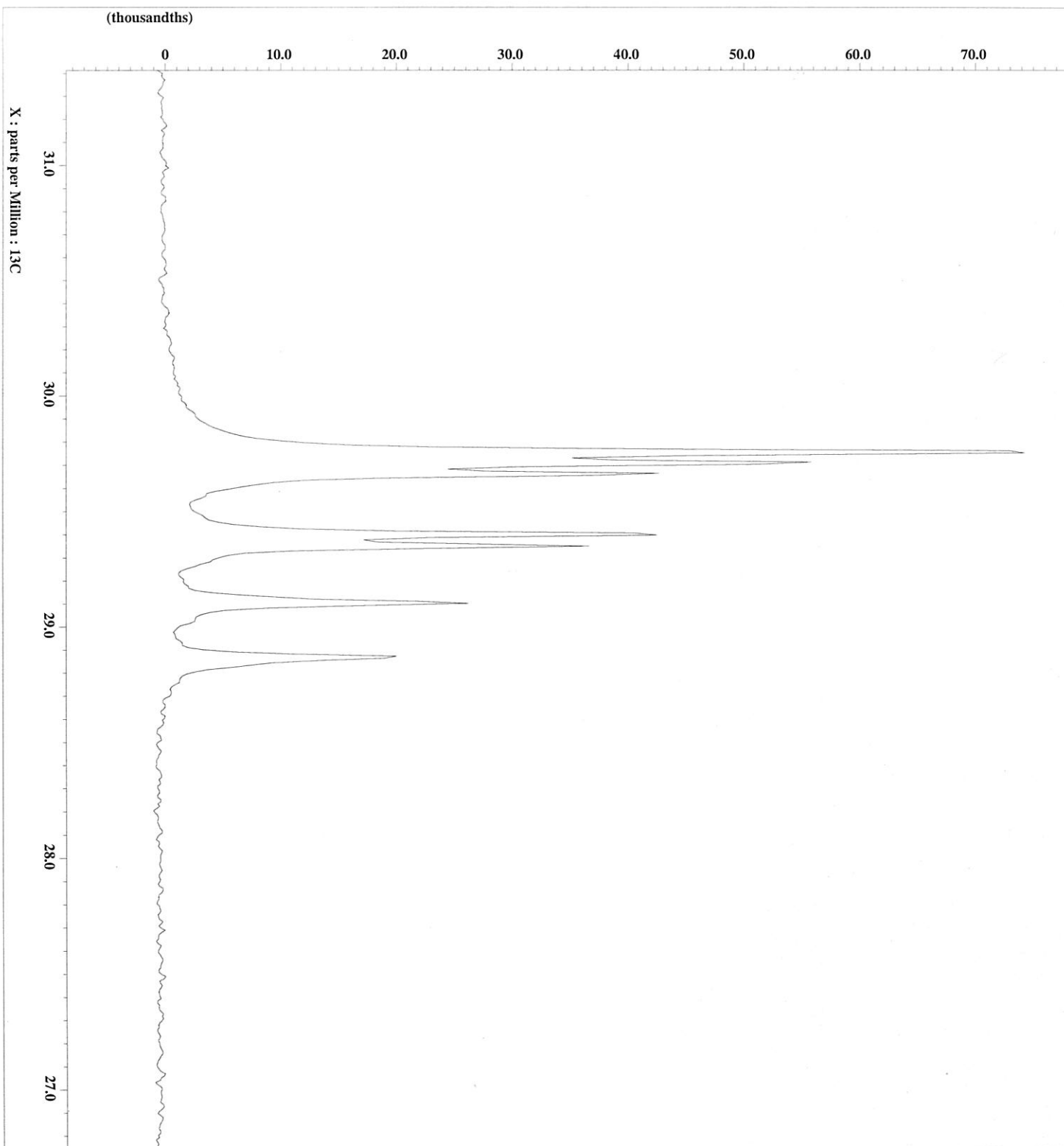


Figure S4. ¹³C NMR of **1b** (Up-field region).



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

Filename = 20161003pna-2.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#804101
Solvent = CHLOROPFORM-D
Creation_time = 3-OCT-2016 21:00:08
Revision_time = 7-JUL-2017 19:04:04
Current_time = 7-JUL-2017 19:04:19

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.384384381 [kHz]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Mod_return = FALSE
Scans = 1
Total_scans = 8

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [dB]
X_pulse = 3.12 [us]
Irr_mode = Off
Irr_offset = Off
Dante_preset = FALSE
Initial_wait = 1 [s]
Recvr_gain = 50
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 21 [C]

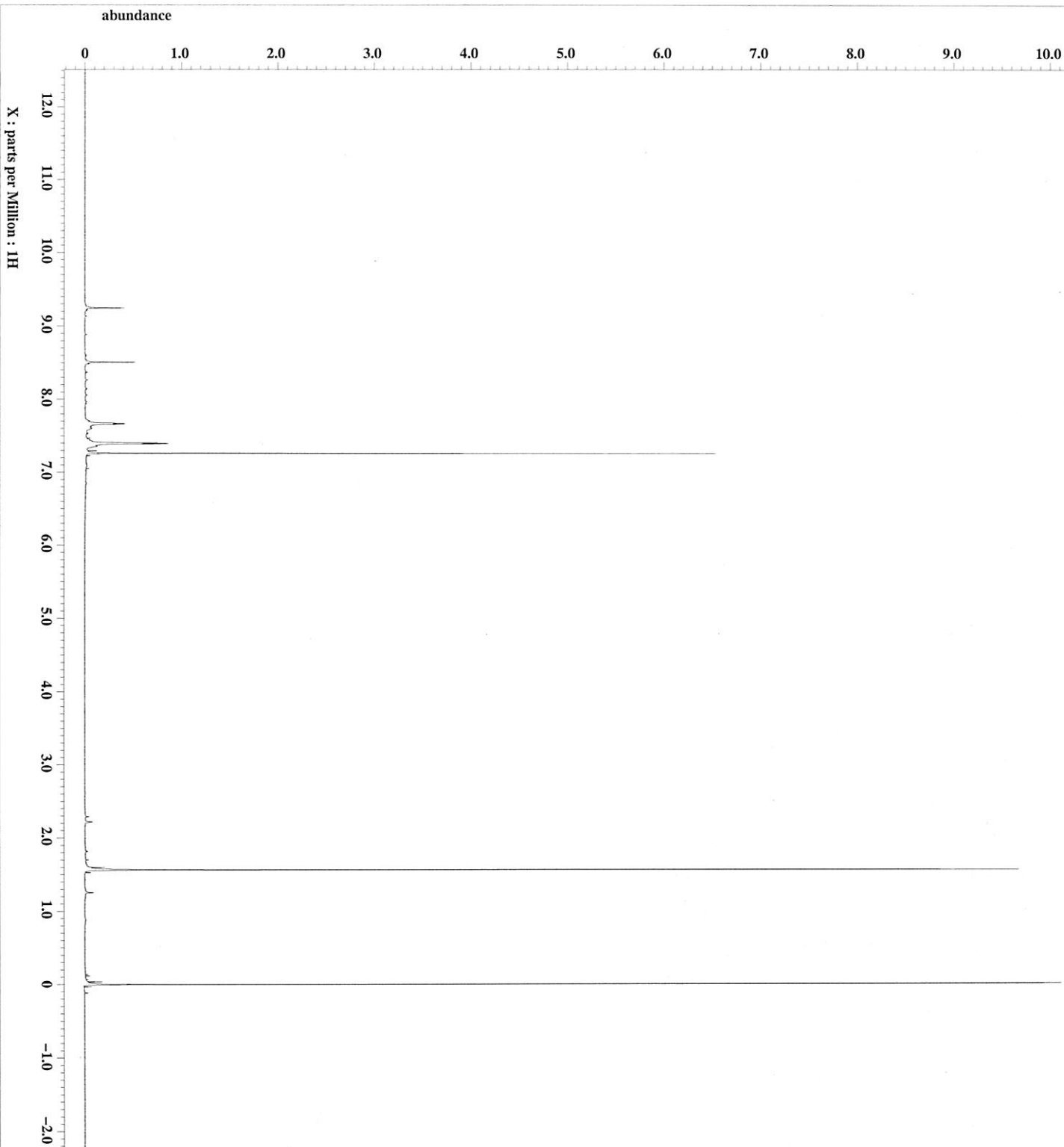


Figure S5. ¹H NMR of 1c.

```

---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2 [Hz] : 0.0 [s]
trapzoid3 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinphase
ppm
  
```

```

Filename = 20170110c3h9ph-cl-2.f
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#592194
Solvent = CHLOROFORM-D
Creation_time = 10-JAN-2017 14:57:58
Revision_time = 7-JUL-2017 20:00:35
Current_time = 7-JUL-2017 20:00:48
  
```

```

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Spectrometer = ECA500
Site = JNM-ECA500
  
```

```

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [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 = 8
Total_scans = 8
  
```

```

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [dB]
X_pulse = 3.12 [us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1 [s]
Recvr_gain = 56
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 20.4 [dC]
  
```

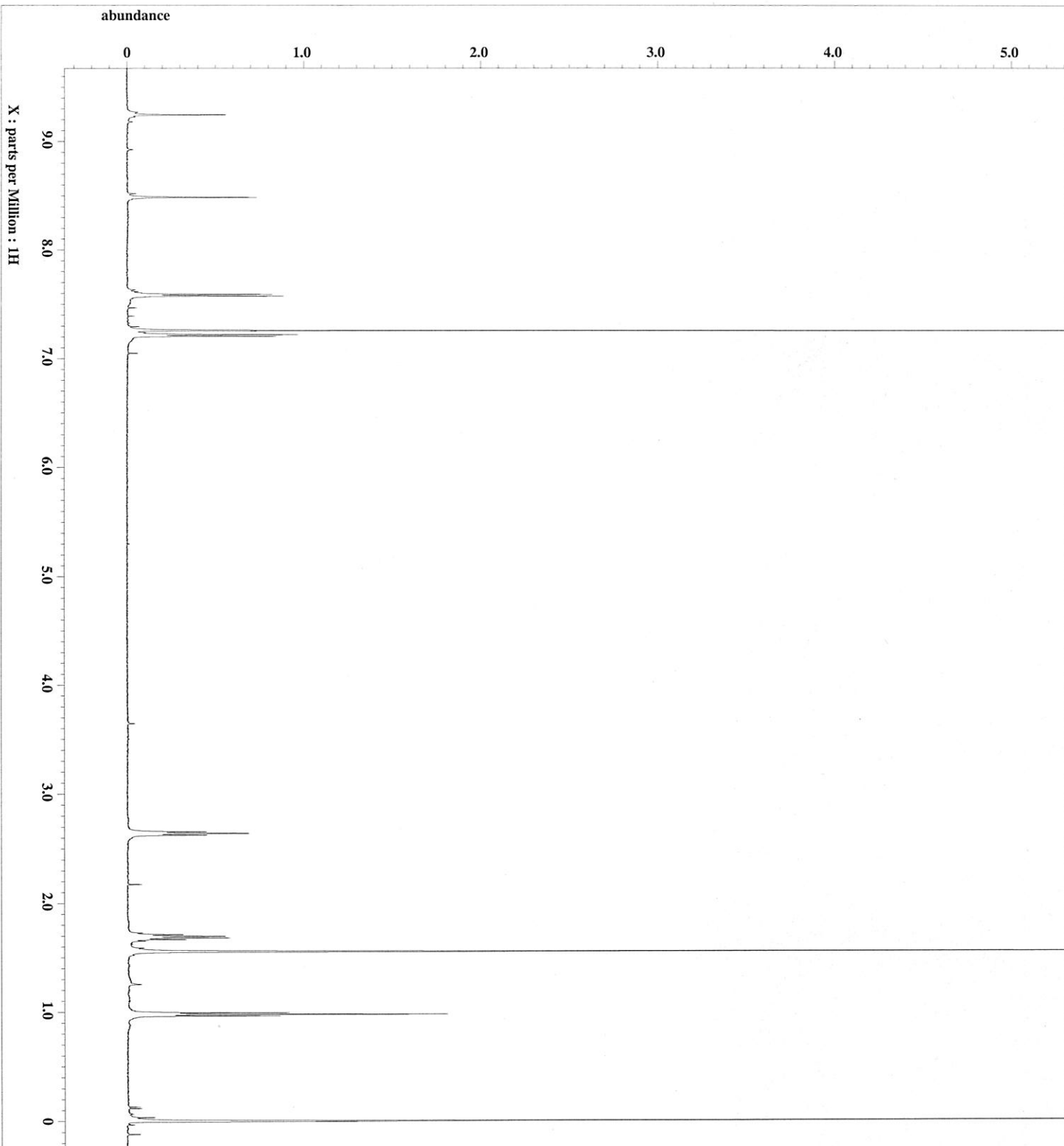


Figure S6. ¹H NMR of **1d**.

```

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

```

Filename = 20170524c6h13ph-cl-2.
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#734695
Solvent = CHLOROFORM-D
Creation_time = 24-MAY-2017 18:42:52
Revision_time = 7-JUL-2017 19:24:41
Current_time = 7-JUL-2017 19:26:09
  
```

```

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Sitr = ECA500
Sitr2 = JNM-ECA500
  
```

```

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.3843838 [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 = 8
Total_scans = 8
  
```

```

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [db]
X_pulse = 3.12 [us]
Irr_mode = Off
Tri_mode = Off
Dance_preset = FALSE
Initial_wait = 1 [s]
Recvr_gain = 56
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 21 [dC]
  
```

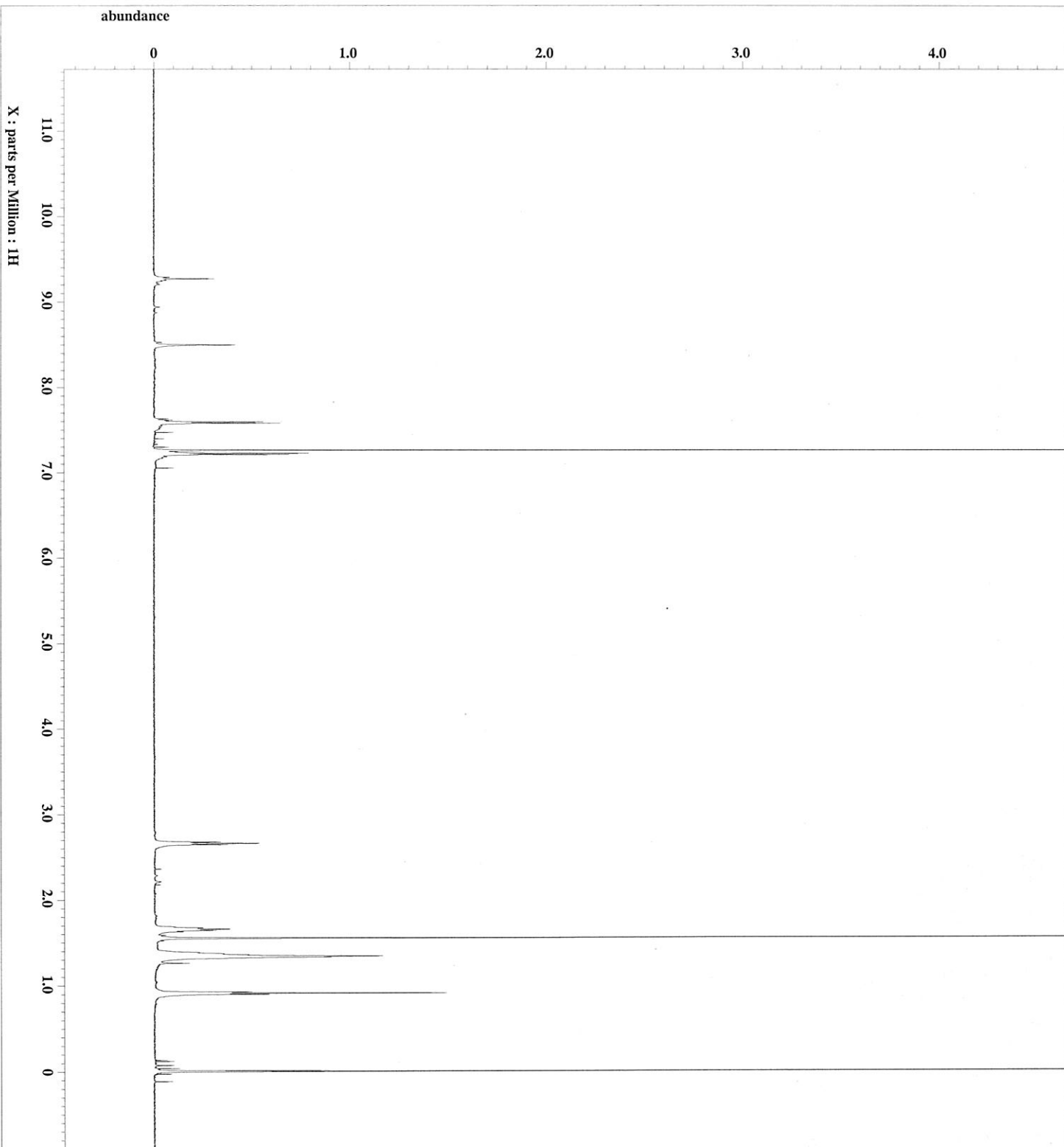


Figure S7. ¹H NMR of **1e**.


```

---- PROCESSING PARAMETERS ----
dc balance : 0 : FALSE
semp : 0.2[Hz] : 0.0[ls]
trapzoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
  
```

```

Filename = 20170524c6h13ph-cl-2.
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#734655
Solvent = CHLOROFORM-D
Creation_time = 24-MAY-2017 18:42:52
Revision_time = 7-JUL-2017 19:24:41
Current_time = 7-JUL-2017 19:26:58
  
```

```

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Spectrometer = ECA500
Site = JNM-ECA500
  
```

```

Field_strength = 11.747379[T] (500[MH
X_acq_duration = 1.74587904[ls]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57227737[Hz]
X_sweep = 9.38438438[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 = 8
Total_scans = 8
  
```

```

X_90_width = 6.24[us]
X_acq_time = 1.74587904[ls]
X_angle = 45[deg]
X_atn = 3.4[db]
X_pulse = 3.12[us]
Irr_mode = Off
Tri_mode = Off
Dance_preset = FALSE
Initial_wait = 1[ls]
Recvr_gain = 56
Relaxation_delay = 5[ls]
Repetition_time = 6.74587904[ls]
Temp_get = 21[dc]
  
```

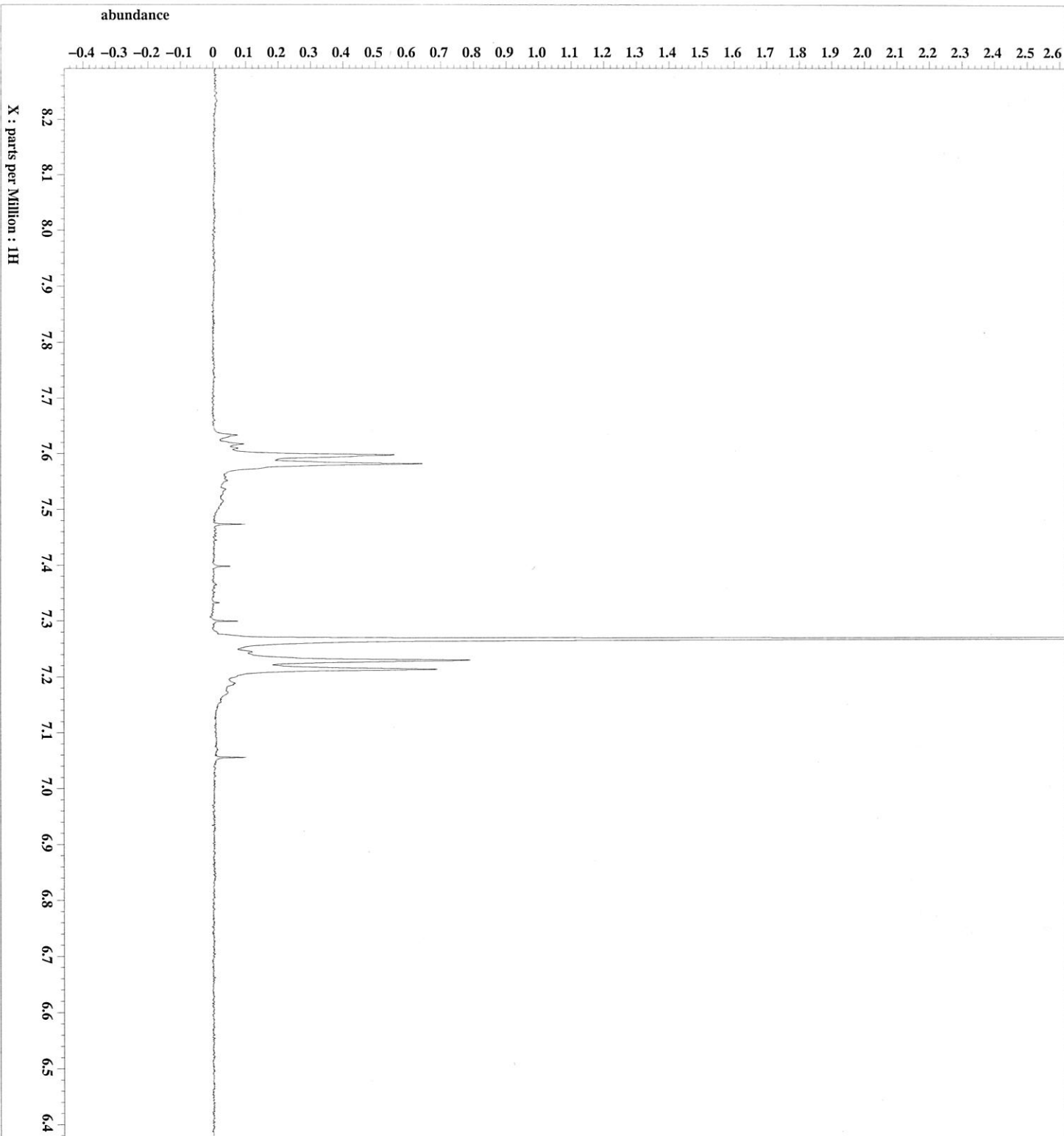


Figure S7. ¹H NMR of **1e** (Low-field region).



----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
sweep : 0.2 [Hz] : 0.0 [s]
trapzoid3 : 0 [%] : 80 [%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename = 20170524c6h13ph-cl-2.
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#734655
Solvent = CHLOROFORM-D
Creation_time = 24-MAY-2017 18:42:52
Revision_time = 7-JUL-2017 19:24:41
Current_time = 7-JUL-2017 19:27:10

Comment = single_pulse
Data_format = ID COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [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 = 8
Total_scans = 8

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [dB]
X_pulse = 3.12 [us]
Tri_mode = Off
Tri_offset = Off
Dante_presat = FALSE
Initial_wait = 1 [s]
Recvr_gain = 56
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 21 [C]



Figure S7. ¹H NMR of **1e** (Up-field region).

```

----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
semp : 0.2 [Hz] : 0.0 [s]
trapzoid3 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
ite : 1 : TRUE : TRUE
machinephase
ppm
  
```

```

Filename = 20161207CGH130Ph-Cl1-
Author =
Experiment = single_pulse.ex2
Sample_id = S#610866
Solvent = CHLOROFORM-D
Creation time = 7-DEC-2016 15:33:02
Revision time = 7-JUL-2017 19:28:42
Current_time = 7-JUL-2017 19:33:00
  
```

```

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500
  
```

```

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_preampl = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [kHz]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
  
```

```

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [dB]
X_pulse = 3.13 [us]
Irr_mode = Off
Irr_mode = FALSE
Dante_presat = 1 [s]
Recvr_gain = 58
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 20.1 [dC]
  
```

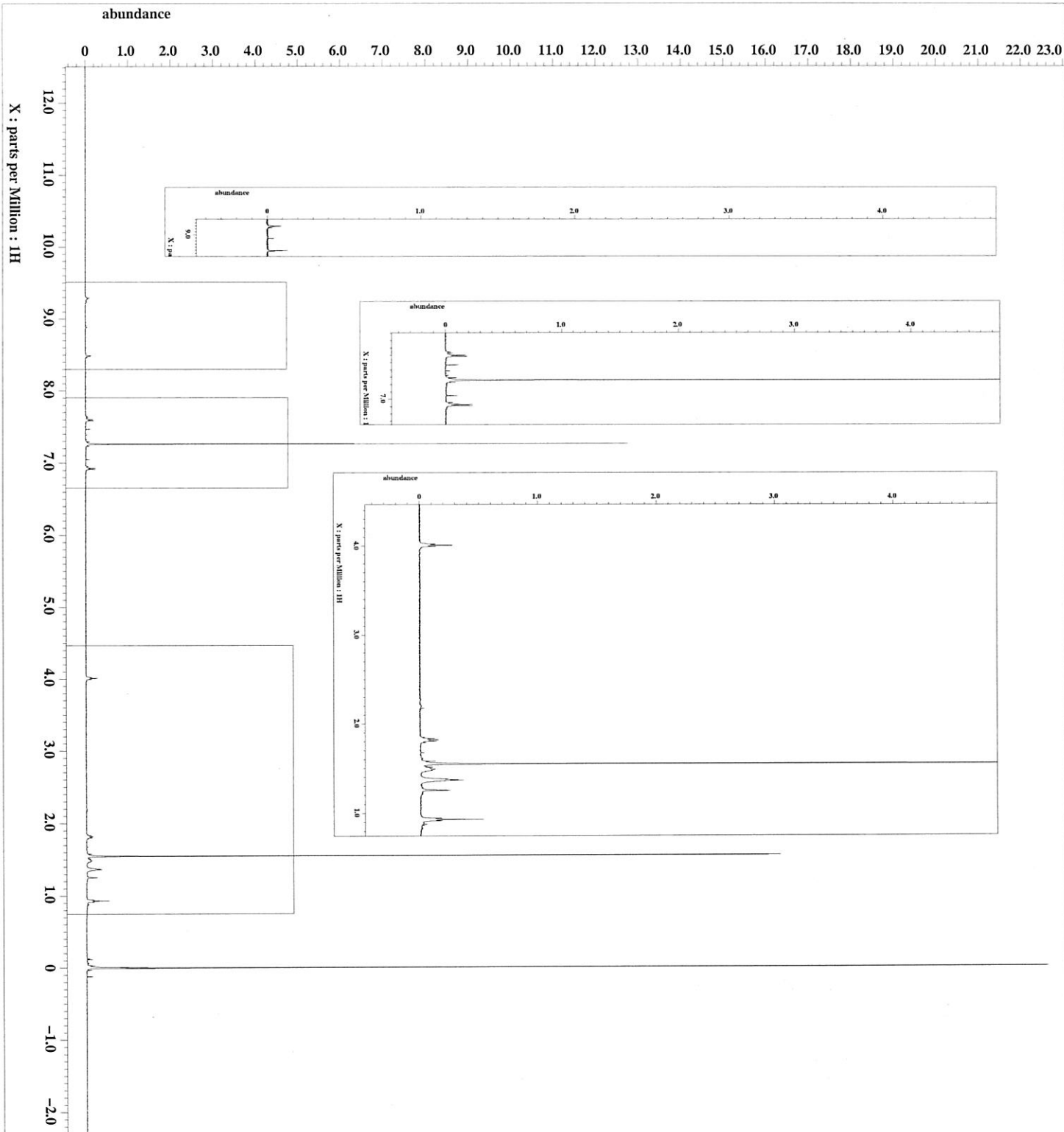


Figure S8. ¹H NMR of **1f**.



----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
smp : 0.2 [Hz] : 0.0 [s]
trepzoid3 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
ife : 1 : TRUE : TRUE
machinepnase
ppm

Filename = 20161215c4h9-f-2.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#447770
Solvent = CHLOROFORM-D
Creation time = 15-DEC-2016 10:59:35
Revision time = 7-JUL-2017 19:34:27
Current_time = 7-JUL-2017 19:35:31

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [kHz]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Mod_return = FALSE
Scans = 1
Total_scans = 8

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [dB]
X_pulse = 3.12 [us]
Irr_mode = Off
Irr_mode = Off
Dante_presat = FALSE
Initial_wait = 1 [s]
Recvr_gain = 54
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 17.1 [dC]

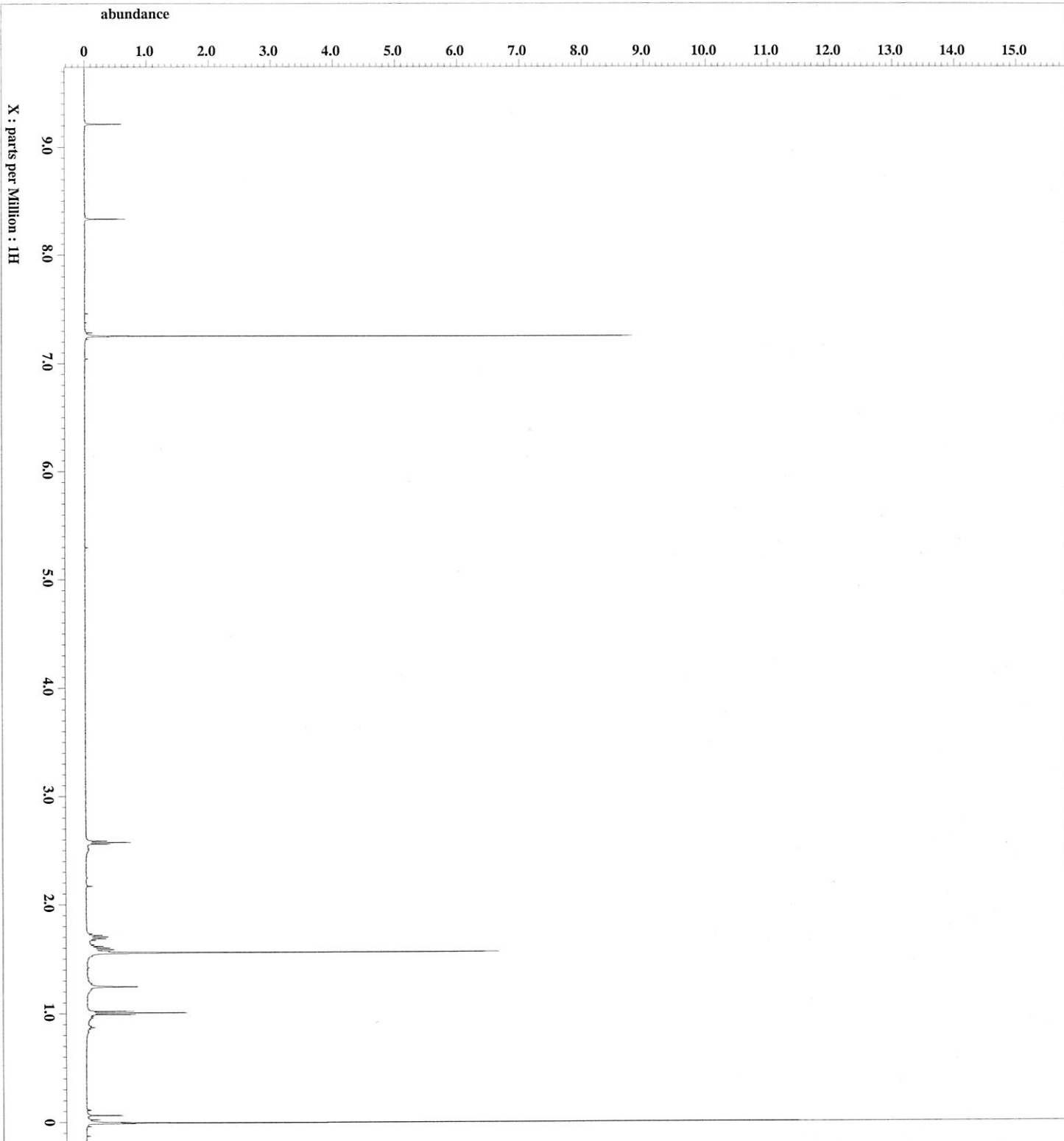


Figure S9. ¹H NMR of 2a.



----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
semp : 0.2 [Hz] : 0.0 [s]
trapzoid3 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinphase
ppm

Filename = 20170531c3h7ph-f-2.fid
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#659624
Solvent = CHLOROFORM-D
Creation_time = 31-MAY-2017 16:35:46
Revision_time = 7-JUL-2017 19:40:39
Current_time = 7-JUL-2017 19:41:07

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Spectrometer = ECA500
Site = JNM-ECA500

Field_strength = 11.743579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [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 = 8
Total_scans = 8

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [db]
X_pulse = 3.12 [us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1 [s]
Recvr_gain = 56
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 19.6 [dc]

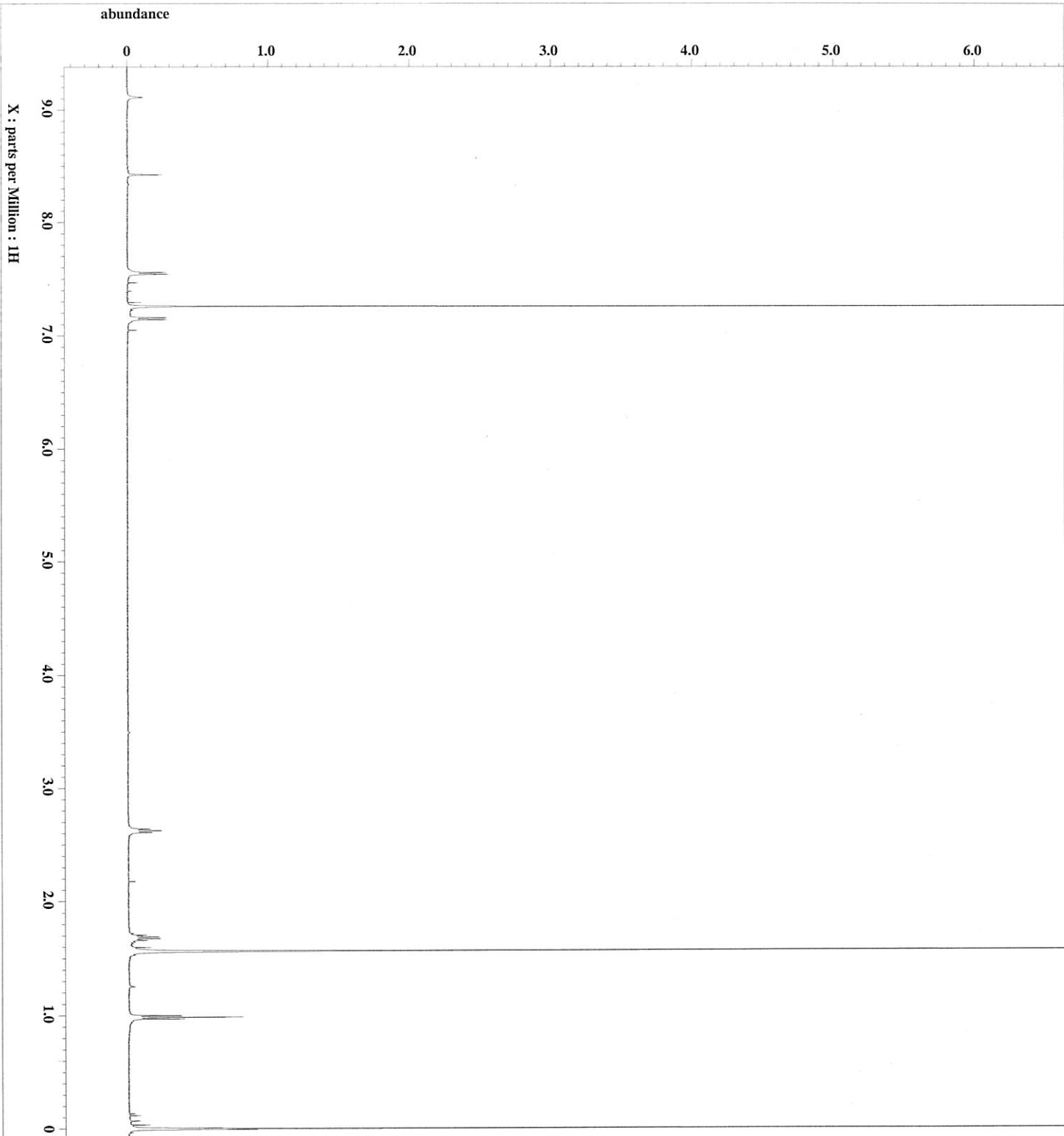


Figure S11. ¹H NMR of 2d.

```

---- PROCESSING PARAMETERS ----
dc balance : 0 : FALSE
smp : 0.2 [Hz] : 0.0 [s]
trapzoid3 : 0 [%] : 80 [%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
  
```

```

Filename = 20170531c3h7ph-f-2.fid
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#559624
Solvent = CHLOROFORM-D
Creation time = 31-MAY-2017 16:35:46
Revision time = 7-JUL-2017 19:40:39
Current_time = 7-JUL-2017 19:41:31
  
```

```

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Spectrum = ECA500
Spectrometer = JNM-ECA500
  
```

```

Field_strength = 11.7473579 [T] (500 [MH])
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [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 = 8
Total_scans = 8
  
```

```

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [dB]
X_pulse = 3.12 [us]
Irr_mode = Off
Tri_mode = FALSE
Dante_presat = 1 [s]
Recvr_gain = 56
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 19.6 [dC]
  
```

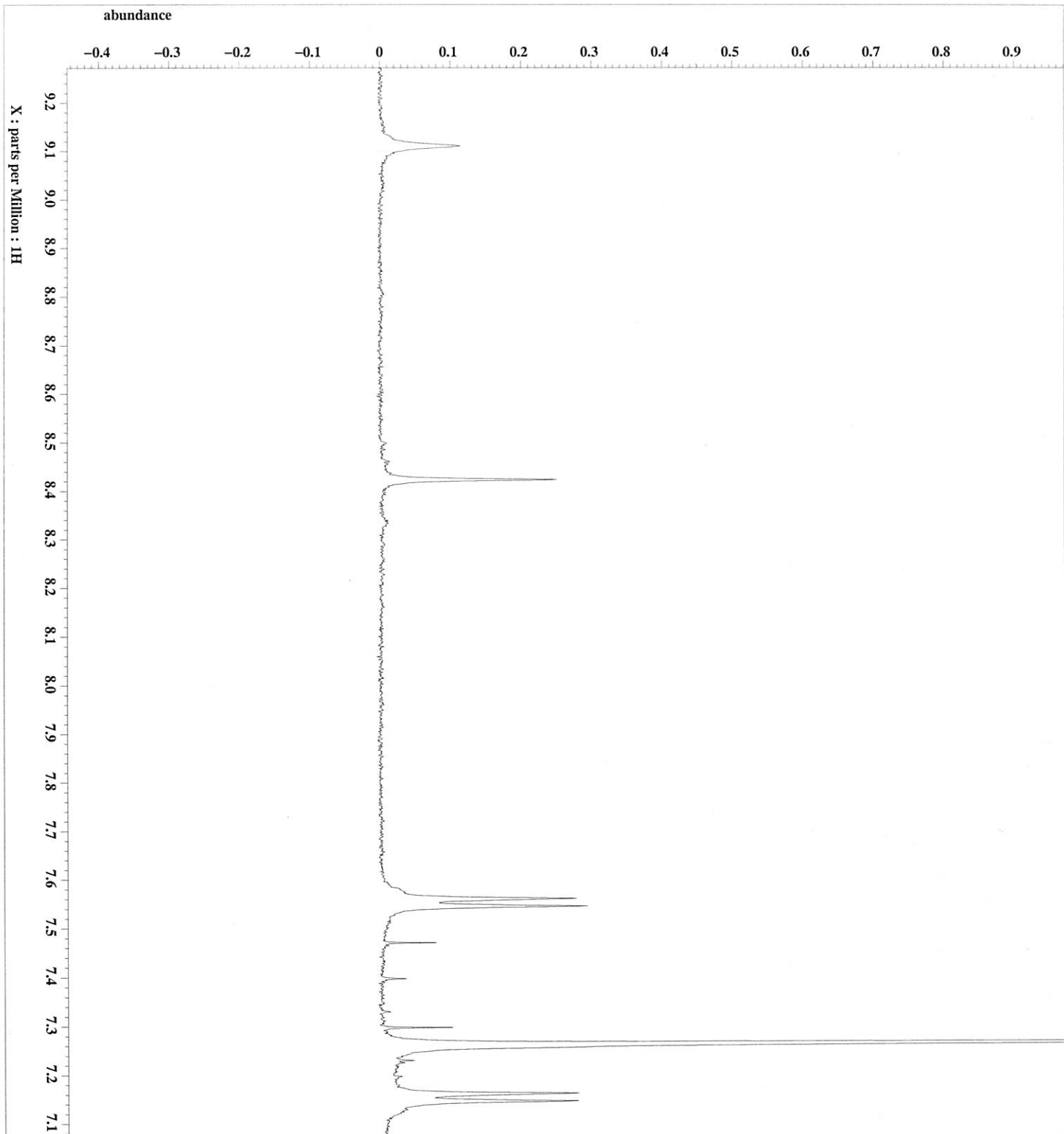


Figure S11. ¹H NMR of 2d (Low-field region).

```

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

```

Filename = 20170531c3h7ph-f-2.fid
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#559624
Solvent = CHLOROFORM-D
Creation time = 31-MAY-2017 16:35:46
Revision time = 7-JUL-2017 19:40:39
Current_time = 7-JUL-2017 19:41:40
  
```

```

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500
  
```

```

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57227737 [Hz]
X_sweep = 9.38438438 [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 = 8
Total_scans = 8
  
```

```

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [db]
X_pulse = 3.12 [us]
Irr_mode = Off
Tri_mode = OFF
Dance_presat = FALSE
Initial_wait = 1 [s]
Recvr_gain = 56
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 19.6 [dcl]
  
```

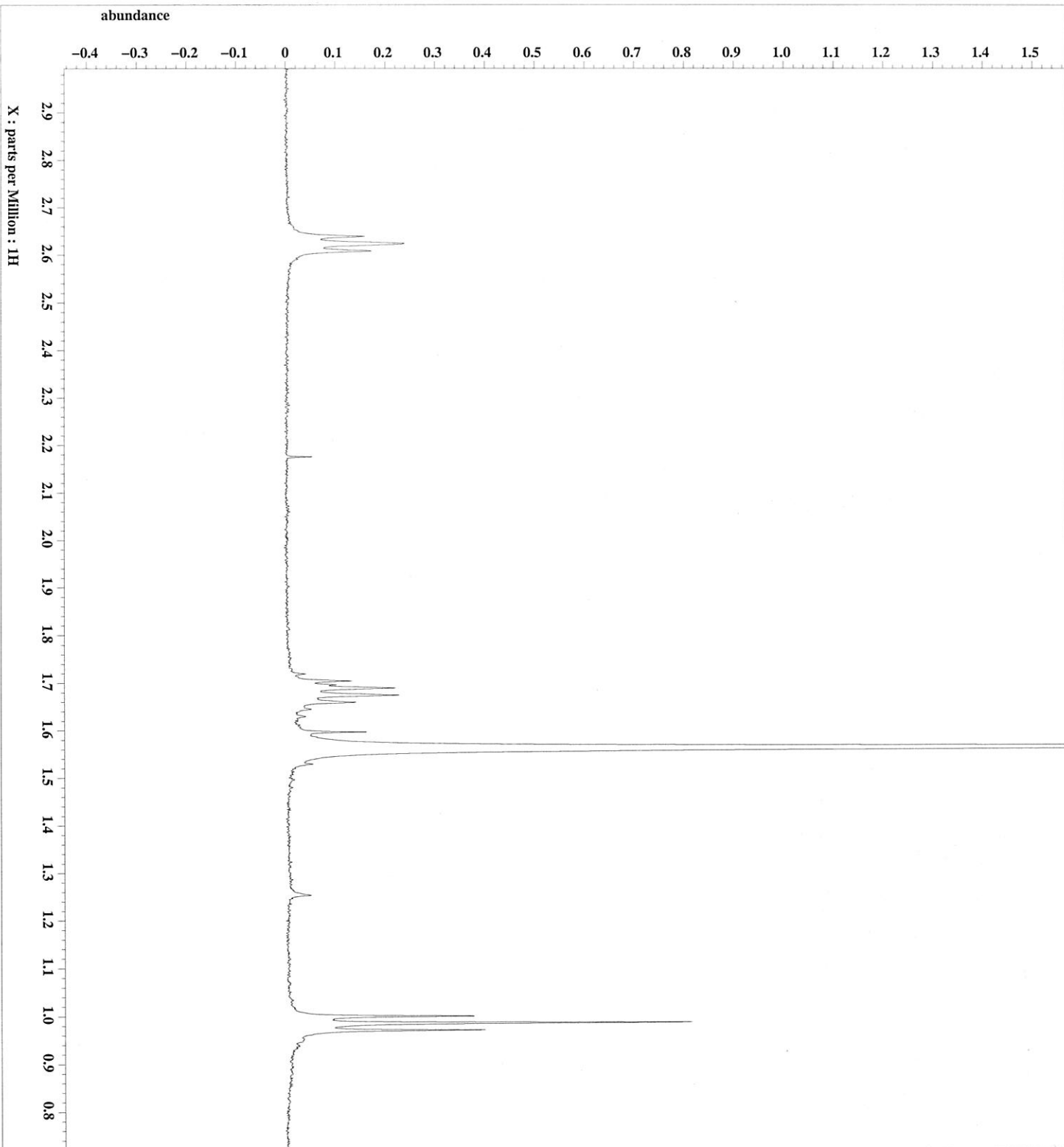


Figure S11. ¹H NMR of **2d** (Up-field region).



----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
semp : 0.2 [Hz] : 0.0 [s]
trapzoid3 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
f1c : 1 : TRUE : TRUE
machinephase
ppm

Filename = 20170510c6h13ph-f-2.f
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#771540
Solvent = CHLOROFORM-D
Creation_time = 10-MAY-2017 19:44:38
Revision_time = 7-JUL-2017 19:44:52
Current_time = 7-JUL-2017 19:46:02

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [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 = 8
Tocal_scans = 8

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [db]
X_pulse = 3.12 [us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1 [s]
Recvr_gain = 54
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 20.9 [dcl]

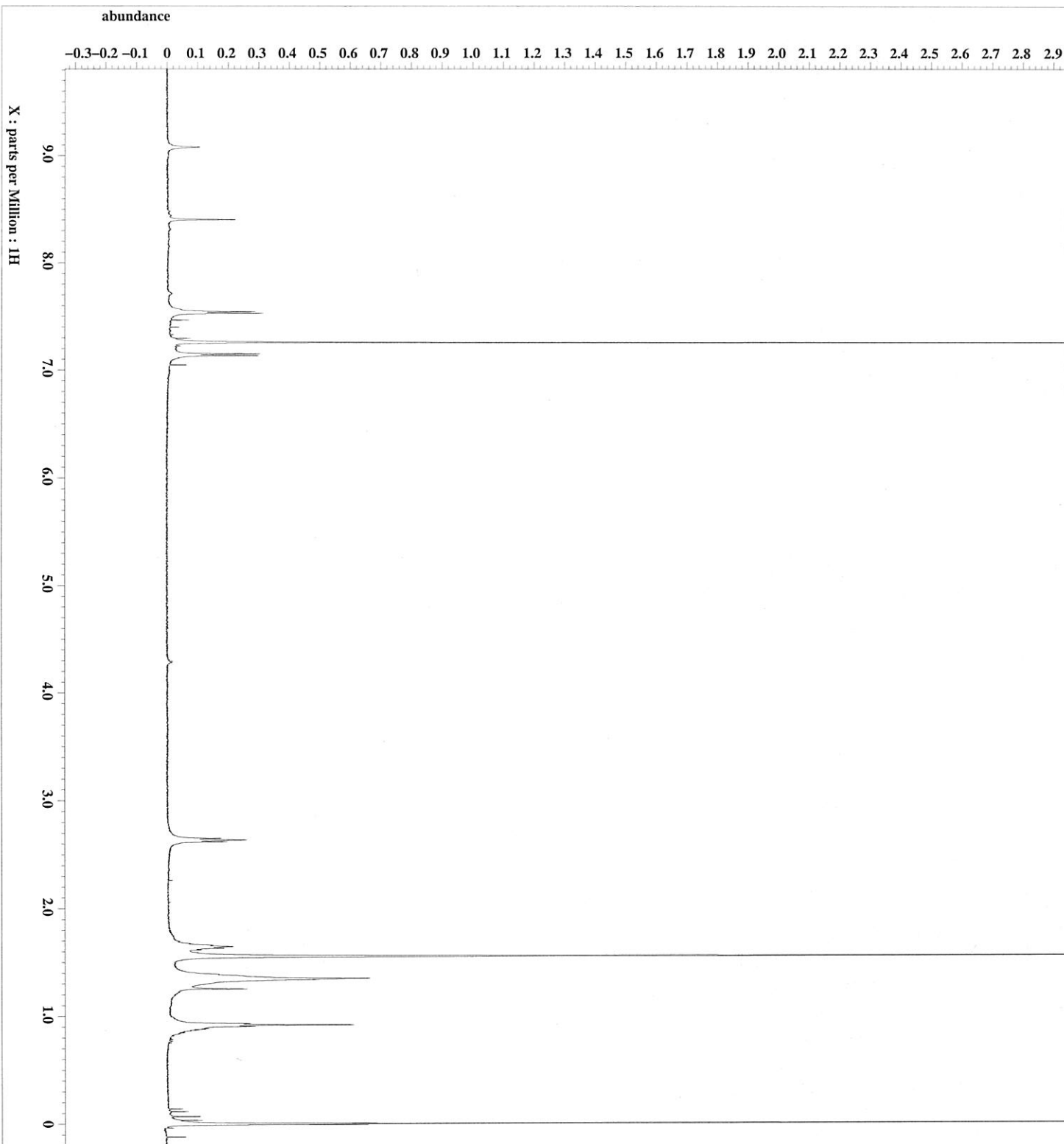


Figure S12. ¹H NMR of 2e.



----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
semp : 0.2 [Hz] : 0.0 [s]
trapzoid3 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename = 20170224CGH130Pn-F-2.
Author = delta
Experiment = single_pulse_ex2
Sample_id = S#630814
Solvent = CHLOROFORM-D
Creation_time = 24-FEB-2017 15:58:37
Revision_time = 7-JUL-2017 19:49:01
Current_time = 7-JUL-2017 19:49:23

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [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 = 8
Total_scans = 8

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [db]
X_pulse = 3.12 [us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1 [s]
Recvr_gain = 54
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_set = 21.1 [dcl]

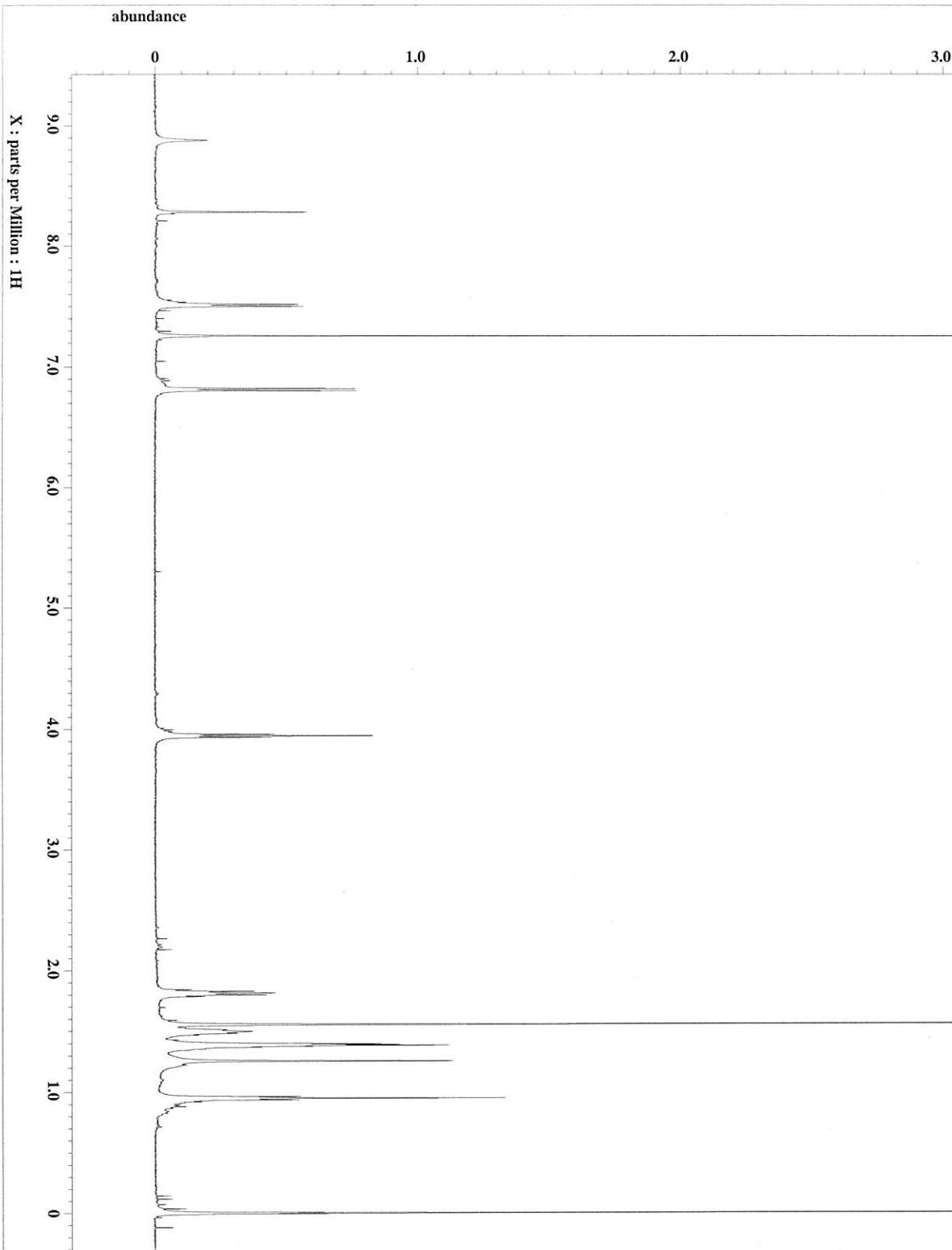


Figure S13. ¹H NMR of 2f.



----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
temp : 0.2 [Hz] : 0.0 [s]
tzero[id3] : 0 [%] : 80 [%] : 100 [%]
zero[fill] : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename = 20170419 (C6H13O) 3Pb-F
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#568103
Solvent = CHLOROFORM-D
Creation time = 19-APR-2017 14:07:47
Revision time = 7-JUL-2017 19:51:20
Current_time = 7-JUL-2017 19:51:50

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [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 = 8
Total_scans = 8
X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [db]
X_pulse = 3.13 [us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1 [s]
Recvr_gain = 40
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 21.7 [dC]

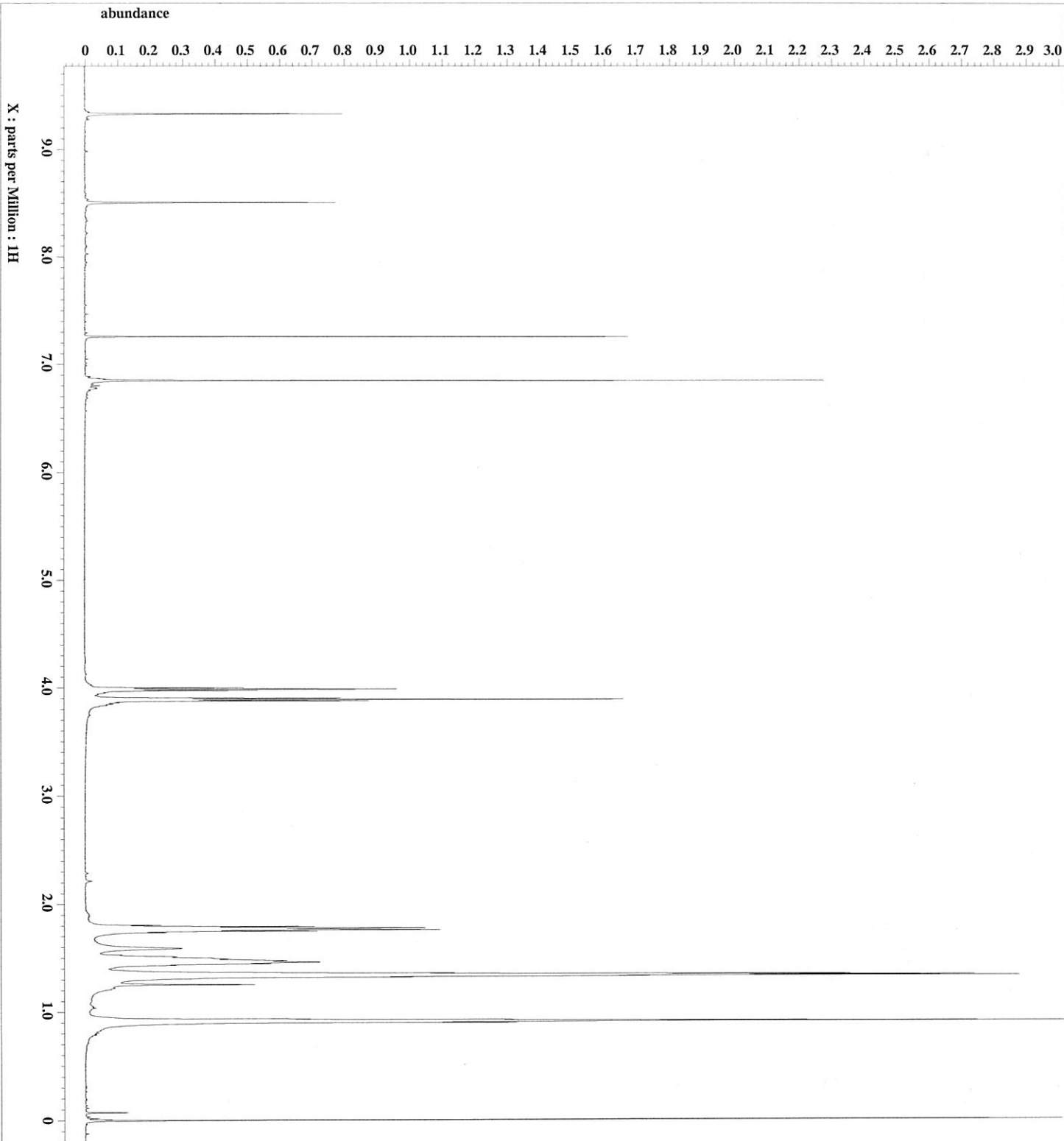


Figure S14. ¹H NMR of 2g.


```

---- PROCESSING PARAMETERS ----
dc balance : 0 : FALSE
sepd : 2.0 [Hz] : 0.0 [s]
t1rho3 : 0 [s] : 80 [%] : 100 [%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
dpm
  
```

```

Filename = 20170419 (C6H13O) 3ph-F
Author = delta
Experiment = single_pulse_dec
Sample_id = S#572271
Solvent = CHLOROFORM-D
Creation_time = 19-APR-2017 15:29:04
Revision_time = 7-JUL-2017 19:52:13
Current_time = 7-JUL-2017 19:53:20
  
```

```

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 = 1600
rotal_scans = 1600
  
```

```

X_90_width = 10.87 [us]
X_acq_time = 0.83361792 [s]
X_angle = 30 [deg]
X_atn = 6.5 [dB]
X_pulse = 3.62333333 [us]
Irr_atn_dec = 26.772 [dB]
Irr_atn_noe = 26.772 [dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1 [s]
Noe_time = TRUE
Noe_wait = 2 [s]
Recvr_gain = 50
Relaxation_delay = 2 [s]
Repetition_time = 2.83361792 [s]
Temp_get = 19.8 [dc]
  
```

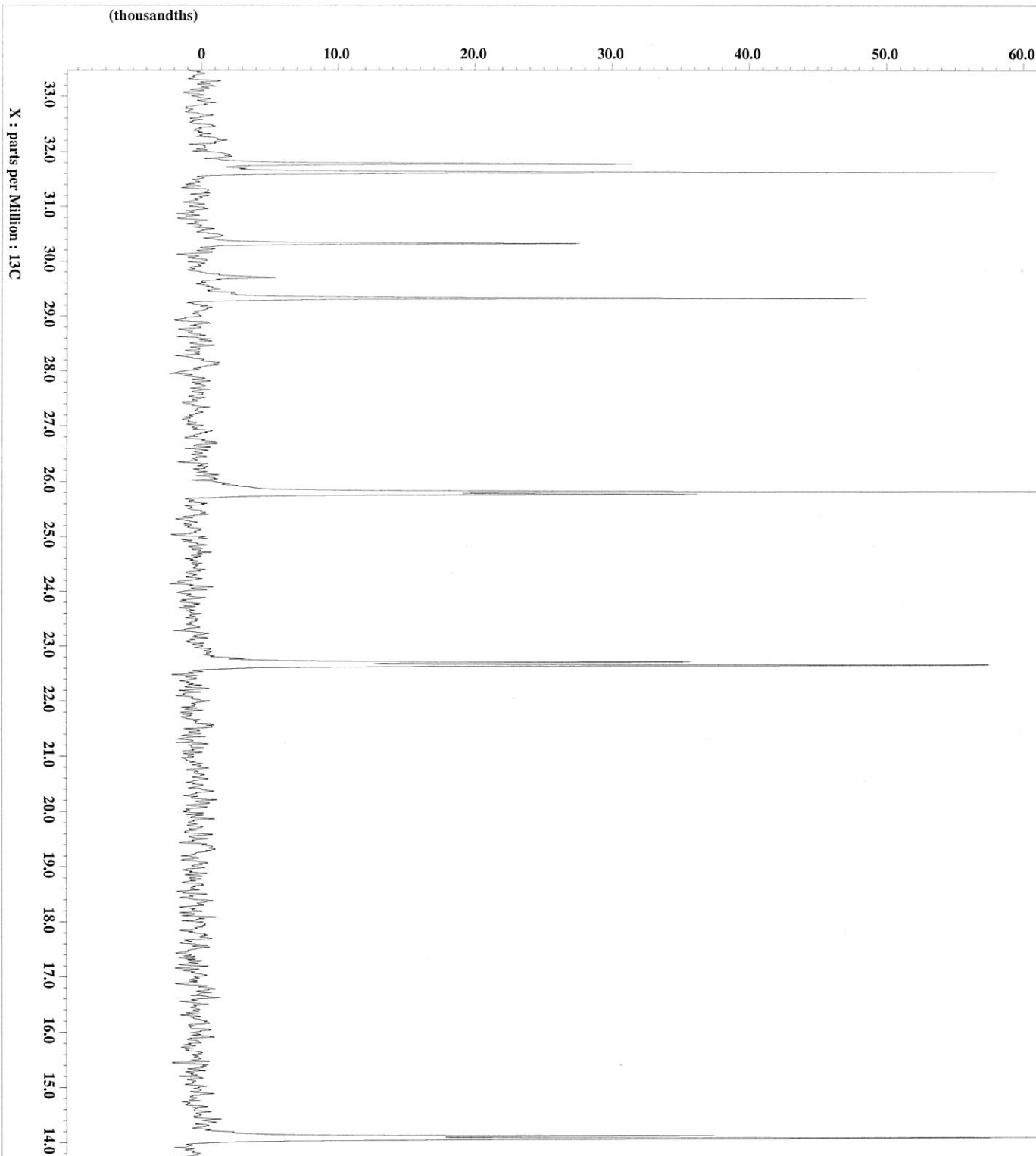


Figure S15. ¹³C NMR of **2g** (Up-field region).



----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
sweep : 0.2 [Hz] : 0.0 [s]
creep_ratio : 0 [%] : 80 [%] : 100 [%]
zero_fill : 1
ift : 1 : TRUE : TRUE
machinephase
ppm

Filename = 20170203BE-Ph345 (OC6H
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#498241
Solvent = CHLOROFORM-D
Creation_time = 3-FEB-2017 12:18:58
Revision_time = 7-JUL-2017 20:05:39
Current_time = 7-JUL-2017 20:06:03

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5727737 [Hz]
X_sweep = 9.38438438 [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 = 8
Total_scans = 8

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [ds]
X_pulse = 3.12 [us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1 [s]
Recur_gain = 32
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 20.5 [dc]

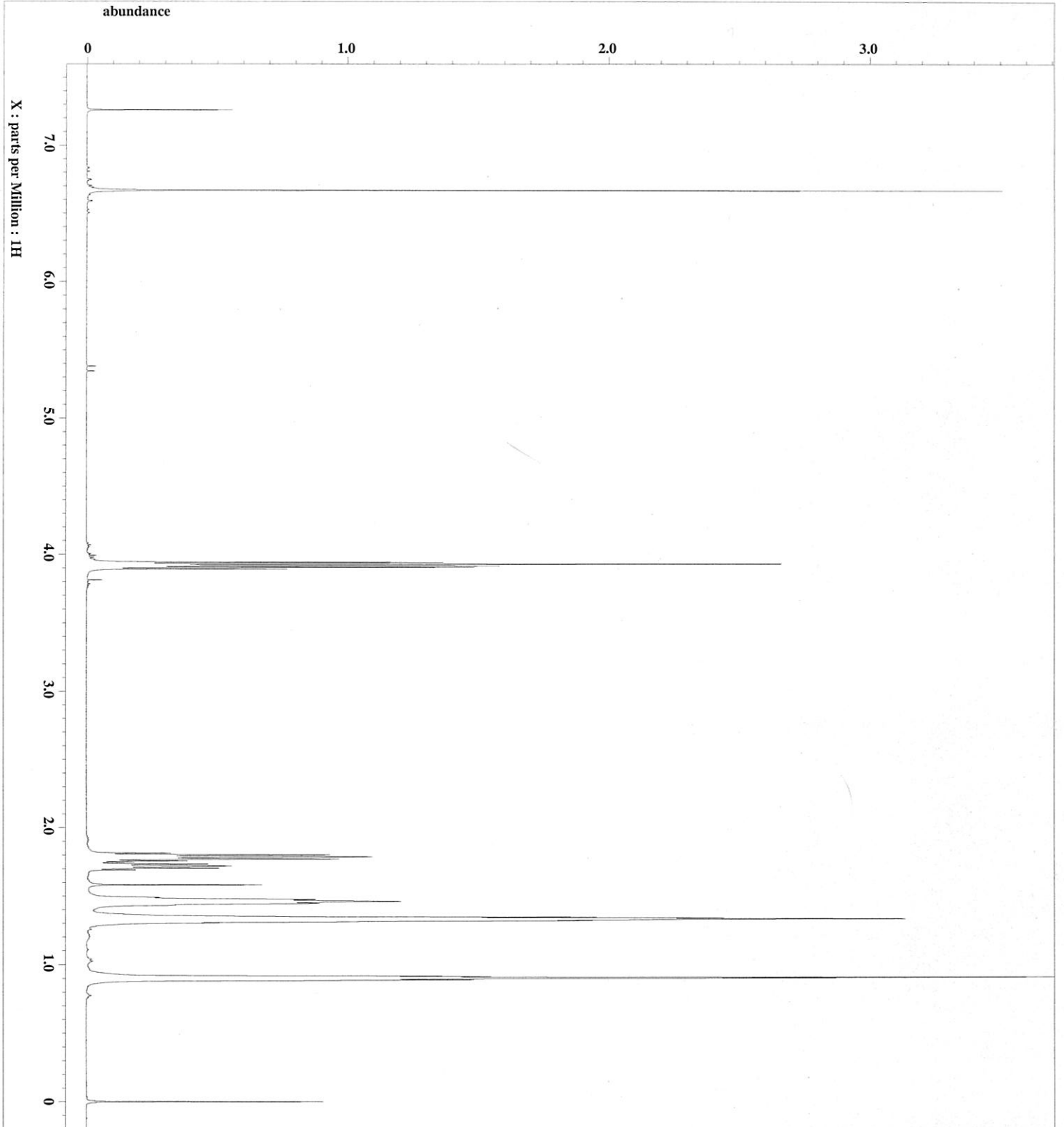


Figure S16. ¹H NMR of 13.



----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
secp : 0.2 [Hz] : 0.0 [Hz]
trapzoid3 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Filename = 20170217trimethyl1SI-P
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#770023
Solvent = CHLOROFORM-D
Creation_time = 17-FEB-2017 19:50:48
Revision_time = 7-JUL-2017 20:08:45
Current_time = 7-JUL-2017 20:09:06

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579 [T] (500 [MH
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [kHz]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [dB]
X_pulse = 3.12 [us]
Irr_mode = Off
Irr_mods = Off
Dante_presat = FALSE
Initial_wait = 1 [s]
Recvr_gain = 42
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 20.5 [dC]

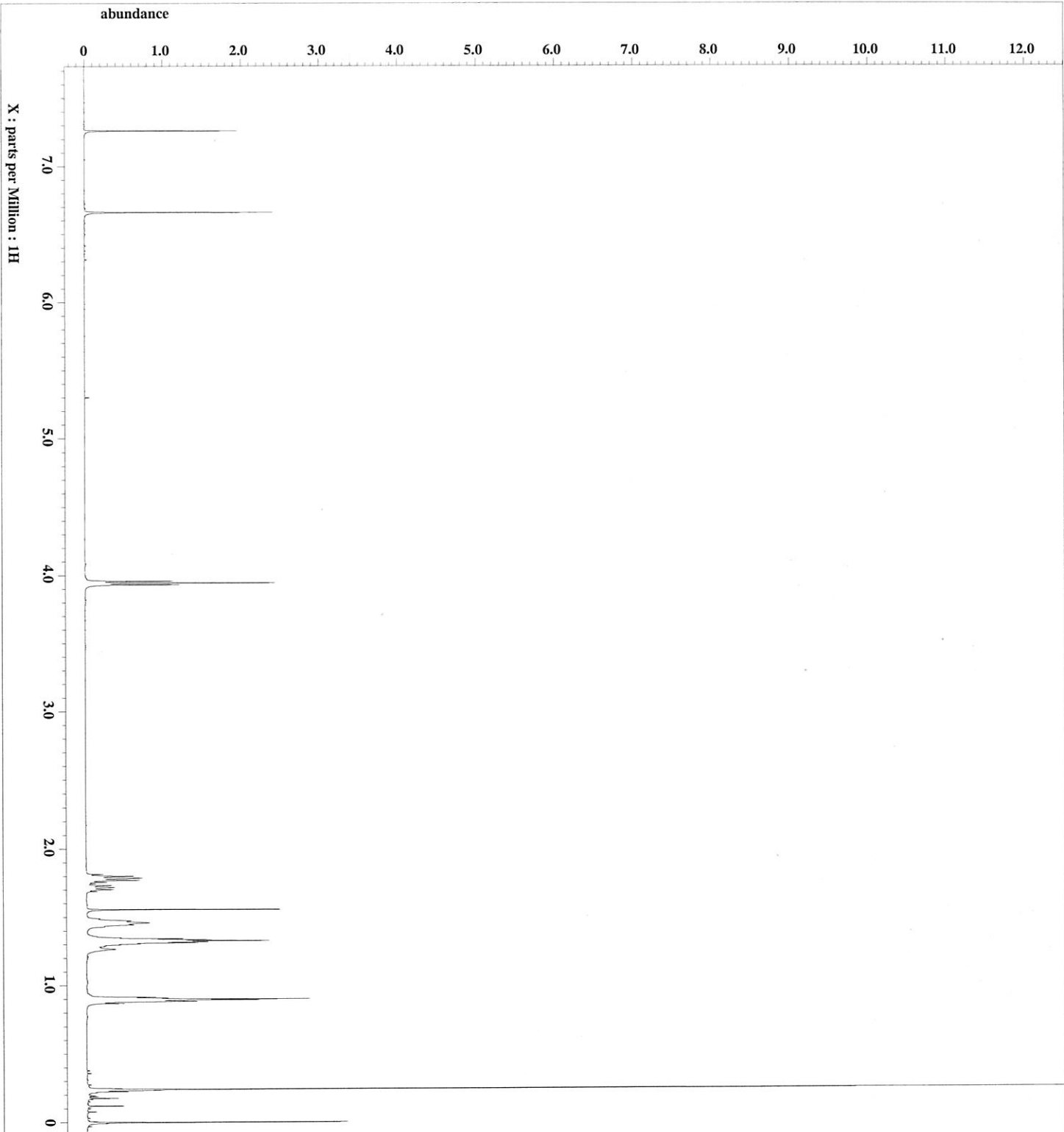


Figure S17. ¹H NMR of 14.



```

----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
sweep : 0.2 [Hz] : 0.0 [s]
curepolds : 0 [%] : 80 [%] : 100 [%]
zerofill : 1
ift : 1 : TRUE : TRUE
machinephase
ppm
  
```

```

Filename = 20170222ADPh345 (CGH130)
Author = delta
Experiment = single_pulse.ex2
Sample_id = #741529
Solvent = CHLOROFORM-D
Creation_time = 22-FEB-2017 19:02:26
Revision_time = 7-JUL-2017 20:11:20
Current_time = 7-JUL-2017 20:11:35
  
```

```

Comment = single_pulse
Data_format = 1D_COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = JNM-ECA500
  
```

```

Field_strength = 11.7473579 [G] (500 [MH]
X_acq_duration = 1.74587904 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 5.0 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737 [Hz]
X_sweep = 9.38438438 [kHz]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Irr_domain = 1H
Irr_freq = 500.15991521 [MHz]
Irr_offset = 5.0 [ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
  
```

```

X_90_width = 6.24 [us]
X_acq_time = 1.74587904 [s]
X_angle = 45 [deg]
X_atn = 3.4 [dB]
X_pulse = 3.12 [us]
Irr_mode = Off
Irr_mode = Off
Dante_presat = FALSE
Initial_wait = 1 [s]
Recvr_gain = 36
Relaxation_delay = 5 [s]
Repetition_time = 6.74587904 [s]
Temp_get = 21.2 [dC]
  
```

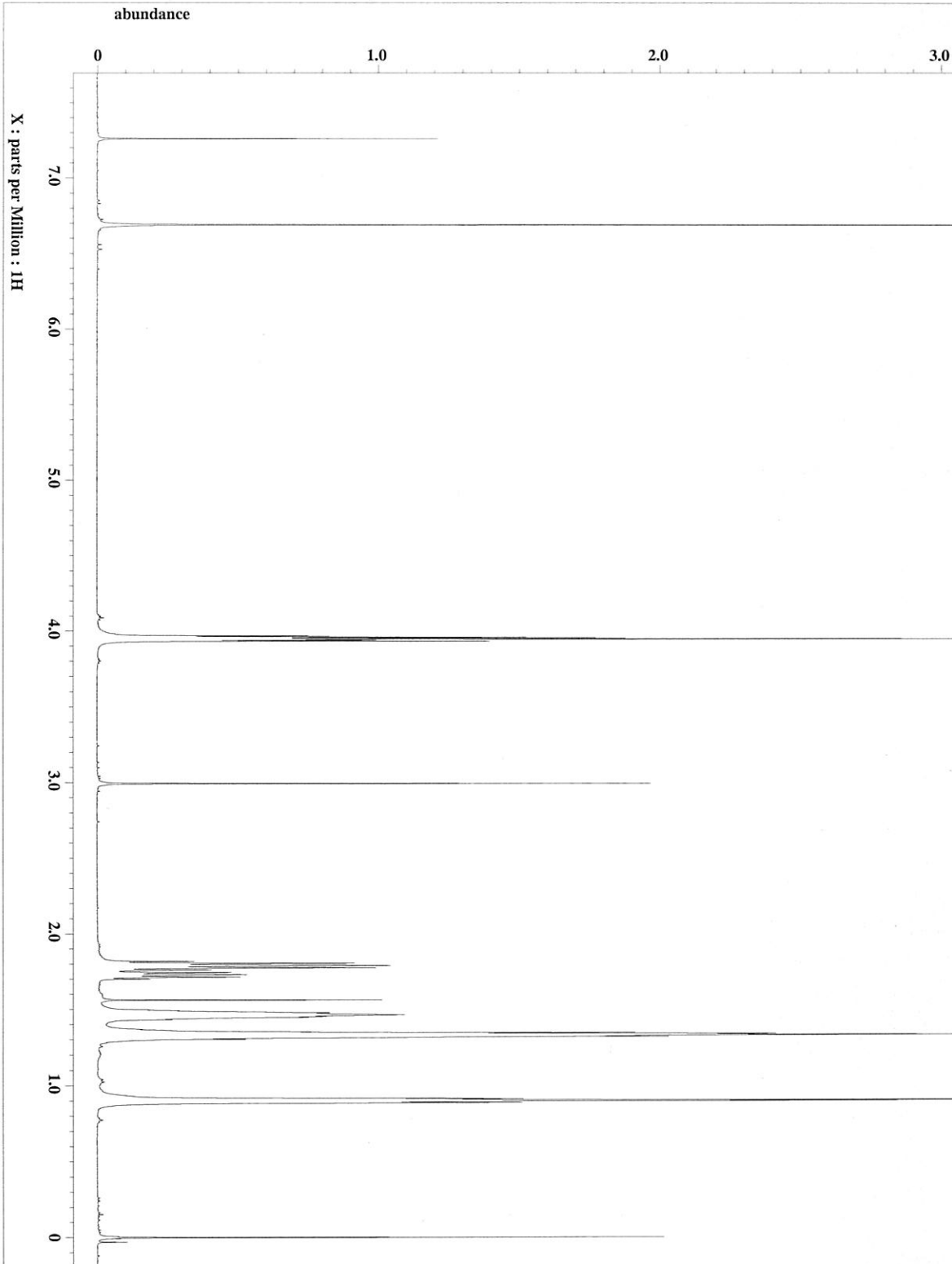
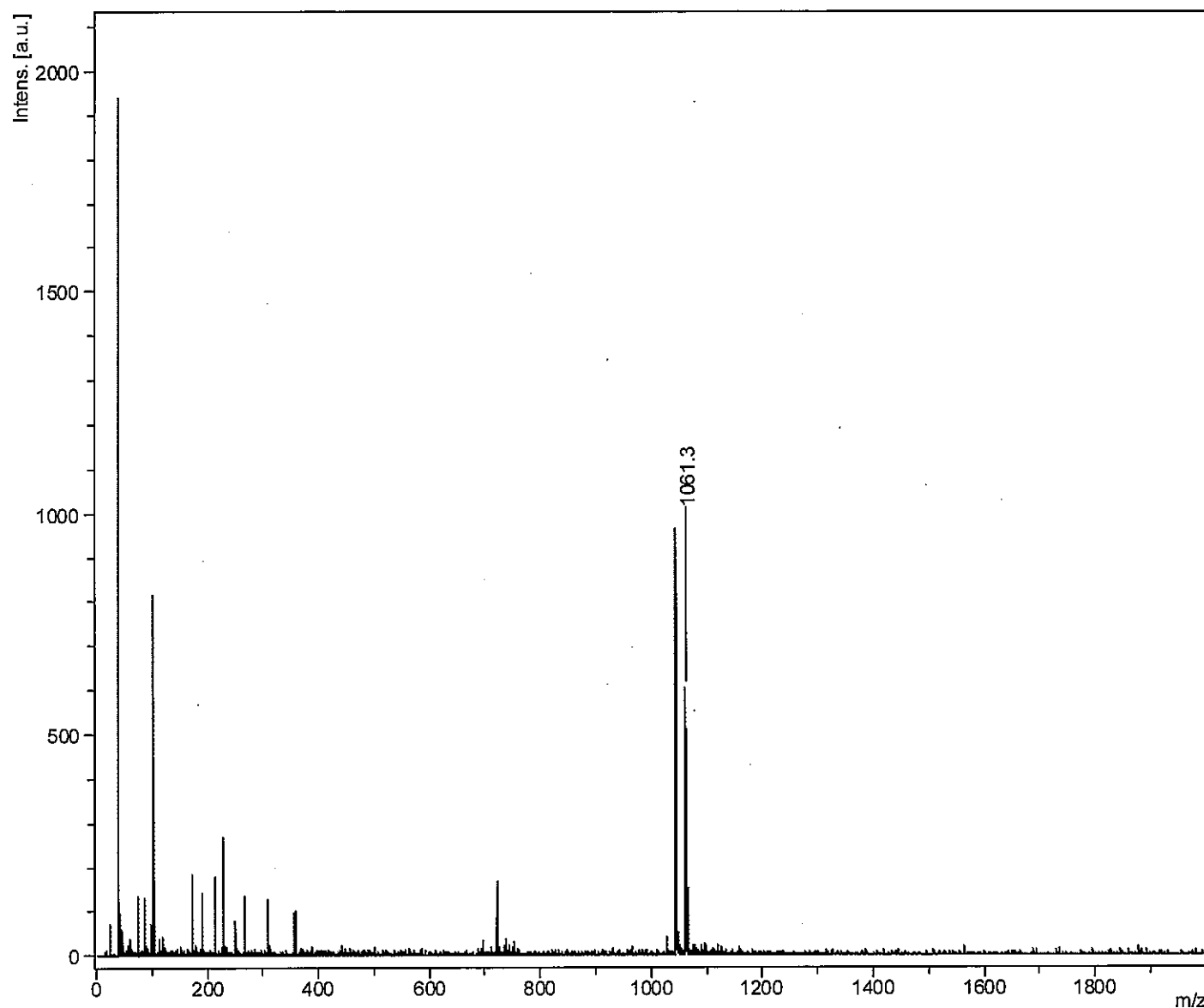


Figure S18. ¹H NMR of 15.

Comment 1

Comment 2



Acquisition Parameter

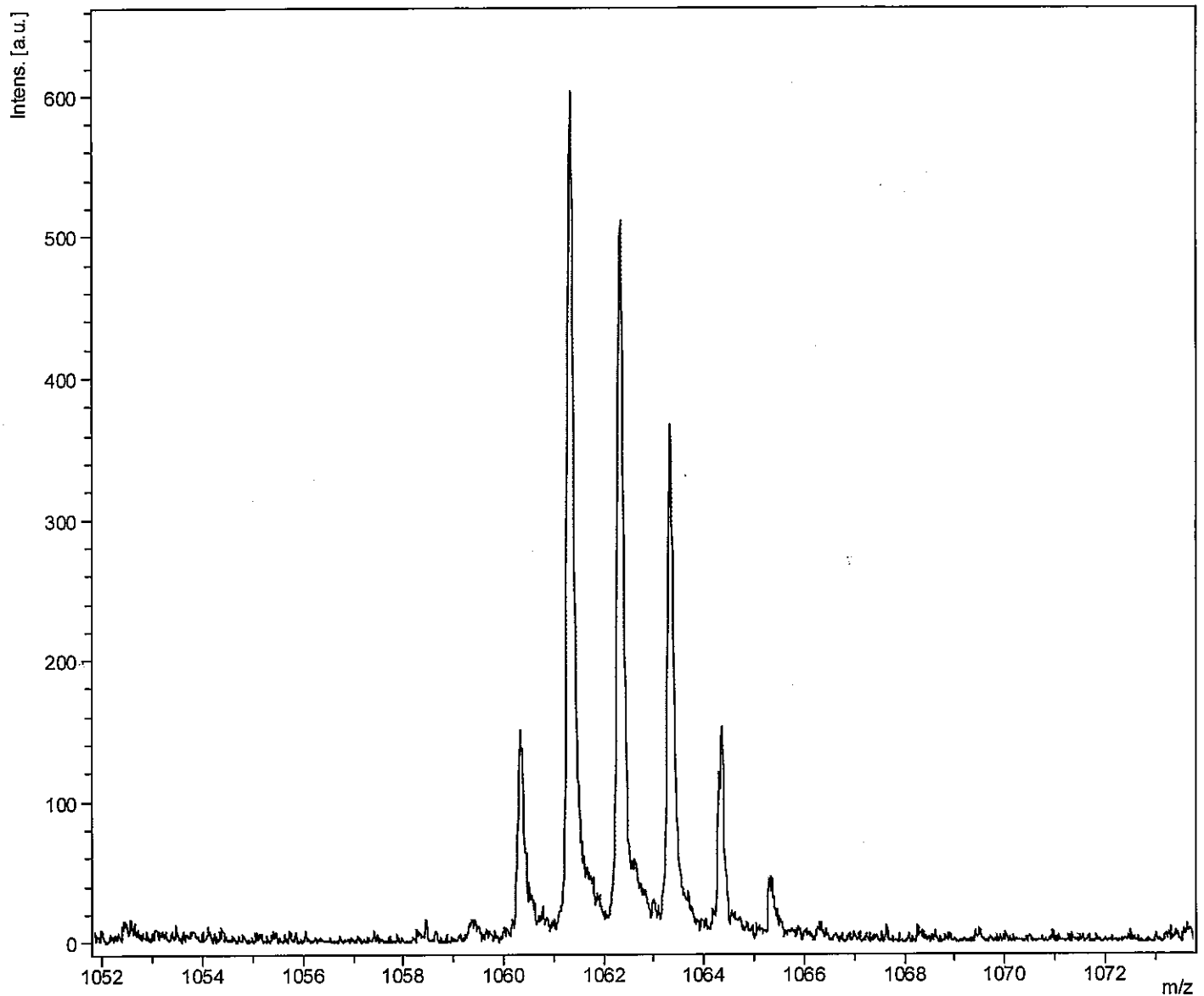
Date of acquisition 2016-10-06T13:29:11.171+09:00
Acquisition method name D:\Methods\flexControl\Methods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

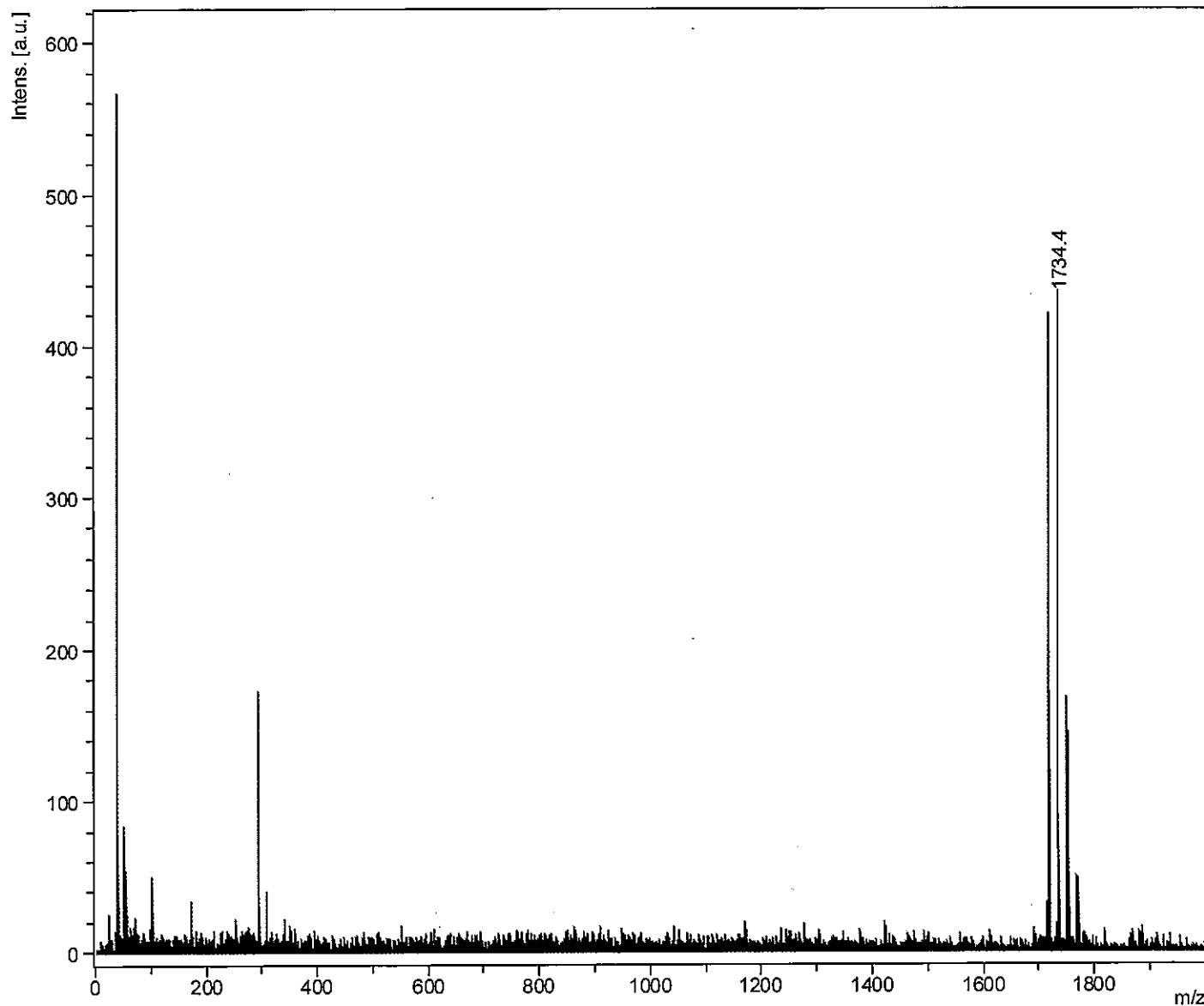
Date of acquisition 2016-10-06T13:29:11.171+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

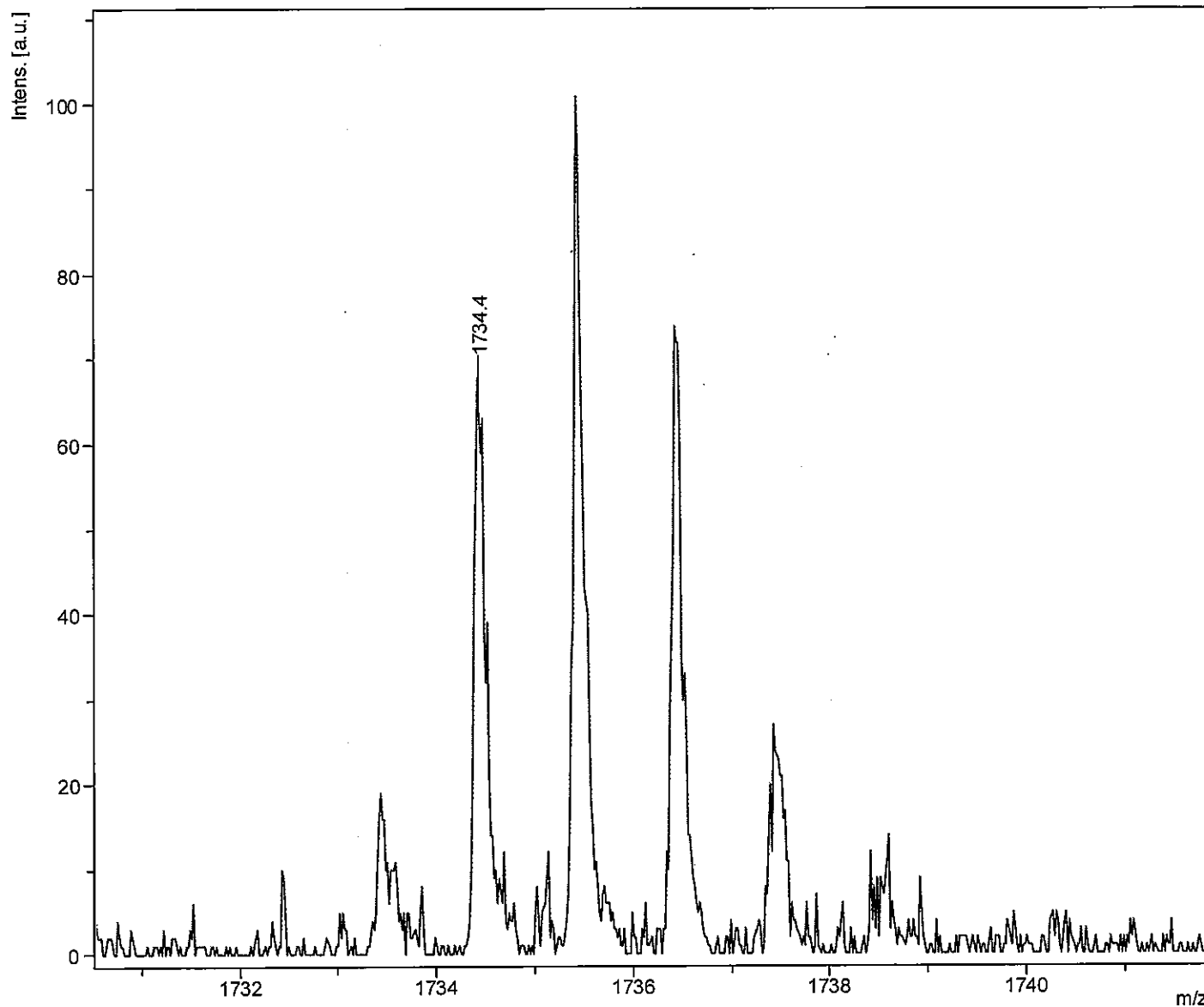
Comment 1

Comment 2



Comment 1

Comment 2



Acquisition Parameter

Date of acquisition 2016-10-06T13:31:10.171+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

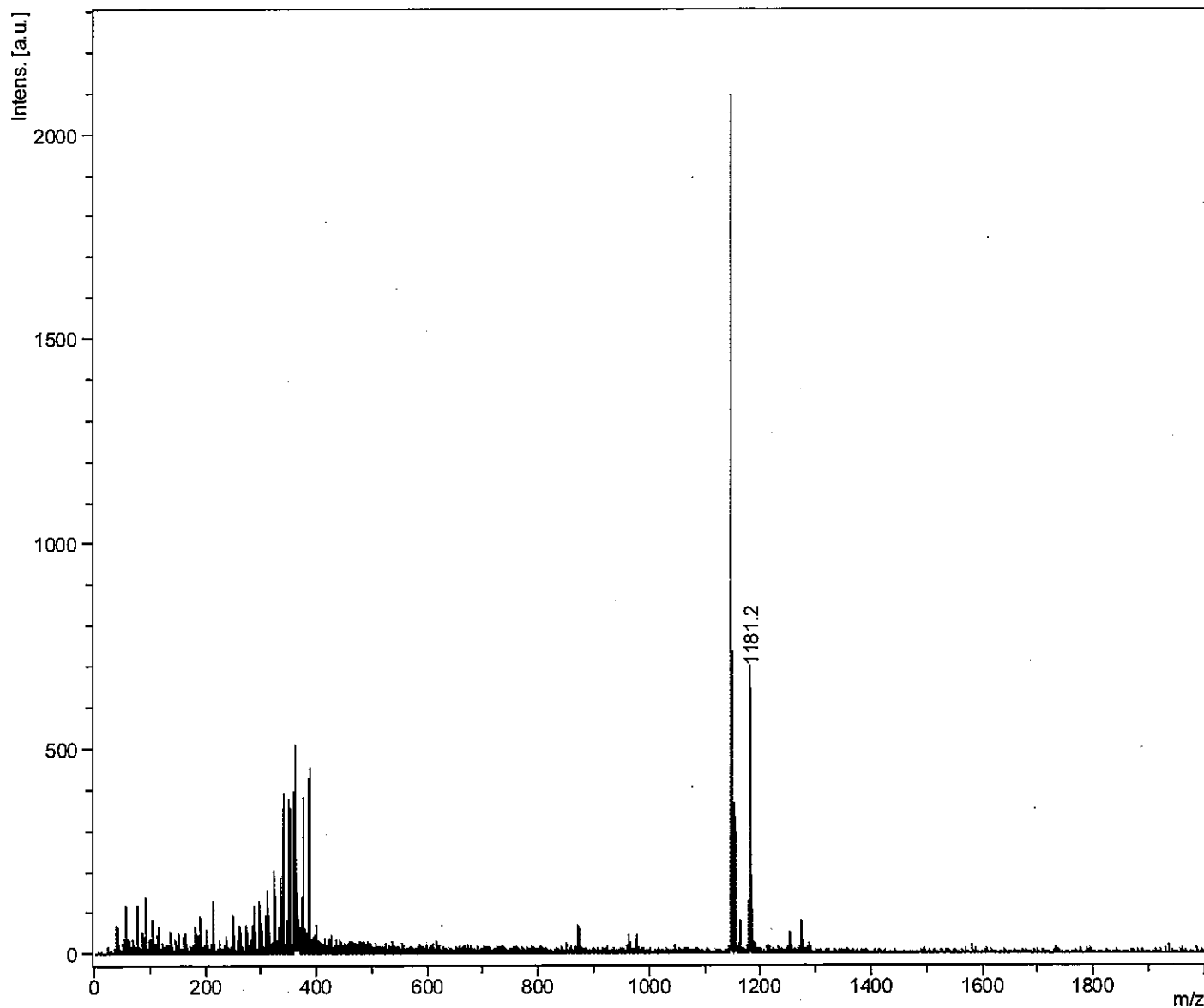
Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Figure S20. MALDI-TOF mass spectrum of 1b (Molecular ion peaks).

Comment 1

Comment 2



Acquisition Parameter

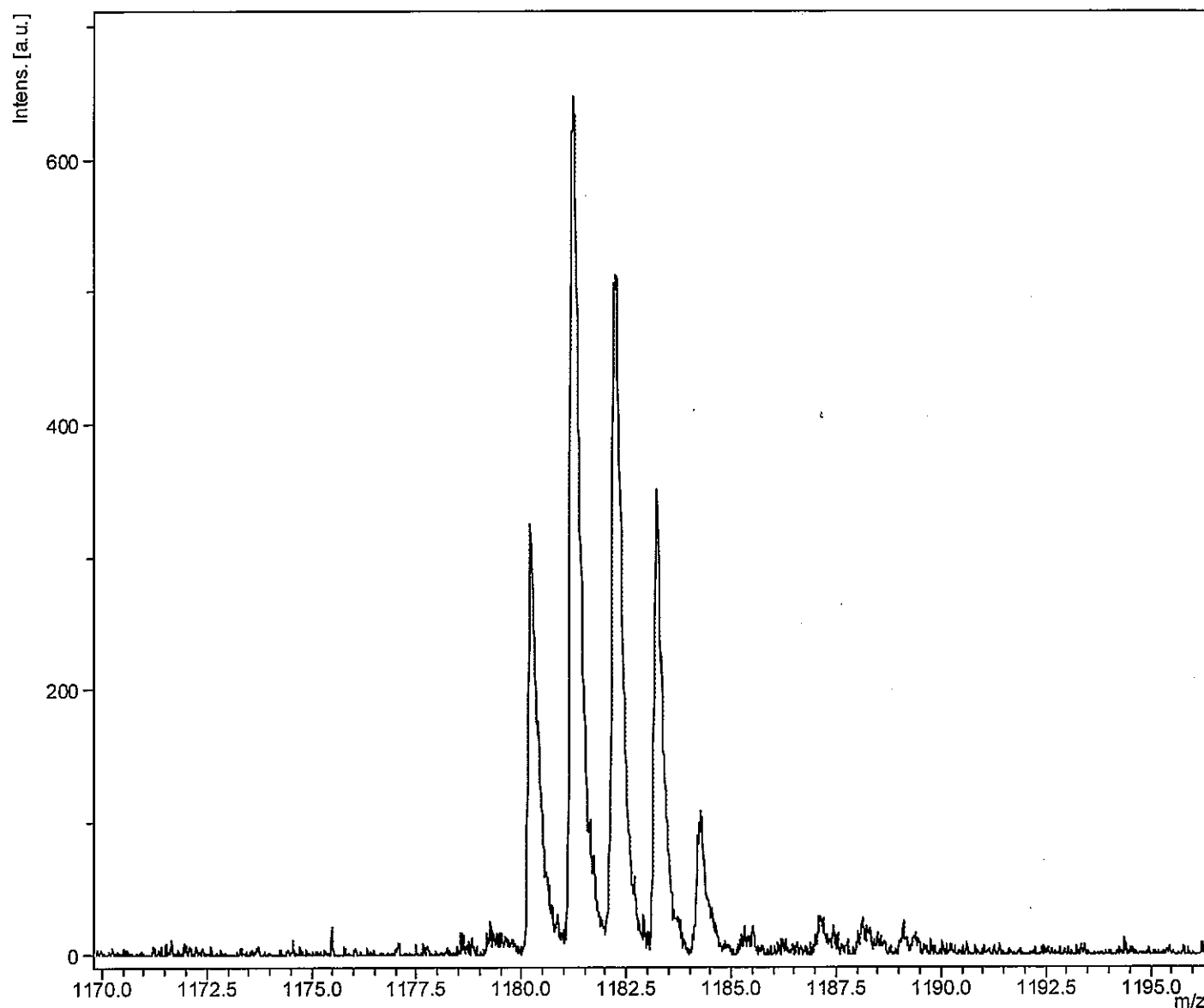
Date of acquisition 2016-10-06T13:32:48.765+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

Date of acquisition 2016-10-06T13:32:48.765+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

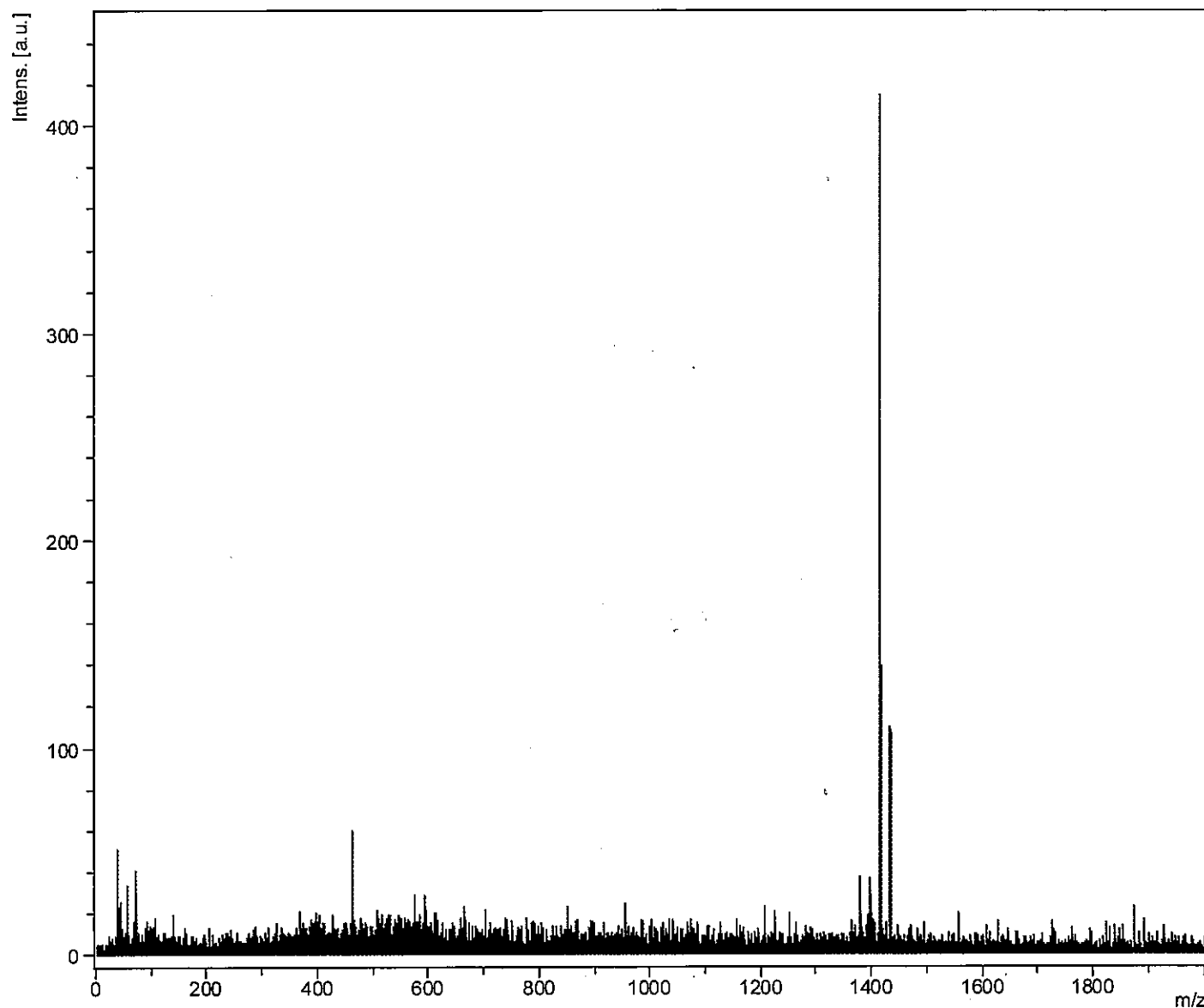
Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Figure S21. MALDI-TOF mass spectrum of **1c** (Molecular ion peaks).

Comment 1

Comment 2



Acquisition Parameter

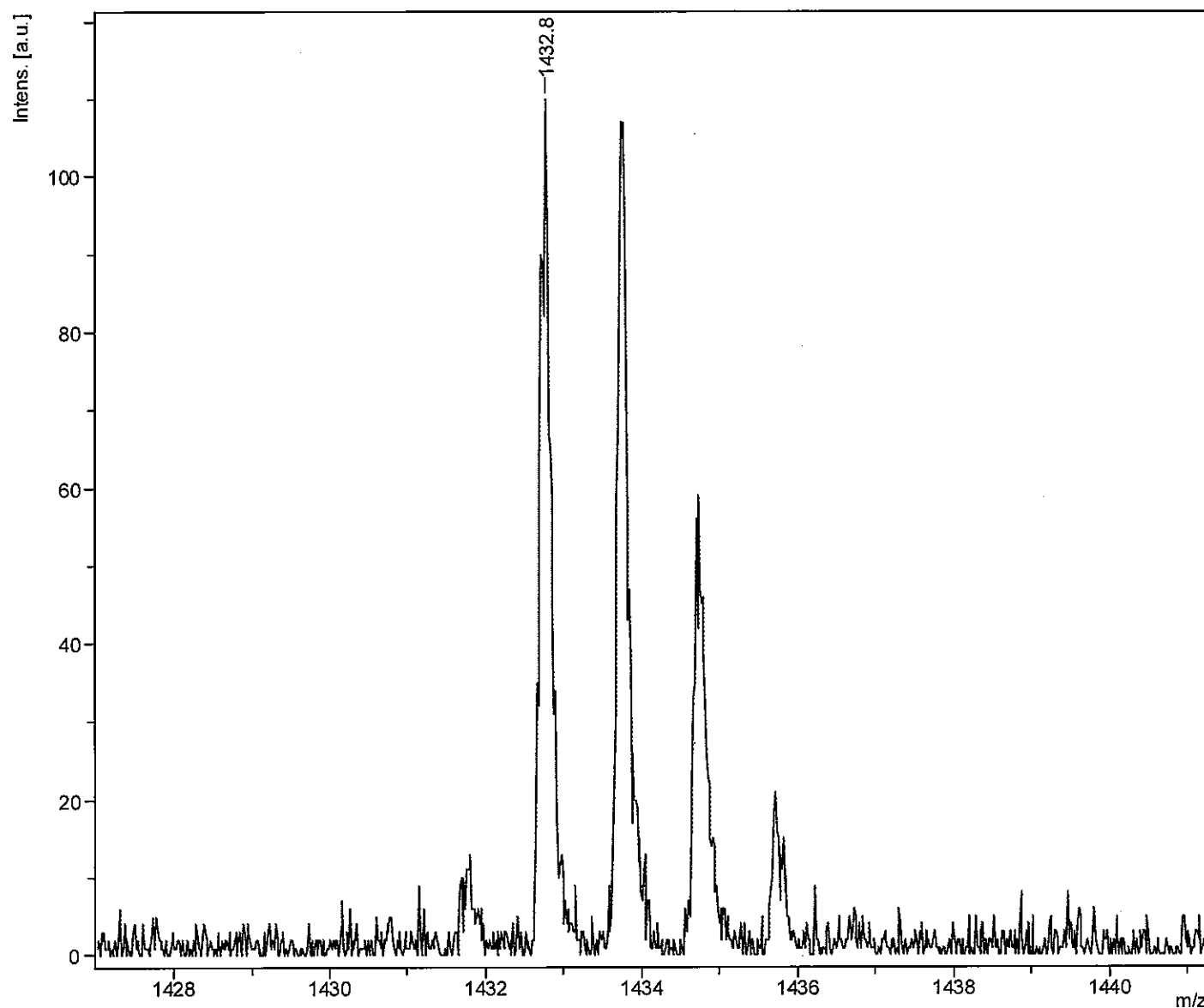
Date of acquisition 2017-01-16T10:50:16.781+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

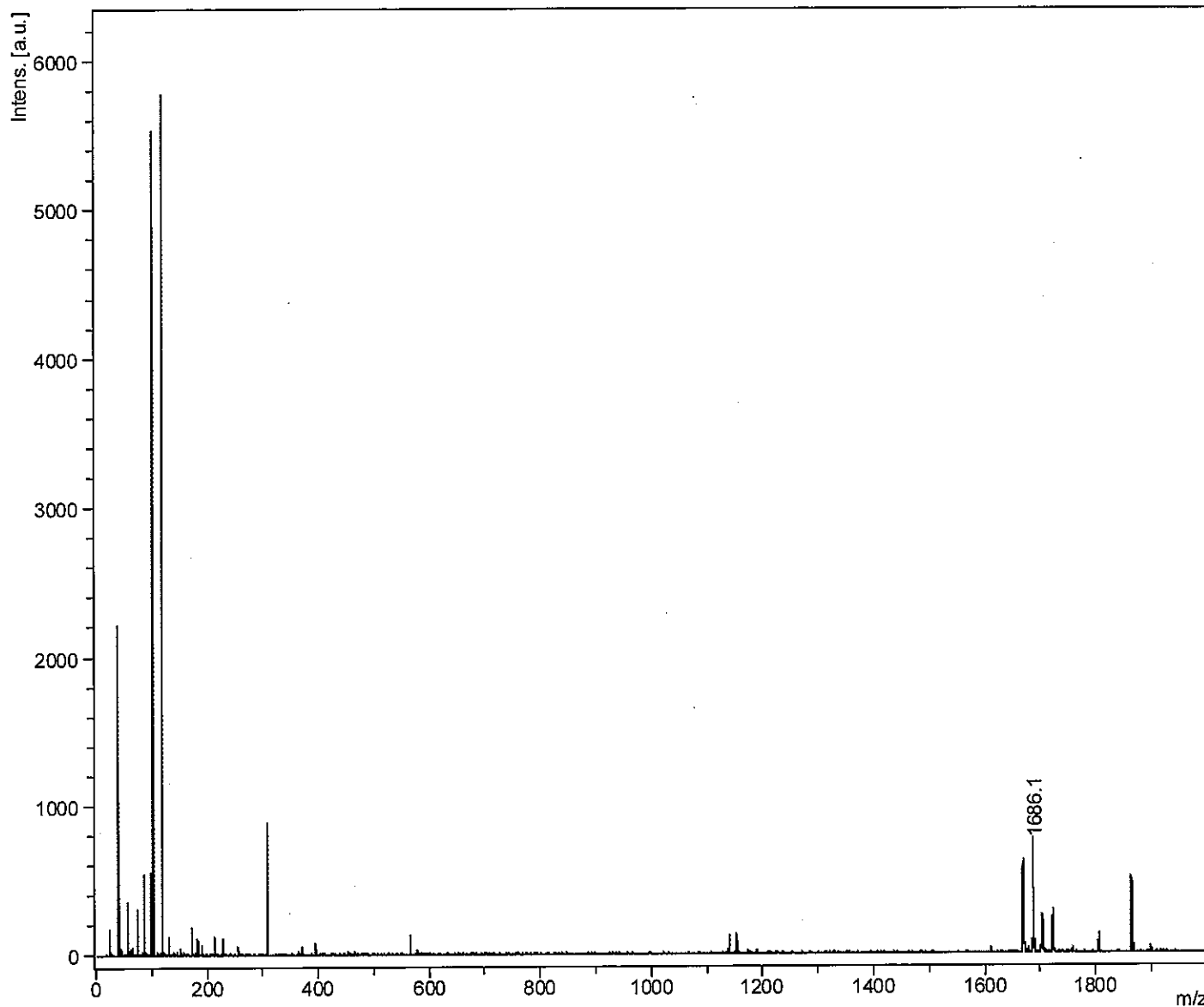
Date of acquisition 2017-01-16T10:50:16.781+09:00
Acquisition method name D:\Methods\flexControl\Methods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

Date of acquisition 2016-10-06T13:34:03.875+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

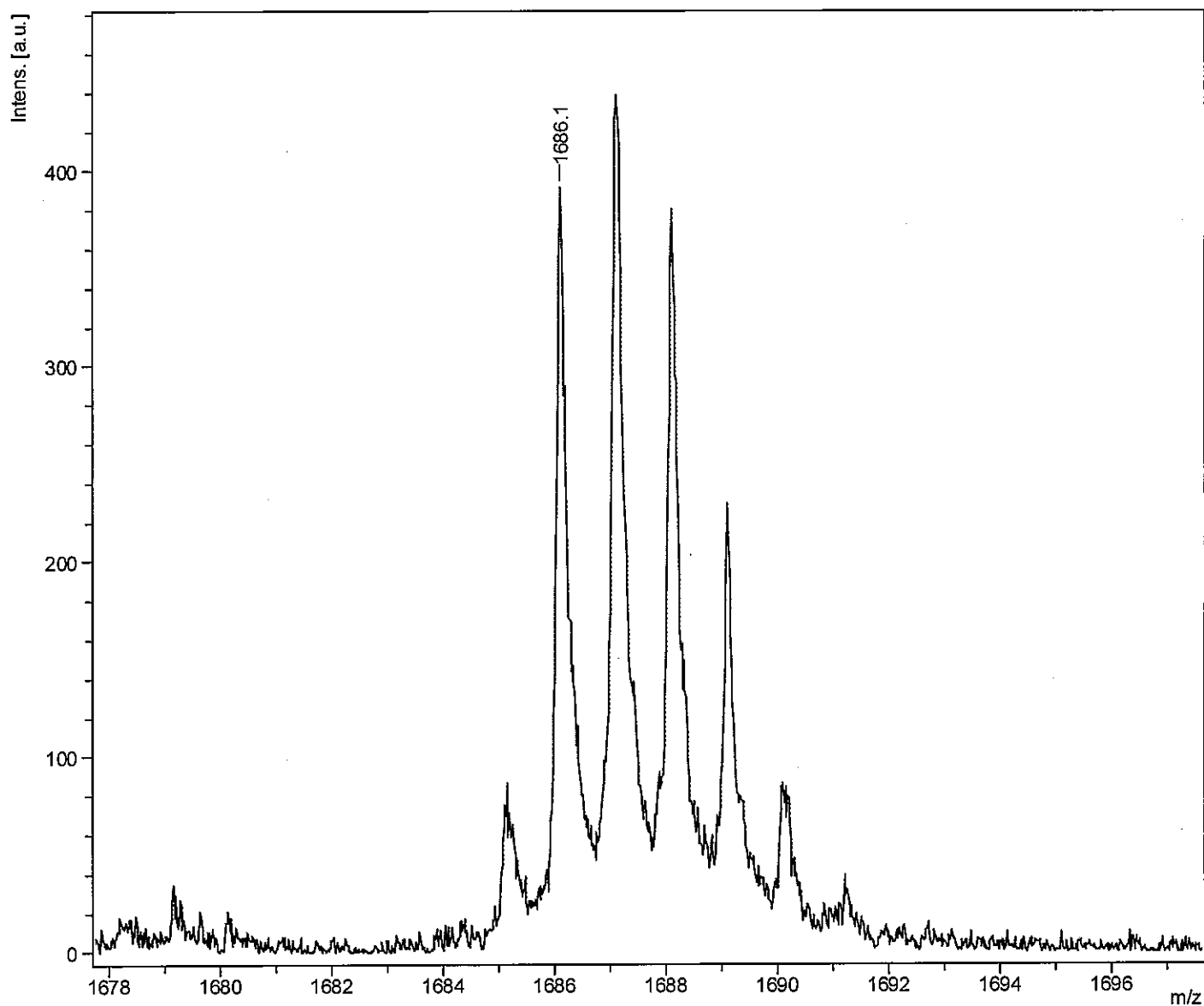
Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Figure S23. MALDI-TOF mass spectrum of **1e**.

Comment 1

Comment 2



Acquisition Parameter

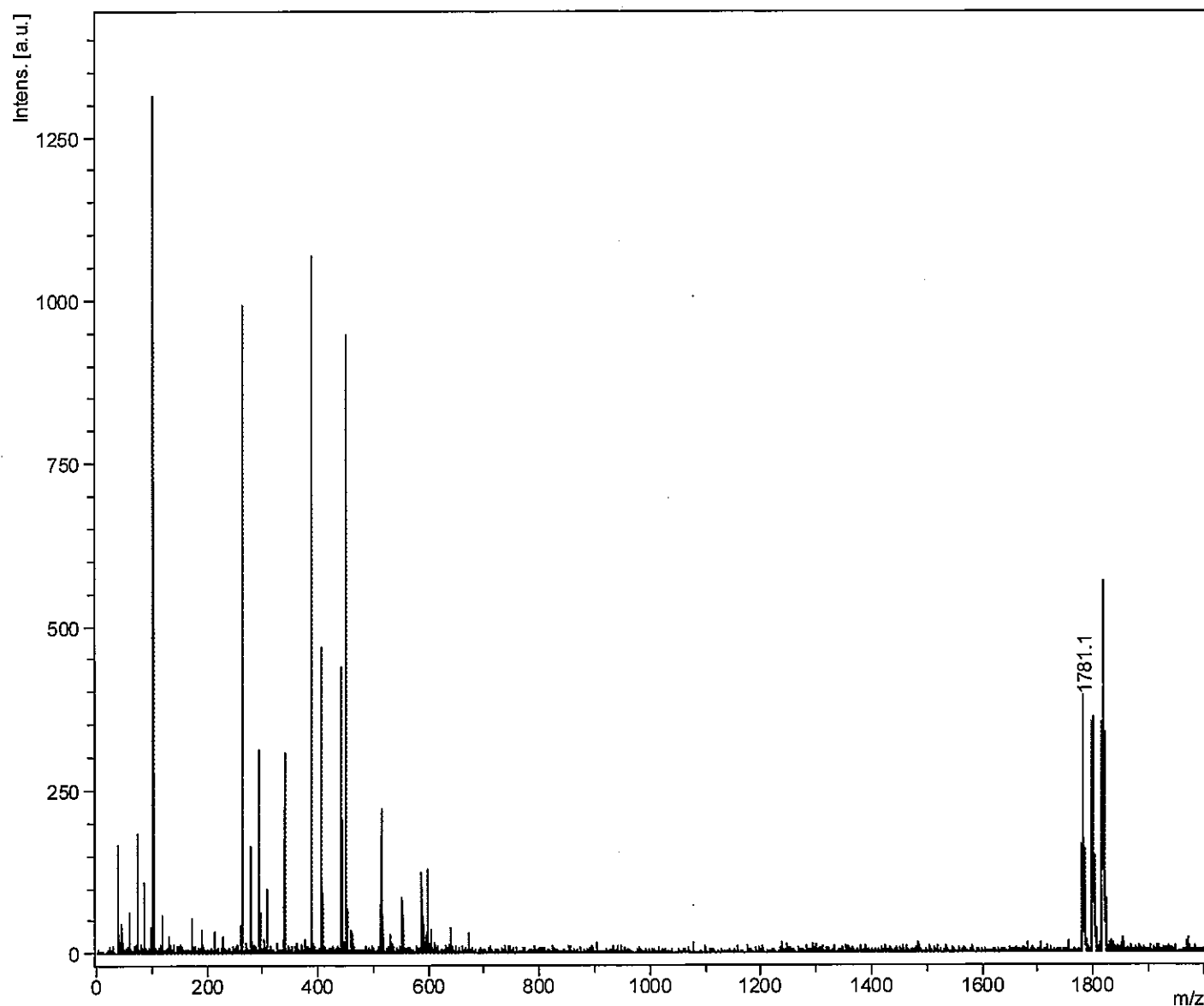
Date of acquisition 2016-10-06T13:34:03.875+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

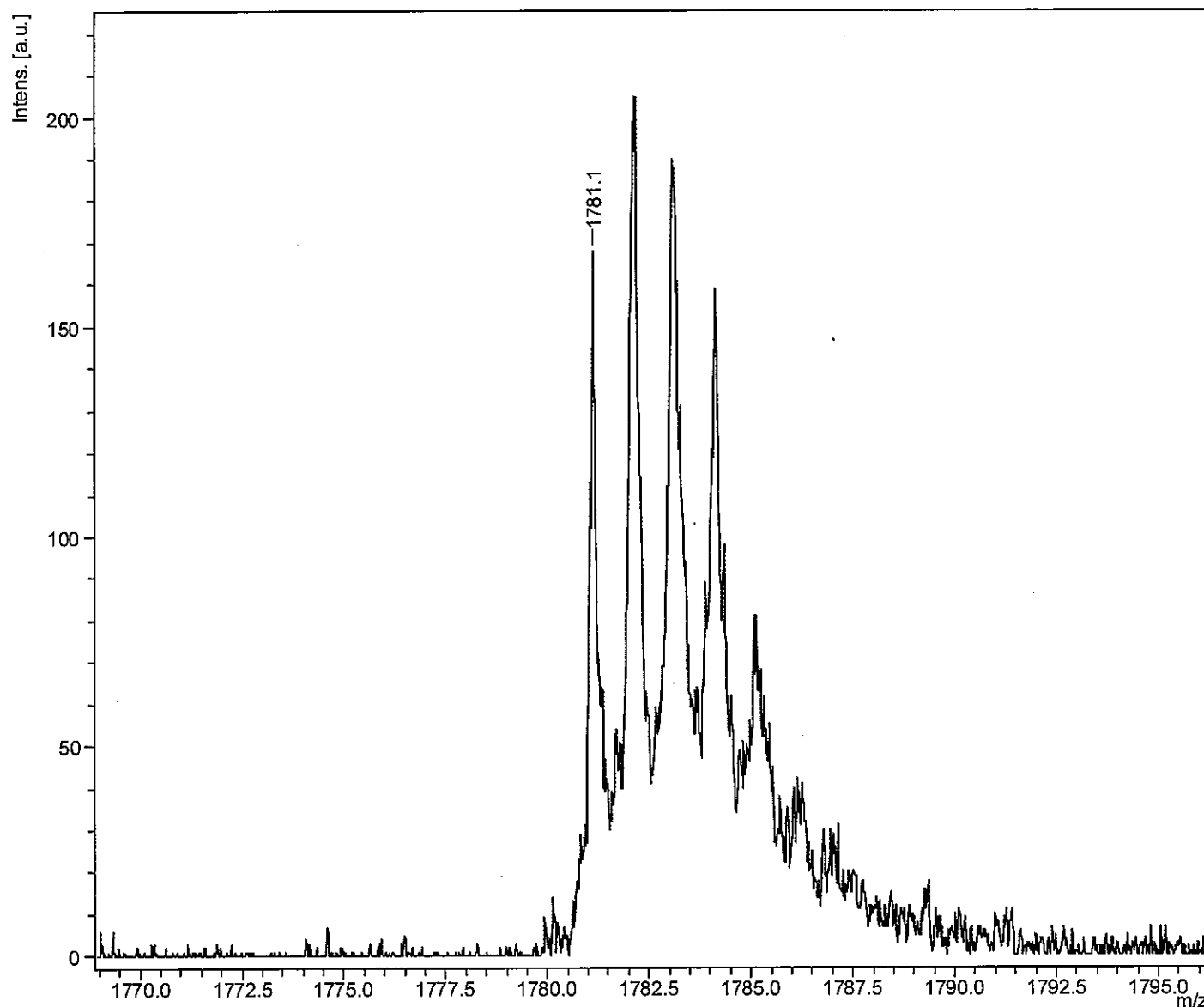
Date of acquisition 2016-10-06T13:37:43.531+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

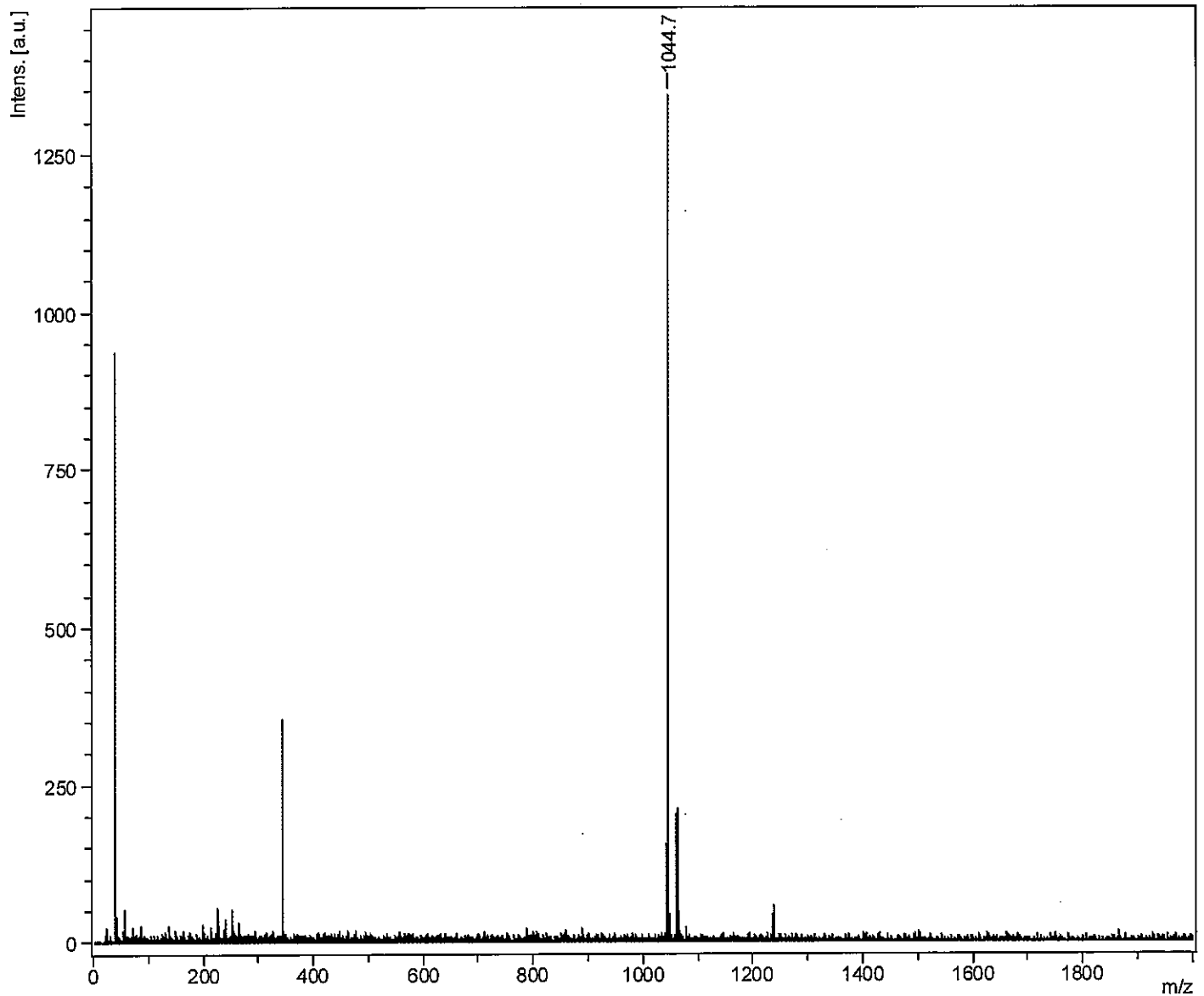
Date of acquisition 2016-10-06T13:37:43.531+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

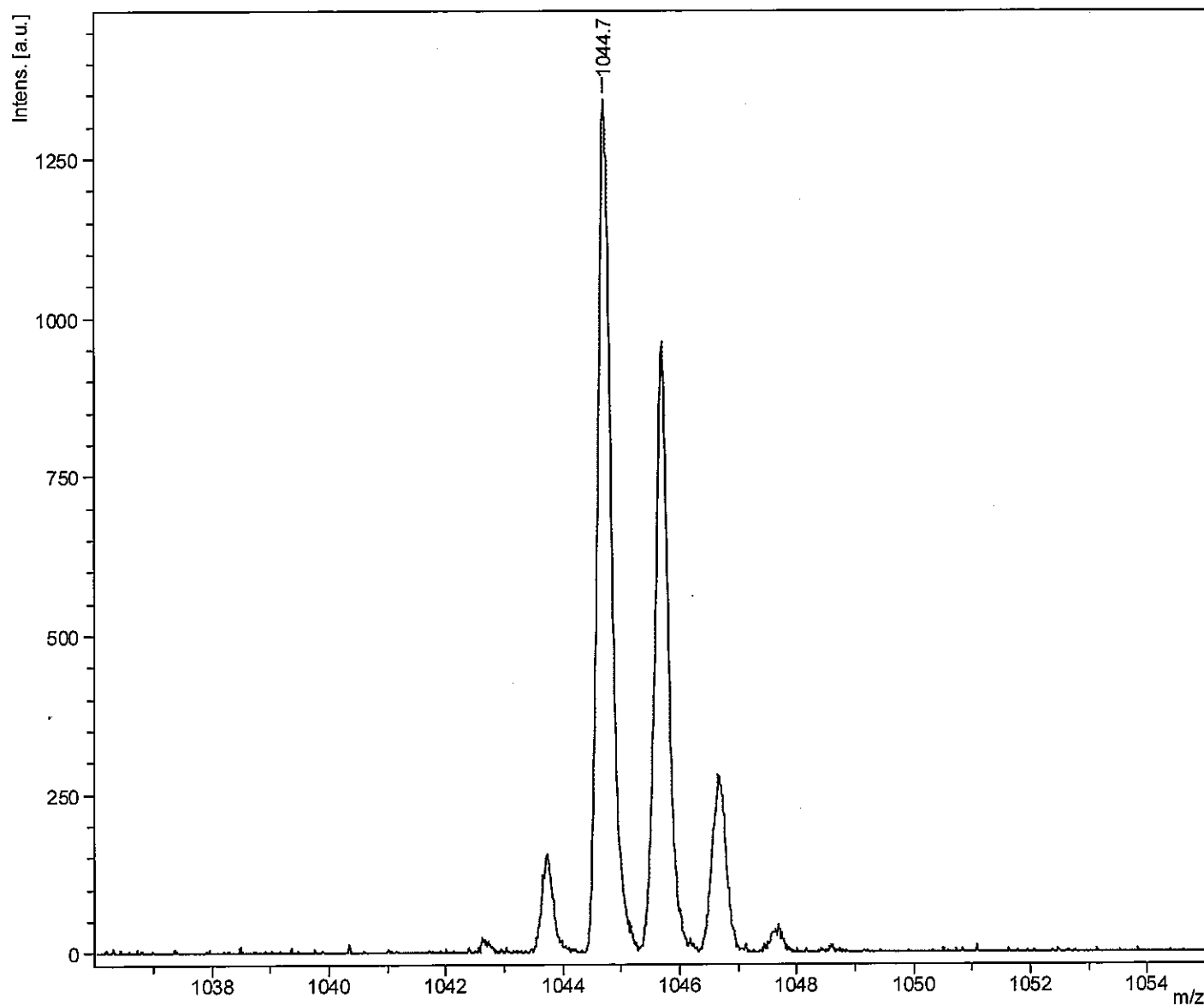
Date of acquisition 2017-01-18T19:38:51.687+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

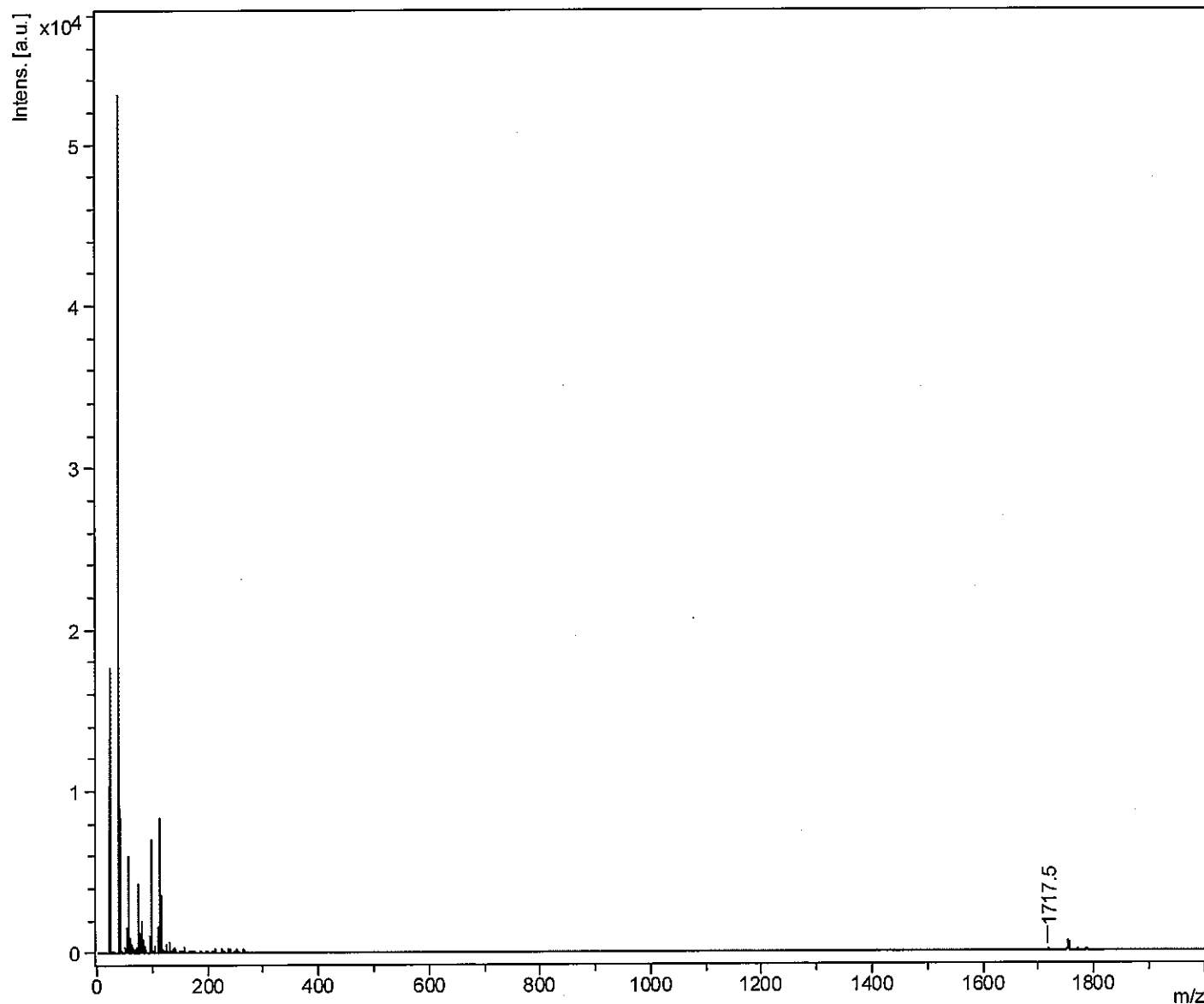
Date of acquisition 2017-01-18T19:38:51.687+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

Date of acquisition 2017-06-05T13:10:35.921+09:00
Acquisition method name D:\Methods\flexControl\Methods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

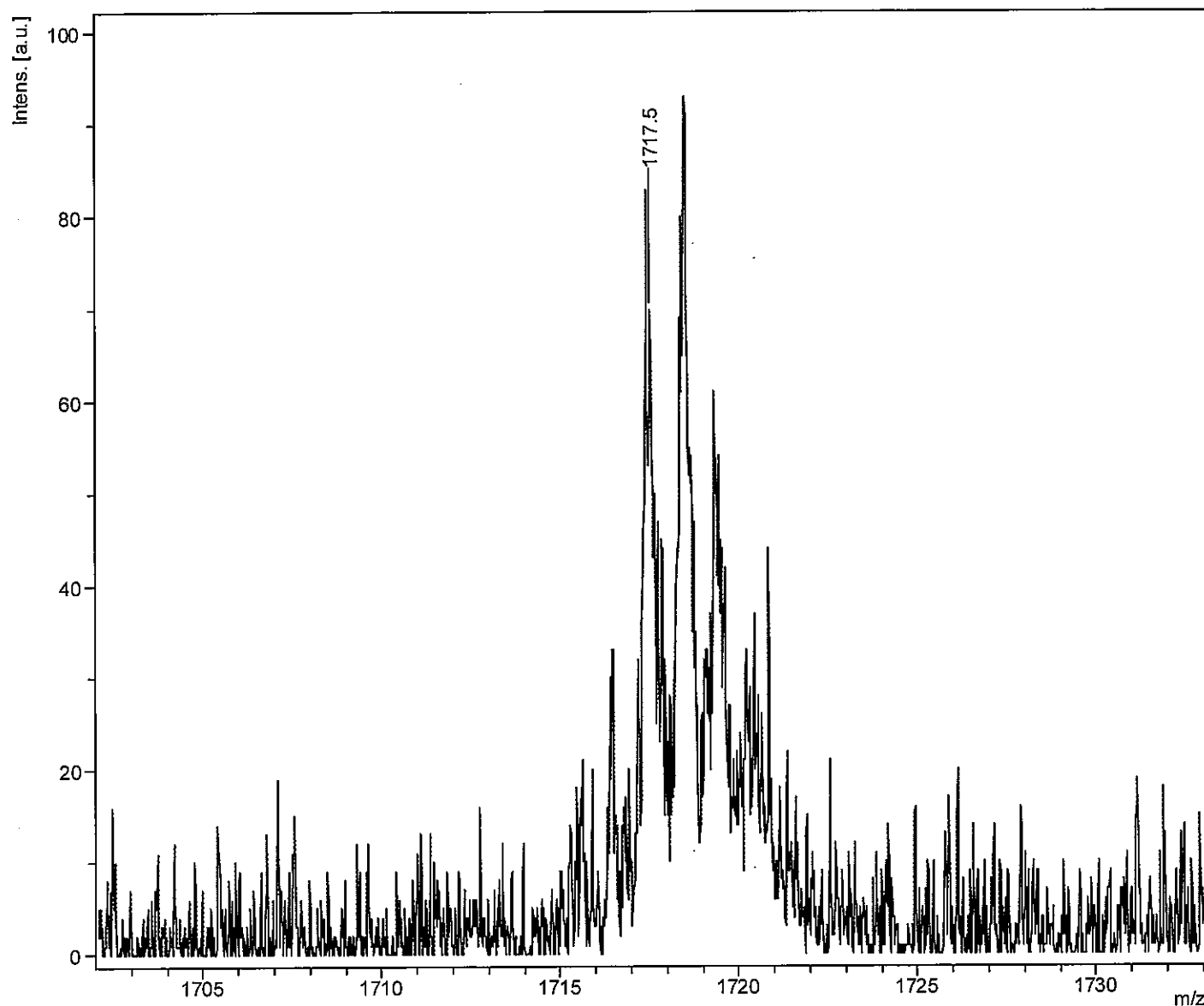
Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Figure S26. MALDI-TOF mass spectrum of **2b**.

Comment 1

Comment 2



Acquisition Parameter

Date of acquisition 2017-06-05T13:10:35.921+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

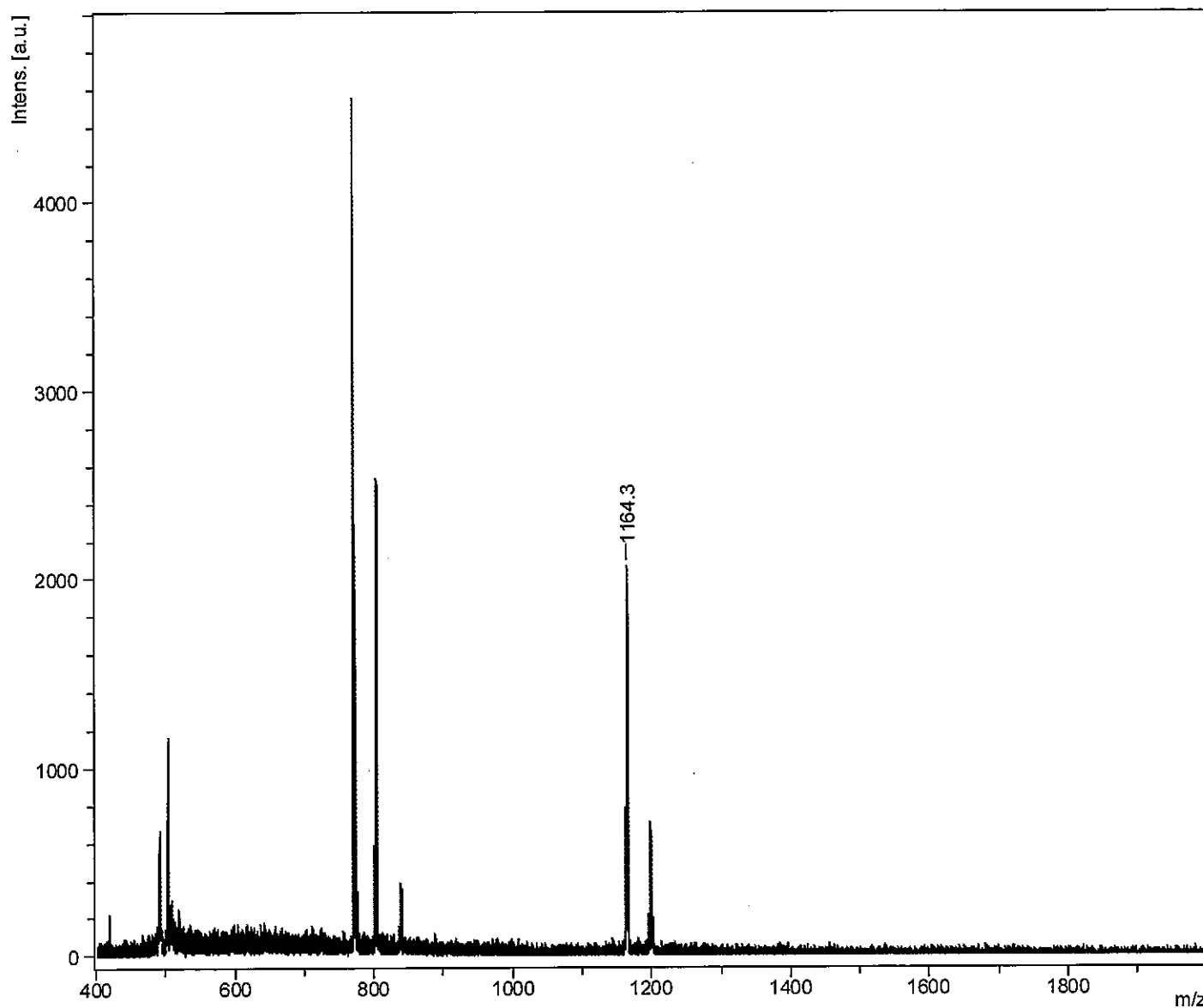
Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Figure S26. MALDI-TOF mass spectrum of **2b** (Molecular ion peaks).

Comment 1

Comment 2



Acquisition Parameter

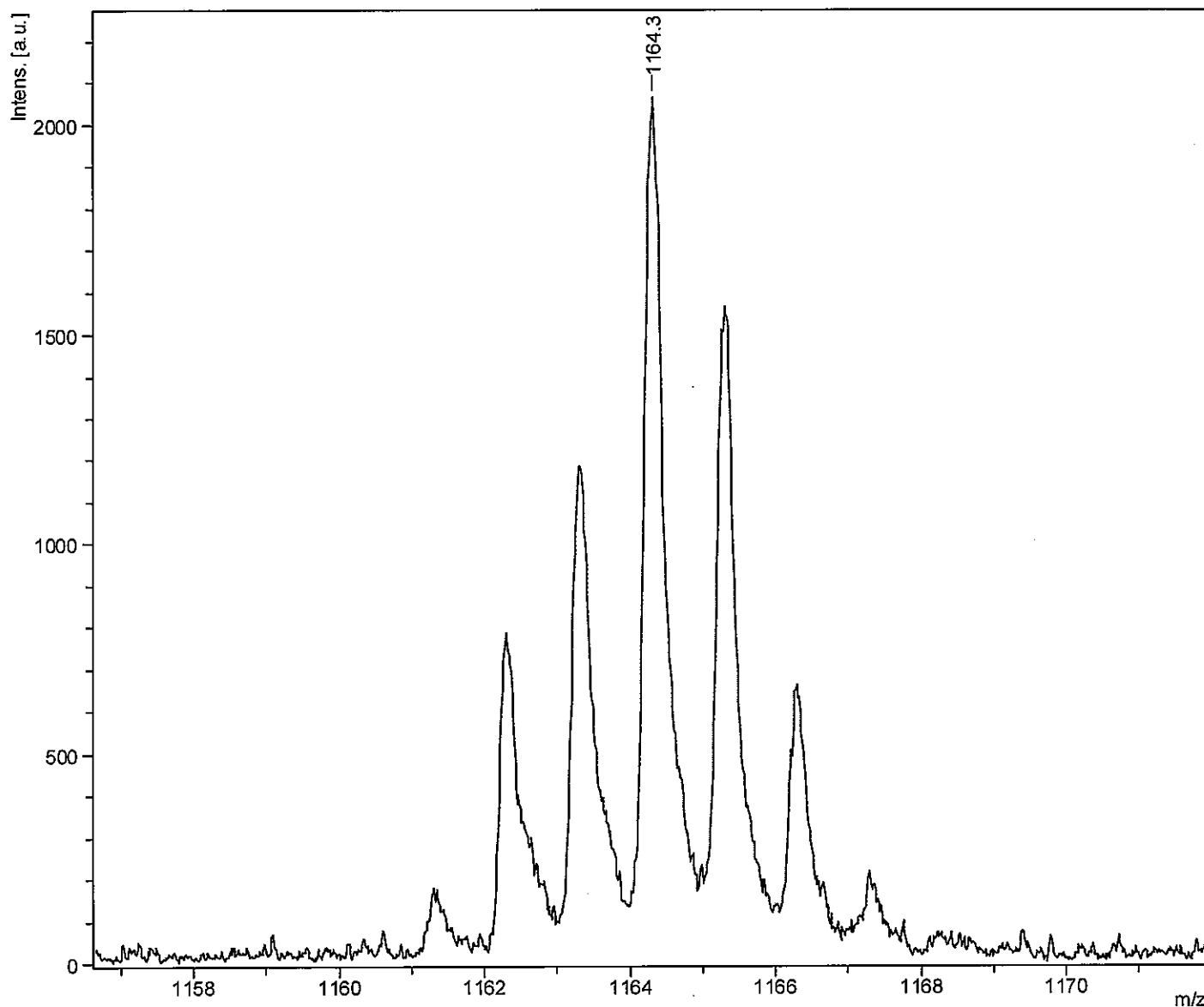
Date of acquisition 2017-06-05T15:56:11.281+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_700-4500_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

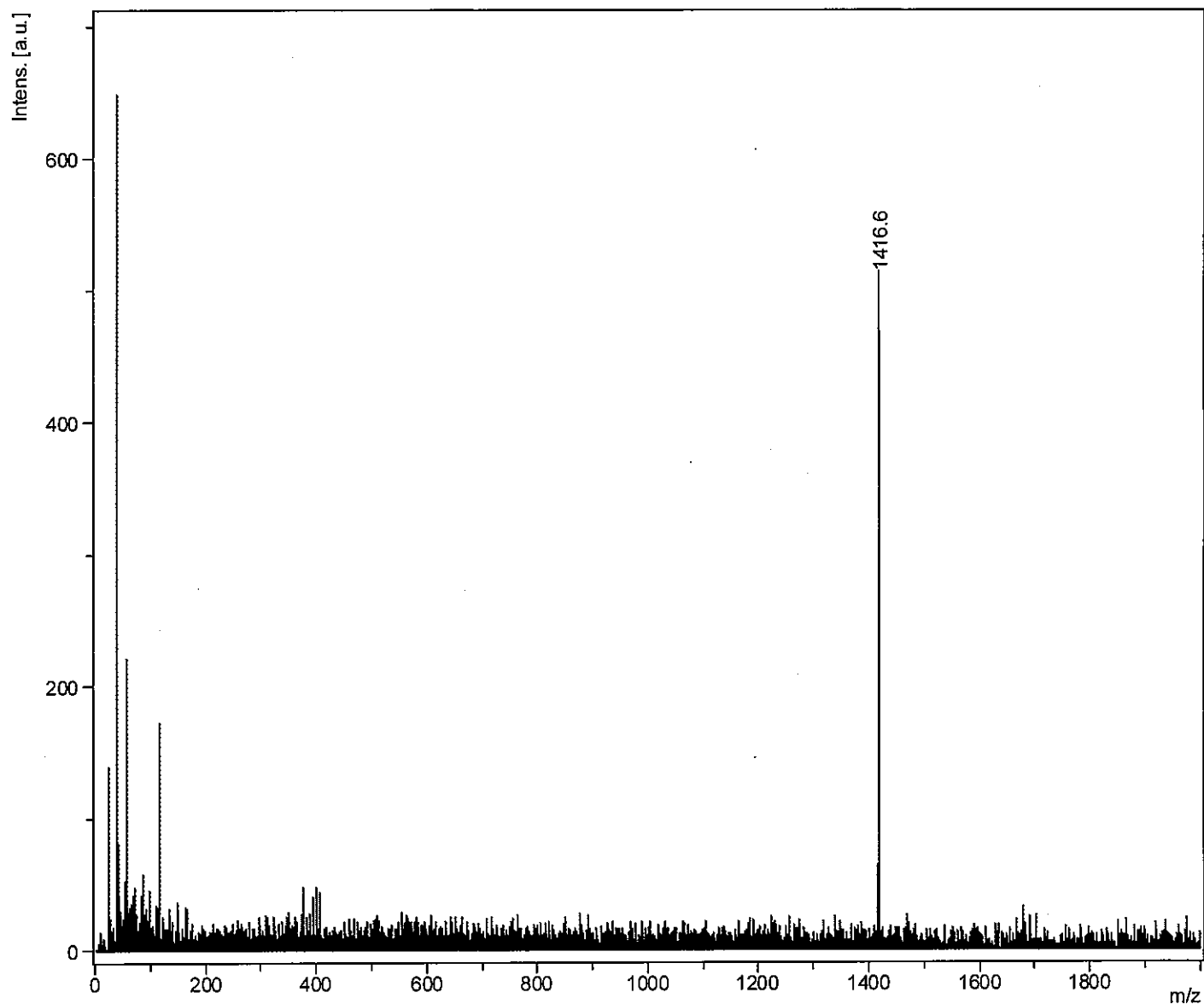
Date of acquisition 2017-06-05T15:56:11.281+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_700-4500_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

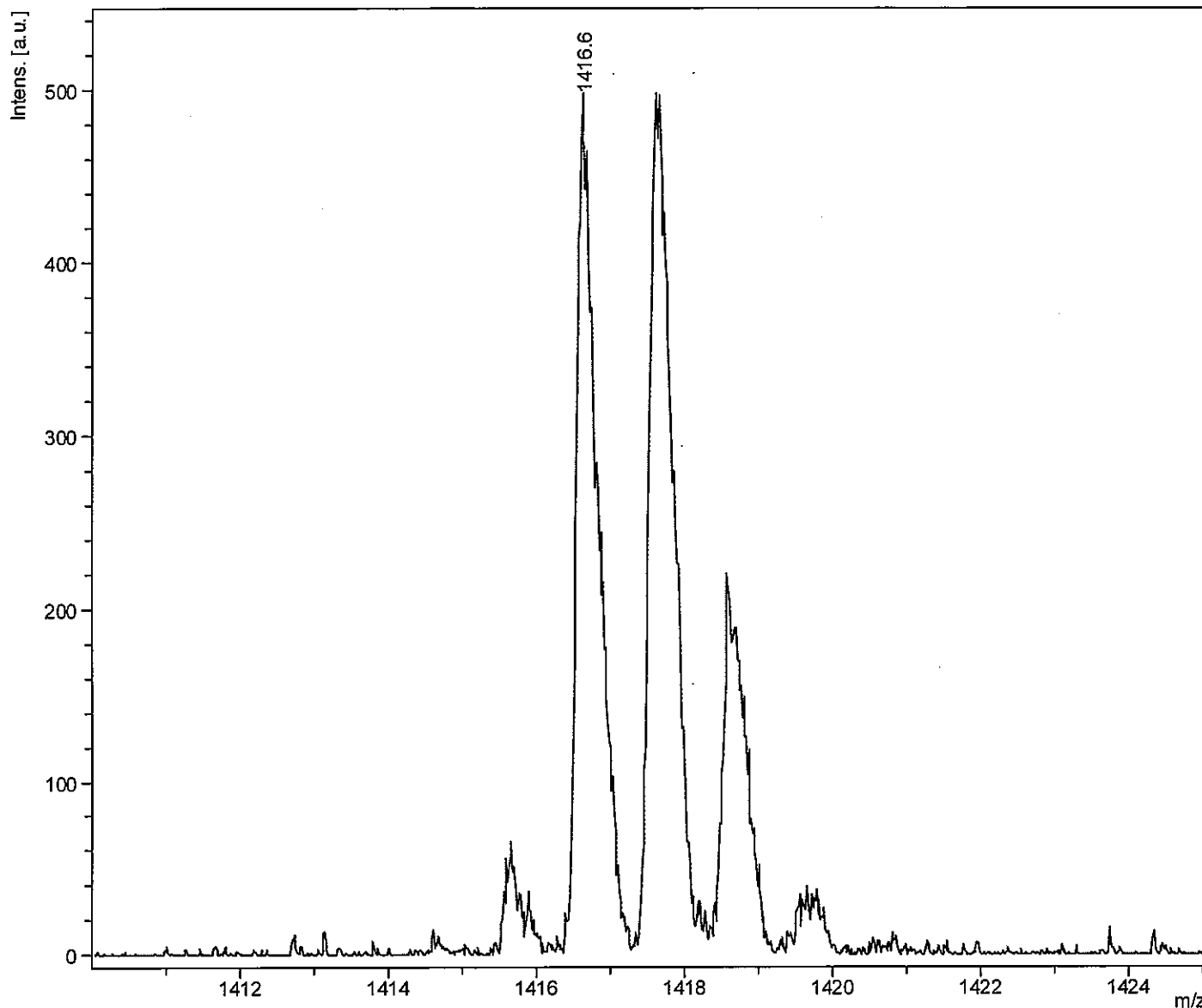
Date of acquisition 2017-06-05T11:26:13.812+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

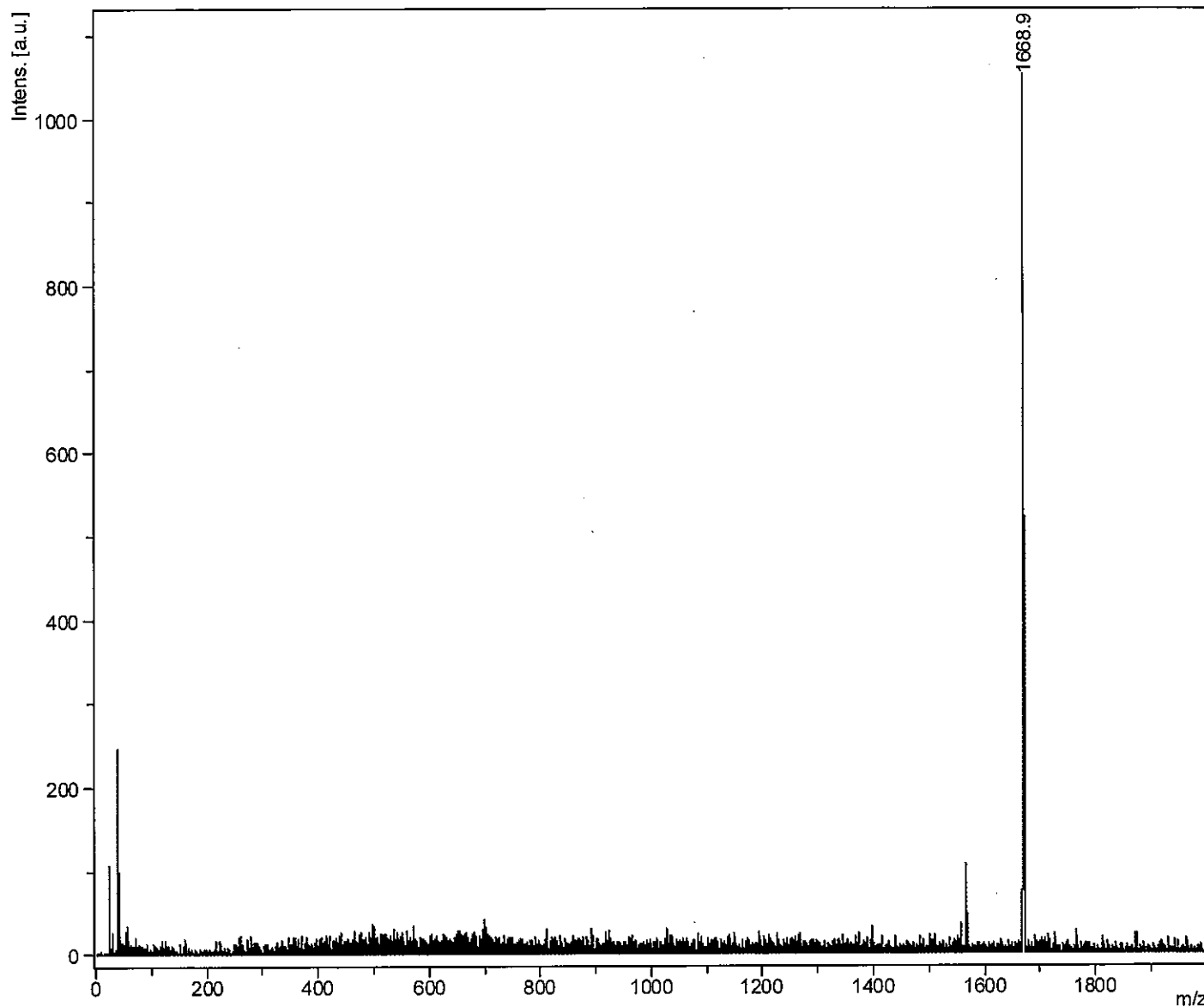
Date of acquisition 2017-06-05T11:26:13.812+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

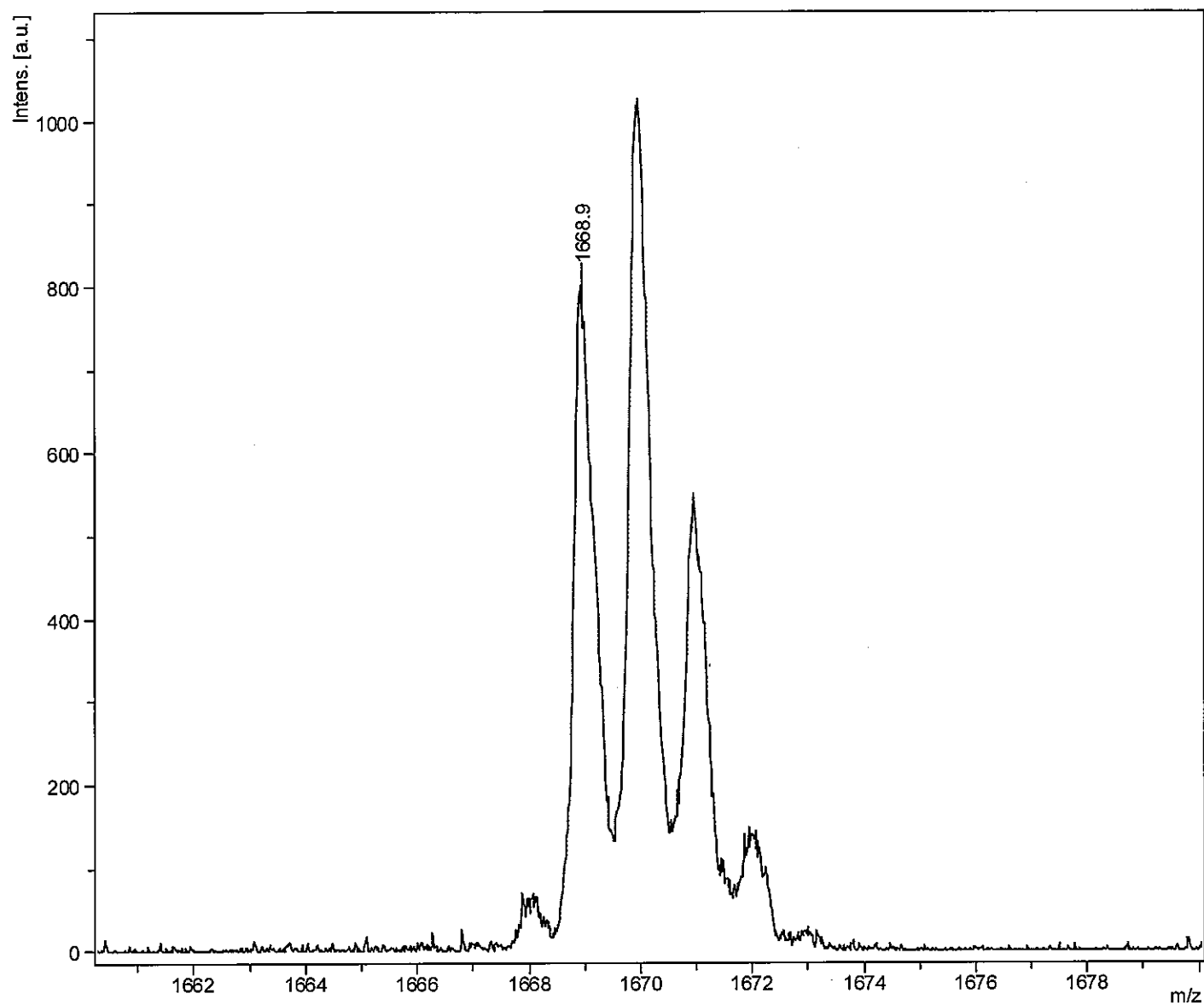
Date of acquisition 2017-06-05T11:28:34.437+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

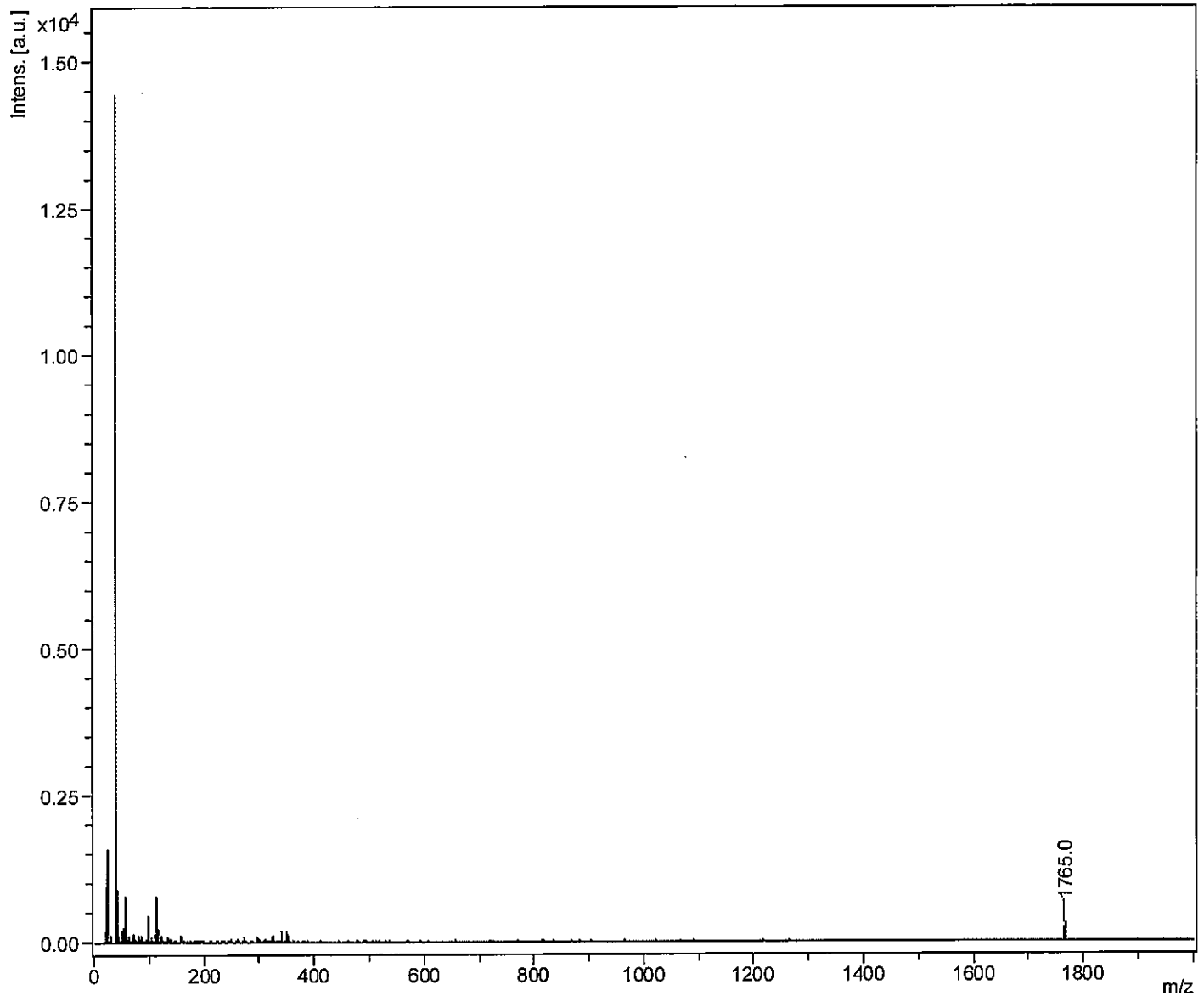
Date of acquisition 2017-06-05T11:28:34.437+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

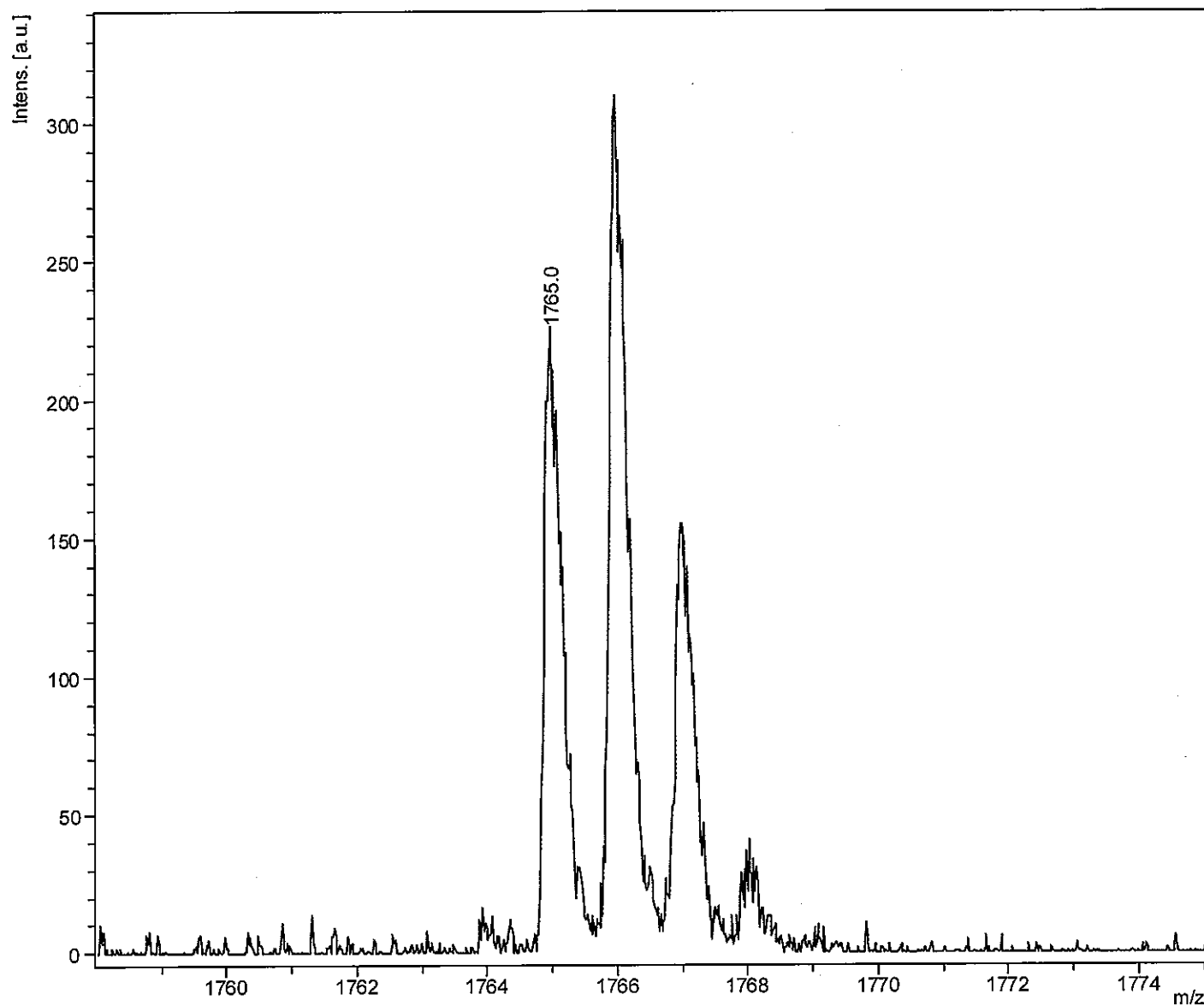
Date of acquisition 2017-06-05T11:32:45.937+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

Date of acquisition 2017-06-05T11:32:45.937+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

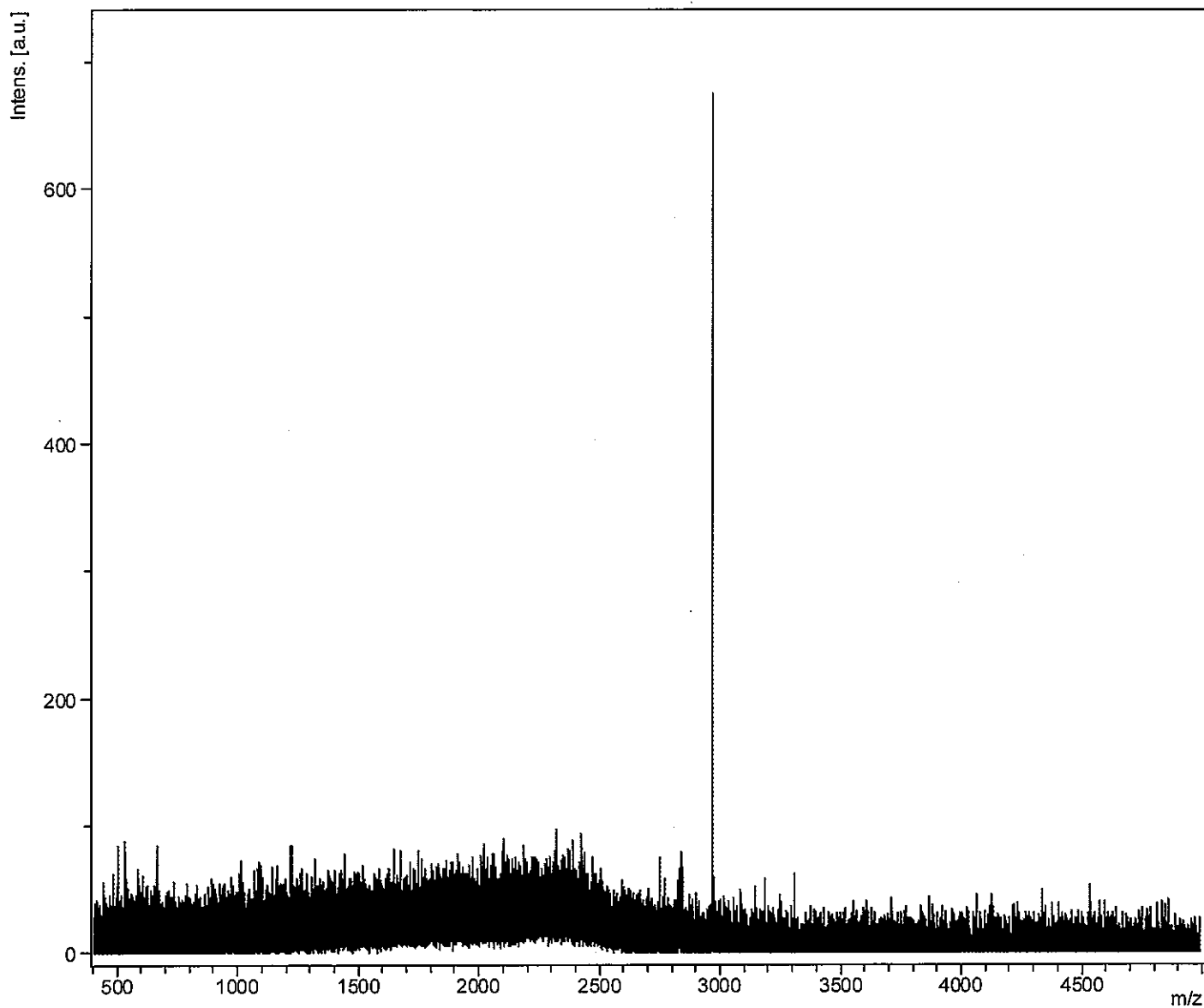
Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Figure S30. MALDI-TOF mass spectrum of 2f (Molecular ion peaks).

Comment 1

Comment 2



Acquisition Parameter

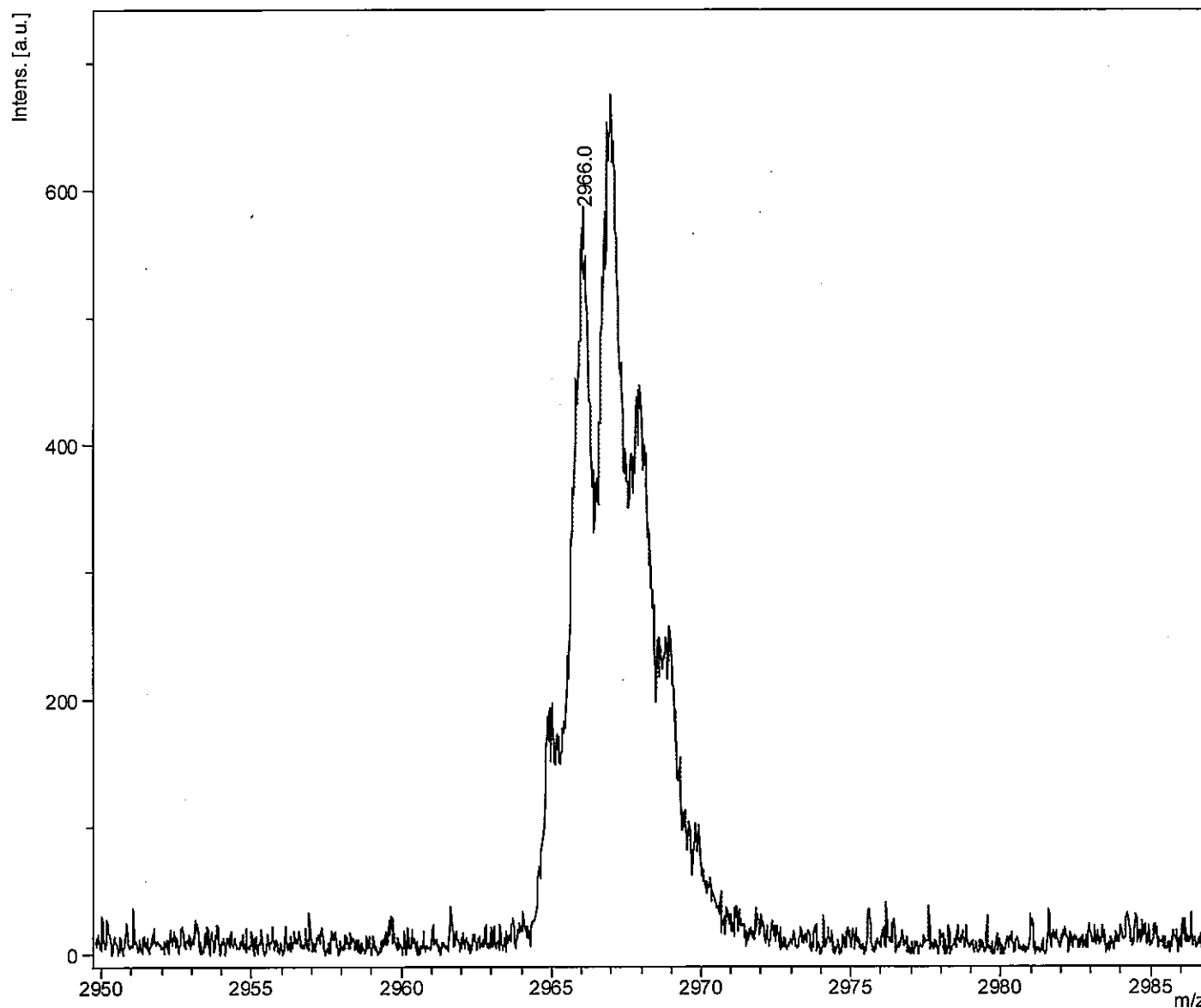
Date of acquisition 2017-06-05T16:14:28.453+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_700-4500_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

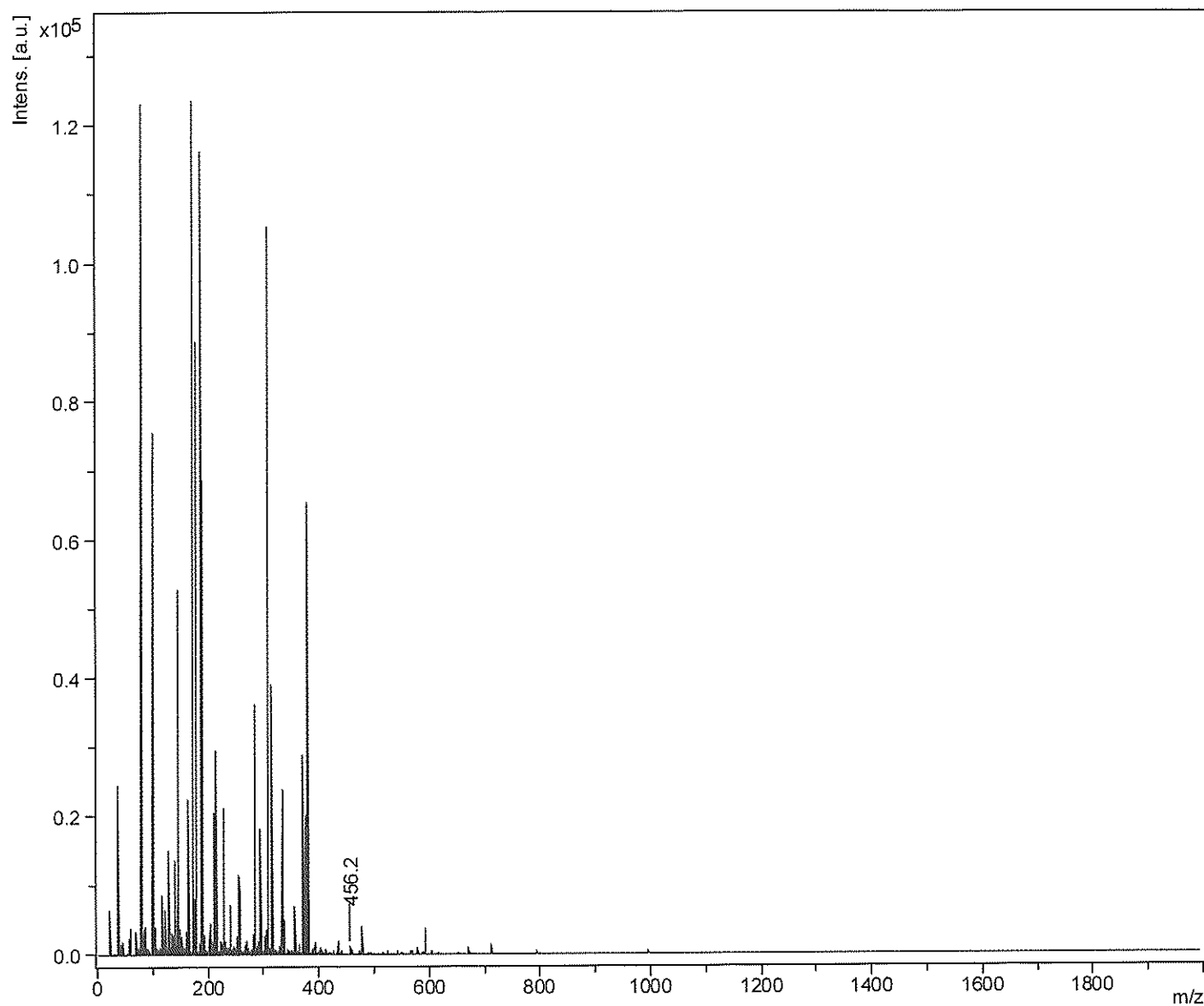
Date of acquisition 2017-06-05T16:14:28.453+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_700-4500_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

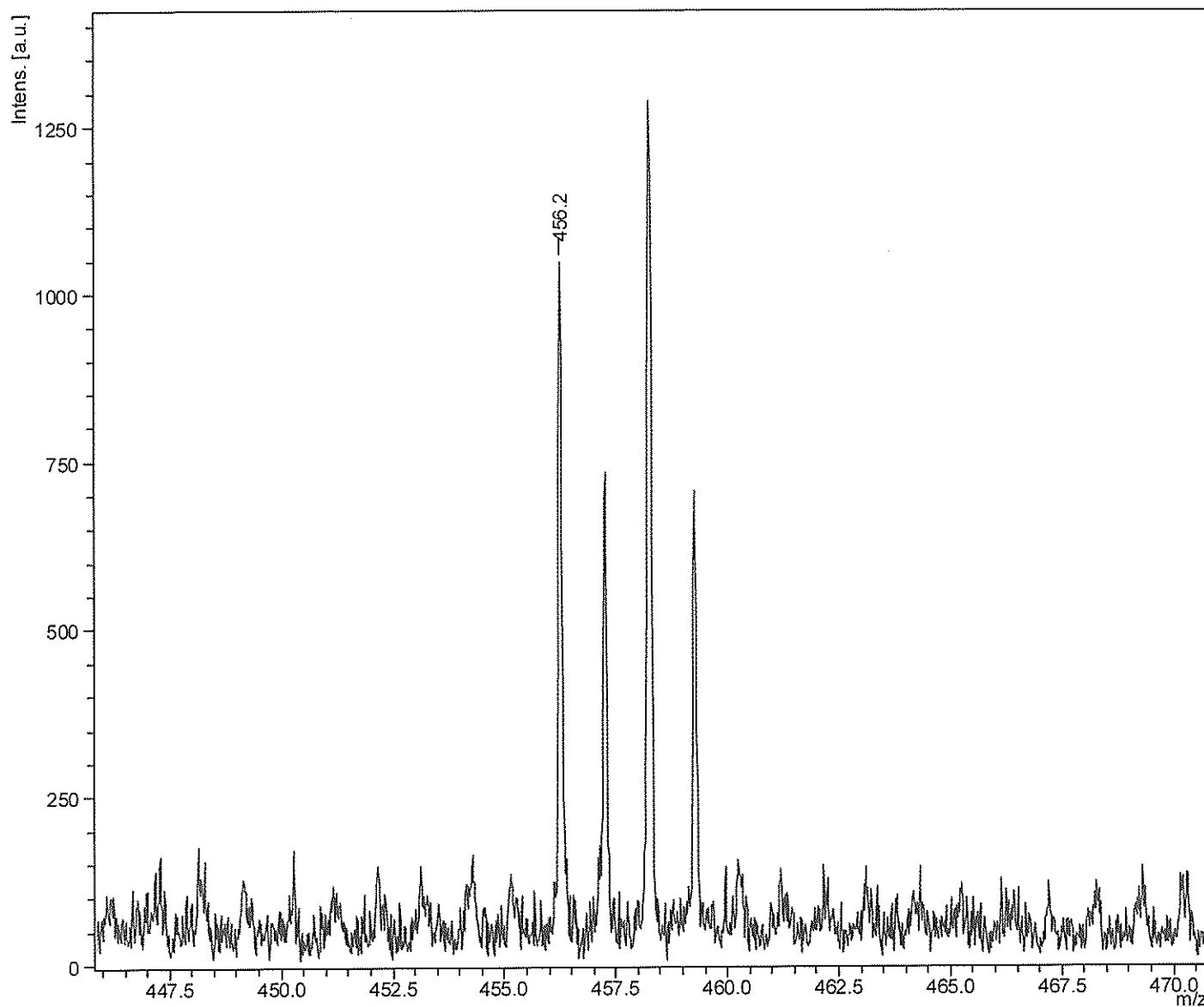
Date of acquisition 2017-07-31T15:26:52.656+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

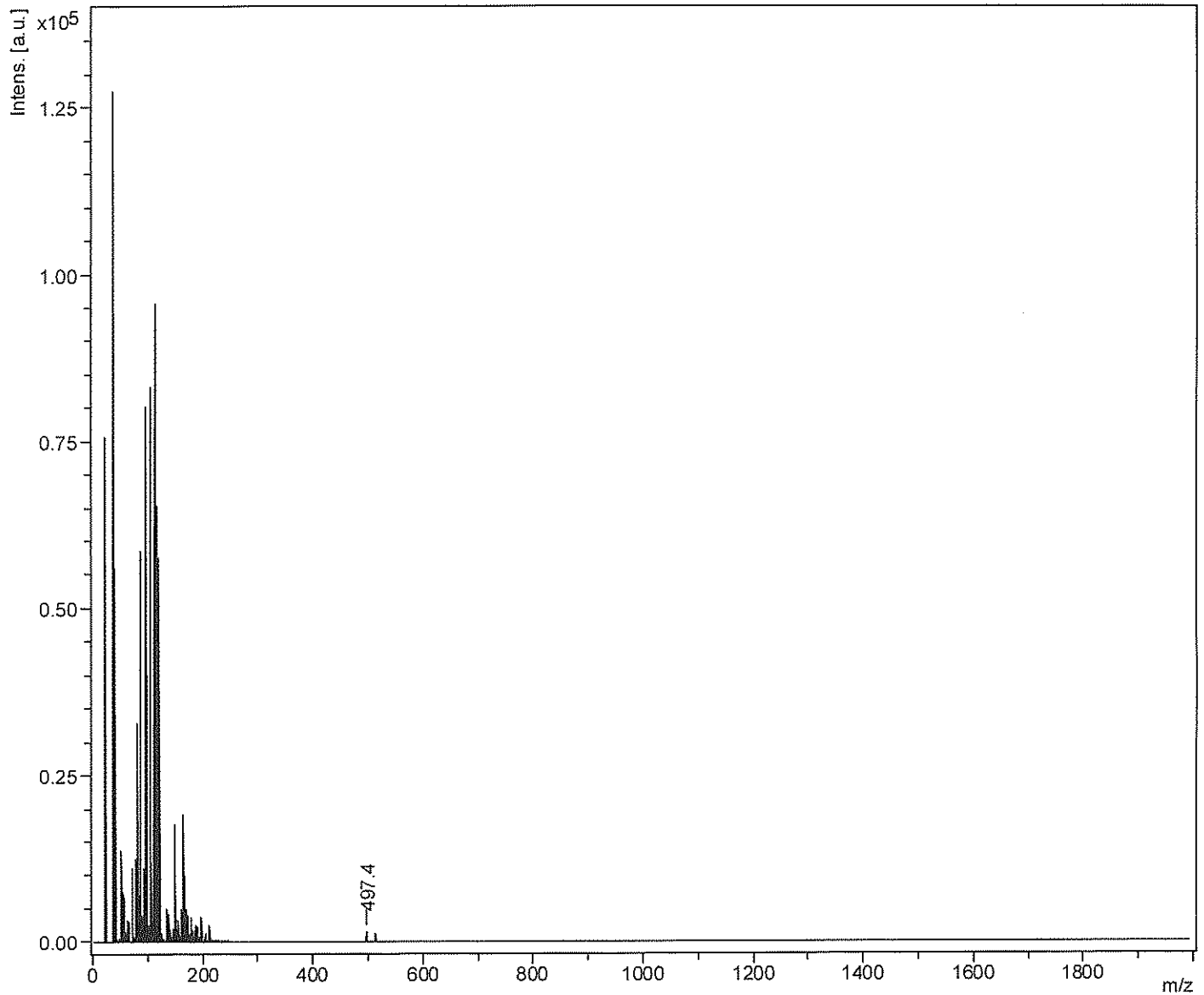
Date of acquisition 2017-07-31T15:26:52.656+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

Date of acquisition 2017-07-31T15:39:46.125+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

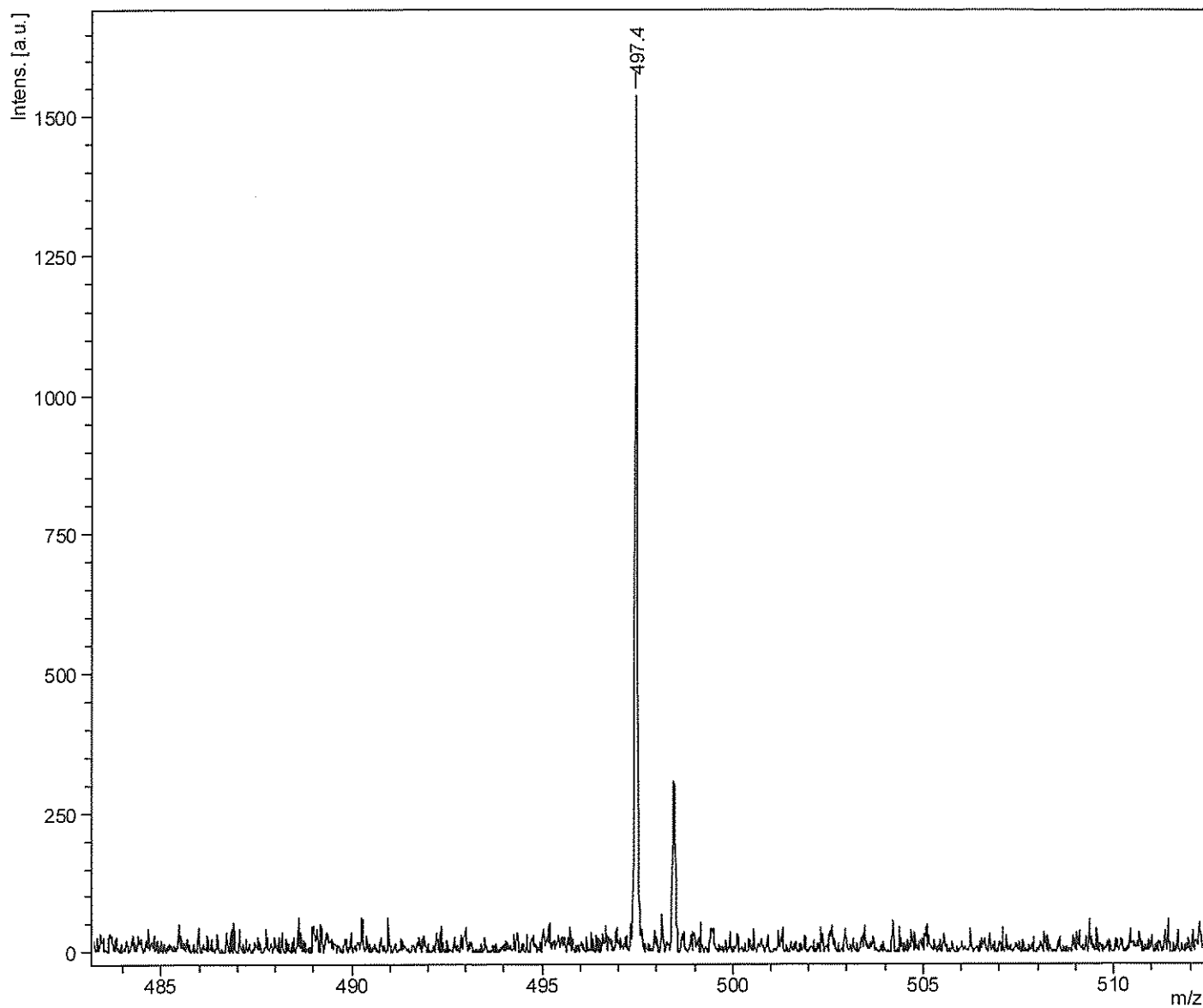
Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Figure S33. MALDI-TOF mass spectrum of 14.

Comment 1

Comment 2



Acquisition Parameter

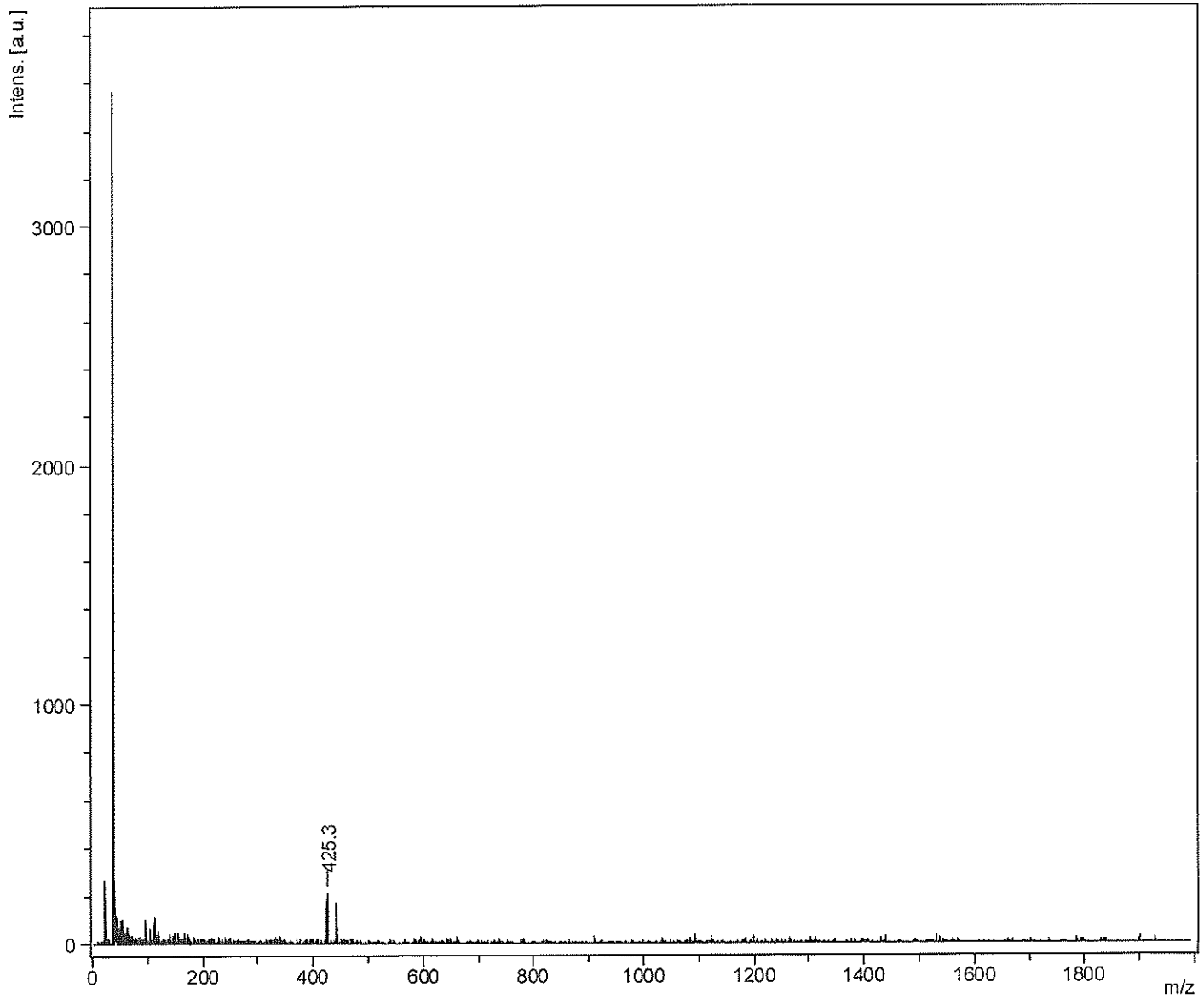
Date of acquisition 2017-07-31T15:39:46.125+09:00
Acquisition method name D:\Methods\flexControl\Methods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Comment 1

Comment 2



Acquisition Parameter

Date of acquisition 2017-07-31T15:34:53.203+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

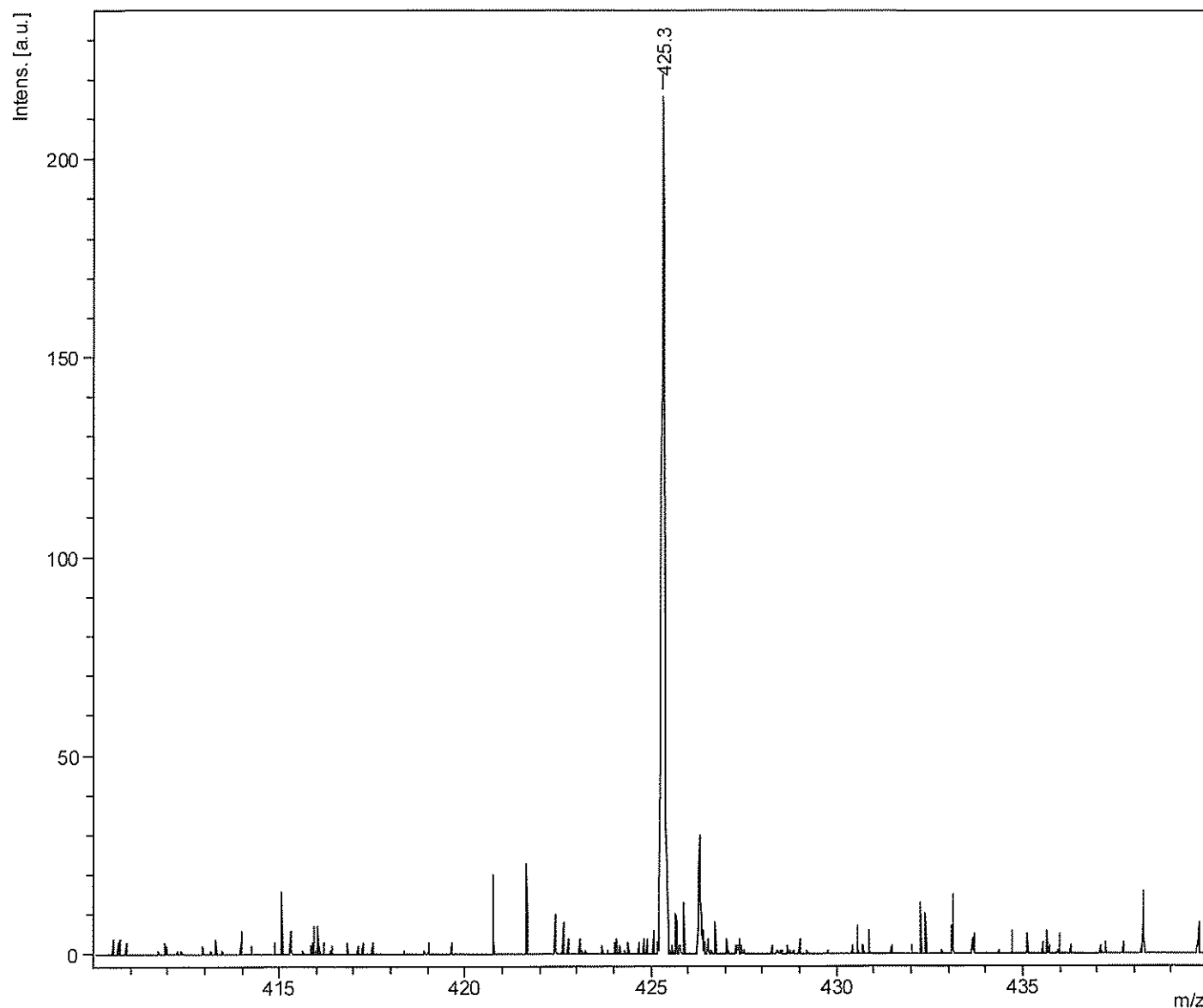
Instrument Info

User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

Figure S34. MALDI-TOF mass spectrum of 15.

Comment 1

Comment 2



Acquisition Parameter

Date of acquisition 2017-07-31T15:34:53.203+09:00
Acquisition method name D:\Methods\flexControlMethods\RP_0-2000_Da.par
Acquisition operation mode Reflector
Voltage polarity POS
Number of shots 500
Name of spectrum used for calibration
Calibration reference list used

Instrument Info

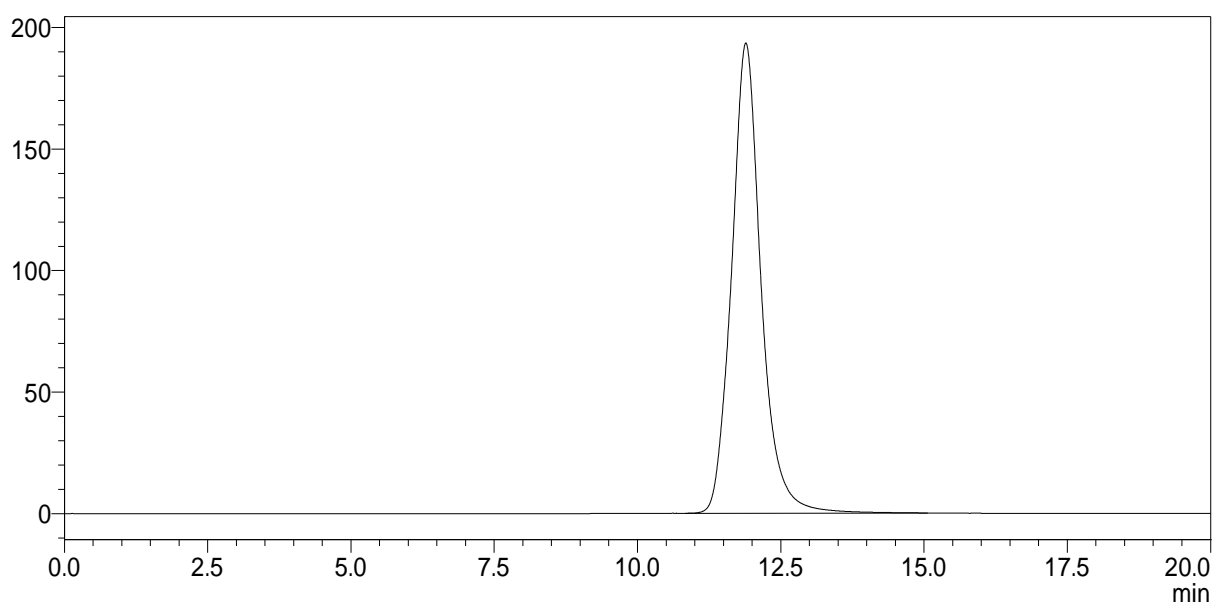
User DoshisyaUniv
Instrument FLEX-PC
Instrument type autoflex

<Sample>

Sample name : **1a**
Sample ID : takagi
File name : 20170529.lcd
Method : SubPc.lcm
: 1-1
Sample vol : 1000 uL
Date Meas. : 2017/06/07 14:37:56
Date Anal. : 2017/06/07 14:57:59

<Chromatogram>

mV



<Peak List>

Detector A Ch2

| Peak# | Ret. time | Area | Hight | Conc. | Area% |
|-------|-----------|---------|--------|-------|---------|
| 1 | 11.890 | 6923546 | 193529 | 0.000 | 100.000 |
| Sum | | 6923546 | 193529 | | 100.000 |

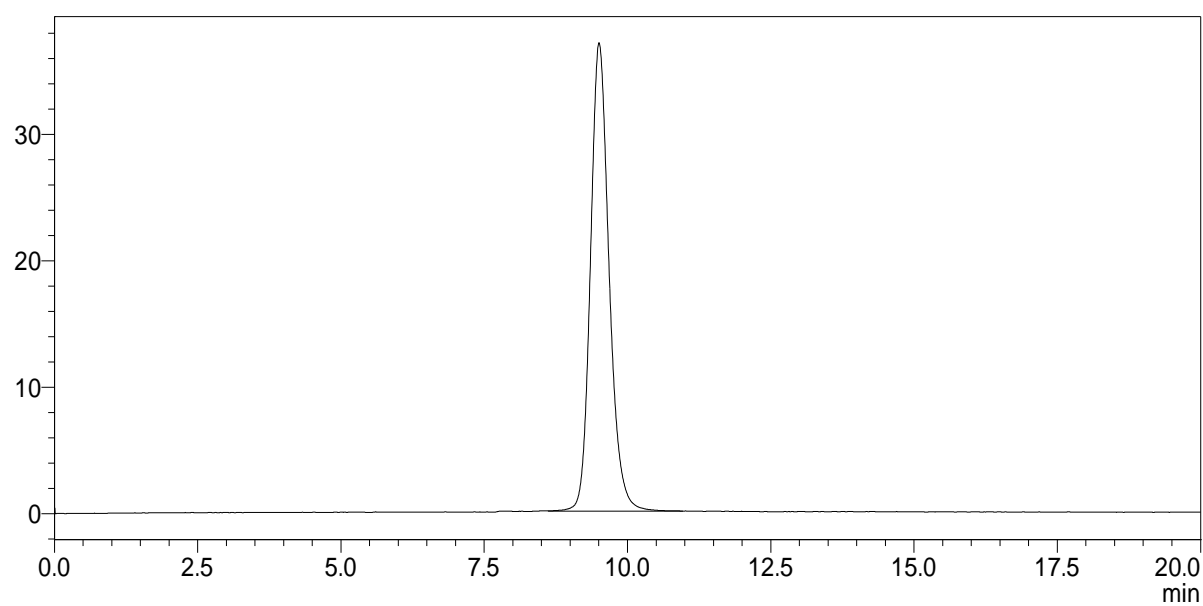
Figure S35. HPLC chart of **1a**.

<Sample>

Sample name : **1b**
 Sample ID : takagi
 File name : 20170545.lcd
 Method : SubPc.lcm
 :
 Sample vol : 1-1
 Date Meas. : 1000 uL
 Date Anal. : 2017/06/08 17:23:10
 : 2017/06/08 17:43:13

<Chromatogram>

mV

**<Peak List>**

Detector A Ch2 680 nm

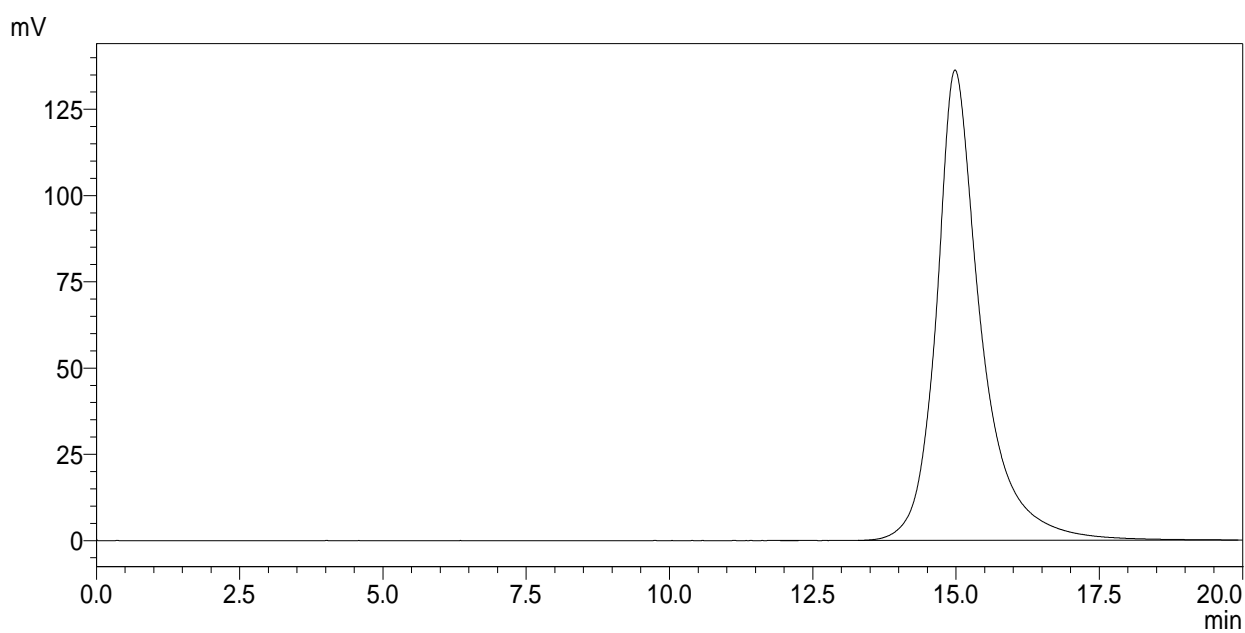
| Peak# | Ret Time | Area | Hight | Conc | Area% |
|-------|----------|--------|-------|-------|---------|
| 1 | 9.505 | 841556 | 37046 | 0.000 | 100.000 |
| Sum | | 841556 | 37046 | | 100.000 |

Figure S36. HPLC chart of **1b**.

<Sample>

Sample name : **1c**
Sample ID : takagi
File name : 20170546.lcd
Method : SubPc.lcm
:
Sample vol : 1-1
Date Meas. : 1000 uL
Date Anal. : 2017/06/08 18:38:10
: 2017/06/08 18:58:12

<Chromatogram>



<Peak List>

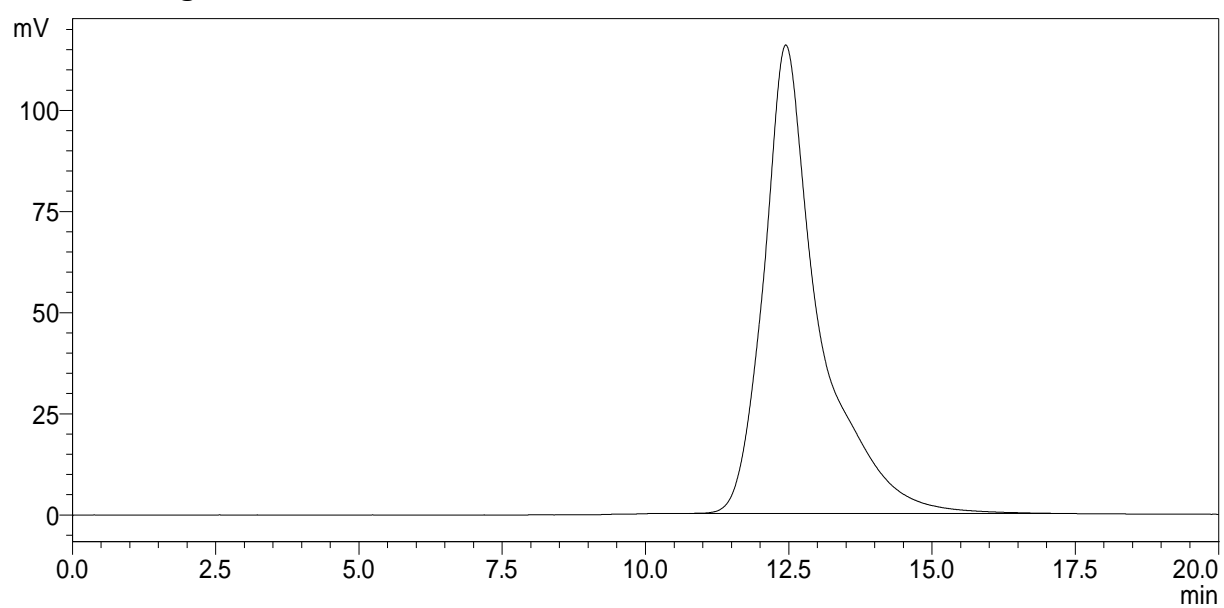
Detector A Ch2 680 nm

| Peak# | Ret. time | Area | Hight | Conc. | Area% |
|-------|-----------|---------|--------|-------|---------|
| 1 | 12.136 | 1124 | 71 | 0.000 | 0.015 |
| 2 | 14.983 | 7442767 | 136370 | 0.000 | 99.985 |
| Sum | | 7443891 | 136441 | | 100.000 |

Figure S37. HPLC chart of **1c**.

<Sample>

Sample name : **1d**
 Sample ID : takagi
 File name : 20170533.lcd
 Method : SubPc.lcm
 :
 Sample vol : 1-1
 Date Meas. : 1000 uL
 Date Anal. : 2017/06/07 17:02:47
 : 2017/06/07 17:22:49

<Chromatogram>**<Peak List>**

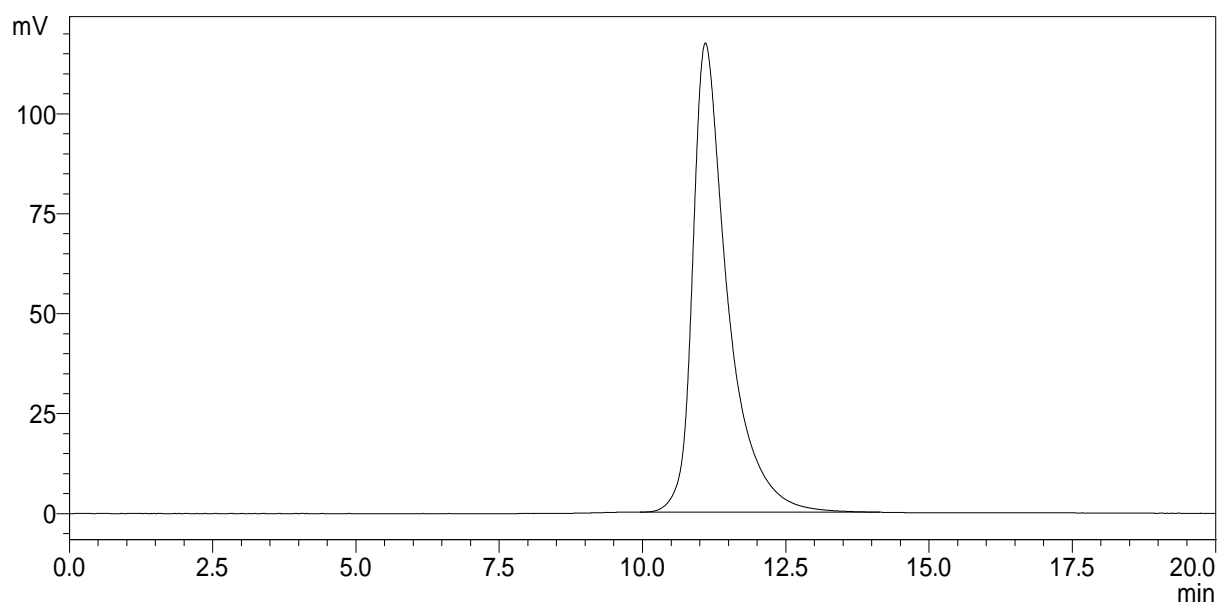
Detector A Ch2 680 nm

| Peak# | Ret. time | Area | Hight | Conc. | Area% |
|-------|-----------|---------|--------|-------|---------|
| 1 | 12.448 | 7782431 | 115829 | 0.000 | 100.000 |
| Sum | | 7782431 | 115829 | | 100.000 |

Figure S38. HPLC chart of **1d**.

<Sample>

Sample name : **1e**
 Sample ID : takagi
 File name : 20170534.lcd
 Method : SubPc.lcm
 :
 Sample vol : 1-1
 Date Meas. : 1000 uL
 Date Anal. : 2017/06/07 17:36:20 :
 2017/06/07 17:56:22

<Chromatogram>**<Peak List>**

Detector A Ch2 680 nm

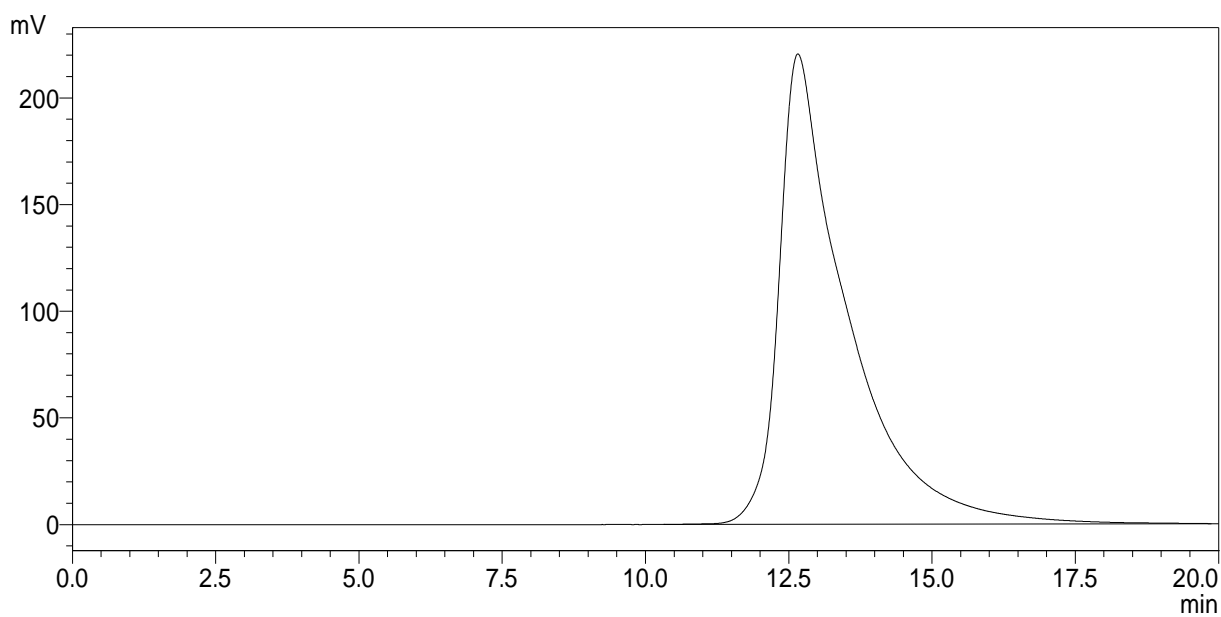
| Peak# | Ret. time | Area | Hight | Conc. | Area% |
|-------|-----------|---------|--------|-------|---------|
| 1 | 11.099 | 5005683 | 117405 | 0.000 | 100.000 |
| Sum | | 5005683 | 117405 | | 100.000 |

Figure S39. HPLC chart of **1e**.

<Sample>

Sample name : **1f**
 Sample ID : takagi
 File name : 20170535.lcd
 Method : SubPc.lcm
 :
 Sample vol : 1-1
 Date Meas. : 1000 uL
 Date Anal. : 2017/06/07 18:01:46 :
 2017/06/07 18:21:48

<Chromatogram>



<Peak List>

Detector A Ch2 680 nm

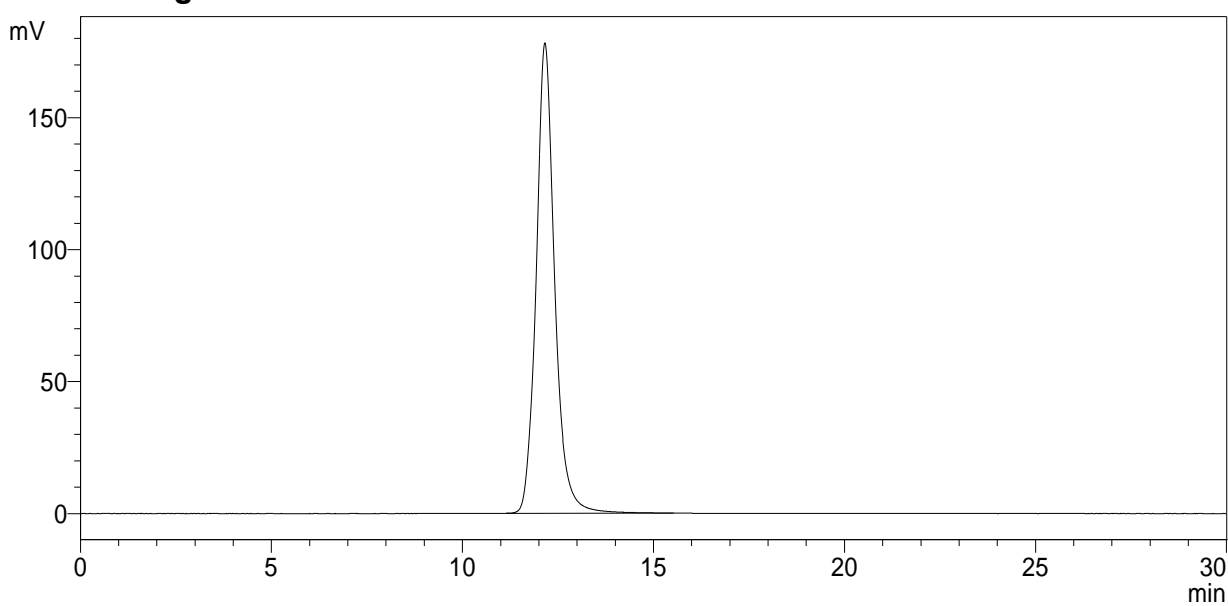
| Peak# | Ret. time | Area | Hight | Conc. | Area% |
|-------|-----------|----------|--------|-------|---------|
| 1 | 10.600 | 1599 | 116 | 0.000 | 0.009 |
| 2 | 12.660 | 18247509 | 220500 | 0.000 | 99.991 |
| Sum | | 18249108 | 220616 | | 100.000 |

Figure S40. HPLC chart of **1f**.

<Sample>

Sample name : **2a**
Sample ID : takagi
File name : 20170543.lcd
Method : SubPc.lcm
:
Sample vol : 1-1
Date Meas. : 1000 uL
Date Anal. : 2017/06/08 14:35:06
: 2017/06/08 15:05:08

<Chromatogram>



<Peak List>

Detector A Ch2 680 nm

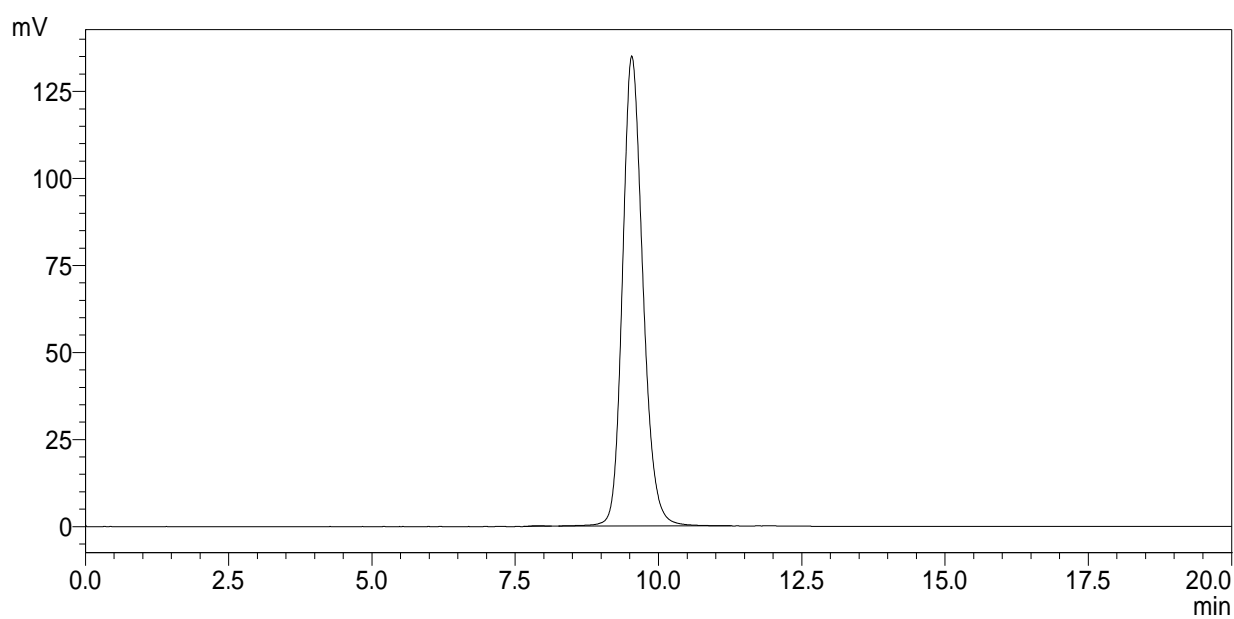
| Peak# | Ret. time | Area | Hight | Conc. | Area% |
|-------|-----------|---------|--------|-------|---------|
| 1 | 12.156 | 5983049 | 178217 | 0.000 | 100.000 |
| Sum | | 5983049 | 178217 | | 100.000 |

Figure S41. HPLC chart of **2a**.

<Sample>

Sample name : **2b**
 Sample ID : takagi
 File name : 20170537.lcd
 Method : SubPc.lcm
 :
 Sample vol : 1-1
 Date Meas. : 1000 uL
 Date Anal. : 2017/06/08 11:13:48
 : 2017/06/08 11:33:51

<Chromatogram>



<Peak List>

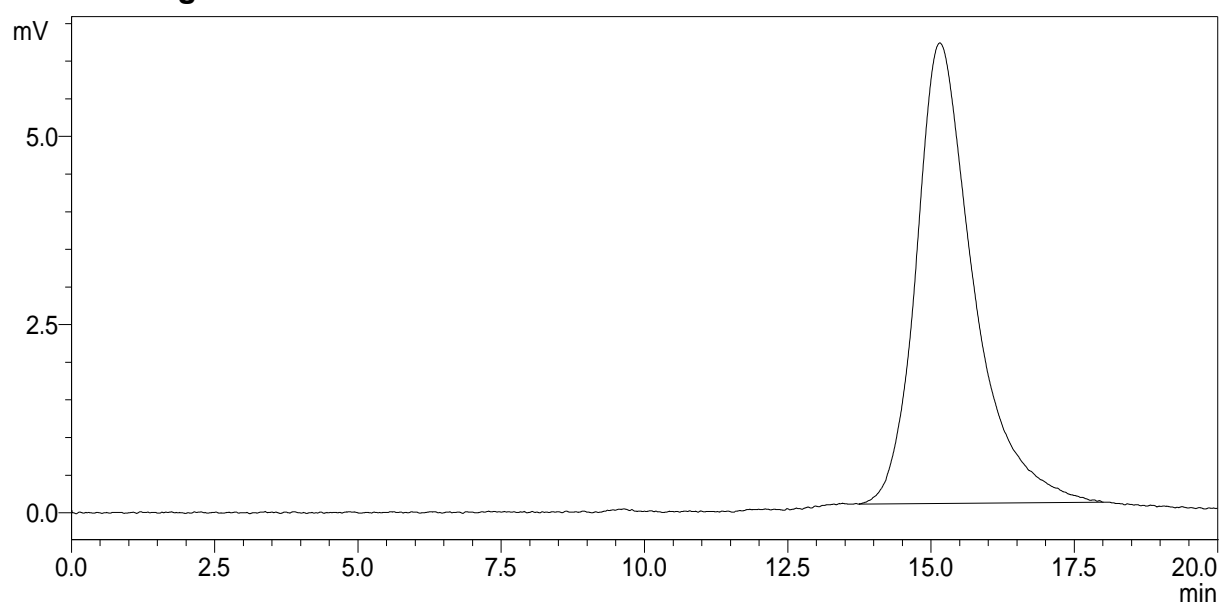
Detector A Ch2 680 nm

| Peak# | Ret. time | Area | Hight | Conc. | Area% |
|-------|-----------|---------|--------|-------|---------|
| 1 | 7.870 | 2564 | 179 | 0.000 | 0.077 |
| 2 | 9.535 | 3343790 | 135032 | 0.000 | 99.923 |
| Sum | | 3346354 | 135211 | | 100.000 |

Figure S42. HPLC chart of **2b**.

<Sample>

Sample name : **2c**
 Sample ID : takagi
 File name : 20170538.lcd
 Method : SubPc.lcm
 :
 Sample vol : 14
 Date Meas. : 1000 uL
 Date Anal. : 2017/06/08 11:38:08
 : 2017/06/08 11:58:10

<Chromatogram>**<Peak List>**

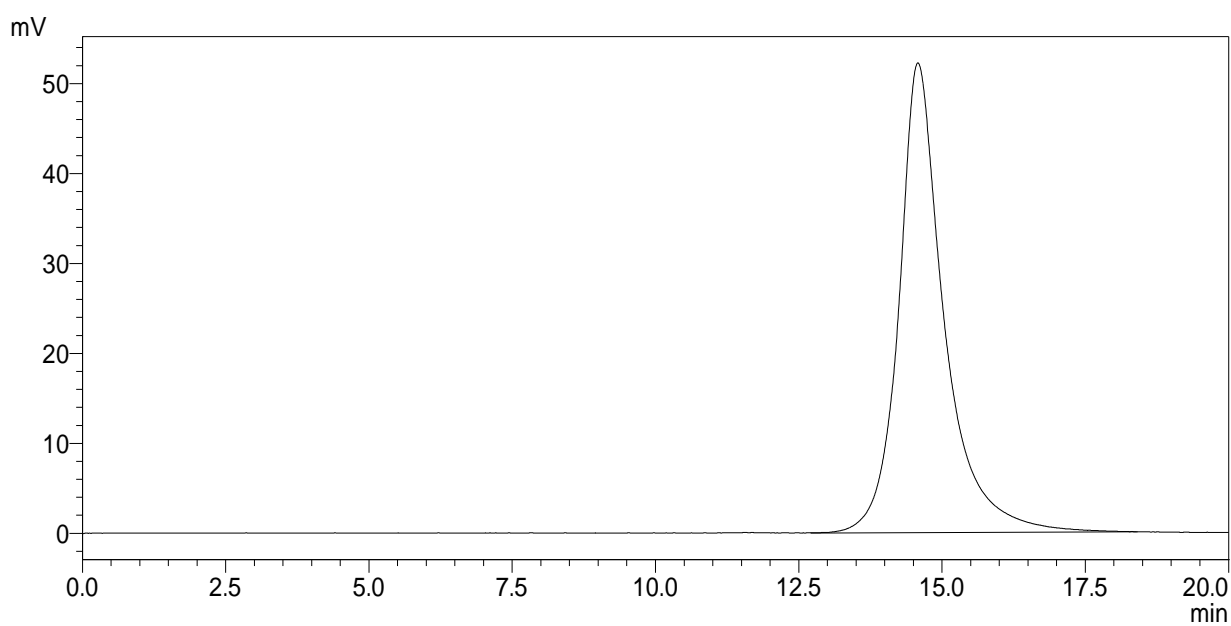
Detector A Ch2 670 nm

| Peak# | Ret. time | Area | Hight | Conc. | Area% |
|-------|-----------|--------|-------|-------|---------|
| 1 | 15.152 | 425471 | 6116 | 0.000 | 100.000 |
| Sum | | 425471 | 6116 | | 100.000 |

Figure S43. HPLC chart of **2c**.

<Sample>

Sample name : **2d**
 Sample ID : takagi
 File name : 20170548.lcd
 Method : SubPc.lcm
 .
 Sample vol : 14
 Date Meas. : 1000 uL
 Date Anal. : 2017/06/08
 20:22:34 : 2017/06/08
 20:42:35

<Chromatogram>**<Peak List>**

Detector A Ch2 670 nm

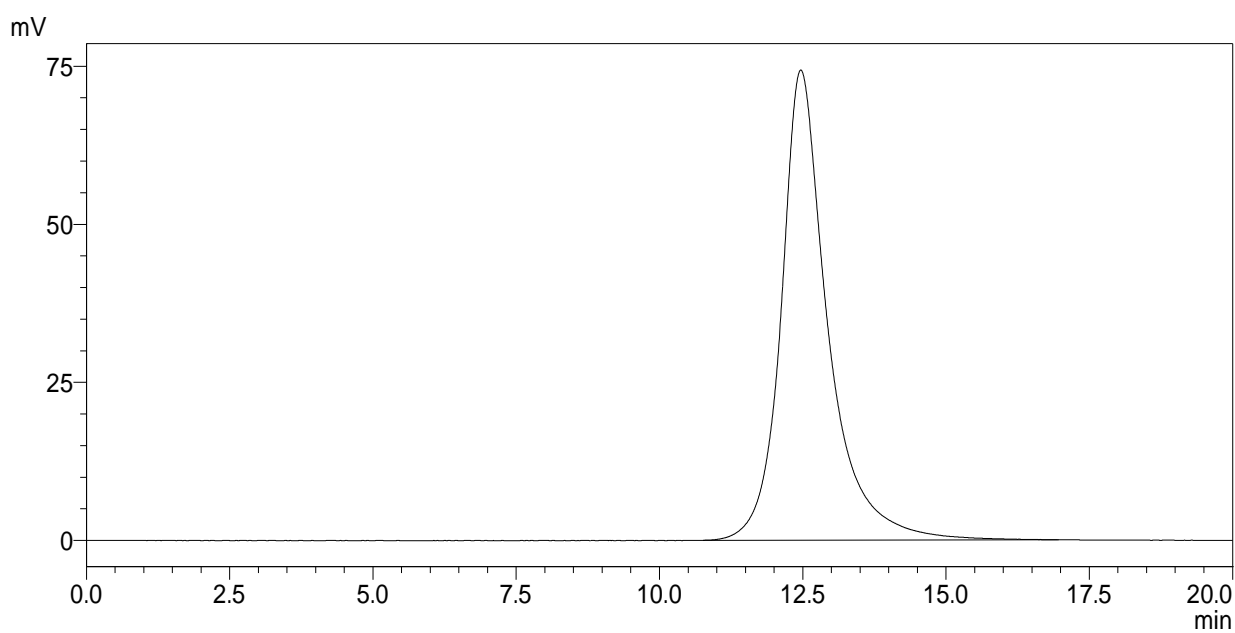
| Peak# | Ret. time | Area | Hight | Conc. | Area% |
|-------|-----------|---------|-------|-------|---------|
| 1 | 14.579 | 2879899 | 52220 | 0.000 | 100.000 |
| Sum | | 2879899 | 52220 | | 100.000 |

Figure S44. HPLC chart of **2d**.

<Sample>

Sample name : **2e**
 Sample ID : takagi
 File name : 20170540.lcd
 Method : SubPc.lcm
 :
 Sample vol : 14
 Date Meas. : 1000 uL
 Date Anal. : 2017/06/08
 12:25:54 : 2017/06/08
 12:45:57

<Chromatogram>



<Peak List>

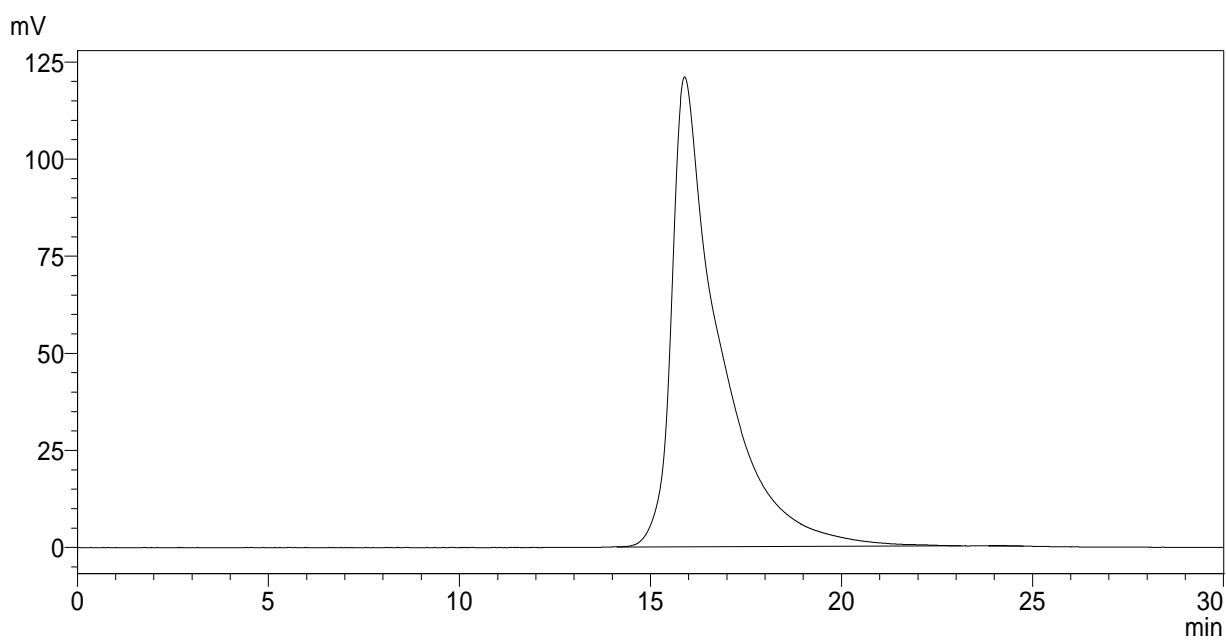
Detector A Ch2 670 nm

| Peak# | Ret. time | Area | Hight | Conc. | Area% |
|-------|-----------|---------|-------|-------|---------|
| 1 | 12.465 | 4231981 | 74397 | 0.000 | 100.000 |
| Sum | | 4231981 | 74397 | | 100.000 |

Figure S45. HPLC chart of **2e**.

<Sample>

Sample name : **2f**
 Sample ID : takagi
 File name : 20170544.lcd
 Method : SubPc.lcm
 .
 Sample vol : 14
 Date Meas. : 1000 uL
 Date Anal. : 2017/06/08 15:28:59 :
 2017/06/08 15:59:01

<Chromatogram>**<Peak List>**

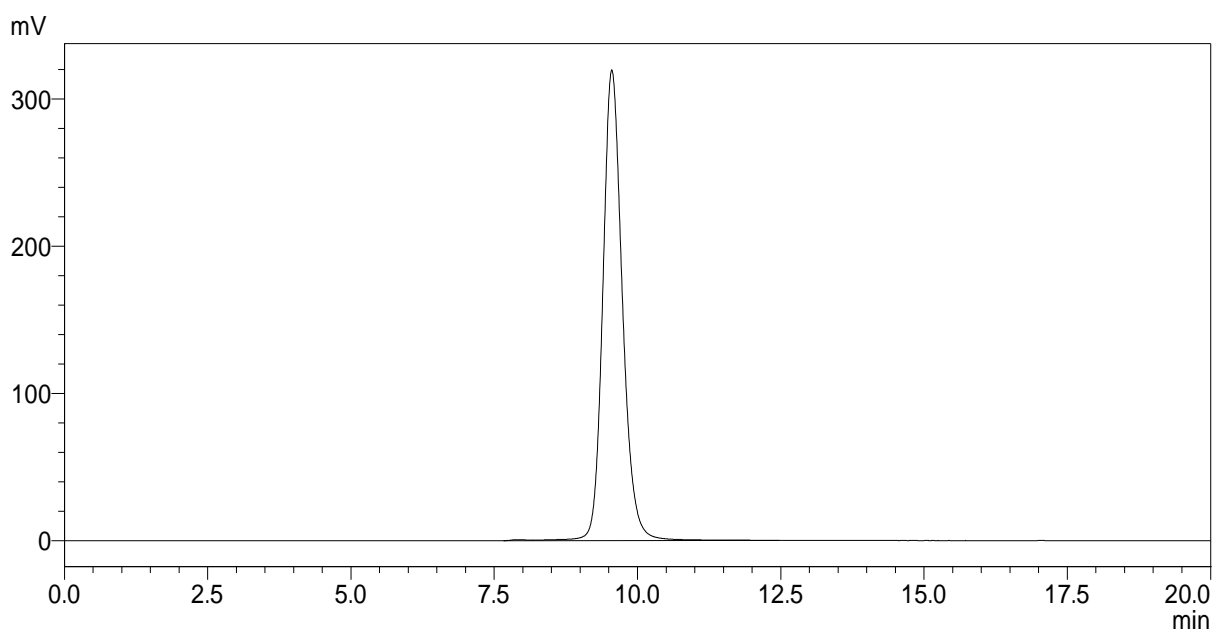
Detector A Ch2 670 nm

| Peak# | Ret. time | Area | Hight | Conc. | Area% |
|-------|-----------|----------|--------|-------|---------|
| 1 | 15.898 | 10609212 | 121020 | 0.000 | 99.954 |
| 2 | 23.967 | 1157 | 114 | 0.000 | 0.011 |
| 3 | 24.073 | 1307 | 129 | 0.000 | 0.012 |
| 4 | 24.250 | 2427 | 117 | 0.000 | 0.023 |
| Sum | | 10614102 | 121380 | | 100.000 |

Figure S46. HPLC chart of **2f**.

<Sample>

Sample name : **2g**
 Sample ID : takagi
 File name : 20170547.lcd
 Method : SubPc.lcm
 :
 Sample vol : 14
 Date Meas. : 1000 uL
 Date Anal. : 2017/06/08 19:26:51
 : 2017/06/08 19:46:53

<Chromatogram>**<Peak List>**

Detector A Ch2 680 nm

| Peak# | Ret. time | Area | Hight | Conc. | Area% |
|-------|-----------|---------|--------|-------|---------|
| 1 | 7.898 | 12560 | 636 | 0.000 | 0.165 |
| 2 | 9.553 | 7616004 | 319771 | 0.000 | 99.835 |
| Sum | | 7628564 | 320407 | | 100.000 |

Figure S47. HPLC chart of **2g**.

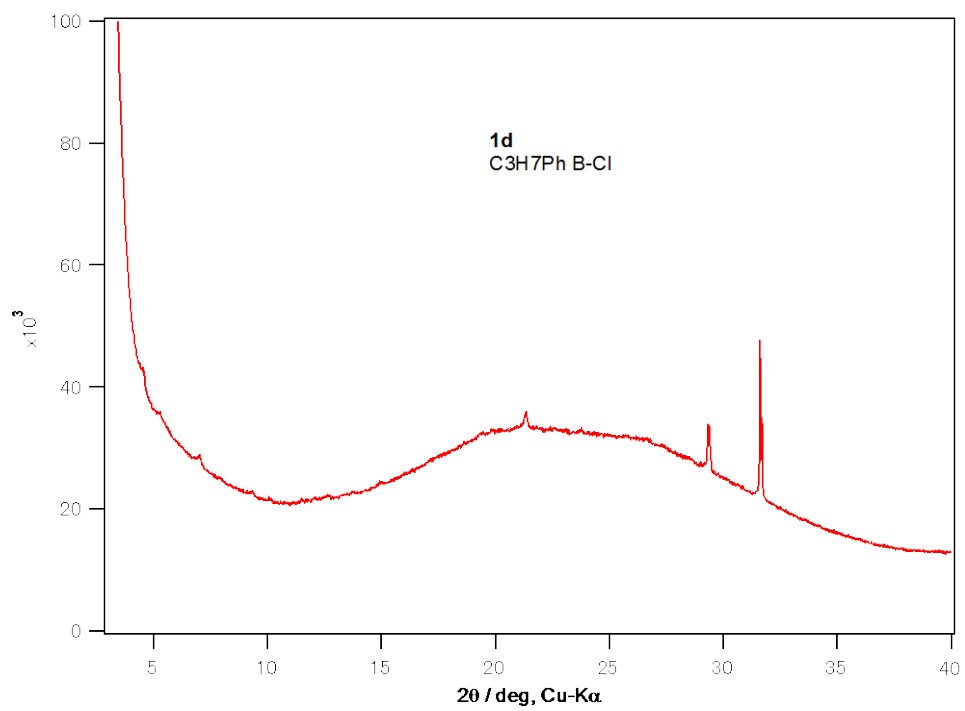


Figure S48 Powder X-ray diffraction patterns of **1d**.

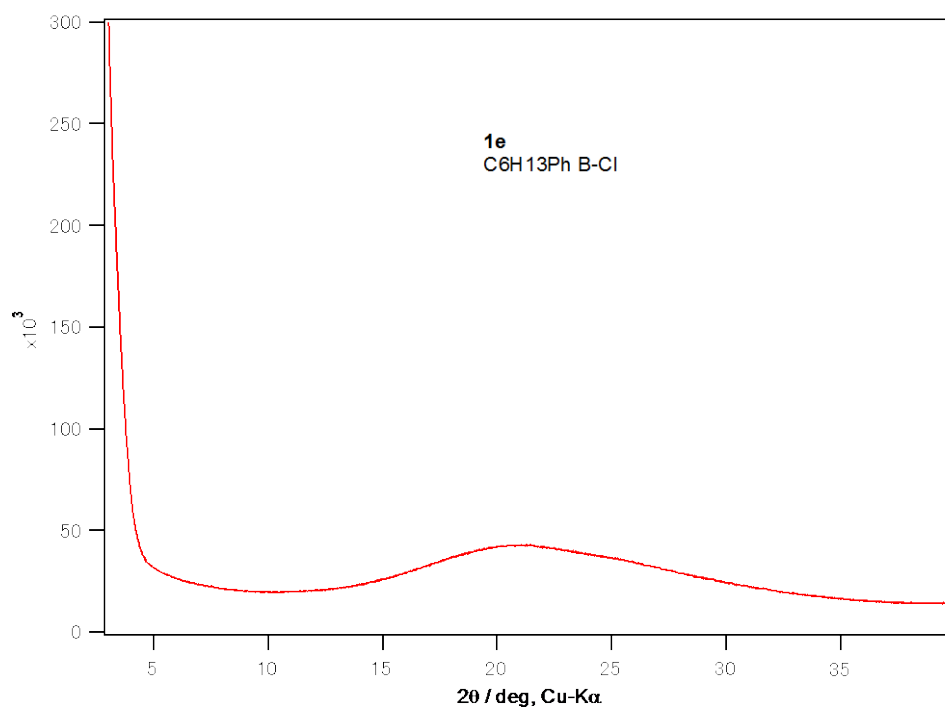


Figure S49 Powder X-ray diffraction patterns of **1e**.

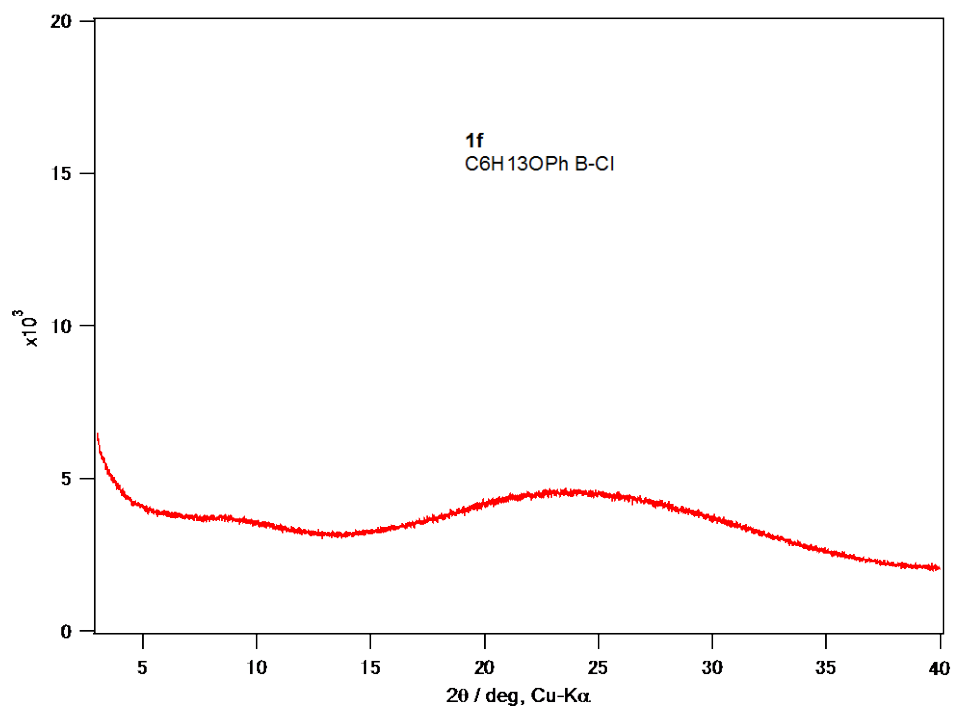


Figure S50 Powder X-ray diffraction patterns of **1f**.

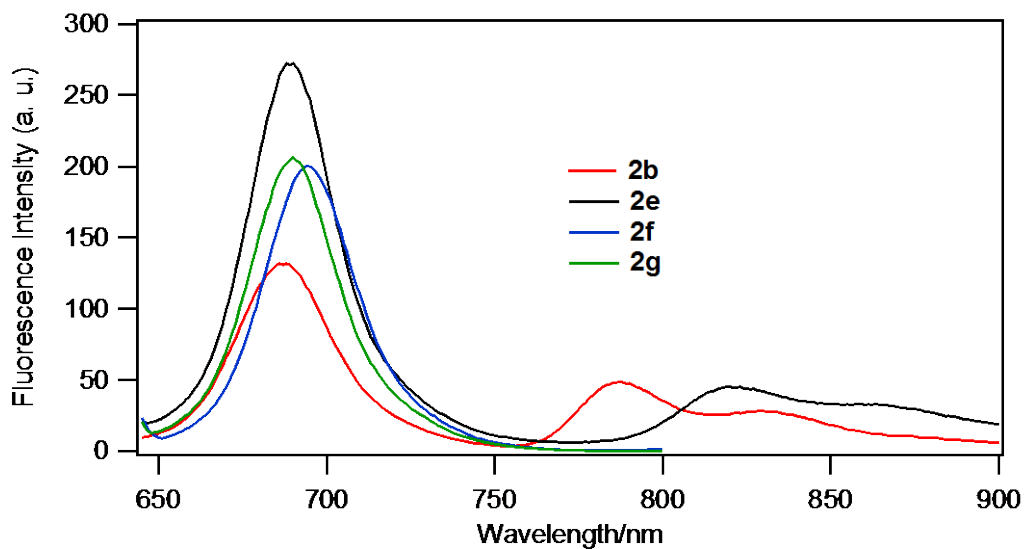


Figure S51. Fluorescence spectra of **2b**, **2e**, **2f** and **2g** in CH₂Cl₂-DMSO (1:1. v/v).

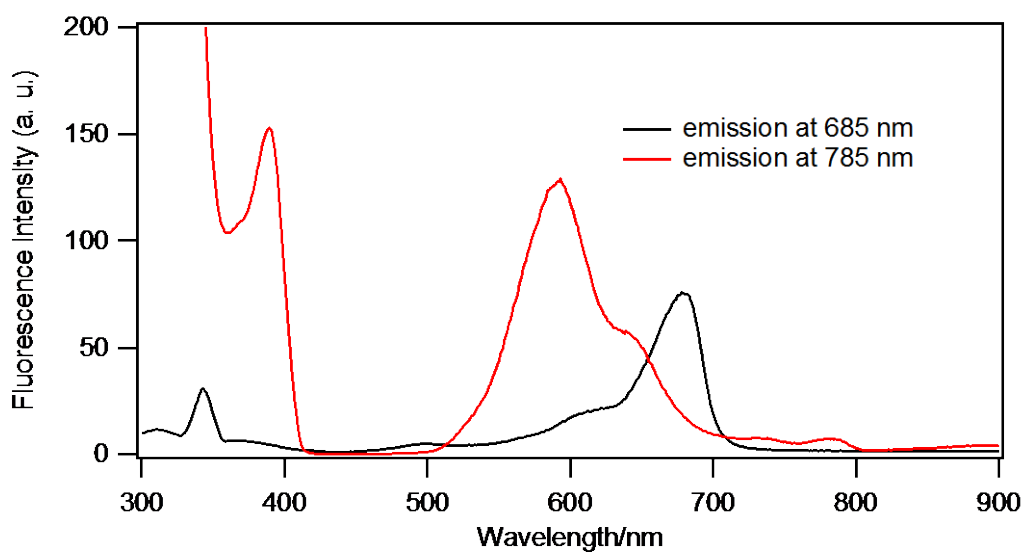


Figure S52. Excitation spectra of **2b** in CH₂Cl₂-DMSO (1:1. v/v).

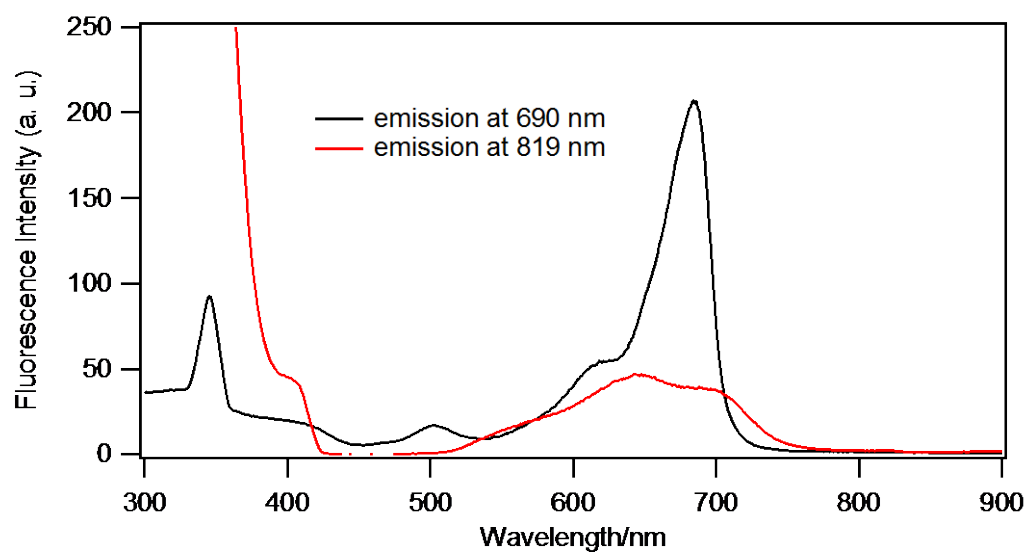


Figure S52. Excitation spectra of **2e** in CH₂Cl₂-DMSO (1:1, v/v).